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Dye Analytical Method Development Report RCRA Facility Investigation at Dye Burial Grounds (Solid Waste Management Unit 2)

Naval Surface Warfare Center Crane Division

Crane, Indiana

Volume I of XI - Summary of Data Package

Southern Division Naval Facilities Engineering Command

Contract Number N62467-94-D-0888

Contract Task Order 0010

May 2001

DEPARTMENT OF THE NAVY
CRANE DIVISION
NAVAL SURFACE WARFARE CENTER
300 HIGHWAY 361
CRANE, INDIANA 47522-5000

IN REPLY REFER TO:

5090
Ser 095/1161
22 MAY 2001

U.S. Environmental Protection Agency, Region V
Waste, Pesticides, & Toxics Division
Waste Management Branch
Illinois, Indiana, and Michigan Section
ATTN: Mr. Peter Ramanauskas (DW-8J)
77 West Jackson Blvd.
Chicago, IL 60604

Dear Mr. Ramanauskas:

Crane Division, Naval Surface Warfare Center (NSWC Crane) submits change pages for the Final Dye Analytical Method Development Report as enclosure (1). This analytical method development is to support the investigative work at Solid Waste Management Unit (SWMU) 02/11, the Dye Burial Grounds. Enclosure (1) contains two copies of the changes to Volume I and one copy of Volumes II through XI. The permit required Certification Statement is provided as enclosure (2).

NAVSURFWARCENDIV Crane point of contact is Mr. Thomas J. Brent, Code 09510, telephone 812-854-6160.

Sincerely,

James M. Hunsicker
Director, Environmental
Protection Department
By direction of the Commander

Encl:
(1) Final Dye Analytical Method Development Report
(2) Certification Statement

Copy to:
ADMINISTRATIVE RECORD (Volume I only)
SOUTHNAVFACENGCOM (Code 1864) (w/o encl)
IDEM (Doug Griffin) (Volume I only)
TTNUS (Ralph Basinski) (w/o encl)

DIRECTIONS FOR REPLACEMENT OF CHANGE PAGES FOR VOLUME I OF XI

Instructions for changes to the Dye Analytical Method Development Report RCRA Facility investigation at Dye Burial Grounds (Solid Waste Management Unit 2)

Volume I of XI

- Replace the existing Cover and Spine dated October 2000 with the enclosed Cover and Spine dated May 2001
- Replace the existing title page dated October 2000 with the enclosed title page dated May 2001.
- Replace Section 10, Attachment X – Reporting Limits and Tables X-1, X-2, X-3, and X-4 with the enclosed Attachment X – Reporting Limits (one page).
- Replace Section 12, SOP CR4052-PD-014 with the enclosed SOP CR4052-PD-2500, Revision 1.

Volume II of XI

- Replace Cover dated October 2000 with Cover dated May 2001.

Volume III of XI

- Replace Cover dated October 2000 with Cover dated May 2001.

Volume IV of XI

- Replace Cover dated October 2000 with Cover dated May 2001.

Volume V of XI

- Replace Cover dated October 2000 with Cover dated May 2001.

Volume VI of XI

- Replace Cover dated October 2000 with Cover dated May 2001.

Volume VII of XI

- Replace Cover dated October 2000 with Cover dated May 2001.

Volume VIII of XI

- Replace Cover dated October 2000 with Cover dated May 2001.

Volume IX of XI

- Replace Cover dated October 2000 with Cover dated May 2001.

Volume X of XI

- Replace Cover dated October 2000 with Cover dated May 2001.

Volume XI of XI

- Replace Cover dated October 2000 with Cover dated May 2001.

5090
Ser 095/1161

22 MAY 2001

The letter Ser 095/1161 was for the submittal of replacement pages for the Draft Dye Burial Grounds Dye Analytical Method Development Report. The Final Dye Burial Grounds Dye Analytical Method Development Report Volume I of XI can be found in its entirety under this item dated 05/22/01.

Dye Analytical Method Development Report RCRA Facility Investigation at Dye Burial Grounds (Solid Waste Management Unit 2)

Naval Surface Warfare Center Crane Division

Crane, Indiana

Volume I of XI - Summary of Data Package

Southern Division Naval Facilities Engineering Command

Contract Number N62467-94-D-0888

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May 2001

DYE ANALYTICAL METHOD DEVELOPMENT
SWMU 2 – DYE BURIAL GROUNDS
NSWC CRANE, INDIANA

The overall objective of this analytical method development effort was to develop methodologies capable of extracting and analyzing a variety of dyes from soil, ground water, surface water, and sediment samples. The methodologies had to produce data of sufficient quality to support a Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) Risk Assessment and Corrective Measures Study. Currently, there are no U.S. EPA-approved analytical methods that address the variety of dyes that could potentially be present at SWMU 2. Because the NSWC Crane Laboratory was previously involved in the dye analysis of the aqueous material for SWMU 2, they continued development of an analytical method for dyes that could be potentially present at the SWMU. The analytical method developed by the laboratory followed the SW-846 Guidance for Method Development and Method Validation for the RCRA Program. This guidance document is provided in Attachment XIV. Also, as shown in the meeting minutes in Attachment XIV, this guidance was approved by U.S. EPA Region 5.

The specific objectives of this project were to:

1. Develop extraction methods capable of recovering dyes from solid and aqueous matrices, and

2. Develop an analytical method capable of separating, identifying, confirming, and quantifying dyes that could potentially be present at SWMU 2 as a result of site operations, and

3. Develop extraction and analytical methods that would meet the requirements for approval by U.S. EPA Region 5.

As demonstrated by the attached documentation and supporting raw data, these objectives have been met.

SITE HISTORY

In the operational history of the Dye Burial Grounds (DBG) (SWMU 2) it is estimated that 50,000 tons of dyes and dye-contaminated materials were deposited in open trenches between 1952 and 1964. Materials reportedly included magnesium, boxes, and rags contaminated with dyes, and about 60 open drums of dyes. There is no record of the specific dyes that were buried in these trenches, although dye characteristics are known to range from water soluble acidic and basic compounds to relatively water insoluble non-polar compounds. The trenches reportedly were back filled to the ground surface with soil in 1972 but were not permanently capped. An interim measures cap was designed based on this

information and construction began in 1996. An additional field investigation was conducted in 1996 by Naval Surface Warfare Center – Environmental Protection Department (NSWC-EPD) and U.S. EPA. Several other areas and trenches of dye contamination were found during this field investigation. These areas were deeper and had a greater width than the original trenches. The interim measures cap was expanded to cover these new areas, thereby delaying the construction of the cap. Those areas were excavated until no dyes were visually observed in the soil. After completion of the excavations, the installation of the multi-layered cap system was completed. Because some of the work during the cap installation was conducted in the winter months, precipitation accumulated in the trench areas. This material was placed in frac tanks. The aqueous waste was qualitatively analyzed for dyes. These analyses were performed by the NSWC Crane Lab with oversight from U.S. EPA Region 5. This is the only dye analysis performed at SWMU 2 to date.

One objective of the field programs is to determine whether dye material was excavated based on visual observation. Additionally, the potential for migration of dyes from soil to ground water is to be evaluated by sampling ground water. To meet these objectives a quantitative analytical method for dyes is necessary.

METHOD DEVELOPMENT HISTORY

Originally, two analytical methodologies were proposed. Thin-layer chromatography (TLC) was proposed for performing screening (qualitative) analyses of soil and sediment samples and high performance liquid chromatography (HPLC) was proposed for identification and quantitation of dyes in aqueous and solid matrices. Both TLC and HPLC were used in the original frac aqueous dye analyses performed by the Crane Lab. Under the original proposals the aqueous method would be used as the basis for developing the new analytical methods.

Original Analyses

In the initial analyses aqueous dye samples were evaporated to dryness using a nitrogen blow down at room temperature. Fourier Transform Infrared Spectroscopy (FT/IR) analysis of the residues after evaporation indicated that sulfur-containing compounds were the major components present. The residues were extracted with ethanol to remove colored components from the sulfurous materials. The extracted solutions were evaporated with nitrogen to dryness and extracted with methanol for chromatographic evaluation. The methanol extract was then subjected to TLC and HPLC analyses. Disodium Fluorescein and an Acid Blue mixture were identified as contaminants present in the frac tank water during this process.

TLC Screening Procedure

Analysis of dyes dissolved in methanol by TLC proved to be very successful. Good separation was achieved for the majority of dyes using two mobile phases for water-soluble and organic-soluble dyes. However, the use of TLC as a screening method for soil samples was abandoned due to difficulties with the extraction procedure. The key difficulty was exchanging the tetrabutylammonium (TBA) hydroxide extraction solvent into the methanol analytical solvent. The decision to abandon TLC as a screening method was based on the time required per sample extraction and the unacceptably low dye recoveries. The TLC extraction method was slower than the HPLC extraction method, and the recoveries for the TLC were substantially lower than those obtained for the HPLC. Therefore, the TLC method could not provide quick turnaround or a reasonable indication of the presence/absence of dye compounds in the soils.

Extraction of Solid Samples for HPLC Analysis

The laboratory used a variety of solvents with both sonication and soxhlet techniques for this evaluation. Based on recommendations from Dr. Baughman (U.S.EPA, Athens Laboratory, retired) a sonication technique using TBA hydroxide (in methanol), organic-free water, and methanol proved to be the most effective extraction approach. After extraction, the samples are filtered through a 0.45 micron nylon filter and analyzed.

Extraction of Aqueous Samples for HPLC Analysis

The laboratory developed two aqueous analysis methods — high-level direct injection HPLC, and low-level extraction followed by HPLC analysis. The high-level method was eliminated for further consideration when the method validation proved that most dye compounds could not be recovered by this method. The poor recoveries were most likely due to the low solubilities of the dyes in water.

Based on recommendations from Dr. Baughman (U.S.EPA, Athens Laboratory, retired) the following low-level procedure was developed. One liter of sample is modified by adding 5-mL of TBA hydroxide (in methanol). The water is then poured through a C18 solid phase extraction (SPE) disk. The dyes are then recovered from the SPE disk by extraction with methanol.

HPLC Analysis

Two HPLC methods were developed using a photodiode array detector (PDA). The first method, short gradient method, uses an approximately 35 minute run time. The second method is a long gradient method, which runs approximately 70 minutes.

For soils, analyte recoveries from the samples are acceptable with the short gradient method. Therefore, *soils and sediments only must be analyzed by that method unless chromatographic coelution occurs.* If coelution occurs, the long gradient method will be used. For aqueous samples, the short gradient method will be used to identify the presence of dyes. However, analyte recoveries from aqueous samples are better using the long gradient method, so aqueous samples will be quantitated using that method.

PRESENTATION OF DATA

The information gathered from the method development is presented in eleven three-ring binders. Volume I (this volume) contains the summary tables and explanatory text. Volumes II through XI contain the supportive raw data. To facilitate review of the method development information, an activities requirement checklist was developed. This checklist is presented as Table 1. There are four columns on *this checklist. The first column identifies the actual activity that corresponds with requirements in the* RCRA method development guidance. The second column is titled "Attachment." This identifies the number of the attachment where summaries of supporting information may be found for the activity. The third column identifies the volume (i.e., the binder) in which the information is located. It should be noted that all the attachments are located in Volume I. The remaining ten volumes provide details (e.g., analytical raw data, spectra, logbook notes, etc.) to support the summary information. The volume in which the applicable detail-specific information is present is listed in column 3. The fourth column presents the actual action items associated with the activity. This entails a brief summary of the work performed for the activity.

TABLE 1

ACTIVITY REQUIRMENTS CHECKLIST FOR SWMU 2 – DYE BURIAL GROUND

Activity	Attachment	Volume	Action Items
Literature Search	I	I	Conduct literature research on potential analytical methods already developed (e.g., SW-846 Method 8321A). Read articles to assist in identifying viable extraction procedures, analytical system requirements, interferences, and corrective actions.
Identify dyes	II	I	Accurately identify dyes. Group all dyes that could be present at the site according to similar chemical structures.
Obtain Standards	III	I	Request dye standards and chromatograms from manufactures. Talk to chemists regarding their experience with dye analyses. Contact companies such as: Aldrich, Aakash Chemicals and Dyestuffs, Crompton and Knowles, Sandoz Chemicals
Calibration Study Retention Times	IV	II, III, and IV	- Determine high and low end of calibration range - Determine linear portion of calibration curve - Establish reporting limit - Establish spiking level - Identify and resolve co-elution problems - Identify and resolve chemical interference problems [NOTE: Initial calibration linearity (%RSD and correlation coefficient) are to be determined using procedures outlined in SW-846 Method 8000.]
Extraction Procedures	V	I	Analyze seven replicate per matrix at two concentrations for each extraction method to be tested.
Optimize Method	VI	I	The following QC items are included in these runs: - Surrogates (benzanthrone and anthracene) - Method Blanks
Validate Analytical Method	VII	V, VI, VII, VIII, IX, X	Initial determination of method accuracy and bias will be done in a spiked clean site-specific matrix.
Interference Effects	VIII	I	Method interferences will be determined in a spiked clean matrix. Both positive and negative interferences will be documented and addressed.
Analyze Site-specific Soil and Groundwater Samples	IX	I	Analyze site–specifc samples to evaluate presence of dyes in the environment.
Method Detection Limits and Reliable Quantification Limits	X	I	Matrix effects may affect the achievable quantitation limits on site-specific samples. Therefore, method detection limits (MDLs) and reliable quantitation limits (RQLs) based on the site-specific samples will be determined. Both MDLs and RQLs will be tabulated and presented as part of the method.
False Positive and Negatives	XI	I	A candidate screening method should produce no more than 10% false positives and should not produce false negatives.
Prepare SOP	XII	I	Prepare SOP.
Sample Calculations	XIII	I	Present example solid and aqueous sample calculations from the beginning to end of the analytical process.
Meeting Notes	XIV	I	Guidance Documents and Meeting Notes
Support Documentation	XV	II thru XI	Raw Data – Calibration, Blanks, and Samples Chromatograms and Spectra

ATTACHMENT I

LITERATURE SEARCH

ATTACHMENT I - LITERATURE SEARCH

A literature search was conducted by both the Crane Laboratory and Tetra Tech NUS, Inc. The Crane Laboratory focused on researching extraction and analytical methodologies through reference books, scientific journals, and professional contacts. TtNUS conducted an Internet search focusing on toxicity information for dye compounds. TtNUS and the Crane Laboratory exchanged information regarding likely sources of information, where appropriate.

The references presented here are those which were used as the basis for developing the final extraction and analytical procedure. The list does not include sources which were consulted but not used for the final method development.

Professional contacts with EPA, Ecological and Toxicological Association of Dyes and Organic Pigment Manufactures (ETAD), and various universities proved to be the richest source of information regarding dye analyses, extraction methods, and sources of dye standards.

1. Randerath, Kurt.Thin-Layer Chromatography.Academic Press, New York, 1964.

2. K. Venkataraman (ed.): The Chemistry of Synthetic Dyes, Vol. III. Academic Press, New York, 1970.

3. Arnold J. Gordon and Richard A. Ford. The Chemist's Companion. John Wiley & Sons, New York, 1972.

4. Gunter Zweig and Joseph Sherman, (eds.): Handbook of Chromatography, Vol.I. CRC Press, Cleveland, 1972.

5. K. Venkataraman, (ed.): The Analytical Chemistry of Synthetic Dyes. John Wiley & Sons, New York, 1977.

6. Dr. Robert E. Keyes Chemist, Abbott Laboratories, in communication.
   -Organic synthesis and screening with TLC

7. Heinrich Zollinger. Color Chemistry, 2nd Ed. Weinheim, New York, 1991.

8. Arthur W. Garrison; George L. Baughman; Eric J. Weber; Rebecca L. Adams; Mary Sue Brewer. *Fate of Colored Smoke Dyes.* 1992.

9. Wegener, J.W., J.C. Klamer, H. Govers, and U.A. Th. Brinkman (1987) Determination of Organic Colorants in Cosmetic Products by High Performance Liquid Chromatography.

10. Chromatographia, 24: 865-875.Garrison, A.W., G.L. Baughman, E.J. Weber, R.L. Adams, and M.S. Brewer (1992) Fate of Colored Smoke Dyes. Army Project Order 88PP8863.

11. Alcantara-Licudine, J.P., M.K. Kawate, and Q.X. Li (1997) Method for the Analysis of Phloxine B, Uranine, and Related Xanthene Dyes in Soil Using Supercritical Fluid Extraction and High Performance Liquid Chromatography. Journal of Agricultural Food Chemistry, 45: 766-773.

12. Gagliardi, L., G. Cavazzutti, A. Amato, A. Basili, and D. Tonelli (1987) Identification of Cosmetic Dyes by Ion Pair Reversed Phase High Performance Liquid C hromatography. Journal of Chromatography, 394: 345-352.

13. Eakes, W., J. Heath, M. Cavit, K. Rimm, W. Powers, C. Connelly, J. Anderson, M. Johnson (1983) Initial Assessment Study of Naval Weapons Support Center Crane, Indiana. NEESA 13-003.

14. U.S. Environmental Protection Agency, Method SW-846-8330.

15. U.S. Environmental Protection Agency, Method SW-846-8000.

16. U.S. Environmental Protection Agency, Method SW-846-3500.

17. U.S. Environmental Protection Agency, Method SW-846-3550.

18. Dr. George Baughman, University of Georgia, in communication.
    -Published multiple articles on dyes in the environment (U.S. EPA Research Laboratory Athens, retired)

SUMMARY OF AVAILABLE TOXICITY INFORMATION
SWMU 2- DYE BURIAL GROUND
NSWC CRANE, INDIANA

	Sources of Information												
Dye Name	NTP C&S	NTP-PUB	OSHA	CAL-EPA	EPA-FACT	SCORECARD	ECDIN	JAPAN	IRAC	IRAC MONO.	RETECS	HSDB	CCRIS
Water Soluble Dyes													
Acid Blue 1	X	X				X	X		G3	X	BP6830000	X	X
Acid Blue 9								X	G3		BQ4725000		
Acid Blue 45													
Acid Red 64													
Acid Yellow 3							X				GC5796000		
Acid Yellow 23						X	X	X			UQ6400000		X
Acid Yellow 73	X	X					X				LM5425000		X
Acid Orange 10	X	X					X		G3	X	QJ6500000	X	X
Basic Violet 10			X	P-65		X	X	X	G3	X	BP3675000	X	X
Basic Yellow 2	X	X			X	X	X	X	G2B		BY3675000	X	X
Organic Soluble Dyes													
1-Aminoanthraquinone							X				CB5075000		
2-Aminoanthraquinone	X	X		P-65/CRICAR	X	X	X	X	G3	X	CB5120000	X	X
Disperse Blue 14							X						
Disperse Red 9							X			X			
Disperse Violet 1							X				CB6300000		X
Pigment Red 1							X				QL4510000		
Smoke Dye											CB6600000		
Solvent Green 3							X				CB5775000		
Solvent Orange 3													
Solvent Orange 7						X	X		G3	X	QL5850000	X	X
Solvent Red 1							X					X	X
Solvent Red 24							X	X	G3	X	QL5775000		X
Solvent Yellow 2	X	X	X	P-65/CRICAR	X	X	X		G2B	X	BX7350000	X	X
Solvent Yellow 3	X	X		P-65/CRICAR		X	X		G2B	X	XU8800000	X	X
Solvent Yellow 14	X	X	X	P-65		X	X		G3	X	QL4900000	X	X
Solvent Yellow 33	X	X					X				GE5925000		X

*NTP C&S & PUB	http://ntp-server.niehs.nih.gov/
*OSHA	http://www.osha.gov/
CAL-EPA	CRIC http://www.oehha.org/docs/covrltrb.htm P-65 http://www.oehha.org/prop65/
EPA FACT	http://www.mail.odsnet.com/TRIFacts/ - TOX!B45
SCORECARD	http://scorecard.org/ - TOX!B47
*ECDIN	http://www.ecdin.etomep.net/
JAPAN	http://www.citi.or.jp/
IRAC	http://193.51.164.11/ - TOX!B53
*TTN-WEB	http://www.epa.gov/ttn/
*HSDB & CCRIS	http://toxnet.nlm.nih.gov/ - TOX!B58
*Chemfinder	http://www.chemfinder.com
*NIST Chembook	http://webbook.nist.gov/chemistry/
*SW-846 On-line	http://www.epa.gov/epaoswer/hazwaste/test/main.htm
*SW-846 Update 4A	http://www.epa.gov/epaoswer/hazwaste/test/up4a.htm
Internet Grateful Med	http://130.14.32.45/cgi-bin/

* Sites which contain physical/chemical/analytical information

Other Professional Contacts:

Pfaltz & Bauer
Aldrich
Aakash Chemicals and Dyestuffs
Sandoz Chemicals

Source: DyeStar

- Dr. Fred Perry Tel: 704-561-3013

Source: University of Leeds

- Dr. Kelvin Tapley, Dept. of Colour Chemistry, University of Leeds, Leeds LS2 9JT, England; Tel: 0113-2336732; Fax: 0113-2332947; e-mail K.Tapley@leeds.ac.uk

Source: Crompton and Knowles

- Technical Help 704-372-5890
- Barry Debinsky 610-582-6634

Source: ETAD

- Lynne Jones; Tel: 202-721-4157; e-mail Lynne.Jones@socma.com
- Have not yet received bibliography

Source: Hewlett Packard

- Cindy Richman e-mail cindy_richman@hp.com

Source: Microtrace, Inc.

- Bill Kerns, 3100 84th Lane N.E., Suite A; Minneapolis, MN 55449-7216; tel: 612-784-9725; Fax: 612-784-3787

Source: 3M

Richard Livesay; Tel: 651-429-8235; Fax: 651-407-0102

Poehlein Sara K CNIN

From: Keyes,Robert [robert.keyes@abbott.com]
nt: Thursday, August 05, 1999 3:02 PM
To: poehlein_sara@crane.navy.mil
Subject: Re: tlc

Sara,

I am glad to hear you like your job. I might be able to help. I will at least offer some suggestions. First the TLC plates we used are coated with Silica Gel 60. The thickness ised is 250 micrometers. They have a fluorescent indicator in the SiO2. With dyes you probably do not need any kind of staining agent since they usually have a strong chromophore so a UV lamp should do. If you do need to stain the plate a general stain which is commonly used is phosphomolybdic acid. It is prepared by dissolving 10% by wt. of phosphomolybdic acid (Aldrich) in 95%EtOH. If you are doing column chromatography use 230 - 400 mesh SiO2. This does not have a fluorescent indicator in it, but that does not matter because the fractions collected will be checked in the manner descriged for TLC above. A good place to start looking for an eluting solvent is with 50% hexanes / 50% ethyl acetate. If you are analyzing dyes you mayneed something more polar such as MeOH / CH2Cl2 mixtures or you might have to go to a reverse phase system, but that is a whole other story. If you can find a book by K.G. Krebs, D. Heusser, and H. Wimmer you will be able to find all the staining agents for all types of molecules. If you have any other questions give me a call. I may be able to better explain it over the phone. Tell Ben I said hello. I will talk to you later.

Rob

Acetone - may get binder
ethyl acetate

10% by weight
Phosphomolybdic
in 95% ethanol
heat gun or hot plate

beaker

blue spots

2,4 DNP

Feigl

200mg DNP
100ml ethanol
1ml Conc. HCl

Aldehyde reactions

general stain

~~3000:200~~
3225ml 95% ethanol
100g para ~~iso~~ anisaldehyde
120ml Conc H_2SO_4
36mL acetic acid
Store in fridge

toluene

Amines

3g Vanillin
100ml absolute ethanol
0.5ml Conc. H_2SO_4

cool

store in fridge

Amines Ninhydrin

Poehlein Sara K CNIN

From: Keyes,Robert [robert.keyes@abbott.com]
Sent: Tuesday, August 10, 1999 12:44 PM
To: poehlein_sara@crane.navy.mil
Subject: Re: TLC

Dr. Robert Keyes
Senior Research Scientist
Abbott Laboratories

Sara,

Why don't you give me a call at work. I think I could be of more help that way. The number is 847-935-2675.

Rob

Rob
Keyes (847) 935-2675
Senior Research Scientist
Synthetic Organic Chemist

~~Moderating~~ Revs.
Monitoring Revs

365 8 KI
35mg bismuth subnitrate
40ml acetic Acid
100 ml H_2O
Alkaloids, Amines
Dilute H_2SO_4 black

10-

Poehlein Sara K CNIN

Spotting concentration limits

From: Keyes,Robert [robert.keyes@abbott.com]
Sent: Tuesday, August 10, 1999 12:44 PM
To: poehlein_sara@crane.navy.mil
Subject: Re: TLC

Sara,

Why don't you give me a call at work. I think I could be of more help that way. The number is 847-935-2675.

Rob

Acid dyes => water soluble
alumina

Fisher Reverse phase TLC plates water / acetonitrile

90% dichloromethane – 70% acetone
10% Methanol 30% Hexanes
1% Ammonium Hydroxide 1-2%

Polar compound – CH_2Cl_2/MeOH (blow 10%) – add acetic acid 1-2% to tighten drops

Nonpolar – CH_2Cl_2
ethyl acetate

basic group → triethylamine 1-2% to prevent streaks

separate w/ funnel to cut the top off

Glass is better => turnover and see better
long λ

iodine crystal of iodine mix with sand in jar

Hexanes with CH_2Cl_2
Ethyl acetate / Hexane 25% 75%

Hexanes Comparison

SG least

most polar

Heptane
Toluene
benzene
Chloroform
ethyl acetate
ethanol
water
acetone

alumina

47 → 100
47

[redacted]

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LE_____ Book No._____

m Page No.__

Result of TLC: Samples moved w/ solvent ∴ no separation
need to try different solvent

Spoke w/ Dr. Reyes @ Abbott Lab
He suggested using hexanes w/ CH_2Cl_2
or 25% ethyl acetate / 75% Hexane

For Polar dyes: 90% dichloromethane
10% MeOH
1% Ammonium Hydroxide (tighten spots)

Prepare and shake in separatory funnel to take off
H_2O layer (top) from Ammonium hydroxide.

or: 70% Acetone
30% Hexane

or CH_2Cl_2
MeOH (<10%)

if streaks acid use 1-2% acetic acid (1-2 drops)

basic use 1-2% triethylamine (1-2 drops)

[redacted]

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Book No._____ TITLE 8-12-99

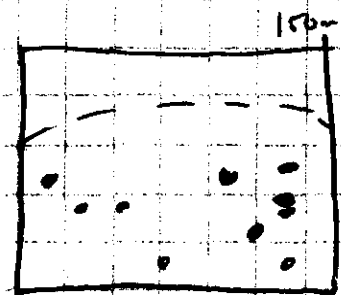
Page No.__

Try Hexane → CH_2Cl_2

100% CH_2Cl_2 → No Separation

75%/25% (CH_2Cl_2/Hex) Not Good
(150 mL/50 mL)

50/50 CH_2Cl_2/Hex Good
150 mL/150 mL



25/75 CH_2Cl_2/Hex
Solvents did not separate as well

50% Ethylacetate / 50% CH_2Cl_2

8/13/99
ISU => Dr. Kjonaas
Dr. Ahn

NMR interpretation

Page No.___ Try

60/40 EA/Hex 120/80

70/30 EA/Hex 140/60 105/45

55/45 EA/HEX 110/90

65/35 EA/Hex 130/70

Try acetone / hexane – does not work due to immiscibility

Stains

1) 10% by weight phosphomolybdic acid in 95% etoh
=> dip plate, then heat with gun or hot plate
=> see blue spots

2). 2,4-DNP
200mg DNP
100 ml Etoh
1 mL conc. HCl
} Aldehydes / Ketones

3). 225 mL 95% Etoh
100g para anisaldehyde
120 mL Conc. H_2SO_4
36 mL glacial acetic acid
} general stain
Store in freezer

4). 3g vanillin
100 ml absolute Etoh (cold)
0.5 mL Conc. H_2SO_4
} amines
Store in freezer

5). Ninhydrin
0.1% solution Ninhydrin in citrate buffer →

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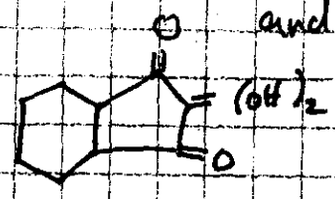
Page No.__

citrate buffer: pH = 5
dissolve 210.1 g of citric acid
in 200 mL of N Sodium hydroxide
and dilute w/ H_2O to 1 liter



<u>Quantify</u>
1) Scrape plate w/ spot
2) dissolve in ethyl acetate
3) filter
4) HPLC

5/19/99
Tried w/ Rodo alone double elution
cellulose or Silica Gel

To Page No.____

Understood by me, | Date | Invented by | Date

Page No.__

The following solvents were tried on Glass TLC

xylene (100%)
cyclohexane/ethyl acetate (20/80), (40/60)
hexane/ethyl acetate (40/60)

Xylene was the best solvent in separating mixture of 5 dyes:

S07
SR24
SY3
SG3
Azoic Dia20

Could see all 5 as separate.

Tip: Dip plate in solvent and allow to separate for a few minutes. then stop and take plate out to air dry completely, then put back in tank to finish separate.

Seem to get better separation

I will try a mixture of solvents w/ Xylene to optimize separation.

Discussion w/ Rob: To separate out further try adding more polar solvent such as THF, Ethyl acetate, Acetone, or MeOH

Try a pure p-xylene, toluene

sed & Understood by me, | Date | Invented by | Date

Page No.__ 8-24-99

Forrest Arnold the auto injector

run 5 samples /mix 50µl on TLC plate
in 2% THF 98% p-Xylene > 50µl

STAND		MIX		
S07	6.0/10.7 = 0.56	1	7.6/10.5 = 0.72	SG3
SR24	5.0/10.6 = 0.47	2	6.4/10.5 = 0.61	S07
SY3	4.0/10.7 = 0.37	3	5.8/10.5 = 0.55	
SG3	7.2/10.6 = 0.68	4	4.6/10.5 = 0.44	SR
AZO	4.1/10.6 = 0.39	5	4.4/10.5 = 0.42	AZO

Discussion w/ Rob: What prevents analyte from completely going to top?
How to preserve TLC plates?

interaction between functional group of analyte and silica (polar, non-polar) over infinite time analyte will rise to top.

take pictures, or scan them in

ed & Understood by me, | Date | Invented by | Date

Sonicate dye standards for ~ 2 hrs.

5% Hexane ; 95% Toluene

line walls with std'd. filter paper

8 std. + Benzanthrone

Prepare Phosphomolybdic Acid Stain

190 mL Etoh
10 mL DI H_2O

Stain SR24 → Blue spot

Observe 5 = AZO, SO 7/SR24, SY33, AZO, Smoko, SY3
3 UV lamp = Benzanthrone, AZO, Smoko
No SG3

Sprayed PA stain on TLC plate
⇒ no blue stain

Called Rob Keyes suggested I need to saturate the TLC plate w/ PA stain

Restained another TLC plate ⇒ SR24 blue

Poehlein Sara K CNIN

From: Calyx11219@aol.com
Sent: Friday, August 13, 1999 10:19 AM
To: poehlein_sara@crane.navy.mil
Subject: Re: help

Dr. Fred Regnier
Purdue University
Chromatography Specialist

Hi Sara! Actually I don't know much about separatiing or analyzing dyes. The thing you have going for you in most cases is that they have a lot of aromaticity and will stick like crazy to reversed phase columns. The other thing is that they will show up well in a diode array detector. You can even get absorbance scans directly from the LC that willl be useful. Finally, they should fly well in LC/MS. Taken together, diode array LC and LC/MS should take you a long way to identifying this substances.

Incidently, because they are water soluble now they have either oxidized to a charged species or had a charge in the first place. This will alllow you to purify them readily by playing with the pH during RPC. I would even guess that they are negatively charged. There are so many silicates in the soil that positively charged species would be strongly adsorbed to the soil and not be very soluble.

Good luck.

FRED

Poehlein Sara K CNIN

From: Jonathan A. Karty [jkarty@indiana.edu]
Sent: Tuesday, August 10, 1999 4:33 PM
To: Poehlein Sara K CNIN
Subject: RE: NMR

Jonathan Karty
I.U. Graduate student
Ran ESI experiments on unknown dyes

could you send jackie home with the FTIR's. I would like to look at them and see if I can come up with anything. (just to reuse some old skills)

jk

--
Jon Karty
Grad Student, Dept. of Chemistry Indiana U.

For with much wisdom comes much sorrow;
the more knowledge, the more grief.
Ecclesiastes 1:18.

jkarty@juliet.ucs.indiana.edu http://ezinfo.ucs.indiana.edu/~jkarty

> -----Original Message-----
> From: Poehlein Sara K CNIN [mailto:poehlein_sara@crane.navy.mil]
> Sent: Tuesday, August 10, 1999 3:08 PM
> To: 'Jonathan A. Karty'
> Subject: RE: NMR
>
>
> Jon,
>
> As of now I don't think we need to do ESI but I may if I can't get enough
> information from the IR and NMR spectra. If you see Steve Kahn
> please tell
> him hello for me.
>
> Sara
>
>
> > -----Original Message-----
> > From: Jonathan A. Karty [SMTP:jkarty@indiana.edu]
> > Sent: Tuesday, August 10, 1999 2:30 PM
> > To: Poehlein Sara K CNIN
> > Subject:RE: NMR
> >
> > I have them here in a manlia folder, will give them to Jackie tonight.
> > Did
> > you want to discuss possibilities of running ESI mass spectra on those
> > samples as well?
> >
> > Nice to hear from you again.
> >
> > jk
> >
> > --
> > Jon Karty
> > Grad Student, Dept. of Chemistry Indiana U.
> >
> > For with much wisdom comes much sorrow;
> > the more knowledge, the more grief.
> > Ecclesiastes 1:18.
> >
> > jkarty@juliet.ucs.indiana.edu
> > http://ezinfo.ucs.indiana.edu/~jkarty
> >
> >

> > > -----Original Message-----
> > > From: Poehlein Sara K CNIN [mailto:poehlein_sara@crane.navy.mil]
> > > Sent: Tuesday, August 10, 1999 2:25 PM
> > > To: 'jkarty@indiana.edu'
> > > Subject: NMR
> >
> > >
> > > Jon,
> > >
> > > Jackie wanted me to remind you to print off those NMR spectra of her
> > dyes.
> > > I will need them by thursday.
> > >
> > > Thanks,
> > > Sara
> > >
>

Dear Sara and Forrest,

This letter should explain the instrument conditions used to generate the NMR spectra and ESI mass spectra I have provided. NMR spectra were taken on a Varian 400 MHz FT NMR spectrometer in 99.9% by atom DMSO-d_6. Prior to being filled with sample, NMR tubes were washed out with normal acetone, the peak at or about 2.1 ppm is most likely do to residual acetone. DMSO is hygroscopic, so a significant water peak (at or about 3.2 ppm) is present. All instrumental parameters are printed on the first spectrum in each series. Samples 4 and 5 (FEAA and M1187) could not be analyzed due to the high viscosity of their solutions. All solutions were approximately 20 g/L. Samples were stored for 4 days in a dessicator while additional dye was obtained. This storage time may have contributed to the size of the water peak in the NMR spectra.

Additional NMR spectra (^{13}C, ^{1}H, COSY, etc.) can be run. Dr. Ulli Werner-Zwanziger (manager of the NMR facility) suggests a less viscous solvent such as acetone or acetonitrile. Either of these can be purchased from the NMR facility or Aldrich. Jackie and I are willing to pay for the ^{1}H spectra that have already been taken. ^{13}C and additional ^{1}H spectra probably need to be paid for by Crane. 400 MHz NMR time runs about $4.48/hr in blocks less than 6 hr, and $2.48/hr for longer blocks. NMR sample tubes are $6.11 each from the stock room (an account would need to be created with our stock room or another method of payment (~~see below~~) may need to be found). Dr. Werner-Zwanziger is very willing to help interpret the spectra if need be. She can also show someone how to use GNMR, a program to simulate NMR spectra so we can confirm our structures.

Mass spectra were recorded on a Finnagin TSQ 700 triple quadrupole mass spectrometer using an electospray ionization (ESI) source. The instrument conditions for the experiments already run were: 3500 volts on the needle, 1000 volts on the electron multiplier, -15,000 volts on the conversion dynode, and the heated capillary was set at 200 degrees C. Samples concentrations are recorded on sheet provided describing the samples. Samples were dissolved in 50 MeOH, 50 H_2O, and 0.1% acetic acid. It should be noted that ion currents were extremely low (<20 nA). This could be improved by increasing the concentration of acid in the sample. This is probably not necessary since there was sufficient ionization of analyte to provide a decent signal to noise ratio. The source is can be operated in the positive or negative ion mode. LC/MS experiments can be run by using our Waters 600e pump and 712 WISP autosampler combination. Samples have been run in a variety of conditions from 0.1 % trifluoracetic acid to 1 mM ammonium bicarbonate, so basic conditions are not a problem.

In addition to the instruments listed above, our lab also has 2 home built matrix assisted laser desorption/ionization time of flight (MALDI-TOF) mass spectrometers with resolutions ($m/\Delta m$) of approximately 1200-2000. We also have a Bruker Reflex III reflectron MALDI-TOF mass spectrometer that is capable of resolutions as high as 11,000 in the reflector mode. This instrument is also capable of performing post-source decay (PSD) structure analysis. The TSQ 700 is a triple quadrupole instrument capable of doing MS/MS and CID experiments. The ESI source can be removed and a traditional EI/CI source can be installed.

I really look forward to helping you out in any way I can. I would really like this to grow into a full-fledged collaboration between our two groups. I have presented the idea in principle to my advisor, Dr. James Reilly, and he seemed very warm towards it. I am sure that this project could provide many opportunities for both of our labs to teach each other a few things as well as solve the problem at hand, developing and testing a method for the analysis of these dye samples. Please feel free to call me at the lab (812) 855-0987 or email me at jkarty@indiana.edu. As I said before, I would really like to help solve the interesting analytical problems presented in this project.

Thank you,

Jonathan A. Karty

Jonathan A. Karty
Research Assistant

Hawman Forrest J CNIN

From: jan.willem.wegener@ivm.vu.nl
Sent: Tuesday, August 31, 1999 6:54 AM
To: hawman_forrest@crane.navy.mil
Subject: re: HPLC Analysis of Dyes

Dear Dr. Forrest,
Unfortunately the publication you reffered was the conclusion of the research project. In other words, no other investigations have been made since then. I feel sorry I am not able to help you. On the other hand I do not think many changes could have been made. Methanol is still to be preferred over acetonitrile when working with buffer salts. Columns of smaller internal diameter are available now, and will of assistance to reduce solvent consumption.
I wish you success with your investigations. If you have any questions do not hesitate to contact me. Although the work was ended long ago I remember it vividly.
Best regards,
Jan Willem Wegener

- - - - - - - - - - - - - - Original Message - - - - - - - - - - - - - -
Dr. Wegener,

Greetings, I am a chemist working for the United States Navy and I have been assigned to a project dealing with dye contaminated soil.

Our task is to develop HPLC methods for the analysis of a multitude of dyes in soil and groundwater. The soil comes from an open pit disposal area which was used in the 1940's to dispose of military dyes. This area has been under remediation for several years now and the main area of the site is under a cap. The dyes in question cover the entire spectrum, from water soluble acid/basic dyes to nonpolar dyes.

I have read your 1987 publication in Chromatographia, Determination of Organic Colorants in Cosmetic Products by High-Performance Liquid Chromatography, and your work was very intriguing. Your study was one of the very few I found that dealt with such a large number of dyes simultaneously.

We are concentrating on TLC for the quick screening methods but these will have to be backed up with HPLC for quantitation.

It was my hopes that since your article was over 10 years old you had since done more research on the subject. If you have done any further research, I would appreciative of any references you may be able to provide, especially in the area of HPLC column and mobile phase selection.

Thank you in advance for any help you may be able to provide.

Forrest Hawman
Chemist
Naval Surface Warfare Center
Explosive Sciences Branch
Code 4052
300 Hwy 361
Crane, IN 47424

(812) 854-5716

Hawman Forrest J CNIN

From: George Baughman [GBAUGHMA@hestia.fcs.uga.edu]
Sent: Tuesday, July 27, 1999 3:57 AM
To: hawman_forrest@crane.navy.mil
Subject: Dye analysis

If you will call me at 706-542-4883, I will help all I can. I love your problem! 8:30-9:00 or 10:30-11:30 am is best chance to catch me. Dr. Parry forwarded me your request for info.

Hawman Forrest J CNIN

›m: George Baughman [GBAUGHMA@hestia.fcs.uga.edu]
.nt: Wednesday, July 28, 1999 8:59 AM
To: Hawman Forrest J CNIN
Subject: RE: Dye analysis

You can call anytime. If I am out just call back a little later. I will be here until 5 or later today. I will try to find the data on the Column for you but than was a long time ago.You might be interested in the report we did for the army. AD-A249-073. "Fate of Colored Smoke Dyes". On second thought, you should get the report.
The column I used was from Bio-Rad Laboratories 2.5cm x 75. and was packed with 100gm of 200-400 mesh Bio-Beads (s-x12) also from Bio-Rad. I don't see the column we used in their 94 catalog so you may need to call and talk to them. The column we used has an adjustable rod at the top and bottom so that you can adjust the pressure on the column that is caused by solvent swelling of the packing.

Hawman Forrest J CNIN

From: George Baughman [GBAUGHMA@hestia.fcs.uga.edu]
Sent: Tuesday, August 03, 1999 5:55 AM
To: hawman_forrest@crane.navy.mil
Subject: Venkataraman

I forgot to tell you that the book is The Analysis of Synthetic Dyes by K. Venkataraman its ISBN no. is ISBN 0-471-90575-5. You should easily find it with that.
Another good source of info. is Dr. Bochner at 201-742-3901ex1332. He is anothe man who has been arround forever. Tell him I told you to call.

Hi Jacquelyn

Apologies for not getting back to you on Friday - several things cropped up.

Now where should I start?? I will annotate a few thoughts in response to your comments actually in your original message. I am afraid it will not all be good news.

I look forward to hearing from you again.

Best Wishes
Kelvin

****

From: Vorhauer Jacquelyn J CNIN <vorhauer_j@crane.navy.mil>
To:ccdknt@west-01.leeds.ac.uk
Subject: Dyes in soil and water
Date: Tue, 13 Jul 1999 11:49:11 -0500

Dr. Tapley,

I am a chemist working for Crane Naval Surface Warfare Center in Crane, Indiana. Linda Karsonovich from Tetra Tech NUS in Pittsburgh, PA e-mailed you about a project we are working on. From your response to her e-mail, it seems you might be able to assist us.

First, let me explain to you exactly what we are dealing with. There is site on our base where a large quantity of scrap dyes and dye-contaminated materials were disposed of in shallow trenches. This went on from the 1930s to the early 1970s. The EPA has since placed a cap over this site. My lab has been give the job of analyzing the soil around the cap to ensure that all the dye-contaminated soil was placed under the cap and to quantify how much dye has made it through to the ground water and near by streams.

Our task has been simplified slightly because our lab has some production line samples of dyes that were taken directly from our manufacturing lines approximately 45 years ago.

Since starting this project, we have encountered several difficulties...

1) We have not been able to develop HPLC procedures that separate the wide variety of dyes we have samples for.

{[You never will - in my opinion. If you consider the chemistry of the dyes then it becomes obvious that you will not be able to

separate and identify them using a single chromatography method (it is like comparing chalk with cheese). You have anionic, cationic and neutral dyes, and also pigments. Note, true pigments are not soluble in solvents (or at most only slightly soluble) and therefore can not be analysed by HPLC.]}

We have done extensive library searches through chromatography journals and other sources. So far we have not come across an article where the scientist has dealt with such a wide variety of dyes. For example, when we try a method that is supposed to separate one of two dyes we have, we find that our other dyes coelute with the target dyes. We have tried modifying some journal article methods and have tried developing some of our own with minimal success. The HPLC we are using is equipped with a Photo Diode Array detector so we do have the capability of spectral matching and we have compiled a spectral library of the dyes we have in house.

{[Note, your lab samples have not been kept under the same conditions as the field samples, therefore the chemistry of the samples may well be very different!]}

2) When the EPA and our environmental department first started this project, they went out with shovels and dug around the area to determine how far the cap would have to extend. They did this by looking for any visible signs of dyes in the soil. We have obtained these samples and have run a preliminary analysis. There are a lot of dyes that UV-vis spectra and retention time do not match the ones we have in-house. Do you have any suggestions how we may go about identifying these dyes?

{[LC-MS or CE-MS]}
{[We are very keen on the technique of Capillary Electrophoresis (CE) (we have six CE instruments!) for the analysis of a wide range of colorants in a wide range of matrices, although CE is not particularly good for trace analysis]}

We do not have access to an LC/MS in our laboratory. We were hoping there might be a UV-vis spectral database out there that may make identification easier.

{[No - not that I am aware of. Although here in the Department of Colour Chemistry we are working on a "Dictionary of Colorants" in collaboration with the SDC.]}

3) What do you foresee as possible interference to the analysis? Supposedly the ground water is highly contaminated with metals.

{[Many metals are able to complex with dyes, especially acid dyes,

and this will change the colour (UV-Vis absorbance profile) of the dye concerned.]}

The soil is yellow clay.

{[Natural colorant to complicate your task further. You shoul do some chemical analysis on this clay!]}

One of my biggest concerns is that some of these dyes may have broken down over the years and we may be seeing break down products in our initial screening of the soil samples instead of the original dye. Any comments you have on this subject would be greatly appreciated.

{[Some of the dyes will have definately broken down - at least in part - and I would be amazed if you did not see any breakdown products (although their detection will obviously be dependent on the method of analysis used and be subject to them not diffusing away from the original area). Several staff (myself included) within the department have been involved in studying/developing natural systems (eg. bacteria or white rot fungi) for breaking down dyes. Under the right conditions certain bacteria can break down dyes in a matter of minutes. Note, trying to establish reaction pathways and identifying break down products is extremely difficult - even if you have access to eg. HPLC-MS.]}

4) The EPA insists that we have an internal standard and a surrogate standard for our analysis. Two distinctively different dyes would be the most logical answer ~~from what we can figure on our part.~~, However, we have no way to judge what dyes we could buy that would not interfere with the dyes we already have on our list. We would like to avoid the costly method of trial and error if at all possible. What do you suggest?

{[The use of standards makes a lot of sense, especially if you hope to be able to achieve some type of quantitative information. The choice of standards would be dependent on a number of factors, not least of which would be the actual sampling, extraction and anlaysis methods used. I would envisage different standards being used with different methods for different groups of colorants/chemicals (including derivatives and breakdown products). Selecting suitable standards appears to be the least of your problems at this stage.]}

I am attaching an Excel file that has all the information on our in-house dyes.

{[Certainly a wide variety of colorants. One further point for you to note is that many commercial dye products actually consist of several different chemicals, for example I have found (by CE

analysis) over twelve different coloured species in one particular "single" commercial dye; there can also be other chemicals present such as diluents, buffers, salts, dispersing agents, anti-dusting agents and impurities (eg dye intermediates, plus oxidation/hydrolysis products - especially if stored for a long period)!]}

Any help you can provide us would be greatly appreciated.

{[To tackle and solve this project properly will require a considerable amount of time, effort and expertise, plus suitable equipment. Therefore, this project is likely to be very expensive - the figure of one million pounds is probably fairly realistic!

If you would like the Department of Colour Chemistry/myself to be involved (either small part or large part) then there will clearly need to be some benefit/incentive for us to do this!

Academics no longer have "free time" - due to pressures from various sources. Please let me know if you would like to utilise the expertise/facilities of the department and also under what terms. If you like I could formulate a proposal to carry out part of the work or act on a consultancy basis.]}

Sincerely,
Jacquelyn Karty
Chemist
Explosive Science Branch, Naval Surface Warfare Center, Crane Division 300 Highway 361 Crane, IN 47522 Phone: 812-854-3143 Fax: 812-854-5054 E-mail: vorhauer_j@crane.navy.mil

<<Storeroom Dye Samples.xls>>

Dr Kelvin N Tapley
Department of Colour Chemistry
University of Leeds
Leeds LS2 9JT
England
Tel 0113 2336732
Fax 0113 2332947
email K.Tapley@leeds.ac.uk

From: Hawman Forrest J CNIN
Sent: Saturday, June 05, 1999 2:08 PM
To: edm@pfaltzandbauer.com
Cc: Vorhauer Jacquelyn J CNIN
Subject: FW: Dye standards

Sir,

Please read the following emails, I had sent them to the wrong email address.

Forrest Hawman
Chemist
Crane Naval Surface Warfare Center
Explosive Sciences Branch
Code 4052, Building 2707
300 Hwy 361
Crane, Indiana 47522

-----Original Message-----

Sent: Saturday, June 05, 1999 2:11 PM
To: 'edm@pfaltzbauer.com'
Cc: Vorhauer Jacquelyn J CNIN
Subject: FW: Dye standards

Sir,

I sent you the following e-mail yesterday. I realized after I sent it that I would be out of town next week, and the information is important. Could you please reply to Jackie Vorhauer at vorhauer_j@crane.navy.mil as well as me. This email is Cc to her so you can get her email address from the header also. If you have any question please feel free to call her at (812) 854-3143.

Thank you,

Forrest Hawman
Chemist
Crane Naval Surface Warfare Center
Explosive Sciences Branch
Code 4052, Building 2707
300 Hwy 361
Crane, Indiana 47522

Phone (812) 854-5716

-----Original Message-----

From: Hawman Forrest J CNIN
Sent: Friday, June 04, 1999 12:17 PM
To: 'edm@pfaltzbauer.com'
Cc: Vorhauer Jacquelyn J CNIN
Subject: Dye standards

Greetings,

Linda Karsonovich

Dye project

412-921-4040

I think this is what you are looking for. Let me know if it isn't.

Jackie Vorhauer

Please note: we are no longer using the same LC system for the disodium fluorescein analysis

All samples will be analyzed using 5 standards as required by Method #8330 that blanket the samples.

A standard from Environmental Resource Associates (ERA) will be obtained for method #8330 for analysis in addition to required blanks, matrix spike samples and laboratory spiked samples. All will be analyzed in accordance with the prescribed procedures.

QUANTITATIVE ANALYSIS OF DISODIUM FLUORESCEIN BY HPLC

PURPOSE

This procedure establishes the activities associated with quantitative analysis of disodium fluorescein by HPLC.

SAMPLE PREPARATION

A sample, a 900-ml aliquot, is evaporated by a dry nitrogen purge to 5 ml.

APPARATUS AND REAGENTS

Anotec Syringe Filter, Catalog # 2001050U, 0.2 micrometers

5 ml volumetric flasks

1000 ml beakers

Shimadzu SIL-9A Auto Injector

LDC Model III Constametric Pump

Spectra-Physics SP4270 Integrator

Alltech 345 Solvent Recycler

Column Packing:	Alltech Econosil, C18, 10 micron
Column Size:	Length: 250 millimeter, Inside Diameter: 4.6 millimeter
Mobile Phase:	Isocratic, 50% Methanol, 50% Deionized Water and a Tetrabutylammonium Phosphate, 0.5 molar, pH 7.5, Kit from Alltech (1 kit per liter of mobile phase).
Flow Rate:	1.0 milliliter per minute

Detector: Ultraviolet at 254 nanometers

Injection Volume: 10 microliters

Standard: Disodium Fluorescein, Eastman Catalog # 735, Lot # AGA, (85% min. by spectro analysis, corrected for water). Disodium Fluorescein standards are prepared in methanol.

Methanol: All methanol used for extraction and analysis is Fisher Scientific, HPLC grade.

Water/Blank: All reagent water used in the analysis procedure is steam distilled and then filtered through a Millipore Model ZD40 (Analytical) Milli-Q-Plus Water System.

Nitrogen: Dry nitrogen gas is supplied by liquid nitrogen cook off.

Glassware: Glassware is rinsed with high quality distilled water.

Filter: Anotop 0.2 micron

Instrument Detection Limits

The limit of detection is dependant on the amount of concentration of the sample. Using a 900 milliliter aliquot of sample concentrated down to 5 milliliters, the limit of detection for Disodium Fluorescein is about 10 parts per billion.

The lowest calibration standard is 1.64 ppm. The reporting limit for the method of 10 ppb, is dependent on the amount of concentrating of the original sample (in this case 180 times). The spectrophotometer analyzes a concentrated water sample. This limit was used because, after concentration from 900 ml into 5 ml, satisfactory results were obtained.

CALIBRATION PROCEDURES

Disodium Fluorescein, Eastman Catalog # 735, Lot # AGA, (85% min. by spectro analysis, corrected for water) is used for reference. Disodium Fluorescein standards are prepared in Fisher Scientific, HPLC grade, methanol. A concentrated standard was prepared and diluted to the range of the sample concentrate (2 ppm, 4.5 ppm, and 9 ppm) in the analysis of the first two samples. For further samples, five standards will be prepared in the range that will blanket the sample values. These standards are in addition to the standard blank. The standards will be evaluated for linear correlation. The linear correlation factor will be at a minimum of 0.995.

The matrix spiking solution should consist of 15 ppb of blue dye standard and 25 ppb of Disodium Fluorescein standard after addition to the neat tank water sample, i.e., approximately double the concentration of each component. Recovery will be 100 +- 20%.

Initial calibration is performed at the start and at the end of a shift. Acceptable criteria for calibration is a stable baseline (correlation factor 0.995).

A laboratory reference standard is prepared independently of the calibration stock using de-ionized laboratory water. It is analyzed by the above procedure and must be within ± 10% of the true value. If the reference is not within ± 10% of the true value, it will be reanalyzed. If the reference is still outside the criteria, the reason will be determined and corrected.

Code 4052 will strive to obtain a certified standard dye sample solution will be obtained from an outside source for further calibration checks/recovery. Recovery will be +- 10% of true value.

INTERFERENCES AND CORRECTIVE MEASURES

Liquid Chromatography separation was used to avoid possible interferences.

ANALYTICAL PROCEDURE

Concentration

The sample bottle containing the dye contaminated water is shaken to put all solids into suspension. A 900 milliliter aliquot of sample is poured into a graduated cylinder. The aliquot is divided between three (1000-milliliter) beakers. A nitrogen line is attached to the lip of each beaker and the beakers are covered with aluminum foil. Nitrogen flow is adjusted to slightly move the surface of the sample liquid. The samples are evaporated to dryness under ambient conditions.

Extraction

The dry sample residues are each extracted with three or more 2-milliliter aliquots of methanol. Each beaker residue is extracted until there is no visible blue color left in the extract or the residue. The combined extracts are quantitatively filtered. The filtered methanol solution is evaporated to less than 2 milliliters. The concentrate is quantitatively transferred to a 5-milliliter volumetric flask and diluted to volume with methanol.

Surrogates? Internal std.

A high performance liquid chromatography (HPLC) procedure for separation of food dyes, found in the Hewlett Packard Catalog, was used on the known and unknown dye samples. The procedure gave a good separation for the Disodium Fluorescein standard and a peak with the same retention time (six minutes) was observed in the unknown sample. (The procedure uses a C18 column with a one milliliter per minute flow rate of 50/50 mixture of methanol and water. The solvent is buffered with, Tetrabutylammonium phosphate solution from Alltech. A 254 nanometer wavelength, detector was used for data collection). This separation procedure can be used for quantitative analysis of the Disodium Fluorescein (uranine) dye. Also, an appropriate secondary confirmation column shall be used to confirm the identify of unknown peaks and all peaks associated with analytes of interest. However, it is the primary column which shall be used for quantitation purposes.

CALCULATIONS

Peak heights of the samples and standards are used for quantitative determination of Disodium Fluorescein. A response factor for the standards is determined from a plot of concentration and peak height of the dye. The peak height of the dye in the sample is multiplied by the response factor and in turn divided by the concentration factor. Results are reported in parts per billion of Disodium Fluorescein in the original water sample.

QUALITY CONTROL

The sample quality control will be handled by in accordance with the Crane NPDES QA/QC plan (Appendix E).

DATA REPORTING

The sample data reporting will be handled in accordance with the Crane NPDES QA/QC plan (Appendix E).

QUANTITATIVE ANALYSIS OF MIL-D-16627, BLUE DYE BY ABSORPTION SPECTROPHOTOMETRY

PURPOSE

This procedure establishes the activities associated with quantitative analysis of MIL-D-16627, Blue Dye by Absorption Spectrophotometry.

SAMPLE PREPARATION

Evaporate the sample, a 900-ml aliquot, without heat under a dry nitrogen purge to 10 ml.

APPARATUS AND REAGENTS

10 ml volumetric flasks

Perking-Elmer model 552 spectrophotometer

one centimeter path length cuvettes

pipettes

Acetic Acid, Fischer Scientific reagent grade

Reference standards are prepared from a sample of Mil-D-16627 dye in methanol. Mil-D-16627 contains only 50 percent dye according to specification. One drop of acetic acid is added to 10 milliliter of the final methanol standard.

Water/Blank: All reagent water used in the analysis procedure is steam distilled and then filtered through a Millipore Model ZD40 (Analytical) Milli-Q-Plus Water System.

INSTRUMENT DETECTION LIMITS

The limit of detection for the blue dye is 5 parts per billion but background interference could change the limit. The lowest standard is 1.6 ppm. The reporting limit for the method of 5 ppb, is dependent on the amount of concentrating of the original sample (in this case 90 times). The spectrophotometer analyzes a concentrated water sample. This limit was used because, after concentration from 900 ml into 10 ml, satisfactory results were obtained. This limit was used because, after concentration from 900 ml into less than 10 ml, interferences from sample background were too overwhelming for the instrument to analyze.

INTERFERENCES AND CORRECTIVE MEASURES

The acetic acid removes the Disodium Fluorescein absorption and slightly increases the blue dye absorption. One drop of acetic acid is added to 10 milliliter of the methanol standard.

CALIBRATION PROCEDURES

Peak heights of the samples and reference standards are used for quantitative determination of the Mil D-16627 blue dye. No attempt was made to distinguish

between the two blue dyes. A response factor for the standards is determined from a plot of concentration and peak height of the dye. The peak height of the dye in the sample is multiplied by the response factor and in turn divided by the concentration factor. Results are reported in parts per billion of blue dye in the original water sample.

A concentrated standard is prepared and diluted to the range of the sample concentrate 1.6 ppm 3.29, and 16.4 ppm for the blue dye in the analysis of the first two samples. For further samples, five standards will be prepared in the range that will blanket the sample values. These standards are in addition to the standard blank. The standards will be evaluated for linear correlation. The linear correlation factor will be at a minimum of 0.995.

The spiking solution should consist of 3 ppm of blue dye standard and 25 ppm of Disodium Fluorescein standard added to a 900 ml tank water sample aliquot before concentration and extraction steps are performed. Recovery will be 100 +- 20%.

Initial calibration is performed at the start and at the end of a shift. Acceptable criteria for calibration is a stable baseline (correlation factor 0.995).

A laboratory reference standard is prepared independently of the calibration stock using de-ionized laboratory water. It is analyzed by the above procedure and must be within ± 10% of the true value. If the reference is not within ± 10% of the true value, it will be reanalyzed. If the reference is still outside the criteria, the reason will be determined and corrected.

Code 4052 will strive to obtain a certified standard dye sample solution from an outside source for further calibration checks/recovery. Recovery will be +- 10% of true value.

ANALYTICAL PROCEDURE

The sample concentrate prepared for Disodium Fluorescein analysis was diluted by half to 90 times concentration using methanol. The background is too high at 180 times concentration. One drop of acetic acid is added to the 10 milliliter volumetric flask. The acetic acid removes the Disodium Fluorescein absorption and slightly increases the blue dye absorption. The Mil-D-16627 blue dye was scanned in the visible range with a spectrophotometer. The blue dyes have a maximum absorbance at 408 nanometer (nm) and a shoulder at 390 nm. The visible spectrophotometer can be used for quantitative analysis of the Mil-D-16627 dye.

CALCULATIONS

The blue dye reference standard weight is corrected for purity. Quantitation was based on the peak height at 408 nm. A response factor for the standards is determined from a

plot of concentration versus peak height of the dye. The peak height of the dye in the sample is multiplied by the response factor and in turn divided by the concentration factor. Results are reported in parts per billion of blue dye in the original water sample.

QUALITY CONTROL

The sample quality control will be handled in accordance with the Crane NPDES QA/QC plan (Appendix E).

DATA REPORTING

The sample data reporting will be handled in accordance with the Crane NPDES QA/QC plan (Appendix E).

TOTAL ORGANIC CARBON (TOC)

Appendix G covers the TOC analysis used in the evaluation of the dye water. The sample will be analyzed as received.

TOTAL SUSPENDED SOLIDS (TSS)

Appendix H covers the procedure for non-filterable residue in drinking or waste water. This test is commonly referred to as TSS. This method will be used for every sample and generate non-soluble particulate samples for FT/IR, SEM, GC-MS, and TLC analysis.

TOXICITY (MICROTOX)

Appendix B covers the procedure for toxicity analysis used in the evaluation of the dye water. The Microtox test is performed on as received samples and on samples representative of the water after treatment. The purpose of the data collection is to show relative toxicity before and after treatment without any correlation to human health toxicity data.

ION-EXCHANGE CHROMATOGRAPHY (IEC)

Appendix I covers the inorganic/organic anion analysis by IEC used in the evaluation of the dye water. Full analysis for all anions will be performed.

MEMORANDUM 11/23/99

Karen

To: Tom Brent
Bill Gates — Please review. Useful?

Bill

From: Peter Ramanauskas

Date: November 19, 1999

Re: Potential DBG Dye Identification Method

Recently, George Bollweg had attended a forensics workshop here at EPA and thought this information might be useful for the Crane DBG Project.

Andrew Grange, a chemist from USEPA's National Exposure Research Lab in Las Vegas, presented at the forensics workshop on a technique called mass peak profiling from selected ion recording data (MPPSIRD). This appears to be a derivative of GC-MS used to help identify unknown compounds of very similar mass. He appeared genuinely interested in providing services to USEPA Regions and the states, in part to help justify what looked like considerable development work on the method.

I sent Andrew a copy of the agenda from our conference call of September 10th to give him an idea of the potential application of this technique to the Crane site. I have included his response for you to review and forward on to Tetra Tech and the Crane lab to see if this may be useful. If so, please contact me and we can make any needed arrangements with Andrew.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
NATIONAL EXPOSURE RESEARCH LABORATORY
P.O. BOX 93478 • LAS VEGAS, NV 89193-3478

NOV 16 1999

OFFICE OF
RESEARCH AND DEVELOPMENT

MEMORANDUM

SUBJECT: Identification of Dyes

FROM: Andrew H. Grange, Ph.D.
Physical Scientist
Environmental Chemistry
Environmental Sciences Division

TO: Peter Ramanauskas (DW-8J)
Environmental Scientist
Corrective Action Section
Waste Management Branch
Waste Pesticides and Toxics Division

It is likely that Mass Peak Profiling from Selected Ion Recording Data (MPPSIRD) and the Profile Generation Model (PGM) could be used to identity dyes and to provide compelling evidence should it be required to encourage a PRP to pay for clean-up or containment.

The attachment indicates two broad categories of dyes are of interest. Those that are insoluble in water would probably provide gas chromatographic peaks. Elemental compositions of the molecular ion (or apparent molecular ion) and fragment ions should lead to identification of some or all of the dyes in the water insoluble fraction.

The ionic or very polar dyes in the water soluble fraction can be studied using electrospray ionization (ESI) or atmospheric pressure chemical ionization (APCI). These are "soft" ionization techniques that produce primarily a molecular ion (or quasi-molecular ion with an extra H or Na atom [+ ions] or lacking an H atom [- ions]). However, increasing voltages within these two sources induces fragmentation to provide ionized portions of the molecule. Our experience with these sources is limited and application of MPPSIRD to liquid sample introduction is a major thrust of our R & D. A disadvantage of these techniques is that in general, they are less sensitive than the gas-phase sample introduction techniques (e.g. GC and probe). But your samples probably contain fairly high levels of dyes, since 50 tons of dyes were deposited in (and perhaps splashed around) the trenches. Your need to identify these dyes provides us with an excellent opportunity to again demonstrate the utility of our new analytical tools for compound identification using gas-phase sample introduction and analytes relevant to the Agency for liquid sample introduction.

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Several years ago, we demonstrated the feasibility of determining elemental compositions of compounds introduced in a liquid using coaxial-flow, secondary ionization mass spectrometry (SIMS). SIMS provides less sensitivity than ESI or APCI and is out of favor. However, we did determine the elemental composition of Acid Orange 8 using MPPSIRD at the time. Therefore, extension of MPPSIRD to liquid sample introduction should be feasible for your dyes as well.

If you could send us portions of your sample extracts (1 mL would be wonderful), we would be pleased to determine elemental compositions of ions produced from the dyes and hopefully identify them for you. If successful, we intend to write a paper on our results for a mass spectrometry journal or the forensics journal mentioned during the course – you and your colleagues would be co-authors. We would perform the analyses without charge. There is always the chance that the samples could prove intractable or that a "hot problem" could demand our attention. As of now, however, we have the time available to investigate your dye samples. If you are interested in our analyzing your samples, let me know. Thank you for your interest.

Attachment

Characterizing Hazardous Waste Constitutents: A New Tool

Andrew H. Grange,[1] William C. Brumley,[1] and John W. Farley[2]*

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[2]Department of Physics, UNLV, Las Vegas, NV 89154-4002
[*]*National Research Council Senior Research Associate*

Problem: Incomplete Characterization of Complex Samples

A typical hazardous waste site accumulates wastes from numerous sources. Over time, these wastes can react to form new compounds, with or without catalysis by solar radiation, components in air, or metals in soil. Hundreds of compounds might be present. To fully characterize each site, every component in the numerous samples would have to be identified and quantified. Typically, wastes are analyzed for priority pollutants listed in appendices to EPA methods. In general, these are compounds know to be toxic that are widely used and for which analytical methods exist. These lists represent but a small fraction of the compounds that might be found in hazardous waste sites. New analytical techniques to analyze the other components are needed to assess risks to humans and the ecosystem and are under development at the Environmental Sciences Division of the EPA's National Exposure Research Laboratory.

Current Practice

Gas chromatography/low resolution mass spectrometry (GC/LRMS) is used to analyze for many of the compounds listed in EPA appendices. When a sample contains dozens of compounds, coelution of multiple components is common, and the mass spectra arise form more than one compound, even after background subtraction. Matches with library mass spectra become poor and compound identification uncertain. This was the case for the extract of a black, viscous sample from a Superfund site in West Virginia; the total ion chromatogram is the upper trace in Figure 1 (1). No credible library matches were found for many mass spectra.

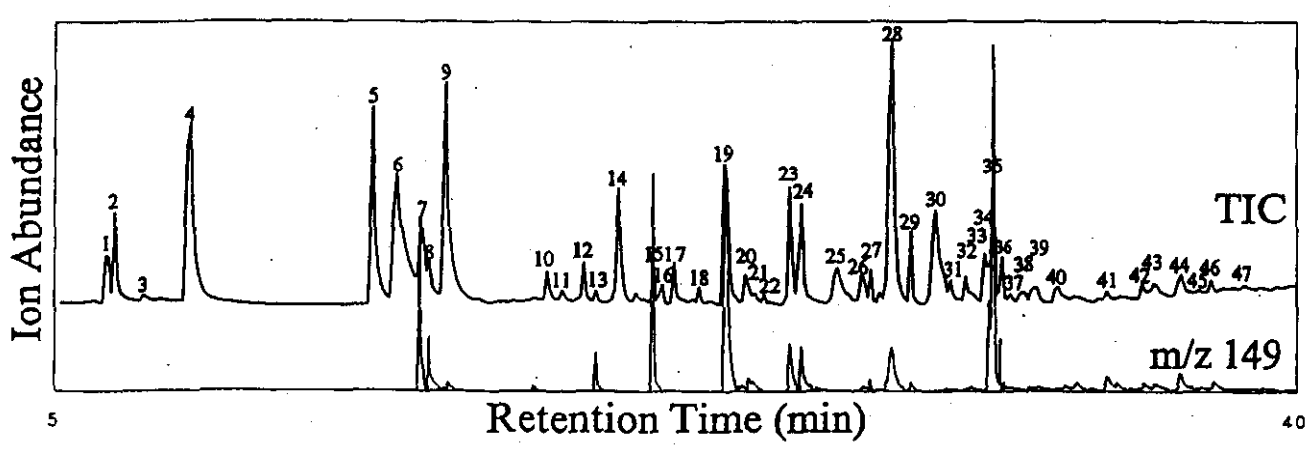

Figure 1.

Additional Information from High Resolution Measurements: Elemental Compositions of Molecular or Fragment Ions

In Table 1 (1) are listed the chromatographic peaks in Figure 1 for the Superfund site sample and the elemental compositions for the largest ion containing only the most abundant isotope of each element produced from the compound most responsible for each peak. Only 12 compounds were identified from library matches of mass spectra, and in some cases, the elemental compositions were needed to select the correct match. The identified compounds account for only a small fraction of the total signal and mass of the sample. The $C_8H_7NS^+$ ion (149.0299 amu), indicative of benzothiazole, was monitored at a mass resolution of 20,000 (lower trace in Figure 1) and indicated that 11 unidentified compounds contained the benzothiazole group. Benzothiazole derivatives are used in the rubber and dye industries. High resolution data also provided the elemental compositions. Table 1 provides far more information about the sample than could be determined by GC/LRMS alone.

The Need for High Mass Resolution

High mass resolution is required for two reasons: to separate the signal due to analyte ions from interferences and to provide narrow error limits for exact mass determinations.

Shown in Figure 2 are calculated mass peak profiles at 1,000 and 20,000 resolution for ions produced from the calibrant, from column bleed, and from an analyte, all with a nominal mass of 281 amu. The overlap of the profiles at the lower resolution indicates all three ions would contribute to the signal observed at m/z 281, distorting the mass spectrum and the exact mass obtained for the analyte ion. At the higher resolution, the profiles are baseline resolved and the analyte ion can be monitored without interferences form the other two ions.

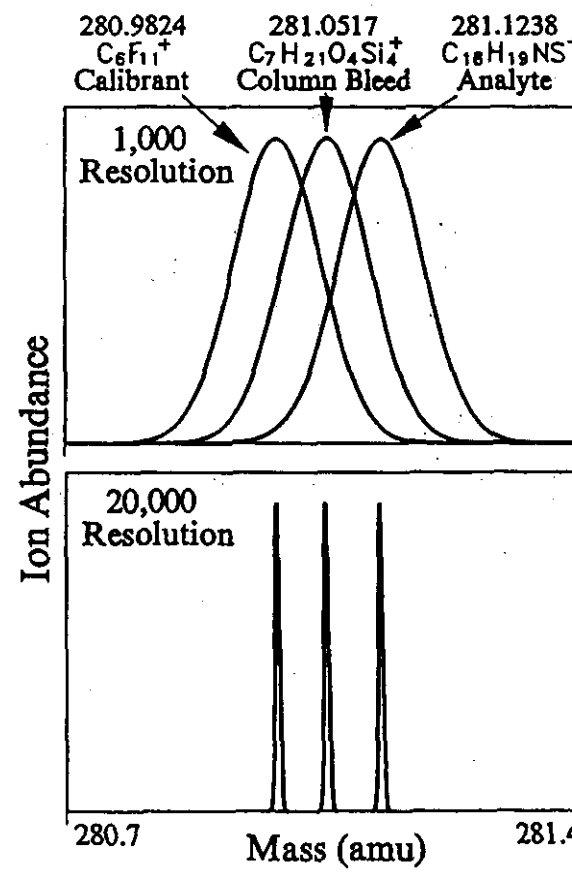

Figure 2.

Using Isotopic Masses and Relative Abundances to Identify Large Ions

The number of elemental compositions possible for an exact mass within the error limits of its determination increases rapidly with the mass of the ion. Only five of the ions in Table 1 were identified form the exact mass of the ion containing only the isotopes with greatest natural abundance (M). The other ions were identified using criteria based on the exact masses and relative abundances of ions with masses 1 or 2 amu greater than M, denoted as M+1 and M+2 (2). The mass peak profiles in Figure 3 were calculated for the last ion in Table 1 and for one of 17 other compositions possible based on the exact mass determined for M. The differences in the exact mass of the M+2 profile and the abundances of the M+1 and M+2 profiles relative to the M profile were large enough to eliminate the bottom composition. Peak broadening of the upper M+2 profile arises from the mass difference between $C_{24}H_{28}N_2S^{34}S^+$ (410.1652 amu) and $^{13}C_2C_{22}H_{28}N_2S_2^+$ (410.1761 amu). The shape and width of the M+2 profile are also useful for distinguishing between possible compositions.

Table 1. Nominal masses, compositions, and tentative identifications of ions corresponding to chromatographic peaks in the total ion chromatogram

TIC Peak #	Nominal Mass	Composition	Criteria Met GC	Criteria Met Probe	Tentative Compound Identification
1	100	$C_6H_{12}O$	na		2-methyl cyclopentanol
2	98	$C_6H_{10}O$	na		
3	113	$C_7H_{15}N$	na		n-methyl cyclohexanamine
4	93	C_6H_7N	na		aniline
5	135	C_7H_5NS	5		benzothiazole
6	127	$C_7H_{13}NO$	na		
7	149	C_8H_7NS	4		2-methyl benzothiazole
8	149	C_8H_7NS	4		3-methyl-1,2-benzisothiazole
9	141	$C_8H_{15}NO$	5	2	
10	173	$C_{12}H_{15}N$	5		
11	178	$C_{12}H_{18}O$	3	2	
12	175	$C_{12}H_{17}N$	5		
13	191	$C_{11}H_{13}NS$	3[a]		2-butylbenzothiazole
14	169	$C_{12}H_{11}N$	5	5	
15	205	$C_{12}H_{15}NS$	5	4	contains benzothiazole group
16	192	$C_{12}H_{16}S$	5	2	1,2,3,4,6,7,8,9-octahydrodibenzothiophene
17	187	$C_{13}H_{17}N$	5		
18	183	$C_{13}H_{13}N$	5		
19	205	$C_{12}H_{15}NS$	5		contains benzothiazole group
20	203	$C_{12}H_{13}NS$	2		
21	219	$C_{13}H_{17}NS$	5		contains benzothiazole group
22	215	$C_{13}H_{13}NS$	5		
23	205	$C_{12}H_{15}NS$	5		alkylated benzothiazole
24	205	$C_{12}H_{15}NS$	5		alkylated benzothiazole
25	199	$C_{12}H_9NS$	4		phenothiazene
26	234	$C_{13}H_{18}N_2S$	4		
27	246	$C_{14}H_{18}N_2S$	5	4	
28	232	$C_{13}H_{16}N_2S$	5		N-cyclohexyl-2-benzothiazolamine
29	246	$C_{14}H_{18}N_2S$	5		methyl-N-cyclohexyl-2-benzothiazolamine
30	226	$C_{13}H_{10}N_2S$	5		o-anilinophenylester thiocyanic acid
31	266	$C_{18}H_{22}N_2$	5		
32	281	$C_{18}H_{19}NS$	5		
33	260	$C_{14}H_{16}ON_2S$	5		
34	260	$C_{18}H_{16}N_2$	5	3	
35	285	$C_{18}N_{23}NS$	5		contains benzothiazole group
36	283	$C_{18}H_{21}NS$	4		contains benzothiazole group
37	280	$C_{16}H_{28}N_2S$	5		
38	286	$C_{18}H_{26}ON_2$	5	3	
39	308	$C_{19}H_{20}N_2S$	5		
40	268	$C_{14}H_8N_2S_2$	5		
41	296	$C_{18}H_{20}N_2S$	5		
42	314	$C_{19}H_{26}N_2S$	5		contains benzothiazole group
43	290	$C_{18}H_{14}N_2S$	5		contains benzothiazole group
44	326	$C_{18}H_{18}N_2S_2$	5	3[c]	contains benzothiazole group
45	354	$C_{20}H_{22}N_2S_2$	5		
46	372	$C_{24}H_{24}N_2S$	4		contains benzothiazole group
47	336	$C_{19}H_{16}N_2S_2$	5		
	301	$C_{19}H_{13}N_2S$		3[b]	
	365	$C_{21}H_{21}N_2S_2$		4[b]	
	402	$C_{24}H_{22}N_2S_2$		4	
	408	$C_{24}H_{28}N_2S_2$		5	

[a]Criteria tested for m/z 162 ion due to low signal
[b]For this ion, an absence of P atoms was assumed
[c]Insufficient to exclude all other compositions not containing P atoms

The New Analytical Technique that Provides Elemental Compositions

Mass Peak Profiling from Selected Ion Recording Data (MPPSIRD) is a new data acquisition technique that increases sensitivity 100-fold and speed by a factor of 6 over conventional scanning modes at a resolution of 20,000. Full scans at high mass resolution have long been used to determine exact masses of synthetic products introduced into the ion source over an extended period of time, but have not been used to obtain exact masses for ions produced from compounds that enter the source as narrow chromatographic peaks, because the scan rate is too slow to track the peaks and because the sensitivity is too low to observe many components in mixtures. In Figure 4, a simulated full scan is shown. Data are acquired as the m/z ratio observed varies continuously across the entire mass range. With Selected Ion Recording (SIR), a set of individual m/z ratios is monitored to provide much greater sensitivity and speed. SIR has been used historically to monitor m/z ratios atop mass peak profiles from target analytes as illustrated by the asterisks in Figure 4.

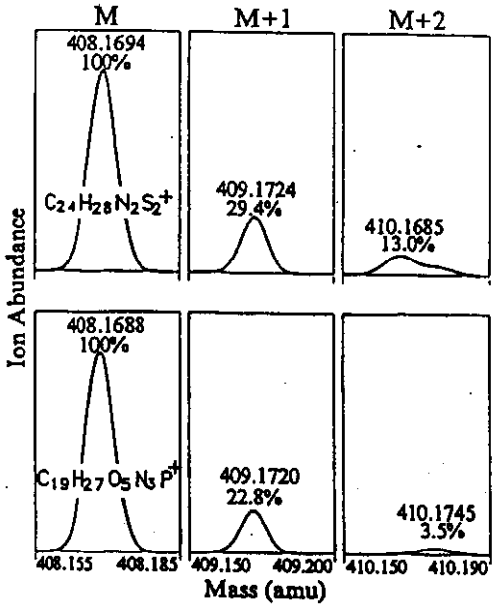

Figure 3.

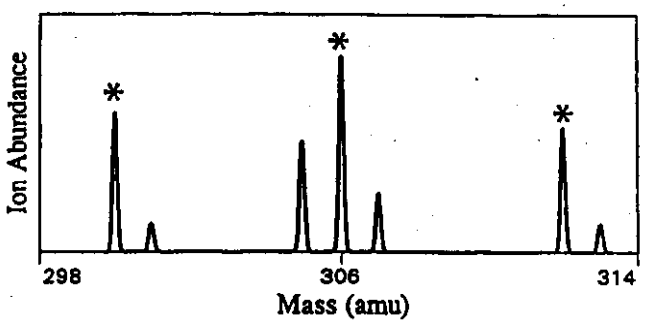

Figure 4.

In Figure 5, the new technique is illustrated. To plot the full mass peak profile, m/z ratios were monitored across a *single* profile (3). To plot the partial profiles, six m/z ratios each were monitored across the top portion of the M, M+1, and M+2 profiles (4). Monitoring of partial profiles provides a rapid cycle time (0.8 sec), which enables tracking of chromatographic peaks. The exact mass of a profile is the weighted average of several m/z ratios across the top of the profile. Triplicate exact mass determinations at 20,000 resolution using a mass increment of 5 ppm provided masses accurate to within 2.5 ppm. The relative abundances are the appropriate ratios of the sums of the six points used to plot the partial profiles. A Profile Generation Model [PGM (2)] calculates the exact masses and relative abundances expected for all possible compositions when six m/z ratios are monitored across M, M+1, and M+2 profiles and applies criteria to reject those compositions that are not consistent with the data. Elemental compositions are determined for ions with masses less than 600 amu and for larger ions in many cases (2).

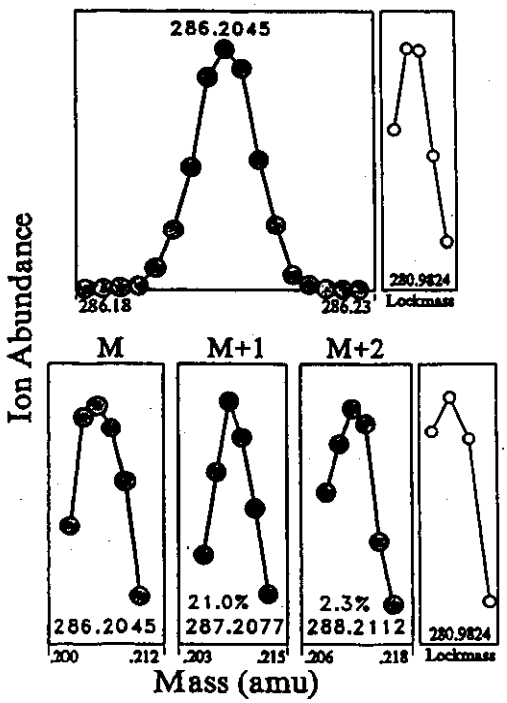

Figure 5.

Screening for Target Compounds

The extract was introduced with a heated probe while the M, M+1, and M+2 partial profiles were monitored for each

of nine ions listed in Table 1 that contained different numbers of N, O, or S atoms to learn if their compositions could be determined rapidly without prior component separation (1). All but the correct compositions were rejected for eight of the nine ions.

A poor library match for strychnine ($C_{21}H_{22}N_2O_2$; 334.1681 amu) was found for a trace component, which was determined to be ($C_{24}H_{18}N_2$; 334.1470 amu). When probe introduction was used to screen for strychnine, no signal was observed for its M, M+1, and M+2 ions. Thus, to a very low detection limit, strychnine was not found in the extract.

Other Applications of MPPSIRD

MPPSIRD is used routinely in our laboratory with GC and probe introduction. The last four ions in Table 1 were identified using the probe, since chromatographic peaks were not observed for these ions. Quantitative analysis of Aroclors (commercial mixtures of PCBs) using a carbon-labeled PCB isomer as the calibration standard has also been demonstrated (5). Many environmental contaminants are too labile, polar, or large to provide chromatographic peaks. Use of MPPSIRD with liquid sample introduction techniques that utilize electrospray ionization (ESI) or atmospheric pressure chemical ionization (APCI) are currently being investigated. These techniques employ "soft" ionization and will provide molecular or quasi-molecular ions for most compounds.

Summary

A new analytical tool using high resolution mass spectrometry (MPPSIRD and the PGM):

- provides elemental compositions for ions with masses up to 600 amu
- helps identify major and trace components in complex mixtures
- quantifies mixture components using an internal standard

Elemental compositions are used to:

- reject library matches with incorrect compositions
- confirm library matches with the correct composition
- limit a compound's identity to a number of isomers

High mass resolution:

- provides high selectivity by separating analyte ion signals from interferences
- permits determination of exact masses within small error limits

Plotting mass peak profiles from selected ion recording data provides:

- high sensitivity enabling use of 20,000 resolution
- a fast cycle time to track chromatographic peaks
- a highly selective, sensitive, and rapid screening technique for target analytes

References

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(2) Grange, A.H.; Brumley, W.C.; *J. Am. Soc. Mass Spectrom.* 1997, *8*, 170-182.
(3) Grange, A.H.; Donnelly, J.R.; Brumley, W.C.; Billets, S.; Sovocool, G.W. *Anal. Chem.* 1994, *66*, 4416-4421.
(4) Grange, A.H.; Donnelly, J.R.; Brumley, W.C.; Sovocool, G.W. *Anal. Chem.* 1996, *68*, 553-560.
(5) Grange, A.H.; Brumley, W.C.; *Proceed. 44th ASMS Conf. on Mass Spectrom. & Allied Topics*, 5/12-16, 1996, Portland, OR, 536.

Well Pollutants Identified With a New Mass Spectrometric Technique

A.H. Grange[1*], G.W. Sovocool[1], J.R. Donnelly[2], and F.A. Genicola[3]

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*National Research Council Senior Research Associate

Abstract. The incidence of cancer observed in children living near a contaminated site in New Jersey was higher than normal. Trace amounts of several isomeric compounds were detected by GC/MS in a concentrated extract of water taken from a well that serviced 50,000 people. Mass Peak Profiling from Selected Ion Recording Data (MPPSIRD) provided the sensitivity and scan speed necessary to acquire mass peak profiles at mass resolutions of 10,000 to 20,000 for the molecular ion ($M^{+\cdot}$) and 10 fragment ions as capillary GC peaks eluted. Exact mass differences between the molecular and fragment ions corresponded to unique combinations of atoms for the neutral losses observed in low resolution mass spectra. Identification of the neutral losses simplified mass spectral interpretation by limiting the number of possible structures for the fragment ions. After inspecting library mass spectra for smaller molecules, isomeric structures were hypothesized with cyano and alkylcyano groups attached to tetralin. A literature search found that six of these isomers were consistent with an industrial polymer synthesis performed by the party that generated the waste. The proposed structures were confirmed by mass spectra and GC retention times for three of the isomers in a standard provided from the polymerization process now used.

Introduction. The Reich Farm Superfund Site near Tom's River, NJ, is a relatively flat, sandy area of approximately three acres, surrounded by commercial establishments and woodland. In 1971, an independent waste hauler leased the site and dumped drums con-taining solvents, still bottoms, and residues from organic chemical manufacturing. More than 100,000 people residing within three miles of the site are served by nearby wells. The NJ Department of Health and Senior Services reported that the incidence of cancer among children in the area was greater than expected. The NJ Department of Environmental Protection found traces of several isomeric compounds in a well that supplied 50,000 people.

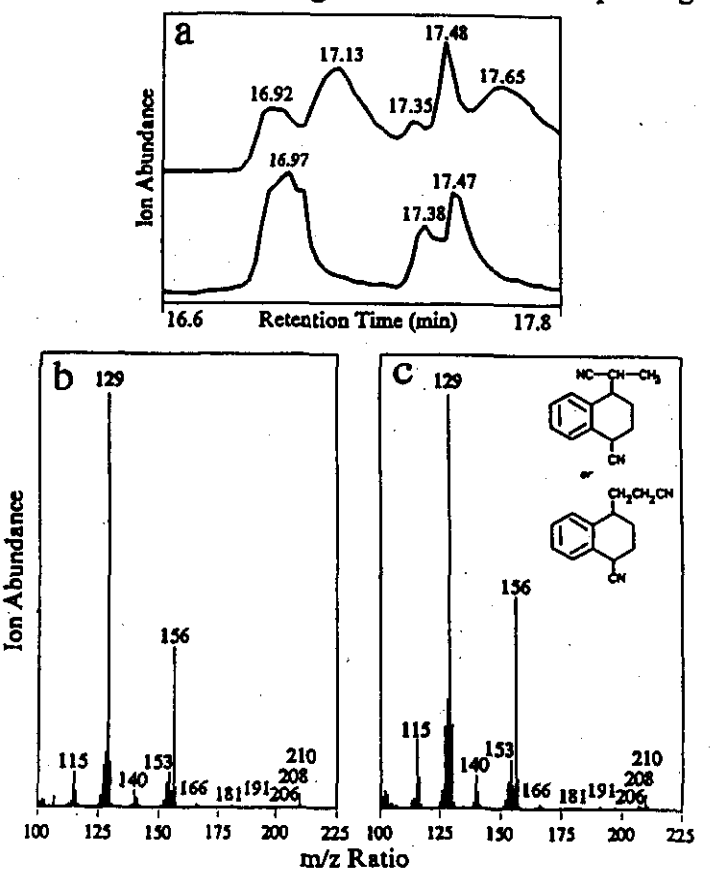

Figure 1. (a) m/z 129 ion chromatogram for well water extract (top) and standard (bottom); (b) mass spectrum of 4th isomer in the extract; and (c) mass spectrum of isomer in the standard with the same retention time.

Neither they nor other laboratories were able to determine the elemental composition of the isomers using conventional mass spectrometric techniques. A concentrated extract was sent to the U.S. EPA's Environmental Sciences Division (ESD) for analysis. FTIR and NMR studies were precluded by the low level of the analytes in the extract.

Industrial chemical syntheses create a myriad of waste compounds. Most of these compounds are not found in mass spectral libraries. When analyzing chemicals dumped at a Superfund site located within 100 miles of numerous industries that generate waste, one does not know *a priori* which elements are contained in the compounds. For industrially generated organic compounds, C, H, O, N, P, S, Si, F, Cl, and Br should all be considered.

Shown in Figure 1a is an ion chromatogram for m/z 129, the base peak in the mass spectra. The mass spectrum for one isomer is shown in Figure 1b. No library matches were found with the Wiley library (262,000 entries) or the NIST library (62,235 entries).

Mass Peak Profiling from Selected Ion Recording Data (MPPSIRD). MPPSIRD was developed by the ESD to determine the elemental compositions of environmental contaminants, a crucial step in their identification [1]. In Figure 2a are several ion chromatograms for m/z ratios that were monitored across a mass peak profile. The areas of the chromatographic peaks were among those plotted to provide the mass peak profile in Figure 2b. The exact mass was calculated as the weighted average of several points near the top of the profile. In Figure 2c are shown partial profiles plotted in the same way. Partial profiles were monitored by a single SIR descriptor to provide exact masses and abundances relative to the $M^{+\cdot}$ partial profile for the $(M+1)^{+\cdot}$ and $(M+2)^{+\cdot}$ partial profiles. SIR provided more than 100 times greater sensitivity than full scanning and 6 times faster cycle times at 20,000 resolution using a VG70-250SE double focusing mass spectrometer. Consequently, MPPSIRD provided exact masses and relative abundances of ions from major or trace components that reside in the mass spectrometer for short times. Data are routinely acquired at 20,000 resolution using capillary GC.

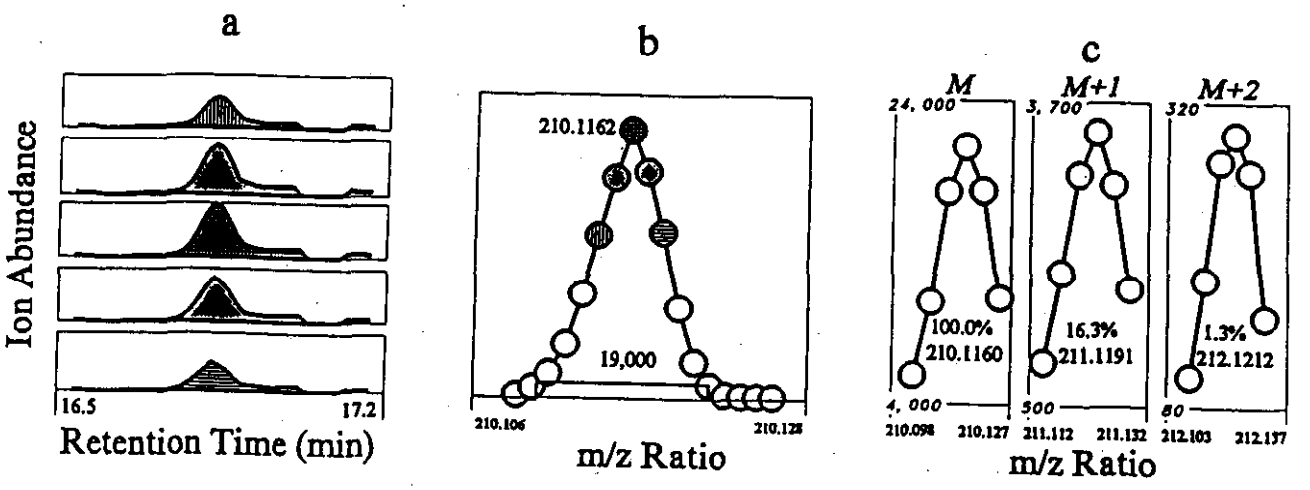

Figure 2. Ion chromatograms (a) for m/z ratios corresponding to the 5 shaded points in (b), a full mass peak profile. In (c) 3 partial profiles are shown.

Determining the Correct Composition Using a Profile Generation Model (PGM). A PGM calculated which elemental compositions were possible that contained C, H, O, N, P, S, Si, and F atoms for 210.1160 Da ± 2.5 ppm, the exact mass of the presumed molecular ion, determined at 20,000 resolution. Cl and Br were not considered, because the isotopic abundance patterns associated with these elements were not observed in the mass spectra. The PGM calculated the exact masses and relative abundances of $(M+1)^{+\cdot}$ and $(M+2)^{+\cdot}$ partial profiles for each composition and applied error limit criteria to the experimental data to reject all but the correct one [2]. Finally, the PGM printed a table similar to Table 1 listing the 5 possible compositions, the mass defects, and relative abundances with an "X" beside each criterion that was failed. The trace levels of the analytes necessitated use of less than 20,000 resolution to study the $(M+1)^{+\cdot}$ and $(M+2)^{+\cdot}$ profiles.

Table 1. Elemental compositions and quantities used to select the correct one

m/z 210.1160 ± 2.5 ppm

Resolution:			20,000	10,000	5,000	10,000		5,000	
#	RDB	Composition	M	M+1	M+2	%M+1	(Range)	%M+2	(Range)
1	1.0	$C_5H_{16}N_6PF$	.1158	.1177	.1190	7.2	(6.2- 8.4) X	0.3	(0.2- 0.3) X
2	0.0	$C_6H_{20}N_4P_2$	.1163	.1187	.1214	8.3	(7.4- 9.3) X	0.3	(0.2- 0.3) X
3	5.5	$C_9H_{13}N_5F$	.1155	.1181	.1204	11.3	(10.0-12.8) X	0.6	(0.5- 0.7) X
4	4.5	$C_{10}H_{17}N_3P$	.1160	.1190	.1217	12.0	(10.8-13.4) X	0.7	(0.6- 0.8) X
5	9.0	$C_{14}H_{14}N_2$	.1157	.1189	.1219	16.2	(14.7-17.7)	1.3	(1.1- 1.4)
Experimental:			.1160	.1187	.1208	16.3		1.3	

Rejecting Functional Groups Based on the Elemental Composition. The nominal mass of the molecular ion indicates whether it contains an even or odd number of N atoms, but provides no other information about the functional groups in a molecule. The elemental composition of the m/z 210 ion, $C_{14}H_{14}N_2^{+\cdot}$, determined from high mass resolution data excluded alcohols, aldehydes, ketones, ethers, esters, carboxylic acids, peroxides, nitro-, nitroso-, or other oxygen, phosphorous, sulfur, halogen, or metal containing compounds. When the formula, $C_{14}H_{14}N_2$ was searched rather than the molecular weight of 210 Da, the number of mass spectra selected was reduced from 1285 to 33 for the Wiley library and from 342 to 13 for the NIST library. Although the mass spectra were not in the library, these reductions illustrate that knowledge of the elemental composition greatly reduced the number of possible compounds.

Limiting the Isomers Possible with Exact Masses of Fragment Ions and Neutral Losses. Exact masses were determined at 10,000 resolution for the 10 fragment ions labeled in Figure 1b and listed in Table 2. The exact masses of the neutral losses listed in Table 3 were determined by subtracting the exact masses of each fragment ion from the exact mass of the presumed molecular ion. For each entry, the observed mass errors in both tables were acceptable for only 1 combination of atoms that contained the elements in the presumed molecular ion. One or two other possibilities were eliminated for 8 of the fragment ions and for the 8 corresponding neutral losses. The correlation of the neutral losses and the fragment ions was consistent with the assumption that $C_{14}H_{14}N_2^{+\cdot}$ was the molecular ion.

The exact masses of the fragment ions and neutral losses eliminated uncertainty about the number of N atoms in the fragments. For example, the composition of the ion corresponding to the base peak at m/z 129 resulted from loss of $C_4H_5N_2$ and not of C_5H_7N or of C_6H_9. $C_{10}H_9^+$, not $C_9H_7N^+$ or $C_8H_5N_2^+$, was consistent with the aromatic substructure shown in Figure 3 that originates from non-

nitrogen containing compounds such as indanes, indenes, and tetralin [3]. Hence, the nitrogen atoms were probably external to the rings. The nominal mass loss of 54 Da could correspond to C_4H_6, C_3H_4N, or $C_2H_2N_2$ leaving 2, 1, or 0 N atoms in the fragment ion. The exact mass of 54.0344 Da indicated loss of C_3H_4N with 2.5 rings-and-double bonds, corresponding to a radical cleavage, either as a composite loss of ($\bullet C_2H_3$ + HCN) or of the intact units, $\bullet CH_2CH_2CN$ or CH_3CHCN.

Table 2. Exact Masses for 10 Fragment Ions from the Isomers

Composition	Experimental Mass	Theoretical Mass	Error (mmu)	C, H & N Combinations with Same Nominal Mass			Errors (mmu)
$C_{14}H_{12}N_2$	208.1001	208.1000	+0.1				
$C_{14}H_{10}N_2$	206.0846	206.0844	+0.2				
$C_{13}H_7N_2$	191.0607	191.0609	-0.2	$C_{14}H_9N$		-12.8	
$C_{13}H_{11}N$	181.0887	181.0891	-0.4	$C_{12}H_9N_2$	$C_{14}H_{13}$	+12.5	-12.6
$C_{12}H_8N$	166.0661	166.0657	+0.4	$C_{11}H_6N_2$	$C_{13}H_{10}$	+13.0	-12.2
$C_{11}H_{10}N$	156.0816	156.0813	+0.3	$C_{10}H_8N_2$	$C_{12}H_{12}$	+12.9	-12.3
$C_{11}H_7N$	153.0578	153.0578	0.0	$C_{10}H_5N_2$	$C_{12}H_9$	+12.5	-12.6
$C_{10}H_6N$	140.0501	140.0500	+0.1	$C_9H_4N_2$	$C_{11}H_8$	+12.7	-12.5
$C_{10}H_9$	129.0707	129.0704	+0.3	$C_8H_5N_2$	C_9H_7N	+25.4	+12.9
C_9H_7	115.0548	115.0548	0.0	$C_7H_3N_2$	C_9H_5N	+25.2	+12.6

Table 3. Exact Masses of Neutral Losses Determined from Mass Differences Between the Molecular and Fragment Ions

Atoms	Experimental Mass	Theoretical Mass	Error (mmu)	C, H & N Combinations with Same Nominal Mass			Errors (mmu)
H_2	2.0159	2.0157	+0.2				
H_4	4.0314	4.0313	+0.1				
CH_7	19.0553	19.0548	+0.5		NH_5		+13.1
CH_5N	29.0273	29.0265	+0.8	C_2H_5	N_2H	-11.8	+13.3
C_2H_6N	44.0499	44.0500	-0.1	C_3H_8	CH_4N_2	-12.7	+12.5
C_3H_4N	54.0344	54.0344	0.0	C_4H_6	$C_2H_2N_2$	-12.6	+12.6
C_3H_7N	57.0582	57.0578	+0.4	C_4H_9	$C_2H_5N_2$	-12.2	+12.9
C_4H_8N	70.0659	70.0657	+0.2	C_5H_{10}	$C_3H_6N_2$	-12.4	+12.8
$C_4H_5N_2$	81.0453	81.0453	0.0	C_6H_9	C_5H_7N	-25.1	-12.5
$C_5H_7N_2$	95.0612	95.0609	+0.3	C_7H_{11}	C_6H_9N	-24.9	-12.3

The library mass spectrum in Figure 4 for tetralin shows sequential loss of several H atoms, similar to the losses observed for the m/z 156 ion in Figure 1b. The neutral loss masses in Table 3 verified that the difference between m/z 153 and m/z 156 was 3 H atoms. After a tetralin core was assumed, 9 rings-and-double bonds required that the 2 N atoms be present as cyano groups. The loss of C_3H_4N then corresponded to an alkylcyano group and the loss of CH_3N corresponded to loss of (2H + HCN). Knowledge of the correct neutral losses led quickly to a limited number of possible isomers, those which contained cyano and alkylcyano groups attached to a tetralin core. Organic chemical considerations of long-term stability in water and solubility were also consistent with this functional group hypothesis. Additional observations that supported this hypothesis will be presented in reference 4.

Searching the Literature for Industrial Processes that Produce Such Isomers. A search of the formula indices of Chemical Abstracts from 1920 to the present found hundreds of citations for compounds with the composition $C_{14}H_{14}N_2$. These were searched for industrial processes that could produce molecules with the expected structural features. Abstracts containing molecules shown in Figure 1c were located and the full papers were later reviewed. These compounds were produced by the copolymerization of styrene and acrylonitrile as 1:2 adducts, and had mass spectra very similar to those acquired for the isomers in the well water.

Establishing Responsibility for the Contaminants. Unknown to us at the time, the party who manufactured these chemicals admitted responsibility after we determined the elemental composition. The party later provided us an authentic standard obtained by distilling the adducts from styrene:acrylonitrile polymer that was prepared by another industrial synthesis. Three of the isomers observed in the standard had the same retention times as isomers in the well water extract and very similar mass spectra as illustrated by Figure 1. The possible structures of these isomers are shown in Figure 5.

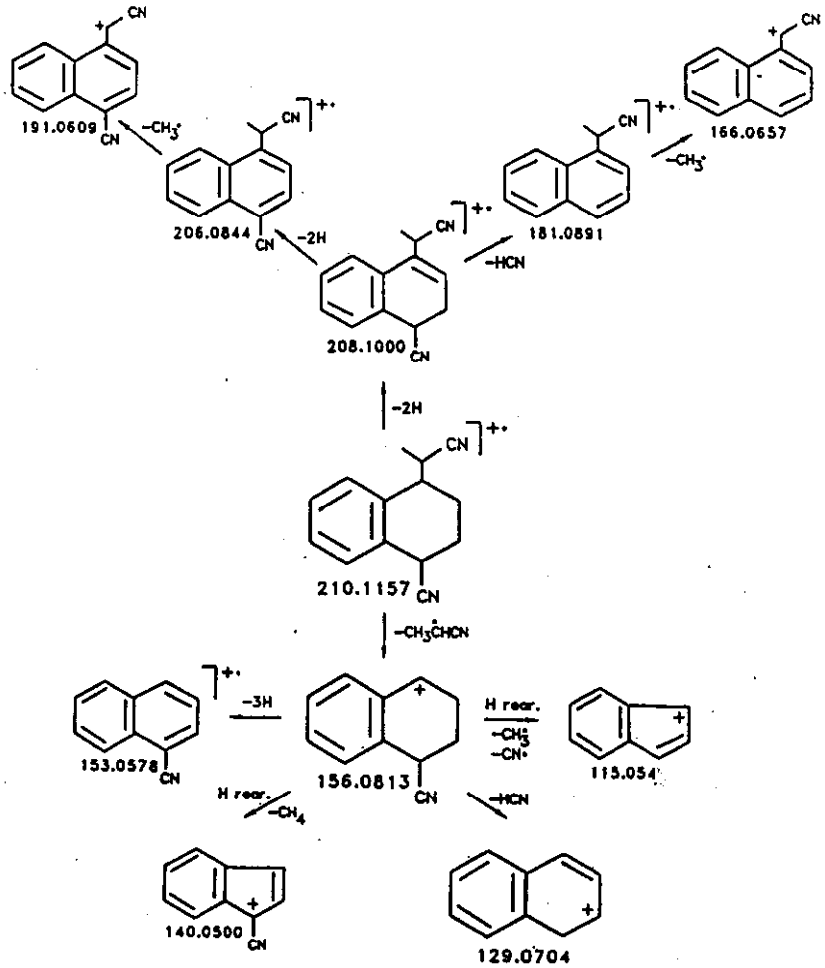

Figure 3. Plausible fragmentation scheme from an isomer with α-cyanoethyl and cyano groups on the saturated ring for the 10 fragment ions studied.

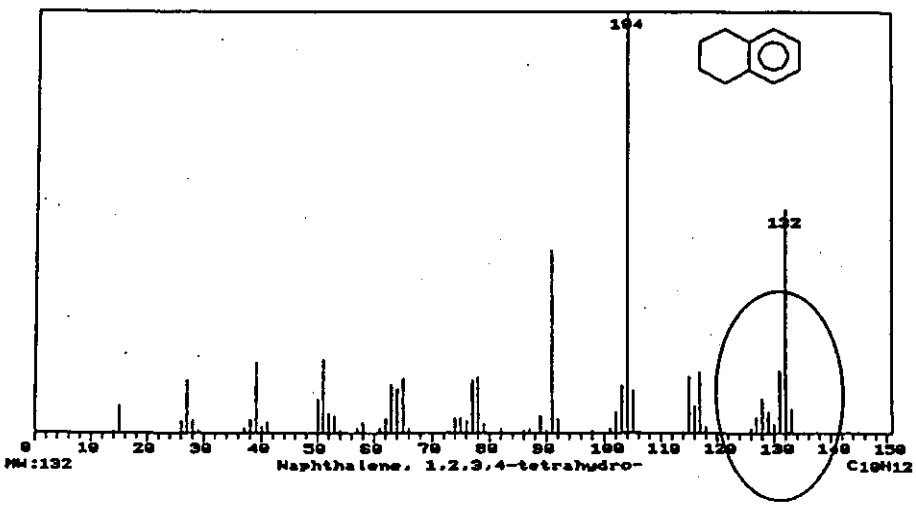

Figure 4. Mass spectrum of tetralin from the NIST library.

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Journal of Chromatography A, 674 (1994) 271–280

JOURNAL OF CHROMATOGRAPHY A

An improved high-performance liquid chromatography system for the analysis of basic dyes in forensic casework

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Abstract

Acrylic fibres are frequently encountered as physical evidence in casework in the Northern Ireland Forensic Science Laboratory. Separation and analysis of the basic dyes, used in the garment industry for the dyeing of acrylic fibres, was carried out by isocratic HPLC with a narrow bore column. The HPLC eluent used was a mixture of methanol and aqueous ammonium acetate solution (pH 9.76) in a ratio of 9:1. The sensitivity of the system is such that the dyes extracted from 0.2 cm of a black acrylic fibre could be detected using three injections on a single channel detector at wavelengths of 400, 500 and 600 nm (for yellow, red and blue dye components respectively).

1. Introduction

Textile fibres contain a number of dyes which may be mixed in different proportions to give varying shades and colours. Methods used by forensic scientists for the comparison of fibres are: (a) an identification of the fibre type by polarising and infrared spectroscopy and (b) examination of the dye components by comparison microscopy, microspectrophotometry and thin-layer chromatography (TLC) [1].

High-performance liquid chromatography (HPLC) systems have been described for the analysis of acid, disperse [2], direct and pigment dyes in forensic science. These include the analysis of fibres [3–7], lipstick smears [8,9], inks [10,11], illicit tablets [12], and counterfeit notes [13].

Acrylic fibres are one of the most frequently occurring fibre types encountered in casework in the Northern Ireland Forensic Science Laboratory. These are found particularly in terrorist incidents where masks and gloves have been used. Basic (cationic) dyes are commonly used in the dyeing of these acrylic fibres.

A few HPLC systems have previously been reported for the analysis of basic dyes. Individual [14,15] or a narrow range of basic dyes [16,17] can be analysed with these systems.

An HPLC method for basic dyes was described previously by this laboratory [18]. It consisted of gradient elution with mixtures of methanol and aqueous ammonium acetate solutions on a normal-phase silica column (160 × 4.5 mm I.D.) and was found to provide acceptable separation of twenty-one basic dyes. However, baseline drift was found to occur in the gradient system due to refractive index changes. This was most noticeable at visible wavelengths, when high sensitivity (0.0005 AUFS) was required for the analysis of the small quantities of extracted fibre dyes available in forensic science casework.

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JOURNAL OF
CHROMATOGRAPHY A

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Identification and determination of red dyes in confectionery by ion-interaction high-performance liquid chromatography

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Abstract

In this paper an ion-interaction HPLC method for the separation and determination of red dyes used to decorate home-made sweets is presented. In particular E122, E123 and E124 dyes are considered, with special interest for E123 (amaranth), prohibited by the US Food and Drug Administration in 1976 and by Italian legislation in 1977 and then again permitted (with restrictions) in the European Community in 1994, (94/36 EEC Directive). A RP-ODS stationary phase is used and an ion-interaction reagent (octylammonium phosphate) is added to the hydro–organic eluent [water–acetonitrile (30:70, v/v), pH=6.4]. Spectrophotometric detection at 520 nm was employed and detection levels below 12 μg/l for the three dyes were achieved. The method is applied to two commercial products, respectively produced in France and in Italy, and the results are discussed in the light of the recent EEC Directive concerning food dyes.

Keywords: Dyes; Amaranth

1. Introduction

In 1960, an Amendment to the Food, Drug and Cosmetic Act permitted colours in use at that time in the USA to be used provisionally until their safety had been established. In the Federal Register of September 23, 1976 (85), the US Food and Drug Administration (FDA) addressed all the colours that were then provisionally listed for food, drug or cosmetic uses [1,2]. Since then many colours have been removed from the provisional list and prohibited, among them, amaranth [3]. In 1977 Italian legislation, DM 21.03.77, [4] banned the use of amaranth (E123) in most food and beverages: the dye could only be used in caviar and caviar succedanea.

On the other hand the dye was still allowed in other European countries. In 1994 the European Community, in order to unify in the member countries the legislation about food dyes, formulated the directive 94/36 [5]. This reports a positive list of the dyes permitted, two lists of foods in which these colours respectively can or cannot be used and, for some of the dyes, the maximum permitted amount. In concern of the time of application, the directive states that all the EC countries had to conform within December 31, 1995 legislations, regulations and administrative provisions to prohibit commercialization and use of all non-conforming products. Art. 9 also states that all the products already on the market or already labelled can be commercialized up to the exhaustion of the stock in hand.

As it particularly concerns red colours, the positive list reports E120, E122, E123 and E124. Among

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JOURNAL OF CHROMATOGRAPHY A

Purification and identification of several sulphonated azo dyes using reversed-phase preparative high-performance liquid chromatography

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Received 28 April 1998; received in revised form 14 August 1998; accepted 17 August 1998

Abstract

Reversed-phase preparative HPLC has been successfully used to isolate several sulphonated azo dyes (Acid Red 1, Acid Red 8, Acid Red 106, Acid Violet 5, Chromotrope 2R, Reactive Orange 16 and Cibacron Brilliant Red 3B-A) from their impurities. The separations were achieved using mobile phases of methanol and ammonium acetate. The major components of these azo dyes were collected and then identified by electrospray mass spectrometry using the characteristic ions $[M-2Na]^{2-}$, $[M-2Na+H]^{-}$ and $[M-Na]^{-}$. The degree of purification, as measured by electrospray mass spectroscopy, was variable. © 1998 Elsevier Science B.V. All rights reserved.

Keywords: Preparative chromatography; Dyes; Azo dyes

1. Introduction

Azo dyes are characterized by the presence of one or more R-N=N-R groups, whose nitrogen atoms are linked to sp^2-hybridized carbon atoms. At least one of these carbon atoms belongs to an aromatic ring (usually a benzene or naphthalene derivative). Commercially, the dyes are synthesized by coupling the aromatic derivatives in an aqueous medium. Given the almost unlimited choice of starting materials, an extremely wide variety of azo dyes is possible. Of the commercial dyes listed in the Color Index (1971) [1], more than 2200 are azo dyes, which are used in the food, cosmetic, textile and printing industries.

Commercially prepared azo dyes are rarely pure initially, because of the presence of impurities in the reactants and the occurrence of side reactions during manufacture. Azo dyes used in the food and cosmetic industries must be purified because ingestion brings with it the possibility of biotransformation. The toxicity of the likely metabolites must be considered. The required purity is dye-specific and ranges from 85% for tartarazine to 96% for Citrus Red Number 2 [2]. Limits are also put on percentages of allowed insoluble matter, ether soluble matter, subsidiary dyes and intermediates. For azo dyes used in the textile and printing industries, where ingestion is less likely, purity is secondary to colour and there is no incentive for manufacturers to further purify these azo dye formulations. The ultimate goal of this project is to investigate the effect of azo dye structure on the ease of bioreduction by intestinal bacteria. Such a study requires that the test compounds be as free from contamination as possible, to allow accurate measurement of the kinetics of reduction. As the eight azo dyes used in this study had

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Method for the Analysis of Phloxine B, Uranine, and Related Xanthene Dyes in Soil Using Supercritical Fluid Extraction and High-Performance Liquid Chromatography

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The use of supercritical fluid (SF) carbon dioxide (CO_2) modified by organic solvents and inorganic salts or chelating reagents was investigated for the extraction of the xanthene dyes phloxine B and uranine from soil. Methanol (MeOH), *n*-butylamine (*n*-BA), and a chelating agent, ethylenediaminetetraacetic acid tetrasodium salt (Na_4EDTA), were the most effective modifiers of SF CO_2 for quantitative recoveries of phloxine B and uranine in soils with 10–20% moisture at 60 °C/476 atm and 60 °C/272 atm, respectively. At these supercritical fluid extraction (SFE) conditions, recoveries of related xanthene dyes (i.e., 2′,7′-dichlorofluorescein, 4,5,6,7-tetrachlorofluorescein, eosin Y lactone, erythrosin B, and rose bengal) fortified at 25 μg/g in Hawaiian soils ranged from 65 to 93%. Good separation of a mixture of these dyes was achieved by HPLC. A mixture of MeOH, *n*-BA, and sodium hexametaphosphate [$(NaPO_3)_6$] was effective for conventional solvent extraction of phloxine B and uranine from fortified soils. However, SFE was more selective and gave cleaner extracts. Recoveries were comparable to those by solvent extraction.

Keywords: *Phloxine B; uranine; xanthene dye; EDTA; SFE; HPLC; soil*

INTRODUCTION

Phloxine B and uranine are xanthene dyes widely used as coloring additives in drugs and cosmetics. Phloxine B has been used as a food-coloring material in Japan since 1973 (Ito et al., 1994). Demand for determining purity led to numerous studies on its analysis, purification, and synthesis (Gandin et al., 1982; Weisz et al., 1992; Pérez-Ruíz et al., 1994). Phloxine B and uranine also have fluorescent properties useful for biological staining (Lillie, 1969) and optical chemical sensing (Nakagama et al., 1990).

The insecticidal property of xanthene dyes was discovered when mortality, retardation of growth, and a decrease in fecundity of female insects were observed while the dyes were being used as biological markers in an insect ecological study (Yoho et al., 1973). Insect bioassays showed that dye-sensitized photooxidation caused mortality in house flies (Carpenter et al., 1984; Yoho et al., 1976) and other insects (Callaham et al., 1975; Fondren and Heitz, 1978). Recent studies showed that phloxine B and uranine effectively suppressed fruit flies (Liquido et al., 1995a,b). USEPA has approved an experimental use permit to conduct field experiments using these dyes in spray or bait formulations to control fruit flies on coffee in Hawaii, oranges in California, and grapefruit in Texas (USEPA, 1995). Currently, malathion is the chief insecticide to combat fruit flies in Hawaii, but malathion and its oxidative product malaoxon are also toxic to honeybees, lady beetles, and other natural enemies of the pests (Mulla et al., 1981). A more environmentally acceptable fruit fly control agent is needed.

Laboratory toxicity tests indicated that phloxine B and uranine were relatively benign to mammals, although phloxine B was reported to have low toxicity to fish (Tonogai et al., 1979). Assessing environmer effects of phloxine B, uranine, and breakdown produ require that reliable analytical methods be establisl to detect these chemicals in environmental sample available methods are intended solely for product determination in foods, drugs, and cosmetics (B 1995). These methods are inadequate for analysis these compounds in complex environmental matri like soil.

In recent years, supercritical fluid extraction (SI has emerged as a valuable technique for rapid a efficient isolation of pesticide residues from envir mental samples (Chester et al., 1994; Clement et 1995; Sherma, 1995). Carbon dioxide (CO_2) has be the supercritical fluid (SF) of choice, being nontoxic a less expensive and having relatively low critical te perature (32 °C) and pressure (72 atm). SF C however, being nonpolar does not efficiently extr polar or ionic organic species. Polar SFs such as wa or MeOH are possible alternatives for extracting po compounds (Capriel et al., 1986; Hawthorne et 1994). However, the required critical temperatures a pressures for water (374 °C/218 atm) and MeOH (2 °C/78.5 atm) are too extreme for routine applicatior

SFE of moderately polar analytes like diuron a atrazine from soil using SF CO_2 required the additi of polar solvent modifiers such as MeOH or etha (McNally et al., 1988). Good SFE recoveries of metl eugenol and labile analytes such as naled and cuel from soil were accomplished (Alcantara-Licudine et 1996). Rochete et al. (1993) reported good SFE recov ies of 2,4-dichlorophenoxyacetic acid (2,4-D) fror with low organic matter by addition of an ionic m and derivatization of the free acid to a methyl es before gas chromatography (GC) determination. Go recovery of 2,4-D was also achieved by *in situ* chemi derivatization and SFE with CO_2 (Hawthorne et 1992). 4-Nitrophenol was efficiently extracted from s by SFE and detected by GC or immunoassay (Wong al., 1991). In general, advances of SFE for highly pol

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Analysis of Dyes in Plastics using HPLC

Application Brief

Polymer/chemcial industry

Angelika Gratzfeld-Huesgen

Abstract

Dyes which are used for coloring plastics have to fulfill special requirements. They have to be heat resistant, resistant against UV-irradiation and weatherproof. In addition, they should show strong coloring power and high brilliance. The analyzed colors in this application brief are soluble in organic solvents but are practically insoluble in water. This is important if the colored plastics are to be used in food packaging materials or in toys. Some of these colors are also used as coloring agents for polyamide fibers and other engineering plastics. The following colors were analyzed:

- Solvent yellow 21
- Filamid violet RB
- Disperse yellow 54
- Solvent red
- Macrolex blue 3R
- Solvent blue 97

Name	Color Index	Structure
Solvent yellow 21		Monoazo 1:2 chromo complex
Filamid violet RB		Monoazo 1:2 chromo complex
Disperse yellow 54	47020	Chinophthalon
Solvent red 52	68210	Anthraquinone
Macrolex blue 3R		Anthraquinone
Solvent blue 97		Anthraquinone

Table 1
Chemical structure of dyes

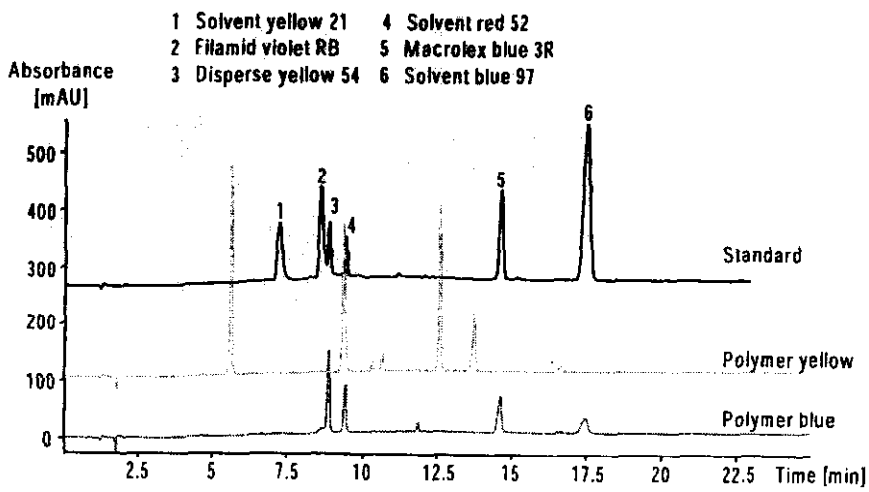

Figure 1
Standard chromatogram

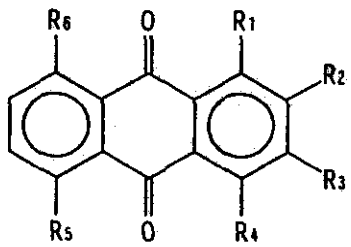

Figure 2
Chemical structure of dyes

Chromatographic conditions

Column:	125 × 3 mm BDS, 3 µm
Mobile Phase:	A = Water + 0.001 m Tetrabutylammoniumhydrogensulfate, pH = 3.0 with H_2SO_4, B = Acetonitrile
Gradient:	Start with 30 % B, to 98 % B in 10 min
Flow Rate:	0.5 ml/min
Injection Vol:	5 µl
Oven Temp:	40 °C
UV-Detector:	DAD, 280/20 nm Reference 900/50 nm 350/40 nm Reference 900/50 nm 465/40 nm Reference 900/50 nm 540/40 nm Reference 900/50 nm 600/40 nm Reference 900/50 nm

Sample preparation
Polymer samples were dissolved in Tetrahydrofurane and filtered after extraction with ultra-sonic bath for 30 min

HEWLETT® PACKARD

Sensitive Analysis of Synthetic Colors using HPLC and Diode-Array Detection at 190–950 nm

Application Note

Food and Flavors
Pharmaceutical
Chemical Processing

Angelika Gratzfeld-Hüsgen
Rainer Schuster

In this study synthetic dyes were analyzed using ion-pair, reversed phase chromatography on a special base-deactivated HPLC column. This separation mechanism was chosen to reduce tailing effects of highly polar colors. Detection was performed using a new design of diode-array detector based on two lamps — a deuterium lamp and a tungsten lamp. This ensured highest light output at 190 - 950 nm, which resulted in lowest detection limits over the entire wavelength range. It was possible to analyze blue, black or green colors in the low ng range at their absorption maxima of 600 - 700 nm. Complete spectra, feasible from 190 - 950 nm, facilitated identification with an automated library search.

Peer Reviewed

UV-Visible System for Identifying Dye Mixtures in the Environment

By *Anne E. Okafor, Chris W. Brown*, and *Martin J. Bide*, University of Rhode Island, Kingston

UV-visible spectroscopy is a powerful tool for quantitative analysis and can detect very low concentrations of analytes. The difficulty encountered with the UV-visible region of the electromagnetic spectrum in compound identification has been in visually recognizing the pattern of these compounds. This is due mainly to the presence of broad and highly overlapping bands. The problem is further compounded with dye samples that have very similar structures and similar UV-visible spectral features. Visual recognition of the pattern is almost impossible.

ABSTRACT

Mixtures of food and fabric dyes in deionized, stream, river, and sea water have been identified using a novel method to correlate UV-visible spectra with library spectra of pure dyes in deionized water. Two-, three-, and five-component mixtures of dyes were successfully identified using the Mix-Match algorithm. This algorithm performs principal component regression (PCR) on an entire library of spectra of pure components treating each of the spectra as a standard mixture having a unit concentration for that component. Approximate concentrations are obtained by projecting the scores of an unknown spectrum onto the principal component basis set and multiplying by the regression coefficients. An adaptive filter is then used to remove all similarities of the first target component from the mixture and the library spectra. PCR is performed on the modified spectra and used to identify the second target component. This process is repeated until the residuals approach zero.

A total of 107 mixtures of food and fabric dyes were searched in a library of 48 pure dyes. The predictions were correct for 100% of deionized water samples, 92% of stream water samples, 89% of river water samples, and 74% of ocean water samples.

KEY TERMS

Acid Dyes
Environmental Media
Mixture Analysis
Principal Component Regression (PCR)
UV-Visible Spectroscopy

Another important problem with dye mixtures in various media is that spectra change in some media due to pH, irradiation, temperature, aging, and ionic strength. These factors can lead to erroneous identification. Dyes are of environmental interest due to their possible health effects and to the fact that they cannot be effectively degraded in waste treatment plants.[1] Dyes are popularly thought to be mutagenic and carcinogenic. While few if any have those properties, correct identification of dyes present in the environment can provide accurate responses to those concerns. Dyes in the environment can also act as fingerprint or tracer compounds and identify their source and thus that of other pollutants.

Previously, it was demonstrated that UV-visible spectra of organic molecules contained enough information so that they can be used for identification and library searches despite all the overlapping and clustered bands.[2] Later, a novel search algorithm, "Mix-Match," was applied to UV-visible spectra for identifying the components in spectra of mixtures.[3] This algorithm, which is based on principal component analysis (PCA), is capable of searching spectra of pure compounds, mixtures of unknowns in medium-to-large size spectral libraries.[4,5]

Earlier methods used for spectral interpretation include computer aided spectral mapping, pattern recognition, factor analysis, cluster analysis, and artificial intelligence.[6-12] Principal component analysis (PCA) and its use for searching spectral libraries was first introduced by Isenhour and coworkers.[13-15] PCA is capable of analyzing large spectral libraries to remove redundant or dependent values leaving only significant information.[16] The concept of searching spectral libraries with spectra of unknown mixtures was first proposed by Nyden and coworkers.[17,18] The PCA and mixture methods were combined and greatly modified by Brown and coworkers.[3,5,19-21] By generating an orthonormal reference library using PCA, spectral dependencies can be eliminated and unknown mixtures can be correctly identified.

The algorithm assumes that a mixture spectrum behaves like a linear combination of pure spectra. Full treatment of this method has been given elsewhere.[3,19,21] The final results are given in terms of a pseudo concentration or composition index. Composition index values range from 0.0 to 1.0 with the highest value corresponding to the top hit. After the first target compound is identified, an adaptive filter is used to make the original reference spectra and the mixture spectrum orthogonal to the target spectrum. A new orthogonal spectral library is generated by PCA and is used to identify the next target component. This process is repeated until the residuals approach zero. Recently, the method was used to successfully identify textiles and dyes on the textiles from their near-IR spectra using a spectral library of pure dyes and textiles.[20]

The method has been used herein to search 107 mixtures of dyes in various environmental media from a UV-visible spectral library of 48 pure dyes dissolved in deionized water. The results demonstrated that dye samples can be successfully identified irrespective of the media in which they were dissolved. Previous knowledge of the sample's composition is unimportant. Despite the fact that some of these dyes show structure similarities, close absorption peaks, and overlapping bands in the UV-visible regions, this algorithm can identify the dyes.

Characterization of Dyes in Environmental Samples by Thermospray High-Performance Liquid Chromatography/Mass Spectrometry

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The use of thermospray HPLC/MS to characterize azo, diazo, and anthraquinone dyes in wastewater and gasoline is demonstrated. Thermospray analysis of these dyes primarily produced $[M + H]^+$ ions with few fragments. "Filament on" operation of the interface aided in the production of fragment ions for the azo dyes analyzed. The commercial diazo and anthraquinone dyes analyzed by HPLC/MS proved to be very complex mixtures of nearly 40 alkyl-substituted dye components, making monitoring and identification of a particular dyestuff very difficult. Thermospray HPLC/MS methods are demonstrated to detect dyes down to 10 ppt (parts per trillion) in wastewater, 100 ppb in soil, and 1 ppm in gasoline. The "filament on" operation of the thermospray interface provided additional fragment ions, aiding the structural elucidation of these dyes.

Dyes play a significant role in the coloration products consumed or used in the United States. They are used in products such as textiles, paper, leather, and foodstuffs. Also, dyes are required by law to be in leaded gasoline to indicate the presence of lead. In addition, unleaded gasoline can contain dyes to identify gasoline manufacturers and to trace a seepage into soil or groundwater.

Wastes generated from coloration processes are of environmental interest due to their possible health effects and the inability of waste treatment processes to remove the dyestuffs. The long-term effects of dyes and their degradation products are unknown. Possible mutagenic and carcinogenic effects of dyes and their byproducts are currently being investigated (*1*).

Waste treatment processes typically do not remove most of the dyes present in wastewater. Biological treatment of the waste only removed 30–68% of the color (*2, 3*), indicating the resistance of dyes to current treatment techniques.

Clearly, analytical methods must be developed to identify and quantitate these dyes to monitor environmental contamination. However, analytical procedures for the determination of dyes in complex mixtures at parts-per-million levels or less, are limited. Gas chromatography (GC) or GC/mass spectrometry (GC/MS) methods cannot be employed due to dye involatility. High-performance liquid chromatography (HPLC) methods proved successful for some dyes at levels less than 1 ppm (*4*) but conventional detectors lack specificity for dye identification. Techniques such as fast atom bombardment mass spectrometry (FAB-MS), field desorption MS, and secondary ion MS proved useful in the identification of a wide variety of dyes (*5–7*). Yet, these techniques lack on-line chromatographic separation necessary for identification and quantitation of a complex mixture containing parts per million or less quantities of dyes. However, HPLC methods are suitable for separations of involatile dyes and MS methods provide specific and sensitive means of detection.

The thermospray (TSP) HPLC/MS interface is well suited for combined HPLC/MS since it can handle high quantities of aqueous solvent without shunting a major portion of the effluent, and the ionization process is soft, usually resulting in molecular ion formation (*8–14*). TSP HPLC/MS analysis has been used to monitor numerous compounds of environmental importance, including pesticides, herbicides, dyes, and nitroso compounds (*15–19*). This paper describes the use of TSP HPLC/MS for identification and quantitation of azo, diazo, and anthraquinone dyes in water, soil, and gasoline at the low parts-per-million levels.

EXPERIMENTAL SECTION

TSP HPLC/MS. Details of the TSP HPLC/MS configuration and operation were given earlier (*15*). The HPLC separation of the azo dyes was performed on a 25 cm × 4.6 mm Zorbax C_{18} column (Du Pont Instruments), Wilmington, DE) at a flow rate of 1.2 mL/min. The separation was accomplished by using a gradient from 70% methanol in water to 90% methanol in water (0.1 M ammonium acetate) in 15 min and holding at the final conditions for 10 min. The diazo and anthraquinone dyes were separated by a 25 cm × 4.6 mm Zorbax TMS column (Du Pont) at 1.2 mL/min. The gradient employed went from 50% methanol in water (0.1 M ammonium acetate) to 90% methanol in water in 15 min and was held at the final conditions for 10 min. Some of the diazo dyes were separated on a 25 cm × 4.6 mm Zorbax C_{18} column (Du Pont) using 100% methanol at 1.0 mL/min. In this separation scheme 0.4 mL/min of 0.3 M aqueous ammonium acetate was added coaxially postcolumn for TSP ionization (*15*). All HPLC/UV chromatograms were recorded at 254 nm. The determination of the percentages of each dye component in a sample by HPLC/MS was based upon peak area measurement on the $[M + H]^+$ ion assuming a response factor of 1 for all components.

"Filament on" TSP HPLC/MS. The majority of the operating conditions for the "filament on" experiments were the same as those reported for TSP HPLC/MS. The filament located at the tip of the thermospray vaporizer was operated with an electron energy of 200 eV and a emission current of 0.05 mA. The TSP jet temperatures optimized lower (near 120 °C for negative ion detection, 180 °C for positive ion detection) but the vaporizer operated at the same temperature. The HPLC separation was performed at a reduced flow rate of 0.3 mL/min using a 25 cm × 2.0 mm C_{18} column and mobile phase conditions reported earlier. The 0.3 mL/min flow rate was employed to lengthen filament lifetime. At this flow rate the filament could last 1 to 2 days.

Dye Samples and Solvents. The dyes were obtained from industrial sources or from Aldrich Chemical Co. (Milwaukee, WI). The stated dye content for the standards ranged from 40% to 90%. Standard solutions were prepared in HPLC grade methanol (Fisher Scientific, Fairlawn, NJ) except for the diazo and anthraquinone dyes which were dissolved in HPLC grade toluene (Fisher Scientific). HPLC grade solvents (Fisher Scientific) were used in this study. The solvents were filtered through a 0.45-μm filter to remove dissolved air and particles which could hinder TSP operation.

Sample Preparation. Gasoline was spiked with 1 ppm of Diazo Red dye to simulate typical dye concentrations found in commercial gas. Twenty milliliters of the gas was eluted thro

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ATTACHMENT II

IDENTIFICATION OF DYES

ATTACHMENT II – IDENTIFICATION OF DYES

The next step was to try to identify dyes that could be potentially present at SWMU 2. The first target compound list consisted of dyes that were identified in the RFI report (USACE, March 1991) and residual dye products archived in the laboratory from past process use.

The Crane Laboratory tried to obtain standards for dye compounds identified in the RFI report. Some dyes were no longer available from either the manufacturer or chemical supply companies. These dyes were eliminated from the target compound list due to lack of a certified standard or known source.

Next, the Crane Laboratory attempted to identify as many of the residual dye products present in the laboratory as possible through literature searches of label information and analysis of the dye products. Many of the dye products could not be identified due to the ages of the dyes and lack of labeling information. Containers for residual dyes known to be used in Base processes were not labeled with a name, Chemical Abstracts Service (CAS) number or Colour Index (CI) number. A range of analytical techniques that are well-established as suitable for identifying and quantifying organic compounds were used; however, they were unsuccessful (See Attachment 1). Those techniques included nuclear magnetic resonance (NMR) and mass spectrometry (MS). An ultraviolet-visible (UV/Vis) spectral library of the archived dyes was developed. Actual spectra of these archived dyes are presented in this attachment.

Site-specific samples taken from the trenches during the capping operation were analyzed. The samples were visibly contaminated with dye compounds. Dyes were detected in site-specific soil samples which could not be matched to the dyes listed in the RFI report or the archived residual dye products. These constituents were categorized as dyes because of their absorbance wavelengths and spectral patterns. These unidentified dyes were also added to the spectral library with labels indicating their status as unidentified materials. Actual spectra of these unidentified dyes are presented in Attachment VII.

After an exhaustive attempt to identify dyes potentially present at the site, the final target compound list included ten aqueous soluble dyes and 14 organic soluble dyes. This comprehensive list of dyes is presented in the standard operating procedure (SOP) located in Attachment XII.

As discussed in the Pre-QAPP meeting (March 9, 2000) for this project, some dyes or dye substances that could potentially be present at the site are not included in the final method for the following reason(s):

No standards could be obtained for them

or

Identification of the dyes or dye substances could not be obtained.

Unknown dyes will be shown as present via a spectral match with the library spectra generated from the analysis of archived residual dye products and site-specific samples.

Color	Vat Yellow 4
Code	Acid Blue 1
	Acid Orange 10
	Acid Yellow 23
	Acid Blue 26
	Acid Blue 34
	Uranine

IR #	ESB Lable	Identity (Ref = CI)	AKA	CI #	CAS#	Wavemax	Type
D0005	3,4,8,9-Dibenzypyrine-10-quinone, MIL-D-50029C, Vat Yellow 4	Vat Yellow 4		59100	128-66-5		Anthraquinone
D0006	Accramine Hydrochloride						
D0007	Acid Blue 1 (3 each) (4/18/98, BB)	Acid Blue 1	Food Blue 3	42045	129-17-9		Triphenylmethane
D0009	Acid Orange 10	Acid Orange 10		16230	1936-15-8		Monoazo
D0069	Acid Orange 10 (McAllister)	Acid Orange 10		16230	1936-15-8		Monoazo
D0008	Acid Yellow 23 (3 each)	Acid Yellow 23			1934-21-0	426	Monoazo
D0016	Blue Dye MIL-D-16627 (1) Old CI 712	Acid Blue 1	Alphazurine 2G	42045	129-17-9		Triphenylmethane
D0067	Blue Dye MIL-D-16627 (5) Blue:	Acid Blue 26	Alphazurine B	N/A			Triphenylmethane
		Acid Blue 34	Acid Fast Violet BG	42561			Triphenylmethane
	Green:	Acid Yellow 23			1934-21-0	426	Monoazo
		Acid Blue 1	Alphazurine 2G	42045	129-17-9		Triphenylmethane
D0020	Calcozine Rhodamine BXP						
D0021							
D0024	Dye Marker. Green Uranine. Contract N19514446, Lot 9721	Uranine		45350		491	
D0025	FEAA, Lot 22242583						
D0026	Green 6820 00 625 3503						
D0027	Green Dye, (1) Acid Yellow 23, (2) Acid Blue 1	Acid Yellow 23			1934-21-0	426	Monoazo
		Acid Blue 1	Alphazurine 2G	42045	129-17-9		Triphenylmethane
D0071	Green - Acid Yellow 23, Acid Blue 1, 3-4-96 (large bottle)	Acid Yellow 23			1934-21-0	426	Monoazo
		Acid Blue 1	Alphazurine 2G	42045	129-17-9		Triphenylmethane
D0058	MIL-D-16627 Dye (Blue Color)	Acid Blue 26	Alphazurine B	N/A			Triphenylmethane
		Acid Blue 34	Acid Fast Violet BG	42561			Triphenylmethane
D0059	MIL-D-16627 Std Dye, Red (light red) (L)						
		Acid Yellow 23			1934-21-0	426	Monoazo
D0060	MIL-D-16627 Dye 146 Blue:	Acid Blue 26	Alphazurine B	N/A			Triphenylmethane
		Acid Blue 34	Acid Fast Violet BG	42561			Triphenylmethane
	Green:	Acid Yellow 23			1934-21-0	426	Monoazo
		Acid Blue 1	Alphazurine 2G	42045	129-17-9		Triphenylmethane
	Red:						
		Acid Yellow 23			1934-21-0	426	Monoazo
	Orange:	Acid Orange 10		16230	1936-15-8		Monoazo
D0061	MIL-D-16627, Red Dye						
		Acid Yellow 23			1934-21-0	426	Monoazo
D0062	MIL-D-16627, Red, ?6820-624OR8-(3 or 2)50(8 or 3)						
		Acid Yellow 23			1934-21-0	426	Monoazo

D0063	MIL-D-16627 Std Dye, 3 pkgs 10 g each Blue:	Acid Blue 26	Alphazurine B	N/A			Triphenylmethane
		Acid Blue 34	Acid Fast Violet BGL	42561			Triphenylmethane
	Green:	Acid Yellow 23	Yellow 4G	19140	1934-21-0	42	Monoazo
		Acid Blue 1	Alphazurine 2G	42045	129-17-9		Triphenylmethane
	Red:						
		Acid Yellow 23	Yellow 4G	19140	1934-21-0	42	Monoazo
	Orange:	Acid Orange 10	Yellow Orange 2G	16230	1936-15-8		Monoazo
D0064	MIL-D-16627 Std Dye, Red (dark red color)						
		Acid Yellow 23	Yellow 4G	19140	1934-21-0	42	Monoazo
D0065	Orange Dye	Probably is Acid	Orange 10 (No Proof	AS	OF	YET)	
D0033	Orange Dye, MIL-D-16627(3), OrangeC, Acid Orange 10, CI#16230	Acid Orange 10	Yellow Orange 2G	16230	1936-15-8		Monoazo
D0070	Orange Dye, MIL-D-16627 Glass Jar	Acid Orange 10	Yellow Orange 2G	16230	1936-15-8		Monoazo
D0034	Para M LA-6-225						
D0035	Pyrazole Anthrone LA-6-228						
D0037	R1187 Violet Dye, DuPont, RDA 435						
D0038	R5054 Benzanthranone RDA 451 Composite						
D0039	Red 6820 00 624 3503						
D0040	Red Dye (McAllister)						
D0041							
D0068	Red Dye, MIL-D-16627 (2)						
		Acid Yellow 23	Yellow 4G	19140	1934-21-0	42	Monoazo
D0043							
D0049	Smoke Yellow I is anthrarufin dry, LA-6-193						
D0055	Violet Dye PA2491, 74.52% Pure, 0.8% VOC @ 70 deg C						

Color		Solvent Yellow 3
Code	1,4-Diamino-2,3-dihydroanthraquinone	Pigment Yellow 23
	Anthracene	
	Disperse Red 9	Vat Yellow 4
		Solvent Yellow 33
	Disperse Blue 14	

File	ESB Lable	Identity (Ref=CI)	AKA	CI#	CAS#	Wavemax	Type
D0001	1,8 Diphenxon Quinon Dry Grd LA-6-209						
D0002	1,4-Diamino-2,3-dihydroanthraquinone, MIL-D-3868		N/A	N/A	[illegible]		Anthraquinone
D0003	1,4-Dimethyl Amino Anthraquinone, MIL-D-21854, Disperse Blue 14	Disperse Blue 14		61500	2475-44-7	644	
D0010	Anthracene, 480 Lot # 63 (Benzene Soluable CRC)	N/A	N/A	N/A	120-12-7		
D0011	Azosol Brillant Yellow GGF Dry, LA-6-223						
D0012	Baghouse Test Dye Fluorscein						
D0013	Benzan Crude B Dry Grd LA-6-207						
D0072	Benzan Crude B, Dry Grd LA-6-207 (large bottle)						
D0014	Benzan Purf Dgrd LA-6-208						
D0018	Brombenzan Grd LA-6-224						
D0019	Calco Oil Scarlet, 69493						
D0023	DuPont Oil Yellow C-1904	Solvent Yellow 3		11160B	97-56-3		
D0028	Helio Fast Yellow 6 GL, LA 6-192	Pigment Yellow 23		60520			
D0029	HVT-Yellow-GK is Indanthrene Yellow GKA Powder, LA-6-191						
D0030	Ind Gold Yel GKAC, LA-6-211						
D0031	Oil Scarlet 1370, Peerless Color						
D0032	[illegible]			[illegible]	[illegible]		
D0036	R1184 Yellow Dye RDA 446, 744-443-505						
D0042	Red Dye, Disperse 9, MIL-D-003284	Disperse Red 9		60505	82-38-2		
D0045	Reference Standard Sample Dye, Disperse 9 99%, MIL-D-3284A, Para 6.6	Disperse Red 9		60505	82-38-2		
D0046	Reference Standard Sample Dye, Disperse 9 99%, MIL-D-3718A, Para 6.5	Disperse Red 9		60505	82-38-2		
D0047	Smoke Yellow B-10						
D0048	Smoke Yellow B-11						
D0050	Solvent Yellow 33 Dye	Solvent Yellow 33		47000	8003-22-3		
D0051	Stearated Yellow Chrome, From MK-12 Marine Location Marker						
D0052	[illegible]			[illegible]	[illegible]		
D0053	[illegible]			[illegible]	[illegible]		
D0054	Transketon Dry, LA-6-227						
D0056	Yellow Dye, MIL-D-50029	Vat Yellow 4		59100	128-66-5		Anthraquinone
D0057	Z4Cr2O7 Pigment (HNO3 Soluable)						

June 16, 1999

RE: DYE PROJECT

Karen,

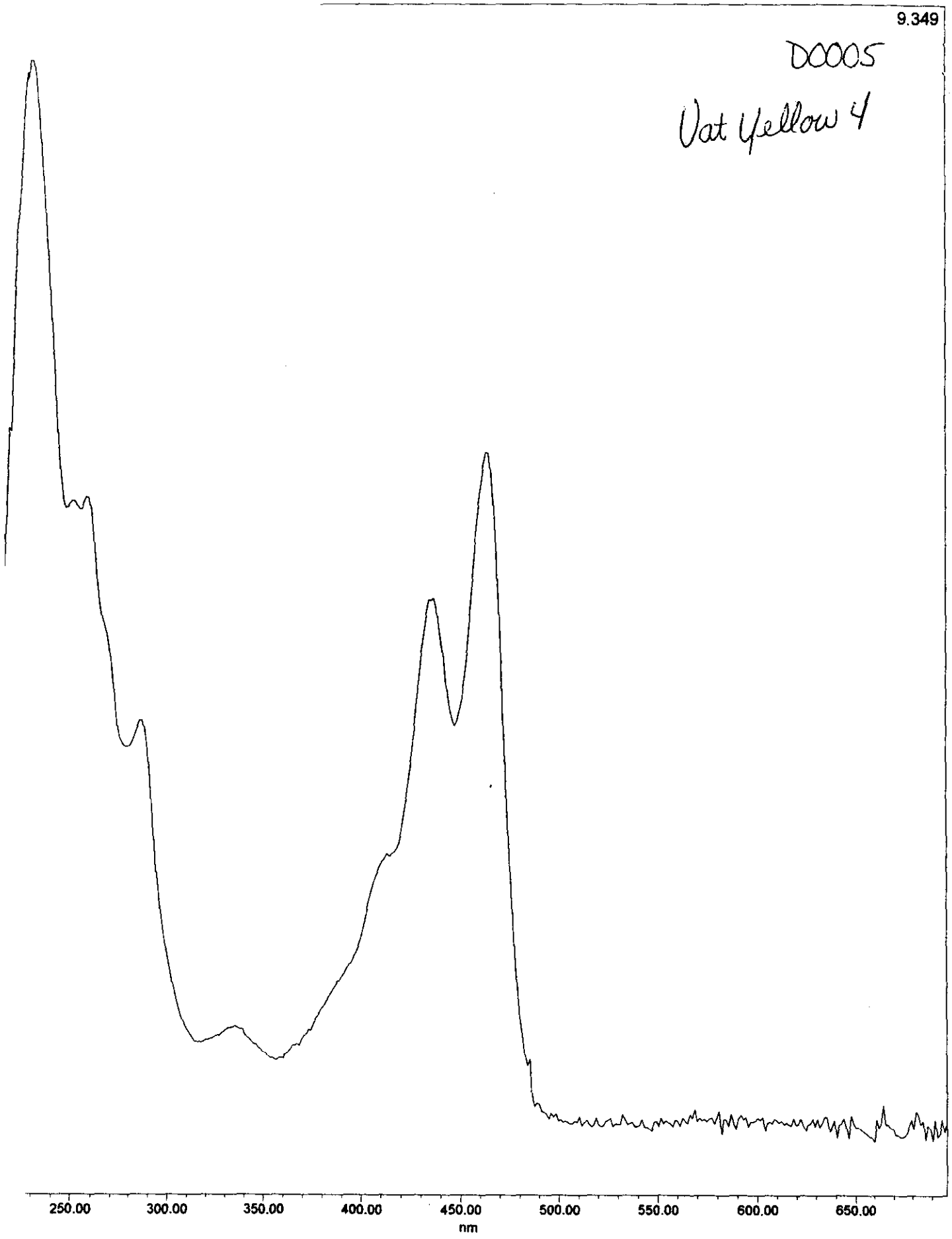
Enclosed is the beginning of the information I am going to send you about what the lab has done so far. This information was gather in the initial evaluation of the dyes in our possession. Analytical conditions are as follows:

Instrumentation:
Waters 996 Photodiode array detector (scan 210-700 nm)
Waters 600 pump
Column: Alltech C8, 4.6 x 250 mm
Flow: 1.0 mL/min
Mobile Phase: 60:40 Acetonitrile: 20 mM KH_2PO_4 @ pH≈3.4

I put dyes that we determined as identical next to each other and labeled the lead chromatogram with the identification, CI #, and CAS #. I didn't staple things together in case you wanted to compare spectra. At the very end are the unknowns without any duplicates. Please note that Sudan Scarlet 6G (D0053) was initially identified as Solvent Orange 7 after a literature search. After comparing the spectra to other dyes identified as Solvent Orange 7, it is obviously not the same thing. I do not know if you ever got the updated list with that error corrected.

More to come.

Jacquelyn Karty
NSWC Crane


9.349
D0005
Vat Yellow 4
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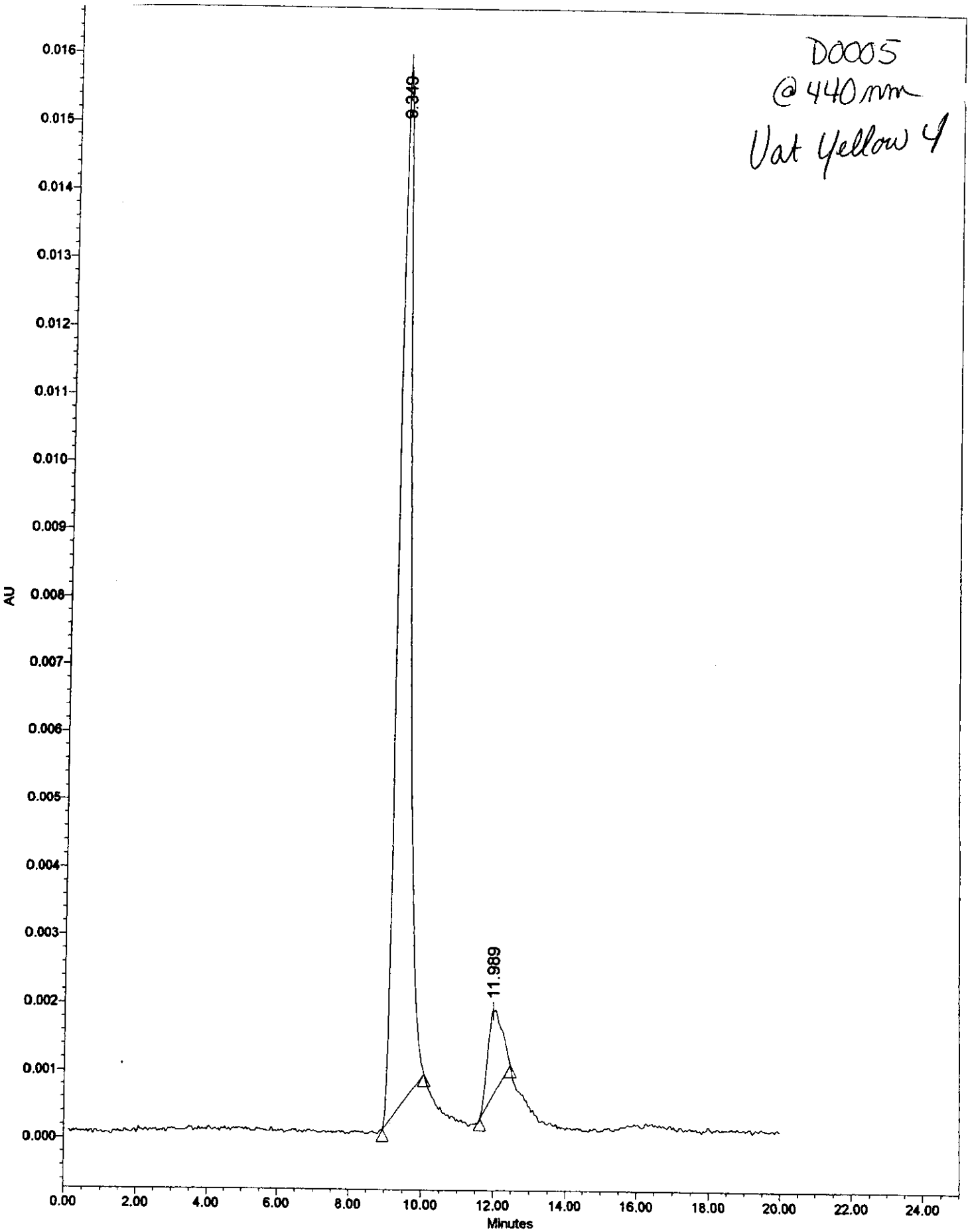
500.00
550.00
600.00
650.00
nm


D0005
@ 440 nm
Vat Yellow 4
9.349
11.989
AU
0.016
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0.011
0.010
0.009
0.008
0.007
0.006
0.005
0.004
0.003
0.002
0.001
0.000
0.00
2.00
4.00
6.00
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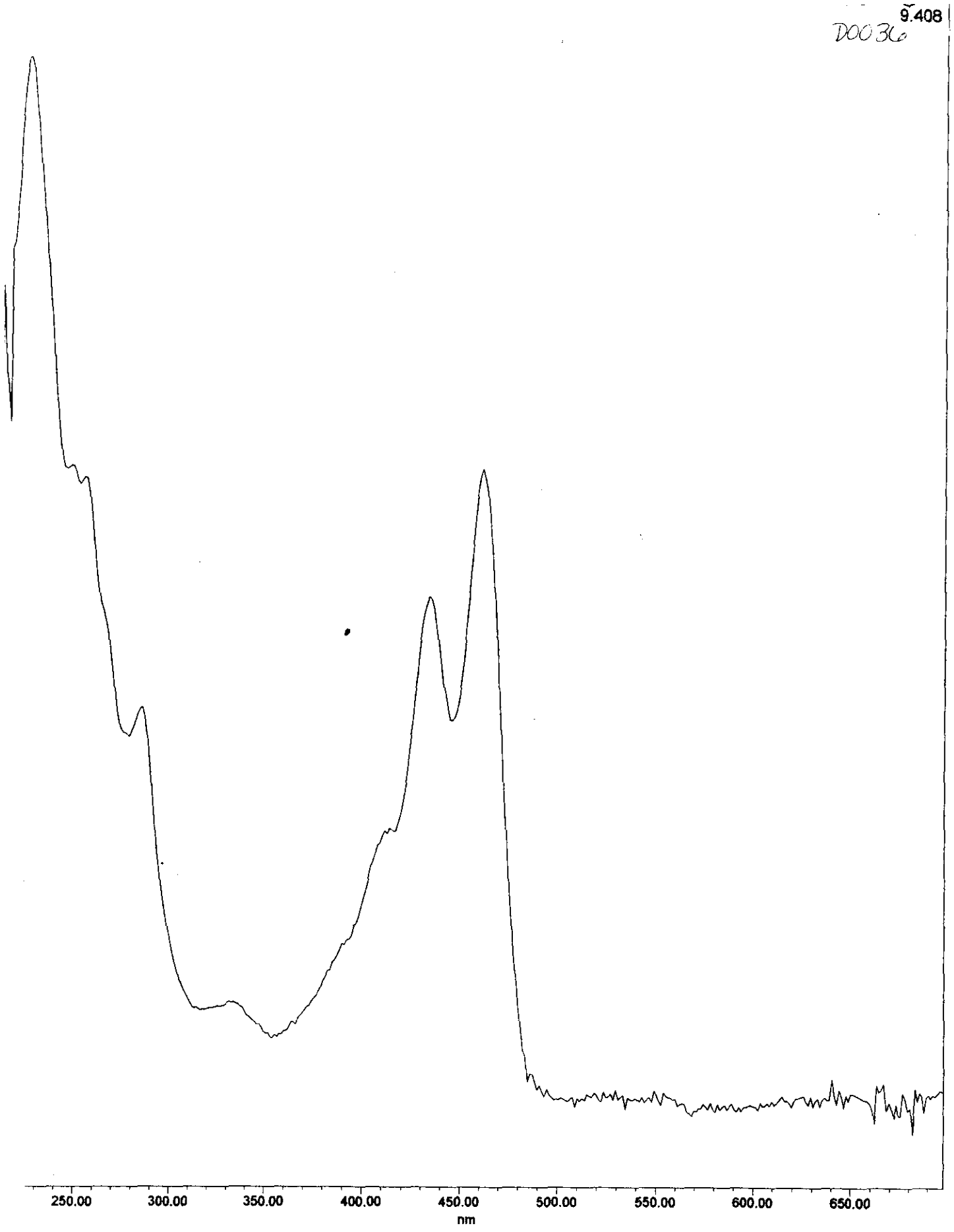
12.00
14.00
16.00
18.00
20.00
22.00
24.00
Minutes


9.408
D0036
250.00
300.00
350.00
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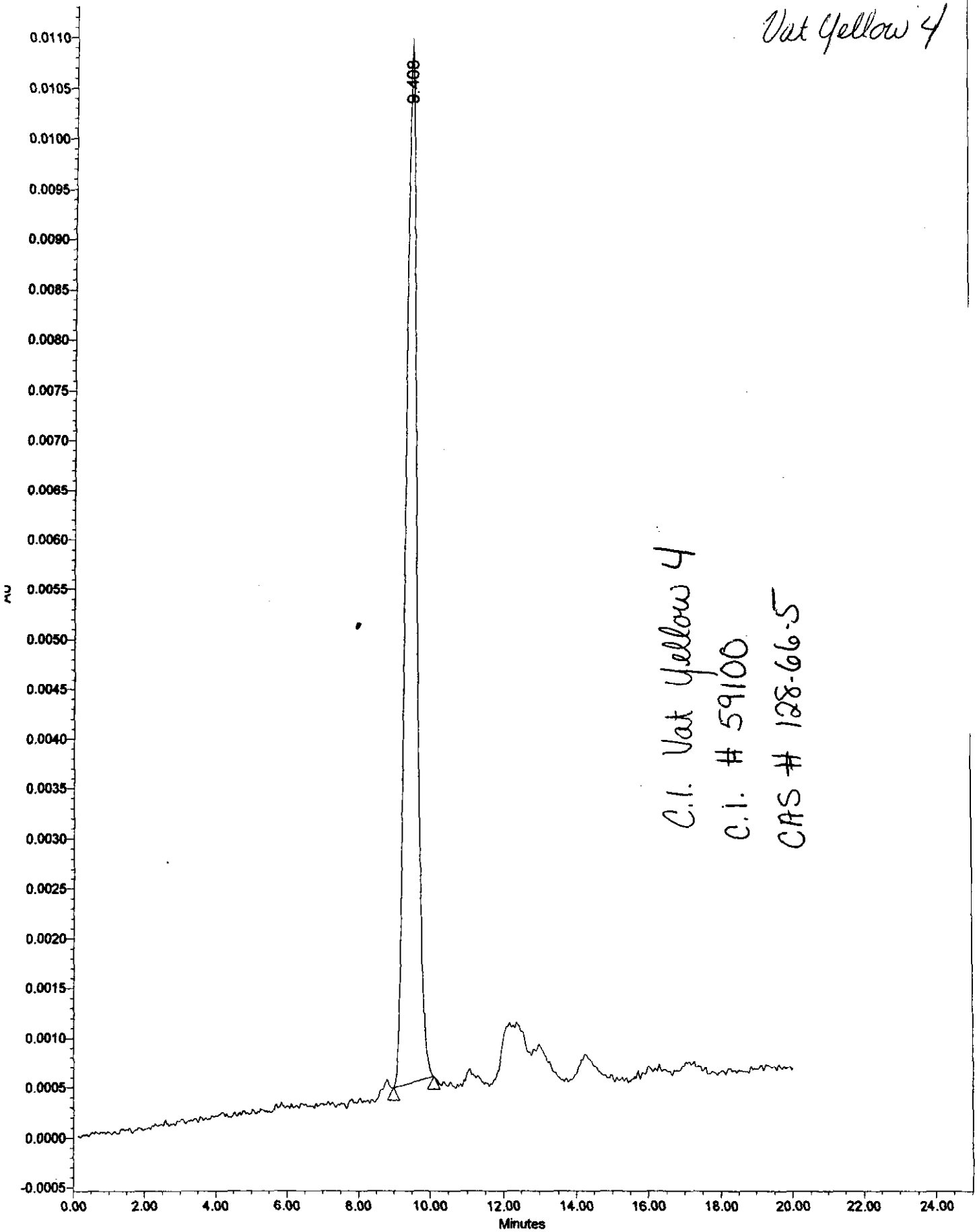
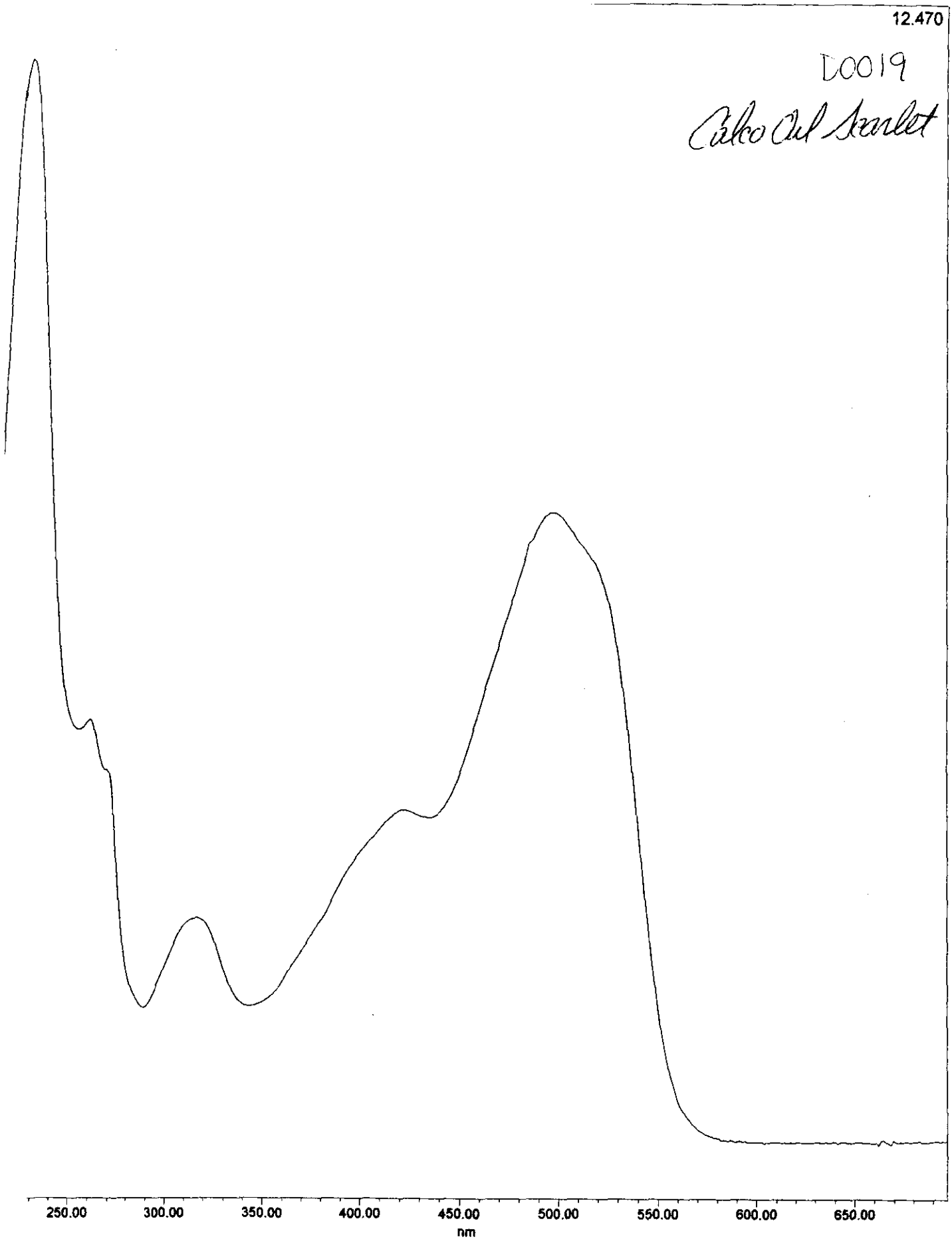
500.00
550.00
600.00
650.00
nm

Vat Yellow 4



C.I. Vat Yellow 4
C.I. # 59100
CAS # 128-66-5


12.470
D0019
Calco Oil Scarlet
250.00
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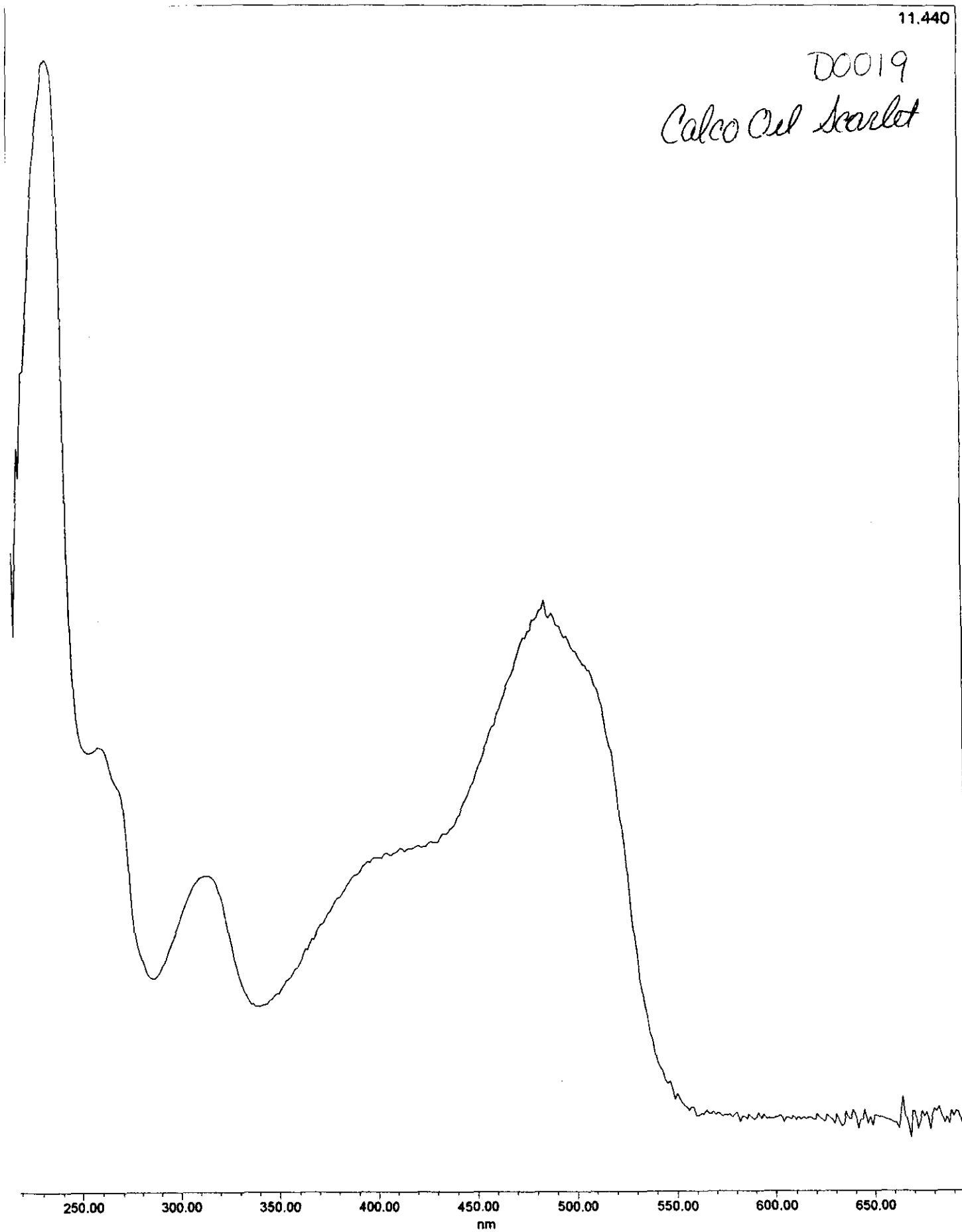
500.00
550.00
600.00
650.00
nm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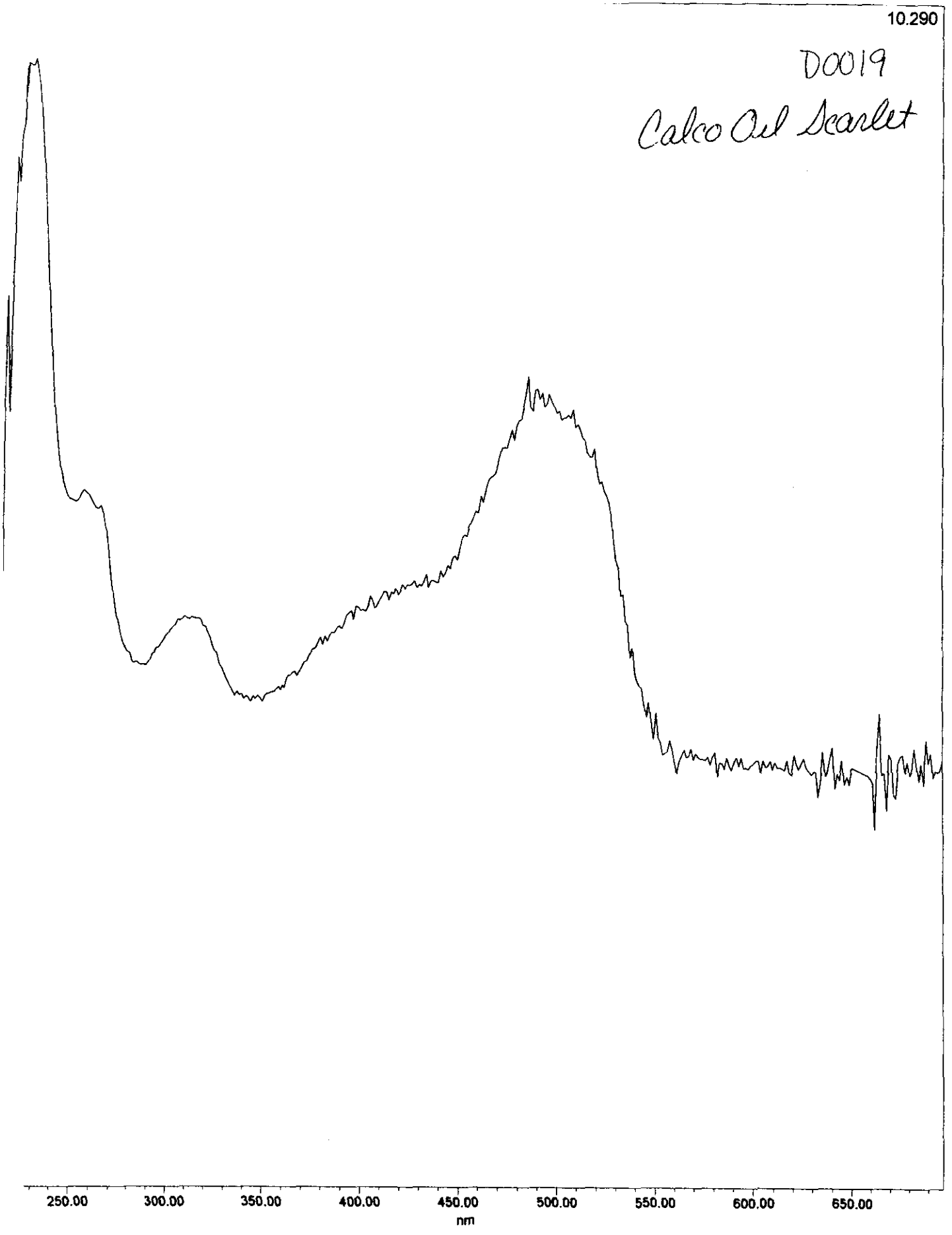
nm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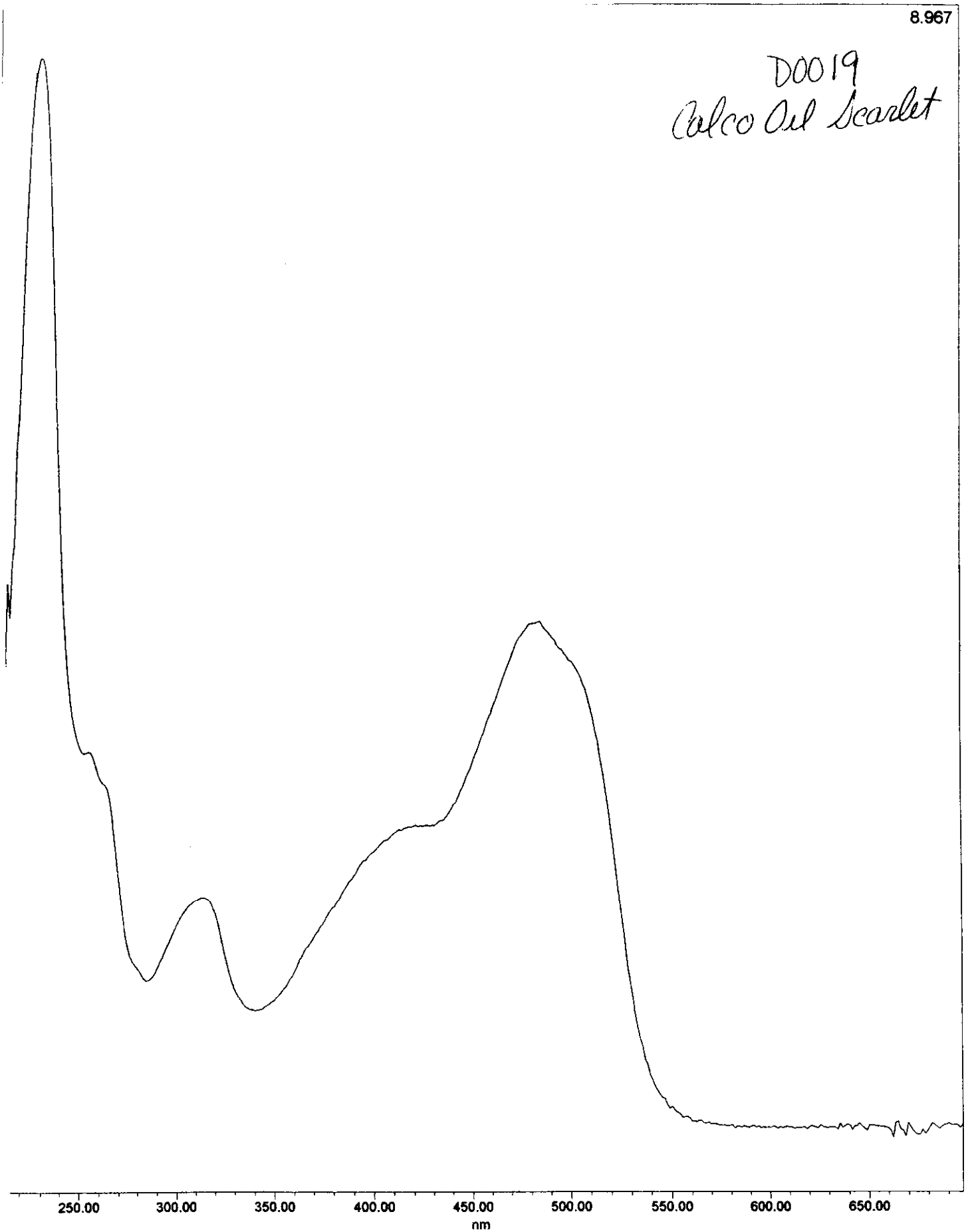
600.00
650.00
nm


8.967
D0019
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300.00
350.00
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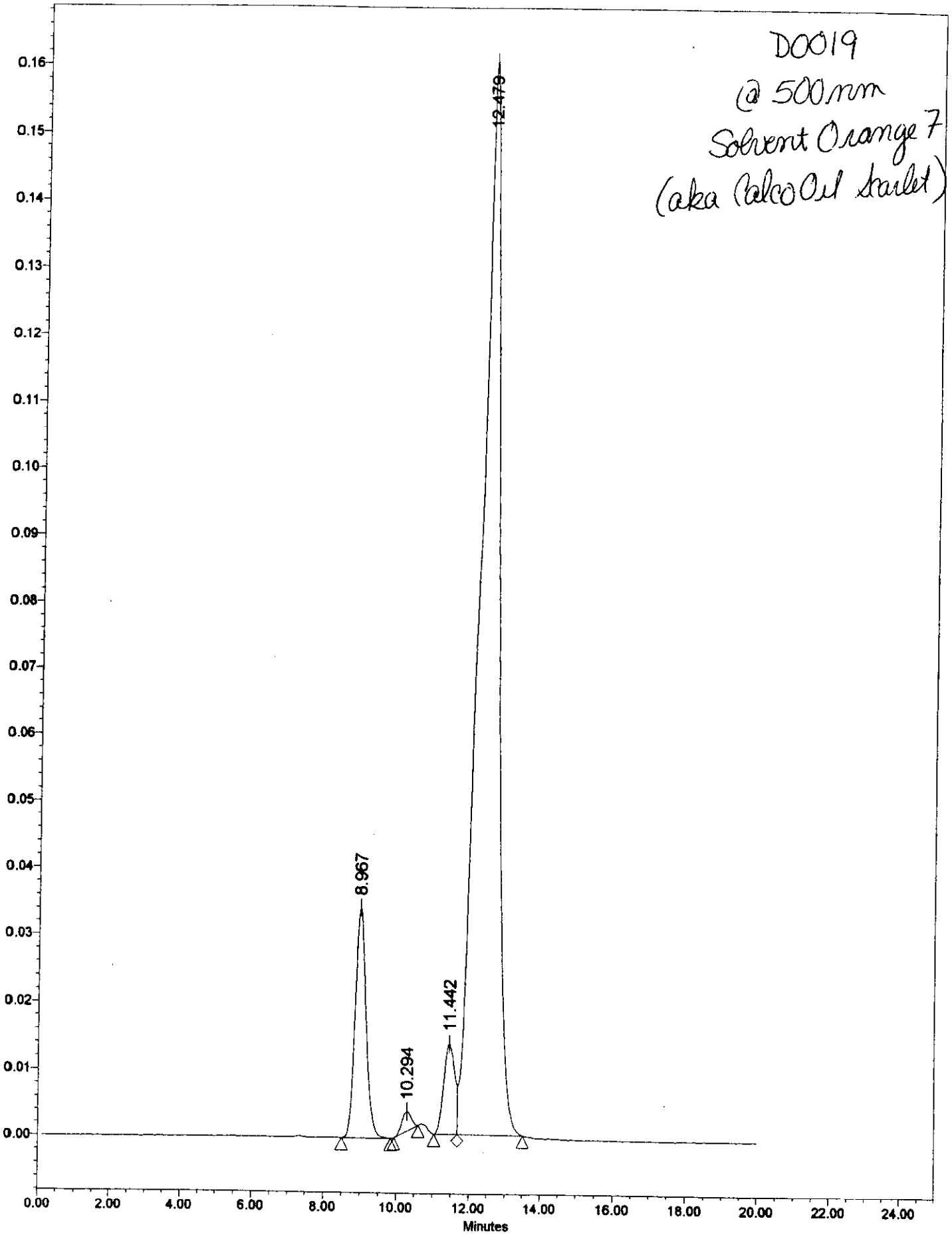
500.00
550.00
600.00
650.00
nm


D0019
@ 500nm
Solvent Orange 7
(aka Calco Oil Scarlet)
12.479
8.967
10.294
11.442
0.16
0.15
0.14
0.13
0.12
0.11
0.10
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0.00
0.00
2.00
4.00
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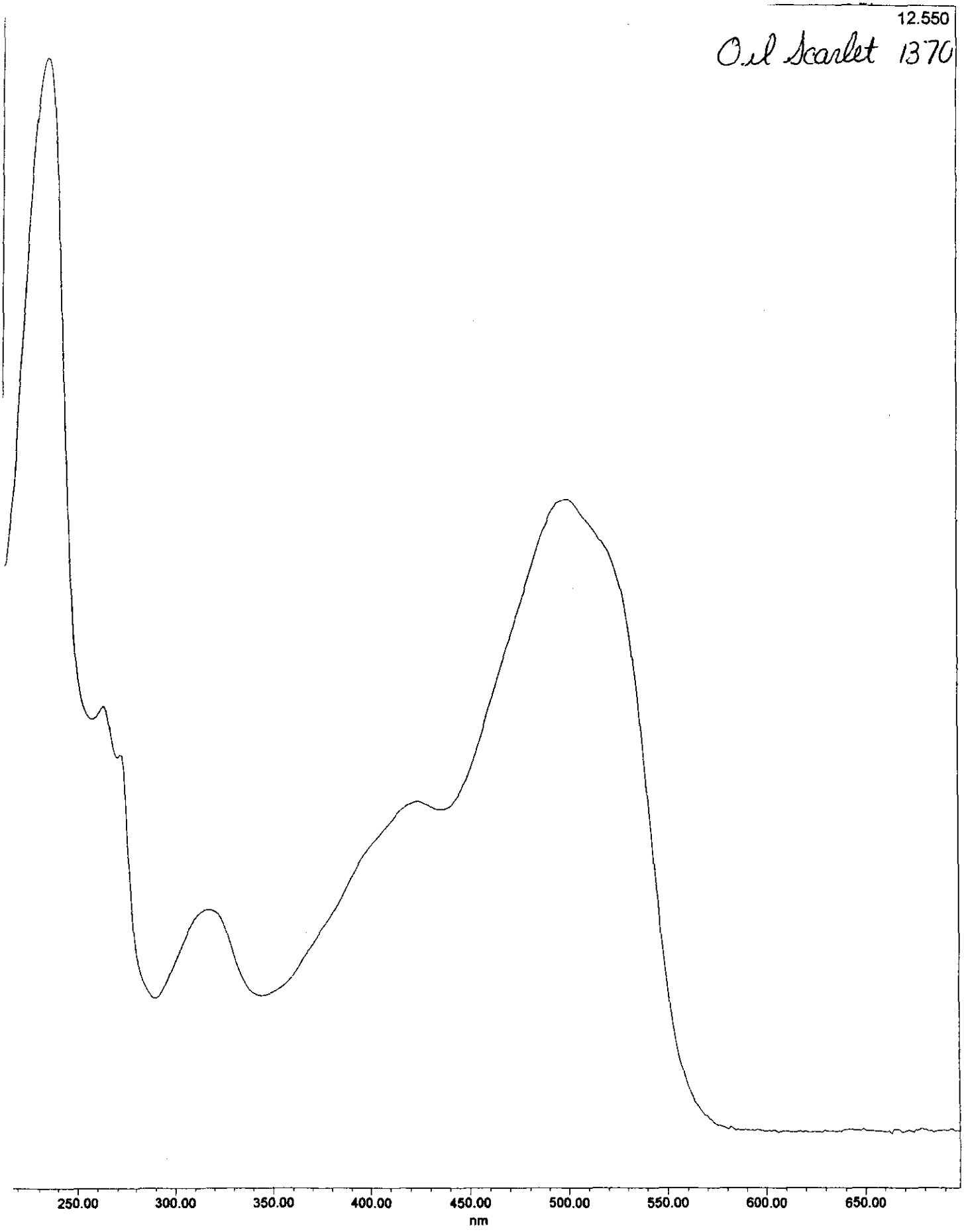
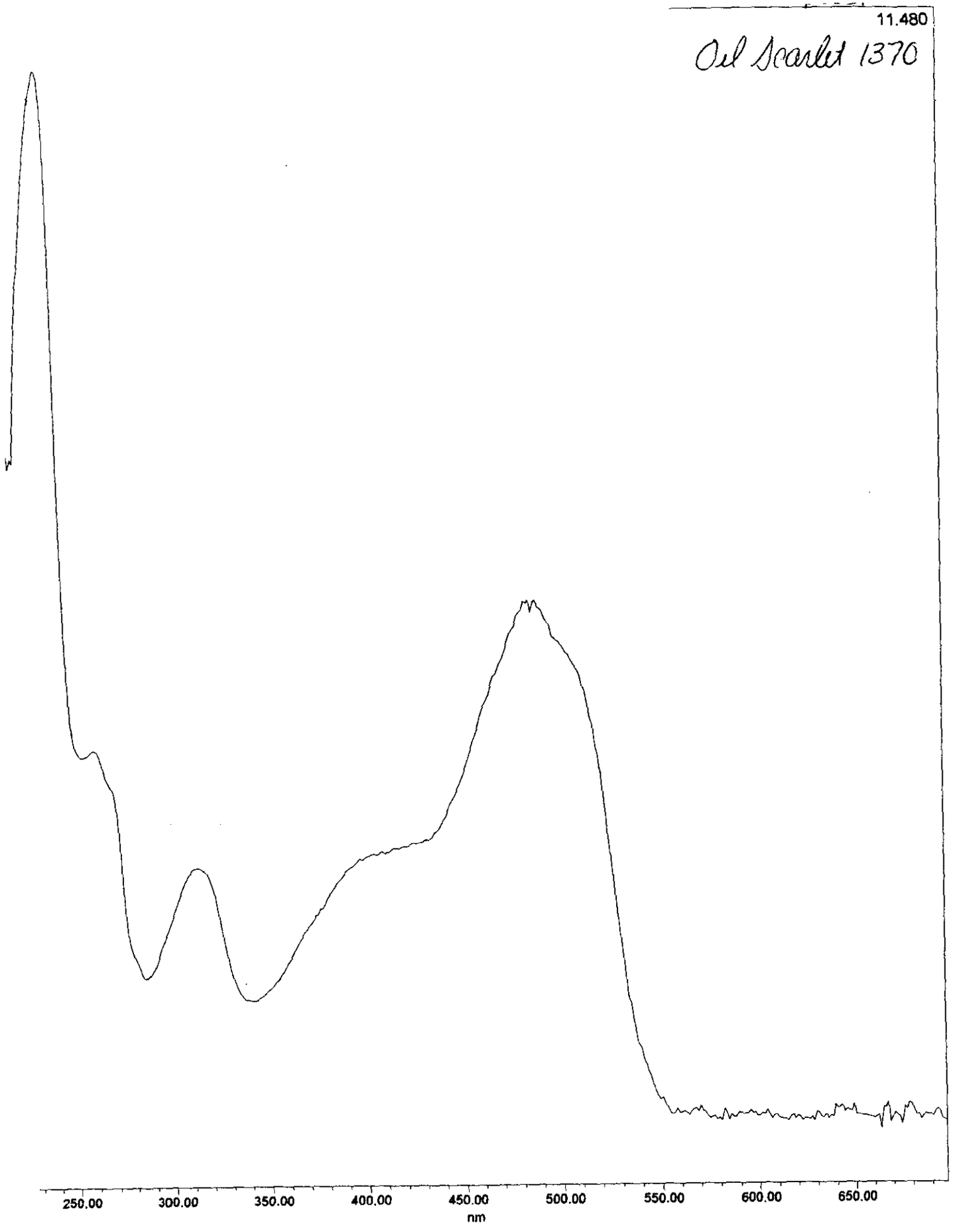
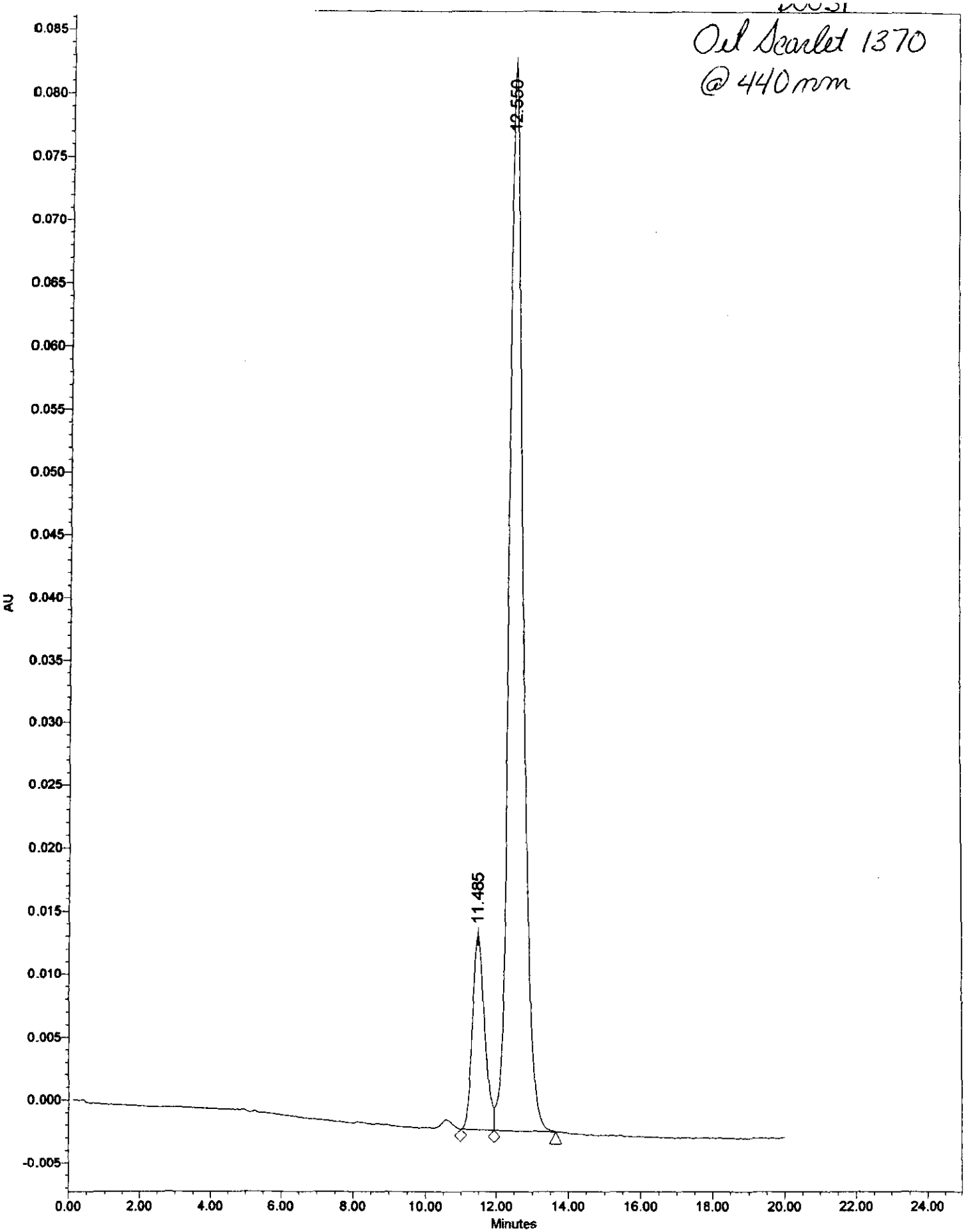
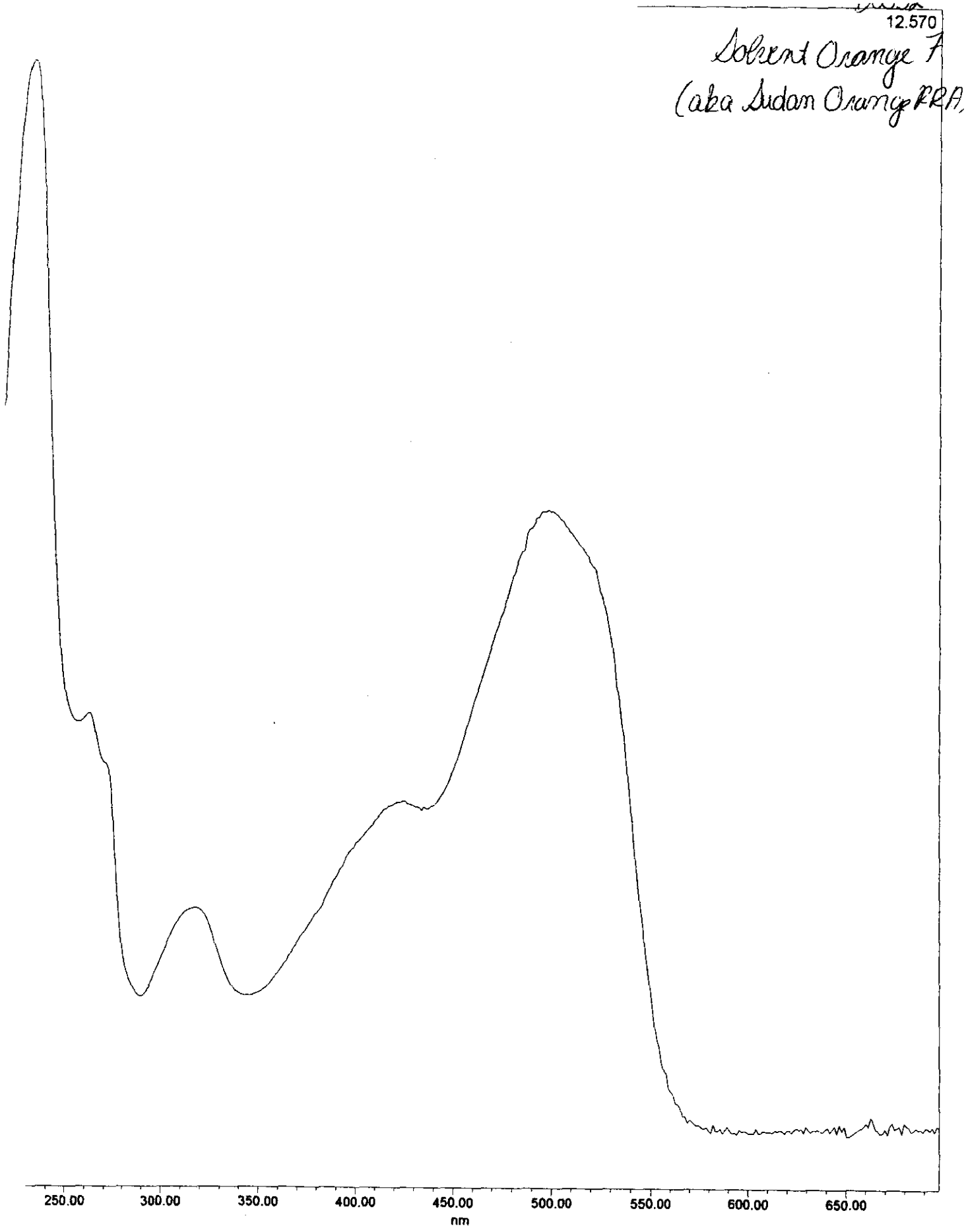
20.00
22.00
24.00
Minutes


12.550
Oil Scarlet 1370
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm


11.480
Oil Scarlet 1370
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

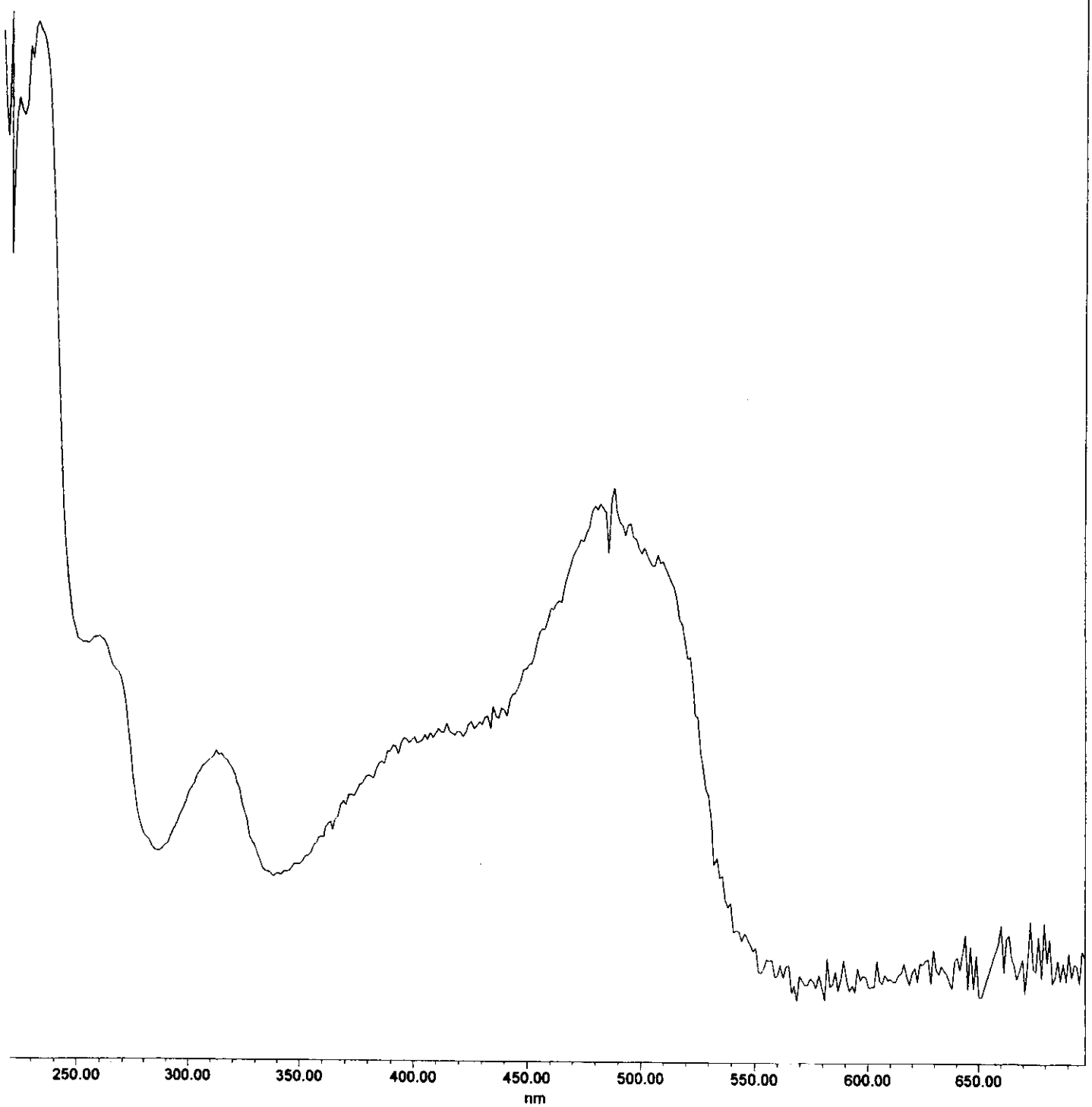
Oil Scarlet 1370
@ 440 nm




12.570
Solvent Orange 7
(aka Sudan Orange RRA)
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

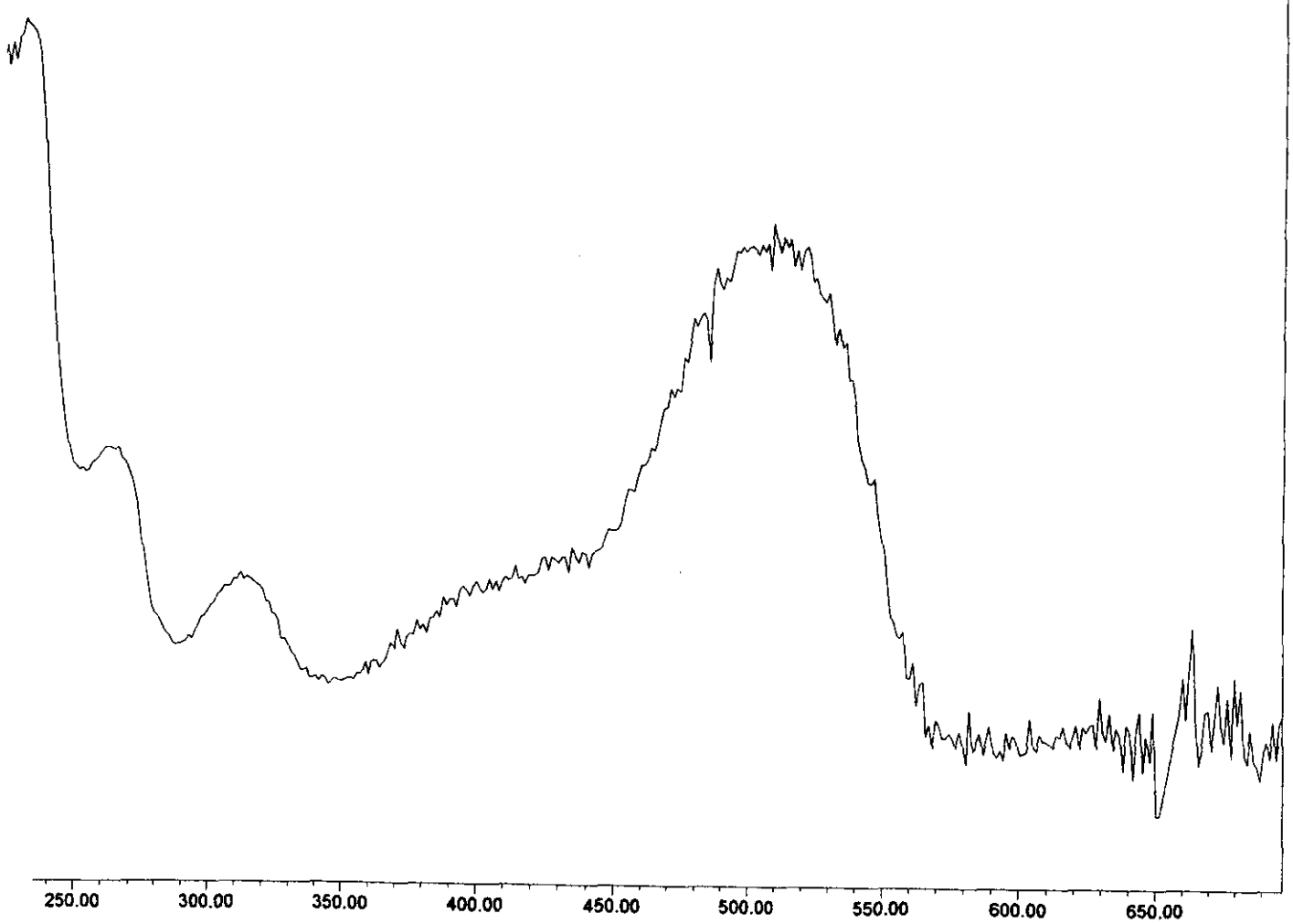
11.530

Solvent Orange 7
(aka Sudan Orange RR)

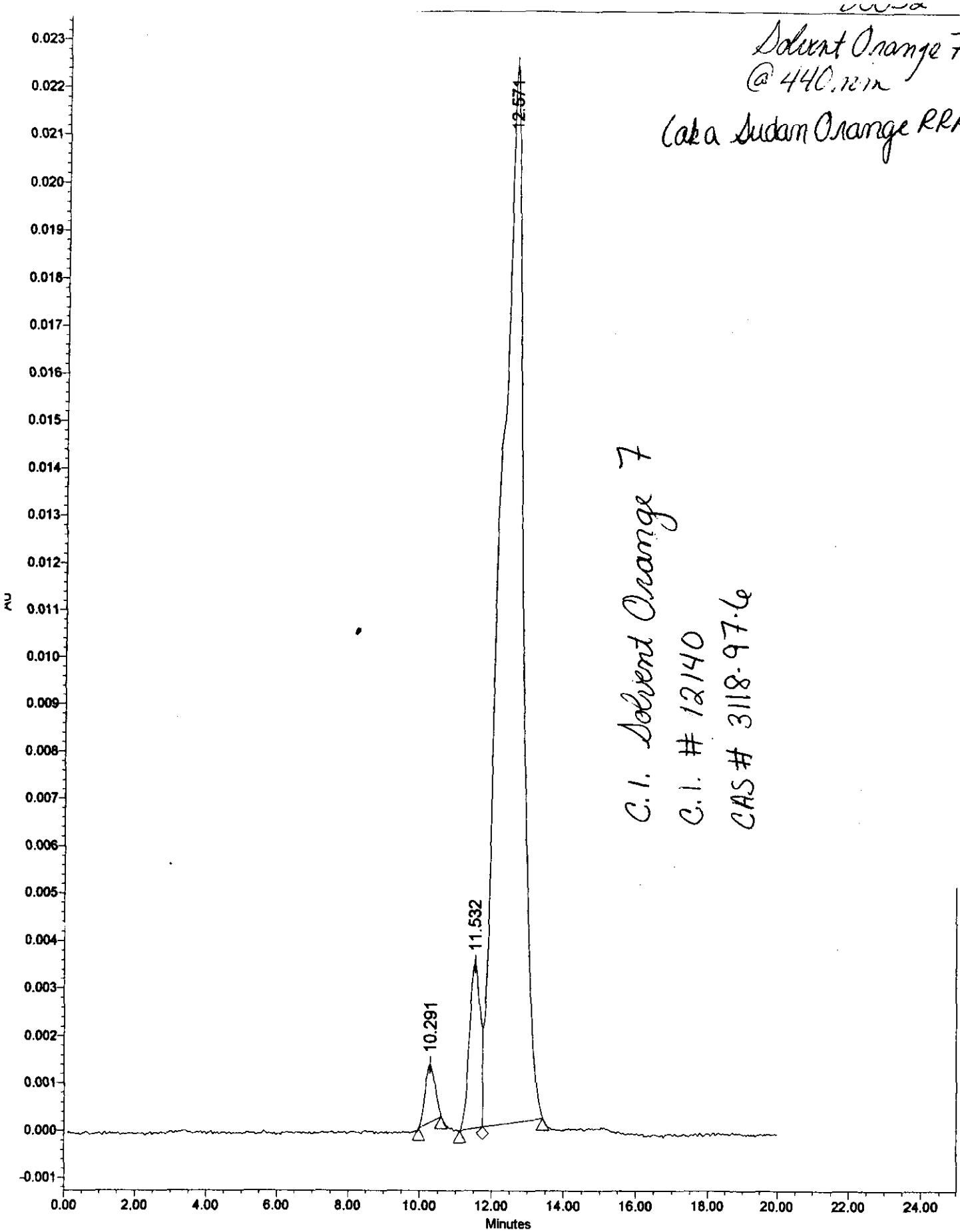


10.290

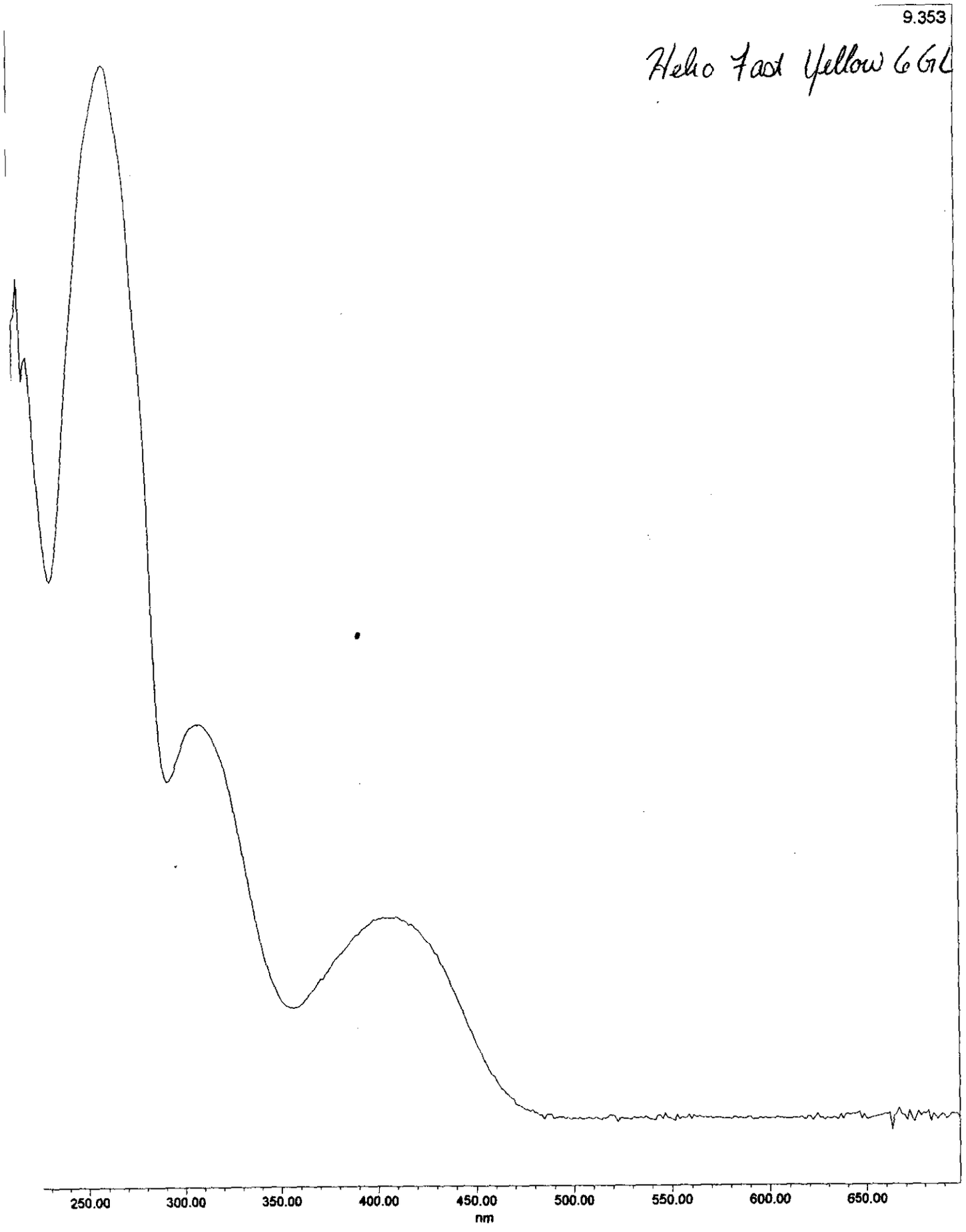
Solvent Orange 7
(aka Sudan Orange RRA)



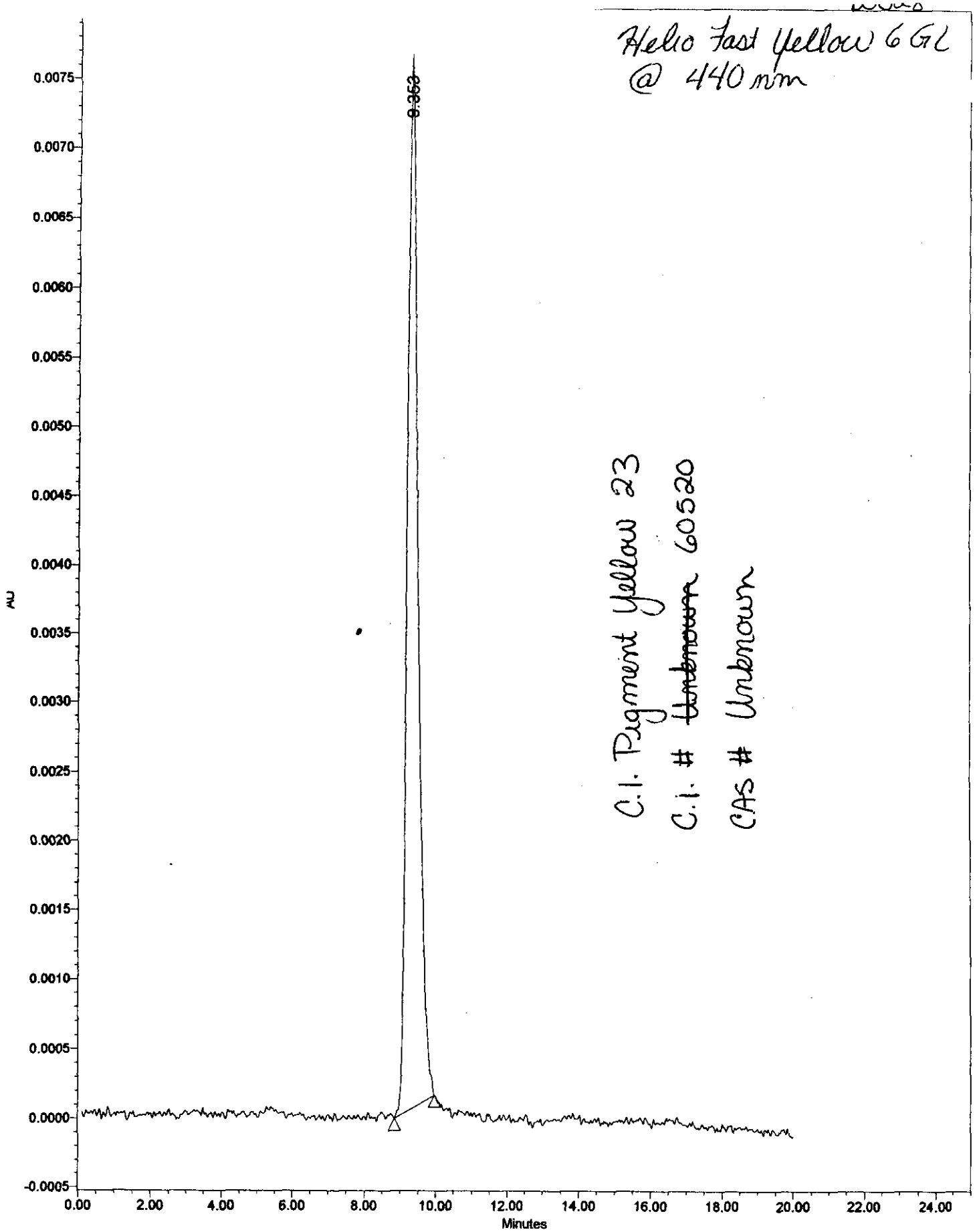
Solvent Orange 7
@ 440 nm
(aka Sudan Orange RRA)



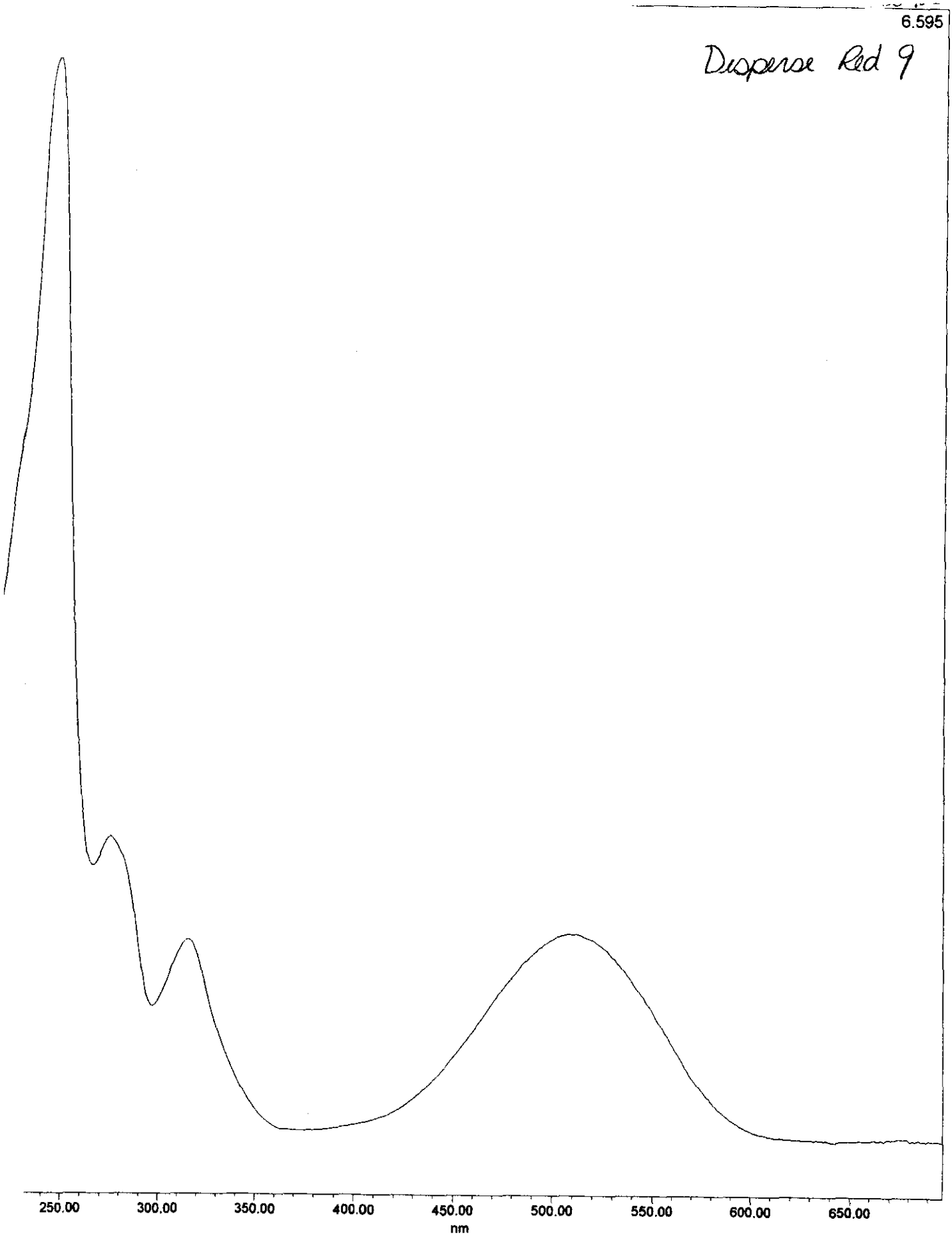
C.I. Solvent Orange 7
C.I. # 12140
CAS # 3118-97-6

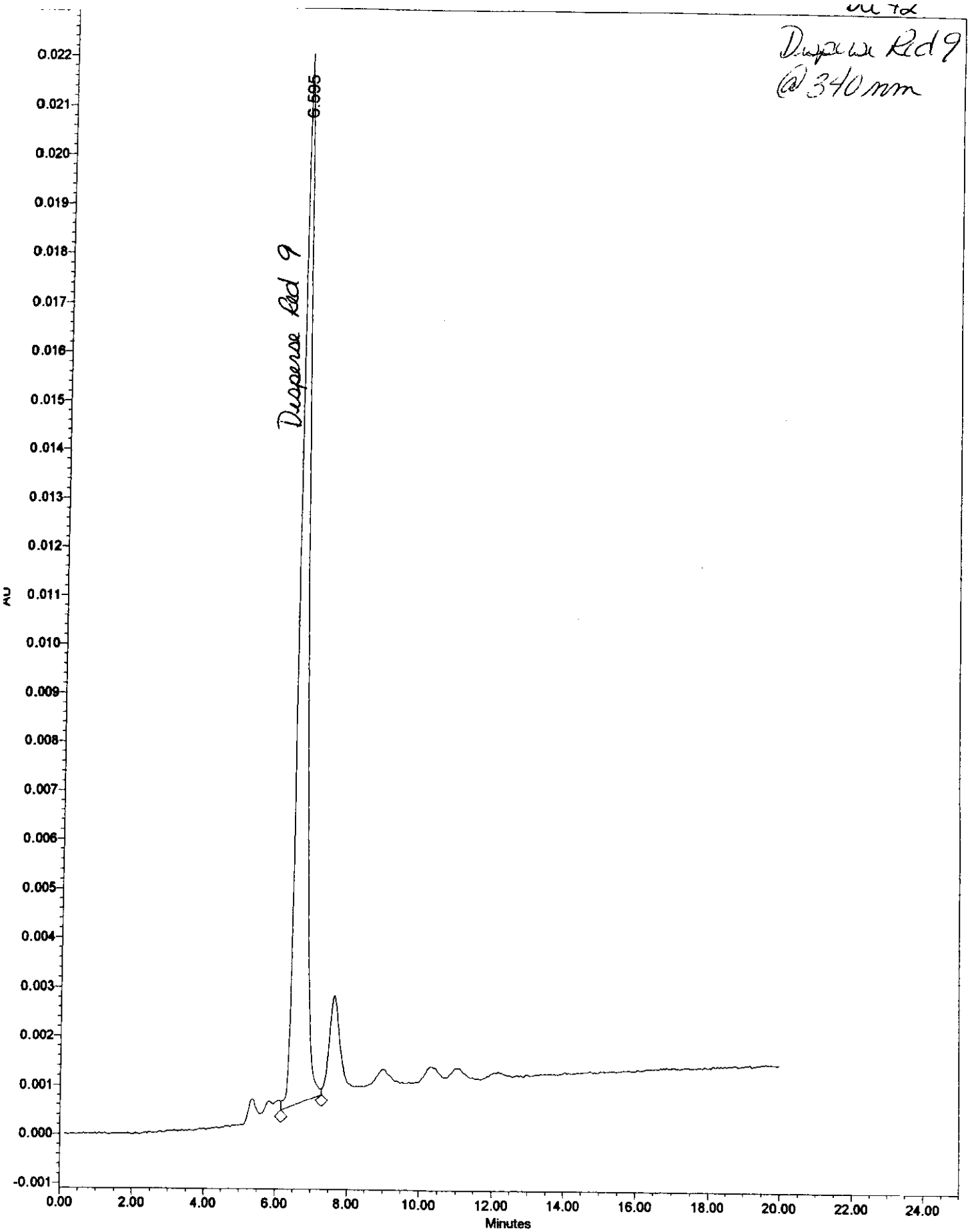

9.353
Helio Fast Yellow 6GL
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

Helio Fast Yellow 6 GL
@ 440 nm



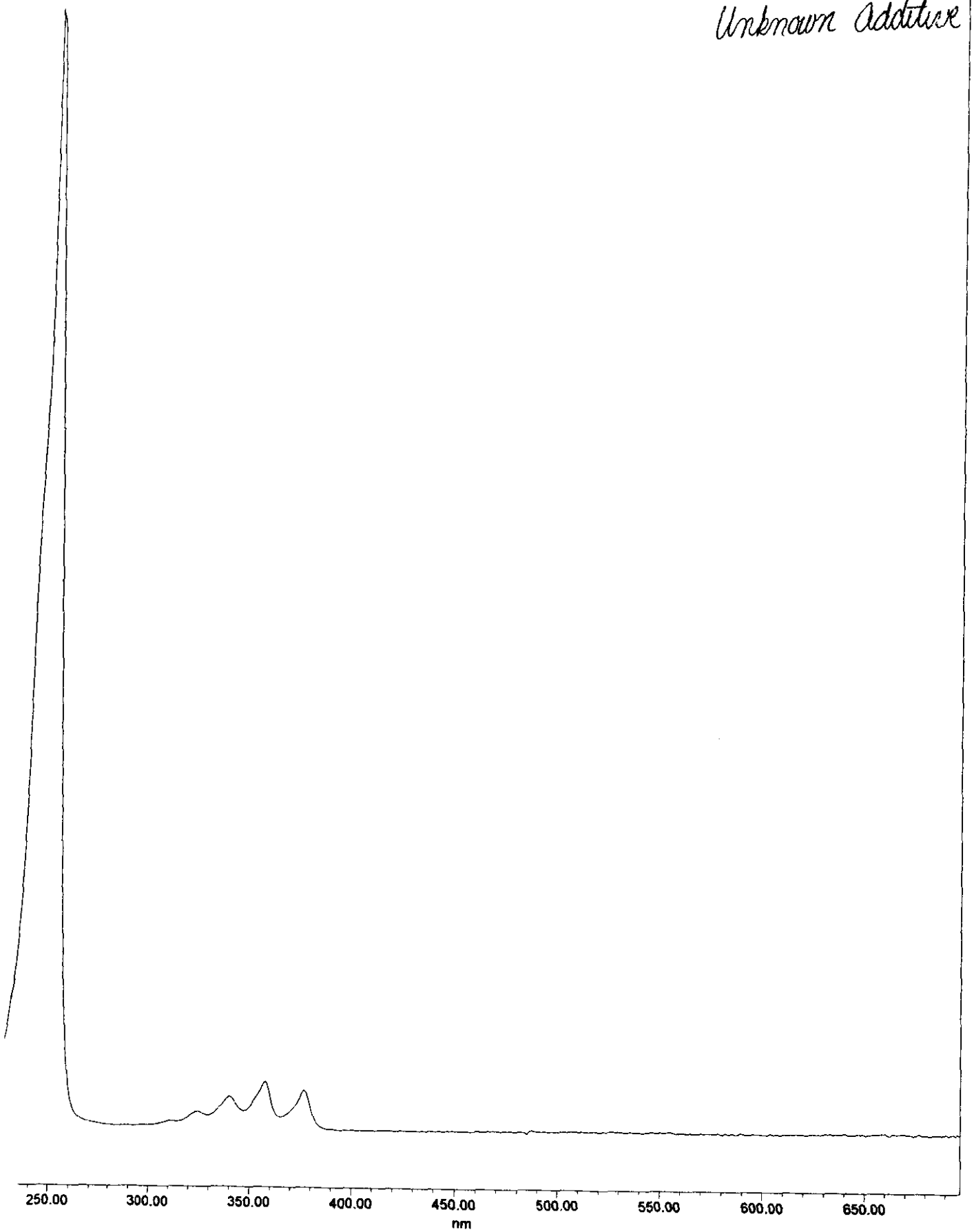
C.I. Pigment Yellow 23
C.I. # ~~Unknown~~ 60520
CAS # Unknown

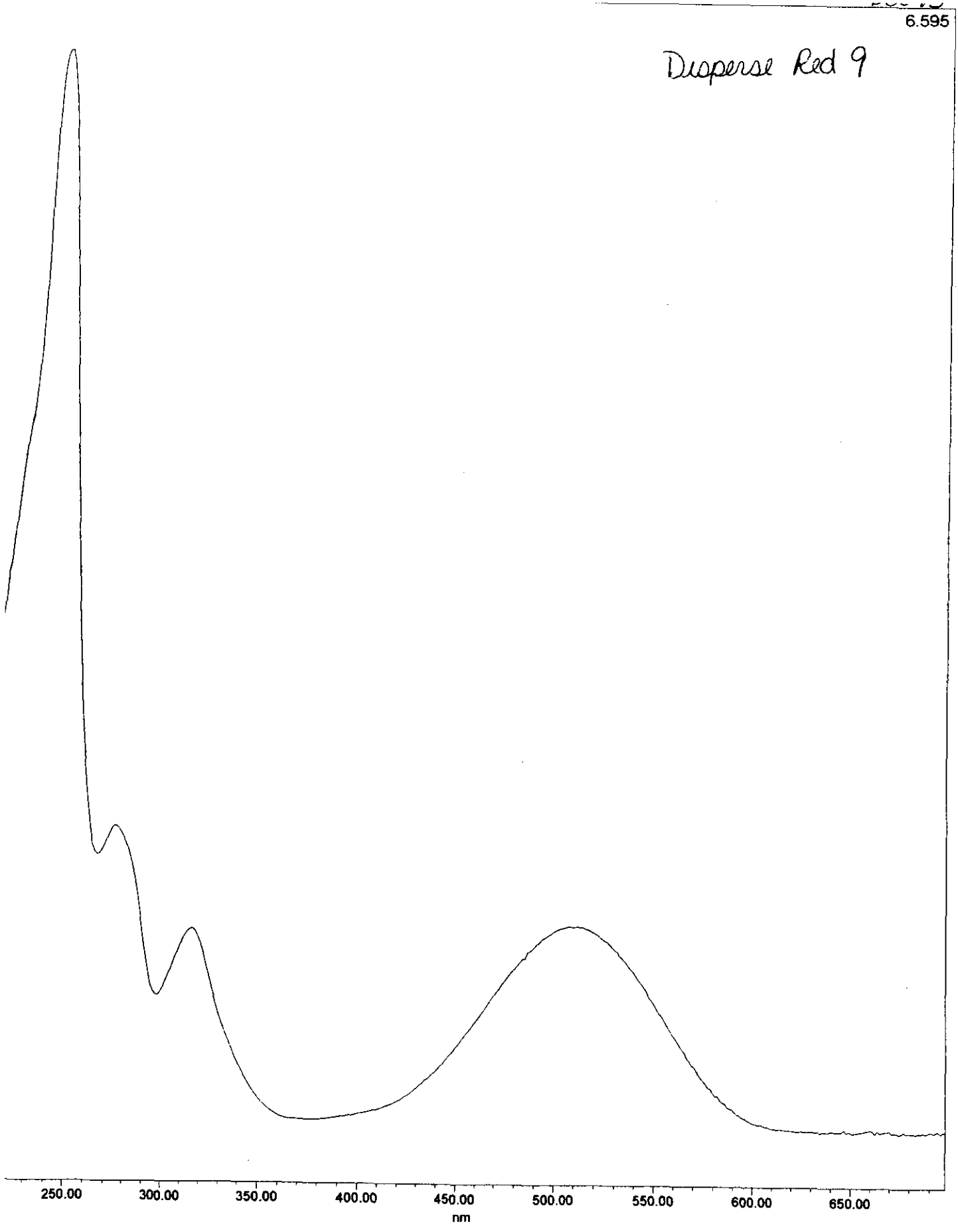

6.595
Disperse Red 9
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

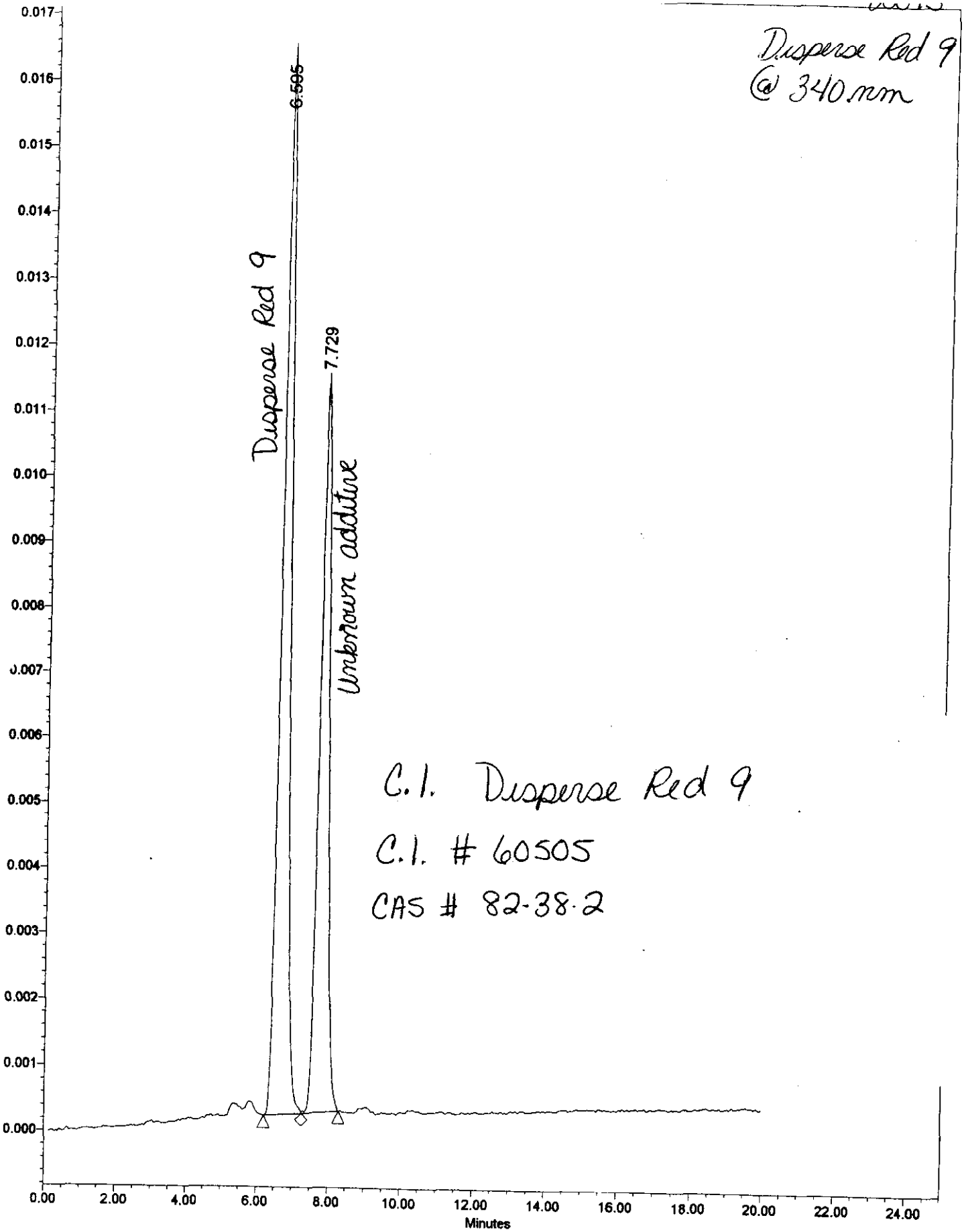

Disperse Red 9
@ 340 nm
Disperse Red 9
6.595
AU
0.022
0.021
0.020
0.019
0.018
0.017
0.016
0.015
0.014
0.013
0.012
0.011
0.010
0.009
0.008
0.007
0.006
0.005
0.004
0.003
0.002
0.001
0.000
-0.001
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
22.00
24.00
Minutes

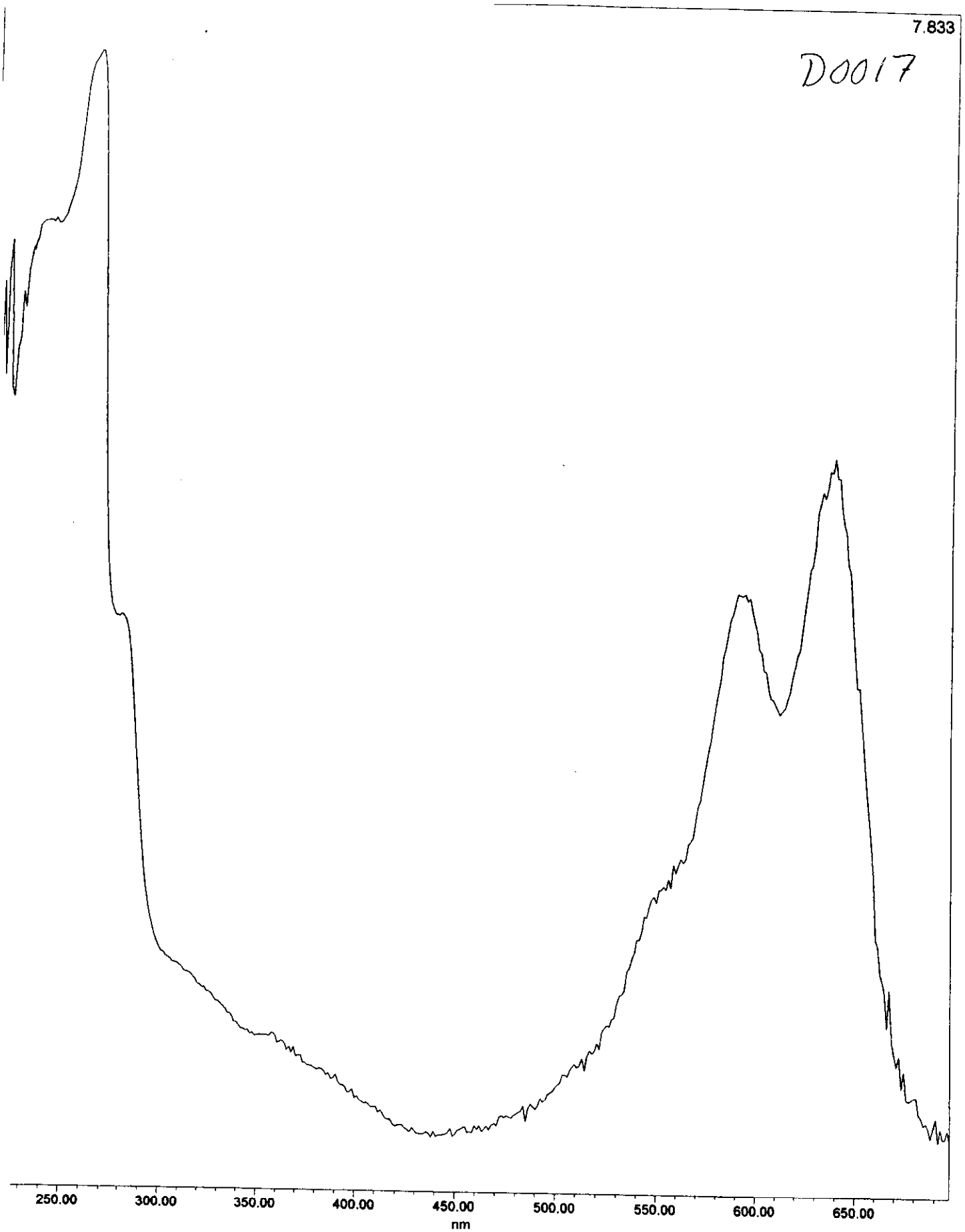
7.729

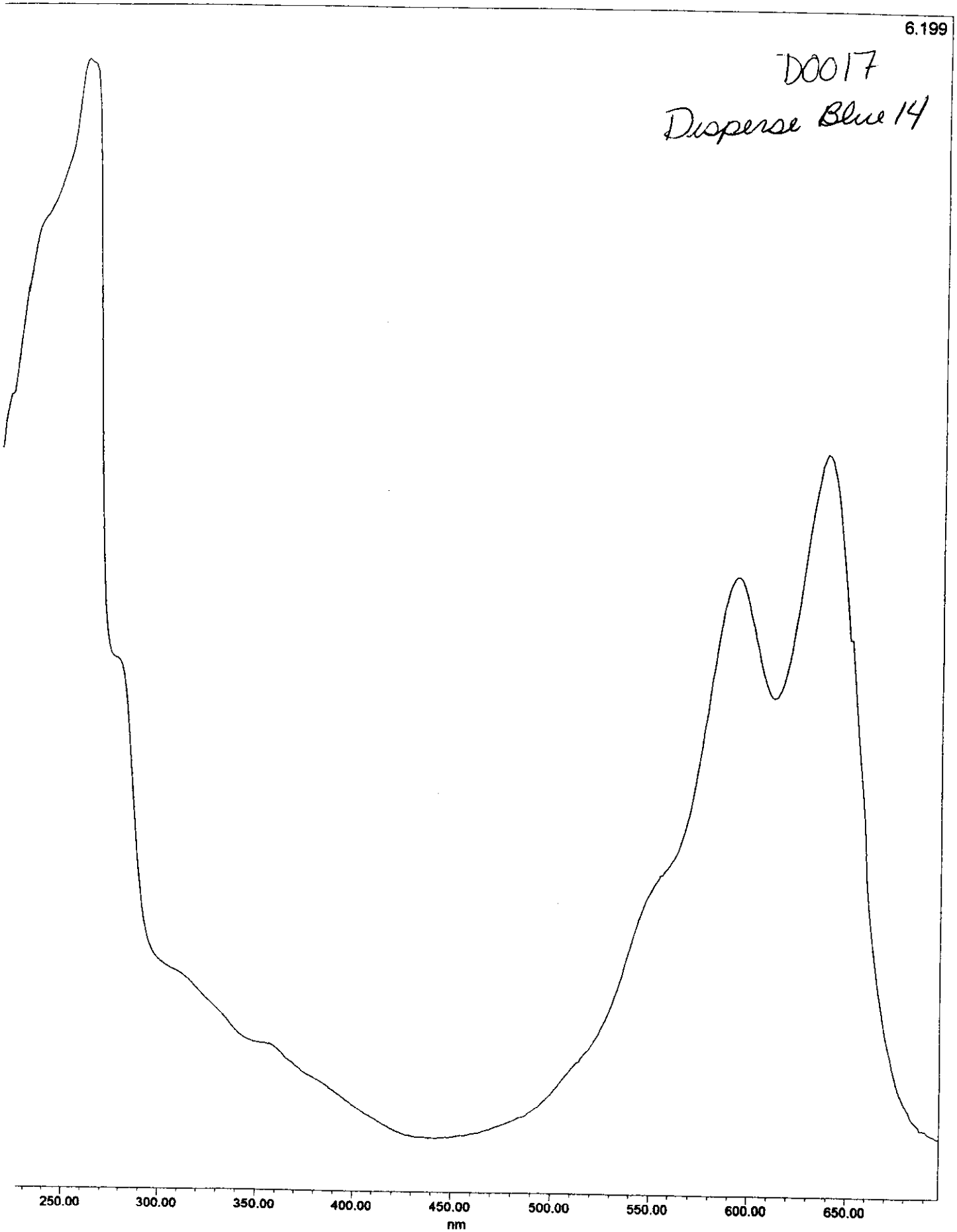
Unknown Additive

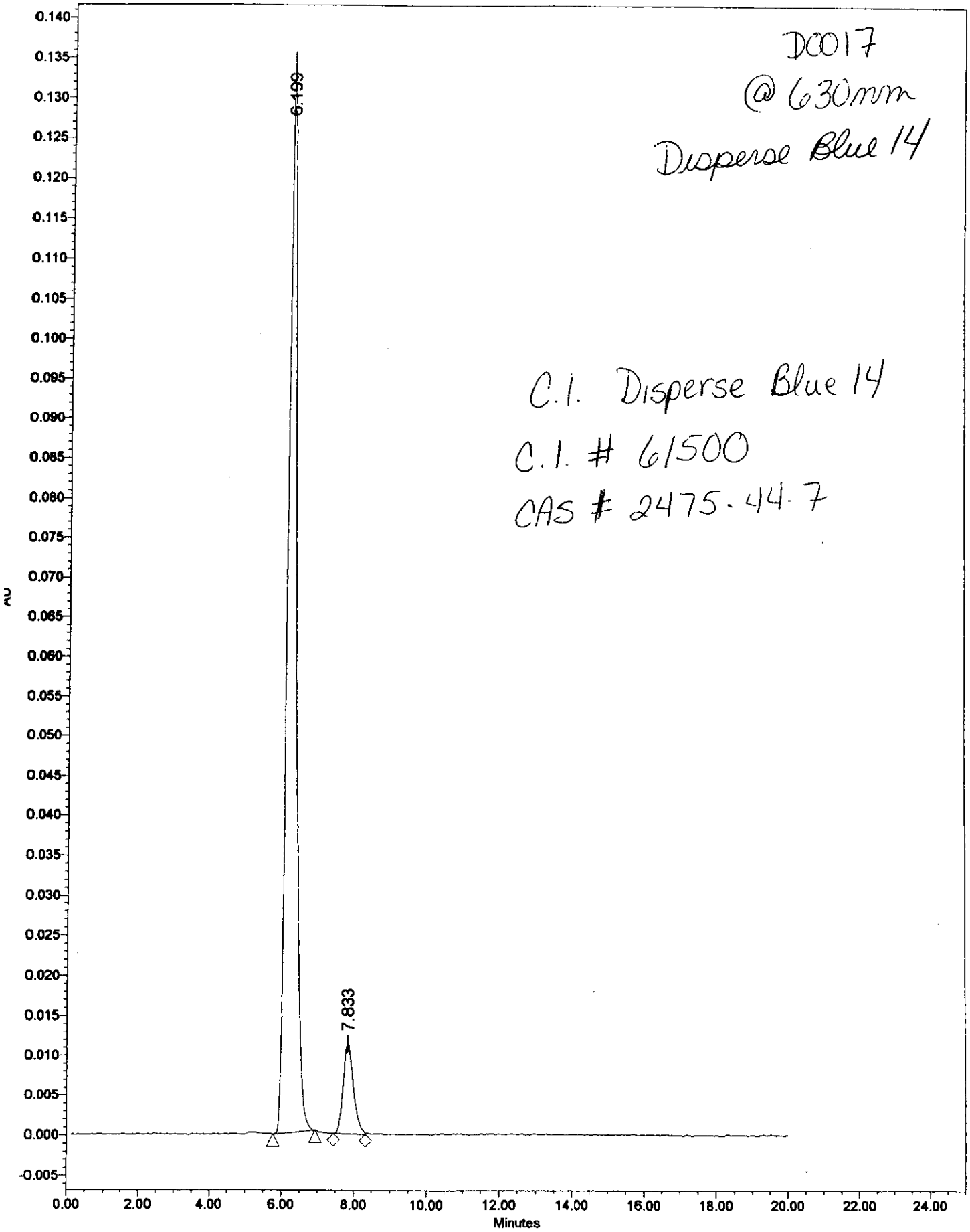


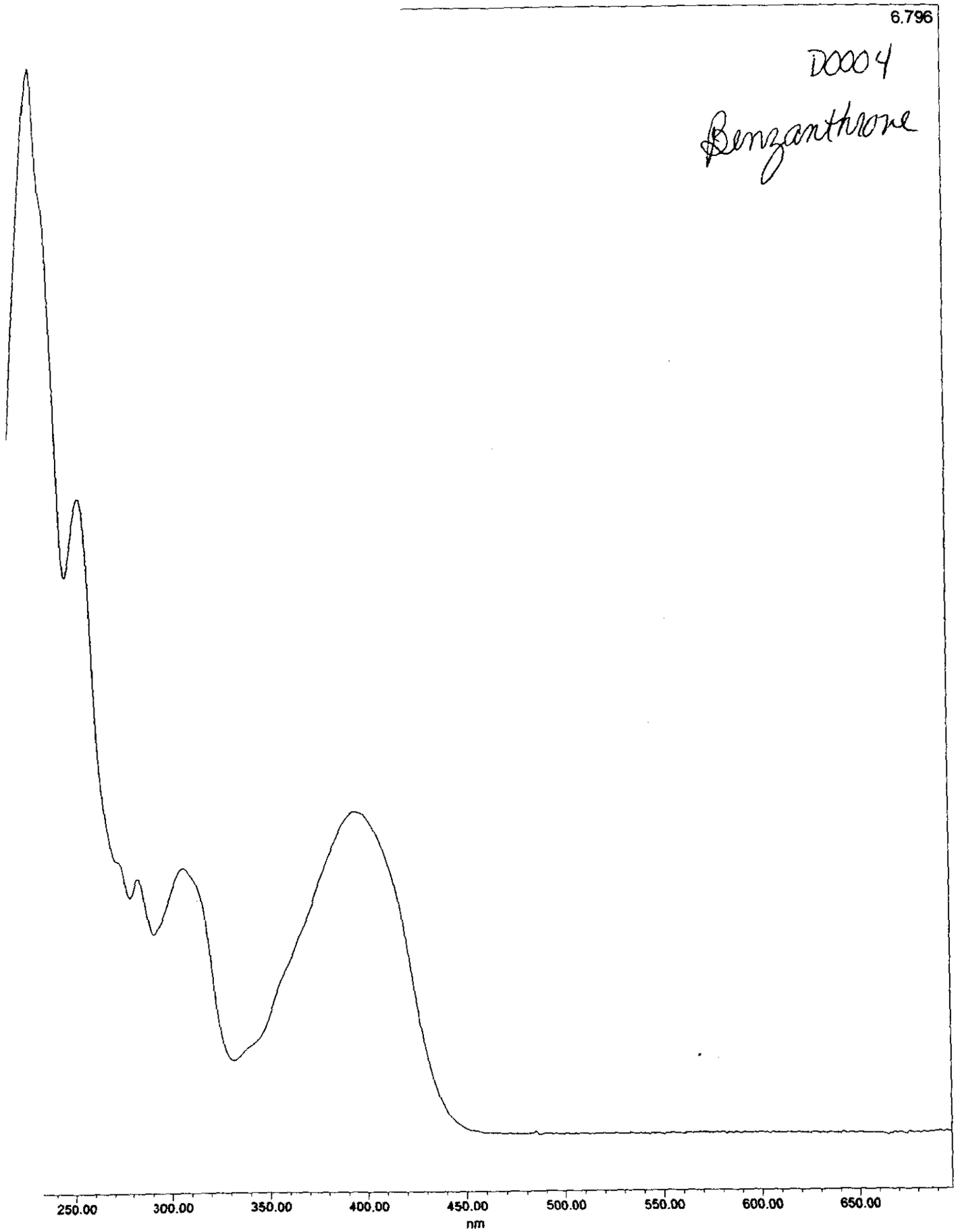

6.595
Disperse Red 9
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

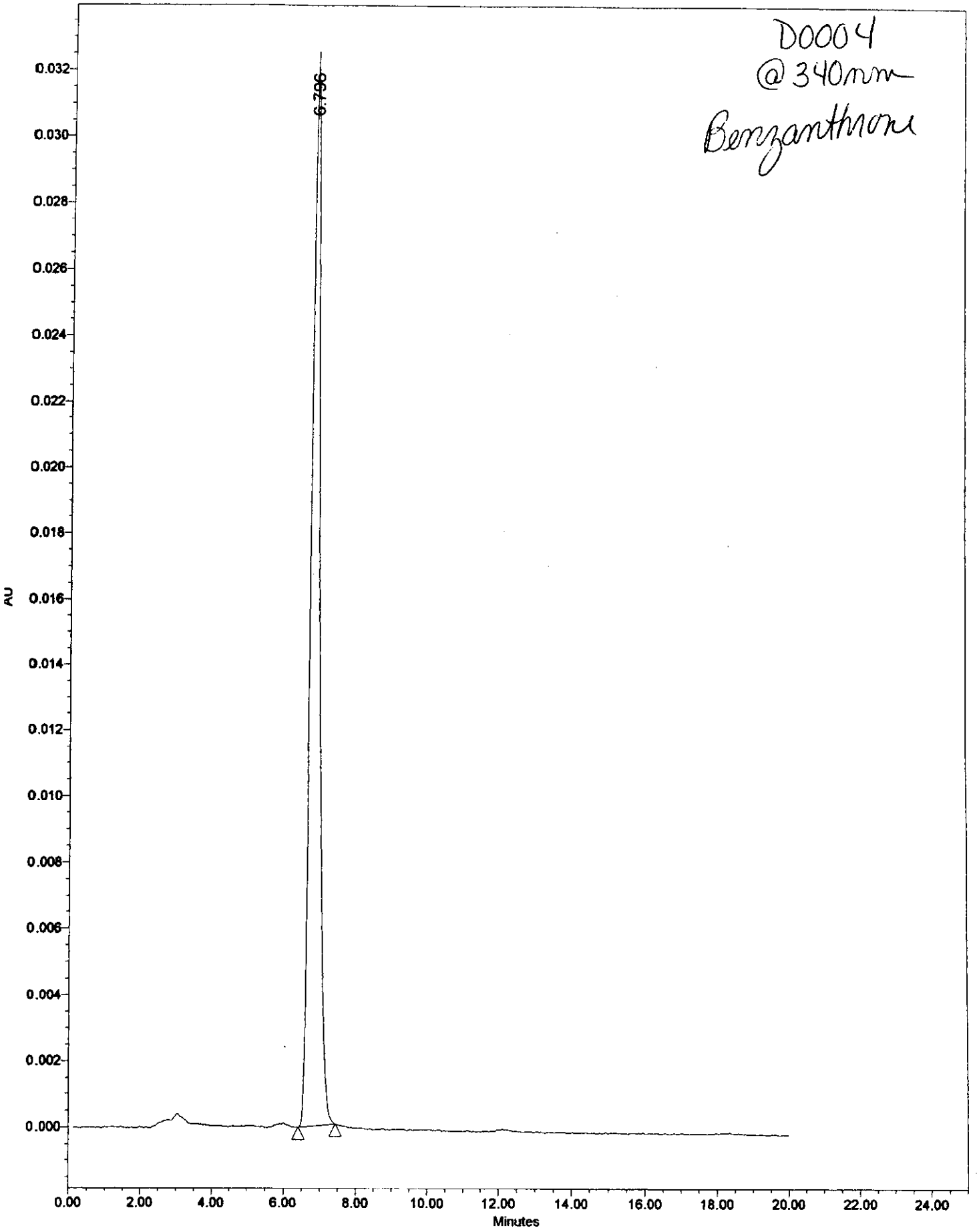

Disperse Red 9
@ 340 nm
6.595
7.729
Disperse Red 9
Unknown additive
C.I. Disperse Red 9
C.I. # 60505
CAS # 82-38-2
0.017
0.016
0.015
0.014
0.013
0.012
0.011
0.010
0.009
0.008
0.007
0.006
0.005
0.004
0.003
0.002
0.001
0.000
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
22.00
24.00
Minutes

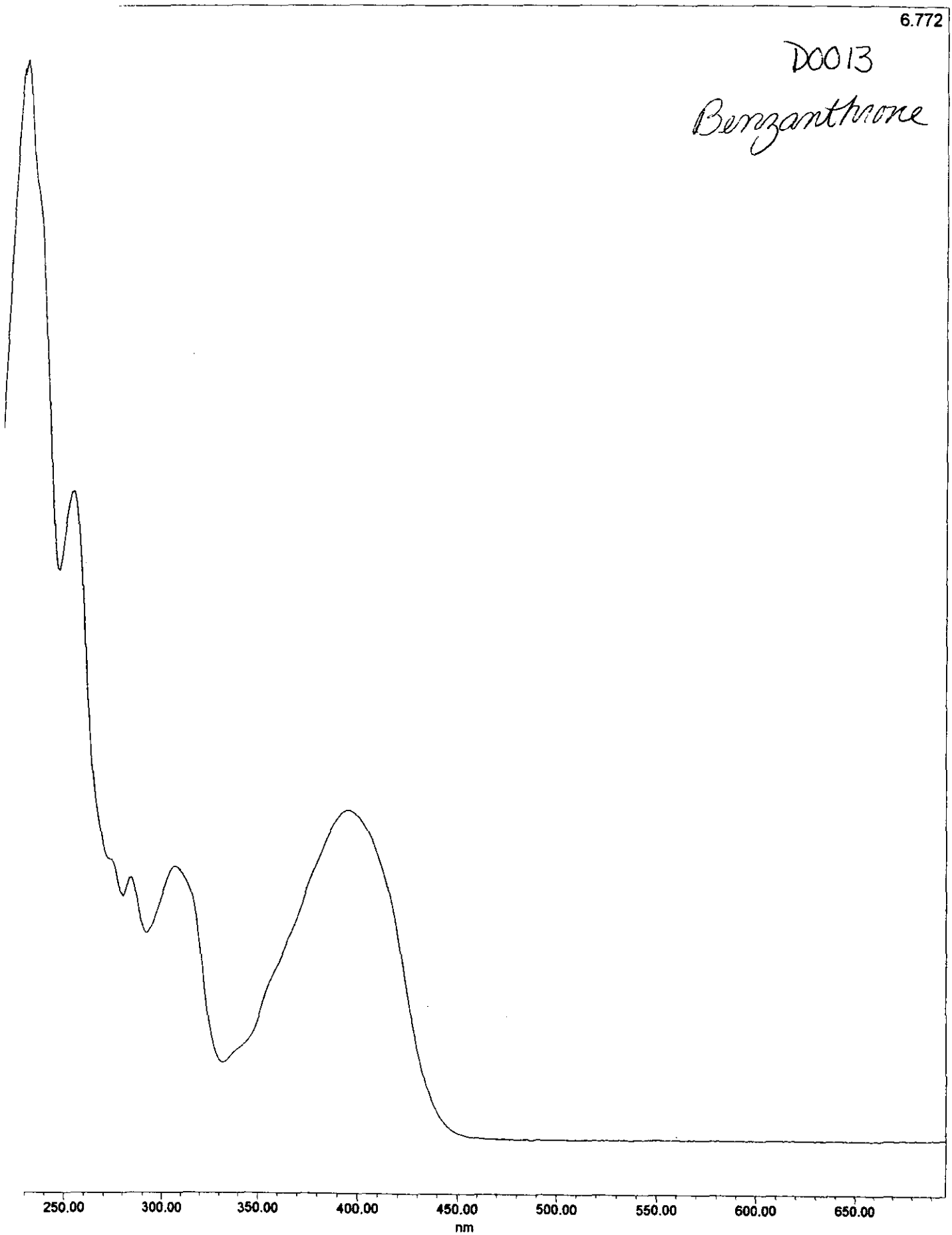

7.833
D0017
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

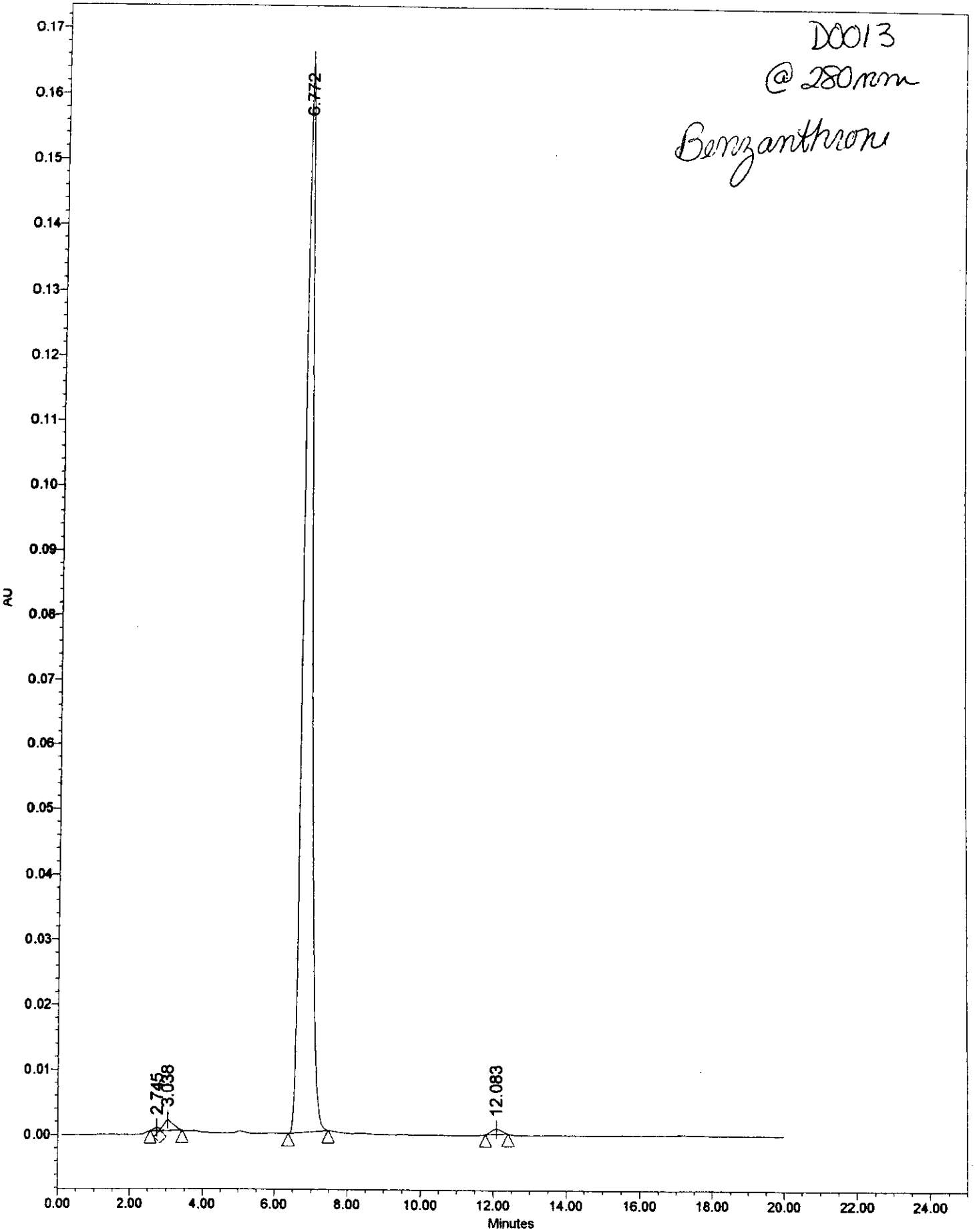

6.199
D0017
Disperse Blue 14
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

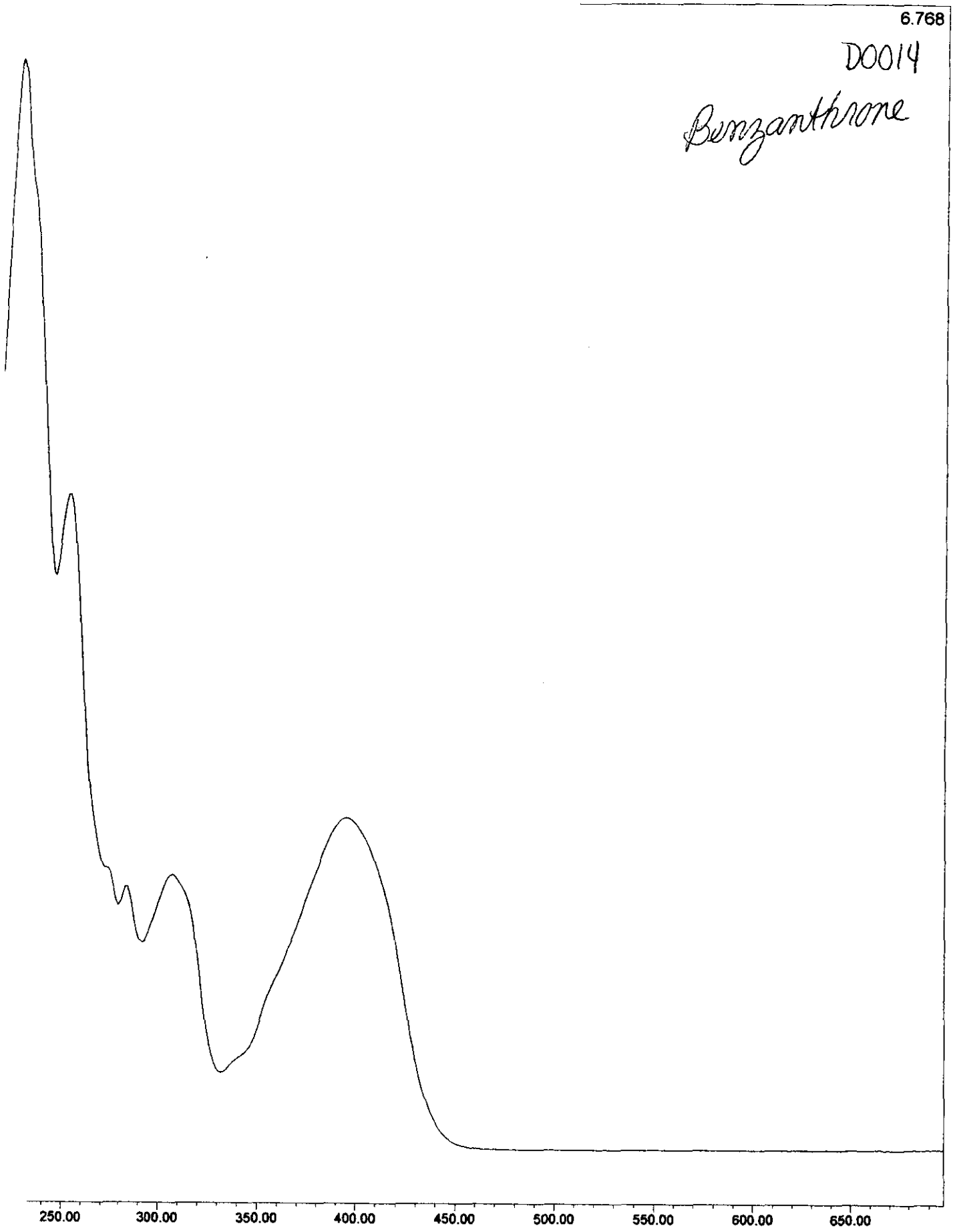

D0017
@ 630nm
Disperse Blue 14
C.I. Disperse Blue 14
C.I. # 61500
CAS # 2475-44-7
6.199
7.833
AU
Minutes

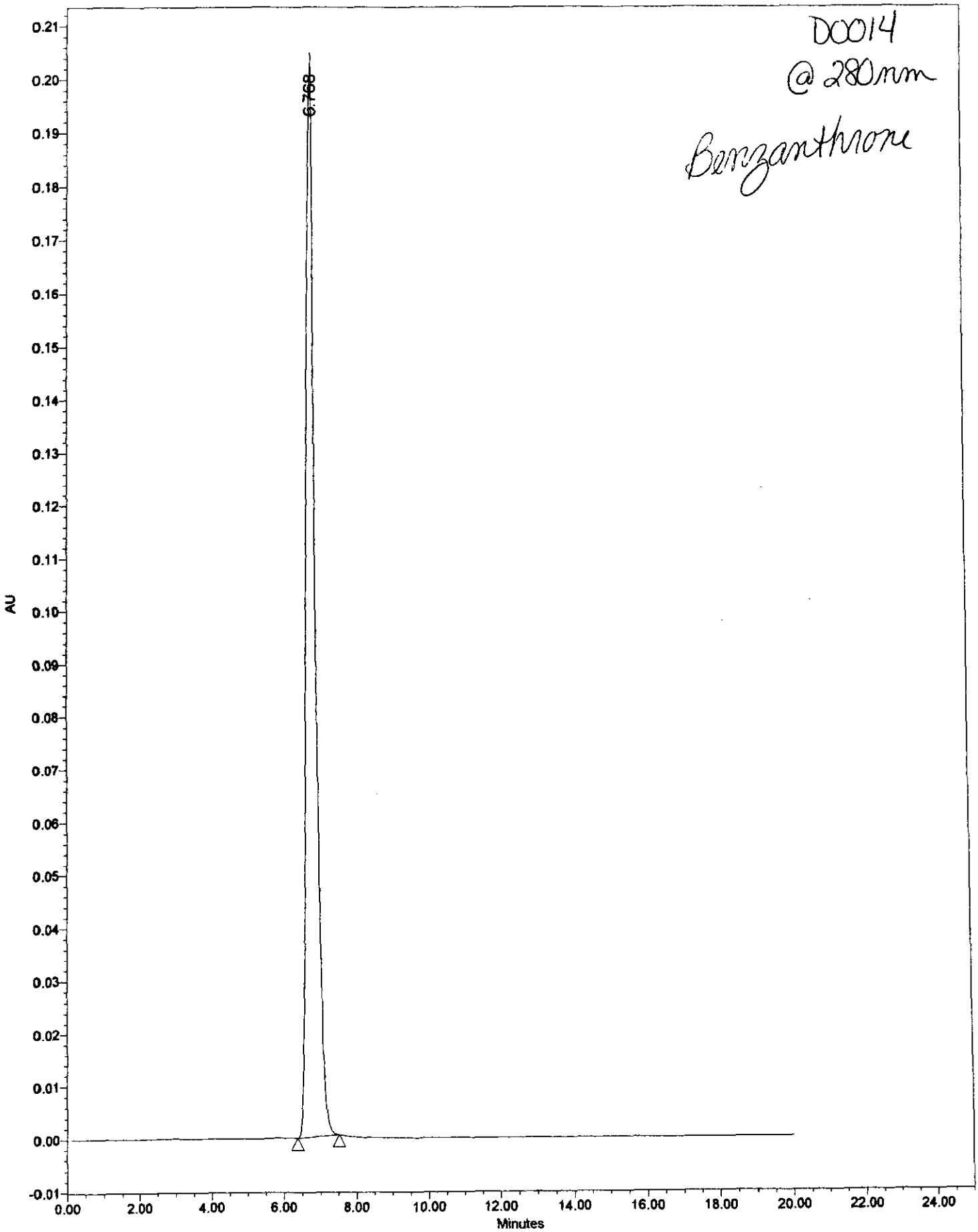

6.796
D0004
Benzanthrone
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

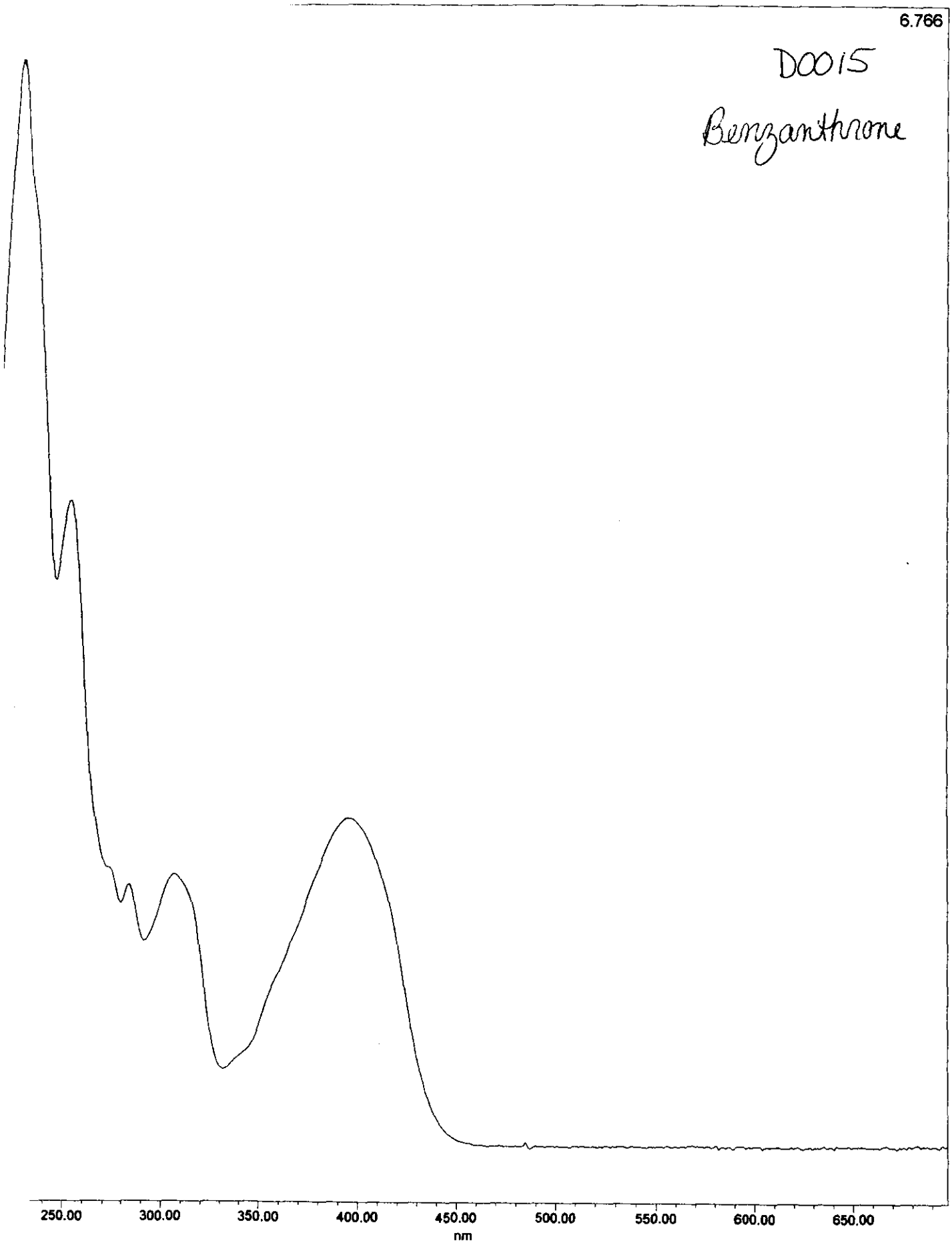

D0004
@ 340nm
Benzanthrone
6.796
AU
0.032
0.030
0.028
0.026
0.024
0.022
0.020
0.018
0.016
0.014
0.012
0.010
0.008
0.006
0.004
0.002
0.000
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
22.00
24.00
Minutes

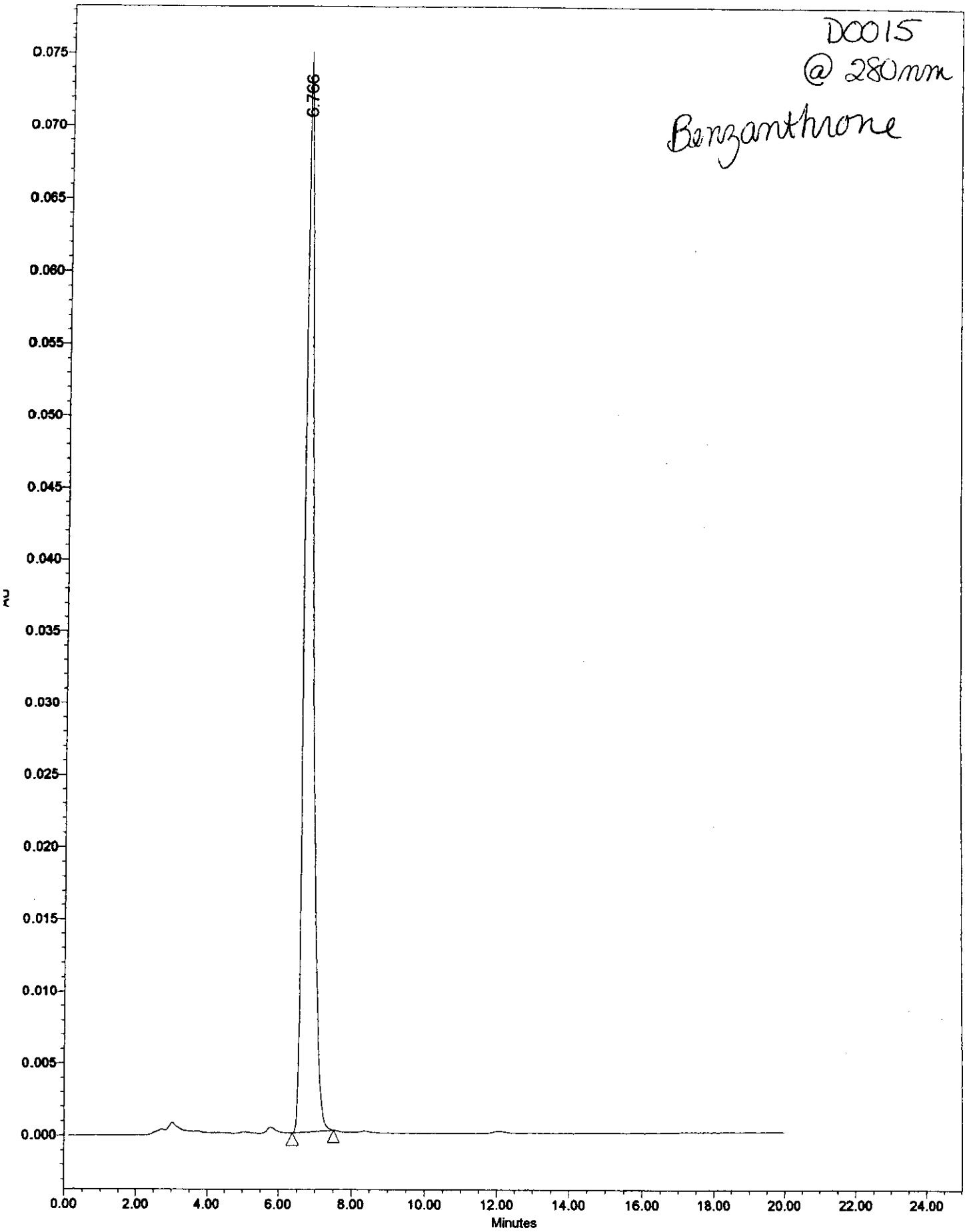

6.772
D0013
Benzanthrone
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

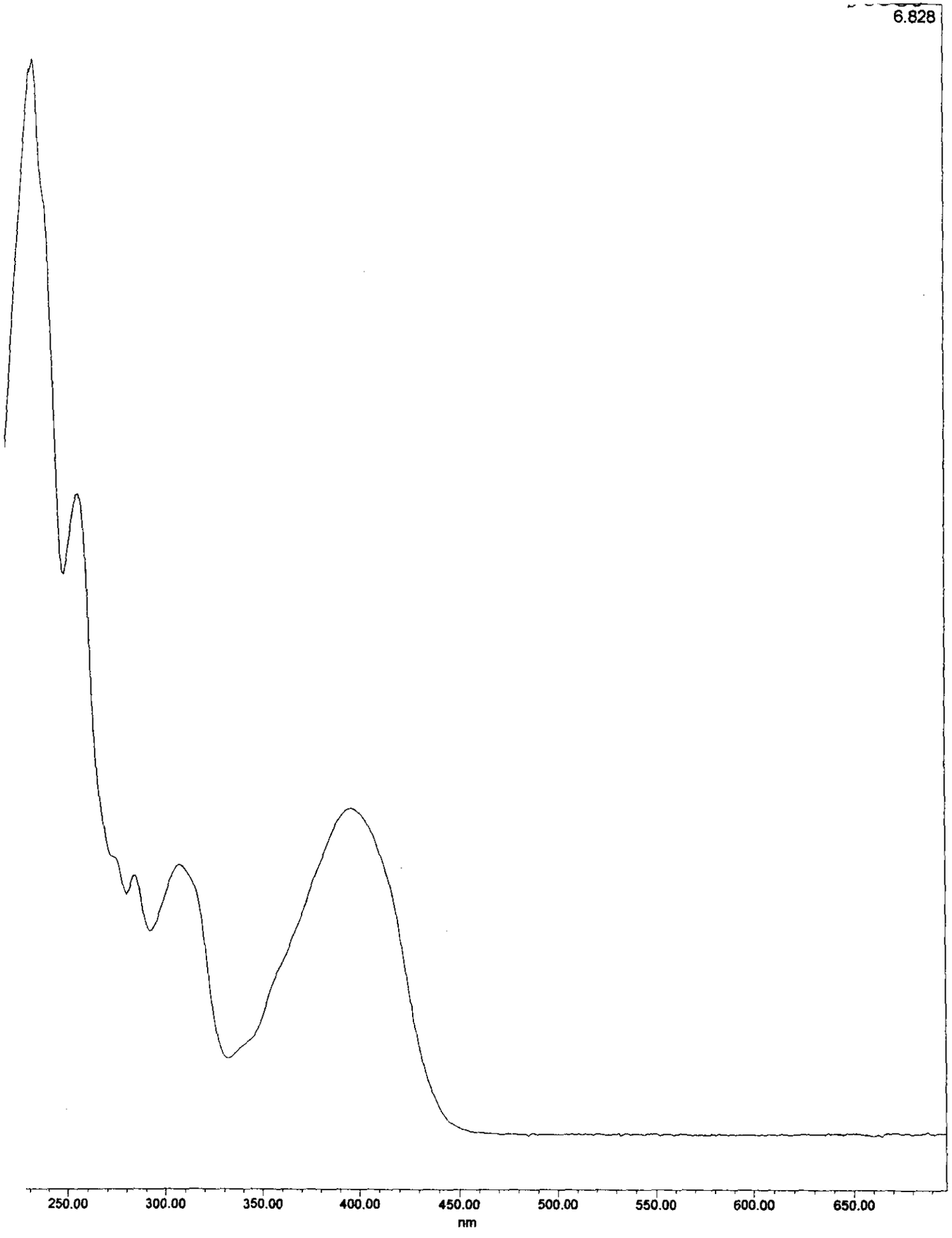

D0013
@ 280nm
Benzanthrone
6.772
2.745
3.038
12.083
AU
Minutes
0.17
0.16
0.15
0.14
0.13
0.12
0.11
0.10
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0.00
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
22.00
24.00

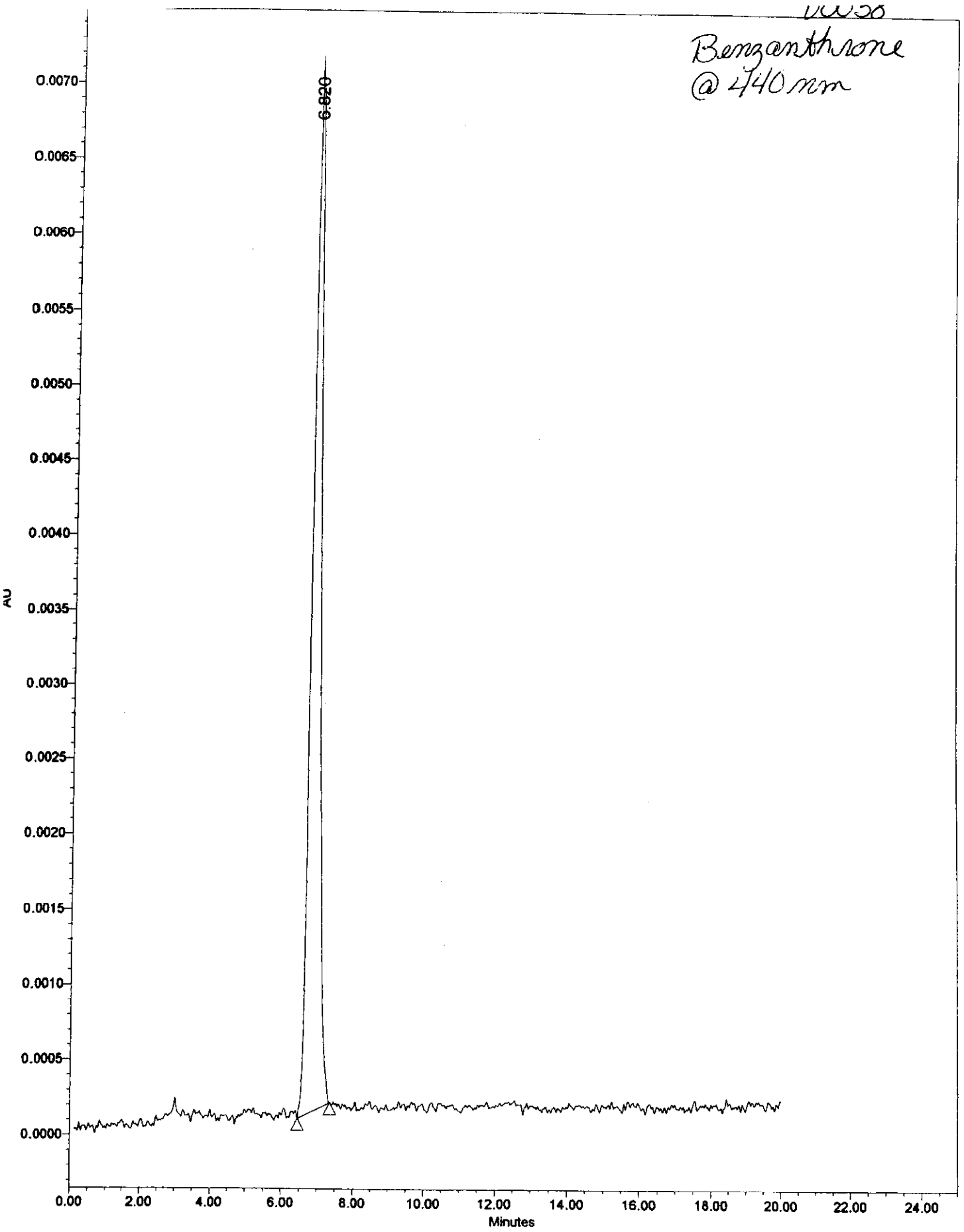

6.768
D0014
Benzanthrone
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

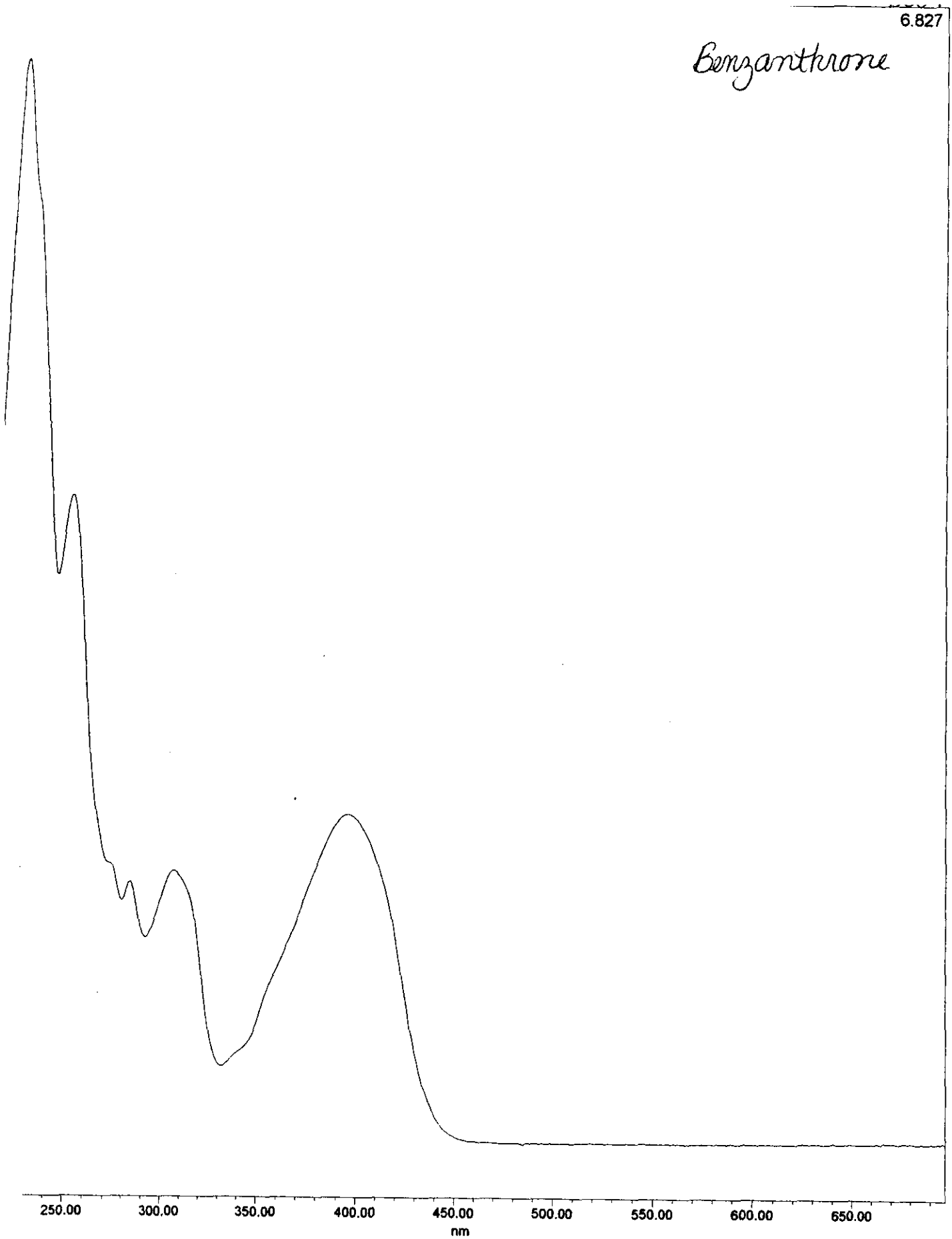

D0014
@ 280nm
Benzanthrone
6.768
AU
Minutes
0.21
0.20
0.19
0.18
0.17
0.16
0.15
0.14
0.13
0.12
0.11
0.10
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0.00
-0.01
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
22.00
24.00


6.766
D0015
Benzanthrone
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm


D0015
@ 280nm
Benzanthrone
6.766
AU
0.075
0.070
0.065
0.060
0.055
0.050
0.045
0.040
0.035
0.030
0.025
0.020
0.015
0.010
0.005
0.000
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
22.00
24.00
Minutes

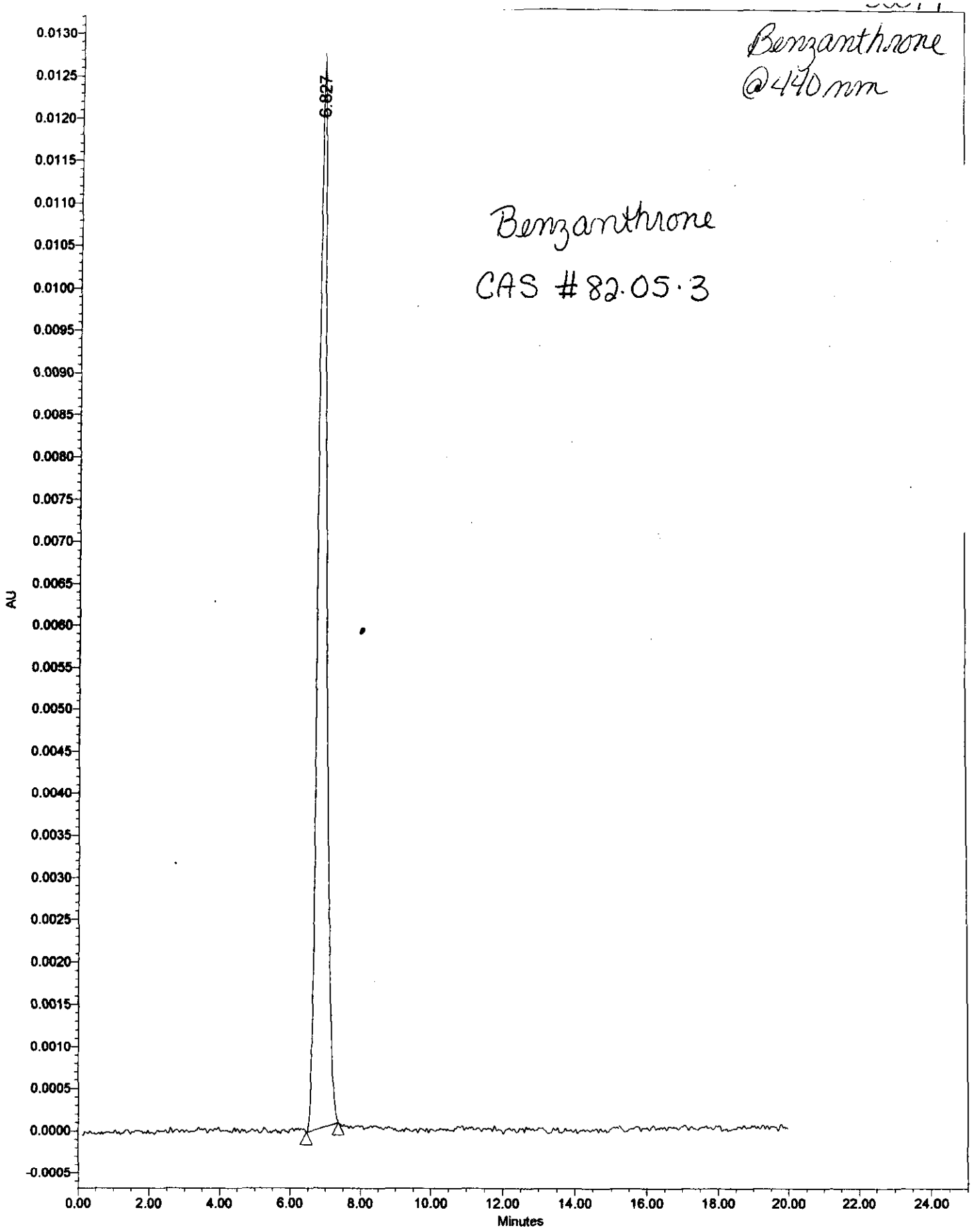

6.828
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

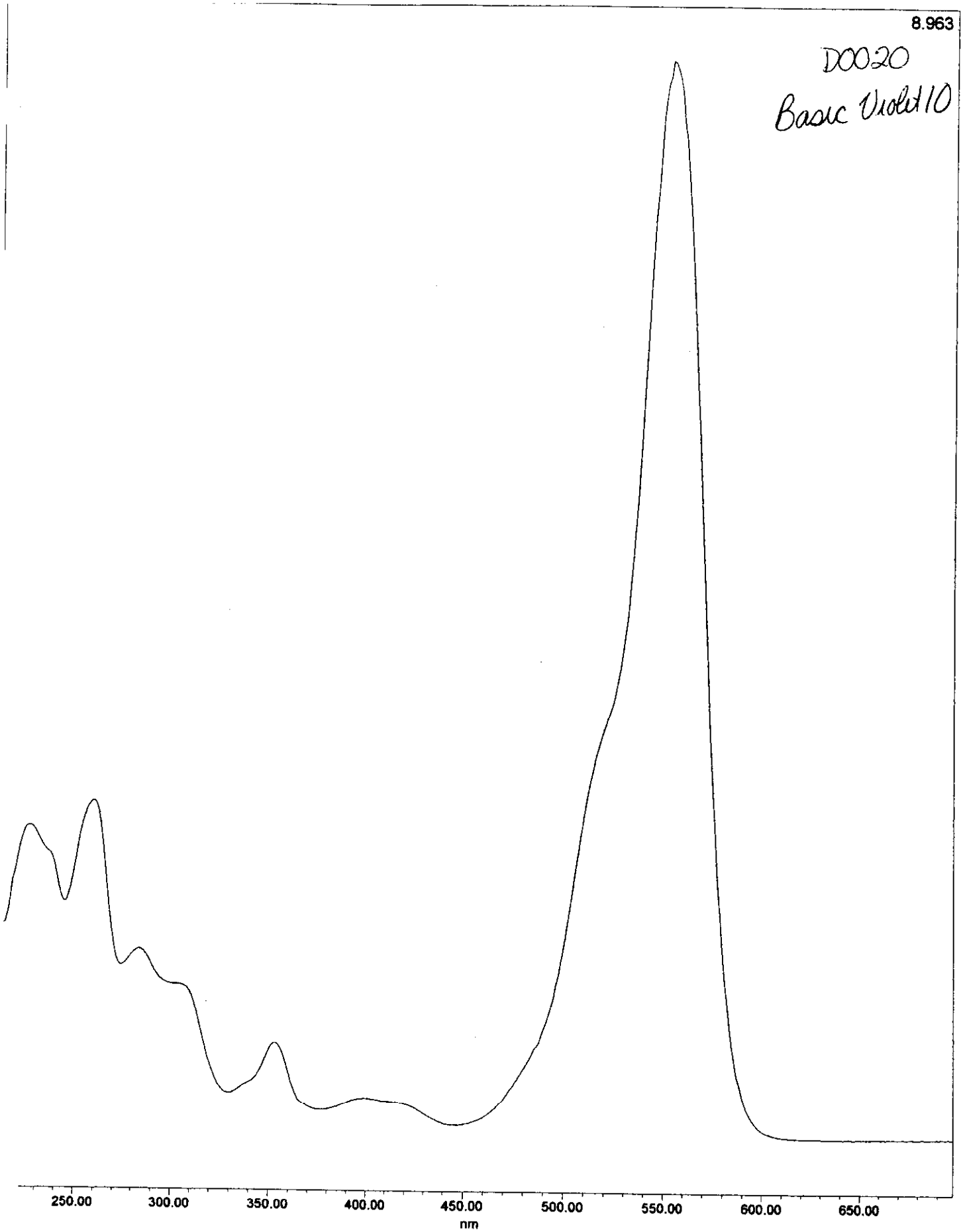

UW30
Benzanthrone
@ 440 nm
6.820
AU
0.0070
0.0065
0.0060
0.0055
0.0050
0.0045
0.0040
0.0035
0.0030
0.0025
0.0020
0.0015
0.0010
0.0005
0.0000
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
22.00
24.00
Minutes

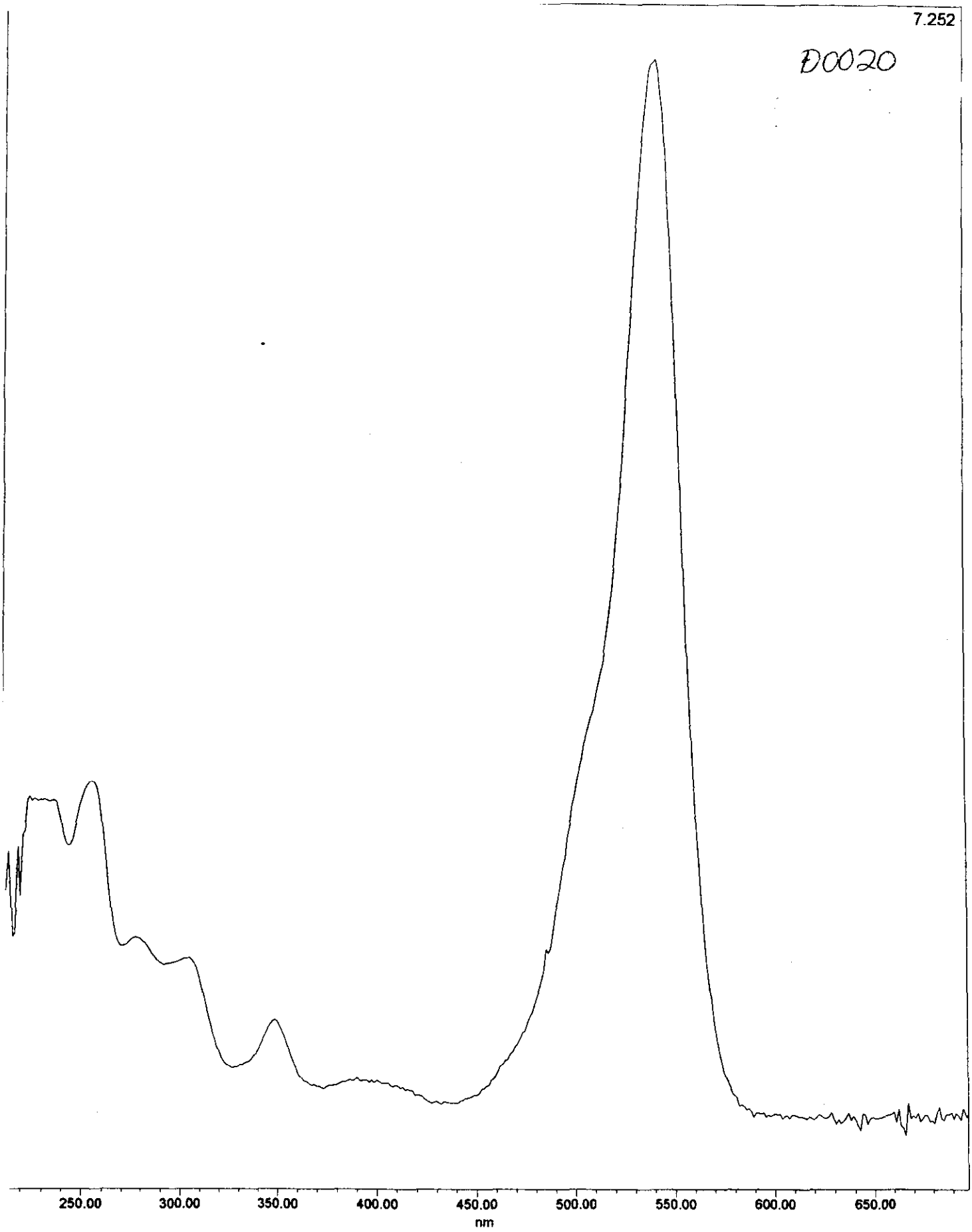

6.827
Benzanthrone
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

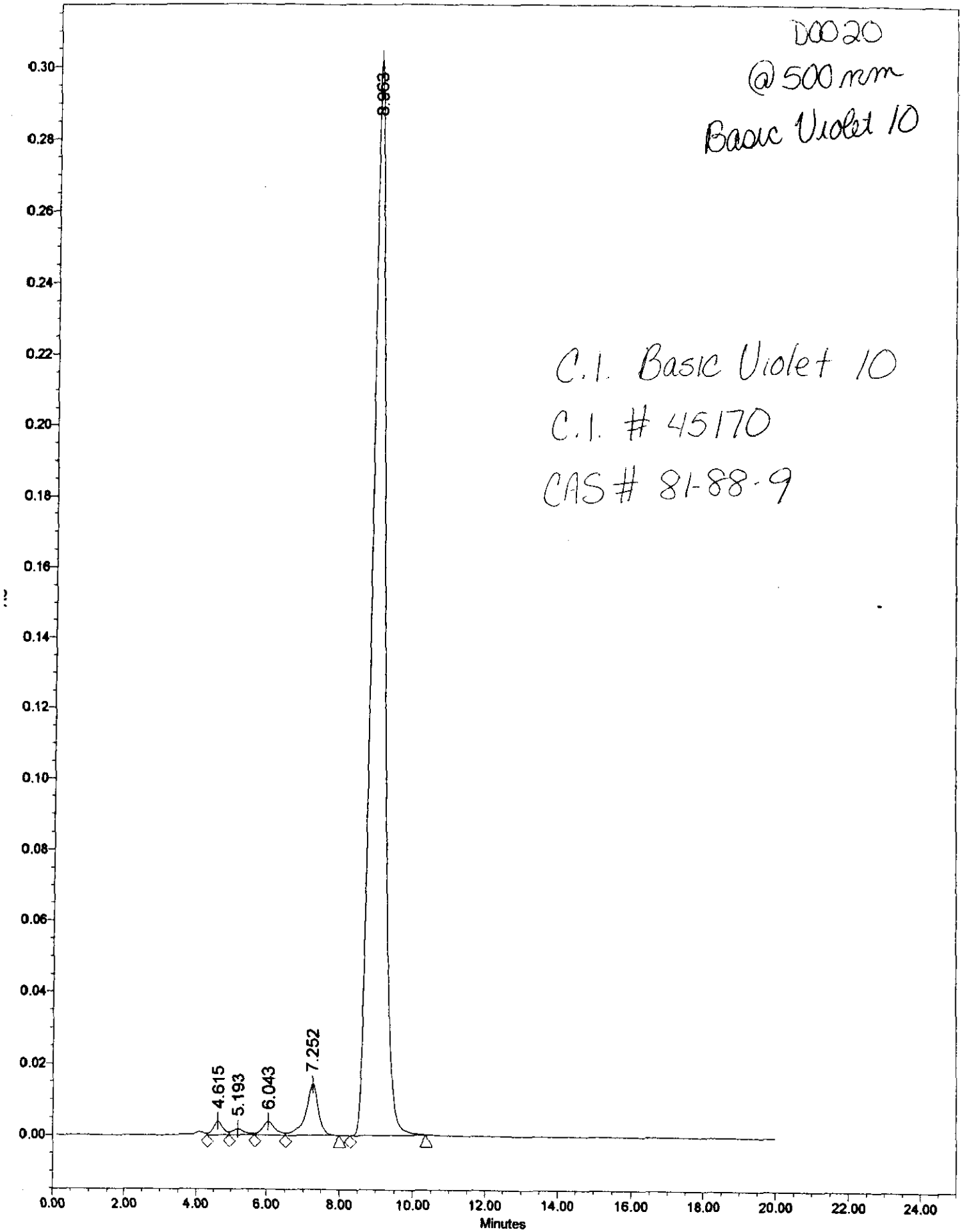
Benzanthrone
@440nm

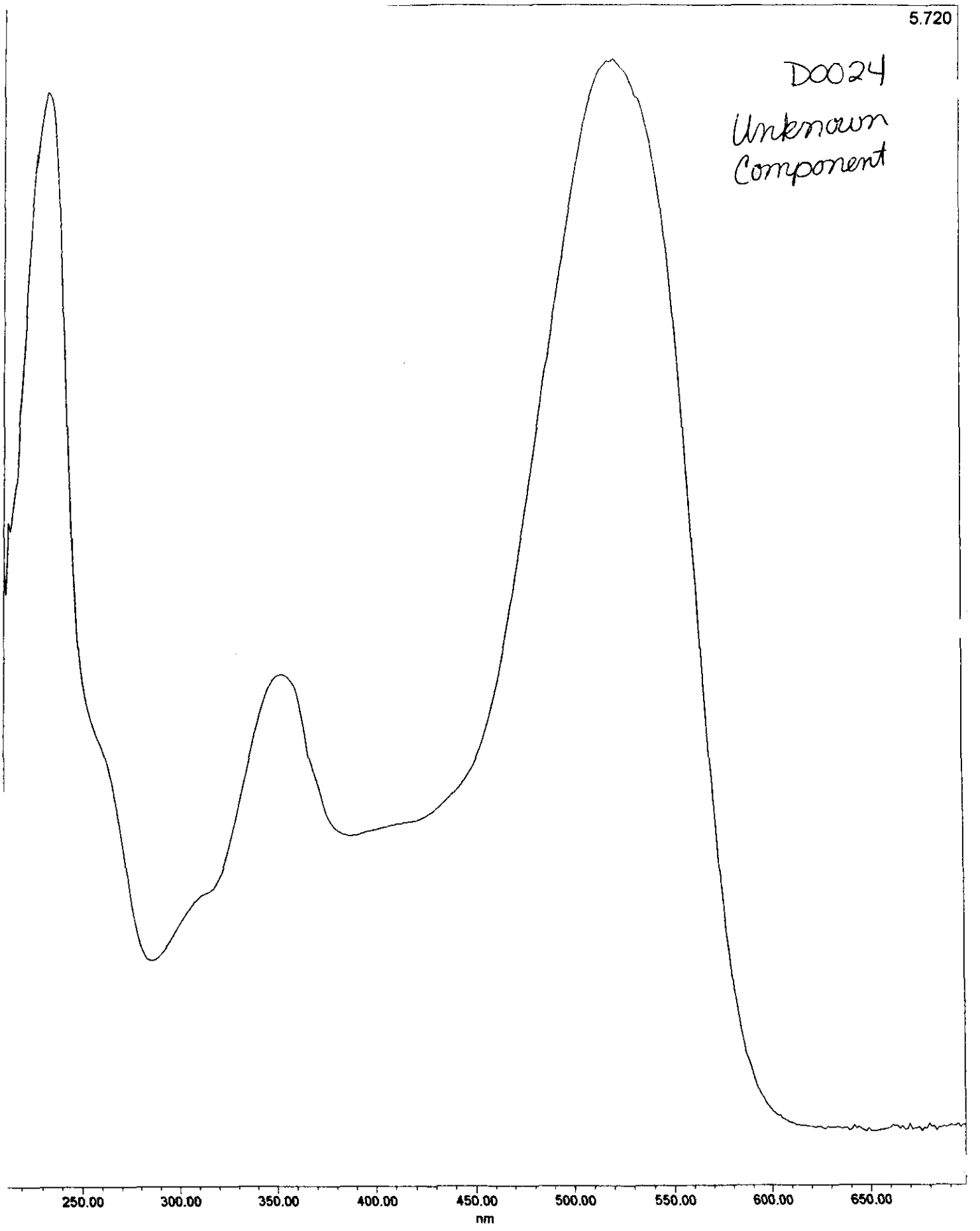
Benzanthrone
CAS #82.05.3

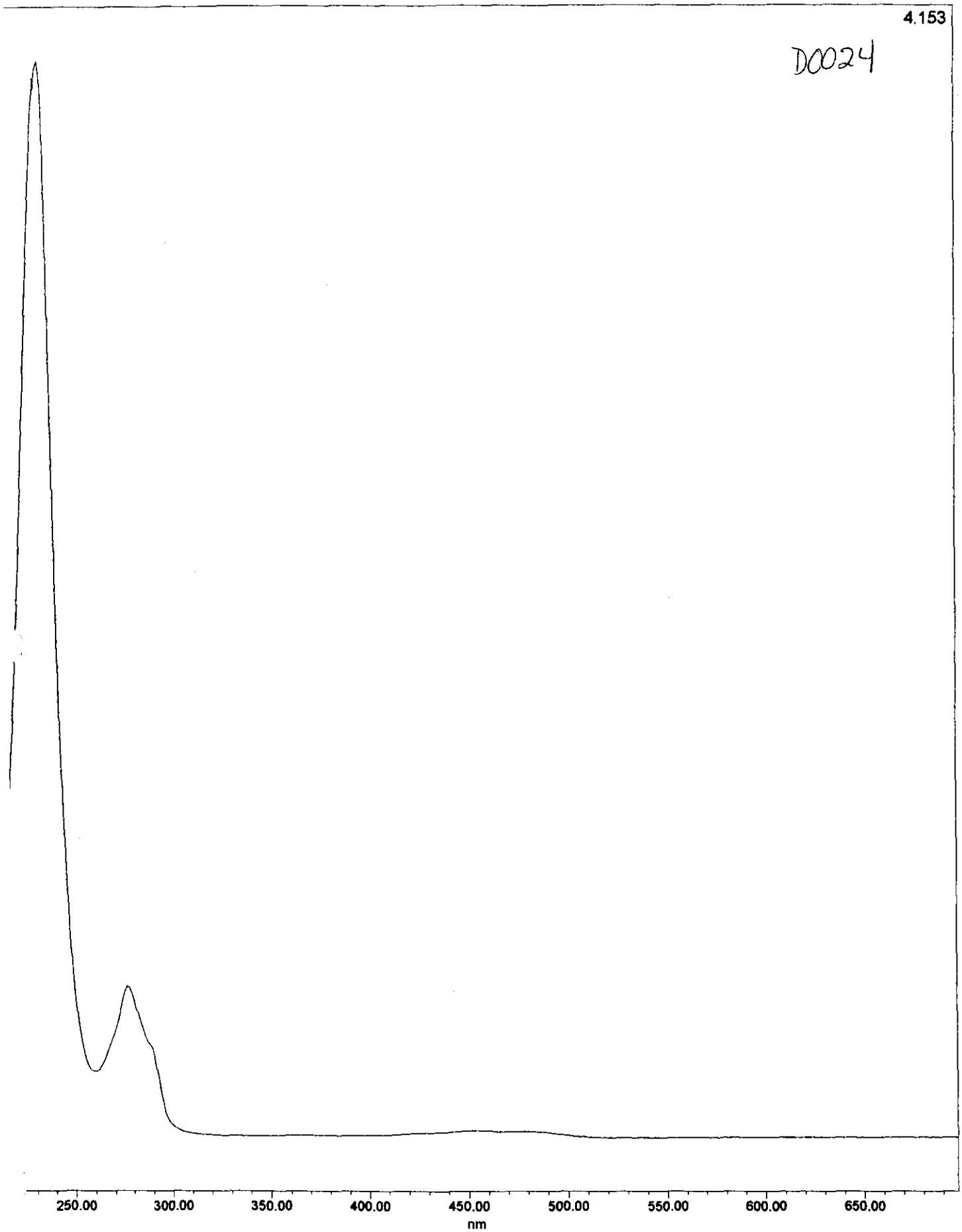



8.963
D0020
Basic Violet 10
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

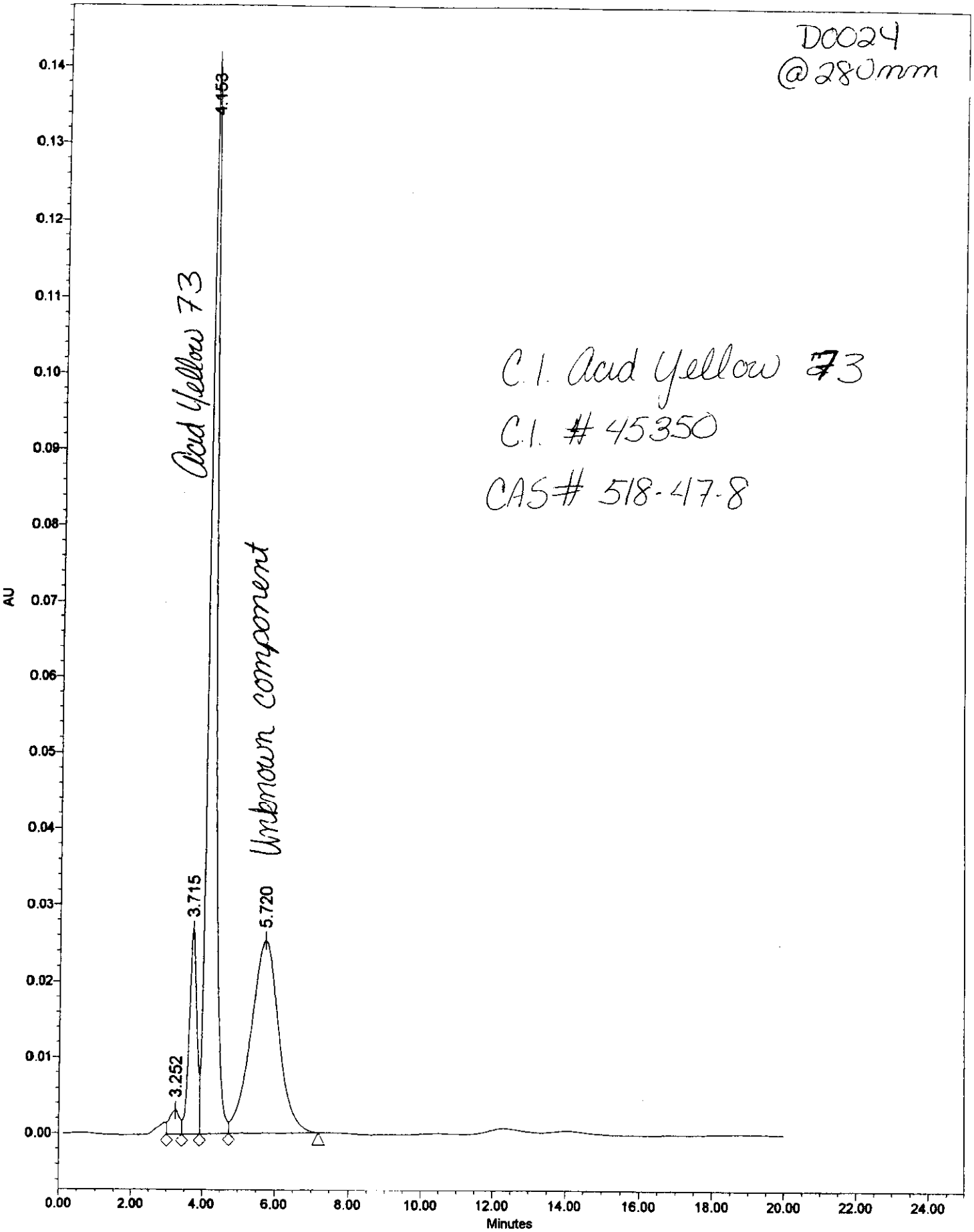

7.252
D0020
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm


D0020
@ 500 nm
Basic Violet 10
C.I. Basic Violet 10
C.I. # 45170
CAS # 81-88-9
8.963
7.252
6.043
5.193
4.615
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
22.00
24.00
Minutes

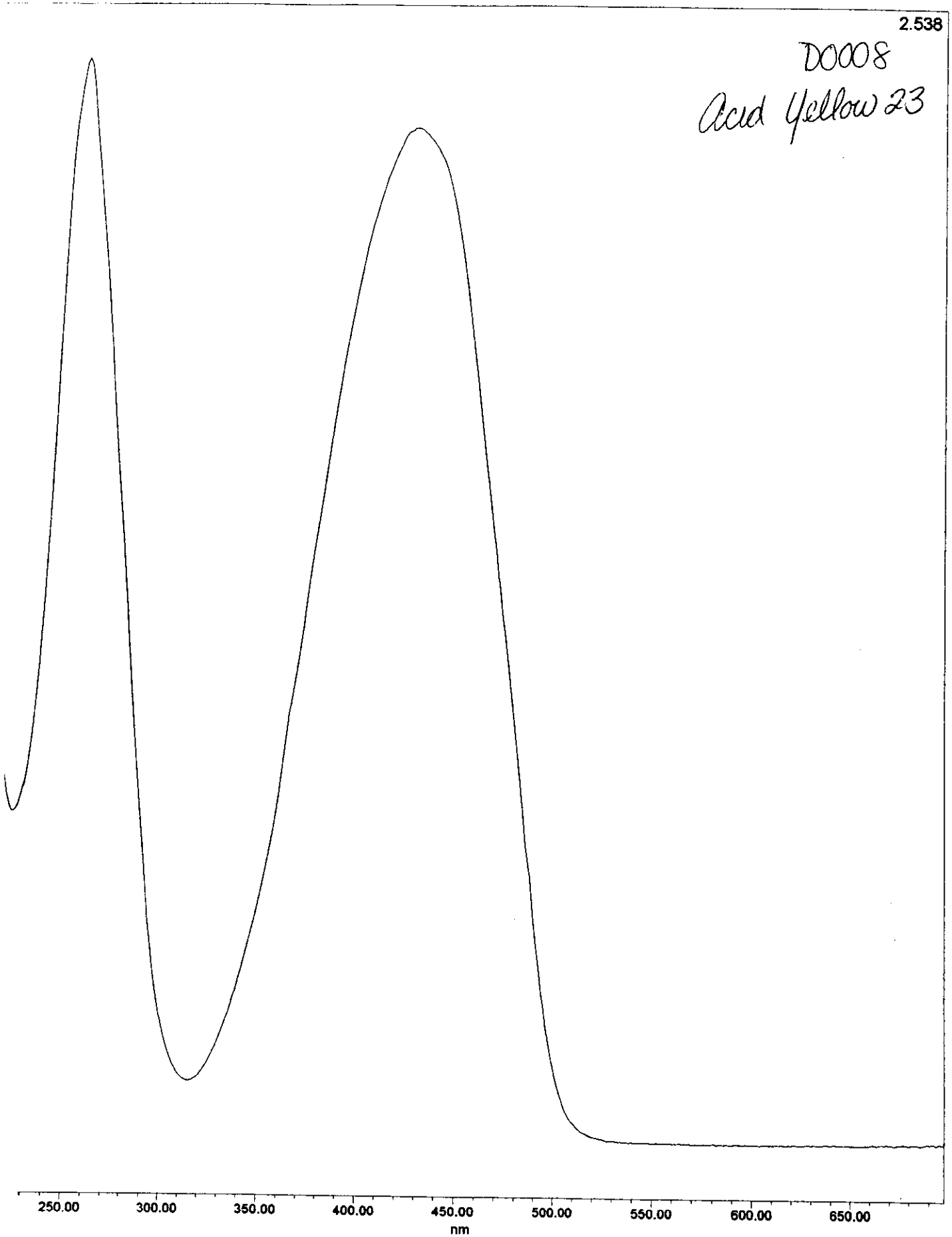

5.720
D0024
Unknown
Component
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

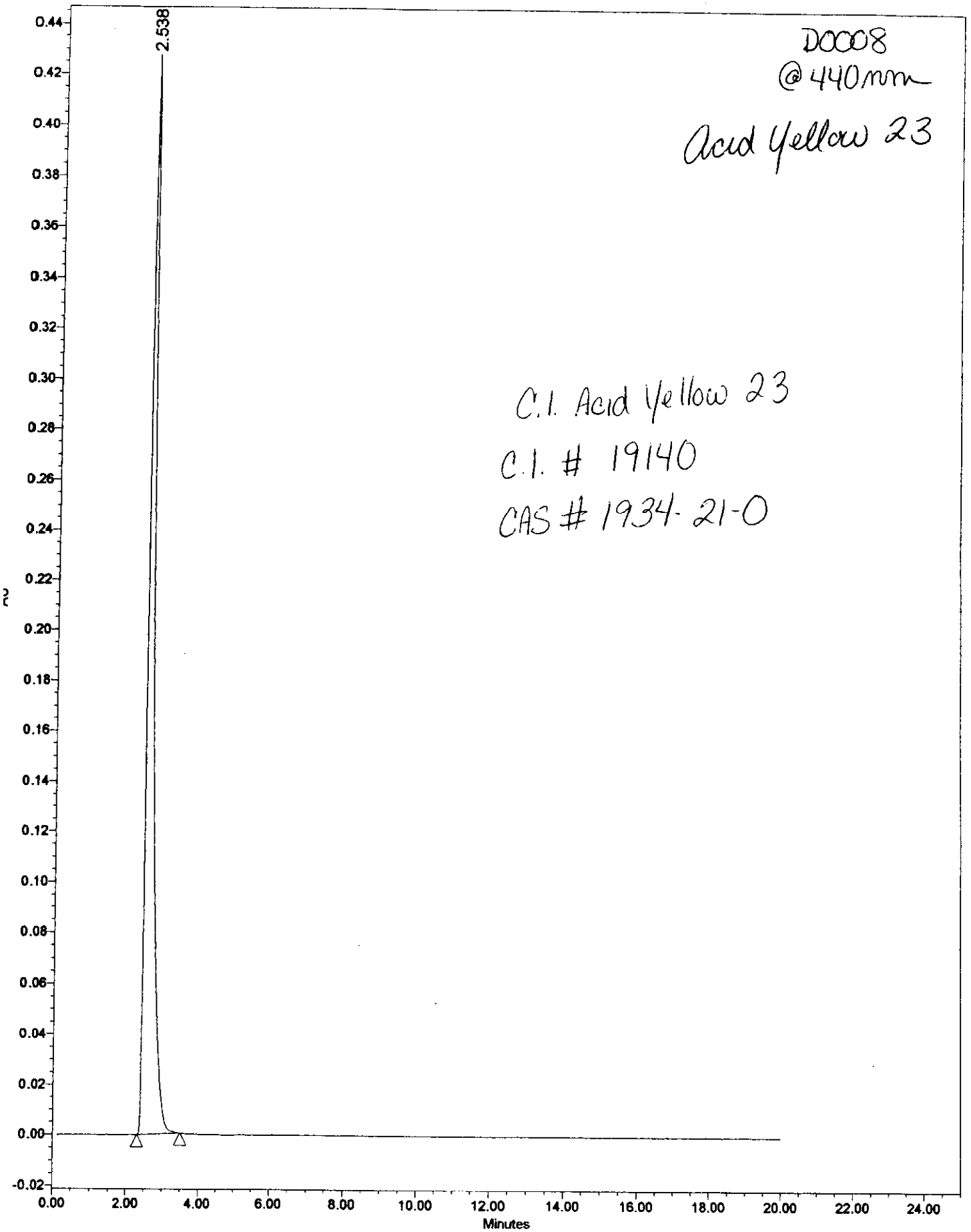

4.153
D0024
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

D0024
@280nm



C.I. Acid Yellow 73
C.I. # 45350
CAS# 518-47-8

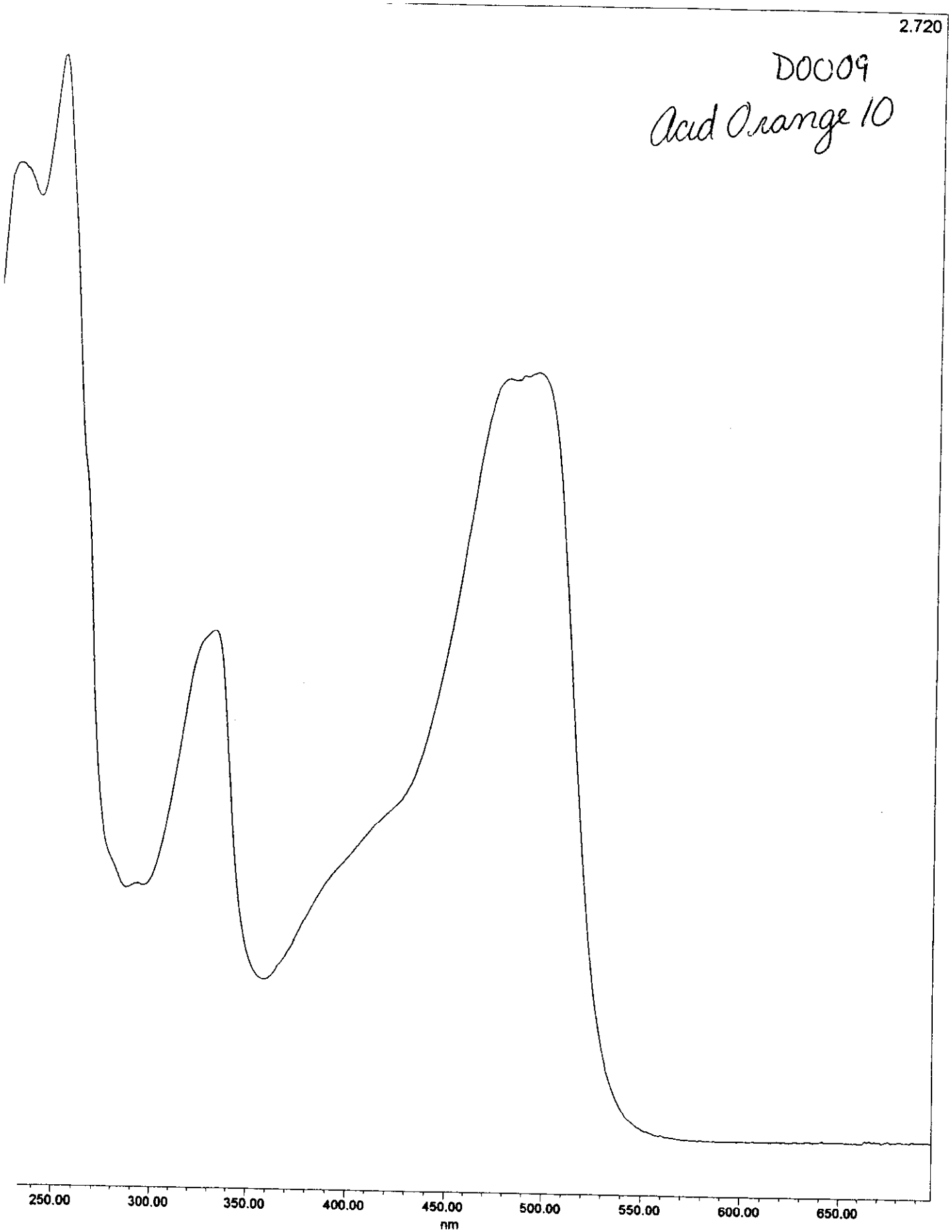

2.538
D0008
Acid Yellow 23
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

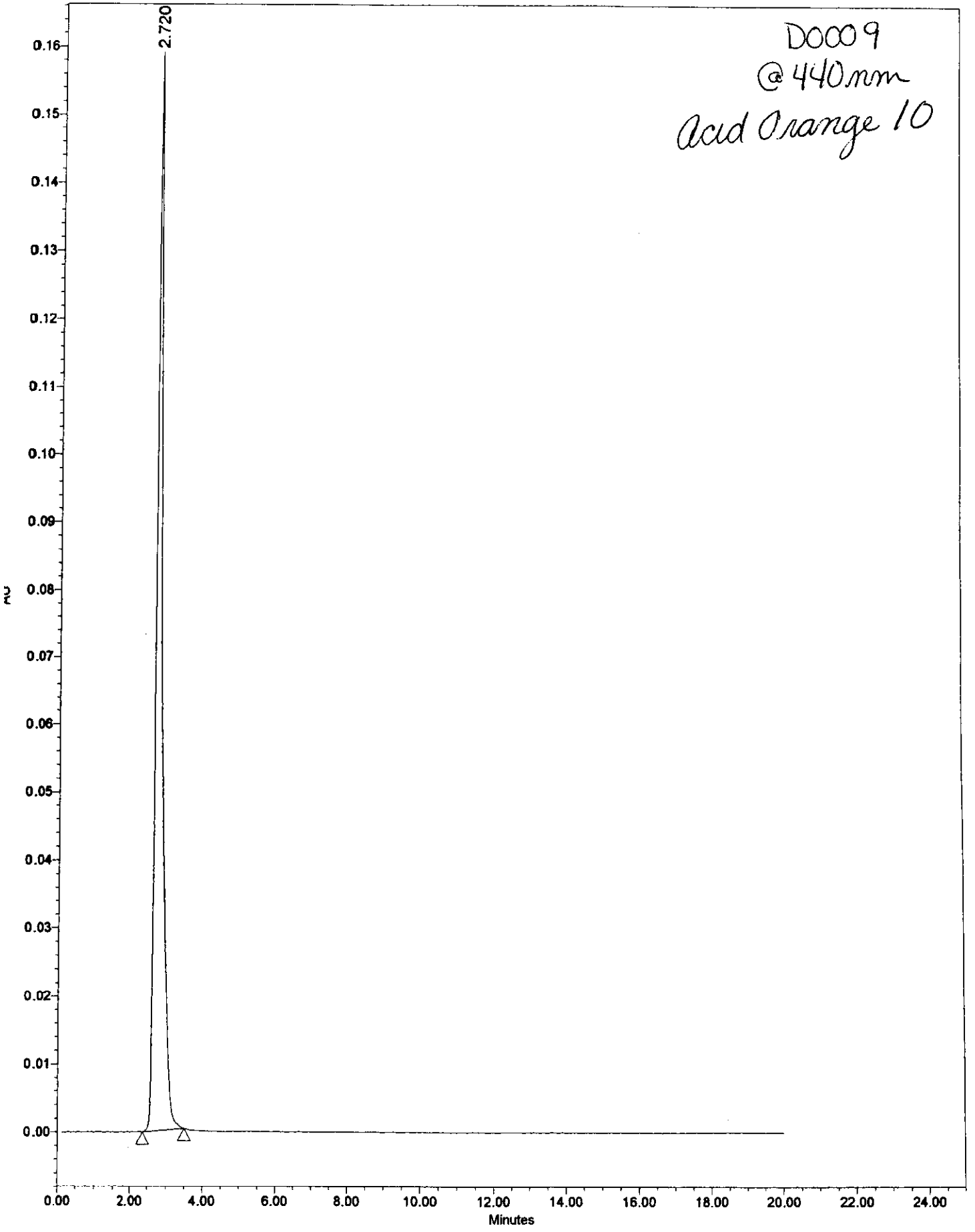


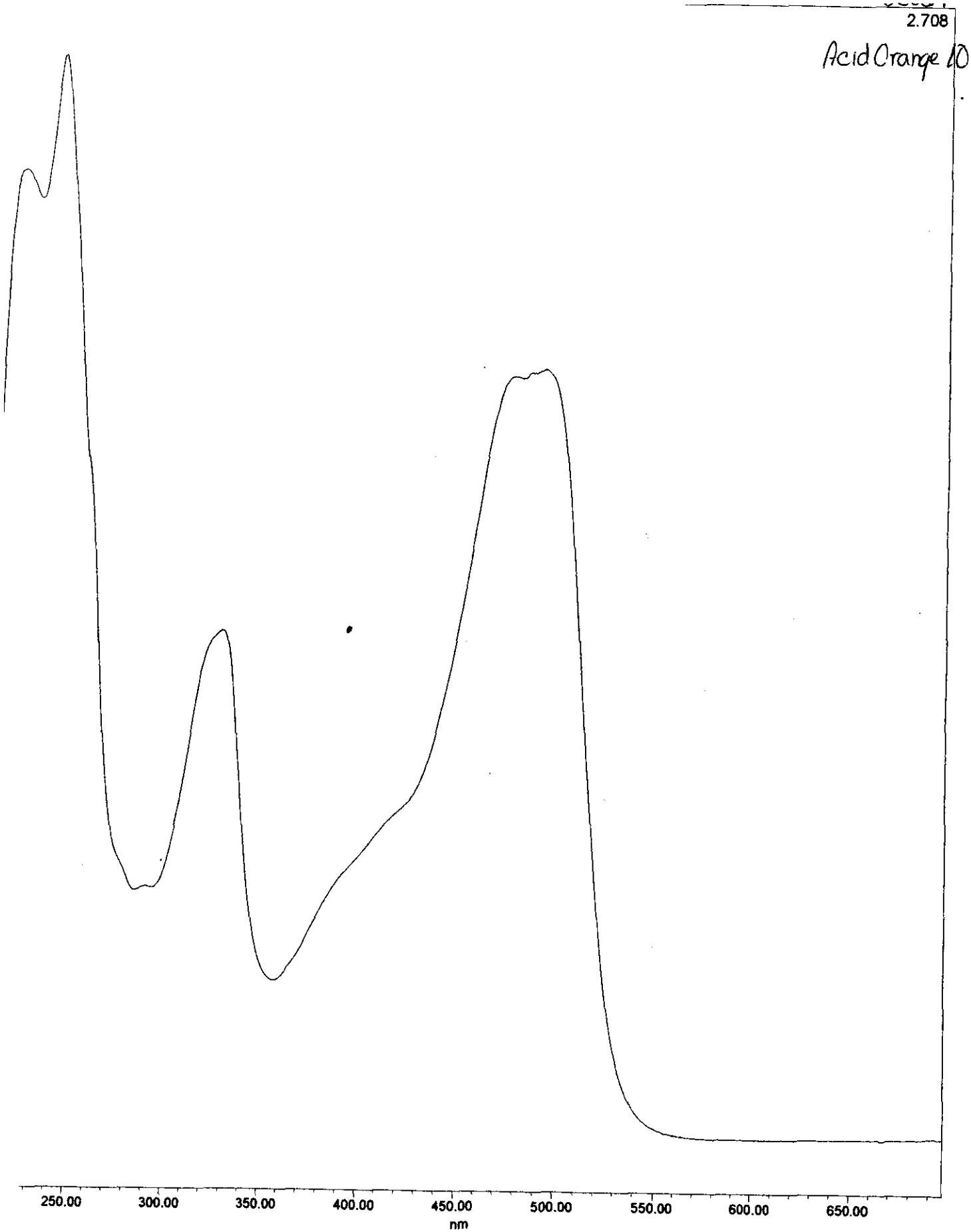
D0008
@ 440nm

Acid Yellow 23

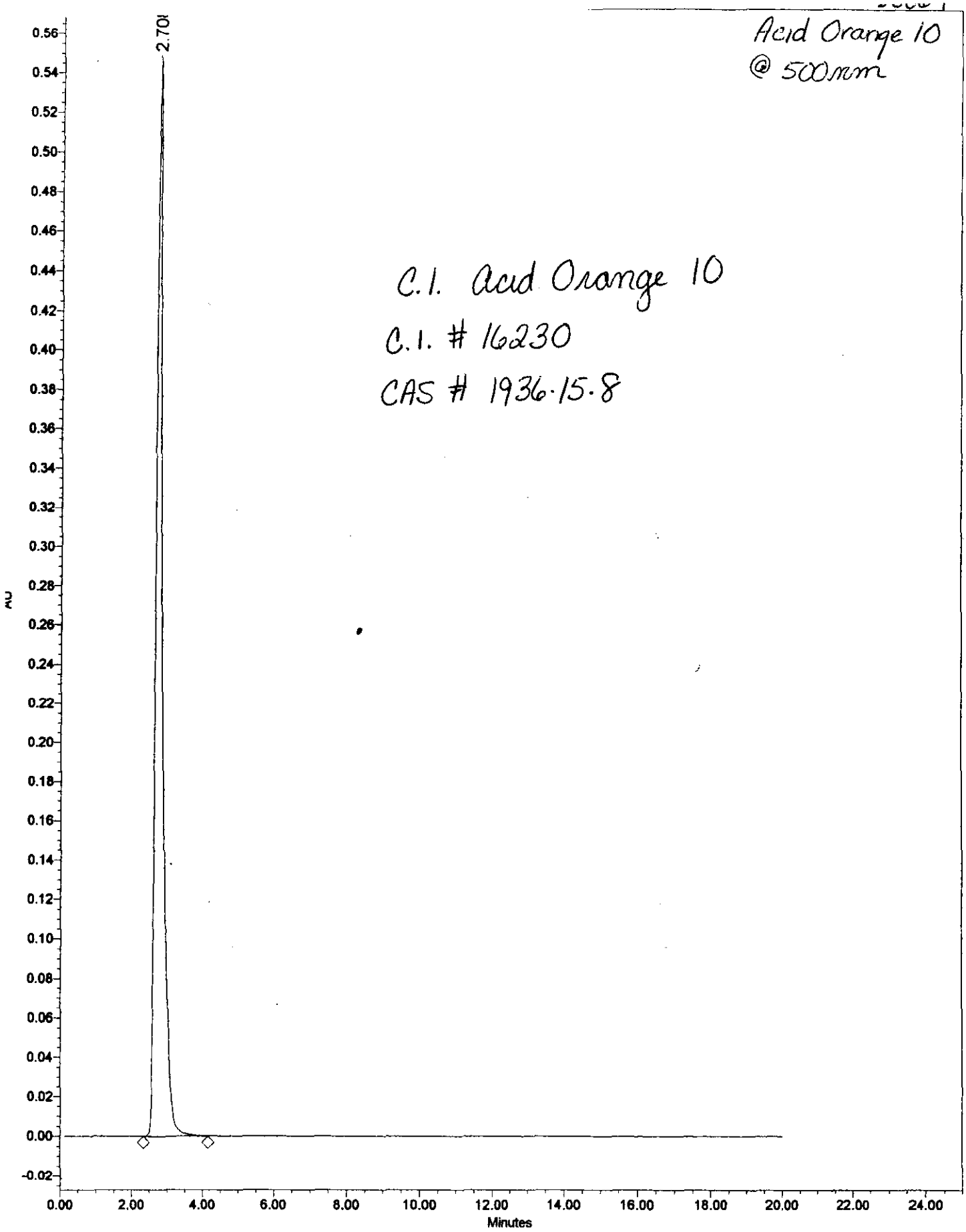
C.I. Acid Yellow 23
C.I. # 19140
CAS # 1934-21-0


2.720
D0009
Acid Orange 10
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

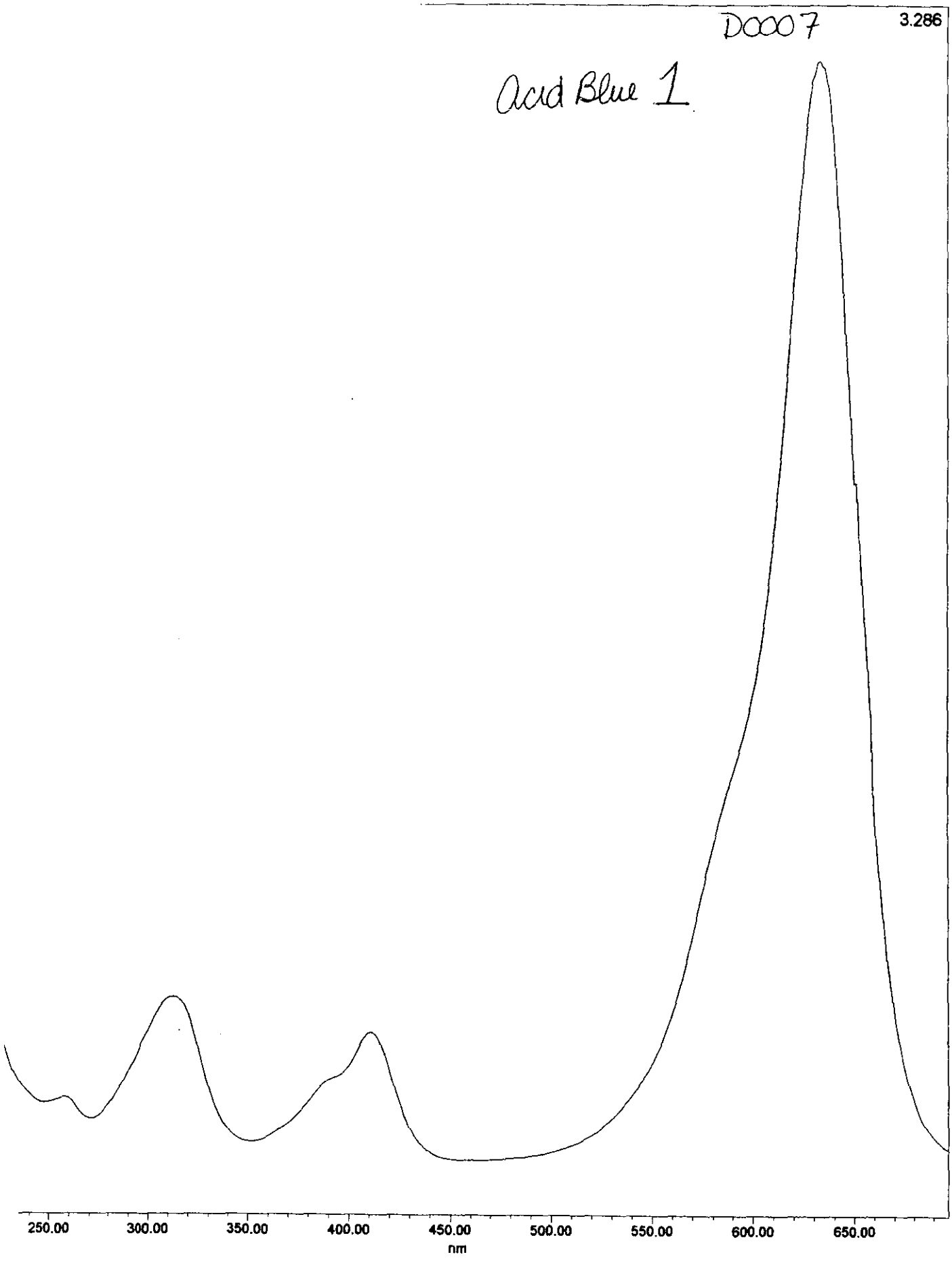

D0009
@440nm
Acid Orange 10
2.720
AU
0.16
0.15
0.14
0.13
0.12
0.11
0.10
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0.00
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
22.00
24.00
Minutes

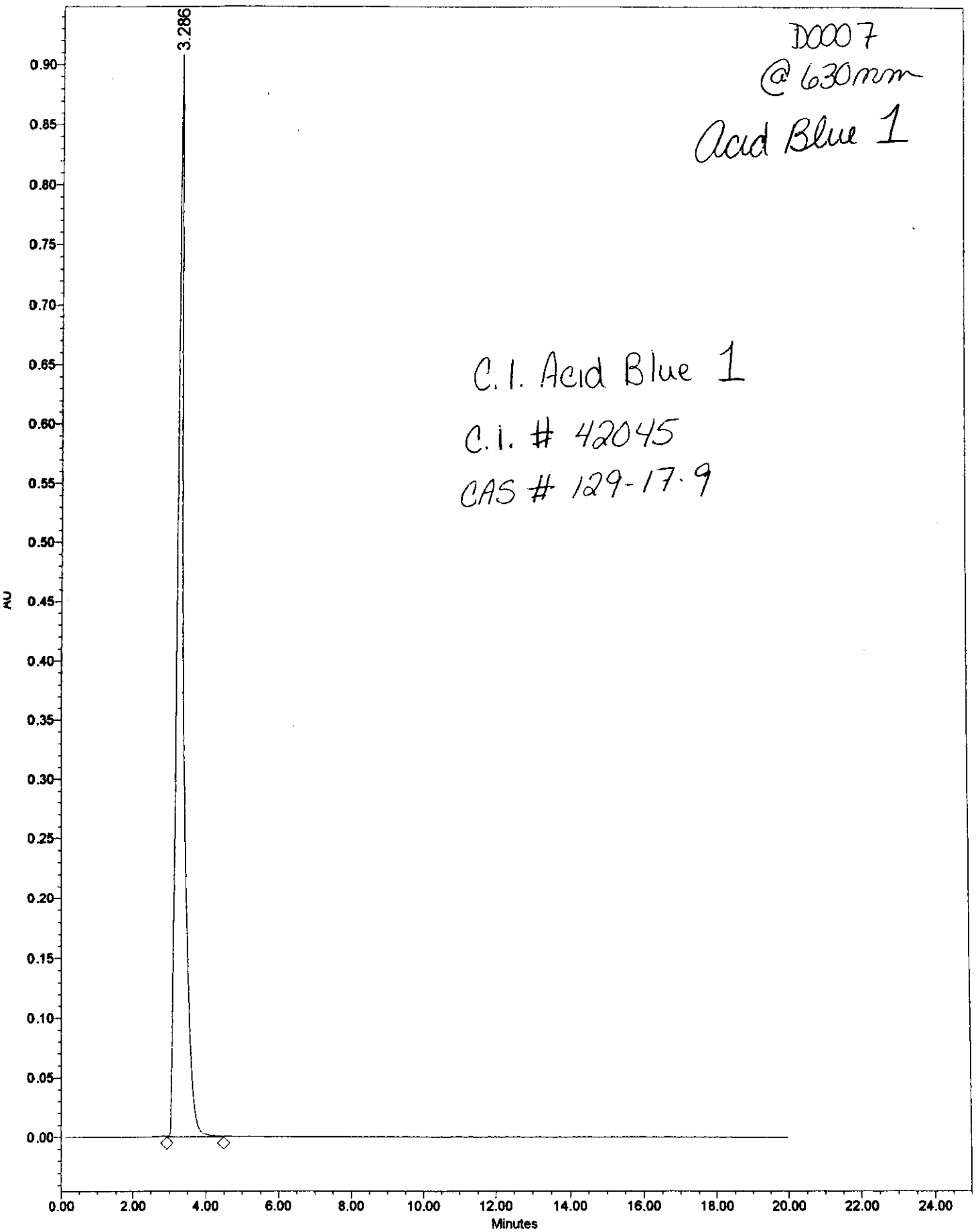

2.708
Acid Orange 10
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

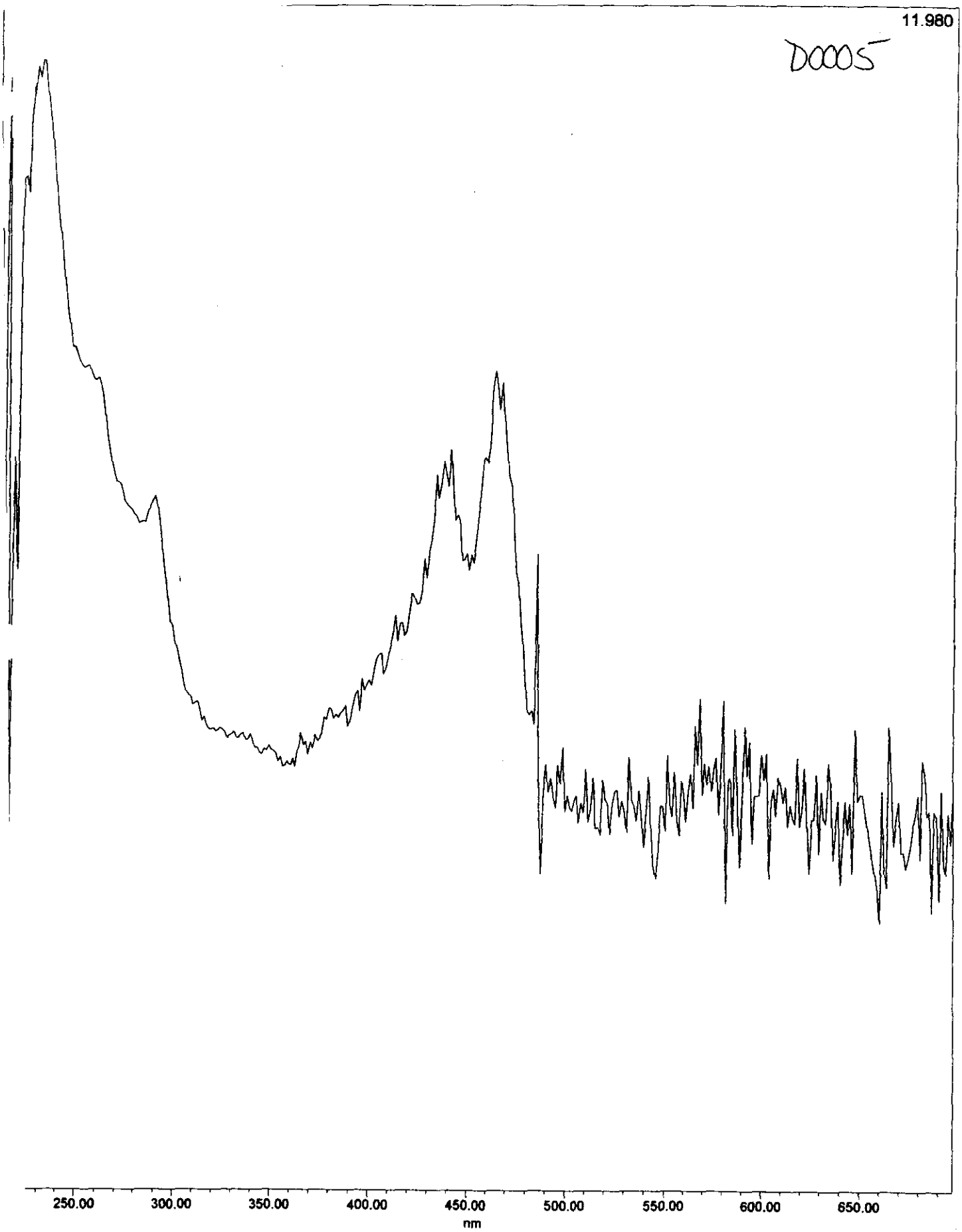
Acid Orange 10
@ 500nm



C.I. Acid Orange 10
C.I. # 16230
CAS # 1936-15-8

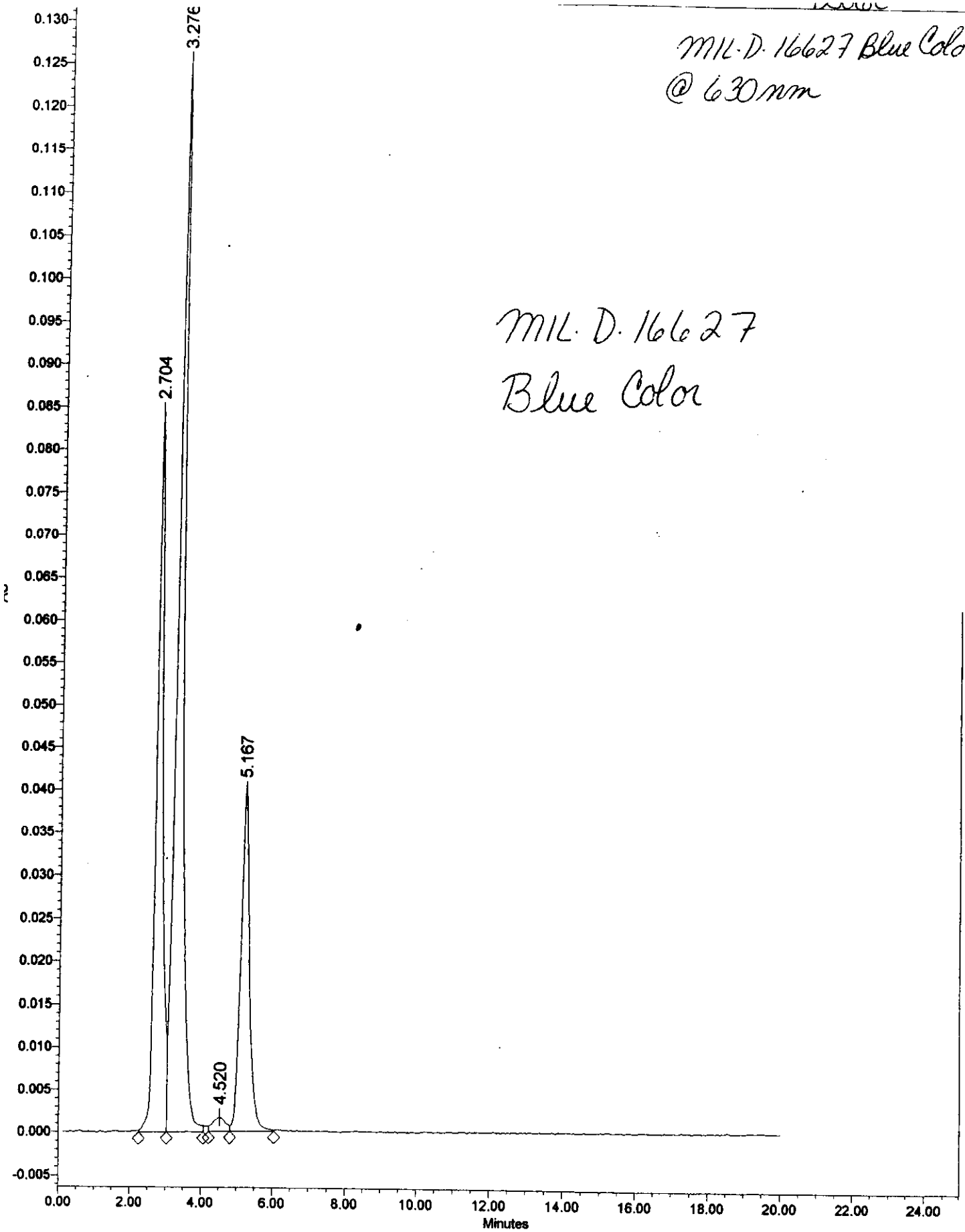

D0007
3.286
Acid Blue 1
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

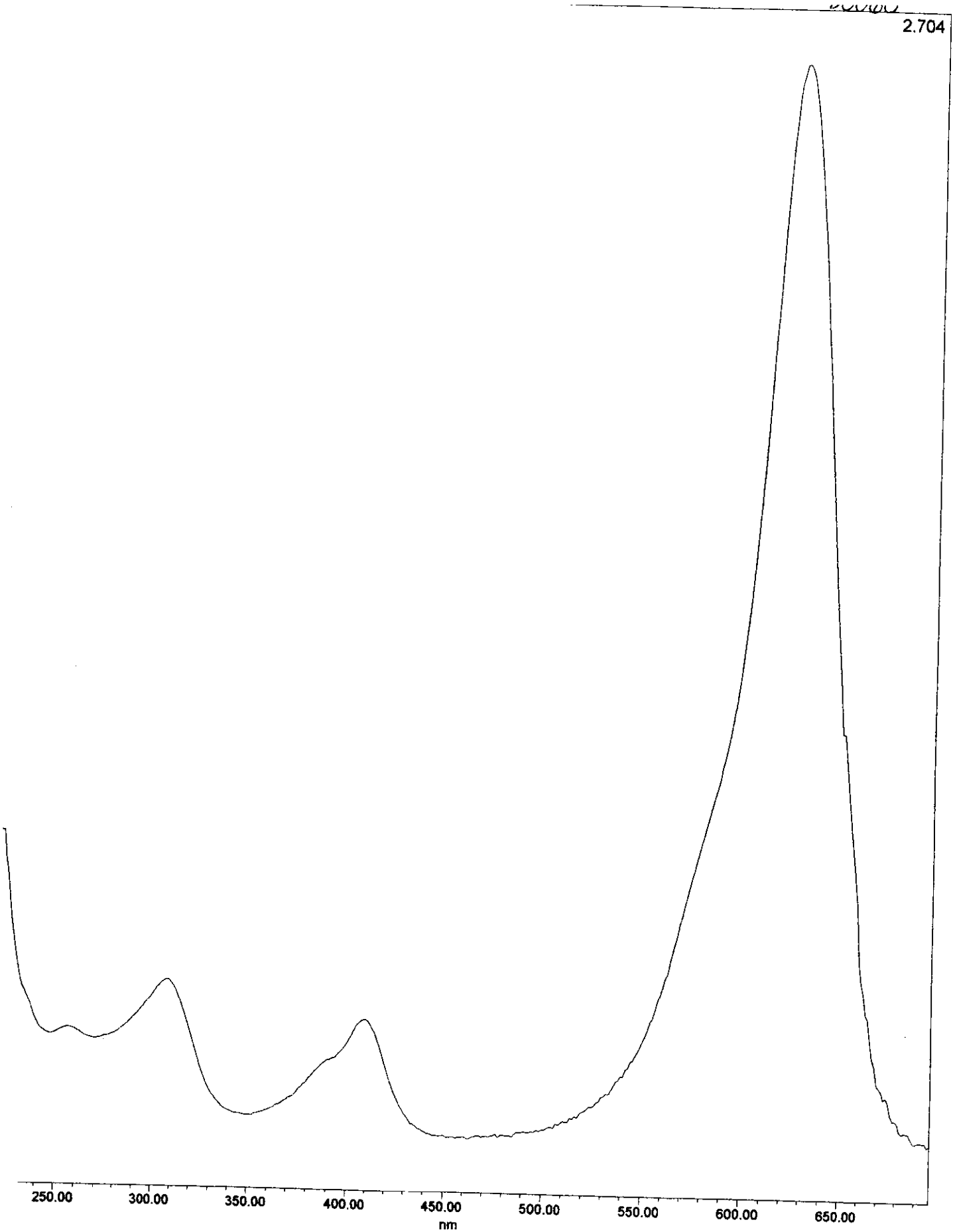

3.286
D0007
@ 630nm
Acid Blue 1
C.I. Acid Blue 1
C.I. # 42045
CAS # 129-17-9
AU
0.90
0.85
0.80
0.75
0.70
0.65
0.60
0.55
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
22.00
24.00
Minutes

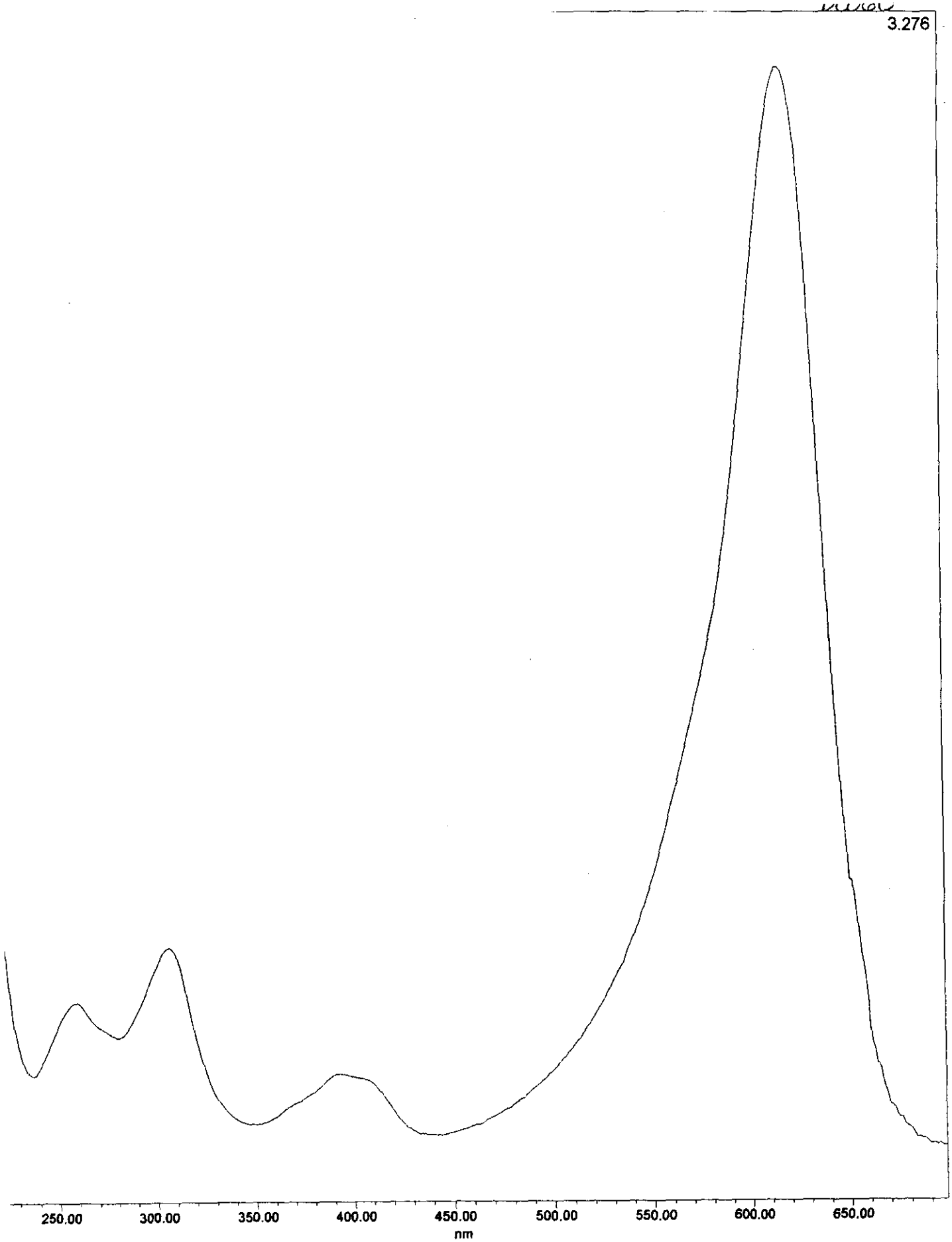

11.980
D0005
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

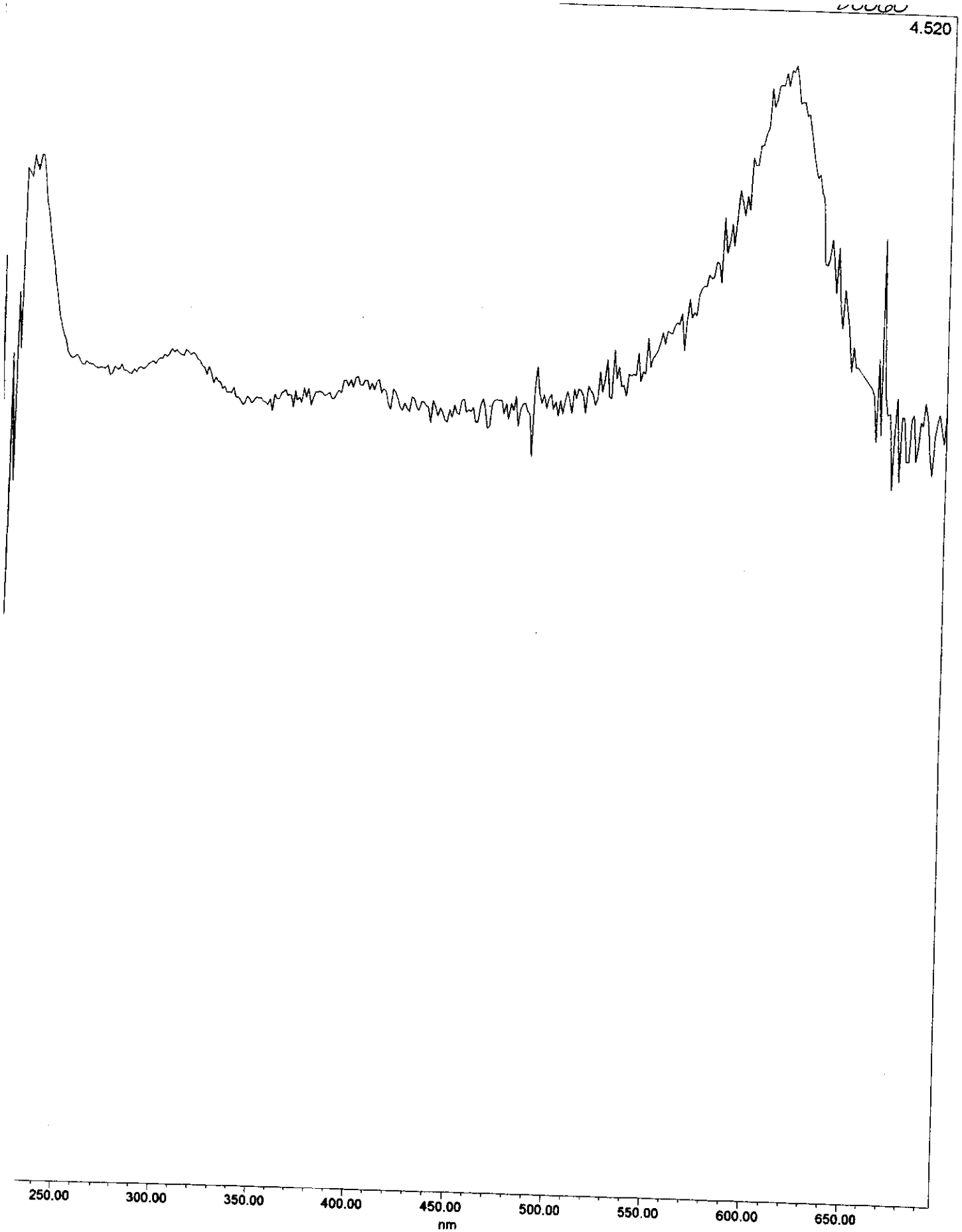
MIL-D-16627 Blue Color
@ 630nm

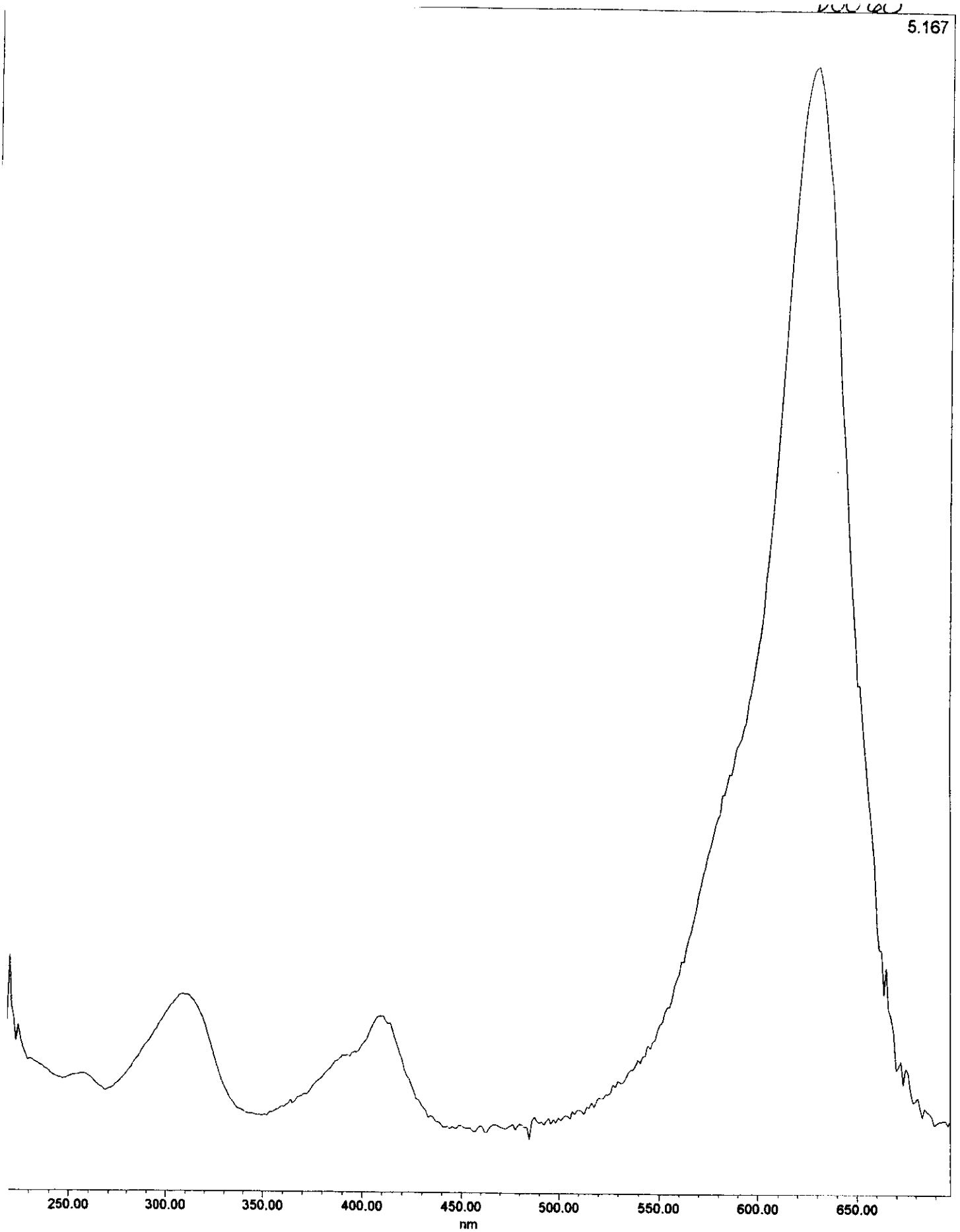
MIL-D-16627
Blue Color




2.704
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

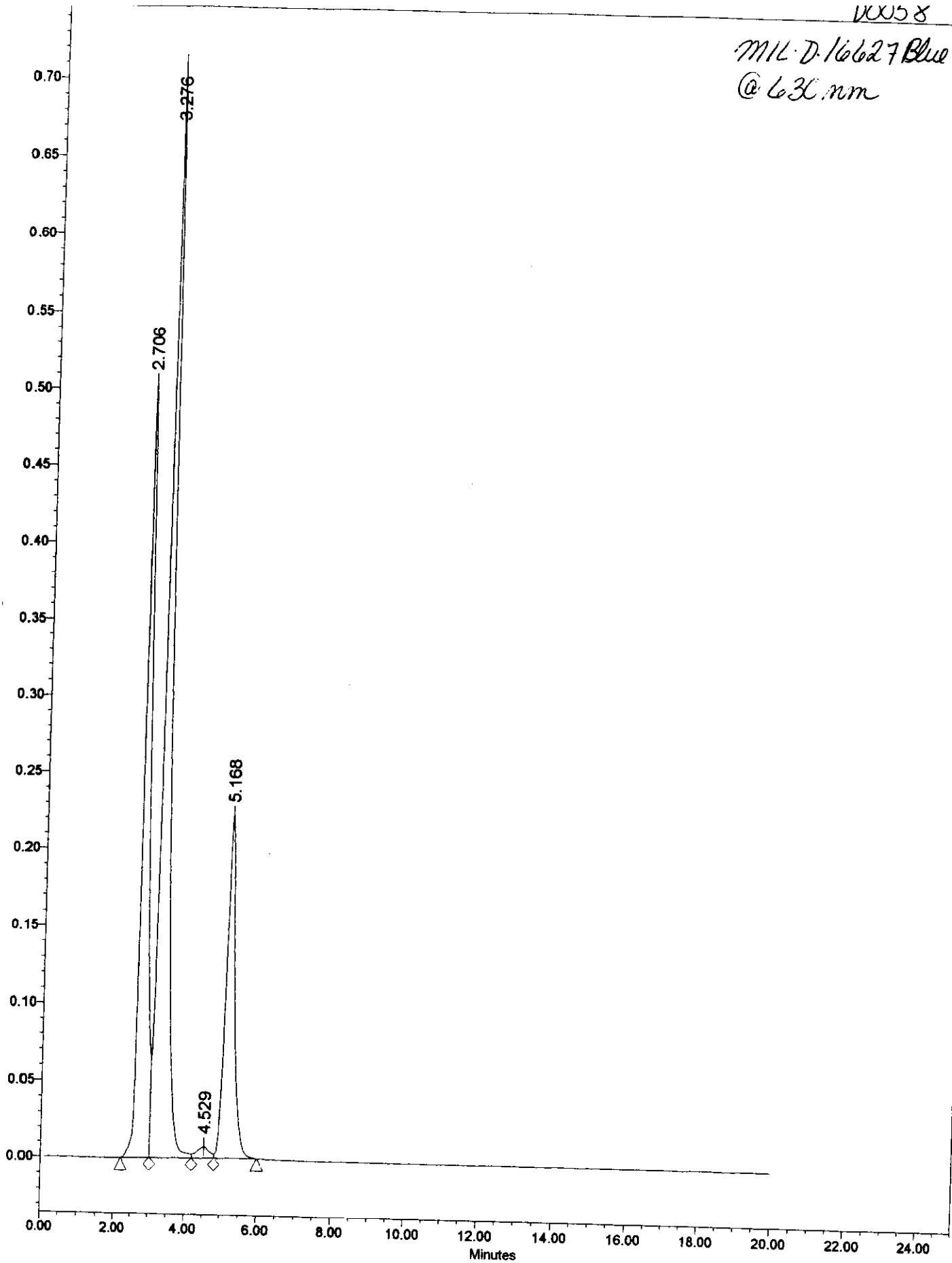

3.276
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

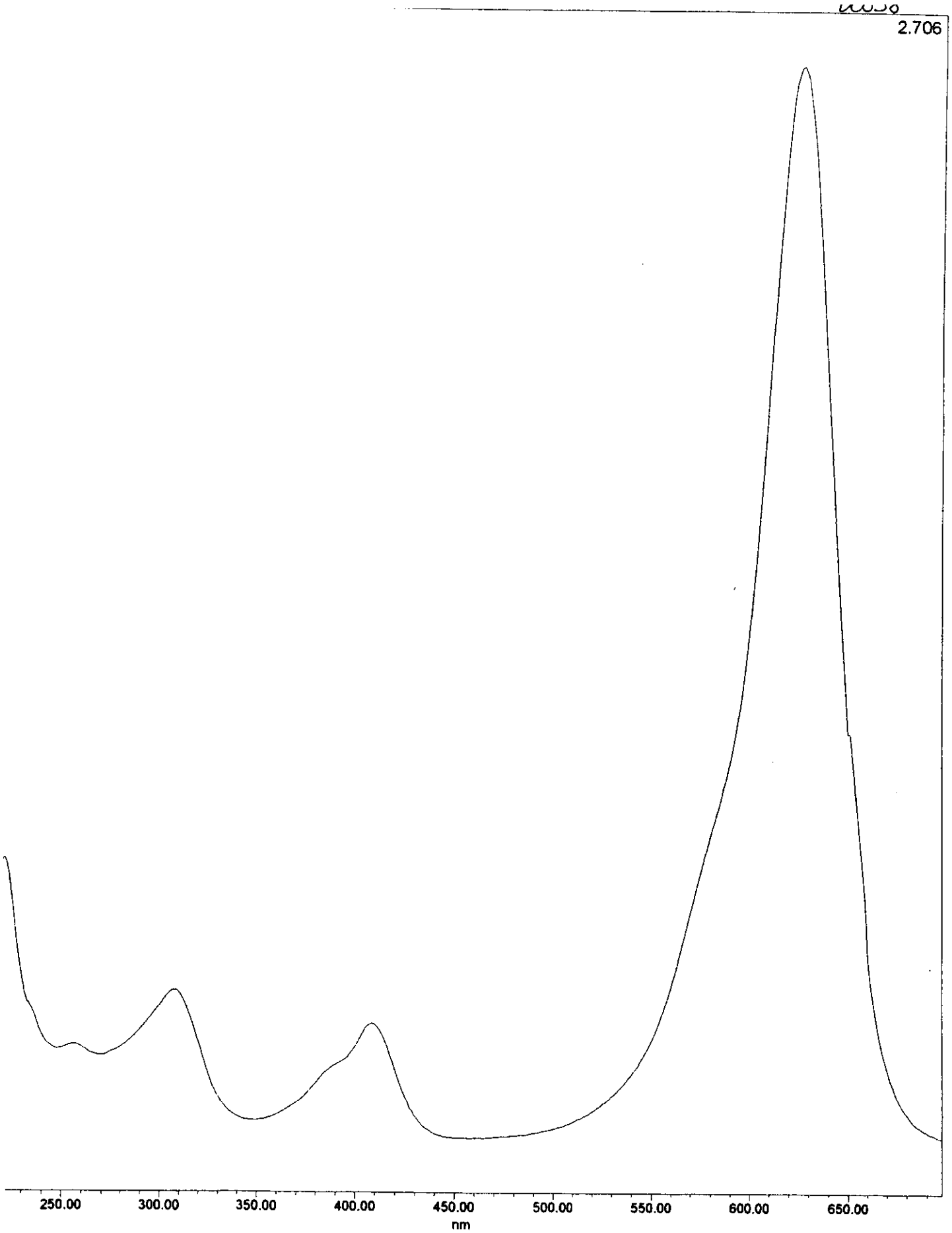

4.520
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

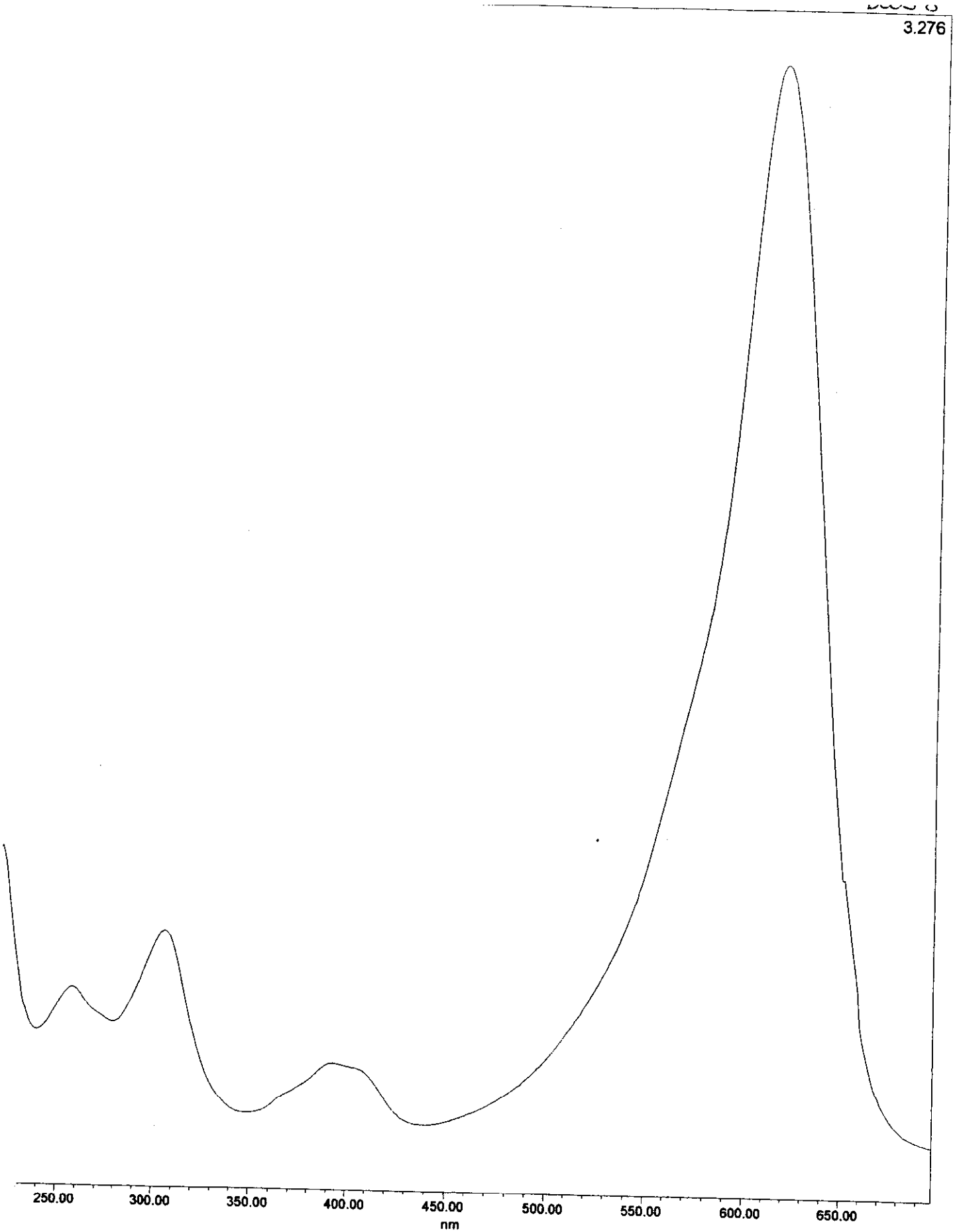

5.167
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

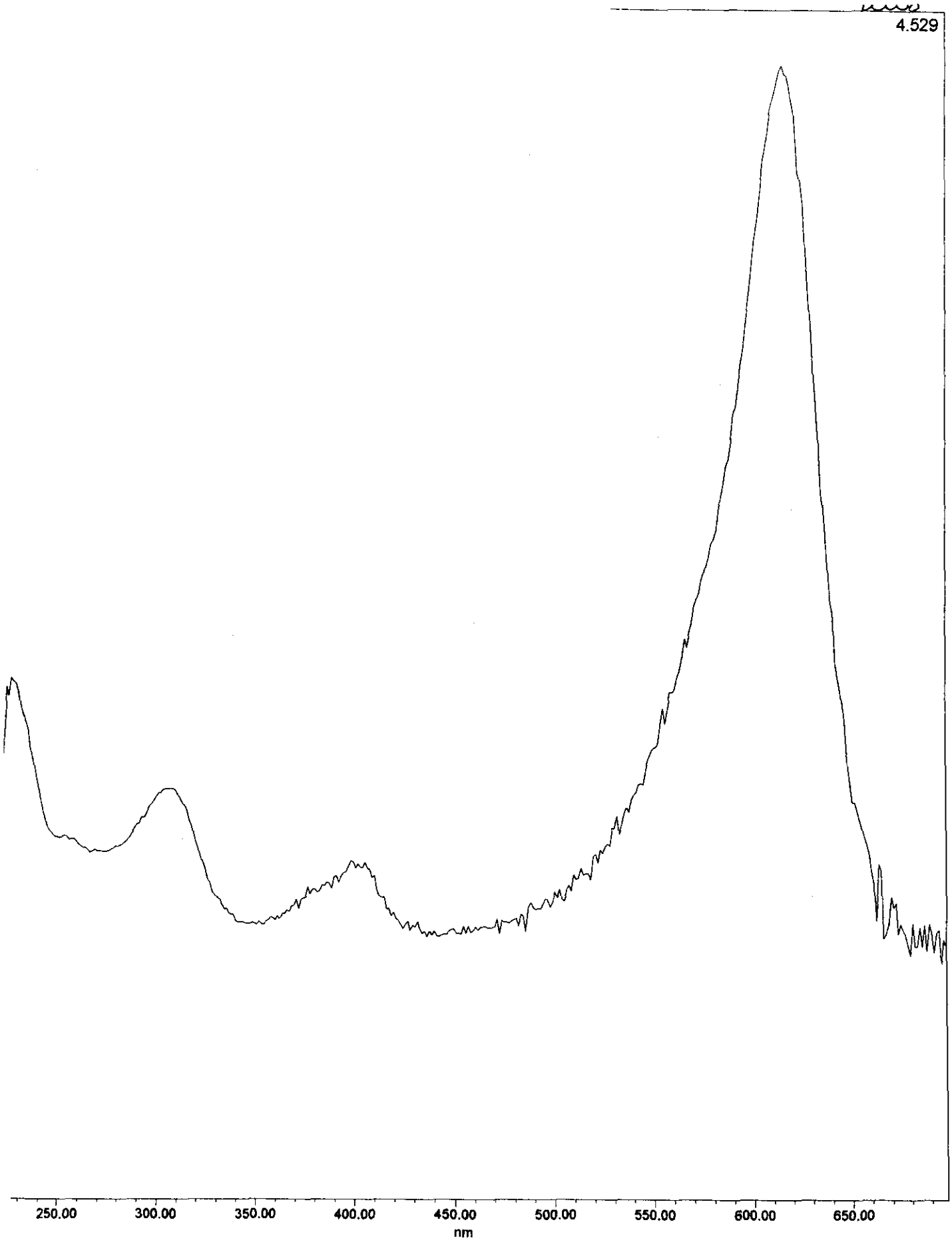
VOOO58

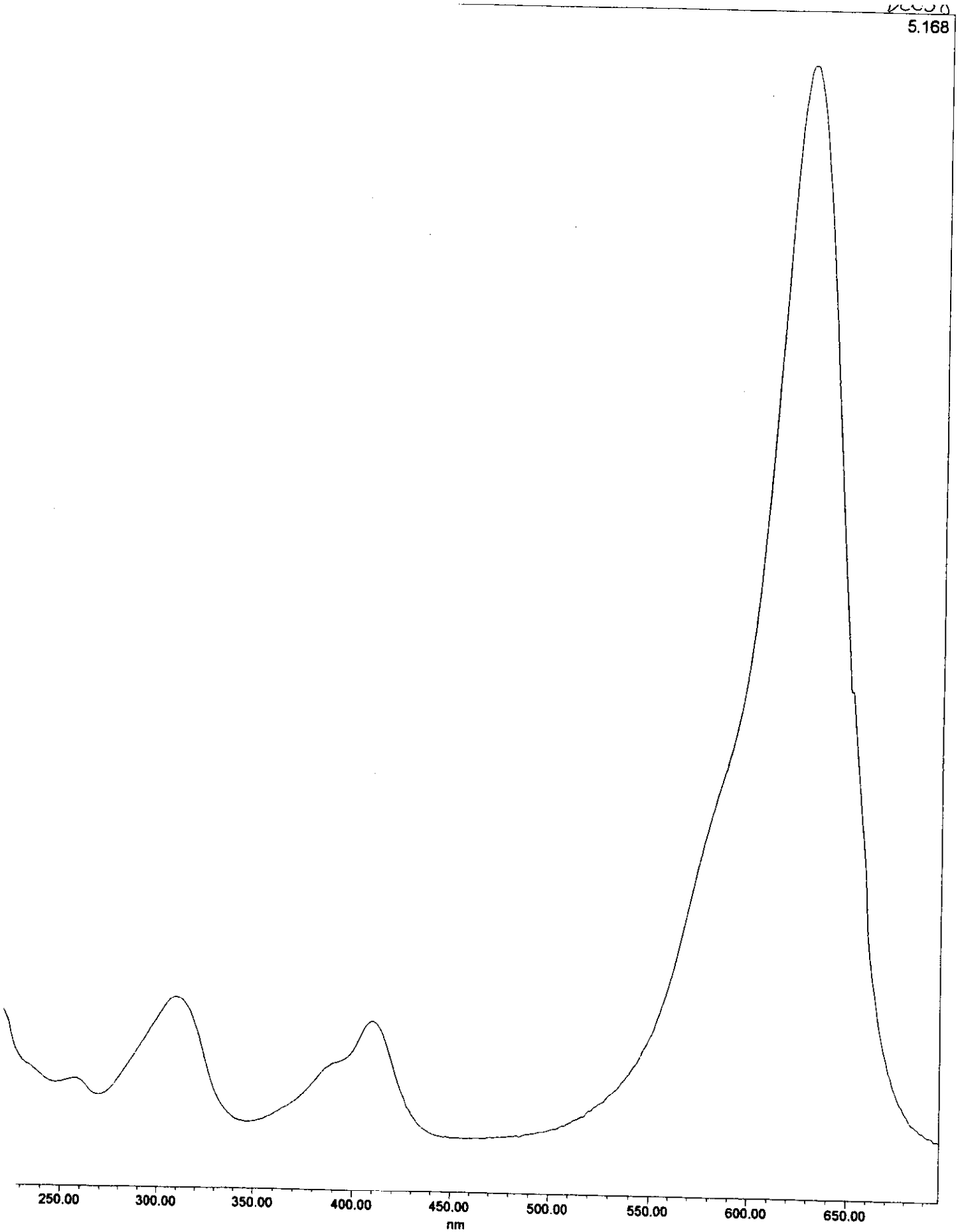
MIL-D-16627 Blue Color
@ 630 nm

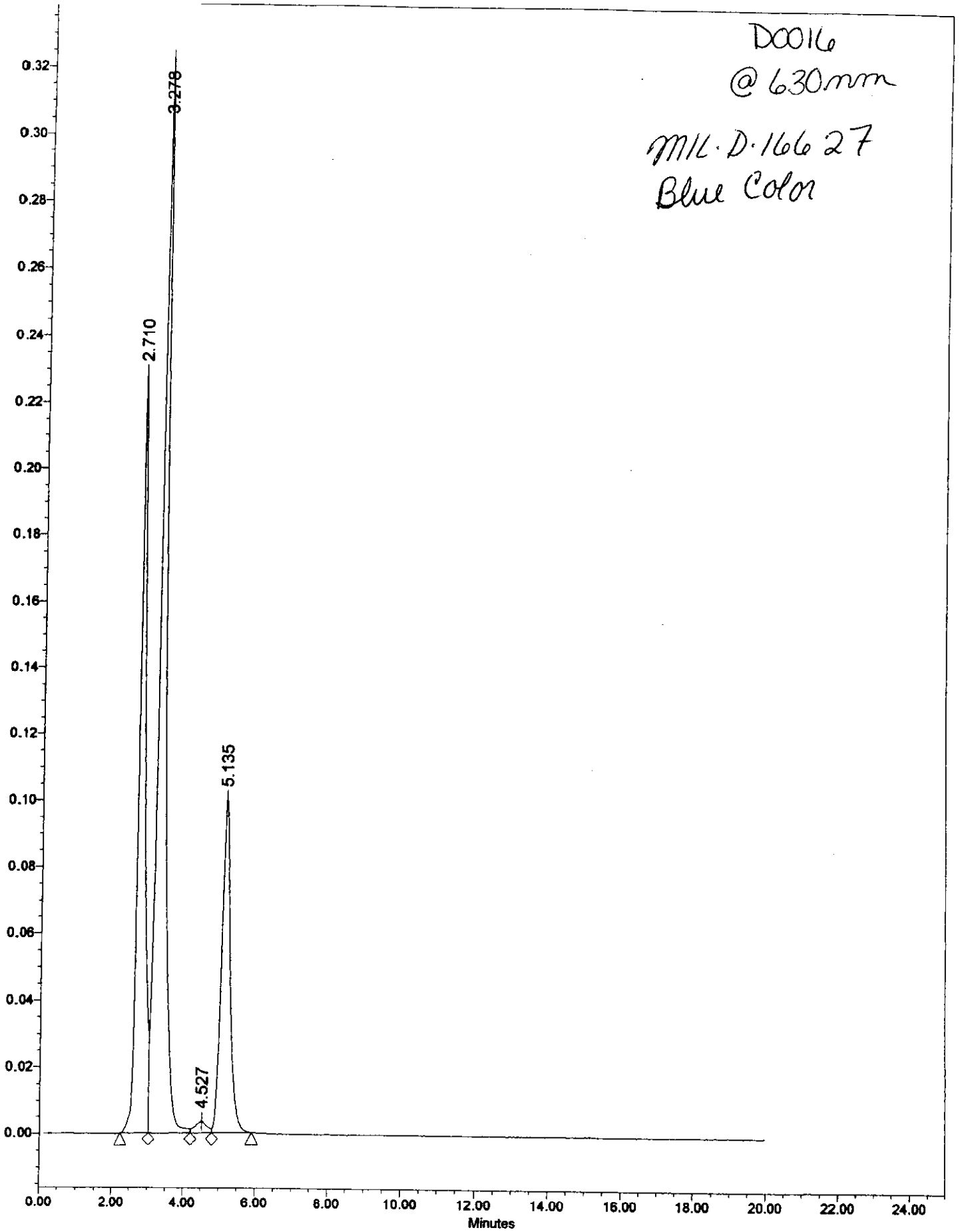


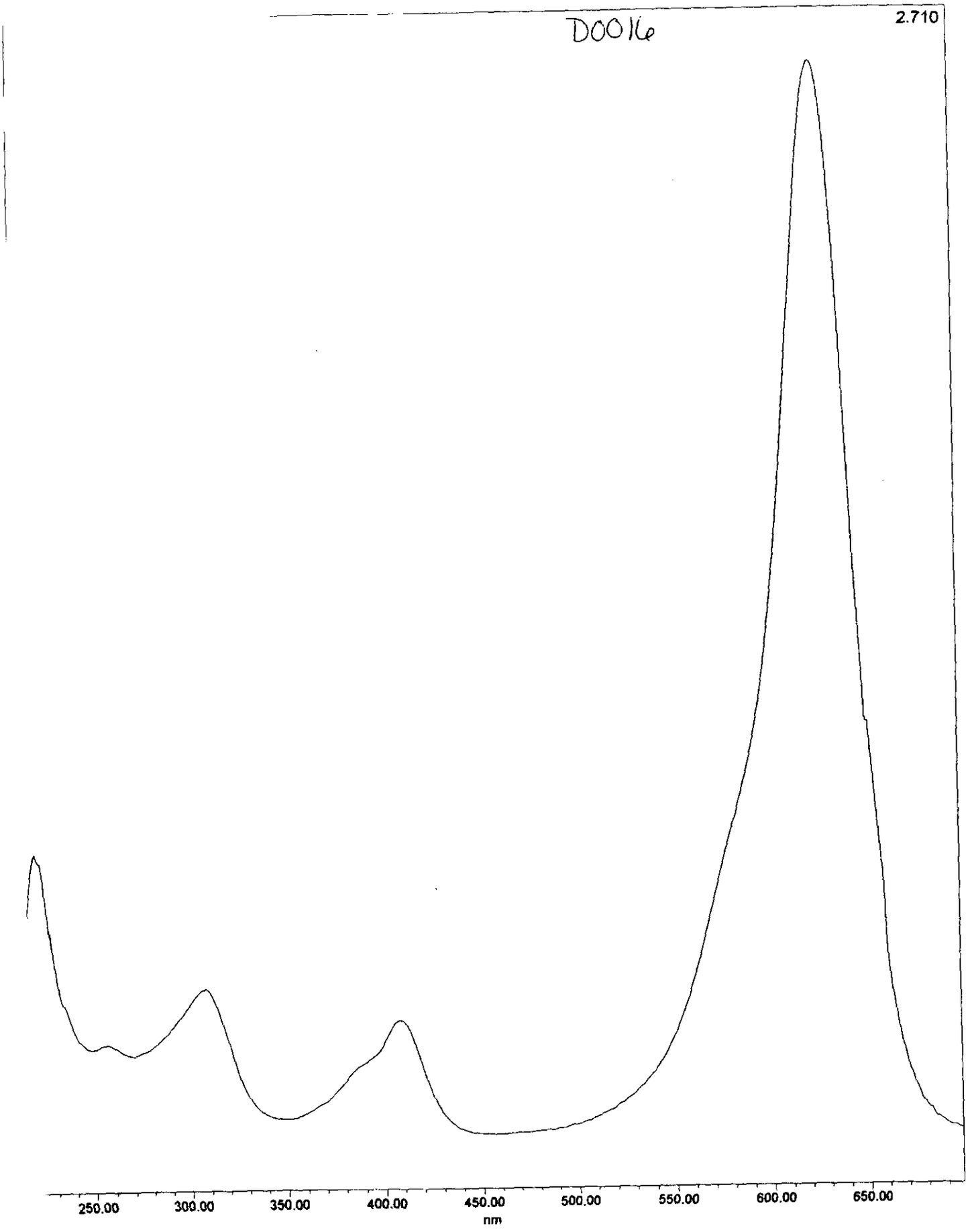

2.706
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

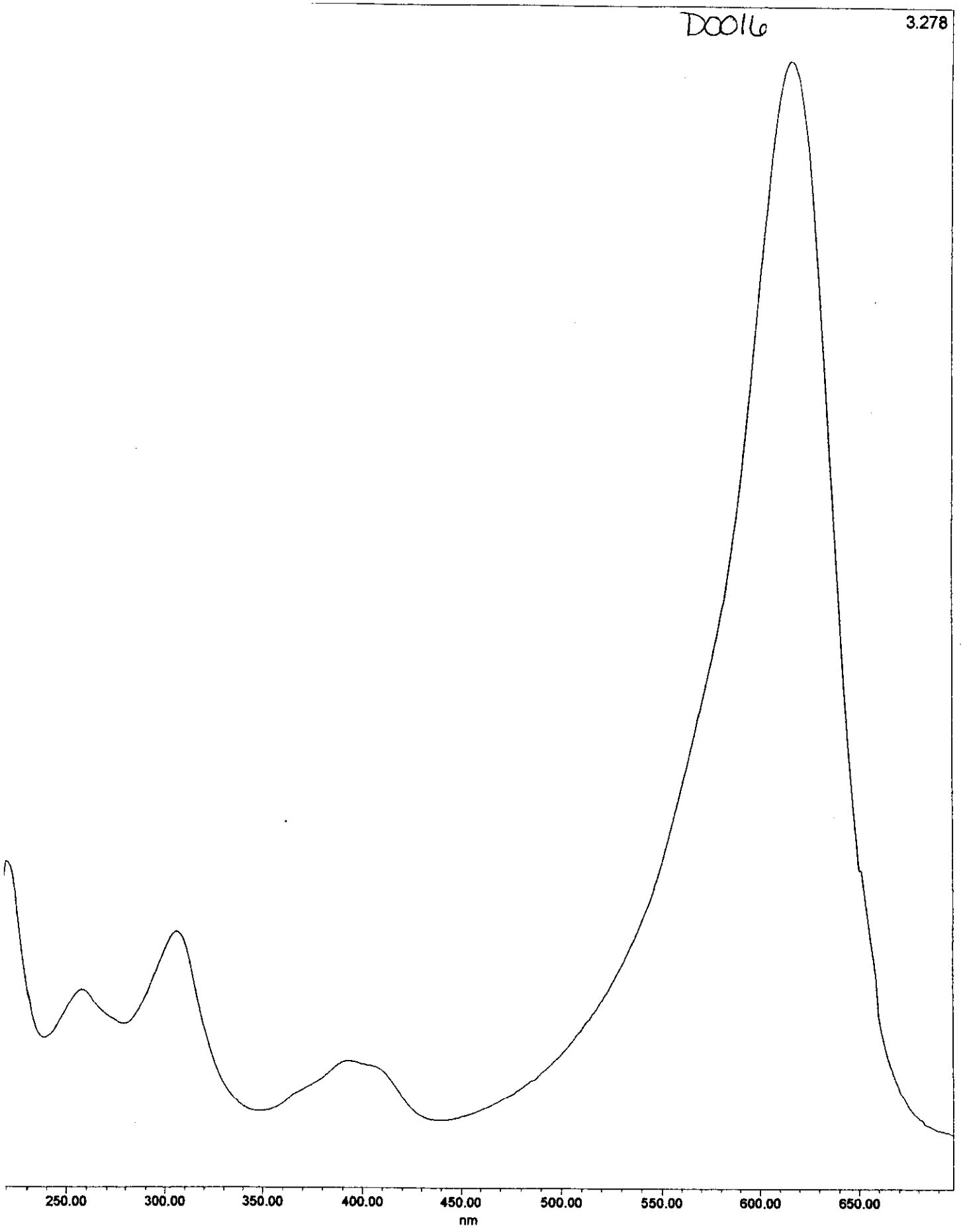

3.276
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

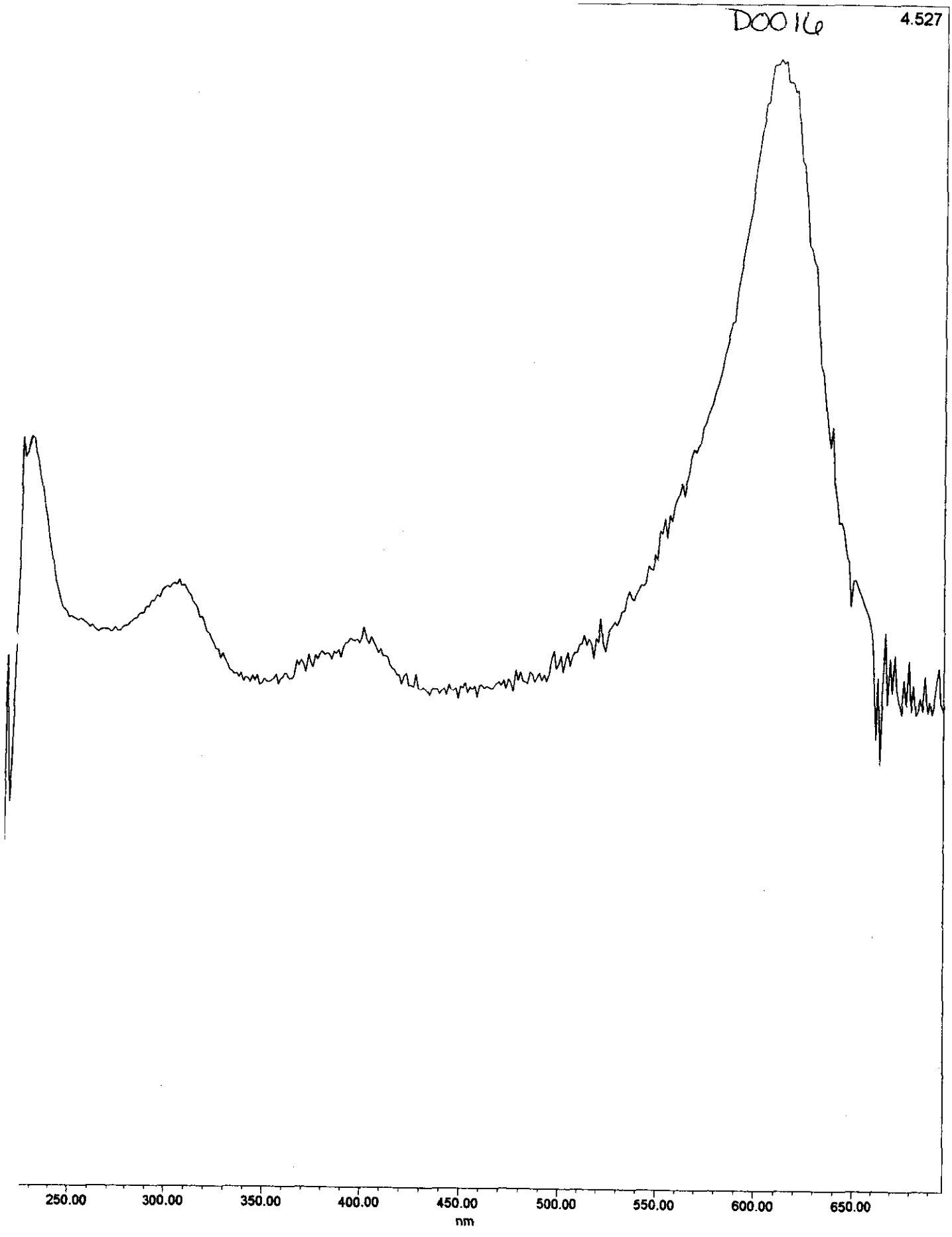

4.529
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

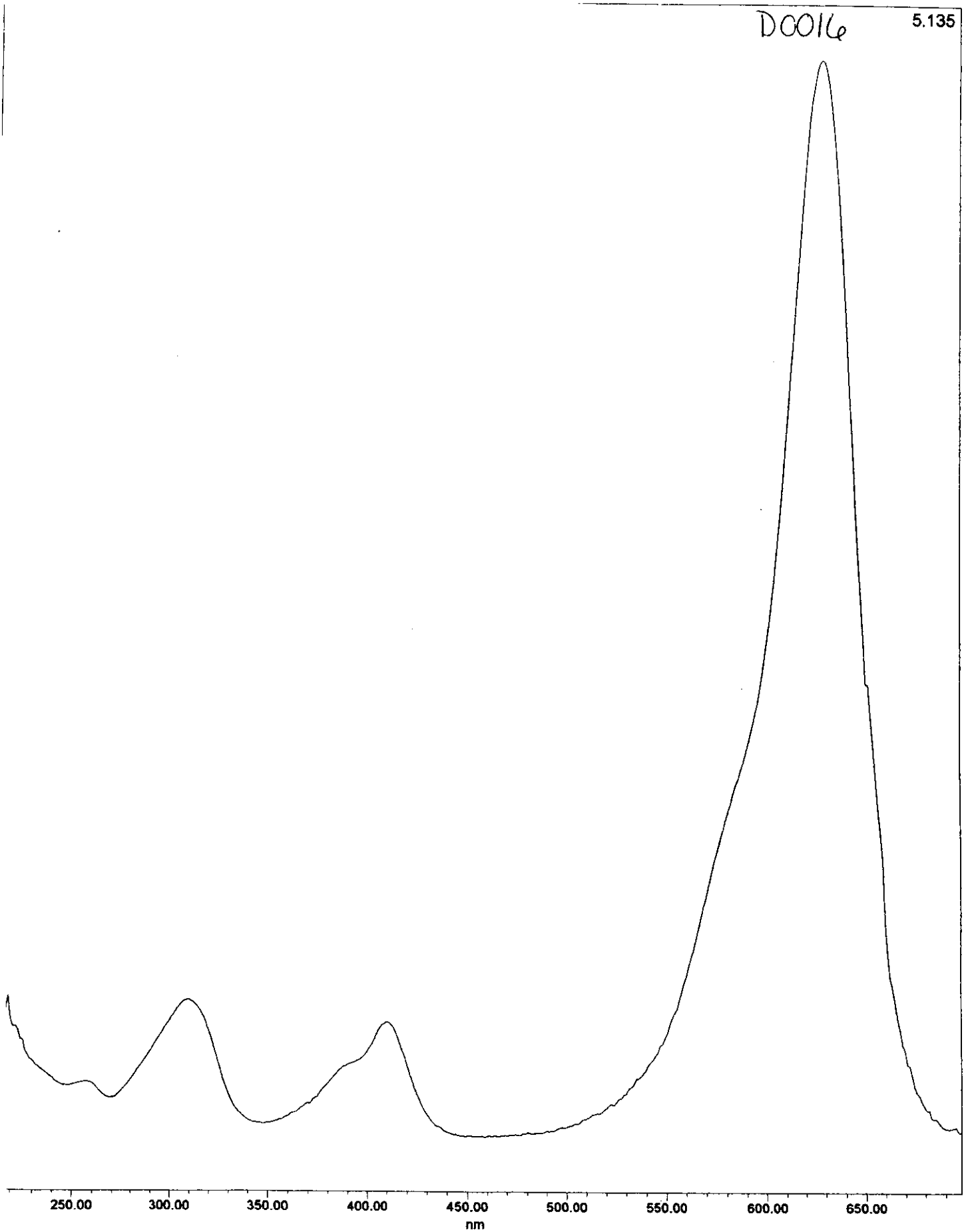

5.168
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm


D0016
@ 630nm
MIL-D-16627
Blue Color
2.710
3.278
4.527
5.135
0.32
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
22.00
24.00
Minutes


D0016
2.710
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

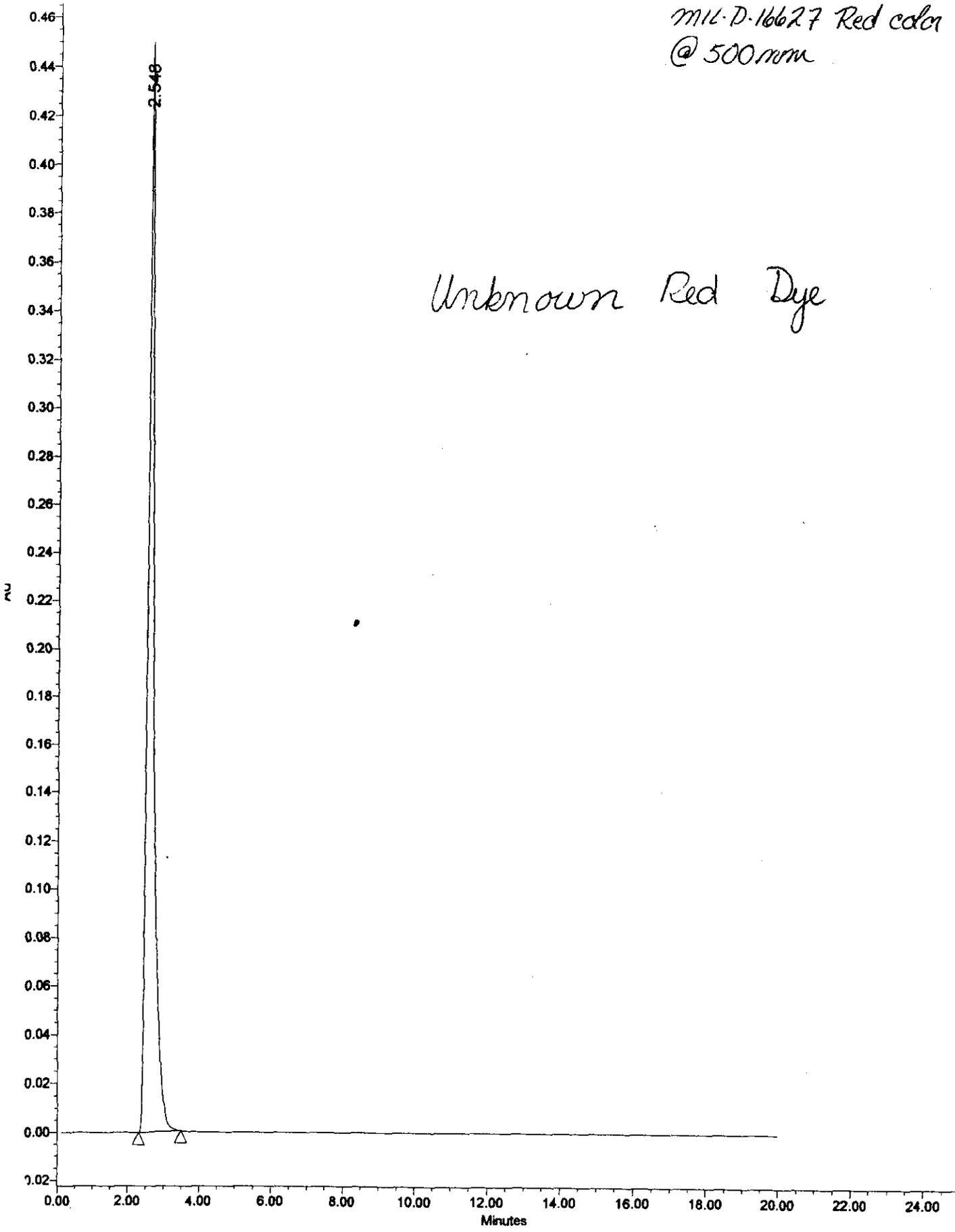

D0016
3.278
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

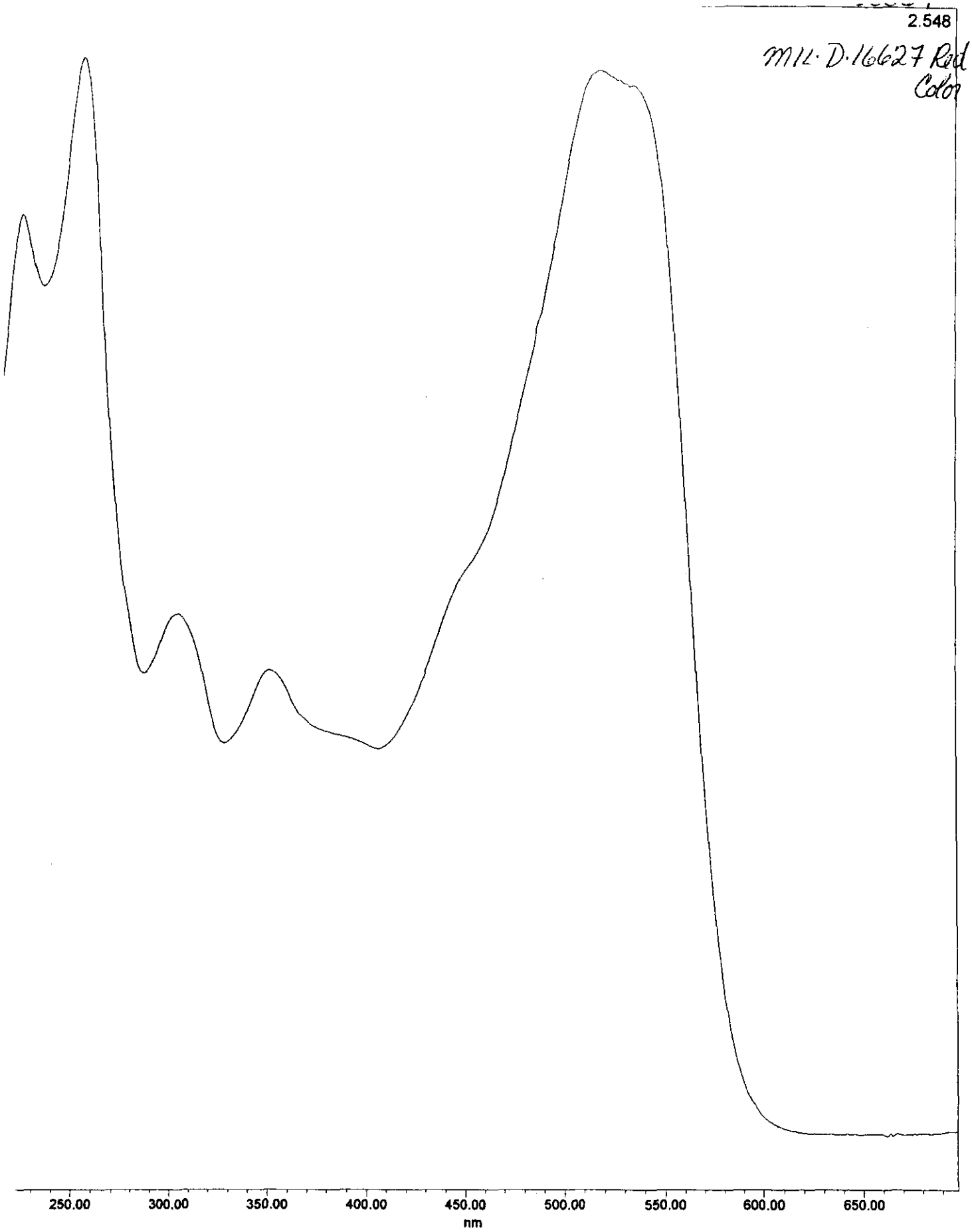

D0016
4.527
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm


D0016
5.135
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

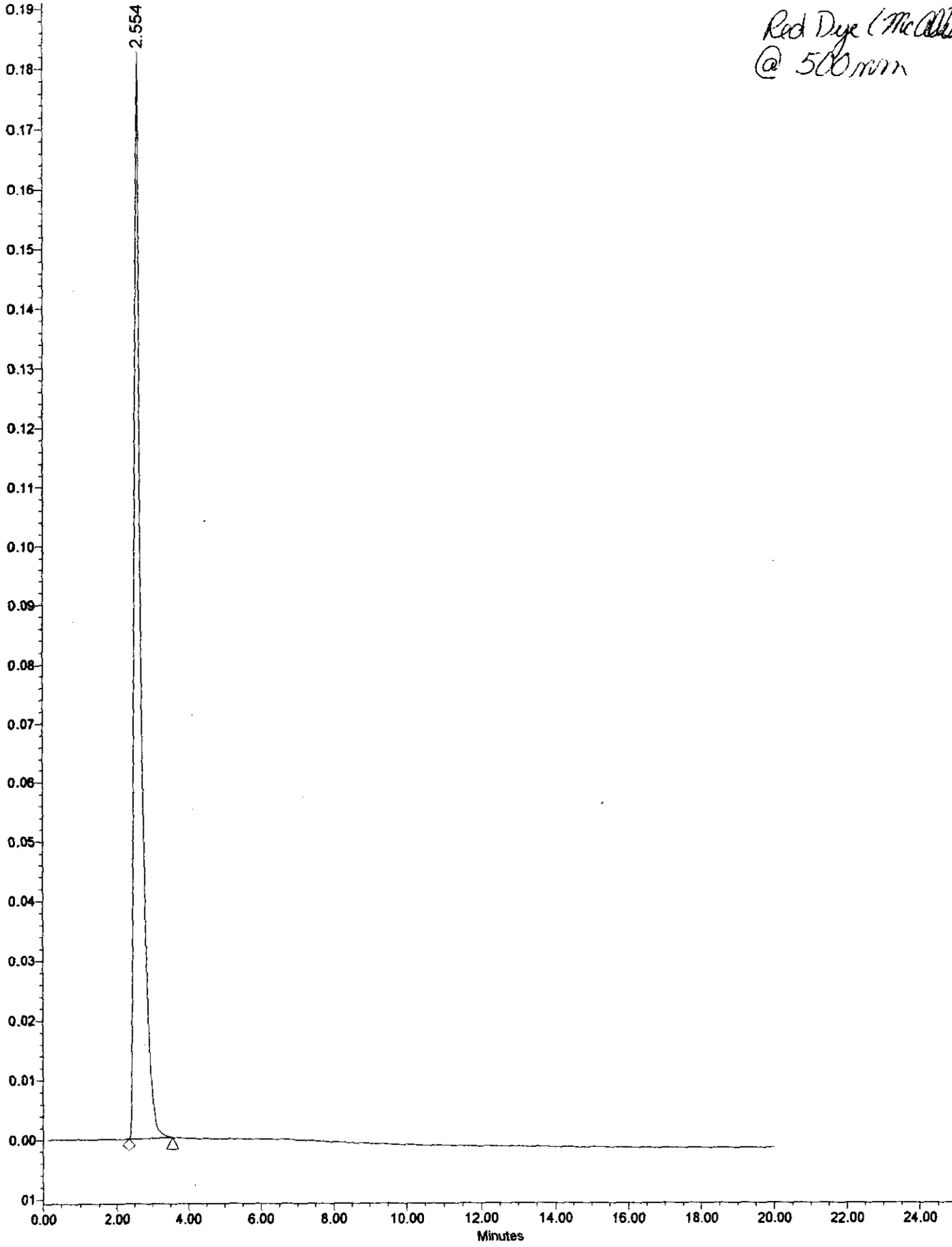
MIL-D-16627 Red color
@ 500nm

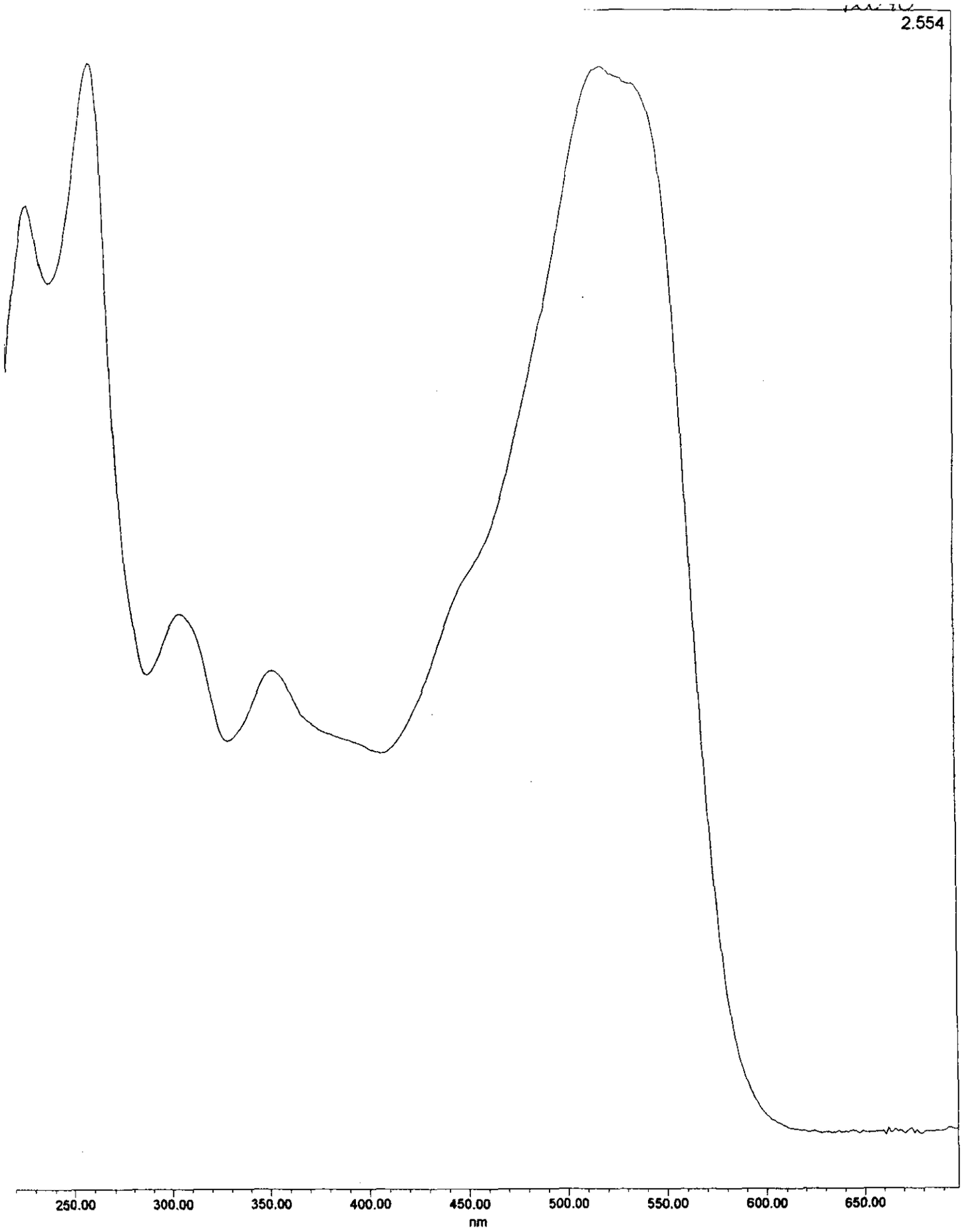
Unknown Red Dye



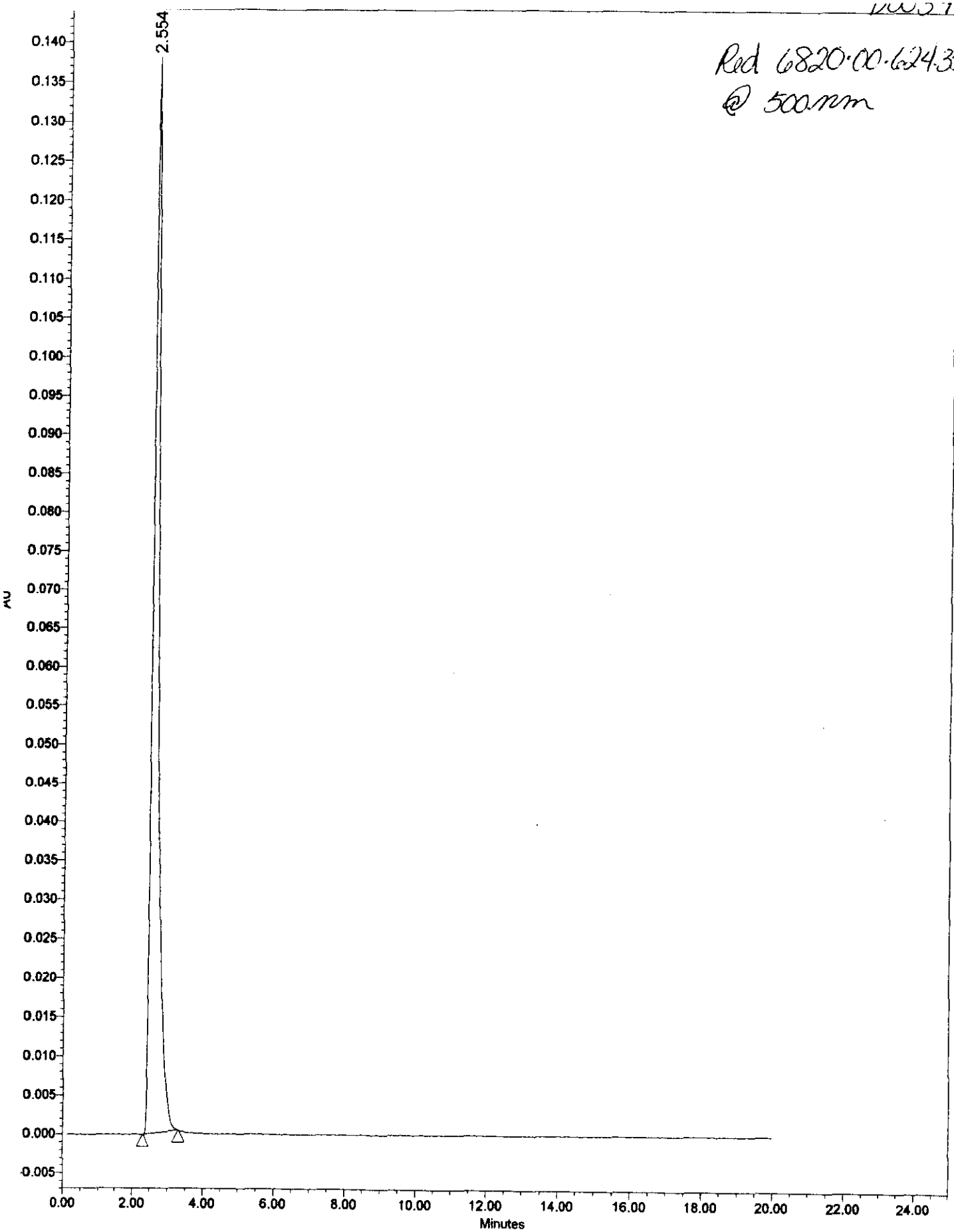

2.548
MIL-D-16627 Red
Color
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

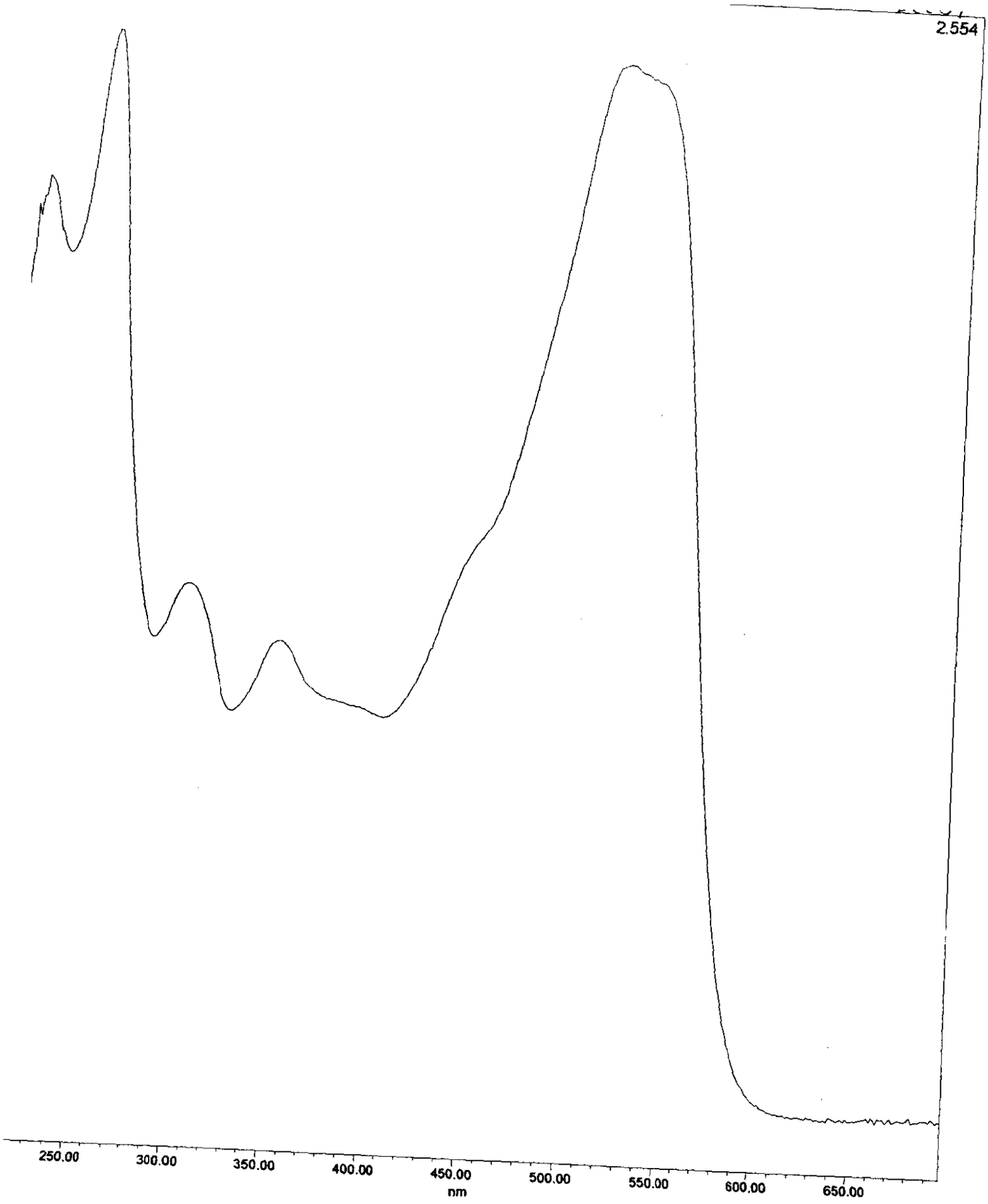
Red Dye (McCallister)
@ 500nm




2.554
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

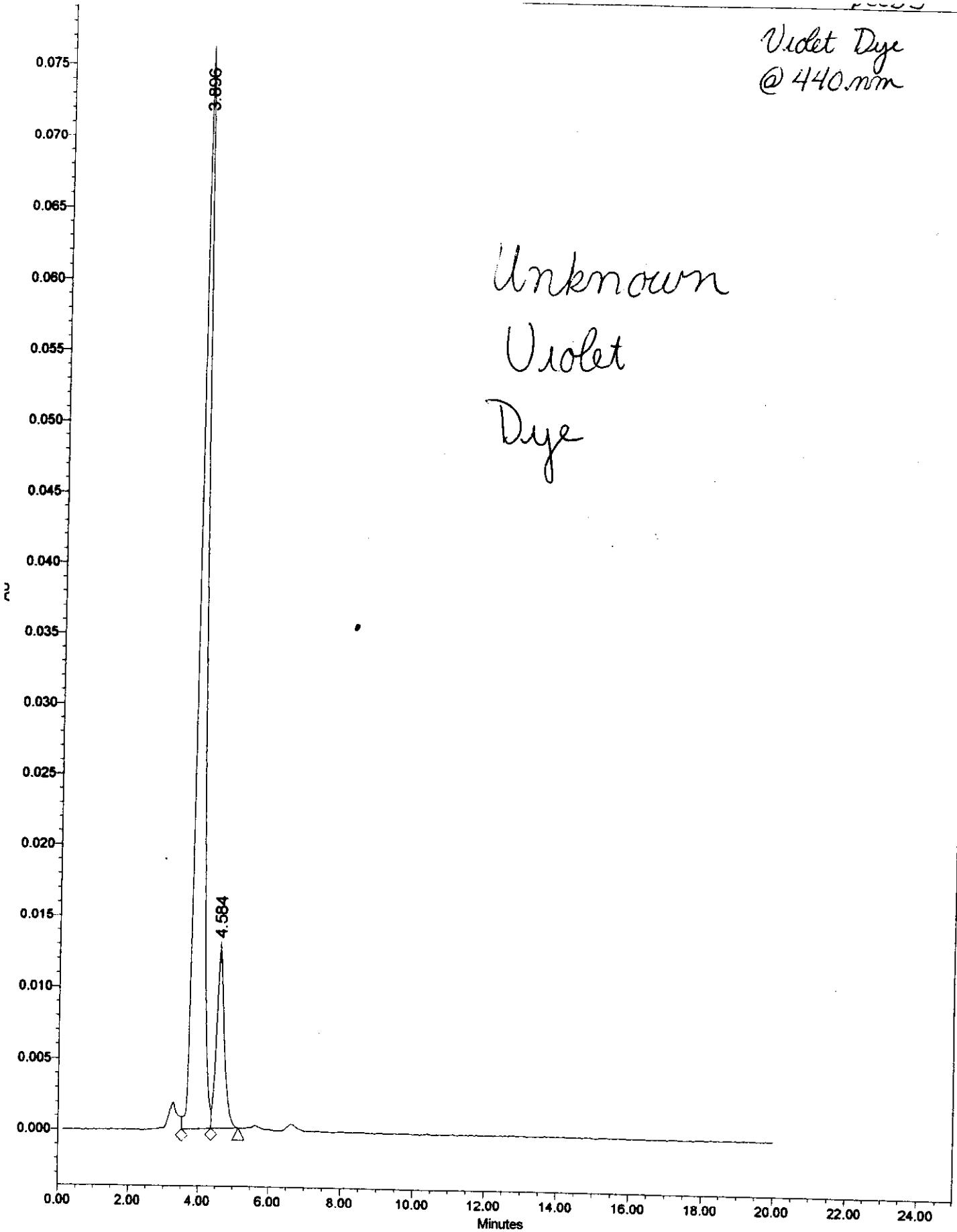
Red 6820-00-624-35
@ 500nm

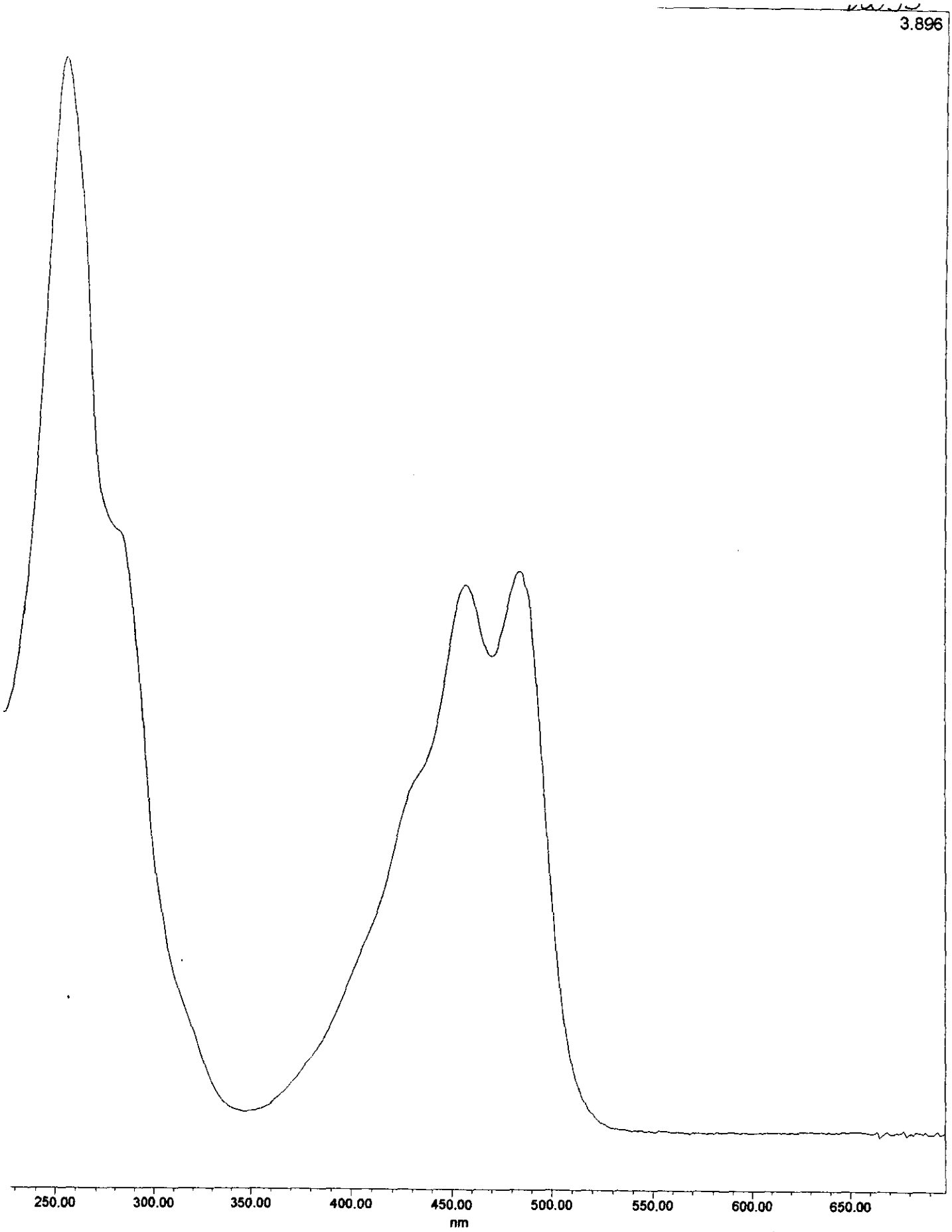


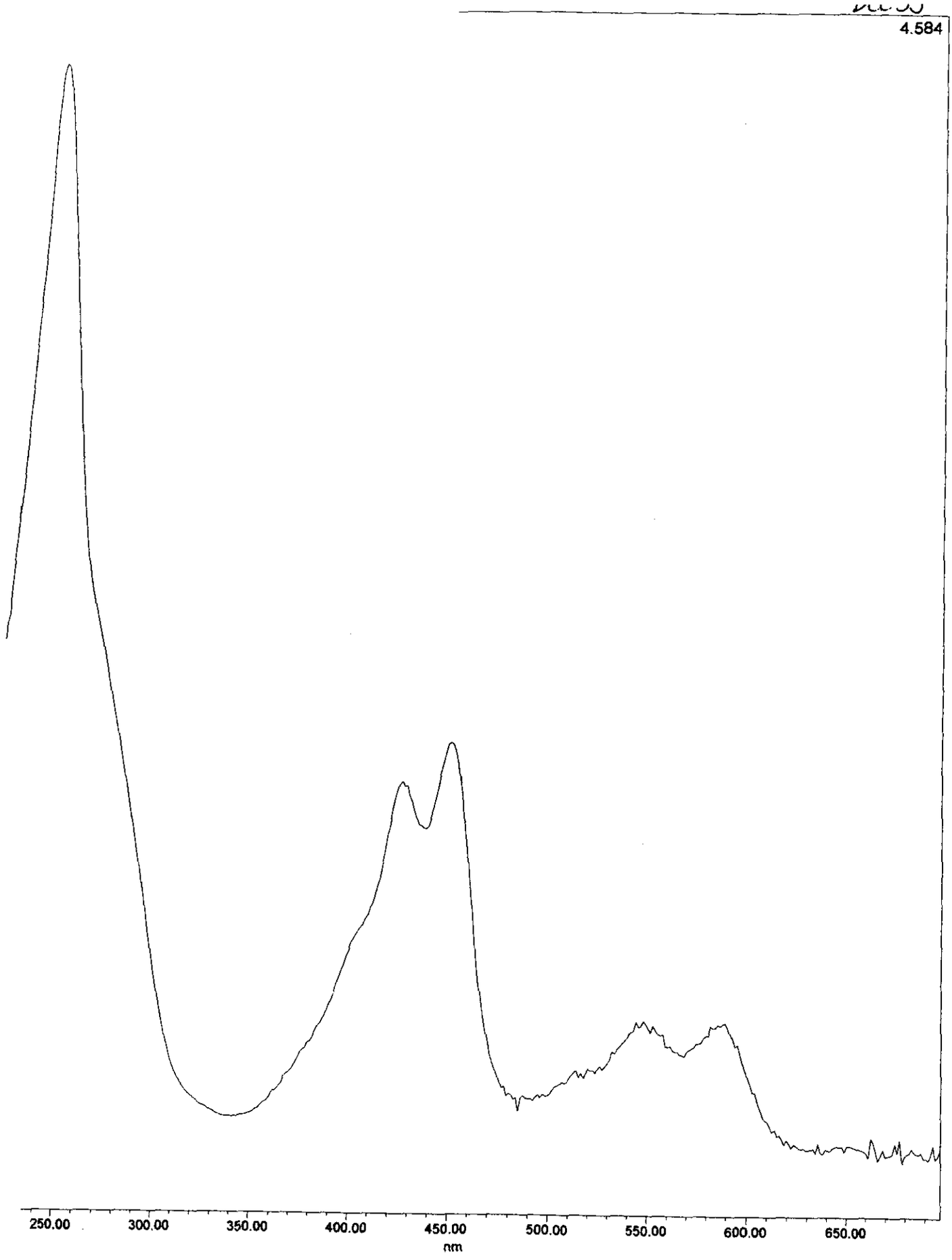

2.554
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

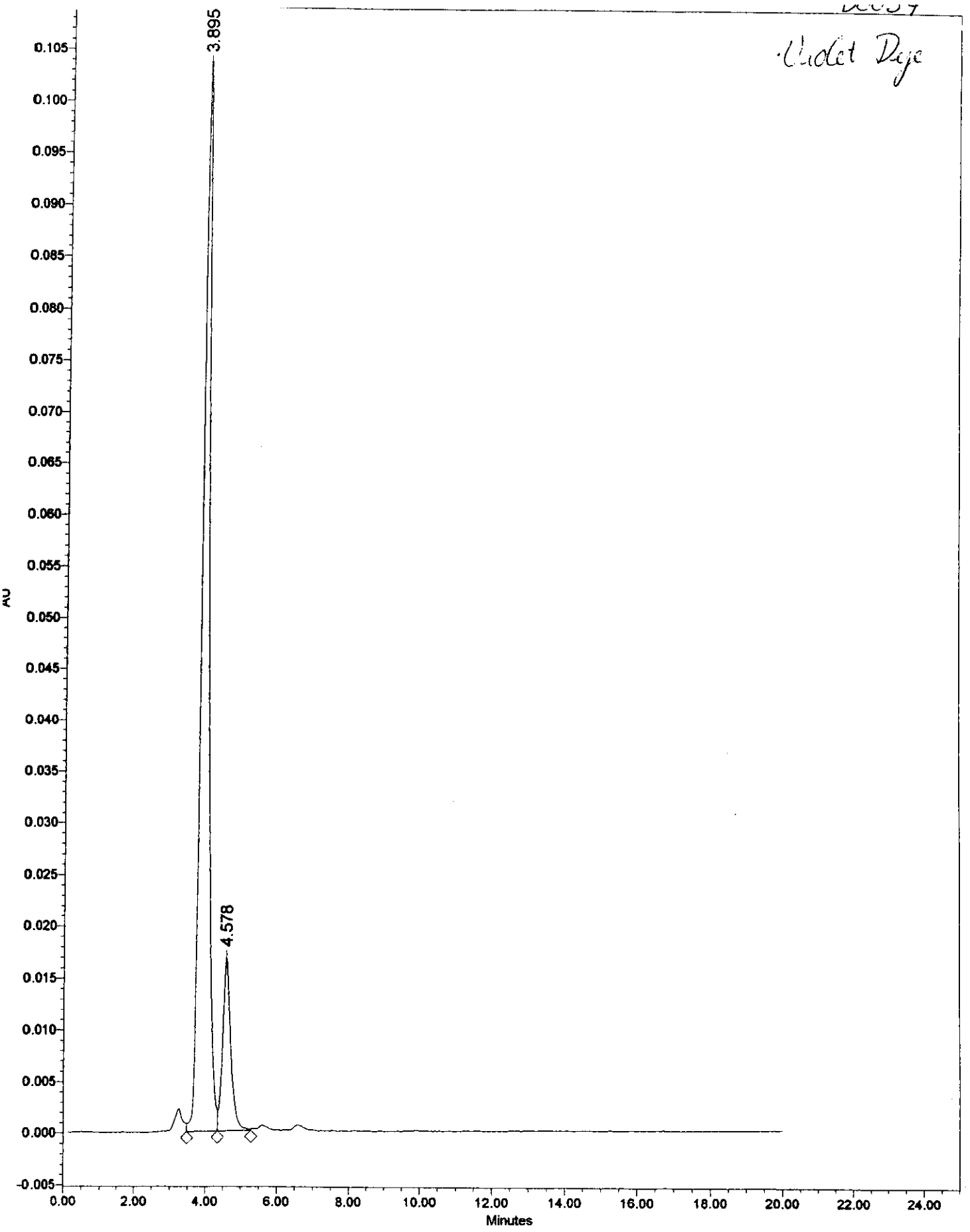
Violet Dye
@ 440nm

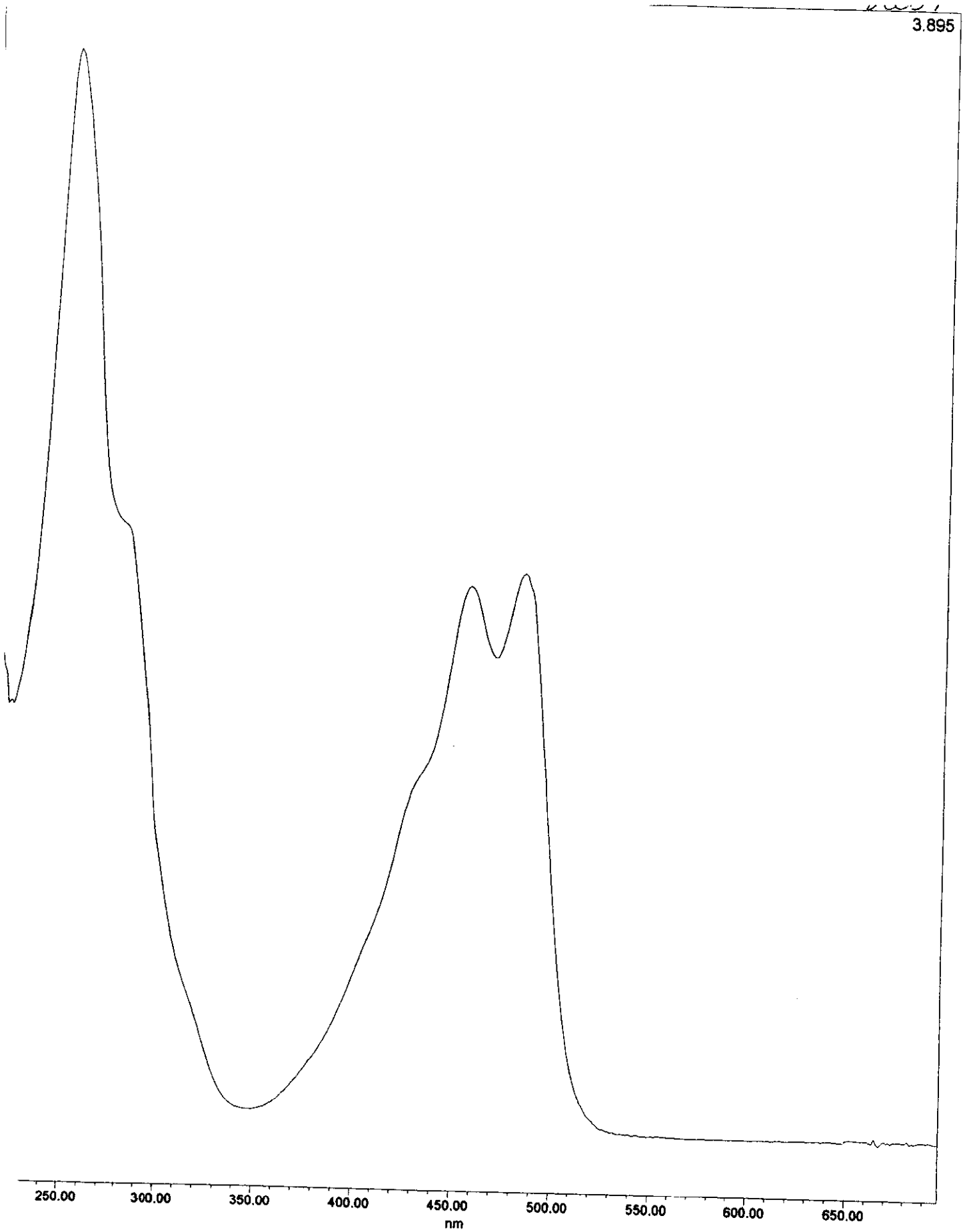
Unknown
Violet
Dye

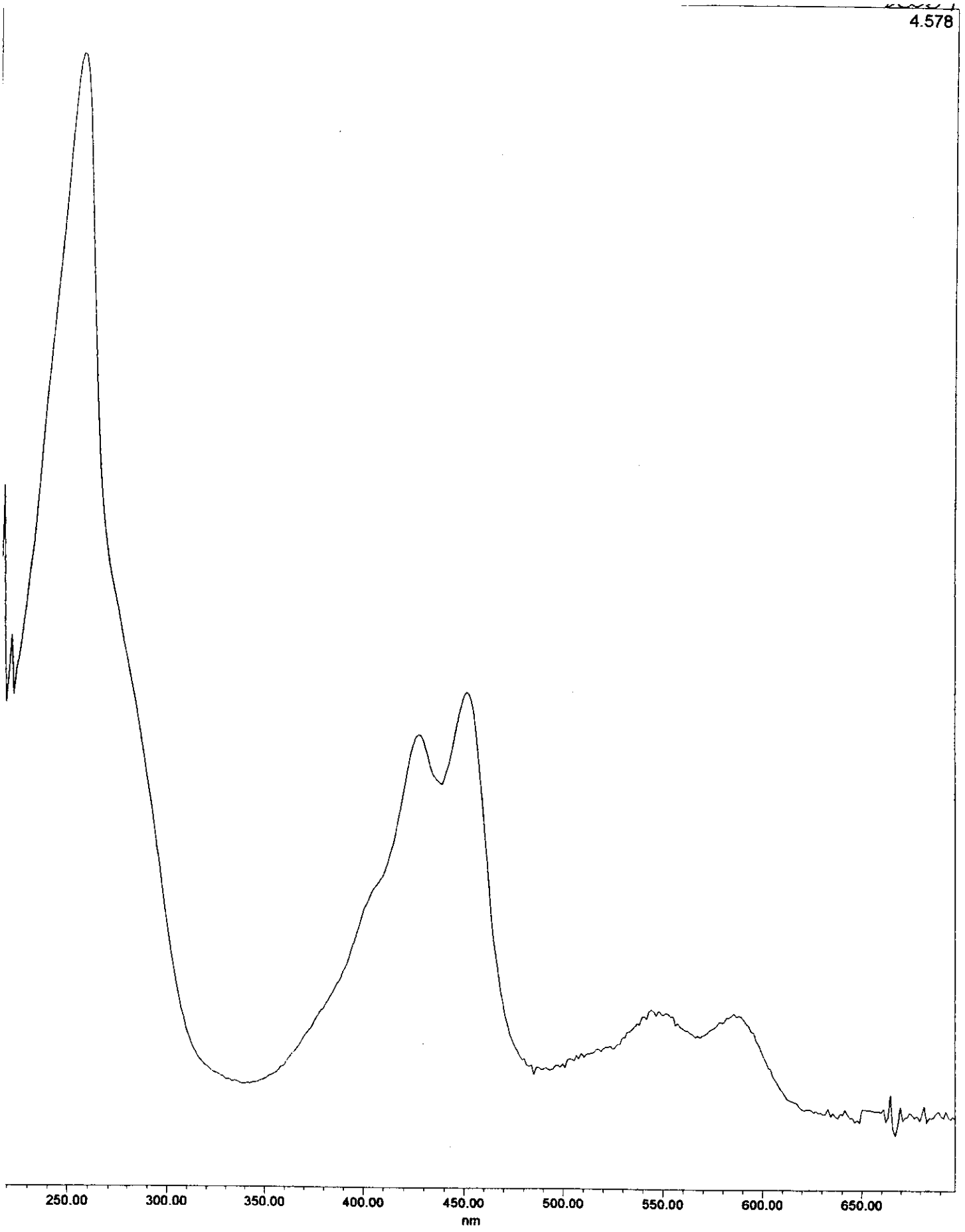



3.896
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm


4.584
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm


Unlabel Dye
3.895
4.578
AU
0.105
0.100
0.095
0.090
0.085
0.080
0.075
0.070
0.065
0.060
0.055
0.050
0.045
0.040
0.035
0.030
0.025
0.020
0.015
0.010
0.005
0.000
-0.005
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
22.00
24.00
Minutes

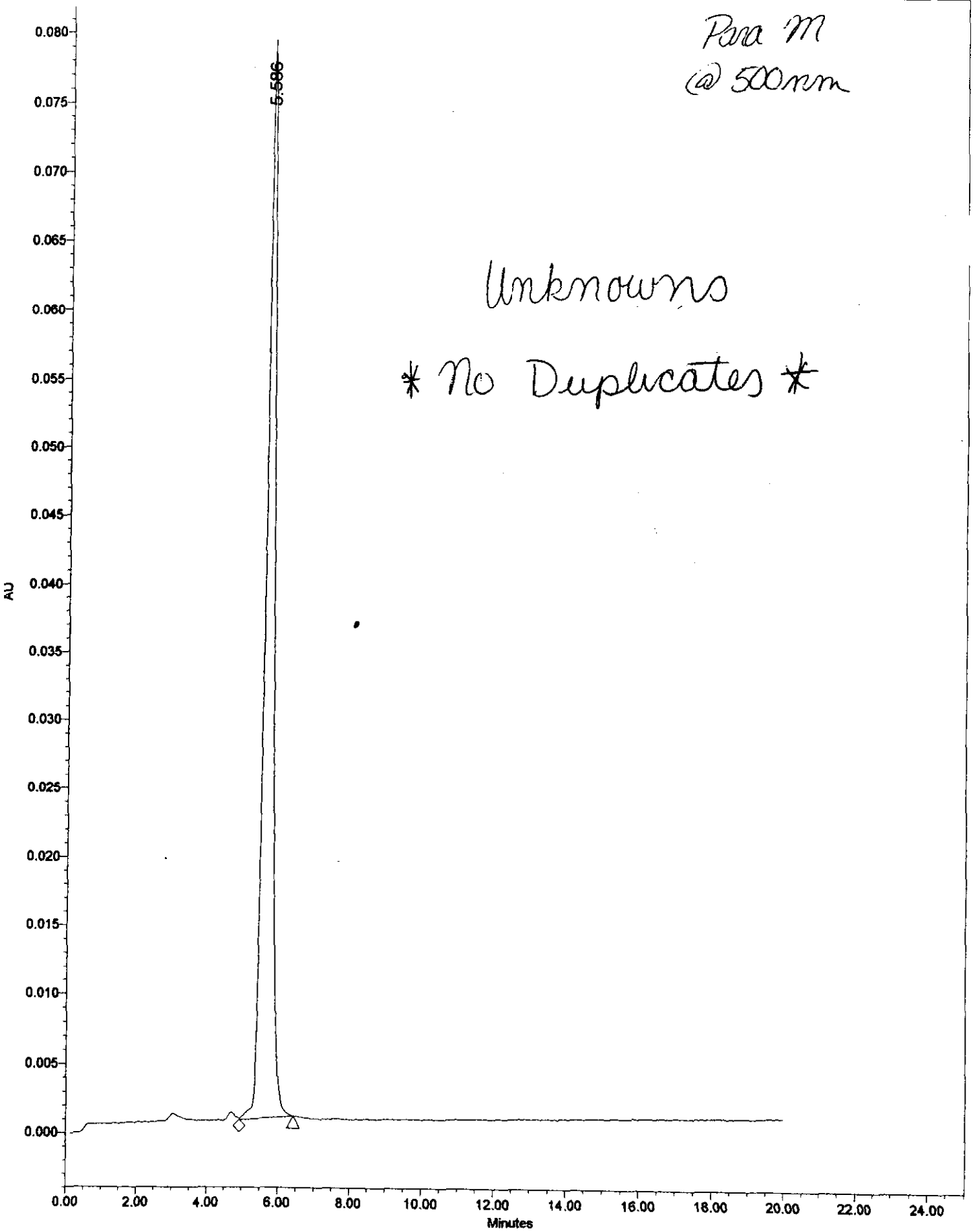

3.895
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

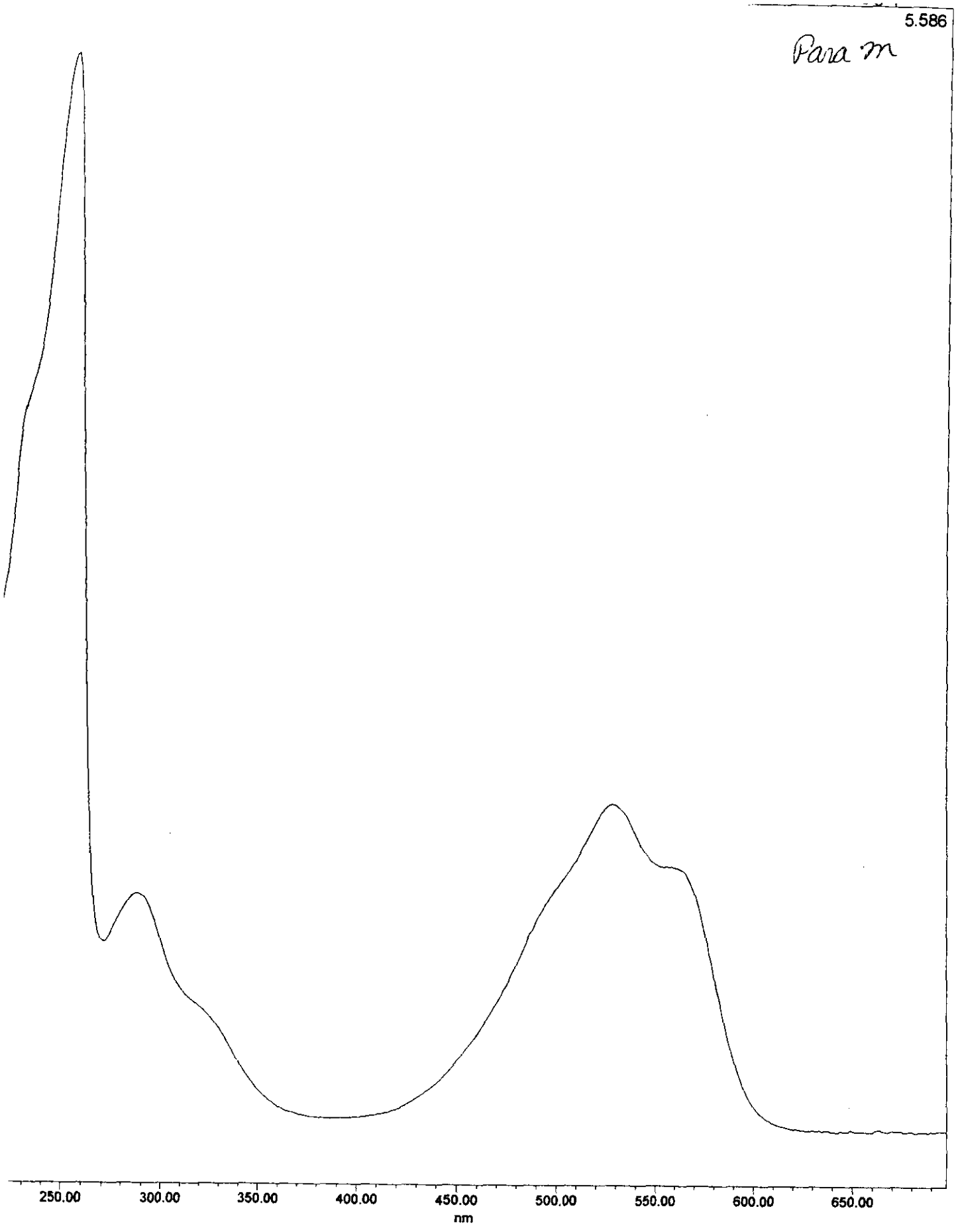

4.578
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

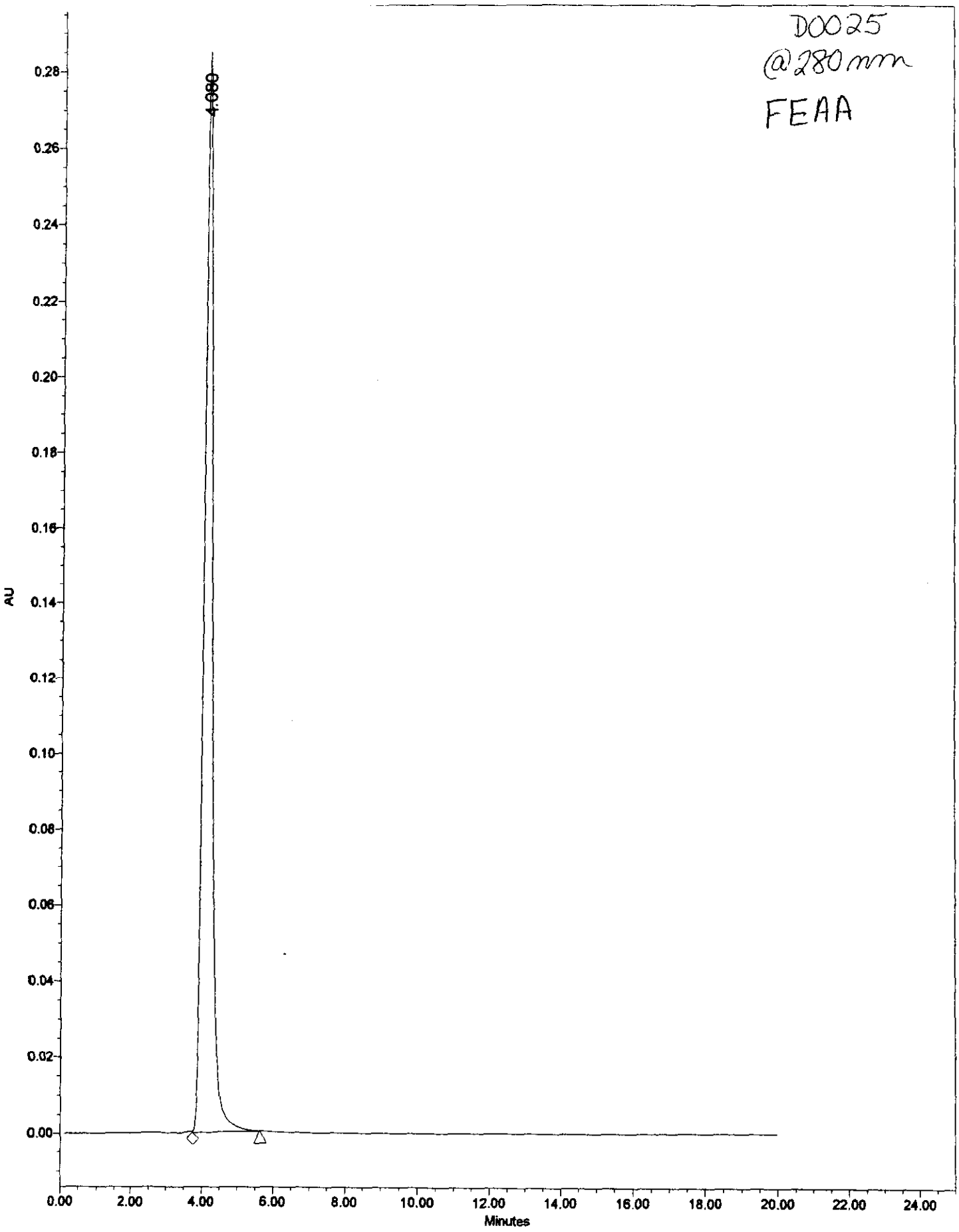
Para M
@ 500nm

Unknowns

* No Duplicates *




5.586
Para m
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm


D0025
@280nm
FEAA
4.080
AU
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
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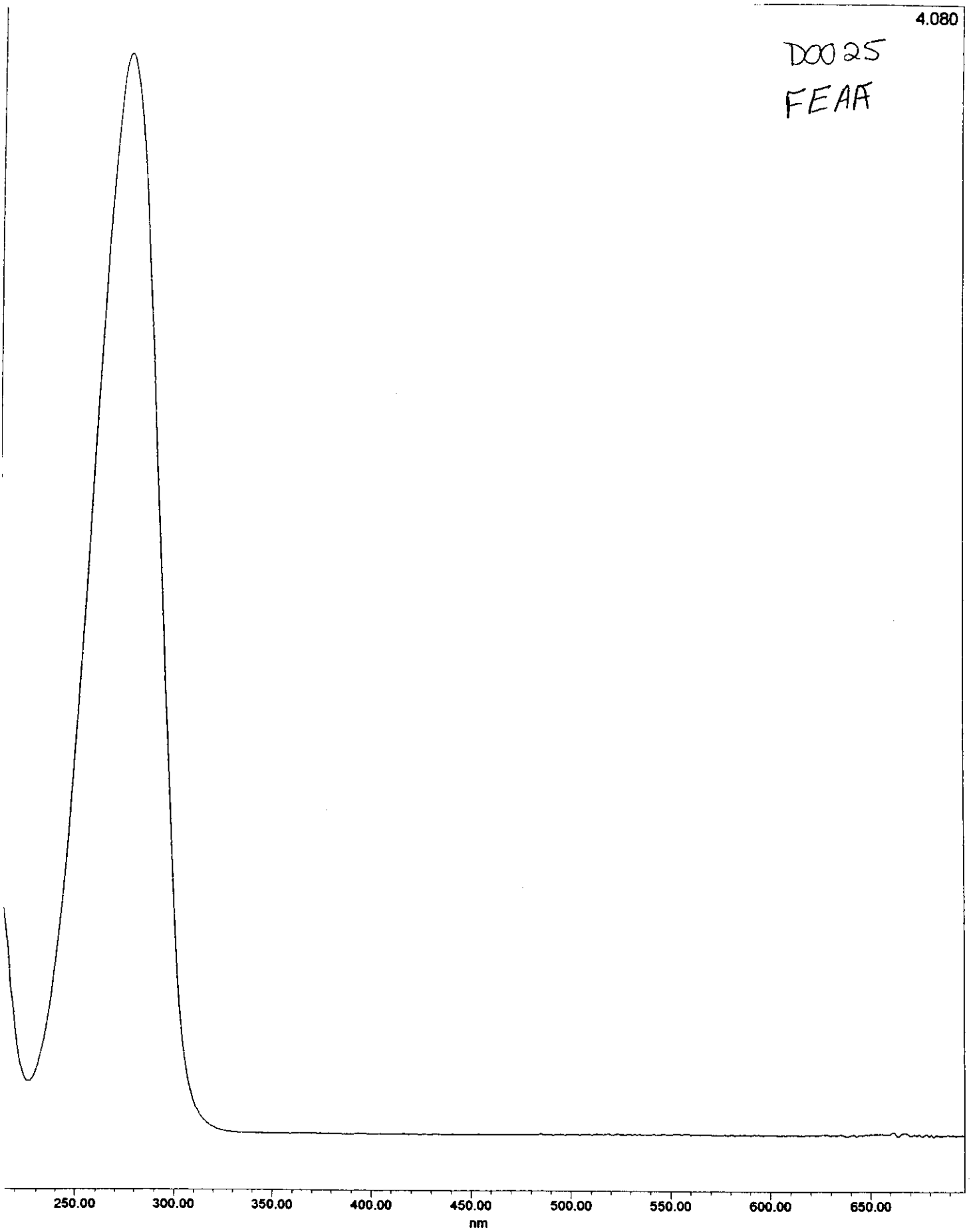
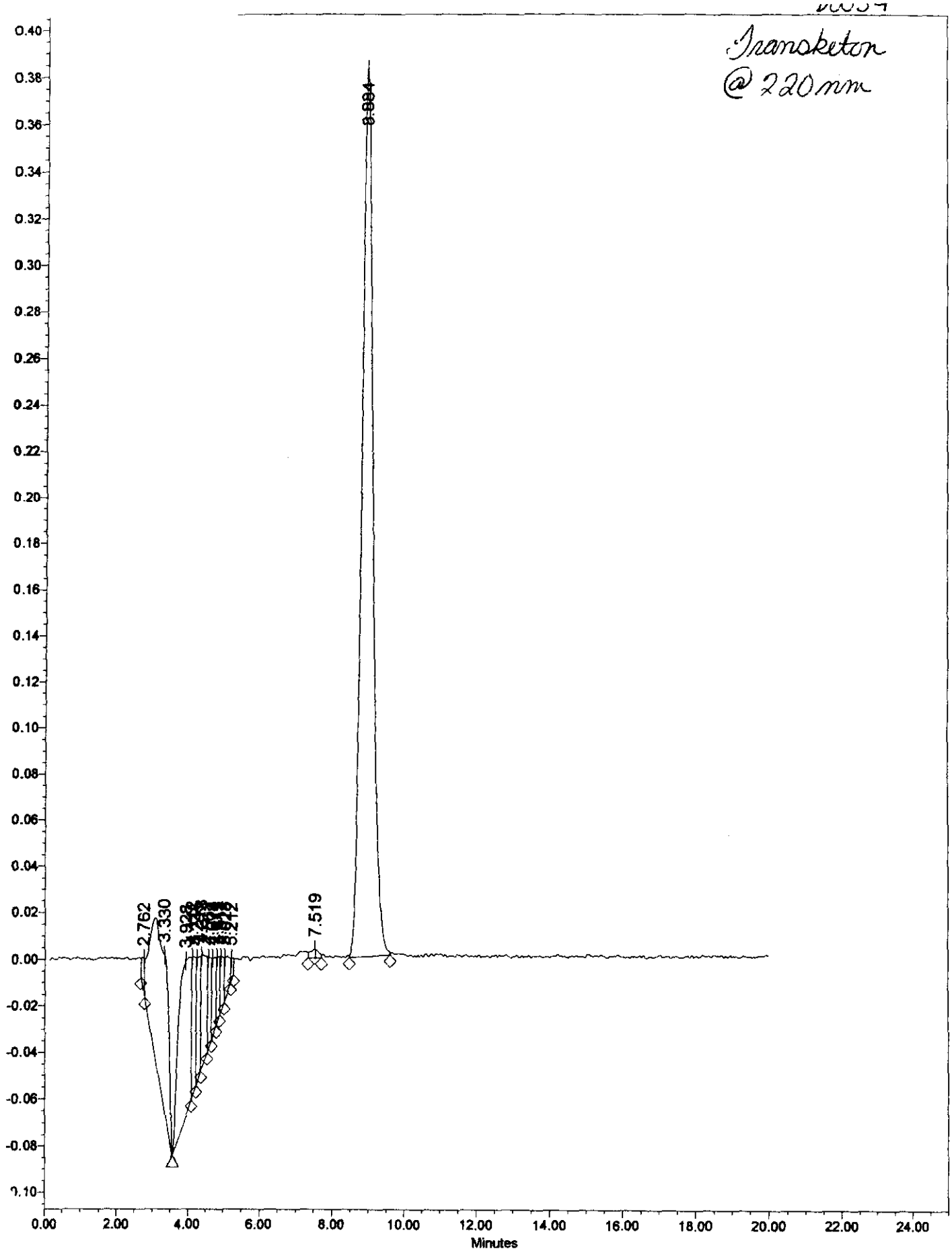
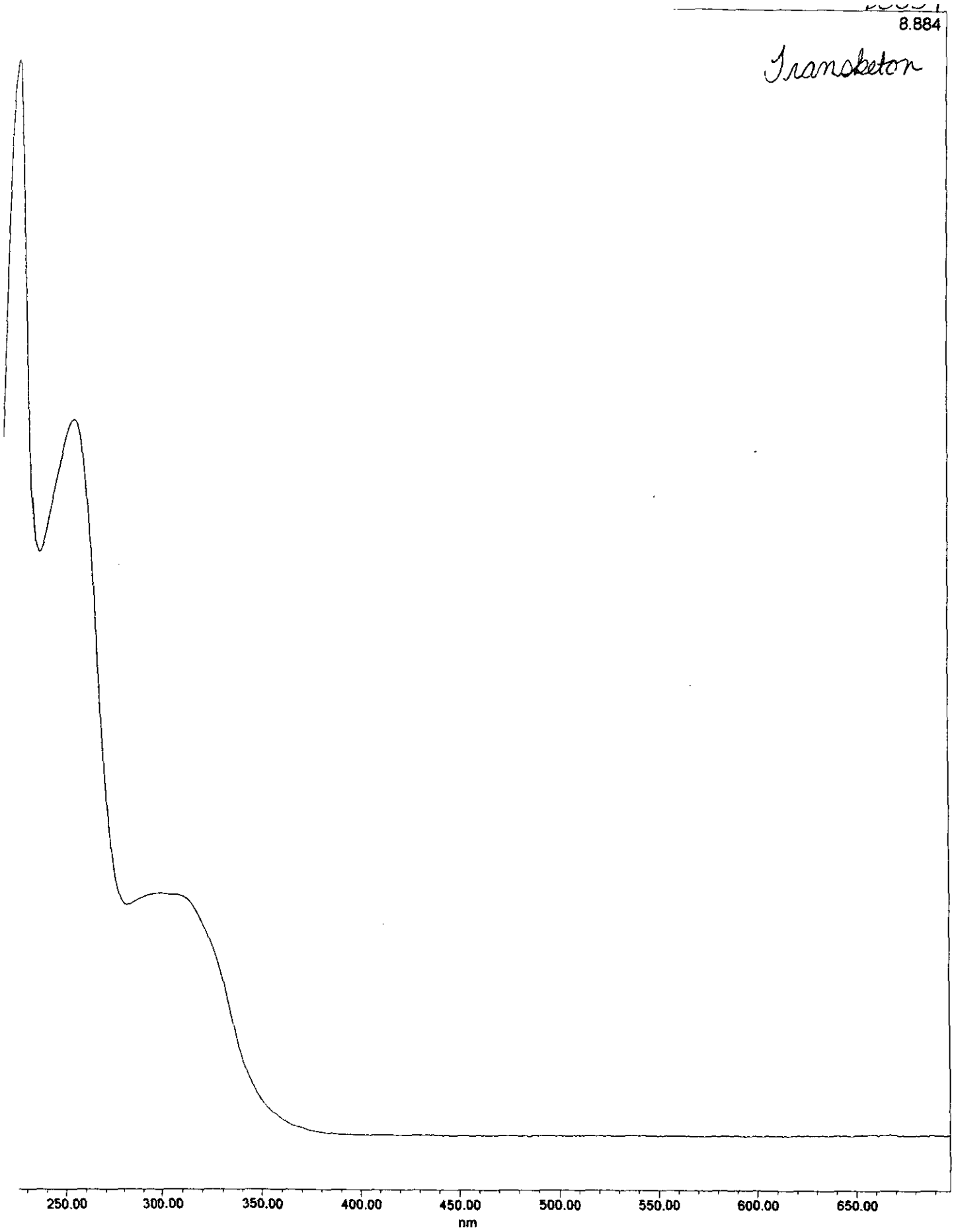
18.00
20.00
22.00
24.00
Minutes

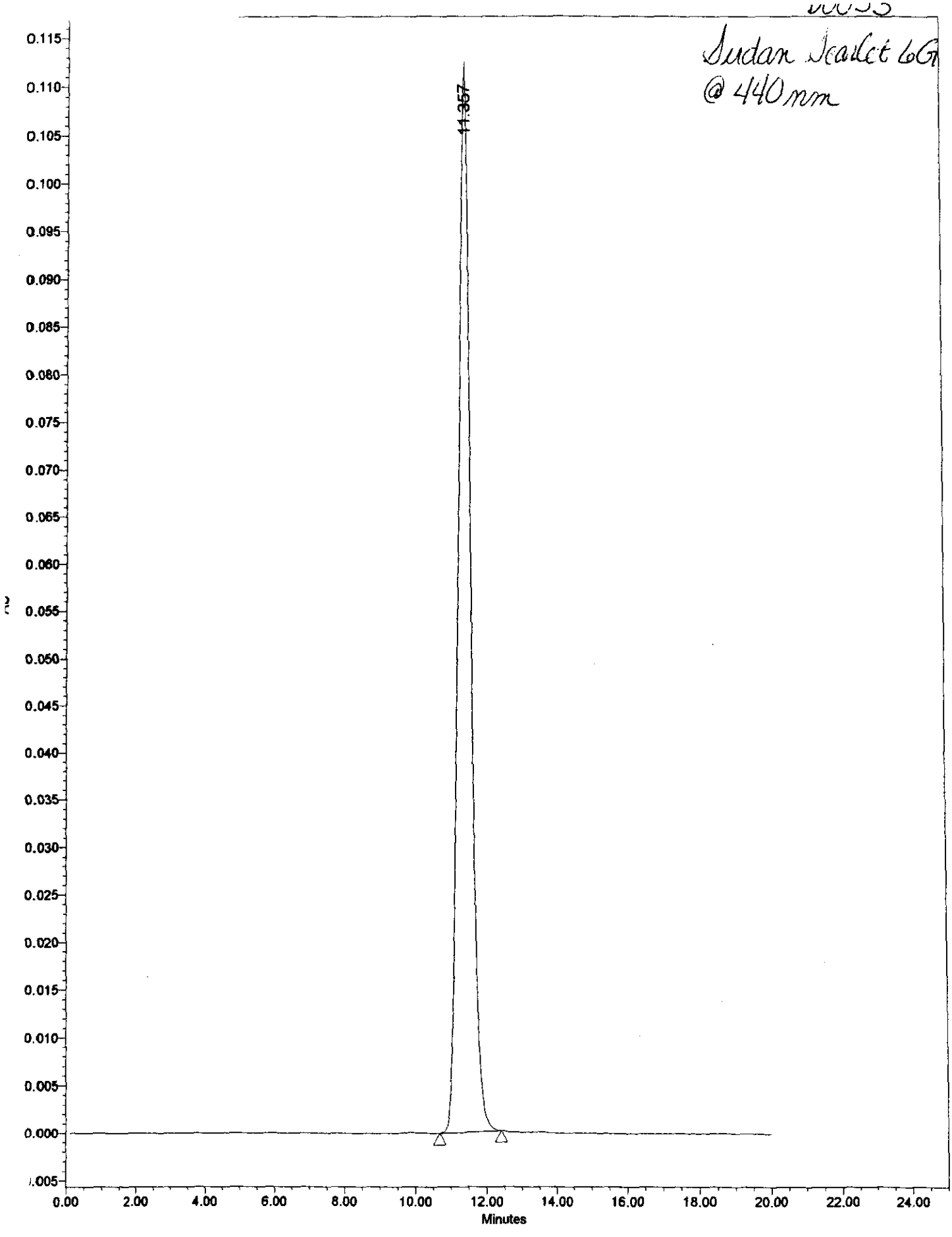

4.080
D0025
FEAA
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

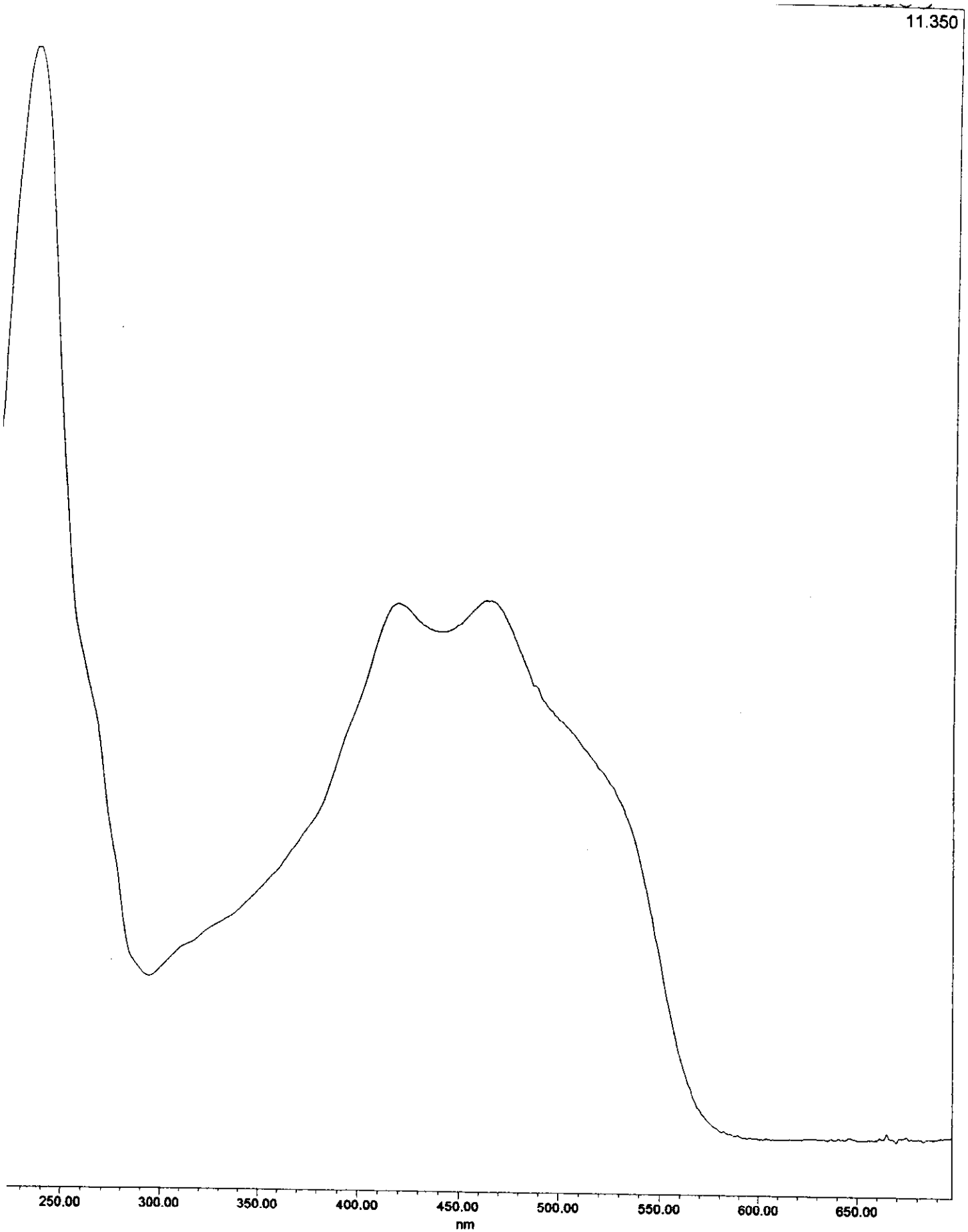
Transketon
@ 220nm

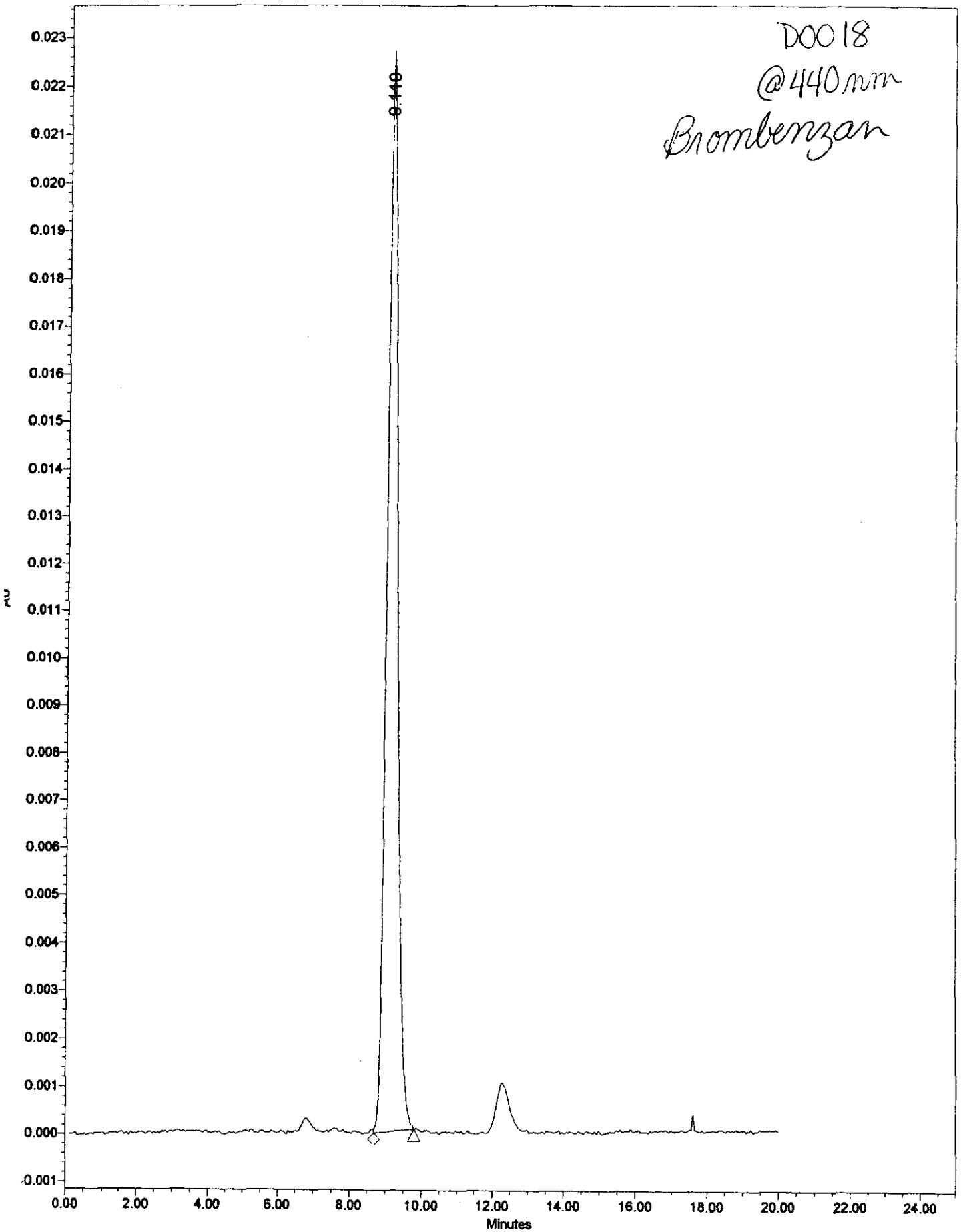


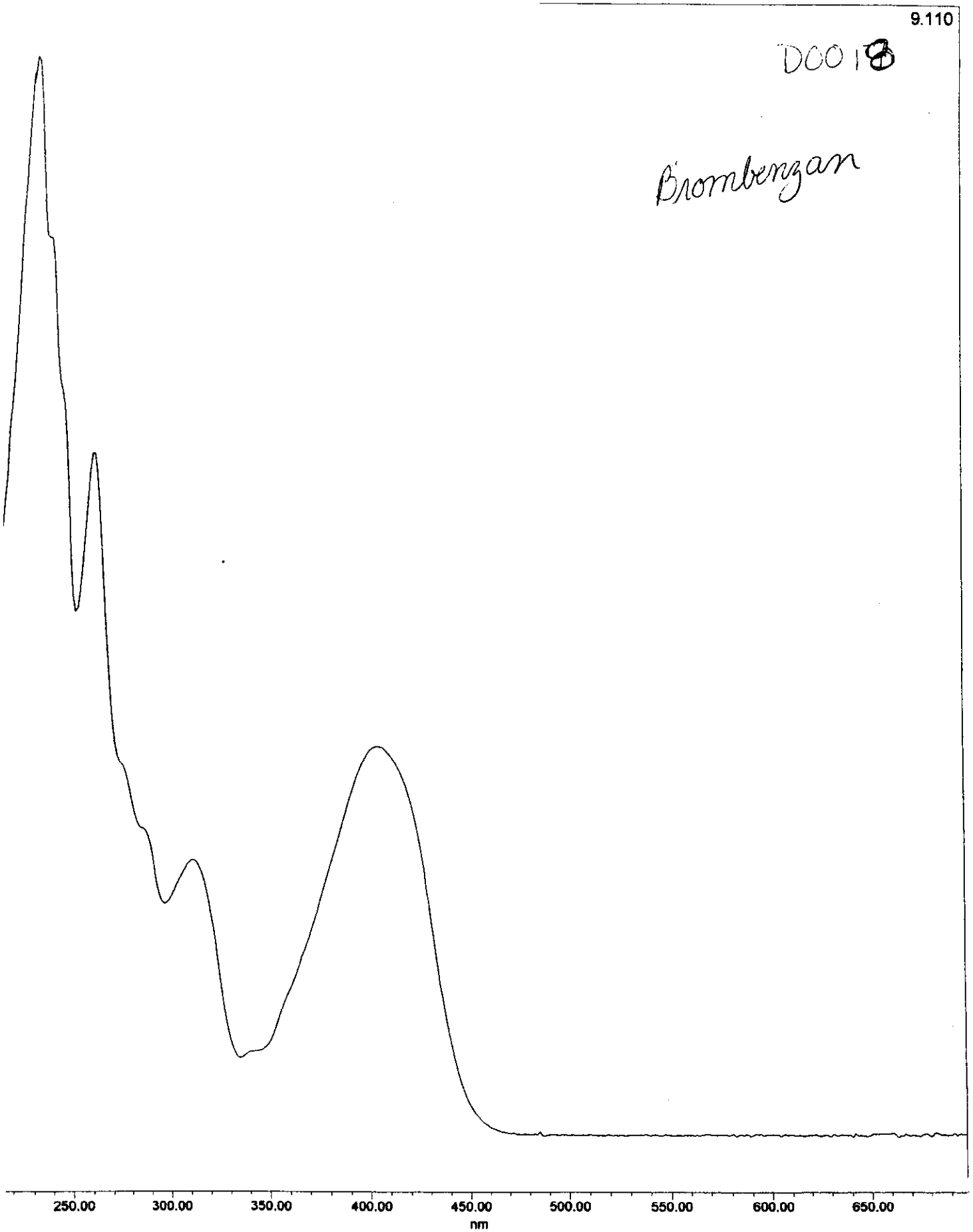

8.884
Transketon
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

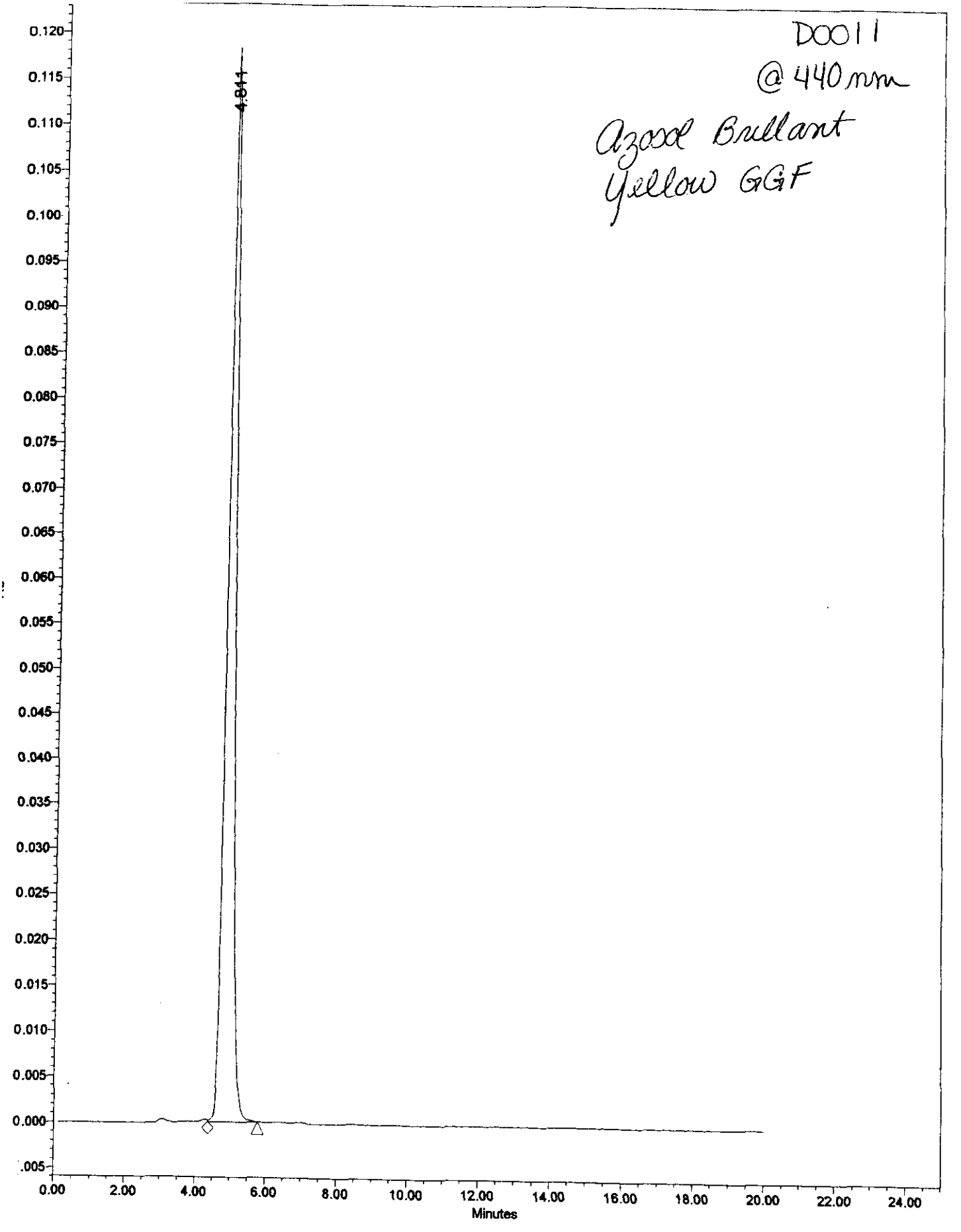
Sudan Scarlet 6G
@ 440nm

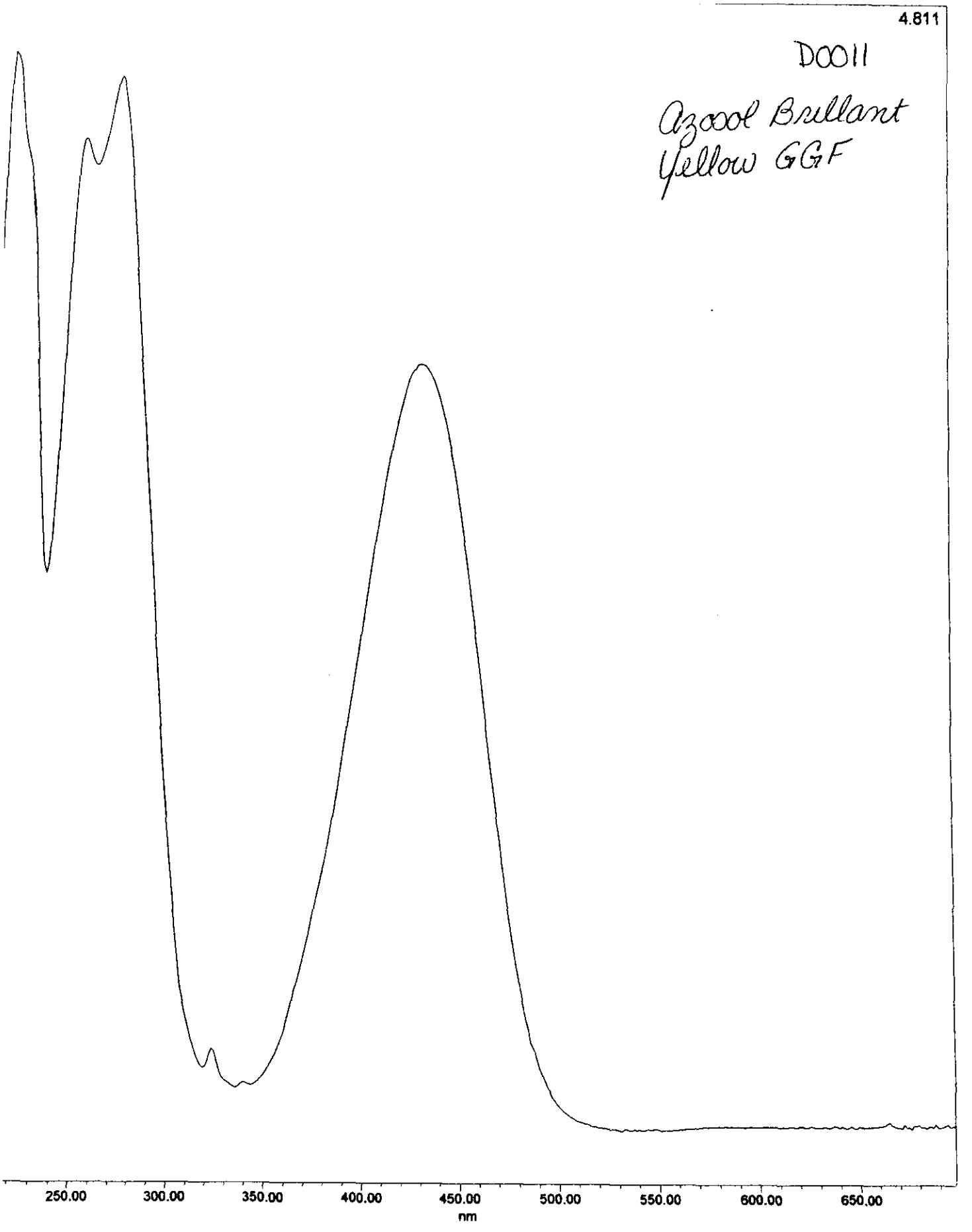



11.350
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm


D0018
@440nm
Brombenzan
9.110
AU
0.023
0.022
0.021
0.020
0.019
0.018
0.017
0.016
0.015
0.014
0.013
0.012
0.011
0.010
0.009
0.008
0.007
0.006
0.005
0.004
0.003
0.002
0.001
0.000
-0.001
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
22.00
24.00
Minutes

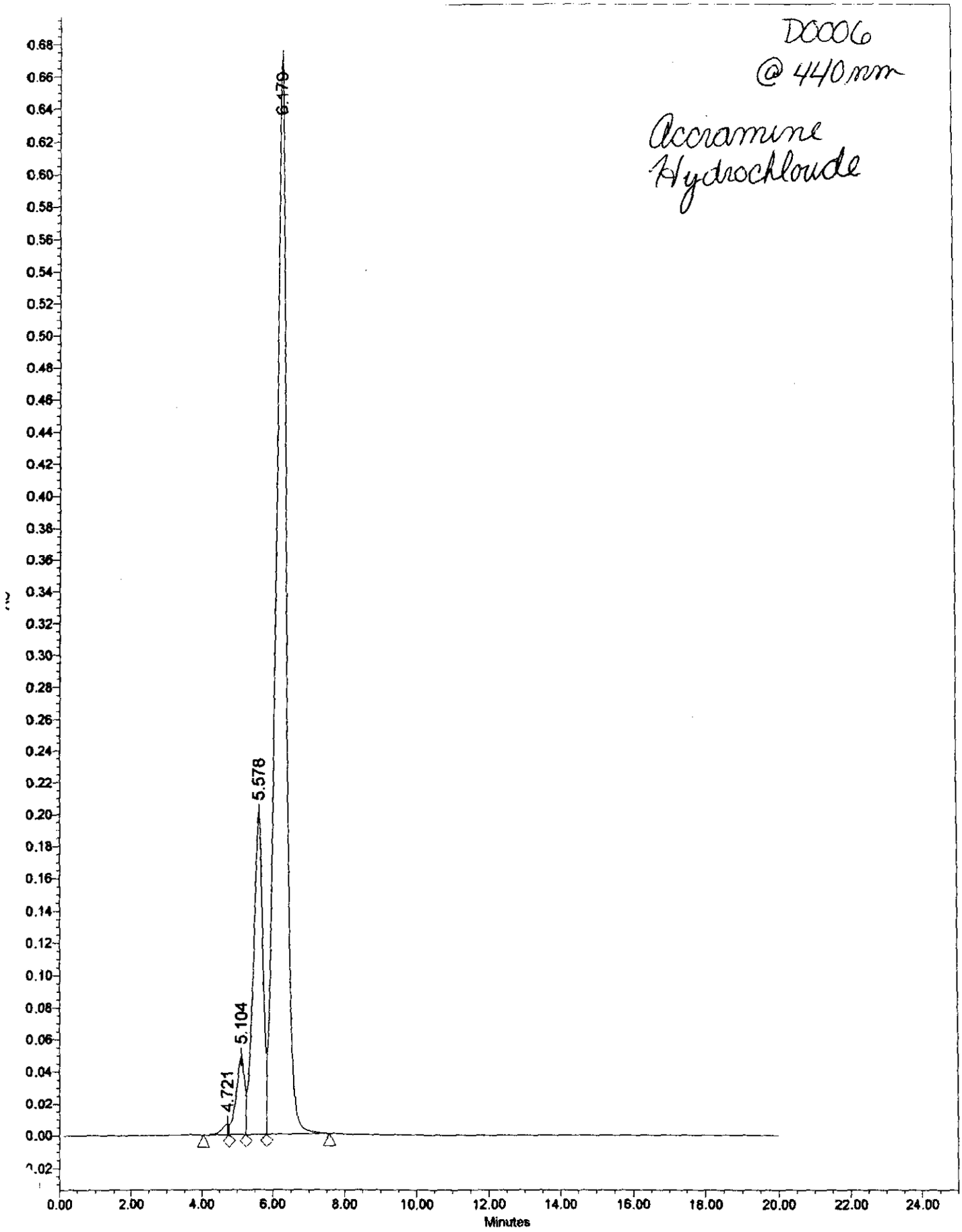

9.110
D001B
Brombenzan
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

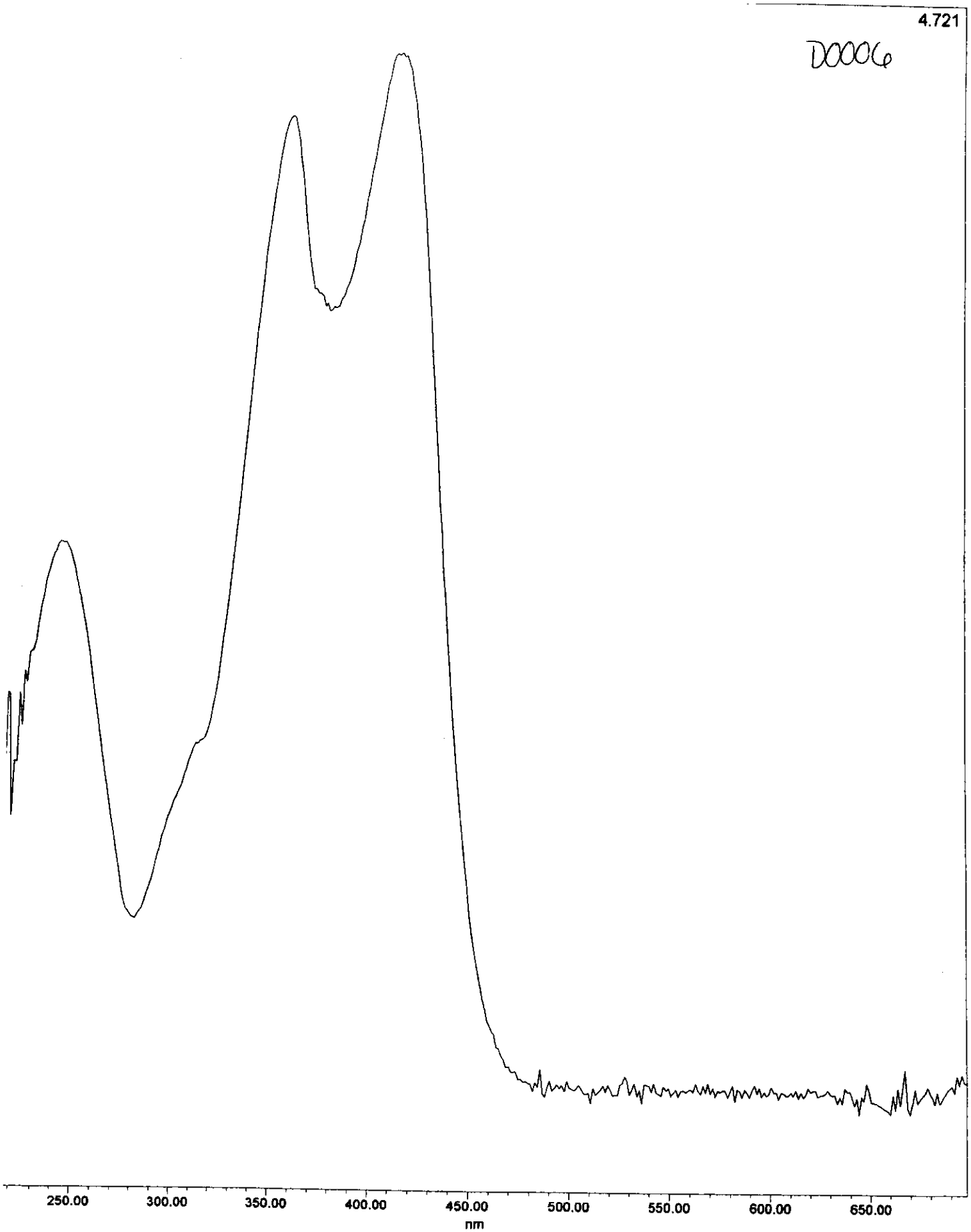

D0011
@ 440 nm
Azoic Brillant
Yellow GGF
4.841
0.120
0.115
0.110
0.105
0.100
0.095
0.090
0.085
0.080
0.075
0.070
0.065
0.060
0.055
0.050
0.045
0.040
0.035
0.030
0.025
0.020
0.015
0.010
0.005
0.000
.005
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
22.00
24.00
Minutes


4.811
D0011
Azoool Brillant
Yellow GGF
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

D0006
@ 440nm

Acramine Hydrochloride

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300.00
350.00
400.00
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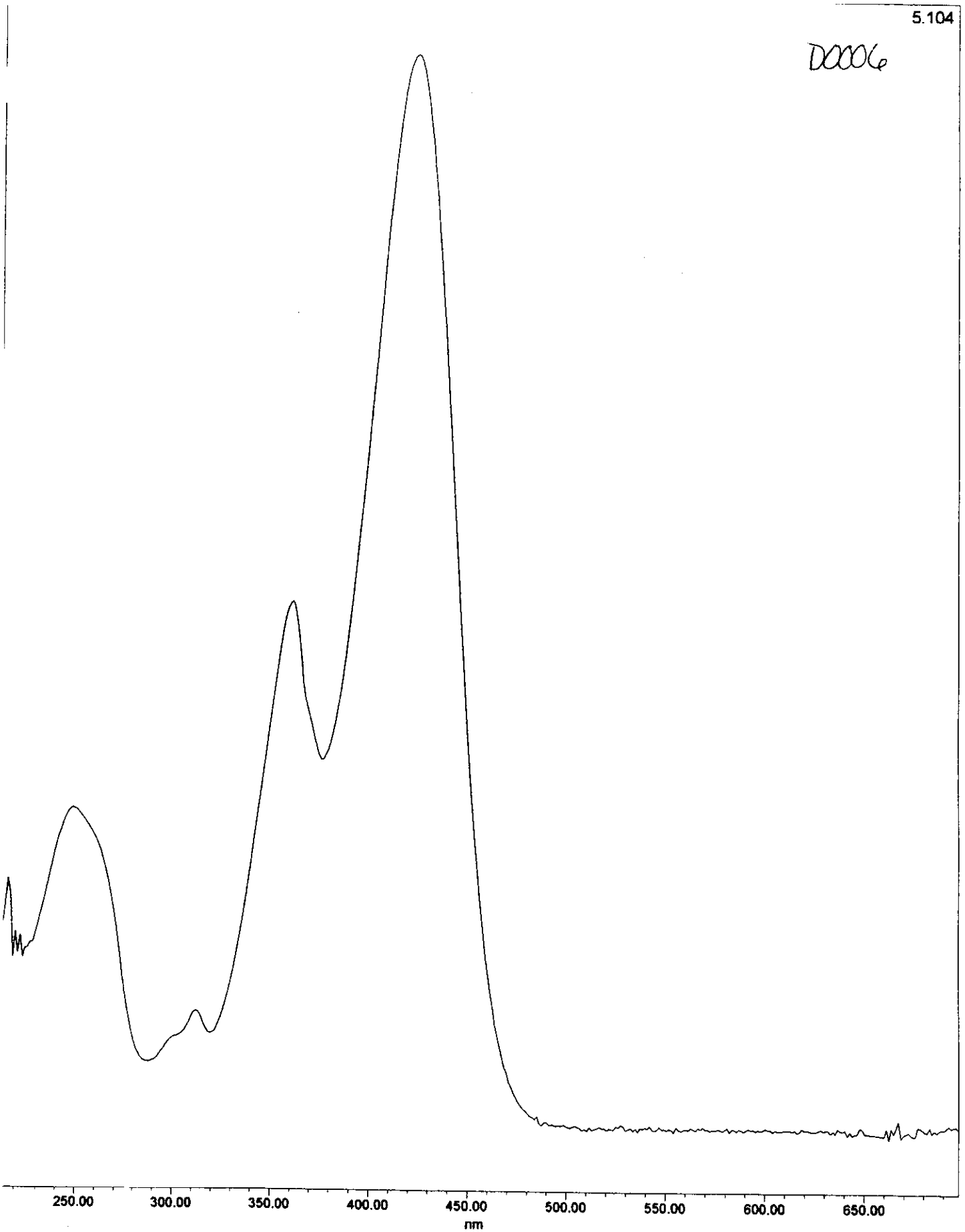
500.00
550.00
600.00
650.00
nm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550.00
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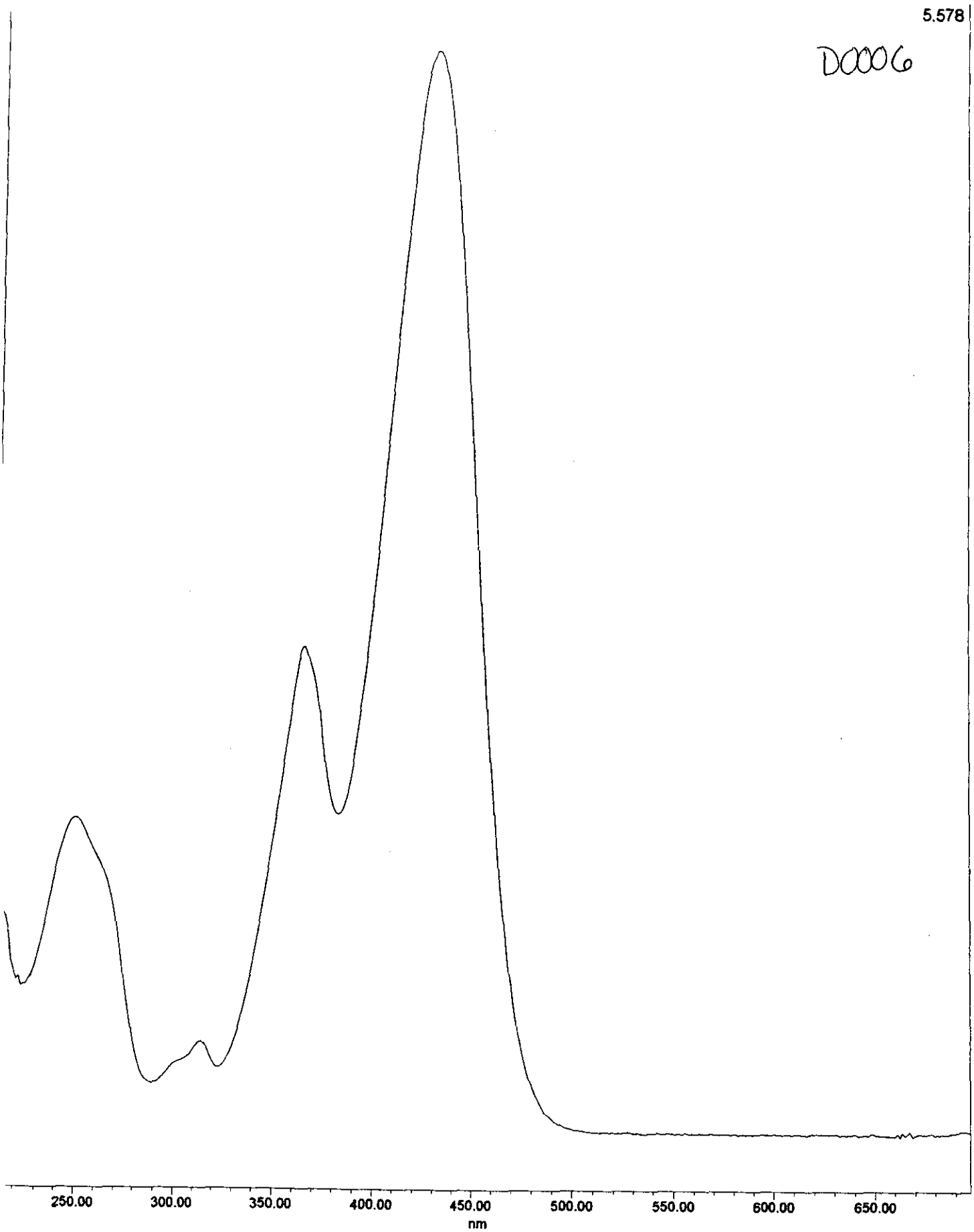
650.00
nm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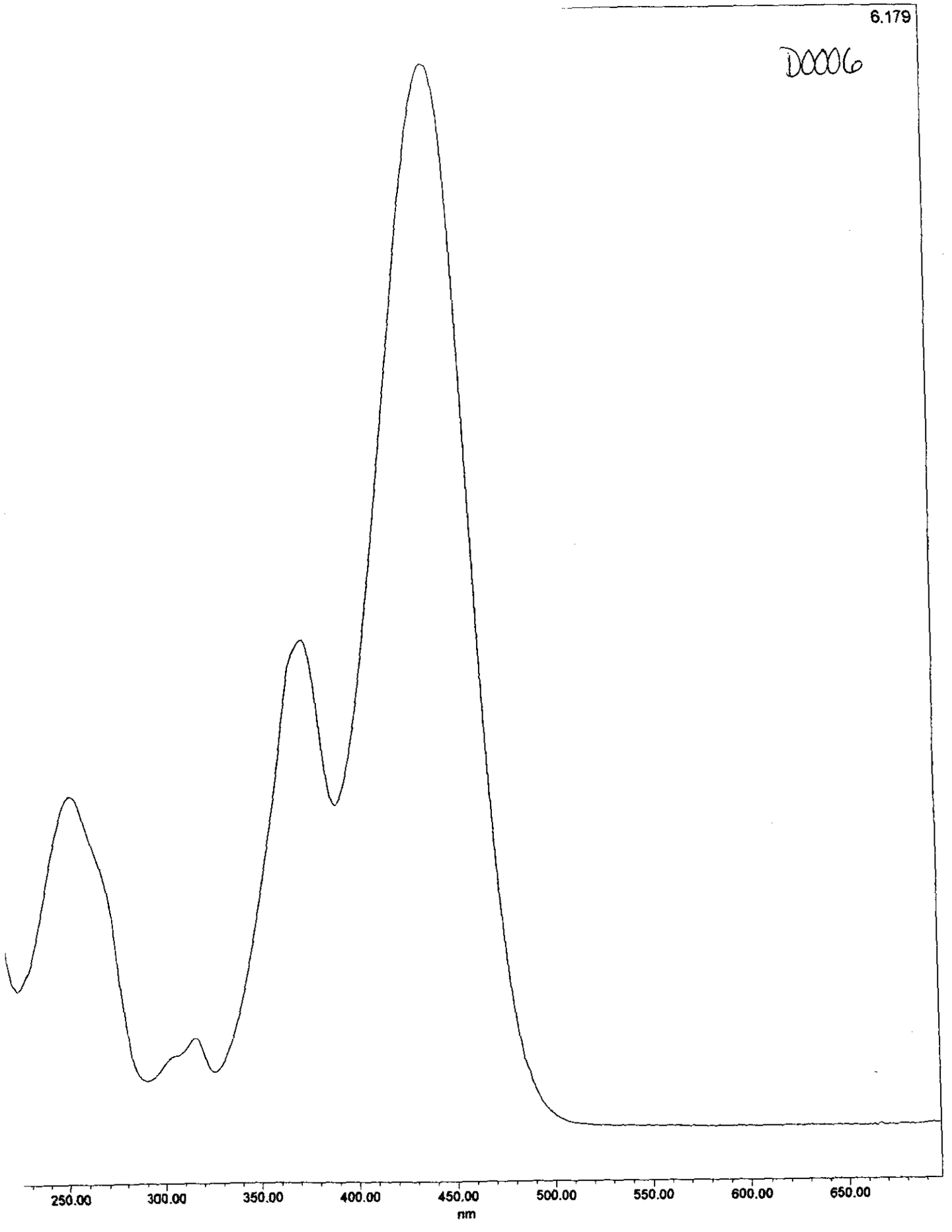
nm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D0006
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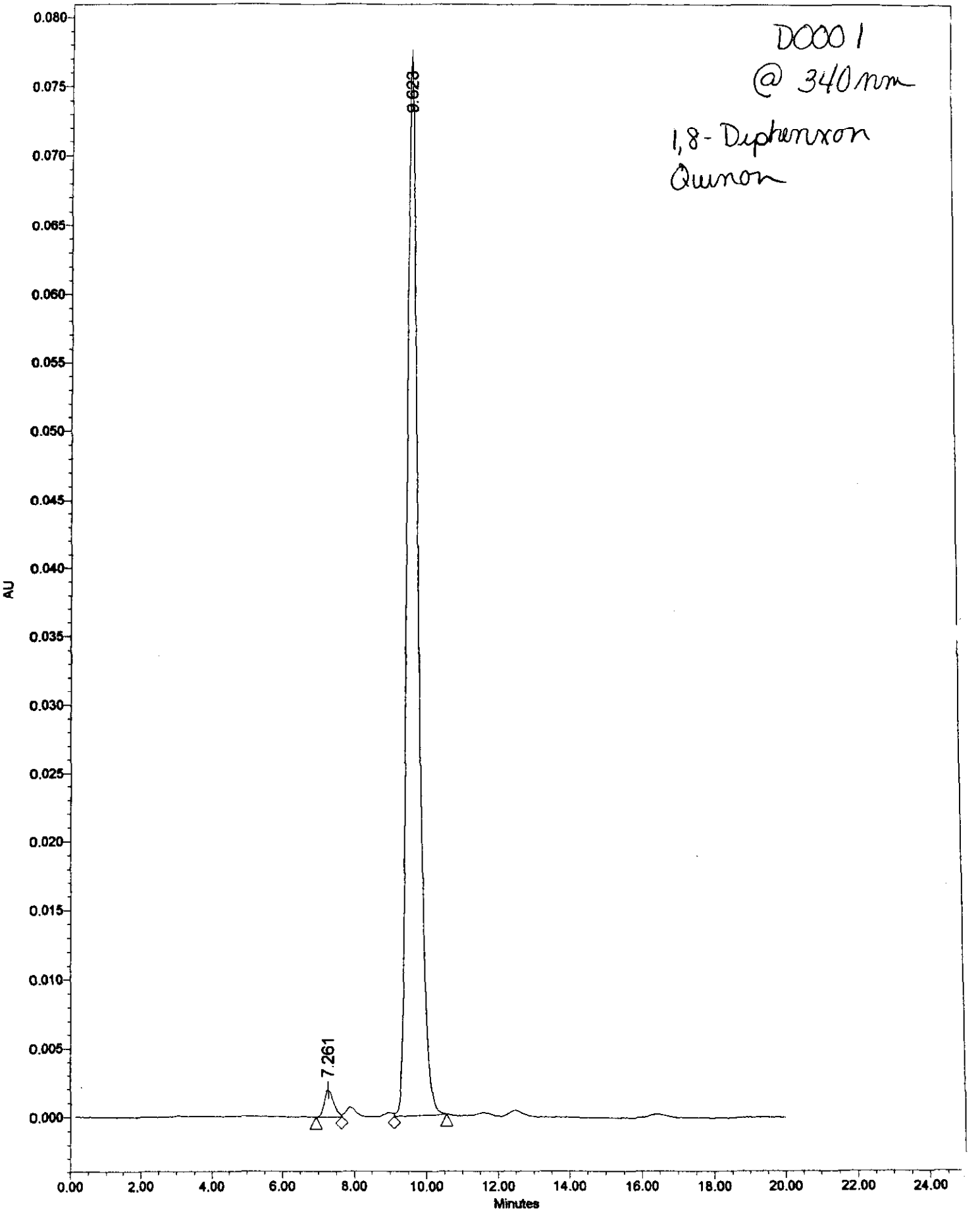
400.00
450.00
500.00
550.00
600.00
650.00
nm


D0001
@ 340nm
1,8-Diphenixon
Quinon
9.623
7.261
AU
Minutes
0.080
0.075
0.070
0.065
0.060
0.055
0.050
0.045
0.040
0.035
0.030
0.025
0.020
0.015
0.010
0.005
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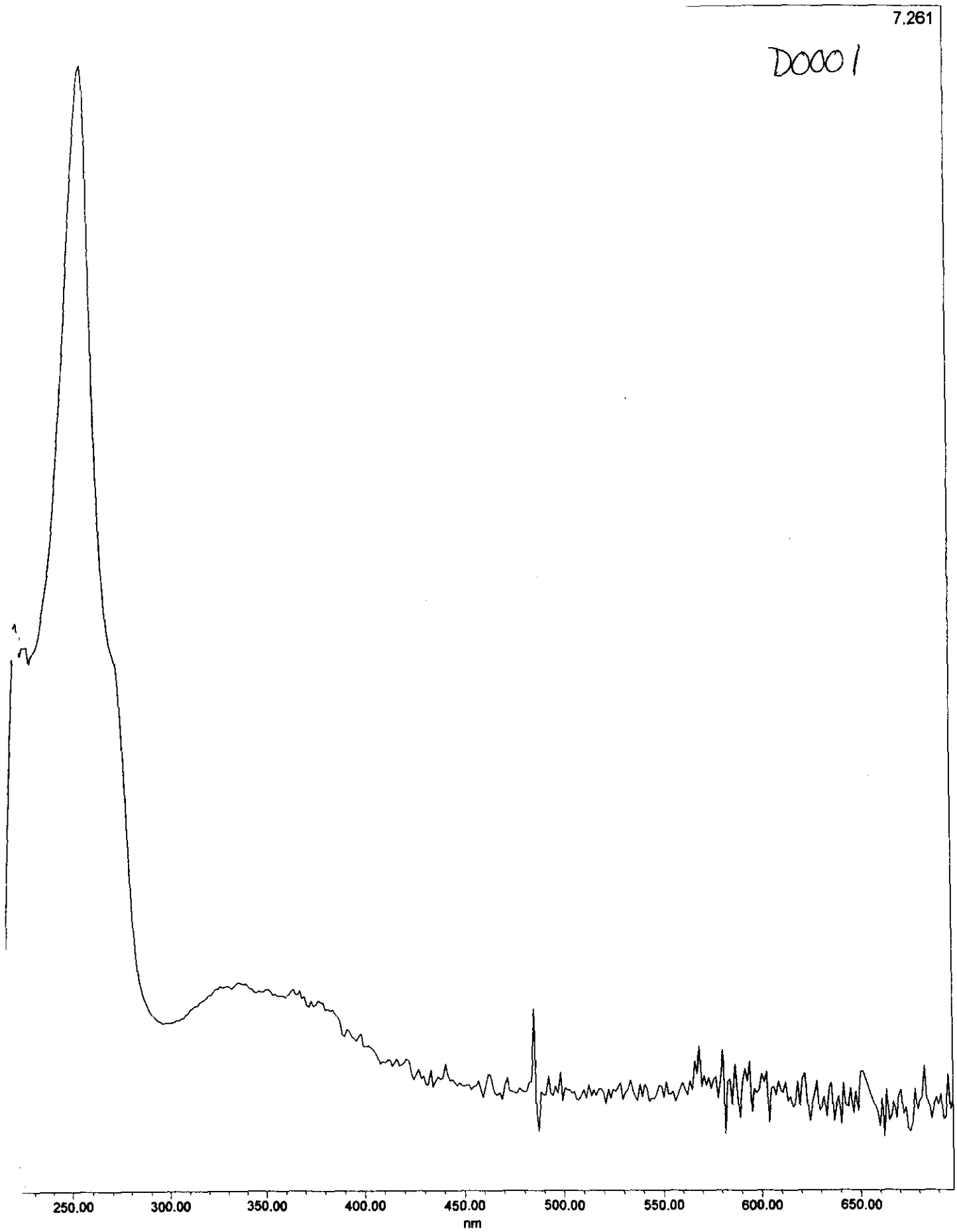
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D0001
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300.00
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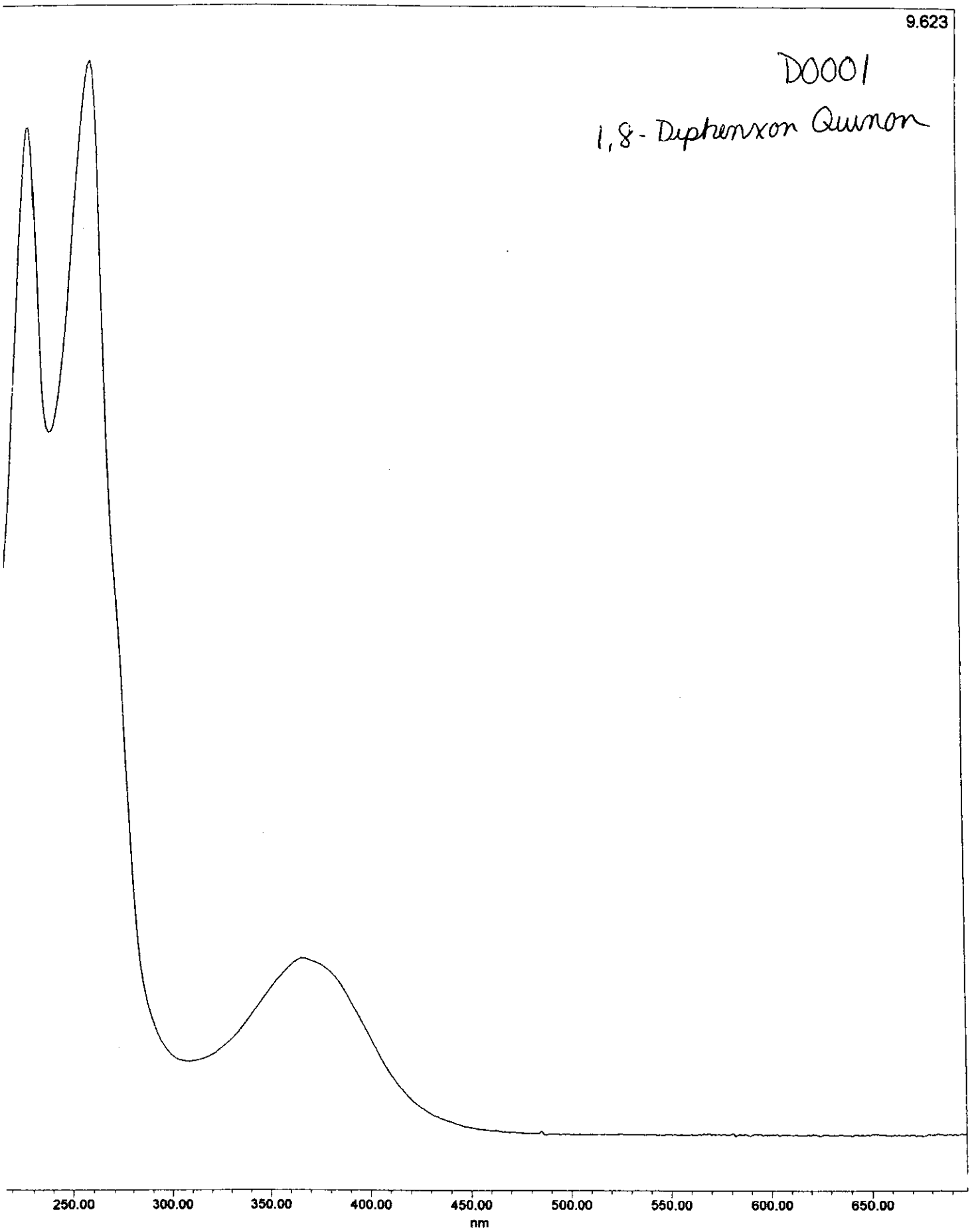
550.00
600.00
650.00
nm


9.623
D0001
1,8- Dipheniron Quinon
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

ATTACHMENT III

STANDARD SEARCH

ATTACHMENT III – STANDARD SEARCH

This attachment details the sources of the standards used during the method development phase. The following dyes were evaluated during method development but were not included in the final target compound list due to poor analytical and/or extraction performance:

Pigment Red 1 (could not be dissolved in measurable concentrations in water)
Smoke Dye (could not be dissolved in measurable concentrations in water)

Spectra for these compounds were added to the spectral library; however, it is not expected that these compounds can be recovered in the samples. If these compounds are present, quantification will be performed using a single point calibration.

Also included in this attachment is specific chemical information found during the Internet search.

SUMMARY OF CHEMICAL INFORMATION FOR DYES
SWMU 2 - DYE BURIAL GROUND
NSWC CRANE, INDIANA

Dye Name	CAS #	Crane Acronym	Color	Color Index #	Dye Classification	Trade/Chemical Name	Formula	Lab Standard Source
Water Soluble Dyes								
Acid Blue 1	129-17-9	AB1	blue	42045	Triarylmethane	Alphazurine 2G	$C_{27}H_{31}N_2NaO_6S_2$	ACROS
Acid Blue 9	3844-45-9	AB9		42090	Triarylmethane	Alphazurine FG	$C_{37}H_{34}N_2Na_2O_9S_3$	Abbey Color
Acid Blue 45	2861-02-1	AB45	blue	63010	Anthraquinone		$C_{14}H_8N_2Na_2O_{10}S_2$	Aldrich
Acid Red 64		AR64	red					ACROS
Acid Yellow 3	8004-92-0	AY3	yellow	47005	Quinoline	Quinoline yellow	$C_{18}H_8NNa_3O_{11}S_3$	ACROS
Acid Yellow 23	1934-21-0	AY23	yellow	19140	Monoazo	Tartrazine	$C_{16}H_9N_4Na_3O_9S_2$	ACROS
Acid Yellow 73	518-47-8	AY73	yellow(F)	45350	Xanthene	Fluorescien	$C_{20}H_{10}Na_2O_5$	ACROS
Acid Orange 10	1936-15-8	AO10	orange	16230	Monoazo	Orange G	$C_{16}H_{10}N_2Na_2O_7S_2$	ACROS
Basic Yellow 2	2465-27-2	BY2	yellow	41000	Diphenylmethane	Auramine hydrochloride	$C_{17}H_{22}ClN_3$	ACROS
Basic Violet 10	81-88-9	BV10	pink(F)	45170	Xanthene	Rhodamine B	$C_{28}H_{31}ClN_2O_3$	ACROS
Organic Soluble Dyes								
1-Aminoanthraquinone	82-45-1	1-AQ	orange	37275	Anthraquinone	1-aminoanthraquinone	$C_{14}H_9NO_2$	ACROS
2-Aminoanthraquinone	117-79-3	2-AQ	orange		Anthraquinone	2-aminoanthraquinone	$C_{14}H_9NO_2$	Aldrich
Disperse Blue 14	2475-44-7	DB14	blue	61500	Anthraquinone	Celliton Fast Blue B	$C_{16}H_{14}N_2O_2$	Aldrich
Disperse Red 9	82-38-2	DR9	red	60505	Anthraquinone	1-(methylamino)anthraquinone	$C_{15}H_{11}NO_2$	Aldrich
Disperse Violet 1	128-95-0	DV1	purple	61100	Anthraquinone	1,4-diaminoanthraquinone	$C_{14}H_{10}N_2O_2$	Aldrich
Pigment Red 1	6410-10-2	PR1	orange	12070	Monoazo	1-(4-nitrophenylazo)-2-naphthol	$C_{16}H_{11}N_3O_3$	Fisher Scientific
Smoke Dye	81-64-1	SD	red streak	58050	Anthraquinone	Quinizarine	$C_{14}H_8O_4$	ACROS
Solvent Green 3	128-80-3	SG3	blue	61565	Anthraquinone	Quinizarine green	$C_{28}H_{22}N_2O_2$	ACROS
Solvent Orange 3	495-54-5	SO3	orange		Monoazo	Chrysoidine Y base	$C_{12}H_{12}N_4$	Abbey Color
Solvent Orange 7	3118-97-6	SO7	red/orange	12140	Monoazo	Calco Oil Scarlet (Sudan II)	$C_{18}H_{16}N_2O$	ACROS
Solvent Red 1	1229-55-6	SR1	red	12150	Monoazo	1-(2-methoxyphenylazo)-2-naphthol	$C_{17}H_{14}N_2O_2$	TCI
Solvent Red 24	85-83-6	SR24	red	26105	Disazo	Oil Red (Sudan IV)	$C_{24}H_{20}N_4O$	ACROS
Solvent Yellow 2	60-11-7	SY2	yellow	11020	Monoazo	Dimethylaminoazobenzene	$C_{14}H_{15}N_3$	Phaltz & Bauer
Solvent Yellow 3	97-56-3	SY3	yellow	11160	Monoazo	Fast Garnet gbc base	$C_{14}H_{15}N_3$	ACROS
Solvent Yellow 14	842-07-9	SY14	orange	12055	Monoazo	Sudan I	$C_{16}H_{12}N_2O$	Aldrich
Solvent Yellow 33	8003-22-3	SY33	yellow	47000	Quinoline	Quinoline yellow	$C_{18}H_{11}NO_2$	ACROS

Organic Dyes

Lab #	Source(Ordered?)	Received (Y/N)	Name	Other Name	CAS#	CI#	Type	Physical Properties
Std(D0003)	Aldrich(YES)	Y	Disperse Blue 14	1,4-bis(methylamino)anthraquinone	2475-44-7	61500	Anthraquinone	266.3
Std(D0045)	Aldrich(YES)	Y	*Disperse Red 9*	1-methylaminoanthraquinone	82-38-2	60505	Anthraquinone	237.3
**	Aldrich(YES)	Y	*Disperse Violet 1*	1,4-diaminoanthraquinone/Solvent violet 11	128-95-0	61100	Anthraquinone	238.3
Std	ACROS(YES)	Y	Anthracene		120-12-7	N/A	Anthracene	178.23
Std	ACROS(YES)	Y	Azoic Diazo Component 36	1-aminoanthraquinone	82-45-1	37275	Anthraquinone	223.23
Std	ACROS(YES)	Y	Benzanthrone		82-05-3	N/A	Anthraquinone	230.27
Std	ACROS(YES)	Y	Smoke Dye (orange)	Quinizarin	81-64-1	58050	Anthraquinone	240.21
Std	ACROS(YES)	Y	*Solvent Green 3*	Quinizarine Green, SS	128-80-3	61565	Anthraquinone	418.5
Std	ACROS(YES)	Y	Solvent Orange 7	Sudan II	3118-97-6	12140	Monoazao	276.34
**	Abbey Color(YES)	Y	Solvent Orange 3		495-54-5	11270B	Monoazao	212
**	ACROS(YES)	Y	Solvent Yellow 3	2-aminoazotoluene	97-56-3	11160	Monoazao	225.29
**	Aldrich(YES)	Y	Solvent Yellow 14	Sudan I	842-07-9	12055	Monoazao	248.28
Std(D0050)	ACROS(YES)	Y	*Solvent Yellow 33*	Quinoline Yellow, spirit soluble	8003-22-3	47000	Quinoline	273.3
**	Pfaltz & Bauer(YES)	Y	Solvent Yellow 2	Butter Yellow	60-11-7	11020	Monoazao	225.29
**	Pfaltz & Bauer(ordered)	N	Solvent Violet 13	Alizurine Purple SS/Quinizarin Blue	81-48-1	60725	Anthraquinone	329.35
	TCI(YES)	Y	*Solvent Red 1*	Sudan R	1229-55-6	12150	Monoazao	278.3
Std	ACROS(YES)	Y	Solvent Red 24	Sudan IV	85-83-6	26105	Disazo	380.45
	TCI(YES)	Y	Indanthrene Yellow		129-09-9		Anthraquinone	474.6
	Fisher Scientific(YES)	Y	Pigment Red 1		6410-10-2	12070	Monoazao	293.28
**	Aldrich(YES)	Y	Vat Blue 1	Indigo	482-89-3	73000	Indigoid	262.27
Std (D0005)	Not found	N	Vat Yellow 4	Anthravat goldent yellow	128-66-5	59100	Anthraquinone	332.36
Std (D0002)	Pfaltz and Bauer(ordered)	N	1,4-Diamino-2,3 dihydroanthraquinone		81-63-0	N/A	Anthraquinone	240.26
**	Aldrich(YES)	Y	2-Aminoanthraquinone		117-79-3	N/A	Anthraquinone	223.23

Italics indicate used as smoke dyes

Water-Soluble Dyes

Lab #	Source(ordered?)	Received (Y/N)	Name	Other Name	CAS#	CI#	Type	
**	Aldrich(YES)	Y	Acid Blue 45		2861-02-1	63010	Anthraquinone	
Std	ACROS(YES)	Y	Acid Blue 1	Blue V	129-17-9	42045	Triphenylmethane	
	Abbey(YES)	Y	Acid Blue 9		3844-45-9	42090		
Std	ACROS(YES)	Y	Acid Orange 10	Orange G	1936-15-8	16230	Monoazao	452
Std	ACROS(YES)	Y	Acid Red 64				Monoazao	
Std	ACROS(YES)	Y	Acid Yellow 3	Quinoline Yellow, water soluble	8004-92-0	47005	Quinoline	579.41
Std	ACROS(YES)	Y	Acid Yellow 23	Tartazine	1934-21-0	19140	Monoazao	534
Std	ACROS(YES)	Y	Acid Yellow 73	Fluoresceine	518-47-8	45350	Xanthene	376
Std	ACROS(YES)	Y	Basic Violet 10	Brilliant Rhodamine	81-88-9	45170	Xanthene	478
Std	ACROS(YES)	Y	Basic Yellow 2	Auramine O	2465-27-2	41000	Ketone Imine	303.83
	Pfaltz and Bauer(ordered)	N	Anthrafurin	1,5-dihydroxyanthraquinone	117-12-4		Anthraquinone	240.21

Unknown Dyes

Organic Dyes

Lab #	Source	Received (Y/N)	Name	Other Name	CAS#	CI#	Type	
D0001	UNK	N	1,8-Diphenol Quinon		UNK	UNK	UNK	392
D0018	UNK	N	Bromobenzan		UNK	UNK	UNK	242
D0034	UNK	N	Para M		UNK	UNK	UNK	240
D0051	UNK	N	Stearated Yellow Chrome		UNK	UNK	UNK	
D0053	UNK	N	Sudan Scarlet 6G	Solvent Orange 7/Sudan II	3118-97-6	12140	Monoazao	276
D0054	UNK	N	Transketon	possibly same as Acid Red 64	UNK	UNK	UNK	222

Water-soluble Dyes

Lab #	Source		Name	Other Name	CAS#	CI#	Type	
D0037	UNK	N	Violet		UNK	UNK	UNK	240
D0036	UNK	N	Yellow		UNK	UNK	UNK	
D0035	UNK	N	Pyrazole Anthrone		UNK	UNK	UNK	
D0016	Abbey Color	Y	MIL-D-16627 Blue	contains Alphazurine B and Acid Fast Violet BG	UNK	UNK	UNK	
D0025	UNK	N	FEAA		UNK	UNK	UNK	

CambridgeSoft ChemFinder.Com ChemACX.Com ChemStore.Com ChemClub.Com ChemNews.Com

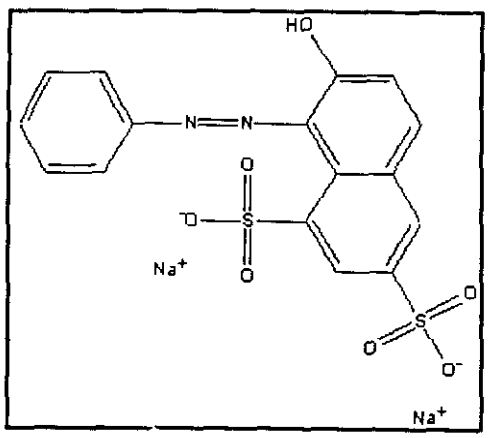
orange G

[1936-15-8]

Synonyms: C.I. 16230; Acid orange 10; C.I. acid orange 10; 7-hydroxy-8-(phenylazo)-1,3-Naphthalenedisulfonic acid, disodium salt; 2-Hydroxy-1-phenylazo-6,8-naphthalenedisulfonic acid, disodium salt; 1-phenylazo-2-naphthol-6,8-disulfonic acid, disodium salt; acidal fast orange; acid fast orange egg; acid fast orange g; acid leather orange kg; acid leather orange pgw; acid light orange g; acid light orange j; acid light orange ja export; acid light orange sx; acid orange g; acid orange 2g; acid orange gg; acilan orange gx; apocid orange 2g; atul acid crystal orange g; brasilan orange 2g; bucacid fast orange g; calcocid fast light orange 2g; certicol orange gs; cetil light orange gg; C.I. 27; C.I. acid orange 10, disodium salt; C.I. food orange 4; colacid orange g; crystal orange 2g; d & c orange no. 3; eniacid light orange g; erio fast orange as; fast light orange ga; fast light orange ga-cf; fast orange g; hexacol orange gg; hidacid fast orange g; hidacid fast orange 2g; hispacid fast orange 2g; java orange 2g; kiton fast orange g; Ink Orange JSN; intracid fast orange g; naphthalene fast orange 2g; naphthalene fast orange 2gs; naphthalene orange solide gg; neklacid fast light orange gg; neklacid fast orange 2g; orange #10; 1370 orange; orange bpc; orange 2g; orange g bpc; orange gg; orange gmp; schultz no. 39; solar light orange gx; standacol orange g; straight orange g; sulfacid light orange j; tertracid light orange g; unitertracid light orange g; vendacid light orange 2g; wool orange g; wool orange 2g; xylene fast orange g; Wood Orange 2G; Orange G, high purity biological stain

$C_{16}H_{10}N_2Na_2O_7S_2$

452.36



This picture is a live chemical image

ChemInfo Ltd. Web Edition offers you the same data as the ChemFinder WebServer, with faster access time and more powerful searching.

BUY

Melting Point (°C)	300-375	Specific Gravity	--
Boiling Point (°C)	--	Vapor Density	--
Evaporation Rate	--	Water Solubility	5-10 g/100 mL at 23.5 C
Flash Point (°C)	--	EPA Code	--
DOT Number	--	RTECS	QJ6500000
Comments	Orange microcrystals		

ChemInfo Searching is faster and more powerful. Click Here.

More information about this compound is available from

Acid Dyes
Acros Chemicals Catalog (with MSDSs)
    Orange G, high purity biological stain
    Orange G, certified
Available Chemicals Exchange
    Information about this particular substance
Berkeley Carcinogenic Potency Database
Dudley Chemical Corporation
    Information about this particular substance
Electron Microscopy Sciences
    Information about this particular substance
IARC Evaluations of Carcinogenicity to Humans
MSDS archive at the University of Utah
    Information about this particular substance
National Toxicology Program (NTP) publications
    Information about this particular substance
NTP Chemical Health and Safety Data
    Information about this particular substance
ProSciTech
    MSDS
    Uses

Return to searching

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Auramine O [2465-27-2]

Synonyms: Basic yellow 2; Auramine hydrochloride; 4,4'-bis(dimethylamino)benzhydrylidenimine hydrochloride; 4:4'-bis(dimethylamino)benzophenone-imine hydrochloride; C.I. 41000; C.I. basic yellow 2; adc auramine o; aizen auramine; aizen auramine oh; auramine a1; auramine fa; auramine lake yellow o; auramine n; auramine ii; auramine fwa; auramine 0-100; auramine chloride; auramine on; auramine oo; auramine ooo; auramine os; auramine sp; auramine yellow; 1,1-bis(p-dimethylaminophenyl)methylenimine hydrochloride; calcozine yellow ox; C.I. basic yellow 2, monohydrochloride; mitsui auramine o; 4,4'-(Imidocarbonyl)bis(N,N-dimethylaniline) monohydrochloride; Pyoctanunum aureum; Pyoktanin Yellow; Canary Yellow; Auramine O, biological stain

$C_{17}H_{22}ClN_3$
303.83

from RFI
auramine hydrochloride

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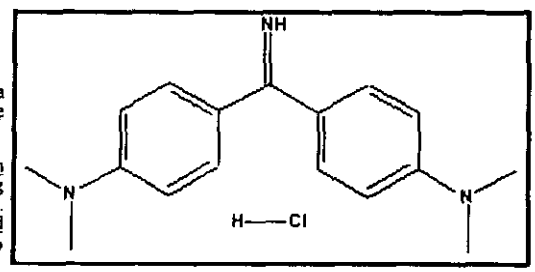
The ChemDraw Plugin lets you search by drawing structures in your web browser. Have you downloaded it yet?



Melting Point (°C)	267	Specific Gravity	--
Boiling Point (°C)	--	Vapor Density	--
Evaporation Rate	--	Water Solubility	<0.1 g/100 mL at 18 C
Flash Point (°C)	--	EPA Code	--
DOT Number	--	RTECS	BY3675000

Comments Yellow flakes or powder. Certified by the Biological Stain Commission for staining acid-fast bacteria in sputum or in paraffin sections of infected tissue.

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More information about this compound is available from

Available Chemicals Exchange
Information about this particular substance
Berkeley Carcinogenic Potency Database
Chemicals Inspection and Testing Service, Japan: Biodegradation and Bioaccumulation Data of Existing Chemicals
Information about this particular substance
Dudley Chemical Corporation
Information about this particular substance
Electron Microscopy Sciences
Information about this particular substance
EPA factsheets for regulated chemicals
Information about this particular substance

Existing Chemicals: Literature Reviews and Evaluations
    Information about this particular substance
Guide to NIOSH/OSHA Air Sampling Methods
    Information about this particular substance
MSDS archive at the University of Utah
    Information about this particular substance
NTP Chemical Health and Safety Data
    Information about this particular substance
Picatinny Arsenal
    Information about this particular substance
ProSciTech
    MSDS
    Uses
UMCP Partial list of mutagens

Return to searching

* not on list from Chem Lab *

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Rhodamine B [81-88-9]

Synonyms: Tetraethylrhodamine,hydrochloride; D&C Red No. 19; C.I. Food Red 15; Rhodamine B Extra; Rhodamine; C.I. Basic Violet 10; Basic Violet 10; C.I. 45170; 9-(2-carboxyphenyl)-3,6-bis (diethylamino)xanthylium chloride; C Red Number 19

$C_{28}H_{31}ClN_2O_3$
479.02

from RFI

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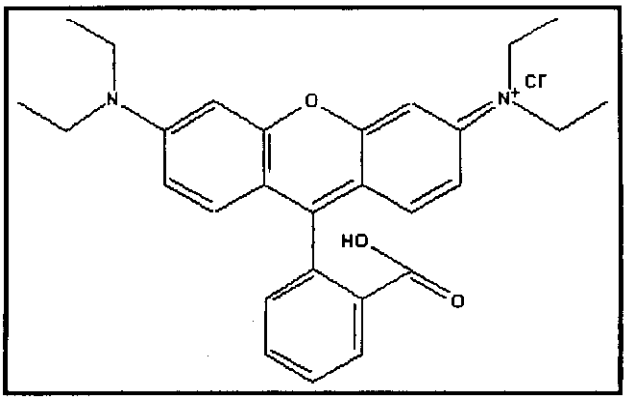

Melting Point (°C)	210-211	Specific Gravity	--
Boiling Point (°C)	--	Vapor Density	--
Evaporation Rate	--	Water Solubility	--
Flash Point (°C)	--	EPA Code	--
DOT Number	--	RTECS	BP3675000
Comments	Efficient laser dye for pulsed and CW operation.		

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More information about this compound is available from

8(e) TRIAGE Chemical Studies Database
ABCR GmbH&Co KG
    Rhodamine B
ATSDR Internet HazDat Site Contaminant Query
    Information about this particular substance
Available Chemicals Exchange
    Information about this particular substance
California EPA List of Lists
Current List of Toxics Release Inventory Chemicals
Dudley Chemical Corporation
    Information about this particular substance
Environmental Science Center database of Experimental Log P coefficients, with Ozone Depletion Potentials and Atmospheric Oxidation Rates
    Information about this particular substance

Existing Chemicals: Literature Reviews and Evaluations
    Information about this particular substance
Fluorochromes
IARC Evaluations of Carcinogenicity to Humans
MSDS archive at the University of Utah
    Information about this particular substance
NASA Department of Environmental Services List Of Lists of Regulated Chemicals
    Information about this particular substance
    Information about this particular substance
National (Canada) Pollutant Release Inventory
NIST Chemistry WebBook
    Information about this particular substance
OSHA Chemical Sampling and Methods
    Information about this particular substance
Picatinny Arsenal
    Information about this particular substance
Title III List of Lists
TRI Chemicals in other Federal Programs: Regulatory Matrix
University of California-Riverside list of compounds that must be reported

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2-Aminoanthraquinone [117-79-3]

Synonyms: AAQ; 2-amino-9,10-Anthracenedione; aminoanthraquinone; 2-amino-9,10-anthraquinone; beta-anthraquinonylamine; beta-aminoanthraquinone

$C_{14}H_9NO_2$
223.23

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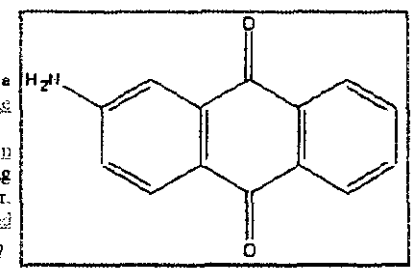

Melting Point (°C)	303-306	Specific Gravity	--
Boiling Point (°C)	(subl)	Vapor Density	--
Evaporation Rate	--	Water Solubility	Insoluble. <0.1 g/100 mL at 17 C
Flash Point (°C)	--	EPA Code	--
DOT Number	--	RTECS	CB5120000
Comments	Red needle-like crystals		

More information about this compound is available from

82 structural descriptors for NTP compounds
Available Chemicals Exchange
    Information about this particular substance
Berkeley Carcinogenic Potency Database
California EPA List of Lists
Chemicals Inspection and Testing Service, Japan: Biodegradation and Bioaccumulation Data of Existing Chemicals
    Information about this particular substance
Current List of Toxics Release Inventory Chemicals
EPA factsheets for regulated chemicals
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IARC Evaluations of Carcinogenicity to Humans
Known Carcinogens, 8th Annual Report on Carcinogens
    Information about this particular substance

NASA Department of Environmental Services List Of Lists of Regulated Chemicals
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National Toxicology Program (NTP) publications
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New Jersey Right to Know Hazardous Substance Fact Sheets
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Picatinny Arsenal
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Procedures for handling carcinogens at Rochester
Protocol Analytical Supplies, Inc. Single-component standards
Spectrum Laboratories, Inc.
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Search

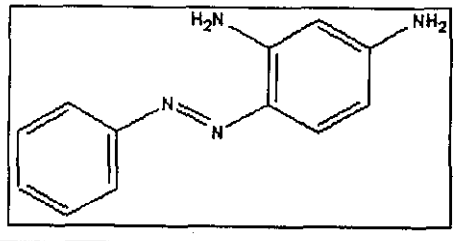
Substructure Query with Plug-In or Substructure Query with Java

2,4-Diaminoazobenzene [495-54-5]

Synonyms: 4-(Phenylazo)-1,3-benzenediamine; Chrysoidine Y base;

$C_{12}H_{12}N_4$

212.2536



ACX Number	I1027140	CAS RN	495-54-5
Melting Point (°C)		Density	
Boiling Point (°C)		Vapor Density	
Refractive Index		Vapor Pressure	
Evaporation Rate		Water Solubility	
Flash Point (°C)		EPA Code	
DOT Number		RTECS	
Comments			

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Sudan II [3118-97-6]

Synonyms: C.I. Solvent Orange 7; 1-[(2,4-dimethylphenyl)azo]-2-Naphthalenol; C.I. 12140; 1-(2,4-Dimethylphenylazo)-2-naphthol; Solvent Orange 7; Oil Red XO; 1-(2-Hydroxy-1-naphthylazo)-2,4-dimethylbenzene

$C_{18}H_{16}N_2O$
276.34

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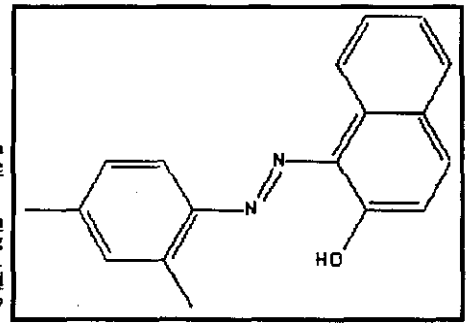

Melting Point (°C)	156-158	Specific Gravity	--
Boiling Point (°C)	--	Vapor Density	--
Evaporation Rate	--	Water Solubility	--
Flash Point (°C)	--	EPA Code	--
DOT Number	--	RTECS	QL5850000
Comments			

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ABCR GmbH&Co KG
Sudan II, 98%
Acros Chemicals Catalog (with MSDSs)
Sudan II
California EPA List of Lists
Current List of Toxics Release Inventory Chemicals
IARC Evaluations of Carcinogenicity to Humans
Japanese Cosmetic Color Index
Information about this particular substance
NASA Department of Environmental Services List Of Lists of Regulated Chemicals
Information about this particular substance
National (Canada) Pollutant Release Inventory
NIST Chemistry WebBook
Information about this particular substance
Title III List of Lists
TRI Chemicals in other Federal Programs: Regulatory Matrix

* not on list from Crane Lab *

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Quinoline Yellow

[8004-92-0]

Synonyms: E 104; C.I. Acid Yellow 3; C.I. 47005; D&C Yellow No. 10; Acid Yellow 3; Food Yellow 13; Quinoline Yellow, water soluble; Quinoline Yellow WS; Sodium 2-(1,3-dioxoindan-2-yl)quinolinedisulfonate; 2-(1,3-Dioxoindan-2-yl)quinolinedisulfonic acid sodium salt

BUY

Melting Point (°C)	--	Specific Gravity	--
Boiling Point (°C)	--	Vapor Density	--
Evaporation Rate	--	Water Solubility	--
Flash Point (°C)	--	EPA Code	--
DOT Number	--	RTECS	GC5796000
Comments			

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Existing Chemicals: Literature Reviews and Evaluations
Information about this particular substance
Japanese Cosmetic Color Index
Information about this particular substance

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from RFI

quinoline yellow

Fluorescein, sodium derivative, sodium salt [518-47-8]

Synonyms: C.I. acid yellow 73; Soap yellow; C.I. 45350 disodium salt; Soluble fluorescein; Fluorescein Sodium; 3',6'-dihydroxyspiro[isobenzofuran-1(3H),9'-(9H)xanthene]-3-one, disodium salt; Fluorone; fluorescein, disodium salt; 9-o-carboxyphenyl-6-hydroxy-3-isoxanthone, disodium salt; disodium 6-hydroxy-3-oxo-9-xanthene-o-benzoate; sodium salt of hydroxy-o-carboxy-phenyl-fluorone; sodium fluoresceinate; aizen uranine; calcocid uranine b4315; certiqual fluoresceine; C.I. 766; C.I. 45350 sodium salt; d&c yellow no. 8; fluorescein sodium b.p; fluorescein, water soluble; fluor-i-strip a.t.; ful-glo; funduscein; furanium; hidacid uranine; resorcinol phthalein sodium; Uranine A Extra; Uranine O; Uranine SS; Uranine USP XII; Uranine WSS; Uranine Yellow; 11824 yellow; 12417 yellow; Acid Yellow 73; Uranine; C.I. 45350; Dihydroxyspiro[isobenzofuran-1 (3H),9'-(9H)xanthen]-3-one disodium salt; Yellow no. 8; 3,6-Dihydroxyxanthene-9-spiro-1'-3'H-isobenzofuran-3'-one; 3,6-Dihydroxyxanthene-9-spiro-1'-3'H-isobenzofuran-3'-one disodium salt; 3',6'-Dihydroxy-spiro[3H-isobenzofuran-1,9'-xanthen]-3-one disodium salt; 9-(o-Carboxyphenyl)-3,6-dihydroxyxanthylium disodium salt; Uranine K; 3,6-Dihydroxyxanthene-9-spiro-1'-3'H-isobenzofuran-3'-one dipotassium salt; 9-(o-Carboxyphenyl)-3,6-dihydroxyxanthylium dipotassium salt; Fluorescein W/S

$$C_{20}H_{10}Na_2O_5$$
376.28

Melting Point (°C)	315-395	Specific Gravity	--
Boiling Point (°C)	--	Vapor Density	--
Evaporation Rate	--	Water Solubility	>=10 g/100 mL at 20 C
Flash Point (°C)	--	EPA Code	--
DOT Number	--	RTECS	LM5425000
Comments	Orange-red powder. HYGROSCOPIC. Water flow patterns.		

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3D Molecular Modeling Homepage of Tokyo University of Pharmacy and Life Science
- Information about this particular substance

ABCR GmbH&Co KG
- Fluorescein, sodium salt
- Fluorescein disodium salt, 98+%

Acid Dyes

Acros Chemicals Catalog (with MSDSs)
- Fluorescein, disodium salt dihydrate, 90% (UV-VIS)
- Fluorescein, disodium salt

ATSDR Internet HazDat Site Contaminant Query
- Information about this particular substance

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Sudan IV

[85-83-6]

Synonyms: Oil Red; Scarlet Red; 1-[[2-methyl-4-[(2-methylphenyl)azo]phenyl]azo]-2-Naphthalenol; C.I. Solvent Red 24; C.I. 26105; Scarlet Red Scharlach; 1-[2-Methyl-4-(2-methylphenylazo)phenylazo]-2-naphthol; Solvent Red 24; Lipid crimson; Oil Red BB; 4-(2-Hydroxy-1-naphthylazo)-2,3-dimethylazobenzene; Scarlet Red N.F

$C_{24}H_{20}N_4O$
380.45

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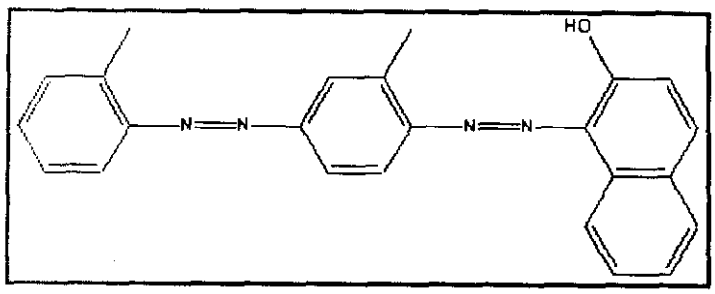

Melting Point (°C)	199	Specific Gravity	--
Boiling Point (°C)	260	Vapor Density	--
Evaporation Rate	--	Water Solubility	--
Flash Point (°C)	--	EPA Code	--
DOT Number	--	RTECS	QL5775000
Comments			

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Scarlet Red
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Sudan IV, high purity biological stain
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ProSciTech
MSDS
User
Proton NMR Chemical Molecular Formula Index

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FD&C Yellow No. 5 [1934-21-0]

Synonyms: Tartrazine; Food Yellow No.4; C.I. 19140; Acid Yellow 23; Filter Yellow; 4,5-dihydro-5-oxo-1-(4-sulfophenyl)-4-[(4-sulfophenyl)azo]-1H-Pyrazole-3-carboxylic acid, trisodium salt; Dihydro-5-oxo-1-(4-sulfophenyl)-4-((4-sulfophenyl)azo)-1H-pyrazole-3-carboxylic acid, trisodium salt; Pyrazole-3-carboxylic acid, 4,5-dihydro-5-oxo-1-(4-sulfophenyl)-4-((4-sulfophenyl)azo)-, trisodium salt; Tartrazine C; Trisodium 1-(4-sulfophenyl)-4-((4-sulfophenyl)azo)-1H-pyrazole-3-carboxylate; Trisodium 1-(4-sulfonatophenyl)-4-(4-sulfonatophenylazo)-5-pyrazolone-3-carboxylate; C.I. Acid Yellow 23; Tartrazine C.I.19140

$C_{16}H_9N_4Na_3O_9S_2$

534.36

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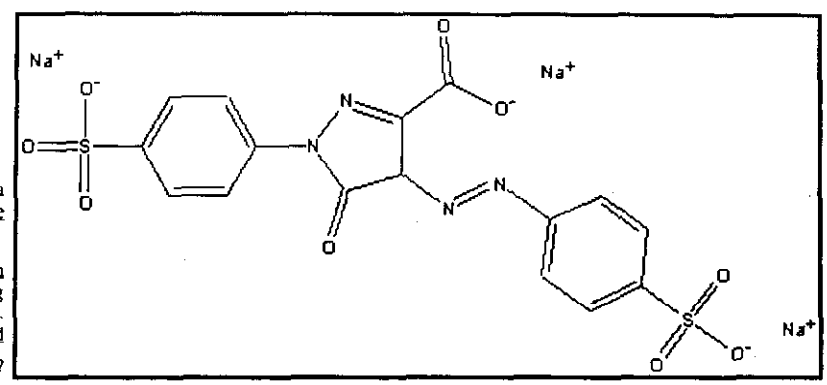

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Melting Point (°C)	--	Specific Gravity	--
Boiling Point (°C)	--	Vapor Density	--
Evaporation Rate	--	Water Solubility	--
Flash Point (°C)	--	EPA Code	--
DOT Number	--	RTECS	UQ6400000

Comments: HYGROSCOPIC.

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California EPA List of Lists
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NASA Department of Environmental Services List Of Lists of Regulated Chemicals
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Search

Substructure Query with Plug-In or Substructure Query with Java

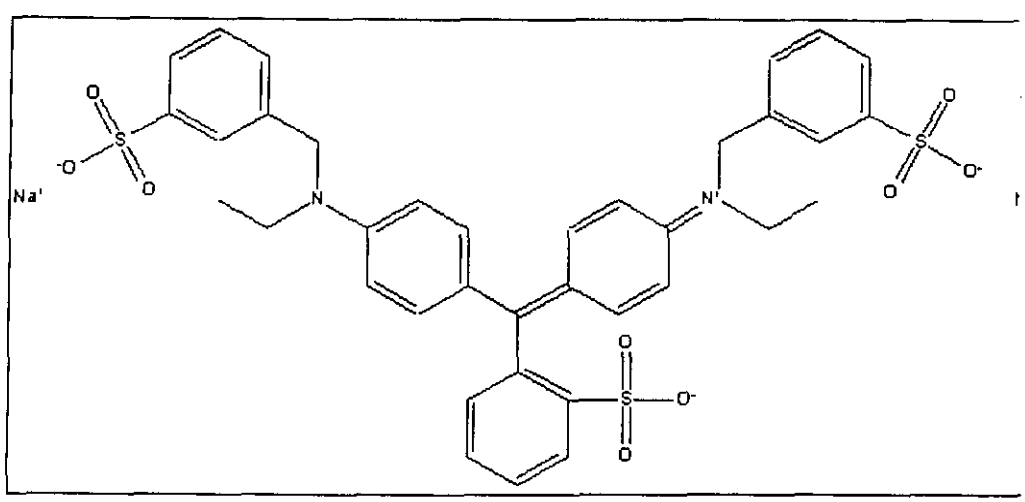
FD&C Blue No. 1

[3844-45-

Synonyms: Brilliant blue FCF; Food Blue No.1; Brilliant blue FCF, disodium salt; Benzenemethanaminium, N-ethyl-N-[4-[[4-[ethyl[(3-sulfophenyl)methyl]amino]phenyl](2-sulfophenyl methylene]-2,5-cyclohexadien-1-ylidene]-3-sulfo-, inner salt, disodium salt; Erioglaucine disodium salt C.I. 42090; Alphazurine FG; Acid Blue 9, disodium salt; Erioglaucine; Food Blue 2; Disodium bis[4-(N ethyl-N-3-sulfonatophenylmethyl)aminophenyl]-2- sulfonatophenylmethylium; Bis[4-(N-ethyl-N-3-sulfophenylmethyl)aminophenyl]-2-sulfophenylmethylium disodium salt; Erioglaucine disodium salt (C.I. 42090);

$C_{37}H_{34}N_2Na_2O_9S_3$

792.84314



BUY

ACX Number	I1005351	CAS RN	3844-45-9
Melting Point (°C)	283	Density	
Boiling Point (°C)		Vapor Density	
Refractive Index		Vapor Pressure	
Evaporation Rate		Water Solubility	
Flash Point (°C)		EPA Code	
DOT Number		RTECS	BQ4725000
Comments	Coloring.		

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Health

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California EPA List of Lists

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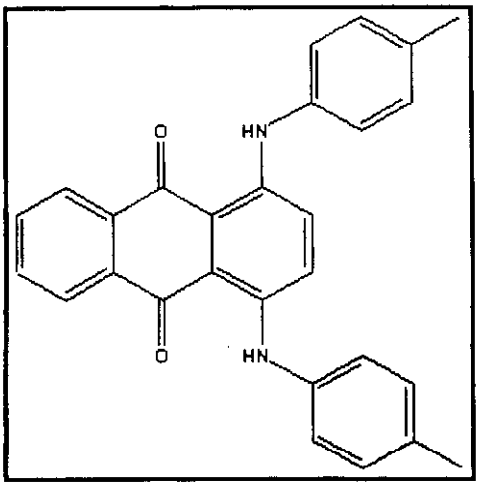
9,10-Anthracenedione, 1,4-bis[(4-methylphenyl) amino]- [128-80-3]

Synonyms: Solvent Green 3; 1,4-Bis(p-tolylamino)anthraquinone; C.I. 61565; Oil Green G; C.I. Solvent Green 3; Quinizarine Green SS; D & C Green No.6; 1,4-Bis(p-toluidino)anthraquinone

$C_{28}H_{22}N_2O_2$
418.49

from RFI:
4-di-p-toluidinoanthraquinone



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Melting Point (°C)	220-221	Specific Gravity	--
Boiling Point (°C)	--	Vapor Density	--
Evaporation Rate	--	Water Solubility	--
Flash Point (°C)	--	EPA Code	--
DOT Number	--	RTECS	CB5775000
Comments			

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Disperse Violet 1 [128-95-0]

Synonyms: 1,4-Diaminoanthraquinone

$C_{14}H_{10}N_2O_2$
238.25

·F1
-diaminoanthraquinone

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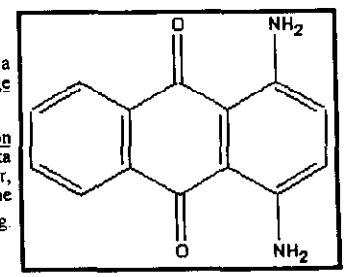

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Melting Point (°C)	--	Specific Gravity	--
Boiling Point (°C)	--	Vapor Density	--
Evaporation Rate	--	Water Solubility	--
Flash Point (°C)	--	EPA Code	--
DOT Number	--	RTECS	--
Comments			

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ABCR GmbH&Co KG
1,4-Diaminoanthraquinone, tech., 90%
Available Chemicals Exchange
Information about this particular substance
Environmental Science Center database of Experimental Log P coefficients, with Ozone Depletion Potentials and Atmospheric Oxidation Rates
Information about this particular substance
Existing Chemicals: Literature Reviews and Evaluations
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NIST Chemistry WebBook
Information about this particular substance

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Disperse Red 9 [82-38-2]

Synonyms: 1-(N-Methylamino)-9.10-anthraquinone; 1-(Methylamino)anthraquinone

$C_{15}H_{11}NO_2$
237.26

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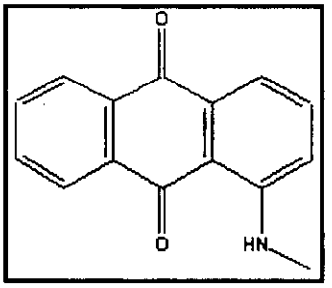

Melting Point (°C)	--	Specific Gravity	--
Boiling Point (°C)	--	Vapor Density	--
Evaporation Rate	--	Water Solubility	--
Flash Point (°C)	--	EPA Code	--
DOT Number	--	RTECS	--
Comments			

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Environmental Science Center database of Experimental Log P coefficients, with Ozone Depletion Potentials and Atmospheric Oxidation Rates
Information about this particular substance
NIST Chemistry WebBook
Information about this particular substance
Proton NMR Spectral Molecular Formula Index
Information about this particular substance

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Celliton Fast Blue B

[2475-44-7]

Synonyms: 1,4-Bis(methylamino)anthraquinone

$C_{16}H_{14}N_2O_2$
266.30

Disperse Blue 14
61500

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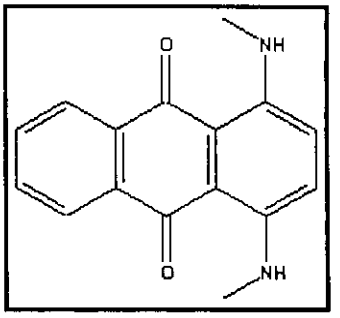

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Melting Point (°C)	--	Specific Gravity	--
Boiling Point (°C)	--	Vapor Density	--
Evaporation Rate	--	Water Solubility	--
Flash Point (°C)	--	EPA Code	--
DOT Number	--	RTECS	--
Comments	A blue dye		

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Available Chemicals Exchange
Information about this particular substance

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Enter a chemical name, CAS Number, molecular formula, or molecular weight

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Substructure Query with Plug-In or Substructure Query with Java

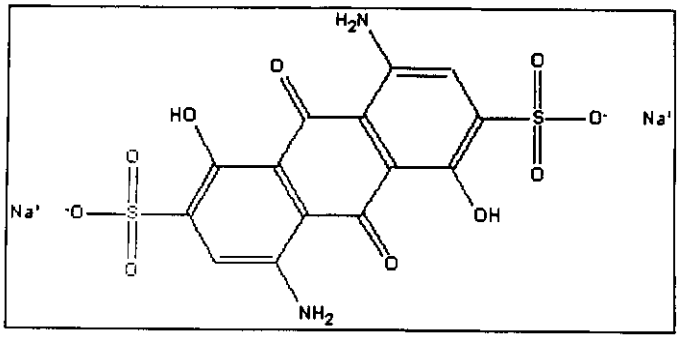
4,8-diamino-9,10-dihydro-1,5-dihydroxy-9,10-dioxo-2,6-Anthracenedisulfonic acid, disodium salt

[2861-02-1]

Synonyms: Acid Blue 45; C.I. 63010; 4,8-Diamino-1,5-dihydroxyanthraquinone-2,6-disulfonic acid, disodium salt; C.I. Acid Blue 45;

$C_{14}H_8N_2Na_2O_{10}S_2$

474.32414



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ACX Number	I1011257	CAS RN	2861-02-1
Melting Point (°C)		Specific Gravity	
Boiling Point (°C)		Vapor Density	
Refractive Index		Vapor Pressure	
Evaporation Rate		Water Solubility	
Flash Point (°C)		EPA Code	
DOT Number		RTECS	CB0548500
Comments			

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Biochemistry

Chemicals Inspection and Testing Service, Japan: Biodegradation and Bioaccumulation Data of Existing Chemicals

Information about this particular compound

Chemical Online Order

Available Chemicals Exchange

Information about this particular compound

Misc

Acid Dyes

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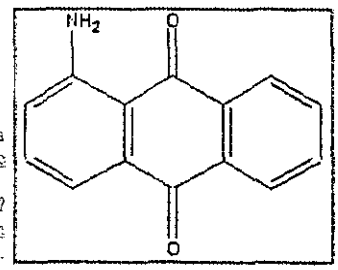
1-Aminoanthraquinone [82-45-1]

Synonyms: 9,10-Anthracenedione, 1-amino-; C.I. 37275

$C_{14}H_9NO_2$

223.23



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Melting Point (°C)	253-255	Specific Gravity	--
Boiling Point (°C)	--	Vapor Density	--
Evaporation Rate	--	Water Solubility	--
Flash Point (°C)	--	EPA Code	--
DOT Number	--	RTECS	CB5075000
Comments			

More information about this compound is available from

Available Chemicals Exchange
Information about this particular substance
Environmental Science Center database of Experimental Log P coefficients, with Ozone Depletion Potentials and Atmospheric Oxidation Rates
Information about this particular substance
Existing Chemicals: Literature Reviews and Evaluations
Information about this particular substance
NIST Chemistry WebBook
Information about this particular substance
UMCP Partial list of mutagens
US Customs Intermediate Chemicals for Dyes Appendix to the Harmonized Tariff Schedule

Poehlein Sara K CNIN

Ms. Betty Puckerin
Research chemist
at Ampacet

From: Puckerin, Betty [BPuckerin@Ampacet.com]
Sent: Tuesday, July 27, 1999 2:25 PM
To: 'Poehlein Sara K CNIN'
Subject: RE: dyes

So far...
The identification does not identify the material. It sounds like an internal code of some sort. I'm still looking for General Dye stuff. One supplier gave the name of a company that disposes their "unknown" stuff.

ADIRONDACK LAB
TCLT test done to determine toxicity
518-434-4546

Testing tends to be expensive. It's a start. They may be able to answer some of your testing questions. Still looking.
Betty

> -----Original Message-----
> From: Poehlein Sara K CNIN [SMTP:poehlein_sara@crane.navy.mil]
> Sent: Tuesday, July 20, 1999 9:39 AM
> To: Betty Puckerin (E-mail)
> Subject: dyes
>
> Betty,
>
> I have some questions about dyes. Respond to this email if you get this
> and
> I will call you.
>
> sara

Hawman Forrest J CNIN

From: George Baughman [GBAUGHMA@hestia.fcs.uga.edu]
Sent: Tuesday, August 03, 1999 4:31 AM
To: hawman_forrest@crane.navy.mil
Subject: Dye Info

Your D0011 is a solvent dye, probably Solvent Yellow 44 which is Azosol Brilliant Yellow 6GF. If so, it is the same as Disperse Yellow 11. The D0053 is probably Sovent Orange 7. I could find nothing on the others. General Dyestuff corp. was a New York Co. It may have become GAF. You might call Dr. Reiff at home 908-349-3230. He is an old guy who will talk your ear off but he knows more about the dye industry than anyone else I know. Don't call Tue or Thur.Tell him I referred you. The other names are probably not commercial names.

Hawman Forrest J CNIN

From: Atul Americas [aai@bellsouth.net]
Sent: Thursday, January 28, 1999 8:51 AM
To: Hawman Forrest J CNIN
Subject: dye analysis information

Dear Sir,

We refer to your E-mail received today. This office is a marketing/sales office for the dyes and chemcials that we manufacture in India. We do not have any manufacturing facilities here in the US and we therefore, do not have any support staff in terms of chemcial analysis, etc. in the US. We regret we were unable to help you in this situation.

Best Regards,

Atul Americas Inc.

Fax

Name:	Dr. Ed Majeski
Organization:	Pfaltz & Bauer
Fax:	203-574-3181
Phone:	203-574-0075
From:	Jacquelyn Kurty
Date:	June 15, 1999
Subject:	Dyes
Pages:	1

Comments:
Dr. Majeski,

Attached is a list of the dyes that we have in our lab. The "Label and Manufacturer" column is what is physically written on the bottles. The "Possible ID" column is what we think they are after going through our 1963 version of the Colour Index. I also included the copies of the Military specifications (MIL-D or MIL-Std). We have used these to help identify some of the dyes. You have been provided with everything we know about the dyes on our shelves.

Back ground:

Between 1940-1965 dyes used in production on center were buried as a means of disposal. The dyes we have in house may or may not be what is actually out there. The dyes we have in house we samples taken off our production lines that were operated at the time.

We are now trying to develop a method for analyzing and quantitating the amount of dye out there. We are trying to use HPLC with a photodiode array detector to do this. We are currently working on a method that will at least separate the dyes we have in house (if you have any suggestions, they would be VERY much appreciated).

Tetra Tech wanted us to contact you about obtaining dyes standards. Their idea is to categorize the dyes by type (i.e. monoazo, diazo, triphenylmethane, anthraquinone, xanthene). They want us to obtain one dye standard for each of categories that is certified (known amount of dye) to do detection limits, method detection limits, and % recovery studies. Do you have any dyes that would meet their requirements? I was specifically interested in Acid Yellow 73, Acid Yellow 23, Acid Blue 1, Solvent Orange 7, Basic Violet 10, and Solvent Red 24.

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1	2035743181	6-16-99	6:49	8'27"	14/ 17	EC	TERMINAL FAULT 9600	0P77 053F
		TOTAL		0:08'27"	14			

NOTE:
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PD : POLLED BY REMOTE SF : STORE & FORWARD RI : RELAY INITIATE RS : RELAY STATION
MB : SEND TO MAILBOX PG : POLLING A REMOTE MP : MULTI-POLLING RM : RECEIVE TO MEMORY

Fax

Name:	Linda Karsonovich
Organization:	Tetra Tech NUS
Fax:	412-921-4040
Phone:	412-921-8729
From:	Jacquelyn Vorhauer
Date:	May 9, 1999
Subject:	Dye Project
Pages:	15

Comments: Attached is the MIL-D-16627 specification. As you can see, MIL-D-16627 is a combination of 5 dyes. The standards we have in house are for the individual colors (i.e. we have MIL-D-16627 blue color which is a mix of Alphazurine B and Acid Fast Violet BG, etc).

Following the specification is a copy of the pages out of the "1963 Supplement for Revised Commercial Names- Colour Index" where I had initially identified Alphazurine B and Acid Fast Violet BG as Acid Blue 26 and 34.

The last attachment is from the 1956 edition of the Colour Index which I did not look through until recently. There again it identifies the trade names as being the ones I listed above. However, it contains the conversion table from the old 3 digit CI numbers to the new 5 digit CI number. It identifies Acid Fast Violet BG (old CI 699) as being Acid Blue 75. Given this discrepancy, I feel we should leave MIL-D-16627 blue color as is and not try to apply a name/CAS # to it without known standards to verify the ID.

Sorry to add to the confusion.

Jackie

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		TOTAL	0:11'24"	16		

NOTE:
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MB : SEND TO MAILBOX PG : POLLING A REMOTE MP : MULTI-POLLING RM : RECEIVE TO MEMORY

Fax

Name:	Dr. Ed Majeski
Organization:	Pfaltz & Bauer
Fax:	203-574-3181
Phone:	203-574-0075
From:	Jacquelyn Karty
Date:	June 15, 1999
Subject:	Dyes
Pages:	1

Comments:
Dr. Majeski,

I think there may have been a misunderstanding on what I meant when I said "certified dyes." We are looking for dyes that you can provide us with a certificate of analysis for. That way we will know exactly how much is dye and how much is by-products/fillers. This will satisfy the EPA's requirement for quantitative analysis. We do not care if the dye was made synthetically or naturally. Just as long as it is the same chemical structure as the ones in our list.

All of the fax that I sent you last week did not go through. I sent the entire documentation through the mail. I hope you have received it by now. If not, let me know.

Thank you,
Jacquelyn Karty
Chemist
ESB-NSWC Crane Division
812-854-3143
vorhauerjj@crane.navy.mil]

ATTACHMENT IV

CALIBRATION STUDY

ATTACHMENT IV – CALIBRATION STUDY

Included in this attachment is an overview of the calibration information from the method validation phase.

The calibration information includes percent relative standard deviations (%RSDs), retention times, continuing calibration verifications (CCVs), and the analytical sequences for both the short and long gradients.

INITIAL CALIBRATIONS - RELATIVE STANDARD DEVIATIONS

SW 846 Method 8000 Section 7.5 specifies a %RSD less than 20% as a criterion for determining linearity. In cases where one or more analytes exceeds 20% RSD, the initial calibration may still be acceptable if the mean of the RSD values for all analytes in the calibration is less than or equal to 20%. Both the long and short gradient calibrations meet this criterion for acceptance.

The laboratory used a linear calibration model for the compounds. SW846 Method 8000 Section 7.5.2 specifies that the correlation coefficient (r) must be greater than or equal to 0.99. Both the long and short gradient calibrations meet this criterion for acceptance.

Included in this section are summary tables for both the long and short gradient highlighting %RSD, correlation coefficient, and coefficient of determination information for each compound. The calculations to support each summary table are also included.

Long Gradient

Two dyes Acid Blue 45 (26.5%) and Solvent Red 24 (47.0%) exceeded the 20% acceptance criterion. However, the mean %RSD is only 7.2%. Correlation coefficients are 0.99 or better for all compounds. The initial calibration is acceptable for identification and quantitation of the compounds.

Short Gradient

Two dyes, Acid Red 64 (43.6%) and Solvent Red 24 (26.7%) exceeded the 20% acceptance criterion. However, the mean %RSD is only 5.9%. Correlation coefficients are 0.99 or better for all compounds. The initial calibration is acceptable for identification and quantitation of the compounds.

CONTINUING CALIBRATION VERIFICATIONS (CCV) – PERCENT DIFFERENCES

SW 846 Method 8000 Section 7.7 specifies an acceptance range of +/- 15% from the initial calibration factors as a criterion for verifying the validity of the initial calibration over time. In keeping with the approach for initial calibrations, if the average of the response for all analytes is within 15% of the initially established values, then the initial calibration has been verified to be acceptable.

Included in this section are four summary tables. The first tables (one each for short and long gradient) depict the percent drift (area of ICAL versus area of CCV) and percent difference (amount ICAL versus calculated amount CCV). Also included in the tables are the retention times and retention time windows. The second tables (one each for short and long gradient) depict the percent differences for each CCV over time. This demonstrates the stability of the initial calibration over the length of the analytical sequence.

Long Gradient

All average continuing calibration percent differences (%Ds) were within the +/- 15% acceptance range. In general, individual %Ds were within the +/- 15% range except for Acid Blue 45 (%D = 31.6%, 24.3%, and 24.2%), Acid Yellow 23 (%D = 34.4% and 40.5%), Disperse Violet I (%D = 23.2% and 25.5%), and Solvent Red 24 (%D = 6.4%). The continuing calibration standards successfully verified the validity of the initial calibration over a seven day period Due to poor response of the compound, the Laboratory remade the standard for Acid Red 64 (AR64) and analyzed the initial calibration curve for this compound at the end of the analytical sequence. Therefore, continuing calibration data for this compound are not available.

Short Gradient

All average continuing calibration %Ds were within the +/- 15% acceptance range. In general, individual %Ds were less than 15% except for Acid Yellow 23 (%D = 15.8% and 73.1%), Disperse Violet I (%D = 23.1%, 24.8%, and 25.7%), Acid Yellow 3 (%D = 26.7%), and Solvent Green 3 (%D = 19.5% and 20.7%). The continuing calibration standards successfully verified the validity of the initial calibration over a 10 day period. Due to poor response of these compounds, the Laboratory remade the standards for AR64, Acid Blue 45 (AB45), and Solvent Red 24 (SR24) and analyzed the initial calibration curve for these compounds at the midpoint of the analytical sequence. Therefore, continuing calibration data for these compounds are not available.

RETENTION TIMES

The laboratory evaluated the performance of two chromatographic columns, Water's Xtera column and Varian's Microsphere column. The Xtera is suitable for use in the pH range of 2 to 10 and is better able than the Microsphere column to tolerate the pH of the sample extracts. The Microsphere column performs well for short periods of time but does not have the ruggedness to last for the length of an entire analytical sequence.

Two graphs are included in this section. The long gradient graph shows the elution order of the compounds on the Xtera column. Compounds with similar heights co-elute or elute very closely on the chromatograph. The short gradient displays the same information. However, included on this graph are the results for the Microsphere column

As shown below, there are 3 pairs of dye constituents that may co-elute for the long and short gradient methods. Co-elution is highly dependent on the age and condition of the analytical column. The co-eluting compounds differ for the two gradient methods. If any of the co-eluting dyes are detected, they will be quantified by the gradient method in which they do not co-elute. The continuing calibration verification standards will be used to assess co-elution throughout the analytical sequence.

Long Gradient

The Crane Laboratory provided a table of the retention times for each dye standard. Resolution of the compounds is good with the possible following exceptions:

Acid Yellow 3 (Peak 1)	14.813
Acid Red 64	14.923
Acid Blue 9	22.626
Disperse Violet I	22.685
Basic Violet 10	36.254
Solvent Yellow 3	36.396

The long gradient method will be used to identify and quantitate any positive results found during the short gradient analysis for aqueous samples and co-eluting dyes for soil samples. If co-eluting peaks are present in the long gradient chromatograms, those compounds will be quantitated using the short gradient results

Short Gradient

Resolution of the compounds is good with the following possible exceptions.

Disperse Red 9	20.419
Benzanthrone (surrogate)	20.5
Solvent Yellow 14	24.530
Solvent Red 1	24.741
Solvent Green 3	31.219
Solvent Red 24	31.255

Resolution of the dye compounds by the short gradient method is sufficient to identify the presence of dye compounds. Where target compounds are present, the sample extracts will be quantitated by the long gradient method for both aqueous samples and co-eluting dyes for soil samples.

Tables Contained in Attachment IV

Table IV - 1	Initial Calibration Summary for the Long Gradient
Table IV - 1A	RSD Calculations for the Long Gradient
Table IV - 2	Initial Calibration Summary for the Short Gradient
Table IV - 2A	RSD Calculations for the Short Gradient
Table IV - 3	HPLC Run Sequence for the Long Gradient
Table IV - 3A	Percent Difference and Percent Drift for the Long Gradient CCVs
Table IV - 3B	Long Gradient Percent Differences Over Time
Table IV - 4	HPLC Run Sequence for the Short Gradient
Table IV - 4A	Percent Difference and Percent Drift for the Short Gradient CCVs
Table IV - 4B	Short Gradient Percent Differences Over Time
Table IV - 5	Retention Time Graph Long Gradient
Table IV - 6	Retention Time Graph Short Graident

TABLE IV-1

INITIAL CALIBRATION SUMMARY FOR THE LONG GRADIENT

DYE	RSD	R^2	R
ACID BLUE 45	26.5	0.992128	0.996056
ACID YELLOW 23	4.5	0.999995	0.999997
ACID YELLOW 73	0.7	0.999983	0.999991
ACID ORANGE 10	3.8	0.999974	0.999987
ACID BLUE 1	0.7	0.999985	0.999992
BASIC YELLOW 2	3.1	0.999917	0.999958
DISPERSE VIOLET 1	1.3	0.999827	0.999914
SOLVENT YELLOW 33	4.4	0.999652	0.999826
DISPERSE BLUE 14	8.4	0.997818	0.998908
ACID RED 64	10.9	0.999007	0.999503
ACID BLUE 9	2.3	0.999878	0.999939
BASIC VIOLET 10	3.6	0.999847	0.999923
2-AMINOANTHRAQUINONE	1.9	0.999865	0.999932
SOLVENT ORANGE 3	2.8	0.999842	0.999921
1-AMINOANTHRAQUINONE	2.6	0.999803	0.999902
SOLVENT YELLOW 14	2.7	0.999876	0.999938
SOLVENT ORANGE 7	1.3	0.999757	0.999878
ACID YELLOW 3	12.4	0.999565	0.999783
SOLVENT YELLOW 3	3.1	0.999607	0.999533
BENZANTHRONE	2.9	0.999306	0.999653
ANTHRACENE	4.4	0.999812	0.999906
SOLVENT RED 1	12.0	0.991517	0.995750
DISPERSE RED 9	4.5	0.999859	0.999929
SOLVENT YELLOW 2	1.6	0.999949	0.999974
SOLVENT GREEN 3	17.8	0.980733	0.990320
SOLVENT RED 24	47.0	0.995607	0.997801

AVERAGE %RSD 7.2

TABLE IV-1A

RSD CALCULATIONS FOR THE LONG GRADIENT
PAGE 1 OF 3

Dye	Units	AB45	AY23	AY73	AO10	AB1	BY2	DV1	SY33	DB14
Level 1	ug/mL	2.325	2.513	2.263	2.513	2.663	2.313	1.626	1.748	1.869
Level 2	ug/mL	4.650	5.025	4.525	5.025	5.325	4.625	3.253	3.495	3.738
Level 3	ug/mL	9.300	10.050	9.050	10.050	10.650	9.250	6.505	6.990	7.475
Level 4	ug/mL	18.600	20.100	18.100	20.100	21.300	18.500	13.010	13.980	14.950
Level 5	ug/mL	37.200	40.200	36.200	40.200	42.600	37.000	26.020	27.960	29.900
Vol Inj	uL	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0
Mass 1	ug	0.02325	0.02513	0.02263	0.02513	0.02663	0.02313	0.01626	0.01748	0.01869
Mass 2	ug	0.0465	0.05025	0.04525	0.05025	0.05325	0.04625	0.03253	0.03495	0.03738
Mass 3	ug	0.093	0.1005	0.0905	0.1005	0.1065	0.0925	0.06505	0.0699	0.07475
Mass 4	ug	0.186	0.201	0.181	0.201	0.213	0.185	0.1301	0.1398	0.1495
Mass 5	ug	0.372	0.402	0.362	0.402	0.426	0.37	0.2602	0.2796	0.299
Area 1		20474	62722	297418	68241	193542	12494	71634	68606	53545
Area 2		75401	136019	589586	144068	383837	25600	141972	142878	105535
Area 3		163913	276126	1184169	293902	766740	52347	283266	290972	221889
Area 4		369962	558545	2386132	594451	1552268	105944	571245	592682	467538
Area 5		650194	1125841	4807228	1207553	3118063	216620	1171309	1235940	1034010
CF (1)		880602.15	2495901.3	13142643	2715519.3	7267818.3	540164.29	4405535.1	3924828.4	2864901
CF (2)		1621526.9	2706845.8	13029525	2867024.9	7208206.6	553513.51	4364340.6	4088068.7	2823301.2
CF (3)		1762505.4	2747522.4	13084740	2924398	7199436.6	565913.51	4354588.8	4162689.6	2968414.7
CF (4)		1989043	2778830.8	13183050	2957467.7	7287643.2	572670.27	4390814.8	4239499.3	3127344.5
CF (5)		1747833.3	2800599.5	13279635	3003863.2	7319396.7	585459.46	4501571.9	4420386.3	3458227.4
Avg CF		1.600E+06	2.706E+06	1.314E+07	2.894E+06	7.257E+06	5.635E+05	4.403E+06	4.167E+06	3.048E+06
(CF (1)-CF (AVG))^2		5.180E+11	4.412E+10	1.628E+06	3.173E+10	1.281E+08	5.466E+08	4.687E+06	5.869E+10	3.369E+10
(CF (2)-CF (AVG))^2		4.505E+08	8.205E+05	1.309E+10	7.091E+08	2.332E+09	1.006E+08	1.523E+09	6.245E+09	5.069E+10
(CF (3)-CF (AVG))^2		2.631E+10	1.729E+09	3.502E+09	9.452E+08	3.256E+09	5.614E+06	2.380E+09	1.940E+07	6.404E+09
(CF (4)-CF (AVG))^2		1.511E+11	5.313E+09	1.531E+09	4.072E+09	9.699E+08	8.328E+07	1.576E+08	5.242E+09	6.226E+09
(CF (5)-CF (AVG))^2		2.177E+10	8.960E+09	1.842E+10	1.215E+10	3.956E+09	4.803E+08	9.644E+09	6.416E+10	1.679E+11
SUM CF-CF(AVG)		7.176E+11	6.012E+10	3.654E+10	4.960E+10	1.064E+10	1.216E+09	1.371E+10	1.344E+11	2.649E+11
SD		4.236E+05	1.226E+05	9.558E+04	1.114E+05	5.158E+04	1.744E+04	5.854E+04	1.833E+05	2.574E+05
RSD		**26.5**	**4.5**	**0.7**	**3.8**	**0.7**	**3.1**	**1.3**	**4.4**	**8.4**

TABLE IV-1A

RSD CALCULATIONS FOR THE LONG GRADIENT
PAGE 2 OF 3

Dye	Units	AR64	AB9	BV10	2AQ	SO3	1AQ	SY14	SO7	AY3
Level 1	ug/mL	6.250	2.263	1.988	2.336	1.599	1.606	1.770	1.800	2.288
Level 2	ug/mL	12.700	4.525	3.975	4.673	3.198	3.213	3.540	3.600	4.575
Level 3	ug/mL	25.400	9.050	7.950	9.345	6.395	6.425	7.080	7.200	9.150
Level 4	ug/mL	40.640	18.100	15.900	18.690	12.790	12.850	14.160	14.400	18.300
Level 5	ug/mL	50.800	36.200	31.800	37.380	25.580	25.700	28.320	28.800	36.600
Vol Inj	uL	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0
Mass 1	ug	0.0625	0.02263	0.01988	0.02336	0.01599	0.01606	0.0177	0.018	0.02288
Mass 2	ug	0.127	0.04525	0.03975	0.04673	0.03198	0.03213	0.0354	0.036	0.04575
Mass 3	ug	0.254	0.0905	0.0795	0.09345	0.06395	0.06425	0.0708	0.072	0.0915
Mass 4	ug	0.4064	0.181	0.159	0.1869	0.1279	0.1285	0.1416	0.144	0.183
Mass 5	ug	0.508	0.362	0.318	0.3738	0.2558	0.257	0.2832	0.288	0.366
Area 1		65898	110739	329081	64214	105092	33312	83139	58578	34252
Area 2		156724	226736	614912	125404	200310	63836	158551	117716	77982
Area 3		348308	471655	1276567	257183	414454	128644	326192	238461	176274
Area 4		543849	924707	2435154	493183	789587	248162	624582	460615	356826
Area 5		707350	1830689	4841233	996485	1587938	506560	1256235	945671	757215
CF (1)		1054368	4893460	16553370	2748887	6572357.7	2074221.7	4697118.6	3254333.3	1497028
CF (2)		1234047.2	5010740.3	15469484	2683586.6	6263602.3	1986803.6	4478841.8	3269888.9	1704524.6
CF (3)		1371291.3	5211657.5	16057447	2752092	6480907	2002241.2	4607231.6	3311958.3	1926491.8
CF (4)		1338211.1	5108878.5	15315434	2638753.3	6173471.5	1931221.8	4410889.8	3198715.3	1949868.9
CF (5)		1392421.3	5057151.9	15224003	2665824	6207732.6	1971050.6	4435858.1	3283579.9	2068893.4
Avg CF		1.278E+06	5.056E+06	1.572E+07	2.698E+06	6.340E+06	1.993E+06	4.526E+06	3.264E+06	1.829E+06
(CF (1)-CF (AVG))^2		5.004E+10	2.654E+10	6.879E+11	2.607E+09	5.417E+10	6.579E+09	2.929E+10	8.764E+07	1.104E+11
(CF (2)-CF (AVG))^2		1.938E+09	2.083E+09	6.475E+10	2.028E+08	5.778E+09	3.974E+07	2.223E+09	3.836E+07	1.558E+10
(CF (3)-CF (AVG))^2		8.691E+09	2.411E+10	1.112E+11	2.945E+09	1.996E+10	8.342E+07	6.601E+09	2.329E+09	9.434E+09
(CF (4)-CF (AVG))^2		3.617E+09	2.756E+09	1.669E+11	3.490E+09	2.760E+10	3.830E+09	1.325E+10	4.222E+09	1.452E+10
(CF (5)-CF (AVG))^2		1.308E+10	5.995E+05	2.499E+11	1.024E+09	1.739E+10	4.865E+08	8.123E+09	3.954E+08	5.738E+10
SUM CF-CF(AVG)		7.736E+10	5.549E+10	1.281E+12	1.027E+10	1.249E+11	1.102E+10	5.948E+10	7.073E+09	2.074E+11
SD		1.391E+05	1.178E+05	5.658E+05	5.067E+04	1.767E+05	5.249E+04	1.219E+05	4.205E+04	2.277E+05
RSD		**10.9**	**2.3**	**3.6**	**1.9**	**2.8**	**2.6**	**2.7**	**1.3**	**12.4**

TABLE IV-1A

RSD CALCULATIONS FOR THE LONG GRADIENT
PAGE 3 OF 3

Dye	Units	SY3	BZ	AN	SR1	DR9	SY2	SG3	SR24
Level 1	ug/mL	1.888	1.520	2.596	2.899	1.651	1.995	1.838	0.710
Level 2	ug/mL	3.775	3.040	5.193	5.798	3.303	3.990	3.675	1.136
Level 3	ug/mL	7.550	6.080	10.385	11.595	6.605	7.980	7.350	1.420
Level 4	ug/mL	15.100	12.160	20.770	23.190	13.210	15.960	14.700	2.840
Level 5	ug/mL	30.200	24.320	41.540	46.380	26.420	31.920	29.400	5.680
Vol Inj	uL	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0
Mass 1	ug	0.01888	0.0152	0.02596	0.02899	0.01651	0.01995	0.01838	0.0071
Mass 2	ug	0.03775	0.0304	0.05193	0.05798	0.03303	0.0399	0.03675	0.01136
Mass 3	ug	0.0755	0.0608	0.10385	0.11595	0.06605	0.0798	0.0735	0.0142
Mass 4	ug	0.151	0.1216	0.2077	0.2319	0.1321	0.1596	0.147	0.0284
Mass 5	ug	0.302	0.2432	0.4154	0.4638	0.2642	0.3192	0.294	0.0568
Area 1		132272	50312	2081462	111250	39277	128853	11307	6932
Area 2		253175	96428	3872277	212507	69625	258753	21901	15439
Area 3		499732	194499	7520808	432962	143151	518465	58249	18842
Area 4		1011441	394239	14996853	918529	293340	1055813	133853	75065
Area 5		2140858	829512	30532340	2245890	587088	2138953	216411	164040
CF (1)		7005932.2	3310000	80179584	3837530.2	2378982.4	6458797	615179.54	976338.03
CF (2)		6706622.5	3171973.7	74567244	3665177.6	2107932.2	6485037.6	595945.58	1359066.9
CF (3)		6618966.9	3198996.7	72419913	3734040.5	2167312.6	6497055.1	792503.4	1326901.4
CF (4)		6698284.8	3242097	72204396	3960884	2220590.5	6615369.7	910564.63	2643133.8
CF (5)		7088933.8	3410822.4	73501059	4842367.4	2222134.7	6700980.6	736091.84	2888028.2
Avg CF		6.824E+06	3.267E+06	7.457E+07	4.008E+06	2.219E+06	6.551E+06	7.301E+05	1.839E+06
(CF (1)-CF (AVG))^2		3.319E+10	1.868E+09	3.142E+13	2.906E+10	2.547E+10	8.584E+09	1.320E+10	7.437E+11
(CF (2)-CF (AVG))^2		1.372E+10	8.988E+09	5.177E+07	1.175E+11	1.242E+10	4.410E+09	1.799E+10	2.300E+11
(CF (3)-CF (AVG))^2		4.194E+10	4.594E+09	4.642E+12	7.505E+10	2.712E+09	2.959E+09	3.900E+09	2.619E+11
(CF (4)-CF (AVG))^2		1.574E+10	6.091E+08	5.617E+12	2.220E+09	1.440E+06	4.086E+09	3.258E+10	6.471E+11
(CF (5)-CF (AVG))^2		7.032E+10	2.075E+10	1.152E+12	6.962E+11	7.531E+06	2.236E+10	3.642E+07	1.101E+12
SUM CF-CF(AVG)		1.749E+11	3.681E+10	4.283E+13	9.200E+11	4.061E+10	4.240E+10	6.770E+10	2.984E+12
SD		2.091E+05	9.593E+04	3.272E+06	4.796E+05	1.008E+05	1.030E+05	1.301E+05	8.637E+05
RSD		**3.1**	**2.9**	**4.4**	**12.0**	**4.5**	**1.6**	**17.8**	**47.0**

TABLE IV-2

INITIAL CALIBRATION SUMMARY FOR THE SHORT GRADIENT

DYE	RSD	R^2	R
ACID BLUE 45	4.9	0.999667	0.999833
ACID YELLOW 23	3.8	0.999927	0.999964
ACID YELLOW 73	3.2	0.999931	0.999965
ACID ORANGE 10	2.5	0.999941	0.999970
ACID BLUE 1	3.4	0.999958	0.999979
BASIC YELLOW 2	3.3	0.999945	0.999973
DISPERSE VIOLET 1	2.0	0.999937	0.999969
SOLVENT YELLOW 33	2.4	0.999844	0.999922
DISPERSE BLUE 14	9.1	0.999258	0.999629
ACID RED 64	43.6	0.999835	0.999918
ACID BLUE 9	2.3	0.999917	0.999958
BASIC VIOLET 10	0.9	0.999980	0.999990
2-AMINOANTHRAQUINONE	1.7	0.999972	0.999986
SOLVENT ORANGE 3	1.9	0.999955	0.999978
1-AMINOANTHRAQUINONE	1.5	0.999962	0.999981
SOLVENT YELLOW 14	2.1	0.999948	0.999974
SOLVENT ORANGE 7	2.0	0.999725	0.999863
ACID YELLOW 3	9.9	0.999651	0.999826
SOLVENT YELLOW 3	0.8	0.999920	0.999960
BENZANTHRONE	1.0	0.999896	0.999948
ANTHRACENE	6.5	0.996361	0.998179
SOLVENT RED 1	5.6	0.996954	0.998476
DISPERSE RED 9	1.7	0.999865	0.999933
SOLVENT YELLOW 2	1.0	0.999881	0.999940
SOLVENT GREEN 3	10.1	0.988542	0.994255
SOLVENT RED 24	26.7	0.998030	0.999015

AVERAGE %RSD 5.9

TABLE IV-2A

RSD CALCULATIONS FOR THE SHORT GRADIENT

Dye	Units	AB45	AY23	AY73	AO10	AB1	BY2
Level 1	ug/mL	4.075	1.620	2.623	3.938	3.189	1.683
Level 2	ug/mL	8.150	3.240	5.245	7.875	6.378	3.365
Level 3	ug/mL	16.300	6.480	10.490	15.750	12.755	6.730
Level 4	ug/mL	32.600	12.690	20.980	31.500	25.510	13.460
Level 5	ug/mL	65.200	25.920	41.960	63.000	51.020	26.920
Vol Inj	uL	10.0	10.0	10.0	10.0	10.0	10.0
Mass 1	ug	0.04075	0.0162	0.02623	0.03938	0.03189	0.01683
Mass 2	ug	0.0815	0.0324	0.05245	0.07875	0.06378	0.03365
Mass 3	ug	0.163	0.0648	0.1049	0.1575	0.12755	0.0673
Mass 4	ug	0.326	0.1269	0.2098	0.315	0.2551	0.1346
Mass 5	ug	0.652	0.2592	0.4196	0.63	0.5102	0.2692
Area 1		20353	34907	240207	44541	159419	6774
Area 2		44068	77312	490525	95159	315802	14798
Area 3		93177	148554	916212	181086	596326	28494
Area 4		180806	297225	1838949	365235	1193297	57860
Area 5		355914	597797	3673558	731506	2383748	116057
CF (1)		499460.1227	2154753.086	9157720.168	1131056.374	4999027.908	402495.5437
CF (2)		540711.6564	2386172.84	9352240.229	1208368.254	4951426.78	439762.2585
CF (3)		571638.0368	2292500	8734146.806	1149752.381	4675233.242	423387.8158
CF (4)		554619.6319	2342198.582	8765247.855	1159476.19	4677761.662	429866.2704
CF (5)		545880.3681	2306315.586	8754904.671	1161120.635	4672183.457	431118.1278
Avg CF		542462.0	2296388.0	8952851.9	1161954.8	4795126.6	425326.0
$(CF (1)-CF (AVG))^2$		1.849E+09	2.006E+10	4.197E+10	9.547E+08	4.158E+10	5.212E+08
$(CF (2)-CF (AVG))^2$		3.064E+06	8.061E+09	1.595E+11	2.154E+09	2.443E+10	2.084E+08
$(CF (3)-CF (AVG))^2$		8.512E+08	1.512E+07	4.783E+10	1.489E+08	1.437E+10	3.757E+06
$(CF (4)-CF (AVG))^2$		1.478E+08	2.099E+09	3.520E+10	6.143E+06	1.377E+10	2.061E+07
$(CF (5)-CF (AVG))^2$		1.169E+07	9.856E+07	3.918E+10	6.958E+05	1.512E+10	3.355E+07
SUM CF-CF(AVG)		2.863E+09	3.033E+10	3.237E+11	3.265E+09	1.093E+11	7.876E+08
SD		2.675E+04	8.708E+04	2.845E+05	2.857E+04	1.653E+05	1.403E+04
RSD		**4.9**	**3.8**	**3.2**	**2.5**	**3.4**	**3.3**

TABLE IV-2A

RSD CALCULATIONS FOR THE SHORT GRADIENT

Dye	Units	DV1	SY33	DB14	AR64	AB9	BV10
Level 1	ug/mL	3.059	1.531	1.873	2.688	2.694	1.846
Level 2	ug/mL	6.118	3.063	3.745	5.375	5.388	3.693
Level 3	ug/mL	12.235	6.125	7.490	10.750	10.775	7.385
Level 4	ug/mL	24.470	12.250	14.980	21.500	21.550	14.770
Level 5	ug/mL	48.940	24.500	29.960	43.000	43.100	29.540
Vol Inj	uL	10.0	10.0	10.0	10.0	10.0	10.0
Mass 1	ug	0.03059	0.01531	0.01873	0.02688	0.02694	0.01846
Mass 2	ug	0.06118	0.03063	0.03745	0.05375	0.05388	0.03693
Mass 3	ug	0.12235	0.06125	0.0749	0.1075	0.10775	0.07385
Mass 4	ug	0.2447	0.1225	0.1498	0.215	0.2155	0.1477
Mass 5	ug	0.4894	0.245	0.2996	0.43	0.431	0.2954
Area 1		54369	59867	29917	9457	84798	181479
Area 2		114417	125526	68731	48700	175671	354721
Area 3		219259	236371	113873	142268	354069	717232
Area 4		443122	492432	224086	306100	722168	1431144
Area 5		892675	987078	448138	655279	1425390	2839805
CF (1)		1777345.538	3910320.052	1597277.096	351822.9167	3147661.47	9830931.744
CF (2)		1870169.99	4098139.079	1835273.698	906046.5116	3260412.027	9605226.103
CF (3)		1792063.752	3859118.367	1520333.778	1323423.256	3286023.202	9712010.833
CF (4)		1810878.627	4019853.061	1495901.202	1423720.93	3351127.61	9689532.837
CF (5)		1824019.207	4028889.796	1495787.717	1523904.651	3307169.374	9613422.478
Avg CF		1814895.4	3983264.1	1588914.7	1105783.7	3270478.7	9690224.8
$(CF (1)-CF (AVG))^2$		1.410E+09	5.321E+09	6.993E+07	5.685E+11	1.508E+10	1.980E+10
$(CF (2)-CF (AVG))^2$		3.055E+09	1.320E+10	6.069E+10	3.989E+10	1.013E+08	7.225E+09
$(CF (3)-CF (AVG))^2$		5.213E+08	1.541E+10	4.703E+09	4.737E+10	2.416E+08	4.746E+08
$(CF (4)-CF (AVG))^2$		1.613E+07	1.339E+09	8.652E+09	1.011E+11	6.504E+09	4.788E+05
$(CF (5)-CF (AVG))^2$		8.324E+07	2.082E+09	8.673E+09	1.748E+11	1.346E+09	5.899E+09
SUM CF-CF(AVG)		5.086E+09	3.735E+10	8.279E+10	9.316E+11	2.328E+10	3.340E+10
SD		3.566E+04	9.663E+04	1.439E+05	4.826E+05	7.628E+04	9.137E+04
RSD		**2.0**	**2.4**	**9.1**	**43.6**	**2.3**	**0.9**

TABLE IV-2A

RSD CALCULATIONS FOR THE SHORT GRADIENT

Dye	Units	2AQ	SO3	1AQ	SY14	SO7	AY3
Level 1	ug/mL	1.656	1.868	2.264	1.769	2.599	1.865
Level 2	ug/mL	3.313	3.735	4.528	3.538	5.198	3.730
Level 3	ug/mL	6.625	7.470	9.055	7.075	10.395	7.460
Level 4	ug/mL	13.250	14.940	18.110	14.150	20.790	14.920
Level 5	ug/mL	26.500	29.880	36.220	28.300	41.580	29.840
Vol Inj	uL	10.0	10.0	10.0	10.0	10.0	10.0
Mass 1	ug	0.01656	0.01868	0.02264	0.01769	0.02599	0.01865
Mass 2	ug	0.03313	0.03735	0.04528	0.03538	0.05198	0.0373
Mass 3	ug	0.06625	0.0747	0.09055	0.07075	0.10395	0.0746
Mass 4	ug	0.1325	0.1494	0.1811	0.1415	0.2079	0.1492
Mass 5	ug	0.265	0.2988	0.3622	0.283	0.4158	0.2984
Area 1		49116	81696	26143	71925	65421	8047
Area 2		94369	157094	51896	136429	125962	19235
Area 3		193120	315344	100584	274309	255823	38427
Area 4		391756	630089	204490	556024	531268	80919
Area 5		784621	1242751	409092	1100042	1036435	169039
CF (1)		2965942.029	4373447.537	1154726.148	4065856.416	2517160.446	431474.5308
CF (2)		2848445.518	4205997.323	1146113.074	3856105.144	2423278.184	515683.6461
CF (3)		2915018.868	4221472.557	1110811.706	3877159.011	2461019.721	515107.2386
CF (4)		2956649.057	4217463.186	1129155.163	3929498.233	2555401.635	542352.5469
CF (5)		2960833.962	4159139.893	1129464.384	3887074.205	2492628.668	566484.5845
Avg CF		2929377.9	4235504.1	1134054.1	3923138.6	2489897.7	514220.5
$(CF (1)-CF (AVG))^2$		1.337E+09	1.903E+10	4.273E+08	2.037E+10	7.433E+08	6.847E+09
$(CF (2)-CF (AVG))^2$		6.550E+09	8.706E+08	1.454E+08	4.493E+09	4.438E+09	2.141E+06
$(CF (3)-CF (AVG))^2$		2.062E+08	1.969E+08	5.402E+08	2.114E+09	8.339E+08	7.863E+05
$(CF (4)-CF (AVG))^2$		7.437E+08	3.255E+08	2.400E+07	4.044E+07	4.291E+09	7.914E+08
$(CF (5)-CF (AVG))^2$		9.895E+08	5.831E+09	2.107E+07	1.301E+09	7.458E+06	2.732E+09
SUM CF-CF(AVG)		9.826E+09	2.625E+10	1.158E+09	2.832E+10	1.031E+10	1.037E+10
SD		4.956E+04	8.101E+04	1.701E+04	8.414E+04	5.078E+04	5.092E+04
RSD		**1.7**	**1.9**	**1.5**	**2.1**	**2.0**	**9.9**

TABLE IV-2A

RSD CALCULATIONS FOR THE SHORT GRADIENT

Dye	Units	SY3	BZ	AN	SR1	DR9	SY2
Level 1	ug/mL	4.286	2.431	2.889	2.115	1.663	1.846
Level 2	ug/mL	8.573	4.863	5.778	4.230	3.325	3.693
Level 3	ug/mL	17.145	9.725	11.555	8.460	6.650	7.385
Level 4	ug/mL	34.290	19.450	23.110	16.920	13.300	14.770
Level 5	ug/mL	68.580	38.900	46.220	33.840	26.600	29.540
Vol Inj	uL	10.0	10.0	10.0	10.0	10.0	10.0
Mass 1	ug	0.04286	0.02431	0.02889	0.02115	0.01663	0.01846
Mass 2	ug	0.08573	0.04863	0.05778	0.0423	0.03325	0.03693
Mass 3	ug	0.17145	0.09725	0.11555	0.0846	0.0665	0.07385
Mass 4	ug	0.3429	0.1945	0.2311	0.1692	0.133	0.1477
Mass 5	ug	0.6858	0.389	0.4622	0.3384	0.266	0.2954
Area 1		105190	40659	1668117	79076	30282	109307
Area 2		209982	81235	3251082	160077	59503	219452
Area 3		423110	163006	6500055	319538	116659	432025
Area 4		841355	326355	12582937	641285	231919	870153
Area 5		1713016	666137	22516633	1438477	472735	1774359
CF (1)		2454269.715	1672521.596	57740290.76	3738817.967	1820926.037	5921289.274
CF (2)		2449340.954	1670470.903	56266562.82	3784326.241	1789563.91	5942377.471
CF (3)		2467833.188	1676154.242	56253180.44	3777044.917	1754270.677	5850033.852
CF (4)		2453645.378	1677917.738	54448018.17	3790100.473	1743751.88	5891354.096
CF (5)		2497836.104	1712434.447	48716211.6	4250818.55[illegible]	1777199.248	6006631.686
Avg CF		2464585.1	1681899.8	54684852.8	3868221.6	1777142.4	5922337.3
$(CF (1)-CF (AVG))^2$		1.064E+08	8.795E+07	9.336E+12	1.675E+10	1.917E+09	1.098E+06
$(CF (2)-CF (AVG))^2$		2.324E+08	1.306E+08	2.502E+12	7.038E+09	1.543E+08	4.016E+08
$(CF (3)-CF (AVG))^2$		1.055E+07	3.301E+07	2.460E+12	8.313E+09	5.231E+08	5.228E+09
$(CF (4)-CF (AVG))^2$		1.197E+08	1.586E+07	5.609E+10	6.103E+09	1.115E+09	9.600E+08
$(CF (5)-CF (AVG))^2$		1.106E+09	9.324E+08	3.562E+13	1.464E+11	3.237E+03	7.106E+09
SUM CF-CF(AVG)		1.575E+09	1.200E+09	4.998E+13	1.846E+11	3.709E+09	1.370E+10
SD		1.984E+04	1.732E+04	3.535E+06	2.148E+05	3.045E+04	5.851E+04
RSD		**0.8**	**1.0**	**6.5**	**5.6**	**1.7**	**1.0**

TABLE IV-2A

RSD CALCULATIONS FOR THE SHORT GRADIENT
PAGE 5 OF 5

Dye	Units	SG3	SR24
Level 1	ug/mL	2.054	0.710
Level 2	ug/mL	4.108	0.852
Level 3	ug/mL	8.215	1.420
Level 4	ug/mL	16.430	2.840
Level 5	ug/mL	32.860	5.680
Vol Inj	uL	10.0	10.0
Mass 1	ug	0.02054	0.0071
Mass 2	ug	0.04108	0.00852
Mass 3	ug	0.08215	0.0142
Mass 4	ug	0.1643	0.0284
Mass 5	ug	0.3286	0.0568
Area 1		14304	7310
Area 2		27088	11099
Area 3		53793	21584
Area 4		104765	50598
Area 5		173087	118757
CF (1)		696397.2736	1029577.465
CF (2)		659396.2999	1302699.531
CF (3)		654814.364	1520000
CF (4)		637644.5526	1781619.718
CF (5)		526740.7182	2090792.254
Avg CF		634998.6	1544937.8
$(CF (1)-CF (AVG))^2$		3.770E+09	2.656E+11
$(CF (2)-CF (AVG))^2$		5.952E+08	5.868E+10
$(CF (3)-CF (AVG))^2$		3.927E+08	6.219E+08
$(CF (4)-CF (AVG))^2$		7.001E+06	5.602E+10
$(CF (5)-CF (AVG))^2$		1.172E+10	2.980E+11
SUM CF-CF(AVG)		1.648E+10	6.789E+11
SD		6.420E+04	4.120E+05
RSD		**10.1**	**26.7**

TABLE IV-3

HPLC RUN SEQUENCE FOR THE LONG GRADIENT
PAGE 1 OF 2

SAMPLE	DATE ANALYZED	TIME ANALYZED	TIME ELAPSED
A L1	7/24/00	15:16:32	0:00:00
A L2	7/24/00	16:28:33	1:12:01
A L3	7/24/00	17:50:31	2:33:59
A L4	7/24/00	18:52:30	3:35:58
A L5	7/24/00	20:04:32	4:48:00
B L1	7/24/00	21:16:32	6:00:00
B L2	7/24/00	22:28:34	7:12:02
B L3	7/24/00	23:40:38	8:24:06
B L4	7/25/00	0:25:37	9:36:05
B L5	7/25/00	2:04:38	10:48:06
C L1	7/25/00	3:16:36	12:00:04
C L2	7/25/00	4:28:40	13:12:08
C L3	7/25/00	5:40:40	14:24:08
C L4	7/25/00	6:52:39	15:36:07
C L5	7/25/00	8:04:39	16:48:07
D L1	7/25/00	9:16:46	18:00:14
D L2	7/25/00	10:28:46	19:12:14
D L3	7/25/00	11:40:47	20:24:15
D L4	7/25/00	12:52:44	21:36:12
D L5	7/25/00	11:16:46	20:00:14
DBG-IN_BLNK-0010	7/25/00		
WLM001LS	7/25/00		
WLM002LS	7/25/00		
WLM003LS	7/25/00		
WLM004LS	7/25/00		
WLM005LS	7/25/00		
WLM006LS	7/25/00		
WLM008LS	7/26/00		
WLM007M_BLNK	7/26/00		
WLM004HS	7/26/00		
WLM006HS	7/26/00		
DBG-IN_BLNK-0011	7/26/00	5:34:05	32:30:29
DBGSTD-A3-0031	7/26/00	6:46:03	33:42:22
WLM007HS	7/26/00		
WLM008HS	7/26/00		
WLM009HS	7/26/00		
WLM010HS	7/26/00		
WLM011HS	7/26/00		
WHM001LS	7/26/00		
WHM002LS	7/26/00		
WHM003LS	7/26/00		
WHM004LS	7/26/00		
WHM005LS	7/26/00		
DBG-IN_BLNK-0012	7/26/00	19:58:55	52:42:23
DBGSTD-B3-0032	7/26/00	21:10:54	53:04:22
WHM006LS	7/26/00		
WHM007LS	7/26/00		

TABLE IV-3

HPLC RUN SEQUENCE FOR THE LONG GRADIENT
PAGE 2 OF 2

SAMPLE	DATE ANALYZED	TIME ANALYZED	TIME ELAPSED
WHM008M_BLNK (LS)	7/27/00		
WHM001HS	7/27/00		
WHM002HS	7/27/00		
WHM003HS	7/27/00		
WHM004HS	7/27/00		
WHM005HS	7/27/00		
WHM006HS	7/27/00		
WHM007HS	7/27/00		
WHM008M_BLNK (HS)	7/27/00		
DBG-IN_BLNK-0013	7/27/00	11:36:24	62:28:04
DBGSTD-C3-0033	7/27/00	12:48:38	63:40:10
S001LS	7/27/00		
S002LS	7/27/00		
S003LS	7/27/00		
S004LS	7/27/00		
S005LS	7/27/00		
S006LS	7/27/00		
DBG-IN_BLNK-0016	7/28/00	20:54:16	101:37:44
DBGSTD-A3-0034	7/28/00	22:06:14	102:49:42
DBGSTD-B3-0035	7/28/00	23:18:13	104:01:41
DBGSTD-C3-0036	7/29/00	0:30:28	104:32:09
DBGSTD-D3-0037	7/29/00	2:54:51	106:46:32
DBG-IN_BLNK-0014	7/29/00		
S007LS	7/29/00		
S008M_BLNK	7/29/00		
S001HS	7/29/00		
S002HS	7/29/00		
S003HS	7/29/00		
S004HS	7/29/00		
S005HS	7/29/00		
S006HS	7/29/00		
S007HS	7/29/00		
DBG-IN_BLNK-0015	7/29/00	14:57:37	118:49:18
DBG-A3-0038	7/29/00	16:09:35	120:53:03
AR64 L3	7/31/00	13:52:25	166:35:53
AR64 L1	7/31/00	16:16:40	169:00:08
AR64 L4	7/31/00	17:28:38	170:07:06
AR64 L5	7/31/00	18:40:38	171:24:06
AR64 40.64	7/31/00	20:47:28	173:30:54

TABLE IV-3A

CCV LONG GRADIENT
PAGE 1 OF 2

CCV FILE NAME	PARAMETER	ICAL AREA	ICAL AMOUNT	CCV AREA	CCV AMOUNT	PCT_DRIFT	PCT_DIFF	RT	RT WINDOW	
DBGSTD-A3-0031	ACID BLUE 45	163913	9.300	110369	6.364	-32.7	-31.6	6.439	0.000	17.486
DBGSTD-A3-0031	ACID YELLOW 23	276127	10.050	294788	10.738	6.8	6.8	14.513	8.913	19.289
DBGSTD-A3-0031	ACID YELLOW 73	1184169	9.050	1272389	9.662	7.4	6.8	17.353	12.976	21.074
DBGSTD-A3-0031	ACID ORANGE 10	293902	10.050	315843	10.753	7.5	7.0	18.752	14.849	22.041
DBGSTD-A3-0031	ACID BLUE 1	766740	10.650	828799	11.405	8.1	7.1	23.733	21.150	25.848
DBGSTD-A3-0031	BASIC YELLOW 2	52347	9.250	53610	9.714	2.4	5.0	35.367	34.194	36.224
DBGSTD-A3-0031	DISPERSE VIOLET 1	283267	6.505	298401	6.769	5.3	4.1	22.770	22.603	22.927
DBGSTD-A3-0031	SOLVENT YELLOW 33	290972	6.990	308204	7.287	5.9	4.2	28.259	26.970	29.294
DBGSTD-A3-0031	DISPERSE BLUE 14	221890	7.475	229882	7.411	3.6	-0.9	34.023	32.878	34.922
DBGSTD-B3-0032	ACID BLUE 9 (*2)	673423	9.050	689843	9.520	2.4	5.2	21.657	19.202	24.392
DBGSTD-B3-0032	BASIC VIOLET 10	1276567	7.950	1291362	8.302	1.2	4.4	36.018	35.205	36.825
DBGSTD-B3-0032	2-AMINOANTHRAQUINONE	257183	9.345	264998	9.875	3.0	5.7	23.758	23.232	24.322
DBGSTD-B3-0032	SOLVENT ORANGE 3	414454	6.395	410466	6.526	-1.0	2.0	24.783	24.636	24.956
DBGSTD-B3-0032	1-AMINOANTHRAQUINONE	128645	6.425	137631	6.966	7.0	8.4	28.853	27.992	29.788
DBGSTD-B3-0032	SOLVENT YELLOW 14	326193	7.080	327798	7.310	0.5	3.2	44.360	43.062	45.752
DBGSTD-B3-0032	SOLVENT ORANGE 7(*3)	294845	7.200	295874	7.368	0.3	2.3	50.894	49.701	52.185
DBGSTD-C3-0033	ACID YELLOW 3(*2)	204012	9.150	218384	9.881	7.0	8.0	13.372	10.265	17.041
DBGSTD-C3-0033	SOLVENT YELLOW 3	499733	7.550	532685	7.832	6.6	3.7	35.998	35.067	37.075
DBGSTD-C3-0033	SOLVENT RED 1	432963	11.595	455839	11.302	5.3	-2.5	44.811	43.780	45.988
DBGSTD-C3-0033	ANTHRACENE	7520809	10.385	7947424	10.830	5.7	4.3	40.641	39.613	41.831
DBGSTD-C3-0033	BENZANTHRONE	194500	6.080	205063	6.270	5.4	3.1	36.724	35.871	37.693
DBGSTD-A3-0034	ACID BLUE 45	163913	9.300	122622	7.042	-25.2	-24.3	4.695	0.000	17.486
DBGSTD-A3-0034	ACID YELLOW 23	276127	10.050	177757	6.588	-35.6	-34.4	12.900	8.913	19.289
DBGSTD-A3-0034	ACID YELLOW 73	1184169	9.050	1289675	9.791	8.9	8.2	16.090	12.976	21.074
DBGSTD-A3-0034	ACID ORANGE 10	293902	10.050	321251	10.932	9.3	8.8	17.615	14.849	22.041
DBGSTD-A3-0034	ACID BLUE 1	766740	10.650	842498	11.592	9.9	8.8	22.959	21.150	25.848
DBGSTD-A3-0034	BASIC YELLOW 2	52347	9.250	53706	9.731	2.6	5.2	34.974	34.194	36.224
DBGSTD-A3-0034	DISPERSE VIOLET 1	283267	6.505	354528	8.013	25.2	23.2	22.793	22.603	22.927
DBGSTD-A3-0034	SOLVENT YELLOW 33	290972	6.990	305546	7.227	5.0	3.4	27.836	26.970	29.294
DBGSTD-A3-0034	DISPERSE BLUE 14	221890	7.475	233777	7.522	5.4	0.6	33.662	32.878	34.922
DBGSTD-B3-0035	ACID BLUE 9 (*2)	673423	9.050	636631	9.071	-5.5	0.2	21.010	19.202	24.392
DBGSTD-B3-0035	BASIC VIOLET 10	1276567	7.950	1218124	7.818	-4.6	-1.7	35.744	35.205	36.825
DBGSTD-B3-0035	2-AMINOANTHRAQUINONE	257183	9.345	246512	9.179	-4.1	-1.8	23.606	23.232	24.322
DBGSTD-B3-0035	SOLVENT ORANGE 3	414454	6.395	389741	6.190	-6.0	-3.2	24.751	24.636	24.956
DBGSTD-B3-0035	1-AMINOANTHRAQUINONE	128645	6.425	125880	6.370	-2.1	-0.9	28.611	27.992	29.788
DBGSTD-B3-0035	SOLVENT YELLOW 14	326193	7.080	309267	6.891	-5.2	-2.7	43.984	43.062	45.752
DBGSTD-B3-0035	SOLVENT ORANGE 7(*3)	294845	7.200	279848	6.945	-5.1	-3.5	50.555	49.701	52.185

TABLE IV-3A

CCV LONG GRADIENT
PAGE 2 OF 2

CCV FILE NAME	PARAMETER	ICAL AREA	ICAL AMOUNT	CCV AREA	CCV AMOUNT	PCT_DRIFT	PCT_DIFF	RT	RT WINDOW	
DBGSTD-C3-0036	ACID YELLOW 3(*2)	204012	9.150	220792	9.663	8.2	5.6	12.690	10.265	17.041
DBGSTD-C3-0036	SOLVENT YELLOW 3	499733	7.550	536359	7.884	7.3	4.4	35.779	35.067	37.075
DBGSTD-C3-0036	SOLVENT RED 1	432963	11.595	456481	11.315	5.4	-2.4	44.558	43.780	45.988
DBGSTD-C3-0036	ANTHRACENE	7520809	10.385	7201381	9.810	-4.2	-5.5	40.399	39.613	41.831
DBGSTD-C3-0036	BENZANTHRONE	194500	6.080	206070	6.299	5.9	3.6	36.511	35.871	37.693
DBGSTD-D3-0037	DISPERSE RED 9	143151	6.605	145312	6.596	1.5	-0.1	36.142	34.980	38.022
DBGSTD-D3-0037	SOLVENT YELLOW 2	518466	7.980	521365	7.926	0.6	-0.7	39.910	38.845	41.633
DBGSTD-D3-0037	SOLVENT GREEN 3	58250	7.350	54461	6.945	-6.5	-5.5	57.448	56.449	59.063
DBGSTD-D3-0037	SOLVENT RED 24	18843	1.420	18140	1.187	-3.7	-16.4	57.994	57.219	59.247
DBGSTD-A3-0038	ACID BLUE 45	163913	9.300	122734	7.048	-25.1	-24.2	4.272	0.000	17.486
DBGSTD-A3-0038	ACID YELLOW 23	276127	10.050	160483	5.976	-41.9	-40.5	12.619	8.913	19.289
DBGSTD-A3-0038	ACID YELLOW 73	1184169	9.050	1297338	9.849	9.6	8.8	15.861	12.976	21.074
DBGSTD-A3-0038	ACID ORANGE 10	293902	10.050	323021	10.990	9.9	9.4	17.404	14.849	22.041
DBGSTD-A3-0038	ACID BLUE 1	766740	10.650	851576	11.716	11.1	10.0	22.801	21.150	25.848
DBGSTD-A3-0038	BASIC YELLOW 2	52347	9.250	53825	9.751	2.8	5.4	34.887	34.194	36.224
DBGSTD-A3-0038	DISPERSE VIOLET 1 (*1)	283267	6.505	361395	8.165	27.6	25.5	22.808	22.603	22.927
DBGSTD-A3-0038	SOLVENT YELLOW 33	290972	6.990	309897	7.325	6.5	4.8	27.809	26.970	29.294
DBGSTD-A3-0038	DISPERSE BLUE 14	221890	7.475	232618	7.489	4.8	0.2	33.591	32.878	34.922

(*1) ACID BLUE 1 INTERFERENCE
(*2) AREA BASED ON 3 PEAKS
(*3) AREA BASED ON 2 PEAKS

TABLE IV-3B

LONG GRADIENT PERCENT DIFFERENCES OVER TIME

CCV FILE NAME	PARAMETER	CCV A	CCV B	CCV C	CCV A	CCV B	CCV C	CCV D	CCV A
		7/26/00	7/26/00	7/27/00	7/28/00	7/28/00	7/29/00	7/29/00	7/29/00
	TIME FROM ICAL:	33 hrs	53 hrs	63 hrs	102 hrs	104 hrs	104.5 hrs	106 hrs	120 hrs
DBGSTD-A3-0031	ACID BLUE 45	31.6			24.3				24.2
DBGSTD-A3-0031	ACID YELLOW 23	6.8			34.4				40.5
DBGSTD-A3-0031	ACID YELLOW 73	6.8			8.2				8.8
DBGSTD-A3-0031	ACID ORANGE 10	7.0			8.8				9.4
DBGSTD-A3-0031	ACID BLUE 1	7.1			8.8				10.0
DBGSTD-A3-0031	BASIC YELLOW 2	5.0			5.2				5.4
DBGSTD-A3-0031	DISPERSE VIOLET 1 (*1)	4.1			23.2				25.5
DBGSTD-A3-0031	SOLVENT YELLOW 33	4.2			3.4				4.8
DBGSTD-A3-0031	DISPERSE BLUE 14	0.9			0.6				0.2
DBGSTD-B3-0032	ACID BLUE 9 (*2)		5.2			0.2			
DBGSTD-B3-0032	BASIC VIOLET 10		4.4			1.7			
DBGSTD-B3-0032	2-AMINOANTHRAQUINONE		5.7			1.8			
DBGSTD-B3-0032	SOLVENT ORANGE 3		2.0			3.2			
DBGSTD-B3-0032	1-AMINOANTHRAQUINONE		8.4			0.9			
DBGSTD-B3-0032	SOLVENT YELLOW 14		3.2			2.7			
DBGSTD-B3-0032	SOLVENT ORANGE 7(*3)		2.3			3.5			
DBGSTD-C3-0033	ACID YELLOW 3(*2)			8.0			5.6		
DBGSTD-C3-0033	SOLVENT YELLOW 3			3.7			4.4		
DBGSTD-C3-0033	SOLVENT RED 1			2.5			2.4		
DBGSTD-C3-0033	ANTHRACENE			4.3			5.5		
DBGSTD-C3-0033	BENZANTHRONE			3.1			3.6		
DBGSTD-D3-0037	DISPERSE RED 9							0.1	
DBGSTD-D3-0037	SOLVENT YELLOW 2							0.7	
DBGSTD-D3-0037	SOLVENT GREEN 3							5.5	
DBGSTD-D3-0037	SOLVENT RED 24							16.4	
Average Pecent Difference		**8.2**	**4.5**	**4.3**	**13.0**	**2.0**	**4.3**	**5.7**	**14.3**

(*1) ACID BLUE 1 INTERFERENCE
(*2) AREA BASED ON 3 PEAKS
(*3) AREA BASED ON 2 PEAKS

TABLE IV-4

HPLC RUN SEQUENCE FOR THE SHORT GRADIENT
PAGE 1 OF 3

SAMPLE	DATE ANALYZED	TIME ANALYZED	TIME ELAPSED
STD A1	7/2/00	14:17:14	0:00:00
STD A2	7/2/00	15:03:08	0:45:54
STD A3	7/2/00	15:49:06	1:31:52
STD A4	7/2/00	16:34:59	2:17:45
STD A5	7/2/00	17:20:56	3:03:42
INSTRUMENT BLANK	7/2/00	18:06:53	3:49:39
STD B1	7/2/00	18:52:50	4:35:36
STD B2	7/2/00	19:38:50	5:21:36
STD B3	7/2/00	20:24:46	6:07:32
STD B4	7/2/00	21:10:44	6:53:30
STD B5	7/2/00	21:56:37	7:39:23
INSTRUMENT BLANK	7/2/00	22:42:34	8:25:20
STD C1	7/2/00	23:28:31	9:11:17
STD C2	7/3/00	0:14:25	9:57:11
STD C3	7/3/00	1:00:25	10:43:11
STD C4	7/3/00	1:46:22	11:29:08
STD C5	7/3/00	2:32:15	12:15:01
INSTRUMENT BLANK	7/3/00	3:18:13	13:00:59
STD D1	7/3/00	4:04:12	13:46:58
STD D2	7/3/00	4:50:05	14:32:51
STD D3	7/3/00	5:36:05	15:18:51
STD D4	7/3/00	6:22:03	16:04:49
STD D5	7/3/00	7:07:59	16:50:45
INSTRUMENT BLANK	7/3/00	7:53:56	17:36:42
DBGSTD-A3-0001	7/3/00	15:13:06	24:55:52
DBGSTD-B3-0002	7/3/00	15:59:05	25:41:51
DBGSTD-C3-0003	7/3/00	16:45:02	26:27:48
DBGSTD-D3-0004	7/3/00		
DBG-IN_BLNK-0001	7/3/00		
WLM004HS	7/3/00		
WLM006HS	7/3/00		
WLM007HS	7/3/00		
WLM008HS	7/3/00		
WLM009HS	7/3/00		
WLM010HS	7/4/00		
DBGSTD-A3-0005	7/4/00	10:08:44	43:51:30
DBGSTD-B3-0006	7/4/00	10:54:38	44:37:24
DBGSTD-C3-0007	7/4/00	11:40:32	45:23:18
DBGSTD-D3-0008	7/4/00	12:26:28	46:09:14
DBG-IN_BLNK-0002	7/4/00		
WLM011HS	7/4/00		
WLM001LS	7/4/00		
WLM002LS	7/4/00		
WLM003LS	7/4/00		
WLM004LS	7/4/00		
WLM005LS	7/4/00		
WLM006LS	7/4/00		

TABLE IV-4

HPLC RUN SEQUENCE FOR THE SHORT GRADIENT
PAGE 2 OF 3

SAMPLE	DATE ANALYZED	TIME ANALYZED	TIME ELAPSED
WHM001HS	7/4/00		
WHM002HS	7/4/00		
WHM003HS	7/4/00		
WHM004HS	7/4/00		
DBG-IN_BLNK-0003	7/4/00		
DBGSTD-A3-0009	7/4/00	23:09:56	57:42:46
WHM005HS	7/4/00		
WHM006HS	7/5/00		
WHM007HS	7/5/00		
WHM008M_BLNK	7/5/00		
DBG-IN_BLNK-0004	7/5/00		
DBGSTD-B3-0010	7/5/00	3:46:25	61:29:11
DBGSTD-A3-0011	7/5/00	8:47:21	66:30:07
DBGSTD-B3-0012	7/5/00	9:33:15	67:30:07
DBGSTD-C3-0013	7/5/00	10:19:13	68:01:59
DBGSTD-D3-0014	7/5/00	11:05:06	68:47:52
DBG-IN_BLNK-0005	7/5/00		
S001LS	7/5/00		
S002LS	7/5/00		
S003LS	7/5/00		
S004LS	7/5/00		
S005LS	7/5/00		
S006LS	7/5/00		
S007LS	7/5/00		
SOIL BLANK	7/5/00		
S001HS	7/5/00		
S002HS	7/5/00		
DBG-IN_BLNK-0006	7/5/00		
DBG-A3-0015	7/5/00	21:12:37	78:55:23
S003HS	7/5/00		
S004HS	7/5/00		
S005HS	7/5/00		
S006HS	7/6/00		
S007HS	7/6/00		
DBG-IN_BLNK-0007	7/6/00		
DBG-B3-0016	7/6/00	2:34:44	84:17:30
DBG-E2-0017	7/6/00	12:23:48	94:06:34
DBG-E1-0018	7/6/00	13:09:43	94:52:29
DBG-E3-0019	7/6/00	13:55:43	95:38:29
DBG-E4-0020	7/6/00	14:41:58	96:24:44
DBG-E5-0021	7/6/00	15:27:58	97:10:44
DBG-E6-0022	7/6/00		
DBGSTD-A3-0023	7/11/00	10:51:20	212:34:06
DBGSTD-B3-0024	7/11/00	11:37:16	213:20:02
DBGSTD-C3-0025	7/11/00	12:23:11	214:05:57
DBGSTD-D3-0026	7/11/00		
DBGSTD-D3-0027	7/11/00	15:20:15	217:03:01

TABLE IV-4

HPLC RUN SEQUENCE FOR THE SHORT GRADIENT

SAMPLE	DATE ANALYZED	TIME ANALYZED	TIME ELAPSED
DBGSTD-E3-0028	7/11/00		
DBG-IN_BLNK-008	7/11/00		
WLM008LS	7/11/00		
WLM007M_BLNK	7/11/00		
WHM001LS	7/11/00		
WHM002LS	7/11/00		
WHM003LS	7/11/00		
WHM004LS	7/11/00		
WHM005LS	7/11/00		
WHM006LS	7/11/00		
WHM007LS	7/11/00		
WHM008M_BLNK	7/12/00		
DBG-IN_BLNK-009	7/12/00		
DBGSTD-A3-0029	7/12/00	2:04:22	227:47:08

TABLE IV-4A

CCV SHORT GRADIENT

CCV FILE NAME	PARAMETER	ICAL AREA	ICAL AMOUNT	CCV AREA	CCV AMOUNT	PCT_DRIFT	PCT_DIFF	RT	RT WINDOW	
DBGSTD-A3-0001	ACID YELLOW 23	148554	6.480	150090	6.529	1.0	0.8	7.525	3.422	10.110
DBGSTD-A3-0001	ACID YELLOW 73	916212	10.490	946075	10.681	3.3	1.8	9.159	6.372	10.926
DBGSTD-A3-0001	ACID ORANGE 10	181087	15.750	188479	16.221	4.1	3.0	9.946	7.433	11.551
DBGSTD-A3-0001	ACID BLUE 1	596326	12.755	621457	13.134	4.2	3.0	12.709	10.893	13.881
DBGSTD-A3-0001	BASIC YELLOW 2	28494	6.730	29790	6.957	4.5	3.4	19.452	18.376	20.158
DBGSTD-A3-0001	DISPERSE VIOLET 1 (*1)	219260	12.235	273486	15.056	24.7	23.1	12.690	12.440	12.880
DBGSTD-A3-0001	SOLVENT YELLOW 33	236372	6.125	245923	6.167	4.0	0.7	15.498	14.518	16.164
DBGSTD-A3-0001	DISPERSE BLUE 14	113874	7.490	116379	7.494	2.2	0.1	18.857	17.914	19.484
DBGSTD-B3-0002	ACID BLUE 9 (*3)	499830	10.775	511269	11.024	2.3	2.3	11.576	9.947	12.647
DBGSTD-B3-0002	BASIC VIOLET 10	717232	7.385	727427	7.518	1.4	1.8	19.691	18.883	20.229
DBGSTD-B3-0002	2-AMINOANTHRAQUINONE	193120	6.625	196430	6.693	1.7	1.0	13.246	12.716	13.620
DBGSTD-B3-0002	SOLVENT ORANGE 3	315344	7.470	320787	7.613	1.7	1.9	13.811	13.609	13.957
DBGSTD-B3-0002	1-AMINOANTHRAQUINONE	100585	9.055	102912	9.116	2.3	0.7	16.108	15.384	16.594
DBGSTD-B3-0002	SOLVENT YELLOW 14	274309	7.075	277157	7.092	1.0	0.2	24.403	23.521	24.975
DBGSTD-B3-0002	SOLVENT ORANGE 7 (*4)	277322	10.395	282626	10.394	1.9	0.0	27.804	27.029	28.299
DBGSTD-C3-0003	ACID YELLOW 3 (*3)	82621	7.460	83385	7.310	0.9	-2.0	7.322	3.913	9.739
DBGSTD-C3-0003	SOLVENT YELLOW 3	423110	17.145	426426	17.289	0.8	0.8	19.962	19.074	20.556
DBGSTD-C3-0003	SOLVENT RED 1	319539	8.460	320803	8.277	0.4	-2.2	24.660	23.675	25.321
DBGSTD-C3-0003	ANTHRACENE	6500055	11.555	6552745	12.181	0.8	5.4	22.537	21.703	23.085
DBGSTD-C3-0003	BENZANTHRONE	163006	9.725	164324	9.759	0.8	0.3	20.438	19.550	21.030
DBGSTD-D3-0004	DISPERSE RED 9	116659	6.650	117898	6.680	1.1	0.5	20.367	19.191	21.161
DBGSTD-D3-0004	SOLVENT YELLOW 2	432026	7.385	438327	7.409	1.5	0.3	22.244	21.221	22.929
DBGSTD-D3-0004	SOLVENT GREEN 3	53794	8.215	55709	9.074	3.6	10.5	31.194	30.327	31.765
DBGSTD-A3-0005	ACID YELLOW 23	148554	6.480	153625	6.683	3.4	3.1	7.307	3.422	10.110
DBGSTD-A3-0005	ACID YELLOW 73	916212	10.490	966238	10.913	5.5	4.0	9.012	6.372	10.926
DBGSTD-A3-0005	ACID ORANGE 10	181087	15.750	192016	16.526	6.0	4.9	9.819	7.433	11.551
DBGSTD-A3-0005	ACID BLUE 1	596326	12.755	631519	13.351	5.9	4.7	12.620	10.893	13.881
DBGSTD-A3-0005	BASIC YELLOW 2	28494	6.730	30465	7.114	6.9	5.7	19.405	18.376	20.158
DBGSTD-A3-0005	DISPERSE VIOLET 1 (*1)	219260	12.235	277384	15.269	26.5	24.8	12.695	12.440	12.880
DBGSTD-A3-0005	SOLVENT YELLOW 33	236372	6.125	250754	6.287	6.1	2.6	15.458	14.518	16.164
DBGSTD-A3-0005	DISPERSE BLUE 14	113874	7.490	115111	7.408	1.1	-1.1	18.821	17.914	19.484

TABLE IV-4A

CCV SHORT GRADIENT

CCV FILE NAME	PARAMETER	ICAL AREA	ICAL AMOUNT	CCV AREA	CCV AMOUNT	PCT_DRIFT	PCT_DIFF	RT	RT WINDOW	
DBGSTD-B3-0006	ACID BLUE 9 (*3)	499830	10.775	506244	11.035	1.3	2.4	11.459	9.947	12.647
DBGSTD-B3-0006	BASIC VIOLET 10	717232	7.385	737812	7.626	2.9	3.3	19.652	18.883	20.229
DBGSTD-B3-0006	2-AMINOANTHRAQUINONE	193120	6.625	197086	6.715	2.1	1.4	13.235	12.716	13.620
DBGSTD-B3-0006	SOLVENT ORANGE 3	315344	7.470	322097	7.645	2.1	2.3	13.819	13.609	13.957
DBGSTD-B3-0006	1-AMINOANTHRAQUINONE	100585	9.055	105921	9.382	5.3	3.6	16.074	15.384	16.594
DBGSTD-B3-0006	SOLVENT YELLOW 14	274309	7.075	278053	7.116	1.4	0.6	24.345	23.251	24.975
DBGSTD-B3-0006	SOLVENT ORANGE 7 (*4)	277322	10.395	282593	10.448	1.9	0.5	27.752	27.029	28.299
DBGSTD-C3-0007	ACID YELLOW 3 (*3)	82621	7.460	144860	7.574	75.3	1.5	7.110	3.913	9.739
DBGSTD-C3-0007	SOLVENT YELLOW 3	423110	17.145	435010	17.632	2.8	2.8	19.884	19.074	20.556
DBGSTD-C3-0007	SOLVENT RED 1	319539	8.460	328139	8.449	2.7	-0.1	24.574	23.675	25.321
DBGSTD-C3-0007	ANTHRACENE	6500055	11.555	6642422	12.367	2.2	7.0	22.453	21.703	23.085
DBGSTD-C3-0007	BENZANTHRONE	163006	9.725	167965	9.971	3.0	2.5	20.359	19.550	21.030
DBGSTD-D3-0008	DISPERSE RED 9	116659	6.650	120667	6.836	3.4	2.8	20.300	19.191	21.161
DBGSTD-D3-0008	SOLVENT YELLOW 2	432026	7.385	443941	7.502	2.8	1.6	22.183	21.221	22.929
DBGSTD-D3-0008	SOLVENT GREEN 3	53794	8.215	54751	8.889	1.8	8.2	31.141	30.327	31.765
DBGSTD-A3-0009	ACID YELLOW 23	148554	6.480	135107	5.880	-9.1	-9.3	7.214	3.442	10.110
DBGSTD-A3-0009	ACID YELLOW 73	916212	10.490	966448	10.915	5.5	4.1	8.905	6.372	10.926
DBGSTD-A3-0009	ACID ORANGE 10	181087	15.750	191572	16.487	5.8	4.7	9.735	7.433	11.551
DBGSTD-A3-0009	ACID BLUE 1	596326	12.755	631829	13.358	6.0	4.7	12.581	10.893	13.881
DBGSTD-A3-0009	BASIC YELLOW 2	28494	6.730	30240	7.062	6.1	4.9	19.402	18.376	20.158
DBGSTD-A3-0009	DISPERSE VIOLET 1 (*1)	219260	12.235	279298	15.374	27.4	25.7	12.699	12.440	12.880
DBGSTD-A3-0009	SOLVENT YELLOW 33	236372	6.125	251045	6.294	6.2	2.8	15.456	14.518	16.164
DBGSTD-A3-0009	DISPERSE BLUE 14	113874	7.490	117119	7.544	2.8	0.7	18.809	17.914	19.484
DBGSTD-B3-0010	ACID BLUE 9 (*3)	499830	10.775	460535	9.935	-7.9	-7.8	11.343	9.947	12.647
DBGSTD-B3-0010	BASIC VIOLET 10	717232	7.385	767678	7.937	7.0	7.5	19.607	18.883	20.229
DBGSTD-B3-0010	2-AMINOANTHRAQUINONE	193120	6.625	205859	7.011	6.6	5.8	13.188	12.716	13.620
DBGSTD-B3-0010	SOLVENT ORANGE 3	315344	7.470	336513	7.992	6.7	7.0	13.784	13.609	13.957
DBGSTD-B3-0010	1-AMINOANTHRAQUINONE	100585	9.055	109877	9.733	9.2	7.5	16.014	15.384	16.594
DBGSTD-B3-0010	SOLVENT YELLOW 14	274309	7.075	290281	7.430	5.8	5.0	24.278	23.521	24.975
DBGSTD-B3-0010	SOLVENT ORANGE 7 (*4)	277322	10.395	273598	9.022	-1.3	-13.2	27.688	27.029	28.299

TABLE IV-4A

CCV SHORT GRADIENT
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CCV FILE NAME	PARAMETER	ICAL AREA	ICAL AMOUNT	CCV AREA	CCV AMOUNT	PCT_DRIFT	PCT_DIFF	RT	RT WINDOW	
DBGSTD-A3-0011	ACID YELLOW 23	148554	6.480	125377	5.458	-15.6	-15.8	6.955	3.422	10.110
DBGSTD-A3-0011	ACID YELLOW 73	916212	10.490	975113	11.015	6.4	5.0	8.757	6.372	10.926
DBGSTD-A3-0011	ACID ORANGE 10	181087	15.750	192044	16.528	6.1	4.9	9.591	7.433	11.551
DBGSTD-A3-0011	ACID BLUE 1	596326	12.755	639454	13.522	7.2	6.0	12.475	10.893	13.881
DBGSTD-A3-0011	BASIC YELLOW 2	28494	6.730	31503	7.354	10.6	9.3	19.355	18.376	20.158
DBGSTD-A3-0011	DISPERSE VIOLET 1	219260	12.235	236009	13.001	7.6	6.3	12.718	12.440	12.880
DBGSTD-A3-0011	SOLVENT YELLOW 33	236372	6.125	251825	6.313	6.5	3.1	15.381	14.518	16.164
DBGSTD-A3-0011	DISPERSE BLUE 14	113874	7.490	118245	7.621	3.8	1.7	18.762	17.914	19.484
DBGSTD-B3-0012	ACID BLUE 9 (*3)	499830	10.775	506358	10.929	1.3	1.4	11.242	9.947	12.647
DBGSTD-B3-0012	BASIC VIOLET 10	717232	7.385	762036	7.878	6.2	6.7	19.568	18.883	20.229
DBGSTD-B3-0012	2-AMINOANTHRAQUINONE	193120	6.625	204128	6.953	5.7	5.0	13.168	12.716	13.620
DBGSTD-B3-0012	SOLVENT ORANGE 3	315344	7.470	335644	7.971	6.4	6.7	13.792	13.609	13.957
DBGSTD-B3-0012	1-AMINOANTHRAQUINONE	100585	9.055	112515	9.967	11.9	10.1	15.981	15.384	16.594
DBGSTD-B3-0012	SOLVENT YELLOW 14	274309	7.075	292667	7.491	6.7	5.9	24.224	23.521	24.975
DBGSTD-B3-0012	SOLVENT ORANGE 7 (*4)	277322	10.395	297768	10.959	7.4	5.4	27.642	27.029	28.299
DBGSTD-C3-0013	ACID YELLOW 3 (*3)	82621	7.460	147129	7.638	78.1	2.4	6.834	3.913	9.739
DBGSTD-C3-0013	SOLVENT YELLOW 3	423110	17.145	457333	18.526	8.1	8.1	19.798	19.074	20.556
DBGSTD-C3-0013	SOLVENT RED 1	319539	8.460	343199	8.801	7.4	4.0	24.477	23.675	25.321
DBGSTD-C3-0013	ANTHRACENE	6500055	11.555	7004915	13.119	7.8	13.5	22.359	21.697	23.091
DBGSTD-C3-0013	BENZANTHRONE	163006	9.725	176405	10.463	8.2	7.6	20.276	19.550	21.030
DBGSTD-D3-0014	DISPERSE RED 9	116659	6.650	124141	7.032	6.4	5.7	20.183	19.191	21.161
DBGSTD-D3-0014	SOLVENT YELLOW 2	432026	7.385	468897	7.917	8.5	7.2	22.081	21.221	22.929
DBGSTD-D3-0014	SOLVENT GREEN 3	53794	8.215	59564	9.821	10.7	19.5	31.042	30.327	31.765
DBGSTD-A3-0015	ACID YELLOW 23	148554	6.480	153013	6.656	3.0	2.7	6.760	3.422	10.110
DBGSTD-A3-0015	ACID YELLOW 73	916212	10.490	974195	11.004	6.3	4.9	8.635	6.372	10.926
DBGSTD-A3-0015	ACID ORANGE 10	181087	15.750	194200	16.714	7.2	6.1	9.494	7.433	11.551
DBGSTD-A3-0015	ACID BLUE 1	596326	12.755	638493	13.501	7.1	5.8	12.422	10.893	13.881
DBGSTD-A3-0015	BASIC YELLOW 2	28494	6.730	29700	6.937	4.2	3.1	19.330	18.376	20.158
DBGSTD-A3-0015	DISPERSE VIOLET 1	219260	12.235	235455	12.970	7.4	6.0	12.718	12.440	12.880
DBGSTD-A3-0015	SOLVENT YELLOW 33	236372	6.125	252671	6.334	6.9	3.4	15.365	14.518	16.164
DBGSTD-A3-0015	DISPERSE BLUE 14	113874	7.490	120398	7.767	5.7	3.7	18.744	17.914	19.484

TABLE IV-4A

CCV SHORT GRADIENT

CCV FILE NAME	PARAMETER	ICAL AREA	ICAL AMOUNT	CCV AREA	CCV AMOUNT	PCT_DRIFT	PCT_DIFF	RT	RT WINDOW	
DBGSTD-B3-0016	ACID BLUE 9 (*3)	499830	10.775	534273	11.532	6.9	7.0	11.173	9.947	12.647
DBGSTD-B3-0016	BASIC VIOLET 10	717232	7.385	775065	8.014	8.1	8.5	19.540	18.883	20.229
DBGSTD-B3-0016	2-AMINOANTHRAQUINONE	193120	6.625	205274	6.991	6.3	5.5	13.172	12.716	13.620
DBGSTD-B3-0016	SOLVENT ORANGE 3	315344	7.470	336578	7.993	6.7	7.0	13.808	13.609	13.957
DBGSTD-B3-0016	1-AMINOANTHRAQUINONE	100585	9.055	111325	9.861	10.7	8.9	15.969	15.384	16.594
DBGSTD-B3-0016	SOLVENT YELLOW 14	274309	7.075	293724	7.519	7.1	6.3	24.190	23.521	24.975
DBGSTD-B3-0016	SOLVENT ORANGE 7 (*4)	277322	10.395	274835	9.118	-0.9	-12.3	27.611	27.029	28.299
DBGSTD-A3-0023	ACID YELLOW 23	148554	6.480	144664	6.294	-2.6	-2.9	5.036	3.422	10.110
DBGSTD-A3-0023	ACID YELLOW 73	916212	10.490	985254	11.131	7.5	6.1	7.493	6.372	10.926
DBGSTD-A3-0023	ACID ORANGE 10	181087	15.750	194945	16.778	7.7	6.5	8.430	7.433	11.551
DBGSTD-A3-0023	ACID BLUE 1	596326	12.755	639001	13.512	7.2	5.9	11.606	10.893	13.881
DBGSTD-A3-0023	BASIC YELLOW 2	28494	6.730	29849	6.971	4.8	3.6	18.791	18.376	20.158
DBGSTD-A3-0023	DISPERSE VIOLET 1	219260	12.235	235166	12.954	7.3	5.9	12.553	12.440	12.880
DBGSTD-A3-0023	SOLVENT YELLOW 33	236372	6.125	253947	6.366	7.4	3.9	14.886	14.518	16.164
DBGSTD-A3-0023	DISPERSE BLUE 14	113874	7.490	118313	7.625	3.9	1.8	18.284	17.914	19.484
DBGSTD-B3-0024	ACID BLUE 9 (*3)	499830	10.775	514972	11.552	3.0	7.2	10.421	9.947	12.647
DBGSTD-B3-0024	BASIC VIOLET 10	717232	7.385	766716	7.927	6.9	7.3	19.075	18.883	20.229
DBGSTD-B3-0024	2-AMINOANTHRAQUINONE	193120	6.625	208951	7.115	8.2	7.4	12.847	12.716	13.620
DBGSTD-B3-0024	SOLVENT ORANGE 3	315344	7.470	333974	7.931	5.9	6.2	13.655	13.609	13.957
DBGSTD-B3-0024	1-AMINOANTHRAQUINONE	100585	9.055	114818	10.171	14.2	12.3	15.572	15.384	16.594
DBGSTD-B3-0024	SOLVENT YELLOW 14	274309	7.075	294647	7.542	7.4	6.6	23.764	23.521	24.975
DBGSTD-B3-0024	SOLVENT ORANGE 7 (*4)	277322	10.395	275914	8.954	-0.5	-13.9	27.243	27.029	28.299
DBGSTD-C3-0025	ACID YELLOW 3 (*3)	82621	7.460	90745	5.469	9.8	-26.7	5.180	3.913	9.739
DBGSTD-C3-0025	SOLVENT YELLOW 3	423110	17.145	460140	18.638	8.8	8.7	19.401	19.074	20.556
DBGSTD-C3-0025	SOLVENT RED 1	319539	8.460	349745	8.954	9.5	5.8	24.040	23.675	25.321
DBGSTD-C3-0025	ANTHRACENE	6500055	11.555	7043855	13.199	8.4	14.2	22.016	21.703	23.085
DBGSTD-C3-0025	BENZANTHRONE	163006	9.725	178334	10.576	9.4	8.8	19.875	19.550	21.030
DBGSTD-D3-0027	DISPERSE RED 9	116659	6.650	125600	7.114	7.7	7.0	19.610	19.191	21.161
DBGSTD-D3-0027	SOLVENT YELLOW 2	432026	7.385	468389	7.909	8.4	7.1	21.584	21.221	22.929
DBGSTD-D3-0027	SOLVENT GREEN 3	53794	8.215	60051	9.915	11.6	20.7	30.635	30.327	31.765

TABLE IV-4A

CCV SHORT GRADIENT

CCV FILE NAME	PARAMETER	ICAL AREA	ICAL AMOUNT	CCV AREA	CCV AMOUNT	PCT_DRIFT	PCT_DIFF	RT	RT WINDOW	
DBGSTD-A3-0029	ACID YELLOW 23 (*2)	148554	6.480	39685	1.743	-73.3	-73.1	5.153	3.422	10.110
DBGSTD-A3-0029	ACID YELLOW 73	916212	10.490	986688	11.148	7.7	6.3	7.562	6.372	10.926
DBGSTD-A3-0029	ACID ORANGE 10	181087	15.750	195098	16.791	7.7	6.6	8.515	7.433	11.551
DBGSTD-A3-0029	ACID BLUE 1	596326	12.755	647246	13.690	8.5	7.3	11.665	10.893	13.881
DBGSTD-A3-0029	BASIC YELLOW 2	28494	6.730	29402	6.868	3.2	2.1	18.813	18.376	20.158
DBGSTD-A3-0029	DISPERSE VIOLET 1	219260	12.235	241847	13.321	10.3	8.9	12.534	12.440	12.880
DBGSTD-A3-0029	SOLVENT YELLOW 33	236372	6.125	257280	6.449	8.8	5.3	14.975	14.518	16.164
DBGSTD-A3-0029	DISPERSE BLUE 14	113874	7.490	119801	7.726	5.2	3.2	18.308	17.914	19.484

(*1) ACID BLUE 1 INTERFERENCE
(*2) PEAK SPLITS
(*3) AREA IS BASED ON 3 PEAKS
(*4) AREA IS BASED ON 2 PEAKS

TABLE IV-4B

SHORT GRADIENT PERCENT DIFFERENCES OVER TIME

CCV FILE NAME	PARAMETER	CCV A, B, C, D	CCV A, B, C, D	CCV A	CCV B	CCV A, B, C, D	CCV A	CCV B	CCV A, B, C, D	CCV A
		7/3/00	7/4/00	7/4/00	7/5/00	7/5/00	7/5/00	7/6/00	7/11/00	7/12/00
	TIME FROM ICAL:	24-27 hrs	43-46 hrs	57 hrs	61 hrs	67-69 hrs	78 hrs	84 hrs	212-217 hrs	227 hrs
DBGSTD-A3-0001	ACID YELLOW 23	0.8	3.1	9.3		15.8	2.7		2.9	73.1 (*4)
DBGSTD-A3-0001	ACID YELLOW 73	1.8	4.0	4.1		5.0	4.9		6.1	6.3
DBGSTD-A3-0001	ACID ORANGE 10	3.0	4.9	4.7		4.9	6.1		6.5	6.6
DBGSTD-A3-0001	ACID BLUE 1	3.0	4.7	4.7		6.0	5.8		5.9	7.3
DBGSTD-A3-0001	BASIC YELLOW 2	3.4	5.7	4.9		9.3	3.1		3.6	2.1
DBGSTD-A3-0001	DISPERSE VIOLET 1 (*1)	23.1	24.8	25.7		6.3	6.0		5.9	8.9
DBGSTD-A3-0001	SOLVENT YELLOW 33	0.7	2.6	2.8		3.1	3.4		3.9	5.3
DBGSTD-A3-0001	DISPERSE BLUE 14	0.1	1.1	0.7		1.7	3.7		1.8	3.2
DBGSTD-B3-0002	ACID BLUE 9 (*2)	2.3	2.4		7.8	1.4		7.0	7.2	
DBGSTD-B3-0002	BASIC VIOLET 10	1.8	3.3		7.5	6.7		8.5	7.3	
DBGSTD-B3-0002	2-AMINOANTHRAQUINONE	1.0	1.4		5.8	5.0		5.5	7.4	
DBGSTD-B3-0002	SOLVENT ORANGE 3	1.9	2.3		7.0	6.7		7.0	6.2	
DBGSTD-B3-0002	1-AMINOANTHRAQUINONE	0.7	3.6		7.5	10.1		8.9	12.3	
DBGSTD-B3-0002	SOLVENT YELLOW 14	0.2	0.6		5.0	5.9		6.3	6.6	
DBGSTD-B3-0002	SOLVENT ORANGE 7 (*3)	0.0	0.5		13.2	5.4		12.3	13.9	
DBGSTD-C3-0003	ACID YELLOW 3 (*2)	2.0	1.5			2.4			26.7	
DBGSTD-C3-0003	SOLVENT YELLOW 3	0.8	2.8			8.1			8.7	
DBGSTD-C3-0003	SOLVENT RED 1	2.2	0.1			4.0			5.8	
DBGSTD-C3-0003	ANTHRACENE	5.4	7.0			13.5			14.2	
DBGSTD-C3-0003	BENZANTHRONE	0.3	2.5			7.6			8.8	
DBGSTD-D3-0004	DISPERSE RED 9	0.5	2.8			5.7			7.0	
DBGSTD-D3-0004	SOLVENT YELLOW 2	0.3	1.6			7.2			7.1	
DBGSTD-D3-0004	SOLVENT GREEN 3	10.5	8.2			19.5			20.7	
Average Pecent Difference		**2.9**	**4.0**	**7.1**	**7.7**	**7.0**	**4.5**	**7.9**	**8.5**	**14.1**

(*1) ACID BLUE 1 INTERFERENCE
(*2) AREA BASED ON 3 PEAKS
(*3) AREA BASED ON 2 PEAKS
(*4) PEAK SPLITS

TABLE IV-5

RETENTION TIMES WATERS' XTERA COLUMN VS VARIAN'S MICROSPHERE COLUMN
LONG GRADIENT

DYE	RT XTERA	RT VARIAN
ACID BLUE 45	11.815	
ACID YELLOW 3 P1	14.813	
ACID RED 64	14.923	
ACID YELLOW 23	16.155	
ACID YELLOW 3 P2	17.610	
ACID YELLOW 73	18.624	
ACID ORANGE 10	19.868	
ACID BLUE 9	22.626	
DISPERSE VIOLET 1	22.685	
2-AMINOANTHRAQUINONE	23.941	
ACID BLUE 1	24.418	
SOLVENT ORANGE 3	24.836	
DISPERSE RED 9	26.420	
SOLVENT YELLOW 33	28.586	
1-AMINOANTHRAQUINONE	29.163	
SOLVENT YELLOW 2	31.920	
DISPERSE BLUE 14	34.292	
BASIC YELLOW 2	35.595	
BASIC VIOLET 10	36.254	
SOLVENT YELLOW 3	36.396	
BENZANTHRONE	37.073	
ANTHRACENE	41.078	
SOLVENT YELLOW 14	44.816	
SOLVENT RED 1	45.241	
SOLVENT ORANGE 7	51.324	
SOLVENT GREEN 3	57.532	
SOLVENT RED 24	58.058	

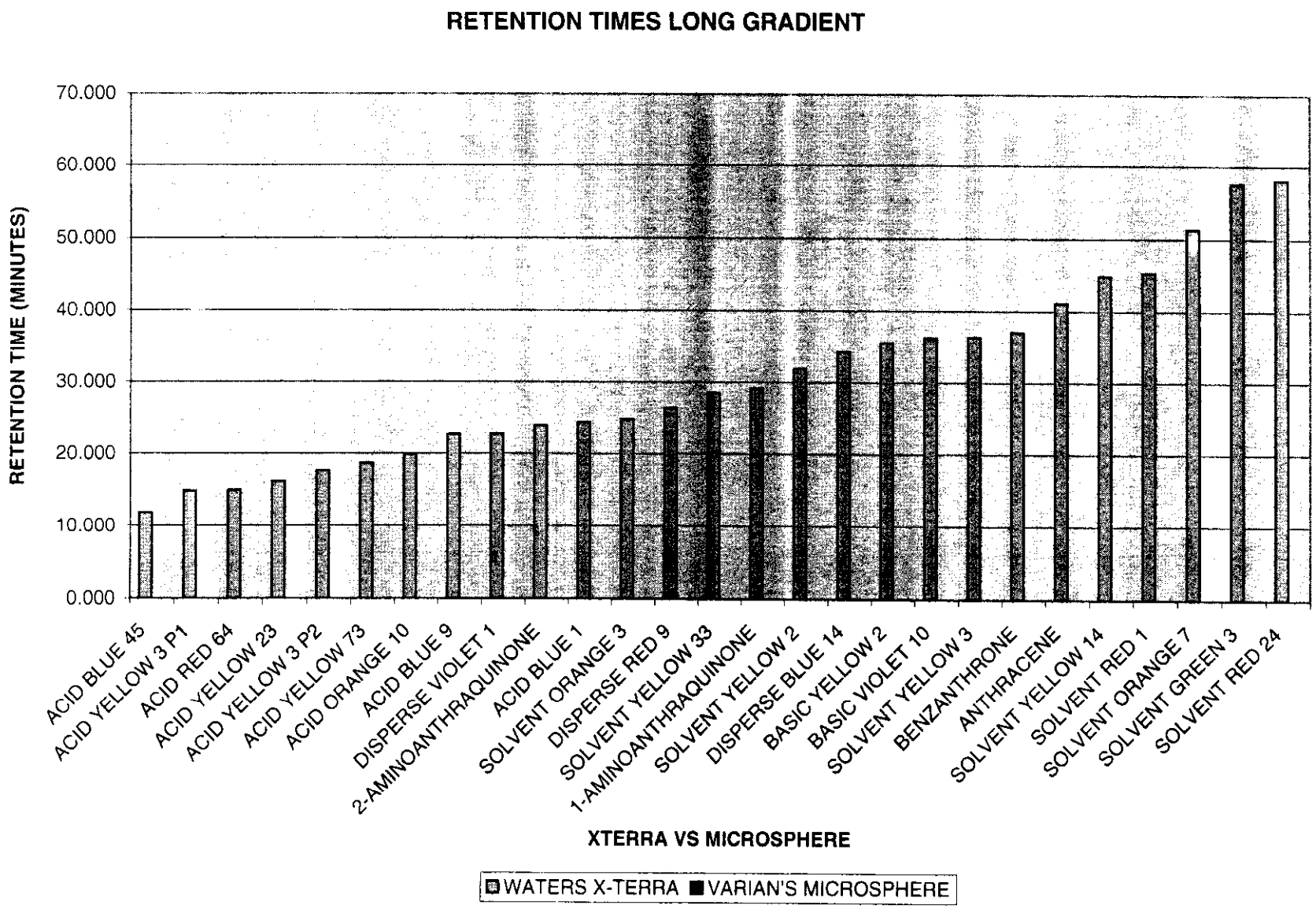
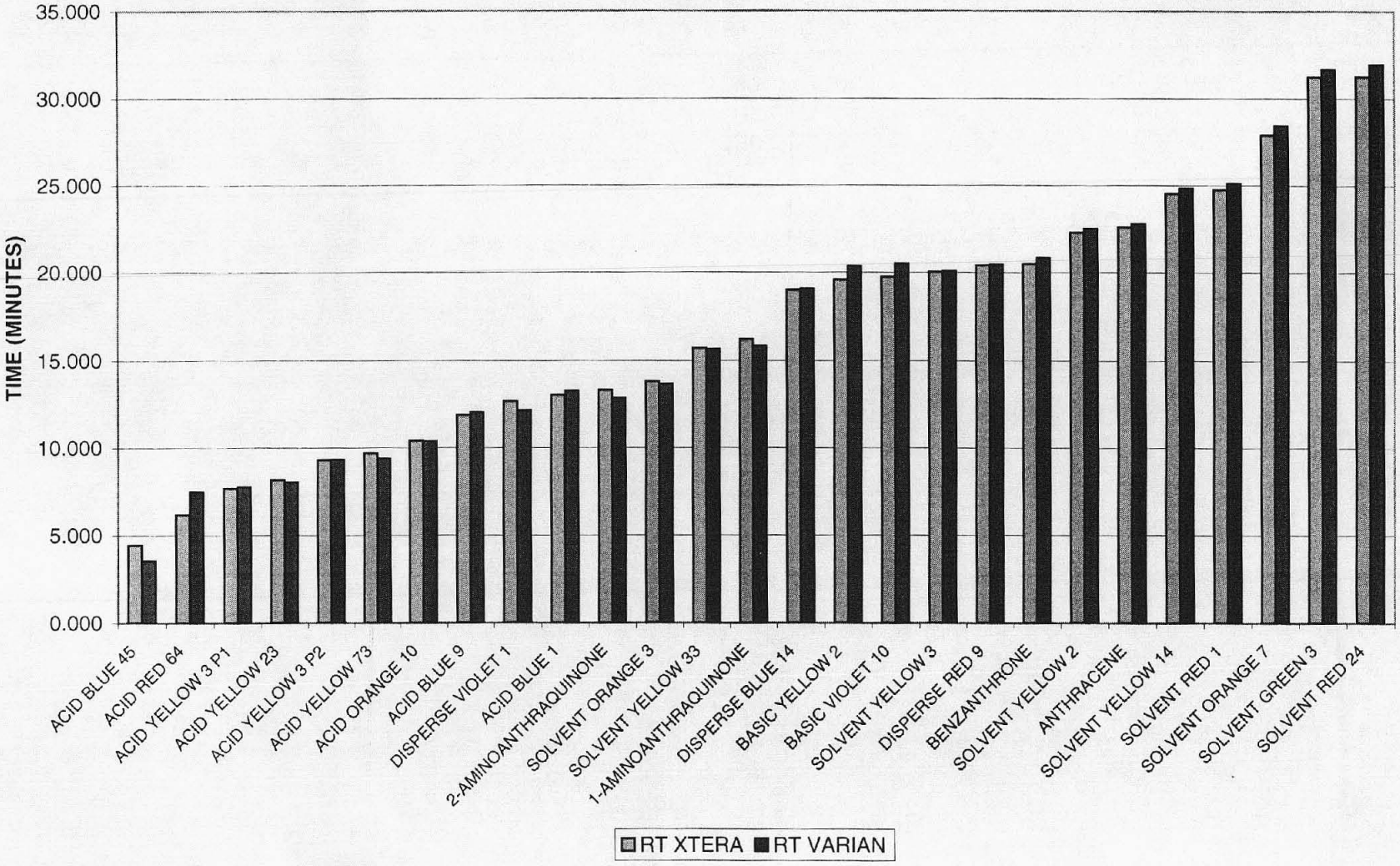

RETENTION TIMES LONG GRADIENT
RETENTION TIME (MINUTES)
70.000
60.000
50.000
40.000
30.000
20.000
10.000
0.000
ACID BLUE 45
ACID YELLOW 3 P1
ACID RED 64
ACID YELLOW 23
ACID YELLOW 3 P2
ACID YELLOW 73
ACID ORANGE 10
ACID BLUE 9
DISPERSE VIOLET 1
2-AMINOANTHRAQUINONE
ACID BLUE 1
SOLVENT ORANGE 3
DISPERSE RED 9
SOLVENT YELLOW 33
1-AMINOANTHRAQUINONE
SOLVENT YELLOW 2
DISPERSE BLUE 14
BASIC YELLOW 2
BASIC VIOLET 10
SOLVENT YELLOW 3
BENZANTHRONE
ANTHRACENE
SOLVENT YELLOW 14
SOLVENT RED 1
SOLVENT ORANGE 7
SOLVENT GREEN 3
SOLVENT RED 24
XTERRA VS MICROSPHERE
WATERS X-TERRA
VARIAN'S MICROSPHERE

TABLE IV-6

RETENTION TIMES WATERS' XTERA COLUMN VS VARIAN'S MICROSPHERE COLUMN
SHORT GRADIENT

DYE	RT XTERA	RT VARIAN
ACID BLUE 45	4.439	3.549
ACID RED 64	6.174	7.462
ACID YELLOW 3 P1	7.684	7.756
ACID YELLOW 23	8.180	8.032
ACID YELLOW 3 P2	9.295	9.306
ACID YELLOW 73	9.672	9.376
ACID ORANGE 10	10.405	10.364
ACID BLUE 9	11.866	12.035
DISPERSE VIOLET 1	12.670	12.136
ACID BLUE 1	13.019	13.251
2-AMINOANTHRAQUINONE	13.317	12.851
SOLVENT ORANGE 3	13.813	13.657
SOLVENT YELLOW 33	15.705	15.642
1-AMINOANTHRAQUINONE	16.208	15.826
DISPERSE BLUE 14	19.010	19.080
BASIC YELLOW 2	19.586	20.349
BASIC VIOLET 10	19.757	20.517
SOLVENT YELLOW 3	20.030	20.078
DISPERSE RED 9	20.419	20.477
BENZANTHRONE	20.500	20.848
SOLVENT YELLOW 2	22.281	22.508
ANTHRACENE	22.604	22.801
SOLVENT YELLOW 14	24.530	24.835
SOLVENT RED 1	24.741	25.127
SOLVENT ORANGE 7	27.909	28.434
SOLVENT GREEN 3	31.219	31.659
SOLVENT RED 24	31.255	31.948

RETENTION TIMES (SHORT GRADIENT): WATERS' XTERA VS VARIAN'S MICROSPHERE



Calibration - May 11, 2000

Project No.
Book No.

No.

	Level 1	Level 2	Level 3	Level 4	R^2	R
*	15959	84465	133426	239094	0.992997	0.996496
	11375	253480	525708	1044488	0.999863	0.999931
	116336	919562	1884570	3704283	0.999867	0.999934
	15384	157299	323463	644054	0.999946	0.999973
	76953	631542	1289547	2524227	0.999858	0.999929
	3771	23780	55578	110147	0.997843	0.999921
	34962 Ignore	156278	345727	923650	~~0.975969~~ 8.931147	~~0.987992~~ 8.933294
	36486	287694	577645	1150159	0.999970	0.999985
	19981 Ignore	94076	219125	509619	0.998718	0.999359
	BDL	87055	235011	520664	0.999923	0.999961
	1731+18133+44390	33350+259982+661748	63197+508082+1319343	122559+2760438+827119		
	115984	1216561	2446379	4865522	0.999983	0.999992
	25823	287243	595491	1236799	0.999500	0.999780
	4809	256998	592685	1190910	0.998907	0.999453
	18942	182345	364704	7726138	0.999996	0.999999
	16795	172436	367959	755048	0.999531	0.999765
	BDL	BDL	BDL	9		
	15521+4731	228149+146704+102585	475171+329952+209340	965842+1391454+430886		
	BDL	BDL	BDL	BDL		
	BDL	BDL	BDL	BDL		
	BDL	19643	39542	77104	0.998790	0.999395
	374425	3828486	7671263	15154506	0.999948	0.999974
	20397	219384	448090	908931	0.999931	0.999965
	12329	105156	207091	414822	0.999986	0.999993
	32746	299032	662077	1370571	0.998852	0.999426
	5313	39567	93423	331653	0.945518	0.972375
	6922	59254	124061	243948	0.999755	0.999878

15: Sewer tailing
lard Injection: 10µL
ple Injection: 80µL

	6182+19556	44927+185269	89996+377149	188165+766476		

~~Extraction (High):~~
~~Extraction (Low):~~
~~Organic Extraction:~~

To Page No.

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		Recorded by	

From Page No.__

Type	Level 1	Level 2	Level 3	Level 4	R^2/R
AB45	BDL	25121	129878	255950	0.981385
AY23	13222 *	254489	512831	1035335	0.990649
AY73	~~2~~ 111180 *	898634	1829845	3669362	0.999969
AO10	15340	157750	319821	641907	0.999965
AB1	73533	608578	~~[illegible]~~ 1229497	2470350	0.999906
BY2	BDL	23108	52254	107244	0.999950
DV1	33854 ■	217168	458077	971515	0.999987
SY33	39542	297060	581618	1166340	0.999993
DB14	15993	113990	240779	509569	0.999929
AR64	5737	114584	249970	536118	0.999765
AB9	1287 / 17614 / 44319 ○	19032 / 164173 / 423791	39203 / 334336 / 866288	80109 / 678565 / 1755420	0.999787
BV10	BDL	29700	59997	118808	0.999973
2AQ	BDL	Not Detected	Not Detected	Not detected	0.998051
SO3	28736 ○	273043 ○	551907 ○	1123233 ○	0.999025
1AQ	11343 △	119705 △	243161 △	488252 △	0.999955
SY14	21106	188112	383442	792695	0.999978
SO7	19022 / 5209	184795 / 43104	379510 / 91471	778411 / 188526	0.998249
SY3	23697 / 4044 ✓	231441 / 7102 / 37711	456805 / 20505 / 76976	972494 / 57234 / 170599	0.999124
9D	BDL	BDL	BDL	BDL	0.998822
SY3	30584	291586	595510	1248904	0.999411
BZ	BDL	19000 ○	~~753 7819~~ 38257 ○	78617 ○	0.999250
AN	441126 ○	3753039 ○	7537819 ○	15298991 ○	0.999625
SR1	21405	222217	456478	953872	0.999745
DR9	BDL	98738	202430	405613	0.999406
SY2	42277	315792	645892	1364272	0.999703
SG3	4729	41780	93687	325976	0.999724
SR24	7432	55511	121631	233895	0.999857
					0.998663
					0.999331
					0.999117
					0.998945
					0.999473

* ~~Did not~~ Software did not ID peak
□ Software miss identified peak
○ IDed peak but no cal pt.
△ 1AQ PM does not see 1AQ peak - only sees it under 2AQ PM

To Page No.

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	

ATTACHMENTS V and VI

EXTRACTION PROCEDURE

and

METHOD OPTIMIZATION

ATTACHMENT V AND VI – EXTRACTION PROCEDURE AND METHOD OPTIMIZATION

The Crane Laboratory performed the extraction and analysis optimization in tandem. Documentation of this portion of the method development is limited and consists primarily of scattered laboratory notes and several Excel tables. During this phase of the development, TLC was abandoned as a screening method, extraction procedures using SPE disks and TBA modifiers were "tweaked," and the HPLC procedure was shortened. TtNUS chemists visited the laboratory and through interviews with the Crane chemists ascertained that method development was proceeding in accordance with the specified criteria despite the spotty nature of the documentation. The findings of the TtNUS lab visit are also included in this attachment.

LABORATORY VISIT SUMMARY

SOUTHDIV CLEAN – NSWC Crane
May 16, 17, and 18, 2000
Recorded by TtNUS

Subject: Summary of visit to the Crane Laboratory for SWMU 2 Dye Burial Grounds

Attendees: Crane Lab – Jackie Karty; Forrest Hawman; TtNUS – Linda Karsonovich; ECT.CON, Inc – Terrie Baranek

Purpose: The purpose of the visit was to assist the Laboratory in assembling documentation needed for EPA approval of the developed methods and to evaluate the Laboratory's current extraction methodologies.

Background: The Laboratory has been working to develop two analytical methods. The first was a screening method for soil samples using thin layer chromatography (TLC). The second method was a quantitative method for soil and water samples using high performance liquid chromatography (HPLC).

Current Status:

- ***TLC*** – The use of TLC as a screening method for soil samples has been abandoned. The key difficulty was exchanging the extraction solvent (Tributylammonium hydroxide [TBA] and water) into the analytical solvent (methanol). The solvent exchange required approximately three hours per sample and dye compound recoveries were low or undetectable. The decision to abandon TLC as a screening method was based on the time required per sample extraction and the low recoveries. The TLC extraction method was slower than the HPLC method, and the recoveries for the TLC were substantially lower than those obtained for the HPLC. Therefore, the TLC method could not provide quick turnaround or a reasonable indication of the presence/absence of dye compounds in the soils.

- ***HPLC*** – The HPLC will be used for both groundwater and soil matrices. The Crane Laboratory is currently developing three analytical scenarios: solid, high-level aqueous, and low-level aqueous. The analytical method will be the same for all sample matrices.

  Solid Extraction and Analysis – The Crane Laboratory has developed a solid extraction method that uses TBA, water, and methanol to pull dye compounds from soils and sediments. Recoveries range from 49% to 400%. The following issues were noted:

  1. Smoke Dye, Solvent Yellow 2, and Solvent Red 24 were not recovered at either the high or low-level spike concentrations. TtNUS does not recommend any further development efforts to obtain these particular compounds. This recommendation is based on the fact that the TBA/water extraction method yields the best recoveries for most compounds of any extraction method tried to date. The key difficulty in developing the extraction method for the HPLC is the varied chemical natures and structures of the dye compounds. Realistically, based on the chemical composition of the compounds, no one extraction method will be able to successfully extract all the compounds from the soils. The Crane Laboratory has used sonication and soxhlet extractions with enough variety of other solvents to eliminate other avenues of investigation. Also, Dr. Baughman agreed that the TBA/water extraction method would be the best avenue for extraction based on his experience.

2. The compound Acid Blue 45 was not recovered at the low-level concentration; but was recovered at the high-level concentration (50%). The reporting level and spiking concentration for this compound will need to be adjusted; the Laboratory will continue to work on this issue. Again, TtNUS does not recommend any further development efforts to increase recovery of this particular compound for the same reasons as specified in Item 1.

3. No recoveries were reported for Pigment Red 1 since the compound was not included in the spiking solution. The compound was not included in the spiking solution due to the low solubility of the compound and the limited size of spiking solution.

4. No recoveries were reported for Solvent Yellow 3 and Solvent Orange 7. These compounds were detected; however, the peaks chromatographed as doublets. The Crane Laboratory had not yet calculated the percent recoveries.

5. Recoveries of Acid Red 64, Basic Yellow 2, 2-Aminoanthraquinone, and Disperse Blue 14 ranged from 190% to 423%. The Crane Laboratory indicated that this was probably due to the need for a new spiking solution and/or new calibration curve. TtNUS concurred. The Crane Laboratory was working on developing new standards at the time of the laboratory visit.

6. Based on the effort thus far, TtNUS recommended that the Crane Laboratory stop experimenting with extraction techniques and method development and move to the method validation or "proof of concept" stage. This consists of spiking the full suite of compounds into seven replicates at a low-level concentration and seven replicates at a high-level concentration. The resulting information will be used to calculate recovery, precision, matrix-specific method detection level (MDL), matrix-specific reliable quantitation limit (RQL), and accuracy (bias) for the soil extraction method.

High-Level Aqueous Analysis – The Crane Laboratory has not performed any experimental test of the high-level procedure. The high-level procedure consists of taking an aliquot of the water sample and directly injecting the sample on the HPLC. Since an extraction procedure is not necessary, solubility of the dyes in aqueous matrices will be the limiting factor for compound recovery. TtNUS recommended that the Crane Laboratory perform the method validation consisting of spiking the full suite of compounds into seven replicates at a low-level concentration and seven replicates at a high-level concentration. The resulting information will be used to calculate recovery, precision, matrix-specific MDL, matrix-specific RQL, and accuracy (bias) for the high-level aqueous analysis method. The following issues were noted:

1. A water solubility test was performed by Crane Laboratory. The following compounds were noted to be insoluble in water: Pigment Red 1, Disperse Red 9, Solvent Green 3, Solvent Orange 7, Disperse Blue 14, Solvent Yellow 14, and Smoke Dye. The following compounds were noted to be slightly soluble in water: Disperse Violet 1, Solvent Orange 3, and Solvent Yellow 33.

2. The following compounds were not included in the water solubility test: 1 and 2-aminoanthraquinone, Solvent Yellow 3, Solvent Red 1, Solvent Yellow 2, and Solvent Red 24.

Low-level Aqueous Extraction and Analysis – The Crane Laboratory was performing experimental tests of the extraction procedure during the laboratory visit. The extraction consists of adding TBA to the aqueous sample, filtering the sample through a solid phase extraction disc to capture the dyes, and subsequently pulling the dyes off the extraction disc with methanol. The following issues were noted with this method:

1. The Laboratory was performing the analyses during the visit. Recoveries should be available during the week of May 22, 2000.

2. The Crane Laboratory noted that the addition of TBA to the aqueous sample was clouding the sample. The Laboratory indicated that this was probably due to the type the TBA they used since the effect was not noted in previous experiments using TBA from a different manufacturer. During the trail and error phase, the Laboratory did not use HPLC grade TBA in order to conserve their supplies. They still have "good" TBA to move on to the calibration, proof of concept, and method validation; therefore, this issue will not impact progress on the method development. When the Laboratory orders additional TBA for the project, they will order the appropriate type of TBA.

- ***Analytical Methodology*** – The Crane Laboratory has successfully modified the HPLC analytical run time from 62 minutes to 35 minutes. The following issues were noted:

  1. Six compounds were noted to co-elute in three pairs. Although coelution occurs, the Laboratory will be able to identify whether these chemicals are present in the environmental samples. If these dyes are determined to be present in the samples, the samples will be re-analyzed using the 62-minute run time in order to achieve separation of these compounds.

  2. Due to the shorter run time, the Laboratory proposed eliminating the use of anthracene as a retention time marker. Since a shorter run time will be used retention time shifting will be drastically reduced and will not be an issue as it was for the 62-minute run. Therefore, to reduce confusion and since it was not longer essential, the Crane Laboratory recommended the elimination of this surrogate. TtNUS concurred. This is not considered to be problematic because benzanthrone will still be used as a surrogate.

  3. In order to document instrument performance over time, the Crane Laboratory will analyze continuing calibration standards over a period of time. This will help to establish percent drift in the retention times and reported amounts of the compounds.

  4. Several compounds consist of two or more peaks in the chromatogram. The Crane Laboratory proposed the use of the primary (biggest) peak as sufficient for quantitation. TtNUS concurred. This approach will be evaluated during method validation.

Related Issues:

- **Minimum Required Supplies/Instrumentation List** – The Crane Laboratory and TtNUS pulled together a minimum required list of supplies and instrumentation that would be needed to ensure the smooth operation of the analytical laboratory during the field event. Please refer to Attachment 1.

- **Schedule** – The Crane Laboratory and TtNUS developed a schedule of events required for completion of method development, method validation, and subsequent submittal of the standard operating procedure (SOP) and support documentation to EPA. Please refer to Attachment 2.

- **Reporting** – In order to streamline the reporting of sample data, TtNUS recommends the use of screening data packages and full data packages. Where dye compounds are present in the samples, the Crane Laboratory would submit full data packages consisting of all applicable forms (Form Is and QA/QC forms) and raw data. Where dye compounds are not

present in the samples, the Crane Laboratory would submit screening data packages consisting of only the applicable forms. Raw data would be stored electronically at the laboratory. Please refer to Attachment 3 for specific details.

- **Bulk Density** – The Crane Laboratory requires funding to purchase a water bath in order to perform the Bulk Density analysis. If funding is approved, the Crane Laboratory will write an SOP.

- **Non-analytical SOPs** – The Crane Laboratory will need to complete an SOP for Percent Moisture Determination. All other non-analytical SOPs have been completed and submitted to TtNUS.

ATTACHMENT 1

Minimum Required Supplies/Instrumentation List
NSWC Crane Explosives Science Branch Laboratory
SWMU 2 - Dye Burial Ground

Item (in priority)	Quantity	Price*
1. Bulk Density Equipment		
Just the water bath	1	$ 2200
The bulk density set up (special price for entire setup)	1	$ 2800
2. TBA	800 mL	$ 207
3. HPLC Columns (sold as 3 for $900)	3	$ 900
4. Manifold System with glassware	1	$ 600
5. HPLC pump with autosampler (to complete a backup system for the current HPLC system)	1	$ 27,000
6. Miscellaneous Glassware		
200 mL volumetric flask ($30 for 1)	5	$ 150
1 L volumetric flask ($50 for 1)	5	$ 250
syringes ($2 for 1)	5	$ 10
7. Column Guards (5 for $175)	5	$ 175

*Prices are estimated

NOTES:

- Without Item 1, the Laboratory will not be able to perform the bulk density analysis. For the number of samples to be analyzed for bulk density, the analytical cost for a commercial laboratory will be similar to the cost of the equipment. By allowing the Crane Laboratory the capability to perform the analysis, it provides more flexibility to the project (i.e., analyze additional samples for this project as well as other projects).
- To ensure that analysis may continue throughout the project, extra TBA (Item 2) should be maintained in the Laboratory. The analyses cannot be completed without the TBA. Because TBA is considered to be a hazardous material, it takes substantial time for the Laboratory to obtain the material. Therefore, a backup of the material will ensure continued analytical progress during the project.
- The current manifold system (Item 4) that the Laboratory has consists of three setups. Two of the setups are dedicated to another project. Only one setup is available for use for the SWMU 2 Dye Burial Ground project. The availability of additional setups to enhance the extraction process will assist in expediting the overall analytical process. This will allow for quick turnaround that will be required for the sampling program designed for SWMU 2.

ATTACHMENT 2

Schedule for Remaining Method Development
NSWC Crane Explosives Science Branch Laboratory
SWMU 2 - Dye Burial Ground

Action	Estimated Date Start	Estimated Completion Date	Date Info Emailed to TtNUS
Finish low-level extraction analysis	5/18/00	5/24/00	
Make new standards	5/19/00	5/19/00	
Run initial calibration	5/19/00	5/22/00	
Develop curves	5/22/00	5/26/00	
Extract high level aqueous hi/low samples (7+7)	5/29/00	6/6/00	
Analyze high level aqueous samples	5/29/00	6/6/00	
Assemble Full Data Package	6/6/00	6/16/00	
Compile Method Validation Table	6/6/00	6/16/00	
Extract soil hi/low samples (7+7)	6/19/00	6/23/00	
Analyze hi/low samples	6/19/00	6/23/00	
Assemble Full Data Package	6/26/00	6/30/00	
Compile Method Validation Table	6/26/00	6/30/00	
Extract low-level aqueous hi/low samples (7+7)	7/10/00	7/14/00	
Analyze low-level aqueous hi/low samples	7/10/00	7/14/00	
Assemble Full Data Package	7/17/00	7/21/00	
Compile Method Validation Table	7/17/00	7/21/00	
Write HPLC SOP	7/24/00	7/28/00	
Write Grain Size SOP	7/31/00	8/4/00	
Write Bulk Density SOP	7/31/00	8/4/00	
Tie up loose ends/vacation and sick time buffer	8/4/00	8/31/00	

ATTACHMENT 3

Reporting Packages
NSWC Crane Explosives Science Branch Laboratory
SWMU 2 - Dye Burial Ground

Full Data Package: A full data package will be compiled and submitted to TtNUS when dyes are detected in the environmental samples. The package will consist of the following:

Case Narrative: Sample and laboratory identification cross reference table, a discussion of any problems encountered during extraction or analysis, signature of laboratory quality assurance officer.

Forms 1: Analytical results for each sample, method blank, blank spike, matrix spike and matrix spike duplicate. Forms should also contain the following: Sample ID, Lab ID, Date Collected, Date Extracted, Date Analyzed, Initial Weight/Volume, Final Volume, Dilution Factor, Percent Moisture, File Number, Reporting Limits, and Qualifiers. The raw data consisting of spectra, chromatogram, and quantitation report must accompany each Form.

Surrogate Recoveries: Tabulate recoveries for each sample, method blank, blank spike, and matrix spike. Include acceptance range for surrogate recoveries for specific matrix.

Blank Spikes: Tabulate amount spiked, percent recoveries, and acceptance ranges for specific matrix.

Matrix Spike/ Matrix Spike Duplicate: Tabulate amount spiked, amount present in native sample, percent recovery, relative percent difference, and acceptance ranges for specific matrix.

Initial Calibration: Summarize all data points from each of the five levels and calculate percent relative standard deviation and/or correlation coefficient. Include linear equations if used. The raw data for each standard consisting of spectra, chromatogram, and quantitation report must accompany the summary.

Continuing Calibration: Calculate and present in tabular form percent difference (percent drift) for each analyte. Calculate and present in tabular form retention time and retention time windows for each analyte. The raw data for each standard consisting of spectra, chromatogram, and quantitation report must accompany the summary.

Support Documentation: Copies of the chain-of-custody, sample check-in sheet, extraction log, HPLC log, and standard prep log must accompany each sample delivery group (SDG).

Screening Level Data Package: A screening level data package will be compiled and submitted when dyes are not detected in the environmental samples. The package will consist of everything submitted for a full data package minus the associated raw data. A chromatogram will be included with each sample and method blank to confirm the absence of peaks; however, all remaining raw data (spectra and quantitation report) will be stored electronically by the Laboratory.

Thin-Layer Chromatography

Thin-layer chromatography is a separation method based on the adsorption of the sample to the stationary phase. The adsorbent layer (stationary phase) is usually formed on a 20 x 20 cm glass plated and consists of silica gel. The sample moisture is applied to the lower edge of the plate. The plate is then placed in a tank with a suitable developing solvent(s). The individual substances from the sample mixture are transported upwards at differing rates. Adsorption, partition, or ion-exchange processes effect the separation. When the solvent has traveled sufficiently up the silica plate, the plated is removed and dried. The separated substances are detected visually, by UV lamp, or modified with a staining reagent. The substances are identified by the ration: rate of migration from the starting point to the distance the solvent traveled from the starting point. This value is called the R_F value.

The thin-layer chromatography (TLC) method was originally proposed as a screening method to rapidly detect dyes in solid matrices. The method was based on spotting a sample dissolved in methanol on a preconcentrated silica plate and detecting the presence or absence of dye visually. The method analyzed up to 16 samples simultaneously and separated dyes rapidly.

The extraction method development phase for the solid matrix evaluated both sonication and soxhlet extractions. It was determined that sonication extraction extracted the dyes more efficiently in a short period of time. This helped to meet the requirement for a 48 hour result turn around time. However, the extraction solvent had to rotary evaporated and exchanged with methanol so the sample would be in 100% methanol to meet TLC requirements. The preparation for TLC analysis was complicated further because the sonication broke the soil down into very fine particles. This made the filtration of the sample prior to rotary evaporation extremely difficult and time consuming. In addition, the rotary evaporation step could only be performed on one sample at a time and took approximately 3 hour per sample to complete the evaporation and solvent exchange.

The use of TLC as a screening method for soil samples has been abandoned. The key difficulty was exchanging the extraction solvent (Tributylammonium hydroxide [TBA] and water) into the analytical solvent (methanol). The solvent exchange required approximately three hours per sample and dye compound recoveries were low or undetectable. The decision to abandon TLC as a screening method was based on the time required per sample extraction and the low recoveries. The TLC extraction method was slower than that required for HPLC and the recoveries were substantially lower. Therefore, the TLC method could not provide quick turnaround or a reasonable indication of the presence/absence of dye compounds in the soils.

Project No.

T.E. High Method - Water MDL Book No. 1

From Page No. High

7 samples total - spike 4 mL of each stock standard dye into 250 mL volumetric flask. (Used standards on pg 133 except for Sample #7 - ran out of DR9, SY2, + SG3; made fresh stock standards). Evaporated methanol under a stream of nitrogen. Diluted to 250 mL with well water.

Note - Sample #2 - AB1 will have spiking error.

New Stock Standard	Weight		ug/mL
SG3	15.7 mg	in 50 mL =	314.0
SY2	19.7 mg		394.0
DR9	15.3 mg		306.0

~~Zero~~

~~took 5 mL of each High sample. Diluted to 10 mL.~~

~~Stored in refrigerator over weekend~~

~~6/2/2000 AK~~

To Page No.

Witnessed & Understood by me, | Date | Invented by | Date

Book No._____ TITLE Soil High Spike IDX

From Page No.__

Sample #	Soil Wt (g)	Vol. Spiked (µL)
1	10.0881	500
2	10.1335	500
3	10.0871	500
4	10.0276	500
5	10.2040	500
6	10.5035	500
7	10.0716	500
Blank	10.0099	N/A

Cleaned volumetric flasks 3 times with methanol. Dried under a gentle stream of nitrogen. Weighed approximately 10 grams dry, meshed soil from dye burial grounds onto a plastic weighing boat. Transferred to volumetric flask. Spiked on 500 µL of each dye from the stock standards. Dried soil under nitrogen after every 5th dye to prevent dye from getting onto sides of volumet

6/5/2000 GK

To Page No

Witnessed & Understood by me, | Date | Invented by | Date

Project No.

TITLE Soil- Low Spike MDL

Book No.

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Sample #	Soil Wt (g)	Vol. Spiked (µL)
1	10.0946	100
2	10.0175	100
3	10.0136	100
4	10.0155	100
5	10.0428	100
6	10.0303	100
7	10.0841	100

Cleaned volumetric flasks 3 times with methanol. Dried under a gentle stream of nitrogen. Weighed approximately 10 grams of tray mixed soil from dye burial gardens onto a plastic weighing boat. Transferred to volumetric flask. Spiked on 100µL of each dye from the stock standards. Dried soil under nitrogen to prevent dye from getting onto sides of volumetric.

6/5/2000 JK

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Sample	Amt (uL) each dye	Date Spiked	Date Ext	Date Analyzed
WLM001 HS	500	6/19/00	6/19/00	No TBA
WLM002 HS	500	6/19/00	6/19/00	No TBA
WLM003 HS	500	6/19/00	6-19-00	No TBA
WLM004 HS	500	6/19/00	6-21-00	5mL TBA (evap down
WLM005 HS	500	6/19/00	6-20-00	No TBA
WLM006 HS	500	6-20-00	6-20-00	5mL Aldrich TBA
WLM007 HS	500	6-20-00	6-21-00	5mL Aldrich TBA
WLM008 HS	500	6-21-00	6-21-00	5mL Aldrich TBA
WLM009 HS	500	6-21-00	6-22-00	5mL Aldrich TBA
WLM010 HS	500	6-21-00	6-22-00	5mL Aldrich TBA
WLM011 HS	500	6-22-00	6-23-00	5mL Aldrich TBA
WLM012 LS	100	6-22-00	6-23-00	5mL Aldrich TBA

$\frac{300\,\mu g}{mL} \times 0.5 = \frac{150\,\mu g}{5\,mL} = \frac{30\,\mu g}{mL}$

Add 5mL TBA to sample prior to extraction

Empore (3M) High performance oil & grease extraction disks — 47mm - Lot 470150

Pre-treatment - 50mL methanol - rinse sides of apparatus - pull thru to waste. 50mL methanol - rinse side of apparatus. Pull thru to collection flask. Rinse flask + dispose of methanol.

Add all of the sample.
Start: 10:10AM End: 10:45A

Dry 5 minutes on vacuum

Extract w/ 30 mL methanol + 20 mL rinse

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TLE Book No.

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MC03HS + OLM004HS - heavily particulated

Sample	Amt (µL) each dye	Date Sealed	Date Ext
OLM002LS	100	6-24-00	6-24-00
OLM003LS	100	6-24-00	6-24-00
OLM004LS	100	6-24-00	6-24-00
OLM005LS	100	6-24-00	6-26-00
OLM006 LS	100	6-26-00	6-26-00
OLM007M_BLNK	0	N/A	7-6-00
OLM008 LS	100	7-7-00	7-7-00

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Spiked 200 µL of each dye into water from DBG wells.
Evaporated under nitrogen until most of the methanol evapo
Diluted to 25 mL with DBG well water.

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mL of 60/40 ACN/H_2O (120mL ACN; 80mL H_2O) in a 250mL
nd bottom flask. Added glass beads as boiling stones.
935 g of dry burial grounds clean soil into a cellulose
traction tube. Added 5 glass beads. Hooked up soxhlet system
ing was not proceeding smoothly so added "boilerzers" (Fisher B-365)
soxhlet until 3:00 PM (approximately 6.5 hours total).
ed system down and removed soxhlet. Place round
tom under a nitrogen purge (no heat) and wrapped
aluminum foil. Left for weekend (2.5 days). On Monday
rning, turned on ~~to~~ hot plate and heated to evaporate to
ness. Added 2mL methanol to round bottom. Filtered using
op 0.2µm filter. Injected 30µL onto HPLC.

ered through 0.45µm glass/nylon filter to remove small
unt of soil particulates.

5- No
3- 16.247 ☆
1- No
P1- 15.580 ☆
- 18.730 ☆
- No
No
- No AB-1
- No BV10
- 30.763 ☆ BY2
No 2AQ
24.797 SO3
28.713 1AQ
45.180 - spectra distorted SY14
51.220 SO7
— AB
36.363 SY3
? BZ
40.080 1AN

SR1 - 45.~~358~~ 680
DR9 - No
SY2 - 40.980
SG3 - 58.630
SR24 - ?
DB14 - 34.363
DV1 - 22.297
SY33 - 29.330
PR1 - No

Notes in blue ink added by the NSWC crane lab
JEJ 10-2-00

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~~H_2O~~ ~~10% ACN~~
~~10 mL~~
~~10 mL~~
~~10 mL~~

Acid Dyes

200 mL total
- 10 mL acetic acid (5%)
- 80 mL H_2O (40%)
- 110 mL ACN (55%)

Dol: 5.0560 g

Spiked AB45, AR64, AO10, AB9, AB1, BV10 (100 µL each stock std)

Organic Dyes

200 mL total
- 80 mL H_2O (40%)
- 120 mL ACN (60%)

Dol: 5.1110 g

2AQ, SY14, BZ, DR9, AN, SR24, PR1 (100 µL each stock std except PR1 - 60

Started soxhlets at 3:30 PM on 4/17/2000. Used teflon boiling chips

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TITLE Dionex Extraction Book No.

From Page No. 108

HPLC Results

Organic Dyes

2AQ - no peak
SY14 - 44.288
BZ - 36.572
DR9 - 36.138
AN - ?
SRZ4 - 50.438
PR1 - 40.055

RT's

Acid Dyes

AB45 - 6.415
AR64 - No
AO10 - 19.082
AB9 - 22.232
i 1 - 24.348
BV10 - 36.565

RTs

10x IDL

pen ink added by the NSWC Crane

TEJ

10-2-00

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Group	Weight (g) Soil
1	4.9497
2	5.0176
3	5.0032
4	5.0533

Dye	TLC 10x IDL (µg/mL)	Stock Standard Spike (µL)
Acid Blue 1	6.6	20
Acid Blue 9	32	100
Acid Blue 45	18	100
Acid Orange 10	21	100
Acid Red 64	45	100
Acid Yellow 23	36	100
Acid Yellow 73	27	100
Basic Violet 10	3.0	10
Basic Yellow 2	21	100
Acid Yellow 3	46	100
1-Aminoanthraquinone	35	100
2-Aminoanthraquinone	31	100
Disperse Blue 14	25	100
Disperse Red 9	~~20~~ 19	100
Disperse Violet 1	2.4	10
Pigment Red 1	88	100
Solvent Orange 3	2.3	10
Solvent Orange 7	24	100
Solvent Red 1	3.8	20
Solvent Red 24	7.7	100
Solvent Green 3	4.1	20
Smoke Dye	2.2	10
Solvent Yellow 3	20	100
Solvent Yellow 14	2	10
Solvent Yellow 2	22	100
Solvent Yellow 33	26	100

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TITLE 10X IDC (TLC) Soxhlet Extraction Book No.

From Page No. 110

Procedure

~~Dr~~ Dried frozen dye burial grounds soil sample in an oven at overnight. Ground soil in a mortar and pestle until it would pass through at Number 18 sieve. Stored in a dessicator in an amber bottle until ready for use.

Weighed out approximately 5 grams of dried soil into a cellulose extraction thimble. Spiked on dyes from stock standards in accordance to the volumes listed on page 110 of this notebc Spiked the dyes in the following groups.

1: AB45, AR64, AY3, BY2, AY73, SY3, SY14, SO3, SD, SY2
2: AY23, AO10, BV10, SR1, SO7, 2AQ, DB14
3: AB1, PR1, DR9, DV1
4: AB9, 1AQ, SG3, SY33, SR24

Extracted using soxhlet extraction for 15 hours. Used 200 mL of 5% acetic acid, 40% water, and 55% acetonitrile (10mL acetic acid, 80mL H_2O, 110mL ACN).

Roto-vap samples down to approximately 20mL, solvent exchanged 3ter with methanol (50mL). Last time, brought down to approximately 2mL.

Analyzed on TLC.

4/20/2000 JK

Sample still in acetic acid after 3rd methanol solvent exchange. Blew sample down to dryness under nitrogen at ~ 70°C. Reconstituted with 1mL methanol.
4/24/2000 JK

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TLC Results

File: JK010.JPG

Group	Rf	Dye
1	0.30	SY3
	0.37	SY14
	0.49	SY2
		Missing: SO3, + SD
2	0.16	DB14
	0.49	SO7
		Missing: SR1 + 2AQ
3	0.41	DR9
		Missing: PR1, + DV1
4	0.13	SY33
	0.28	1-AQ
	0.37	SR24
	0.47	SG3

4/24/2000 [illegible]

Diluted ~ 1 to 2 to run on HPLC

HPLC Results

Extraction		ml Spiked	Conc 10x IDL (TLC) in 1 m
1	AY3: 15.443	100	46
	AY73: 18.193	100	27
	BY2: 35.710	100	21
	SY3: 35.460	100	20
	SY2: 40.027	100	22
	AB45: Missing	100	18
	AR64: Missing	100	45
	SY14: Missing	~~2~~ 10	~~10~~ 2
	SO3: Missing	~~2.3~~ 10	~~10~~ 2.3
	SD: Missing	~~2.2~~ 10	~~10~~ 2.2

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Extraction		mL Spiked	Conc 10x IDL (TLC) in 1mL M
2	AY23 : Missing	100	36
	AO10 : 17.727	100	21
	BV10 : Missing	10	3.0
	SR1 : 44.593	20	3.8
	SO7 : 50.943	100	24
	2AQ : Missing	100	31
	DB14 : 32.643	100	25
3	AB1 : 22.985	20	6.6
	PR1 : Missing	100	88
	DR9 : 35.468	100	19
	DV1 : Missing	10	2.4
4	AB9 : 20.745	100	32
	1AQ : 25.778	100	35
	SG3 : 57.828	20	4.1
	SY33 : 27.078	100	26
	SR24 : 58.362	100	7.7

4/25/2000 JK

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Page No.

Spiking Solution

Group #	Dye	Concentration (ppm)
1	Acid Blue 45	7.136
	Acid Red 64	17.936
	Acid Yellow 3	18.432
	Basic Yellow 2	8.584
	Acid Yellow 73	10.688
2	Acid Yellow 23	14.528
	Acid Blue 1	1.988
	Basic Violet 10	1.202
3	Acid Orange 10	8.36
4	Acid Blue 9	12.888

d Well 0201 water as matrix. Spiked ~~3~~ 4 mL water w/ 400 µL 2/17/00 GK
of each dye. Filtered spiked water through 0.45 µm Nylon filter
prior to evaporation. (Osmonics Cat # DGR04T2550, Lot 76922)
Evaporated to dryness under a gentle stream of air.
Note - group 4 clearly visible at this stage.
17/2000 GK

centration After Evaporation & Dilution W/ 1mL MeOH

Group #	Dye	Concentration (ppm ≠ µg/mL)
1	Acid Blue 45	2.854 *
	Acid Red 64	7.174
	Acid Yellow 3	7.373
	Basic Yellow 2	3.434
	Acid Yellow 73	4.275
2	Acid Yellow 23	5.811
	Acid Blue 1	0.795
	Basic Violet 10	0.481
3	Acid Orange 10	3.344
		~~5.155~~
4	Acid Blue 9	5.155

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AB45

$(0.4\,mL)(7.136\,\mu g/mL) = x(1.0\,mL)$

$x = 2.854\,\mu g/mL$

AR64

$(0.4\,mL)(17.936\,\mu g/mL) = x(1.0\,mL)$

$x = 7.174\,\mu g/mL$

$4\overline{)770}$ = 192.5; 4, 37, 36, 10, 8, 2.0

$4\overline{)250}$ = 62.5; 24, 10, 8, 20

$4\overline{)164}$ = 41; 16

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B9

$(X\,mL)(322.2\,\mu g/mL) = (16.0\,\mu g/mL)(1\,mL)$

$X = 49.66\,\mu L$

Spiking Groups

Dyes	Volume (Stock) µL	TLC File	Results
d Blue 45	50		Not detected
d Red 64	50	NK003	Not detected
d Yellow 3	50		Not detected
xc Yellow 2	50		Rf = 0.58
d Yellow 73	50		Rf = 0.76
d Yellow 23	50		
d Orange 10	50		
xc Violet 10	5		
d Blue 1	2		
d Blue 9	50		

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LE Water Solubility Tests Book No.

m Page No. Cross-reference of recoveries.

'01 Totally Water Insoluble

✓PR1 ✓SO7 ✓BZ
✓DR9 ✓DB14 ✓SD
✓SG3 ✓SY14 ✓AN

Missing from solubility test

2AQ
1AQ
SY3
SR1
SY2
SR24

Slightly Water Soluble

✓DV1
✓SO3
✓SY33

Very Water Soluble [Acid Dye]

~~AB1~~

3/17/2000 GK

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Witnessed & Understood by me,	Date	Invented by	Date
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Extraction Date: 11-May-00 Std Inj Vol:10 uL Notebook Pg: 127
Analysis Date: 12-May-00 Sample Inj Vol: 30 uL
Extraction Procedure: 5 mL TBA, 5 mL water sonicate for 0.5 hour. Add 50 mL MeOH. Sonicate for 1 hour.

DYE	STOCK STD ug/mL	SPIKE mL	Conc on Soil mg/kg	CONC IN 60 mL ug/mL	Recovered ug/mL	Correction for Inj Vol (ug/mL)	Amt Rec on Soil mg/kg	Recovery %
ACID BLUE 45	178.4	0	0	0.000	N/A	N/A	N/A	N/A
ACID RED 64	448.4	0	0	0.000	N/A	N/A	N/A	N/A
ACID YELLOW 3	460.8	0	0	0.000	N/A	N/A	N/A	N/A
BASIC YELLOW 2	214.6	0.5	106.71	1.788	22.74	7.58	45.23	423.86
ACID YELLOW 73	267.2	0	0	0.000	N/A	N/A	N/A	N/A
SOLVENT YELLOW 3	197.6	0.5	98.26	1.647		0.00	0.00	0.00
SOLVENT YELLOW 14	199.4	0.5	99.15	1.662	4.119	1.37	8.19	82.63
SOLVENT ORANGE 3	232.4	0.5	115.56	1.937	5.682	1.89	11.30	97.80
SMOKE DYE	346	0.5	172.05	2.883	ND	ND	ND	ND
SOLVENT YELLOW 2	220	0.5	109.40	1.833	?	ND	ND	ND
ACID YELLOW 23	363.2	0	0	0.000	N/A	N/A	N/A	N/A
ACID ORANGE 10	209	0	0	0.000	N/A	N/A	N/A	N/A
BASIC VIOLET 10	300.6	0.5	149.48	2.505	7.867	2.62	15.65	104.68
SOLVENT RED 1	190.6	0.5	94.78	1.588	3.522	1.17	7.01	73.91
SOLVENT ORANGE 7	244	0.5	121.33	2.033		0.00	0.00	0.00
2-AMINOANTHRAQUINONE	356	0.5	177.03	2.967	17.09	5.70	33.99	192.02
DISPERESE BLUE 14	254.8	0.5	126.70	2.123	12.16	4.05	24.19	190.89
ACID BLUE 1	331.4	0	0	0.000	N/A	N/A	N/A	N/A
PIGMENT RED 1	8.8	0	0	0.000	N/A	N/A	N/A	N/A
DISPERSE RED 9	191	0.5	94.98	1.592	4.165	1.39	8.28	87.23
DISPERSE VIOLET 1	237.8	0.5	118.25	1.982	5.402	1.80	10.74	90.87
ACID BLUE 9	322.2	0	0	0.000	N/A	N/A	N/A	N/A
1-AMINOANTHRAQUINONE	354.4	0.5	176.23	2.953	7.527	2.51	14.97	84.95
SOLVENT GREEN 3	207.2	0.5	103.03	1.727	6.095	2.03	12.12	117.66
SOLVENT YELLOW 33	255.6	0.5	127.10	2.130	6.583	2.19	13.09	103.02
SOLVENT RED 24	76.8	0.5	38.19	0.640	ND	ND	ND	ND

peak wrong not calibrate for.

double peak calibrate by hand

Low Level Acid Dyes
Soil

(2)

Extraction Date: 8-May-00 Std Inj Vol: 10 uL Notebook Pg: 122
Analysis Date: 11-May-00 Sample Inj Vol: 30 uL
Extraction Procedure: 5 mL TBA, 5 mL water sonicate for 0.5 hour. Add 50 mL MeOH. Sonicate for 1 hour.

DYE	STOCK STD ug/mL	SPIKE mL	CONC IN 60 mL ug/mL	Conc on Soil mg/kg	Recovered ug/mL	Correction for Inj Vol (ug/mL)	Amt Rec on Soil mg/kg	Recovery %
ACID BLUE 45	178.4	0.1	0.297	1.761	BDL	BDL	BDL	BDL
ACID RED 64	448.4	0.1	0.747	4.426	4.82	1.61	9.52	214.99
ACID YELLOW 3	460.8	0.1	0.768	4.549	2.82	0.94	5.57	122.40
BASIC YELLOW 2	214.6	0	0.000	0.000	N/A	N/A	N/A	N/A
ACID YELLOW 73	267.2	0.1	0.445	2.638	1.201	0.40	2.37	89.90
SOLVENT YELLOW 3	197.6	0	0.000	0.000	N/A	N/A	N/A	N/A
SOLVENT YELLOW 14	199.4	0	0.000	0.000	N/A	N/A	N/A	N/A
SOLVENT ORANGE 3	232.4	0	0.000	0.000	N/A	N/A	N/A	N/A
SMOKE DYE	346	0	0.000	0.000	N/A	N/A	N/A	N/A
SOLVENT YELLOW 2	220	0	0.000	0.000	N/A	N/A	N/A	N/A
ACID YELLOW 23	363.2	0.1	0.605	3.585	2.316	0.77	4.57	127.53
ACID ORANGE 10	209	0.1	0.348	2.063	1.048	0.35	2.07	100.29
BASIC VIOLET 10	300.6	0	0.000	0.000	N/A	N/A	N/A	N/A
SOLVENT RED 1	190.6	0	0.000	0.000	N/A	N/A	N/A	N/A
SOLVENT ORANGE 7	244	0	0.000	0.000	N/A	N/A	N/A	N/A
2-AMINOANTHRAQUINONE	356	0	0.000	0.000	N/A	N/A	N/A	N/A
DISPERESE BLUE 14	254.8	0	0.000	0.000	N/A	N/A	N/A	N/A
ACID BLUE 1	331.4	0.1	0.552	3.271	1.439	0.48	2.84	86.84
PIGMENT RED 1	8.8	0.0	0.000	0.000	N/A	N/A	N/A	N/A
DISPERSE RED 9	191	0	0.000	0.000	N/A	N/A	N/A	N/A
DISPERSE VIOLET 1	237.8	0	0.000	0.000	N/A	N/A	N/A	N/A
ACID BLUE 9	322.2	0.1	0.537	3.180	1.29	0.43	2.55	80.07
1-AMINOANTHRAQUINONE	354.4	0	0.000	0.000	N/A	N/A	N/A	N/A
SOLVENT GREEN 3	207.2	0	0.000	0.000	N/A	N/A	N/A	N/A
SOLVENT YELLOW 33	255.6	0	0.000	0.000	N/A	N/A	N/A	N/A
SOLVENT RED 24	76.8	0	0.000	0.000	N/A	N/A	N/A	N/A

Hi level O-d dyes

①

Extraction Date: 4-May-00 Std Inj Vol: 10 uL Notebook Pg: 121
Analysis Date: 11-May-00 Sample Inj Vol: 30 uL
Extraction Procedure: 5 mL TBA, 5 mL water sonicate for 0.5 hour. Add 50 mL MeOH. Sonicate for 1 hour.

dried soil after spiking stds

DYE	STOCK STD ug/mL	SPIKE mL	CONC IN 60 mL ug/mL	Conc on Soil mg/kg	Recovered ug/mL	Correction for Inj Vol (ug/mL)	Amt Rec on Soil mg/kg	Recovery %
ACID BLUE 45	178.4	0.5	1.487	8.807	2.222	0.74	4.39	49.82
ACID RED 64	448.4	0.5	3.737	22.136	12.67	4.22	25.02	113.02
ACID YELLOW 3	460.8	0.5	3.840	22.748	12.06	4.02	23.81	104.69
BASIC YELLOW 2	214.6	0	0.000	0.000	N/A	N/A	N/A	N/A
ACID YELLOW 73	267.2	0.5	2.227	13.191	5.884	1.96	11.62	88.08
SOLVENT YELLOW 3	197.6	0	0.000	0.000	N/A	N/A	N/A	N/A
SOLVENT YELLOW 14	199.4	0	0.000	0.000	N/A	N/A	N/A	N/A
SOLVENT ORANGE 3	232.4	0	0.000	0.000	N/A	N/A	N/A	N/A
SMOKE DYE	346	0	0.000	0.000	N/A	N/A	N/A	N/A
SOLVENT YELLOW 2	220	0	0.000	0.000	N/A	N/A	N/A	N/A
ACID YELLOW 23	363.2	0.5	3.027	17.930	8.723	2.91	17.23	96.07
ACID ORANGE 10	209	0.5	1.742	10.318	4.821	1.61	9.52	92.27
BASIC VIOLET 10	300.6	0	0.000	0.000	N/A	N/A	N/A	N/A
SOLVENT RED 1	190.6	0	0.000	0.000	N/A	N/A	N/A	N/A
SOLVENT ORANGE 7	244	0	0.000	0.000	N/A	N/A	N/A	N/A
2-AMINOANTHRAQUINONE	356	0	0.000	0.000	N/A	N/A	N/A	N/A
DISPERESE BLUE 14	254.8	0	0.000	0.000	N/A	N/A	N/A	N/A
ACID BLUE 1	331.4	0.5	2.762	16.360	7.508	2.50	14.83	90.62
PIGMENT RED 1	8.8	0.0	0.000	0.000	N/A	N/A	N/A	N/A
DISPERSE RED 9	191	0	0.000	0.000	N/A	N/A	N/A	N/A
DISPERSE VIOLET 1	237.8	0	0.000	0.000	N/A	N/A	N/A	N/A
ACID BLUE 9	322.2	0.5	2.685	15.906	4.03	1.34	7.96	50.03
1-AMINOANTHRAQUINONE	354.4	0	0.000	0.000	N/A	N/A	N/A	N/A
SOLVENT GREEN 3	207.2	0	0.000	0.000	N/A	N/A	N/A	N/A
SOLVENT YELLOW 33	255.6	0	0.000	0.000	N/A	N/A	N/A	N/A
SOLVENT RED 24	76.8	0	0.000	0.000	N/A	N/A	N/A	N/A

High level

#

Extraction Date: 3-May-00 Std Inj Vol: 10 uL Notebook Pg: 119

Analysis Date: 6-May-00 Sample Inj Vol: 30 uL

Extraction Procedure: 100 mL of 60/40 Acetonitrile/Water sonicated for 1.5 hours (Dried soil after spiking dyes.) — given to make sure stds on sats N_2 blowdown

DYE	STOCK STD ug/mL	SPIKE mL	CONC IN 100 mL ug/mL	Conc on Soil mg/kg	Recovered ug/mL	Amt Rec on Soil mg/kg	Recovery %
ACID BLUE 45	178.4	0.5	0.892	8.410	BDL	BDL	BDL
ACID RED 64	448.4	0.5	2.242	21.138	BDL	BDL	BDL
ACID YELLOW 3	460.8	0.5	2.304	21.722	1.69	15.93	73.35
BASIC YELLOW 2	214.6	0.5	1.073	10.116	BDL	BDL	BDL
ACID YELLOW 73	267.2	0.5	1.336	12.596	0.687	6.48	51.42
SOLVENT YELLOW 3	197.6	0.5	0.988	9.315	0.619	5.84	62.65
SOLVENT YELLOW 14	199.4	0.5	0.997	9.400	0.487	4.59	48.85
SOLVENT ORANGE 3	232.4	0.5	1.162	10.955	0.119	1.12	10.24
SMOKE DYE	346	0.5	1.730	16.310	BDL	BDL	BDL
SOLVENT YELLOW 2	220	0.5	1.100	10.371	0.527	4.97	47.91
ACID YELLOW 23	363.2	0.5	1.816	18.004	0.609	6.04	33.54
ACID ORANGE 10	209	0.5	1.045	10.360	0.366	3.63	35.02
BASIC VIOLET 10	300.6	0.5	1.503	14.901	BDL	BDL	BDL
SOLVENT RED 1	190.6	0.5	0.953	9.448	0.249	2.47	26.13
SOLVENT ORANGE 7	244	0.5	1.220	12.095	0.552	5.47	45.25
2-AMINOANTHRAQUINONE	356	0.5	1.780	17.647	BDL	BDL	BDL
DISPERESE BLUE 14	254.8	0.5	1.274	12.630	0.65	6.44	51.02
ACID BLUE 1	331.4	0.5	1.657	16.194	0.664	6.49	40.07
PIGMENT RED 1	8.8	5.0	0.440	4.300	BDL	BDL	BDL
DISPERSE RED 9	191	0.5	0.955	9.333	BDL	BDL	BDL
DISPERSE VIOLET 1	237.8	0.5	1.189	11.620	BDL	BDL	BDL
ACID BLUE 9	322.2	0.5	1.611	16.017	BDL	BDL	BDL
1-AMINOANTHRAQUINONE	354.4	0.5	1.772	17.618	BDL	BDL	BDL
SOLVENT GREEN 3	207.2	0.5	1.036	10.300	1.493	14.84	144.11
SOLVENT YELLOW 33	255.6	0.5	1.278	12.706	0.817	8.12	63.93
SOLVENT RED 24	76.8	0.5	0.384	3.818	BDL	BDL	BDL

after this consulted Dr. Bauchman suggested higher concentration TL

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TLC Results

Organic Dyes

Column	Rf	Possible Dyes
H_2O Filtrate	0.73	SY14, SY2, SO7, SG3, SR24
	0.77	SY2, SG3, A
60/40 ACN/H_2O	0.22	SO3, 2-AQ, DV1
80/20	0.21	SO3, 2-AQ, DV1
	0.40	~~SY3,~~ ?
	0.48	SY3, 1-AQ, DB14
	0.54	SY3, SR1, 1-AQ, DB14
	0.59	~~G~~. SR1, PR1,
	0.73	SY14, SY2, SO7, SG3, SR24
	0.76	SY2, SG3, A
ACN	0.54	SY3, SR1, 1-AQ, DB14
	0.58	SR1, PR1
	0.69	SY14, SY2, SO7, DR9, SR24
Ethyl Acetate	0.54	SY3, SR1, 1-AQ, DB14
	0.58	SR1, PR1
	0.69	SY14, SY2, SO7, DR9, SR24
	0.75	SY2, SG3, A

Water Dyes

Water-60/40	0.34	AY3,
	0.54	BV10, BY2,
Water-80/20	0.50	BV10, BY2
	0.53	
TBA-Filtrate	none	
TBA-60/40	0.03	AR64
	0.23	AY23
	0.28	AY3
	0.31	AY3, AB1, AB9, AO10
	0.50	BV10, BY2,

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Results

Inner - TBA

TB45	11.871 min
AY3	16.129 min
Y23	17.133 min
Y73	18.583 min
TB1	24.817 min
B9	P1 - 22.498 P2 - 22.884 P3 - 23.230
O10	20.233
R64	16.183 (coelution)
SY10	36.46
SY2	35

Organic - Mixed fractions

2-AQ	not sure → ~~27.2~~ ~~26.9 (coelutes w/ SY33)~~
SR	44.2
-AQ	26.823 (~~possible coelution~~ interference) w/ SY33)
SY3	34.907
DB14	32.723
SD	not sure
SO3	22.973
SG3	57.623
SZ	36.073
SR9	35.446
SY2	not sure. 39.607 (interference w/ AN)
SV1	20.140
O7	51.040 (Problems w/ SR1)
Y33	26.940 (~~possible coelution~~ interference) w/ 1-AQ)
R24	not sure
R1	not sure
N	39.690 (possible coelution)
Y14	43.573

SR1 + SO7 have very similar spectra. Computer can not tell the difference.

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Diluted 1mL organic dye mix in 200mL Hi-Q water
Diluted 1mL water dye mix in 200mL Hi-Q water
Diluted 1mL water dye + 1mL reconstituted FBA (0.005M) in 200mL Hi-Q water
1M before dilution after dilution
Waters Sep-Pak Vac 6cc C18 cartridge

Conditioned w/ 25mL MeOH then ~~50~~ 20mL Hi-Q H_2O (time constraints)
Pulled thru 200mL spike H_2O sample (organic used syringe to pull thru faster)
~~10mL 60/40 ACN/H2O~~

Organic

1st extraction: 10mL 60-40 acetonitrile-water: red color to solvent. Dyes still present on cartridge
2nd extraction: 10mL 80-20 acetonitrile-water - yellow tint to solvent. Still see color on cartridge
3rd extraction: 10mL acetonitrile - yellow tint to solvent. Still see faint yellow tint on cartridge
4th extraction: 20mL ethyl acetate - yellow tint to solvent. Most yellow tint to cartridge appears to be gone

Water - TBA
1st extraction: 15mL 60-40 acetonitrile-water. Dirty purple color to solvent. All color appears to be removed from cartridge

Water

Appears yellow dye broke through. Will analyze water collected after passing thru cartridge to determine dye.

1st extraction - 60-40 ACN-water. Dirty purple color to water. May be slight yellow tinge left on cartridge

2nd extraction - 80-20 ACN-water. Slight yellow tint to solvent.

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TLC Results

Hexane ⇒ JK001
Could see...
DV1 ⇒ Rf = 0.21
SY2 ⇒ Rf = 0.64 (slightly low)

Butanol JK002
Could see...
BV10 ⇒ Rf = 0.516 (slightly low)
SY2 ⇒ Rf = 0.72 (did not expect to move w/mobile phase)
Note: Made new mobile phase solution 200 (BuOH) : 50 H_2O : 50 Acetic Acid
Could not see AB1
03/03/2000 JK

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iked 770 mL of well water with the following dyes

ye	Stock Std Conc	Volume Spiked (uL)	Concentration After Dilution in 1mL MeOH
MC	300.6 ppm	10	3.006 ppm
B1	331.4 ppm	10	3.314 ppm
1	237.8 ppm	10	2.378 ppm
2	220.0 ppm	50	11.00 ppm

11.0 PROCEDURE

For aqueous solutions, it is highly recommended that process waste samples be screened with the high-level method to determine if the low-level method (1 to 50 microg/L) is required; the high-level method will not be necessary in most cases.

For samples requiring the low-level method, there are three equally acceptable options: salting-out extraction (Section 11.1), cartridge solid-phase extraction (Section 11.2), or membrane solid-phase extraction (Section 11.3).

11.1 Aqueous Samples, Low-Level Method No. 1: Salting-out extraction

HPLC

11.1.7 - notable to get all 84mL in volumetric

11.1.16 Vt = 20.1mL

TLC

Unable to see separation of phases due to high level of sediment present in sample. Guessed as to where phases might be.

11.1.1 Place 251.3 g of sodium chloride into a 1-L round volumetric flask.

11.1.2 Using a 1-L graduated cylinder, measure out 770 mL of a water sample and transfer it to the volumetric flask containing the sodium chloride. Add a stir bar and mix the contents at maximum speed on a magnetic stirrer until the salt is completely dissolved.

11.1.3 Add 164 mL of acetonitrile (measured with a 250-mL graduated cylinder) while the solution is being stirred, and stir for an additional 15 minutes. Turn off the stirrer and allow the phases to separate for 10 minutes.

11.1.4 Remove the acetonitrile (upper) layer with a Pasteur pipette, approximately 8 mL, and transfer it to a 100-mL round volumetric flask.

11.1.5 Add 10 mL of fresh acetonitrile to the water sample in the 1-L flask. Again, stir the contents of the flask for 15 minutes followed by 10 minutes for phase separation.

11.1.6 Combine the second acetonitrile portion with the initial extract in the 100-mL flask. The inclusion of a few drops of salt water at this point is unimportant.

11.1.7 Add 84 mL of salt water (325 g NaCl per 1,000 mL of reagent water) to the acetonitrile extract in the 100-mL volumetric flask. Add a stir bar and stir the contents on a magnetic stirrer for 10 minutes, followed by 10 minutes of phase separation.
15?

11.1.8 Carefully transfer the acetonitrile phase to a 10-mL graduated cylinder using a Pasteur pipette. At this stage, the amount of water transferred with the acetonitrile must be minimized; the water contains a high concentration of NaCl that produces a large peak at the beginning of the chromatogram, ~~where it could interfere with the HMX determination~~.

11.1.9 Add an additional 1.0 mL of acetonitrile to the 100-mL volumetric flask. Once again, stir the contents of the flask for 15 minutes, followed by 10 minutes of phase separation.

11.1.10 Combine the second acetonitrile portion with the initial extract in the 10-mL graduated cylinder (transfer to a 25-mL graduated cylinder if the volume exceeds 5 mL). Record the total volume of acetonitrile extract to the nearest 0.1 mL; use this as the volume of total extract [V_t] in the calculation of concentration after converting it to microL.

11.1.11 Evaporated to dryness then diluted w/ 1 mL methanol

~~**11.1.11** The resulting extract, about 5 to 6 mL, is then diluted 1:1 with organic-free reagent water (with pH < 3 if tetryl is a suspected analyte) prior to analysis.~~

11.1.12 If the diluted extract is turbid, filter it through a 0.45-microm ~~Teflon~~ Anotop filter using a disposable syringe. Discard the first 0.5 mL of filtrate, and retain the remainder in a Teflon-capped ~~vial for RP-HPLC analysis (Section 12)~~.

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ATTACHMENT VII

METHOD VALIDATION

ATTACHMENT VII – METHOD VALIDATION

The Crane Laboratory performed a method validation study using the following procedures:

Aqueous	Low-level Extraction Procedure	High Spike	Long Gradient	Short Gradient
		Low Spike	Long Gradient	Short Gradient
Aqueous	High-level Extraction Procedure	High Spike	Long Gradient	Short Gradient
		Low Spike	Long Gradient	Short Gradient
Solids	Extraction Procedure	High Spike	Long Gradient	Short Gradient
		Low Spike	Long Gradient	Short Gradient

The laboratory used the same sample extract on both the short and long gradient sequences. Stock standards for each individual target compound were made. Samples designated low spike had 100 µL of each stock standard added to the sample matrix prior to extraction using an Ependorf pipet. Samples designated "high spike" had 500 µL of each stock standard added to the sample matrix prior to extraction using the same technique. Due to the number of spikes required, this method of adding compounds to the samples is more prone to errors such as missed spiking or double-spiking of target compounds. Instances where these situations may have occurred have been noted in the summaries below.

Tables 1 through 4 give an overview of the percent recoveries for the Low-level aqueous extraction procedure and the Solids extraction procedure. Results for the compound Basic Yellow 2 were consistently high regardless of matrix or analytical method. This may indicate a problem with the spiking solution.

Percent Recoveries Low-Level Aqueous Method High Spike

As stated previously recovery of Basic Yellow 2 was consistently high. Results for 2-Aminoanthraquinone were taken from the short gradient analysis due to co-elution with Acid Blue 1 using the long gradient method. Recovery of Acid Blue 45 was less than 10% in five of the seven standards. Results of sample WLM004HS were not used to calculate accuracy or method detection limits due to problems encountered during extraction.

Percent Recoveries Low-Level Aqueous Method Low Spike

As stated previously, recovery of Basic Yellow 2 was consistently high. Two samples (WLM004LS and WLM008LS) had recoveries for Basic Yellow 2 in excess of 1000%. These samples may have been double-spiked for this compound. Results for 2-Aminoanthraquinone were taken from the short gradient analysis due to co-elution with Acid Blue 1 using the long gradient method. Recovery of Solvent Green 3 and Solvent Red 24 were 0% in sample WLM005LS. This may indicate that these compounds were not spiked into the sample since the remaining six samples have significantly higher recoveries. Recovery of the surrogate Anthracene was less than 10% in sample WLM004LS. This may indicate that this compound was not spiked into the sample. Recovery of Acid Blue 45 was improved over the recoveries reported for the high spike. This may be a function of the solubility of the compound.

Percent Recoveries Soil Method High Spike

As stated previously, recovery of Basic Yellow 2 was consistently high. Results for 2-aminoanthraquinone and Acid Blue 1 were taken from the short gradient analysis due to co-elution using the long gradient method. Recoveries of Acid Yellow 3, Acid Blue 9, Acid Blue 1, and Solvent Red 24 were less than 10% on both the long and short gradient analyses.

Percent Recoveries Soil Method Low Spike

As stated previously, recovery of Basic Yellow 2 was consistently high. Results for 2-aminoanthraquinone and Acid Blue 1 were taken from the short gradient analysis due to co-elution using the long gradient method. Recoveries of Acid Blue 9 and Solvent Red 24 were less than 10% on both the long and short gradient analyses. Acid Yellow 3 and Disperse Violet 1 were not recovered in sample S007LS. This may be an indication that the compounds were not spiked onto the sample since the remaining six samples have significantly higher results.

The remaining percent recovery tables depict the percent recoveries for each method and each gradient. Retention times have been included and the compounds are presented in elution order to highlight possible co-elution/interference problems.

The remainder of this section includes calculation of mean percent recoveries, standard deviations, method detection limits, and reliable quantitation limits. Excel tables presenting the laboratory calculations from the raw data are given with each table to support the findings.

Tables Contained in Attachment VII

Table VII - 1	Solid Extraction Method, Long Gradient, Low Spike – Bias and Precision
Table VII - 1A	Solid Extraction Method, Long Gradient, Low Spike – Quality Control Limits
Table VII - 1B	Solid Extraction Method, Long Gradient, Low Spike – Percent Recovery
Table VII - 2	Solid Extraction Method, Long Gradient, High Spike – Bias and Precision
Table VII - 2A	Solid Extraction Method, Long Gradient, High Spike – Percent Recovery
Table VII - 3	Solid Extraction Method, Short Gradient, Low Spike – Bias and Precision
Table VII - 3A	Solid Extraction Method, Short Gradient, Low Spike – Quality Control Limits
Table VII - 3B	Solid Extraction Method, Short Gradient, Low Spike – Percent Recovery
Table VII - 4	Solid Extraction Method, Short Gradient, High Spike – Bias and Precision
Table VII - 4A	Solid Extraction Method, Short Gradient, High Spike – Percent Recovery
Table VII - 5	Low-Level Aqueous Extraction Method, Long Gradient, Low Spike – Bias and Precision
Table VII - 5A	Low-Level Aqueous Extraction Method, Long Gradient, Low Spike – Quality Control Limits
Table VII - 5B	Low-Level Aqueous Extraction Method, Long Gradient, Low Spike – Percent Recovery
Table VII - 6	Low-Level Aqueous Extraction Method, Long Gradient, High Spike – Bias and Precision
Table VII - 6A	Low-Level Aqueous Extraction Method, Long Gradient, High Spike – Percent Recovery
Table VII - 7	Low-Level Aqueous Extraction Method, Short Gradient, Low Spike – Bias and Precision
Table VII - 7A	Low-Level Aqueous Extraction Method, Short Gradient, Low Spike – Quality Control Limits
Table VII - 7B	Low-Level Aqueous Extraction Method, Short Gradient, Low Spike – Percent Recovery
Table VII - 8	Low-Level Aqueous Extraction Method, Short Gradient, High Spike – Bias and Precision
Table VII - 8A	Low-Level Aqueous Extraction Method, Short Gradient, High Spike – Percent Recovery
Table VII - 9	High Level Aqueous Extraction Method, Long Gradient, Low Spike – Bias and Precision
Table VII - 9A	High Level Aqueous Extraction Method, Long Gradient, Low Spike – Percent Recovery
Table VII - 10	High Level Aqueous Extraction Method, Long Gradient, High Spike – Bias and Precision
Table VII - 10A	High Level Aqueous Extraction Method, Long Gradient, High Spike – Percent Recovery
Table VII - 11	High Level Aqueous Extraction Method, Short Gradient, Low Spike – Bias and Precision
Table VII - 11A	High Level Aqueous Extraction Method, Short Gradient, Low Spike – Percent Recovery
Table VII - 12	High Level Aqueous Extraction Method, Short Gradient, High Spike – Bias and Precision
Table VII - 12A	High Level Aqueous Extraction Method, Short Gradient, High Spike – Percent Recovery

TABLE VII-1

Solid Extraction Method, Long Gradient, Low Spike

		Sample Results (µg/Kg)							Mean Result µg/Kg	Method Bias (Accuracy) %R	Standard Deviation	Method Precision (%RSD)	MDL µg/Kg	RQL µg/Kg
Compounds	Sample Conc µg/Kg	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS	average sample result	(mean result/sample conc)*100	std. dev. sample results	(std.dev/mean result)*100	std.dev. *3.14	MDL*4
ACID BLUE 45	3444	1196.7	1089.1	1232.3	1217.6	1172.0	900.3	973.8	1111.7	32.3	129.6	11.7	407.1	1628.4
ACID YELLOW 23	2812	1500.8	1656.1	1507.9	1564.1	1490.6	1440.6	1918.9	1582.7	56.3	163.1	10.3	512.3	2049.1
ACID YELLOW 73	3510	2033.3	2154.7	2143.6	2038.3	2126.9	2080.7	2035.4	2087.6	59.5	53.8	2.6	168.8	675.2
ACID ORANGE 10	3074	3289.4	3414.0	3345.9	3167.1	3325.3	3239.7	3223.9	3286.5	106.9	83.3	2.5	261.7	1046.8
ACID BLUE 1	3692	416.6	445.2	295.6	342.0	370.4	515.9	0.0	340.8	9.2	166.5	48.9	522.9	2091.5
BASIC YELLOW 2	2642	12021.3	13142.5	13197.6	12468.7	12943.1	12464.7	12810.3	12721.2	481.5	424.5	3.3	1332.8	5331.3
DISPERSE VIOLET 1	2602	627.1	885.9	725.5	781.3	863.8	681.9	0.0	652.2	25.1	302.3	46.4	949.2	3797.0
SOLVENT YELLOW 33	2796	1220.5	1346.6	1350.2	1303.0	1329.3	1311.5	1319.4	1311.5	46.9	43.7	3.3	137.3	549.2
DISPERSE BLUE 14	2990	1131.8	1275.3	1329.2	1445.8	1494.1	1370.3	1445.3	1356.0	45.4	124.4	9.2	390.6	1562.2
ACID RED 64	3618	2721.8	2951.3	2877.1	2672.4	2851.8	2910.2	2899.6	2840.6	78.5	103.7	3.6	325.5	1302.0
ACID BLUE 9	3280	97.1	117.3	103.4	77.9	67.7	36.9	55.0	79.3	2.4	28.5	35.9	89.4	357.6
BASIC VIOLET 10	2084	1434.9	1511.9	1455.0	1366.4	1435.9	1390.8	1429.0	1432.0	68.7	46.4	3.2	145.8	583.4
2-AMINOANTHRAQUINONE	3738	2148.7	2189.2	2271.4	2169.1	2257.8	2165.4	2052.2	2179.1	58.3	73.2	3.4	229.8	919.1
SOLVENT ORANGE 3	2558	1065.9	1083.1	1081.0	1069.3	1109.8	1063.3	1055.1	1075.4	42.0	18.1	1.7	56.7	226.8
1-AMINOANTHRAQUINONE	2570	1289.8	1484.9	1532.9	1465.7	1472.2	1412.2	1453.8	1444.5	56.2	77.2	5.3	242.3	969.2
SOLVENT YELLOW 14	2832	1178.4	1207.4	1236.3	1167.2	1242.7	1239.2	1209.3	1211.5	42.8	30.1	2.5	94.6	378.3
SOLVENT ORANGE 7	2880	1494.9	1636.6	1658.2	1600.0	1652.4	1595.7	1631.3	1609.9	55.9	56.1	3.5	176.0	704.0
ACID YELLOW 3	3348	1659.8	1772.9	1699.7	1690.9	1728.6	1682.4	0.0	1462.0	43.7	645.7	44.2	2027.6	8110.3
SOLVENT YELLOW 3	3020	2772.3	2929.9	2954.5	2850.6	2931.0	2902.2	2884.7	2889.3	95.7	61.9	2.1	194.5	777.8
SOLVENT RED 1	4638	2564.7	2678.3	3741.4	4441.1	2877.7	2627.0	2592.2	3074.6	66.3	730.4	23.8	2293.4	9173.7
DISPERSE RED 9	3060	1248.7	1346.1	1408.1	1294.5	1310.4	1371.3	1385.3	1337.8	43.7	56.2	4.2	176.5	706.1
SOLVENT YELLOW 2	3940	1604.3	1789.4	1790.1	1734.8	1820.2	1753.7	1760.7	1750.5	44.4	70.3	4.0	220.8	883.1
SOLVENT GREEN 3	3140	1793.0	1550.3	1765.1	1688.9	1463.7	1519.9	1145.4	1560.9	49.7	222.1	14.2	697.3	2789.1
SOLVENT RED 24	568	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ANTHRACENE	4154	1694.0	1904.2	1912.4	1860.6	1893.9	1861.4	1884.7	1858.7	44.7	75.3	4.1	236.4	945.6
BENZANTHRONE	2432	1507.2	1568.8	1557.4	1554.1	1593.7	1555.3	1572.3	1558.4	64.1	26.4	1.7	83.0	331.9

TABLE VII - 1A

PERCENT RECOVERIES SOIL METHOD LOW SPIKE, LONG GRADIENT

Compounds		Ret. Time	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS	Average (p)	Std. Deviation (s)	Lower Control Limit (p-2s)	Upper Control Limit (p+2s)	Average (on six points)	Std. Dev (s) (on six points)	Lower Control Limit (p-2s)	Upper Control Limit (p+2s)
% RECOVERY AB45	%	6.439	35	32	36	35	34	26	29	32	4	25	40				
% RECOVERY AY3	%	13.372	50	53	51	51	52	50	0	44	19	5	83	51	1	49	53
% RECOVERY AY23	%	14.513	54	59	54	56	53	51	69	57	6	45	68				
% RECOVERY AR64	%	14.9	76	82	80	74	79	81	81	79	3	73	85				
% RECOVERY AY73	%	17.353	58	61	61	58	61	59	58	60	1	57	63				
% RECOVERY AO10	%	18.752	108	111	109	103	109	106	106	107	3	102	113				
% RECOVERY AB9	%	21.657	3	4	3	2	2	1	2	2	1	1	4				
% RECOVERY DV1	%	22.77	24	34	28	30	33	26	0	25	12	2	48	29	4	22	37
% RECOVERY AB1	%	23.733	11	12	8	9	10	14	0	9	5	0	18	11	2	7	15
% RECOVERY 2AQ	%	23.758	58	59	61	58	61	58	55	59	2	0	62				
% RECOVERY SO3	%	24.783	42	42	42	42	44	42	42	42	1	41	44				
% RECOVERY SY33	%	28.259	44	48	48	47	48	47	48	47	1	44	50				
% RECOVERY 1AQ	%	28.853	51	58	60	57	58	55	57	56	3	51	62				
% RECOVERY DB14	%	34.023	38	43	45	48	50	46	49	46	4	37	54				
% RECOVERY BY2	%	35.367	459	498	500	473	492	473	489	484	15	0	514				
% RECOVERY SY3	%	35.998	93	97	98	95	97	96	96	96	2	92	100				
% RECOVERY BV10	%	36.018	70	73	70	66	69	67	69	69	2	65	73				
% RECOVERY DR9	%	36.142	41	44	46	42	43	45	46	44	2	40	48				
% RECOVERY BZ	%	36.724	63	65	64	64	66	64	65	64	1	61	67				
% RECOVERY SY2	%	39.91	41	45	45	44	46	45	45	45	2	41	48				
% RECOVERY AN	%	40.641	41	46	46	45	46	45	46	45	2	0	50				
% RECOVERY SY14	%	44.36	42	43	44	41	44	44	43	43	1	41	45				
% RECOVERY SR1	%	44.811	56	58	81	96	62	57	56	67	16	35	98				
% RECOVERY SO7	%	50.894	52	57	58	56	58	56	57	56	2	52	60				
% RECOVERY SG3	%	57.448	58	49	56	54	47	49	37	50	7	0	64				
% RECOVERY SR24	%	57.994	0	0	0	0	0	0	0	0	0	0	0				

Table VII - 1B

Percent Recoveries Soil Method Low Spike, Long Gradient

Compounds		Ret. Time	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
% RECOVERY AB45	%	6.439	35	32	36	35	34	26	29
% RECOVERY AY3	%	13.372	50	53	51	51	52	50	0
% RECOVERY AY23	%	14.513	54	59	54	56	53	51	69
% RECOVERY AR64	%	14.9	76	82	80	74	79	81	81
% RECOVERY AY73	%	17.353	58	61	61	58	61	59	58
% RECOVERY AO10	%	18.752	108	111	109	103	109	106	106
% RECOVERY AB9	%	21.657	3	4	3	2	2	1	2
% RECOVERY DV1	%	22.77	24	34	28	30	33	26	0
% RECOVERY AB1	%	23.733	11	12	8	9	10	14	0
% RECOVERY 2AQ	%	23.758	58	59	61	58	61	58	55
% RECOVERY SO3	%	24.783	42	42	42	42	44	42	42
% RECOVERY SY33	%	28.259	44	48	48	47	48	47	48
% RECOVERY 1AQ	%	28.853	51	58	60	57	58	55	57
% RECOVERY DB14	%	34.023	38	43	45	48	50	46	49
% RECOVERY BY2	%	35.367	459	498	500	473	492	473	489
% RECOVERY SY3	%	35.998	93	97	98	95	97	96	96
% RECOVERY BV10	%	36.018	70	73	70	66	69	67	69
% RECOVERY DR9	%	36.142	41	44	46	42	43	45	46
% RECOVERY BZ	%	36.724	63	65	64	64	66	64	65
% RECOVERY SY2	%	39.91	41	45	45	44	46	45	45
% RECOVERY AN	%	40.641	41	46	46	45	46	45	46
% RECOVERY SY14	%	44.36	42	43	44	41	44	44	43
% RECOVERY SR1	%	44.811	56	58	81	96	62	57	56
% RECOVERY SO7	%	50.894	52	57	58	56	58	56	57
% RECOVERY SG3	%	57.448	58	49	56	54	47	49	37
% RECOVERY SR24	%	57.994	0	0	0	0	0	0	0

Percent Recovery

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
	Stock Standard Concentrations						
ACID BLUE 45 STD	344.4	344.4	344.4	344.4	344.4	344.4	344.4
ACID YELLOW 23 STD	281.2	281.2	281.2	281.2	281.2	281.2	281.2
ACID YELLOW 73 STD	351.0	351.0	351.0	351.0	351.0	351.0	351.0
ACID ORANGE 10 STD	307.4	307.4	307.4	307.4	307.4	307.4	307.4
ACID BLUE 1 STD	369.2	369.2	369.2	369.2	369.2	369.2	369.2
BASIC YELLOW 2 STD	264.2	264.2	264.2	264.2	264.2	264.2	264.2
DISPERSE VIOLET 1 STD	260.2	260.2	260.2	260.2	260.2	260.2	260.2
SOLVENT YELLOW 33 STD	279.6	279.6	279.6	279.6	279.6	279.6	279.6
DISPERSE BLUE 14 STD	299.0	299.0	299.0	299.0	299.0	299.0	299.0
ACID RED 64 STD	361.8	361.8	361.8	361.8	361.8	361.8	361.8
ACID BLUE 9 STD	328.0	328.0	328.0	328.0	328.0	328.0	328.0
BASIC VIOLET 10 STD	208.4	208.4	208.4	208.4	208.4	208.4	208.4
2-AMINOANTHRAQUINONE STD	373.8	373.8	373.8	373.8	373.8	373.8	373.8
SOLVENT ORANGE 3 STD	255.8	255.8	255.8	255.8	255.8	255.8	255.8
1-AMINOANTHRAQUINONE STD	257.0	257.0	257.0	257.0	257.0	257.0	257.0
SOLVENT YELLOW 14 STD	283.2	283.2	283.2	283.2	283.2	283.2	283.2
SOLVENT ORANGE 7 STD	288.0	288.0	288.0	288.0	288.0	288.0	288.0
ACID YELLOW 3 STD	334.8	334.8	334.8	334.8	334.8	334.8	334.8
SOLVENT YELLOW 3 STD	302.0	302.0	302.0	302.0	302.0	302.0	302.0
SOLVENT RED 1 STD	463.8	463.8	463.8	463.8	463.8	463.8	463.8
DISPERSE RED 9 STD	306.0	306.0	306.0	306.0	306.0	306.0	306.0
SOLVENT YELLOW 2 STD	394.0	394.0	394.0	394.0	394.0	394.0	394.0
SOLVENT GREEN 3 STD	314.0	314.0	314.0	314.0	314.0	314.0	314.0
SOLVENT RED 24 STD	56.8	56.8	56.8	56.8	56.8	56.8	56.8
ANTHRACENE	415.4	415.4	415.4	415.4	415.4	415.4	415.4
BENZANTHRONE	243.2	243.2	243.2	243.2	243.2	243.2	243.2

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
	Weight of Stock Standard Spiked						
SPIKE VOLUME EACH DYE	100.0	100.0	100.0	100.0	100.0	100.0	100.0
AB45 SPIKE WEIGHT	34.4	34.4	34.4	34.4	34.4	34.4	34.4
AY23 SPIKE WEIGHT	28.1	28.1	28.1	28.1	28.1	28.1	28.1
AY73 SPIKE WEIGHT	35.1	35.1	35.1	35.1	35.1	35.1	35.1
AO10 SPIKE WEIGHT	30.7	30.7	30.7	30.7	30.7	30.7	30.7
AB1 SPIKE WEIGHT	36.9	36.9	36.9	36.9	36.9	36.9	36.9
BY2 SPIKE WEIGHT	26.4	26.4	26.4	26.4	26.4	26.4	26.4
DV1 SPIKE WEIGHT	26.0	26.0	26.0	26.0	26.0	26.0	26.0
SY33 SPIKE WEIGHT	28.0	28.0	28.0	28.0	28.0	28.0	28.0
DB14 SPIKE WEIGHT	29.9	29.9	29.9	29.9	29.9	29.9	29.9
AR64 SPIKE WEIGHT	36.2	36.2	36.2	36.2	36.2	36.2	36.2
AB9 SPIKE WEIGHT	32.8	32.8	32.8	32.8	32.8	32.8	32.8
BV10 SPIKE WEIGHT	20.8	20.8	20.8	20.8	20.8	20.8	20.8
2AQ SPIKE WEIGHT	37.4	37.4	37.4	37.4	37.4	37.4	37.4
SO3 SPIKE WEIGHT	25.6	25.6	25.6	25.6	25.6	25.6	25.6
1AQ SPIKE WEIGHT	25.7	25.7	25.7	25.7	25.7	25.7	25.7
SY14 SPIKE WEIGHT	28.3	28.3	28.3	28.3	28.3	28.3	28.3
SO7 SPIKE WEIGHT	28.8	28.8	28.8	28.8	28.8	28.8	28.8
AY3 SPIKE WEIGHT	33.5	33.5	33.5	33.5	33.5	33.5	33.5
SY3 SPIKE WEIGHT	30.2	30.2	30.2	30.2	30.2	30.2	30.2
SR1 SPIKE WEIGHT	46.4	46.4	46.4	46.4	46.4	46.4	46.4
DR9 SPIKE WEIGHT	30.6	30.6	30.6	30.6	30.6	30.6	30.6
SY2 SPIKE WEIGHT	39.4	39.4	39.4	39.4	39.4	39.4	39.4
SG3 SPIKE WEIGHT	31.4	31.4	31.4	31.4	31.4	31.4	31.4
SR24 SPIKE WEIGHT	5.7	5.7	5.7	5.7	5.7	5.7	5.7
AN SPIKE WEIGHT	41.5	41.5	41.5	41.5	41.5	41.5	41.5
BZ SPIKE WEIGHT	24.3	24.3	24.3	24.3	24.3	24.3	24.3

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
	Weight Standard per Volume Sample						
MATRIX VOLUME (WATER)	NA	NA	NA	NA	NA	NA	NA
MATRIX WEIGHT (SOIL)	0.0100946	0.0100175	0.0100136	0.0100155	0.0100428	0.0100303	0.0100841
SPIKE CONC AB45	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY23	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY73	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AO10	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BY2	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DV1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY33	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DB14	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AR64	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB9	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BV10	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 2AQ	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 1AQ	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY14	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO7	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DR9	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY2	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SG3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR24	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AN	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BZ	NA	NA	NA	NA	NA	NA	NA

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
SPIKE CONC AB45	3411.73	3437.98	3439.32	3438.67	3429.32	3433.60	3415.28
SPIKE CONC AY23	2785.65	2807.09	2808.18	2807.65	2800.02	2803.51	2788.55
SPIKE CONC AY73	3477.11	3503.87	3505.23	3504.57	3495.04	3499.40	3480.73
SPIKE CONC AO10	3045.19	3068.63	3069.83	3069.24	3060.90	3064.71	3048.36
SPIKE CONC AB1	3657.40	3685.55	3686.99	3686.29	3676.27	3680.85	3661.21
SPIKE CONC BY2	2617.24	2637.38	2638.41	2637.91	2630.74	2634.02	2619.97
SPIKE CONC DV1	2577.62	2597.45	2598.47	2597.97	2590.91	2594.14	2580.30
SPIKE CONC SY33	2769.80	2791.12	2792.20	2791.67	2784.08	2787.55	2772.68
SPIKE CONC DB14	2961.98	2984.78	2985.94	2985.37	2977.26	2980.97	2965.06
SPIKE CONC AR64	3584.09	3611.68	3613.09	3612.40	3602.58	3607.07	3587.83
SPIKE CONC AB9	3249.26	3274.27	3275.55	3274.92	3266.02	3270.09	3252.65
SPIKE CONC BV10	2064.47	2080.36	2081.17	2080.77	2075.12	2077.70	2066.62
SPIKE CONC 2AQ	3702.97	3731.47	3732.92	3732.22	3722.07	3726.71	3706.83
SPIKE CONC SO3	2534.03	2553.53	2554.53	2554.04	2547.10	2550.27	2536.67
SPIKE CONC 1AQ	2545.92	2565.51	2566.51	2566.02	2559.05	2562.24	2548.57
SPIKE CONC SY14	2805.46	2827.05	2828.15	2827.62	2819.93	2823.44	2808.38
SPIKE CONC SO7	2853.01	2874.97	2876.09	2875.54	2867.73	2871.30	2855.98
SPIKE CONC AY3	3316.62	3342.15	3343.45	3342.82	3333.73	3337.89	3320.08
SPIKE CONC SY3	2991.70	3014.72	3015.90	3015.33	3007.13	3010.88	2994.81
SPIKE CONC SR1	4594.54	4629.90	4631.70	4630.82	4618.23	4623.99	4599.32
SPIKE CONC DR9	3031.32	3054.65	3055.84	3055.26	3046.96	3050.76	3034.48
SPIKE CONC SY2	3903.08	3933.12	3934.65	3933.90	3923.21	3928.10	3907.14
SPIKE CONC SG3	3110.57	3134.51	3135.74	3135.14	3126.62	3130.51	3113.81
SPIKE CONC SR24	562.68	567.01	567.23	567.12	565.58	566.28	563.26
SPIKE CONC AN	4115.07	4146.74	4148.36	4147.57	4136.30	4141.45	4119.36
SPIKE CONC BZ	2409.21	2427.75	2428.70	2428.24	2421.64	2424.65	2411.72

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
	Results from HPLC						
HPLC RESULT AB45	2.416	2.182	2.468	2.439	2.354	1.806	1.964
HPLC RESULT AY23	3.030	3.318	3.020	3.133	2.994	2.890	3.870
HPLC RESULT AY73	4.105	4.317	4.293	4.083	4.272	4.174	4.105
HPLC RESULT AO10	6.641	6.840	6.701	6.344	6.679	6.499	6.502
HPLC RESULT AB1	0.841	0.892	0.592	0.685	0.744	1.035	0.000
HPLC RESULT BY2	24.270	26.331	26.431	24.976	25.997	25.005	25.836
HPLC RESULT DV1	1.266	1.775	1.453	1.565	1.735	1.368	0.000
HPLC RESULT SY33	2.464	2.698	2.704	2.610	2.670	2.631	2.661
HPLC RESULT DB14	2.285	2.555	2.662	2.896	3.001	2.749	2.915
HPLC RESULT AR64	5.495	5.913	5.762	5.353	5.728	5.838	5.848
HPLC RESULT AB9	0.196	0.235	0.207	0.156	0.136	0.074	0.111
HPLC RESULT BV10	2.897	3.029	2.914	2.737	2.884	2.790	2.882
HPLC RESULT 2AQ	4.338	4.386	4.549	4.345	4.535	4.344	4.139
HPLC RESULT SO3	2.152	2.170	2.165	2.142	2.229	2.133	2.128
HPLC RESULT 1AQ	2.604	2.975	3.070	2.936	2.957	2.833	2.932
HPLC RESULT SY14	2.379	2.419	2.476	2.338	2.496	2.486	2.439
HPLC RESULT SO7	3.018	3.279	3.321	3.205	3.319	3.201	3.290
HPLC RESULT AY3	3.351	3.552	3.404	3.387	3.472	3.375	0.000
HPLC RESULT SY3	5.597	5.870	5.917	5.710	5.887	5.822	5.818
HPLC RESULT SR1	5.178	5.366	7.493	8.896	5.780	5.270	5.228
HPLC RESULT DR9	2.521	2.697	2.820	2.593	2.632	2.751	2.794
HPLC RESULT SY2	3.239	3.585	3.585	3.475	3.656	3.518	3.551
HPLC RESULT SG3	3.620	3.106	3.535	3.383	2.940	3.049	2.310
HPLC RESULT SR24	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT AN	3.420	3.815	3.83	3.727	3.804	3.734	3.801
HPLC RESULT BZ	3.043	3.143	3.119	3.113	3.201	3.120	3.171

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
	Final Sample Results						
CF (STD INJ VOL VS SAM INJ VOL)	2	2	2	2	2	2	2
FINAL VOLUME	10.000	10.000	10.000	10.000	10.000	10.000	10.000
CORRECTED RESULT AB45	1196.68	1089.09	1232.32	1217.61	1171.98	900.27	973.81
CORRECTED RESULT AY23	1500.80	1656.10	1507.95	1564.08	1490.62	1440.63	1918.86
CORRECTED RESULT AY73	2033.27	2154.73	2143.58	2038.34	2126.90	2080.70	2035.38
CORRECTED RESULT AO10	3289.38	3414.03	3345.95	3167.09	3325.27	3239.68	3223.89
CORRECTED RESULT AB1	416.56	445.22	295.60	341.97	370.41	515.94	0.00
CORRECTED RESULT BY2	12021.28	13142.50	13197.55	12468.67	12943.10	12464.73	12810.27
CORRECTED RESULT DV1	627.07	885.95	725.51	781.29	863.80	681.93	0.00
CORRECTED RESULT SY33	1220.45	1346.64	1350.16	1302.98	1329.31	1311.53	1319.40
CORRECTED RESULT DB14	1131.79	1275.27	1329.19	1445.76	1494.11	1370.35	1445.34
CORRECTED RESULT AR64	2721.75	2951.34	2877.09	2672.36	2851.79	2910.18	2899.61
CORRECTED RESULT AB9	97.08	117.29	103.36	77.88	67.71	36.89	55.04
CORRECTED RESULT BV10	1434.93	1511.85	1455.02	1366.38	1435.85	1390.79	1428.98
CORRECTED RESULT 2AQ	2148.67	2189.17	2271.41	2169.14	2257.84	2165.44	2052.24
CORRECTED RESULT SO3	1065.92	1083.10	1081.03	1069.34	1109.75	1063.28	1055.13
CORRECTED RESULT 1AQ	1289.80	1484.90	1532.92	1465.73	1472.20	1412.22	1453.77
CORRECTED RESULT SY14	1178.35	1207.39	1236.32	1167.19	1242.68	1239.25	1209.33
CORRECTED RESULT SO7	1494.86	1636.64	1658.24	1600.02	1652.43	1595.67	1631.28
CORRECTED RESULT AY3	1659.80	1772.90	1699.69	1690.88	1728.60	1682.40	0.00
CORRECTED RESULT SY3	2772.27	2929.87	2954.48	2850.58	2930.96	2902.21	2884.74
CORRECTED RESULT SR1	2564.74	2678.31	3741.41	4441.12	2877.68	2627.04	2592.20
CORRECTED RESULT DR9	1248.69	1346.14	1408.09	1294.49	1310.39	1371.34	1385.35
CORRECTED RESULT SY2	1604.32	1789.37	1790.07	1734.81	1820.21	1753.69	1760.69
CORRECTED RESULT SG3	1793.04	1550.29	1765.10	1688.88	1463.74	1519.89	1145.37
CORRECTED RESULT SR24	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT AN	1693.97	1904.17	1912.40	1860.62	1893.89	1861.36	1884.65
CORRECTED RESULT BZ	1507.24	1568.75	1557.38	1554.09	1593.68	1555.29	1572.28

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
	Percent Recoveries						
% RECOVERY AB45	35.08	31.68	35.83	35.41	34.18	26.22	28.51
% RECOVERY AY23	53.88	59.00	53.70	55.71	53.24	51.39	68.81
% RECOVERY AY73	58.48	61.50	61.15	58.16	60.85	59.46	58.48
% RECOVERY AO10	108.02	111.26	108.99	103.19	108.64	105.71	105.76
% RECOVERY AB1	11.39	12.08	8.02	9.28	10.08	14.02	0.00
% RECOVERY BY2	459.31	498.32	500.21	472.67	491.99	473.22	488.95
% RECOVERY DV1	24.33	34.11	27.92	30.07	33.34	26.29	0.00
% RECOVERY SY33	44.06	48.25	48.35	46.67	47.75	47.05	47.59
% RECOVERY DB14	38.21	42.73	44.52	48.43	50.18	45.97	48.75
% RECOVERY AR64	75.94	81.72	79.63	73.98	79.16	80.68	80.82
% RECOVERY AB9	2.99	3.58	3.16	2.38	2.07	1.13	1.69
% RECOVERY BV10	69.51	72.67	69.91	65.67	69.19	66.94	69.15
% RECOVERY 2AQ	58.03	58.67	60.85	58.12	60.66	58.11	55.36
% RECOVERY SO3	42.06	42.42	42.32	41.87	43.57	41.69	41.59
% RECOVERY 1AQ	50.66	57.88	59.73	57.12	57.53	55.12	57.04
% RECOVERY SY14	42.00	42.71	43.71	41.28	44.07	43.89	43.06
% RECOVERY SO7	52.40	56.93	57.66	55.64	57.62	55.57	57.12
% RECOVERY AY3	50.04	53.05	50.84	50.58	51.85	50.40	0.00
% RECOVERY SY3	92.67	97.19	97.96	94.54	97.47	96.39	96.32
% RECOVERY SR1	55.82	57.85	80.78	95.90	62.31	56.81	56.36
% RECOVERY DR9	41.19	44.07	46.08	42.37	43.01	44.95	45.65
% RECOVERY SY2	41.10	45.49	45.49	44.10	46.40	44.64	45.06
% RECOVERY SG3	57.64	49.46	56.29	53.87	46.82	48.55	36.78
% RECOVERY SR24	0.00	0.00	0.00	0.00	0.00	0.00	0.00
% RECOVERY AN	41.17	45.92	46.10	44.86	45.79	44.94	45.75
% RECOVERY BZ	62.56	64.62	64.12	64.00	65.81	64.14	65.19

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
	Square of Corrected Results						
N	7	7	7	7	7	7	7
Xi^2 AB45	1432041.6	1186125.9	1518622.5	1482580.7	1373546.3	810490.0	948306.4
Xi^2 AY23	2252407.9	2742673.2	2273910.8	2446332.7	2221948.4	2075428.8	3682032.8
Xi^2 AY73	4134167.8	4642858.0	4594955.5	4154832.3	4523690.3	4329293.7	4142781.7
Xi^2 AO10	1.082E+07	1.166E+07	1.120E+07	1.003E+07	1.106E+07	1.050E+07	1.039E+07
Xi^2 AB1	173521.7	198221.6	87378.2	116943.4	137207.0	266190.7	0.0
Xi^2 BY2	1.445E+08	1.727E+08	1.742E+08	1.555E+08	1.675E+08	1.554E+08	1.641E+08
Xi^2 DV1	393214.2	784906.7	526369.6	610412.5	746155.5	465033.6	0.0
Xi^2 SY33	1489509.2	1813448.4	1822942.2	1697757.9	1767066.5	1720100.6	1740826.4
Xi^2 DB14	1280955.9	1626309.2	1766752.2	2090219.3	2232350.4	1877853.2	2089021.2
Xi^2 AR64	7407935.2	8710379.2	8277630.5	7141496.5	8132730.8	8469160.1	8407762.8
Xi^2 AB9	9424.8	13758.1	10683.2	6065.2	4584.7	1360.7	3029.1
Xi^2 BV10	2059011.5	2285703.3	2117086.6	1867000.1	2061678.3	1934285.5	2041990.3
Xi^2 2AQ	4616798.0	4792460.7	5159307.4	4705159.0	5097825.5	4689124.9	4211691.7
Xi^2 SO3	1136177.8	1173115.5	1168625.4	1143493.4	1231545.7	1130560.7	1113291.7
Xi^2 1AQ	1663580.2	2204932.2	2349829.1	2148358.9	2167369.9	1994368.1	2113458.2
Xi^2 SY14	1388515.3	1457783.5	1528483.7	1362334.5	1544256.9	1535728.4	1462477.9
Xi^2 SO7	2234602.3	2678577.0	2749775.8	2560063.9	2730517.0	2546147.2	2661077.5
Xi^2 AY3	2754930.4	3143165.3	2888940.7	2859072.3	2988063.4	2830477.6	0.0
Xi^2 SY3	7685504.7	8584154.2	8728963.3	8125815.4	8590500.2	8422801.5	8321721.1
Xi^2 SR1	6577878.9	7173360.3	13998161.4	19723513.7	8281062.4	6901339.5	6719498.8
Xi^2 DR9	1559220.3	1812104.3	1982703.4	1675713.5	1717125.9	1880586.6	1919192.4
Xi^2 SY2	2573852.6	3201840.0	3204334.5	3009569.4	3313162.6	3075415.7	3100038.3
Xi^2 SG3	3214984.8	2403389.8	3115576.1	2852323.2	2142520.8	2310080.0	1311866.6
Xi^2 SR24	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 AN	2869551.3	3625854.7	3657270.5	3461892.1	3586835.0	3464661.3	3551906.0
Xi^2 BZ	2271776.9	2460991.2	2425438.6	2415199.3	2539812.9	2418919.1	2472055.4

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
	Sum of the Square of Corrected Results						
E Xi^2 AB45	8.752E+06						
E Xi^2 AY23	1.769E+07						
E Xi^2 AY73	3.052E+07						
E Xi^2 AO10	7.565E+07						
E Xi^2 AB1	9.795E+05						
E Xi^2 BY2	1.134E+09						
E Xi^2 DV1	3.526E+06						
E Xi^2 SY33	1.205E+07						
E Xi^2 DB14	1.296E+07						
E Xi^2 AR64	5.655E+07						
E Xi^2 AB9	4.891E+04						
E Xi^2 BV10	1.437E+07						
E Xi^2 2AQ	3.327E+07						
E Xi^2 SO3	8.097E+06						
E Xi^2 1AQ	1.464E+07						
E Xi^2 SY14	1.028E+07						
E Xi^2 SO7	1.816E+07						
E Xi^2 AY3	1.746E+07						
E Xi^2 SY3	5.846E+07						
E Xi^2 SR1	6.937E+07						
E Xi^2 DR9	1.255E+07						
E Xi^2 SY2	2.148E+07						
E Xi^2 SG3	1.735E+07						
E Xi^2 SR24	0.000E+00						
E Xi^2 AN	2.422E+07						
E Xi^2 BZ	1.700E+07						

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
	Sum of the Corrected Results Squared						
(E Xi)^2 AB45	6.056E+07						
(E Xi)^2 AY23	1.227E+08						
(E Xi)^2 AY73	2.135E+08						
(E Xi)^2 AO10	5.292E+08						
(E Xi)^2 AB1	5.692E+06						
(E Xi)^2 BY2	7.930E+09						
(E Xi)^2 DV1	2.084E+07						
(E Xi)^2 SY33	8.428E+07						
(E Xi)^2 DB14	9.009E+07						
(E Xi)^2 AR64	3.954E+08						
(E Xi)^2 AB9	3.083E+05						
(E Xi)^2 BV10	1.005E+08						
(E Xi)^2 2AQ	2.327E+08						
(E Xi)^2 SO3	5.666E+07						
(E Xi)^2 1AQ	1.022E+08						
(E Xi)^2 SY14	7.192E+07						
(E Xi)^2 SO7	1.270E+08						
(E Xi)^2 AY3	1.047E+08						
(E Xi)^2 SY3	4.091E+08						
(E Xi)^2 SR1	4.632E+08						
(E Xi)^2 DR9	8.769E+07						
(E Xi)^2 SY2	1.501E+08						
(E Xi)^2 SG3	1.194E+08						
(E Xi)^2 SR24	0.000E+00						
(E Xi)^2 AN	1.693E+08						
(E Xi)^2 BZ	1.190E+08						

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
	Variance						
S^2: VARIANCE AB45	16808.3						
S^2: VARIANCE AY23	26616.0						
S^2: VARIANCE AY73	2889.7						
S^2: VARIANCE AO10	6946.5						
S^2: VARIANCE AB1	27730.4						
S^2: VARIANCE BY2	180169.0						
S^2: VARIANCE DV1	91389.0						
S^2: VARIANCE SY33	1911.9						
S^2: VARIANCE DB14	15470.5						
S^2: VARIANCE AR64	10745.6						
S^2: VARIANCE AB9	810.4						
S^2: VARIANCE BV10	2157.3						
S^2: VARIANCE 2AQ	5354.5						
S^2: VARIANCE SO3	325.9						
S^2: VARIANCE 1AQ	5954.6						
S^2: VARIANCE SY14	906.9						
S^2: VARIANCE SO7	3142.1						
S^2: VARIANCE AY3	416960.1						
S^2: VARIANCE SY3	3835.3						
S^2: VARIANCE SR1	533467.3						
S^2: VARIANCE DR9	3160.6						
S^2: VARIANCE SY2	4943.9						
S^2: VARIANCE SG3	49311.2						
S^2: VARIANCE SR24	0.0						
S^2: VARIANCE AN	5668.0						
S^2: VARIANCE BZ	698.5						

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
	Standard Deviation						
STD DEVIATION AB45	129.6						
STD DEVIATION AY23	163.1						
STD DEVIATION AY73	53.8						
STD DEVIATION AO10	83.3						
STD DEVIATION AB1	166.5						
STD DEVIATION BY2	424.5						
STD DEVIATION DV1	302.3						
STD DEVIATION SY33	43.7						
STD DEVIATION DB14	124.4						
STD DEVIATION AR64	103.7						
STD DEVIATION AB9	28.5						
STD DEVIATION BV10	46.4						
STD DEVIATION 2AQ	73.2						
STD DEVIATION SO3	18.1						
STD DEVIATION 1AQ	77.2						
STD DEVIATION SY14	30.1						
STD DEVIATION SO7	56.1						
STD DEVIATION AY3	645.7						
STD DEVIATION SY3	61.9						
STD DEVIATION SR1	730.4						
STD DEVIATION DR9	56.2						
STD DEVIATION SY2	70.3						
STD DEVIATION SG3	222.1						
STD DEVIATION SR24	0.0						
STD DEVIATION AN	75.3						
STD DEVIATION BZ	26.4						

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
	Method Detection Limit						
T (STUDENT'S T @ 99% FOR 6 DF)	3.140						
MDL AB45	407.1						
MDL AY23	512.3						
MDL AY73	168.8						
MDL AO10	261.7						
MDL AB1	522.9						
MDL BY2	1332.8						
MDL DV1	949.2						
MDL SY33	137.3						
MDL DB14	390.6						
MDL AR64	325.5						
MDL AB9	89.4						
MDL BV10	145.8						
MDL 2AQ	229.8						
MDL SO3	56.7						
MDL 1AQ	242.3						
MDL SY14	94.6						
MDL SO7	176.0						
MDL AY3	2027.6						
MDL SY3	194.5						
MDL SR1	2293.4						
MDL DR9	176.5						
MDL SY2	220.8						
MDL SG3	697.3						
MDL SR24	0.0						
MDL AN	236.4						
MDL BZ	83.0						

TABLE VII-2

Solid Extraction Method, Long Gradient, High Spike

		Sample Results (µg/Kg)							Mean Result µg/Kg	Method Bias (Accuracy) %R	Standard Deviation	Method Precision (%RSD)
Compounds	Sample Conc µg/Kg	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS	average sample result	(mean result/sample conc)*100	std. dev. sample results	(std.dev/mean result)*100
ACID BLUE 45	17220	3665.7	5124.1	5273.1	4659.1	5307.2	5165.4	4834.4	4861.3	28.2	577.5	11.9
ACID YELLOW 23	14060	5030.7	5180.8	5092.6	5117.4	4676.6	4728.9	5054.8	4983.1	35.4	197.9	4.0
ACID YELLOW 73	17550	5457.9	5473.9	5290.4	5449.5	5002.5	5068.8	5349.2	5298.9	30.2	192.4	3.6
ACID ORANGE 10	15370	8295.9	8464.0	8279.4	8302.1	7679.3	7746.5	8177.0	8134.9	52.9	300.9	3.7
ACID BLUE 1	18460	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
BASIC YELLOW 2	13210	34471.8	35455.7	34294.8	34672.8	30980.0	31076.8	33344.8	33470.9	253.4	1780.3	5.3
DISPERSE VIOLET 1	13010	3908.6	3688.8	3657.6	3943.1	3464.8	3366.0	3883.7	3701.8	28.5	225.5	6.1
SOLVENT YELLOW 33	13980	3182.0	3250.6	3243.3	3198.7	2937.6	2981.9	3143.5	3133.9	22.4	125.1	4.0
DISPERSE BLUE 14	14950	2781.5	3569.8	3339.9	3018.2	2737.2	2770.5	2836.2	3007.6	20.1	325.7	10.8
ACID RED 64	18090	6263.8	6367.5	6289.2	6375.9	5858.0	5742.4	6100.8	6142.5	34.0	253.0	4.1
ACID BLUE 9	16400	478.3	476.6	473.9	514.1	475.8	403.2	349.5	453.1	2.8	56.4	12.4
BASIC VIOLET 10	10420	4015.1	4052.4	3986.3	3968.5	3689.2	3723.5	3955.7	3913.0	37.6	145.0	3.7
2-AMINOANTHRAQUINONE	18690	6018.0	6402.5	6111.3	6159.5	5442.5	5683.3	6007.5	5974.9	32.0	318.1	5.3
SOLVENT ORANGE 3	12790	2963.9	2926.4	2906.2	2899.0	2679.3	2720.5	2914.6	2858.6	22.4	111.0	3.9
1-AMINOANTHRAQUINONE	12850	4090.5	4253.7	4105.2	4139.1	3640.7	3817.8	4103.1	4021.4	31.3	213.2	5.3
SOLVENT YELLOW 14	14160	3307.4	3414.9	3341.9	3349.8	3058.1	3102.3	3304.3	3268.4	23.1	134.2	4.1
SOLVENT ORANGE 7	14400	4303.6	4793.5	4605.9	4660.6	3915.1	4390.0	4558.9	4461.1	31.0	291.2	6.5
ACID YELLOW 3	16740	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT YELLOW 3	15100	7448.9	7751.5	7569.1	7537.2	6911.0	7036.2	7430.8	7383.5	48.9	301.1	4.1
SOLVENT RED 1	23190	5303.3	5167.5	6168.8	6139.1	5241.1	4752.7	4954.5	5389.6	23.2	554.2	10.3
DISPERSE RED 9	15300	3349.0	3508.7	3433.1	3310.4	3062.5	3152.8	3386.8	3314.7	21.7	157.0	4.7
SOLVENT YELLOW 2	19700	4495.4	4594.7	4532.5	4450.7	4083.2	4132.0	4422.8	4387.3	22.3	199.5	4.5
SOLVENT GREEN 3	15700	3360.4	2235.2	13683.3	11536.7	10682.6	8903.7	6624.1	8146.6	51.9	4268.5	52.4
SOLVENT RED 24	2840	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ANTHRACENE	20770	4877.0	5055.0	4955.3	4868.6	4560.0	4622.7	4913.3	4836.0	23.3	179.2	3.7
BENZANTHRONE	12160	3896.7	4099.3	3976.4	3998.0	3610.8	3760.7	3976.5	3902.6	32.1	165.3	4.2

TABLE VII - 2A

PERCENT RECOVERIES
SOIL METHOD
HIGH SPIKE, LONG GRADIENT

Compounds		Ret. Time	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
% RECOVERY AB45	%	6.439	21	30	31	27	31	32	28
% RECOVERY AY3	%	13.372	0	0	0	0	0	0	0
% RECOVERY AY23	%	14.513	36	37	37	36	34	35	36
% RECOVERY AR64	%	14.9	35	36	35	35	33	33	34
% RECOVERY AY73	%	17.353	31	32	30	31	29	30	31
% RECOVERY AO10	%	18.752	54	56	54	54	51	53	54
% RECOVERY AB9	%	21.657	3	3	3	3	3	3	2
% RECOVERY DV1	%	22.77	30	29	28	30	27	27	30
% RECOVERY AB1	%	23.733	0	0	0	0	0	0	0
% RECOVERY 2AQ	%	23.758	32	35	33	33	30	32	32
% RECOVERY SO3	%	24.783	23	23	23	23	21	22	23
% RECOVERY SY33	%	28.259	23	24	23	23	21	22	23
% RECOVERY 1AQ	%	28.853	32	34	32	32	29	31	32
% RECOVERY DB14	%	34.023	19	24	23	20	19	19	19
% RECOVERY BY2	%	35.367	263	272	262	263	239	247	254
% RECOVERY SY3	%	35.998	50	52	51	50	47	49	50
% RECOVERY BV10	%	36.018	39	39	39	38	36	38	38
% RECOVERY DR9	%	36.142	22	23	23	22	20	22	22
% RECOVERY BZ	%	36.724	32	34	33	33	30	32	33
% RECOVERY SY2	%	39.91	23	24	23	23	21	22	23
% RECOVERY AN	%	40.641	24	25	24	24	22	23	24
% RECOVERY SY14	%	44.36	24	24	24	24	22	23	24
% RECOVERY SR1	%	44.811	23	23	27	27	23	22	22
% RECOVERY SO7	%	50.894	30	34	32	32	28	32	32
% RECOVERY SG3	%	57.448	22	14	88	74	69	60	42
% RECOVERY SR24	%	57.994	0	0	0	0	0	0	0

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Stock Standard Concentrations						
ACID BLUE 45 STD	344.4	344.4	344.4	344.4	344.4	344.4	344.4
ACID YELLOW 23 STD	281.2	281.2	281.2	281.2	281.2	281.2	281.2
ACID YELLOW 73 STD	351.0	351.0	351.0	351.0	351.0	351.0	351.0
ACID ORANGE 10 STD	307.4	307.4	307.4	307.4	307.4	307.4	307.4
ACID BLUE 1 STD	369.2	369.2	369.2	369.2	369.2	369.2	369.2
BASIC YELLOW 2 STD	264.2	264.2	264.2	264.2	264.2	264.2	264.2
DISPERSE VIOLET 1 STD	260.2	260.2	260.2	260.2	260.2	260.2	260.2
SOLVENT YELLOW 33 STD	279.6	279.6	279.6	279.6	279.6	279.6	279.6
DISPERSE BLUE 14 STD	299.0	299.0	299.0	299.0	299.0	299.0	299.0
ACID RED 64 STD	361.8	361.8	361.8	361.8	361.8	361.8	361.8
ACID BLUE 9 STD	328.0	328.0	328.0	328.0	328.0	328.0	328.0
BASIC VIOLET 10 STD	208.4	208.4	208.4	208.4	208.4	208.4	208.4
2-AMINOANTHRAQUINONE STD	373.8	373.8	373.8	373.8	373.8	373.8	373.8
SOLVENT ORANGE 3 STD	255.8	255.8	255.8	255.8	255.8	255.8	255.8
1-AMINOANTHRAQUINONE STD	257.0	257.0	257.0	257.0	257.0	257.0	257.0
SOLVENT YELLOW 14 STD	283.2	283.2	283.2	283.2	283.2	283.2	283.2
SOLVENT ORANGE 7 STD	288.0	288.0	288.0	288.0	288.0	288.0	288.0
ACID YELLOW 3 STD	334.8	334.8	334.8	334.8	334.8	334.8	334.8
SOLVENT YELLOW 3 STD	302.0	302.0	302.0	302.0	302.0	302.0	302.0
SOLVENT RED 1 STD	463.8	463.8	463.8	463.8	463.8	463.8	463.8
DISPERSE RED 9 STD	306.0	306.0	306.0	306.0	306.0	306.0	306.0
SOLVENT YELLOW 2 STD	394.0	394.0	394.0	394.0	394.0	394.0	394.0
SOLVENT GREEN 3 STD	314.0	314.0	314.0	314.0	314.0	314.0	314.0
SOLVENT RED 24 STD	56.8	56.8	56.8	56.8	56.8	56.8	56.8
ANTHRACENE	415.4	415.4	415.4	415.4	415.4	415.4	415.4
BENZANTHRONE	243.2	243.2	243.2	243.2	243.2	243.2	243.2

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Weight of Stock Standard Spiked						
SPIKE VOLUME EACH DYE	500.0	500.0	500.0	500.0	500.0	500.0	500.0
AB45 SPIKE WEIGHT	172.2	172.2	172.2	172.2	172.2	172.2	172.2
AY23 SPIKE WEIGHT	140.6	140.6	140.6	140.6	140.6	140.6	140.6
AY73 SPIKE WEIGHT	175.5	175.5	175.5	175.5	175.5	175.5	175.5
AO10 SPIKE WEIGHT	153.7	153.7	153.7	153.7	153.7	153.7	153.7
AB1 SPIKE WEIGHT	184.6	184.6	184.6	184.6	184.6	184.6	184.6
BY2 SPIKE WEIGHT	132.1	132.1	132.1	132.1	132.1	132.1	132.1
DV1 SPIKE WEIGHT	130.1	130.1	130.1	130.1	130.1	130.1	130.1
SY33 SPIKE WEIGHT	139.8	139.8	139.8	139.8	139.8	139.8	139.8
DB14 SPIKE WEIGHT	149.5	149.5	149.5	149.5	149.5	149.5	149.5
AR64 SPIKE WEIGHT	180.9	180.9	180.9	180.9	180.9	180.9	180.9
AB9 SPIKE WEIGHT	164.0	164.0	164.0	164.0	164.0	164.0	164.0
BV10 SPIKE WEIGHT	104.2	104.2	104.2	104.2	104.2	104.2	104.2
2AQ SPIKE WEIGHT	186.9	186.9	186.9	186.9	186.9	186.9	186.9
SO3 SPIKE WEIGHT	127.9	127.9	127.9	127.9	127.9	127.9	127.9
1AQ SPIKE WEIGHT	128.5	128.5	128.5	128.5	128.5	128.5	128.5
SY14 SPIKE WEIGHT	141.6	141.6	141.6	141.6	141.6	141.6	141.6
SO7 SPIKE WEIGHT	144.0	144.0	144.0	144.0	144.0	144.0	144.0
AY3 SPIKE WEIGHT	167.4	167.4	167.4	167.4	167.4	167.4	167.4
SY3 SPIKE WEIGHT	151.0	151.0	151.0	151.0	151.0	151.0	151.0
SR1 SPIKE WEIGHT	231.9	231.9	231.9	231.9	231.9	231.9	231.9
DR9 SPIKE WEIGHT	153.0	153.0	153.0	153.0	153.0	153.0	153.0
SY2 SPIKE WEIGHT	197.0	197.0	197.0	197.0	197.0	197.0	197.0
SG3 SPIKE WEIGHT	157.0	157.0	157.0	157.0	157.0	157.0	157.0
SR24 SPIKE WEIGHT	28.4	28.4	28.4	28.4	28.4	28.4	28.4
AN SPIKE WEIGHT	207.7	207.7	207.7	207.7	207.7	207.7	207.7
BZ SPIKE WEIGHT	121.6	121.6	121.6	121.6	121.6	121.6	121.6

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Weight Standard per Volume Sample						
MATRIX VOLUME (WATER)	NA	NA	NA	NA	NA	NA	NA
MATRIX WEIGHT (SOIL)	0.0100881	0.0101335	0.0100871	0.0100276	0.0102040	0.0105035	0.0100716
SPIKE CONC AB45	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY23	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY73	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AO10	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BY2	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DV1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY33	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DB14	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AR64	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB9	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BV10	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 2AQ	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 1AQ	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY14	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO7	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DR9	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY2	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SG3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR24	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AN	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BZ	NA	NA	NA	NA	NA	NA	NA

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
SPIKE CONC AB45	17069.62	16993.14	17071.31	17172.60	16875.74	16394.54	17097.58
SPIKE CONC AY23	13937.21	13874.77	13938.59	14021.30	13778.91	13386.01	13960.05
SPIKE CONC AY73	17396.73	17318.79	17398.46	17501.70	17199.14	16708.72	17425.24
SPIKE CONC AO10	15235.77	15167.51	15237.28	15327.70	15062.72	14633.22	15260.73
SPIKE CONC AB1	18298.79	18216.81	18300.60	18409.19	18090.94	17575.09	18328.77
SPIKE CONC BY2	13094.64	13035.97	13095.93	13173.64	12945.90	12576.76	13116.09
SPIKE CONC DV1	12896.38	12838.60	12897.66	12974.19	12749.90	12386.35	12917.51
SPIKE CONC SY33	13857.91	13795.83	13859.29	13941.52	13700.51	13309.85	13880.61
SPIKE CONC DB14	14819.44	14753.05	14820.91	14908.85	14651.12	14233.35	14843.72
SPIKE CONC AR64	17932.02	17851.68	17933.80	18040.21	17728.34	17222.83	17961.40
SPIKE CONC AB9	16256.78	16183.94	16258.39	16354.86	16072.13	15613.84	16283.41
SPIKE CONC BV10	10329.00	10282.73	10330.03	10391.32	10211.68	9920.50	10345.92
SPIKE CONC 2AQ	18526.78	18443.78	18528.62	18638.56	18316.35	17794.07	18557.13
SPIKE CONC SO3	12678.30	12621.50	12679.56	12754.80	12534.30	12176.89	12699.07
SPIKE CONC 1AQ	12737.78	12680.71	12739.04	12814.63	12593.10	12234.02	12758.65
SPIKE CONC SY14	14036.34	13973.45	14037.73	14121.03	13876.91	13481.22	14059.34
SPIKE CONC SO7	14274.24	14210.29	14275.66	14360.37	14112.11	13709.72	14297.63
SPIKE CONC AY3	16593.81	16519.47	16595.45	16693.92	16405.33	15937.54	16620.99
SPIKE CONC SY3	14968.13	14901.07	14969.61	15058.44	14798.12	14376.16	14992.65
SPIKE CONC SR1	22987.48	22884.49	22989.76	23126.17	22726.38	22078.35	23025.14
SPIKE CONC DR9	15166.38	15098.44	15167.89	15257.89	14994.12	14566.57	15191.23
SPIKE CONC SY2	19527.96	19440.47	19529.89	19645.78	19306.15	18755.65	19559.95
SPIKE CONC SG3	15562.89	15493.17	15564.43	15656.79	15386.12	14947.40	15588.39
SPIKE CONC SR24	2815.20	2802.59	2815.48	2832.18	2783.22	2703.86	2819.81
SPIKE CONC AN	20588.61	20496.37	20590.66	20712.83	20354.76	19774.36	20622.34
SPIKE CONC BZ	12053.81	11999.80	12055.00	12126.53	11916.90	11577.09	12073.55

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Results from HPLC						
HPLC RESULT AB45	7.396	10.385	10.638	9.344	10.831	10.851	9.738
HPLC RESULT AY23	10.150	10.500	10.274	10.263	9.544	9.934	10.182
HPLC RESULT AY73	11.012	11.094	10.673	10.929	10.209	10.648	10.775
HPLC RESULT AO10	16.738	17.154	16.703	16.650	15.672	16.273	16.471
HPLC RESULT AB1	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT BY2	69.551	71.858	69.187	69.537	63.224	65.283	67.167
HPLC RESULT DV1	7.886	7.476	7.379	7.908	7.071	7.071	7.823
HPLC RESULT SY33	6.420	6.588	6.543	6.415	5.995	6.264	6.332
HPLC RESULT DB14	5.612	7.235	6.738	6.053	5.586	5.820	5.713
HPLC RESULT AR64	12.638	12.905	12.688	12.787	11.955	12.063	12.289
HPLC RESULT AB9	0.965	0.966	0.956	1.031	0.971	0.847	0.704
HPLC RESULT BV10	8.101	8.213	8.042	7.959	7.529	7.822	7.968
HPLC RESULT 2AQ	12.142	12.976	12.329	12.353	11.107	11.939	12.101
HPLC RESULT SO3	5.980	5.931	5.863	5.814	5.468	5.715	5.871
HPLC RESULT 1AQ	8.253	8.621	8.282	8.301	7.430	8.020	8.265
HPLC RESULT SY14	6.673	6.921	6.742	6.718	6.241	6.517	6.656
HPLC RESULT SO7	8.683	9.715	9.292	9.347	7.990	9.222	9.183
HPLC RESULT AY3	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SY3	15.029	15.710	15.270	15.116	14.104	14.781	14.968
HPLC RESULT SR1	10.700	10.473	12.445	12.312	10.696	9.984	9.980
HPLC RESULT DR9	6.757	7.111	6.926	6.639	6.250	6.623	6.822
HPLC RESULT SY2	9.070	9.312	9.144	8.926	8.333	8.680	8.909
HPLC RESULT SG3	6.780	4.530	27.605	23.137	21.801	18.704	13.343
HPLC RESULT SR24	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT AN	9.840	10.245	9.997	9.764	9.306	9.711	9.897
HPLC RESULT BZ	7.862	8.308	8.022	8.018	7.369	7.900	8.010

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Final Sample Results						
CF (STD INJ VOL VS SAM INJ VOL)	2	2	2	2	2	2	2
FINAL VOLUME	10.000	10.000	10.000	10.000	10.000	10.000	10.000
CORRECTED RESULT AB45	3665.71	5124.09	5273.07	4659.14	5307.23	5165.42	4834.39
CORRECTED RESULT AY23	5030.68	5180.84	5092.64	5117.38	4676.60	4728.90	5054.81
CORRECTED RESULT AY73	5457.92	5473.92	5290.42	5449.46	5002.45	5068.79	5349.20
CORRECTED RESULT AO10	8295.91	8464.01	8279.39	8302.09	7679.34	7746.47	8176.95
CORRECTED RESULT AB1	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT BY2	34471.80	35455.67	34294.79	34672.80	30980.01	31076.78	33344.75
CORRECTED RESULT DV1	3908.57	3688.76	3657.64	3943.12	3464.82	3366.02	3883.69
CORRECTED RESULT SY33	3181.97	3250.60	3243.25	3198.67	2937.57	2981.86	3143.49
CORRECTED RESULT DB14	2781.50	3569.84	3339.91	3018.17	2737.16	2770.51	2836.19
CORRECTED RESULT AR64	6263.82	6367.49	6289.22	6375.90	5858.00	5742.37	6100.82
CORRECTED RESULT AB9	478.29	476.64	473.87	514.08	475.79	403.20	349.50
CORRECTED RESULT BV10	4015.13	4052.40	3986.28	3968.55	3689.24	3723.52	3955.68
CORRECTED RESULT 2AQ	6017.98	6402.53	6111.27	6159.50	5442.47	5683.34	6007.49
CORRECTED RESULT SO3	2963.89	2926.43	2906.19	2899.00	2679.34	2720.52	2914.63
CORRECTED RESULT 1AQ	4090.46	4253.71	4105.24	4139.08	3640.73	3817.78	4103.12
CORRECTED RESULT SY14	3307.36	3414.91	3341.89	3349.75	3058.11	3102.30	3304.34
CORRECTED RESULT SO7	4303.59	4793.51	4605.88	4660.64	3915.13	4389.97	4558.86
CORRECTED RESULT AY3	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SY3	7448.88	7751.52	7569.07	7537.20	6911.02	7036.23	7430.80
CORRECTED RESULT SR1	5303.28	5167.51	6168.77	6139.06	5241.08	4752.70	4954.53
CORRECTED RESULT DR9	3349.00	3508.66	3433.10	3310.36	3062.52	3152.76	3386.75
CORRECTED RESULT SY2	4495.40	4594.66	4532.52	4450.72	4083.20	4131.96	4422.83
CORRECTED RESULT SG3	3360.39	2235.16	13683.32	11536.66	10682.58	8903.70	6624.07
CORRECTED RESULT SR24	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT AN	4877.03	5055.02	4955.34	4868.56	4559.98	4622.74	4913.32
CORRECTED RESULT BZ	3896.67	4099.27	3976.37	3997.97	3610.84	3760.65	3976.53

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Percent Recoveries						
% RECOVERY AB45	21.48	30.15	30.89	27.13	31.45	31.51	28.28
% RECOVERY AY23	36.10	37.34	36.54	36.50	33.94	35.33	36.21
% RECOVERY AY73	31.37	31.61	30.41	31.14	29.09	30.34	30.70
% RECOVERY AO10	54.45	55.80	54.34	54.16	50.98	52.94	53.58
% RECOVERY AB1	0.00	0.00	0.00	0.00	0.00	0.00	0.00
% RECOVERY BY2	263.25	271.98	261.87	263.20	239.30	247.10	254.23
% RECOVERY DV1	30.31	28.73	28.36	30.39	27.18	27.18	30.07
% RECOVERY SY33	22.96	23.56	23.40	22.94	21.44	22.40	22.65
% RECOVERY DB14	18.77	24.20	22.54	20.24	18.68	19.46	19.11
% RECOVERY AR64	34.93	35.67	35.07	35.34	33.04	33.34	33.97
% RECOVERY AB9	2.94	2.95	2.91	3.14	2.96	2.58	2.15
% RECOVERY BV10	38.87	39.41	38.59	38.19	36.13	37.53	38.23
% RECOVERY 2AQ	32.48	34.71	32.98	33.05	29.71	31.94	32.37
% RECOVERY SO3	23.38	23.19	22.92	22.73	21.38	22.34	22.95
% RECOVERY 1AQ	32.11	33.54	32.23	32.30	28.91	31.21	32.16
% RECOVERY SY14	23.56	24.44	23.81	23.72	22.04	23.01	23.50
% RECOVERY SO7	30.15	33.73	32.26	32.45	27.74	32.02	31.89
% RECOVERY AY3	0.00	0.00	0.00	0.00	0.00	0.00	0.00
% RECOVERY SY3	49.76	52.02	50.56	50.05	46.70	48.94	49.56
% RECOVERY SR1	23.07	22.58	26.83	26.55	23.06	21.53	21.52
% RECOVERY DR9	22.08	23.24	22.63	21.70	20.42	21.64	22.29
% RECOVERY SY2	23.02	23.63	23.21	22.65	21.15	22.03	22.61
% RECOVERY SG3	21.59	14.43	87.91	73.68	69.43	59.57	42.49
% RECOVERY SR24	0.00	0.00	0.00	0.00	0.00	0.00	0.00
% RECOVERY AN	23.69	24.66	24.07	23.51	22.40	23.38	23.83
% RECOVERY BZ	32.33	34.16	32.99	32.97	30.30	32.48	32.94

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Square of Corrected Results						
N	7	7	7	7	7	7	7
Xi^2 AB45	13437394.2	2.626E+07	2.781E+07	21707592.7	28166716.4	26681574.6	23371286.0
Xi^2 AY23	25307738.4	26841060.0	25935013.5	26187537.6	21870563.4	22362494.2	25551079.6
Xi^2 AY73	29788844.5	29963834.4	27988548.4	29696608.8	25024506.2	25692597.5	28613937.8
Xi^2 AO10	6.882E+07	7.164E+07	6.855E+07	6.892E+07	5.897E+07	6.001E+07	6.686E+07
Xi^2 AB1	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 BY2	1.188E+09	1.257E+09	1.176E+09	1.202E+09	9.598E+08	9.658E+08	1.112E+09
Xi^2 DV1	15276884.6	13606914.3	13378344.6	15548171.7	12004961.8	11330096.4	15083069.5
Xi^2 SY33	10124913.2	10566429.2	10518678.9	10231500.4	8629338.1	8891508.1	9881545.7
Xi^2 DB14	7736714.6	12743776.2	11154994.7	9109349.3	7492055.3	7675698.3	8043989.9
Xi^2 AR64	39235388.2	40544979.3	39554299.4	40652132.8	34316127.3	32974831.5	37219982.0
Xi^2 AB9	228757.8	227182.7	224555.2	264279.4	226379.7	162569.4	122148.6
Xi^2 BV10	16121242.7	16421949.4	15890424.3	15749363.8	13610488.2	13864606.6	15647383.3
Xi^2 2AQ	36216102.3	40992342.7	37347631.2	37939437.5	29620518.2	32300395.0	36089892.8
Xi^2 SO3	8784632.9	8564005.0	8445923.5	8404193.8	7178870.5	7401238.5	8495075.3
Xi^2 1AQ	16731887.7	18094073.7	16853022.8	17131951.4	13254908.6	14575406.2	16835607.3
Xi^2 SY14	10938644.3	11661616.7	11168242.9	11220856.4	9352064.1	9624260.5	10918668.9
Xi^2 SO7	18520847.4	22977706.3	21214156.0	21721533.9	15328253.3	19271794.9	20783191.5
Xi^2 AY3	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SY3	5.549E+07	6.009E+07	5.729E+07	5.681E+07	4.776E+07	4.951E+07	5.522E+07
Xi^2 SR1	28124758.8	26703197.8	38053723.5	37688011.1	27468939.8	22588171.4	24547323.9
Xi^2 DR9	11215769.9	12310690.8	11786159.9	10958505.8	9379056.3	9939886.8	11470081.4
Xi^2 SY2	20208581.3	21110912.2	20543753.3	19808873.1	16672544.0	17073060.5	19561447.5
Xi^2 SG3	11292254.0	4995942.9	187233199.6	133094496.8	114117418.5	79275851.7	43878325.2
Xi^2 SR24	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 AN	23785454.2	25553182.1	24555384.6	23702903.4	20793385.5	21369769.5	24140719.6
Xi^2 BZ	15184039.7	16804052.9	15811485.4	15983729.1	13038157.5	14142497.5	15812775.4

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Sum of the Square of Corrected Results						
E Xi^2 AB45	1.674E+08						
E Xi^2 AY23	1.741E+08						
E Xi^2 AY73	1.968E+08						
E Xi^2 AO10	4.638E+08						
E Xi^2 AB1	0.000E+00						
E Xi^2 BY2	7.861E+09						
E Xi^2 DV1	9.623E+07						
E Xi^2 SY33	6.884E+07						
E Xi^2 DB14	6.396E+07						
E Xi^2 AR64	2.645E+08						
E Xi^2 AB9	1.456E+06						
E Xi^2 BV10	1.073E+08						
E Xi^2 2AQ	2.505E+08						
E Xi^2 SO3	5.727E+07						
E Xi^2 1AQ	1.135E+08						
E Xi^2 SY14	7.488E+07						
E Xi^2 SO7	1.398E+08						
E Xi^2 AY3	0.000E+00						
E Xi^2 SY3	3.822E+08						
E Xi^2 SR1	2.052E+08						
E Xi^2 DR9	7.706E+07						
E Xi^2 SY2	1.350E+08						
E Xi^2 SG3	5.739E+08						
E Xi^2 SR24	0.000E+00						
E Xi^2 AN	1.639E+08						
E Xi^2 BZ	1.068E+08						

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Sum of the Corrected Results Squared						
(E Xi)^2 AB45	1.158E+09						
(E Xi)^2 AY23	1.217E+09						
(E Xi)^2 AY73	1.376E+09						
(E Xi)^2 AO10	3.243E+09						
(E Xi)^2 AB1	0.000E+00						
(E Xi)^2 BY2	5.489E+10						
(E Xi)^2 DV1	6.715E+08						
(E Xi)^2 SY33	4.813E+08						
(E Xi)^2 DB14	4.432E+08						
(E Xi)^2 AR64	1.849E+09						
(E Xi)^2 AB9	1.006E+07						
(E Xi)^2 BV10	7.503E+08						
(E Xi)^2 2AQ	1.749E+09						
(E Xi)^2 SO3	4.004E+08						
(E Xi)^2 1AQ	7.924E+08						
(E Xi)^2 SY14	5.234E+08						
(E Xi)^2 SO7	9.752E+08						
(E Xi)^2 AY3	0.000E+00						
(E Xi)^2 SY3	2.671E+09						
(E Xi)^2 SR1	1.423E+09						
(E Xi)^2 DR9	5.384E+08						
(E Xi)^2 SY2	9.432E+08						
(E Xi)^2 SG3	3.252E+09						
(E Xi)^2 SR24	0.000E+00						
(E Xi)^2 AN	1.146E+09						
(E Xi)^2 BZ	7.463E+08						

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Variance						
S^2: VARIANCE AB45	333500.2						
S^2: VARIANCE AY23	39182.8						
S^2: VARIANCE AY73	37004.0						
S^2: VARIANCE AO10	90541.1						
S^2: VARIANCE AB1	0.0						
S^2: VARIANCE BY2	3169454.3						
S^2: VARIANCE DV1	50849.9						
S^2: VARIANCE SY33	15639.1						
S^2: VARIANCE DB14	106085.4						
S^2: VARIANCE AR64	64021.0						
S^2: VARIANCE AB9	3179.5						
S^2: VARIANCE BV10	21018.4						
S^2: VARIANCE 2AQ	101156.5						
S^2: VARIANCE SO3	12320.4						
S^2: VARIANCE 1AQ	45444.7						
S^2: VARIANCE SY14	18017.4						
S^2: VARIANCE SO7	84796.8						
S^2: VARIANCE AY3	0.0						
S^2: VARIANCE SY3	90641.5						
S^2: VARIANCE SR1	307091.9						
S^2: VARIANCE DR9	24640.3						
S^2: VARIANCE SY2	39788.8						
S^2: VARIANCE SG3	18220514.3						
S^2: VARIANCE SR24	0.0						
S^2: VARIANCE AN	32099.8						
S^2: VARIANCE BZ	27331.9						

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Standard Deviation						
STD DEVIATION AB45	577.5						
STD DEVIATION AY23	197.9						
STD DEVIATION AY73	192.4						
STD DEVIATION AO10	300.9						
STD DEVIATION AB1	0.0						
STD DEVIATION BY2	1780.3						
STD DEVIATION DV1	225.5						
STD DEVIATION SY33	125.1						
STD DEVIATION DB14	325.7						
STD DEVIATION AR64	253.0						
STD DEVIATION AB9	56.4						
STD DEVIATION BV10	145.0						
STD DEVIATION 2AQ	318.1						
STD DEVIATION SO3	111.0						
STD DEVIATION 1AQ	213.2						
STD DEVIATION SY14	134.2						
STD DEVIATION SO7	291.2						
STD DEVIATION AY3	0.0						
STD DEVIATION SY3	301.1						
STD DEVIATION SR1	554.2						
STD DEVIATION DR9	157.0						
STD DEVIATION SY2	199.5						
STD DEVIATION SG3	4268.5						
STD DEVIATION SR24	0.0						
STD DEVIATION AN	179.2						
STD DEVIATION BZ	165.3						

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Method Detection Limit						
T (STUDENT'S T @ 99% FOR 6 DF)	3.140						
MDL AB45	1813.3						
MDL AY23	621.6						
MDL AY73	604.0						
MDL AO10	944.8						
MDL AB1	0.0						
MDL BY2	5590.1						
MDL DV1	708.1						
MDL SY33	392.7						
MDL DB14	1022.7						
MDL AR64	794.5						
MDL AB9	177.1						
MDL BV10	455.2						
MDL 2AQ	998.7						
MDL SO3	348.5						
MDL 1AQ	669.4						
MDL SY14	421.5						
MDL SO7	914.4						
MDL AY3	0.0						
MDL SY3	945.4						
MDL SR1	1740.1						
MDL DR9	492.9						
MDL SY2	626.3						
MDL SG3	13403.2						
MDL SR24	0.0						
MDL AN	562.6						
MDL BZ	519.1						

Table VII - 3B

Percent Recoveries Soil Method Low Spike, Short Gradient

Compounds		Ret. Time	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
% RECOVERY AB45	%	4.439	50	65	66	66	66	55	53
% RECOVERY AR64	%	6.174	64	66	68	62	66	64	66
% RECOVERY AY3	%	7.322	30	31	31	33	30	29	32
% RECOVERY AY23	%	7.525	74	67	63	59	64	62	63
% RECOVERY AY73	%	9.159	64	67	66	62	65	64	63
% RECOVERY AO10	%	9.946	205	209	205	193	203	198	199
% RECOVERY AB9	%	11.576	5	6	8	6	7	4	5
% RECOVERY DV1	%	12.69	49	65	54	59	61	50	56
% RECOVERY AB1	%	12.709	16	17	8	11	15	21	20
% RECOVERY 2AQ	%	13.246	44	43	46	43	45	43	43
% RECOVERY SO3	%	13.811	53	53	52	51	54	52	52
% RECOVERY SY33	%	15.498	36	40	40	38	39	38	39
% RECOVERY 1AQ	%	16.108	73	88	87	84	87	83	84
% RECOVERY DB14	%	18.857	38	45	51	64	62	53	63
% RECOVERY BY2	%	19.452	483	521	517	488	513	489	499
% RECOVERY BV10	%	19.691	101	104	99	95	99	97	99
% RECOVERY SY3	%	19.962	211	221	221	214	219	219	219
% RECOVERY DR9	%	20.367	40	46	43	43	46	45	45
% RECOVERY BZ	%	20.438	102	108	106	104	107	105	109
% RECOVERY SY2	%	22.244	41	45	45	44	45	44	45
% RECOVERY AN	%	22.537	45	52	51	49	51	49	51
% RECOVERY SY14	%	24.403	43	43	42	41	44	43	43
% RECOVERY SR1	%	24.66	46	48	71	86	51	47	46
% RECOVERY SO7	%	27.804	64	83	84	67	69	82	83
% RECOVERY SG3	%	31.194	57	47	61	62	44	41	30
% RECOVERY SR24	%	31.255	0	0	0	0	0	0	0

Percent Recovery

TABLE VII - 3A

PERCENT RECOVERIES SOIL METHOD LOW SPIKE, SHORT GRADIENT

Compounds		Ret. Time	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS	Average (p)	Std. Deviation (s)	Lower Control Limit (p-2s)	Upper Control Limit (p+2s)
% RECOVERY AB45	%	4.439	50	65	66	66	66	55	53	60	7	46	75
% RECOVERY AR64	%	6.174	64	66	68	62	66	64	66	65	2	61	69
% RECOVERY AY3	%	7.322	30	31	31	33	30	29	32	31	1	28	34
% RECOVERY AY23	%	7.525	74	67	63	59	64	62	63	65	5	55	74
% RECOVERY AY73	%	9.159	64	67	66	62	65	64	63	65	2	61	68
% RECOVERY AO10	%	9.946	205	209	205	193	203	198	199	202	5	191	213
% RECOVERY AB9	%	11.576	5	6	8	6	7	4	5	6	1	3	9
% RECOVERY DV1	%	12.69	49	65	54	59	61	50	56	56	6	45	68
% RECOVERY AB1	%	12.709	16	17	8	11	15	21	20	16	5	6	25
% RECOVERY 2AQ	%	13.246	44	43	46	43	45	43	43	44	1	42	46
% RECOVERY SO3	%	13.811	53	53	52	51	54	52	52	52	1	51	54
% RECOVERY SY33	%	15.498	36	40	40	38	39	38	39	38	1	35	41
% RECOVERY 1AQ	%	16.108	73	88	87	84	87	83	84	84	5	73	94
% RECOVERY DB14	%	18.857	38	45	51	64	62	53	63	54	10	34	73
% RECOVERY BY2	%	19.452	483	521	517	488	513	489	499	501	15	470	532
% RECOVERY BV10	%	19.691	101	104	99	95	99	97	99	99	3	94	105
% RECOVERY SY3	%	19.962	211	221	221	214	219	219	219	218	4	210	225
% RECOVERY DR9	%	20.367	40	46	43	43	46	45	45	44	2	40	48
% RECOVERY BZ	%	20.438	102	108	106	104	107	105	109	106	2	99	113
% RECOVERY SY2	%	22.244	41	45	45	44	45	44	45	44	1	41	47
% RECOVERY AN	%	22.537	45	52	51	49	51	49	51	50	2	42	57
% RECOVERY SY14	%	24.403	43	43	42	41	44	43	43	43	1	41	44
% RECOVERY SR1	%	24.66	46	48	71	86	51	47	46	56	16	25	88
% RECOVERY SO7	%	27.804	64	83	84	67	69	82	83	76	9	58	94
% RECOVERY SG3	%	31.194	57	47	61	62	44	41	30	49	12	25	72
% RECOVERY SR24	%	31.255	0	0	0	0	0	0	0	0	0	0	0

TABLE VII-3

Solid Extraction Method, Short Gradient, Low Spike

		Sample Results (µg/Kg)							Mean Result µg/Kg	Method Bias (Accuracy)	Standard Deviation	Method Precision (%RSD)	MDL	RQL
Compounds	Sample Conc µg/Kg	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS	average sample result	(mean result/sample conc)*100	std. dev. sample results	(std.dev/mean result)*100	std.dev. *3.14	MDL*4
ACID BLUE 45	3444	1705.5	2232.5	2278.5	2280.0	2274.0	1890.0	1824.0	2069.2	60.1	252.1	12.2	791.6	3166.5
ACID YELLOW 23	2812	2062.0	1879.5	1775.5	1657.0	1790.5	1746.0	1768.0	1811.2	64.4	128.6	7.1	403.7	1614.7
ACID YELLOW 73	3510	2215.5	2346.5	2302.5	2190.0	2284.0	2245.5	2206.5	2255.8	64.3	57.3	2.5	179.9	719.6
ACID ORANGE 10	3074	6243.0	6420.0	6294.0	5915.5	6205.5	6079.5	6074.5	6176.0	200.9	166.5	2.7	522.7	2091.0
ACID BLUE 1	3692	593.0	640.0	279.0	415.5	542.5	783.5	742.5	570.9	15.5	178.0	31.2	558.9	2235.5
BASIC YELLOW 2	2642	12632.5	13740.0	13636.0	12878.5	13498.0	12885.0	13076.0	13192.3	499.3	430.1	3.3	1350.6	5402.5
DISPERSE VIOLET 1	2602	1272.5	1684.5	1402.5	1529.5	1582.5	1305.0	1439.0	1459.4	56.1	149.0	10.2	467.8	1871.1
SOLVENT YELLOW 33	2796	984.5	1110.5	1104.0	1051.0	1097.5	1063.0	1078.5	1069.9	38.3	43.5	4.1	136.5	546.2
DISPERSE BLUE 14	2990	1138.5	1344.5	1509.0	1909.0	1837.5	1586.5	1861.0	1598.0	53.4	290.6	18.2	912.5	3650.0
ACID RED 64	3618	2287.0	2381.0	2467.5	2241.0	2395.5	2301.5	2365.5	2348.4	64.9	76.7	3.3	240.7	962.9
ACID BLUE 9	3280	170.5	208.5	255.5	211.0	232.5	129.5	152.0	194.2	5.9	45.2	23.3	141.8	567.3
BASIC VIOLET 10	2084	2075.5	2167.0	2057.5	1982.0	2055.5	2024.5	2052.0	2059.1	98.8	56.4	2.7	177.2	709.0
2-AMINOANTHRAQUINONE	3738	1614.5	1616.0	1698.5	1613.5	1687.0	1619.5	1575.5	1632.1	43.7	44.2	2.7	138.7	554.6
SOLVENT ORANGE 3	2558	1332.5	1343.0	1327.5	1301.5	1367.0	1318.5	1320.0	1330.0	52.0	20.8	1.6	65.3	261.2
1-AMINOANTHRAQUINONE	2570	1856.5	2255.0	2230.5	2145.5	2229.5	2120.5	2137.0	2139.2	83.2	135.5	6.3	425.6	1702.3
SOLVENT YELLOW 14	2832	1197.5	1213.5	1190.5	1148.5	1227.5	1214.5	1208.0	1200.0	42.4	25.7	2.1	80.7	322.7
SOLVENT ORANGE 7	2880	1814.5	2383.5	2408.5	1939.0	1979.5	2345.0	2370.0	2177.1	75.6	254.5	11.7	799.3	3197.1
ACID YELLOW 3	3348	983.0	1035.5	1020.5	1118.5	1016.0	978.5	1052.5	1029.2	30.7	47.5	4.6	149.0	596.0
SOLVENT YELLOW 3	3020	6320.0	6654.5	6670.0	6443.5	6585.5	6594.5	6569.0	6548.1	216.8	124.6	1.9	391.2	1564.9
SOLVENT RED 1	4638	2120.5	2208.0	3286.5	3967.5	2347.5	2151.0	2109.5	2598.6	56.0	733.8	28.2	2304.2	9216.6
DISPERSE RED 9	3060	1208.5	1393.0	1328.0	1321.5	1411.0	1383.0	1355.0	1342.9	43.9	67.9	5.1	213.1	852.6
SOLVENT YELLOW 2	3940	1599.0	1769.5	1760.5	1714.0	1780.0	1727.5	1743.5	1727.7	43.9	61.3	3.5	192.5	770.2
SOLVENT GREEN 3	3140	1770.0	1476.5	1909.5	1942.0	1369.5	1284.0	935.0	1526.6	48.6	368.4	24.1	1156.9	4627.5
SOLVENT RED 24	568	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ANTHRACENE	4154	1849.0	2170.0	2129.0	2021.5	2110.5	2029.0	2087.0	2056.6	49.5	105.7	5.1	331.9	1327.5
BENZANTHRONE	2432	2465.5	2617.5	2569.0	2532.0	2603.0	2537.0	2619.0	2563.3	105.4	56.1	2.2	176.2	704.6

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Method Detection Limit						
T (STUDENT'S T @ 99% FOR 6 DF)	3.140						
MDL AB45	1813.3						
MDL AY23	621.6						
MDL AY73	604.0						
MDL AO10	944.8						
MDL AB1	0.0						
MDL BY2	5590.1						
MDL DV1	708.1						
MDL SY33	392.7						
MDL DB14	1022.7						
MDL AR64	794.5						
MDL AB9	177.1						
MDL BV10	455.2						
MDL 2AQ	998.7						
MDL SO3	348.5						
MDL 1AQ	669.4						
MDL SY14	421.5						
MDL SO7	914.4						
MDL AY3	0.0						
MDL SY3	945.4						
MDL SR1	1740.1						
MDL DR9	492.9						
MDL SY2	626.3						
MDL SG3	13403.2						
MDL SR24	0.0						
MDL AN	562.6						
MDL BZ	519.1						

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
	Stock Standard Concentrations						
ACID BLUE 45 STD	344.4	344.4	344.4	344.4	344.4	344.4	344.4
ACID YELLOW 23 STD	281.2	281.2	281.2	281.2	281.2	281.2	281.2
ACID YELLOW 73 STD	351.0	351.0	351.0	351.0	351.0	351.0	351.0
ACID ORANGE 10 STD	307.4	307.4	307.4	307.4	307.4	307.4	307.4
ACID BLUE 1 STD	369.2	369.2	369.2	369.2	369.2	369.2	369.2
BASIC YELLOW 2 STD	264.2	264.2	264.2	264.2	264.2	264.2	264.2
DISPERSE VIOLET 1 STD	260.2	260.2	260.2	260.2	260.2	260.2	260.2
SOLVENT YELLOW 33 STD	279.6	279.6	279.6	279.6	279.6	279.6	279.6
DISPERSE BLUE 14 STD	299.0	299.0	299.0	299.0	299.0	299.0	299.0
ACID RED 64 STD	361.8	361.8	361.8	361.8	361.8	361.8	361.8
ACID BLUE 9 STD	328.0	328.0	328.0	328.0	328.0	328.0	328.0
BASIC VIOLET 10 STD	208.4	208.4	208.4	208.4	208.4	208.4	208.4
2-AMINOANTHRAQUINONE STD	373.8	373.8	373.8	373.8	373.8	373.8	373.8
SOLVENT ORANGE 3 STD	255.8	255.8	255.8	255.8	255.8	255.8	255.8
1-AMINOANTHRAQUINONE STD	257.0	257.0	257.0	257.0	257.0	257.0	257.0
SOLVENT YELLOW 14 STD	283.2	283.2	283.2	283.2	283.2	283.2	283.2
SOLVENT ORANGE 7 STD	288.0	288.0	288.0	288.0	288.0	288.0	288.0
ACID YELLOW 3 STD	334.8	334.8	334.8	334.8	334.8	334.8	334.8
SOLVENT YELLOW 3 STD	302.0	302.0	302.0	302.0	302.0	302.0	302.0
SOLVENT RED 1 STD	463.8	463.8	463.8	463.8	463.8	463.8	463.8
DISPERSE RED 9 STD	306.0	306.0	306.0	306.0	306.0	306.0	306.0
SOLVENT YELLOW 2 STD	394.0	394.0	394.0	394.0	394.0	394.0	394.0
SOLVENT GREEN 3 STD	314.0	314.0	314.0	314.0	314.0	314.0	314.0
SOLVENT RED 24 STD	56.8	56.8	56.8	56.8	56.8	56.8	56.8
ANTHRACENE	415.4	415.4	415.4	415.4	415.4	415.4	415.4
BENZANTHRONE	243.2	243.2	243.2	243.2	243.2	243.2	243.2

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
	Weight of Stock Standard Spiked						
SPIKE VOLUME EACH DYE	100.0	100.0	100.0	100.0	100.0	100.0	100.0
AB45 SPIKE WEIGHT	34.4	34.4	34.4	34.4	34.4	34.4	34.4
AY23 SPIKE WEIGHT	28.1	28.1	28.1	28.1	28.1	28.1	28.1
AY73 SPIKE WEIGHT	35.1	35.1	35.1	35.1	35.1	35.1	35.1
AO10 SPIKE WEIGHT	30.7	30.7	30.7	30.7	30.7	30.7	30.7
AB1 SPIKE WEIGHT	36.9	36.9	36.9	36.9	36.9	36.9	36.9
BY2 SPIKE WEIGHT	26.4	26.4	26.4	26.4	26.4	26.4	26.4
DV1 SPIKE WEIGHT	26.0	26.0	26.0	26.0	26.0	26.0	26.0
SY33 SPIKE WEIGHT	28.0	28.0	28.0	28.0	28.0	28.0	28.0
DB14 SPIKE WEIGHT	29.9	29.9	29.9	29.9	29.9	29.9	29.9
AR64 SPIKE WEIGHT	36.2	36.2	36.2	36.2	36.2	36.2	36.2
AB9 SPIKE WEIGHT	32.8	32.8	32.8	32.8	32.8	32.8	32.8
BV10 SPIKE WEIGHT	20.8	20.8	20.8	20.8	20.8	20.8	20.8
2AQ SPIKE WEIGHT	37.4	37.4	37.4	37.4	37.4	37.4	37.4
SO3 SPIKE WEIGHT	25.6	25.6	25.6	25.6	25.6	25.6	25.6
1AQ SPIKE WEIGHT	25.7	25.7	25.7	25.7	25.7	25.7	25.7
SY14 SPIKE WEIGHT	28.3	28.3	28.3	28.3	28.3	28.3	28.3
SO7 SPIKE WEIGHT	28.8	28.8	28.8	28.8	28.8	28.8	28.8
AY3 SPIKE WEIGHT	33.5	33.5	33.5	33.5	33.5	33.5	33.5
SY3 SPIKE WEIGHT	30.2	30.2	30.2	30.2	30.2	30.2	30.2
SR1 SPIKE WEIGHT	46.4	46.4	46.4	46.4	46.4	46.4	46.4
DR9 SPIKE WEIGHT	30.6	30.6	30.6	30.6	30.6	30.6	30.6
SY2 SPIKE WEIGHT	39.4	39.4	39.4	39.4	39.4	39.4	39.4
SG3 SPIKE WEIGHT	31.4	31.4	31.4	31.4	31.4	31.4	31.4
SR24 SPIKE WEIGHT	5.7	5.7	5.7	5.7	5.7	5.7	5.7
AN SPIKE WEIGHT	41.5	41.5	41.5	41.5	41.5	41.5	41.5
BZ SPIKE WEIGHT	24.3	24.3	24.3	24.3	24.3	24.3	24.3

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
	Weight Standard per Volume Sample						
MATRIX VOLUME (WATER)	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB45	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY23	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY73	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AO10	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BY2	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DV1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY33	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DB14	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AR64	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB9	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BV10	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 2AQ	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 1AQ	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY14	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO7	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DR9	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY2	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SG3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR24	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AN	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BZ	NA	NA	NA	NA	NA	NA	NA

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
SPIKE CONC AB45	3411.73	3437.98	3439.32	3438.67	3429.32	3433.60	3415.28
SPIKE CONC AY23	2785.65	2807.09	2808.18	2807.65	2800.02	2803.51	2788.55
SPIKE CONC AY73	3477.11	3503.87	3505.23	3504.57	3495.04	3499.40	3480.73
SPIKE CONC AO10	3045.19	3068.63	3069.83	3069.24	3060.90	3064.71	3048.36
SPIKE CONC AB1	3657.40	3685.55	3686.99	3686.29	3676.27	3680.85	3661.21
SPIKE CONC BY2	2617.24	2637.38	2638.41	2637.91	2630.74	2634.02	2619.97
SPIKE CONC DV1	2577.62	2597.45	2598.47	2597.97	2590.91	2594.14	2580.30
SPIKE CONC SY33	2769.80	2791.12	2792.20	2791.67	2784.08	2787.55	2772.68
SPIKE CONC DB14	2961.98	2984.78	2985.94	2985.37	2977.26	2980.97	2965.06
SPIKE CONC AR64	3584.09	3611.68	3613.09	3612.40	3602.58	3607.07	3587.83
SPIKE CONC AB9	3249.26	3274.27	3275.55	3274.92	3266.02	3270.09	3252.65
SPIKE CONC BV10	2064.47	2080.36	2081.17	2080.77	2075.12	2077.70	2066.62
SPIKE CONC 2AQ	3702.97	3731.47	3732.92	3732.22	3722.07	3726.71	3706.83
SPIKE CONC SO3	2534.03	2553.53	2554.53	2554.04	2547.10	2550.27	2536.67
SPIKE CONC 1AQ	2545.92	2565.51	2566.51	2566.02	2559.05	2562.24	2548.57
SPIKE CONC SY14	2805.46	2827.05	2828.15	2827.62	2819.93	2823.44	2808.38
SPIKE CONC SO7	2853.01	2874.97	2876.09	2875.54	2867.73	2871.30	2855.98
SPIKE CONC AY3	3316.62	3342.15	3343.45	3342.82	3333.73	3337.89	3320.08
SPIKE CONC SY3	2991.70	3014.72	3015.90	3015.33	3007.13	3010.88	2994.81
SPIKE CONC SR1	4594.54	4629.90	4631.70	4630.82	4618.23	4623.99	4599.32
SPIKE CONC DR9	3031.32	3054.65	3055.84	3055.26	3046.96	3050.76	3034.48
SPIKE CONC SY2	3903.08	3933.12	3934.65	3933.90	3923.21	3928.10	3907.14
SPIKE CONC SG3	3110.57	3134.51	3135.74	3135.14	3126.62	3130.51	3113.81
SPIKE CONC SR24	562.68	567.01	567.23	567.12	565.58	566.28	563.26
SPIKE CONC AN	4115.07	4146.74	4148.36	4147.57	4136.30	4141.45	4119.36
SPIKE CONC BZ	2409.21	2427.75	2428.70	2428.24	2421.64	2424.65	2411.72

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
	Results from HPLC						
HPLC RESULT AB45	3.411	4.465	4.557	4.560	4.548	3.780	3.648
HPLC RESULT AY23	4.124	3.759	3.551	3.314	3.581	3.492	3.536
HPLC RESULT AY73	4.431	4.693	4.605	4.380	4.568	4.491	4.413
HPLC RESULT AO10	12.486	12.840	12.588	11.831	12.411	12.159	12.149
HPLC RESULT AB1	1.186	1.280	0.558	0.831	1.085	1.567	1.485
HPLC RESULT BY2	25.265	27.480	27.272	25.757	26.996	25.770	26.152
HPLC RESULT DV1	2.545	3.369	2.805	3.059	3.165	2.610	2.878
HPLC RESULT SY33	1.969	2.221	2.208	2.102	2.195	2.126	2.157
HPLC RESULT DB14	2.277	2.689	3.018	3.818	3.675	3.173	3.722
HPLC RESULT AR64	4.574	4.762	4.935	4.482	4.791	4.603	4.731
HPLC RESULT AB9	0.341	0.417	0.511	0.422	0.465	0.259	0.304
HPLC RESULT BV10	4.151	4.334	4.115	3.964	4.111	4.049	4.104
HPLC RESULT 2AQ	3.229	3.232	3.397	3.227	3.374	3.239	3.151
HPLC RESULT SO3	2.665	2.686	2.655	2.603	2.734	2.637	2.640
HPLC RESULT 1AQ	3.713	4.510	4.461	4.291	4.459	4.241	4.274
HPLC RESULT SY14	2.395	2.427	2.381	2.297	2.455	2.429	2.416
HPLC RESULT SO7	3.629	4.767	4.817	3.878	3.959	4.690	4.740
HPLC RESULT AY3	1.966	2.071	2.041	2.237	2.032	1.957	2.105
HPLC RESULT SY3	12.640	13.309	13.340	12.887	13.171	13.189	13.138
HPLC RESULT SR1	4.241	4.416	6.573	7.935	4.695	4.302	4.219
HPLC RESULT DR9	2.417	2.786	2.656	2.643	2.822	2.766	2.710
HPLC RESULT SY2	3.198	3.539	3.521	3.428	3.560	3.455	3.487
HPLC RESULT SG3	3.540	2.953	3.819	3.884	2.739	2.568	1.870
HPLC RESULT SR24	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT AN	3.698	4.34	4.258	4.043	4.221	4.058	4.174
HPLC RESULT BZ	4.931	5.235	5.138	5.064	5.206	5.074	5.238

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
	Final Sample Results						
CF (STD INJ VOL VS SAM INJ VOL)	2	2	2	2	2	2	2
FINAL VOLUME	10.0946000	10.0175000	10.0136000	10.0155000	10.0428000	10.0303000	10.0841000
CORRECTED RESULT AB45	1705.50	2232.50	2278.50	2280.00	2274.00	1890.00	1824.00
CORRECTED RESULT AY23	2062.00	1879.50	1775.50	1657.00	1790.50	1746.00	1768.00
CORRECTED RESULT AY73	2215.50	2346.50	2302.50	2190.00	2284.00	2245.50	2206.50
CORRECTED RESULT AO10	6243.00	6420.00	6294.00	5915.50	6205.50	6079.50	6074.50
CORRECTED RESULT AB1	593.00	640.00	279.00	415.50	542.50	783.50	742.50
CORRECTED RESULT BY2	12632.50	13740.00	13636.00	12878.50	13498.00	12885.00	13076.00
CORRECTED RESULT DV1	1272.50	1684.50	1402.50	1529.50	1582.50	1305.00	1439.00
CORRECTED RESULT SY33	984.50	1110.50	1104.00	1051.00	1097.50	1063.00	1078.50
CORRECTED RESULT DB14	1138.50	1344.50	1509.00	1909.00	1837.50	1586.50	1861.00
CORRECTED RESULT AR64	2287.00	2381.00	2467.50	2241.00	2395.50	2301.50	2365.50
CORRECTED RESULT AB9	170.50	208.50	255.50	211.00	232.50	129.50	152.00
CORRECTED RESULT BV10	2075.50	2167.00	2057.50	1982.00	2055.50	2024.50	2052.00
CORRECTED RESULT 2AQ	1614.50	1616.00	1698.50	1613.50	1687.00	1619.50	1575.50
CORRECTED RESULT SO3	1332.50	1343.00	1327.50	1301.50	1367.00	1318.50	1320.00
CORRECTED RESULT 1AQ	1856.50	2255.00	2230.50	2145.50	2229.50	2120.50	2137.00
CORRECTED RESULT SY14	1197.50	1213.50	1190.50	1148.50	1227.50	1214.50	1208.00
CORRECTED RESULT SO7	1814.50	2383.50	2408.50	1939.00	1979.50	2345.00	2370.00
CORRECTED RESULT AY3	983.00	1035.50	1020.50	1118.50	1016.00	978.50	1052.50
CORRECTED RESULT SY3	6320.00	6654.50	6670.00	6443.50	6585.50	6594.50	6569.00
CORRECTED RESULT SR1	2120.50	2208.00	3286.50	3967.50	2347.50	2151.00	2109.50
CORRECTED RESULT DR9	1208.50	1393.00	1328.00	1321.50	1411.00	1383.00	1355.00
CORRECTED RESULT SY2	1599.00	1769.50	1760.50	1714.00	1780.00	1727.50	1743.50
CORRECTED RESULT SG3	1770.00	1476.50	1909.50	1942.00	1369.50	1284.00	935.00
CORRECTED RESULT SR24	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT AN	1849.00	2170.00	2129.00	2021.50	2110.50	2029.00	2087.00
CORRECTED RESULT BZ	2465.50	2617.50	2569.00	2532.00	2603.00	2537.00	2619.00

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
	Percent Recoveries						
% RECOVERY AB45	49.99	64.94	66.25	66.30	66.31	55.04	53.41
% RECOVERY AY23	74.02	66.96	63.23	59.02	63.95	62.28	63.40
% RECOVERY AY73	63.72	66.97	65.69	62.49	65.35	64.17	63.39
% RECOVERY AO10	205.01	209.21	205.03	192.73	202.73	198.37	199.27
% RECOVERY AB1	16.21	17.37	7.57	11.27	14.76	21.29	20.28
% RECOVERY BY2	482.66	520.97	516.83	488.21	513.09	489.18	499.09
% RECOVERY DV1	49.37	64.85	53.97	58.87	61.08	50.31	55.77
% RECOVERY SY33	35.54	39.79	39.54	37.65	39.42	38.13	38.90
% RECOVERY DB14	38.44	45.05	50.54	63.95	61.72	53.22	62.76
% RECOVERY AR64	63.81	65.93	68.29	62.04	66.49	63.81	65.93
% RECOVERY AB9	5.25	6.37	7.80	6.44	7.12	3.96	4.67
% RECOVERY BV10	100.53	104.16	98.86	95.25	99.05	97.44	99.29
% RECOVERY 2AQ	43.60	43.31	45.50	43.23	45.32	43.46	42.50
% RECOVERY SO3	52.58	52.59	51.97	50.96	53.67	51.70	52.04
% RECOVERY 1AQ	72.92	87.90	86.91	83.61	87.12	82.76	83.85
% RECOVERY SY14	42.68	42.92	42.09	40.62	43.53	43.01	43.01
% RECOVERY SO7	63.60	82.91	83.74	67.43	69.03	81.67	82.98
% RECOVERY AY3	29.64	30.98	30.52	33.46	30.48	29.31	31.70
% RECOVERY SY3	211.25	220.73	221.16	213.69	219.00	219.02	219.35
% RECOVERY SR1	46.15	47.69	70.96	85.68	50.83	46.52	45.87
% RECOVERY DR9	39.87	45.60	43.46	43.25	46.31	45.33	44.65
% RECOVERY SY2	40.97	44.99	44.74	43.57	45.37	43.98	44.62
% RECOVERY SG3	56.90	47.10	60.89	61.94	43.80	41.02	30.03
% RECOVERY SR24	0.00	0.00	0.00	0.00	0.00	0.00	0.00
% RECOVERY AN	44.93	52.33	51.32	48.74	51.02	48.99	50.66
% RECOVERY BZ	102.34	107.82	105.78	104.27	107.49	104.63	108.59

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
	Square of Corrected Results						
N	7	7	7	7	7	7	7
Xi^2 AB45	2908730.3	4984056.3	5191562.3	5198400.0	5171076.0	3572100.0	3326976.0
Xi^2 AY23	4251844.0	3532520.3	3152400.3	2745649.0	3205890.3	3048516.0	3125824.0
Xi^2 AY73	4908440.3	5506062.3	5301506.3	4796100.0	5216656.0	5042270.3	4868642.3
Xi^2 AO10	3.898E+07	4.122E+07	3.961E+07	3.499E+07	3.851E+07	3.696E+07	3.690E+07
Xi^2 AB1	351649.0	409600.0	77841.0	172640.3	294306.3	613872.3	551306.3
Xi^2 BY2	1.596E+08	1.888E+08	1.859E+08	1.659E+08	1.822E+08	1.660E+08	1.710E+08
Xi^2 DV1	1619256.3	2837540.3	1967006.3	2339370.3	2504306.3	1703025.0	2070721.0
Xi^2 SY33	969240.3	1233210.3	1218816.0	1104601.0	1204506.3	1129969.0	1163162.3
Xi^2 DB14	1296182.3	1807680.3	2277081.0	3644281.0	3376406.3	2516982.3	3463321.0
Xi^2 AR64	5230369.0	5669161.0	6088556.3	5022081.0	5738420.3	5296902.3	5595590.3
Xi^2 AB9	29070.3	43472.3	65280.3	44521.0	54056.3	16770.3	23104.0
Xi^2 BV10	4307700.3	4695889.0	4233306.3	3928324.0	4225080.3	4098600.3	4210704.0
Xi^2 2AQ	2606610.3	2611456.0	2884902.3	2603382.3	2845969.0	2622780.3	2482200.3
Xi^2 SO3	1775556.3	1803649.0	1762256.3	1693902.3	1868689.0	1738442.3	1742400.0
Xi^2 1AQ	3446592.3	5085025.0	4975130.3	4603170.3	4970670.3	4496520.3	4566769.0
Xi^2 SY14	1434006.3	1472582.3	1417290.3	1319052.3	1506756.3	1475010.3	1459264.0
Xi^2 SO7	3292410.3	5681072.3	5800872.3	3759721.0	3918420.3	5499025.0	5616900.0
Xi^2 AY3	966289.0	1072260.3	1041420.3	1251042.3	1032256.0	957462.3	1107756.3
Xi^2 SY3	39942400.0	44282370.3	44488900.0	41518692.3	43368810.3	43487430.3	43151761.0
Xi^2 SR1	4496520.3	4875264.0	10801082.3	15741056.3	5510756.3	4626801.0	4449990.3
Xi^2 DR9	1460472.3	1940449.0	1763584.0	1746362.3	1990921.0	1912689.0	1836025.0
Xi^2 SY2	2556801.0	3131130.3	3099360.3	2937796.0	3168400.0	2984256.3	3039792.3
Xi^2 SG3	3132900.0	2180052.3	3646190.3	3771364.0	1875530.3	1648656.0	874225.0
Xi^2 SR24	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 AN	3418801.0	4708900.0	4532641.0	4086462.3	4454210.3	4116841.0	4355569.0
Xi^2 BZ	6078690.3	6851306.3	6599761.0	6411024.0	6775609.0	6436369.0	6859161.0

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
	Sum of the Square of Corrected Results						
E Xi^2 AB45	3.035E+07						
E Xi^2 AY23	2.306E+07						
E Xi^2 AY73	3.564E+07						
E Xi^2 AO10	2.672E+08						
E Xi^2 AB1	2.471E+06						
E Xi^2 BY2	1.219E+09						
E Xi^2 DV1	1.504E+07						
E Xi^2 SY33	8.024E+06						
E Xi^2 DB14	1.838E+07						
E Xi^2 AR64	3.864E+07						
E Xi^2 AB9	2.763E+05						
E Xi^2 BV10	2.970E+07						
E Xi^2 2AQ	1.866E+07						
E Xi^2 SO3	1.238E+07						
E Xi^2 1AQ	3.214E+07						
E Xi^2 SY14	1.008E+07						
E Xi^2 SO7	3.357E+07						
E Xi^2 AY3	7.428E+06						
E Xi^2 SY3	3.002E+08						
E Xi^2 SR1	5.050E+07						
E Xi^2 DR9	1.265E+07						
E Xi^2 SY2	2.092E+07						
E Xi^2 SG3	1.713E+07						
E Xi^2 SR24	0.000E+00						
E Xi^2 AN	2.967E+07						
E Xi^2 BZ	4.601E+07						

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
	Sum of the Corrected Results Squared						
(E Xi)^2 AB45	2.098E+08						
(E Xi)^2 AY23	1.607E+08						
(E Xi)^2 AY73	2.493E+08						
(E Xi)^2 AO10	1.869E+09						
(E Xi)^2 AB1	1.597E+07						
(E Xi)^2 BY2	8.528E+09						
(E Xi)^2 DV1	1.044E+08						
(E Xi)^2 SY33	5.609E+07						
(E Xi)^2 DB14	1.251E+08						
(E Xi)^2 AR64	2.702E+08						
(E Xi)^2 AB9	1.848E+06						
(E Xi)^2 BV10	2.078E+08						
(E Xi)^2 2AQ	1.305E+08						
(E Xi)^2 SO3	8.668E+07						
(E Xi)^2 1AQ	2.242E+08						
(E Xi)^2 SY14	7.056E+07						
(E Xi)^2 SO7	2.323E+08						
(E Xi)^2 AY3	5.190E+07						
(E Xi)^2 SY3	2.101E+09						
(E Xi)^2 SR1	3.309E+08						
(E Xi)^2 DR9	8.836E+07						
(E Xi)^2 SY2	1.463E+08						
(E Xi)^2 SG3	1.142E+08						
(E Xi)^2 SR24	0.000E+00						
(E Xi)^2 AN	2.072E+08						
(E Xi)^2 BZ	3.220E+08						

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
				Variance			
S^2: VARIANCE AB45	63561.1						
S^2: VARIANCE AY23	16527.2						
S^2: VARIANCE AY73	3282.2						
S^2: VARIANCE AO10	27715.7						
S^2: VARIANCE AB1	31678.3						
S^2: VARIANCE BY2	185017.2						
S^2: VARIANCE DV1	22193.7						
S^2: VARIANCE SY33	1890.8						
S^2: VARIANCE DB14	84451.0						
S^2: VARIANCE AR64	5877.1						
S^2: VARIANCE AB9	2040.0						
S^2: VARIANCE BV10	3186.5						
S^2: VARIANCE 2AQ	1950.0						
S^2: VARIANCE SO3	432.5						
S^2: VARIANCE 1AQ	18368.8						
S^2: VARIANCE SY14	660.3						
S^2: VARIANCE SO7	64794.0						
S^2: VARIANCE AY3	2252.0						
S^2: VARIANCE SY3	15523.3						
S^2: VARIANCE SR1	538476.2						
S^2: VARIANCE DR9	4607.6						
S^2: VARIANCE SY2	3759.9						
S^2: VARIANCE SG3	135741.5						
S^2: VARIANCE SR24	0.0						
S^2: VARIANCE AN	11170.4						
S^2: VARIANCE BZ	3147.5						

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
	Standard Deviation						
STD DEVIATION AB45	252.1						
STD DEVIATION AY23	128.6						
STD DEVIATION AY73	57.3						
STD DEVIATION AO10	166.5						
STD DEVIATION AB1	178.0						
STD DEVIATION BY2	430.1						
STD DEVIATION DV1	149.0						
STD DEVIATION SY33	43.5						
STD DEVIATION DB14	290.6						
STD DEVIATION AR64	76.7						
STD DEVIATION AB9	45.2						
STD DEVIATION BV10	56.4						
STD DEVIATION 2AQ	44.2						
STD DEVIATION SO3	20.8						
STD DEVIATION 1AQ	135.5						
STD DEVIATION SY14	25.7						
STD DEVIATION SO7	254.5						
STD DEVIATION AY3	47.5						
STD DEVIATION SY3	124.6						
STD DEVIATION SR1	733.8						
STD DEVIATION DR9	67.9						
STD DEVIATION SY2	61.3						
STD DEVIATION SG3	368.4						
STD DEVIATION SR24	0.0						
STD DEVIATION AN	105.7						
STD DEVIATION BZ	56.1						

	S001LS	S002LS	S003LS	S004LS	S005LS	S006LS	S007LS
	Method Detection Limit						
T (STUDENT'S T @ 99% FOR 6 DF)	3.140						
MDL AB45	791.6						
MDL AY23	403.7						
MDL AY73	179.9						
MDL AO10	522.7						
MDL AB1	558.9						
MDL BY2	1350.6						
MDL DV1	467.8						
MDL SY33	136.5						
MDL DB14	912.5						
MDL AR64	240.7						
MDL AB9	141.8						
MDL BV10	177.2						
MDL 2AQ	138.7						
MDL SO3	65.3						
MDL 1AQ	425.6						
MDL SY14	80.7						
MDL SO7	799.3						
MDL AY3	149.0						
MDL SY3	391.2						
MDL SR1	2304.2						
MDL DR9	213.1						
MDL SY2	192.5						
MDL SG3	1156.9						
MDL SR24	0.0						
MDL AN	331.9						
MDL BZ	176.2						

TABLE VII - 4A

PERCENT RECOVERIES
SOIL METHOD
HIGH SPIKE, SHORT GRADIENT

Compounds		Ret. Time	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
% RECOVERY AB45	%	4.439	41	61	62	57	57	63	55
% RECOVERY AR64	%	6.174	27	27	27	27	26	27	26
% RECOVERY AY3	%	7.322	7	6	6	6	6	7	7
% RECOVERY AY23	%	7.525	34	35	34	34	32	34	33
% RECOVERY AY73	%	9.159	36	36	35	35	33	36	35
% RECOVERY AO10	%	9.946	106	110	106	106	101	108	104
% RECOVERY AB9	%	11.576	4	5	5	5	4	4	3
% RECOVERY DV1	%	12.69	41	37	36	39	35	38	40
% RECOVERY AB1	%	12.709	2	2	1	2	2	5	7
% RECOVERY 2AQ	%	13.246	24	25	24	24	22	24	23
% RECOVERY SO3	%	13.811	29	29	28	28	27	29	28
% RECOVERY SY33	%	15.498	20	21	20	20	19	20	20
% RECOVERY 1AQ	%	16.108	47	49	48	48	45	48	48
% RECOVERY DB14	%	18.857	32	40	41	34	32	35	33
% RECOVERY BY2	%	19.452	284	300	293	293	267	286	278
% RECOVERY BV10	%	19.691	54	56	54	53	51	55	53
% RECOVERY SY3	%	19.962	117	123	119	118	111	120	116
% RECOVERY DR9	%	20.367	23	25	23	24	21	24	24
% RECOVERY BZ	%	20.438	57	60	58	58	54	60	58
% RECOVERY SY2	%	22.244	23	24	23	23	21	23	23
% RECOVERY AN	%	22.537	32	34	32	32	31	33	32
% RECOVERY SY14	%	24.403	23	24	24	23	22	24	23
% RECOVERY SR1	%	24.66	22	22	26	25	22	21	20
% RECOVERY SO7	%	27.804	38	42	48	48	42	41	47
% RECOVERY SG3	%	31.194	30	18	121	99	97	83	57
% RECOVERY SR24	%	31.255	0	0	0	0	0	0	0

TABLE VII-4

Solid Extraction Method, Short Gradient, High Spike

		Sample Results (µg/Kg)							Mean Result µg/Kg	Method Bias (Accuracy) %R	Standard Deviation	Method Precision (%RSD)
Compounds	Sample Conc µg/Kg	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS	average sample result	(mean result/sample conc)*100	std. dev. sample results	(std.dev/mean result)*100
ACID BLUE 45	17220	6982.5	10366.0	10499.0	9734.5	9631.0	10277.0	9404.5	9556.4	55.5	1207.5	12.6
ACID YELLOW 23	14060	4695.0	4804.0	4700.5	4720.5	4406.5	4616.5	4649.5	4656.1	33.1	124.8	2.7
ACID YELLOW 73	17550	6210.5	6265.0	6107.5	6175.0	5749.5	6000.0	6031.0	6076.9	34.6	172.7	2.8
ACID ORANGE 10	15370	16190.5	16636.5	16159.5	16240.0	15205.5	15839.0	15903.0	16024.9	104.3	444.7	2.8
ACID BLUE 1	18460	433.5	371.5	219.5	317.0	295.0	864.0	1366.5	552.4	3.0	416.2	75.3
BASIC YELLOW 2	13210	37221.0	39097.0	38334.5	38639.5	34557.5	35959.5	36438.0	37178.1	281.4	1635.9	4.4
DISPERSE VIOLET 1	13010	5286.0	4706.0	4657.0	5109.0	4456.5	4703.5	5187.0	4872.1	37.4	316.7	6.5
SOLVENT YELLOW 33	13980	2750.0	2840.5	2815.0	2767.0	2576.0	2701.5	2723.5	2739.1	19.6	86.8	3.2
DISPERSE BLUE 14	14950	4813.0	5840.0	6103.5	5134.5	4639.0	5031.5	4921.0	5211.8	34.9	547.7	10.5
ACID RED 64	18090	4858.0	4908.0	4904.0	4873.5	4566.5	4705.5	4741.5	4793.9	26.5	127.7	2.7
ACID BLUE 9	16400	711.0	732.5	732.5	738.5	684.5	622.5	514.0	676.5	4.1	82.4	12.2
BASIC VIOLET 10	10420	5594.0	5710.0	5560.0	5552.5	5219.0	5443.5	5496.5	5510.8	52.9	153.1	2.8
2-AMINOANTHRAQUINONE	18690	4365.5	4674.5	4450.5	4458.5	4026.5	4327.0	4313.0	4373.6	23.4	195.8	4.5
SOLVENT ORANGE 3	12790	3623.0	3601.0	3555.5	3539.0	3323.0	3514.0	3554.5	3530.0	27.6	98.4	2.8
1-AMINOANTHRAQUINONE	12850	6020.5	6272.5	6057.5	6144.5	5622.5	5872.5	6133.5	6017.6	46.8	213.4	3.5
SOLVENT YELLOW 14	14160	3225.5	3421.5	3302.0	3292.0	3041.0	3225.5	3234.0	3248.8	22.9	114.8	3.5
SOLVENT ORANGE 7	14400	5356.5	5939.5	6884.0	6884.5	5957.5	5593.5	6724.0	6191.4	43.0	634.6	10.3
ACID YELLOW 3	16740	1083.0	1010.0	920.5	1062.5	1033.5	1102.5	1208.5	1060.1	6.3	88.6	8.4
SOLVENT YELLOW 3	15100	17475.5	18371.0	17794.5	17696.0	16468.0	17316.5	17351.0	17496.1	115.9	578.1	3.3
SOLVENT RED 1	23190	5011.0	4941.5	5896.0	5840.0	4986.5	4611.0	4608.5	5127.8	22.1	532.7	10.4
DISPERSE RED 9	15300	3494.0	3704.0	3540.5	3591.5	3181.0	3444.0	3602.0	3508.1	22.9	166.6	4.7
SOLVENT YELLOW 2	19700	4505.0	4632.0	4546.5	4477.0	4139.5	4343.0	4406.5	4435.6	22.5	160.5	3.6
SOLVENT GREEN 3	15700	4629.5	2736.5	18881.5	15449.0	14901.0	12414.0	8840.0	11121.6	70.8	5952.0	53.5
SOLVENT RED 24	2840	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ANTHRACENE	20770	6594.5	6908.5	6683.5	6642.5	6223.0	6562.0	6636.0	6607.1	31.8	203.5	3.1
BENZANTHRONE	12160	6873.0	7249.0	7033.0	7032.0	6418.0	6984.0	6945.0	6933.4	57.0	255.4	3.7

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Stock Standard Concentrations						
ACID BLUE 45 STD	344.4	344.4	344.4	344.4	344.4	344.4	344.4
ACID YELLOW 23 STD	281.2	281.2	281.2	281.2	281.2	281.2	281.2
ACID YELLOW 73 STD	351.0	351.0	351.0	351.0	351.0	351.0	351.0
ACID ORANGE 10 STD	307.4	307.4	307.4	307.4	307.4	307.4	307.4
ACID BLUE 1 STD	369.2	369.2	369.2	369.2	369.2	369.2	369.2
BASIC YELLOW 2 STD	264.2	264.2	264.2	264.2	264.2	264.2	264.2
DISPERSE VIOLET 1 STD	260.2	260.2	260.2	260.2	260.2	260.2	260.2
SOLVENT YELLOW 33 STD	279.6	279.6	279.6	279.6	279.6	279.6	279.6
DISPERSE BLUE 14 STD	299.0	299.0	299.0	299.0	299.0	299.0	299.0
ACID RED 64 STD	361.8	361.8	361.8	361.8	361.8	361.8	361.8
ACID BLUE 9 STD	328.0	328.0	328.0	328.0	328.0	328.0	328.0
BASIC VIOLET 10 STD	208.4	208.4	208.4	208.4	208.4	208.4	208.4
2-AMINOANTHRAQUINONE STD	373.8	373.8	373.8	373.8	373.8	373.8	373.8
SOLVENT ORANGE 3 STD	255.8	255.8	255.8	255.8	255.8	255.8	255.8
1-AMINOANTHRAQUINONE STD	257.0	257.0	257.0	257.0	257.0	257.0	257.0
SOLVENT YELLOW 14 STD	283.2	283.2	283.2	283.2	283.2	283.2	283.2
SOLVENT ORANGE 7 STD	288.0	288.0	288.0	288.0	288.0	288.0	288.0
ACID YELLOW 3 STD	334.8	334.8	334.8	334.8	334.8	334.8	334.8
SOLVENT YELLOW 3 STD	302.0	302.0	302.0	302.0	302.0	302.0	302.0
SOLVENT RED 1 STD	463.8	463.8	463.8	463.8	463.8	463.8	463.8
DISPERSE RED 9 STD	306.0	306.0	306.0	306.0	306.0	306.0	306.0
SOLVENT YELLOW 2 STD	394.0	394.0	394.0	394.0	394.0	394.0	394.0
SOLVENT GREEN 3 STD	314.0	314.0	314.0	314.0	314.0	314.0	314.0
SOLVENT RED 24 STD	56.8	56.8	56.8	56.8	56.8	56.8	56.8
ANTHRACENE	415.4	415.4	415.4	415.4	415.4	415.4	415.4
BENZANTHRONE	243.2	243.2	243.2	243.2	243.2	243.2	243.2

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Weight of Stock Standard Spiked						
SPIKE VOLUME EACH DYE	500.0	500.0	500.0	500.0	500.0	500.0	500.0
AB45 SPIKE WEIGHT	172.2	172.2	172.2	172.2	172.2	172.2	172.2
AY23 SPIKE WEIGHT	140.6	140.6	140.6	140.6	140.6	140.6	140.6
AY73 SPIKE WEIGHT	175.5	175.5	175.5	175.5	175.5	175.5	175.5
AO10 SPIKE WEIGHT	153.7	153.7	153.7	153.7	153.7	153.7	153.7
AB1 SPIKE WEIGHT	184.6	184.6	184.6	184.6	184.6	184.6	184.6
BY2 SPIKE WEIGHT	132.1	132.1	132.1	132.1	132.1	132.1	132.1
DV1 SPIKE WEIGHT	130.1	130.1	130.1	130.1	130.1	130.1	130.1
SY33 SPIKE WEIGHT	139.8	139.8	139.8	139.8	139.8	139.8	139.8
DB14 SPIKE WEIGHT	149.5	149.5	149.5	149.5	149.5	149.5	149.5
AR64 SPIKE WEIGHT	180.9	180.9	180.9	180.9	180.9	180.9	180.9
AB9 SPIKE WEIGHT	164.0	164.0	164.0	164.0	164.0	164.0	164.0
BV10 SPIKE WEIGHT	104.2	104.2	104.2	104.2	104.2	104.2	104.2
2AQ SPIKE WEIGHT	186.9	186.9	186.9	186.9	186.9	186.9	186.9
SO3 SPIKE WEIGHT	127.9	127.9	127.9	127.9	127.9	127.9	127.9
1AQ SPIKE WEIGHT	128.5	128.5	128.5	128.5	128.5	128.5	128.5
SY14 SPIKE WEIGHT	141.6	141.6	141.6	141.6	141.6	141.6	141.6
SO7 SPIKE WEIGHT	144.0	144.0	144.0	144.0	144.0	144.0	144.0
AY3 SPIKE WEIGHT	167.4	167.4	167.4	167.4	167.4	167.4	167.4
SY3 SPIKE WEIGHT	151.0	151.0	151.0	151.0	151.0	151.0	151.0
SR1 SPIKE WEIGHT	231.9	231.9	231.9	231.9	231.9	231.9	231.9
DR9 SPIKE WEIGHT	153.0	153.0	153.0	153.0	153.0	153.0	153.0
SY2 SPIKE WEIGHT	197.0	197.0	197.0	197.0	197.0	197.0	197.0
SG3 SPIKE WEIGHT	157.0	157.0	157.0	157.0	157.0	157.0	157.0
SR24 SPIKE WEIGHT	28.4	28.4	28.4	28.4	28.4	28.4	28.4
AN SPIKE WEIGHT	207.7	207.7	207.7	207.7	207.7	207.7	207.7
BZ SPIKE WEIGHT	121.6	121.6	121.6	121.6	121.6	121.6	121.6

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Weight Standard per Volume Sample						
MATRIX VOLUME (WATER)	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB45	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY23	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY73	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AO10	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BY2	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DV1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY33	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DB14	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AR64	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB9	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BV10	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 2AQ	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 1AQ	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY14	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO7	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DR9	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY2	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SG3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR24	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AN	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BZ	NA	NA	NA	NA	NA	NA	NA

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
SPIKE CONC AB45	17069.62	16993.14	17071.31	17172.60	16875.74	16394.54	17097.58
SPIKE CONC AY23	13937.21	13874.77	13938.59	14021.30	13778.91	13386.01	13960.05
SPIKE CONC AY73	17396.73	17318.79	17398.46	17501.70	17199.14	16708.72	17425.24
SPIKE CONC AO10	15235.77	15167.51	15237.28	15327.70	15062.72	14633.22	15260.73
SPIKE CONC AB1	18298.79	18216.81	18300.60	18409.19	18090.94	17575.09	18328.77
SPIKE CONC BY2	13094.64	13035.97	13095.93	13173.64	12945.90	12576.76	13116.09
SPIKE CONC DV1	12896.38	12838.60	12897.66	12974.19	12749.90	12386.35	12917.51
SPIKE CONC SY33	13857.91	13795.83	13859.29	13941.52	13700.51	13309.85	13880.61
SPIKE CONC DB14	14819.44	14753.05	14820.91	14908.85	14651.12	14233.35	14843.72
SPIKE CONC AR64	17932.02	17851.68	17933.80	18040.21	17728.34	17222.83	17961.40
SPIKE CONC AB9	16256.78	16183.94	16258.39	16354.86	16072.13	15613.84	16283.41
SPIKE CONC BV10	10329.00	10282.73	10330.03	10391.32	10211.68	9920.50	10345.92
SPIKE CONC 2AQ	18526.78	18443.78	18528.62	18638.56	18316.35	17794.07	18557.13
SPIKE CONC SO3	12678.30	12621.50	12679.56	12754.80	12534.30	12176.89	12699.07
SPIKE CONC 1AQ	12737.78	12680.71	12739.04	12814.63	12593.10	12234.02	12758.65
SPIKE CONC SY14	14036.34	13973.45	14037.73	14121.03	13876.91	13481.22	14059.34
SPIKE CONC SO7	14274.24	14210.29	14275.66	14360.37	14112.11	13709.72	14297.63
SPIKE CONC AY3	16593.81	16519.47	16595.45	16693.92	16405.33	15937.54	16620.99
SPIKE CONC SY3	14968.13	14901.07	14969.61	15058.44	14798.12	14376.16	14992.65
SPIKE CONC SR1	22987.48	22884.49	22989.76	23126.17	22726.38	22078.35	23025.14
SPIKE CONC DR9	15166.38	15098.44	15167.89	15257.89	14994.12	14566.57	15191.23
SPIKE CONC SY2	19527.96	19440.47	19529.89	19645.78	19306.15	18755.65	19559.95
SPIKE CONC SG3	15562.89	15493.17	15564.43	15656.79	15386.12	14947.40	15588.39
SPIKE CONC SR24	2815.20	2802.59	2815.48	2832.18	2783.22	2703.86	2819.81
SPIKE CONC AN	20588.61	20496.37	20590.66	20712.83	20354.76	19774.36	20622.34
SPIKE CONC BZ	12053.81	11999.80	12055.00	12126.53	11916.90	11577.09	12073.55

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Results from HPLC						
HPLC RESULT AB45	13.965	20.732	20.998	19.469	19.262	20.554	18.809
HPLC RESULT AY23	9.390	9.608	9.401	9.441	8.813	9.233	9.299
HPLC RESULT AY73	12.421	12.530	12.215	12.350	11.499	12.000	12.062
HPLC RESULT AO10	32.381	33.273	32.319	32.480	30.411	31.678	31.806
HPLC RESULT AB1	0.867	0.743	0.439	0.634	0.590	1.728	2.733
HPLC RESULT BY2	74.442	78.194	76.669	77.279	69.115	71.919	72.876
HPLC RESULT DV1	10.572	9.412	9.314	10.218	8.913	9.407	10.374
HPLC RESULT SY33	5.500	5.681	5.630	5.534	5.152	5.403	5.447
HPLC RESULT DB14	9.626	11.680	12.207	10.269	9.278	10.063	9.842
HPLC RESULT AR64	9.716	9.816	9.808	9.747	9.133	9.411	9.483
HPLC RESULT AB9	1.422	1.465	1.465	1.477	1.369	1.245	1.028
HPLC RESULT BV10	11.188	11.420	11.120	11.105	10.438	10.887	10.993
HPLC RESULT 2AQ	8.731	9.349	8.901	8.917	8.053	8.654	8.626
HPLC RESULT SO3	7.246	7.202	7.111	7.078	6.646	7.028	7.109
HPLC RESULT 1AQ	12.041	12.545	12.115	12.289	11.245	11.745	12.267
HPLC RESULT SY14	6.451	6.843	6.604	6.584	6.082	6.451	6.468
HPLC RESULT SO7	10.713	11.879	13.768	13.769	11.915	11.187	13.448
HPLC RESULT AY3	2.166	2.020	1.841	2.125	2.067	2.205	2.417
HPLC RESULT SY3	34.951	36.742	35.589	35.392	32.936	34.633	34.702
HPLC RESULT SR1	10.022	9.883	11.792	11.680	9.973	9.222	9.217
HPLC RESULT DR9	6.988	7.408	7.081	7.183	6.362	6.888	7.204
HPLC RESULT SY2	9.010	9.264	9.093	8.954	8.279	8.686	8.813
HPLC RESULT SG3	9.259	5.473	37.763	30.898	29.802	24.828	17.680
HPLC RESULT SR24	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT AN	13.189	13.817	13.367	13.285	12.446	13.124	13.272
HPLC RESULT BZ	13.746	14.498	14.066	14.064	12.836	13.968	13.890

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Final Sample Results						
CF (STD INJ VOL VS SAM INJ VOL)	2	2	2	2	2	2	2
FINAL VOLUME	10.0881	10.1335000	10.0871000	10.0276000	10.2040000	10.5035000	10.0716000
CORRECTED RESULT AB45	6982.50	10366.00	10499.00	9734.50	9631.00	10277.00	9404.50
CORRECTED RESULT AY23	4695.00	4804.00	4700.50	4720.50	4406.50	4616.50	4649.50
CORRECTED RESULT AY73	6210.50	6265.00	6107.50	6175.00	5749.50	6000.00	6031.00
CORRECTED RESULT AO10	16190.50	16636.50	16159.50	16240.00	15205.50	15839.00	15903.00
CORRECTED RESULT AB1	433.50	371.50	219.50	317.00	295.00	864.00	1366.50
CORRECTED RESULT BY2	37221.00	39097.00	38334.50	38639.50	34557.50	35959.50	36438.00
CORRECTED RESULT DV1	5286.00	4706.00	4657.00	5109.00	4456.50	4703.50	5187.00
CORRECTED RESULT SY33	2750.00	2840.50	2815.00	2767.00	2576.00	2701.50	2723.50
CORRECTED RESULT DB14	4813.00	5840.00	6103.50	5134.50	4639.00	5031.50	4921.00
CORRECTED RESULT AR64	4858.00	4908.00	4904.00	4873.50	4566.50	4705.50	4741.50
CORRECTED RESULT AB9	711.00	732.50	732.50	738.50	684.50	622.50	514.00
CORRECTED RESULT BV10	5594.00	5710.00	5560.00	5552.50	5219.00	5443.50	5496.50
CORRECTED RESULT 2AQ	4365.50	4674.50	4450.50	4458.50	4026.50	4327.00	4313.00
CORRECTED RESULT SO3	3623.00	3601.00	3555.50	3539.00	3323.00	3514.00	3554.50
CORRECTED RESULT 1AQ	6020.50	6272.50	6057.50	6144.50	5622.50	5872.50	6133.50
CORRECTED RESULT SY14	3225.50	3421.50	3302.00	3292.00	3041.00	3225.50	3234.00
CORRECTED RESULT SO7	5356.50	5939.50	6884.00	6884.50	5957.50	5593.50	6724.00
CORRECTED RESULT AY3	1083.00	1010.00	920.50	1062.50	1033.50	1102.50	1208.50
CORRECTED RESULT SY3	17475.50	18371.00	17794.50	17696.00	16468.00	17316.50	17351.00
CORRECTED RESULT SR1	5011.00	4941.50	5896.00	5840.00	4986.50	4611.00	4608.50
CORRECTED RESULT DR9	3494.00	3704.00	3540.50	3591.50	3181.00	3444.00	3602.00
CORRECTED RESULT SY2	4505.00	4632.00	4546.50	4477.00	4139.50	4343.00	4406.50
CORRECTED RESULT SG3	4629.50	2736.50	18881.50	15449.00	14901.00	12414.00	8840.00
CORRECTED RESULT SR24	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT AN	6594.50	6908.50	6683.50	6642.50	6223.00	6562.00	6636.00
CORRECTED RESULT BZ	6873.00	7249.00	7033.00	7032.00	6418.00	6984.00	6945.00

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Percent Recoveries						
% RECOVERY AB45	40.91	61.00	61.50	56.69	57.07	62.69	55.00
% RECOVERY AY23	33.69	34.62	33.72	33.67	31.98	34.49	33.31
% RECOVERY AY73	35.70	36.17	35.10	35.28	33.43	35.91	34.61
% RECOVERY AO10	106.27	109.69	106.05	105.95	100.95	108.24	104.21
% RECOVERY AB1	2.37	2.04	1.20	1.72	1.63	4.92	7.46
% RECOVERY BY2	284.25	299.92	292.72	293.31	266.94	285.92	277.81
% RECOVERY DV1	40.99	36.66	36.11	39.38	34.95	37.97	40.15
% RECOVERY SY33	19.84	20.59	20.31	19.85	18.80	20.30	19.62
% RECOVERY DB14	32.48	39.59	41.18	34.44	31.66	35.35	33.15
% RECOVERY AR64	27.09	27.49	27.35	27.01	25.76	27.32	26.40
% RECOVERY AB9	4.37	4.53	4.51	4.52	4.26	3.99	3.16
% RECOVERY BV10	54.16	55.53	53.82	53.43	51.11	54.87	53.13
% RECOVERY 2AQ	23.56	25.34	24.02	23.92	21.98	24.32	23.24
% RECOVERY SO3	28.58	28.53	28.04	27.75	26.51	28.86	27.99
% RECOVERY 1AQ	47.26	49.46	47.55	47.95	44.65	48.00	48.07
% RECOVERY SY14	22.98	24.49	23.52	23.31	21.91	23.93	23.00
% RECOVERY SO7	37.53	41.80	48.22	47.94	42.22	40.80	47.03
% RECOVERY AY3	6.53	6.11	5.55	6.36	6.30	6.92	7.27
% RECOVERY SY3	116.75	123.29	118.87	117.52	111.28	120.45	115.73
% RECOVERY SR1	21.80	21.59	25.65	25.25	21.94	20.88	20.02
% RECOVERY DR9	23.04	24.53	23.34	23.54	21.21	23.64	23.71
% RECOVERY SY2	23.07	23.83	23.28	22.79	21.44	23.16	22.53
% RECOVERY SG3	29.75	17.66	121.31	98.67	96.85	83.05	56.71
% RECOVERY SR24	0.00	0.00	0.00	0.00	0.00	0.00	0.00
% RECOVERY AN	32.03	33.71	32.46	32.07	30.57	33.18	32.18
% RECOVERY BZ	57.02	60.41	58.34	57.99	53.86	60.33	57.52

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Square of Corrected Results						
N	7	7	7	7	7	7	7
Xi^2 AB45	48755306.3	1.075E+08	1.102E+08	94760490.3	92756161.0	105616729.0	88444620.3
Xi^2 AY23	22043025.0	23078416.0	22094700.3	22283120.3	19417242.3	21312072.3	21617850.3
Xi^2 AY73	38570310.3	39250225.0	37301556.3	38130625.0	33056750.3	36000000.0	36372961.0
Xi^2 AO10	2.621E+08	2.768E+08	2.611E+08	2.637E+08	2.312E+08	2.509E+08	2.529E+08
Xi^2 AB1	187922.3	138012.3	48180.3	100489.0	87025.0	746496.0	1867322.3
Xi^2 BY2	1.385E+09	1.529E+09	1.470E+09	1.493E+09	1.194E+09	1.293E+09	1.328E+09
Xi^2 DV1	27941796.0	22146436.0	21687649.0	26101881.0	19860392.3	22122912.3	26904969.0
Xi^2 SY33	7562500.0	8068440.3	7924225.0	7656289.0	6635776.0	7298102.3	7417452.3
Xi^2 DB14	23164969.0	34105600.0	37252712.3	26363090.3	21520321.0	25315992.3	24216241.0
Xi^2 AR64	23600164.0	24088464.0	24049216.0	23751002.3	20852922.3	22141730.3	22481822.3
Xi^2 AB9	505521.0	536556.3	536556.3	545382.3	468540.3	387506.3	264196.0
Xi^2 BV10	31292836.0	32604100.0	30913600.0	30830256.3	27237961.0	29631692.3	30211512.3
Xi^2 2AQ	19057590.3	21850950.3	19806950.3	19878222.3	16212702.3	18722929.0	18601969.0
Xi^2 SO3	13126129.0	12967201.0	12641580.3	12524521.0	11042329.0	12348196.0	12634470.3
Xi^2 1AQ	36246420.3	39344256.3	36693306.3	37754880.3	31612506.3	34486256.3	37619822.3
Xi^2 SY14	10403850.3	11706662.3	10903204.0	10837264.0	9247681.0	10403850.3	10458756.0
Xi^2 SO7	28692092.3	35277660.3	47389456.0	47396340.3	35491806.3	31287242.3	45212176.0
Xi^2 AY3	1172889.0	1020100.0	847320.3	1128906.3	1068122.3	1215506.3	1460472.3
Xi^2 SY3	3.054E+08	3.375E+08	3.166E+08	3.131E+08	2.712E+08	2.999E+08	3.011E+08
Xi^2 SR1	25110121.0	24418422.3	34762816.0	34105600.0	24865182.3	21261321.0	21238272.3
Xi^2 DR9	12208036.0	13719616.0	12535140.3	12898872.3	10118761.0	11861136.0	12974404.0
Xi^2 SY2	20295025.0	21455424.0	20670662.3	20043529.0	17135460.3	18861649.0	19417242.3
Xi^2 SG3	21432270.3	7488432.3	356511042.3	238671601.0	222039801.0	154107396.0	78145600.0
Xi^2 SR24	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 AN	43487430.3	47727372.3	44669172.3	44122806.3	38725729.0	43059844.0	44036496.0
Xi^2 BZ	47238129.0	52548001.0	49463089.0	49449024.0	41190724.0	48776256.0	48233025.0

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Sum of the Square of Corrected Results						
E Xi^2 AB45	6.480E+08						
E Xi^2 AY23	1.518E+08						
E Xi^2 AY73	2.587E+08						
E Xi^2 AO10	1.799E+09						
E Xi^2 AB1	3.175E+06						
E Xi^2 BY2	9.692E+09						
E Xi^2 DV1	1.668E+08						
E Xi^2 SY33	5.256E+07						
E Xi^2 DB14	1.919E+08						
E Xi^2 AR64	1.610E+08						
E Xi^2 AB9	3.244E+06						
E Xi^2 BV10	2.127E+08						
E Xi^2 2AQ	1.341E+08						
E Xi^2 SO3	8.728E+07						
E Xi^2 1AQ	2.538E+08						
E Xi^2 SY14	7.396E+07						
E Xi^2 SO7	2.707E+08						
E Xi^2 AY3	7.913E+06						
E Xi^2 SY3	2.145E+09						
E Xi^2 SR1	1.858E+08						
E Xi^2 DR9	8.632E+07						
E Xi^2 SY2	1.379E+08						
E Xi^2 SG3	1.078E+09						
E Xi^2 SR24	0.000E+00						
E Xi^2 AN	3.058E+08						
E Xi^2 BZ	3.369E+08						

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Sum of the Corrected Results Squared						
(E Xi)^2 AB45	4.475E+09						
(E Xi)^2 AY23	1.062E+09						
(E Xi)^2 AY73	1.810E+09						
(E Xi)^2 AO10	1.258E+10						
(E Xi)^2 AB1	1.495E+07						
(E Xi)^2 BY2	6.773E+10						
(E Xi)^2 DV1	1.163E+09						
(E Xi)^2 SY33	3.676E+08						
(E Xi)^2 DB14	1.331E+09						
(E Xi)^2 AR64	1.126E+09						
(E Xi)^2 AB9	2.242E+07						
(E Xi)^2 BV10	1.488E+09						
(E Xi)^2 2AQ	9.373E+08						
(E Xi)^2 SO3	6.106E+08						
(E Xi)^2 1AQ	1.774E+09						
(E Xi)^2 SY14	5.172E+08						
(E Xi)^2 SO7	1.878E+09						
(E Xi)^2 AY3	5.506E+07						
(E Xi)^2 SY3	1.500E+10						
(E Xi)^2 SR1	1.288E+09						
(E Xi)^2 DR9	6.030E+08						
(E Xi)^2 SY2	9.641E+08						
(E Xi)^2 SG3	6.061E+09						
(E Xi)^2 SR24	0.000E+00						
(E Xi)^2 AN	2.139E+09						
(E Xi)^2 BZ	2.356E+09						

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
				Variance			
S^2: VARIANCE AB45	1458088.5						
S^2: VARIANCE AY23	15569.7						
S^2: VARIANCE AY73	29833.6						
S^2: VARIANCE AO10	197783.0						
S^2: VARIANCE AB1	173201.0						
S^2: VARIANCE BY2	2676207.8						
S^2: VARIANCE DV1	100267.2						
S^2: VARIANCE SY33	7533.1						
S^2: VARIANCE DB14	299992.2						
S^2: VARIANCE AR64	16309.5						
S^2: VARIANCE AB9	6782.1						
S^2: VARIANCE BV10	23440.6						
S^2: VARIANCE 2AQ	38341.7						
S^2: VARIANCE SO3	9687.7						
S^2: VARIANCE 1AQ	45544.8						
S^2: VARIANCE SY14	13167.9						
S^2: VARIANCE SO7	402741.7						
S^2: VARIANCE AY3	7842.7						
S^2: VARIANCE SY3	334196.1						
S^2: VARIANCE SR1	283738.4						
S^2: VARIANCE DR9	27750.2						
S^2: VARIANCE SY2	25749.8						
S^2: VARIANCE SG3	35426594.0						
S^2: VARIANCE SR24	0.0						
S^2: VARIANCE AN	41415.5						
S^2: VARIANCE BZ	65204.3						

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Standard Deviation						
STD DEVIATION AB45	1207.5						
STD DEVIATION AY23	124.8						
STD DEVIATION AY73	172.7						
STD DEVIATION AO10	444.7						
STD DEVIATION AB1	416.2						
STD DEVIATION BY2	1635.9						
STD DEVIATION DV1	316.7						
STD DEVIATION SY33	86.8						
STD DEVIATION DB14	547.7						
STD DEVIATION AR64	127.7						
STD DEVIATION AB9	82.4						
STD DEVIATION BV10	153.1						
STD DEVIATION 2AQ	195.8						
STD DEVIATION SO3	98.4						
STD DEVIATION 1AQ	213.4						
STD DEVIATION SY14	114.8						
STD DEVIATION SO7	634.6						
STD DEVIATION AY3	88.6						
STD DEVIATION SY3	578.1						
STD DEVIATION SR1	532.7						
STD DEVIATION DR9	166.6						
STD DEVIATION SY2	160.5						
STD DEVIATION SG3	5952.0						
STD DEVIATION SR24	0.0						
STD DEVIATION AN	203.5						
STD DEVIATION BZ	255.4						

Table VII - 5B

Percent Recoveries Low-Level Aqueous Method Low Spike, Long Gradient

Compounds		Ret. Time	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
% RECOVERY AB45	%	6.439	35	43	22	44	37	52	18
% RECOVERY AY3	%	13.372	312	252	298	312	262	284	284
% RECOVERY AY23	%	14.513	267	220	260	258	216	251	230
% RECOVERY AR64	%	14.9	164	138	159	214	178	131	137
% RECOVERY AY73	%	17.353	229	203	238	245	201	218	203
% RECOVERY AO10	%	18.752	279	222	268	266	223	256	253
% RECOVERY AB9	%	21.657	293	242	287	282	237	277	286
% RECOVERY DV1	%	22.77	221	199	196	194	177	204	187
% RECOVERY AB1	%	23.733	300	188	233	279	238	272	276
% RECOVERY 2AQ	%	23.758	4	0	0	0	0	0	0
% RECOVERY SO3	%	24.783	169	160	155	186	114	218	186
% RECOVERY SY33	%	28.259	264	233	257	200	196	255	246
% RECOVERY 1AQ	%	28.853	246	201	244	221	209	237	230
% RECOVERY DB14	%	34.023	148	121	152	133	115	212	173
% RECOVERY BY2	%	35.367	526	469	468	1084	537	442	1346
% RECOVERY SY3	%	35.998	258	200	233	220	193	251	249
% RECOVERY BV10	%	36.018	282	220	264	239	228	264	270
% RECOVERY DR9	%	36.142	204	168	201	167	152	199	190
% RECOVERY BZ	%	36.724	247	217	250	201	190	248	244
% RECOVERY SY2	%	39.91	169	144	175	135	121	170	162
% RECOVERY AN	%	40.641	154	156	150	4	25	154	174
% RECOVERY SY14	%	44.36	202	178	203	167	73	173	164
% RECOVERY SR1	%	44.811	156	236	182	171	40	222	178
% RECOVERY SO7	%	50.894	220	231	245	183	23	235	232
% RECOVERY SG3	%	57.448	58	54	100	110	0	68	76
% RECOVERY SR24	%	57.994	297	331	180	217	0	345	162

Percent Recovery

TABLE VII - 5A

PERCENT RECOVERIES LOW-LEVEL AQUEOUS METHOD LOW SPIKE, LONG GRADIENT

Compounds		Ret. Time	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS	Average (p)	Std. Deviation (s)	Lower Control Limit (p-2s)	Upper Control Limit (p+2s)	Average (on six points)	Std. Dev (s) (on six points)	Lower Control Limit (p-2s)	Upper Control Limit (p+2s)
% RECOVERY AB45	%	6.439	35	43	22	44	37	52	18	36	12	12	60				
% RECOVERY AY3	%	13.372	312	252	298	312	262	284	284	286	23	240	333				
% RECOVERY AY23	%	14.513	267	220	260	258	216	251	230	243	21	202	284				
% RECOVERY AR64	%	14.9	164	138	159	214	178	131	137	160	29	102	218				
% RECOVERY AY73	%	17.353	229	203	238	245	201	218	203	219	18	183	255				
% RECOVERY AO10	%	18.752	279	222	268	266	223	256	253	252	22	208	297				
% RECOVERY AB9	%	21.657	293	242	287	282	237	277	286	272	23	227	318				
% RECOVERY DV1	%	22.77	221	199	196	194	177	204	187	197	14	169	225				
% RECOVERY AB1	%	23.733	300	188	233	279	238	272	276	255	38	180	331				
% RECOVERY 2AQ	%	23.758	4	0	0	0	0	0	0	1	1	0	4				
% RECOVERY SO3	%	24.783	169	160	155	186	114	218	186	170	32	105	234				
% RECOVERY SY33	%	28.259	264	233	257	200	196	255	246	236	28	181	291				
% RECOVERY 1AQ	%	28.853	246	201	244	221	209	237	230	227	17	192	261				
% RECOVERY DB14	%	34.023	148	121	152	133	115	212	173	151	34	83	218				
% RECOVERY BY2	%	35.367	526	469	468	1084	537	442	1346	696	364	0	1424	588	246	95	1080
% RECOVERY SY3	%	35.998	258	200	233	220	193	251	249	229	26	178	280				
% RECOVERY BV10	%	36.018	282	220	264	239	228	264	270	252	23	206	299				
% RECOVERY DR9	%	36.142	204	168	201	167	152	199	190	183	20	142	224				
% RECOVERY BZ	%	36.724	247	217	250	201	190	248	244	228	25	153	304				
% RECOVERY SY2	%	39.91	169	144	175	135	121	170	162	154	20	113	195				
% RECOVERY AN	%	40.641	154	156	150	4	25	154	174	117	71	0	329	135	55	0	300
% RECOVERY SY14	%	44.36	202	178	203	167	73	173	164	166	44	78	253				
% RECOVERY SR1	%	44.811	156	236	182	171	40	222	178	169	64	42	297				
% RECOVERY SO7	%	50.894	220	231	245	183	23	235	232	196	79	39	353				
% RECOVERY SG3	%	57.448	58	54	100	110	0	68	76	66	36	0	138	78	23	32	123
% RECOVERY SR24	%	57.994	297	331	180	217	0	345	162	219	121	0	460	255	79	97	413

TABLE VII-5

Low-Level Aqueous Extraction Method, Long Gradient, Low Spike

		Sample Results (µg/L)							Mean Result µg/L	Method Bias (Accuracy) %R	Standard Deviation	Method Precision (%RSD)	MDL µg/L	RQL µg/L
Compounds	Sample Conc µg/L	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS	average sample result	(mean result/sample conc)*100	std. dev. sample results	(std.dev/mean result)*100	std.dev. *3.14	MDL*4
ACID BLUE 45	34.44	12.2	14.8	7.7	15.1	12.9	18.0	6.3	12.4	36.1	4.2	33.5	13.1	52.3
ACID YELLOW 23	28.12	75.0	61.9	73.0	72.5	60.6	70.7	64.8	68.4	243.1	5.8	8.5	18.3	73.1
ACID YELLOW 73	35.1	80.2	71.3	83.5	85.9	70.5	76.5	71.3	77.0	219.5	6.3	8.2	19.8	79.3
ACID ORANGE 10	30.74	85.7	68.2	82.3	81.9	68.5	78.7	77.9	77.6	252.5	6.8	8.8	21.4	85.5
ACID BLUE 1	36.92	110.6	69.4	86.1	103.0	88.0	100.3	101.9	94.2	255.1	13.9	14.8	43.7	175.0
BASIC YELLOW 2	26.42	138.8	124.0	123.6	286.5	141.9	116.7	355.5	183.9	695.9	96.2	52.3	302.0	1208.1
DISPERSE VIOLET 1	26.02	57.6	51.8	50.9	50.5	46.0	53.2	48.6	51.2	196.9	3.6	7.1	11.5	45.8
SOLVENT YELLOW 33	27.96	73.8	65.1	71.9	56.0	54.8	71.2	68.7	65.9	235.8	7.7	11.7	24.3	97.0
DISPERSE BLUE 14	29.9	44.3	36.0	45.6	39.8	34.3	63.5	51.8	45.0	150.6	10.1	22.4	31.7	126.8
ACID RED 64	36.18	59.5	50.0	57.5	77.3	64.5	47.5	49.7	58.0	160.3	10.5	18.1	32.9	131.5
ACID BLUE 9	32.8	96.2	79.4	94.1	92.5	77.9	90.8	93.9	89.3	272.1	7.5	8.4	23.4	93.7
BASIC VIOLET 10	20.84	58.7	45.8	55.1	49.9	47.5	55.1	56.2	52.6	252.5	4.9	9.3	15.4	61.4
2-AMINOANTHRAQUINONE	37.38	1.5	0.0	0.0	0.0	0.0	0.0	0.0	0.2	0.6	0.6	264.6	1.7	6.9
SOLVENT ORANGE 3	25.58	43.3	40.9	39.7	47.6	29.2	55.7	47.6	43.4	169.8	8.3	19.0	25.9	103.7
1-AMINOANTHRAQUINONE	25.7	63.2	51.7	62.7	56.9	53.6	60.8	59.2	58.3	226.8	4.4	7.6	13.9	55.6
SOLVENT YELLOW 14	28.32	57.2	50.5	57.5	47.3	20.8	48.9	46.5	47.0	165.8	12.4	26.4	38.9	155.6
SOLVENT ORANGE 7	28.8	63.4	66.6	70.5	52.7	6.7	67.8	66.9	56.4	195.8	22.6	40.1	71.1	284.3
ACID YELLOW 3	33.48	104.5	84.4	99.7	104.6	87.7	94.9	95.2	95.9	286.3	7.8	8.1	24.5	98.1
SOLVENT YELLOW 3	30.2	77.8	60.5	70.4	66.3	58.2	75.7	75.1	69.2	229.0	7.7	11.2	24.2	96.9
SOLVENT RED 1	46.38	72.6	109.4	84.3	79.3	18.4	102.8	82.6	78.5	169.2	29.6	37.7	92.9	371.5
DISPERSE RED 9	30.6	62.5	51.6	61.4	51.3	46.5	60.8	58.2	56.0	183.1	6.2	11.1	19.6	78.2
SOLVENT YELLOW 2	39.4	66.6	56.9	68.8	53.2	47.7	66.8	64.0	60.6	153.7	8.0	13.3	25.3	101.1
SOLVENT GREEN 3	31.4	18.2	16.9	31.3	34.6	0.0	21.3	23.8	20.9	66.5	11.3	54.0	35.4	141.7
SOLVENT RED 24	5.68	16.9	18.8	10.2	12.3	0.0	19.6	9.2	12.4	218.9	6.8	55.1	21.5	86.0
ANTHRACENE	41.54	63.9	64.8	62.4	1.5	10.3	64.2	72.2	48.4	116.6	29.4	60.6	92.2	368.7
BENZANTHRONE	24.32	60.1	52.7	60.0	48.8	46.3	60.3	59.4	55.5	228.1	6.1	11.0	19.2	76.9

	S001HS	S002HS	S003HS	S004HS	S005HS	S006HS	S007HS
	Method Detection Limit						
T (STUDENT'S T @ 99% FOR 6 DF)	3.140						
MDL AB45	3791.6						
MDL AY23	391.8						
MDL AY73	542.4						
MDL AO10	1396.4						
MDL AB1	1306.8						
MDL BY2	5136.8						
MDL DV1	994.3						
MDL SY33	272.5						
MDL DB14	1719.8						
MDL AR64	401.0						
MDL AB9	258.6						
MDL BV10	480.7						
MDL 2AQ	614.8						
MDL SO3	309.1						
MDL 1AQ	670.1						
MDL SY14	360.3						
MDL SO7	1992.7						
MDL AY3	278.1						
MDL SY3	1815.2						
MDL SR1	1672.6						
MDL DR9	523.1						
MDL SY2	503.9						
MDL SG3	18689.4						
MDL SR24	0.0						
MDL AN	639.0						
MDL BZ	801.8						

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Stock Standard Concentrations						
ACID BLUE 45 STD	344.4	344.4	344.4	344.4	344.4	344.4	344.4
ACID YELLOW 23 STD	281.2	281.2	281.2	281.2	281.2	281.2	281.2
ACID YELLOW 73 STD	351.0	351.0	351.0	351.0	351.0	351.0	351.0
ACID ORANGE 10 STD	307.4	307.4	307.4	307.4	307.4	307.4	307.4
ACID BLUE 1 STD	369.2	369.2	369.2	369.2	369.2	369.2	369.2
BASIC YELLOW 2 STD	264.2	264.2	264.2	264.2	264.2	264.2	264.2
DISPERSE VIOLET 1 STD	260.2	260.2	260.2	260.2	260.2	260.2	260.2
SOLVENT YELLOW 33 STD	279.6	279.6	279.6	279.6	279.6	279.6	279.6
DISPERSE BLUE 14 STD	299.0	299.0	299.0	299.0	299.0	299.0	299.0
ACID RED 64 STD	361.8	361.8	361.8	361.8	361.8	361.8	361.8
ACID BLUE 9 STD	328.0	328.0	328.0	328.0	328.0	328.0	328.0
BASIC VIOLET 10 STD	208.4	208.4	208.4	208.4	208.4	208.4	208.4
2-AMINOANTHRAQUINONE STD	373.8	373.8	373.8	373.8	373.8	373.8	373.8
SOLVENT ORANGE 3 STD	255.8	255.8	255.8	255.8	255.8	255.8	255.8
1-AMINOANTHRAQUINONE STD	257.0	257.0	257.0	257.0	257.0	257.0	257.0
SOLVENT YELLOW 14 STD	283.2	283.2	283.2	283.2	283.2	283.2	283.2
SOLVENT ORANGE 7 STD	288.0	288.0	288.0	288.0	288.0	288.0	288.0
ACID YELLOW 3 STD	334.8	334.8	334.8	334.8	334.8	334.8	334.8
SOLVENT YELLOW 3 STD	302.0	302.0	302.0	302.0	302.0	302.0	302.0
SOLVENT RED 1 STD	463.8	463.8	463.8	463.8	463.8	463.8	463.8
DISPERSE RED 9 STD	306.0	306.0	306.0	306.0	306.0	306.0	306.0
SOLVENT YELLOW 2 STD	394.0	394.0	394.0	394.0	394.0	394.0	394.0
SOLVENT GREEN 3 STD	314.0	314.0	314.0	314.0	314.0	314.0	314.0
SOLVENT RED 24 STD	56.8	56.8	56.8	56.8	56.8	56.8	56.8
ANTHRACENE	415.4	415.4	415.4	415.4	415.4	415.4	415.4
BENZANTHRONE	243.2	243.2	243.2	243.2	243.2	243.2	243.2

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Weight of Stock Standard Spiked						
SPIKE VOLUME EACH DYE	100.0	100.0	100.0	100.0	100.0	100.0	100.0
AB45 SPIKE WEIGHT	34.4	34.4	34.4	34.4	34.4	34.4	34.4
AY23 SPIKE WEIGHT	28.1	28.1	28.1	28.1	28.1	28.1	28.1
AY73 SPIKE WEIGHT	35.1	35.1	35.1	35.1	35.1	35.1	35.1
AO10 SPIKE WEIGHT	30.7	30.7	30.7	30.7	30.7	30.7	30.7
AB1 SPIKE WEIGHT	36.9	36.9	36.9	36.9	36.9	36.9	36.9
BY2 SPIKE WEIGHT	26.4	26.4	26.4	26.4	26.4	26.4	26.4
DV1 SPIKE WEIGHT	26.0	26.0	26.0	26.0	26.0	26.0	26.0
SY33 SPIKE WEIGHT	28.0	28.0	28.0	28.0	28.0	28.0	28.0
DB14 SPIKE WEIGHT	29.9	29.9	29.9	29.9	29.9	29.9	29.9
AR64 SPIKE WEIGHT	36.2	36.2	36.2	36.2	36.2	36.2	36.2
AB9 SPIKE WEIGHT	32.8	32.8	32.8	32.8	32.8	32.8	32.8
BV10 SPIKE WEIGHT	20.8	20.8	20.8	20.8	20.8	20.8	20.8
2AQ SPIKE WEIGHT	37.4	37.4	37.4	37.4	37.4	37.4	37.4
SO3 SPIKE WEIGHT	25.6	25.6	25.6	25.6	25.6	25.6	25.6
1AQ SPIKE WEIGHT	25.7	25.7	25.7	25.7	25.7	25.7	25.7
SY14 SPIKE WEIGHT	28.3	28.3	28.3	28.3	28.3	28.3	28.3
SO7 SPIKE WEIGHT	28.8	28.8	28.8	28.8	28.8	28.8	28.8
AY3 SPIKE WEIGHT	33.5	33.5	33.5	33.5	33.5	33.5	33.5
SY3 SPIKE WEIGHT	30.2	30.2	30.2	30.2	30.2	30.2	30.2
SR1 SPIKE WEIGHT	46.4	46.4	46.4	46.4	46.4	46.4	46.4
DR9 SPIKE WEIGHT	30.6	30.6	30.6	30.6	30.6	30.6	30.6
SY2 SPIKE WEIGHT	39.4	39.4	39.4	39.4	39.4	39.4	39.4
SG3 SPIKE WEIGHT	31.4	31.4	31.4	31.4	31.4	31.4	31.4
SR24 SPIKE WEIGHT	5.7	5.7	5.7	5.7	5.7	5.7	5.7
AN SPIKE WEIGHT	41.5	41.5	41.5	41.5	41.5	41.5	41.5
BZ SPIKE WEIGHT	24.3	24.3	24.3	24.3	24.3	24.3	24.3

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Weight Standard per Volume Sample						
MATRIX VOLUME (WATER)	1.000	1.000	1.000	1.000	1.000	1.000	1.000
MATRIX WEIGHT (SOIL)	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB45	34.4	34.4	34.4	34.4	34.4	34.4	34.4
SPIKE CONC AY23	28.1	28.1	28.1	28.1	28.1	28.1	28.1
SPIKE CONC AY73	35.1	35.1	35.1	35.1	35.1	35.1	35.1
SPIKE CONC AO10	30.7	30.7	30.7	30.7	30.7	30.7	30.7
SPIKE CONC AB1	36.9	36.9	36.9	36.9	36.9	36.9	36.9
SPIKE CONC BY2	26.4	26.4	26.4	26.4	26.4	26.4	26.4
SPIKE CONC DV1	26.0	26.0	26.0	26.0	26.0	26.0	26.0
SPIKE CONC SY33	28.0	28.0	28.0	28.0	28.0	28.0	28.0
SPIKE CONC DB14	29.9	29.9	29.9	29.9	29.9	29.9	29.9
SPIKE CONC AR64	36.2	36.2	36.2	36.2	36.2	36.2	36.2
SPIKE CONC AB9	32.8	32.8	32.8	32.8	32.8	32.8	32.8
SPIKE CONC BV10	20.8	20.8	20.8	20.8	20.8	20.8	20.8
SPIKE CONC 2AQ	37.4	37.4	37.4	37.4	37.4	37.4	37.4
SPIKE CONC SO3	25.6	25.6	25.6	25.6	25.6	25.6	25.6
SPIKE CONC 1AQ	25.7	25.7	25.7	25.7	25.7	25.7	25.7
SPIKE CONC SY14	28.3	28.3	28.3	28.3	28.3	28.3	28.3
SPIKE CONC SO7	28.8	28.8	28.8	28.8	28.8	28.8	28.8
SPIKE CONC AY3	33.5	33.5	33.5	33.5	33.5	33.5	33.5
SPIKE CONC SY3	30.2	30.2	30.2	30.2	30.2	30.2	30.2
SPIKE CONC SR1	46.4	46.4	46.4	46.4	46.4	46.4	46.4
SPIKE CONC DR9	30.6	30.6	30.6	30.6	30.6	30.6	30.6
SPIKE CONC SY2	39.4	39.4	39.4	39.4	39.4	39.4	39.4
SPIKE CONC SG3	31.4	31.4	31.4	31.4	31.4	31.4	31.4
SPIKE CONC SR24	5.7	5.7	5.7	5.7	5.7	5.7	5.7
SPIKE CONC AN	41.5	41.5	41.5	41.5	41.5	41.5	41.5
SPIKE CONC BZ	24.3	24.3	24.3	24.3	24.3	24.3	24.3

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
SPIKE CONC AB45	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY23	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY73	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AO10	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BY2	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DV1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY33	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DB14	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AR64	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB9	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BV10	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 2AQ	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 1AQ	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY14	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO7	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DR9	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY2	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SG3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR24	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AN	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BZ	NA	NA	NA	NA	NA	NA	NA

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Results from HPLC						
HPLC RESULT AB45	2.437	2.969	1.542	3.012	2.578	3.593	1.253
HPLC RESULT AY23	14.993	12.372	14.602	14.504	12.124	14.142	12.957
HPLC RESULT AY73	16.048	14.253	16.698	17.177	14.101	15.304	14.260
HPLC RESULT AO10	17.133	13.648	16.468	16.382	13.700	15.746	15.570
HPLC RESULT AB1	22.126	13.871	17.228	20.594	17.605	20.068	20.387
HPLC RESULT BY2	27.768	24.807	24.726	57.300	28.377	23.332	71.104
HPLC RESULT DV1	11.524	10.356	10.181	10.092	9.196	10.635	9.728
HPLC RESULT SY33	14.754	13.023	14.377	11.190	10.951	14.245	13.745
HPLC RESULT DB14	8.854	7.207	9.114	7.950	6.866	12.704	10.350
HPLC RESULT AR64	11.895	10.007	11.494	15.452	12.906	9.509	9.934
HPLC RESULT AB9	19.247	15.876	18.811	18.503	15.571	18.161	18.785
HPLC RESULT BV10	11.744	9.151	11.021	9.975	9.501	11.023	11.248
HPLC RESULT 2AQ	0.292	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SO3	8.667	8.182	7.937	9.510	5.842	11.149	9.523
HPLC RESULT 1AQ	12.630	10.331	12.530	11.383	10.719	12.163	11.837
HPLC RESULT SY14	11.449	10.096	11.508	9.456	4.152	9.786	9.294
HPLC RESULT SO7	12.687	13.315	14.097	10.546	1.345	13.562	13.386
HPLC RESULT AY3	20.908	16.871	19.933	20.921	17.544	18.985	19.043
HPLC RESULT SY3	15.569	12.097	14.080	13.269	11.649	15.141	15.015
HPLC RESULT SR1	14.513	21.872	16.866	15.866	3.672	20.567	16.527
HPLC RESULT DR9	12.509	10.311	12.276	10.250	9.293	12.154	11.644
HPLC RESULT SY2	13.313	11.383	13.761	10.636	9.533	13.360	12.798
HPLC RESULT SG3	3.649	3.384	6.251	6.926	0.000	4.259	4.761
HPLC RESULT SR24	3.374	3.755	2.047	2.468	0.000	3.920	1.842
HPLC RESULT AN	12.776	12.95	12.473	0.293	2.061	12.831	14.442
HPLC RESULT BZ	12.01	10.544	12.157	9.755	9.252	12.056	11.877

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Final Sample Results						
CF (STD INJ VOL VS SAM INJ VOL)	2	2	2	2	2	2	2
FINAL VOLUME	10.00	10.00	10.00	10.00	10.00	10.00	10.00
CORRECTED RESULT AB45	12.19	14.85	7.71	15.06	12.89	17.97	6.27
CORRECTED RESULT AY23	74.97	61.86	73.01	72.52	60.62	70.71	64.79
CORRECTED RESULT AY73	80.24	71.27	83.49	85.89	70.51	76.52	71.30
CORRECTED RESULT AO10	85.67	68.24	82.34	81.91	68.50	78.73	77.85
CORRECTED RESULT AB1	110.63	69.36	86.14	102.97	88.03	100.34	101.94
CORRECTED RESULT BY2	138.84	124.04	123.63	286.50	141.89	116.66	355.52
CORRECTED RESULT DV1	57.62	51.78	50.91	50.46	45.98	53.18	48.64
CORRECTED RESULT SY33	73.77	65.12	71.89	55.95	54.76	71.23	68.73
CORRECTED RESULT DB14	44.27	36.04	45.57	39.75	34.33	63.52	51.75
CORRECTED RESULT AR64	59.48	50.04	57.47	77.26	64.53	47.55	49.67
CORRECTED RESULT AB9	96.24	79.38	94.06	92.52	77.86	90.81	93.93
CORRECTED RESULT BV10	58.72	45.76	55.11	49.88	47.51	55.12	56.24
CORRECTED RESULT 2AQ	1.46	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SO3	43.34	40.91	39.69	47.55	29.21	55.75	47.62
CORRECTED RESULT 1AQ	63.15	51.66	62.65	56.92	53.60	60.82	59.19
CORRECTED RESULT SY14	57.25	50.48	57.54	47.28	20.76	48.93	46.47
CORRECTED RESULT SO7	63.44	66.58	70.49	52.73	6.73	67.81	66.93
CORRECTED RESULT AY3	104.54	84.36	99.67	104.61	87.72	94.93	95.22
CORRECTED RESULT SY3	77.85	60.49	70.40	66.35	58.25	75.71	75.08
CORRECTED RESULT SR1	72.57	109.36	84.33	79.33	18.36	102.84	82.64
CORRECTED RESULT DR9	62.55	51.56	61.38	51.25	46.47	60.77	58.22
CORRECTED RESULT SY2	66.57	56.92	68.81	53.18	47.67	66.80	63.99
CORRECTED RESULT SG3	18.25	16.92	31.26	34.63	0.00	21.30	23.81
CORRECTED RESULT SR24	16.87	18.78	10.24	12.34	0.00	19.60	9.21
CORRECTED RESULT AN	63.88	64.75	62.37	1.47	10.31	64.16	72.21
CORRECTED RESULT BZ	60.05	52.72	60.79	48.78	46.26	60.28	59.39

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Percent Recoveries						
% RECOVERY AB45	35.38	43.10	22.39	43.73	37.43	52.16	18.19
% RECOVERY AY23	266.6	220.0	259.6	257.9	215.6	251.5	230.4
% RECOVERY AY73	228.6	203.0	237.9	244.7	200.9	218.0	203.1
% RECOVERY AO10	278.7	222.0	267.9	266.5	222.8	256.1	253.3
% RECOVERY AB1	299.6	187.9	233.3	278.9	238.4	271.8	276.1
% RECOVERY BY2	525.5	469.5	467.9	1084.4	537.0	441.6	1345.6
% RECOVERY DV1	221.4	199.0	195.6	193.9	176.7	204.4	186.9
% RECOVERY SY33	263.8	232.9	257.1	200.1	195.8	254.7	245.8
% RECOVERY DB14	148.1	120.5	152.4	132.9	114.8	212.4	173.1
% RECOVERY AR64	164.4	138.3	158.8	213.5	178.4	131.4	137.3
% RECOVERY AB9	293.4	242.0	286.8	282.1	237.4	276.8	286.4
% RECOVERY BV10	281.8	219.6	264.4	239.3	228.0	264.5	269.9
% RECOVERY 2AQ	3.9	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SO3	169.4	159.9	155.1	185.9	114.2	217.9	186.1
% RECOVERY 1AQ	245.7	201.0	243.8	221.5	208.5	236.6	230.3
% RECOVERY SY14	202.1	178.2	203.2	166.9	73.3	172.8	164.1
% RECOVERY SO7	220.3	231.2	244.7	183.1	23.4	235.5	232.4
% RECOVERY AY3	312.2	252.0	297.7	312.4	262.0	283.5	284.4
% RECOVERY SY3	257.8	200.3	233.1	219.7	192.9	250.7	248.6
% RECOVERY SR1	156.5	235.8	181.8	171.0	39.6	221.7	178.2
% RECOVERY DR9	204.4	168.5	200.6	167.5	151.8	198.6	190.3
% RECOVERY SY2	168.9	144.5	174.6	135.0	121.0	169.5	162.4
% RECOVERY SG3	58.1	53.9	99.5	110.3	0.0	67.8	75.8
% RECOVERY SR24	297.0	330.5	180.2	217.3	0.0	345.1	162.1
% RECOVERY AN	153.8	155.9	150.1	3.5	24.8	154.4	173.8
% RECOVERY BZ	246.9	216.8	249.9	200.6	190.2	247.9	244.2

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Square of Corrected Results						
N	7	7	7	7	7	7	7
Xi^2 AB45	148.5	220.4	59.4	226.8	166.2	322.7	39.3
Xi^2 AY23	5619.8	3826.7	5330.5	5259.2	3674.8	4999.9	4197.1
Xi^2 AY73	6438.5	5078.7	6970.6	7376.2	4971.0	5855.3	5083.7
Xi^2 AO10	7338.5	4656.7	6779.9	6709.2	4692.3	6198.4	6060.6
Xi^2 AB1	12239.0	4810.1	7420.1	10602.8	7748.4	10068.1	10390.7
Xi^2 BY2	19276.5	15384.7	15284.4	82082.3	20131.4	13609.6	126394.5
Xi^2 DV1	3320.1	2681.2	2591.3	2546.2	2114.2	2827.6	2365.8
Xi^2 SY33	5442.0	4240.0	5167.5	3130.4	2998.1	5073.0	4723.1
Xi^2 DB14	1959.8	1298.5	2076.6	1580.1	1178.5	4034.8	2678.1
Xi^2 AR64	3537.3	2503.5	3302.8	5969.1	4164.1	2260.5	2467.1
Xi^2 AB9	9261.2	6301.2	8846.3	8559.0	6061.4	8245.5	8821.9
Xi^2 BV10	3448.0	2093.5	3036.6	2487.5	2256.7	3037.7	3162.9
Xi^2 2AQ	2.1	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SO3	1877.9	1673.6	1574.9	2261.0	853.2	3107.5	2267.2
Xi^2 1AQ	3987.9	2668.2	3925.0	3239.3	2872.4	3698.5	3502.9
Xi^2 SY14	3277.0	2548.2	3310.9	2235.4	431.0	2394.1	2159.5
Xi^2 SO7	4024.0	4432.2	4968.1	2780.5	45.2	4598.2	4479.6
Xi^2 AY3	10928.6	7115.8	9933.1	10942.2	7694.8	9010.8	9065.9
Xi^2 SY3	6059.8	3658.4	4956.2	4401.7	3392.5	5731.2	5636.3
Xi^2 SR1	5265.7	11959.6	7111.5	6293.2	337.1	10575.0	6828.5
Xi^2 DR9	3911.9	2657.9	3767.5	2626.6	2159.0	3693.0	3389.6
Xi^2 SY2	4430.9	3239.3	4734.1	2828.1	2272.0	4462.2	4094.7
Xi^2 SG3	332.9	286.3	976.9	1199.2	0.0	453.5	566.7
Xi^2 SR24	284.6	352.5	104.8	152.3	0.0	384.2	84.8
Xi^2 AN	4080.7	4192.6	3889.4	2.1	106.2	4115.9	5214.3
Xi^2 BZ	3606.0	2779.4	3694.8	2379.0	2140.0	3633.7	3526.6

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Sum of the Square of Corrected Results						
E Xi^2 AB45	1183.2						
E Xi^2 AY23	32907.8						
E Xi^2 AY73	41773.9						
E Xi^2 AO10	42435.6						
E Xi^2 AB1	63279.3						
E Xi^2 BY2	292163.2						
E Xi^2 DV1	18446.4						
E Xi^2 SY33	30774.1						
E Xi^2 DB14	14806.4						
E Xi^2 AR64	24204.4						
E Xi^2 AB9	56096.6						
E Xi^2 BV10	19523.0						
E Xi^2 2AQ	2.1						
E Xi^2 SO3	13615.4						
E Xi^2 1AQ	23894.3						
E Xi^2 SY14	16356.1						
E Xi^2 SO7	25327.9						
E Xi^2 AY3	64691.1						
E Xi^2 SY3	33836.1						
E Xi^2 SR1	48370.8						
E Xi^2 DR9	22205.4						
E Xi^2 SY2	26061.4						
E Xi^2 SG3	3815.4						
E Xi^2 SR24	1363.1						
E Xi^2 AN	21601.1						
E Xi^2 BZ	21759.5						

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Sum of the Corrected Results Squared						
(E Xi)^2 AB45	7555.1						
(E Xi)^2 AY23	228933.5						
(E Xi)^2 AY73	290742.0						
(E Xi)^2 AO10	295104.3						
(E Xi)^2 AB1	434801.8						
(E Xi)^2 BY2	1656549.2						
(E Xi)^2 DV1	128565.3						
(E Xi)^2 SY33	212913.0						
(E Xi)^2 DB14	99366.8						
(E Xi)^2 AR64	164823.8						
(E Xi)^2 AB9	390337.6						
(E Xi)^2 BV10	135655.9						
(E Xi)^2 2AQ	2.1						
(E Xi)^2 SO3	92446.4						
(E Xi)^2 1AQ	166435.4						
(E Xi)^2 SY14	108047.0						
(E Xi)^2 SO7	155780.2						
(E Xi)^2 AY3	450274.6						
(E Xi)^2 SY3	234352.8						
(E Xi)^2 SR1	301856.8						
(E Xi)^2 DR9	153809.1						
(E Xi)^2 SY2	179708.2						
(E Xi)^2 SG3	21359.8						
(E Xi)^2 SR24	7574.2						
(E Xi)^2 AN	115009.2						
(E Xi)^2 BZ	150741.9						

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Variance						
S^2: VARIANCE AB45	17.3						
S^2: VARIANCE AY23	33.8						
S^2: VARIANCE AY73	39.9						
S^2: VARIANCE AO10	46.3						
S^2: VARIANCE AB1	194.1						
S^2: VARIANCE BY2	9252.2						
S^2: VARIANCE DV1	13.3						
S^2: VARIANCE SY33	59.7						
S^2: VARIANCE DB14	101.9						
S^2: VARIANCE AR64	109.7						
S^2: VARIANCE AB9	55.7						
S^2: VARIANCE BV10	23.9						
S^2: VARIANCE 2AQ	0.3						
S^2: VARIANCE SO3	68.1						
S^2: VARIANCE 1AQ	19.6						
S^2: VARIANCE SY14	153.5						
S^2: VARIANCE SO7	512.3						
S^2: VARIANCE AY3	61.0						
S^2: VARIANCE SY3	59.5						
S^2: VARIANCE SR1	874.7						
S^2: VARIANCE DR9	38.8						
S^2: VARIANCE SY2	64.8						
S^2: VARIANCE SG3	127.3						
S^2: VARIANCE SR24	46.8						
S^2: VARIANCE AN	861.9						
S^2: VARIANCE BZ	37.5						

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Standard Deviation						
STD DEVIATION AB45	4.2						
STD DEVIATION AY23	5.8						
STD DEVIATION AY73	6.3						
STD DEVIATION AO10	6.8						
STD DEVIATION AB1	13.9						
STD DEVIATION BY2	96.2						
STD DEVIATION DV1	3.6						
STD DEVIATION SY33	7.7						
STD DEVIATION DB14	10.1						
STD DEVIATION AR64	10.5						
STD DEVIATION AB9	7.5						
STD DEVIATION BV10	4.9						
STD DEVIATION 2AQ	0.6						
STD DEVIATION SO3	8.3						
STD DEVIATION 1AQ	4.4						
STD DEVIATION SY14	12.4						
STD DEVIATION SO7	22.6						
STD DEVIATION AY3	7.8						
STD DEVIATION SY3	7.7						
STD DEVIATION SR1	29.6						
STD DEVIATION DR9	6.2						
STD DEVIATION SY2	8.0						
STD DEVIATION SG3	11.3						
STD DEVIATION SR24	6.8						
STD DEVIATION AN	29.4						
STD DEVIATION BZ	6.1						

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Method Detection Limit						
T (STUDENT'S T @ 99% FOR 6 DF)	3.140						
MDL AB45	13.1						
MDL AY23	18.3						
MDL AY73	19.8						
MDL AO10	21.4						
MDL AB1	43.7						
MDL BY2	302.0						
MDL DV1	11.5						
MDL SY33	24.3						
MDL DB14	31.7						
MDL AR64	32.9						
MDL AB9	23.4						
MDL BV10	15.4						
MDL 2AQ	1.7						
MDL SO3	25.9						
MDL 1AQ	13.9						
MDL SY14	38.9						
MDL SO7	71.1						
MDL AY3	24.5						
MDL SY3	24.2						
MDL SR1	92.9						
MDL DR9	19.6						
MDL SY2	25.3						
MDL SG3	35.4						
MDL SR24	21.5						
MDL AN	92.2						
MDL BZ	19.2						

TABLE VII - 6A

PERCENT RECOVERIES
LOW-LEVEL AQUEOUS METHOD
HIGH SPIKE, LONG GRADIENT

Compounds		Ret. Time	WLM004HS [1]	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
% RECOVERY AB45	%	6.439	0	3	2	11	19	7	8
% RECOVERY AY3	%	13.372	127	152	125	144	135	131	139
% RECOVERY AY23	%	14.513	113	99	103	118	124	122	107
% RECOVERY AR64	%	14.9	74	52	68	55	102	86	51
% RECOVERY AY73	%	17.353	95	83	80	109	113	99	101
% RECOVERY AO10	%	18.752	122	128	120	133	132	127	130
% RECOVERY AB9	%	21.657	116	150	140	148	145	139	133
% RECOVERY DV1	%	22.77	13	90	89	98	107	95	95
% RECOVERY AB1	%	23.733	132	146	136	144	144	138	141
% RECOVERY 2AQ	%	23.758	0	0	0	0	0	0	0
% RECOVERY SO3	%	24.783	14	96	90	97	102	72	75
% RECOVERY SY33	%	28.259	110	129	126	133	132	125	126
% RECOVERY 1AQ	%	28.853	107	124	122	127	118	109	120
% RECOVERY DB14	%	34.023	19	86	71	70	87	81	97
% RECOVERY BY2	%	35.367	274	437	708	403	357	276	280
% RECOVERY SY3	%	35.998	95	132	124	130	125	113	121
% RECOVERY BV10	%	36.018	117	143	132	139	138	130	136
% RECOVERY DR9	%	36.142	84	106	99	103	99	89	98
% RECOVERY BZ	%	36.724	103	133	126	130	128	117	121
% RECOVERY SY2	%	39.91	43	93	90	90	93	74	87
% RECOVERY AN	%	40.641	65	115	104	113	97	67	83
% RECOVERY SY14	%	44.36	94	113	109	109	113	90	105
% RECOVERY SR1	%	44.811	68	94	115	112	97	91	83
% RECOVERY SO7	%	50.894	103	133	130	132	127	112	123
% RECOVERY SG3	%	57.448	45	59	55	77	51	58	82
% RECOVERY SR24	%	57.994	111	175	161	173	169	152	128

1 Sample not used due to problems during extraction

TABLE VII-6

Low-Level Aqueous Extraction Method, Long Gradient, High Spike

		Sample Results (µg/L)							Mean Result µg/L	Method Bias (Accuracy) %R	Standard Deviation	Method Precision (%RSD)
Compounds	Sample Conc µg/L	WLM004HS [1]	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS	average sample result	(mean result/sample conc)*100	std. dev. sample results	(std.dev/mean result)*100
ACID BLUE 45	172.2	0	5.0	4.2	19.5	32.7	11.2	14.5	14.5	8.4	10.6	73.2
ACID YELLOW 23	140.6	158.3	139.2	144.5	165.4	173.7	171.9	150.6	157.5	112.1	14.7	9.3
ACID YELLOW 73	175.5	165.9	145.9	141.0	190.6	199.0	173.4	177.1	171.2	97.5	23.4	13.7
ACID ORANGE 10	153.7	187.9	197.4	184.1	204.2	203.5	194.5	199.8	197.3	128.3	7.4	3.7
ACID BLUE 1	184.6	243.5	269.9	251.3	265.9	265.1	254.9	260.8	261.3	141.6	7.1	2.7
BASIC YELLOW 2	132.1	362.1	577.6	935.1	533.0	472.0	364.9	369.6	542.0	410.3	210.7	38.9
DISPERSE VIOLET 1	130.1	16.565	117.6	115.5	127.7	139.8	123.1	123.1	124.5	95.7	8.7	7.0
SOLVENT YELLOW 33	139.8	154.0	180.4	175.8	186.2	184.2	174.4	176.8	179.6	128.5	4.8	2.7
DISPERSE BLUE 14	149.5	29.0	128.1	106.4	104.6	130.1	121.2	145.5	122.6	82.0	15.5	12.6
ACID RED 64	180.9	133.9	94.2	123.5	99.1	184.1	155.6	92.1	124.8	69.0	37.8	30.3
ACID BLUE 9	164	189.5	246.4	229.9	242.6	238.3	227.5	218.3	233.8	142.6	10.5	4.5
BASIC VIOLET 10	104.2	122.2	149.2	137.9	144.4	143.9	135.8	141.4	142.1	136.4	4.9	3.4
2-AMINOANTHRAQUINONE	186.9	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT ORANGE 3	127.9	17.9	122.9	115.5	124.6	130.6	92.3	96.2	113.7	88.9	15.9	14.0
1-AMINOANTHRAQUINONE	128.5	137.1	159.0	156.1	162.9	151.7	139.6	154.4	154.0	119.8	8.0	5.2
SOLVENT YELLOW 14	141.6	132.4	160.5	154.9	154.5	159.8	127.2	148.9	151.0	106.6	12.4	8.2
SOLVENT ORANGE 7	144	148.7	191.9	187.6	189.4	183.5	162.0	176.6	181.8	126.3	11.1	6.1
ACID YELLOW 3	167.4	212.0	254.5	208.8	240.6	226.7	218.6	233.5	230.4	137.7	16.2	7.0
SOLVENT YELLOW 3	151	144.0	199.4	187.4	195.7	189.0	170.3	182.3	187.3	124.1	10.3	5.5
SOLVENT RED 1	231.9	158.5	217.7	266.3	260.7	225.4	210.4	193.5	229.0	98.8	28.8	12.6
DISPERSE RED 9	153	128.7	162.4	151.9	157.5	151.4	135.4	150.4	151.5	99.0	9.1	6.0
SOLVENT YELLOW 2	197	84.7	183.7	177.8	176.8	182.3	145.7	170.6	172.8	87.7	14.1	8.1
SOLVENT GREEN 3	157	71.0	92.2	86.3	121.5	79.7	90.5	129.0	99.9	63.6	20.3	20.3
SOLVENT RED 24	28.4	31.4	49.6	45.7	49.1	47.9	43.2	36.3	45.3	159.4	5.0	11.1
ANTHRACENE	207.7	135.6	238.8	215.4	235.1	200.7	138.2	172.9	200.2	96.4	38.8	19.4
BENZANTHRONE	121.6	124.9	162.0	153.8	157.9	155.6	142.8	147.4	153.3	126.0	7.0	4.6

1 Sample results not used for calculations due to problems encountered during extraction.

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
		Stock Standard Concentrations						
ACID BLUE 45 STD	UG/ML	344.4	344.4	344.4	344.4	344.4	344.4	344.4
ACID YELLOW 23 STD	UG/ML	281.2	281.2	281.2	281.2	281.2	281.2	281.2
ACID YELLOW 73 STD	UG/ML	351.0	351.0	351.0	351.0	351.0	351.0	351.0
ACID ORANGE 10 STD	UG/ML	307.4	307.4	307.4	307.4	307.4	307.4	307.4
ACID BLUE 1 STD	UG/ML	369.2	369.2	369.2	369.2	369.2	369.2	369.2
BASIC YELLOW 2 STD	UG/ML	264.2	264.2	264.2	264.2	264.2	264.2	264.2
DISPERSE VIOLET 1 STD	UG/ML	260.2	260.2	260.2	260.2	260.2	260.2	260.2
SOLVENT YELLOW 33 STD	UG/ML	279.6	279.6	279.6	279.6	279.6	279.6	279.6
DISPERSE BLUE 14 STD	UG/ML	299.0	299.0	299.0	299.0	299.0	299.0	299.0
ACID RED 64 STD	UG/ML	361.8	361.8	361.8	361.8	361.8	361.8	361.8
ACID BLUE 9 STD	UG/ML	328.0	328.0	328.0	328.0	328.0	328.0	328.0
BASIC VIOLET 10 STD	UG/ML	208.4	208.4	208.4	208.4	208.4	208.4	208.4
2-AMINOANTHRAQUINONE STD	UG/ML	373.8	373.8	373.8	373.8	373.8	373.8	373.8
SOLVENT ORANGE 3 STD	UG/ML	255.8	255.8	255.8	255.8	255.8	255.8	255.8
1-AMINOANTHRAQUINONE STD	UG/ML	257.0	257.0	257.0	257.0	257.0	257.0	257.0
SOLVENT YELLOW 14 STD	UG/ML	283.2	283.2	283.2	283.2	283.2	283.2	283.2
SOLVENT ORANGE 7 STD	UG/ML	288.0	288.0	288.0	288.0	288.0	288.0	288.0
ACID YELLOW 3 STD	UG/ML	334.8	334.8	334.8	334.8	334.8	334.8	334.8
SOLVENT YELLOW 3 STD	UG/ML	302.0	302.0	302.0	302.0	302.0	302.0	302.0
SOLVENT RED 1 STD	UG/ML	463.8	463.8	463.8	463.8	463.8	463.8	463.8
DISPERSE RED 9 STD	UG/ML	306.0	306.0	306.0	306.0	306.0	306.0	306.0
SOLVENT YELLOW 2 STD	UG/ML	394.0	394.0	394.0	394.0	394.0	394.0	394.0
SOLVENT GREEN 3 STD	UG/ML	314.0	314.0	314.0	314.0	314.0	314.0	314.0
SOLVENT RED 24 STD	UG/ML	56.8	56.8	56.8	56.8	56.8	56.8	56.8
ANTHRACENE	UG/ML	415.4	415.4	415.4	415.4	415.4	415.4	415.4
BENZANTHRONE	UG/ML	243.2	243.2	243.2	243.2	243.2	243.2	243.2

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
		Weight of Stock Standard Spiked						
SPIKE VOLUME EACH DYE	UL	500.0	500.0	500.0	500.0	500.0	500.0	500.0
AB45 SPIKE WEIGHT	UG	172.2	172.2	172.2	172.2	172.2	172.2	172.2
AY23 SPIKE WEIGHT	UG	140.6	140.6	140.6	140.6	140.6	140.6	140.6
AY73 SPIKE WEIGHT	UG	175.5	175.5	175.5	175.5	175.5	175.5	175.5
AO10 SPIKE WEIGHT	UG	153.7	153.7	153.7	153.7	153.7	153.7	153.7
AB1 SPIKE WEIGHT	UG	184.6	184.6	184.6	184.6	184.6	184.6	184.6
BY2 SPIKE WEIGHT	UG	132.1	132.1	132.1	132.1	132.1	132.1	132.1
DV1 SPIKE WEIGHT	UG	130.1	130.1	130.1	130.1	130.1	130.1	130.1
SY33 SPIKE WEIGHT	UG	139.8	139.8	139.8	139.8	139.8	139.8	139.8
DB14 SPIKE WEIGHT	UG	149.5	149.5	149.5	149.5	149.5	149.5	149.5
AR64 SPIKE WEIGHT	UG	180.9	180.9	180.9	180.9	180.9	180.9	180.9
AB9 SPIKE WEIGHT	UG	164.0	164.0	164.0	164.0	164.0	164.0	164.0
BV10 SPIKE WEIGHT	UG	104.2	104.2	104.2	104.2	104.2	104.2	104.2
2AQ SPIKE WEIGHT	UG	186.9	186.9	186.9	186.9	186.9	186.9	186.9
SO3 SPIKE WEIGHT	UG	127.9	127.9	127.9	127.9	127.9	127.9	127.9
1AQ SPIKE WEIGHT	UG	128.5	128.5	128.5	128.5	128.5	128.5	128.5
SY14 SPIKE WEIGHT	UG	141.6	141.6	141.6	141.6	141.6	141.6	141.6
SO7 SPIKE WEIGHT	UG	144.0	144.0	144.0	144.0	144.0	144.0	144.0
AY3 SPIKE WEIGHT	UG	167.4	167.4	167.4	167.4	167.4	167.4	167.4
SY3 SPIKE WEIGHT	UG	151.0	151.0	151.0	151.0	151.0	151.0	151.0
SR1 SPIKE WEIGHT	UG	231.9	231.9	231.9	231.9	231.9	231.9	231.9
DR9 SPIKE WEIGHT	UG	153.0	153.0	153.0	153.0	153.0	153.0	153.0
SY2 SPIKE WEIGHT	UG	197.0	197.0	197.0	197.0	197.0	197.0	197.0
SG3 SPIKE WEIGHT	UG	157.0	157.0	157.0	157.0	157.0	157.0	157.0
SR24 SPIKE WEIGHT	UG	28.4	28.4	28.4	28.4	28.4	28.4	28.4
AN SPIKE WEIGHT	UG	207.7	207.7	207.7	207.7	207.7	207.7	207.7
BZ SPIKE WEIGHT	UG	121.6	121.6	121.6	121.6	121.6	121.6	121.6

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
		Weight Standard per Volume Sample						
MATRIX VOLUME (WATER)	L	1.000	1.000	1.000	1.000	1.000	1.000	1.000
MATRIX WEIGHT (SOIL)	KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB45	UG/L	172.2	172.2	172.2	172.2	172.2	172.2	172.2
SPIKE CONC AY23	UG/L	140.6	140.6	140.6	140.6	140.6	140.6	140.6
SPIKE CONC AY73	UG/L	175.5	175.5	175.5	175.5	175.5	175.5	175.5
SPIKE CONC AO10	UG/L	153.7	153.7	153.7	153.7	153.7	153.7	153.7
SPIKE CONC AB1	UG/L	184.6	184.6	184.6	184.6	184.6	184.6	184.6
SPIKE CONC BY2	UG/L	132.1	132.1	132.1	132.1	132.1	132.1	132.1
SPIKE CONC DV1	UG/L	130.1	130.1	130.1	130.1	130.1	130.1	130.1
SPIKE CONC SY33	UG/L	139.8	139.8	139.8	139.8	139.8	139.8	139.8
SPIKE CONC DB14	UG/L	149.5	149.5	149.5	149.5	149.5	149.5	149.5
SPIKE CONC AR64	UG/L	180.9	180.9	180.9	180.9	180.9	180.9	180.9
SPIKE CONC AB9	UG/L	164.0	164.0	164.0	164.0	164.0	164.0	164.0
SPIKE CONC BV10	UG/L	104.2	104.2	104.2	104.2	104.2	104.2	104.2
SPIKE CONC 2AQ	UG/L	186.9	186.9	186.9	186.9	186.9	186.9	186.9
SPIKE CONC SO3	UG/L	127.9	127.9	127.9	127.9	127.9	127.9	127.9
SPIKE CONC 1AQ	UG/L	128.5	128.5	128.5	128.5	128.5	128.5	128.5
SPIKE CONC SY14	UG/L	141.6	141.6	141.6	141.6	141.6	141.6	141.6
SPIKE CONC SO7	UG/L	144.0	144.0	144.0	144.0	144.0	144.0	144.0
SPIKE CONC AY3	UG/L	167.4	167.4	167.4	167.4	167.4	167.4	167.4
SPIKE CONC SY3	UG/L	151.0	151.0	151.0	151.0	151.0	151.0	151.0
SPIKE CONC SR1	UG/L	231.9	231.9	231.9	231.9	231.9	231.9	231.9
SPIKE CONC DR9	UG/L	153.0	153.0	153.0	153.0	153.0	153.0	153.0
SPIKE CONC SY2	UG/L	197.0	197.0	197.0	197.0	197.0	197.0	197.0
SPIKE CONC SG3	UG/L	157.0	157.0	157.0	157.0	157.0	157.0	157.0
SPIKE CONC SR24	UG/L	28.4	28.4	28.4	28.4	28.4	28.4	28.4
SPIKE CONC AN	UG/L	207.7	207.7	207.7	207.7	207.7	207.7	207.7
SPIKE CONC BZ	UG/L	121.6	121.6	121.6	121.6	121.6	121.6	121.6

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
SPIKE CONC AB45	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY23	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY73	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AO10	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB1	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BY2	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DV1	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY33	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DB14	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AR64	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB9	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BV10	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 2AQ	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO3	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 1AQ	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY14	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO7	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY3	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY3	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR1	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DR9	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY2	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SG3	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR24	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AN	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BZ	UG/KG	NA	NA	NA	NA	NA	NA	NA

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
		Results from HPLC						
HPLC RESULT AB45	UG/ML	0.000	0.995	0.843	3.896	6.545	2.245	2.896
HPLC RESULT AY23	UG/ML	31.665	27.837	28.907	33.080	34.740	34.374	30.117
HPLC RESULT AY73	UG/ML	33.188	29.185	28.193	38.121	39.801	34.677	35.415
HPLC RESULT AO10	UG/ML	37.571	39.473	36.828	40.843	40.695	38.905	39.967
HPLC RESULT AB1	UG/ML	48.691	53.970	50.263	53.189	53.018	50.981	52.169
HPLC RESULT BY2	UG/ML	72.411	115.523	187.021	106.590	94.405	72.977	73.912
HPLC RESULT DV1	UG/ML	3.313	23.526	23.104	25.545	27.964	24.618	24.622
HPLC RESULT SY33	UG/ML	30.801	36.073	35.166	37.243	36.838	34.886	35.358
HPLC RESULT DB14	UG/ML	5.799	25.614	21.277	20.913	26.015	24.246	29.102
HPLC RESULT AR64	UG/ML	26.770	18.841	24.702	19.810	36.829	31.124	18.413
HPLC RESULT AB9	UG/ML	37.905	49.288	45.974	48.529	47.667	45.500	43.655
HPLC RESULT BV10	UG/ML	24.441	29.847	27.586	28.886	28.788	27.151	28.278
HPLC RESULT 2AQ	UG/ML	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SO3	UG/ML	3.586	24.572	23.103	24.919	26.118	18.450	19.235
HPLC RESULT 1AQ	UG/ML	27.425	31.797	31.227	32.580	30.330	27.920	30.889
HPLC RESULT SY14	UG/ML	26.486	32.098	30.976	30.897	31.950	25.441	29.788
HPLC RESULT SO7	UG/ML	29.740	38.371	37.529	37.873	36.702	32.390	35.318
HPLC RESULT AY3	UG/ML	42.409	50.900	41.758	48.114	45.341	43.726	46.692
HPLC RESULT SY3	UG/ML	28.800	39.874	37.480	39.147	37.790	34.057	36.466
HPLC RESULT SR1	UG/ML	31.696	43.549	53.255	52.130	45.087	42.077	38.708
HPLC RESULT DR9	UG/ML	25.738	32.480	30.378	31.493	30.272	27.084	30.079
HPLC RESULT SY2	UG/ML	16.943	36.740	35.559	35.362	36.465	29.142	34.127
HPLC RESULT SG3	UG/ML	14.202	18.437	17.269	24.297	15.931	18.102	25.807
HPLC RESULT SR24	UG/ML	6.283	9.919	9.130	9.815	9.586	8.632	7.255
HPLC RESULT AN	UG/ML	27.125	47.752	43.087	47.018	40.142	27.649	34.577
HPLC RESULT BZ	UG/ML	24.973	32.404	30.753	31.583	31.126	28.565	29.48

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
		Final Sample Results						
CF (STD INJ VOL VS SAM INJ VOL)		2	2	2	2	2	2	2
FINAL VOLUME	ML	10.00	10.00	10.00	10.00	10.00	10.00	10.00
CORRECTED RESULT AB45	UG/L or UG/KG	0.00	4.98	4.22	19.48	32.73	11.23	14.48
CORRECTED RESULT AY23	UG/L or UG/KG	158.33	139.19	144.54	165.40	173.70	171.87	150.59
CORRECTED RESULT AY73	UG/L or UG/KG	165.94	145.93	140.97	190.61	199.01	173.39	177.08
CORRECTED RESULT AO10	UG/L or UG/KG	187.86	197.37	184.14	204.22	203.48	194.53	199.84
CORRECTED RESULT AB1	UG/L or UG/KG	243.46	269.85	251.32	265.95	265.09	254.91	260.85
CORRECTED RESULT BY2	UG/L or UG/KG	362.06	577.62	935.11	532.95	472.03	364.89	369.56
CORRECTED RESULT DV1	UG/L or UG/KG	16.57	117.63	115.52	127.73	139.82	123.09	123.11
CORRECTED RESULT SY33	UG/L or UG/KG	154.01	180.37	175.83	186.22	184.19	174.43	176.79
CORRECTED RESULT DB14	UG/L or UG/KG	29.00	128.07	106.39	104.57	130.08	121.23	145.51
CORRECTED RESULT AR64	UG/L or UG/KG	133.85	94.21	123.51	99.05	184.15	155.62	92.07
CORRECTED RESULT AB9	UG/L or UG/KG	189.53	246.44	229.87	242.65	238.34	227.50	218.28
CORRECTED RESULT BV10	UG/L or UG/KG	122.21	149.24	137.93	144.43	143.94	135.76	141.39
CORRECTED RESULT 2AQ	UG/L or UG/KG	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SO3	UG/L or UG/KG	17.93	122.86	115.52	124.60	130.59	92.25	96.18
CORRECTED RESULT 1AQ	UG/L or UG/KG	137.13	158.99	156.14	162.90	151.65	139.60	154.45
CORRECTED RESULT SY14	UG/L or UG/KG	132.43	160.49	154.88	154.49	159.75	127.21	148.94
CORRECTED RESULT SO7	UG/L or UG/KG	148.70	191.86	187.65	189.37	183.51	161.95	176.59
CORRECTED RESULT AY3	UG/L or UG/KG	212.05	254.50	208.79	240.57	226.71	218.63	233.46
CORRECTED RESULT SY3	UG/L or UG/KG	144.00	199.37	187.40	195.74	188.95	170.29	182.33
CORRECTED RESULT SR1	UG/L or UG/KG	158.48	217.75	266.28	260.65	225.44	210.39	193.54
CORRECTED RESULT DR9	UG/L or UG/KG	128.69	162.40	151.89	157.47	151.36	135.42	150.40
CORRECTED RESULT SY2	UG/L or UG/KG	84.72	183.70	177.80	176.81	182.33	145.71	170.63
CORRECTED RESULT SG3	UG/L or UG/KG	71.01	92.19	86.35	121.49	79.66	90.51	129.04
CORRECTED RESULT SR24	UG/L or UG/KG	31.42	49.60	45.65	49.08	47.93	43.16	36.28
CORRECTED RESULT AN	UG/L or UG/KG	135.63	238.76	215.44	235.09	200.71	138.25	172.89
CORRECTED RESULT BZ	UG/L or UG/KG	124.87	162.02	153.77	157.92	155.63	142.83	147.40

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
		Percent Recoveries						
% RECOVERY AB45	%	0.00	2.89	2.45	11.31	19.00	6.52	8.41
% RECOVERY AY23	%	112.6	99.0	102.8	117.6	123.5	122.2	107.1
% RECOVERY AY73	%	94.6	83.1	80.3	108.6	113.4	98.8	100.9
% RECOVERY AO10	%	122.2	128.4	119.8	132.9	132.4	126.6	130.0
% RECOVERY AB1	%	131.9	146.2	136.1	144.1	143.6	138.1	141.3
% RECOVERY BY2	%	274.1	437.3	707.9	403.4	357.3	276.2	279.8
% RECOVERY DV1	%	12.7	90.4	88.8	98.2	107.5	94.6	94.6
% RECOVERY SY33	%	110.2	129.0	125.8	133.2	131.8	124.8	126.5
% RECOVERY DB14	%	19.4	85.7	71.2	69.9	87.0	81.1	97.3
% RECOVERY AR64	%	74.0	52.1	68.3	54.8	101.8	86.0	50.9
% RECOVERY AB9	%	115.6	150.3	140.2	148.0	145.3	138.7	133.1
% RECOVERY BV10	%	117.3	143.2	132.4	138.6	138.1	130.3	135.7
% RECOVERY 2AQ	%	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SO3	%	14.0	96.1	90.3	97.4	102.1	72.1	75.2
% RECOVERY 1AQ	%	106.7	123.7	121.5	126.8	118.0	108.6	120.2
% RECOVERY SY14	%	93.5	113.3	109.4	109.1	112.8	89.8	105.2
% RECOVERY SO7	%	103.3	133.2	130.3	131.5	127.4	112.5	122.6
% RECOVERY AY3	%	126.7	152.0	124.7	143.7	135.4	130.6	139.5
% RECOVERY SY3	%	95.4	132.0	124.1	129.6	125.1	112.8	120.7
% RECOVERY SR1	%	68.3	93.9	114.8	112.4	97.2	90.7	83.5
% RECOVERY DR9	%	84.1	106.1	99.3	102.9	98.9	88.5	98.3
% RECOVERY SY2	%	43.0	93.2	90.3	89.8	92.6	74.0	86.6
% RECOVERY SG3	%	45.2	58.7	55.0	77.4	50.7	57.6	82.2
% RECOVERY SR24	%	110.6	174.6	160.7	172.8	168.8	152.0	127.7
% RECOVERY AN	%	65.3	115.0	103.7	113.2	96.6	66.6	83.2
% RECOVERY BZ	%	102.7	133.2	126.5	129.9	128.0	117.5	121.2

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
		Square of Corrected Results						
N		6	6	6	6	6	6	6
Xi^2 AB45		0.0	24.8	17.8	379.5	1070.9	126.0	209.7
Xi^2 AY23		25066.8	19372.5	20890.4	27357.2	30171.7	29539.3	22675.8
Xi^2 AY73		27536.1	21294.1	19871.1	36330.3	39603.0	30062.4	31355.6
Xi^2 AO10		35289.5	38952.9	33907.5	41703.8	41402.1	37840.0	39934.0
Xi^2 AB1		59270.3	72819.0	63159.2	70726.7	70272.7	64976.6	68040.1
Xi^2 BY2		131083.8	333639.1	874421.4	284035.7	222807.6	133141.1	136574.6
Xi^2 DV1		274.4	13836.8	13344.9	16313.7	19549.6	15151.1	15156.1
Xi^2 SY33		23717.5	32531.5	30916.2	34676.0	33926.0	30425.8	31254.7
Xi^2 DB14		840.7	16401.9	11317.8	10933.8	16919.5	14696.7	21173.2
Xi^2 AR64		17915.8	8874.6	15254.7	9810.9	33909.4	24217.6	8476.0
Xi^2 AB9		35919.7	60732.7	52840.2	58876.6	56803.6	51756.3	47644.0
Xi^2 BV10		14934.1	22271.1	19024.7	20860.0	20718.7	18429.4	19991.1
Xi^2 2AQ		0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SO3		321.5	15094.6	13343.7	15523.9	17053.7	8510.1	9249.6
Xi^2 1AQ		18803.3	25276.2	24378.1	26536.4	22997.7	19488.2	23853.3
Xi^2 SY14		17537.7	25757.0	23987.8	23865.6	25520.1	16181.1	22183.1
Xi^2 SO7		22111.7	36808.3	35210.6	35859.1	33675.9	26227.8	31184.0
Xi^2 AY3		44963.1	64770.3	43593.3	57873.9	51395.2	47799.1	54503.6
Xi^2 SY3		20736.0	39748.4	35118.8	38312.2	35702.1	28997.0	33244.2
Xi^2 SR1		25115.9	47412.9	70902.4	67938.4	50820.9	44261.8	37457.7
Xi^2 DR9		16561.1	26373.8	23070.6	24795.2	22909.8	18338.6	22618.7
Xi^2 SY2		7176.6	33745.7	31611.1	31261.8	33242.4	21231.4	29115.5
Xi^2 SG3		5042.4	8498.1	7455.5	14758.6	6344.9	8192.1	16650.0
Xi^2 SR24		986.9	2459.7	2083.9	2408.4	2297.3	1862.8	1315.9
Xi^2 AN		18394.1	57006.3	46412.2	55267.3	40284.5	19111.7	29889.2
Xi^2 BZ		15591.3	26250.5	23643.7	24937.1	24220.7	20399.0	21726.8

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
		Sum of the Square of Corrected Results						
E Xi^2 AB45		1828.6						
E Xi^2 AY23		150006.8						
E Xi^2 AY73		178516.4						
E Xi^2 AO10		233740.3						
E Xi^2 AB1		409994.4						
E Xi^2 BY2		1984619.4						
E Xi^2 DV1		93352.2						
E Xi^2 SY33		193730.2						
E Xi^2 DB14		91442.9						
E Xi^2 AR64		100543.1						
E Xi^2 AB9		328653.3						
E Xi^2 BV10		121295.1						
E Xi^2 2AQ		0.0						
E Xi^2 SO3		78775.7						
E Xi^2 1AQ		142529.9						
E Xi^2 SY14		137494.8						
E Xi^2 SO7		198965.8						
E Xi^2 AY3		319935.2						
E Xi^2 SY3		211122.7						
E Xi^2 SR1		318794.2						
E Xi^2 DR9		138106.6						
E Xi^2 SY2		180207.8						
E Xi^2 SG3		61899.1						
E Xi^2 SR24		12427.9						
E Xi^2 AN		247971.3						
E Xi^2 BZ		141177.7						

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
		Sum of the Corrected Results Squared						
(E Xi)^2 AB45		7586.4						
(E Xi)^2 AY23		893544.8						
(E Xi)^2 AY73		1054646.8						
(E Xi)^2 AO10		1400802.4						
(E Xi)^2 AB1		2458467.2						
(E Xi)^2 BY2		10576414.6						
(E Xi)^2 DV1		557852.1						
(E Xi)^2 SY33		1161696.0						
(E Xi)^2 DB14		541453.1						
(E Xi)^2 AR64		560394.5						
(E Xi)^2 AB9		1968591.4						
(E Xi)^2 BV10		727063.2						
(E Xi)^2 2AQ		0.0						
(E Xi)^2 SO3		465103.5						
(E Xi)^2 1AQ		853249.4						
(E Xi)^2 SY14		820383.1						
(E Xi)^2 SO7		1190095.5						
(E Xi)^2 AY3		1911734.8						
(E Xi)^2 SY3		1263533.4						
(E Xi)^2 SR1		1887958.4						
(E Xi)^2 DR9		826153.7						
(E Xi)^2 SY2		1075312.0						
(E Xi)^2 SG3		359058.6						
(E Xi)^2 SR24		73812.7						
(E Xi)^2 AN		1442701.3						
(E Xi)^2 BZ		845581.4						

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
					Variance			
S^2: VARIANCE AB45		112.8						
S^2: VARIANCE AY23		216.5						
S^2: VARIANCE AY73		548.4						
S^2: VARIANCE AO10		54.7						
S^2: VARIANCE AB1		50.0						
S^2: VARIANCE BY2		44376.7						
S^2: VARIANCE DV1		75.4						
S^2: VARIANCE SY33		22.8						
S^2: VARIANCE DB14		240.1						
S^2: VARIANCE AR64		1428.8						
S^2: VARIANCE AB9		110.9						
S^2: VARIANCE BV10		23.6						
S^2: VARIANCE 2AQ		0.0						
S^2: VARIANCE SO3		251.7						
S^2: VARIANCE 1AQ		64.3						
S^2: VARIANCE SY14		152.9						
S^2: VARIANCE SO7		123.3						
S^2: VARIANCE AY3		262.6						
S^2: VARIANCE SY3		106.8						
S^2: VARIANCE SR1		826.9						
S^2: VARIANCE DR9		82.9						
S^2: VARIANCE SY2		197.8						
S^2: VARIANCE SG3		411.2						
S^2: VARIANCE SR24		25.2						
S^2: VARIANCE AN		1504.2						
S^2: VARIANCE BZ		49.5						

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
		Standard Deviation						
STD DEVIATION AB45		10.6						
STD DEVIATION AY23		14.7						
STD DEVIATION AY73		23.4						
STD DEVIATION AO10		7.4						
STD DEVIATION AB1		7.1						
STD DEVIATION BY2		210.7						
STD DEVIATION DV1		8.7						
STD DEVIATION SY33		4.8						
STD DEVIATION DB14		15.5						
STD DEVIATION AR64		37.8						
STD DEVIATION AB9		10.5						
STD DEVIATION BV10		4.9						
STD DEVIATION 2AQ		0.0						
STD DEVIATION SO3		15.9						
STD DEVIATION 1AQ		8.0						
STD DEVIATION SY14		12.4						
STD DEVIATION SO7		11.1						
STD DEVIATION AY3		16.2						
STD DEVIATION SY3		10.3						
STD DEVIATION SR1		28.8						
STD DEVIATION DR9		9.1						
STD DEVIATION SY2		14.1						
STD DEVIATION SG3		20.3						
STD DEVIATION SR24		5.0						
STD DEVIATION AN		38.8						
STD DEVIATION BZ		7.0						

Table VII - 7B

Percent Recoveries Low-Level Aqueous Method Low Spike, Short Gradient

Compounds		Ret. Time	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
% RECOVERY AB45	%	4.439	142	194	95	177	60	209	15
% RECOVERY AR64	%	6.174	133	120	134	121	144	98	134
% RECOVERY AY3	%	7.322	174	127	149	213	211	135	415
% RECOVERY AY23	%	7.525	370	298	355	105	139	248	327
% RECOVERY AY73	%	9.159	255	224	264	272	231	242	235
% RECOVERY AO10	%	9.946	533	422	513	511	442	488	505
% RECOVERY AB9	%	11.576	350	284	340	329	289	328	346
% RECOVERY DV1	%	12.69	503	418	485	489	462	511	397
% RECOVERY AB1	%	12.709	379	239	296	350	313	344	362
% RECOVERY 2AQ	%	13.246	147	131	159	158	149	151	150
% RECOVERY SO3	%	13.811	202	188	184	220	146	256	228
% RECOVERY SY33	%	15.498	226	197	220	170	175	218	221
% RECOVERY 1AQ	%	16.108	346	283	340	309	304	330	338
% RECOVERY DB14	%	18.857	256	201	256	223	188	374	306
% RECOVERY BY2	%	19.452	534	473	473	1294	508	416	1538
% RECOVERY BV10	%	19.691	374	290	349	317	313	350	372
% RECOVERY SY3	%	19.962	583	449	527	497	453	569	576
% RECOVERY DR9	%	20.367	218	177	211	174	165	206	210
% RECOVERY BZ	%	20.438	409	358	415	331	328	411	410
% RECOVERY SY2	%	22.244	164	139	169	130	122	164	162
% RECOVERY AN	%	22.537	183	185	178	0	19	183	216
% RECOVERY SY14	%	24.403	191	166	193	155	79	161	160
% RECOVERY SR1	%	24.66	143	223	171	159	45	206	166
% RECOVERY SO7	%	27.804	311	328	347	215	44	334	281
% RECOVERY SG3	%	31.194	57	50	133	125	0	61	81
% RECOVERY SR24	%	31.255	356	444	224	262	0	429	175

Percent Recovery

TABLE VII - 7A

PERCENT RECOVERIES LOW-LEVEL AQUEOUS METHOD LOW SPIKE, SHORT GRADIENT

Compounds		Ret. Time	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS	Average (p)	Std. Deviation (s)	Lower Control Limit (p-2s)	Upper Control Limit (p+2s)	Average (on six points)	Std. Dev (s) (on six points)	Lower Control Limit (p-2s)	Upper Control Limit (p+2s)
% RECOVERY AB45	%	4.439	142	194	95	177	60	209	15	127	73	0	273				
% RECOVERY AR64	%	6.174	133	120	134	121	144	98	134	126	15	96	156				
% RECOVERY AY3	%	7.322	174	127	149	213	211	135	415	204	99	5	402				
% RECOVERY AY23	%	7.525	370	298	355	105	139	248	327	263	105	53	473				
% RECOVERY AY73	%	9.159	255	224	264	272	231	242	235	246	18	210	282				
% RECOVERY AO10	%	9.946	533	422	513	511	442	488	505	488	41	407	569				
% RECOVERY AB9	%	11.576	350	284	340	329	289	328	346	324	27	270	377				
% RECOVERY DV1	%	12.69	503	418	485	489	462	511	397	466	43	380	553				
% RECOVERY AB1	%	12.709	379	239	296	350	313	344	362	326	48	231	421				
% RECOVERY 2AQ	%	13.246	147	131	159	158	149	151	150	149	9	131	168				
% RECOVERY SO3	%	13.811	202	188	184	220	146	256	228	203	36	132	275				
% RECOVERY SY33	%	15.498	226	197	220	170	175	218	221	204	23	157	251				
% RECOVERY 1AQ	%	16.108	346	283	340	309	304	330	338	321	23	275	367				
% RECOVERY DB14	%	18.857	256	201	256	223	188	374	306	258	65	128	387				
% RECOVERY BY2	%	19.452	534	473	473	1294	508	416	1538	748	463	0	1674	616	334	0	1285
% RECOVERY BV10	%	19.691	374	290	349	317	313	350	372	338	32	274	402				
% RECOVERY SY3	%	19.962	583	449	527	497	453	569	576	522	57	408	636				
% RECOVERY DR9	%	20.367	216	177	211	174	165	206	210	194	22	151	237				
% RECOVERY BZ	%	20.438	409	358	415	331	328	411	410	380	40	261	500				
% RECOVERY SY2	%	22.244	164	139	169	130	122	164	162	150	19	112	188				
% RECOVERY AN	%	22.537	183	185	178	0	19	183	216	138	89	0	404	161	71	19	373
% RECOVERY SY14	%	24.403	191	166	193	155	79	161	160	158	38	62	234				
% RECOVERY SR1	%	24.66	143	223	171	159	43	206	166	159	58	43	275				
% RECOVERY SO7	%	27.804	311	328	347	215	44	334	281	266	108	51	481				
% RECOVERY SG3	%	31.194	57	50	133	125	0	61	61	73	46	0	164	85	36	13	156
% RECOVERY SR24	%	31.255	356	444	224	262	0	429	175	270	157	0	583	315	111	92	538

TABLE VII-7

Low-Level Aqueous Extraction Method, Short Gradient, Low Spike

		Sample Results (µg/L)							Mean Result µg/L	Method Bias (Accuracy) %R	Standard Deviation	Method Precision (%RSD)	MDL µg/L	RQL µg/L
Compounds	Sample Conc µg/L	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS	average sample result	(mean result/sample conc)*100	std. dev. sample results	(std.dev/mean result)*100	std.dev. *3.14	MDL*4
ACID BLUE 45	34.44	48.8	67.0	32.6	61.0	20.5	71.9	5.3	43.9	127.4	25.2	57.3	79.0	316.0
ACID YELLOW 23	28.12	103.9	83.8	99.9	29.4	39.1	69.6	92.1	74.0	263.1	29.5	39.8	92.6	370.3
ACID YELLOW 73	35.1	89.3	78.7	92.8	95.4	81.1	84.9	82.4	86.4	246.1	6.3	7.3	19.7	78.7
ACID ORANGE 10	30.74	164.0	129.8	157.7	157.0	135.9	150.1	155.1	150.0	487.8	12.5	8.3	39.2	157.0
ACID BLUE 1	36.92	139.8	88.1	109.3	129.3	115.5	126.9	133.6	120.3	326.0	17.6	14.6	55.3	221.1
BASIC YELLOW 2	26.42	141.2	124.9	124.9	341.9	134.3	109.9	406.4	197.6	748.0	122.4	61.9	384.3	1537.0
DISPERSE VIOLET 1	26.02	130.8	108.9	126.2	127.3	120.3	132.9	103.2	121.4	466.5	11.3	9.3	35.5	142.0
SOLVENT YELLOW 33	27.96	63.2	55.2	61.6	47.6	48.9	60.8	61.8	57.0	203.9	6.5	11.5	20.5	82.0
DISPERSE BLUE 14	29.9	76.5	60.1	76.4	66.7	56.3	111.8	91.4	77.0	257.6	19.3	25.1	60.7	242.7
ACID RED 64	36.18	48.2	43.4	48.5	43.8	52.0	35.4	48.6	45.7	126.3	5.4	11.9	17.1	68.4
ACID BLUE 9	32.8	114.9	93.3	111.6	107.9	94.6	107.4	113.4	106.2	323.7	8.7	8.2	27.5	109.8
BASIC VIOLET 10	20.84	78.0	60.5	72.7	66.1	65.3	73.0	77.5	70.4	338.0	6.6	9.4	20.8	83.1
2-AMINOANTHRAQUINONE	37.38	55.1	48.9	59.4	59.1	55.7	56.6	56.2	55.8	149.4	3.5	6.2	10.9	43.6
SOLVENT ORANGE 3	25.58	51.5	48.0	47.0	56.4	37.3	65.6	58.2	52.0	203.3	9.1	17.5	28.6	114.5
1-AMINOANTHRAQUINONE	25.7	88.9	72.8	87.4	79.4	78.0	84.7	86.8	82.6	321.3	5.9	7.2	18.6	74.6
SOLVENT YELLOW 14	28.32	54.2	46.9	54.6	44.0	22.4	45.6	45.4	44.7	158.0	10.7	24.0	33.7	134.7
SOLVENT ORANGE 7	28.8	89.6	94.4	100.1	61.8	12.6	96.3	81.1	76.6	265.8	31.0	40.5	97.3	389.3
ACID YELLOW 3	33.48	58.4	42.6	49.9	71.2	70.6	45.4	138.8	68.1	203.5	33.2	48.7	104.2	416.9
SOLVENT YELLOW 3	30.2	176.0	135.7	159.3	150.0	136.7	171.9	173.8	157.6	521.9	17.2	10.9	54.1	216.3
SOLVENT RED 1	46.38	66.5	103.2	79.2	73.9	19.8	95.4	77.1	73.6	158.6	26.9	36.6	84.5	338.0
DISPERSE RED 9	30.6	66.7	54.1	64.7	53.1	50.6	63.0	64.1	59.5	194.4	6.6	11.1	20.7	82.8
SOLVENT YELLOW 2	39.4	64.7	54.8	66.5	51.3	48.0	64.4	63.9	59.1	150.0	7.5	12.7	23.6	94.5
SOLVENT GREEN 3	31.4	18.0	15.7	41.7	39.2	0.0	19.2	25.5	22.8	72.5	14.4	63.1	45.1	180.5
SOLVENT RED 24	5.68	20.2	25.2	12.7	14.9	0.0	24.3	9.9	15.3	269.8	8.9	58.0	27.9	111.7
ANTHRACENE	41.54	76.1	76.7	74.1	0.0	7.8	76.2	89.7	57.2	137.7	36.9	64.4	115.7	463.0
BENZANTHRONE	24.32	99.4	86.9	100.8	80.6	79.8	100.0	99.8	92.5	380.2	9.7	10.5	30.4	121.7

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
		Method Detection Limit						
T (STUDENT'S T @ 99% FOR 6 DF)		3.360						
MDL AB45	UG/L or UG/KG	35.7						
MDL AY23	UG/L or UG/KG	49.4						
MDL AY73	UG/L or UG/KG	78.7						
MDL AO10	UG/L or UG/KG	24.8						
MDL AB1	UG/L or UG/KG	23.8						
MDL BY2	UG/L or UG/KG	707.8						
MDL DV1	UG/L or UG/KG	29.2						
MDL SY33	UG/L or UG/KG	16.1						
MDL DB14	UG/L or UG/KG	52.1						
MDL AR64	UG/L or UG/KG	127.0						
MDL AB9	UG/L or UG/KG	35.4						
MDL BV10	UG/L or UG/KG	16.3						
MDL 2AQ	UG/L or UG/KG	0.0						
MDL SO3	UG/L or UG/KG	53.3						
MDL 1AQ	UG/L or UG/KG	27.0						
MDL SY14	UG/L or UG/KG	41.5						
MDL SO7	UG/L or UG/KG	37.3						
MDL AY3	UG/L or UG/KG	54.4						
MDL SY3	UG/L or UG/KG	34.7						
MDL SR1	UG/L or UG/KG	96.6						
MDL DR9	UG/L or UG/KG	30.6						
MDL SY2	UG/L or UG/KG	47.3						
MDL SG3	UG/L or UG/KG	68.1						
MDL SR24	UG/L or UG/KG	16.9						
MDL AN	UG/L or UG/KG	130.3						
MDL BZ	UG/L or UG/KG	23.6						

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Stock Standard Concentrations						
ACID BLUE 45 STD	344.4	344.4	344.4	344.4	344.4	344.4	344.4
ACID YELLOW 23 STD	281.2	281.2	281.2	281.2	281.2	281.2	281.2
ACID YELLOW 73 STD	351.0	351.0	351.0	351.0	351.0	351.0	351.0
ACID ORANGE 10 STD	307.4	307.4	307.4	307.4	307.4	307.4	307.4
ACID BLUE 1 STD	369.2	369.2	369.2	369.2	369.2	369.2	369.2
BASIC YELLOW 2 STD	264.2	264.2	264.2	264.2	264.2	264.2	264.2
DISPERSE VIOLET 1 STD	260.2	260.2	260.2	260.2	260.2	260.2	260.2
SOLVENT YELLOW 33 STD	279.6	279.6	279.6	279.6	279.6	279.6	279.6
DISPERSE BLUE 14 STD	299.0	299.0	299.0	299.0	299.0	299.0	299.0
ACID RED 64 STD	361.8	361.8	361.8	361.8	361.8	361.8	361.8
ACID BLUE 9 STD	328.0	328.0	328.0	328.0	328.0	328.0	328.0
BASIC VIOLET 10 STD	208.4	208.4	208.4	208.4	208.4	208.4	208.4
2-AMINOANTHRAQUINONE STD	373.8	373.8	373.8	373.8	373.8	373.8	373.8
SOLVENT ORANGE 3 STD	255.8	255.8	255.8	255.8	255.8	255.8	255.8
1-AMINOANTHRAQUINONE STD	257.0	257.0	257.0	257.0	257.0	257.0	257.0
SOLVENT YELLOW 14 STD	283.2	283.2	283.2	283.2	283.2	283.2	283.2
SOLVENT ORANGE 7 STD	288.0	288.0	288.0	288.0	288.0	288.0	288.0
ACID YELLOW 3 STD	334.8	334.8	334.8	334.8	334.8	334.8	334.8
SOLVENT YELLOW 3 STD	302.0	302.0	302.0	302.0	302.0	302.0	302.0
SOLVENT RED 1 STD	463.8	463.8	463.8	463.8	463.8	463.8	463.8
DISPERSE RED 9 STD	306.0	306.0	306.0	306.0	306.0	306.0	306.0
SOLVENT YELLOW 2 STD	394.0	394.0	394.0	394.0	394.0	394.0	394.0
SOLVENT GREEN 3 STD	314.0	314.0	314.0	314.0	314.0	314.0	314.0
SOLVENT RED 24 STD	56.8	56.8	56.8	56.8	56.8	56.8	56.8
ANTHRACENE	415.4	415.4	415.4	415.4	415.4	415.4	415.4
BENZANTHRONE	243.2	243.2	243.2	243.2	243.2	243.2	243.2

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Weight of Stock Standard Spiked						
SPIKE VOLUME EACH DYE	100.0	100.0	100.0	100.0	100.0	100.0	100.0
AB45 SPIKE WEIGHT	34.4	34.4	34.4	34.4	34.4	34.4	34.4
AY23 SPIKE WEIGHT	28.1	28.1	28.1	28.1	28.1	28.1	28.1
AY73 SPIKE WEIGHT	35.1	35.1	35.1	35.1	35.1	35.1	35.1
AO10 SPIKE WEIGHT	30.7	30.7	30.7	30.7	30.7	30.7	30.7
AB1 SPIKE WEIGHT	36.9	36.9	36.9	36.9	36.9	36.9	36.9
BY2 SPIKE WEIGHT	26.4	26.4	26.4	26.4	26.4	26.4	26.4
DV1 SPIKE WEIGHT	26.0	26.0	26.0	26.0	26.0	26.0	26.0
SY33 SPIKE WEIGHT	28.0	28.0	28.0	28.0	28.0	28.0	28.0
DB14 SPIKE WEIGHT	29.9	29.9	29.9	29.9	29.9	29.9	29.9
AR64 SPIKE WEIGHT	36.2	36.2	36.2	36.2	36.2	36.2	36.2
AB9 SPIKE WEIGHT	32.8	32.8	32.8	32.8	32.8	32.8	32.8
BV10 SPIKE WEIGHT	20.8	20.8	20.8	20.8	20.8	20.8	20.8
2AQ SPIKE WEIGHT	37.4	37.4	37.4	37.4	37.4	37.4	37.4
SO3 SPIKE WEIGHT	25.6	25.6	25.6	25.6	25.6	25.6	25.6
1AQ SPIKE WEIGHT	25.7	25.7	25.7	25.7	25.7	25.7	25.7
SY14 SPIKE WEIGHT	28.3	28.3	28.3	28.3	28.3	28.3	28.3
SO7 SPIKE WEIGHT	28.8	28.8	28.8	28.8	28.8	28.8	28.8
AY3 SPIKE WEIGHT	33.5	33.5	33.5	33.5	33.5	33.5	33.5
SY3 SPIKE WEIGHT	30.2	30.2	30.2	30.2	30.2	30.2	30.2
SR1 SPIKE WEIGHT	46.4	46.4	46.4	46.4	46.4	46.4	46.4
DR9 SPIKE WEIGHT	30.6	30.6	30.6	30.6	30.6	30.6	30.6
SY2 SPIKE WEIGHT	39.4	39.4	39.4	39.4	39.4	39.4	39.4
SG3 SPIKE WEIGHT	31.4	31.4	31.4	31.4	31.4	31.4	31.4
SR24 SPIKE WEIGHT	5.7	5.7	5.7	5.7	5.7	5.7	5.7
AN SPIKE WEIGHT	41.5	41.5	41.5	41.5	41.5	41.5	41.5
BZ SPIKE WEIGHT	24.3	24.3	24.3	24.3	24.3	24.3	24.3

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Weight Standard per Volume Sample						
MATRIX VOLUME (WATER)	1.000	1.000	1.000	1.000	1.000	1.000	1.000
SPIKE CONC AB45	34.4	34.4	34.4	34.4	34.4	34.4	34.4
SPIKE CONC AY23	28.1	28.1	28.1	28.1	28.1	28.1	28.1
SPIKE CONC AY73	35.1	35.1	35.1	35.1	35.1	35.1	35.1
SPIKE CONC AO10	30.7	30.7	30.7	30.7	30.7	30.7	30.7
SPIKE CONC AB1	36.9	36.9	36.9	36.9	36.9	36.9	36.9
SPIKE CONC BY2	26.4	26.4	26.4	26.4	26.4	26.4	26.4
SPIKE CONC DV1	26.0	26.0	26.0	26.0	26.0	26.0	26.0
SPIKE CONC SY33	28.0	28.0	28.0	28.0	28.0	28.0	28.0
SPIKE CONC DB14	29.9	29.9	29.9	29.9	29.9	29.9	29.9
SPIKE CONC AR64	36.2	36.2	36.2	36.2	36.2	36.2	36.2
SPIKE CONC AB9	32.8	32.8	32.8	32.8	32.8	32.8	32.8
SPIKE CONC BV10	20.8	20.8	20.8	20.8	20.8	20.8	20.8
SPIKE CONC 2AQ	37.4	37.4	37.4	37.4	37.4	37.4	37.4
SPIKE CONC SO3	25.6	25.6	25.6	25.6	25.6	25.6	25.6
SPIKE CONC 1AQ	25.7	25.7	25.7	25.7	25.7	25.7	25.7
SPIKE CONC SY14	28.3	28.3	28.3	28.3	28.3	28.3	28.3
SPIKE CONC SO7	28.8	28.8	28.8	28.8	28.8	28.8	28.8
SPIKE CONC AY3	33.5	33.5	33.5	33.5	33.5	33.5	33.5
SPIKE CONC SY3	30.2	30.2	30.2	30.2	30.2	30.2	30.2
SPIKE CONC SR1	46.4	46.4	46.4	46.4	46.4	46.4	46.4
SPIKE CONC DR9	30.6	30.6	30.6	30.6	30.6	30.6	30.6
SPIKE CONC SY2	39.4	39.4	39.4	39.4	39.4	39.4	39.4
SPIKE CONC SG3	31.4	31.4	31.4	31.4	31.4	31.4	31.4
SPIKE CONC SR24	5.7	5.7	5.7	5.7	5.7	5.7	5.7
SPIKE CONC AN	41.5	41.5	41.5	41.5	41.5	41.5	41.5
SPIKE CONC BZ	24.3	24.3	24.3	24.3	24.3	24.3	24.3

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
SPIKE CONC AB45	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY23	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY73	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AO10	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BY2	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DV1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY33	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DB14	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AR64	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB9	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BV10	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 2AQ	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 1AQ	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY14	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO7	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DR9	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY2	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SG3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR24	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AN	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BZ	NA	NA	NA	NA	NA	NA	NA

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Results from HPLC						
HPLC RESULT AB45	9.767	13.392	6.519	12.203	4.106	14.374	1.055
HPLC RESULT AY23	20.782	16.764	19.989	5.888	7.817	13.921	18.416
HPLC RESULT AY73	17.867	15.744	18.557	19.086	16.229	16.982	16.481
HPLC RESULT AO10	32.799	25.964	31.547	31.407	27.181	30.019	31.020
HPLC RESULT AB1	27.967	17.629	21.854	25.854	23.090	25.373	26.711
HPLC RESULT BY2	28.240	24.971	24.970	. 68.373	26.864	21.984	81.272
HPLC RESULT DV1	26.161	21.775	25.244	25.461	24.059	26.581	20.649
HPLC RESULT SY33	12.641	11.035	12.315	9.514	9.772	12.166	12.368
HPLC RESULT DB14	15.309	12.017	15.283	13.330	11.252	22.351	18.282
HPLC RESULT AR64	9.646	8.673	9.701	8.755	10.398	7.078	9.720
HPLC RESULT AB9	22.978	18.653	22.322	21.571	18.950	21.485	22.675
HPLC RESULT BV10	15.594	12.100	14.542	13.211	13.060	14.602	15.502
HPLC RESULT 2AQ	11.023	9.777	11.870	11.812	11.136	11.323	11.237
HPLC RESULT SO3	10.309	9.608	9.406	11.270	7.457	13.114	11.641
HPLC RESULT 1AQ	17.775	14.557	17.473	15.886	15.602	16.944	17.355
HPLC RESULT SY14	10.845	9.378	10.914	8.793	4.489	9.128	9.087
HPLC RESULT SO7	17.926	18.886	20.011	12.359	2.516	19.260	16.214
HPLC RESULT AY3	11.684	8.529	9.975	14.247	14.111	9.071	27.769
HPLC RESULT SY3	35.194	27.141	31.856	30.001	27.348	34.370	34.769
HPLC RESULT SR1	13.301	20.647	15.846	14.771	3.952	19.080	15.415
HPLC RESULT DR9	13.340	10.829	12.934	10.627	10.114	12.594	12.826
HPLC RESULT SY2	12.948	10.967	13.296	10.265	9.597	12.889	12.773
HPLC RESULT SG3	3.604	3.149	8.333	7.847	0.000	3.840	5.102
HPLC RESULT SR24	4.042	5.046	2.539	2.974	0.000	4.868	1.986
HPLC RESULT AN	15.225	15.34	14.814	0.000	1.555	15.24	17.934
HPLC RESULT BZ	19.887	17.389	20.162	16.11	15.951	20.004	19.958

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Final Sample Results						
CF (STD INJ VOL VS SAM INJ VOL)	2	2	2	2	2	2	2
FINAL VOLUME	10.00	10.00	10.00	10.00	10.00	10.00	10.00
CORRECTED RESULT AB45	48.84	66.96	32.60	61.02	20.53	71.87	5.28
CORRECTED RESULT AY23	103.91	83.82	99.95	29.44	39.09	69.61	92.08
CORRECTED RESULT AY73	89.34	78.72	92.79	95.43	81.15	84.91	82.41
CORRECTED RESULT AO10	164.00	129.82	157.74	157.04	135.91	150.10	155.10
CORRECTED RESULT AB1	139.84	88.15	109.27	129.27	115.45	126.87	133.56
CORRECTED RESULT BY2	141.20	124.86	124.85	341.87	134.32	109.92	406.36
CORRECTED RESULT DV1	130.81	108.88	126.22	127.31	120.30	132.91	103.25
CORRECTED RESULT SY33	63.21	55.18	61.58	47.57	48.86	60.83	61.84
CORRECTED RESULT DB14	76.55	60.09	76.42	66.65	56.26	111.76	91.41
CORRECTED RESULT AR64	48.23	43.37	48.51	43.78	51.99	35.39	48.60
CORRECTED RESULT AB9	114.89	93.27	111.61	107.86	94.75	107.43	113.38
CORRECTED RESULT BV10	77.97	60.50	72.71	66.06	65.30	73.01	77.51
CORRECTED RESULT 2AQ	55.12	48.89	59.35	59.06	55.68	56.62	56.19
CORRECTED RESULT SO3	51.55	48.04	47.03	56.35	37.29	65.57	58.21
CORRECTED RESULT 1AQ	88.88	72.79	87.37	79.43	78.01	84.72	86.78
CORRECTED RESULT SY14	54.23	46.89	54.57	43.97	22.45	45.64	45.44
CORRECTED RESULT SO7	89.63	94.43	100.06	61.80	12.58	96.30	81.07
CORRECTED RESULT AY3	58.42	42.65	49.88	71.24	70.56	45.36	138.85
CORRECTED RESULT SY3	175.97	135.71	159.28	150.01	136.74	171.85	173.85
CORRECTED RESULT SR1	66.51	103.24	79.23	73.86	19.76	95.40	77.08
CORRECTED RESULT DR9	66.70	54.15	64.67	53.14	50.57	62.97	64.13
CORRECTED RESULT SY2	64.74	54.84	66.48	51.33	47.99	64.45	63.87
CORRECTED RESULT SG3	18.02	15.75	41.67	39.24	0.00	19.20	25.51
CORRECTED RESULT SR24	20.21	25.23	12.70	14.87	0.00	24.34	9.93
CORRECTED RESULT AN	76.13	76.70	74.07	0.00	7.78	76.20	89.67
CORRECTED RESULT BZ	99.44	86.95	100.81	80.55	79.76	100.02	99.79

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Percent Recoveries						
% RECOVERY AB45	141.80	194.43	94.64	177.16	59.61	208.68	15.32
% RECOVERY AY23	369.5	298.1	355.4	104.7	139.0	247.5	327.5
% RECOVERY AY73	254.5	224.3	264.3	271.9	231.2	241.9	234.8
% RECOVERY AO10	533.5	422.3	513.1	510.8	442.1	488.3	504.6
% RECOVERY AB1	378.8	238.7	296.0	350.1	312.7	343.6	361.7
% RECOVERY BY2	534.4	472.6	472.6	1294.0	508.4	416.0	1538.1
% RECOVERY DV1	502.7	418.4	485.1	489.3	462.3	510.8	396.8
% RECOVERY SY33	226.1	197.3	220.2	170.1	174.7	217.6	221.2
% RECOVERY DB14	256.0	201.0	255.6	222.9	188.2	373.8	305.7
% RECOVERY AR64	133.3	119.9	134.1	121.0	143.7	97.8	134.3
% RECOVERY AB9	350.3	284.3	340.3	328.8	288.9	327.5	345.7
% RECOVERY BV10	374.1	290.3	348.9	317.0	313.3	350.3	371.9
% RECOVERY 2AQ	147.4	130.8	158.8	158.0	149.0	151.5	150.3
% RECOVERY SO3	201.5	187.8	183.9	220.3	145.8	256.3	227.5
% RECOVERY 1AQ	345.8	283.2	339.9	309.1	303.5	329.6	337.6
% RECOVERY SY14	191.5	165.6	192.7	155.2	79.3	161.2	160.4
% RECOVERY SO7	311.2	327.9	347.4	214.6	43.7	334.4	281.5
% RECOVERY AY3	174.5	127.4	149.0	212.8	210.7	135.5	414.7
% RECOVERY SY3	582.7	449.4	527.4	496.7	452.8	569.0	575.6
% RECOVERY SR1	143.4	222.6	170.8	159.2	42.6	205.7	166.2
% RECOVERY DR9	218.0	176.9	211.3	173.6	165.3	205.8	209.6
% RECOVERY SY2	164.3	139.2	168.7	130.3	121.8	163.6	162.1
% RECOVERY SG3	57.4	50.1	132.7	125.0	0.0	61.1	81.2
% RECOVERY SR24	355.8	444.2	223.5	261.8	0.0	428.5	174.8
% RECOVERY AN	183.3	184.6	178.3	0.0	18.7	183.4	215.9
% RECOVERY BZ	408.9	357.5	414.5	331.2	327.9	411.3	410.3

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Square of Corrected Results						
N	7	7	7	7	7	7	7
Xi^2 AB45	2384.9	4483.6	1062.4	3722.8	421.5	5165.3	27.8
Xi^2 AY23	10797.3	7025.8	9989.0	866.7	1527.6	4844.9	8478.7
Xi^2 AY73	7980.7	6196.8	8609.1	9106.9	6584.5	7209.7	6790.6
Xi^2 AO10	26894.4	16853.2	24880.3	24660.0	18470.2	22528.5	24056.0
Xi^2 AB1	19553.8	7769.5	11939.9	16710.7	13328.7	16094.7	17836.9
Xi^2 BY2	19937.4	15588.8	15587.5	116871.7	18041.9	12082.4	165128.4
Xi^2 DV1	17109.9	11853.8	15931.5	16206.6	14470.9	17663.7	10659.5
Xi^2 SY33	3994.9	3044.3	3791.5	2262.9	2387.3	3700.3	3824.2
Xi^2 DB14	5859.1	3610.2	5839.3	4442.2	3165.2	12489.2	8355.8
Xi^2 AR64	2326.1	1880.5	2352.7	1916.3	2703.0	1252.5	2362.0
Xi^2 AB9	13199.7	8698.4	12456.8	11632.7	8977.6	11540.1	12853.9
Xi^2 BV10	6079.3	3660.3	5286.7	4363.3	4264.1	5330.5	6007.8
Xi^2 2AQ	3037.7	2389.7	3522.4	3488.1	3100.3	3205.3	3156.8
Xi^2 SO3	2656.9	2307.8	2211.8	3175.3	1390.2	4299.4	3387.8
Xi^2 1AQ	7898.8	5297.7	7632.6	6309.1	6085.6	7177.5	7529.9
Xi^2 SY14	2940.4	2198.7	2977.9	1932.9	503.8	2083.0	2064.3
Xi^2 SO7	8033.5	8917.0	10011.0	3818.6	158.3	9273.7	6572.3
Xi^2 AY3	3412.9	1818.6	2487.5	5074.4	4978.0	2057.1	19277.9
Xi^2 SY3	30965.4	18415.8	25370.1	22501.5	18697.8	29532.4	30222.1
Xi^2 SR1	4422.9	10657.5	6277.4	5454.6	390.5	9101.2	5940.6
Xi^2 DR9	4448.9	2931.7	4182.2	2823.3	2557.3	3965.2	4112.7
Xi^2 SY2	4191.3	3006.9	4419.6	2634.3	2302.6	4153.2	4078.7
Xi^2 SG3	324.7	247.9	1736.0	1539.4	0.0	368.6	650.8
Xi^2 SR24	408.4	636.6	161.2	221.1	0.0	592.4	98.6
Xi^2 AN	5795.0	5882.9	5486.4	0.0	60.5	5806.4	8040.7
Xi^2 BZ	9887.3	7559.4	10162.7	6488.3	6360.9	10004.0	9958.0

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Sum of the Square of Corrected Results						
E Xi^2 AB45	17268.4						
E Xi^2 AY23	43530.0						
E Xi^2 AY73	52478.3						
E Xi^2 AO10	158342.6						
E Xi^2 AB1	103234.4						
E Xi^2 BY2	363238.1						
E Xi^2 DV1	103895.9						
E Xi^2 SY33	23005.3						
E Xi^2 DB14	43761.0						
E Xi^2 AR64	14793.0						
E Xi^2 AB9	79359.1						
E Xi^2 BV10	34991.9						
E Xi^2 2AQ	21900.2						
E Xi^2 SO3	19429.3						
E Xi^2 1AQ	47931.1						
E Xi^2 SY14	14701.0						
E Xi^2 SO7	46784.5						
E Xi^2 AY3	39106.5						
E Xi^2 SY3	175705.2						
E Xi^2 SR1	42244.5						
E Xi^2 DR9	25021.3						
E Xi^2 SY2	24786.4						
E Xi^2 SG3	4867.4						
E Xi^2 SR24	2118.3						
E Xi^2 AN	31071.9						
E Xi^2 BZ	60420.6						

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Sum of the Corrected Results Squared						
(E Xi)^2 AB45	94298.1						
(E Xi)^2 AY23	268204.9						
(E Xi)^2 AY73	365698.4						
(E Xi)^2 AO10	1101838.6						
(E Xi)^2 AB1	709620.9						
(E Xi)^2 BY2	1913712.6						
(E Xi)^2 DV1	721905.1						
(E Xi)^2 SY33	159244.9						
(E Xi)^2 DB14	290650.4						
(E Xi)^2 AR64	102307.2						
(E Xi)^2 AB9	552301.6						
(E Xi)^2 BV10	243103.2						
(E Xi)^2 2AQ	152795.0						
(E Xi)^2 SO3	132514.2						
(E Xi)^2 1AQ	334037.8						
(E Xi)^2 SY14	98075.4						
(E Xi)^2 SO7	287145.9						
(E Xi)^2 AY3	227462.2						
(E Xi)^2 SY3	1217480.5						
(E Xi)^2 SR1	265286.8						
(E Xi)^2 DR9	173322.3						
(E Xi)^2 SY2	171127.0						
(E Xi)^2 SG3	25400.4						
(E Xi)^2 SR24	11507.9						
(E Xi)^2 AN	160432.3						
(E Xi)^2 BZ	419003.8						

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Variance						
S^2: VARIANCE AB45	632.9						
S^2: VARIANCE AY23	869.2						
S^2: VARIANCE AY73	39.3						
S^2: VARIANCE AO10	156.2						
S^2: VARIANCE AB1	310.0						
S^2: VARIANCE BY2	14975.1						
S^2: VARIANCE DV1	127.8						
S^2: VARIANCE SY33	42.7						
S^2: VARIANCE DB14	373.2						
S^2: VARIANCE AR64	29.6						
S^2: VARIANCE AB9	76.5						
S^2: VARIANCE BV10	43.8						
S^2: VARIANCE 2AQ	12.1						
S^2: VARIANCE SO3	83.1						
S^2: VARIANCE 1AQ	35.2						
S^2: VARIANCE SY14	115.0						
S^2: VARIANCE SO7	960.6						
S^2: VARIANCE AY3	1102.0						
S^2: VARIANCE SY3	296.6						
S^2: VARIANCE SR1	724.4						
S^2: VARIANCE DR9	43.5						
S^2: VARIANCE SY2	56.6						
S^2: VARIANCE SG3	206.5						
S^2: VARIANCE SR24	79.1						
S^2: VARIANCE AN	1358.8						
S^2: VARIANCE BZ	93.8						

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Standard Deviation						
STD DEVIATION AB45	25.2						
STD DEVIATION AY23	29.5						
STD DEVIATION AY73	6.3						
STD DEVIATION AO10	12.5						
STD DEVIATION AB1	17.6						
STD DEVIATION BY2	122.4						
STD DEVIATION DV1	11.3						
STD DEVIATION SY33	6.5						
STD DEVIATION DB14	19.3						
STD DEVIATION AR64	5.4						
STD DEVIATION AB9	8.7						
STD DEVIATION BV10	6.6						
STD DEVIATION 2AQ	3.5						
STD DEVIATION SO3	9.1						
STD DEVIATION 1AQ	5.9						
STD DEVIATION SY14	10.7						
STD DEVIATION SO7	31.0						
STD DEVIATION AY3	33.2						
STD DEVIATION SY3	17.2						
STD DEVIATION SR1	26.9						
STD DEVIATION DR9	6.6						
STD DEVIATION SY2	7.5						
STD DEVIATION SG3	14.4						
STD DEVIATION SR24	8.9						
STD DEVIATION AN	36.9						
STD DEVIATION BZ	9.7						

	WLM001LS	WLM002LS	WLM003LS	WLM004LS	WLM005LS	WLM006LS	WLM008LS
	Method Detection Limit						
T (STUDENT'S T @ 99% FOR 6 DF)	3.140						
MDL AB45	79.0						
MDL AY23	92.6						
MDL AY73	19.7						
MDL AO10	39.2						
MDL AB1	55.3						
MDL BY2	384.3						
MDL DV1	35.5						
MDL SY33	20.5						
MDL DB14	60.7						
MDL AR64	17.1						
MDL AB9	27.5						
MDL BV10	20.8						
MDL 2AQ	10.9						
MDL SO3	28.6						
MDL 1AQ	18.6						
MDL SY14	33.7						
MDL SO7	97.3						
MDL AY3	104.2						
MDL SY3	54.1						
MDL SR1	84.5						
MDL DR9	20.7						
MDL SY2	23.6						
MDL SG3	45.1						
MDL SR24	27.9						
MDL AN	115.7						
MDL BZ	30.4						

TABLE VII-8

Low-Level Aqueous Extraction Method, Short Gradient, High Spike

		Sample Results (µg/L)							Mean Result µg/L	Method Bias (Accuracy) %R	Standard Deviation	Method Precision (%RSD)
Compounds	Sample Conc µg/L	WLM004HS [1]	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS	average sample result	(mean result/sample conc)*100	std. dev. sample results	(std.dev/mean result)*100
ACID BLUE 45	172.2	0	18.0	10.6	99.6	137.3	40.3	65.9	61.9	36.0	49.3	79.6
ACID YELLOW 23	140.6	158.3	164.1	139.4	181.3	244.6	181.5	229.4	190.0	135.2	39.8	20.9
ACID YELLOW 73	175.5	175.7	158.3	156.4	210.0	225.0	194.8	199.1	190.6	108.6	27.8	14.6
ACID ORANGE 10	153.7	343.0	367.0	350.8	387.1	397.1	377.6	387.8	377.9	245.9	16.8	4.4
ACID BLUE 1	184.6	286.7	329.4	316.9	331.4	338.6	324.2	331.3	328.6	178.0	7.4	2.2
BASIC YELLOW 2	132.1	385.9	593.0	1104.4	566.5	496.0	388.1	401.0	591.5	447.8	264.7	44.8
DISPERSE VIOLET 1	130.1	0	300.1	298.9	323.5	358.1	319.0	320.2	320.0	245.9	21.5	6.7
SOLVENT YELLOW 33	139.8	125.0	151.1	150.8	157.8	160.2	150.6	153.2	154.0	110.1	4.1	2.7
DISPERSE BLUE 14	149.5	47.1	229.2	193.4	188.5	242.5	224.0	272.2	225.0	150.5	31.3	13.9
ACID RED 64	180.9	74.0	78.8	102.6	98.7	95.4	85.5	88.0	91.5	50.6	8.9	9.8
ACID BLUE 9	164	206.8	278.9	267.5	282.3	279.6	266.7	259.5	272.4	166.1	9.1	3.3
BASIC VIOLET 10	104.2	151.2	189.1	179.8	186.3	190.5	179.1	186.1	185.2	177.7	4.7	2.6
2-AMINOANTHRAQUINONE	186.9	139.5	124.7	142.2	158.0	167.4	139.6	136.1	144.7	77.4	15.5	10.7
SOLVENT ORANGE 3	127.9	19.8	139.3	133.5	143.3	153.9	109.3	115.4	132.5	103.6	17.0	12.9
1-AMINOANTHRAQUINONE	128.5	181.4	227.7	217.3	223.9	214.1	195.9	216.0	215.6	167.9	11.0	5.1
SOLVENT YELLOW 14	141.6	116.5	147.2	142.8	140.4	151.5	120.3	141.7	140.6	99.3	10.8	7.7
SOLVENT ORANGE 7	144	164.6	220.2	218.8	264.3	262.1	192.6	251.7	235.0	163.2	28.8	12.3
ACID YELLOW 3	167.4	114.8	136.9	162.1	142.9	153.5	141.4	138.2	145.8	87.1	9.9	6.8
SOLVENT YELLOW 3	151	309.2	441.5	426.3	439.3	434.2	388.0	417.4	424.5	281.1	19.9	4.7
SOLVENT RED 1	231.9	147.1	206.1	260.8	255.0	225.3	209.0	187.2	224.1	96.6	29.3	13.1
DISPERSE RED 9	153	127.5	163.5	158.6	162.5	158.8	141.7	157.1	157.0	102.6	7.9	5.0
SOLVENT YELLOW 2	197	77.9	173.1	171.7	168.7	178.6	142.2	166.4	166.8	84.7	12.7	7.6
SOLVENT GREEN 3	157	80.6	119.7	111.2	160.9	105.7	124.9	170.5	132.2	84.2	27.0	20.4
SOLVENT RED 24	28.4	38.1	65.8	61.8	67.6	67.1	58.9	49.3	61.7	217.4	7.0	11.3
ANTHRACENE	207.7	154.9	257.1	243.5	256.9	232.4	168.8	205.8	227.4	109.5	34.4	15.1
BENZANTHRONE	121.6	198.0	261.7	253.9	258.2	261.9	239.7	248.5	254.0	208.8	8.7	3.4

1 Sample results not used for calculations due to problems encountered during extraction.

TABLE VII - 8A

PERCENT RECOVERIES
LOW-LEVEL AQUEOUS METHOD
HIGH SPIKE, SHORT GRADIENT

Compounds		Ret. Time	WLM004HS [1]	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
% RECOVERY AB45	%	4.439	0	10	6	58	80	23	38
% RECOVERY AR64	%	6.174	41	44	57	55	53	47	49
% RECOVERY AY3	%	7.322	69	82	97	85	92	84	83
% RECOVERY AY23	%	7.525	113	117	99	129	174	129	163
% RECOVERY AY73	%	9.159	100	90	89	120	128	111	113
% RECOVERY AO10	%	9.946	223	239	228	252	258	246	252
% RECOVERY AB9	%	11.576	126	170	163	172	170	163	158
% RECOVERY DV1	%	12.69	0	231	230	249	275	245	246
% RECOVERY AB1	%	12.709	155	178	172	180	183	176	179
% RECOVERY 2AQ	%	13.246	75	67	76	85	90	75	73
% RECOVERY SO3	%	13.811	16	109	104	112	120	85	90
% RECOVERY SY33	%	15.498	89	108	108	113	115	108	110
% RECOVERY 1AQ	%	16.108	141	177	169	174	167	152	168
% RECOVERY DB14	%	18.857	31	153	129	126	162	150	182
% RECOVERY BY2	%	19.452	292	449	836	429	376	294	304
% RECOVERY BV10	%	19.691	145	181	173	179	183	172	179
% RECOVERY SY3	%	19.962	205	292	282	291	288	257	276
% RECOVERY DR9	%	20.367	83	107	104	106	104	93	103
% RECOVERY BZ	%	20.438	163	215	208	212	215	197	204
% RECOVERY SY2	%	22.244	40	88	87	86	91	72	84
% RECOVERY AN	%	22.537	75	124	117	124	112	81	99
% RECOVERY SY14	%	24.403	82	104	101	99	107	85	100
% RECOVERY SR1	%	24.66	63	89	112	110	97	90	81
% RECOVERY SO7	%	27.804	114	153	152	184	182	134	175
% RECOVERY SG3	%	31.194	51	76	71	102	67	80	109
% RECOVERY SR24	%	31.255	134	232	219	238	236	207	173

1 Sample not used due to problems during extraction

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
		Stock Standard Concentrations						
ACID BLUE 45 STD	UG/ML	344.4	344.4	344.4	344.4	344.4	344.4	344.4
ACID YELLOW 23 STD	UG/ML	281.2	281.2	281.2	281.2	281.2	281.2	281.2
ACID YELLOW 73 STD	UG/ML	351.0	351.0	351.0	351.0	351.0	351.0	351.0
ACID ORANGE 10 STD	UG/ML	307.4	307.4	307.4	307.4	307.4	307.4	307.4
ACID BLUE 1 STD	UG/ML	369.2	369.2	369.2	369.2	369.2	369.2	369.2
BASIC YELLOW 2 STD	UG/ML	264.2	264.2	264.2	264.2	264.2	264.2	264.2
DISPERSE VIOLET 1 STD	UG/ML	260.2	260.2	260.2	260.2	260.2	260.2	260.2
SOLVENT YELLOW 33 STD	UG/ML	279.6	279.6	279.6	279.6	279.6	279.6	279.6
DISPERSE BLUE 14 STD	UG/ML	299.0	299.0	299.0	299.0	299.0	299.0	299.0
ACID RED 64 STD	UG/ML	361.8	361.8	361.8	361.8	361.8	361.8	361.8
ACID BLUE 9 STD	UG/ML	328.0	328.0	328.0	328.0	328.0	328.0	328.0
BASIC VIOLET 10 STD	UG/ML	208.4	208.4	208.4	208.4	208.4	208.4	208.4
2-AMINOANTHRAQUINONE STD	UG/ML	373.8	373.8	373.8	373.8	373.8	373.8	373.8
SOLVENT ORANGE 3 STD	UG/ML	255.8	255.8	255.8	255.8	255.8	255.8	255.8
1-AMINOANTHRAQUINONE STD	UG/ML	257.0	257.0	257.0	257.0	257.0	257.0	257.0
SOLVENT YELLOW 14 STD	UG/ML	283.2	283.2	283.2	283.2	283.2	283.2	283.2
SOLVENT ORANGE 7 STD	UG/ML	288.0	288.0	288.0	288.0	288.0	288.0	288.0
ACID YELLOW 3 STD	UG/ML	334.8	334.8	334.8	334.8	334.8	334.8	334.8
SOLVENT YELLOW 3 STD	UG/ML	302.0	302.0	302.0	302.0	302.0	302.0	302.0
SOLVENT RED 1 STD	UG/ML	463.8	463.8	463.8	463.8	463.8	463.8	463.8
DISPERSE RED 9 STD	UG/ML	306.0	306.0	306.0	306.0	306.0	306.0	306.0
SOLVENT YELLOW 2 STD	UG/ML	394.0	394.0	394.0	394.0	394.0	394.0	394.0
SOLVENT GREEN 3 STD	UG/ML	314.0	314.0	314.0	314.0	314.0	314.0	314.0
SOLVENT RED 24 STD	UG/ML	56.8	56.8	56.8	56.8	56.8	56.8	56.8
ANTHRACENE	UG/ML	415.4	415.4	415.4	415.4	415.4	415.4	415.4
BENZANTHRONE	UG/ML	243.2	243.2	243.2	243.2	243.2	243.2	243.2

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
		Weight of Stock Standard Spiked						
SPIKE VOLUME EACH DYE	UL	500.0	500.0	500.0	500.0	500.0	500.0	500.0
AB45 SPIKE WEIGHT	UG	172.2	172.2	172.2	172.2	172.2	172.2	172.2
AY23 SPIKE WEIGHT	UG	140.6	140.6	140.6	140.6	140.6	140.6	140.6
AY73 SPIKE WEIGHT	UG	175.5	175.5	175.5	175.5	175.5	175.5	175.5
AO10 SPIKE WEIGHT	UG	153.7	153.7	153.7	153.7	153.7	153.7	153.7
AB1 SPIKE WEIGHT	UG	184.6	184.6	184.6	184.6	184.6	184.6	184.6
BY2 SPIKE WEIGHT	UG	132.1	132.1	132.1	132.1	132.1	132.1	132.1
DV1 SPIKE WEIGHT	UG	130.1	130.1	130.1	130.1	130.1	130.1	130.1
SY33 SPIKE WEIGHT	UG	139.8	139.8	139.8	139.8	139.8	139.8	139.8
DB14 SPIKE WEIGHT	UG	149.5	149.5	149.5	149.5	149.5	149.5	149.5
AR64 SPIKE WEIGHT	UG	180.9	180.9	180.9	180.9	180.9	180.9	180.9
AB9 SPIKE WEIGHT	UG	164.0	164.0	164.0	164.0	164.0	164.0	164.0
BV10 SPIKE WEIGHT	UG	104.2	104.2	104.2	104.2	104.2	104.2	104.2
2AQ SPIKE WEIGHT	UG	186.9	186.9	186.9	186.9	186.9	186.9	186.9
SO3 SPIKE WEIGHT	UG	127.9	127.9	127.9	127.9	127.9	127.9	127.9
1AQ SPIKE WEIGHT	UG	128.5	128.5	128.5	128.5	128.5	128.5	128.5
SY14 SPIKE WEIGHT	UG	141.6	141.6	141.6	141.6	141.6	141.6	141.6
SO7 SPIKE WEIGHT	UG	144.0	144.0	144.0	144.0	144.0	144.0	144.0
AY3 SPIKE WEIGHT	UG	167.4	167.4	167.4	167.4	167.4	167.4	167.4
SY3 SPIKE WEIGHT	UG	151.0	151.0	151.0	151.0	151.0	151.0	151.0
SR1 SPIKE WEIGHT	UG	231.9	231.9	231.9	231.9	231.9	231.9	231.9
DR9 SPIKE WEIGHT	UG	153.0	153.0	153.0	153.0	153.0	153.0	153.0
SY2 SPIKE WEIGHT	UG	197.0	197.0	197.0	197.0	197.0	197.0	197.0
SG3 SPIKE WEIGHT	UG	157.0	157.0	157.0	157.0	157.0	157.0	157.0
SR24 SPIKE WEIGHT	UG	28.4	28.4	28.4	28.4	28.4	28.4	28.4
AN SPIKE WEIGHT	UG	207.7	207.7	207.7	207.7	207.7	207.7	207.7
BZ SPIKE WEIGHT	UG	121.6	121.6	121.6	121.6	121.6	121.6	121.6

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
		Weight Standard per Volume Sample						
MATRIX VOLUME (WATER)	L	1.000	1.000	1.000	1.000	1.000	1.000	1.000
SPIKE CONC AB45	UG/L	172.2	172.2	172.2	172.2	172.2	172.2	172.2
SPIKE CONC AY23	UG/L	140.6	140.6	140.6	140.6	140.6	140.6	140.6
SPIKE CONC AY73	UG/L	175.5	175.5	175.5	175.5	175.5	175.5	175.5
SPIKE CONC AO10	UG/L	153.7	153.7	153.7	153.7	153.7	153.7	153.7
SPIKE CONC AB1	UG/L	184.6	184.6	184.6	184.6	184.6	184.6	184.6
SPIKE CONC BY2	UG/L	132.1	132.1	132.1	132.1	132.1	132.1	132.1
SPIKE CONC DV1	UG/L	130.1	130.1	130.1	130.1	130.1	130.1	130.1
SPIKE CONC SY33	UG/L	139.8	139.8	139.8	139.8	139.8	139.8	139.8
SPIKE CONC DB14	UG/L	149.5	149.5	149.5	149.5	149.5	149.5	149.5
SPIKE CONC AR64	UG/L	180.9	180.9	180.9	180.9	180.9	180.9	180.9
SPIKE CONC AB9	UG/L	164.0	164.0	164.0	164.0	164.0	164.0	164.0
SPIKE CONC BV10	UG/L	104.2	104.2	104.2	104.2	104.2	104.2	104.2
SPIKE CONC 2AQ	UG/L	186.9	186.9	186.9	186.9	186.9	186.9	186.9
SPIKE CONC SO3	UG/L	127.9	127.9	127.9	127.9	127.9	127.9	127.9
SPIKE CONC 1AQ	UG/L	128.5	128.5	128.5	128.5	128.5	128.5	128.5
SPIKE CONC SY14	UG/L	141.6	141.6	141.6	141.6	141.6	141.6	141.6
SPIKE CONC SO7	UG/L	144.0	144.0	144.0	144.0	144.0	144.0	144.0
SPIKE CONC AY3	UG/L	167.4	167.4	167.4	167.4	167.4	167.4	167.4
SPIKE CONC SY3	UG/L	151.0	151.0	151.0	151.0	151.0	151.0	151.0
SPIKE CONC SR1	UG/L	231.9	231.9	231.9	231.9	231.9	231.9	231.9
SPIKE CONC DR9	UG/L	153.0	153.0	153.0	153.0	153.0	153.0	153.0
SPIKE CONC SY2	UG/L	197.0	197.0	197.0	197.0	197.0	197.0	197.0
SPIKE CONC SG3	UG/L	157.0	157.0	157.0	157.0	157.0	157.0	157.0
SPIKE CONC SR24	UG/L	28.4	28.4	28.4	28.4	28.4	28.4	28.4
SPIKE CONC AN	UG/L	207.7	207.7	207.7	207.7	207.7	207.7	207.7
SPIKE CONC BZ	UG/L	121.6	121.6	121.6	121.6	121.6	121.6	121.6

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
SPIKE CONC AB45	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY23	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY73	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AO10	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB1	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BY2	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DV1	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY33	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DB14	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AR64	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB9	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BV10	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 2AQ	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO3	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 1AQ	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY14	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO7	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY3	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY3	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR1	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DR9	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY2	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SG3	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR24	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AN	UG/KG	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BZ	UG/KG	NA	NA	NA	NA	NA	NA	NA

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
		Results from HPLC						
HPLC RESULT AB45	UG/ML	0.000	3.595	2.114	19.915	27.454	8.052	13.181
HPLC RESULT AY23	UG/ML	31.665	32.827	27.877	36.250	48.910	36.303	45.884
HPLC RESULT AY73	UG/ML	35.147	31.661	31.271	41.998	45.004	38.919	39.819
HPLC RESULT AO10	UG/ML	68.604	73.404	70.166	77.417	79.422	75.521	77.568
HPLC RESULT AB1	UG/ML	57.336	65.881	63.379	66.276	67.719	64.848	66.264
HPLC RESULT BY2	UG/ML	77.183	118.608	220.873	113.297	99.208	77.629	80.206
HPLC RESULT DV1	UG/ML	0.000	60.029	59.779	64.692	71.627	63.803	64.041
HPLC RESULT SY33	UG/ML	24.997	30.222	30.165	31.567	32.042	30.115	30.641
HPLC RESULT DB14	UG/ML	9.415	45.831	38.675	37.699	48.491	44.802	54.448
HPLC RESULT AR64	UG/ML	14.805	15.762	20.512	19.744	19.080	17.095	17.592
HPLC RESULT AB9	UG/ML	41.365	55.785	53.509	56.452	55.920	53.335	51.897
HPLC RESULT BV10	UG/ML	30.246	37.815	35.966	37.260	38.109	35.810	37.223
HPLC RESULT 2AQ	UG/ML	27.901	24.949	28.448	31.607	33.484	27.927	27.228
HPLC RESULT SO3	UG/ML	3.965	27.855	26.695	28.668	30.782	21.868	23.072
HPLC RESULT 1AQ	UG/ML	36.276	45.533	43.458	44.779	42.815	39.183	43.202
HPLC RESULT SY14	UG/ML	23.298	29.436	28.556	28.075	30.304	24.060	28.339
HPLC RESULT SO7	UG/ML	32.920	44.035	43.769	52.868	52.429	38.523	50.337
HPLC RESULT AY3	UG/ML	22.967	27.378	32.413	28.572	30.692	28.289	27.633
HPLC RESULT SY3	UG/ML	61.840	88.300	85.268	87.858	86.830	77.600	83.484
HPLC RESULT SR1	UG/ML	29.425	41.212	52.168	51.204	45.057	41.792	37.432
HPLC RESULT DR9	UG/ML	25.506	32.705	31.717	32.496	31.762	28.341	31.418
HPLC RESULT SY2	UG/ML	15.589	34.627	34.336	33.737	35.725	28.446	33.279
HPLC RESULT SG3	UG/ML	16.111	23.946	22.241	32.177	21.137	24.977	34.105
HPLC RESULT SR24	UG/ML	7.616	13.165	12.368	13.510	13.425	11.779	9.851
HPLC RESULT AN	UG/ML	30.986	51.410	48.706	51.37	46.471	33.768	41.165
HPLC RESULT BZ	UG/ML	39.590	52.332	50.776	51.641	52.375	47.931	49.69

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
		Final Sample Results						
CF (STD INJ VOL VS SAM INJ VOL)		2	2	2	2	2	2	2
FINAL VOLUME	ML	10.00	10.00	10.00	10.00	10.00	10.00	10.00
CORRECTED RESULT AB45	UG/L or UG/KG	0.00	17.98	10.57	99.58	137.27	40.26	65.91
CORRECTED RESULT AY23	UG/L or UG/KG	158.33	164.14	139.39	181.25	244.55	181.52	229.42
CORRECTED RESULT AY73	UG/L or UG/KG	175.74	158.31	156.36	209.99	225.02	194.60	199.10
CORRECTED RESULT AO10	UG/L or UG/KG	343.02	367.02	350.83	387.09	397.11	377.61	387.84
CORRECTED RESULT AB1	UG/L or UG/KG	286.68	329.41	316.90	331.38	338.60	324.24	331.32
CORRECTED RESULT BY2	UG/L or UG/KG	385.92	593.04	1104.37	566.49	496.04	388.15	401.03
CORRECTED RESULT DV1	UG/L or UG/KG	0.00	300.15	298.90	323.46	358.14	319.02	320.21
CORRECTED RESULT SY33	UG/L or UG/KG	124.99	151.11	150.83	157.84	160.21	150.58	153.21
CORRECTED RESULT DB14	UG/L or UG/KG	47.08	229.16	193.38	188.50	242.46	224.01	272.24
CORRECTED RESULT AR64	UG/L or UG/KG	74.03	78.81	102.56	98.72	95.40	85.48	87.96
CORRECTED RESULT AB9	UG/L or UG/KG	206.83	278.93	267.55	282.26	279.60	266.68	259.49
CORRECTED RESULT BV10	UG/L or UG/KG	151.23	189.08	179.83	186.30	190.55	179.05	186.12
CORRECTED RESULT 2AQ	UG/L or UG/KG	139.51	124.75	142.24	158.04	167.42	139.64	136.14
CORRECTED RESULT SO3	UG/L or UG/KG	19.83	139.28	133.48	143.34	153.91	109.34	115.36
CORRECTED RESULT 1AQ	UG/L or UG/KG	181.38	227.67	217.29	223.90	214.08	195.92	216.01
CORRECTED RESULT SY14	UG/L or UG/KG	116.49	147.18	142.78	140.38	151.52	120.30	141.70
CORRECTED RESULT SO7	UG/L or UG/KG	164.60	220.18	218.85	264.34	262.15	192.62	251.69
CORRECTED RESULT AY3	UG/L or UG/KG	114.84	136.89	162.07	142.86	153.46	141.45	138.17
CORRECTED RESULT SY3	UG/L or UG/KG	309.20	441.50	426.34	439.29	434.15	388.00	417.42
CORRECTED RESULT SR1	UG/L or UG/KG	147.13	206.06	260.84	256.02	225.29	208.96	187.16
CORRECTED RESULT DR9	UG/L or UG/KG	127.53	163.53	158.59	162.48	158.81	141.71	157.09
CORRECTED RESULT SY2	UG/L or UG/KG	77.95	173.14	171.68	168.69	178.63	142.23	166.40
CORRECTED RESULT SG3	UG/L or UG/KG	80.56	119.73	111.21	160.89	105.69	124.89	170.53
CORRECTED RESULT SR24	UG/L or UG/KG	38.08	65.83	61.84	67.55	67.13	58.90	49.26
CORRECTED RESULT AN	UG/L or UG/KG	154.93	257.05	243.53	256.85	232.36	168.84	205.83
CORRECTED RESULT BZ	UG/L or UG/KG	197.95	261.66	253.88	258.21	261.88	239.66	248.45

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
		Percent Recoveries						
% RECOVERY AB45	%	0.00	10.44	6.14	57.83	79.72	23.38	38.27
% RECOVERY AY23	%	112.6	116.7	99.1	128.9	173.9	129.1	163.2
% RECOVERY AY73	%	100.1	90.2	89.1	119.7	128.2	110.9	113.4
% RECOVERY AO10	%	223.2	238.8	228.3	251.8	258.4	245.7	252.3
% RECOVERY AB1	%	155.3	178.4	171.7	179.5	183.4	175.6	179.5
% RECOVERY BY2	%	292.1	448.9	836.0	428.8	375.5	293.8	303.6
% RECOVERY DV1	%	0.0	230.7	229.7	248.6	275.3	245.2	246.1
% RECOVERY SY33	%	89.4	108.1	107.9	112.9	114.6	107.7	109.6
% RECOVERY DB14	%	31.5	153.3	129.3	126.1	162.2	149.8	182.1
% RECOVERY AR64	%	40.9	43.6	56.7	54.6	52.7	47.2	48.6
% RECOVERY AB9	%	126.1	170.1	163.1	172.1	170.5	162.6	158.2
% RECOVERY BV10	%	145.1	181.5	172.6	178.8	182.9	171.8	178.6
% RECOVERY 2AQ	%	74.6	66.7	76.1	84.6	89.6	74.7	72.8
% RECOVERY SO3	%	15.5	108.9	104.4	112.1	120.3	85.5	90.2
% RECOVERY 1AQ	%	141.2	177.2	169.1	174.2	166.6	152.5	168.1
% RECOVERY SY14	%	82.3	103.9	100.8	99.1	107.0	85.0	100.1
% RECOVERY SO7	%	114.3	152.9	152.0	183.6	182.0	133.8	174.8
% RECOVERY AY3	%	68.6	81.8	96.8	85.3	91.7	84.5	82.5
% RECOVERY SY3	%	204.8	292.4	282.3	290.9	287.5	257.0	276.4
% RECOVERY SR1	%	63.4	88.9	112.5	110.4	97.1	90.1	80.7
% RECOVERY DR9	%	83.4	106.9	103.7	106.2	103.8	92.6	102.7
% RECOVERY SY2	%	39.6	87.9	87.1	85.6	90.7	72.2	84.5
% RECOVERY SG3	%	51.3	76.3	70.8	102.5	67.3	79.5	108.6
% RECOVERY SR24	%	134.1	231.8	217.7	237.9	236.4	207.4	173.4
% RECOVERY AN	%	74.6	123.8	117.3	123.7	111.9	81.3	99.1
% RECOVERY BZ	%	162.8	215.2	208.8	212.3	215.4	197.1	204.3

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
		Square of Corrected Results						
N		6	6	6	6	6	6	6
Xi^2 AB45		0.0	323.1	111.7	9915.2	18843.1	1620.9	4343.5
Xi^2 AY23		25066.8	26940.3	19428.2	32851.6	59804.7	32947.7	52633.5
Xi^2 AY73		30882.8	25060.5	24446.9	44095.8	50634.0	37867.2	39638.8
Xi^2 AO10		117662.7	134703.7	123081.7	149834.8	157696.4	142585.5	150419.9
Xi^2 AB1		82185.4	108507.7	100422.4	109812.7	114646.6	105131.6	109772.9
Xi^2 BY2		148930.4	351696.4	1219622.1	320905.3	246055.7	150656.5	160825.1
Xi^2 DV1		0.0	90087.0	89338.2	104626.4	128260.7	101770.6	102531.2
Xi^2 SY33		15621.3	22834.2	22748.2	24911.9	25667.2	22672.8	23471.8
Xi^2 DB14		2216.1	52512.0	37393.9	35530.4	58784.4	50180.5	74114.6
Xi^2 AR64		5479.7	6211.0	10518.6	9745.6	9101.2	7306.0	7737.0
Xi^2 AB9		42776.6	77799.2	71580.3	79670.7	78176.2	71115.6	67332.5
Xi^2 BV10		22870.5	35749.4	32338.8	34707.7	36307.4	32058.9	34638.8
Xi^2 2AQ		19461.6	15561.3	20232.2	24975.1	28029.5	19497.9	18534.1
Xi^2 SO3		393.0	19397.5	17815.6	20546.4	23688.3	11955.2	13307.9
Xi^2 1AQ		32898.7	51831.4	47214.9	50129.0	45828.1	38382.7	46660.3
Xi^2 SY14		13569.9	21662.0	20386.1	19705.1	22958.3	14472.1	20077.5
Xi^2 SO7		27093.2	48477.0	47893.1	69875.6	68720.0	37100.5	63345.3
Xi^2 AY3		13187.1	18738.9	26265.1	20409.0	23550.0	20006.7	19089.6
Xi^2 SY3		95604.6	194922.3	181765.8	192975.7	188486.2	150544.0	174239.5
Xi^2 SR1		21645.8	42460.7	68037.5	65546.2	50753.3	43664.3	35028.9
Xi^2 DR9		16263.9	26740.4	25149.2	26399.8	25220.6	20080.3	24677.3
Xi^2 SY2		6075.4	29975.7	29474.0	28454.6	31906.9	20229.4	27687.3
Xi^2 SG3		6489.1	14335.3	12366.6	25884.0	11169.3	15596.3	29078.8
Xi^2 SR24		1450.1	4332.9	3824.2	4563.0	4505.8	3468.6	2426.1
Xi^2 AN		24003.3	66074.7	59306.9	65971.9	53988.8	28506.9	42363.9
Xi^2 BZ		39184.2	68466.0	64455.1	66669.8	68578.5	57434.5	61727.4

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
		Sum of the Square of Corrected Results						
E Xi^2 AB45		35157.4						
E Xi^2 AY23		224606.0						
E Xi^2 AY73		221743.2						
E Xi^2 AO10		858321.9						
E Xi^2 AB1		648293.9						
E Xi^2 BY2		2449761.0						
E Xi^2 DV1		616614.1						
E Xi^2 SY33		142306.1						
E Xi^2 DB14		308515.8						
E Xi^2 AR64		50619.3						
E Xi^2 AB9		445674.4						
E Xi^2 BV10		205801.0						
E Xi^2 2AQ		126830.1						
E Xi^2 SO3		106710.9						
E Xi^2 1AQ		280046.4						
E Xi^2 SY14		119261.1						
E Xi^2 SO7		335411.7						
E Xi^2 AY3		128059.1						
E Xi^2 SY3		1082933.4						
E Xi^2 SR1		305490.9						
E Xi^2 DR9		148267.6						
E Xi^2 SY2		167727.9						
E Xi^2 SG3		108430.2						
E Xi^2 SR24		23120.6						
E Xi^2 AN		316213.2						
E Xi^2 BZ		387331.3						

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
		Sum of the Corrected Results Squared						
(E Xi)^2 AB45		138053.1						
(E Xi)^2 AY23		1300181.5						
(E Xi)^2 AY73		1307272.1						
(E Xi)^2 AO10		5141510.9						
(E Xi)^2 AB1		3888133.3						
(E Xi)^2 BY2		12596146.3						
(E Xi)^2 DV1		3685843.2						
(E Xi)^2 SY33		853332.5						
(E Xi)^2 DB14		1821771.1						
(E Xi)^2 AR64		301318.7						
(E Xi)^2 AB9		2671557.6						
(E Xi)^2 BV10		1234132.1						
(E Xi)^2 2AQ		753797.3						
(E Xi)^2 SO3		631548.1						
(E Xi)^2 1AQ		1676636.5						
(E Xi)^2 SY14		712082.8						
(E Xi)^2 SO7		1987550.1						
(E Xi)^2 AY3		765423.8						
(E Xi)^2 SY3		6485680.9						
(E Xi)^2 SR1		1807209.7						
(E Xi)^2 DR9		887731.4						
(E Xi)^2 SY2		1001500.6						
(E Xi)^2 SG3		628714.2						
(E Xi)^2 SR24		137262.8						
(E Xi)^2 AN		1861723.8						
(E Xi)^2 BZ		2321737.9						

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
		Variance						
S^2: VARIANCE AB45		2429.7						
S^2: VARIANCE AY23		1581.8						
S^2: VARIANCE AY73		772.9						
S^2: VARIANCE AO10		280.7						
S^2: VARIANCE AB1		54.3						
S^2: VARIANCE BY2		70080.7						
S^2: VARIANCE DV1		461.4						
S^2: VARIANCE SY33		16.8						
S^2: VARIANCE DB14		977.5						
S^2: VARIANCE AR64		79.9						
S^2: VARIANCE AB9		83.0						
S^2: VARIANCE BV10		22.5						
S^2: VARIANCE 2AQ		239.4						
S^2: VARIANCE SO3		290.6						
S^2: VARIANCE 1AQ		121.4						
S^2: VARIANCE SY14		116.1						
S^2: VARIANCE SO7		830.7						
S^2: VARIANCE AY3		97.7						
S^2: VARIANCE SY3		397.3						
S^2: VARIANCE SR1		857.9						
S^2: VARIANCE DR9		62.5						
S^2: VARIANCE SY2		162.2						
S^2: VARIANCE SG3		728.9						
S^2: VARIANCE SR24		48.7						
S^2: VARIANCE AN		1185.2						
S^2: VARIANCE BZ		75.0						

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
		Standard Deviation						
STD DEVIATION AB45		49.3						
STD DEVIATION AY23		39.8						
STD DEVIATION AY73		27.8						
STD DEVIATION AO10		16.8						
STD DEVIATION AB1		7.4						
STD DEVIATION BY2		264.7						
STD DEVIATION DV1		21.5						
STD DEVIATION SY33		4.1						
STD DEVIATION DB14		31.3						
STD DEVIATION AR64		8.9						
STD DEVIATION AB9		9.1						
STD DEVIATION BV10		4.7						
STD DEVIATION 2AQ		15.5						
STD DEVIATION SO3		17.0						
STD DEVIATION 1AQ		11.0						
STD DEVIATION SY14		10.8						
STD DEVIATION SO7		28.8						
STD DEVIATION AY3		9.9						
STD DEVIATION SY3		19.9						
STD DEVIATION SR1		29.3						
STD DEVIATION DR9		7.9						
STD DEVIATION SY2		12.7						
STD DEVIATION SG3		27.0						
STD DEVIATION SR24		7.0						
STD DEVIATION AN		34.4						
STD DEVIATION BZ		8.7						

	UNITS	WLM004HS	WLM006HS	WLM007HS	WLM008HS	WLM009HS	WLM010HS	WLM011HS
		Method Detection Limit						
T (STUDENT'S T @ 99% FOR 6 DF)		3.360						
MDL AB45	UG/L or UG/KG	165.6						
MDL AY23	UG/L or UG/KG	133.6						
MDL AY73	UG/L or UG/KG	93.4						
MDL AO10	UG/L or UG/KG	56.3						
MDL AB1	UG/L or UG/KG	24.8						
MDL BY2	UG/L or UG/KG	889.5						
MDL DV1	UG/L or UG/KG	72.2						
MDL SY33	UG/L or UG/KG	13.8						
MDL DB14	UG/L or UG/KG	105.0						
MDL AR64	UG/L or UG/KG	30.0						
MDL AB9	UG/L or UG/KG	30.6						
MDL BV10	UG/L or UG/KG	15.9						
MDL 2AQ	UG/L or UG/KG	52.0						
MDL SO3	UG/L or UG/KG	57.3						
MDL 1AQ	UG/L or UG/KG	37.0						
MDL SY14	UG/L or UG/KG	36.2						
MDL SO7	UG/L or UG/KG	96.8						
MDL AY3	UG/L or UG/KG	33.2						
MDL SY3	UG/L or UG/KG	67.0						
MDL SR1	UG/L or UG/KG	98.4						
MDL DR9	UG/L or UG/KG	26.6						
MDL SY2	UG/L or UG/KG	42.8						
MDL SG3	UG/L or UG/KG	90.7						
MDL SR24	UG/L or UG/KG	23.4						
MDL AN	UG/L or UG/KG	115.7						
MDL BZ	UG/L or UG/KG	29.1						

TABLE VII-9

High-Level Aqueous Extraction Method, Long Gradient, Low Spike

	Sample Conc µg/L	Sample Results (µg/L)							Mean Result µg/L	Method Bias (Accuracy) %R	Standard Deviation	Method Precision (%RSD)	MDL µg/L	RQL µg/L
		WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS						
ACID BLUE 45	2755.2	2123.0	3136.5	2845.5	2960.5	1573.0	1899.5	1558.0	2299.4	83.5	671.4	29.2	2108.1	8432.3
ACID YELLOW 23	2249.6	3827.0	3944.5	3999.0	3948.0	3536.0	3424.5	3123.5	3686.1	163.9	331.8	9.0	1041.9	4167.6
ACID YELLOW 73	2808	4377.5	5082.5	5097.5	5158.5	0.0	745.0	5626.0	3726.7	132.7	2330.1	62.5	7316.6	29266.4
ACID ORANGE 10	2459.2	4395.5	4415.5	4460.0	4533.0	3571.5	3713.5	3247.0	4048.0	164.6	523.0	12.9	1642.3	6569.2
ACID BLUE 1	2953.6	5878.5	5639.0	5769.5	5478.0	4982.5	5790.0	795.0	4904.6	166.1	1836.8	37.5	5767.6	23070.5
BASIC YELLOW 2	2113.6	4336.0	2158.0	2232.5	1424.5	1128.5	4595.0	1334.5	2458.4	116.3	1433.7	58.3	4501.7	18006.9
DISPERSE VIOLET 1	2081.6	1430.0	0.0	0.0	0.0	0.0	1188.0	0.0	374.0	18.0	642.5	171.8	2017.6	8070.2
SOLVENT YELLOW 33	2236.8	300.0	0.0	0.0	0.0	0.0	0.0	0.0	42.9	1.9	113.4	264.6	356.0	1424.2
DISPERSE BLUE 14	2392	647.5	0.0	0.0	0.0	0.0	598.0	0.0	177.9	7.4	304.2	171.0	955.2	3820.8
ACID RED 64	2894.4	5280.0	4983.5	5293.0	5014.5	4663.0	4097.5	4368.0	4814.2	166.3	456.5	9.5	1433.6	5734.2
ACID BLUE 9	2624	4865.0	4769.5	5095.0	4881.0	3893.5	4833.5	3807.0	4592.1	175.0	517.2	11.3	1624.0	6496.1
BASIC VIOLET 10	1667.2	2749.5	2206.0	2366.5	1955.0	1136.0	2661.0	1411.5	2069.4	124.1	610.5	29.5	1917.1	7668.5
2-AMINOANTHRAQUINONE	2990.4	0.0	0.0	0.0	0.0	0.0	2980.0	0.0	425.7	14.2	1126.3	264.6	3536.7	14146.8
SOLVENT ORANGE 3	2046.4	2456.5	181.5	292.0	0.0	527.0	3139.0	642.5	1034.1	50.5	1239.0	119.8	3890.6	15562.3
1-AMINOANTHRAQUINONE	2056	1628.0	0.0	0.0	0.0	0.0	1384.5	0.0	430.4	20.9	738.3	171.6	2318.4	9273.4
SOLVENT YELLOW 14	2265.6	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT ORANGE 7	2304	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ACID YELLOW 3	2678.4	4961.0	5104.0	5065.0	5026.5	2326.5	4236.5	2324.0	4149.1	154.9	1280.6	30.9	4021.2	16084.8
SOLVENT YELLOW 3	2416	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT RED 1	3710.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
DISPERSE RED 9	2448	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT YELLOW 2	3152	813.5	0.0	0.0	0.0	0.0	712.0	0.0	217.9	6.9	373.3	171.3	1172.3	4689.1
SOLVENT GREEN 3	2512	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT RED 24	454.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ANTHRACENE	3323.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
BENZANTHRONE	1945.6	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

TABLE VII - 9A

PERCENT RECOVERIES
HIGH-LEVEL AQUEOUS METHOD
LOW SPIKE, LONG GRADIENT

Compounds		Ret. Time	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
% RECOVERY AB45	%	6.439	77	114	103	107	57	69	57
% RECOVERY AY3	%	13.372	185	191	189	188	87	158	87
% RECOVERY AY23	%	14.513	170	175	178	175	157	152	139
% RECOVERY AR64	%	14.9	182	172	183	173	161	142	151
% RECOVERY AY73	%	17.353	156	181	182	184	0	27	200
% RECOVERY AO10	%	18.752	179	180	181	184	145	151	132
% RECOVERY AB9	%	21.657	185	182	194	186	148	184	145
% RECOVERY DV1	%	22.77	69	0	0	0	0	57	0
% RECOVERY AB1	%	23.733	199	191	195	185	169	196	27
% RECOVERY 2AQ	%	23.758	0	0	0	0	0	100	0
% RECOVERY SO3	%	24.783	120	9	14	0	26	153	31
% RECOVERY SY33	%	28.259	13	0	0	0	0	0	0
% RECOVERY 1AQ	%	28.853	79	0	0	0	0	67	0
% RECOVERY DB14	%	34.023	27	0	0	0	0	25	0
% RECOVERY BY2	%	35.367	205	102	106	67	53	217	63
% RECOVERY SY3	%	35.998	0	0	0	0	0	0	0
% RECOVERY BV10	%	36.018	165	132	142	117	68	160	85
% RECOVERY DR9	%	36.142	0	0	0	0	0	0	0
% RECOVERY BZ	%	36.724	0	0	0	0	0	0	0
% RECOVERY SY2	%	39.91	26	0	0	0	0	23	0
% RECOVERY AN	%	40.641	0	0	0	0	0	0	0
% RECOVERY SY14	%	44.36	0	0	0	0	0	0	0
% RECOVERY SR1	%	44.811	0	0	0	0	0	0	0
% RECOVERY SO7	%	50.894	0	0	0	0	0	0	0
% RECOVERY SG3	%	57.448	0	0	0	0	0	0	0
% RECOVERY SR24	%	57.994	0	0	0	0	0	0	0

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Stock Standard Concentrations						
ACID BLUE 45 STD	344.4	344.4	344.4	344.4	344.4	344.4	344.4
ACID YELLOW 23 STD	281.2	281.2	281.2	281.2	281.2	281.2	281.2
ACID YELLOW 73 STD	351.0	351.0	351.0	351.0	351.0	351.0	351.0
ACID ORANGE 10 STD	307.4	307.4	307.4	307.4	307.4	307.4	307.4
ACID BLUE 1 STD	369.2	369.2	369.2	369.2	369.2	369.2	369.2
BASIC YELLOW 2 STD	264.2	264.2	264.2	264.2	264.2	264.2	264.2
DISPERSE VIOLET 1 STD	260.2	260.2	260.2	260.2	260.2	260.2	260.2
SOLVENT YELLOW 33 STD	279.6	279.6	279.6	279.6	279.6	279.6	279.6
DISPERSE BLUE 14 STD	299.0	299.0	299.0	299.0	299.0	299.0	299.0
ACID RED 64 STD	361.8	361.8	361.8	361.8	361.8	361.8	361.8
ACID BLUE 9 STD	328.0	328.0	328.0	328.0	328.0	328.0	328.0
BASIC VIOLET 10 STD	208.4	208.4	208.4	208.4	208.4	208.4	208.4
2-AMINOANTHRAQUINONE STD	373.8	373.8	373.8	373.8	373.8	373.8	373.8
SOLVENT ORANGE 3 STD	255.8	255.8	255.8	255.8	255.8	255.8	255.8
1-AMINOANTHRAQUINONE STD	257.0	257.0	257.0	257.0	257.0	257.0	257.0
SOLVENT YELLOW 14 STD	283.2	283.2	283.2	283.2	283.2	283.2	283.2
SOLVENT ORANGE 7 STD	288.0	288.0	288.0	288.0	288.0	288.0	288.0
ACID YELLOW 3 STD	334.8	334.8	334.8	334.8	334.8	334.8	334.8
SOLVENT YELLOW 3 STD	302.0	302.0	302.0	302.0	302.0	302.0	302.0
SOLVENT RED 1 STD	463.8	463.8	463.8	463.8	463.8	463.8	463.8
DISPERSE RED 9 STD	306.0	306.0	306.0	306.0	306.0	306.0	306.0
SOLVENT YELLOW 2 STD	394.0	394.0	394.0	394.0	394.0	394.0	394.0
SOLVENT GREEN 3 STD	314.0	314.0	314.0	314.0	314.0	314.0	314.0
SOLVENT RED 24 STD	56.8	56.8	56.8	56.8	56.8	56.8	56.8
ANTHRACENE	415.4	415.4	415.4	415.4	415.4	415.4	415.4
BENZANTHRONE	243.2	243.2	243.2	243.2	243.2	243.2	243.2

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Weight of Stock Standard Spiked						
SPIKE VOLUME EACH DYE	200.0	200.0	200.0	200.0	200.0	200.0	200.0
AB45 SPIKE WEIGHT	68.9	68.9	68.9	68.9	68.9	68.9	68.9
AY23 SPIKE WEIGHT	56.2	56.2	56.2	56.2	56.2	56.2	56.2
AY73 SPIKE WEIGHT	70.2	70.2	70.2	70.2	70.2	70.2	70.2
AO10 SPIKE WEIGHT	61.5	61.5	61.5	61.5	61.5	61.5	61.5
AB1 SPIKE WEIGHT	73.8	73.8	73.8	73.8	73.8	73.8	73.8
BY2 SPIKE WEIGHT	52.8	52.8	52.8	52.8	52.8	52.8	52.8
DV1 SPIKE WEIGHT	52.0	52.0	52.0	52.0	52.0	52.0	52.0
SY33 SPIKE WEIGHT	55.9	55.9	55.9	55.9	55.9	55.9	55.9
DB14 SPIKE WEIGHT	59.8	59.8	59.8	59.8	59.8	59.8	59.8
AR64 SPIKE WEIGHT	72.4	72.4	72.4	72.4	72.4	72.4	72.4
AB9 SPIKE WEIGHT	65.6	65.6	65.6	65.6	65.6	65.6	65.6
BV10 SPIKE WEIGHT	41.7	41.7	41.7	41.7	41.7	41.7	41.7
2AQ SPIKE WEIGHT	74.8	74.8	74.8	74.8	74.8	74.8	74.8
SO3 SPIKE WEIGHT	51.2	51.2	51.2	51.2	51.2	51.2	51.2
1AQ SPIKE WEIGHT	51.4	51.4	51.4	51.4	51.4	51.4	51.4
SY14 SPIKE WEIGHT	56.6	56.6	56.6	56.6	56.6	56.6	56.6
SO7 SPIKE WEIGHT	57.6	57.6	57.6	57.6	57.6	57.6	57.6
AY3 SPIKE WEIGHT	67.0	67.0	67.0	67.0	67.0	67.0	67.0
SY3 SPIKE WEIGHT	60.4	60.4	60.4	60.4	60.4	60.4	60.4
SR1 SPIKE WEIGHT	92.8	92.8	92.8	92.8	92.8	92.8	92.8
DR9 SPIKE WEIGHT	61.2	61.2	61.2	61.2	61.2	61.2	61.2
SY2 SPIKE WEIGHT	78.8	78.8	78.8	78.8	78.8	78.8	78.8
SG3 SPIKE WEIGHT	62.8	62.8	62.8	62.8	62.8	62.8	62.8
SR24 SPIKE WEIGHT	11.4	11.4	11.4	11.4	11.4	11.4	11.4
AN SPIKE WEIGHT	83.1	83.1	83.1	83.1	83.1	83.1	83.1
BZ SPIKE WEIGHT	48.6	48.6	48.6	48.6	48.6	48.6	48.6

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Weight Standard per Volume Sample						
MATRIX VOLUME (WATER)	0.025	0.025	0.025	0.025	0.025	0.025	0.025
MATRIX WEIGHT (SOIL)	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB45	2755.2	2755.2	2755.2	2755.2	2755.2	2755.2	2755.2
SPIKE CONC AY23	2249.6	2249.6	2249.6	2249.6	2249.6	2249.6	2249.6
SPIKE CONC AY73	2808.0	2808.0	2808.0	2808.0	2808.0	2808.0	2808.0
SPIKE CONC AO10	2459.2	2459.2	2459.2	2459.2	2459.2	2459.2	2459.2
SPIKE CONC AB1	2953.6	2953.6	2953.6	2953.6	2953.6	2953.6	2953.6
SPIKE CONC BY2	2113.6	2113.6	2113.6	2113.6	2113.6	2113.6	2113.6
SPIKE CONC DV1	2081.6	2081.6	2081.6	2081.6	2081.6	2081.6	2081.6
SPIKE CONC SY33	2236.8	2236.8	2236.8	2236.8	2236.8	2236.8	2236.8
SPIKE CONC DB14	2392.0	2392.0	2392.0	2392.0	2392.0	2392.0	2392.0
SPIKE CONC AR64	2894.4	2894.4	2894.4	2894.4	2894.4	2894.4	2894.4
SPIKE CONC AB9	2624.0	2624.0	2624.0	2624.0	2624.0	2624.0	2624.0
SPIKE CONC BV10	1667.2	1667.2	1667.2	1667.2	1667.2	1667.2	1667.2
SPIKE CONC 2AQ	2990.4	2990.4	2990.4	2990.4	2990.4	2990.4	2990.4
SPIKE CONC SO3	2046.4	2046.4	2046.4	2046.4	2046.4	2046.4	2046.4
SPIKE CONC 1AQ	2056.0	2056.0	2056.0	2056.0	2056.0	2056.0	2056.0
SPIKE CONC SY14	2265.6	2265.6	2265.6	2265.6	2265.6	2265.6	2265.6
SPIKE CONC SO7	2304.0	2304.0	2304.0	2304.0	2304.0	2304.0	2304.0
SPIKE CONC AY3	2678.4	2678.4	2678.4	2678.4	2678.4	2678.4	2678.4
SPIKE CONC SY3	2416.0	2416.0	2416.0	2416.0	2416.0	2416.0	2416.0
SPIKE CONC SR1	3710.4	3710.4	3710.4	3710.4	3710.4	3710.4	3710.4
SPIKE CONC DR9	2448.0	2448.0	2448.0	2448.0	2448.0	2448.0	2448.0
SPIKE CONC SY2	3152.0	3152.0	3152.0	3152.0	3152.0	3152.0	3152.0
SPIKE CONC SG3	2512.0	2512.0	2512.0	2512.0	2512.0	2512.0	2512.0
SPIKE CONC SR24	454.4	454.4	454.4	454.4	454.4	454.4	454.4
SPIKE CONC AN	3323.2	3323.2	3323.2	3323.2	3323.2	3323.2	3323.2
SPIKE CONC BZ	1945.6	1945.6	1945.6	1945.6	1945.6	1945.6	1945.6

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
SPIKE CONC AB45	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY23	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY73	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AO10	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BY2	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DV1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY33	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DB14	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AR64	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB9	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BV10	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 2AQ	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 1AQ	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY14	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO7	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DR9	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY2	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SG3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR24	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AN	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BZ	NA	NA	NA	NA	NA	NA	NA

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Results from HPLC						
HPLC RESULT AB45	4.246	6.273	5.691	5.921	3.146	3.799	3.116
HPLC RESULT AY23	7.654	7.889	7.998	7.896	7.072	6.849	6.247
HPLC RESULT AY73	8.755	10.165	10.195	10.317	0.000	1.490	11.252
HPLC RESULT AO10	8.791	8.831	8.920	9.066	7.143	7.427	6.494
HPLC RESULT AB1	11.757	11.278	11.539	10.956	9.965	11.580	1.590
HPLC RESULT BY2	8.672	4.316	4.465	2.849	2.257	9.190	2.669
HPLC RESULT DV1	2.860	0.000	0.000	0.000	0.000	2.376	0.000
HPLC RESULT SY33	0.600	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT DB14	1.295	0.000	0.000	0.000	0.000	1.196	0.000
HPLC RESULT AR64	10.560	9.967	10.586	10.029	9.326	8.195	8.736
HPLC RESULT AB9	9.730	9.539	10.190	9.762	7.787	9.667	7.614
HPLC RESULT BV10	5.499	4.412	4.733	3.910	2.272	5.322	2.823
HPLC RESULT 2AQ	0.000	0.000	0.000	0.000	0.000	5.960	0.000
HPLC RESULT SO3	4.913	0.363	0.584	0.000	1.054	6.278	1.285
HPLC RESULT 1AQ	3.256	0.000	0.000	0.000	0.000	2.769	0.000
HPLC RESULT SY14	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SO7	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT AY3	9.922	10.208	10.130	10.053	4.653	8.473	4.648
HPLC RESULT SY3	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SR1	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT DR9	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SY2	1.627	0.000	0.000	0.000	0.000	1.424	0.000
HPLC RESULT SG3	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SR24	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT AN	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT BZ	0.000	0.000	0.000	0.000	0.000	0.000	0.000

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Final Sample Results						
CF (STD INJ VOL VS SAM INJ VOL)	2	2	2	2	2	2	2
FINAL VOLUME	25.000	25.000	25.000	25.000	25.000	25.000	25.000
CORRECTED RESULT AB45	2123.00	3136.50	2845.50	2960.50	1573.00	1899.50	1558.00
CORRECTED RESULT AY23	3827.00	3944.50	3999.00	3948.00	3536.00	3424.50	3123.50
CORRECTED RESULT AY73	4377.50	5082.50	5097.50	5158.50	0.00	745.00	5626.00
CORRECTED RESULT AO10	4395.50	4415.50	4460.00	4533.00	3571.50	3713.50	3247.00
CORRECTED RESULT AB1	5878.50	5639.00	5769.50	5478.00	4982.50	5790.00	795.00
CORRECTED RESULT BY2	4336.00	2158.00	2232.50	1424.50	1128.50	4595.00	1334.50
CORRECTED RESULT DV1	1430.00	0.00	0.00	0.00	0.00	1188.00	0.00
CORRECTED RESULT SY33	300.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT DB14	647.50	0.00	0.00	0.00	0.00	598.00	0.00
CORRECTED RESULT AR64	5280.00	4983.50	5293.00	5014.50	4663.00	4097.50	4368.00
CORRECTED RESULT AB9	4865.00	4769.50	5095.00	4881.00	3893.50	4833.50	3807.00
CORRECTED RESULT BV10	2749.50	2206.00	2366.50	1955.00	1136.00	2661.00	1411.50
CORRECTED RESULT 2AQ	0.00	0.00	0.00	0.00	0.00	2980.00	0.00
CORRECTED RESULT SO3	2456.50	181.50	292.00	0.00	527.00	3139.00	642.50
CORRECTED RESULT 1AQ	1628.00	0.00	0.00	0.00	0.00	1384.50	0.00
CORRECTED RESULT SY14	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SO7	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT AY3	4961.00	5104.00	5065.00	5026.50	2326.50	4236.50	2324.00
CORRECTED RESULT SY3	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SR1	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT DR9	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SY2	813.50	0.00	0.00	0.00	0.00	712.00	0.00
CORRECTED RESULT SG3	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SR24	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT AN	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT BZ	0.00	0.00	0.00	0.00	0.00	0.00	0.00

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Percent Recoveries						
% RECOVERY AB45	77.05	113.84	103.28	107.45	57.09	68.94	56.55
% RECOVERY AY23	170.1	175.3	177.8	175.5	157.2	152.2	138.8
% RECOVERY AY73	155.9	181.0	181.5	183.7	0.0	26.5	200.4
% RECOVERY AO10	178.7	179.6	181.4	184.3	145.2	151.0	132.0
% RECOVERY AB1	199.0	190.9	195.3	185.5	168.7	196.0	26.9
% RECOVERY BY2	205.1	102.1	105.6	67.4	53.4	217.4	63.1
% RECOVERY DV1	68.7	0.0	0.0	0.0	0.0	57.1	0.0
% RECOVERY SY33	13.4	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY DB14	27.1	0.0	0.0	0.0	0.0	25.0	0.0
% RECOVERY AR64	182.4	172.2	182.9	173.2	161.1	141.6	150.9
% RECOVERY AB9	185.4	181.8	194.2	186.0	148.4	184.2	145.1
% RECOVERY BV10	164.9	132.3	141.9	117.3	68.1	159.6	84.7
% RECOVERY 2AQ	0.0	0.0	0.0	0.0	0.0	99.7	0.0
% RECOVERY SO3	120.0	8.9	14.3	0.0	25.8	153.4	31.4
% RECOVERY 1AQ	79.2	0.0	0.0	0.0	0.0	67.3	0.0
% RECOVERY SY14	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SO7	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY AY3	185.2	190.6	189.1	187.7	86.9	158.2	86.8
% RECOVERY SY3	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SR1	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY DR9	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SY2	25.8	0.0	0.0	0.0	0.0	22.6	0.0
% RECOVERY SG3	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SR24	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY AN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY BZ	0.0	0.0	0.0	0.0	0.0	0.0	0.0

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Square of Corrected Results						
N	7	7	7	7	7	7	7
Xi^2 AB45	4507129.0	9837632.3	8096870.3	8764560.3	2474329.0	3608100.3	2427364.0
Xi^2 AY23	14645929.0	15559080.3	15992001.0	15586704.0	12503296.0	11727200.3	9756252.3
Xi^2 AY73	19162506.3	25831806.3	25984506.3	26610122.3	0.0	555025.0	31651876.0
Xi^2 AO10	1.932E+07	1.950E+07	1.989E+07	2.055E+07	1.276E+07	1.379E+07	1.054E+07
Xi^2 AB1	34556762.3	31798321.0	33287130.3	30008484.0	24825306.3	33524100.0	632025.0
Xi^2 BY2	18800896.0	4656964.0	4984056.3	2029200.3	1273512.3	21114025.0	1780890.3
Xi^2 DV1	2044900.0	0.0	0.0	0.0	0.0	1411344.0	0.0
Xi^2 SY33	90000.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 DB14	419256.3	0.0	0.0	0.0	0.0	357604.0	0.0
Xi^2 AR64	27878400.0	24835272.3	28015849.0	25145210.3	21743569.0	16789506.3	19079424.0
Xi^2 AB9	23668225.0	22748130.3	25959025.0	23824161.0	15159342.3	23362722.3	14493249.0
Xi^2 BV10	7559750.3	4866436.0	5600322.3	3822025.0	1290496.0	7080921.0	1992332.3
Xi^2 2AQ	0.0	0.0	0.0	0.0	0.0	8880400.0	0.0
Xi^2 SO3	6034392.3	32942.3	85264.0	0.0	277729.0	9853321.0	412806.3
Xi^2 1AQ	2650384.0	0.0	0.0	0.0	0.0	1916840.3	0.0
Xi^2 SY14	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SO7	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 AY3	24611521.0	26050816.0	25654225.0	25265702.3	5412602.3	17947932.3	5400976.0
Xi^2 SY3	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SR1	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 DR9	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SY2	661782.3	0.0	0.0	0.0	0.0	506944.0	0.0
Xi^2 SG3	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SR24	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 AN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 BZ	0.0	0.0	0.0	0.0	0.0	0.0	0.0

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Sum of the Square of Corrected Results						
E Xi^2 AB45	3.972E+07						
E Xi^2 AY23	9.577E+07						
E Xi^2 AY73	1.298E+08						
E Xi^2 AO10	1.163E+08						
E Xi^2 AB1	1.886E+08						
E Xi^2 BY2	5.464E+07						
E Xi^2 DV1	3.456E+06						
E Xi^2 SY33	9.000E+04						
E Xi^2 DB14	7.769E+05						
E Xi^2 AR64	1.635E+08						
E Xi^2 AB9	1.492E+08						
E Xi^2 BV10	3.221E+07						
E Xi^2 2AQ	8.880E+06						
E Xi^2 SO3	1.670E+07						
E Xi^2 1AQ	4.567E+06						
E Xi^2 SY14	0.000E+00						
E Xi^2 SO7	0.000E+00						
E Xi^2 AY3	1.303E+08						
E Xi^2 SY3	0.000E+00						
E Xi^2 SR1	0.000E+00						
E Xi^2 DR9	0.000E+00						
E Xi^2 SY2	1.169E+06						
E Xi^2 SG3	0.000E+00						
E Xi^2 SR24	0.000E+00						
E Xi^2 AN	0.000E+00						
E Xi^2 BZ	0.000E+00						

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Sum of the Corrected Results Squared						
(E Xi)^2 AB45	2.591E+08						
(E Xi)^2 AY23	6.658E+08						
(E Xi)^2 AY73	6.805E+08						
(E Xi)^2 AO10	8.029E+08						
(E Xi)^2 AB1	1.179E+09						
(E Xi)^2 BY2	2.961E+08						
(E Xi)^2 DV1	6.854E+06						
(E Xi)^2 SY33	9.000E+04						
(E Xi)^2 DB14	1.551E+06						
(E Xi)^2 AR64	1.136E+09						
(E Xi)^2 AB9	1.033E+09						
(E Xi)^2 BV10	2.098E+08						
(E Xi)^2 2AQ	8.880E+06						
(E Xi)^2 SO3	5.240E+07						
(E Xi)^2 1AQ	9.075E+06						
(E Xi)^2 SY14	0.000E+00						
(E Xi)^2 SO7	0.000E+00						
(E Xi)^2 AY3	8.435E+08						
(E Xi)^2 SY3	0.000E+00						
(E Xi)^2 SR1	0.000E+00						
(E Xi)^2 DR9	0.000E+00						
(E Xi)^2 SY2	2.327E+06						
(E Xi)^2 SG3	0.000E+00						
(E Xi)^2 SR24	0.000E+00						
(E Xi)^2 AN	0.000E+00						
(E Xi)^2 BZ	0.000E+00						

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Variance						
S^2: VARIANCE AB45	450730.5						
S^2: VARIANCE AY23	110100.8						
S^2: VARIANCE AY73	5429507.7						
S^2: VARIANCE AO10	273554.2						
S^2: VARIANCE AB1	3373913.0						
S^2: VARIANCE BY2	2055407.8						
S^2: VARIANCE DV1	412852.0						
S^2: VARIANCE SY33	12857.1						
S^2: VARIANCE DB14	92541.7						
S^2: VARIANCE AR64	208436.1						
S^2: VARIANCE AB9	267502.5						
S^2: VARIANCE BV10	372768.3						
S^2: VARIANCE 2AQ	1268628.6						
S^2: VARIANCE SO3	1535221.5						
S^2: VARIANCE 1AQ	545128.9						
S^2: VARIANCE SY14	0.0						
S^2: VARIANCE SO7	0.0						
S^2: VARIANCE AY3	1640036.5						
S^2: VARIANCE SY3	0.0						
S^2: VARIANCE SR1	0.0						
S^2: VARIANCE DR9	0.0						
S^2: VARIANCE SY2	139379.4						
S^2: VARIANCE SG3	0.0						
S^2: VARIANCE SR24	0.0						
S^2: VARIANCE AN	0.0						
S^2: VARIANCE BZ	0.0						

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Standard Deviation						
STD DEVIATION AB45	671.4						
STD DEVIATION AY23	331.8						
STD DEVIATION AY73	2330.1						
STD DEVIATION AO10	523.0						
STD DEVIATION AB1	1836.8						
STD DEVIATION BY2	1433.7						
STD DEVIATION DV1	642.5						
STD DEVIATION SY33	113.4						
STD DEVIATION DB14	304.2						
STD DEVIATION AR64	456.5						
STD DEVIATION AB9	517.2						
STD DEVIATION BV10	610.5						
STD DEVIATION 2AQ	1126.3						
STD DEVIATION SO3	1239.0						
STD DEVIATION 1AQ	738.3						
STD DEVIATION SY14	0.0						
STD DEVIATION SO7	0.0						
STD DEVIATION AY3	1280.6						
STD DEVIATION SY3	0.0						
STD DEVIATION SR1	0.0						
STD DEVIATION DR9	0.0						
STD DEVIATION SY2	373.3						
STD DEVIATION SG3	0.0						
STD DEVIATION SR24	0.0						
STD DEVIATION AN	0.0						
STD DEVIATION BZ	0.0						

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Method Detection Limit						
T (STUDENT'S T @ 99% FOR 6 DF)	3.140						
MDL AB45	2108.1						
MDL AY23	1041.9						
MDL AY73	7316.6						
MDL AO10	1642.3						
MDL AB1	5767.6						
MDL BY2	4501.7						
MDL DV1	2017.6						
MDL SY33	356.0						
MDL DB14	955.2						
MDL AR64	1433.6						
MDL AB9	1624.0						
MDL BV10	1917.1						
MDL 2AQ	3536.7						
MDL SO3	3890.6						
MDL 1AQ	2318.4						
MDL SY14	0.0						
MDL SO7	0.0						
MDL AY3	4021.2						
MDL SY3	0.0						
MDL SR1	0.0						
MDL DR9	0.0						
MDL SY2	1172.3						
MDL SG3	0.0						
MDL SR24	0.0						
MDL AN	0.0						
MDL BZ	0.0						

TABLE VII-10

High-Level Aqueous Extraction Method, Long Gradient, High Spike

	Sample Conc µg/L	Sample Results (µg/L)							Mean Result µg/L	Method Bias (Accuracy) %R	Standard Deviation	Method Precision (%RSD)	MDL µg/L	RQL µg/L
		WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS						
ACID BLUE 45	5510.4	2236.5	1760.5	3465.5	1885.0	2680.0	2069.0	2122.5	2317.0	42.0	584.9	25.2	1836.7	7346.9
ACID YELLOW 23	4499.2	3713.5	3493.5	3242.5	3497.0	3616.0	3579.5	3569.5	3530.2	78.5	147.3	4.2	462.5	1849.9
ACID YELLOW 73	5616	5987.0	5816.5	5828.0	5914.0	5718.5	5653.5	5626.0	5791.9	103.1	133.5	2.3	419.2	1676.9
ACID ORANGE 10	4918.4	8880.0	8281.5	8077.0	8205.5	8290.0	8392.0	7983.5	8301.4	168.8	289.9	3.5	910.2	3640.7
ACID BLUE 1	5907.2	7408.0	6595.0	6620.5	6903.5	6869.0	7018.5	6934.5	6907.0	116.9	272.4	3.9	855.2	3420.8
BASIC YELLOW 2	4227.2	1447.0	0.0	0.0	0.0	0.0	0.0	0.0	206.7	4.9	546.9	264.6	1717.3	6869.2
DISPERSE VIOLET 1	4163.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT YELLOW 33	4473.6	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
DISPERSE BLUE 14	4784	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ACID RED 64	5788.8	7007.5	7037.5	6908.0	6958.0	7135.5	6925.5	6858.0	6975.7	120.5	92.8	1.3	291.3	1165.2
ACID BLUE 9	5248	6688.0	6127.0	5866.5	6026.0	6046.5	6123.5	5980.0	6122.5	116.7	264.8	4.3	831.5	3326.0
BASIC VIOLET 10	3334.4	2542.0	1532.5	1354.5	1505.0	1625.0	1684.0	1559.0	1686.0	50.6	391.3	23.2	1228.8	4915.0
2-AMINOANTHRAQUINONE	5980.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT ORANGE 3	4092.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
1-AMINOANTHRAQUINONE	4112	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT YELLOW 14	4531.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT ORANGE 7	4608	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ACID YELLOW 3	5356.8	4233.5	3832.0	3529.5	3655.0	3663.5	3649.5	3528.5	3727.4	69.6	245.4	6.6	770.4	3081.7
SOLVENT YELLOW 3	4832	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT RED 1	7420.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
DISPERSE RED 9	4896	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT YELLOW 2	6304	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT GREEN 3	5024	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT RED 24	908.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ANTHRACENE	6646.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
BENZANTHRONE	3891.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

TABLE VII - 10A

PERCENT RECOVERIES
HIGH-LEVEL AQUEOUS METHOD
HIGH SPIKE, LONG GRADIENT

Compounds		Ret. Time	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
% RECOVERY AB45	%	6.439	41	32	63	34	49	38	39
% RECOVERY AY3	%	13.372	79	72	66	68	68	68	66
% RECOVERY AY23	%	14.513	83	78	72	78	80	80	79
% RECOVERY AR64	%	14.9	121	122	119	120	123	120	118
% RECOVERY AY73	%	17.353	107	104	104	105	102	101	100
% RECOVERY AO10	%	18.752	181	168	164	167	169	171	162
% RECOVERY AB9	%	21.657	127	117	112	115	115	117	114
% RECOVERY DV1	%	22.77	0	0	0	0	0	0	0
% RECOVERY AB1	%	23.733	125	112	112	117	116	119	117
% RECOVERY 2AQ	%	23.758	0	0	0	0	0	0	0
% RECOVERY SO3	%	24.783	0	0	0	0	0	0	0
% RECOVERY SY33	%	28.259	0	0	0	0	0	0	0
% RECOVERY 1AQ	%	28.853	0	0	0	0	0	0	0
% RECOVERY DB14	%	34.023	0	0	0	0	0	0	0
% RECOVERY BY2	%	35.367	34	0	0	0	0	0	0
% RECOVERY SY3	%	35.998	0	0	0	0	0	0	0
% RECOVERY BV10	%	36.018	76	46	41	45	49	51	47
% RECOVERY DR9	%	36.142	0	0	0	0	0	0	0
% RECOVERY BZ	%	36.724	0	0	0	0	0	0	0
% RECOVERY SY2	%	39.91	0	0	0	0	0	0	0
% RECOVERY AN	%	40.641	0	0	0	0	0	0	0
% RECOVERY SY14	%	44.36	0	0	0	0	0	0	0
% RECOVERY SR1	%	44.811	0	0	0	0	0	0	0
% RECOVERY SO7	%	50.894	0	0	0	0	0	0	0
% RECOVERY SG3	%	57.448	0	0	0	0	0	0	0
% RECOVERY SR24	%	57.994	0	0	0	0	0	0	0

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Stock Standard Concentrations						
ACID BLUE 45 STD	344.4	344.4	344.4	344.4	344.4	344.4	344.4
ACID YELLOW 23 STD	281.2	281.2	281.2	281.2	281.2	281.2	281.2
ACID YELLOW 73 STD	351.0	351.0	351.0	351.0	351.0	351.0	351.0
ACID ORANGE 10 STD	307.4	307.4	307.4	307.4	307.4	307.4	307.4
ACID BLUE 1 STD	369.2	369.2	369.2	369.2	369.2	369.2	369.2
BASIC YELLOW 2 STD	264.2	264.2	264.2	264.2	264.2	264.2	264.2
DISPERSE VIOLET 1 STD	260.2	260.2	260.2	260.2	260.2	260.2	260.2
SOLVENT YELLOW 33 STD	279.6	279.6	279.6	279.6	279.6	279.6	279.6
DISPERSE BLUE 14 STD	299.0	299.0	299.0	299.0	299.0	299.0	299.0
ACID RED 64 STD	361.8	361.8	361.8	361.8	361.8	361.8	361.8
ACID BLUE 9 STD	328.0	328.0	328.0	328.0	328.0	328.0	328.0
BASIC VIOLET 10 STD	208.4	208.4	208.4	208.4	208.4	208.4	208.4
2-AMINOANTHRAQUINONE STD	373.8	373.8	373.8	373.8	373.8	373.8	373.8
SOLVENT ORANGE 3 STD	255.8	255.8	255.8	255.8	255.8	255.8	255.8
1-AMINOANTHRAQUINONE STD	257.0	257.0	257.0	257.0	257.0	257.0	257.0
SOLVENT YELLOW 14 STD	283.2	283.2	283.2	283.2	283.2	283.2	283.2
SOLVENT ORANGE 7 STD	288.0	288.0	288.0	288.0	288.0	288.0	288.0
ACID YELLOW 3 STD	334.8	334.8	334.8	334.8	334.8	334.8	334.8
SOLVENT YELLOW 3 STD	302.0	302.0	302.0	302.0	302.0	302.0	302.0
SOLVENT RED 1 STD	463.8	463.8	463.8	463.8	463.8	463.8	463.8
DISPERSE RED 9 STD	306.0	306.0	306.0	306.0	306.0	306.0	306.0
SOLVENT YELLOW 2 STD	394.0	394.0	394.0	394.0	394.0	394.0	394.0
SOLVENT GREEN 3 STD	314.0	314.0	314.0	314.0	314.0	314.0	314.0
SOLVENT RED 24 STD	56.8	56.8	56.8	56.8	56.8	56.8	56.8
ANTHRACENE	415.4	415.4	415.4	415.4	415.4	415.4	415.4
BENZANTHRONE	243.2	243.2	243.2	243.2	243.2	243.2	243.2

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Weight of Stock Standard Spiked						
SPIKE VOLUME EACH DYE	4000.0	4000.0	4000.0	4000.0	4000.0	4000.0	4000.0
AB45 SPIKE WEIGHT	1377.6	1377.6	1377.6	1377.6	1377.6	1377.6	1377.6
AY23 SPIKE WEIGHT	1124.8	1124.8	1124.8	1124.8	1124.8	1124.8	1124.8
AY73 SPIKE WEIGHT	1404.0	1404.0	1404.0	1404.0	1404.0	1404.0	1404.0
AO10 SPIKE WEIGHT	1229.6	1229.6	1229.6	1229.6	1229.6	1229.6	1229.6
AB1 SPIKE WEIGHT	1476.8	1476.8	1476.8	1476.8	1476.8	1476.8	1476.8
BY2 SPIKE WEIGHT	1056.8	1056.8	1056.8	1056.8	1056.8	1056.8	1056.8
DV1 SPIKE WEIGHT	1040.8	1040.8	1040.8	1040.8	1040.8	1040.8	1040.8
SY33 SPIKE WEIGHT	1118.4	1118.4	1118.4	1118.4	1118.4	1118.4	1118.4
DB14 SPIKE WEIGHT	1196.0	1196.0	1196.0	1196.0	1196.0	1196.0	1196.0
AR64 SPIKE WEIGHT	1447.2	1447.2	1447.2	1447.2	1447.2	1447.2	1447.2
AB9 SPIKE WEIGHT	1312.0	1312.0	1312.0	1312.0	1312.0	1312.0	1312.0
BV10 SPIKE WEIGHT	833.6	833.6	833.6	833.6	833.6	833.6	833.6
2AQ SPIKE WEIGHT	1495.2	1495.2	1495.2	1495.2	1495.2	1495.2	1495.2
SO3 SPIKE WEIGHT	1023.2	1023.2	1023.2	1023.2	1023.2	1023.2	1023.2
1AQ SPIKE WEIGHT	1028.0	1028.0	1028.0	1028.0	1028.0	1028.0	1028.0
SY14 SPIKE WEIGHT	1132.8	1132.8	1132.8	1132.8	1132.8	1132.8	1132.8
SO7 SPIKE WEIGHT	1152.0	1152.0	1152.0	1152.0	1152.0	1152.0	1152.0
AY3 SPIKE WEIGHT	1339.2	1339.2	1339.2	1339.2	1339.2	1339.2	1339.2
SY3 SPIKE WEIGHT	1208.0	1208.0	1208.0	1208.0	1208.0	1208.0	1208.0
SR1 SPIKE WEIGHT	1855.2	1855.2	1855.2	1855.2	1855.2	1855.2	1855.2
DR9 SPIKE WEIGHT	1224.0	1224.0	1224.0	1224.0	1224.0	1224.0	1224.0
SY2 SPIKE WEIGHT	1576.0	1576.0	1576.0	1576.0	1576.0	1576.0	1576.0
SG3 SPIKE WEIGHT	1256.0	1256.0	1256.0	1256.0	1256.0	1256.0	1256.0
SR24 SPIKE WEIGHT	227.2	227.2	227.2	227.2	227.2	227.2	227.2
AN SPIKE WEIGHT	1661.6	1661.6	1661.6	1661.6	1661.6	1661.6	1661.6
BZ SPIKE WEIGHT	972.8	972.8	972.8	972.8	972.8	972.8	972.8

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Weight Standard per Volume Sample						
MATRIX VOLUME (WATER)	0.250	0.250	0.250	0.250	0.250	0.250	0.250
MATRIX WEIGHT (SOIL)	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB45	5510.4	5510.4	5510.4	5510.4	5510.4	5510.4	5510.4
SPIKE CONC AY23	4499.2	4499.2	4499.2	4499.2	4499.2	4499.2	4499.2
SPIKE CONC AY73	5616.0	5616.0	5616.0	5616.0	5616.0	5616.0	5616.0
SPIKE CONC AO10	4918.4	4918.4	4918.4	4918.4	4918.4	4918.4	4918.4
SPIKE CONC AB1	5907.2	5907.2	5907.2	5907.2	5907.2	5907.2	5907.2
SPIKE CONC BY2	4227.2	4227.2	4227.2	4227.2	4227.2	4227.2	4227.2
SPIKE CONC DV1	4163.2	4163.2	4163.2	4163.2	4163.2	4163.2	4163.2
SPIKE CONC SY33	4473.6	4473.6	4473.6	4473.6	4473.6	4473.6	4473.6
SPIKE CONC DB14	4784.0	4784.0	4784.0	4784.0	4784.0	4784.0	4784.0
SPIKE CONC AR64	5788.8	5788.8	5788.8	5788.8	5788.8	5788.8	5788.8
SPIKE CONC AB9	5248.0	5248.0	5248.0	5248.0	5248.0	5248.0	5248.0
SPIKE CONC BV10	3334.4	3334.4	3334.4	3334.4	3334.4	3334.4	3334.4
SPIKE CONC 2AQ	5980.8	5980.8	5980.8	5980.8	5980.8	5980.8	5980.8
SPIKE CONC SO3	4092.8	4092.8	4092.8	4092.8	4092.8	4092.8	4092.8
SPIKE CONC 1AQ	4112.0	4112.0	4112.0	4112.0	4112.0	4112.0	4112.0
SPIKE CONC SY14	4531.2	4531.2	4531.2	4531.2	4531.2	4531.2	4531.2
SPIKE CONC SO7	4608.0	4608.0	4608.0	4608.0	4608.0	4608.0	4608.0
SPIKE CONC AY3	5356.8	5356.8	5356.8	5356.8	5356.8	5356.8	5356.8
SPIKE CONC SY3	4832.0	4832.0	4832.0	4832.0	4832.0	4832.0	4832.0
SPIKE CONC SR1	7420.8	7420.8	7420.8	7420.8	7420.8	7420.8	7420.8
SPIKE CONC DR9	4896.0	4896.0	4896.0	4896.0	4896.0	4896.0	4896.0
SPIKE CONC SY2	6304.0	6304.0	6304.0	6304.0	6304.0	6304.0	6304.0
SPIKE CONC SG3	5024.0	5024.0	5024.0	5024.0	5024.0	5024.0	5024.0
SPIKE CONC SR24	908.8	908.8	908.8	908.8	908.8	908.8	908.8
SPIKE CONC AN	6646.4	6646.4	6646.4	6646.4	6646.4	6646.4	6646.4
SPIKE CONC BZ	3891.2	3891.2	3891.2	3891.2	3891.2	3891.2	3891.2

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
SPIKE CONC AB45	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY23	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY73	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AO10	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BY2	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DV1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY33	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DB14	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AR64	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB9	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BV10	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 2AQ	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 1AQ	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY14	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO7	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DR9	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY2	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SG3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR24	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AN	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BZ	NA	NA	NA	NA	NA	NA	NA

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Results from HPLC						
HPLC RESULT AB45	4.473	3.521	6.931	3.770	5.360	4.138	4.245
HPLC RESULT AY23	7.427	6.987	6.485	6.994	7.232	7.159	7.139
HPLC RESULT AY73	11.974	11.633	11.656	11.828	11.437	11.307	11.252
HPLC RESULT AO10	17.760	16.563	16.154	16.411	16.580	16.784	15.967
HPLC RESULT AB1	14.816	13.190	13.241	13.807	13.738	14.037	13.869
HPLC RESULT BY2	2.894	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT DV1	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SY33	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT DB14	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT AR64	14.015	14.075	13.816	13.916	14.271	13.851	13.716
HPLC RESULT AB9	13.376	12.254	11.733	12.052	12.093	12.247	11.960
HPLC RESULT BV10	5.084	3.065	2.709	3.010	3.250	3.368	3.118
HPLC RESULT 2AQ	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SO3	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT 1AQ	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SY14	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SO7	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT AY3	8.467	7.664	7.059	7.310	7.327	7.299	7.057
HPLC RESULT SY3	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SR1	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT DR9	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SY2	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SG3	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SR24	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT AN	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT BZ	0.000	0.000	0.000	0.000	0.000	0.000	0.000

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Final Sample Results						
CF (STD INJ VOL VS SAM INJ VOL)	2	2	2	2	2	2	2
FINAL VOLUME	250.000	250.000	250.000	250.000	250.000	250.000	250.000
CORRECTED RESULT AB45	2236.50	1760.50	3465.50	1885.00	2680.00	2069.00	2122.50
CORRECTED RESULT AY23	3713.50	3493.50	3242.50	3497.00	3616.00	3579.50	3569.50
CORRECTED RESULT AY73	5987.00	5816.50	5828.00	5914.00	5718.50	5653.50	5626.00
CORRECTED RESULT AO10	8880.00	8281.50	8077.00	8205.50	8290.00	8392.00	7983.50
CORRECTED RESULT AB1	7408.00	6595.00	6620.50	6903.50	6869.00	7018.50	6934.50
CORRECTED RESULT BY2	1447.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT DV1	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SY33	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT DB14	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT AR64	7007.50	7037.50	6908.00	6958.00	7135.50	6925.50	6858.00
CORRECTED RESULT AB9	6688.00	6127.00	5866.50	6026.00	6046.50	6123.50	5980.00
CORRECTED RESULT BV10	2542.00	1532.50	1354.50	1505.00	1625.00	1684.00	1559.00
CORRECTED RESULT 2AQ	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SO3	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT 1AQ	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SY14	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SO7	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT AY3	4233.50	3832.00	3529.50	3655.00	3663.50	3649.50	3528.50
CORRECTED RESULT SY3	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SR1	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT DR9	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SY2	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SG3	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SR24	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT AN	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT BZ	0.00	0.00	0.00	0.00	0.00	0.00	0.00

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Percent Recoveries						
% RECOVERY AB45	40.59	31.95	62.89	34.21	48.64	37.55	38.52
% RECOVERY AY23	82.5	77.6	72.1	77.7	80.4	79.6	79.3
% RECOVERY AY73	106.6	103.6	103.8	105.3	101.8	100.7	100.2
% RECOVERY AO10	180.5	168.4	164.2	166.8	168.6	170.6	162.3
% RECOVERY AB1	125.4	111.6	112.1	116.9	116.3	118.8	117.4
% RECOVERY BY2	34.2	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY DV1	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SY33	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY DB14	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY AR64	121.1	121.6	119.3	120.2	123.3	119.6	118.5
% RECOVERY AB9	127.4	116.7	111.8	114.8	115.2	116.7	113.9
% RECOVERY BV10	76.2	46.0	40.6	45.1	48.7	50.5	46.8
% RECOVERY 2AQ	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SO3	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY 1AQ	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SY14	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SO7	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY AY3	79.0	71.5	65.9	68.2	68.4	68.1	65.9
% RECOVERY SY3	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SR1	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY DR9	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SY2	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SG3	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SR24	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY AN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY BZ	0.0	0.0	0.0	0.0	0.0	0.0	0.0

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Square of Corrected Results						
N	7	7	7	7	7	7	7
Xi^2 AB45	5001932.3	3099360.3	12009690.3	3553225.0	7182400.0	4280761.0	4505006.3
Xi^2 AY23	13790082.3	12204542.3	10513806.3	12229009.0	13075456.0	12812820.3	12741330.3
Xi^2 AY73	35844169.0	33831672.3	33965584.0	34975396.0	32701242.3	31962062.3	31651876.0
Xi^2 AO10	7.885E+07	6.858E+07	6.524E+07	6.733E+07	6.872E+07	7.043E+07	6.374E+07
Xi^2 AB1	54878464.0	43494025.0	43831020.3	47658312.3	47183161.0	49259342.3	48087290.3
Xi^2 BY2	2093809.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 DV1	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SY33	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 DB14	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 AR64	49105056.3	49526406.3	47720464.0	48413764.0	50915360.3	47962550.3	47032164.0
Xi^2 AB9	44729344.0	37540129.0	34415822.3	36312676.0	36560162.3	37497252.3	35760400.0
Xi^2 BV10	6461764.0	2348556.3	1834670.3	2265025.0	2640625.0	2835856.0	2430481.0
Xi^2 2AQ	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SO3	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 1AQ	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SY14	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SO7	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 AY3	17922522.3	14684224.0	12457370.3	13359025.0	13421232.3	13318850.3	12450312.3
Xi^2 SY3	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SR1	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 DR9	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SY2	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SG3	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SR24	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 AN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 BZ	0.0	0.0	0.0	0.0	0.0	0.0	0.0

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Sum of the Square of Corrected Results						
E Xi^2 AB45	3.963E+07						
E Xi^2 AY23	8.737E+07						
E Xi^2 AY73	2.349E+08						
E Xi^2 AO10	4.829E+08						
E Xi^2 AB1	3.344E+08						
E Xi^2 BY2	2.094E+06						
E Xi^2 DV1	0.000E+00						
E Xi^2 SY33	0.000E+00						
E Xi^2 DB14	0.000E+00						
E Xi^2 AR64	3.407E+08						
E Xi^2 AB9	2.628E+08						
E Xi^2 BV10	2.082E+07						
E Xi^2 2AQ	0.000E+00						
E Xi^2 SO3	0.000E+00						
E Xi^2 1AQ	0.000E+00						
E Xi^2 SY14	0.000E+00						
E Xi^2 SO7	0.000E+00						
E Xi^2 AY3	9.761E+07						
E Xi^2 SY3	0.000E+00						
E Xi^2 SR1	0.000E+00						
E Xi^2 DR9	0.000E+00						
E Xi^2 SY2	0.000E+00						
E Xi^2 SG3	0.000E+00						
E Xi^2 SR24	0.000E+00						
E Xi^2 AN	0.000E+00						
E Xi^2 BZ	0.000E+00						

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Sum of the Corrected Results Squared						
(E Xi)^2 AB45	2.631E+08						
(E Xi)^2 AY23	6.107E+08						
(E Xi)^2 AY73	1.644E+09						
(E Xi)^2 AO10	3.377E+09						
(E Xi)^2 AB1	2.338E+09						
(E Xi)^2 BY2	2.094E+06						
(E Xi)^2 DV1	0.000E+00						
(E Xi)^2 SY33	0.000E+00						
(E Xi)^2 DB14	0.000E+00						
(E Xi)^2 AR64	2.384E+09						
(E Xi)^2 AB9	1.837E+09						
(E Xi)^2 BV10	1.393E+08						
(E Xi)^2 2AQ	0.000E+00						
(E Xi)^2 SO3	0.000E+00						
(E Xi)^2 1AQ	0.000E+00						
(E Xi)^2 SY14	0.000E+00						
(E Xi)^2 SO7	0.000E+00						
(E Xi)^2 AY3	6.808E+08						
(E Xi)^2 SY3	0.000E+00						
(E Xi)^2 SR1	0.000E+00						
(E Xi)^2 DR9	0.000E+00						
(E Xi)^2 SY2	0.000E+00						
(E Xi)^2 SG3	0.000E+00						
(E Xi)^2 SR24	0.000E+00						
(E Xi)^2 AN	0.000E+00						
(E Xi)^2 BZ	0.000E+00						

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Variance						
S^2: VARIANCE AB45	342158.7						
S^2: VARIANCE AY23	21692.7						
S^2: VARIANCE AY73	17824.3						
S^2: VARIANCE AO10	84020.8						
S^2: VARIANCE AB1	74178.7						
S^2: VARIANCE BY2	299115.6						
S^2: VARIANCE DV1	0.0						
S^2: VARIANCE SY33	0.0						
S^2: VARIANCE DB14	0.0						
S^2: VARIANCE AR64	8606.1						
S^2: VARIANCE AB9	70123.7						
S^2: VARIANCE BV10	153134.3						
S^2: VARIANCE 2AQ	0.0						
S^2: VARIANCE SO3	0.0						
S^2: VARIANCE 1AQ	0.0						
S^2: VARIANCE SY14	0.0						
S^2: VARIANCE SO7	0.0						
S^2: VARIANCE AY3	60199.6						
S^2: VARIANCE SY3	0.0						
S^2: VARIANCE SR1	0.0						
S^2: VARIANCE DR9	0.0						
S^2: VARIANCE SY2	0.0						
S^2: VARIANCE SG3	0.0						
S^2: VARIANCE SR24	0.0						
S^2: VARIANCE AN	0.0						
S^2: VARIANCE BZ	0.0						

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Standard Deviation						
STD DEVIATION AB45	584.9						
STD DEVIATION AY23	147.3						
STD DEVIATION AY73	133.5						
STD DEVIATION AO10	289.9						
STD DEVIATION AB1	272.4						
STD DEVIATION BY2	546.9						
STD DEVIATION DV1	0.0						
STD DEVIATION SY33	0.0						
STD DEVIATION DB14	0.0						
STD DEVIATION AR64	92.8						
STD DEVIATION AB9	264.8						
STD DEVIATION BV10	391.3						
STD DEVIATION 2AQ	0.0						
STD DEVIATION SO3	0.0						
STD DEVIATION 1AQ	0.0						
STD DEVIATION SY14	0.0						
STD DEVIATION SO7	0.0						
STD DEVIATION AY3	245.4						
STD DEVIATION SY3	0.0						
STD DEVIATION SR1	0.0						
STD DEVIATION DR9	0.0						
STD DEVIATION SY2	0.0						
STD DEVIATION SG3	0.0						
STD DEVIATION SR24	0.0						
STD DEVIATION AN	0.0						
STD DEVIATION BZ	0.0						

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Method Detection Limit						
T (STUDENT'S T @ 99% FOR 6 DF)	3.140						
MDL AB45	1836.7						
MDL AY23	462.5						
MDL AY73	419.2						
MDL AO10	910.2						
MDL AB1	855.2						
MDL BY2	1717.3						
MDL DV1	0.0						
MDL SY33	0.0						
MDL DB14	0.0						
MDL AR64	291.3						
MDL AB9	831.5						
MDL BV10	1228.8						
MDL 2AQ	0.0						
MDL SO3	0.0						
MDL 1AQ	0.0						
MDL SY14	0.0						
MDL SO7	0.0						
MDL AY3	770.4						
MDL SY3	0.0						
MDL SR1	0.0						
MDL DR9	0.0						
MDL SY2	0.0						
MDL SG3	0.0						
MDL SR24	0.0						
MDL AN	0.0						
MDL BZ	0.0						

TABLE VII-11

High-Level Aqueous Extraction Method, Short Gradient, Low Spike

	Sample Conc µg/L	Sample Results (µg/L)							Mean Result µg/L	Method Bias (Accuracy) %R	Standard Deviation	Method Precision (%RSD)	MDL µg/L	RQL µg/L
		WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS						
ACID BLUE 45	2755.2	3560.0	4778.5	4036.5	3774.0	951.5	1212.0	976.5	2755.6	100.0	1644.2	59.7	5162.7	20650.7
ACID YELLOW 23	2249.6	4864.5	3962.0	2746.0	2678.0	2522.0	1950.5	2919.0	3091.7	137.4	987.2	31.9	3099.9	12399.5
ACID YELLOW 73	2808	5067.5	5897.5	5936.0	5983.0	0.0	734.5	0.0	3374.1	120.2	2953.6	87.5	9274.2	37096.8
ACID ORANGE 10	2459.2	8718.5	8781.5	8967.0	9086.0	6988.0	7272.5	6347.5	8023.0	326.2	1119.7	14.0	3515.8	14063.0
ACID BLUE 1	2953.6	7714.0	7319.0	7592.0	7217.5	6342.5	7527.5	6798.5	7215.9	244.3	488.9	6.8	1535.2	6141.0
BASIC YELLOW 2	2113.6	3433.5	0.0	906.0	0.0	0.0	3670.5	0.0	1144.3	54.1	1679.1	146.7	5272.4	21089.7
DISPERSE VIOLET 1	2081.6	2998.5	0.0	0.0	0.0	0.0	2186.5	0.0	740.7	35.6	1286.5	173.7	4039.7	16159.0
SOLVENT YELLOW 33	2236.8	143.0	0.0	0.0	0.0	0.0	0.0	0.0	20.4	0.9	54.0	264.6	169.7	678.9
DISPERSE BLUE 14	2392	351.5	0.0	0.0	0.0	0.0	171.5	0.0	74.7	3.1	137.8	184.4	432.6	1730.4
ACID RED 64	2894.4	3543.0	3480.0	3441.0	3428.0	3199.5	3128.0	3200.5	3345.7	115.6	164.7	4.9	517.0	2068.1
ACID BLUE 9	2624	6094.0	5927.0	6323.5	6010.0	4614.5	5851.0	4517.0	5619.6	214.2	735.6	13.1	2309.9	9239.6
BASIC VIOLET 10	1667.2	3993.5	3185.5	3367.5	2807.0	1687.5	3862.5	2095.5	2999.9	179.9	863.9	28.8	2712.8	10851.2
2-AMINOANTHRAQUINONE	2990.4	2413.5	173.5	197.0	0.0	0.0	2166.5	0.0	707.2	23.6	1086.8	153.7	3412.5	13650.0
SOLVENT ORANGE 3	2046.4	3356.0	555.5	576.0	113.0	814.0	4181.0	935.0	1504.4	73.5	1586.0	105.4	4979.9	19919.8
1-AMINOANTHRAQUINONE	2056	2461.0	0.0	0.0	0.0	0.0	2130.0	0.0	655.9	31.9	1124.2	171.4	3529.9	14119.4
SOLVENT YELLOW 14	2265.6	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT ORANGE 7	2304	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ACID YELLOW 3	2678.4	7442.5	2432.5	2746.0	1494.5	1754.5	3724.0	1845.5	3062.8	114.4	2072.4	67.7	6507.4	26029.6
SOLVENT YELLOW 3	2416	3724.5	0.0	0.0	0.0	0.0	3160.0	0.0	983.5	40.7	1687.5	171.6	5298.9	21195.4
SOLVENT RED 1	3710.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
DISPERSE RED 9	2448	834.0	0.0	0.0	0.0	0.0	0.0	0.0	119.1	4.9	315.2	264.6	989.8	3959.2
SOLVENT YELLOW 2	3152	806.5	0.0	0.0	0.0	0.0	708.5	0.0	216.4	6.9	370.7	171.3	1164.0	4656.0
SOLVENT GREEN 3	2512	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT RED 24	454.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ANTHRACENE	3323.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
BENZANTHRONE	1945.6	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

TABLE VII - 11A

PERCENT RECOVERIES
HIGH-LEVEL AQUEOUS METHOD
LOW SPIKE, SHORT GRADIENT

Compounds		Ret. Time	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
% RECOVERY AB45	%	4.439	129	173	147	137	35	44	35
% RECOVERY AR64	%	6.174	122	120	119	118	111	108	111
% RECOVERY AY3	%	7.322	278	91	103	56	66	139	69
% RECOVERY AY23	%	7.525	216	176	122	119	112	87	130
% RECOVERY AY73	%	9.159	180	210	211	213	0	26	0
% RECOVERY AO10	%	9.946	355	357	365	369	284	296	258
% RECOVERY AB9	%	11.576	232	226	241	229	176	223	172
% RECOVERY DV1	%	12.69	144	0	0	0	0	105	0
% RECOVERY AB1	%	12.709	261	248	257	244	215	255	230
% RECOVERY 2AQ	%	13.246	81	6	7	0	0	72	0
% RECOVERY SO3	%	13.811	164	27	28	6	40	204	46
% RECOVERY SY33	%	15.498	6	0	0	0	0	0	0
% RECOVERY 1AQ	%	16.108	120	0	0	0	0	104	0
% RECOVERY DB14	%	18.857	15	0	0	0	0	7	0
% RECOVERY BY2	%	19.452	162	0	43	0	0	174	0
% RECOVERY BV10	%	19.691	240	191	202	168	101	232	126
% RECOVERY SY3	%	19.962	154	0	0	0	0	131	0
% RECOVERY DR9	%	20.367	34	0	0	0	0	0	0
% RECOVERY BZ	%	20.438	0	0	0	0	0	0	0
% RECOVERY SY2	%	22.244	26	0	0	0	0	22	0
% RECOVERY AN	%	22.537	0	0	0	0	0	0	0
% RECOVERY SY14	%	24.403	0	0	0	0	0	0	0
% RECOVERY SR1	%	24.66	0	0	0	0	0	0	0
% RECOVERY SO7	%	27.804	0	0	0	0	0	0	0
% RECOVERY SG3	%	31.194	0	0	0	0	0	0	0
% RECOVERY SR24	%	31.255	0	0	0	0	0	0	0

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Stock Standard Concentrations						
ACID BLUE 45 STD	344.4	344.4	344.4	344.4	344.4	344.4	344.4
ACID YELLOW 23 STD	281.2	281.2	281.2	281.2	281.2	281.2	281.2
ACID YELLOW 73 STD	351.0	351.0	351.0	351.0	351.0	351.0	351.0
ACID ORANGE 10 STD	307.4	307.4	307.4	307.4	307.4	307.4	307.4
ACID BLUE 1 STD	369.2	369.2	369.2	369.2	369.2	369.2	369.2
BASIC YELLOW 2 STD	264.2	264.2	264.2	264.2	264.2	264.2	264.2
DISPERSE VIOLET 1 STD	260.2	260.2	260.2	260.2	260.2	260.2	260.2
SOLVENT YELLOW 33 STD	279.6	279.6	279.6	279.6	279.6	279.6	279.6
DISPERSE BLUE 14 STD	299.0	299.0	299.0	299.0	299.0	299.0	299.0
ACID RED 64 STD	361.8	361.8	361.8	361.8	361.8	361.8	361.8
ACID BLUE 9 STD	328.0	328.0	328.0	328.0	328.0	328.0	328.0
BASIC VIOLET 10 STD	208.4	208.4	208.4	208.4	208.4	208.4	208.4
2-AMINOANTHRAQUINONE STD	373.8	373.8	373.8	373.8	373.8	373.8	373.8
SOLVENT ORANGE 3 STD	255.8	255.8	255.8	255.8	255.8	255.8	255.8
1-AMINOANTHRAQUINONE STD	257.0	257.0	257.0	257.0	257.0	257.0	257.0
SOLVENT YELLOW 14 STD	283.2	283.2	283.2	283.2	283.2	283.2	283.2
SOLVENT ORANGE 7 STD	288.0	288.0	288.0	288.0	288.0	288.0	288.0
ACID YELLOW 3 STD	334.8	334.8	334.8	334.8	334.8	334.8	334.8
SOLVENT YELLOW 3 STD	302.0	302.0	302.0	302.0	302.0	302.0	302.0
SOLVENT RED 1 STD	463.8	463.8	463.8	463.8	463.8	463.8	463.8
DISPERSE RED 9 STD	306.0	306.0	306.0	306.0	306.0	306.0	306.0
SOLVENT YELLOW 2 STD	394.0	394.0	394.0	394.0	394.0	394.0	394.0
SOLVENT GREEN 3 STD	314.0	314.0	314.0	314.0	314.0	314.0	314.0
SOLVENT RED 24 STD	56.8	56.8	56.8	56.8	56.8	56.8	56.8
ANTHRACENE	415.4	415.4	415.4	415.4	415.4	415.4	415.4
BENZANTHRONE	243.2	243.2	243.2	243.2	243.2	243.2	243.2

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Weight of Stock Standard Spiked						
SPIKE VOLUME EACH DYE	200.0	200.0	200.0	200.0	200.0	200.0	200.0
AB45 SPIKE WEIGHT	68.9	68.9	68.9	68.9	68.9	68.9	68.9
AY23 SPIKE WEIGHT	56.2	56.2	56.2	56.2	56.2	56.2	56.2
AY73 SPIKE WEIGHT	70.2	70.2	70.2	70.2	70.2	70.2	70.2
AO10 SPIKE WEIGHT	61.5	61.5	61.5	61.5	61.5	61.5	61.5
AB1 SPIKE WEIGHT	73.8	73.8	73.8	73.8	73.8	73.8	73.8
BY2 SPIKE WEIGHT	52.8	52.8	52.8	52.8	52.8	52.8	52.8
DV1 SPIKE WEIGHT	52.0	52.0	52.0	52.0	52.0	52.0	52.0
SY33 SPIKE WEIGHT	55.9	55.9	55.9	55.9	55.9	55.9	55.9
DB14 SPIKE WEIGHT	59.8	59.8	59.8	59.8	59.8	59.8	59.8
AR64 SPIKE WEIGHT	72.4	72.4	72.4	72.4	72.4	72.4	72.4
AB9 SPIKE WEIGHT	65.6	65.6	65.6	65.6	65.6	65.6	65.6
BV10 SPIKE WEIGHT	41.7	41.7	41.7	41.7	41.7	41.7	41.7
2AQ SPIKE WEIGHT	74.8	74.8	74.8	74.8	74.8	74.8	74.8
SO3 SPIKE WEIGHT	51.2	51.2	51.2	51.2	51.2	51.2	51.2
1AQ SPIKE WEIGHT	51.4	51.4	51.4	51.4	51.4	51.4	51.4
SY14 SPIKE WEIGHT	56.6	56.6	56.6	56.6	56.6	56.6	56.6
SO7 SPIKE WEIGHT	57.6	57.6	57.6	57.6	57.6	57.6	57.6
AY3 SPIKE WEIGHT	67.0	67.0	67.0	67.0	67.0	67.0	67.0
SY3 SPIKE WEIGHT	60.4	60.4	60.4	60.4	60.4	60.4	60.4
SR1 SPIKE WEIGHT	92.8	92.8	92.8	92.8	92.8	92.8	92.8
DR9 SPIKE WEIGHT	61.2	61.2	61.2	61.2	61.2	61.2	61.2
SY2 SPIKE WEIGHT	78.8	78.8	78.8	78.8	78.8	78.8	78.8
SG3 SPIKE WEIGHT	62.8	62.8	62.8	62.8	62.8	62.8	62.8
SR24 SPIKE WEIGHT	11.4	11.4	11.4	11.4	11.4	11.4	11.4
AN SPIKE WEIGHT	83.1	83.1	83.1	83.1	83.1	83.1	83.1
BZ SPIKE WEIGHT	48.6	48.6	48.6	48.6	48.6	48.6	48.6

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Weight Standard per Volume Sample						
MATRIX VOLUME (WATER)	0.025	0.025	0.025	0.025	0.025	0.025	0.025
SPIKE CONC AB45	2755.2	2755.2	2755.2	2755.2	2755.2	2755.2	2755.2
SPIKE CONC AY23	2249.6	2249.6	2249.6	2249.6	2249.6	2249.6	2249.6
SPIKE CONC AY73	2808.0	2808.0	2808.0	2808.0	2808.0	2808.0	2808.0
SPIKE CONC AO10	2459.2	2459.2	2459.2	2459.2	2459.2	2459.2	2459.2
SPIKE CONC AB1	2953.6	2953.6	2953.6	2953.6	2953.6	2953.6	2953.6
SPIKE CONC BY2	2113.6	2113.6	2113.6	2113.6	2113.6	2113.6	2113.6
SPIKE CONC DV1	2081.6	2081.6	2081.6	2081.6	2081.6	2081.6	2081.6
SPIKE CONC SY33	2236.8	2236.8	2236.8	2236.8	2236.8	2236.8	2236.8
SPIKE CONC DB14	2392.0	2392.0	2392.0	2392.0	2392.0	2392.0	2392.0
SPIKE CONC AR64	2894.4	2894.4	2894.4	2894.4	2894.4	2894.4	2894.4
SPIKE CONC AB9	2624.0	2624.0	2624.0	2624.0	2624.0	2624.0	2624.0
SPIKE CONC BV10	1667.2	1667.2	1667.2	1667.2	1667.2	1667.2	1667.2
SPIKE CONC 2AQ	2990.4	2990.4	2990.4	2990.4	2990.4	2990.4	2990.4
SPIKE CONC SO3	2046.4	2046.4	2046.4	2046.4	2046.4	2046.4	2046.4
SPIKE CONC 1AQ	2056.0	2056.0	2056.0	2056.0	2056.0	2056.0	2056.0
SPIKE CONC SY14	2265.6	2265.6	2265.6	2265.6	2265.6	2265.6	2265.6
SPIKE CONC SO7	2304.0	2304.0	2304.0	2304.0	2304.0	2304.0	2304.0
SPIKE CONC AY3	2678.4	2678.4	2678.4	2678.4	2678.4	2678.4	2678.4
SPIKE CONC SY3	2416.0	2416.0	2416.0	2416.0	2416.0	2416.0	2416.0
SPIKE CONC SR1	3710.4	3710.4	3710.4	3710.4	3710.4	3710.4	3710.4
SPIKE CONC DR9	2448.0	2448.0	2448.0	2448.0	2448.0	2448.0	2448.0
SPIKE CONC SY2	3152.0	3152.0	3152.0	3152.0	3152.0	3152.0	3152.0
SPIKE CONC SG3	2512.0	2512.0	2512.0	2512.0	2512.0	2512.0	2512.0
SPIKE CONC SR24	454.4	454.4	454.4	454.4	454.4	454.4	454.4
SPIKE CONC AN	3323.2	3323.2	3323.2	3323.2	3323.2	3323.2	3323.2
SPIKE CONC BZ	1945.6	1945.6	1945.6	1945.6	1945.6	1945.6	1945.6

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
SPIKE CONC AB45	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY23	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY73	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AO10	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BY2	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DV1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY33	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DB14	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AR64	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB9	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BV10	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 2AQ	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 1AQ	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY14	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO7	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DR9	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY2	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SG3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR24	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AN	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BZ	NA	NA	NA	NA	NA	NA	NA

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Results from HPLC						
HPLC RESULT AB45	7.120	9.557	8.073	7.548	1.903	2.424	1.953
HPLC RESULT AY23	9.729	7.924	5.492	5.356	5.044	3.901	5.838
HPLC RESULT AY73	10.135	11.795	11.872	11.966	0.000	1.469	0.000
HPLC RESULT AO10	17.437	17.563	17.934	18.172	13.976	14.545	12.695
HPLC RESULT AB1	15.428	14.638	15.184	14.435	12.685	15.055	13.597
HPLC RESULT BY2	6.867	0.000	1.812	0.000	0.000	7.341	0.000
HPLC RESULT DV1	5.997	0.000	0.000	0.000	0.000	4.373	0.000
HPLC RESULT SY33	0.286	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT DB14	0.703	0.000	0.000	0.000	0.000	0.343	0.000
HPLC RESULT AR64	7.086	6.960	6.882	6.856	6.399	6.256	6.401
HPLC RESULT AB9	12.188	11.854	12.647	12.020	9.229	11.702	9.034
HPLC RESULT BV10	7.987	6.371	6.735	5.614	3.375	7.725	4.191
HPLC RESULT 2AQ	4.827	0.347	0.394	0.000	0.000	4.333	0.000
HPLC RESULT SO3	6.712	1.111	1.152	0.226	1.628	8.362	1.870
HPLC RESULT 1AQ	4.922	0.000	0.000	0.000	0.000	4.260	0.000
HPLC RESULT SY14	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SO7	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT AY3	14.885	4.865	5.492	2.989	3.509	7.448	3.691
HPLC RESULT SY3	7.449	0.000	0.000	0.000	0.000	6.320	0.000
HPLC RESULT SR1	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT DR9	1.668	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SY2	1.613	0.000	0.000	0.000	0.000	1.417	0.000
HPLC RESULT SG3	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SR24	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT AN	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT BZ	0.000	0.000	0.000	0.000	0.000	0.000	0.000

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Final Sample Results						
CF (STD INJ VOL VS SAM INJ VOL)	2	2	2	2	2	2	2
FINAL VOLUME	25.000	25.000	25.000	25.000	25.000	25.000	25.000
CORRECTED RESULT AB45	3560.00	4778.50	4036.50	3774.00	951.50	1212.00	976.50
CORRECTED RESULT AY23	4864.50	3962.00	2746.00	2678.00	2522.00	1950.50	2919.00
CORRECTED RESULT AY73	5067.50	5897.50	5936.00	5983.00	0.00	734.50	0.00
CORRECTED RESULT AO10	8718.50	8781.50	8967.00	9086.00	6988.00	7272.50	6347.50
CORRECTED RESULT AB1	7714.00	7319.00	7592.00	7217.50	6342.50	7527.50	6798.50
CORRECTED RESULT BY2	3433.50	0.00	906.00	0.00	0.00	3670.50	0.00
CORRECTED RESULT DV1	2998.50	0.00	0.00	0.00	0.00	2186.50	0.00
CORRECTED RESULT SY33	143.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT DB14	351.50	0.00	0.00	0.00	0.00	171.50	0.00
CORRECTED RESULT AR64	3543.00	3480.00	3441.00	3428.00	3199.50	3128.00	3200.50
CORRECTED RESULT AB9	6094.00	5927.00	6323.50	6010.00	4614.50	5851.00	4517.00
CORRECTED RESULT BV10	3993.50	3185.50	3367.50	2807.00	1687.50	3862.50	2095.50
CORRECTED RESULT 2AQ	2413.50	173.50	197.00	0.00	0.00	2166.50	0.00
CORRECTED RESULT SO3	3356.00	555.50	576.00	113.00	814.00	4181.00	935.00
CORRECTED RESULT 1AQ	2461.00	0.00	0.00	0.00	0.00	2130.00	0.00
CORRECTED RESULT SY14	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SO7	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT AY3	7442.50	2432.50	2746.00	1494.50	1754.50	3724.00	1845.50
CORRECTED RESULT SY3	3724.50	0.00	0.00	0.00	0.00	3160.00	0.00
CORRECTED RESULT SR1	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT DR9	834.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SY2	806.50	0.00	0.00	0.00	0.00	708.50	0.00
CORRECTED RESULT SG3	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SR24	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT AN	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT BZ	0.00	0.00	0.00	0.00	0.00	0.00	0.00

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Percent Recoveries						
% RECOVERY AB45	129.21	173.44	146.50	136.98	34.53	43.99	35.44
% RECOVERY AY23	216.2	176.1	122.1	119.0	112.1	86.7	129.8
% RECOVERY AY73	180.5	210.0	211.4	213.1	0.0	26.2	0.0
% RECOVERY AO10	354.5	357.1	364.6	369.5	284.2	295.7	258.1
% RECOVERY AB1	261.2	247.8	257.0	244.4	214.7	254.9	230.2
% RECOVERY BY2	162.4	0.0	42.9	0.0	0.0	173.7	0.0
% RECOVERY DV1	144.0	0.0	0.0	0.0	0.0	105.0	0.0
% RECOVERY SY33	6.4	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY DB14	14.7	0.0	0.0	0.0	0.0	7.2	0.0
% RECOVERY AR64	122.4	120.2	118.9	118.4	110.5	108.1	110.6
% RECOVERY AB9	232.2	225.9	241.0	229.0	175.9	223.0	172.1
% RECOVERY BV10	239.5	191.1	202.0	168.4	101.2	231.7	125.7
% RECOVERY 2AQ	80.7	5.8	6.6	0.0	0.0	72.4	0.0
% RECOVERY SO3	164.0	27.1	28.1	5.5	39.8	204.3	45.7
% RECOVERY 1AQ	119.7	0.0	0.0	0.0	0.0	103.6	0.0
% RECOVERY SY14	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SO7	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY AY3	277.9	90.8	102.5	55.8	65.5	139.0	68.9
% RECOVERY SY3	154.2	0.0	0.0	0.0	0.0	130.8	0.0
% RECOVERY SR1	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY DR9	34.1	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SY2	25.6	0.0	0.0	0.0	0.0	22.5	0.0
% RECOVERY SG3	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SR24	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY AN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY BZ	0.0	0.0	0.0	0.0	0.0	0.0	0.0

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Square of Corrected Results						
N	7	7	7	7	7	7	7
Xi^2 AB45	12673600.0	22834062.3	16293332.3	14243076.0	905352.3	1468944.0	953552.3
Xi^2 AY23	23663360.3	15697444.0	7540516.0	7171684.0	6360484.0	3804450.3	8520561.0
Xi^2 AY73	25679556.3	34780506.3	35236096.0	35796289.0	0.0	539490.3	0.0
Xi^2 AO10	7.601E+07	7.711E+07	8.041E+07	8.256E+07	4.883E+07	5.289E+07	4.029E+07
Xi^2 AB1	59505796.0	53567761.0	57638464.0	52092306.3	40227306.3	56663256.3	46219602.3
Xi^2 BY2	11788922.3	0.0	820836.0	0.0	0.0	13472570.3	0.0
Xi^2 DV1	8991002.3	0.0	0.0	0.0	0.0	4780782.3	0.0
Xi^2 SY33	20449.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 DB14	123552.3	0.0	0.0	0.0	0.0	29412.3	0.0
Xi^2 AR64	12552849.0	12110400.0	11840481.0	11751184.0	10236800.3	9784384.0	10243200.3
Xi^2 AB9	37136836.0	35129329.0	39986652.3	36120100.0	21293610.3	34234201.0	20403289.0
Xi^2 BV10	15948042.3	10147410.3	11340056.3	7879249.0	2847656.3	14918906.3	4391120.3
Xi^2 2AQ	5824982.3	30102.3	38809.0	0.0	0.0	4693722.3	0.0
Xi^2 SO3	11262736.0	308580.3	331776.0	12769.0	662596.0	17480761.0	874225.0
Xi^2 1AQ	6056521.0	0.0	0.0	0.0	0.0	4536900.0	0.0
Xi^2 SY14	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SO7	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 AY3	55390806.3	5917056.3	7540516.0	2233530.3	3078270.3	13868176.0	3405870.3
Xi^2 SY3	13871900.3	0.0	0.0	0.0	0.0	9985600.0	0.0
Xi^2 SR1	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 DR9	695556.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SY2	650442.3	0.0	0.0	0.0	0.0	501972.3	0.0
Xi^2 SG3	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SR24	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 AN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 BZ	0.0	0.0	0.0	0.0	0.0	0.0	0.0

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Sum of the Square of Corrected Results						
E Xi^2 AB45	6.937E+07						
E Xi^2 AY23	7.276E+07						
E Xi^2 AY73	1.320E+08						
E Xi^2 AO10	4.581E+08						
E Xi^2 AB1	3.659E+08						
E Xi^2 BY2	2.608E+07						
E Xi^2 DV1	1.377E+07						
E Xi^2 SY33	2.045E+04						
E Xi^2 DB14	1.530E+05						
E Xi^2 AR64	7.852E+07						
E Xi^2 AB9	2.243E+08						
E Xi^2 BV10	6.747E+07						
E Xi^2 2AQ	1.059E+07						
E Xi^2 SO3	3.093E+07						
E Xi^2 1AQ	1.059E+07						
E Xi^2 SY14	0.000E+00						
E Xi^2 SO7	0.000E+00						
E Xi^2 AY3	9.143E+07						
E Xi^2 SY3	2.386E+07						
E Xi^2 SR1	0.000E+00						
E Xi^2 DR9	6.956E+05						
E Xi^2 SY2	1.152E+06						
E Xi^2 SG3	0.000E+00						
E Xi^2 SR24	0.000E+00						
E Xi^2 AN	0.000E+00						
E Xi^2 BZ	0.000E+00						

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Sum of the Corrected Results Squared						
(E Xi)^2 AB45	3.721E+08						
(E Xi)^2 AY23	4.684E+08						
(E Xi)^2 AY73	5.578E+08						
(E Xi)^2 AO10	3.154E+09						
(E Xi)^2 AB1	2.551E+09						
(E Xi)^2 BY2	6.416E+07						
(E Xi)^2 DV1	2.688E+07						
(E Xi)^2 SY33	2.045E+04						
(E Xi)^2 DB14	2.735E+05						
(E Xi)^2 AR64	5.485E+08						
(E Xi)^2 AB9	1.547E+09						
(E Xi)^2 BV10	4.410E+08						
(E Xi)^2 2AQ	2.451E+07						
(E Xi)^2 SO3	1.109E+08						
(E Xi)^2 1AQ	2.108E+07						
(E Xi)^2 SY14	0.000E+00						
(E Xi)^2 SO7	0.000E+00						
(E Xi)^2 AY3	4.597E+08						
(E Xi)^2 SY3	4.740E+07						
(E Xi)^2 SR1	0.000E+00						
(E Xi)^2 DR9	6.956E+05						
(E Xi)^2 SY2	2.295E+06						
(E Xi)^2 SG3	0.000E+00						
(E Xi)^2 SR24	0.000E+00						
(E Xi)^2 AN	0.000E+00						
(E Xi)^2 BZ	0.000E+00						

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Variance						
S^2: VARIANCE AB45	2703283.6						
S^2: VARIANCE AY23	974603.2						
S^2: VARIANCE AY73	8723572.0						
S^2: VARIANCE AO10	1253653.8						
S^2: VARIANCE AB1	239055.3						
S^2: VARIANCE BY2	2819433.3						
S^2: VARIANCE DV1	1655196.8						
S^2: VARIANCE SY33	2921.3						
S^2: VARIANCE DB14	18981.5						
S^2: VARIANCE AR64	27111.7						
S^2: VARIANCE AB9	541156.0						
S^2: VARIANCE BV10	746406.7						
S^2: VARIANCE 2AQ	1181091.9						
S^2: VARIANCE SO3	2515301.7						
S^2: VARIANCE 1AQ	1263730.1						
S^2: VARIANCE SY14	0.0						
S^2: VARIANCE SO7	0.0						
S^2: VARIANCE AY3	4294938.5						
S^2: VARIANCE SY3	2847765.8						
S^2: VARIANCE SR1	0.0						
S^2: VARIANCE DR9	99365.1						
S^2: VARIANCE SY2	137420.9						
S^2: VARIANCE SG3	0.0						
S^2: VARIANCE SR24	0.0						
S^2: VARIANCE AN	0.0						
S^2: VARIANCE BZ	0.0						

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Standard Deviation						
STD DEVIATION AB45	1644.2						
STD DEVIATION AY23	987.2						
STD DEVIATION AY73	2953.6						
STD DEVIATION AO10	1119.7						
STD DEVIATION AB1	488.9						
STD DEVIATION BY2	1679.1						
STD DEVIATION DV1	1286.5						
STD DEVIATION SY33	54.0						
STD DEVIATION DB14	137.8						
STD DEVIATION AR64	164.7						
STD DEVIATION AB9	735.6						
STD DEVIATION BV10	863.9						
STD DEVIATION 2AQ	1086.8						
STD DEVIATION SO3	1586.0						
STD DEVIATION 1AQ	1124.2						
STD DEVIATION SY14	0.0						
STD DEVIATION SO7	0.0						
STD DEVIATION AY3	2072.4						
STD DEVIATION SY3	1687.5						
STD DEVIATION SR1	0.0						
STD DEVIATION DR9	315.2						
STD DEVIATION SY2	370.7						
STD DEVIATION SG3	0.0						
STD DEVIATION SR24	0.0						
STD DEVIATION AN	0.0						
STD DEVIATION BZ	0.0						

	WHM001LS	WHM002LS	WHM003LS	WHM004LS	WHM005LS	WHM006LS	WHM007LS
	Method Detection Limit						
T (STUDENT'S T @ 99% FOR 6 DF)	3.140						
MDL AB45	5162.7						
MDL AY23	3099.9						
MDL AY73	9274.2						
MDL AO10	3515.8						
MDL AB1	1535.2						
MDL BY2	5272.4						
MDL DV1	4039.7						
MDL SY33	169.7						
MDL DB14	432.6						
MDL AR64	517.0						
MDL AB9	2309.9						
MDL BV10	2712.8						
MDL 2AQ	3412.5						
MDL SO3	4979.9						
MDL 1AQ	3529.9						
MDL SY14	0.0						
MDL SO7	0.0						
MDL AY3	6507.4						
MDL SY3	5298.9						
MDL SR1	0.0						
MDL DR9	989.8						
MDL SY2	1164.0						
MDL SG3	0.0						
MDL SR24	0.0						
MDL AN	0.0						
MDL BZ	0.0						

TABLE VII-12

High-Level Aqueous Extraction Method, Short Gradient, High Spike

	Sample Conc µg/L	Sample Results (µg/L)							Mean Result µg/L	Method Bias (Accuracy) %R	Standard Deviation	Method Precision (%RSD)	MDL µg/L	RQL µg/L
		WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS						
ACID BLUE 45	5510.4	4429.0	1381.0	0.0	0.0	2833.5	0.0	0.0	1234.8	22.4	1773.8	143.7	5569.9	22279.4
ACID YELLOW 23	4499.2	3153.0	3302.5	2965.5	2929.0	2520.5	2809.0	2875.0	2936.4	65.3	249.9	8.5	784.8	3139.2
ACID YELLOW 73	5616	6899.5	6714.5	6711.5	6822.0	6624.0	6555.0	6546.5	6696.1	119.2	132.5	2.0	415.9	1663.8
ACID ORANGE 10	4918.4	17712.5	16457.0	15991.5	16255.0	16491.5	16739.0	15897.0	16506.2	335.6	607.1	3.7	1906.2	7624.7
ACID BLUE 1	5907.2	9621.0	8507.5	8534.5	8910.5	8863.0	9080.0	9038.5	8936.4	151.3	376.7	4.2	1182.7	4730.9
BASIC YELLOW 2	4227.2	1220.0	0.0	0.0	0.0	0.0	0.0	251.0	210.1	5.0	455.0	216.5	1428.8	5715.1
DISPERSE VIOLET 1	4163.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT YELLOW 33	4473.6	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
DISPERSE BLUE 14	4784	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ACID RED 64	5788.8	4643.5	4600.5	3979.0	4229.0	4411.0	4144.0	4334.5	4334.5	74.9	240.1	5.5	753.9	3015.7
ACID BLUE 9	5248	8053.0	7397.5	6823.5	6810.5	7117.5	6684.0	5772.5	6951.2	132.5	699.7	10.1	2197.1	8788.5
BASIC VIOLET 10	3334.4	3603.0	2234.0	1989.0	2191.0	2371.5	2497.0	2381.5	2466.7	74.0	527.1	21.4	1655.0	6619.9
2-AMINOANTHRAQUINONE	5980.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT ORANGE 3	4092.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
1-AMINOANTHRAQUINONE	4112	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT YELLOW 14	4531.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT ORANGE 7	4608	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ACID YELLOW 3	5356.8	5160.5	2927.5	2239.5	2596.0	4297.0	2565.5	2528.0	3187.7	59.5	1100.1	34.5	3454.4	13817.6
SOLVENT YELLOW 3	4832	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT RED 1	7420.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
DISPERSE RED 9	4896	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT YELLOW 2	6304	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT GREEN 3	5024	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SOLVENT RED 24	908.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ANTHRACENE	6646.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
BENZANTHRONE	3891.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

TABLE VII - 12A

PERCENT RECOVERIES
HIGH-LEVEL AQUEOUS METHOD
HIGH SPIKE, SHORT GRADIENT

Compounds		Ret. Time	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
% RECOVERY AB45	%	4.439	80	25	0	0	51	0	0
% RECOVERY AR64	%	6.174	80	79	69	73	76	72	75
% RECOVERY AY3	%	7.322	96	55	42	48	80	48	47
% RECOVERY AY23	%	7.525	70	73	66	65	56	62	64
% RECOVERY AY73	%	9.159	123	120	120	121	118	117	117
% RECOVERY AO10	%	9.946	360	335	325	330	335	340	323
% RECOVERY AB9	%	11.576	153	141	130	130	136	127	110
% RECOVERY DV1	%	12.69	0	0	0	0	0	0	0
% RECOVERY AB1	%	12.709	163	144	144	151	150	154	153
% RECOVERY 2AQ	%	13.246	0	0	0	0	0	0	0
% RECOVERY SO3	%	13.811	0	0	0	0	0	0	0
% RECOVERY SY33	%	15.498	0	0	0	0	0	0	0
% RECOVERY 1AQ	%	16.108	0	0	0	0	0	0	0
% RECOVERY DB14	%	18.857	0	0	0	0	0	0	0
% RECOVERY BY2	%	19.452	29	0	0	0	0	0	6
% RECOVERY BV10	%	19.691	108	67	60	66	71	75	71
% RECOVERY SY3	%	19.962	0	0	0	0	0	0	0
% RECOVERY DR9	%	20.367	0	0	0	0	0	0	0
% RECOVERY BZ	%	20.438	0	0	0	0	0	0	0
% RECOVERY SY2	%	22.244	0	0	0	0	0	0	0
% RECOVERY AN	%	22.537	0	0	0	0	0	0	0
% RECOVERY SY14	%	24.403	0	0	0	0	0	0	0
% RECOVERY SR1	%	24.66	0	0	0	0	0	0	0
% RECOVERY SO7	%	27.804	0	0	0	0	0	0	0
% RECOVERY SG3	%	31.194	0	0	0	0	0	0	0
% RECOVERY SR24	%	31.255	0	0	0	0	0	0	0

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Stock Standard Concentrations						
ACID BLUE 45 STD	344.4	344.4	344.4	344.4	344.4	344.4	344.4
ACID YELLOW 23 STD	281.2	281.2	281.2	281.2	281.2	281.2	281.2
ACID YELLOW 73 STD	351.0	351.0	351.0	351.0	351.0	351.0	351.0
ACID ORANGE 10 STD	307.4	307.4	307.4	307.4	307.4	307.4	307.4
ACID BLUE 1 STD	369.2	369.2	369.2	369.2	369.2	369.2	369.2
BASIC YELLOW 2 STD	264.2	264.2	264.2	264.2	264.2	264.2	264.2
DISPERSE VIOLET 1 STD	260.2	260.2	260.2	260.2	260.2	260.2	260.2
SOLVENT YELLOW 33 STD	279.6	279.6	279.6	279.6	279.6	279.6	279.6
DISPERSE BLUE 14 STD	299.0	299.0	299.0	299.0	299.0	299.0	299.0
ACID RED 64 STD	361.8	361.8	361.8	361.8	361.8	361.8	361.8
ACID BLUE 9 STD	328.0	328.0	328.0	328.0	328.0	328.0	328.0
BASIC VIOLET 10 STD	208.4	208.4	208.4	208.4	208.4	208.4	208.4
2-AMINOANTHRAQUINONE STD	373.8	373.8	373.8	373.8	373.8	373.8	373.8
SOLVENT ORANGE 3 STD	255.8	255.8	255.8	255.8	255.8	255.8	255.8
1-AMINOANTHRAQUINONE STD	257.0	257.0	257.0	257.0	257.0	257.0	257.0
SOLVENT YELLOW 14 STD	283.2	283.2	283.2	283.2	283.2	283.2	283.2
SOLVENT ORANGE 7 STD	288.0	288.0	288.0	288.0	288.0	288.0	288.0
ACID YELLOW 3 STD	334.8	334.8	334.8	334.8	334.8	334.8	334.8
SOLVENT YELLOW 3 STD	302.0	302.0	302.0	302.0	302.0	302.0	302.0
SOLVENT RED 1 STD	463.8	463.8	463.8	463.8	463.8	463.8	463.8
DISPERSE RED 9 STD	306.0	306.0	306.0	306.0	306.0	306.0	306.0
SOLVENT YELLOW 2 STD	394.0	394.0	394.0	394.0	394.0	394.0	394.0
SOLVENT GREEN 3 STD	314.0	314.0	314.0	314.0	314.0	314.0	314.0
SOLVENT RED 24 STD	56.8	56.8	56.8	56.8	56.8	56.8	56.8
ANTHRACENE	415.4	415.4	415.4	415.4	415.4	415.4	415.4
BENZANTHRONE	243.2	243.2	243.2	243.2	243.2	243.2	243.2

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Weight of Stock Standard Spiked						
SPIKE VOLUME EACH DYE	4000.0	4000.0	4000.0	4000.0	4000.0	4000.0	4000.0
AB45 SPIKE WEIGHT	1377.6	1377.6	1377.6	1377.6	1377.6	1377.6	1377.6
AY23 SPIKE WEIGHT	1124.8	1124.8	1124.8	1124.8	1124.8	1124.8	1124.8
AY73 SPIKE WEIGHT	1404.0	1404.0	1404.0	1404.0	1404.0	1404.0	1404.0
AO10 SPIKE WEIGHT	1229.6	1229.6	1229.6	1229.6	1229.6	1229.6	1229.6
AB1 SPIKE WEIGHT	1476.8	1476.8	1476.8	1476.8	1476.8	1476.8	1476.8
BY2 SPIKE WEIGHT	1056.8	1056.8	1056.8	1056.8	1056.8	1056.8	1056.8
DV1 SPIKE WEIGHT	1040.8	1040.8	1040.8	1040.8	1040.8	1040.8	1040.8
SY33 SPIKE WEIGHT	1118.4	1118.4	1118.4	1118.4	1118.4	1118.4	1118.4
DB14 SPIKE WEIGHT	1196.0	1196.0	1196.0	1196.0	1196.0	1196.0	1196.0
AR64 SPIKE WEIGHT	1447.2	1447.2	1447.2	1447.2	1447.2	1447.2	1447.2
AB9 SPIKE WEIGHT	1312.0	1312.0	1312.0	1312.0	1312.0	1312.0	1312.0
BV10 SPIKE WEIGHT	833.6	833.6	833.6	833.6	833.6	833.6	833.6
2AQ SPIKE WEIGHT	1495.2	1495.2	1495.2	1495.2	1495.2	1495.2	1495.2
SO3 SPIKE WEIGHT	1023.2	1023.2	1023.2	1023.2	1023.2	1023.2	1023.2
1AQ SPIKE WEIGHT	1028.0	1028.0	1028.0	1028.0	1028.0	1028.0	1028.0
SY14 SPIKE WEIGHT	1132.8	1132.8	1132.8	1132.8	1132.8	1132.8	1132.8
SO7 SPIKE WEIGHT	1152.0	1152.0	1152.0	1152.0	1152.0	1152.0	1152.0
AY3 SPIKE WEIGHT	1339.2	1339.2	1339.2	1339.2	1339.2	1339.2	1339.2
SY3 SPIKE WEIGHT	1208.0	1208.0	1208.0	1208.0	1208.0	1208.0	1208.0
SR1 SPIKE WEIGHT	1855.2	1855.2	1855.2	1855.2	1855.2	1855.2	1855.2
DR9 SPIKE WEIGHT	1224.0	1224.0	1224.0	1224.0	1224.0	1224.0	1224.0
SY2 SPIKE WEIGHT	1576.0	1576.0	1576.0	1576.0	1576.0	1576.0	1576.0
SG3 SPIKE WEIGHT	1256.0	1256.0	1256.0	1256.0	1256.0	1256.0	1256.0
SR24 SPIKE WEIGHT	227.2	227.2	227.2	227.2	227.2	227.2	227.2
AN SPIKE WEIGHT	1661.6	1661.6	1661.6	1661.6	1661.6	1661.6	1661.6
BZ SPIKE WEIGHT	972.8	972.8	972.8	972.8	972.8	972.8	972.8

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Weight Standard per Volume Sample						
MATRIX VOLUME (WATER)	0.250	0.250	0.250	0.250	0.250	0.250	0.250
SPIKE CONC AB45	5510.4	5510.4	5510.4	5510.4	5510.4	5510.4	5510.4
SPIKE CONC AY23	4499.2	4499.2	4499.2	4499.2	4499.2	4499.2	4499.2
SPIKE CONC AY73	5616.0	5616.0	5616.0	5616.0	5616.0	5616.0	5616.0
SPIKE CONC AO10	4918.4	4918.4	4918.4	4918.4	4918.4	4918.4	4918.4
SPIKE CONC AB1	5907.2	5907.2	5907.2	5907.2	5907.2	5907.2	5907.2
SPIKE CONC BY2	4227.2	4227.2	4227.2	4227.2	4227.2	4227.2	4227.2
SPIKE CONC DV1	4163.2	4163.2	4163.2	4163.2	4163.2	4163.2	4163.2
SPIKE CONC SY33	4473.6	4473.6	4473.6	4473.6	4473.6	4473.6	4473.6
SPIKE CONC DB14	4784.0	4784.0	4784.0	4784.0	4784.0	4784.0	4784.0
SPIKE CONC AR64	5788.8	5788.8	5788.8	5788.8	5788.8	5788.8	5788.8
SPIKE CONC AB9	5248.0	5248.0	5248.0	5248.0	5248.0	5248.0	5248.0
SPIKE CONC BV10	3334.4	3334.4	3334.4	3334.4	3334.4	3334.4	3334.4
SPIKE CONC 2AQ	5980.8	5980.8	5980.8	5980.8	5980.8	5980.8	5980.8
SPIKE CONC SO3	4092.8	4092.8	4092.8	4092.8	4092.8	4092.8	4092.8
SPIKE CONC 1AQ	4112.0	4112.0	4112.0	4112.0	4112.0	4112.0	4112.0
SPIKE CONC SY14	4531.2	4531.2	4531.2	4531.2	4531.2	4531.2	4531.2
SPIKE CONC SO7	4608.0	4608.0	4608.0	4608.0	4608.0	4608.0	4608.0
SPIKE CONC AY3	5356.8	5356.8	5356.8	5356.8	5356.8	5356.8	5356.8
SPIKE CONC SY3	4832.0	4832.0	4832.0	4832.0	4832.0	4832.0	4832.0
SPIKE CONC SR1	7420.8	7420.8	7420.8	7420.8	7420.8	7420.8	7420.8
SPIKE CONC DR9	4896.0	4896.0	4896.0	4896.0	4896.0	4896.0	4896.0
SPIKE CONC SY2	6304.0	6304.0	6304.0	6304.0	6304.0	6304.0	6304.0
SPIKE CONC SG3	5024.0	5024.0	5024.0	5024.0	5024.0	5024.0	5024.0
SPIKE CONC SR24	908.8	908.8	908.8	908.8	908.8	908.8	908.8
SPIKE CONC AN	6646.4	6646.4	6646.4	6646.4	6646.4	6646.4	6646.4
SPIKE CONC BZ	3891.2	3891.2	3891.2	3891.2	3891.2	3891.2	3891.2

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
SPIKE CONC AB45	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY23	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY73	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AO10	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BY2	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DV1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY33	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DB14	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AR64	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AB9	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BV10	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 2AQ	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC 1AQ	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY14	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SO7	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AY3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR1	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC DR9	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SY2	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SG3	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC SR24	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC AN	NA	NA	NA	NA	NA	NA	NA
SPIKE CONC BZ	NA	NA	NA	NA	NA	NA	NA

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Results from HPLC						
HPLC RESULT AB45	8.858	2.762	0.000	0.000	5.667	0.000	0.000
HPLC RESULT AY23	6.306	6.605	5.931	5.858	5.041	5.618	5.750
HPLC RESULT AY73	13.799	13.429	13.423	13.644	13.248	13.110	13.093
HPLC RESULT AO10	35.425	32.914	31.983	32.510	32.983	33.478	31.794
HPLC RESULT AB1	19.242	17.015	17.069	17.821	17.726	18.160	18.077
HPLC RESULT BY2	2.440	0.000	0.000	0.000	0.000	0.000	0.502
HPLC RESULT DV1	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SY33	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT DB14	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT AR64	9.287	9.201	7.958	8.458	8.822	8.288	8.669
HPLC RESULT AB9	16.106	14.795	13.647	13.621	14.235	13.368	11.545
HPLC RESULT BV10	7.206	4.468	3.978	4.382	4.743	4.994	4.763
HPLC RESULT 2AQ	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SO3	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT 1AQ	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SY14	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SO7	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT AY3	10.321	5.855	4.479	5.192	8.594	5.131	5.056
HPLC RESULT SY3	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SR1	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT DR9	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SY2	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SG3	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT SR24	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT AN	0.000	0.000	0.000	0.000	0.000	0.000	0.000
HPLC RESULT BZ	0.000	0.000	0.000	0.000	0.000	0.000	0.000

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Final Sample Results						
CF (STD INJ VOL VS SAM INJ VOL)	2	2	2	2	2	2	2
FINAL VOLUME	250.000	250.000	250.000	250.000	250.000	250.000	250.000
CORRECTED RESULT AB45	4429.00	1381.00	0.00	0.00	2833.50	0.00	0.00
CORRECTED RESULT AY23	3153.00	3302.50	2965.50	2929.00	2520.50	2809.00	2875.00
CORRECTED RESULT AY73	6899.50	6714.50	6711.50	6822.00	6624.00	6555.00	6546.50
CORRECTED RESULT AO10	17712.50	16457.00	15991.50	16255.00	16491.50	16739.00	15897.00
CORRECTED RESULT AB1	9621.00	8507.50	8534.50	8910.50	8863.00	9080.00	9038.50
CORRECTED RESULT BY2	1220.00	0.00	0.00	0.00	0.00	0.00	251.00
CORRECTED RESULT DV1	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SY33	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT DB14	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT AR64	4643.50	4600.50	3979.00	4229.00	4411.00	4144.00	4334.50
CORRECTED RESULT AB9	8053.00	7397.50	6823.50	6810.50	7117.50	6684.00	5772.50
CORRECTED RESULT BV10	3603.00	2234.00	1989.00	2191.00	2371.50	2497.00	2381.50
CORRECTED RESULT 2AQ	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SO3	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT 1AQ	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SY14	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SO7	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT AY3	5160.50	2927.50	2239.50	2596.00	4297.00	2565.50	2528.00
CORRECTED RESULT SY3	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SR1	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT DR9	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SY2	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SG3	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT SR24	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT AN	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CORRECTED RESULT BZ	0.00	0.00	0.00	0.00	0.00	0.00	0.00

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Percent Recoveries						
% RECOVERY AB45	80.38	25.06	0.00	0.00	51.42	0.00	0.00
% RECOVERY AY23	70.1	73.4	65.9	65.1	56.0	62.4	63.9
% RECOVERY AY73	122.9	119.6	119.5	121.5	117.9	116.7	116.6
% RECOVERY AO10	360.1	334.6	325.1	330.5	335.3	340.3	323.2
% RECOVERY AB1	162.9	144.0	144.5	150.8	150.0	153.7	153.0
% RECOVERY BY2	28.9	0.0	0.0	0.0	0.0	0.0	5.9
% RECOVERY DV1	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SY33	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY DB14	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY AR64	80.2	79.5	68.7	73.1	76.2	71.6	74.9
% RECOVERY AB9	153.4	141.0	130.0	129.8	135.6	127.4	110.0
% RECOVERY BV10	108.1	67.0	59.7	65.7	71.1	74.9	71.4
% RECOVERY 2AQ	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SO3	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY 1AQ	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SY14	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SO7	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY AY3	96.3	54.7	41.8	48.5	80.2	47.9	47.2
% RECOVERY SY3	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SR1	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY DR9	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SY2	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SG3	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY SR24	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY AN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
% RECOVERY BZ	0.0	0.0	0.0	0.0	0.0	0.0	0.0

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Square of Corrected Results						
N	7	7	7	7	7	7	7
Xi^2 AB45	19616041.0	1907161.0	0.0	0.0	8028722.3	0.0	0.0
Xi^2 AY23	9941409.0	10906506.3	8794190.3	8579041.0	6352920.3	7890481.0	8265625.0
Xi^2 AY73	47603100.3	45084510.3	45044232.3	46539684.0	43877376.0	42968025.0	42856662.3
Xi^2 AO10	3.137E+08	2.708E+08	2.557E+08	2.642E+08	2.720E+08	2.802E+08	2.527E+08
Xi^2 AB1	92563641.0	72377556.3	72837690.3	79397010.3	78552769.0	82446400.0	81694482.3
Xi^2 BY2	1488400.0	0.0	0.0	0.0	0.0	0.0	63001.0
Xi^2 DV1	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SY33	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 DB14	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 AR64	21562092.3	21164600.3	15832441.0	17884441.0	19456921.0	17172736.0	18787890.3
Xi^2 AB9	64850809.0	54723006.3	46560152.3	46382910.3	50658806.3	44675856.0	33321756.3
Xi^2 BV10	12981609.0	4990756.0	3956121.0	4800481.0	5624012.3	6235009.0	5671542.3
Xi^2 2AQ	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SO3	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 1AQ	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SY14	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SO7	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 AY3	26630760.3	8570256.3	5015360.3	6739216.0	18464209.0	6581790.3	6390784.0
Xi^2 SY3	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SR1	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 DR9	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SY2	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SG3	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 SR24	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 AN	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Xi^2 BZ	0.0	0.0	0.0	0.0	0.0	0.0	0.0

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Sum of the Square of Corrected Results						
E Xi^2 AB45	2.955E+07						
E Xi^2 AY23	6.073E+07						
E Xi^2 AY73	3.140E+08						
E Xi^2 AO10	1.909E+09						
E Xi^2 AB1	5.599E+08						
E Xi^2 BY2	1.551E+06						
E Xi^2 DV1	0.000E+00						
E Xi^2 SY33	0.000E+00						
E Xi^2 DB14	0.000E+00						
E Xi^2 AR64	1.319E+08						
E Xi^2 AB9	3.412E+08						
E Xi^2 BV10	4.426E+07						
E Xi^2 2AQ	0.000E+00						
E Xi^2 SO3	0.000E+00						
E Xi^2 1AQ	0.000E+00						
E Xi^2 SY14	0.000E+00						
E Xi^2 SO7	0.000E+00						
E Xi^2 AY3	7.839E+07						
E Xi^2 SY3	0.000E+00						
E Xi^2 SR1	0.000E+00						
E Xi^2 DR9	0.000E+00						
E Xi^2 SY2	0.000E+00						
E Xi^2 SG3	0.000E+00						
E Xi^2 SR24	0.000E+00						
E Xi^2 AN	0.000E+00						
E Xi^2 BZ	0.000E+00						

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Sum of the Corrected Results Squared						
(E Xi)^2 AB45	7.471E+07						
(E Xi)^2 AY23	4.225E+08						
(E Xi)^2 AY73	2.197E+09						
(E Xi)^2 AO10	1.335E+10						
(E Xi)^2 AB1	3.913E+09						
(E Xi)^2 BY2	2.164E+06						
(E Xi)^2 DV1	0.000E+00						
(E Xi)^2 SY33	0.000E+00						
(E Xi)^2 DB14	0.000E+00						
(E Xi)^2 AR64	9.206E+08						
(E Xi)^2 AB9	2.368E+09						
(E Xi)^2 BV10	2.981E+08						
(E Xi)^2 2AQ	0.000E+00						
(E Xi)^2 SO3	0.000E+00						
(E Xi)^2 1AQ	0.000E+00						
(E Xi)^2 SY14	0.000E+00						
(E Xi)^2 SO7	0.000E+00						
(E Xi)^2 AY3	4.979E+08						
(E Xi)^2 SY3	0.000E+00						
(E Xi)^2 SR1	0.000E+00						
(E Xi)^2 DR9	0.000E+00						
(E Xi)^2 SY2	0.000E+00						
(E Xi)^2 SG3	0.000E+00						
(E Xi)^2 SR24	0.000E+00						
(E Xi)^2 AN	0.000E+00						
(E Xi)^2 BZ	0.000E+00						

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Variance						
S^2: VARIANCE AB45	3146509.0						
S^2: VARIANCE AY23	62470.0						
S^2: VARIANCE AY73	17547.6						
S^2: VARIANCE AO10	368522.4						
S^2: VARIANCE AB1	141876.6						
S^2: VARIANCE BY2	207046.8						
S^2: VARIANCE DV1	0.0						
S^2: VARIANCE SY33	0.0						
S^2: VARIANCE DB14	0.0						
S^2: VARIANCE AR64	57648.3						
S^2: VARIANCE AB9	489606.0						
S^2: VARIANCE BV10	277795.8						
S^2: VARIANCE 2AQ	0.0						
S^2: VARIANCE SO3	0.0						
S^2: VARIANCE 1AQ	0.0						
S^2: VARIANCE SY14	0.0						
S^2: VARIANCE SO7	0.0						
S^2: VARIANCE AY3	1210286.6						
S^2: VARIANCE SY3	0.0						
S^2: VARIANCE SR1	0.0						
S^2: VARIANCE DR9	0.0						
S^2: VARIANCE SY2	0.0						
S^2: VARIANCE SG3	0.0						
S^2: VARIANCE SR24	0.0						
S^2: VARIANCE AN	0.0						
S^2: VARIANCE BZ	0.0						

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Standard Deviation						
STD DEVIATION AB45	1773.8						
STD DEVIATION AY23	249.9						
STD DEVIATION AY73	132.5						
STD DEVIATION AO10	607.1						
STD DEVIATION AB1	376.7						
STD DEVIATION BY2	455.0						
STD DEVIATION DV1	0.0						
STD DEVIATION SY33	0.0						
STD DEVIATION DB14	0.0						
STD DEVIATION AR64	240.1						
STD DEVIATION AB9	699.7						
STD DEVIATION BV10	527.1						
STD DEVIATION 2AQ	0.0						
STD DEVIATION SO3	0.0						
STD DEVIATION 1AQ	0.0						
STD DEVIATION SY14	0.0						
STD DEVIATION SO7	0.0						
STD DEVIATION AY3	1100.1						
STD DEVIATION SY3	0.0						
STD DEVIATION SR1	0.0						
STD DEVIATION DR9	0.0						
STD DEVIATION SY2	0.0						
STD DEVIATION SG3	0.0						
STD DEVIATION SR24	0.0						
STD DEVIATION AN	0.0						
STD DEVIATION BZ	0.0						

	WHM001HS	WHM002HS	WHM003HS	WHM004HS	WHM005HS	WHM006HS	WHM007HS
	Method Detection Limit						
T (STUDENT'S T @ 99% FOR 6 DF)	3.140						
MDL AB45	5569.9						
MDL AY23	784.8						
MDL AY73	415.9						
MDL AO10	1906.2						
MDL AB1	1182.7						
MDL BY2	1428.8						
MDL DV1	0.0						
MDL SY33	0.0						
MDL DB14	0.0						
MDL AR64	753.9						
MDL AB9	2197.1						
MDL BV10	1655.0						
MDL 2AQ	0.0						
MDL SO3	0.0						
MDL 1AQ	0.0						
MDL SY14	0.0						
MDL SO7	0.0						
MDL AY3	3454.4						
MDL SY3	0.0						
MDL SR1	0.0						
MDL DR9	0.0						
MDL SY2	0.0						
MDL SG3	0.0						
MDL SR24	0.0						
MDL AN	0.0						
MDL BZ	0.0						

Project No.

F. High Method - Water MDL

Book No.

n Page No. High

samples total - spike 4 mL of each stock standard dye into 250 mL volumetric flask. (Used standards on pg 133 except for Sample #7 - ran out of DR9, SY2, + SG3; made fresh stock standards). Evaporated methanol under a stream of nitrogen. Diluted to 250 mL with well water.

Note - Sample #2 - AB1 will have spiking error.

New Stock Standard	Weight		µg/mL
SG3	15.7 mg	in 50 mL =	314.0
SY2	19.7 mg		394.0
DR9	15.3 mg		306.0

~~Took 5 mL of each High sample. Diluted to 10 mL~~

~~Stored in refrigerator over weekend~~

~~12/2000 GK~~

To Page No.

essed & Understood by me, | Date | Invented by | Date

Book No._____ TITLE Soil High Spike MDL

From Page No.__

Sample #	Soil Wt (g)	Vol. Spiked (µL)
1	10.0881	500
2	10.1335	500
3	10.0871	500
4	10.0276	500
5	10.2040	500
6	10.5035	500
7	10.0716	500
Blank	10.0099	N/A

Cleaned volumetric flasks 3 times with methanol. Dried under a gentle stream of nitrogen. Weighed approximately 10 grams of dry, meshed soil from dye burial grounds onto a plastic weighing boat. Transferred to volumetric flask. Spiked on 500 µL of each dye from the stock standards. Dried soil under nitrogen after every 5th dye to prevent dye from getting onto sides of volumet

6/5/2000 GK

To Page No.

Witnessed & Understood by me, | Date | Invented by | Date

LE Soil - Low Spike MDL

Project No.

Book No.

om Page No.

Sample #	Soil Wt (g)	Vol. Spiked (µL)
1	10.0946	100
2	10.0175	100
3	10.0186	100
4	10.0155	100
5	10.0428	100
6	10.0303	100
7	10.0841	100

leaned volumetric flasks 3 times with methanol. Dried under gentle stream of nitrogen. Weighed approximately 10 grams of ry mesked soil from dye burial grounds onto a plastic weighing eet. Transferred to volumetric flask. Spiked on 100µL each ye from the stock standards. Dried soil under nitrogen to revent dye from getting onto sides of volumetric

2/5/2000 JK

ssed & Understood by me,

Date

Invented by

Date

To Page No.

BOOK NO._____ TITLE Water - OOW 1164Hoc1

From Page No.__

Sample	Amt (uL) each dye	Date Spiked	Date Ext	Date Analyzed
WLM001 HS	500	6/19/00	6/19/00	No TBA
WLM002 HS	500	6/19/00	6/19/00	No TBA
WLM003 HS	500	6/19/00	6-19-00	No TBA
WLM004 HS	500	6/19/00	6-21-00	+ 5mL TBA (evap down
WLM005 HS	500	6/19/00	6-20-00	No TBA
WLM006 HS	500	6-20-00	6-20-00	5mL Aldrich TBA
WLM007 HS	500	6-20-00	6-21-00	5mL Aldrich TBA
WLM008 HS	500	6-21-00	6-21-00	5mL Aldrich TBA
WLM009 HS	500	6-21-00	6-22-00	5mL Aldrich TBA
WLM010 HS	500	6-21-00	6-22-00	5mL Aldrich TBA
WLM011 HS	500	6-22-00	6-23-00	5mL Aldrich TBA
WLM012 LS	100	6-22-00	6-23-00	5mL Aldrich TBA

$300 \frac{\mu g}{mL} \times 0.5 = \frac{150 \mu g}{5 mL} = 30 \frac{\mu g}{mL}$

Add 5mL TBA to sample prior to extraction

Empore (3M) High performance oil & grease extraction disks — 47mm - Lot 470150

Pretreatment - 50mL methanol - rinse sides of apparatus - pull thru to waste. 50mL methanol - rinse side of apparatus. Pull thru to collection flask. Rinse flask + dispose of methanol.

Add all of the sample
Start: 10:10AM End: 10:45A

Dry 5 minutes on vacuum

Extract w/ 30 mL methanol + 20mL rinse

To Page No.

Witnessed & Understood by me,	Date	Invented by	Date

LE _____ Project No. _____

Book No. _____

m Page No. __

_MC03HS + LMC004HS - heavily particulated

Sample	Amt (µL) each dye	Date Sealed	Date Ext
-M002LS	100	6-24-00	6-24-00
LM003LS	100	6-24-00	6-24-00
-M004LS	100	6-24-00	6-24-00
LM005LS	100	6-24-00	6-26-00
-M006 LS	100	6-26-00	6-26-00
-M007M_BLNK	0	N/A	7-6-00
M008 LS	100	7-7-00	7-7-00

From Page No.

Spiked 200 µL of each dye into water from DBG wells.
Evaporated under nitrogen until most of the methanol evapo
Diluted to 25 mL with DBG well water.

To Page No

Witnessed & Understood by me,	Date	Invented by	Date

ATTACHMENT VIII

INTERFERENCES

ATTACHMENT VIII – INTERFERENCES

No major interferences were noted during the analysis of soil and groundwater samples from the Crane site. Minor interferences present eluted during the first -10 minutes of the analytical run and did not adversely affect sample quantitation. Therefore, no clean-up procedures were developed for the dye analysis.

The analyst also noted that dye compounds absorb mainly in the region above 600 nm. Few other organic compounds absorb in this range. This will also be an important tool in discriminating between interferences and the presence of unknown dye compounds.

ATTACHMENT IX

SITE-SPECIFIC ANALYSES

ATTACHMENT IX – SITE-SPECIFIC ANALYSES

During the capping phase of the remediation effort at Crane, soil samples visibly contaminated with dye compounds were collected opportunistically by field investigators. The Crane Laboratory analyzed the samples using early extraction and analytical procedures in order to identify dyes present at the site. The laboratory was able to successfully identify several dyes by matching their absorbance spectra with the absorbance spectra of known standards. Dyes which could not be identified were added to the spectral library as unknown dyes.

To: Karen Smecker
From: Jacquelyn Karty
Subject: Dye Burial Grounds

Karen,

Enclosed are the results from our analysis of Tom Brent's samples. I included the chromatograms and W-vis spectra.

In the results table, when I refer to a dye as being "unknown," it means we got a strong W-vis spectra and it did not match any of our in-house dye samples. When I refer to a dye as being "unidentifiable," it means we did not get a strong W-vis spectra and therefore were not able to match it to either an in-house sample or one of the unknowns in the other samples.

If you have any questions, please feel free to call me at 812-85X-3143.

Sincerely,
Jacquelyn Karty.

Extraction

Some samples appeared to be close to pure dye. Approximately 20 mg of the sample was weighed out and dissolved in a suitable solvent. No particulates appeared to be visable after the dye dissolved so it was assumed everything went into solution.

Samples DBGS- 2.3. 5. 6. 7. 9, & 10 were heavily contaminated with soil. The samples were dried in an oven at approximately 50°C for approximately 2 hours. The sample was then ground in a mortar and pestle. 2 grams of sample was weighed into a screw cap test tube. 10 mL of HPLC grade water was added. The sample was sonicated approximately 2 hours. The water was poured off into a PTFE capped bottle for storage leaving as much of the soil behind in the test tube as possible. The procedure was repeated with HPLC grade methanol and acetonitrile. The samples were then centrifuged until the majority of particulates fell out of solution. The sample volume was reduced under a gentle stream of nitrogen to approximately 4 mL. The samples were filtered through 0.45 μ nylon syringe filters prior to injection on the HPLC.

Instrumental Conditions

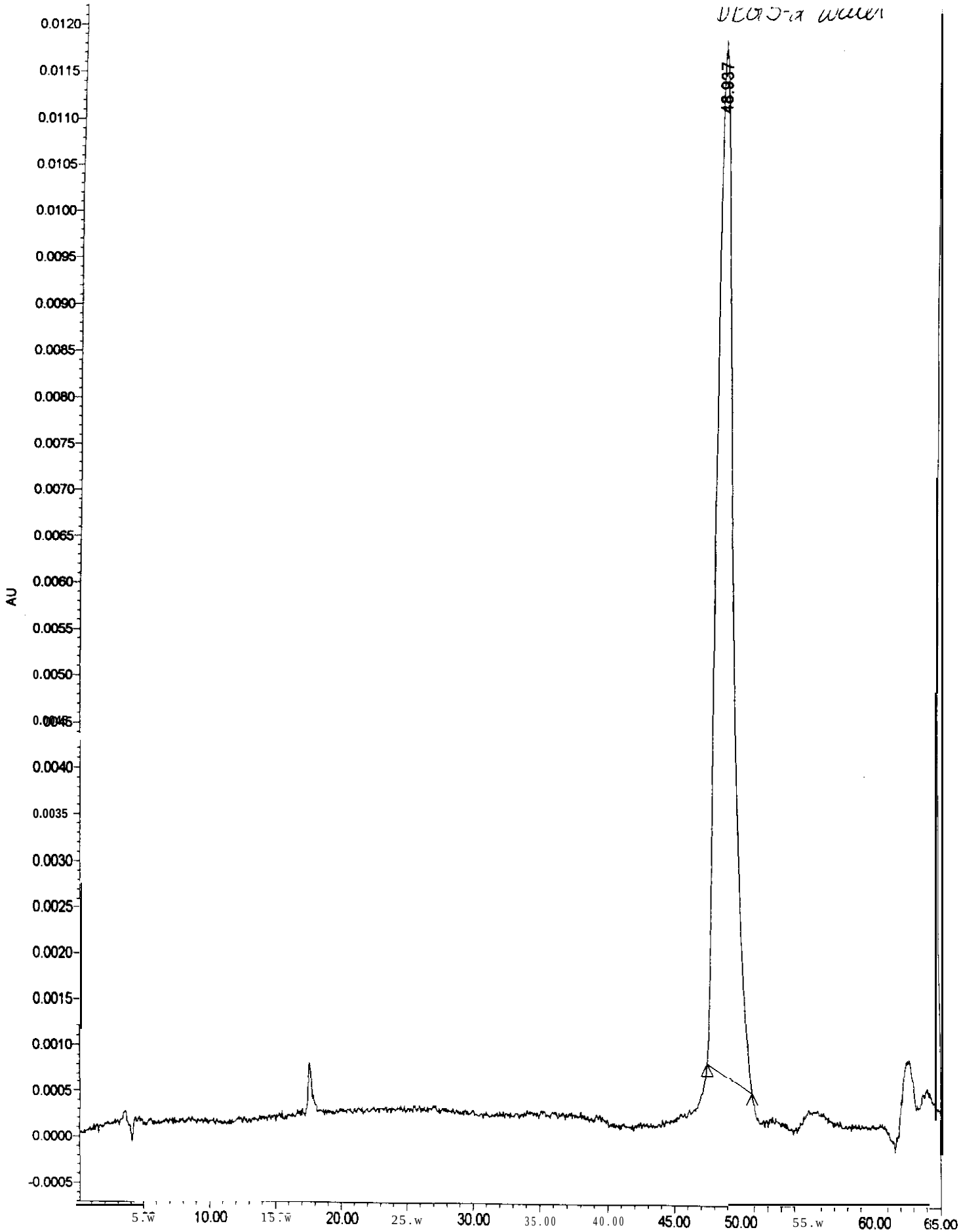
All analyses were performed on the following system: Waters 600E multi-solvent pump, Waters 717 autosampler, and Waters 996 photodiode array detector. Mobile phase was 60:40 acetonitrile:20 mM KH_2PO_4 pH=3.4. Flow rate was 1 .O mL/min on a Alltech C8 column (10 μ pore size, 4.6 x 250 mm). The UV-vis spectrum was scanned from 210.1 to 700 nm.

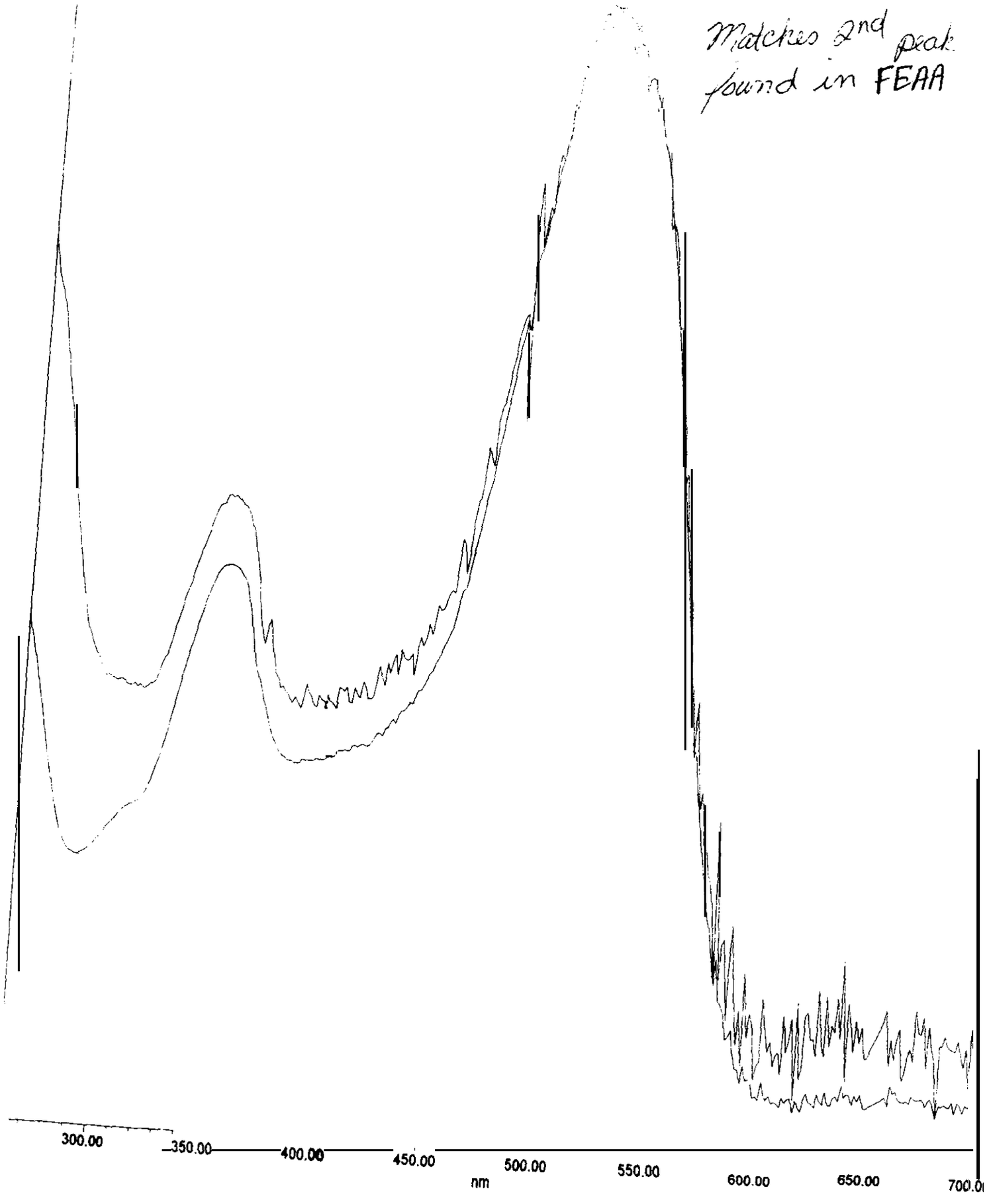
Results

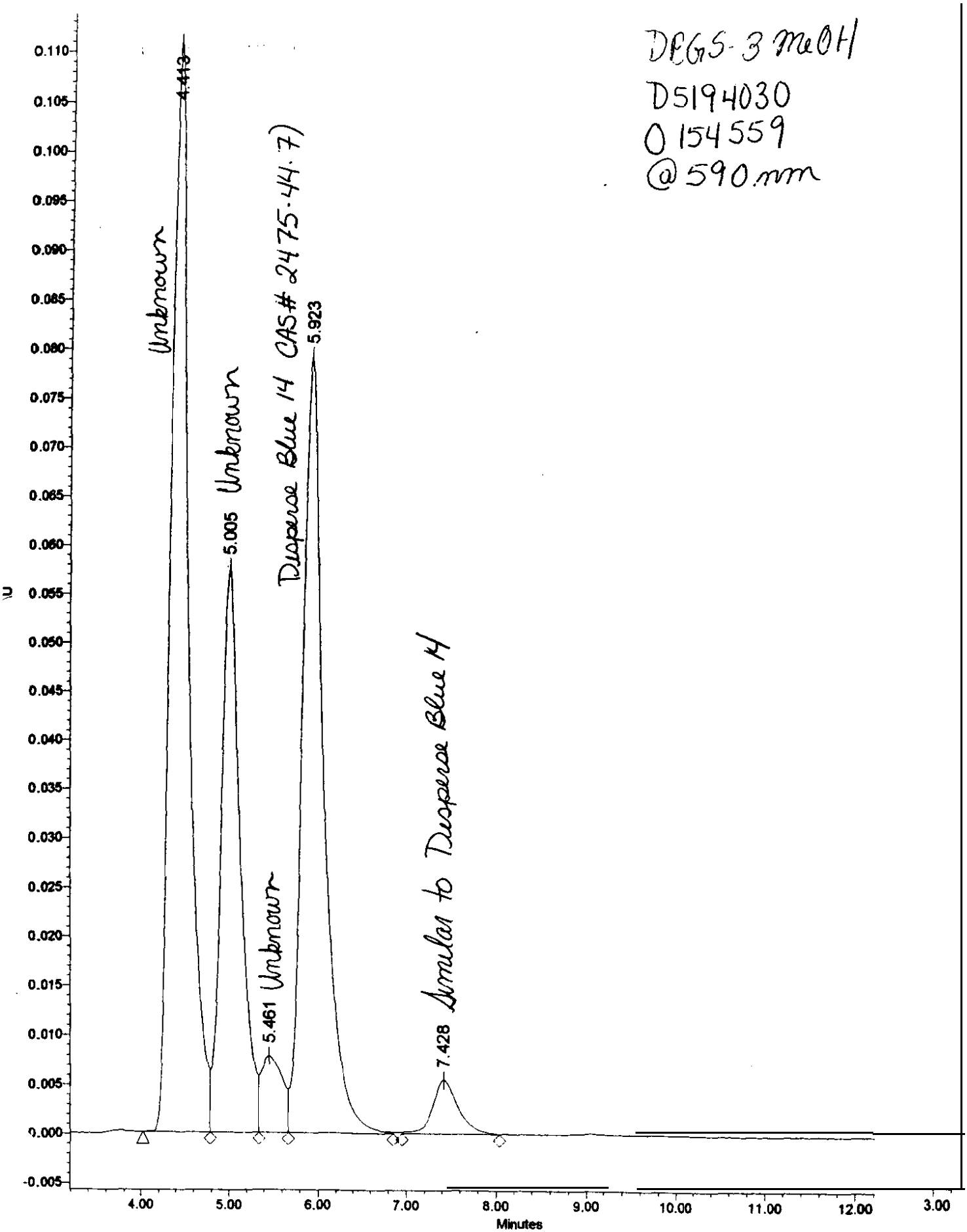
Results are listed in the table below. No dyes were detected in samples DBGS-6, and 10. Sample DBGS-19 was not soluble in water, methanol, ethanol, tetrahydrofuran. isopropanol, acetonitrile. pentane, dichlommethane, ethyl ether, acetone, toluene, ethyl acetate, or benzene. We are currently investigating other solvents. Sample DBGS-9 was accidently skipped during analysis. It will be run in the near future.

Laboratory Code	Lot #	Serial #	Description of Jar	Results
DBGS-1	D 5194030	O 154577	5S10	Dye visible. Soluble only in methanol. Could not get to elute off column.
DBGS-2	D 5194030	O 154578	1CS17	FEAA - peak 2
DBGS-3	D 5194030	0 154559	P D	Unknown #1; Unknown #2; Unknown #3; Disperse Blue 14; Unknown #4; Vat Yellow 4; Benzanthrone; Unidentifiable #5
DBGS-4	D 5194030	0 154561	Dk Purple, Very Blueberry 5ES	Unknown #6; Unidentifiable #7; Unknown #8; Disperse Red 9 additive; Unknown #9; Solvent Orange 7; Unknown #12
DBGS-5	D 5194030	O 154539	Purpleish blue, "Fair Skies"	Unknown #11; Unknown #12; Disperse Red 9 additive; Unidentifiable #13; Solvent Orange 7; MILD-16637 peak #3
DBGS-6	D 5194030	0 154556	LG	No dye detected
DBGS-7	D 5194030	0 154565	Blue 21; Eagle Picher label "241 blue hue 08/07/96 1611 Sampling site L49 96RW01 535 grab"	Unknown #13
DBGS-8	D 5194030	O 154574	1.51 Yellow; Eagle Picher label "174 Yellow 08/07/96 1611 Sampling site L49 96RW01536 grab"	Sample appears (by sight) to be mostly dye. Only slightly soluble in DMSO. Not able to detect on HPLC. Eiiher did not elute or below detection limits @ the injection concentration
DBGS-9	D 5194030	O 154568	LS	

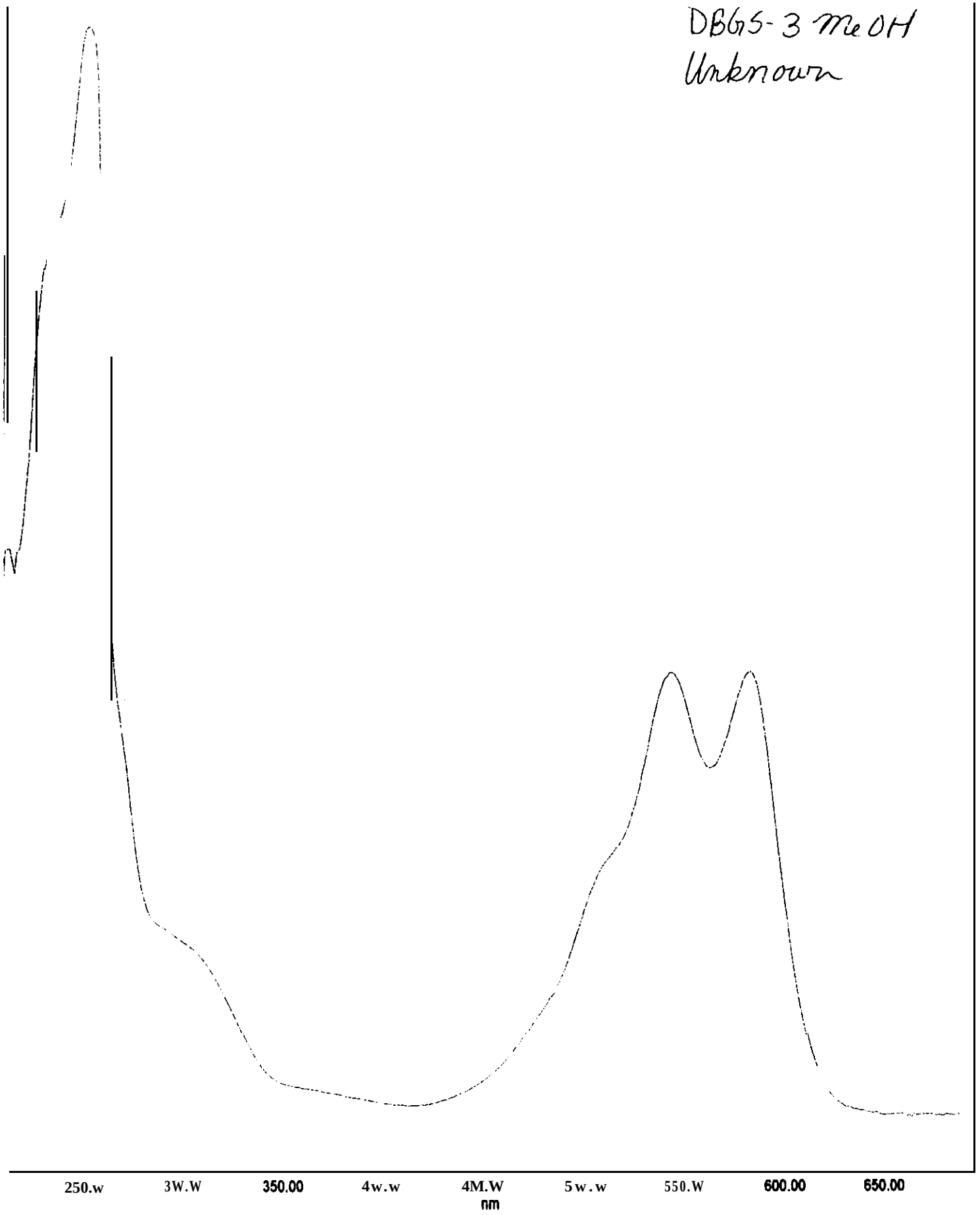
DBGS-10	D 5194030	O 154571	1CS18	No dye detected
DBGS-11	none	none	Collected 1100 on 6/27/96 SWMU 02/11 Southwest Trench #1	Unknown #20
DBGS-12	F 5150040	N 354803	9	Accramine Hydrochloride Unidentifiable #14
DBGS-13	F 5150040	N 354821	SWMU 02/11 6/27/96 1515 Southwest of #3 trench in roadway	FEAA
DBGS-14	F 5150040	N 354811	3	Disperse Red 9; Vat Yellow 4; Unidentifiable #15
DBGS-15	F 5150040	N 354824	7/10/96 Blue dye? DBG	Disperse Blue 14
DBGS-16	F 5150040	N 354809	1 SWMU 02/11 1645 6/26/96 N.W. #13 40'	Unknown #16; Unknown #17; Solvent Orange 7
DBGS-17	F 5150040	N 354805	2	Unknown #16; Unknown #17; Benzanthrone; Solvent Orange 7; Vat Yellow 4; Unidentifiable #18
DBGS-18	F 5150040	N 354806	6/29/96 dye from SWMU 02/11 1000	Unknown #1; Unknown #19; Disperse Blue 14
DBGS-19	D 5194030	O 154557	Packing; Eagle Picher label "Packing material 08/07/96 1611 L49 96RW01536 grab"	Could not get "dye" on packing material to dissolve. Continuing with effort.

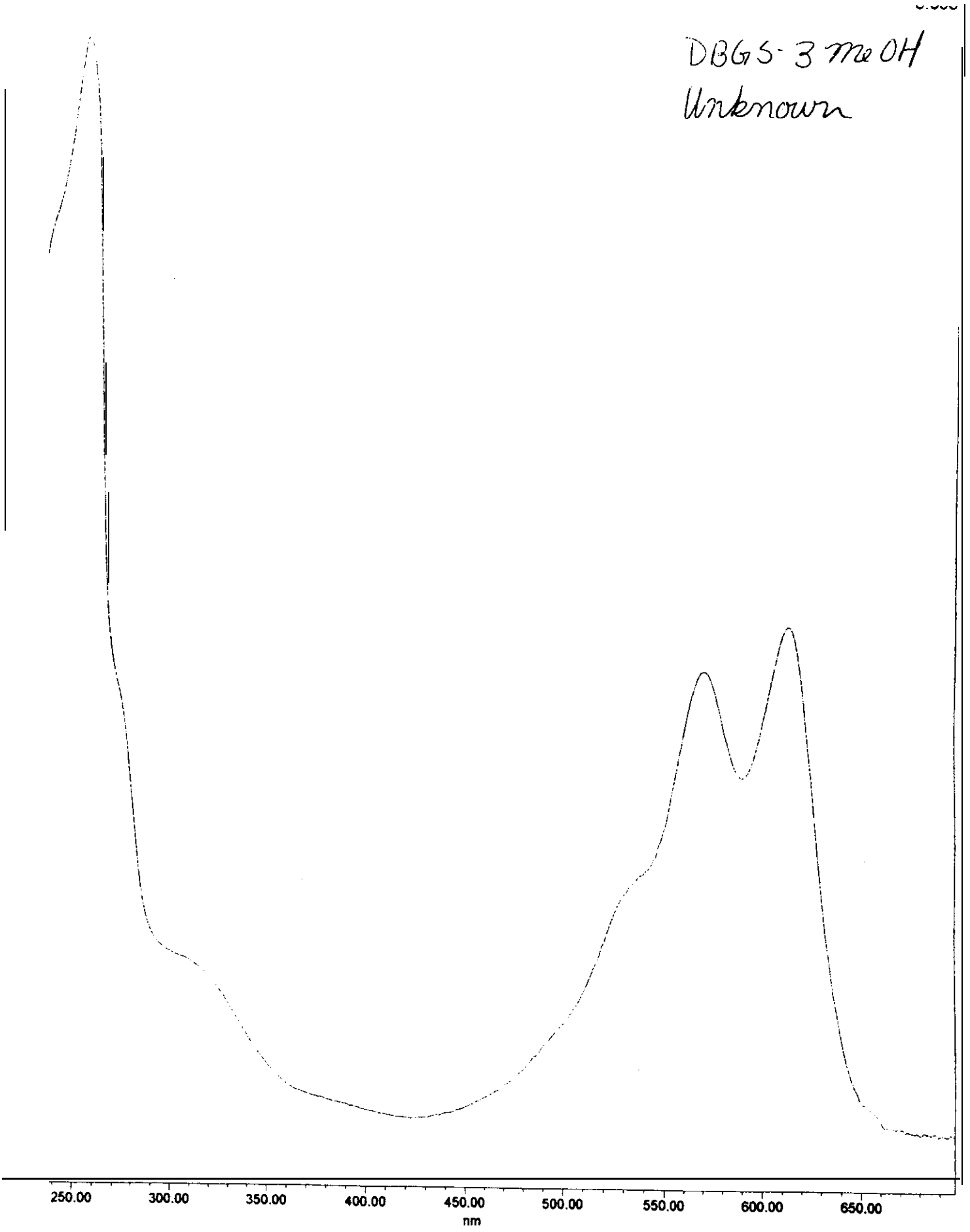

48.007
AU
0.0120
0.0115
0.0110
0.0105
0.0100
0.0095
0.0090
0.0085
0.0080
0.0075
0.0070
0.0065
0.0060
0.0055
0.0050
0.0045
0.0040
0.0035
0.0030
0.0025
0.0020
0.0015
0.0010
0.0005
0.0000
-0.0005
5.00
10.00
15.00
20.00
25.00
30.00
35.00
40.00
45.00
50.00
55.00
60.00
65.00


Matches 2nd peak found in FEAA
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
700.00
nm

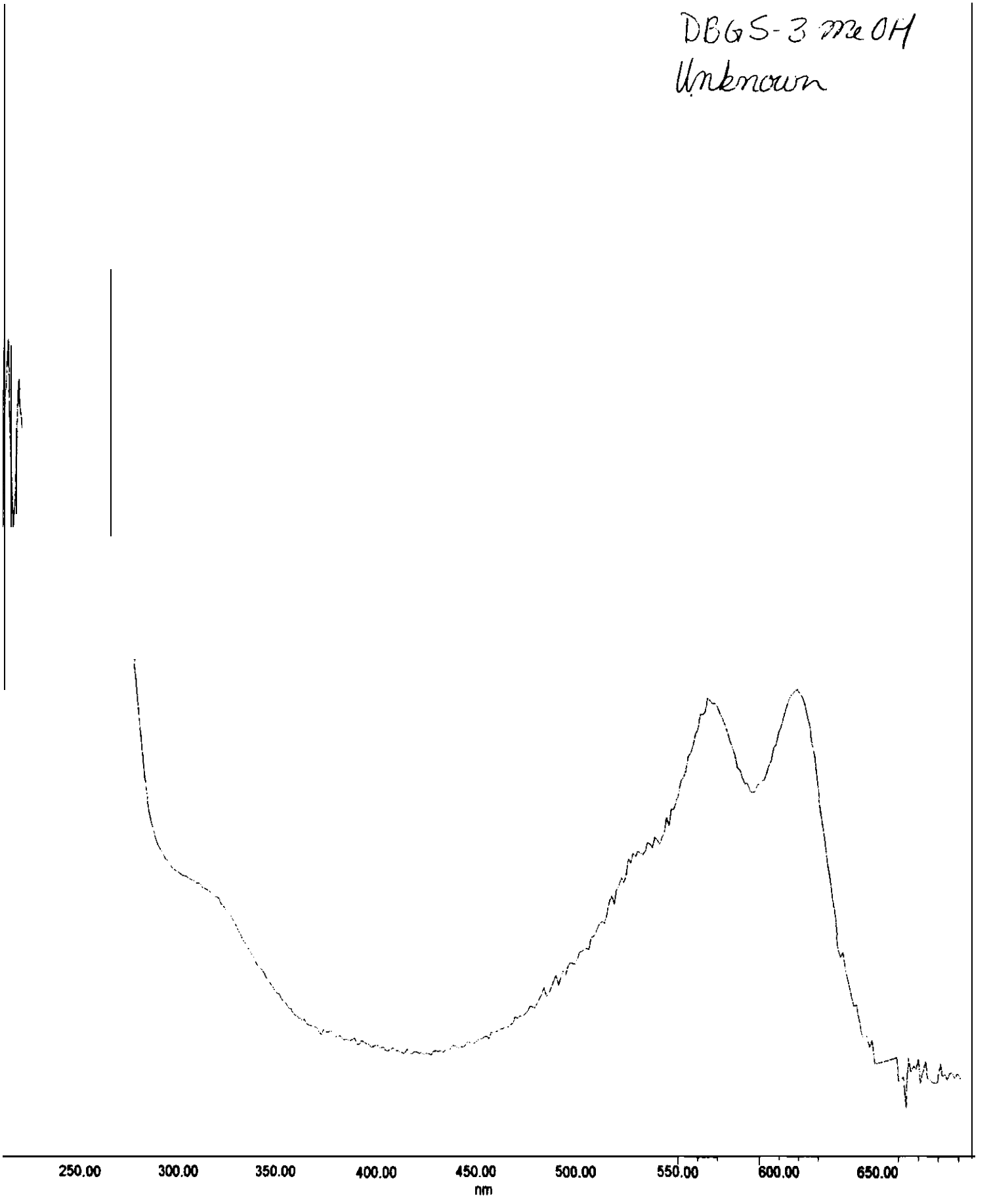

DEGS-3 MeOH
D5194030
0154559
@590 nm
Unknown
4.413
Unknown
5.005
Unknown
5.461
Disperse Blue 14 CAS# 2475-44-7)
5.923
Similar to Disperse Blue 14
7.428
0.110
0.105
0.100
0.095
0.090
0.085
0.080
0.075
0.070
0.065
0.060
0.055
0.050
0.045
0.040
0.035
0.030
0.025
0.020
0.015
0.010
0.005
0.000
-0.005
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
3.00
Minutes

DBGS-3 MeOH
Unknown



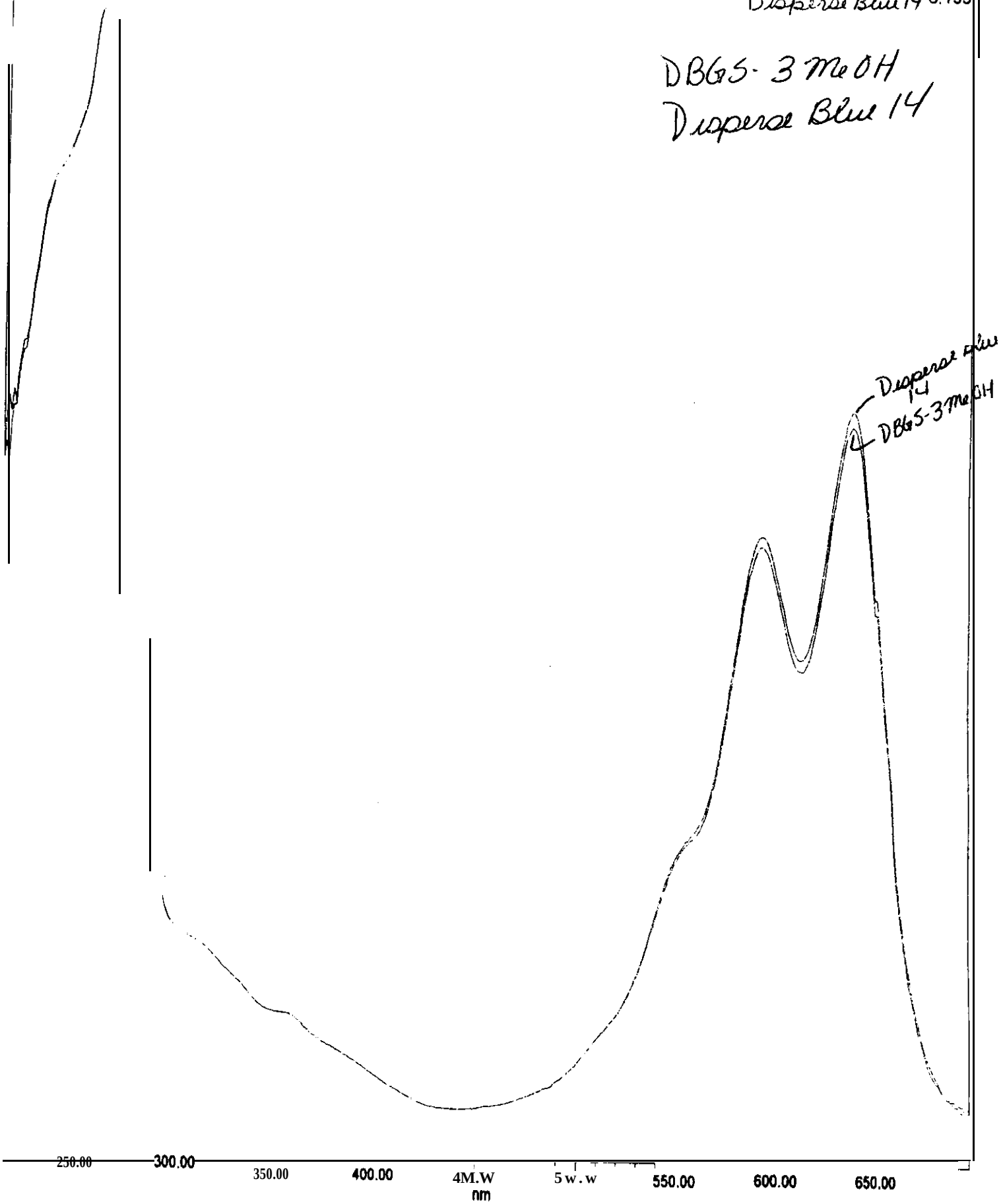

DBGS-3 MeOH
Unknown
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

DBGS-3 MeOH
Unknown

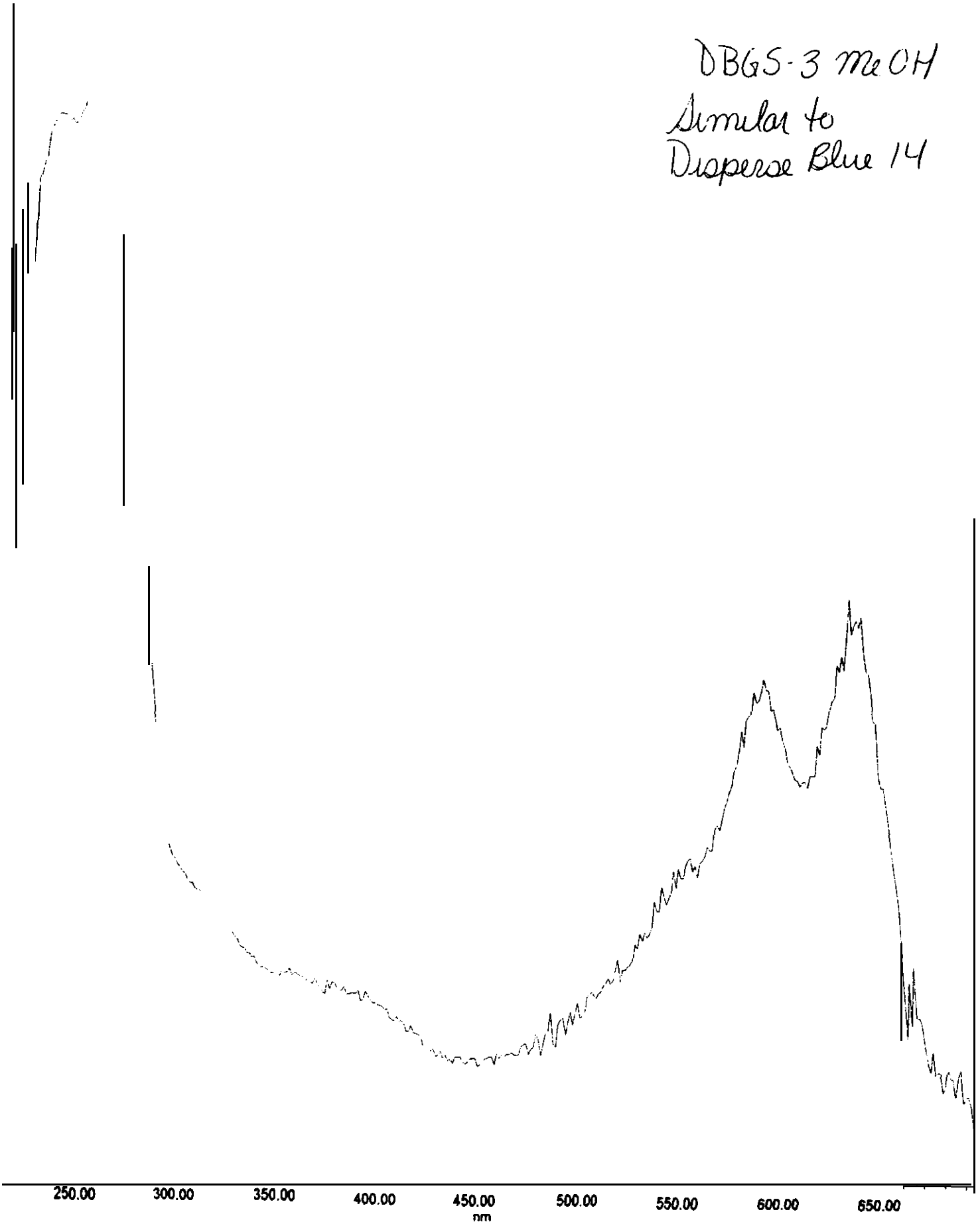


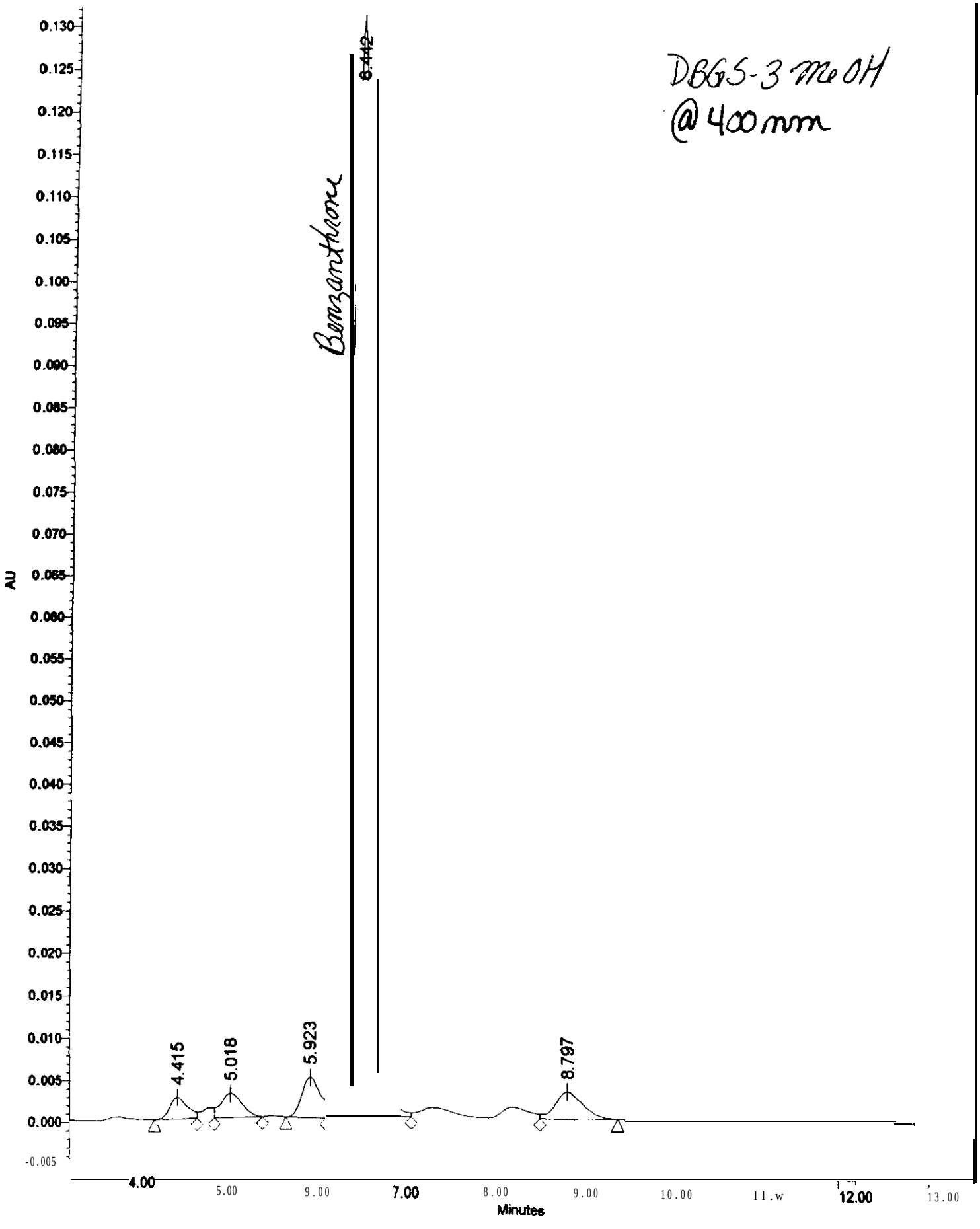
Disperse Blue 14 6.199

DBGS-3 MeOH
Disperse Blue 14

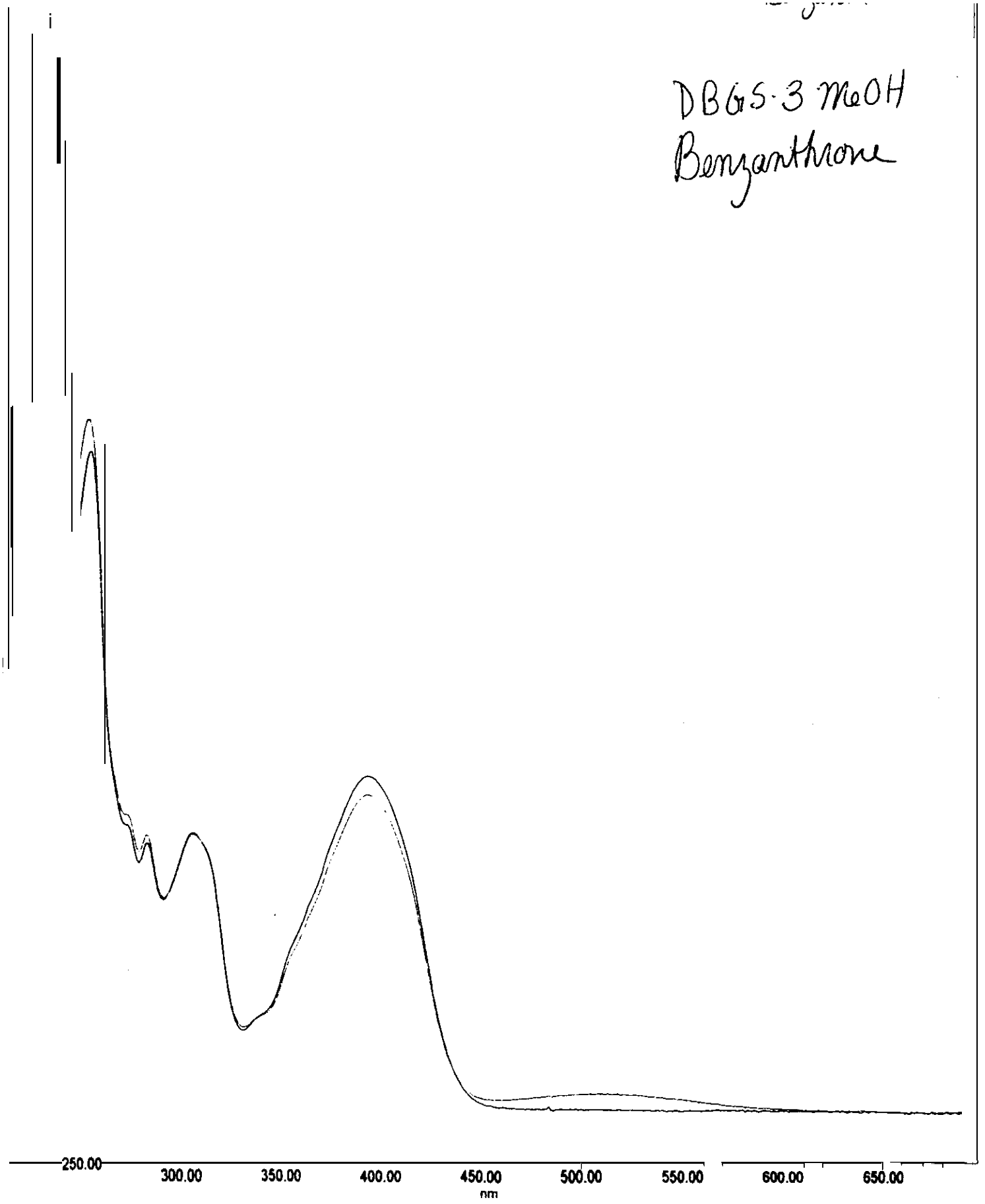


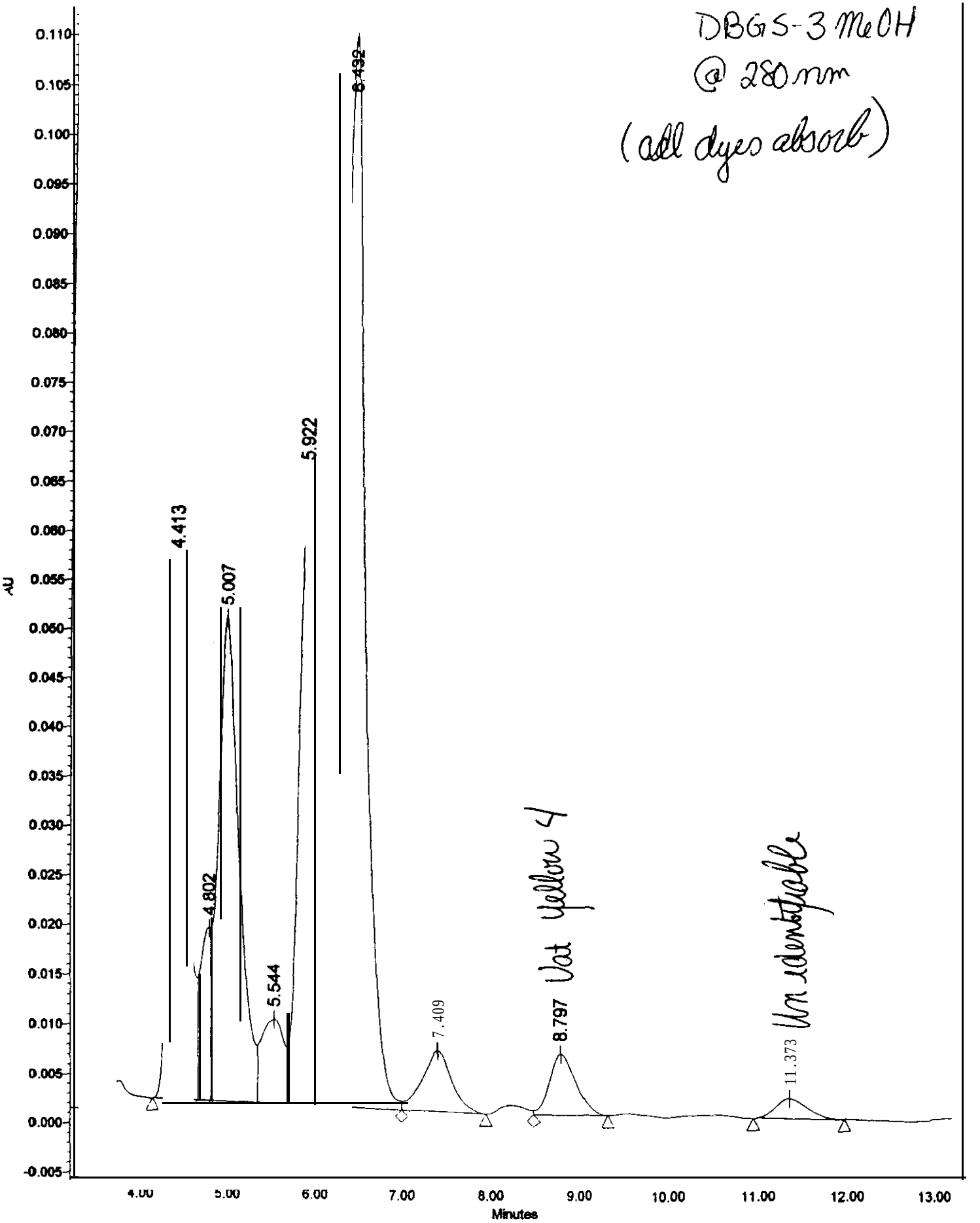
DBGS-3 MeOH
Similar to
Disperse Blue 14




DBGS-3 MeOH
@400nm
Benzanthrone
~~6.442~~
AU
0.130
0.125
0.120
0.115
0.110
0.105
0.100
0.095
0.090
0.085
0.080
0.075
0.070
0.065
0.060
0.055
0.050
0.045
0.040
0.035
0.030
0.025
0.020
0.015
0.010
0.005
0.000
-0.005
4.415
5.018
5.923
8.797
4.00
5.00
9.00
7.00
8.00
9.00
10.00
11.w
12.00
13.00
Minutes

DBGS-3 MeOH
Benzanthrone

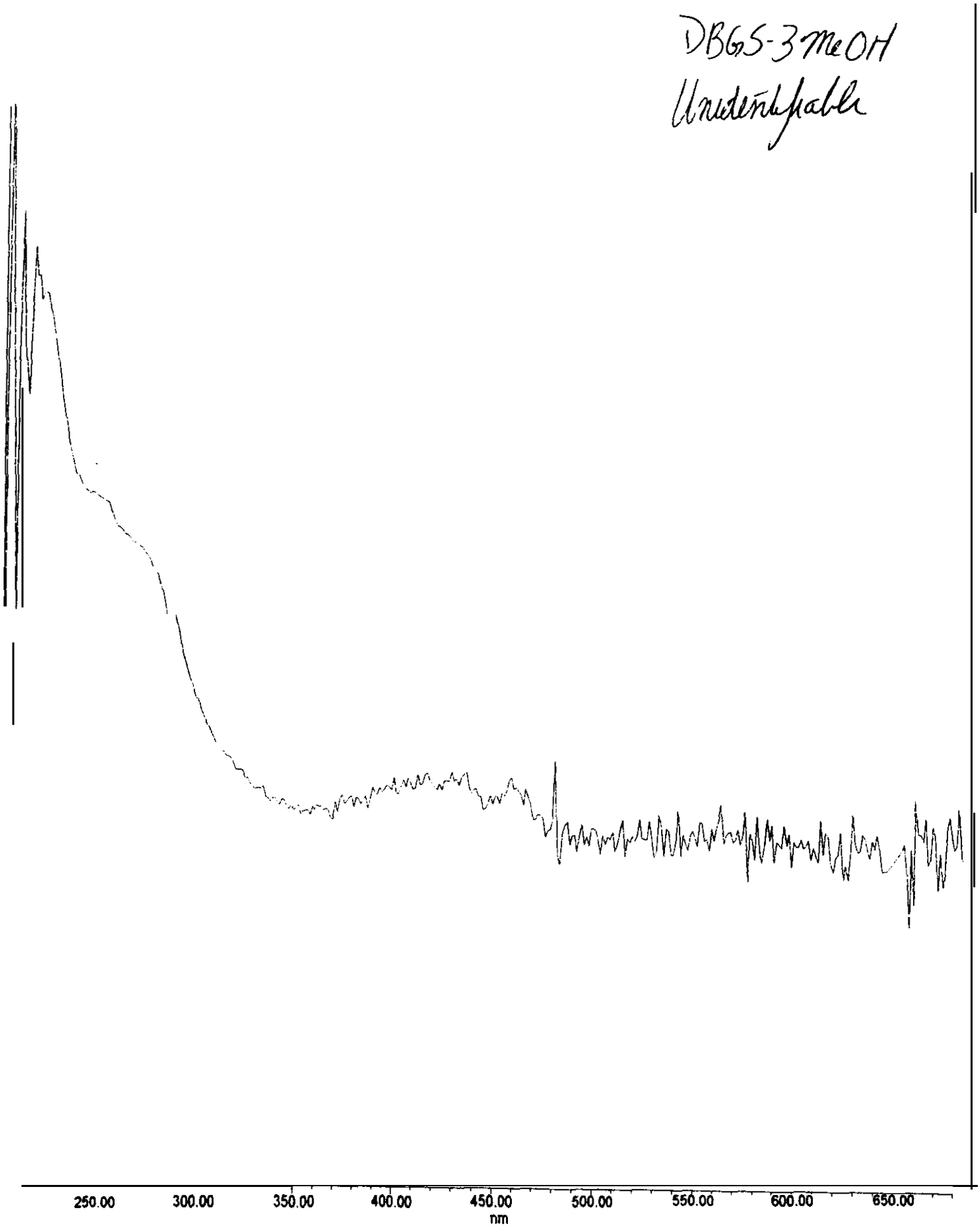



DBGS-3 MeOH
@ 280 nm
(all dyes absorb)
AU
Minutes
4.413
4.802
5.007
5.544
5.922
6.432
7.409
8.797
Vat Yellow 4
11.373
Unidentifiable

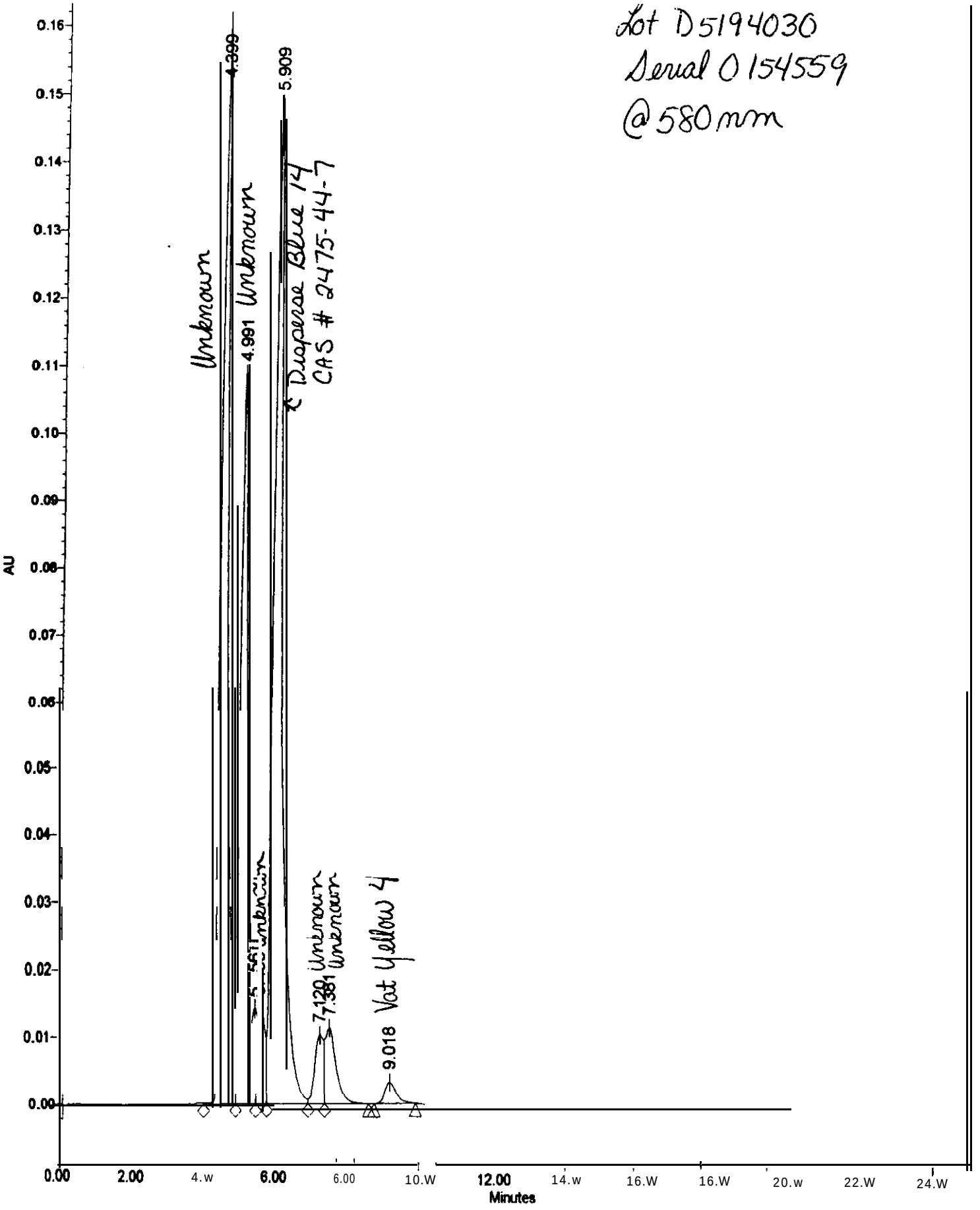
Vat Yellow 4 – 9.340

DBGS-3 MeOH
Vat Yellow 4

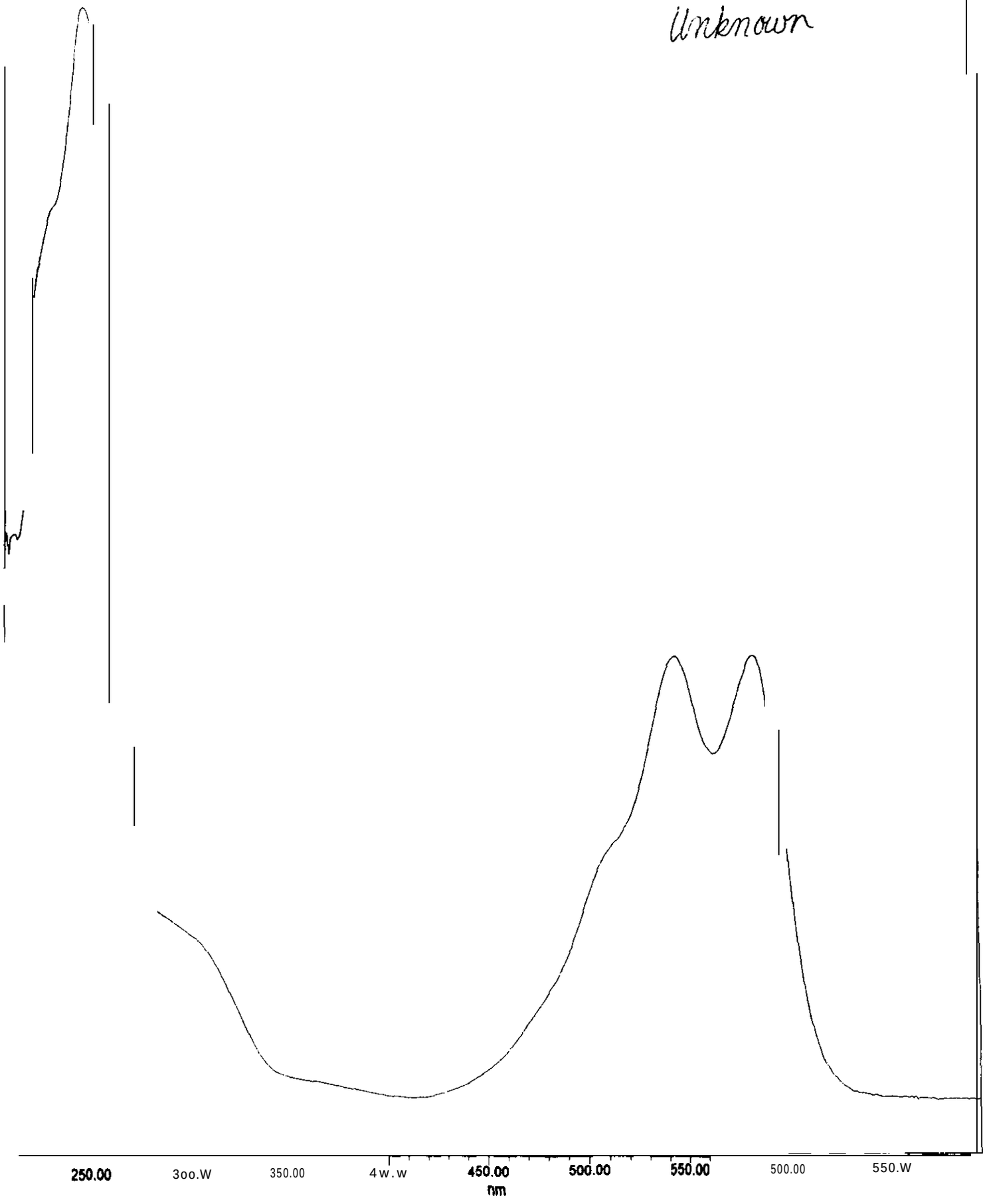
DBGS-3 MeOH
Unidentifiable



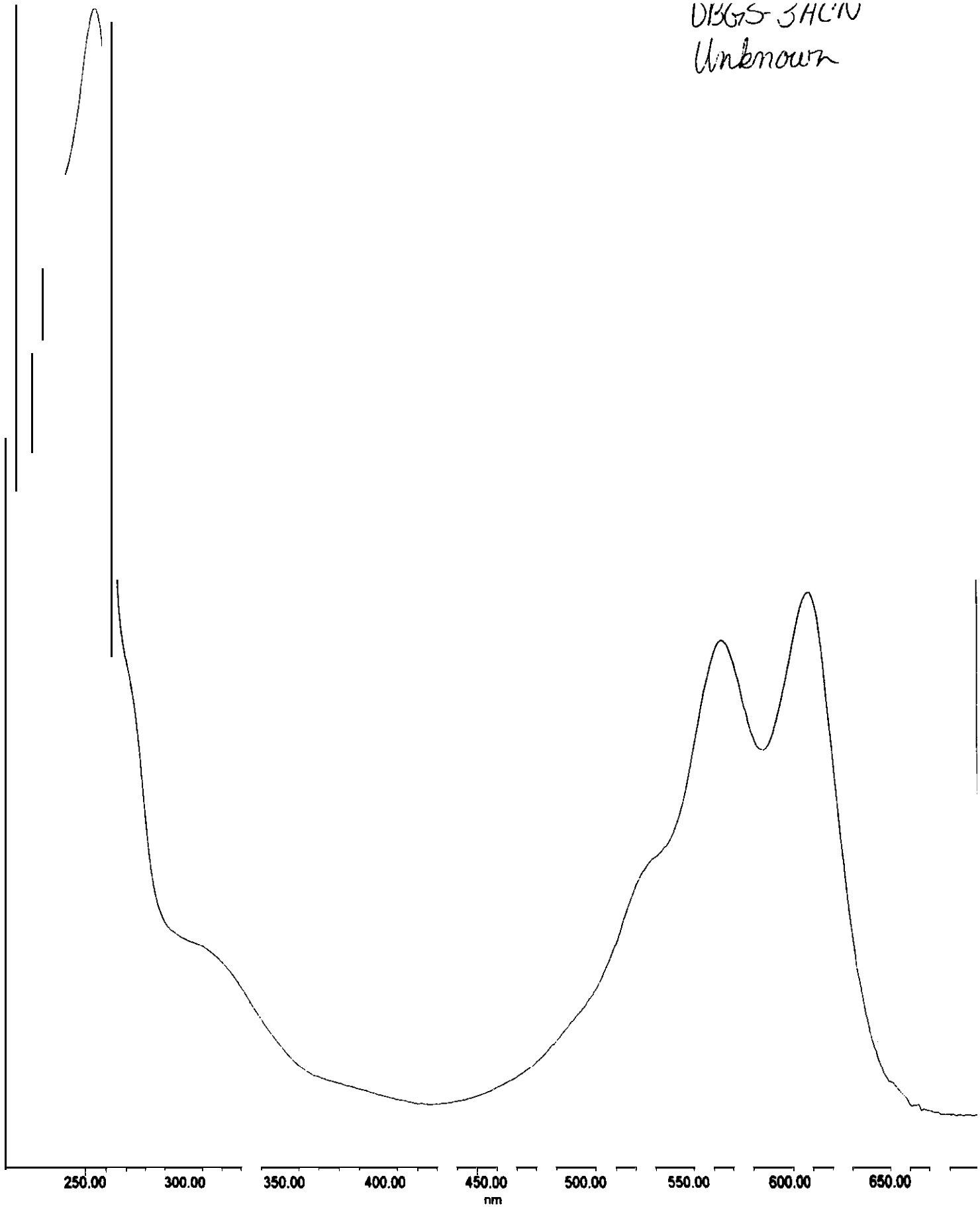
Lot D5194030
Serial 0154559
@580nm

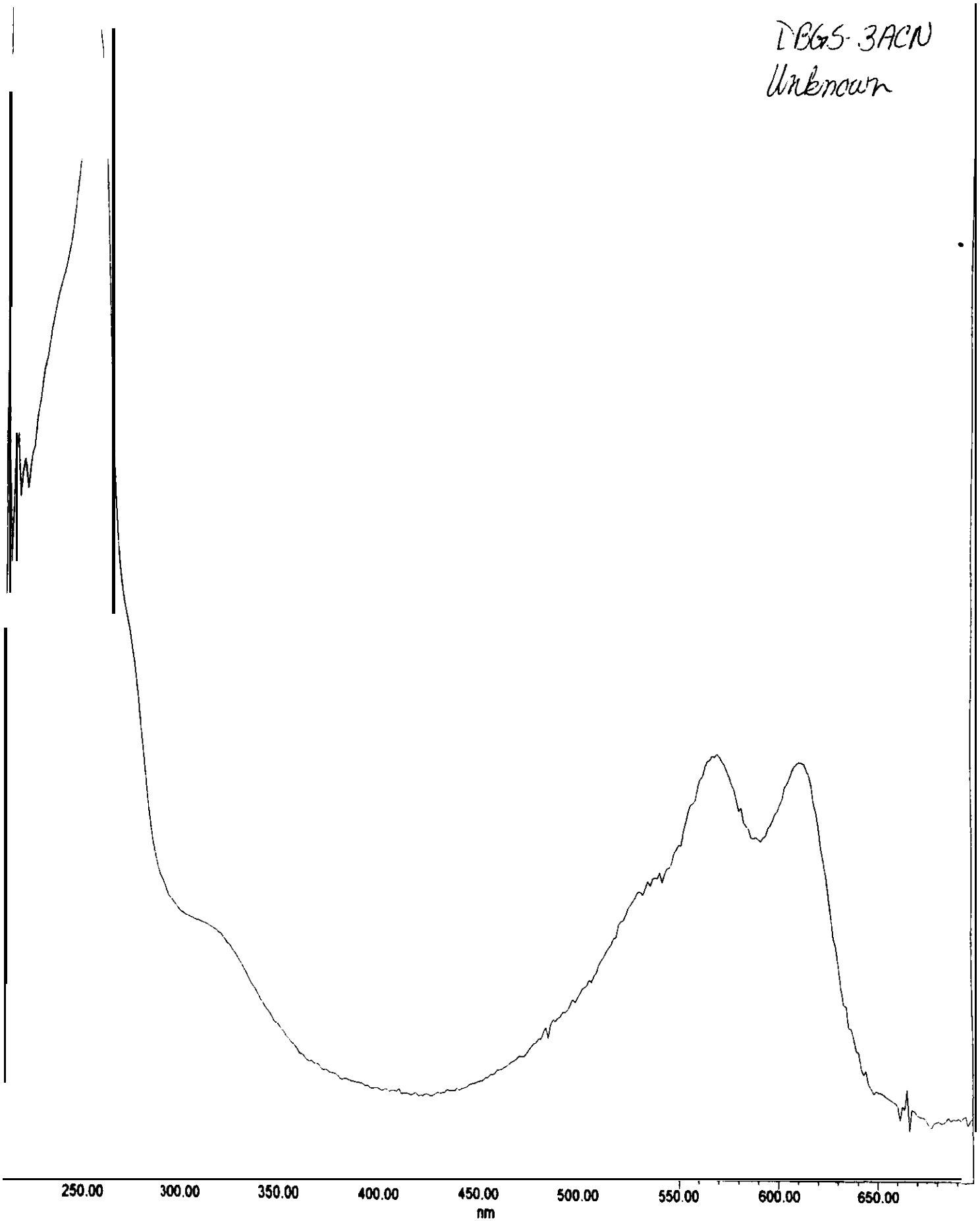


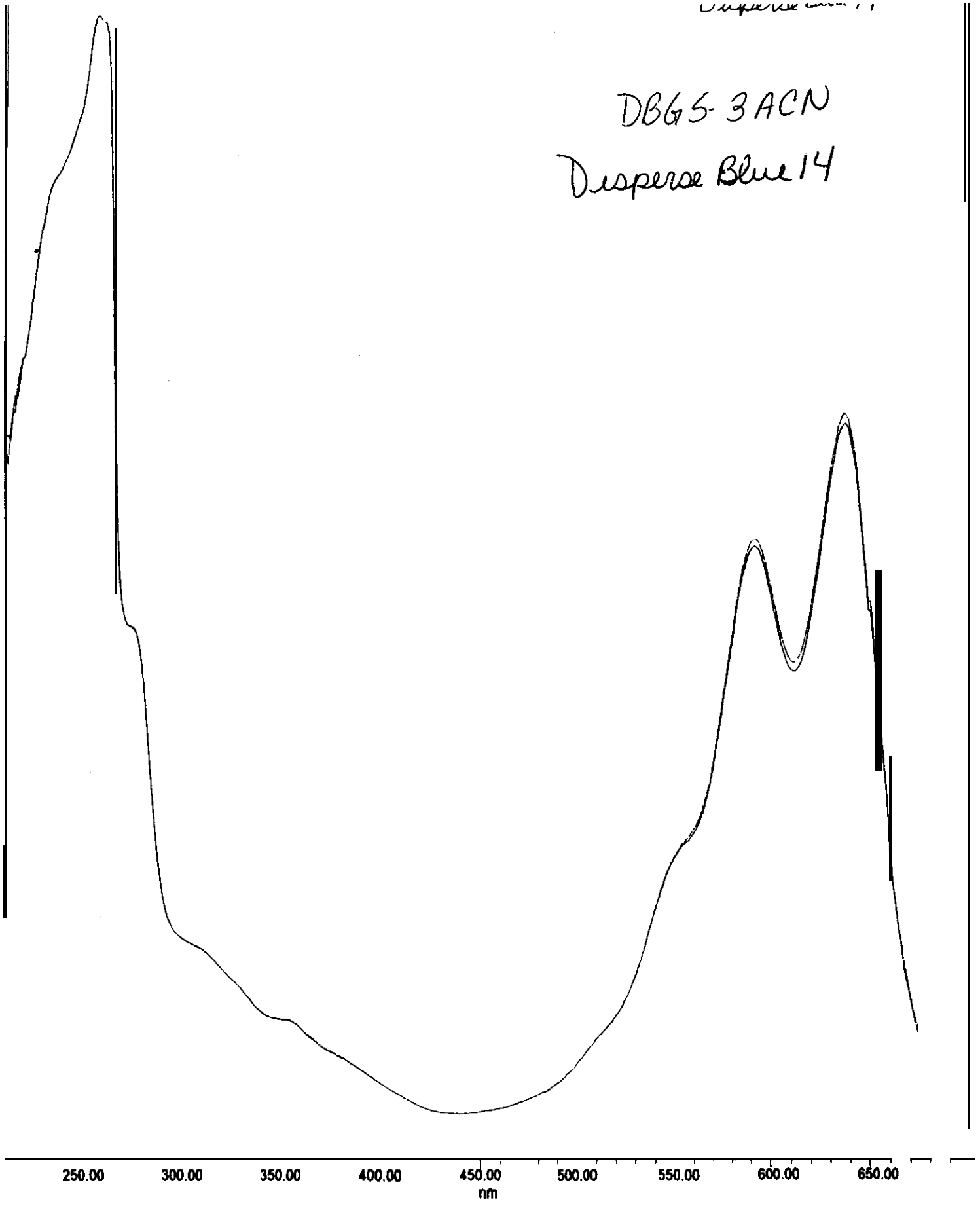
DBGS-3ACN
Unknown



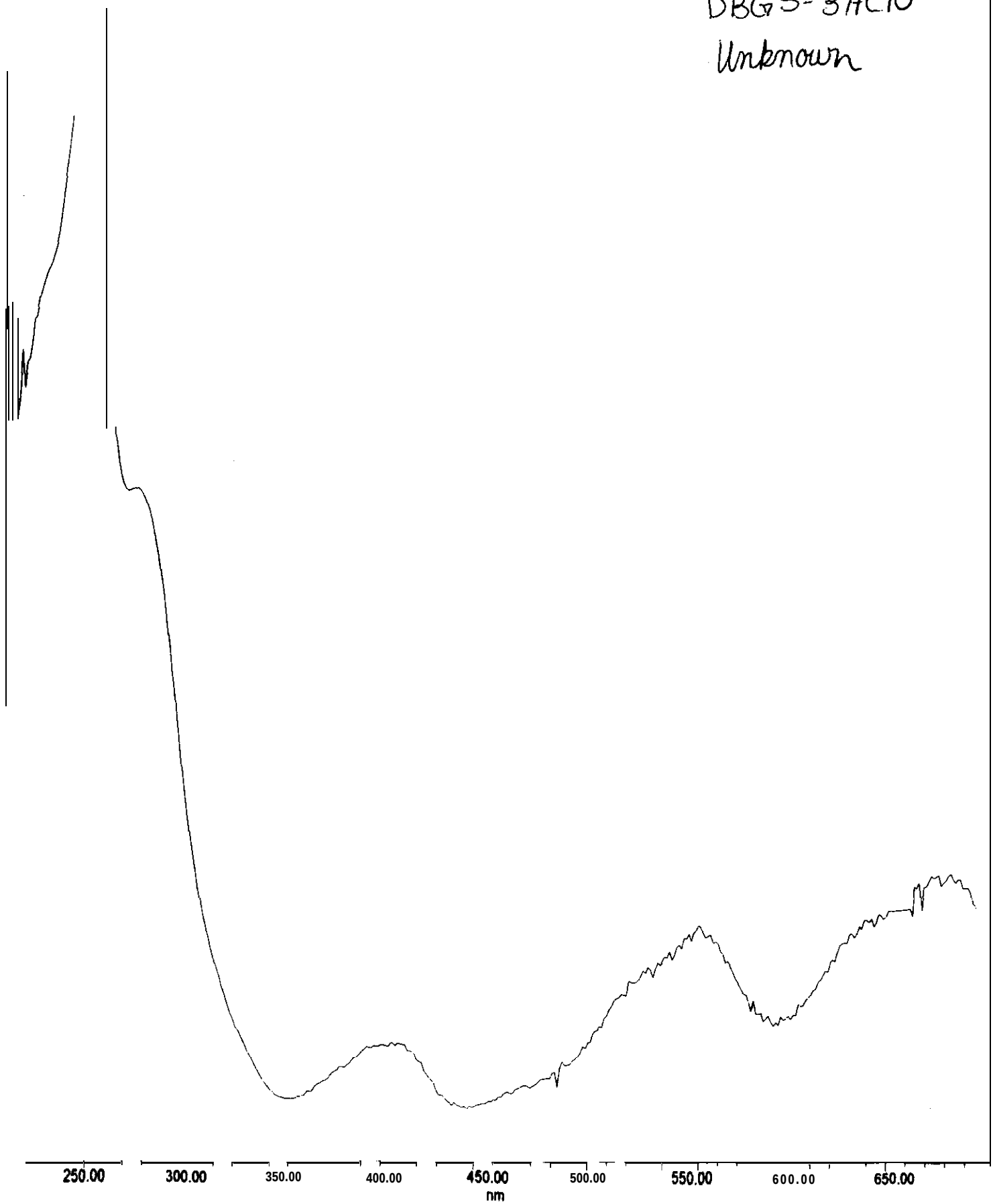
DIGGS-JALIN
Unknown

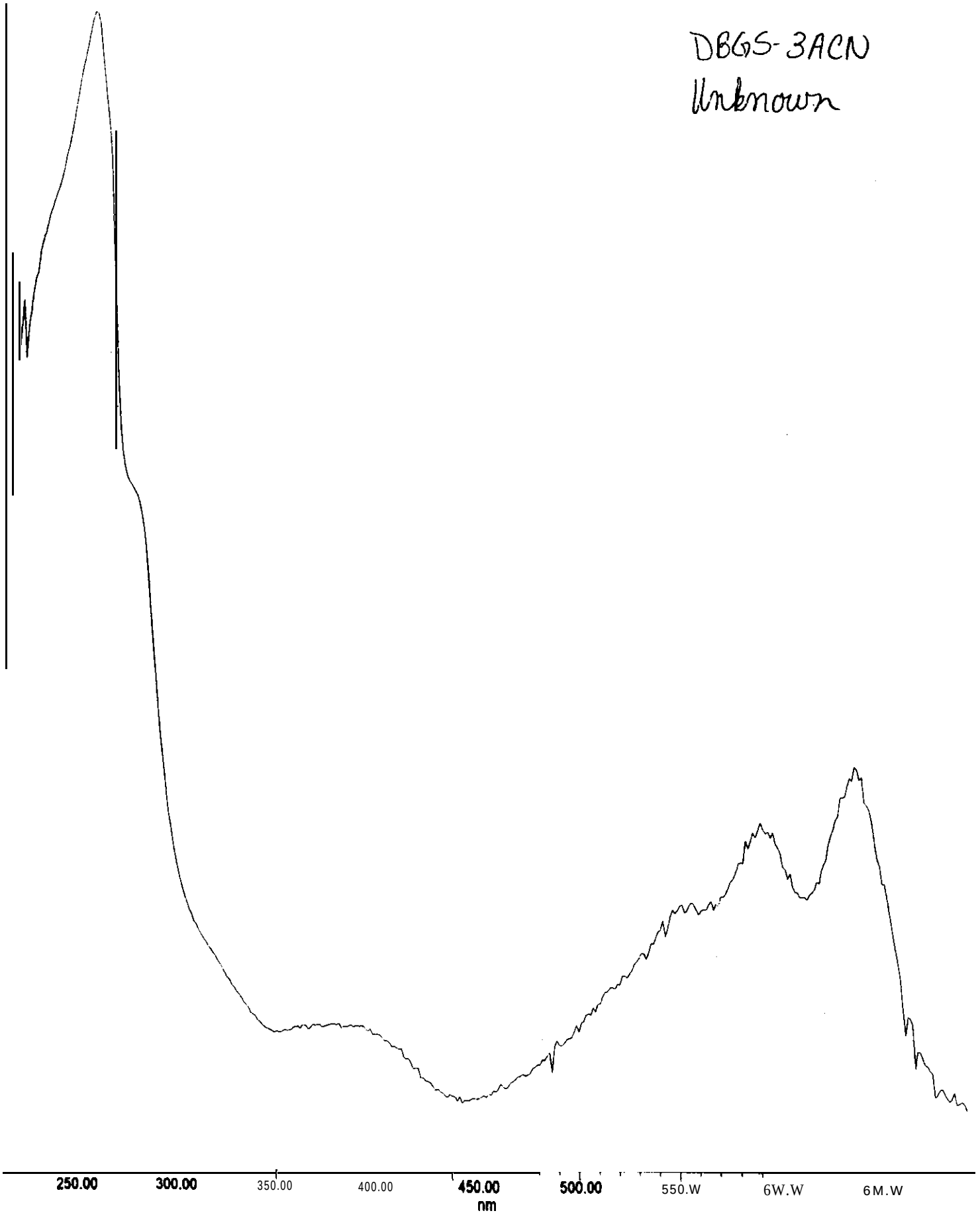


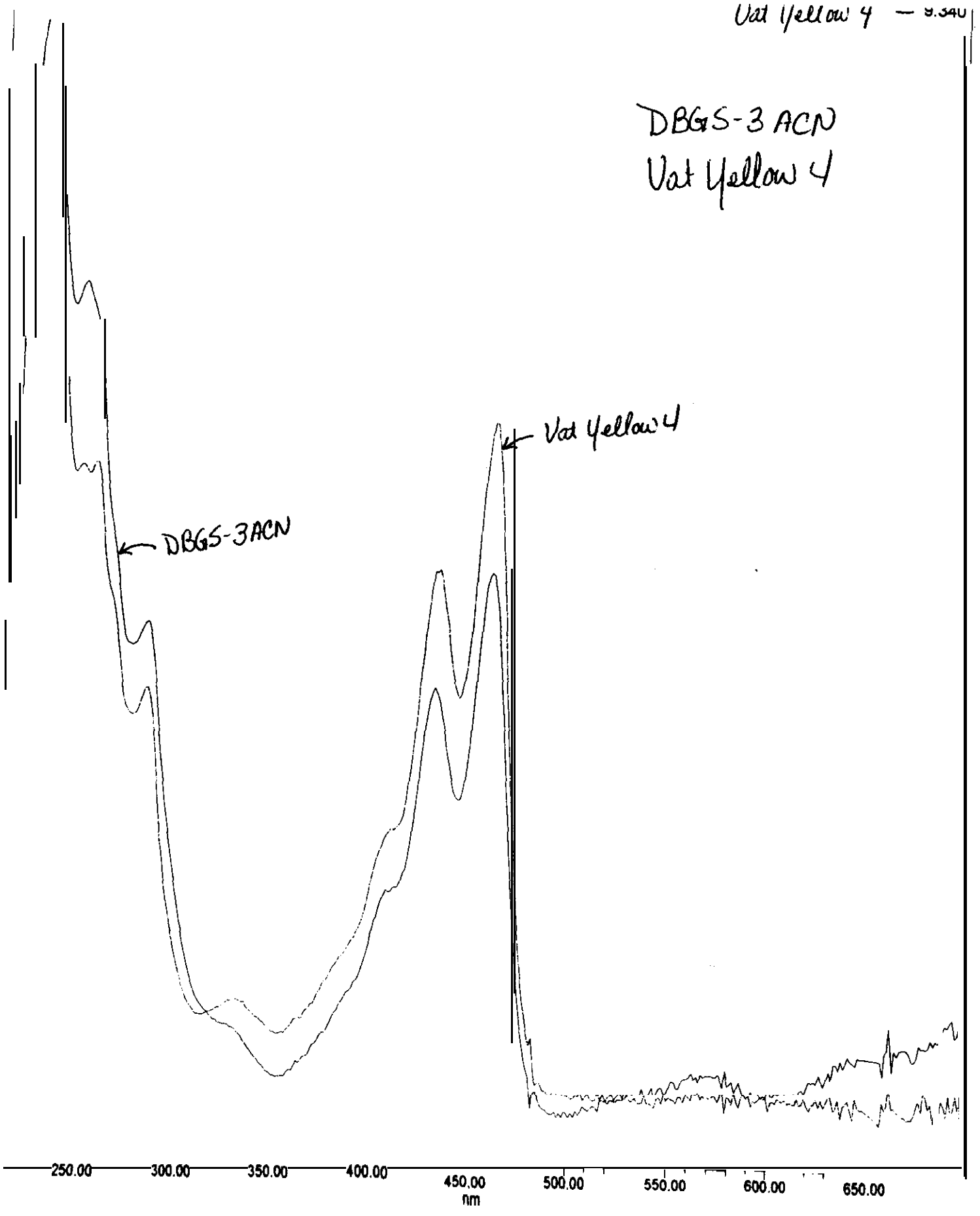

DBGS-3ACN
Unknown
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

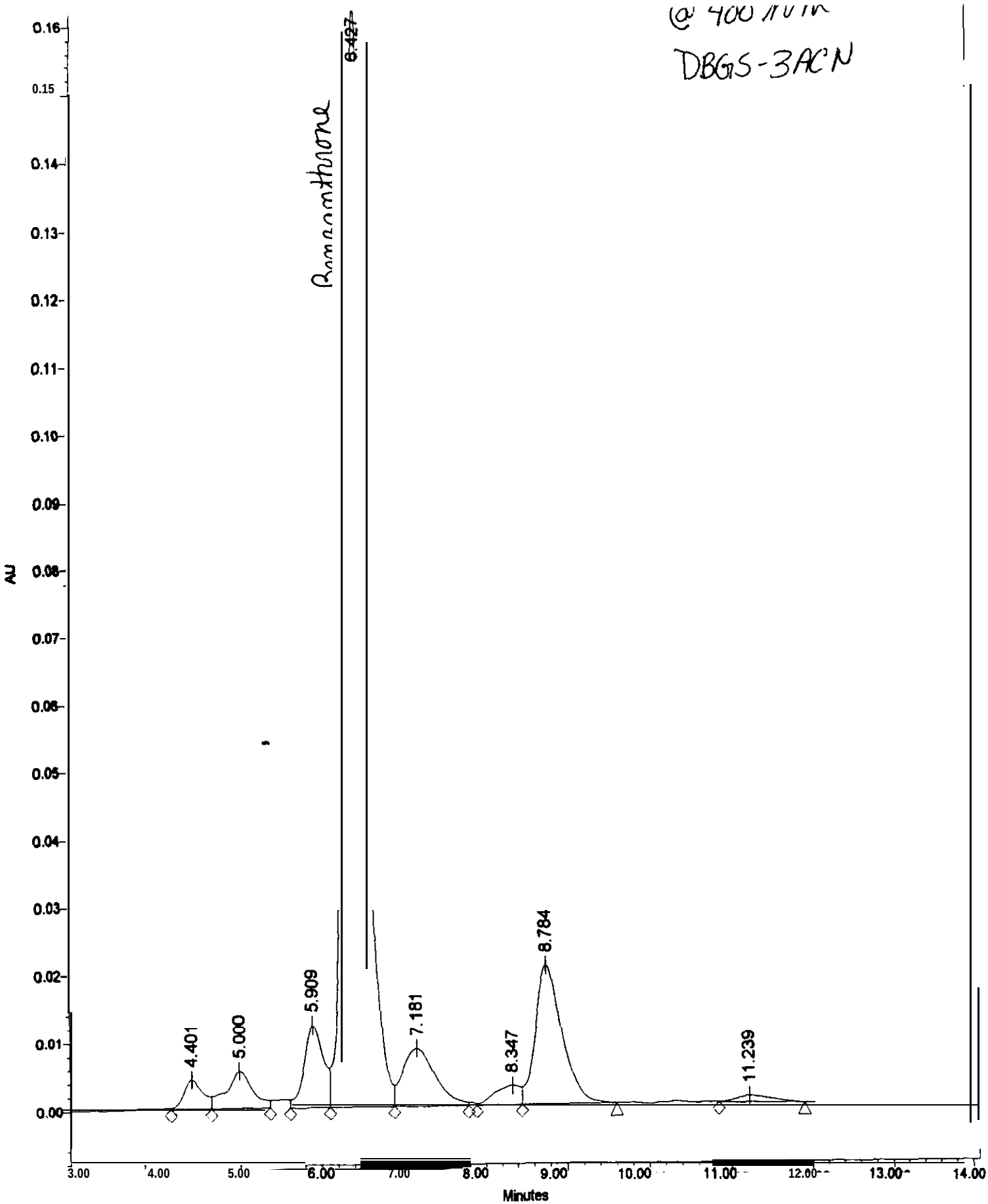

DBGS-3ACN
Disperse Blue 14
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

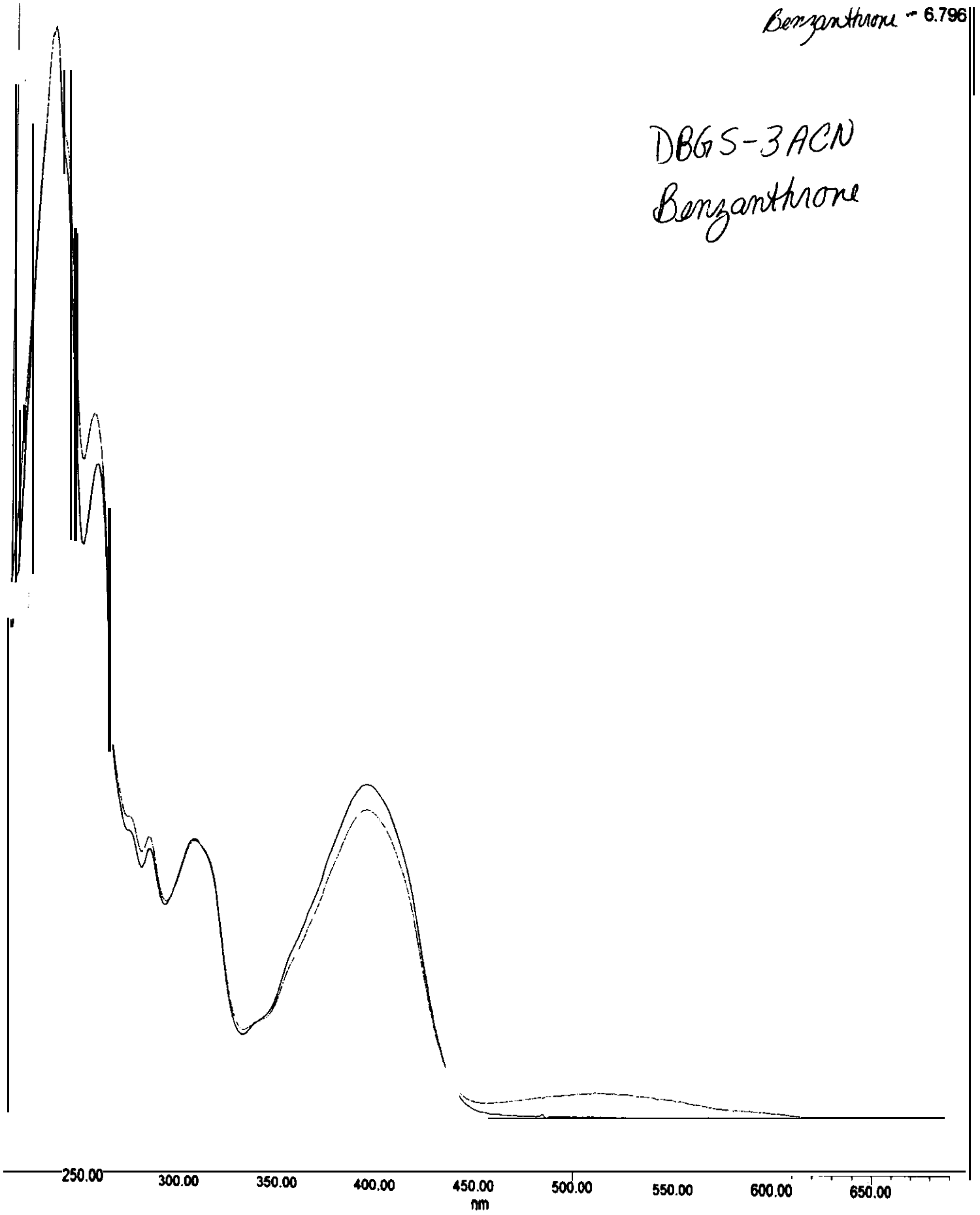
DBGS-3ACN
Unknown

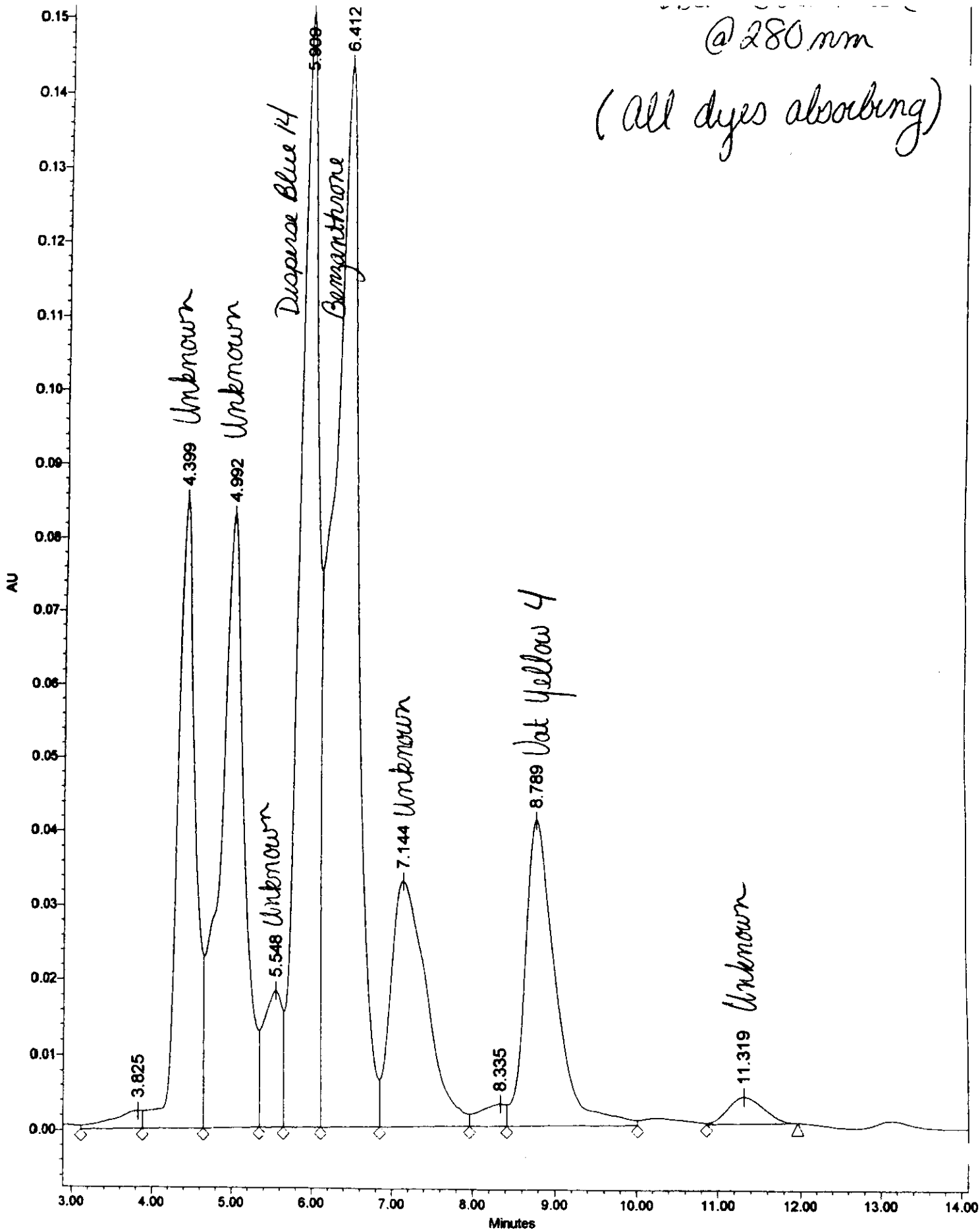


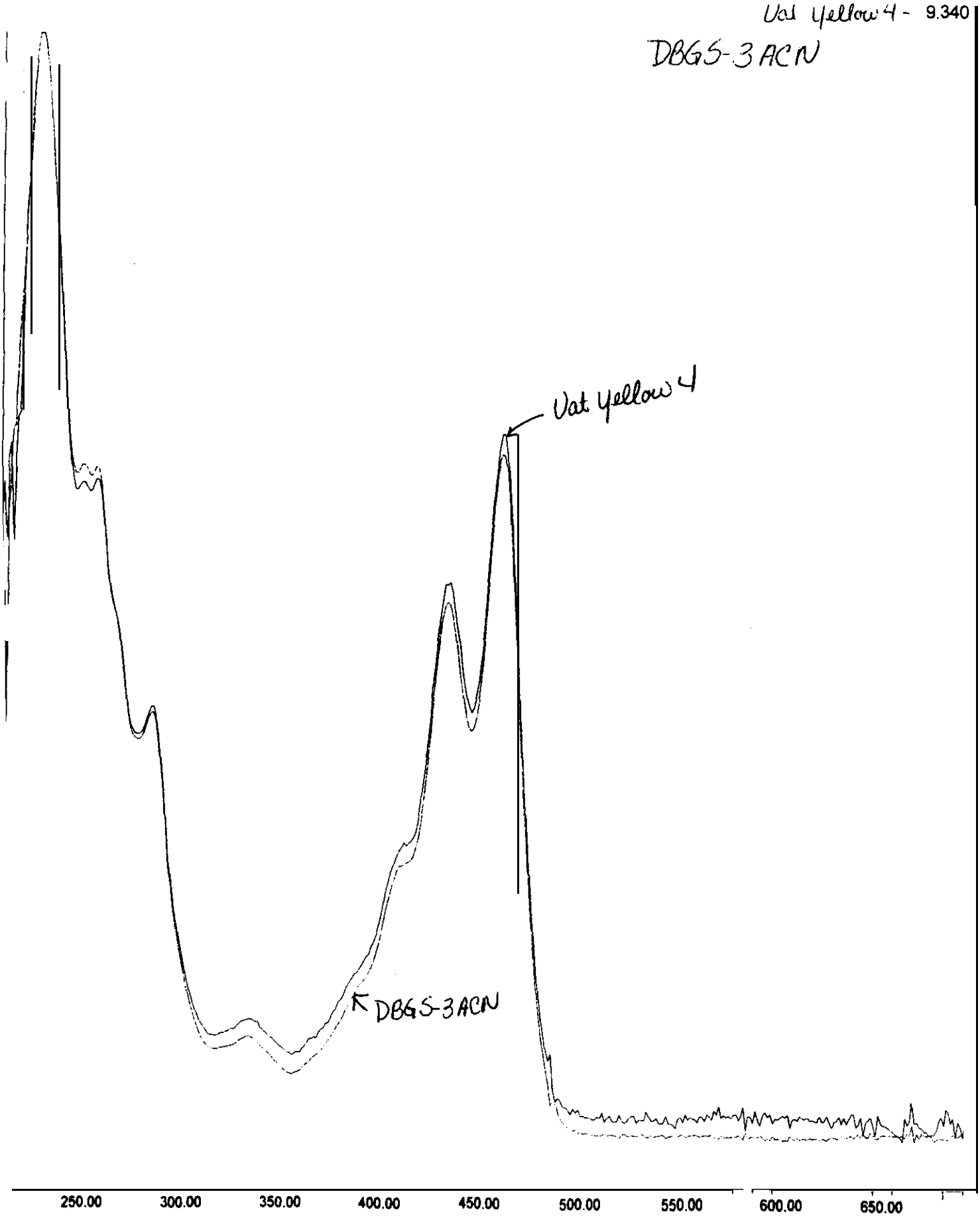

DBGS-3ACN
Unknown
250.00
300.00
350.00
400.00
450.00
nm
500.00
550.W
6W.W
6M.W


Vat Yellow 4 — 9.340
DBGS-3 ACN
Vat Yellow 4
Vat Yellow 4
DBGS-3ACN
250.00
300.00
350.00
400.00
450.00
nm
500.00
550.00
600.00
650.00

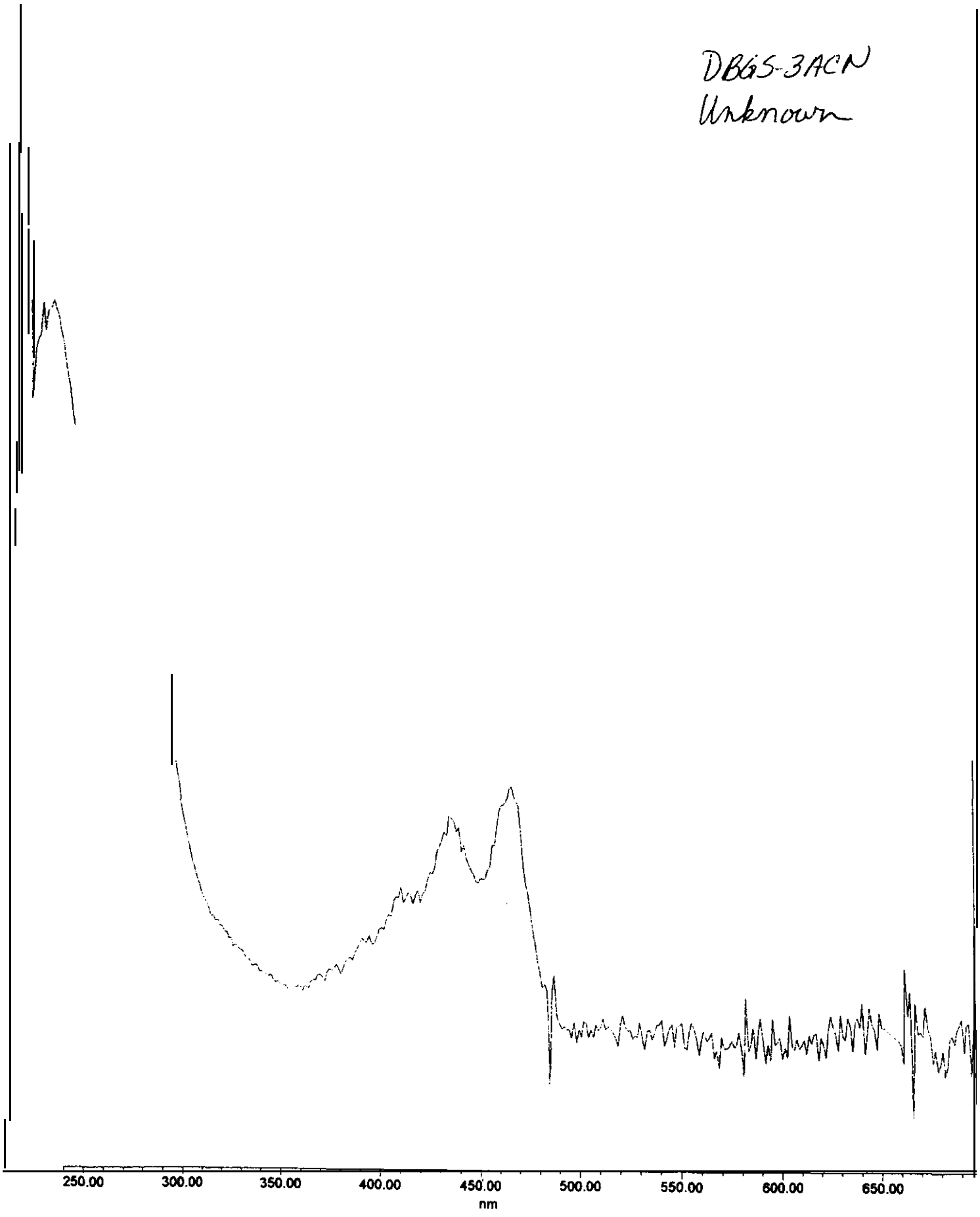

@ 400 nm
DBGS-3ACN
Ramanathone
8.427
4.401
5.000
5.909
7.181
8.347
8.784
11.239
AU
Minutes
0.16
0.15
0.14
0.13
0.12
0.11
0.10
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0.00
3.00
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.00

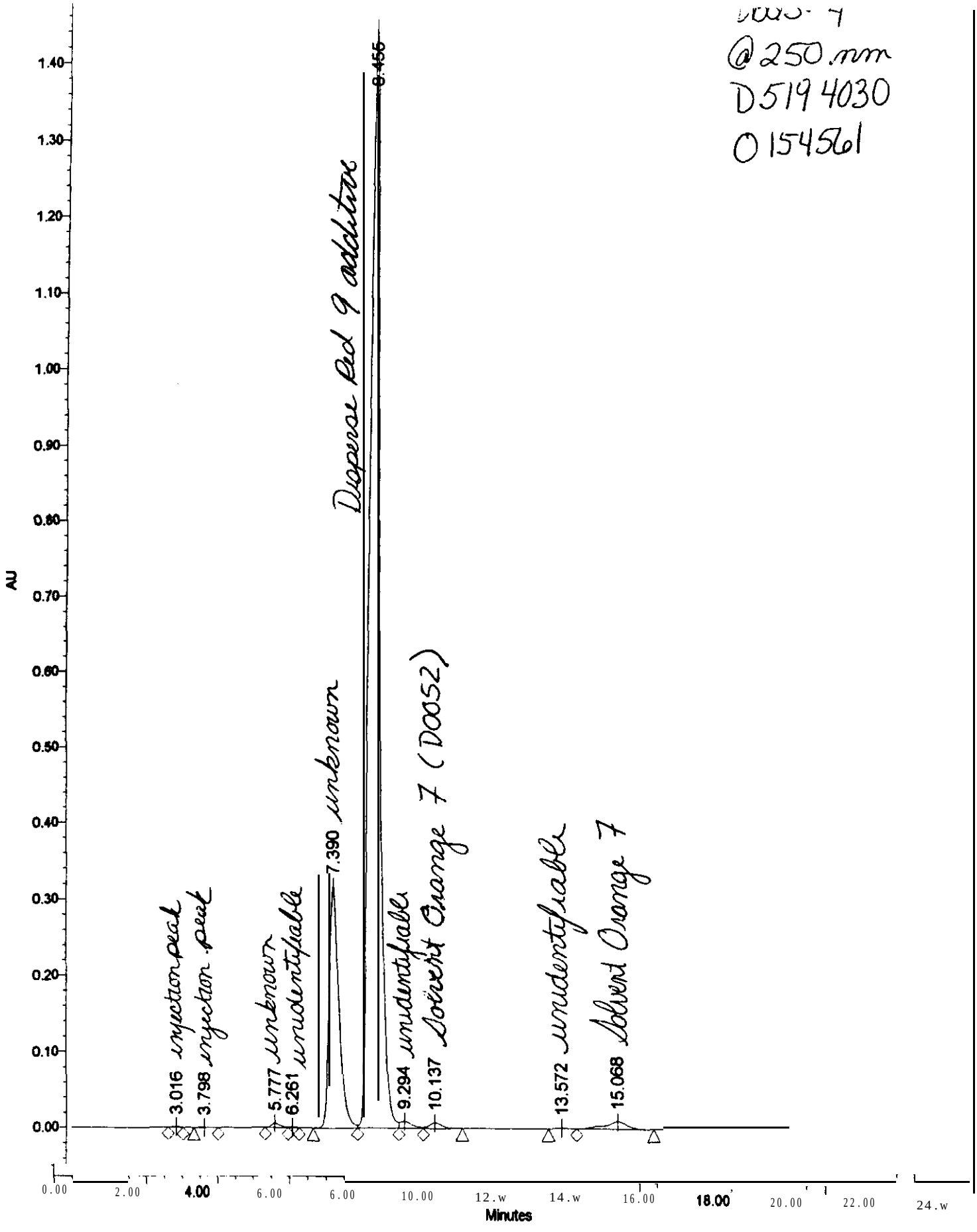

Benzanthrone - 6.796
DBGS-3ACN
Benzanthrone
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm


@280 nm
(All dyes absorbing)
Unknown
Unknown
Dupont Blue 14
Benzanthrone
Unknown
Unknown
Vat Yellow 4
Unknown
3.825
4.399
4.992
5.548
5.909
6.412
7.144
8.335
8.789
11.319
AU
Minutes


Vat Yellow 4 - 9.340
DBGS-3ACN
Vat Yellow 4
DBGS-3ACN
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00

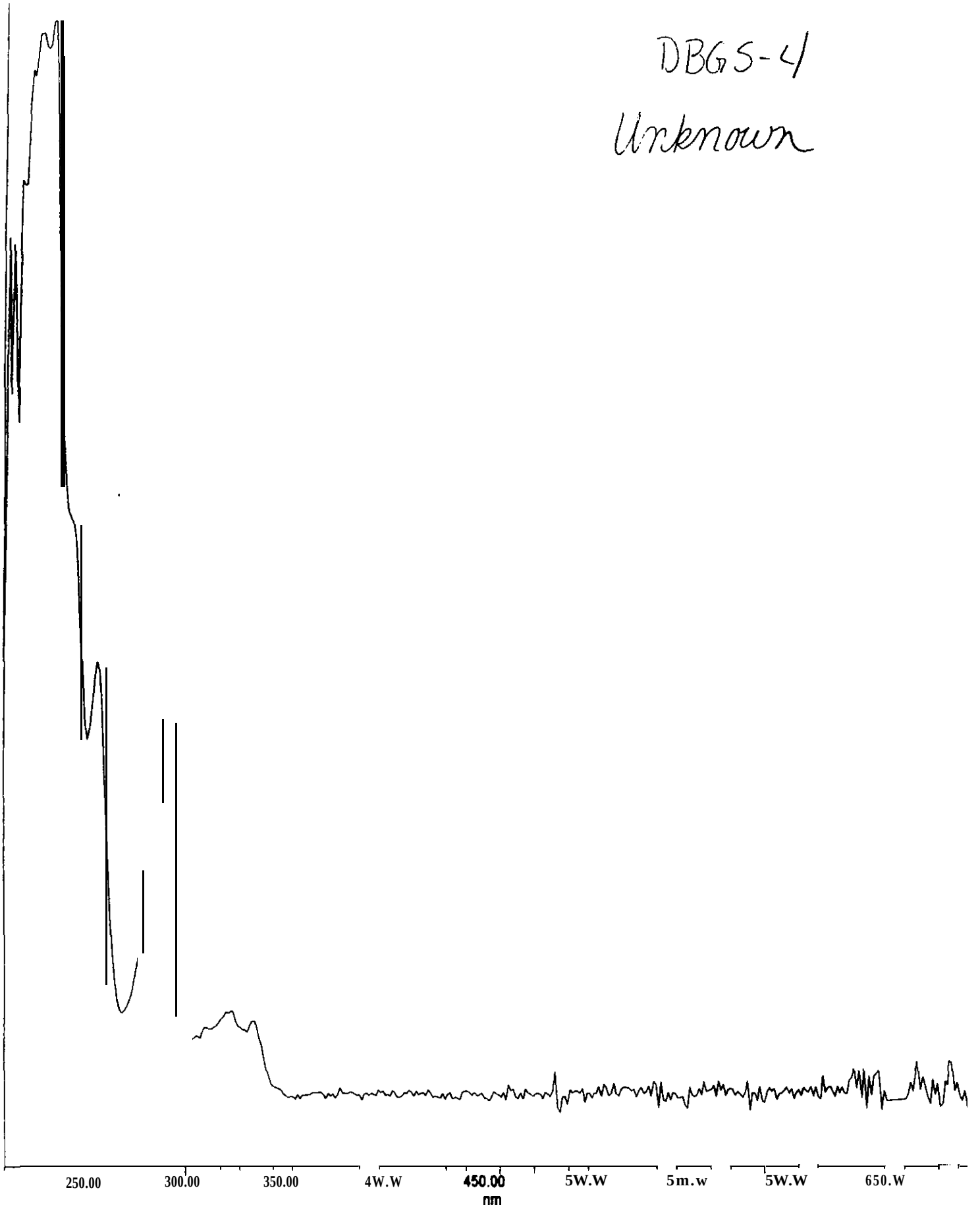
DBGS-3ACN
Unknown




D005-7
@250 nm
D5194030
0154561
AU
1.40
1.30
1.20
1.10
1.00
0.90
0.80
0.70
0.60
0.50
0.40
0.30
0.20
0.10
0.00
3.016 injection peak
3.798 injection peak
5.777 unknown
6.261 unidentifiable
7.390 unknown
8.455 Disperse Red 9 additive
9.294 unidentifiable
10.137 Solvent Orange 7 (D0052)
13.572 unidentifiable
15.068 Solvent Orange 7
0.00
2.00
4.00
6.00
6.00
10.00
12.w
14.w
16.00
18.00
20.00
22.00
24.w
Minutes

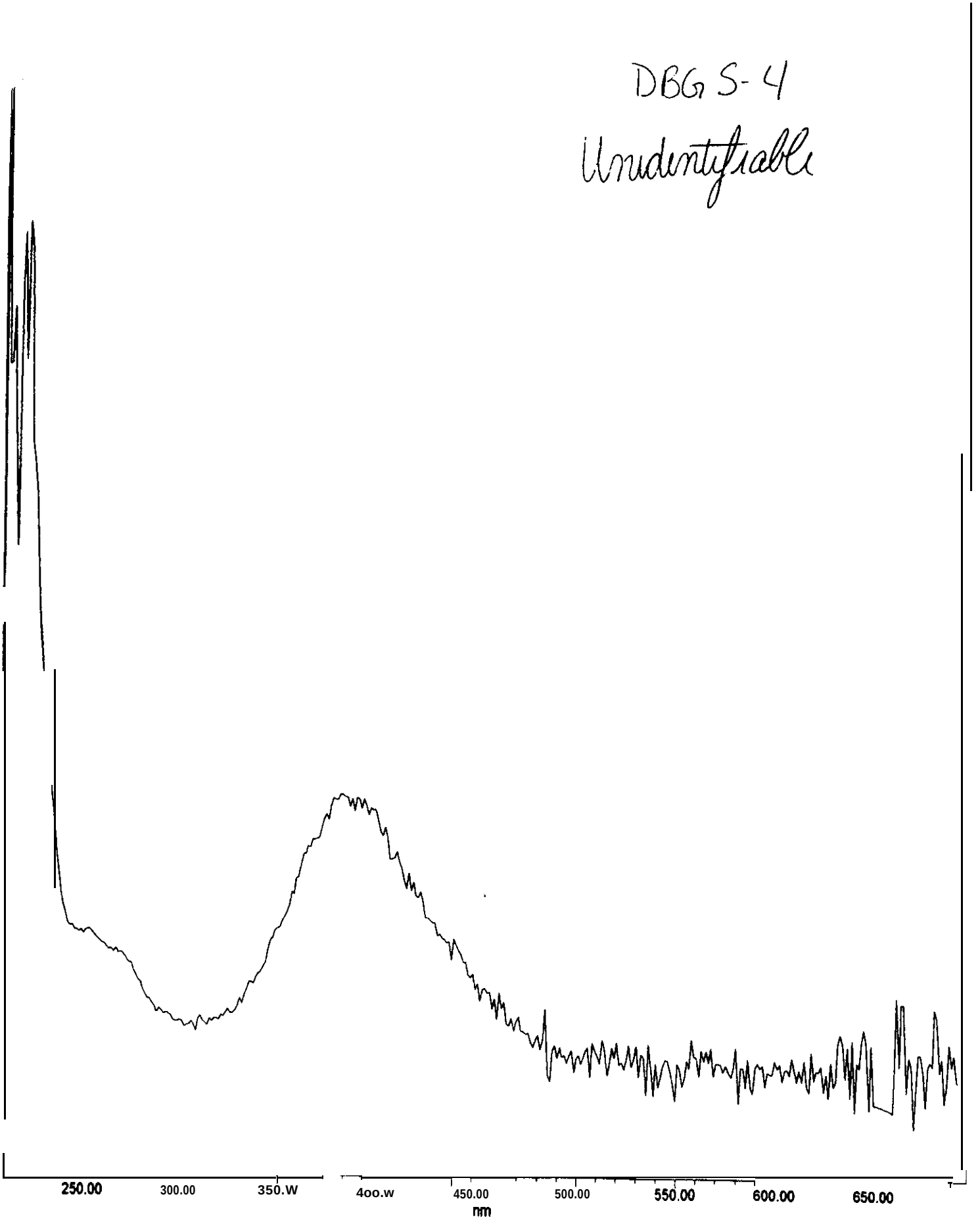
DBGS-4

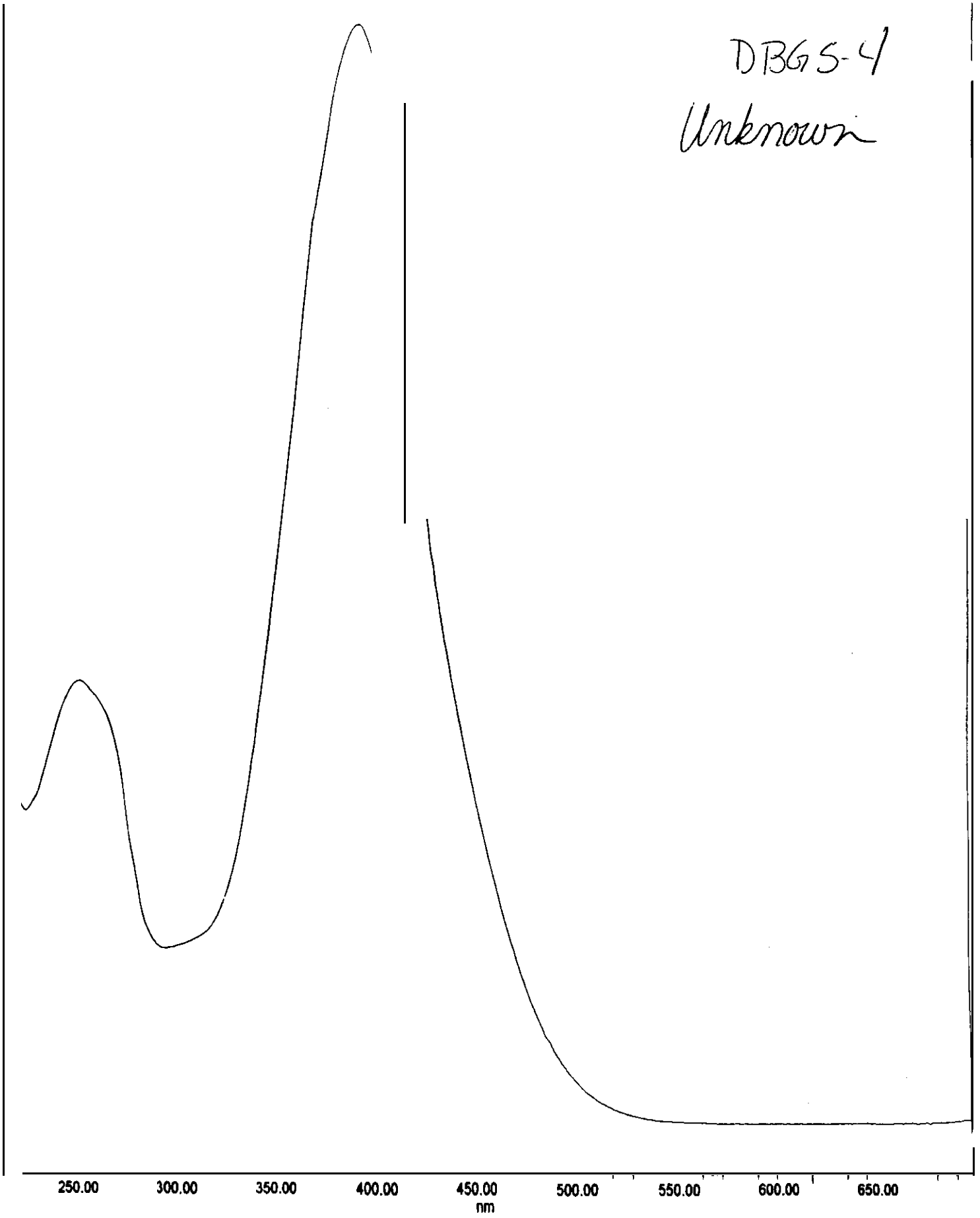
Unknown



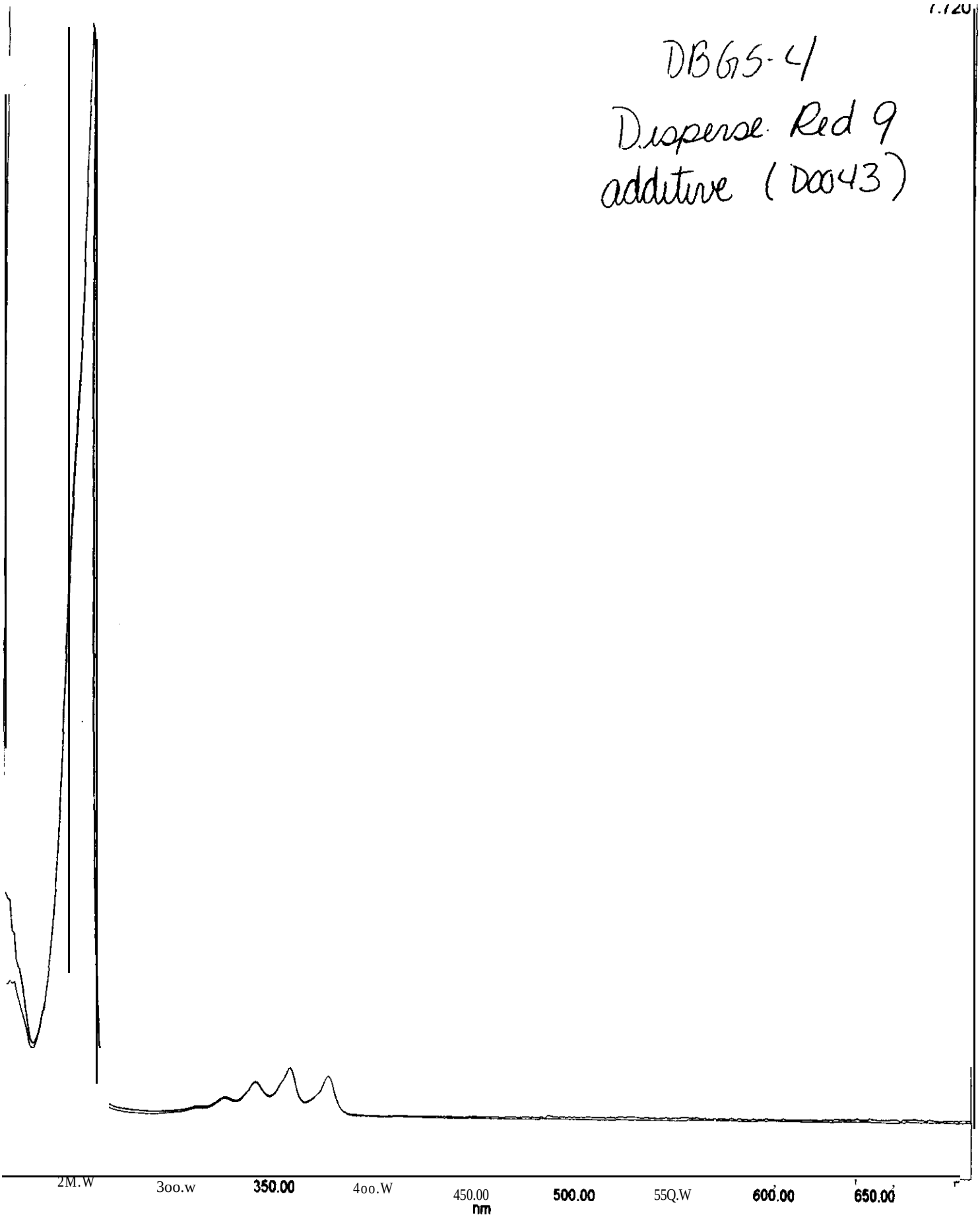
DBG S-4

Unidentifiable

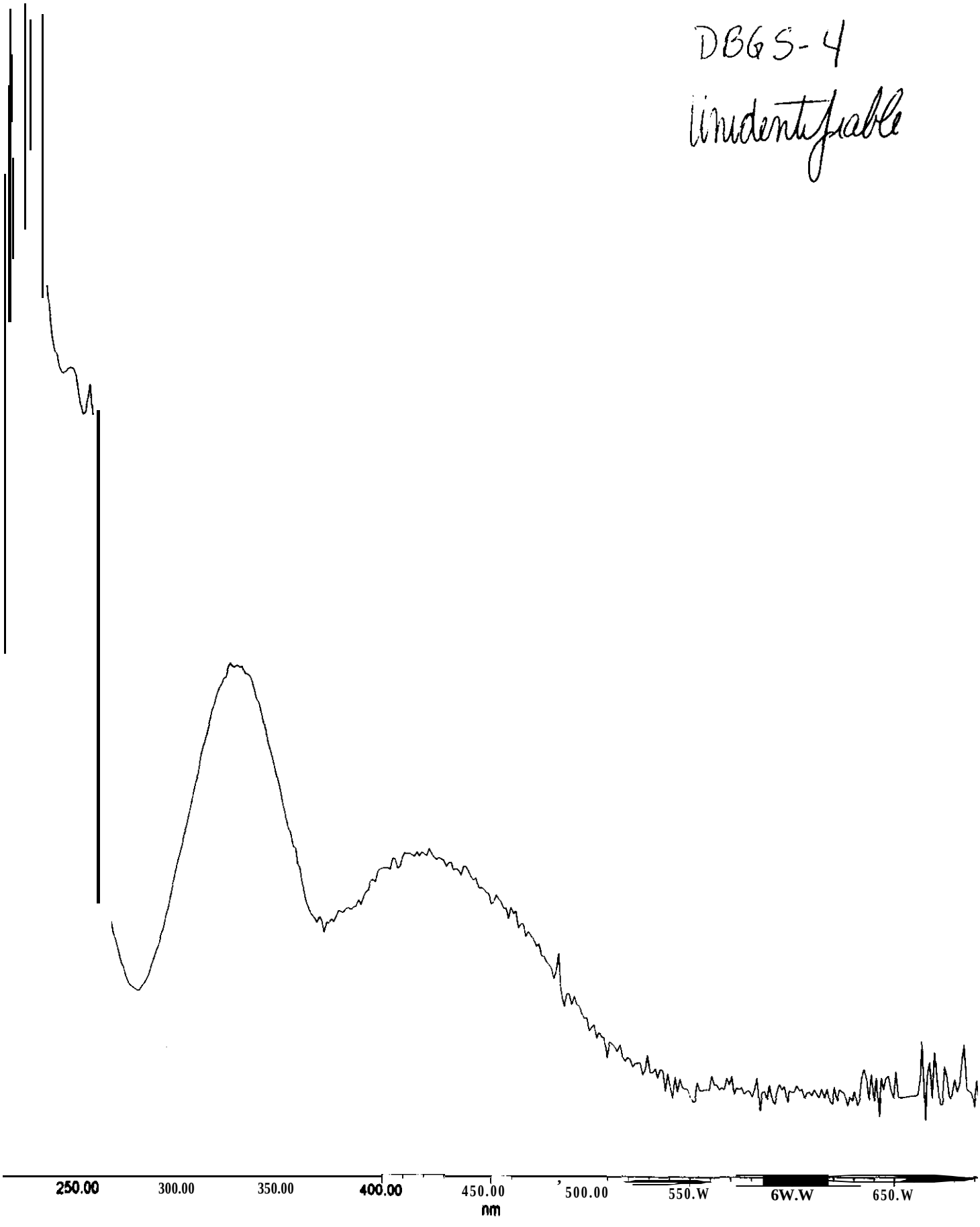



DBGS-4
Unknown
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

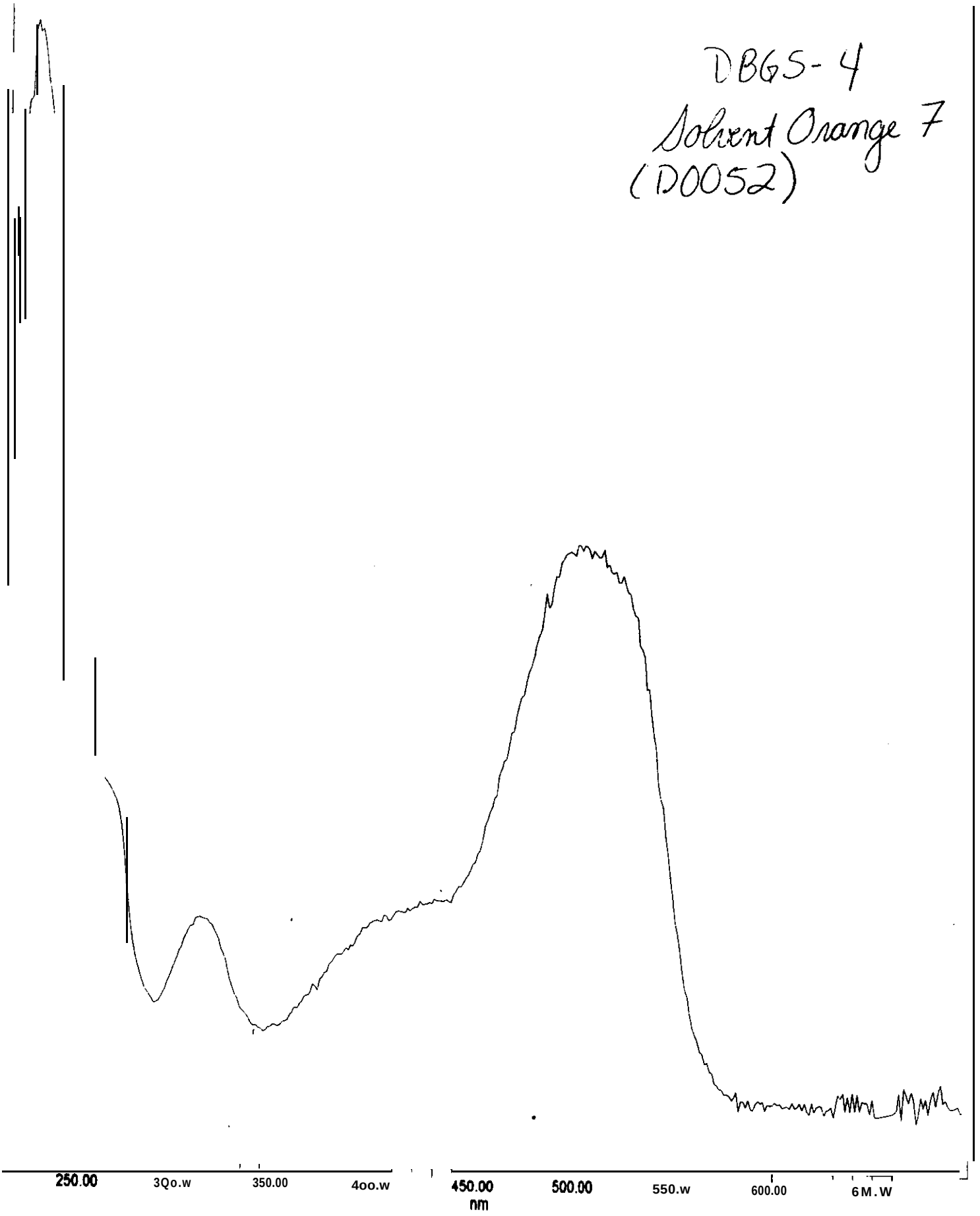
DBGS-4
Disperse Red 9
additive (D0043)



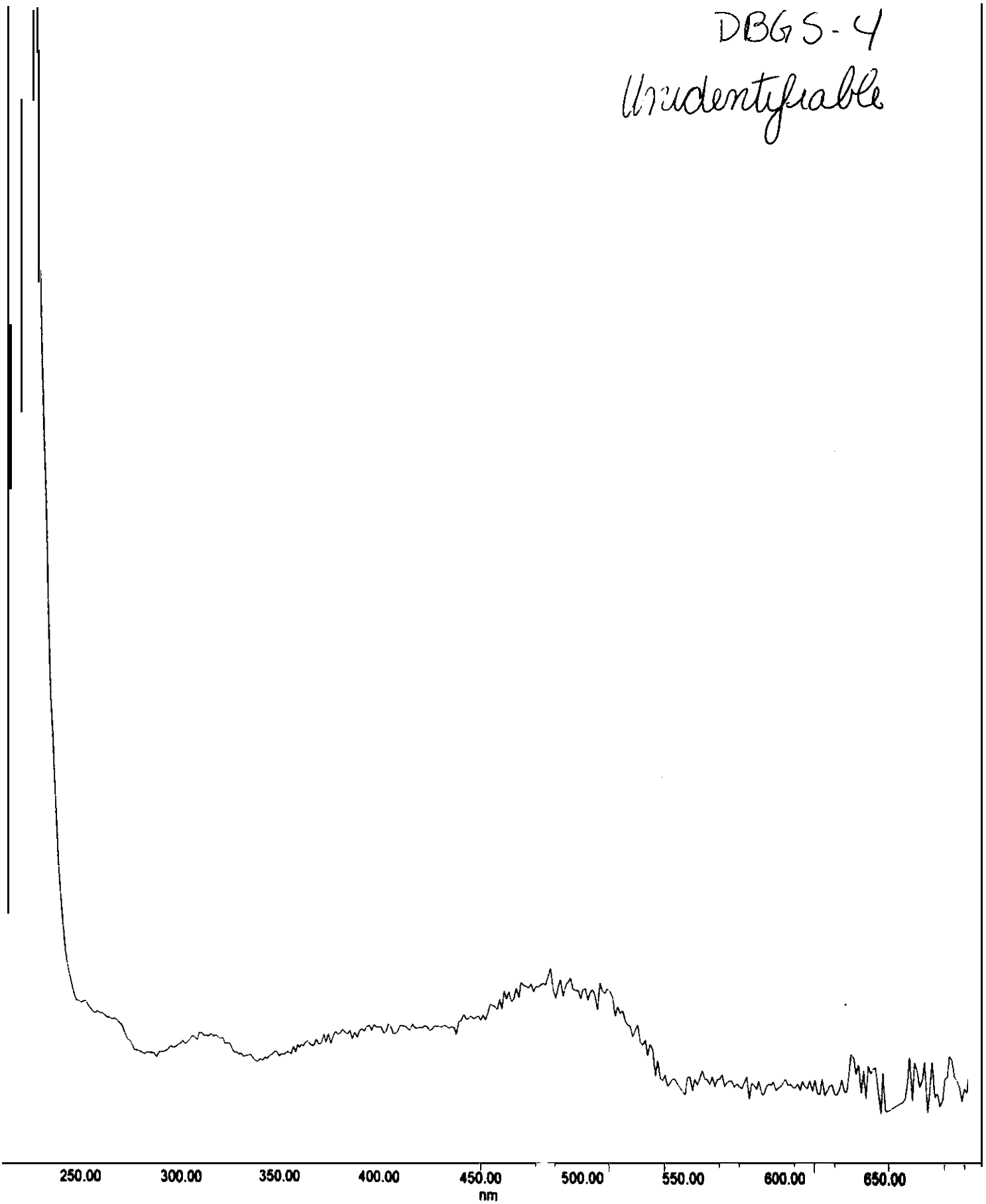
DBGS-4
Unidentifiable

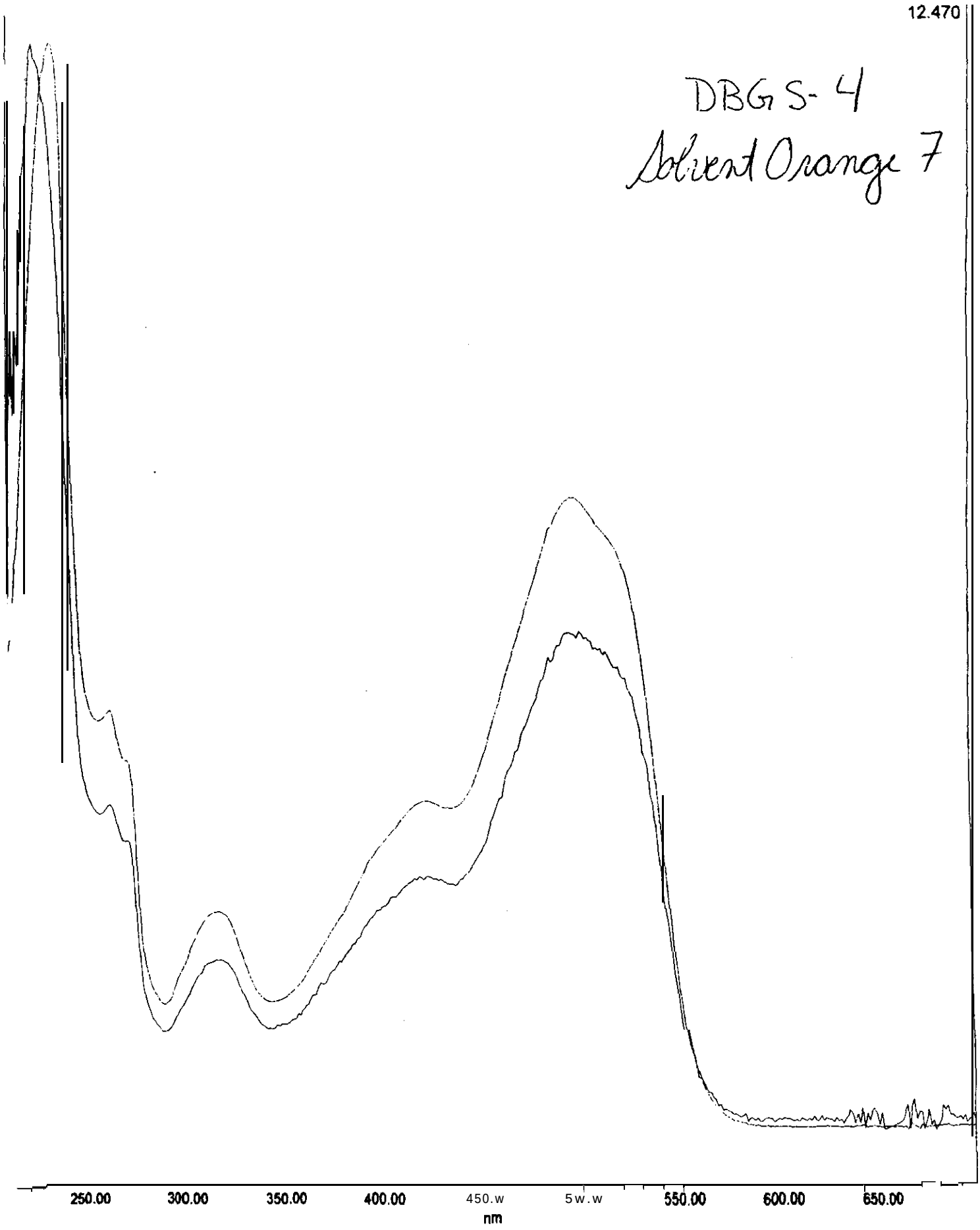


DBGS-4
Solvent Orange 7
(D0052)

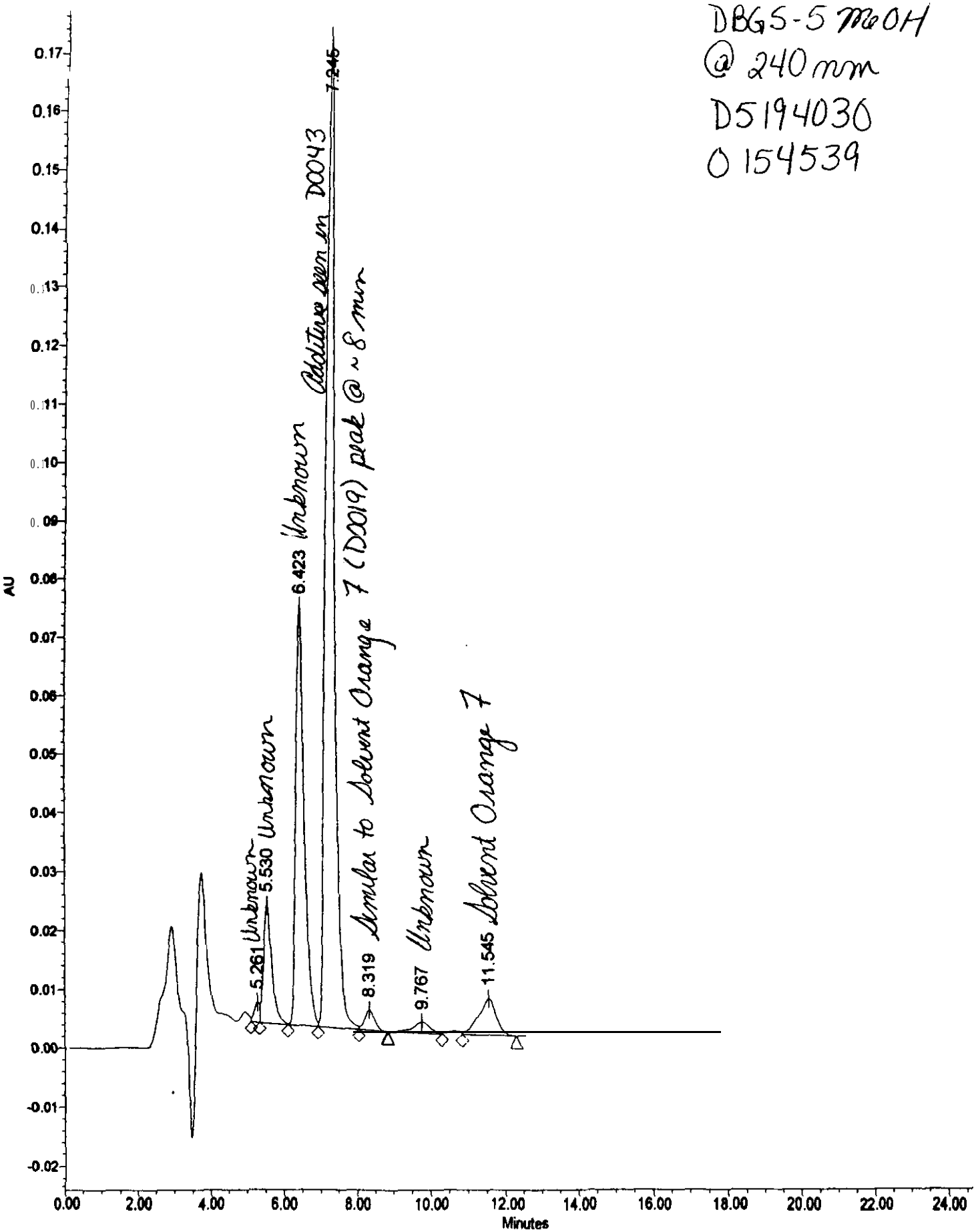


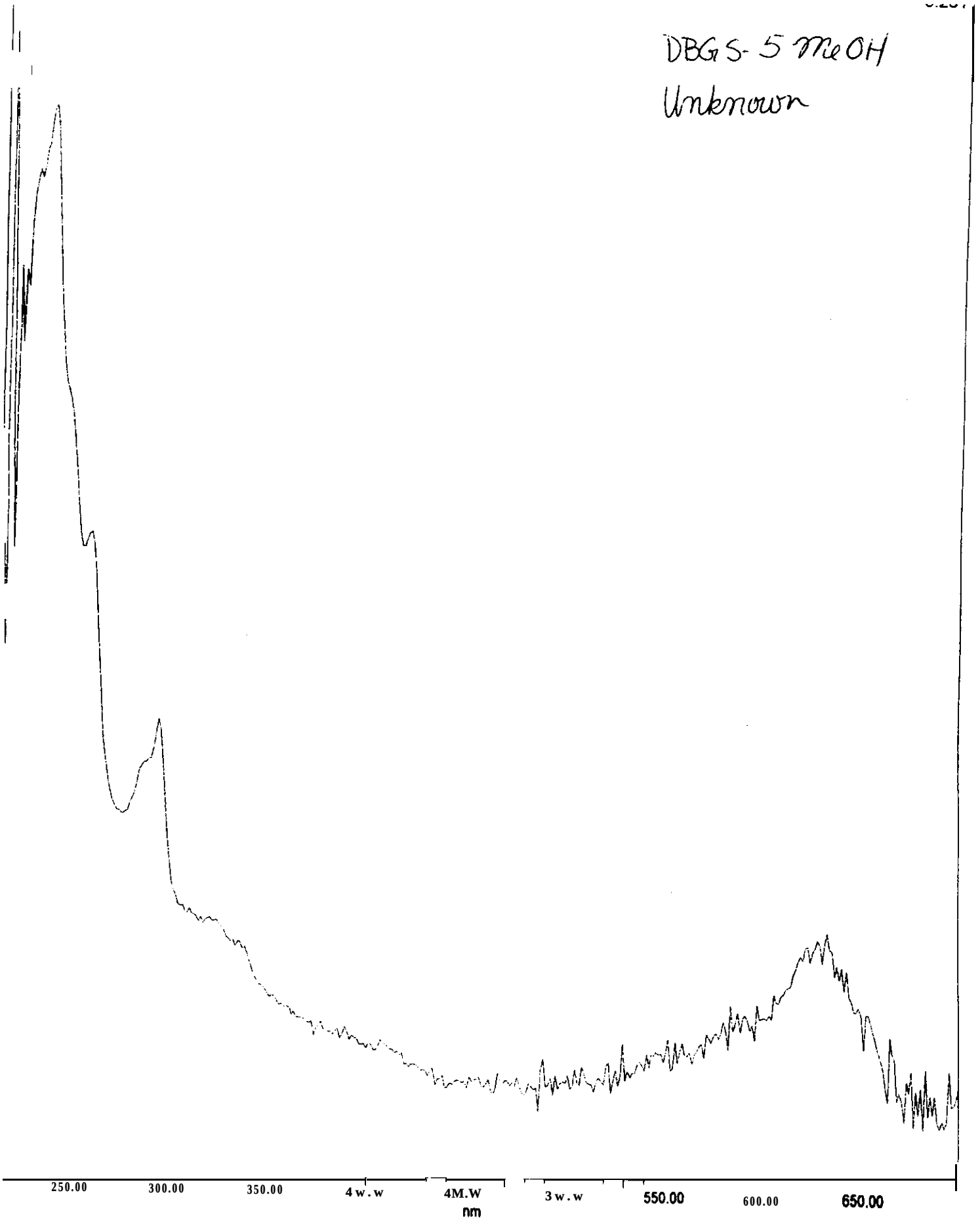
DBGS-4
Unidentifiable

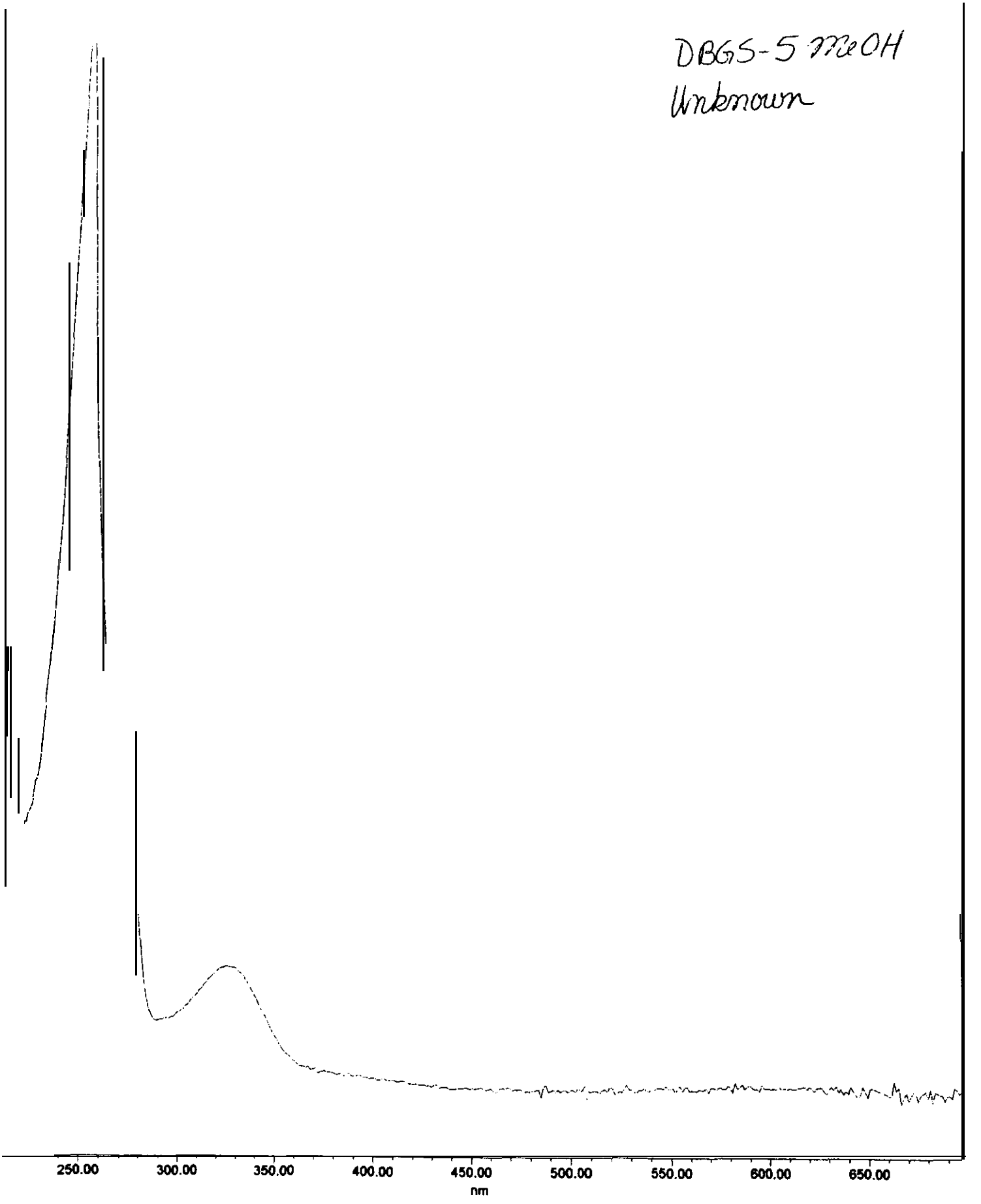


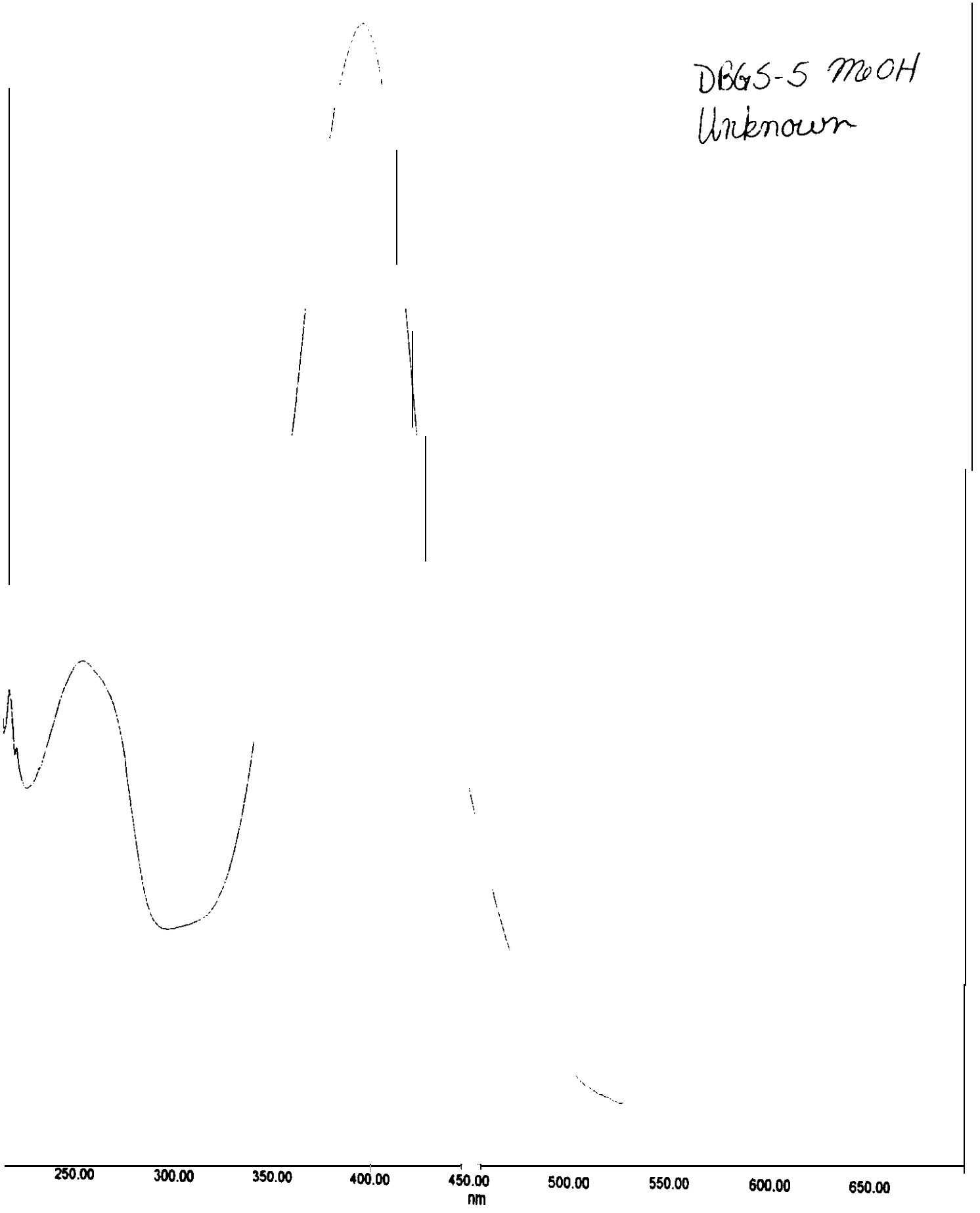

12.470
DBG S-4
Solvent Orange 7
250.00
300.00
350.00
400.00
450.w
5w.w
550.00
600.00
650.00
nm

DBGS-5 MeOH
@ 240 nm
D5194030
0154539




DBGS-5 MeOH
Unknown
250.00
300.00
350.00
4w.w
4M.W
nm
3w.w
550.00
600.00
650.00

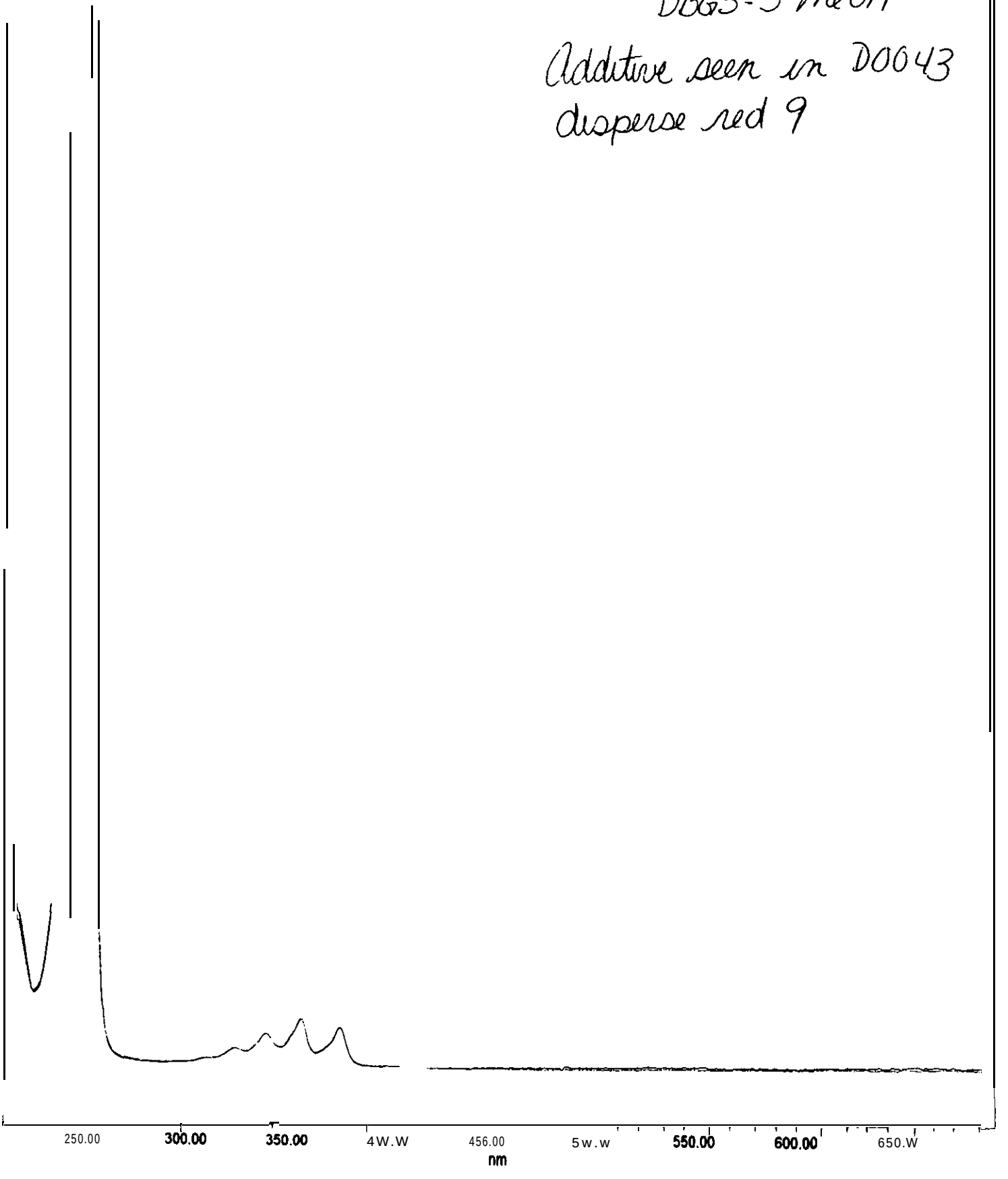

DBGS-5 MeOH
Unknown
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm


DBGS-5 MeOH
Unknown
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

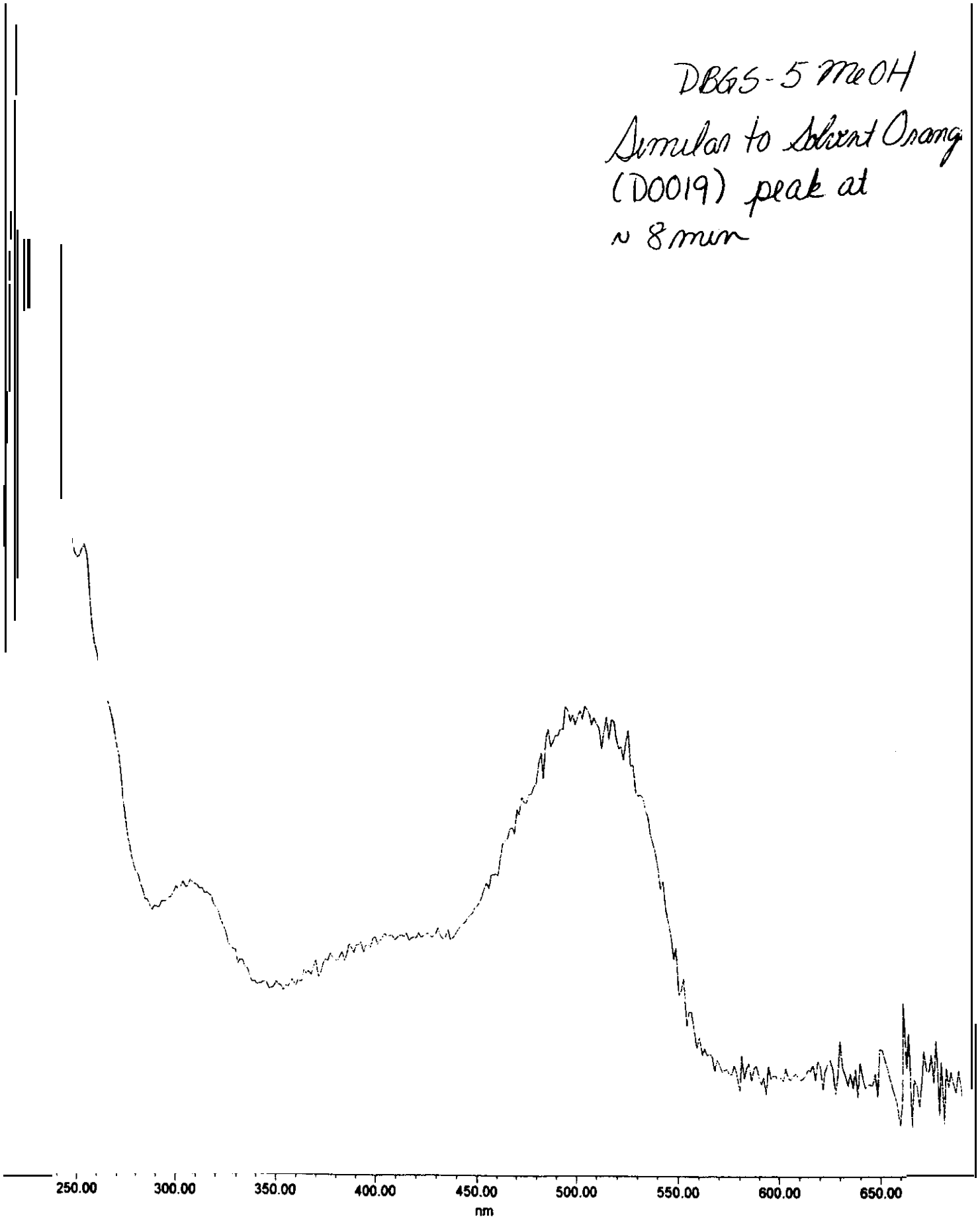
DBGS-5 MeOH

Additive seen in D0043

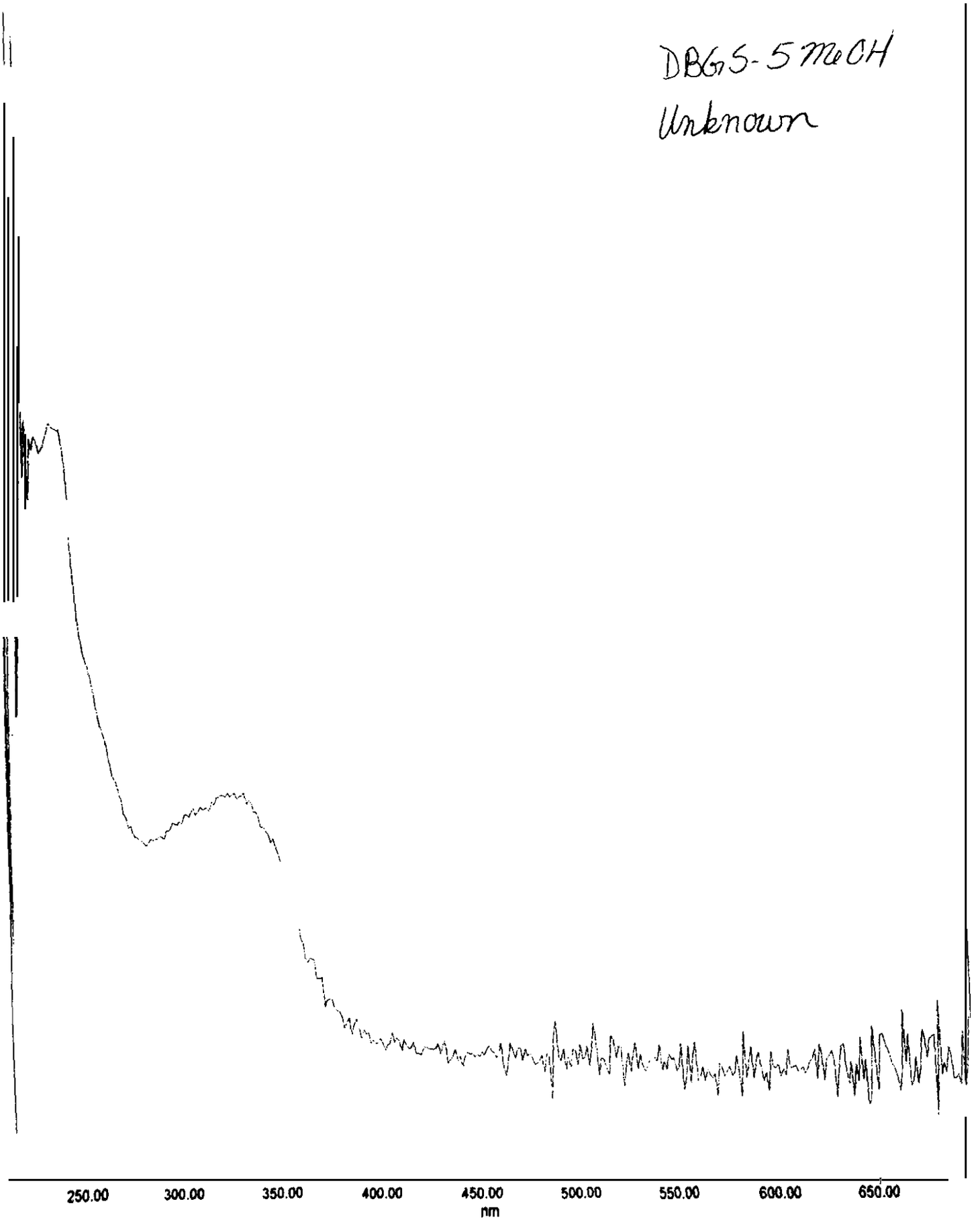
disperse red 9



DBGS-5 MeOH
Similar to Solvent Orange 7
(D0019) peak at
~ 8 min

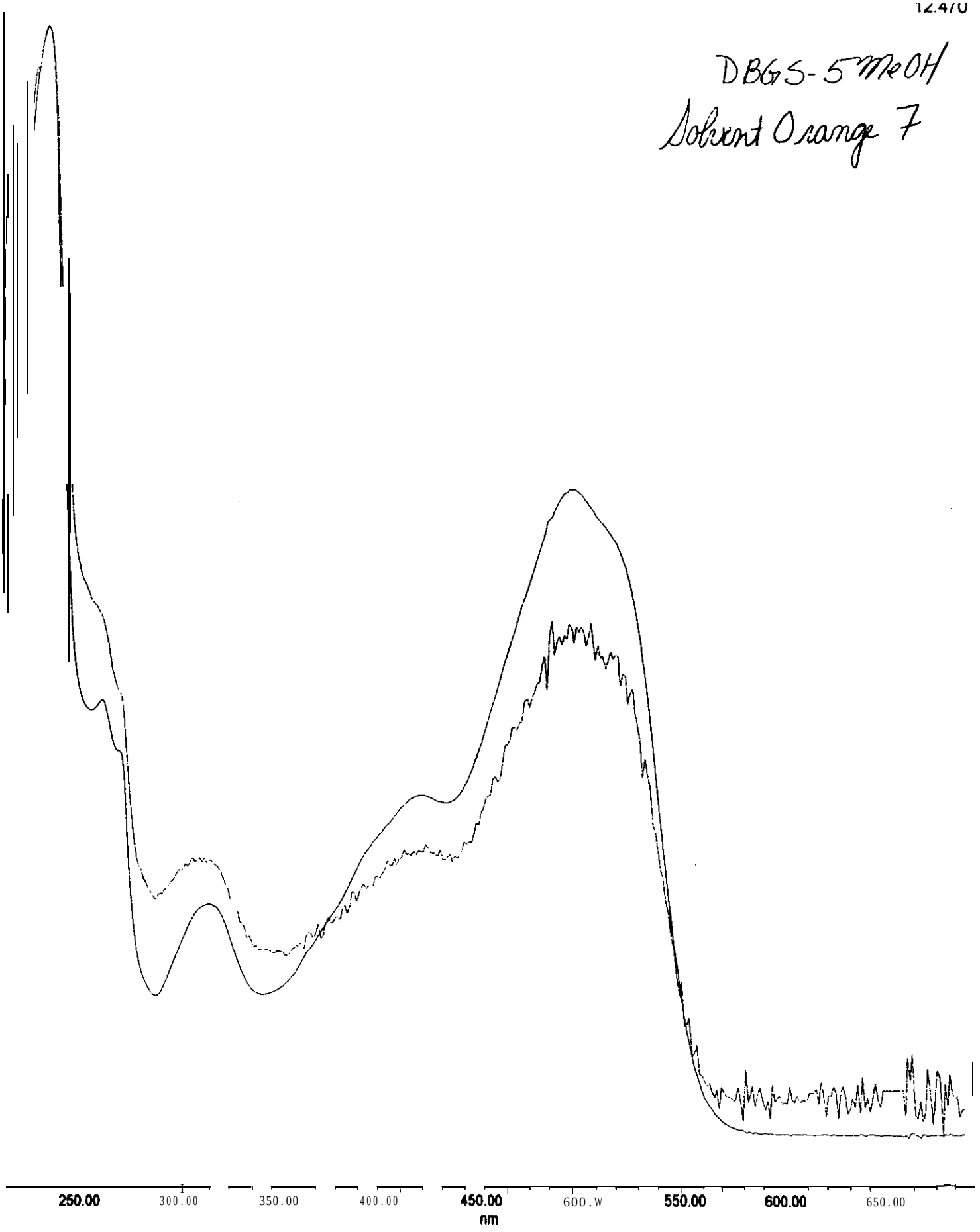


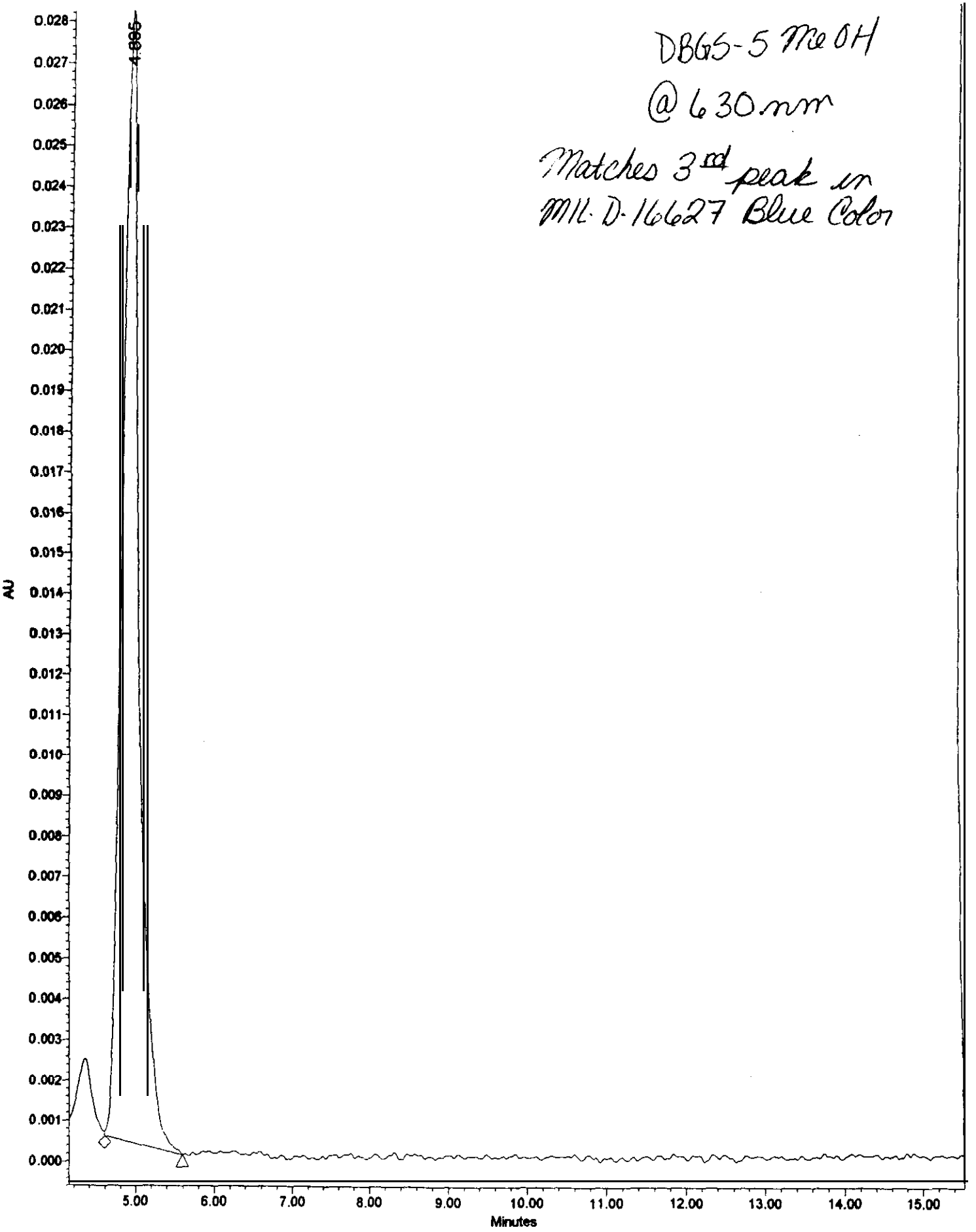
DBGS-5 MeOH
Unknown



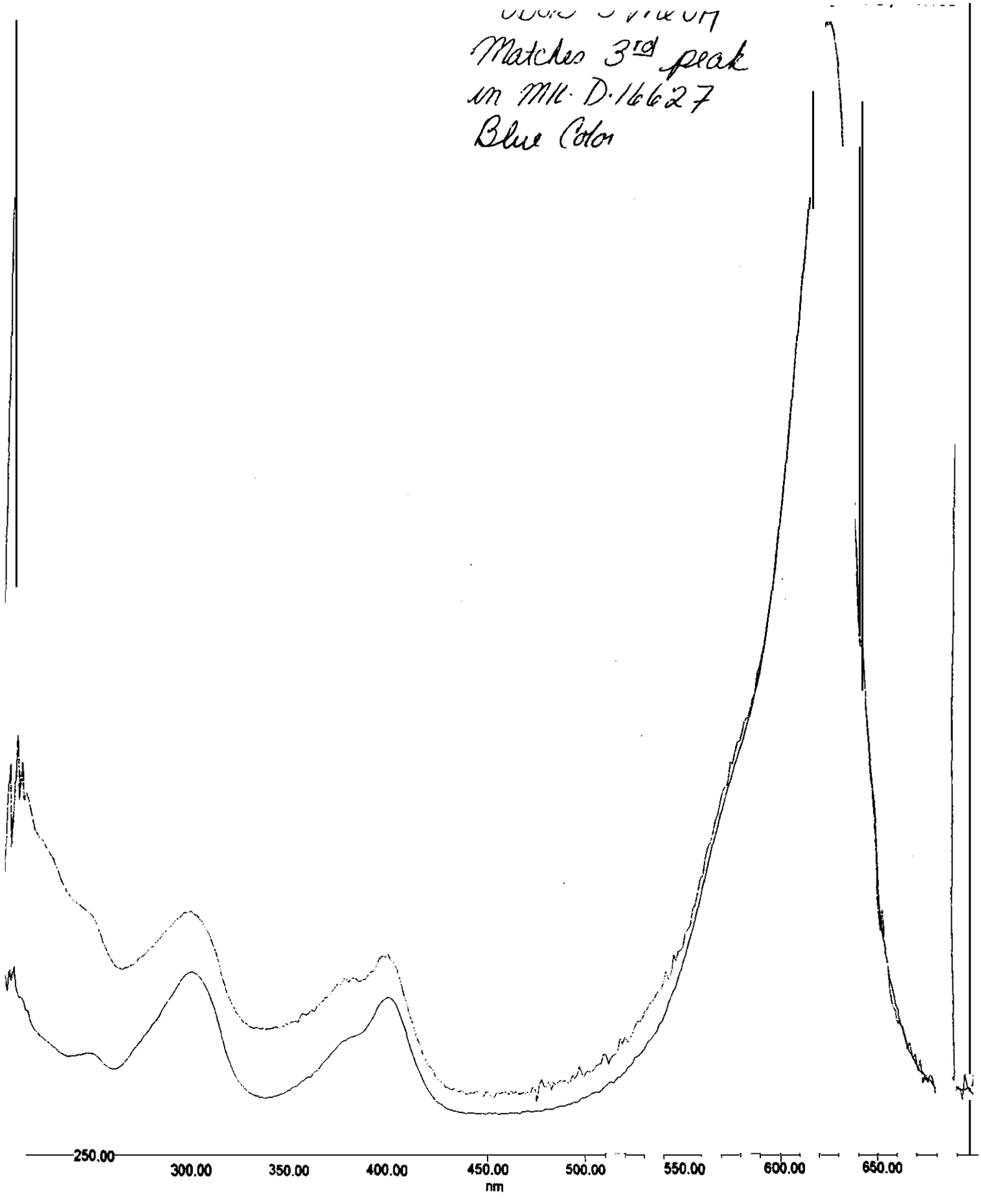
12.470

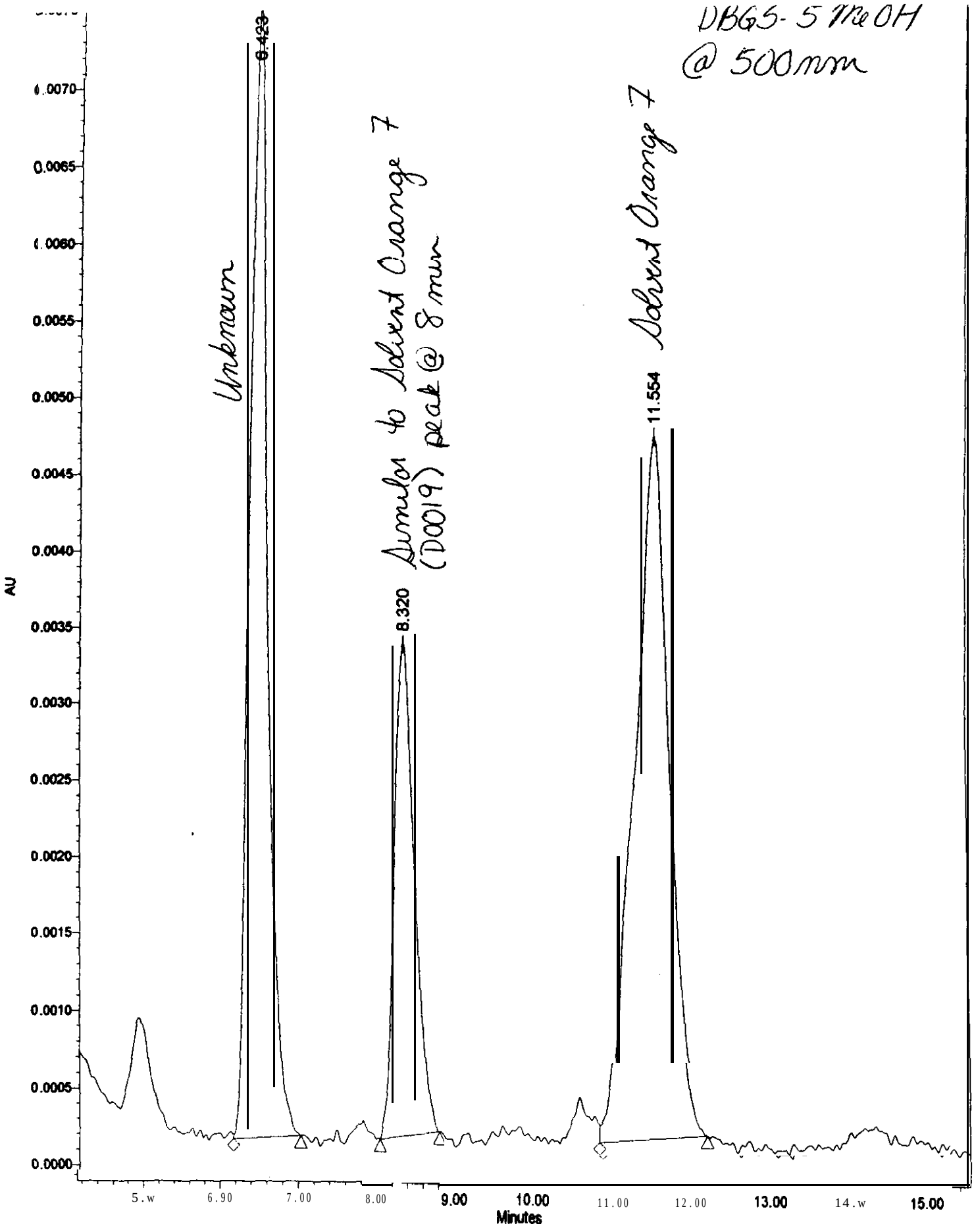
DBGS-5 MeOH
Solvent Orange 7

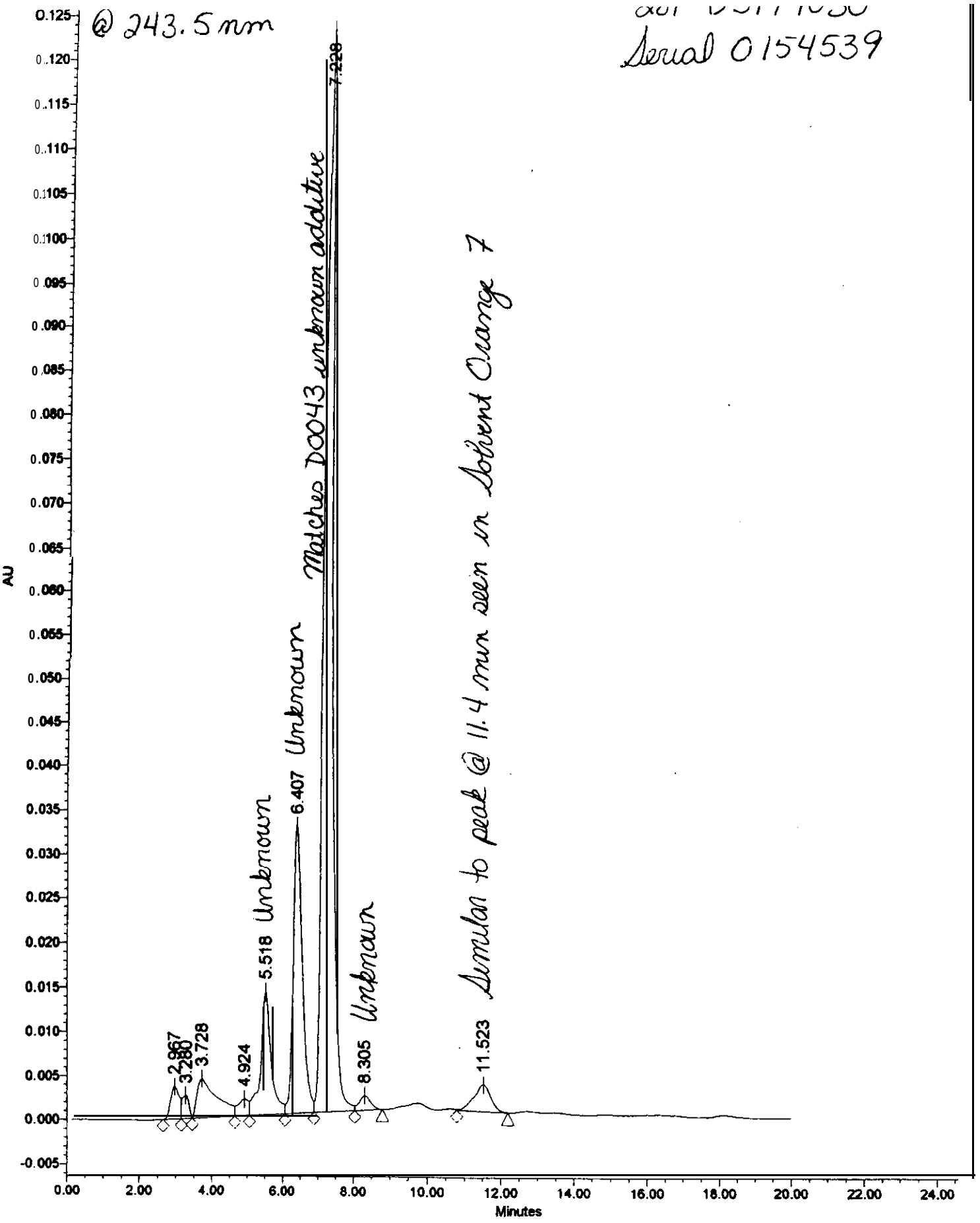




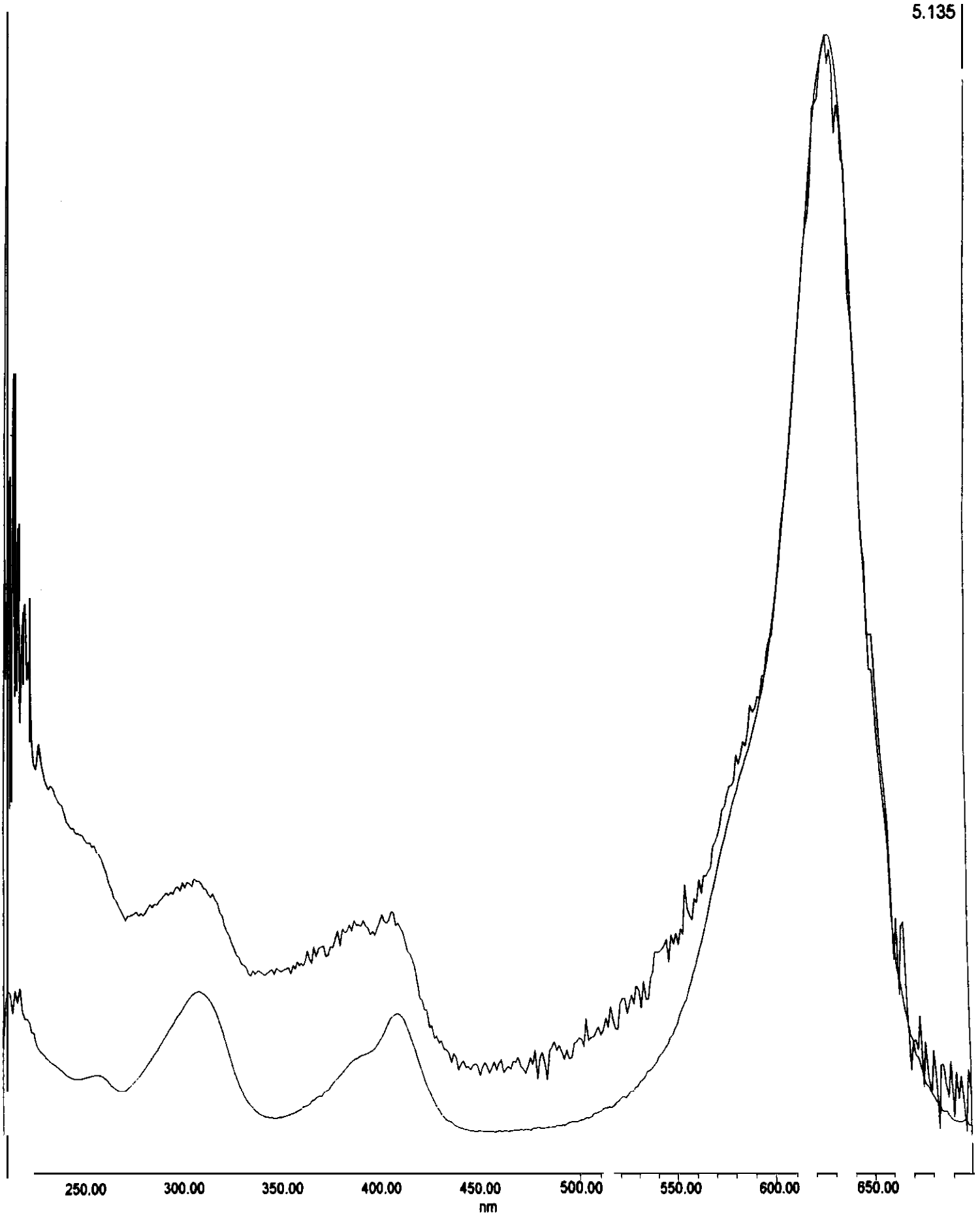
DBG5-5 MeOH
@ 630 nm
Matches 3rd peak in
MIL-D-16627 Blue Color

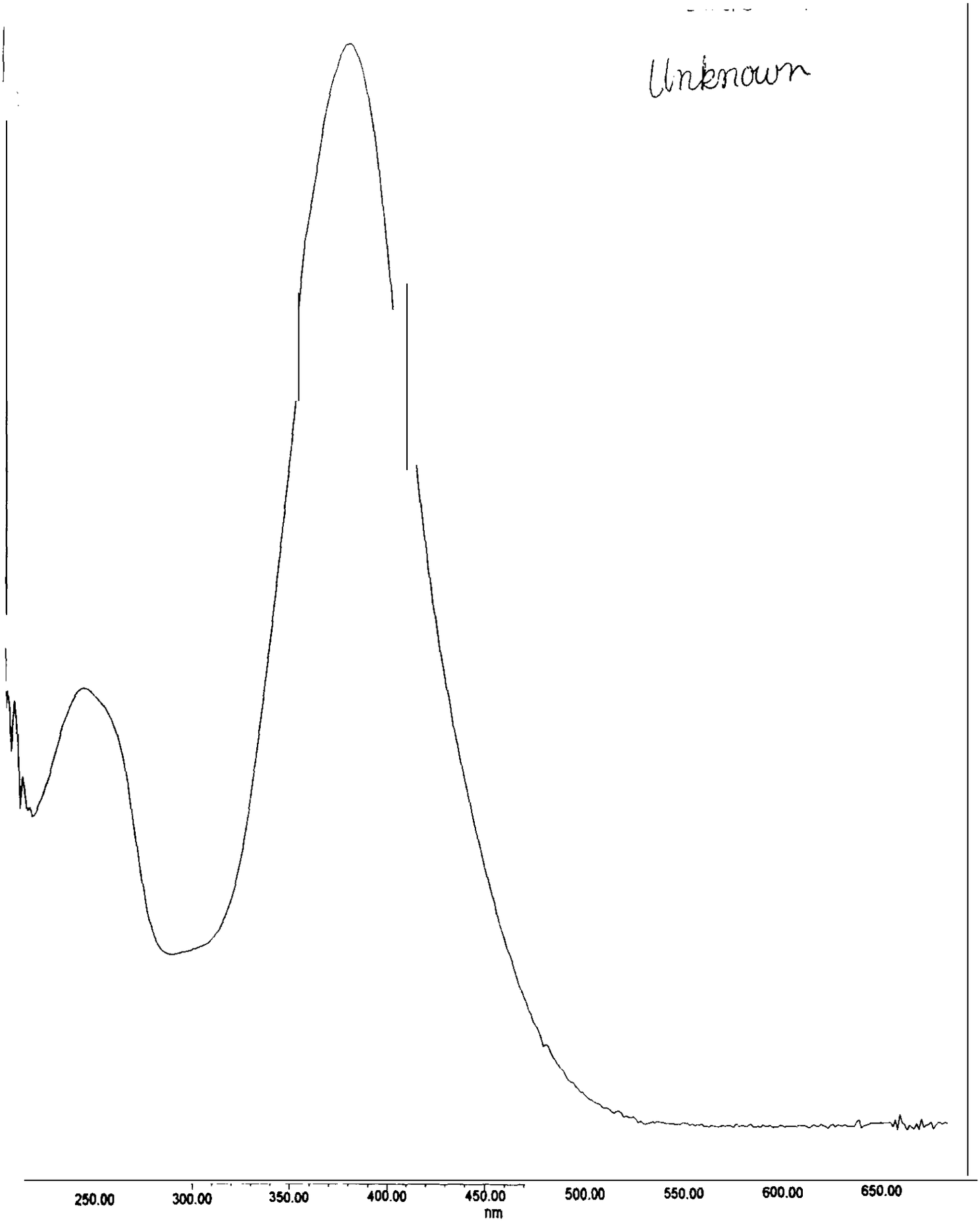

Matches 3rd peak
in Mil. D-16627
Blue Color
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm


DBGS-5 MeOH
@ 500nm
Unknown
6.423
Similar to Solvent Orange 7
(D0019) peak @ 8 min
8.320
Solvent Orange 7
11.554
AU
0.0070
0.0065
0.0060
0.0055
0.0050
0.0045
0.0040
0.0035
0.0030
0.0025
0.0020
0.0015
0.0010
0.0005
0.0000
5.w
6.90
7.00
8.00
9.00
10.00
11.00
12.00
13.00
14.w
15.00
Minutes

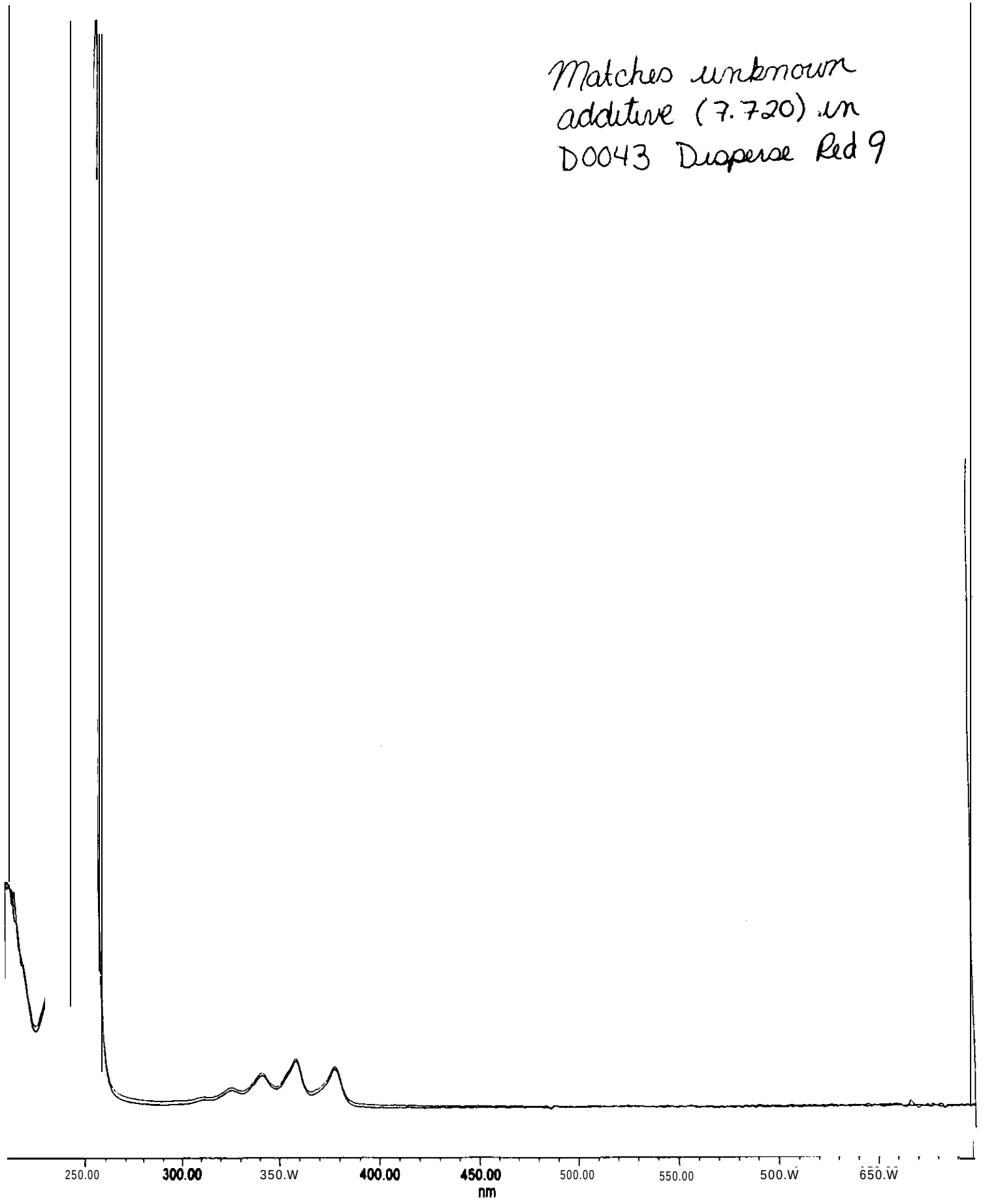

@ 243.5 nm
Serial 0154539
Matches D0043 unknown additive
Unknown
Unknown
Unknown
Similar to peak @ 11.4 min seen in Solvent Orange 7
2.967
3.280
3.728
4.924
5.518
6.407
7.228
8.305
11.523
AU
Minutes

Solvent Orange 7 — 12.470
DBGS-5 MeOH
Solvent Orange 7
Solvent Orange 7
DBGS-5 MeOH
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

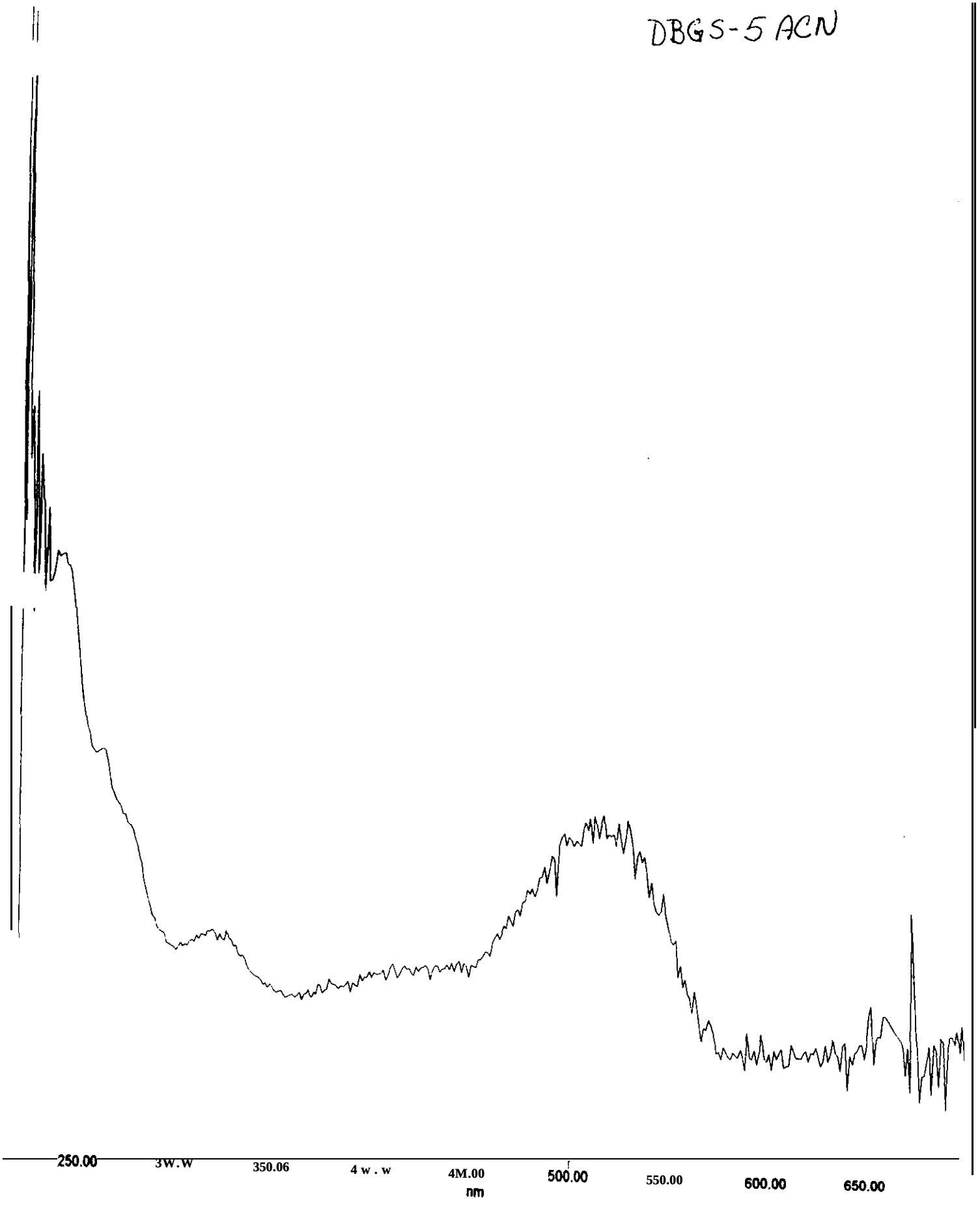

5.135
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm


Unknown
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

Matches unknown additive (7.720) in D0043 Disperse Red 9

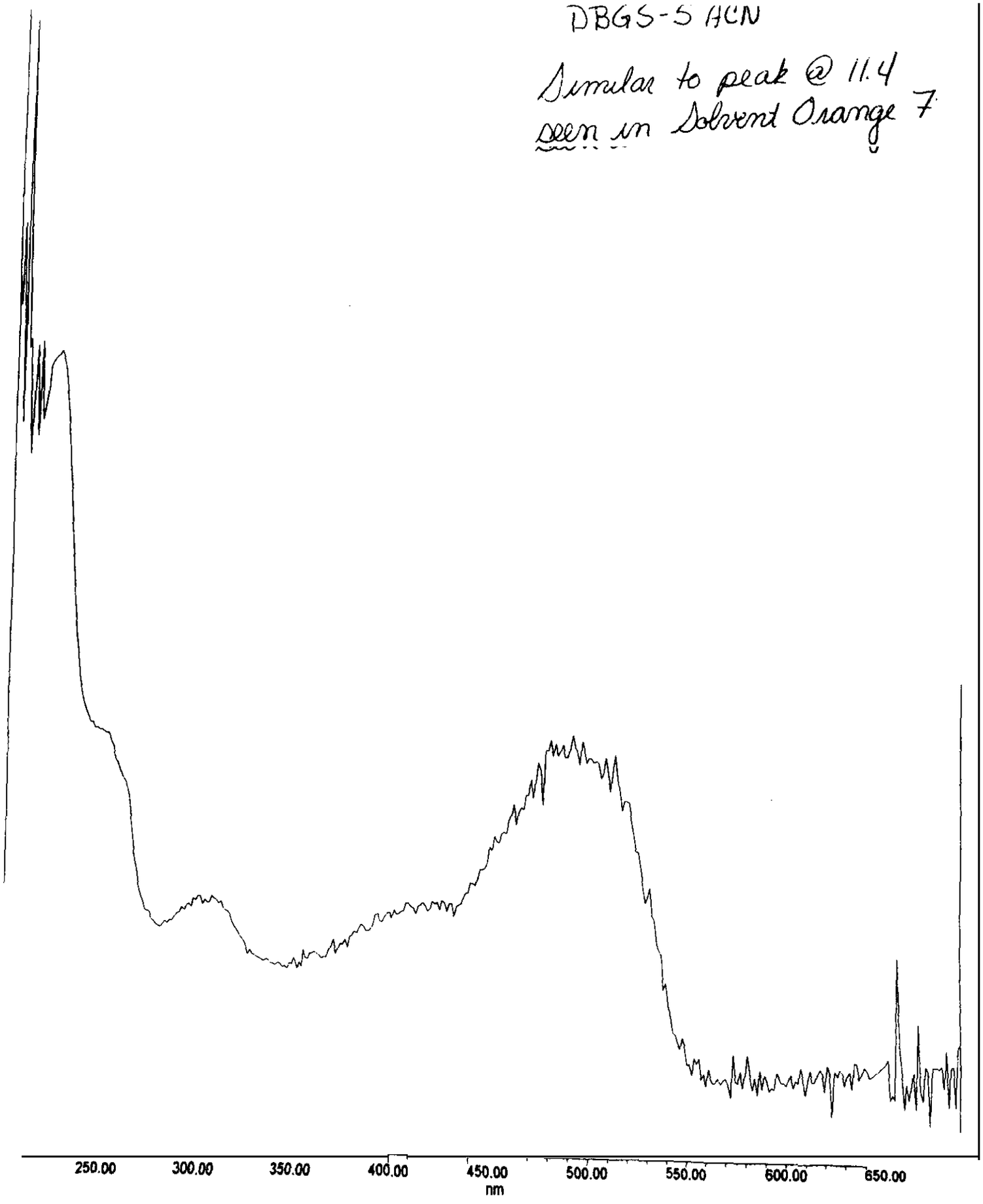


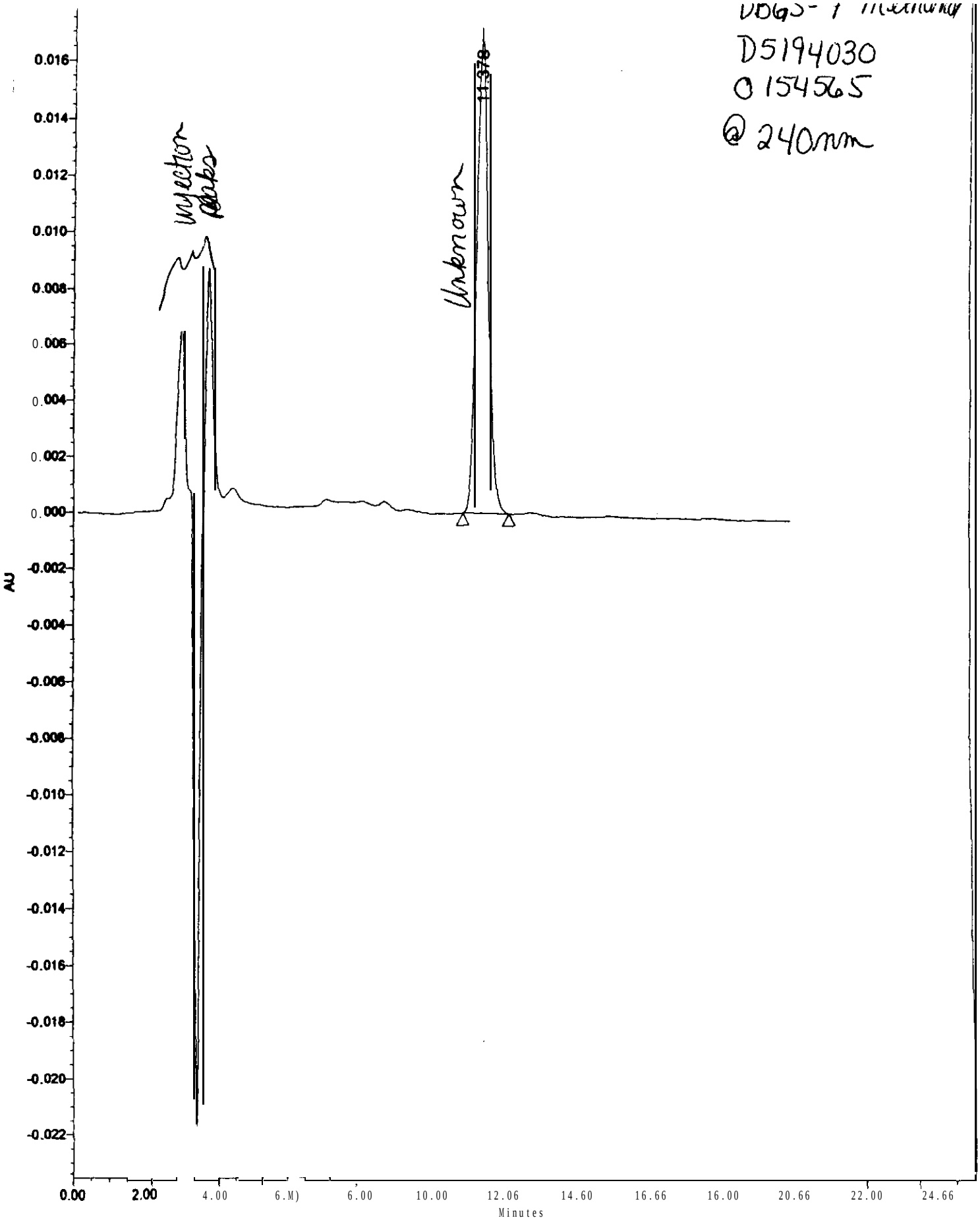
DBGS-5 ACN

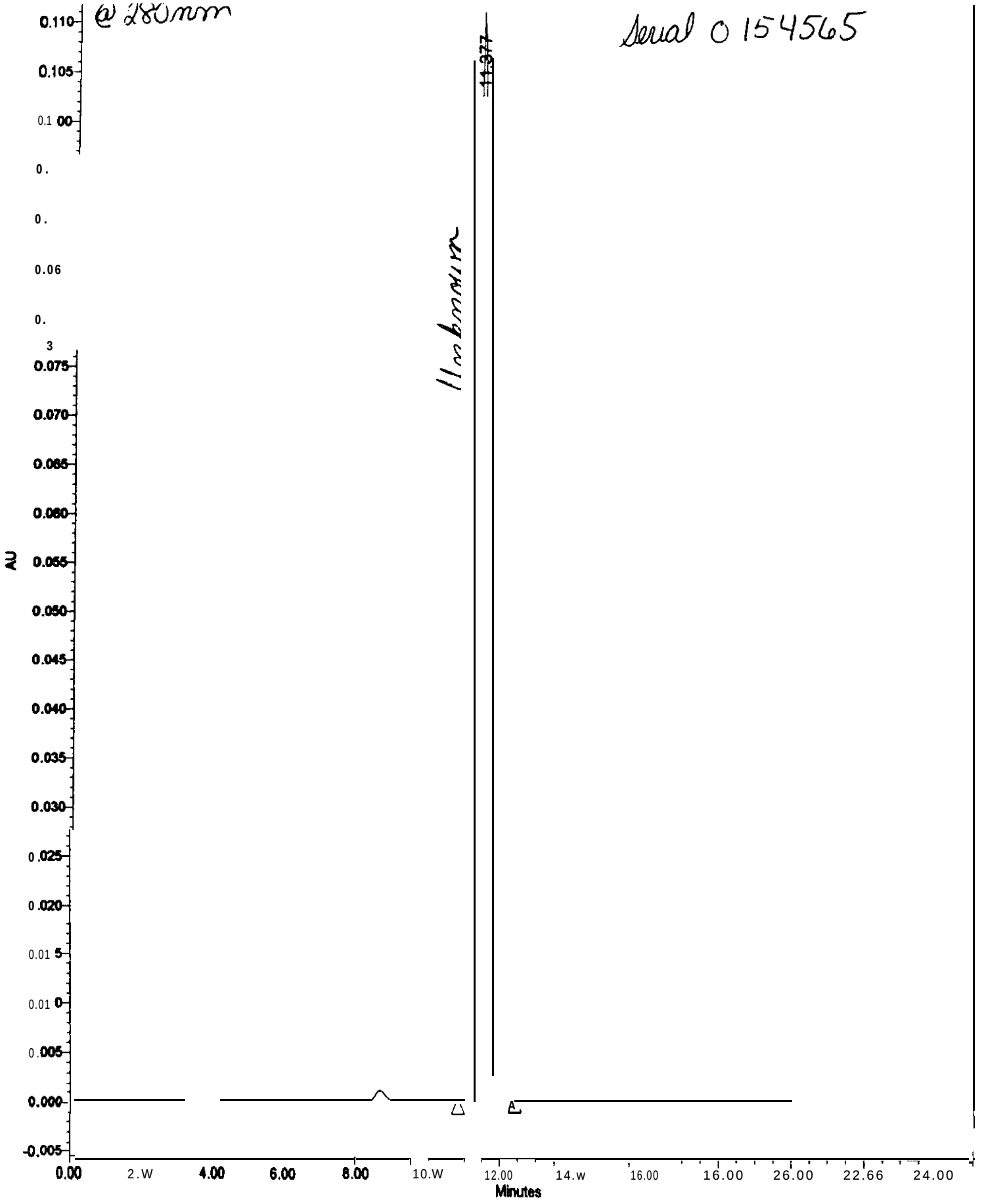


DBGS-5 ACN

Similar to peak @ 11.4 seen in Solvent Orange 7

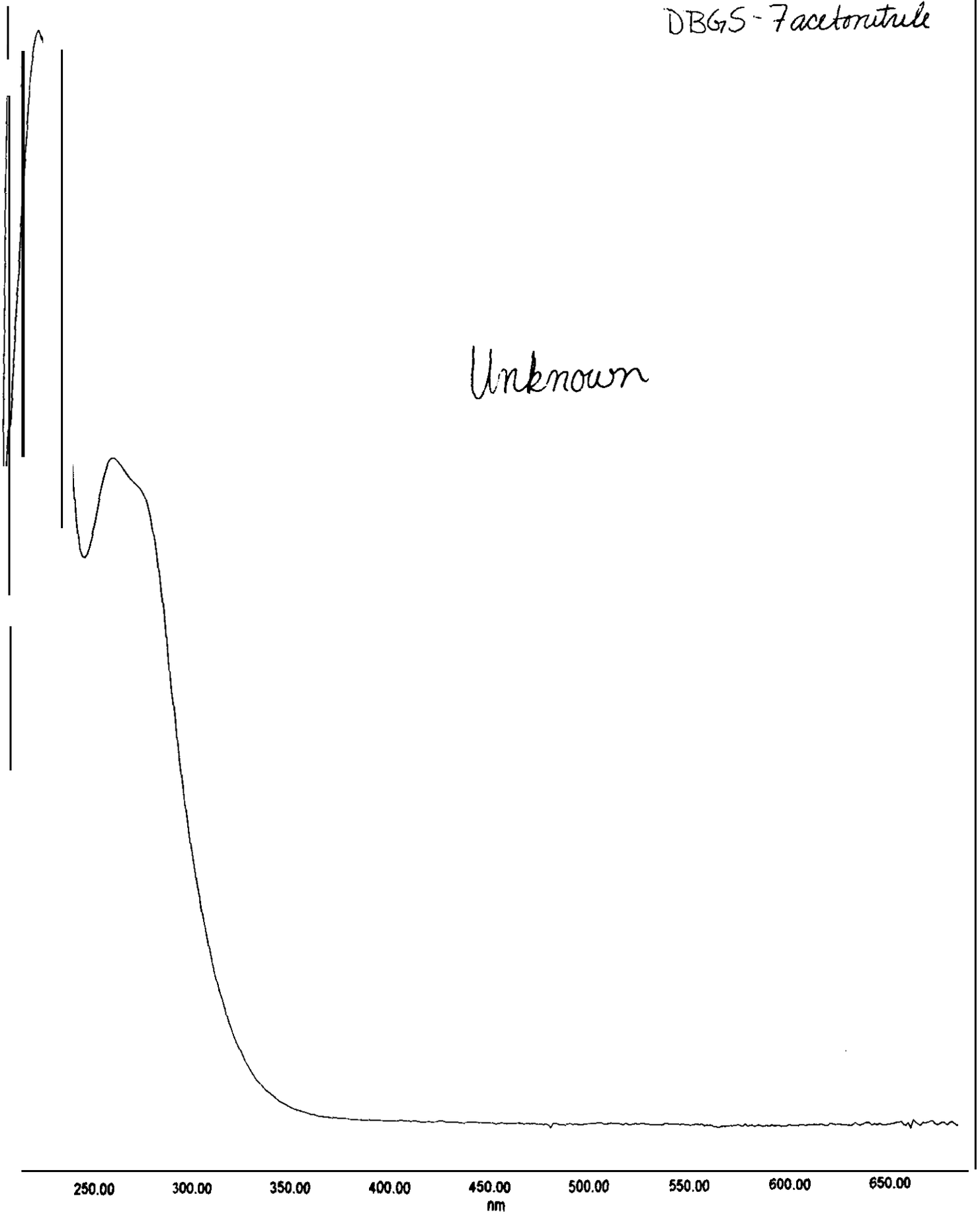



D5194030
0154565
@ 240nm
Injection Blank
Unknown
11.378
AU
0.016
0.014
0.012
0.010
0.008
0.006
0.004
0.002
0.000
-0.002
-0.004
-0.006
-0.008
-0.010
-0.012
-0.014
-0.016
-0.018
-0.020
-0.022
0.00
2.00
4.00
6.00
10.00
12.06
14.60
16.66
16.00
20.66
22.00
24.66
Minutes

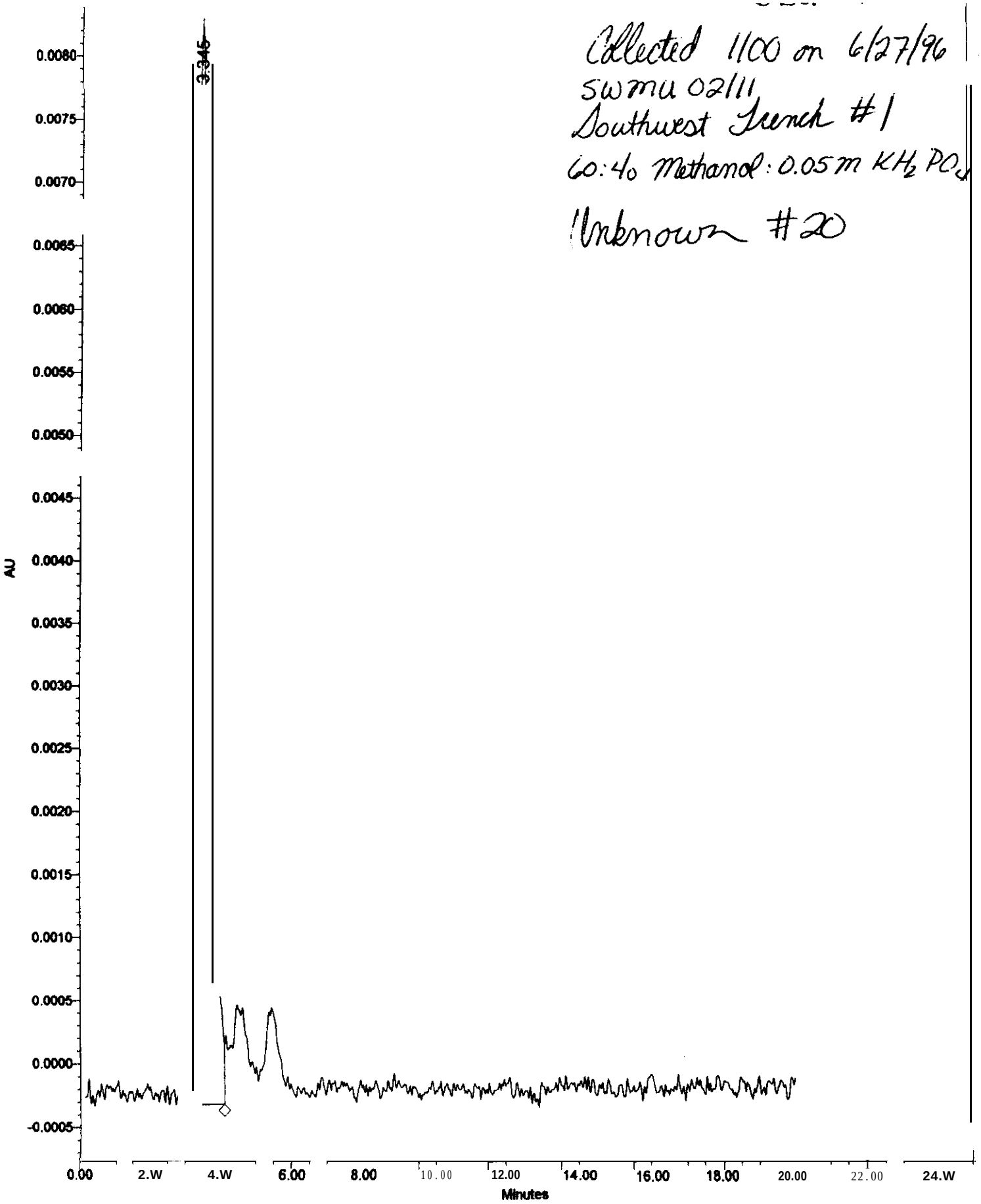

@ 280nm
Serial 0 154565
11.377
Minimum
AU
0.110
0.105
0.100
0.
0.
0.06
0.
3
0.075
0.070
0.065
0.060
0.055
0.050
0.045
0.040
0.035
0.030
0.025
0.020
0.015
0.010
0.005
0.000
-0.005
0.00
2.W
4.00
6.00
8.00
10.W
12.00
14.w
16.00
16.00
26.00
22.66
24.00
Minutes

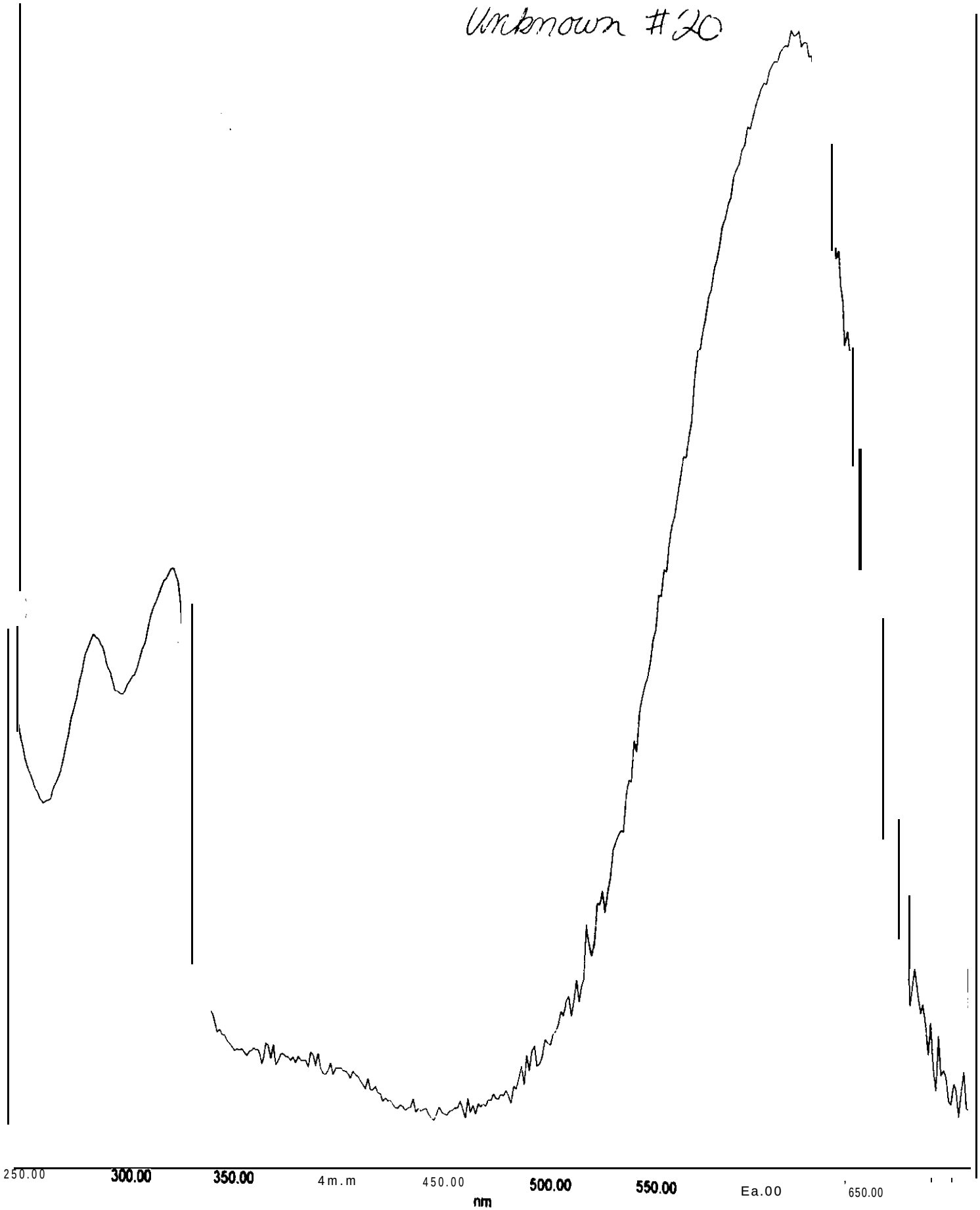
DBGS - 7 acetonitrile

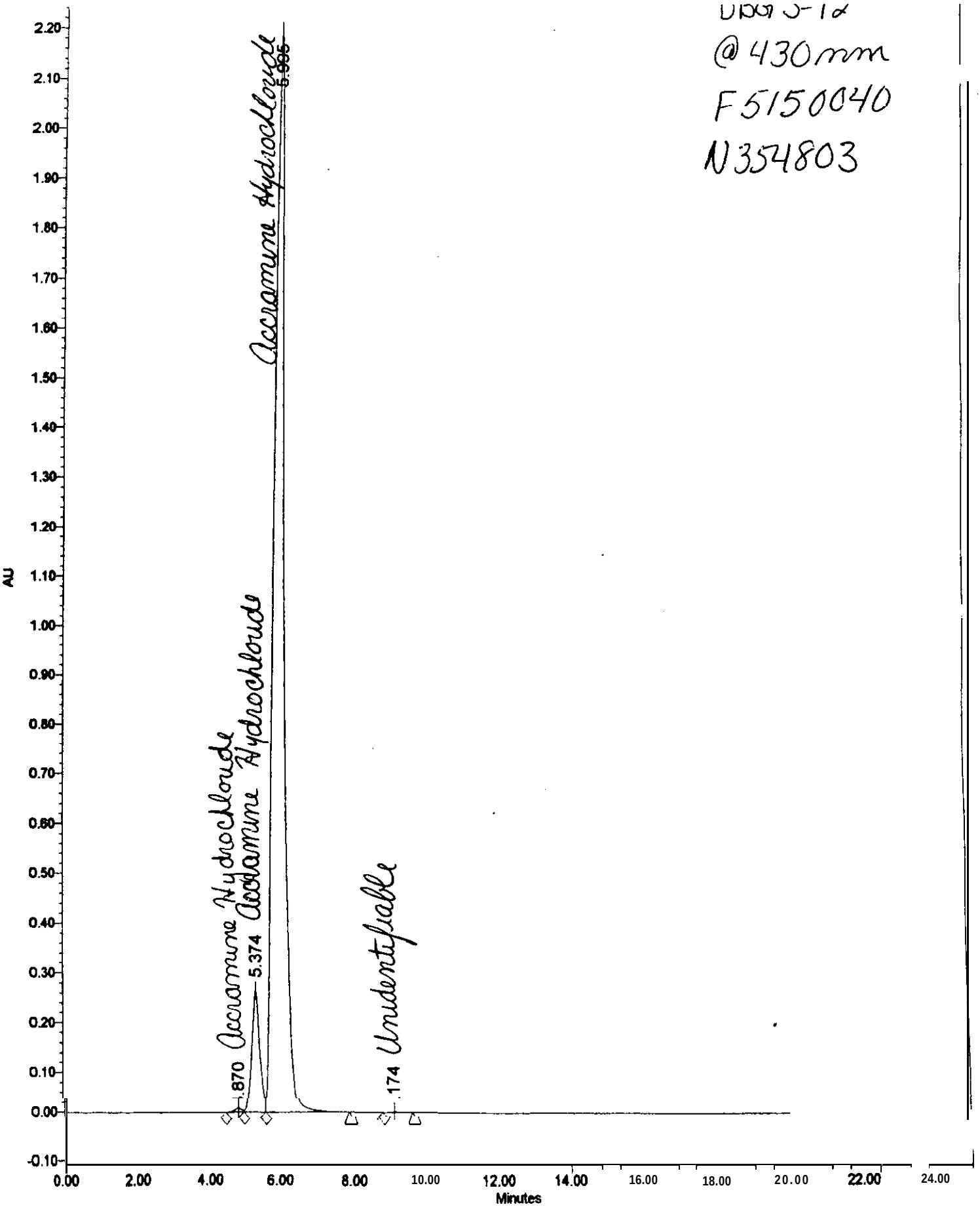
Unknown

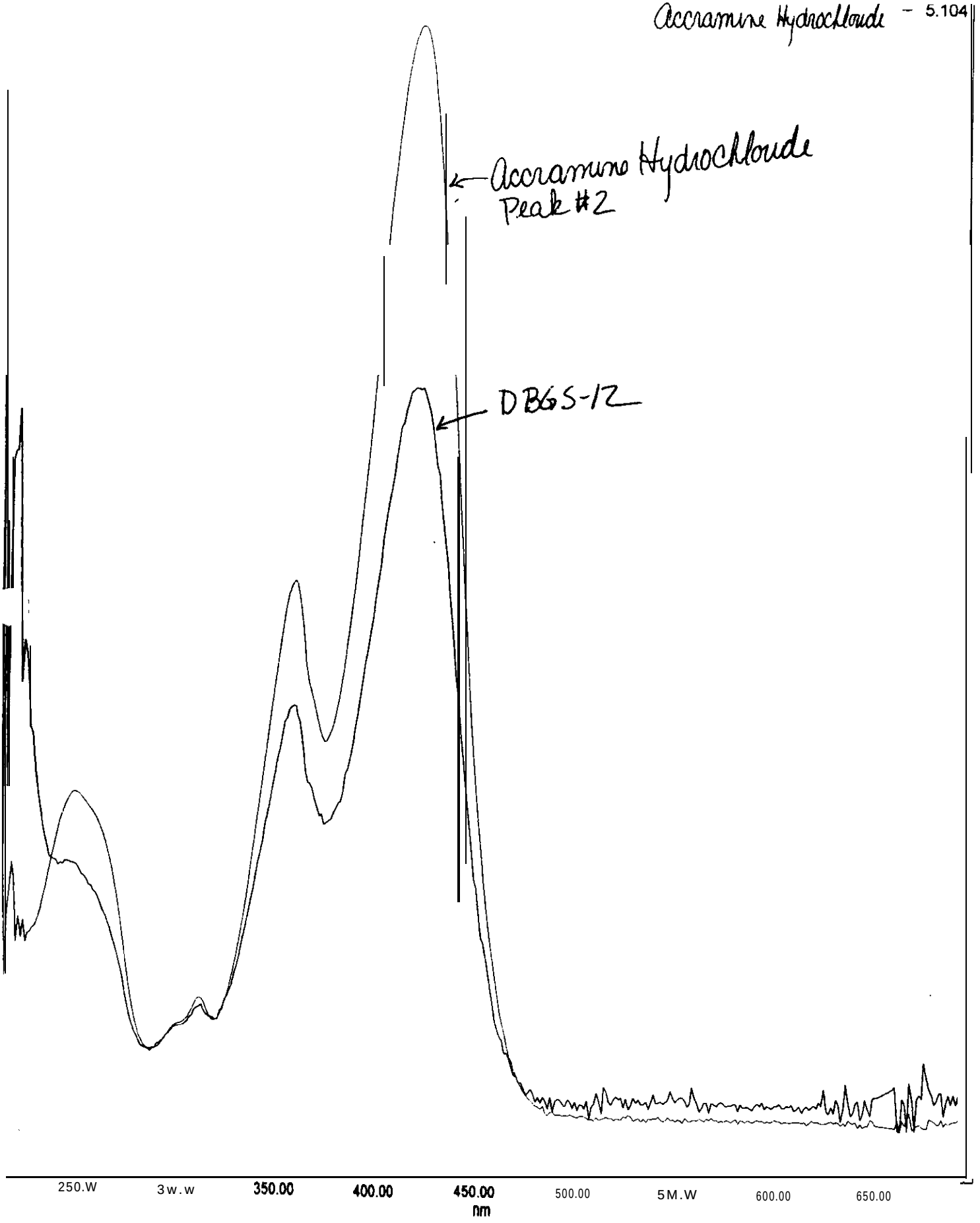


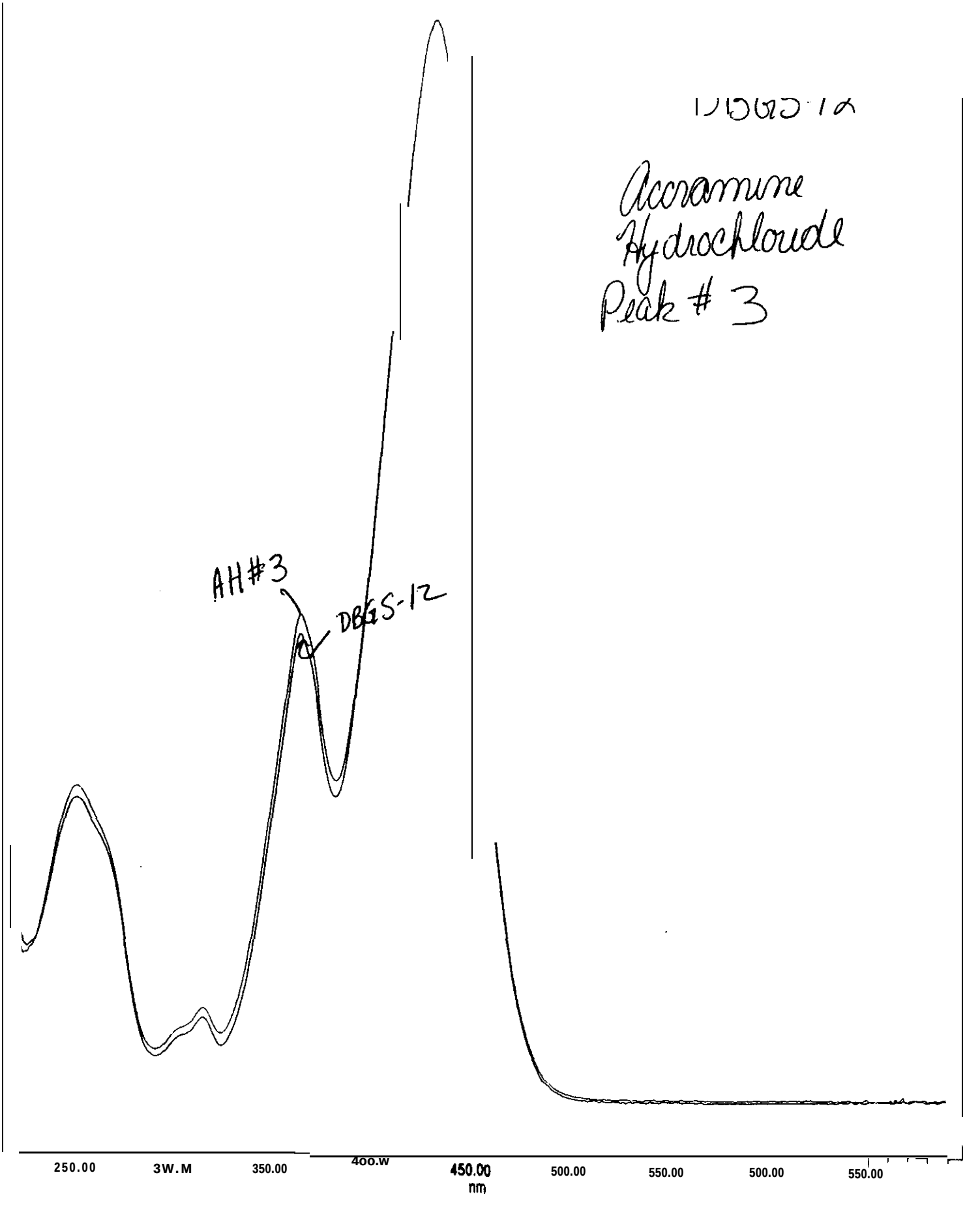
Collected 1100 on 6/27/96
SWMU 02/11
Southwest Trench #1
60:40 Methanol: 0.05m KH_2PO_4
Unknown #20

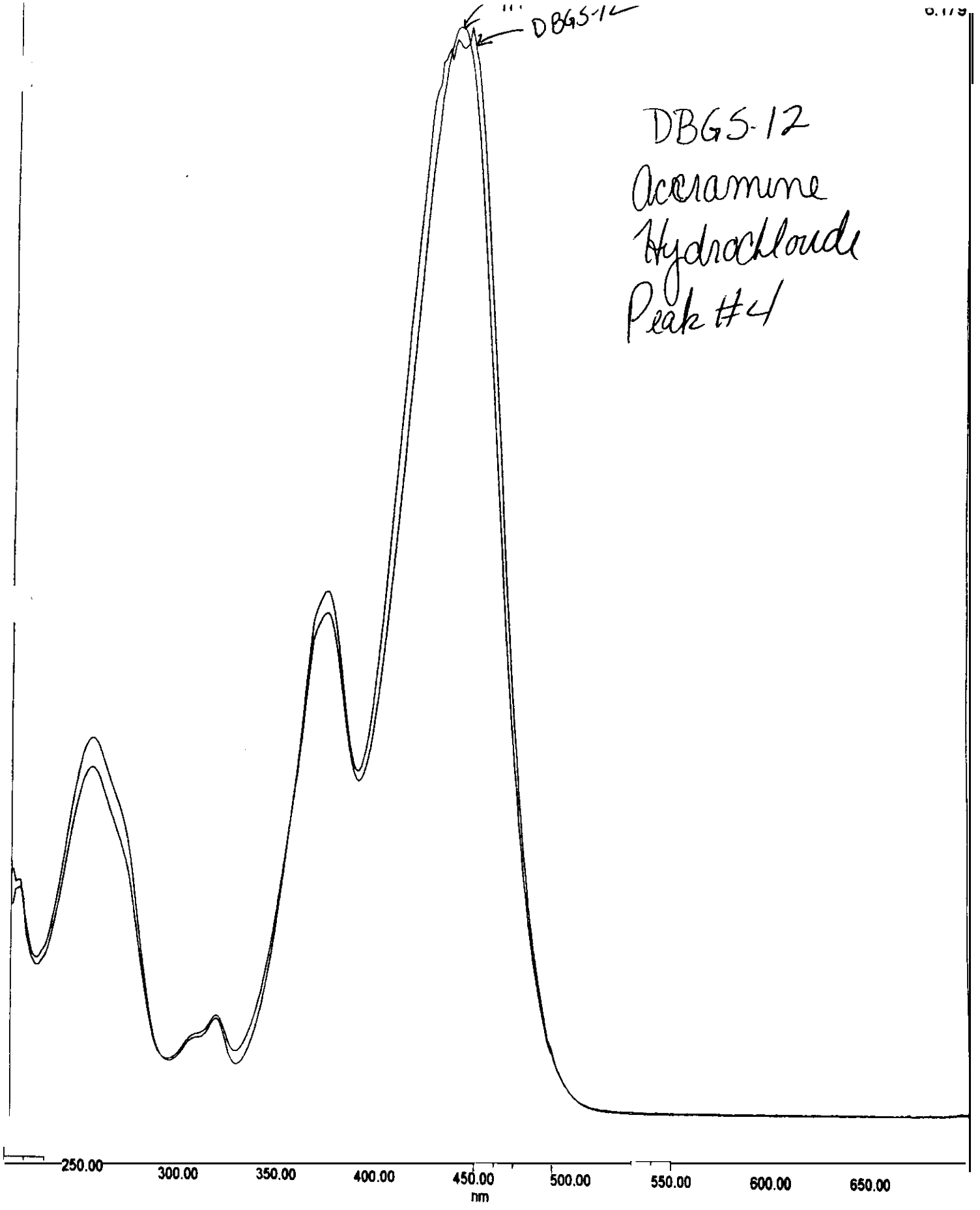



Unknown #20
250.00
300.00
350.00
4m.m
450.00
500.00
550.00
Ea.00
650.00
nm


UDG 5-1α
@430nm
F5150040
N354803
Acramine Hydrochloride
5.995
Acramine Hydrochloride
5.374
Acramine Hydrochloride
.870
Unidentifiable
.174
AU
Minutes

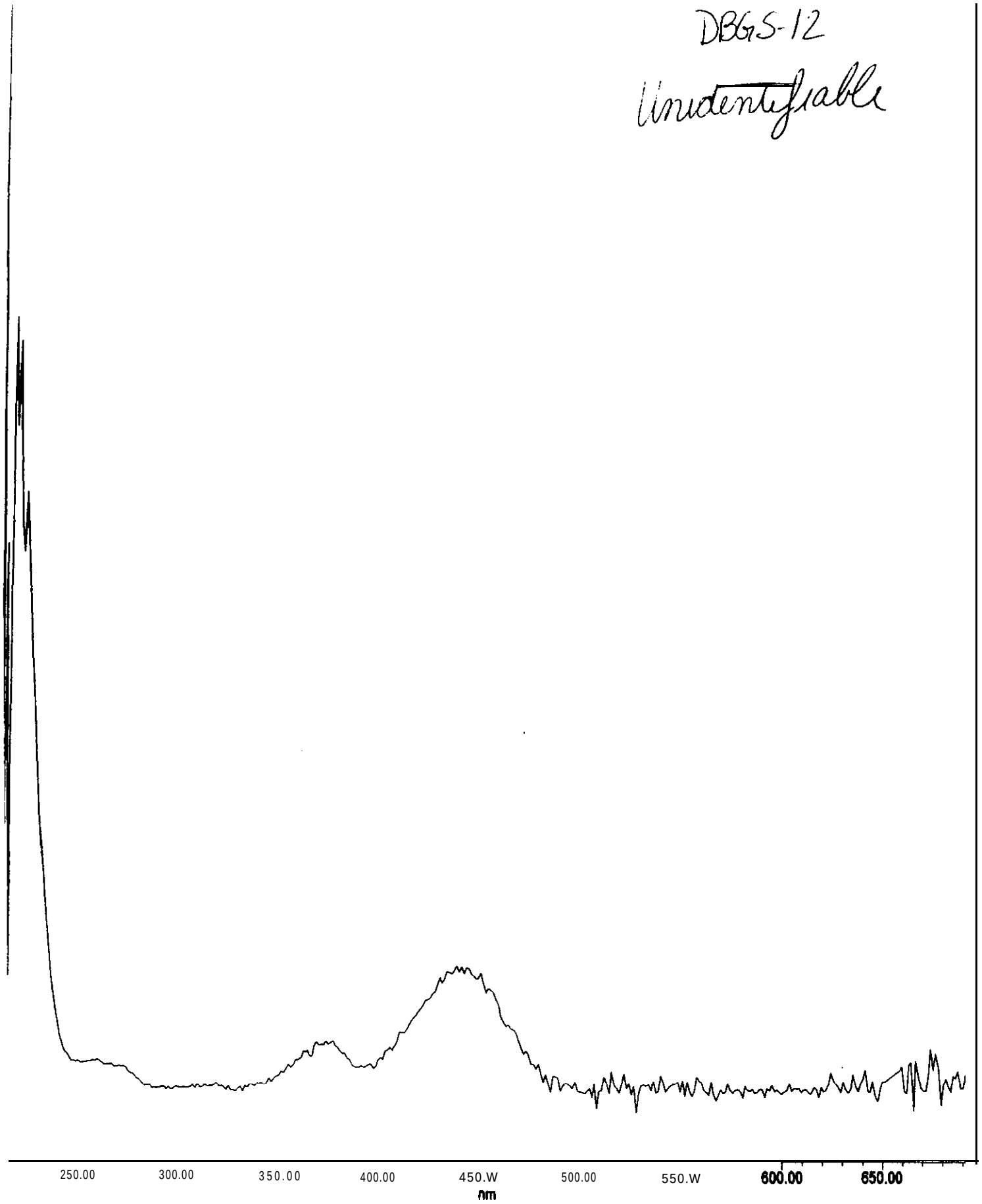

Accramine Hydrochloride - 5.104
Accramine Hydrochloride
Peak #2
DBGS-12
250.W
3w.w
350.00
400.00
450.00
nm
500.00
5M.W
600.00
650.00

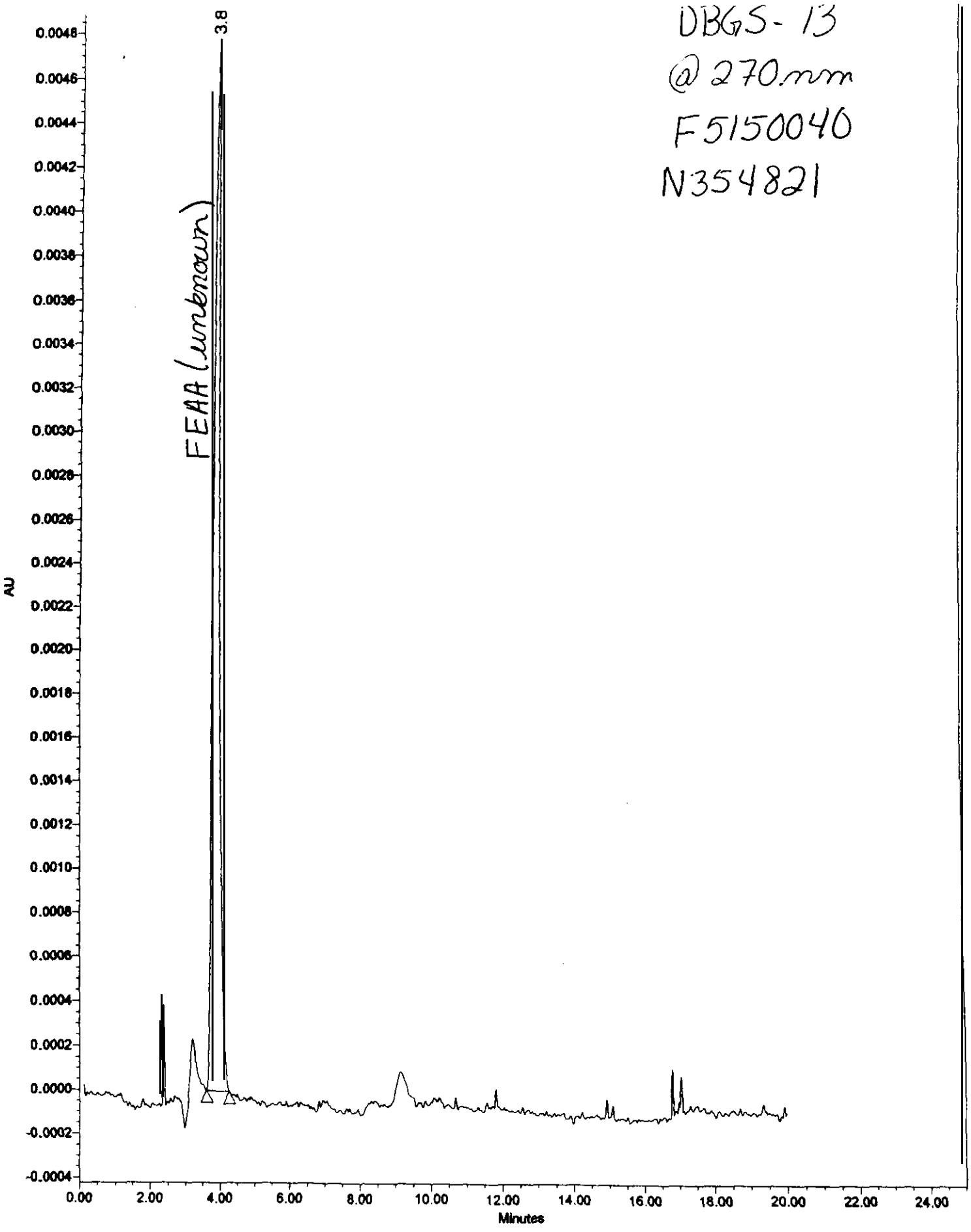

DBGS-12
Acramine
Hydrochloride
Peak # 3
AH#3
DBGS-12
250.00
3W.M
350.00
400.W
450.00
nm
500.00
550.00
500.00
550.00

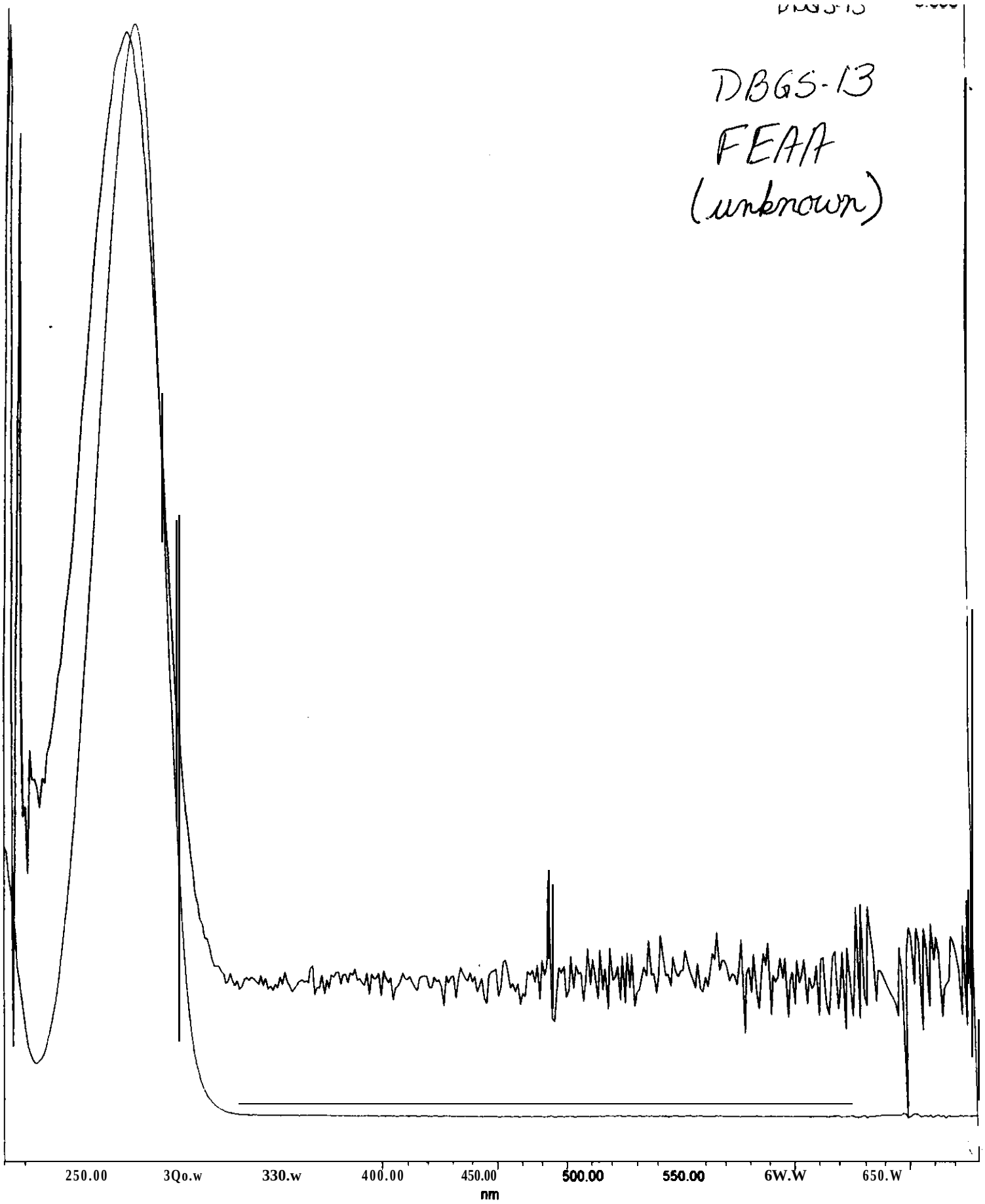

DBGS-12
DBGS-12
Acridamine
Hydrochloride
Peak #4
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

DBGS-12

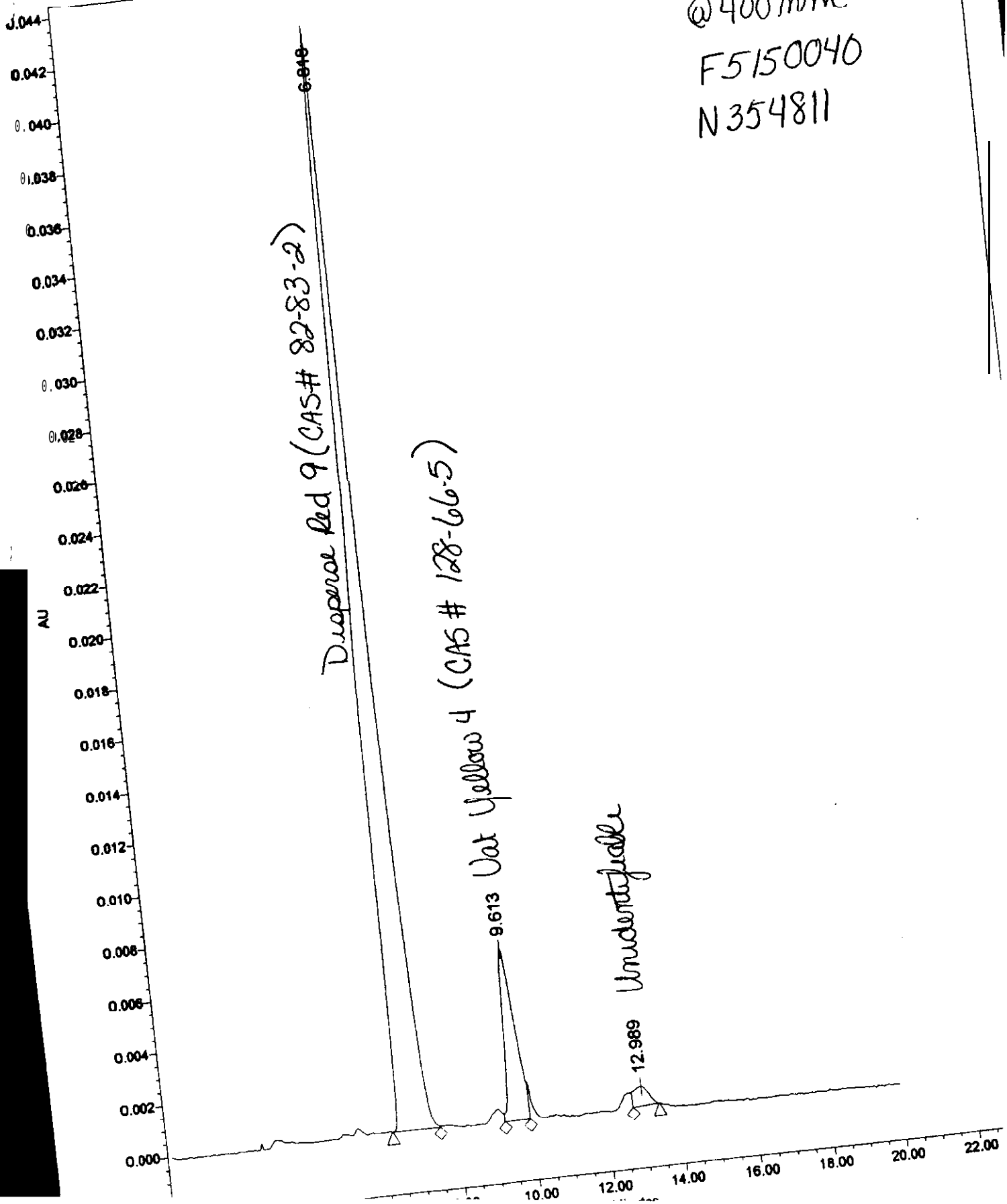
Unidentifiable




DBGS-13
@ 270nm
F5150040
N354821
FEAA (unknown)
3.8
AU
Minutes
0.0048
0.0046
0.0044
0.0042
0.0040
0.0038
0.0036
0.0034
0.0032
0.0030
0.0028
0.0026
0.0024
0.0022
0.0020
0.0018
0.0016
0.0014
0.0012
0.0010
0.0008
0.0006
0.0004
0.0002
0.0000
-0.0002
-0.0004
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
22.00
24.00


DBGS-13
FEAA
(unknown)
250.00
3Qo.w
330.w
400.00
450.00
500.00
550.00
6W.W
650.W
nm

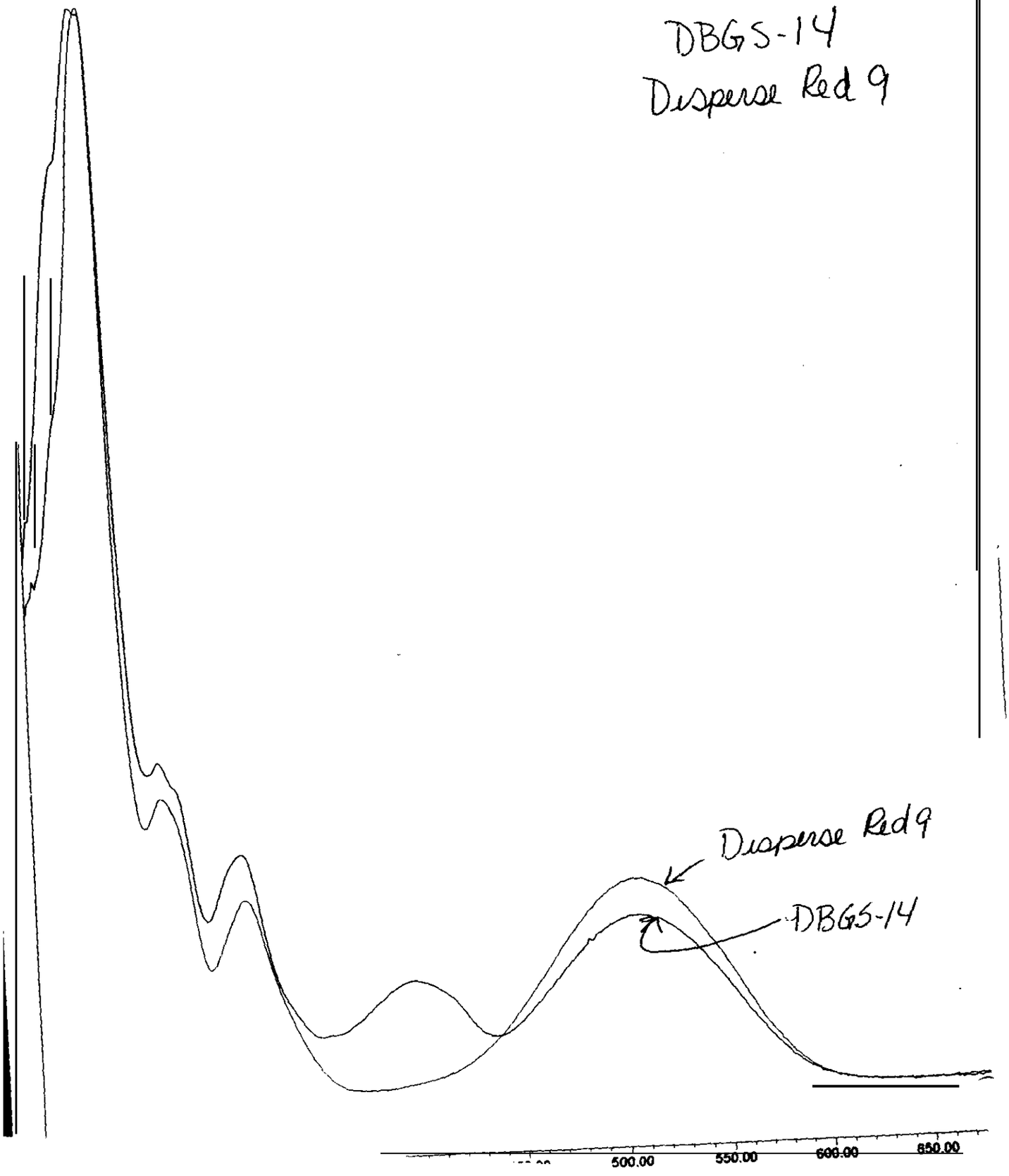
DBGS- 14
@400 mm.
F5150040
N354811



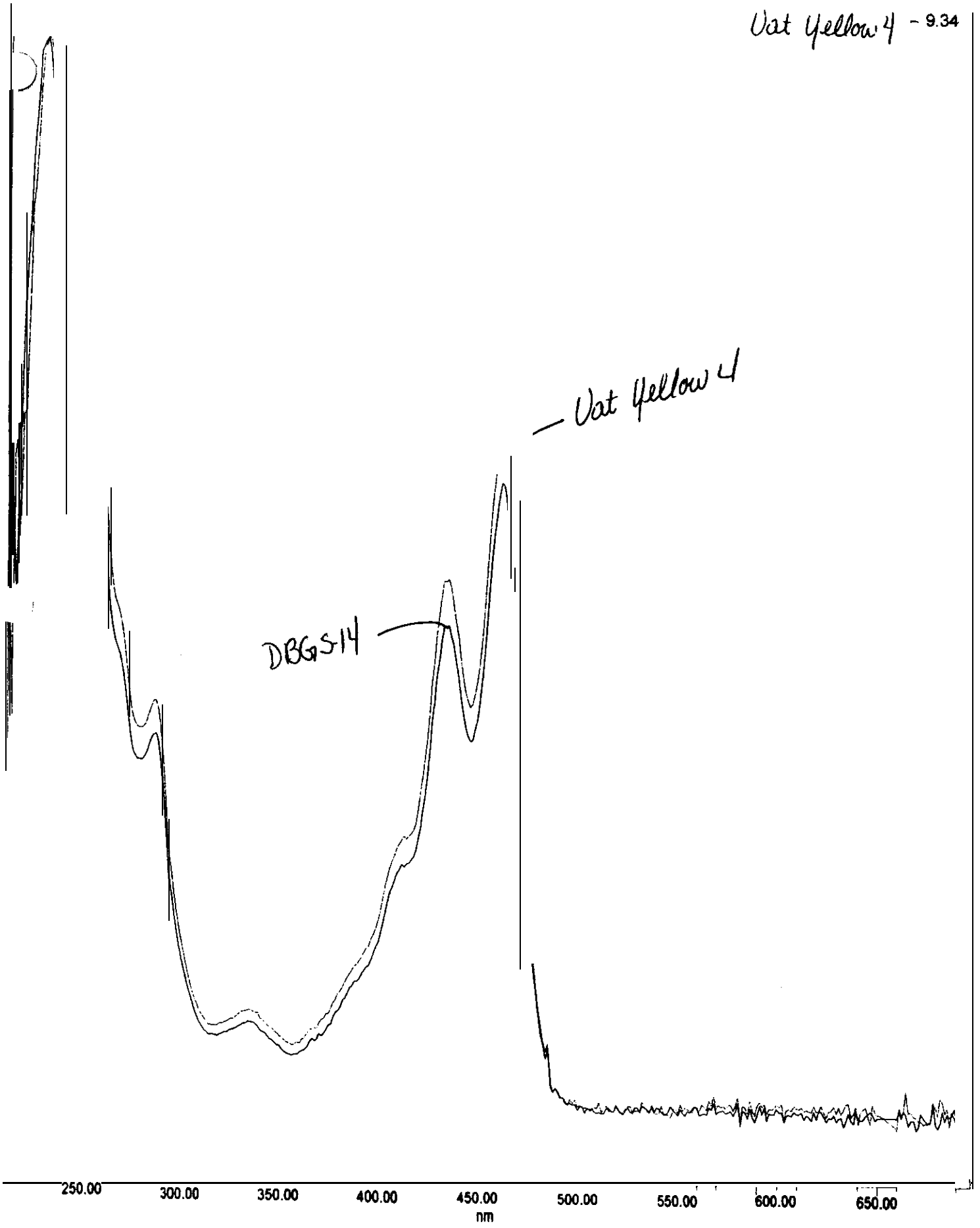
[redacted]

Disperse Red 9 - 6.595

DBGS-14
Disperse Red 9

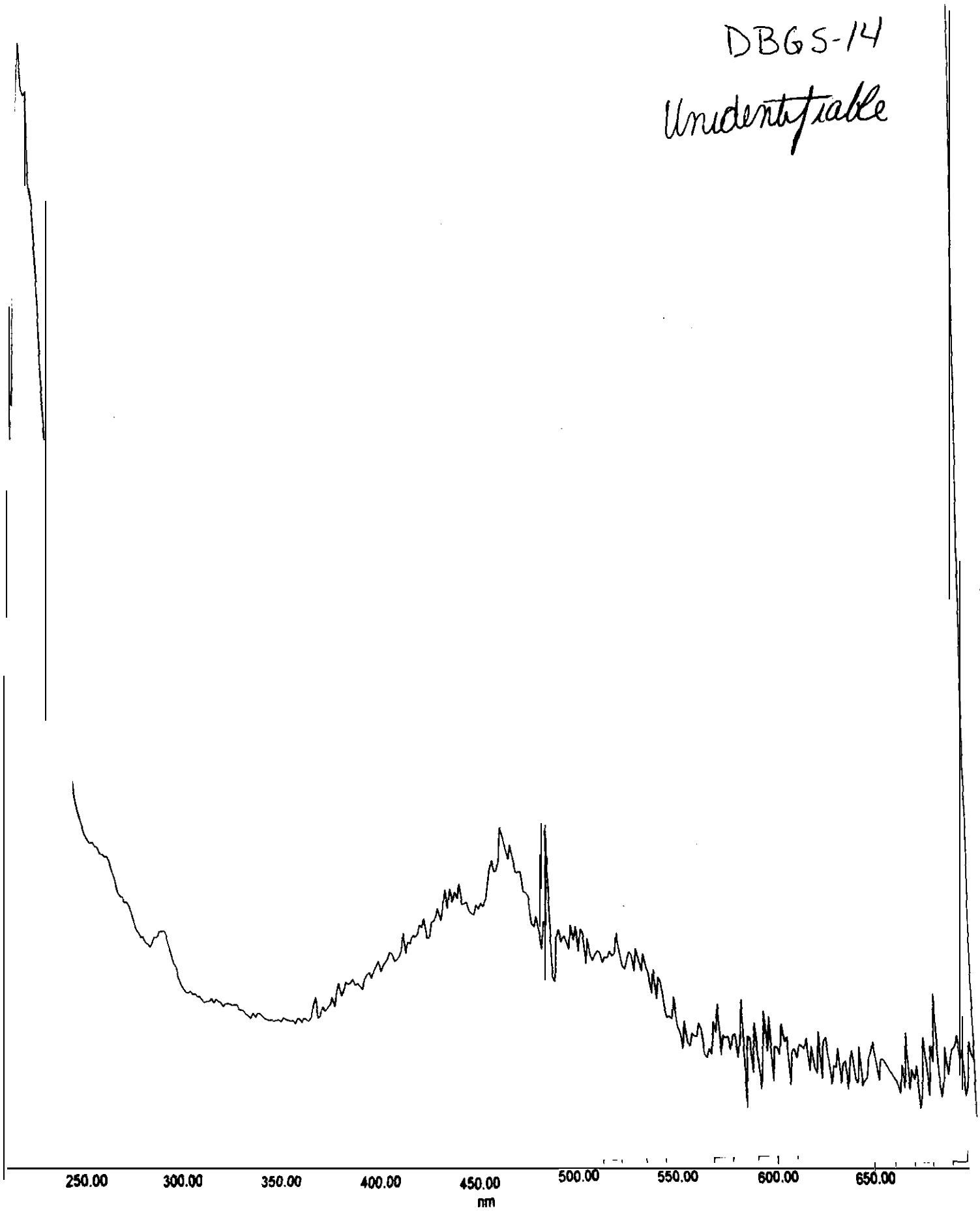


[redacted]

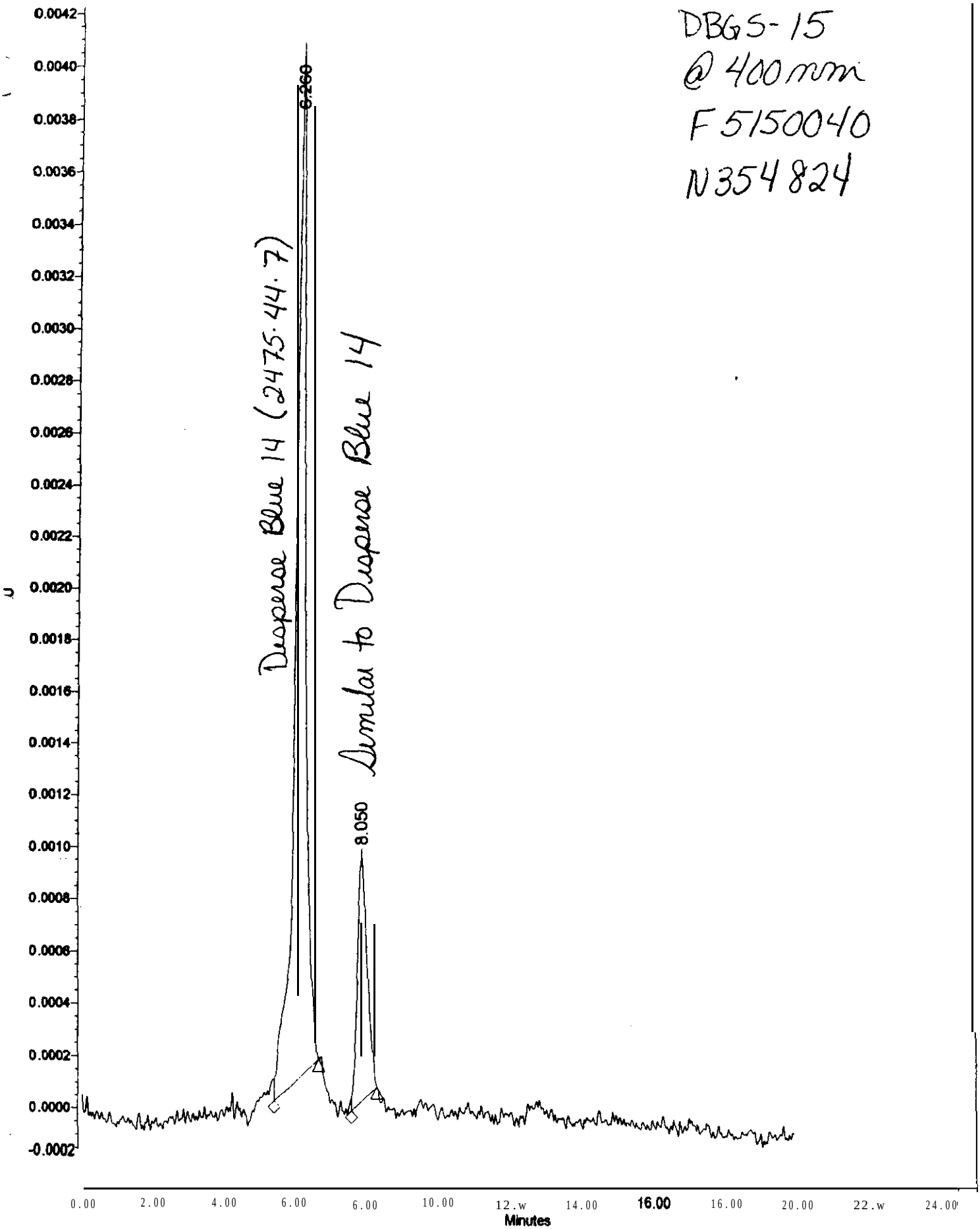

Vat Yellow 4 - 9.34
Vat Yellow 4
DBGS14
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

DBGS-14

Unidentifiable

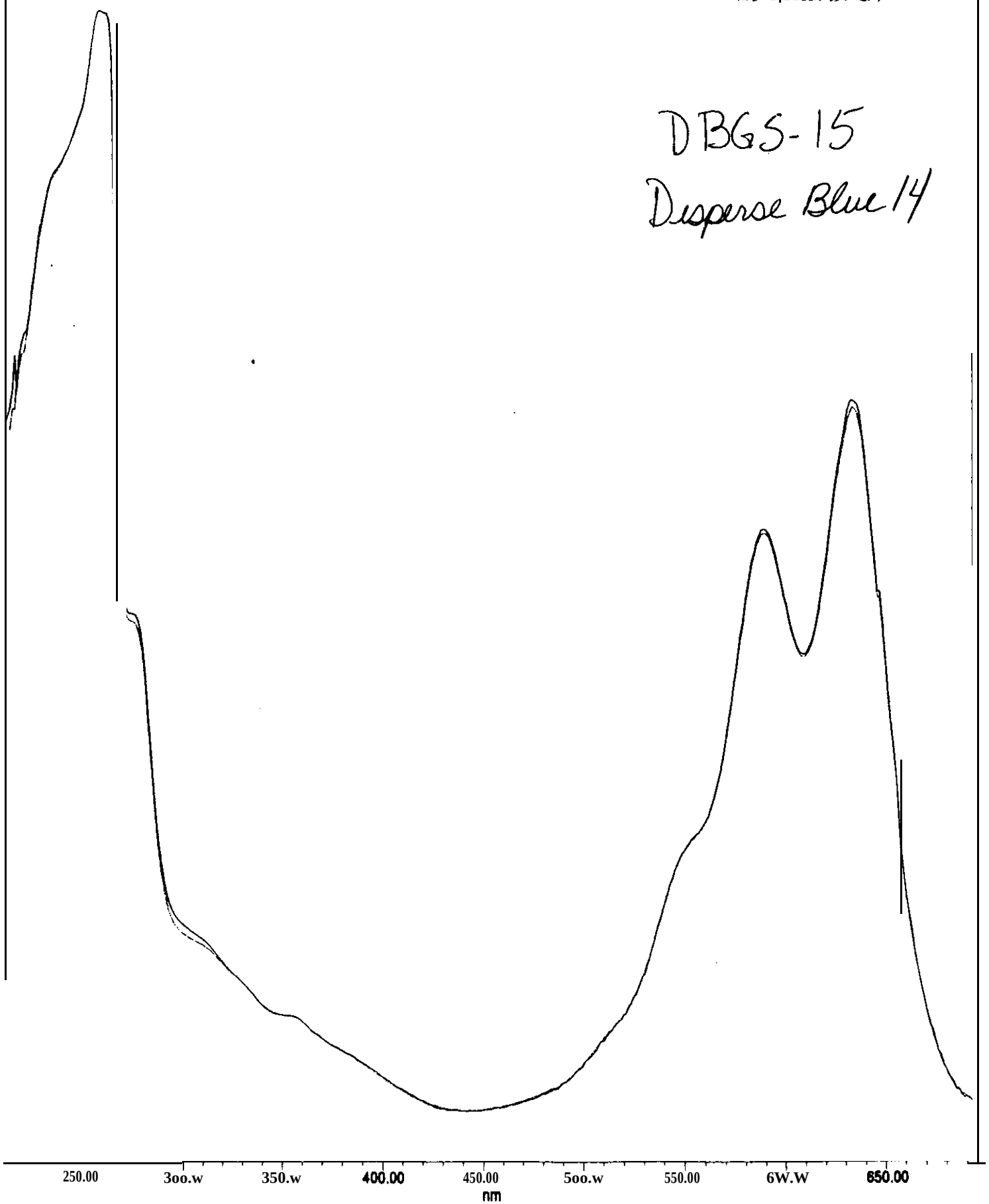


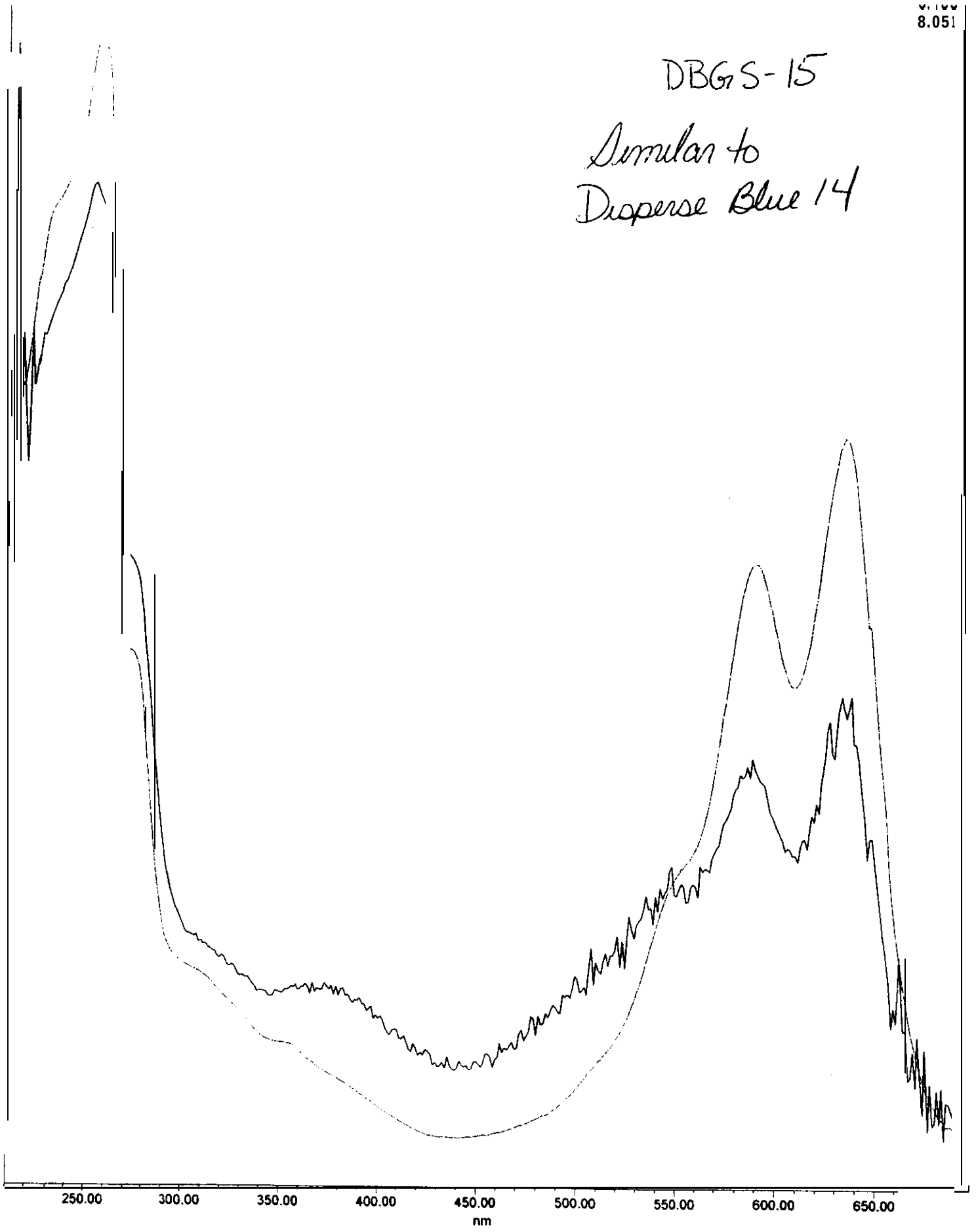
DBGS-15
@ 400nm
F5150040
N354824

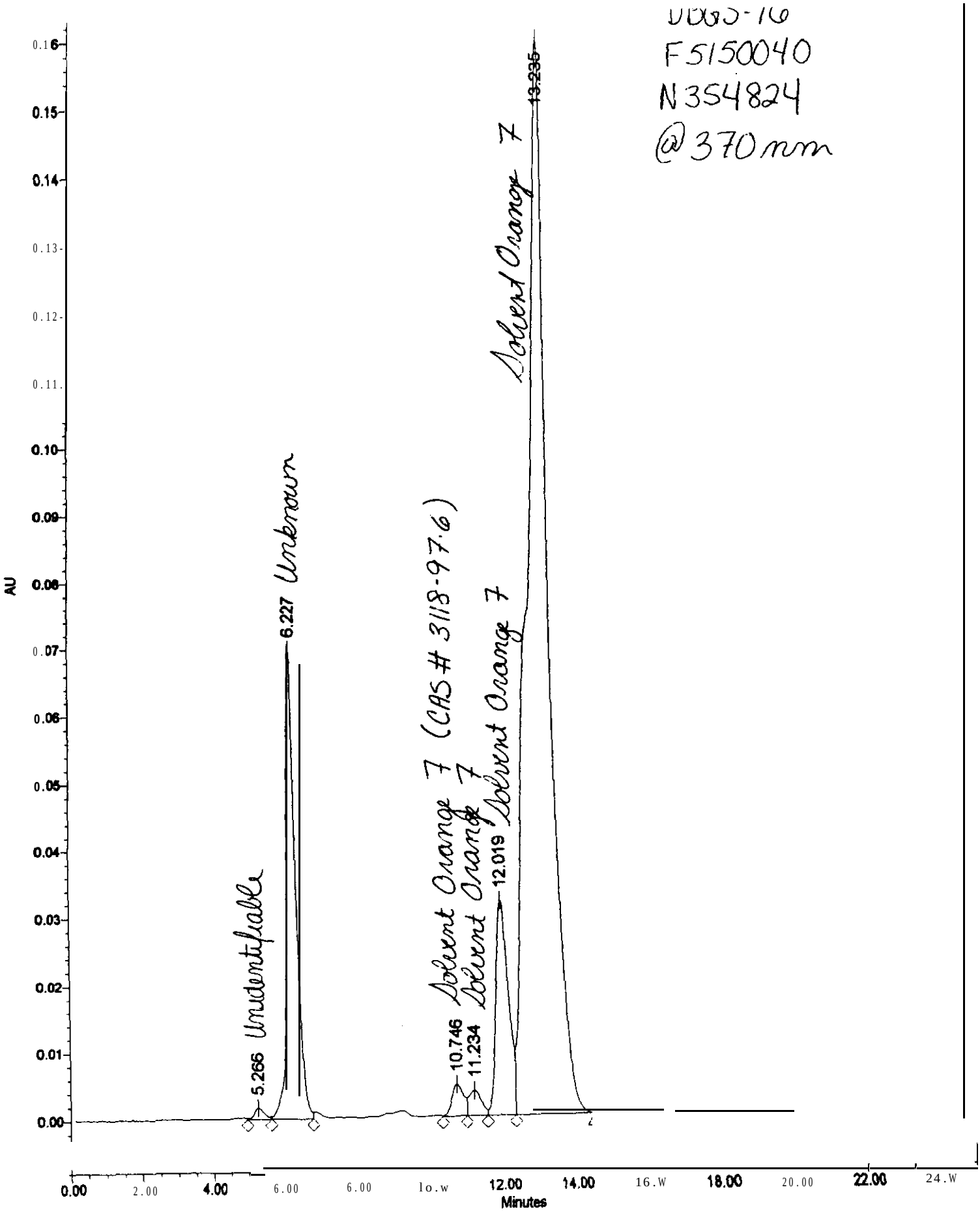


Disperse Blue 14 - 6.199

DBGS-15
Disperse Blue 14

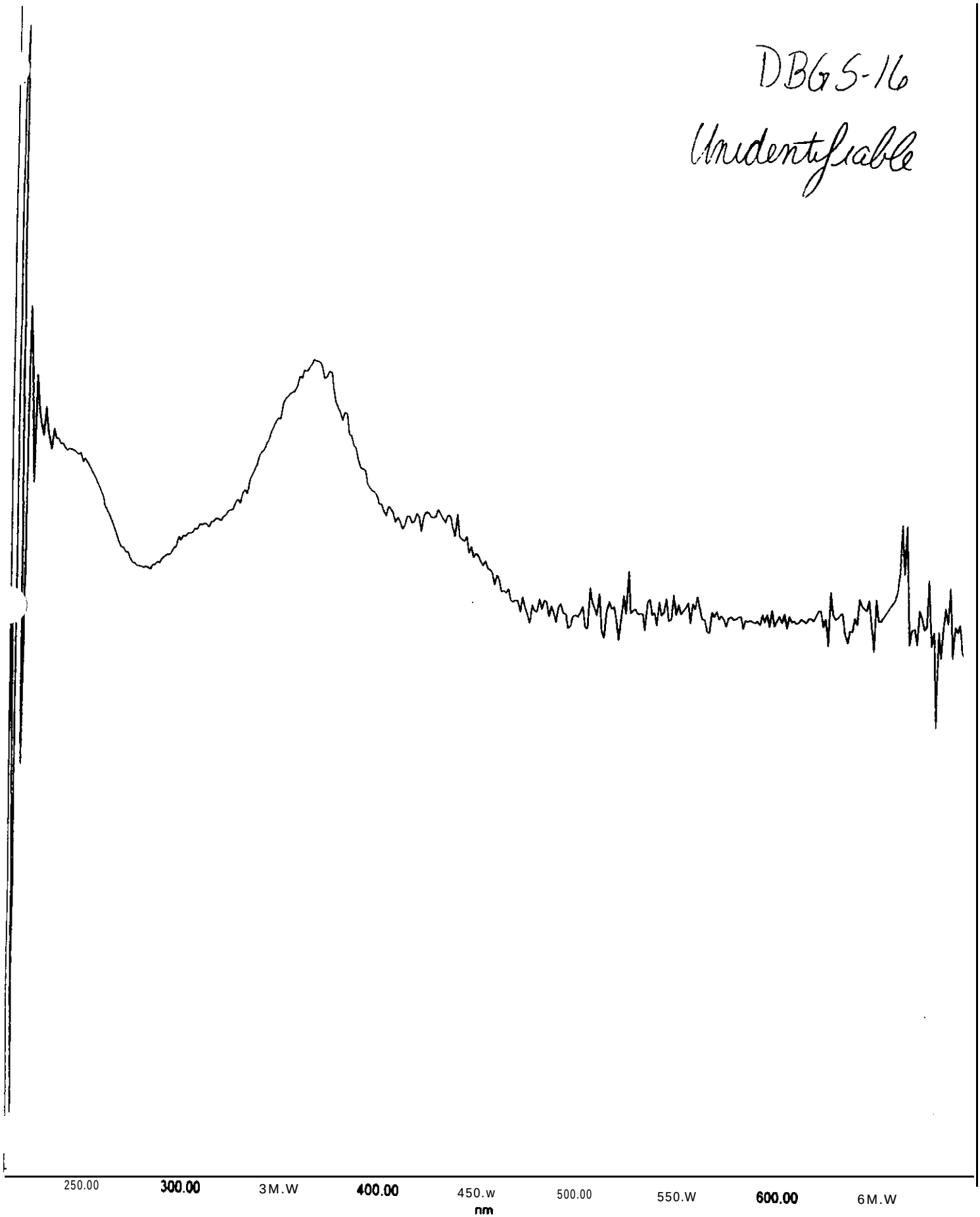


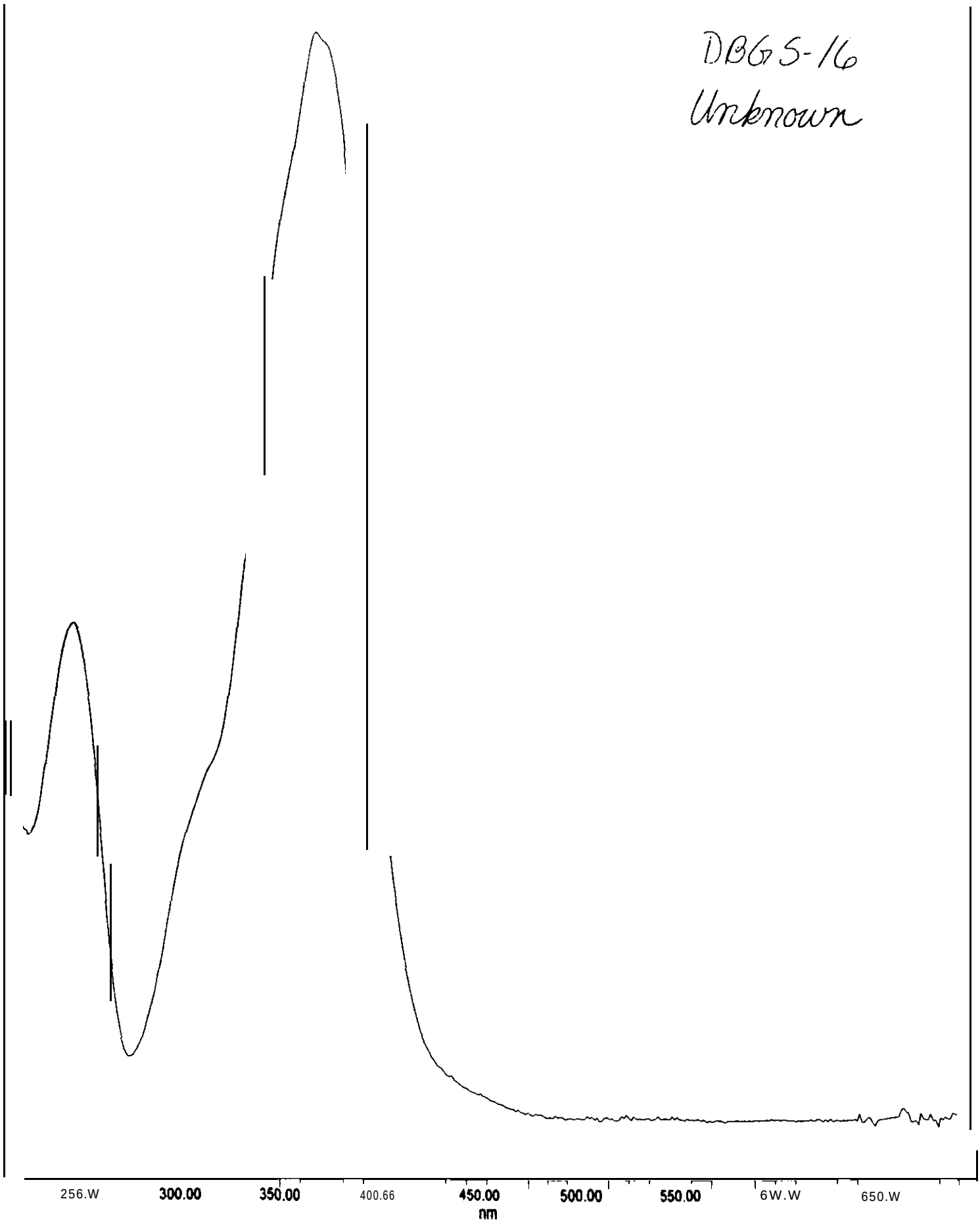

8.051
DBGS-15
Similar to
Disperse Blue 14
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

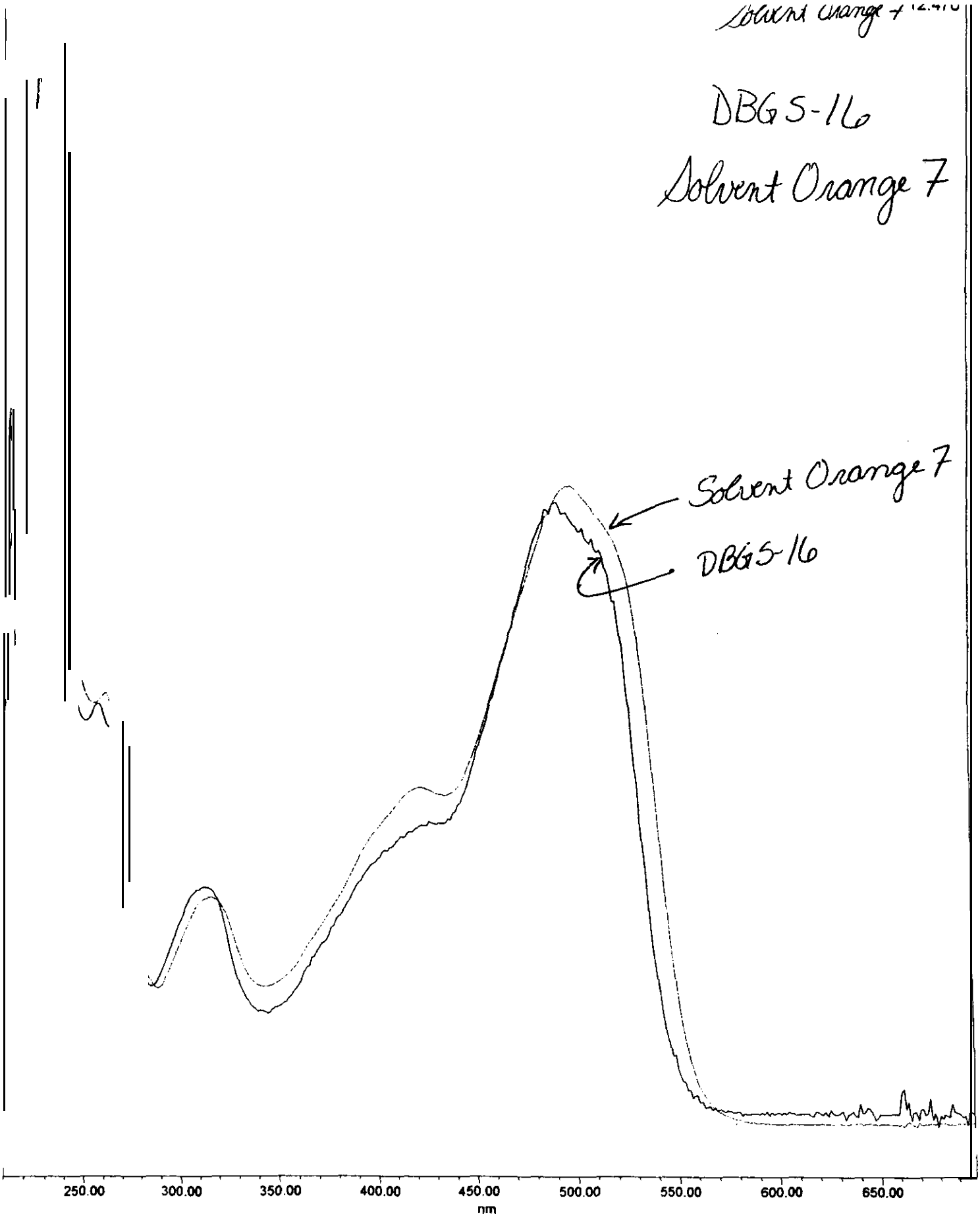

D003-16
F5150040
N354824
@370nm
AU
0.16
0.15
0.14
0.13
0.12
0.11
0.10
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0.00
5.266 Unidentifiable
6.227 Unknown
10.746 Solvent Orange 7 (CAS # 3118-97-6)
11.234 Solvent Orange 7
12.019 Solvent Orange 7
13.235 Solvent Orange 7
0.00
2.00
4.00
6.00
6.00
lo.w
12.00
14.00
16.W
18.00
20.00
22.00
24.W
Minutes

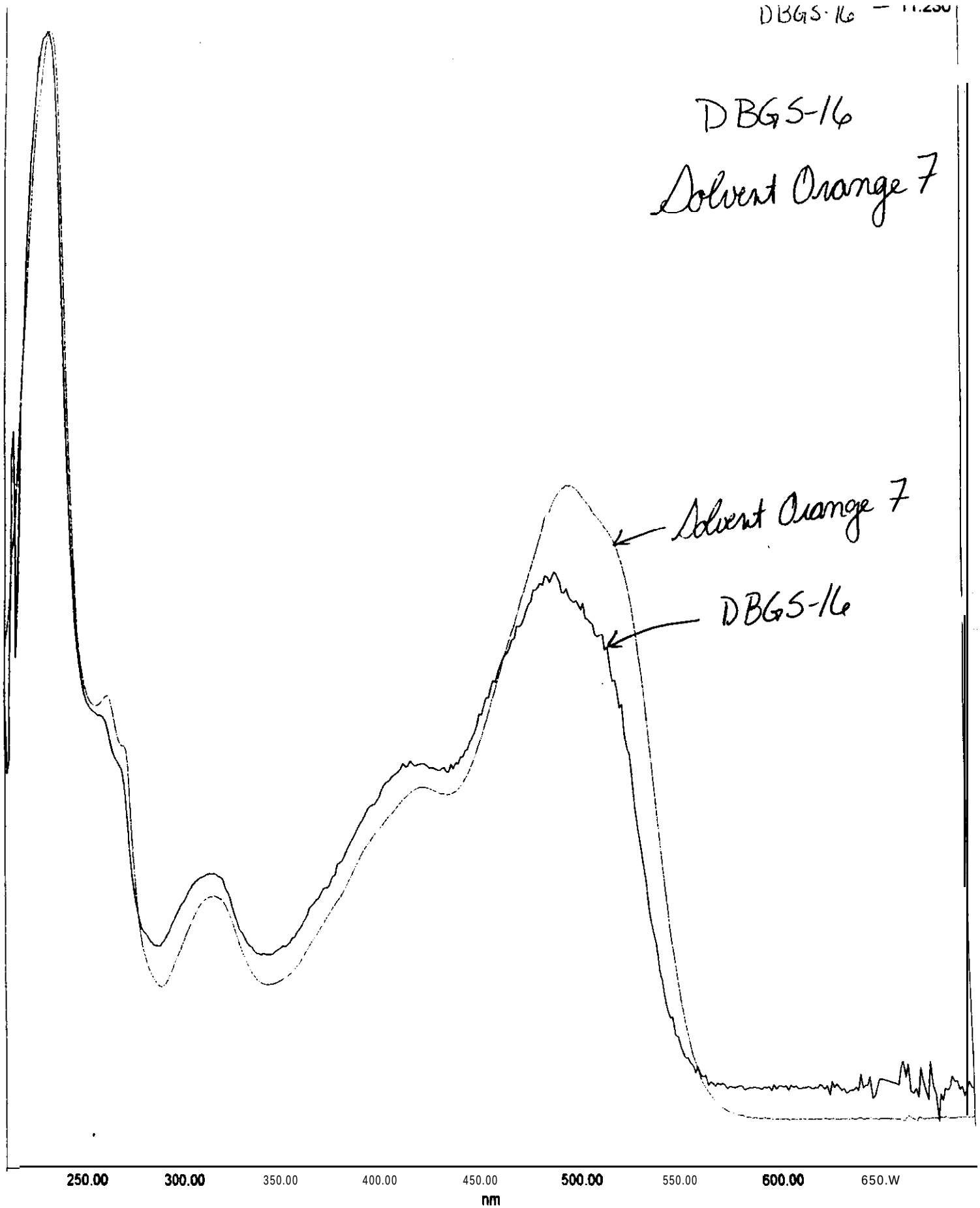
DBGS-16

Unidentifiable



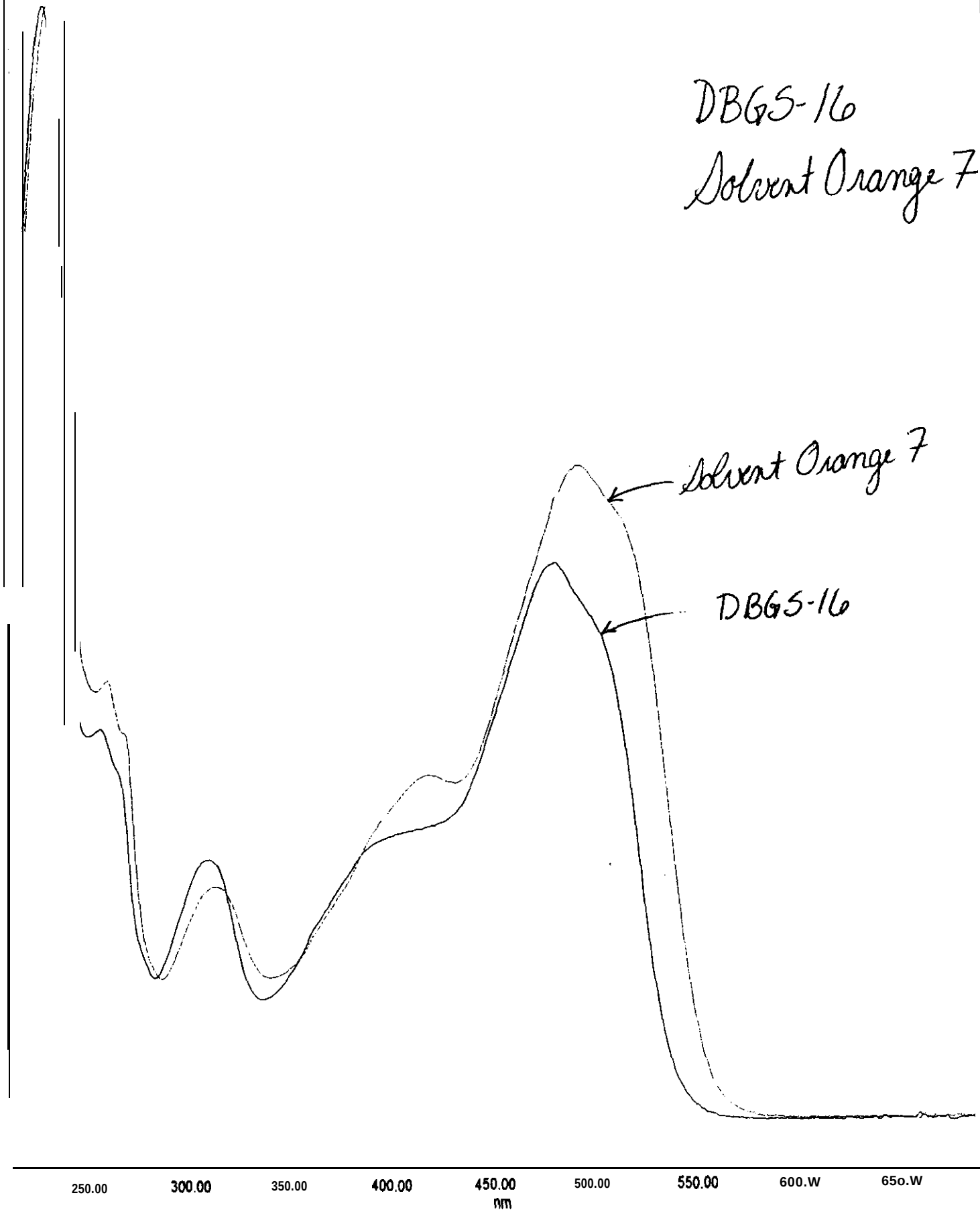

DBGS-16
Unknown
256.W
300.00
350.00
400.66
450.00
500.00
550.00
6W.W
650.W
nm

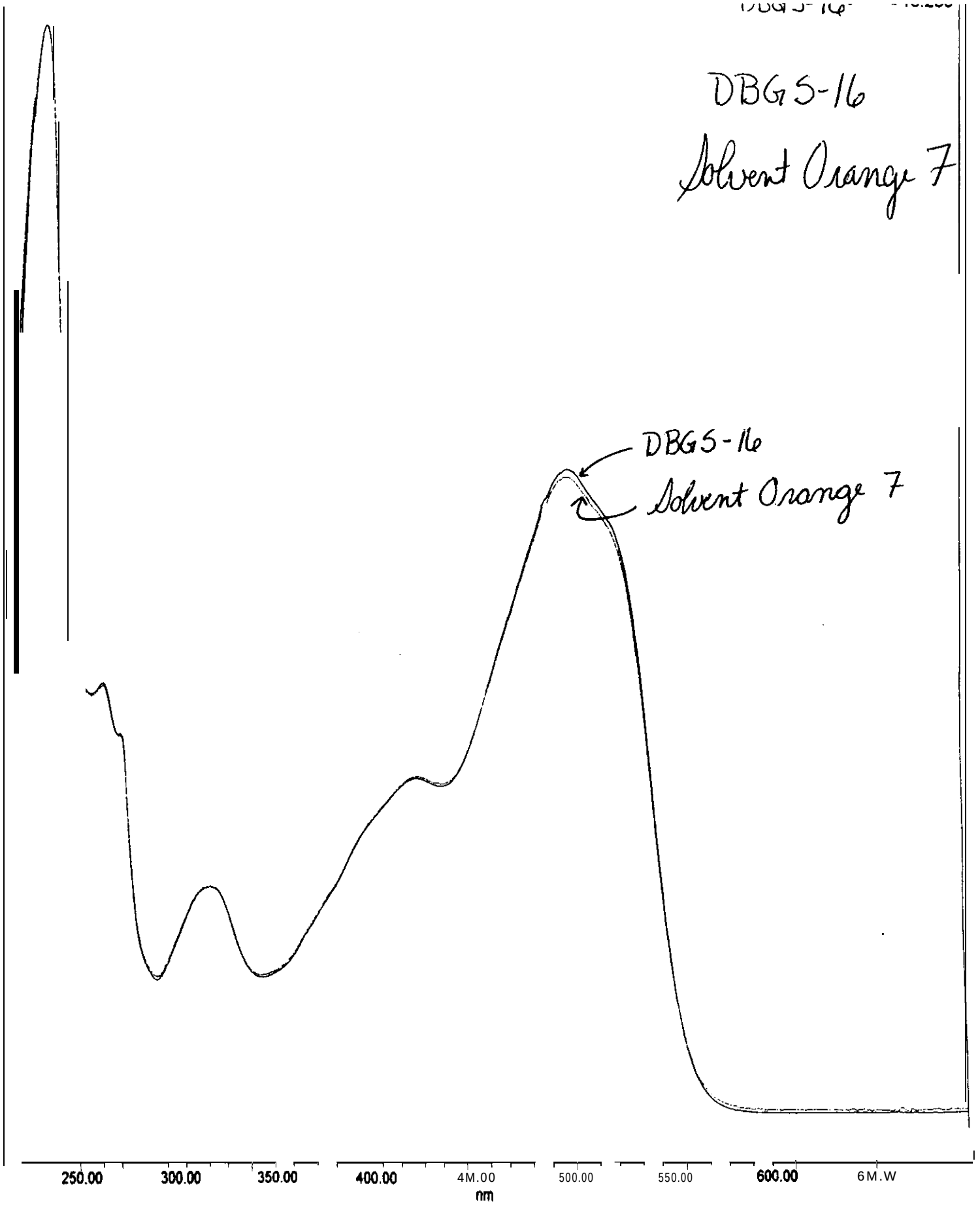

Solvent Orange 7 + 12.410
DBG S-16
Solvent Orange 7
Solvent Orange 7
DBG S-16
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

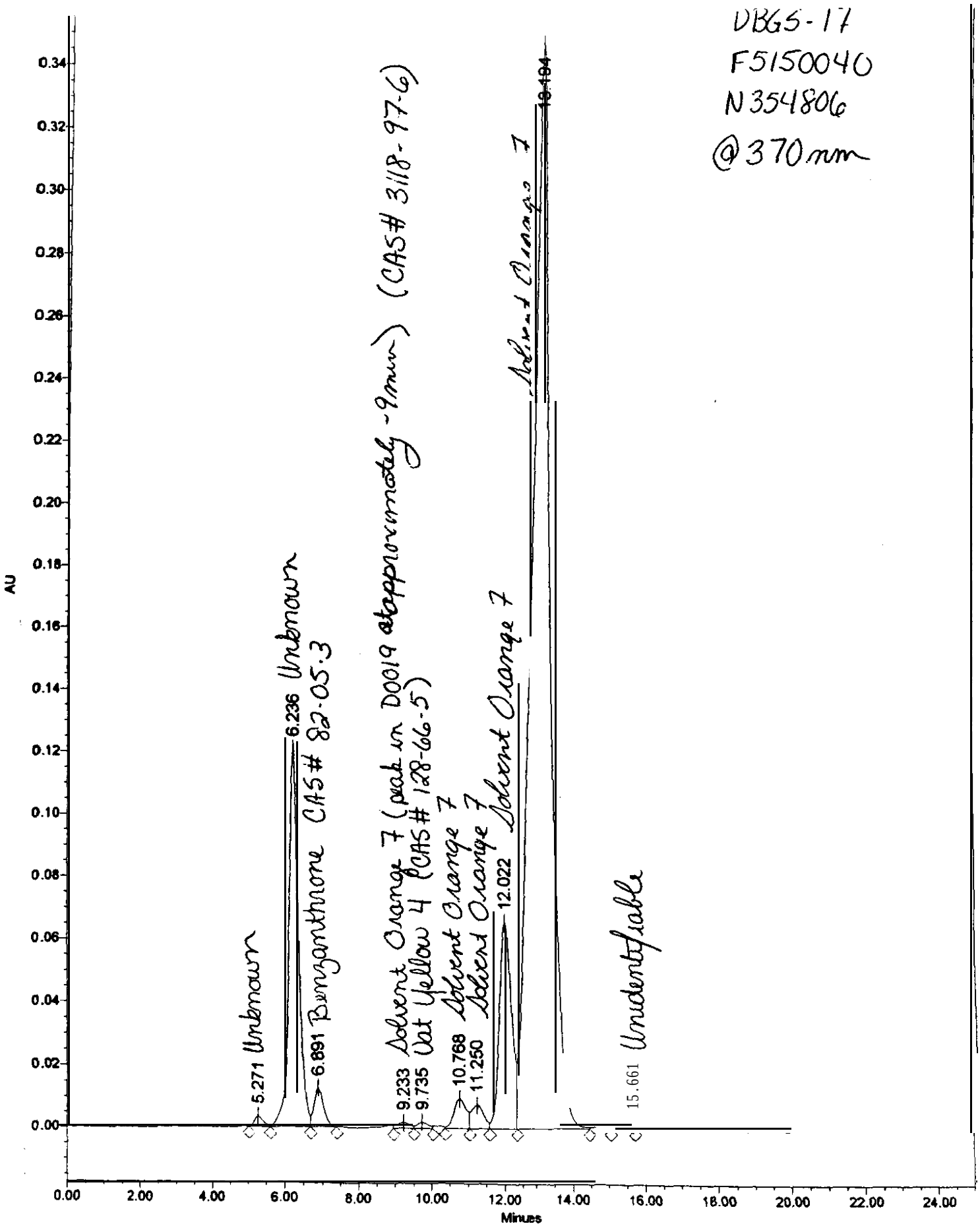

DBGS-16
Solvent Orange 7
Solvent Orange 7
DBGS-16
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.W
nm

DBGS-16 - 12.010

DBGS-16
Solvent Orange 7

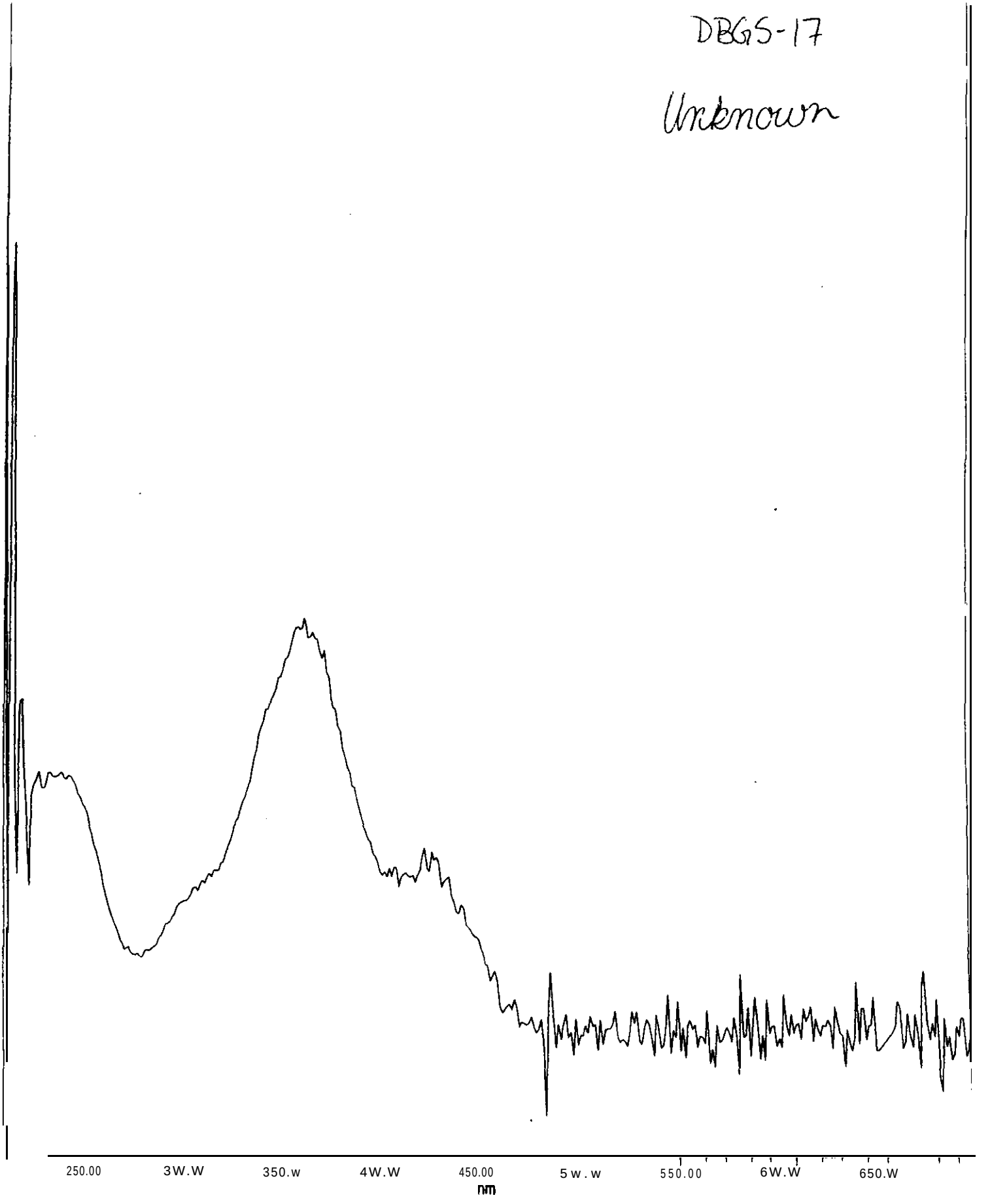


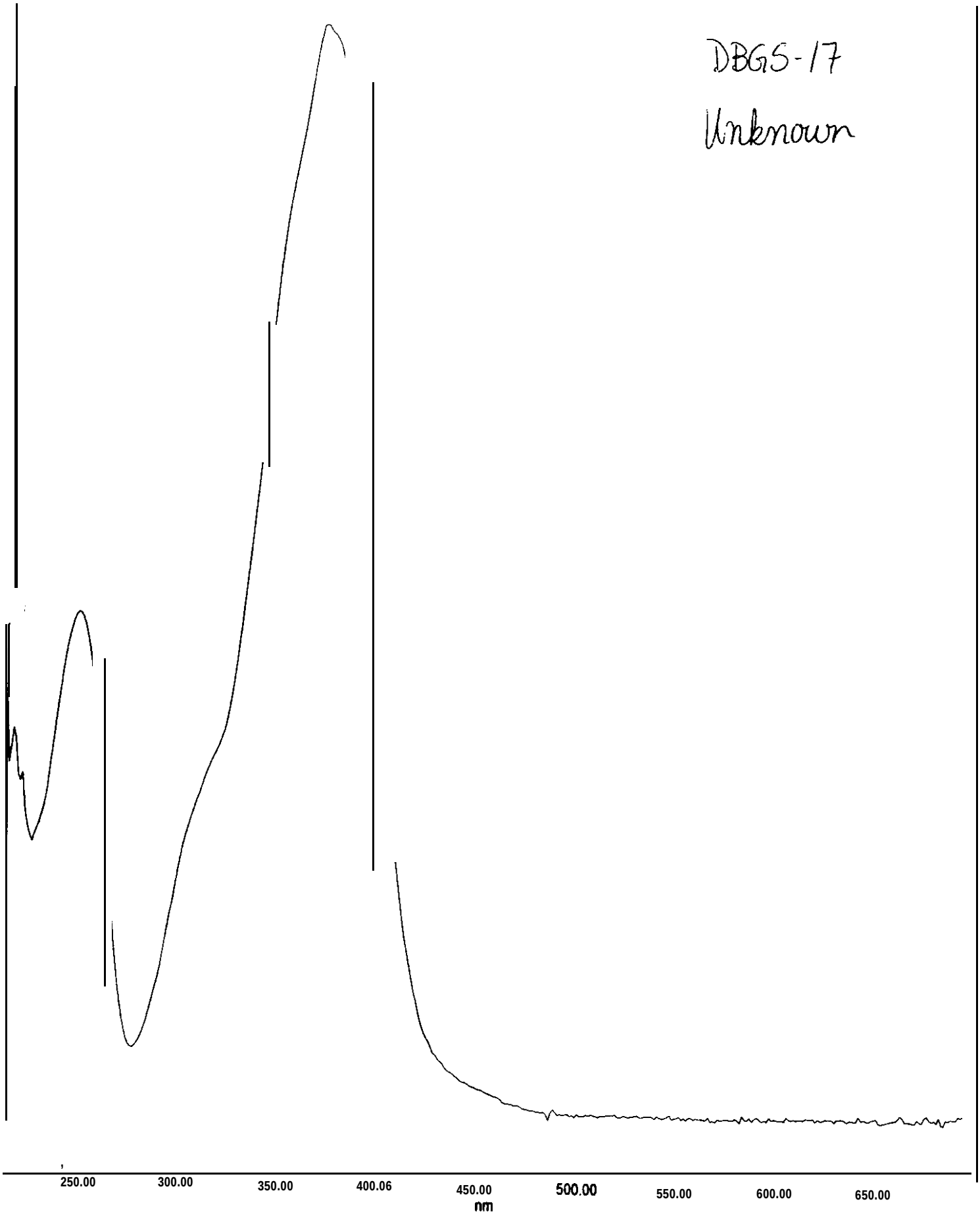

DBGS-16
Solvent Orange 7
DBGS-16
Solvent Orange 7
250.00
300.00
350.00
400.00
4M.00
500.00
550.00
600.00
6M.W
nm

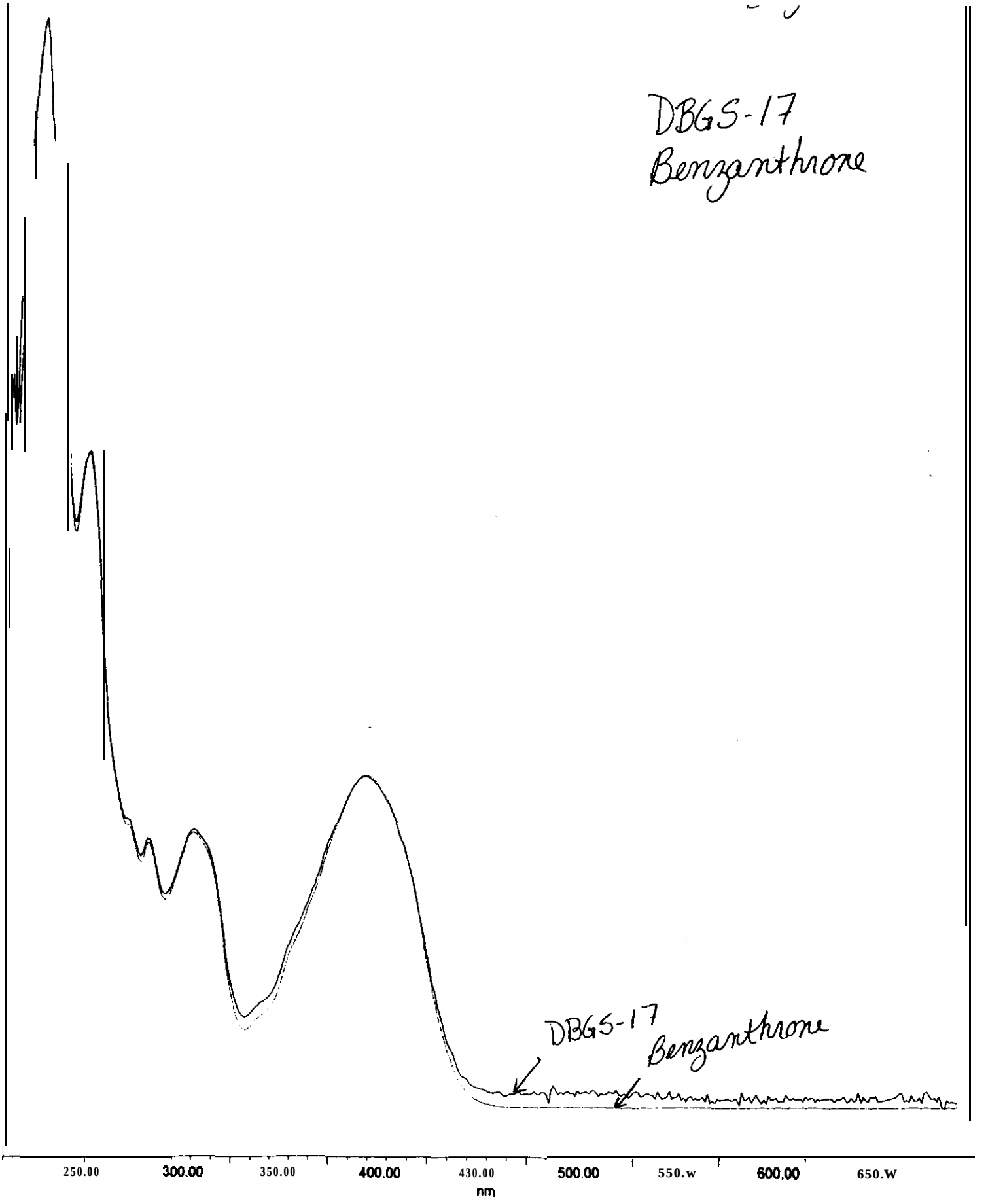

DBG5-17
F5150040
N354806
@370nm
AU
Minutes
5.271 Unknown
6.236 Unknown
6.891 Benzanthrone CAS# 82-05-3
9.233 Solvent Orange 7 (peak in D0019 approximately -9min) (CAS# 3118-97-6)
9.735 Oil Yellow 4 (CAS# 128-66-5)
10.768 Solvent Orange 7
11.250 Solvent Orange 7
12.022 Solvent Orange 7
Solvent Orange 7
15.661 Unidentifiable

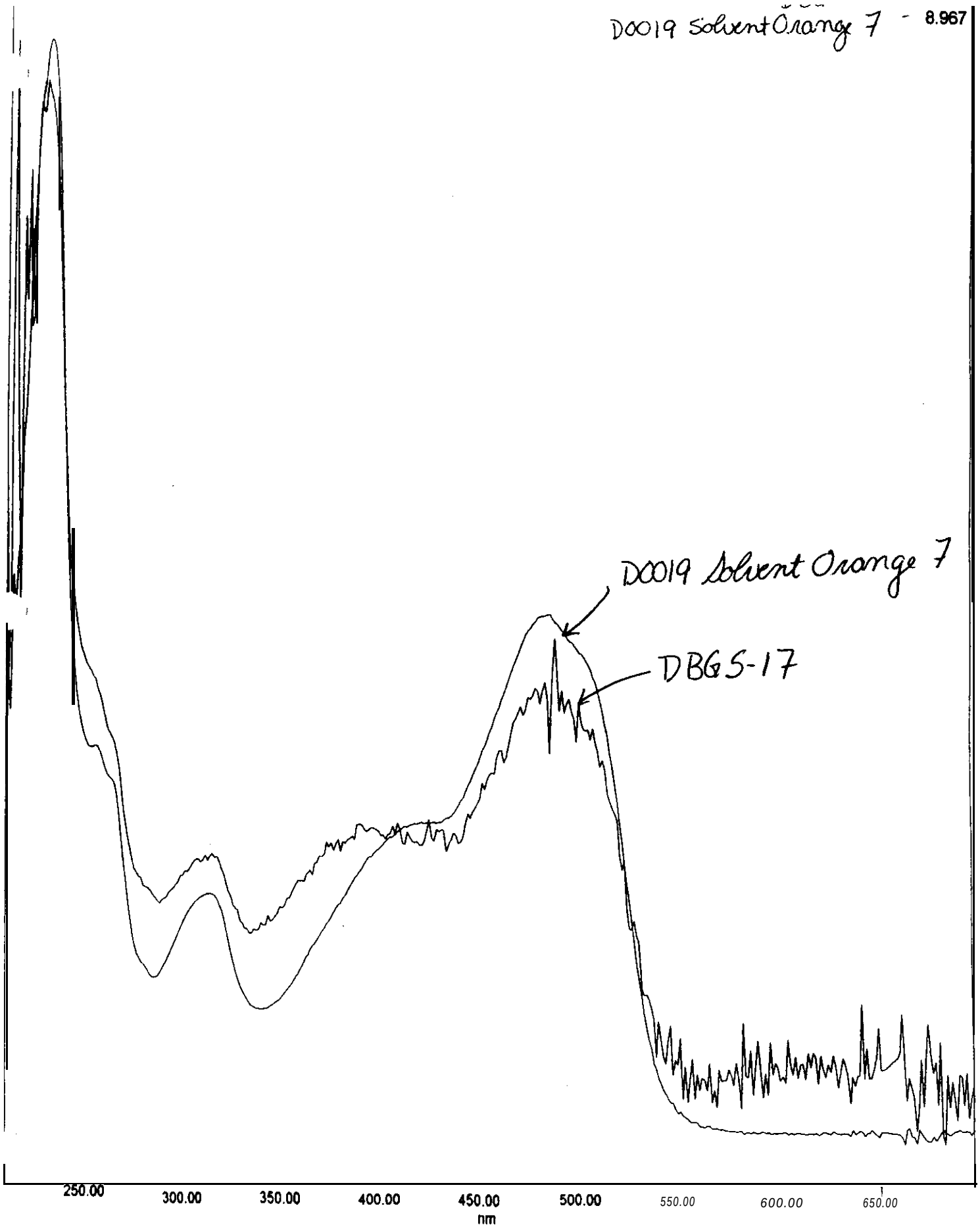
DBGS-17

Unknown




DBGS-17
Unknown
250.00
300.00
350.00
400.06
450.00
500.00
550.00
600.00
650.00
nm


DBGS-17
Benzanthrone
DBGS-17
Benzanthrone
250.00
300.00
350.00
400.00
430.00
500.00
550.w
600.00
650.W
nm


D0019 Solvent Orange 7 - 8.967
D0019 Solvent Orange 7
DBGS-17
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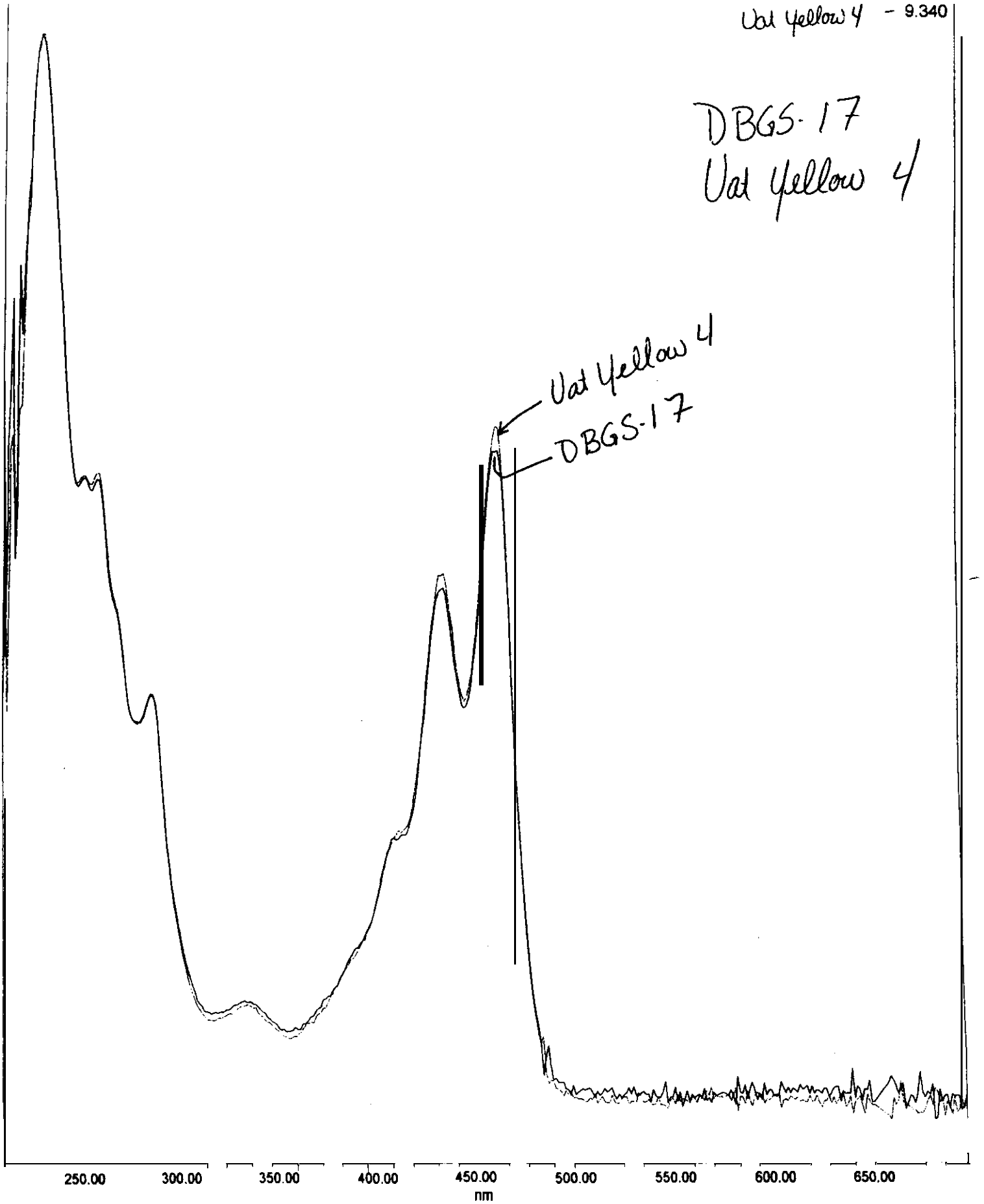
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm


Vat Yellow 4 - 9.340
DBGS-17
Vat Yellow 4
Vat Yellow 4
DBGS-17
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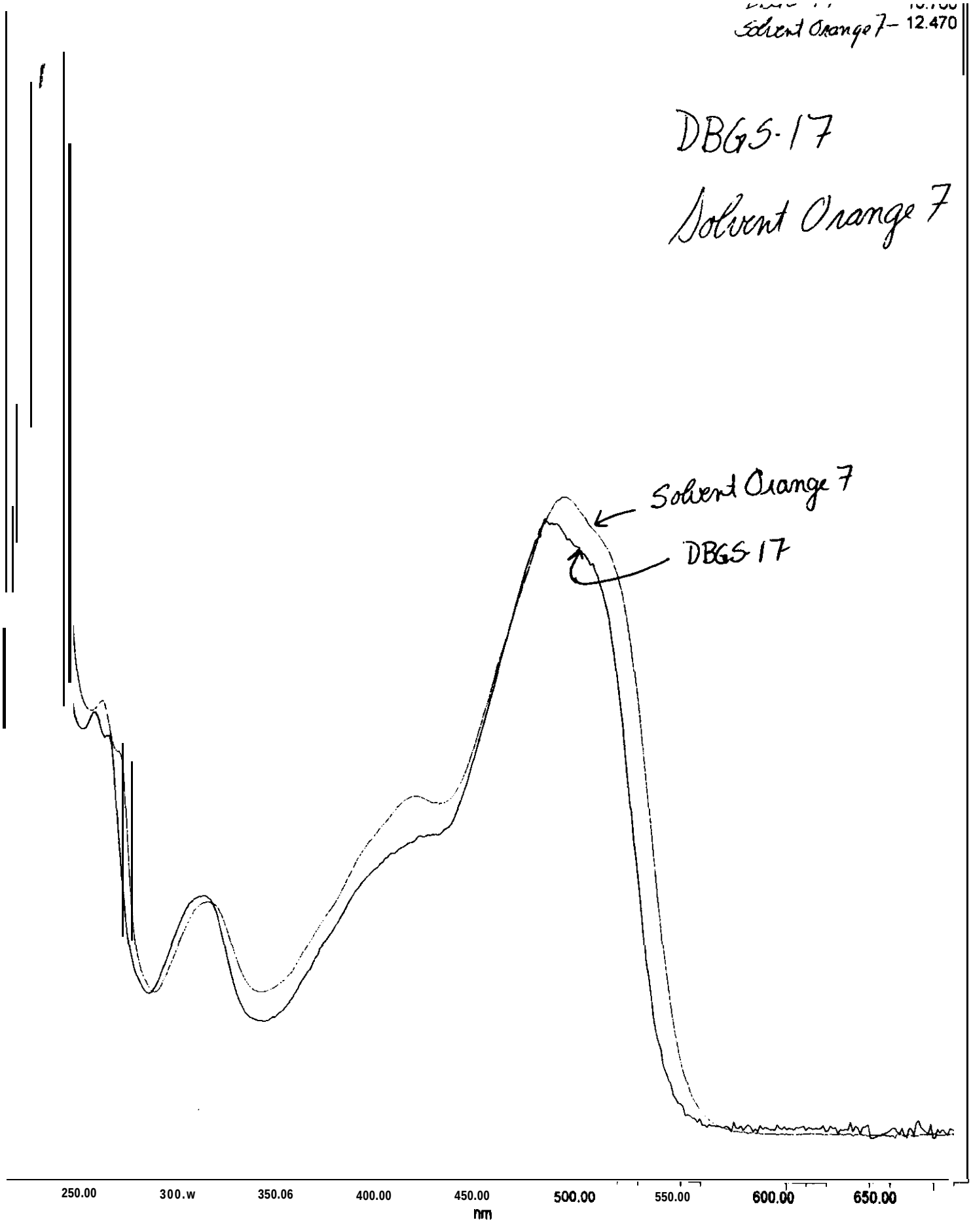
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm


Solvent Orange 7– 12.470
DBGS-17
Solvent Orange 7
Solvent Orange 7
DBGS 17
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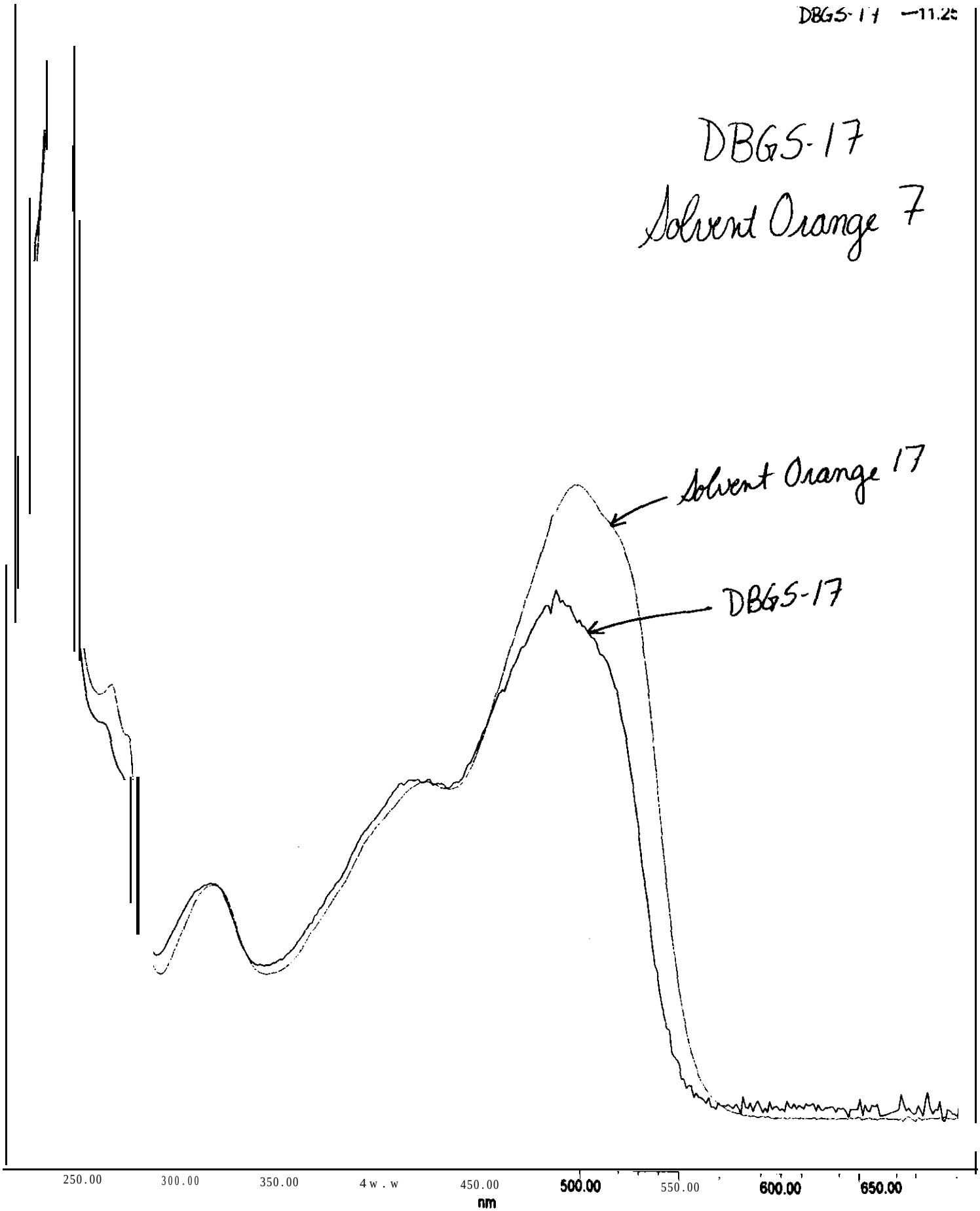
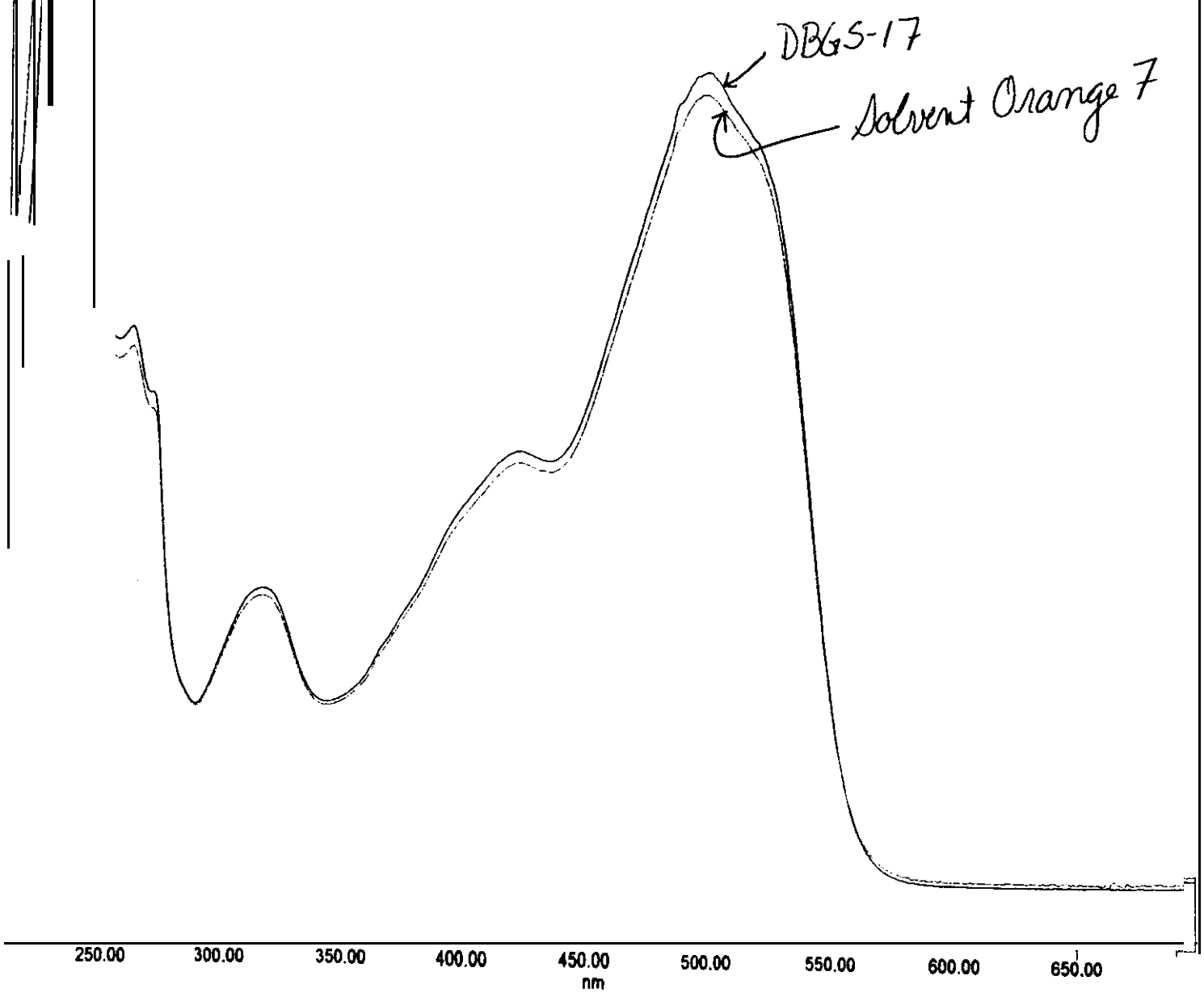
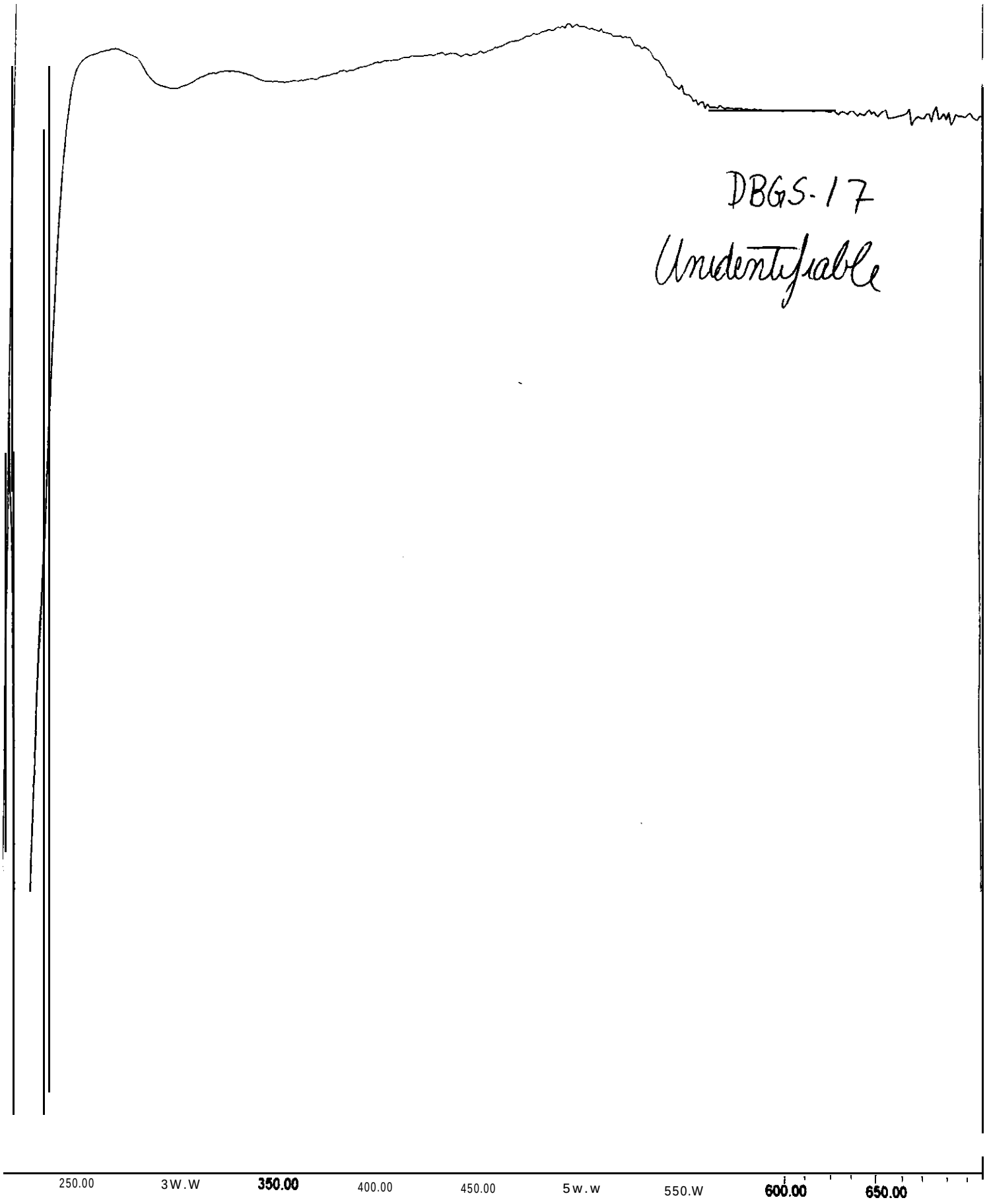
300.w
350.06
400.00
450.00
500.00
550.00
600.00
650.00
nm


DBGS-17 —11.25
DBGS-17
Solvent Orange 7
Solvent Orange 17
DBGS-17
250.00
300.00
350.00
4w.w
450.00
500.00
550.00
600.00
650.00
nm

DBGS-17 – 13.190

DBGS-17
Solvent Orange 7

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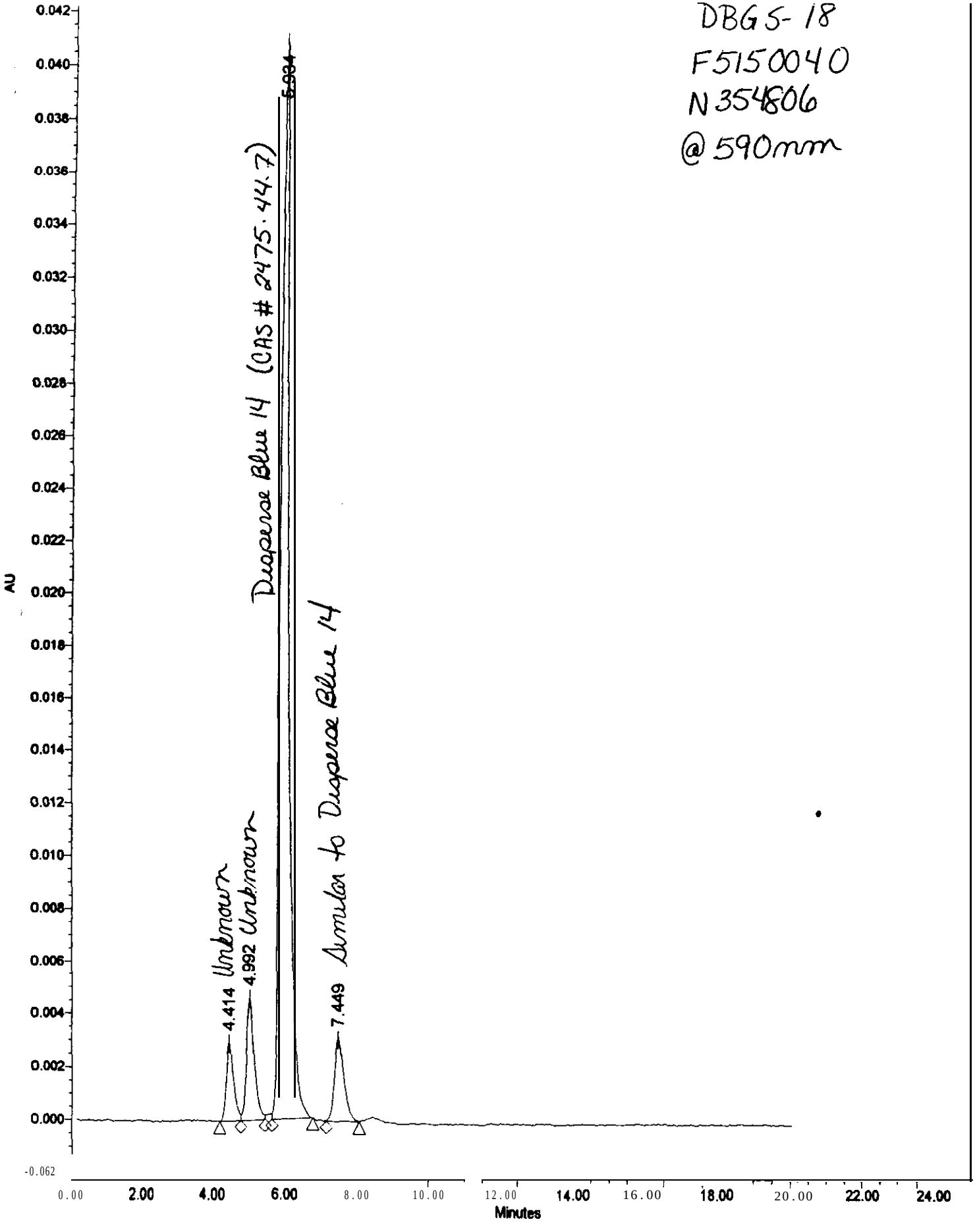
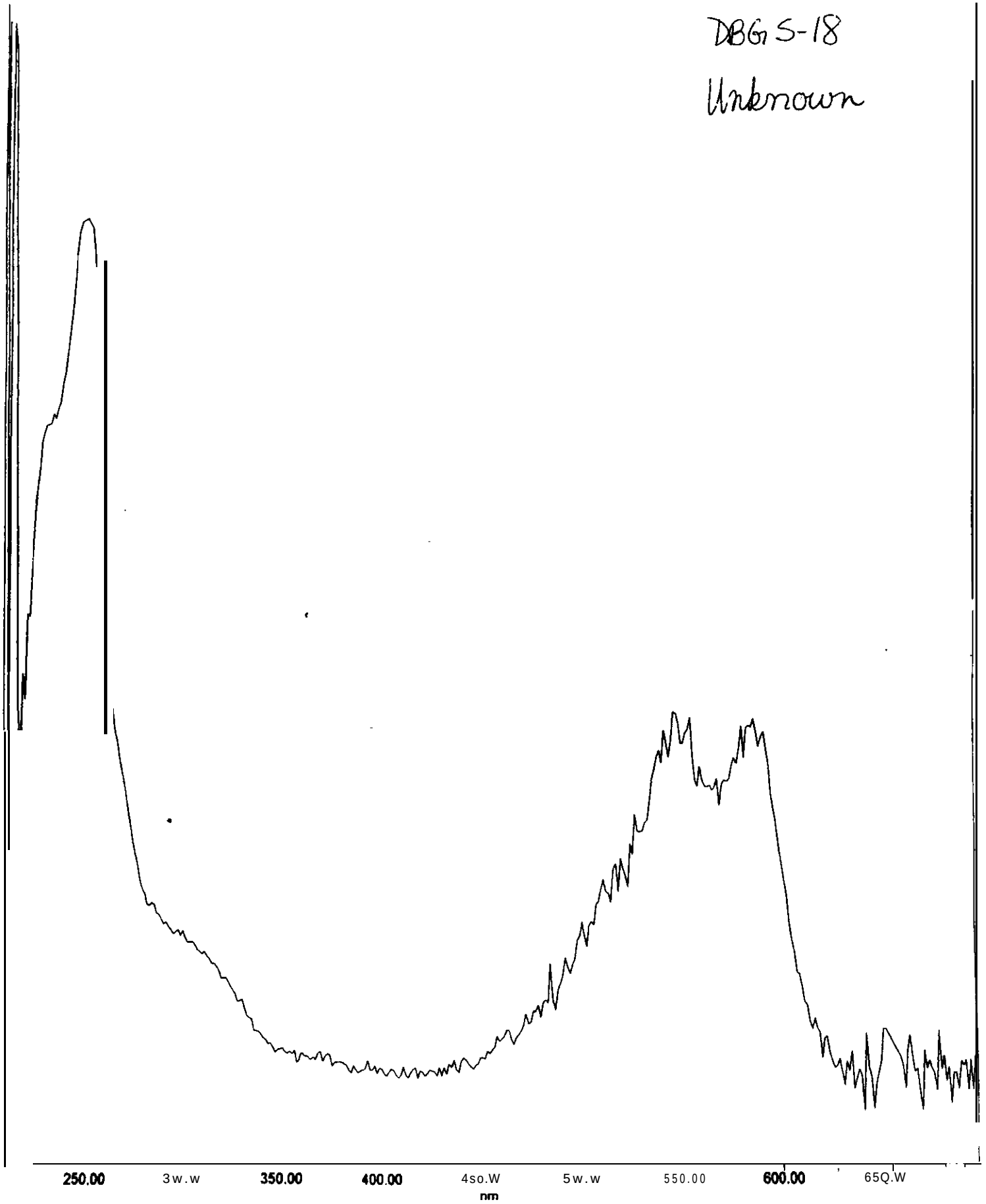
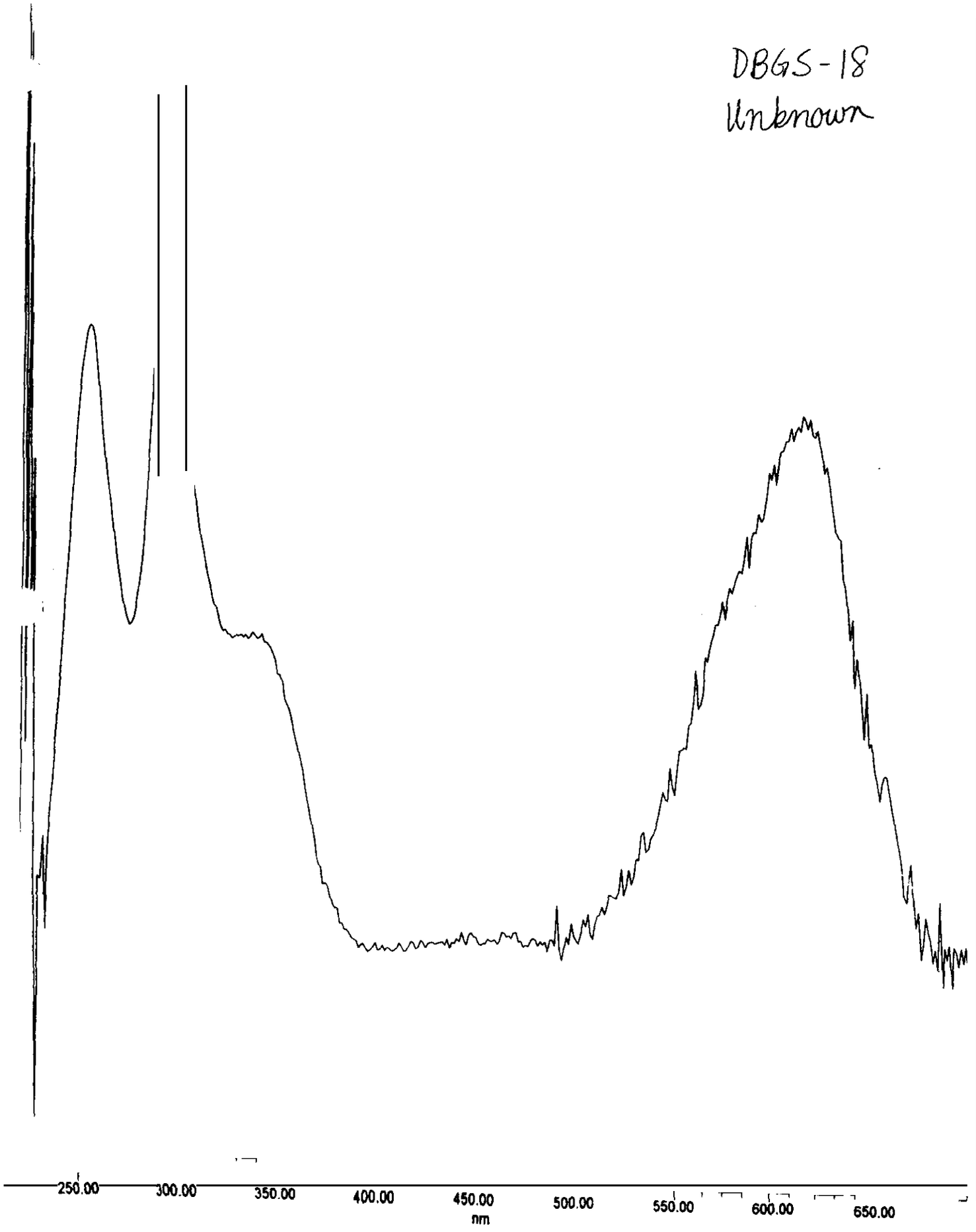
3W.W
350.00
400.00
450.00
5w.w
550.W
600.00
650.00


DBG5-18
F5150040
N354806
@590nm
Disperse Blue 14 (CAS # 2475-44-7)
6.934
Unknown
4.414
Unknown
4.992
Similar to Disperse Blue 14
7.449
AU
0.042
0.040
0.038
0.036
0.034
0.032
0.030
0.028
0.026
0.024
0.022
0.020
0.018
0.016
0.014
0.012
0.010
0.008
0.006
0.004
0.002
0.000
-0.062
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
22.00
24.00
Minutes

DBG S-18

Unknown




DBGS-18
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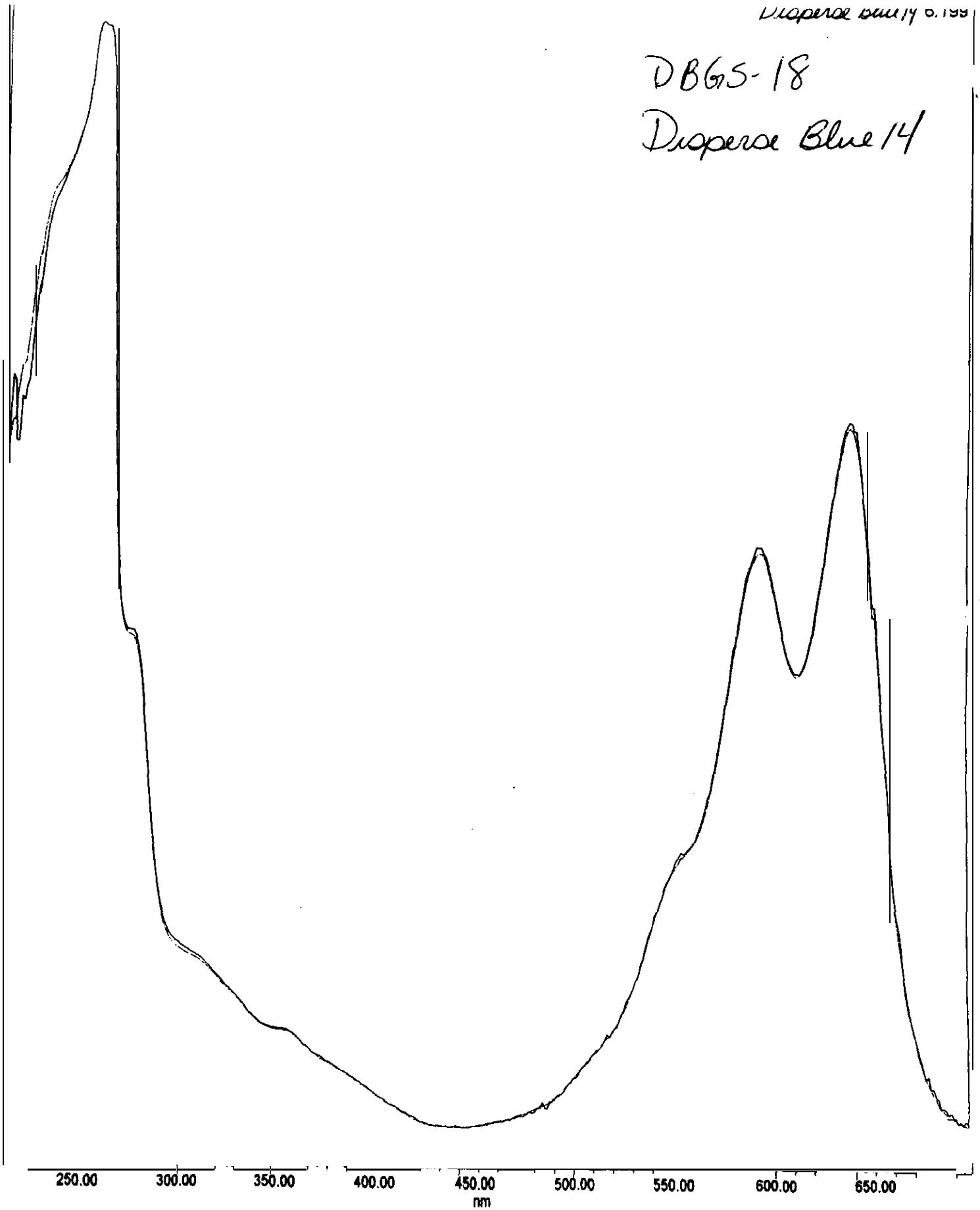
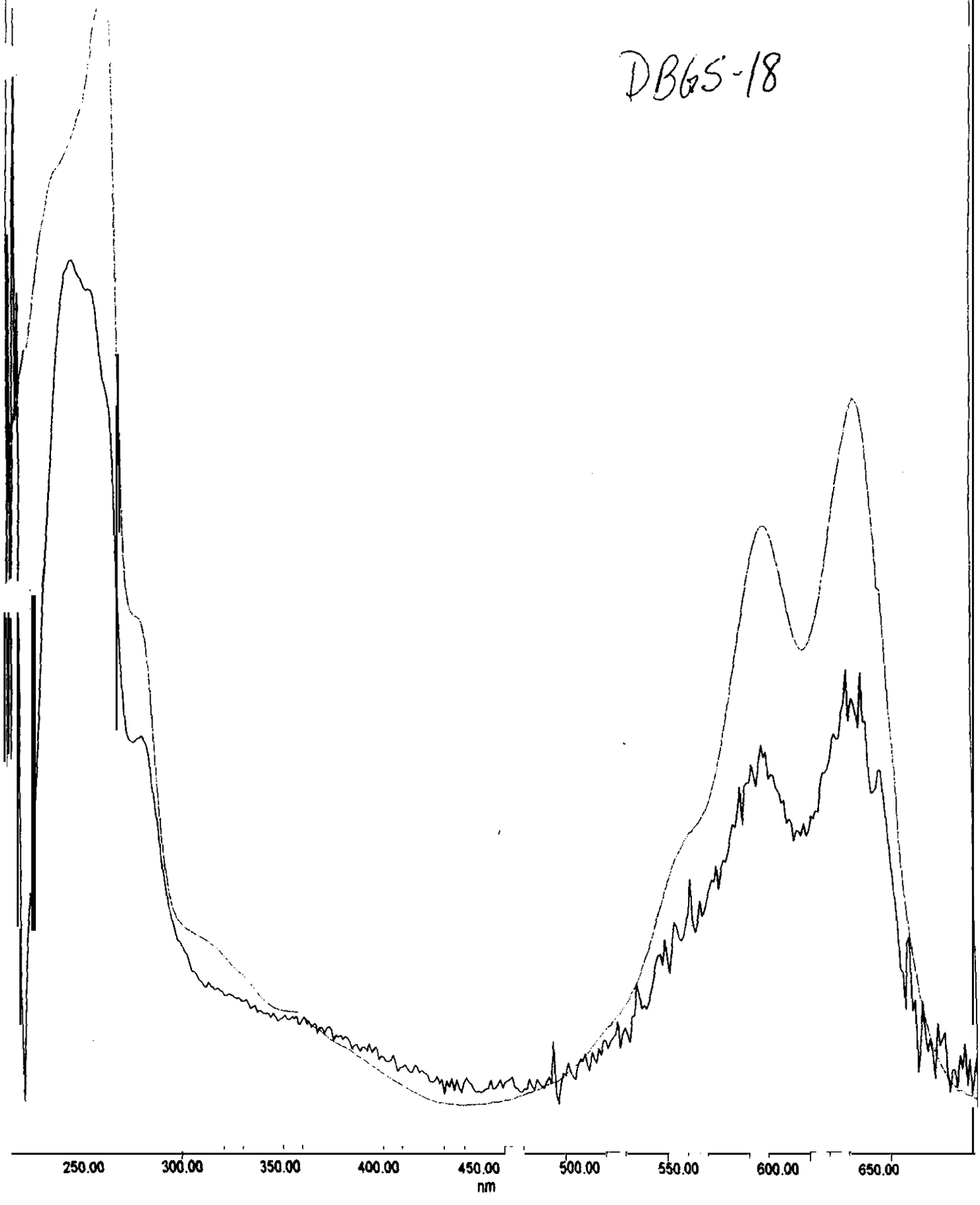
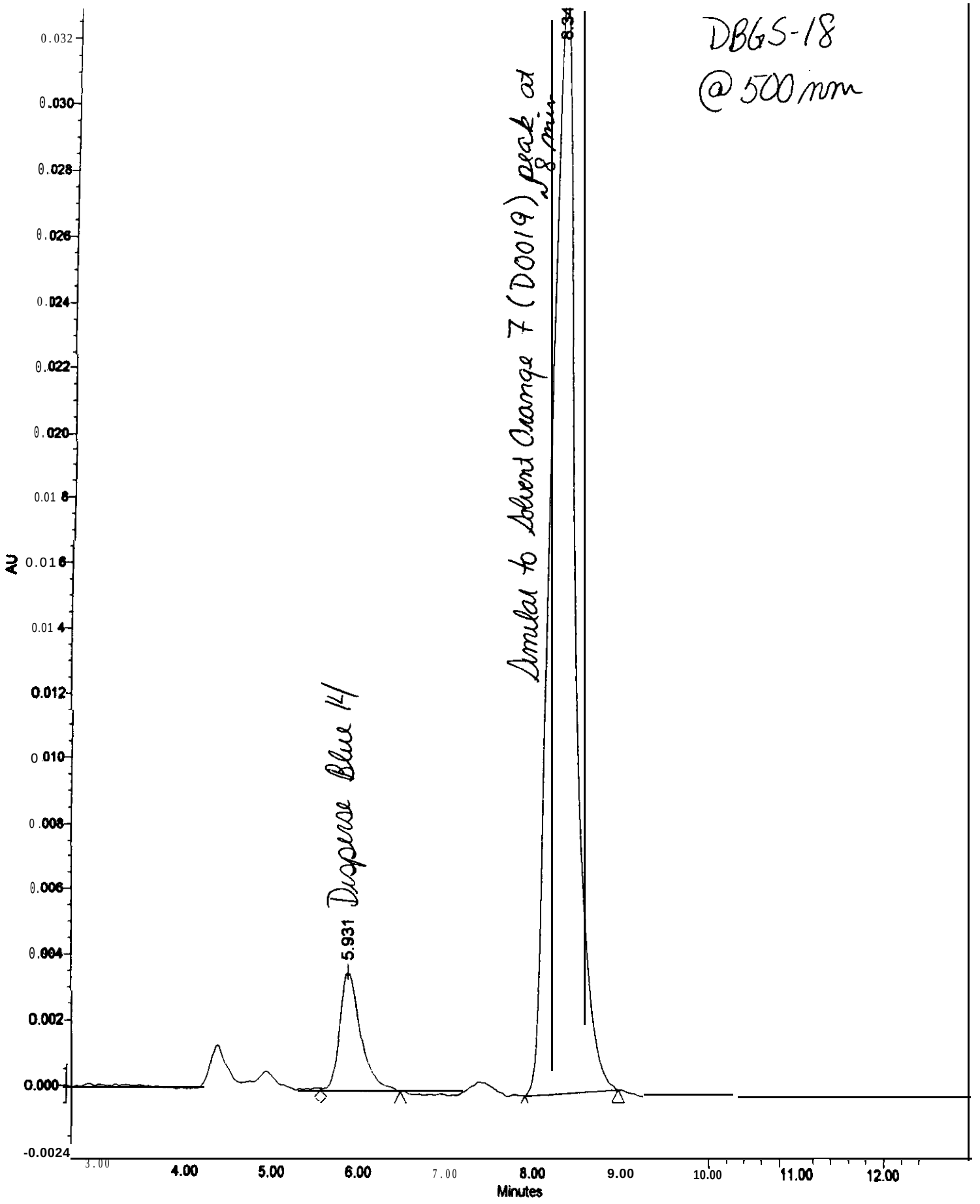
250.00
300.00
350.00
400.00
450.00
nm
500.00
550.00
600.00
650.00


Disperse Blue 14 0.199
DBGS-18
Disperse Blue 14
250.00
300.00
350.00
400.00
450.00
500.00
550.00
600.00
650.00
nm

DBGS-18 – 7.449

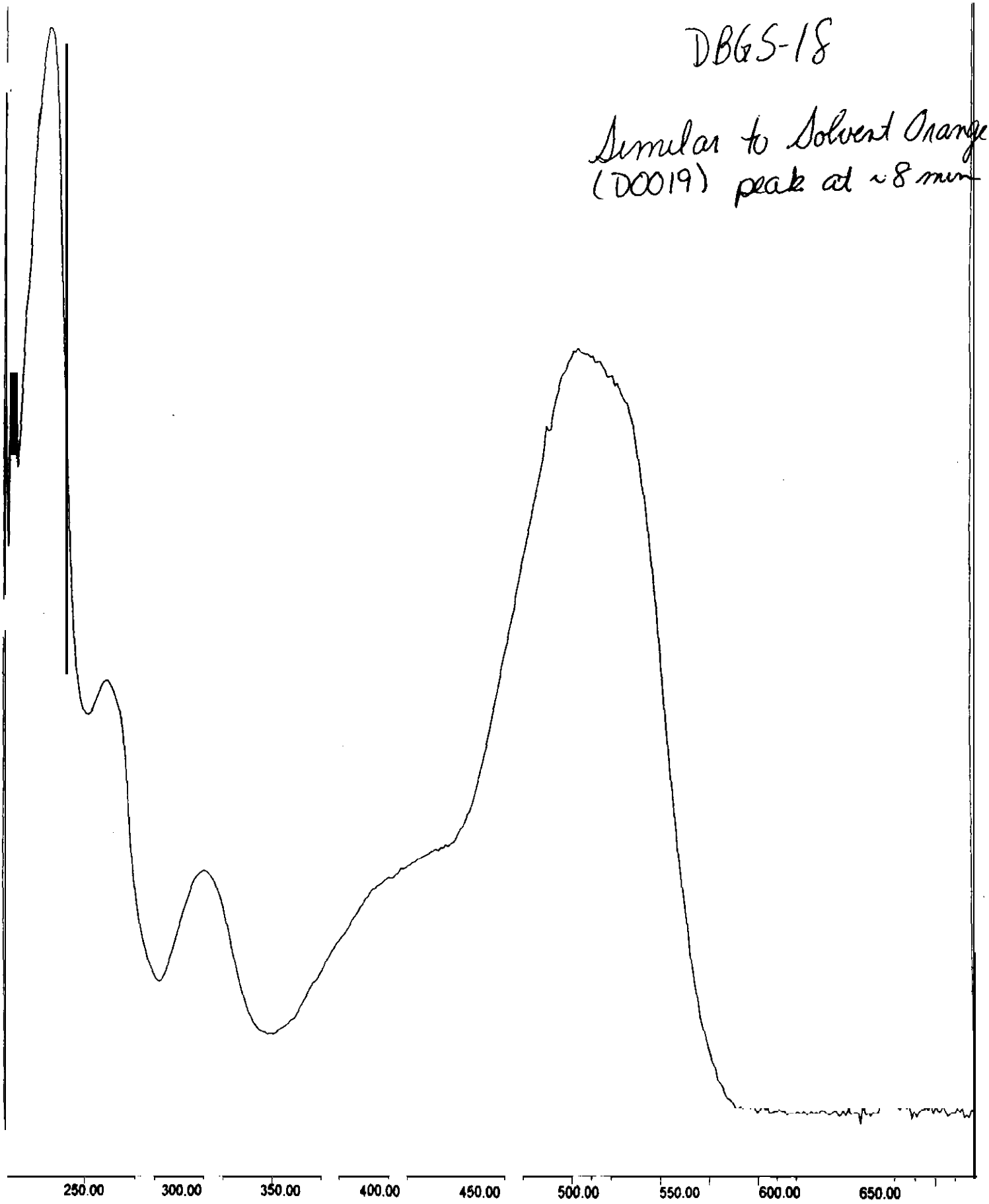
DBGS-18

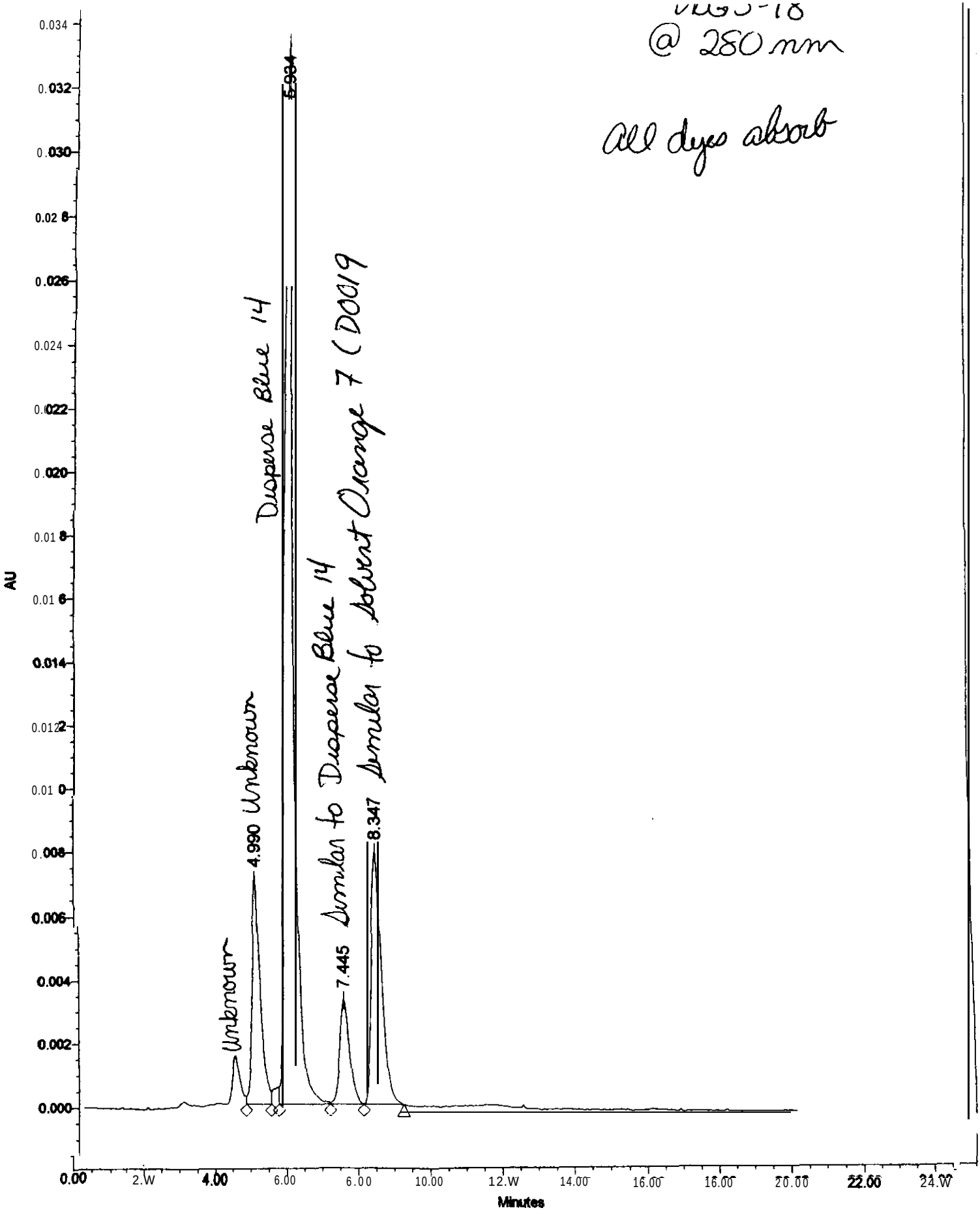



DBGS-18
@ 500 nm
Similar to Solvent Orange 7 (D0019) peak at 8 min
Disperse Blue 14
5.931
8.34
AU
Minutes
0.032
0.030
0.028
0.026
0.024
0.022
0.020
0.018
0.016
0.014
0.012
0.010
0.008
0.006
0.004
0.002
0.000
-0.0024
3.00
4.00
5.00
6.00
7.00
8.00
9.00
10.00
11.00
12.00

DBGS-18

Similar to Solvent Orange (D0019) peak at ~8 min




@ 280 nm
All dyes absorb
Unknown
4.990
Unknown
Disperse Blue 14
5.934
Similar to Disperse Blue 14
7.445
Similar to Solvent Orange 7 (D0019
8.347
AU
Minutes

ATTACHMENT X

MDLs and RQLS

ATTACHMENT X - REPORTING LIMITS

Refer to Tables 3 and 4 in SOP CR4052-PD-2500 Revision 1 in Section 12 of this report

ATTACHMENT X – REPORTING LIMITS

Several different "reporting limits" were developed for the dye methodology.

MDL – Method Detection Limit, Calculated from the low level spikes for each extraction procedure and long and short gradient analyses.

RQL - Reliable Quantitation Limit. Calculated as 4X the MDL as per the Guideline for Method Development.

RL – Reporting Limit. Calculated from the lowest analytical standard adjusted for sample aliquot, percent solids, and dilution factors.

SCL - Spectral Confirmation Limit. The lowest concentration which can be confirmed spectrally.

RBL – Risk Based Limit. Guidelines will be developed by EPA. TtNUS developed some preliminary limits as an aid to method development. The RBLs will be updated pending EPA approval. More RBLs will be added as information becomes available.

Tables Contained in Attachment X

Table X - 1	Limits of Detection, Aqueous Low Method, Short Gradient
Table X - 2	Limits of Detection, Aqueous Low Method, Long Gradient
Table X - 3	Limits of Detection, Solids, Short Gradient
Table X - 4	Limits of Detection, Solids, Long Gradient

TABLE X-I

LIMITS OF DETECTION
AQUEOUS LOW
METHOD
SHORT GRADIENT

Aqueous Low Method					
Chemical	MDL (ug/L)	RQL (ug/L)	RL (ug/L)	SCL (ug/L)	RBL (ug/L)
Acid Blue 1	55.3	221	3189.0	2663.0	450
Acid Blue 9	27.5	110	2694.0	2263.0	230,000
Acid Blue 45	79.1	316	4075.0	2325.0	
Acid Orange 10	39.3	157	3938.0	2513.0	100
Acid Red 64	17.1	68	2688.0	2688.0	
Acid Yellow 3	104.3	417	1865.0	1865.0	
Acid Yellow 23	92.7	370	1620.0	1620.0	
Acid Yellow 73	19.7	79	2623.0	2263.0	
1-Amino-anthraquinone	18.7	75	2264.0	1606.0	
2-Amino-anthraquinone	10.9	44	1656.0	1656.0	2
Basic Violet 10	20.8	83	1846.0	1846.0	570
Basic Yellow 2	384.6	1537	1683.0	2313.0	60
Disperse Blue 14	60.7	843	1873.0	1869.0	
Disperse Red 9	20.7	83	1663.0	1651.0	
Disperse Violet 1	35.5	142	3059.0	1626.0	
Solvent Green 3	45.2	180	2054.0	3675.0	
Solvent Orange 3	28.7	115	1868.0	1599.0	600
Solvent Orange 7	97.4	389	2599.0	1800.0	730
Solvent Red 1	84.6	338	2115.0	2115.0	
Solvent Red 24	27.9	112	710.0	1420.0	
Solvent Yellow 2	23.7	95	1846.0	3990.0	0.01
Solvent Yellow 3	54.1	216	4286.0	1880.0	0.02
Solvent Yellow 14	33.7	135	1769.0	1769.0	460
Solvent Yellow 33	20.5	82	1531.0	1531.0	
Anthracene	115.9	463	2889.0	2596.0	
Benzoanthrone	30.4	122	2431.0	1520.0	

TABLE X-2

LIMITS OF DETECTION
AQUEOUS LOW METHOD
LONG GRADIENT

Aqueous Low Method					
Chemical	MDL (ug/L)	RQL (ug/L)	RL (ug/L)	SCL (ug/L)	RBL (ug/L)
Acid Blue 1	43.8	175	2663.0	2663.0	450
Acid Blue 9	23.5	94	2263.0	2263.0	230,000
Acid Blue 45	13.1	52	2325.0	2325.0	
Acid Orange 10	21.4	85	2513.0	2513.0	100
Acid Red 64	32.9	132	6250.0	2688.0	
Acid Yellow 3	24.6	98	2288.0	1865.0	
Acid Yellow 23	18.3	73	2513.0	1620.0	
Acid Yellow 73	19.9	79	2263.0	2263.0	
1-Amino-anthraquinone	13.9	56	1606.0	1606.0	
2-Amino-anthraquinone	1.7	7	2336.0	1656.0	2
Basic Violet 10	15.4	61	1988.0	1846.0	570
Basic Yellow 2	302.3	1208	2313.0	2313.0	60
Disperse Blue 14	31.7	127	1869.0	1869.0	
Disperse Red 9	19.6	78	1651.0	1651.0	
Disperse Violet 1	11.5	46	1626.0	1626.0	
Solvent Green 3	35.5	142	1636.0	3675.0	
Solvent Orange 3	25.9	104	1599.0	1599.0	600
Solvent Orange 7	71.1	284	1800.0	1800.0	730
Solvent Red 1	93.0	371	2899.0	2115.0	
Solvent Red 24	21.5	86	710.0	1420.0	
Solvent Yellow 2	25.3	101	1995.0	3990.0	0.01
Solvent Yellow 3	24.2	97	1888.0	1880.0	0.02
Solvent Yellow 14	38.9	156	1770.0	1769.0	460
Solvent Yellow 33	24.3	97	1748.0	1531.0	
Anthracene	92.3	389	2596.0	2596.0	
Benzoanthrone	19.2	77	1520.0	1520.0	

TABLE X-3

LIMITS OF DETECTION
SOLIDS
SHORT GRADIENT

Solids					
Chemical	MDL (µg/kg)	RQL (µg/kg)	RL (µg/kg)	SCL (µg/kg)	RBL (µg/kg)
Acid Blue 1	555.0	2219	3189.0	2663.0	10,000
Acid Blue 9	142.5	568	2694.0	2263.0	1,630,000
Acid Blue 45	806.1	3173	4075.0	2325.0	
Acid Orange 10	529.9	2160	3938.0	2513.0	6,460
Acid Red 64	246.6	948	2688.0	2688.0	
Acid Yellow 3	152.5	590	1865.0	1865.0	
Acid Yellow 23	390.3	1638	1620.0	1620.0	
Acid Yellow 73	192.5	738	2623.0	2263.0	
1-Amino-anthraquinone	439.6	1681	2264.0	1606.0	
2-Amino-anthraquinone	146.8	574	1656.0	1656.0	14,700
Basic Violet 10	177.6	720	1846.0	1846.0	12,000
Basic Yellow 2	1427.2	5382	1683.0	2313.0	1,300
Disperse Blue 14	911.4	3607	1873.0	1869.0	
Disperse Red 9	219.5	830	1663.0	1651.0	
Disperse Violet 1	473.8	1862	3059.0	1626.0	
Solvent Green 3	1158.5	4652	2054.0	3675.0	
Solvent Orange 3	65.3	262	1868.0	1599.0	129,000
Solvent Orange 7	804.3	3163	2599.0	1800.0	52,000
Solvent Red 1	2316.1	9228	2115.0	2115.0	
Solvent Red 24		0	710.0	1420.0	
Solvent Yellow 2	202.9	745	1846.0	399.0	110
Solvent Yellow 3	433.3	1506	4286.0	1880.0	130
Solvent Yellow 14	78.2	309	1769.0	1769.0	3,230
Solvent Yellow 33	143	534	1531.0	1531.0	
Anthracene	343.6	1302	2889.0	2596.0	
Benzoanthrone	184.3	648	2431.0	1520.0	

TABLE X-4

LIMITS OF DETECTION
SOLIDS
LONG GRADIENT

Solids					
Chemical	**MDL (ug/kg)**	**RQL (ug/kg)**	**RL (ug/kg)**	**SCL (ug/kg)**	**RBL (ug/kg)**
Acid Blue 1	522.9	2092	2663.0	2663.0	10,000
Acid Blue 9	89.5	358	2263.0	2263.0	1,630,000
Acid Blue 45	407.1	1628	2325.0	2325.0	
Acid Orange 10	261.7	1047	2513.0	2513.0	6,460
Acid Red 64	325.8	1302	6250.0	2688.0	
Acid Yellow 3	2029.5	8110	2288.0	1865.0	
Acid Yellow 23	512.3	2049	2513.0	1620.0	
Acid Yellow 73	168.8	675	2263.0	2263.0	
1-Amino-anthraquinone	242.5	969	1606.0	1606.0	
2-Amino-anthraquinone	230.0	919	2336.0	1656.0	14,700
Basic Violet 10	146.0	583	1988.0	1846.0	12,000
Basic Yellow 2	1332.8	5331	2313.0	2313.0	1,300
Disperse Blue 14	390.9	1562	1869.0	1869.0	
Disperse Red 9	176.7	706	1651.0	1651.0	
Disperse Violet 1	949.2	3797	1626.0	1626.0	
Solvent Green 3	697.9	2789	1838.0	3675.0	
Solvent Orange 3	56.7	227	1599.0	1599.0	129,000
Solvent Orange 7	176.2	704	1800.0	1800.0	52,000
Solvent Red 1	2295.6	9174	2899.0	2115.0	
Solvent Red 24		0	710.0	1420.0	
Solvent Yellow 2	221.0	883	1995.0	3990.0	110
Solvent Yellow 3	194.6	778	1888.0	1880.0	130
Solvent Yellow 14	94.7	378	1770.0	1769.0	3,230
Solvent Yellow 33	137.4	549	1748.0	1531.0	
Anthracene	236.6	946	2596.0	2596.0	
Benzoanthrone	83.1	332	1520.0	1520.0	

ATTACHMENT XI

FALSE POSITIVES AND NEGATIVES

ATTACHMENT XI – FALSE POSITIVES AND NEGATIVES

The false negative/false positive test was not performed since TLC was abandoned as a screening procedure.

ATTACHMENT XII

STANDARD OPERATING PROCEDURE

Determination of Organic Colorants in Environmental Matrices by High-Performance Liquid Chromatography

Explosive Sciences Branch
Naval Surface Warfare Center Crane
300 Highway 361
Crane, IN 47522

May 9, 2001

Approvals:

Name: Nancey J. Maegerlein
Title: Branch Manager
Signature

Name: Jacquelyn J. Karty
Title: Quality Assurance Officer
Signature

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1.0 SCOPE AND APPLICABILITY

This method is intended for the analysis of organic colorants by high performance liquid chromatography (HPLC) using a Photodiode Array Detector (PDA). This method is used to determine the concentration of the following dyes in aqueous and solid matrices:

Aqueous Soluble			Organic Soluble		
Compound Name	Abbr.	CAS #	Compound Name	Abbr.	CAS #
Acid Blue 1	AB1	129-17-g	Disperse Blue 14	DB14	2475-44-7
Acid Blue 9	AB9	3844-45-g	Disperse Red 9	DR9	82-38-2
Acid Blue 45	AB45	2861-02-l	Disperse Violet 1	DV1	128-95-0
Acid Orange 10	A010	1936-15-8	Solvent Green 3	SG3	128-80-3
Acid Red 64	AR64	N A	Solventt Orange 3	SO3	495-54-5
Acid Yellow 3	AY3	8004-92-o	Solvent Orange 7	SO7	3118-97-6
Acid Yellow 23	AY23	1934-21-o	Solvent Red 1	SR1	1229-55-6
Acid Yellow 73	AY73	518-47-8	Solvent Red 24	SR24	85-83-6
Basic Violet 10	BV10	81-88-9	Solventt Yellow 2	SY2	60-11-7
Basic Yellow 2	BY2	2465-27-2	Solvent Yellow 3	SY3	97-56-3
			Solvent Yellow 14	SY14	842-07-g
			Solvent Yellow 33	SY33	8003-22-3
			1-Aminoanthraquinone	1AQ	82-45-l
			2-Aminoanthraquinone	2AQ	117-79-3

1.1 This method provides a solid phase extraction procedure for dyes in surface or ground water.

1.2 These dyes may be dangerous. When handling samples and stock solutions, follow safety precautions listed in Section 4.0 and 5.0.

2.0 SUMMARY OF THE METHOD

2.1 This method provides HPLC conditions for the detection of ppb and low ppm levels of dyes in aqueous and solid matrices. Prior to using this method, appropriate sample preparation must be used.

2.2 This method utilizes a dual gradient, where flow and mobile phase composition are varied over time, to achieve sufficient separation of the target analytes.

2.3 This method provides two individual gradient methods for the separation of the analytes. The short gradient method (40 minutes total) provides for rapid separation of the analytes with the loss of resolution between two coeluting pairs of analytes (Acid Yellow 3/Acid Yellow 23 and Acid Blue l/Disperse Violet 1). The long gradient method (70 minutes total) provides for the separation of the two coeluting pairs from the short

gradient method. For soils, the long gradient method will only be run if one of the analytes in the coeluting pairs is detected in the short gradient method. The long gradient is used for aqueous samples because the percent recovery is biased high in the short gradient. Therefore, quantification of aqueous samples will be based on the long gradient.

2.4 Aqueous Sample Extraction: This method is an adaptation of a membrane solid phase extraction procedure for nonvolatile organics described in EPA Method SW-846-8330. Aqueous samples are extracted using a Cl8 extraction disk and tetrabutylammonium hydroxide. A small volume of methanol extracts the analytes from the C18 disk and is concentrated by rotary evaporation.

2.5 Soil and Sediment Sample Extraction: Solid samples are extracted with a 5:5:50 v/v mixture of tetrabutylammonium hydroxide (in methanol): water:methanol and filtered prior to analysis.

2.6 Acid Blue 45 exhibits significantly reduced recoveries in particulate-contaminated aqueous matrices. This is due to the preferential binding of Acid Blue 45 to patticulates because of its high polarity.

2.1 Basic Yellow 2 results are biased high in both solid and aqueous matrices. While this method can confirm the presence of basic yellow 2 in a sample matrix, the quantitative results will be significantly higher than the actual amount in the matrix.

2.8 Acid Yellow 23 deviates significantly in ICV and CCV despite considerable effort to correct the problem. Therefore, Acid Yellow 23 is exempt from ICV and CCV criteria and corrective action.

3.0 DEFINITIONS

This section defines the terms and acronyms as used in this SOP.

PDA	Photodiode Array Detector
c c v	Continuing Calibration Verification - Standard that is injected at some prescribed frequency during the analytical run sequence to determine if the instrument is still calibrated.
ICV	Initial Calibration Verification – Standards that are injected at the beginning of a 24 analytical run sequence to determine if the instrument is still calibrated.
LC	Liquid Chromatography

RT	Retention Time The time (in minutes) at which a target analyte elutes from the LC column.
RT Window	Retention Time Window - The +/- value which is applied to the ICV to establish the time range used to make tentative compound identifications.
UVNIS	Ultraviolet/Visible absorption spectra

4.0 HEALTH AND SAFETY-WARNINGS

4.1 Standard laboratory protective clothing and eye covering is required during the execution of this method.

5.0 CAUTIONS

5.1 Refer to the instrument manufacturer's manual for routine instrument precautions. This includes an awareness of moving parts and the potential for electrical shock.

5.2 Waste Disposal -- All waste will be disposed of according to federal, state, and local regulations.

5.3 Routine and Preventative Maintenance

PREVENTIVE MAINTENANCE FOR ANALYTICAL INSTRUMENTS

Instrument	Preventive Maintenance	Maintenance Frequency
HPLC	Change filter frit in mixer.	As needed (when pressure builds)
	Change column pre-filter.	As needed (2-3 months)
	Rinse water pump with methanol, filter water, sonicate water intake filter frit.	Approximately weekly.
	Change pump seals.	As needed.

6.0 INTERFERENCES

6.1 Solvent, reagents, glassware and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences.

6.2 Several dye compounds decompose rapidly when exposed to light. Standards must be kept in a dark cool area until used. Samples and sample extracts must be stored in a dark cool area until analyzed.

7.0 PERSONNEL QUALIFICATIONS

This method is restricted to use by or under the supervision of analysts experienced in the use of HPLC, skilled in the interpretation of chromatograms, and experienced in handling dye materials. Each analyst must demonstrate the ability to generate acceptable results with this method.

8.0 APPARATUS AND MATERIALS

8.1 HPLC System

- Waters 2690 Separations Module (auto-sampler, gradient pump)
- Waters 996 Photodiode Array Detector
- Waters Temperature Control Module
- 30 uL minimum volume sample loop
- Waters Millennium[32] Chromatography Software

The system is equipped with a Waters 2690 Separation Module. The pump is capable of achieving 5000 psi. For the low concentration option, the detector must be capable of a stable baseline at 0.001 absorbance units full scale.

8.2 Other Equipment
- 8.2.1 Temperature controlled ultrasonic bath
- 8.2.2 Balance ± 0.0001 g
- 8.2.3 Oven

8.3 Materials
- 8.3.1 Disposable cartridge filters – 0.45 urn teflon filter
- 8.3.2 Pipettes -Class A, glass, appropriate sizes
- 8.3.3 Pasteur pipettes
- 8.3.4 Vials – appropriate sizes with Teflon-lined cap
- 8.3.5 Disposable syringes – plastipak, 5 cc or equivalent
- 8.3.6 Volumetric flasks – appropriate sizes with ground glass stoppers, Class A.
- 8.3.7 Vacuum desiccator – glass

8.3.8 Mortar and pestle-ceramic
8.3.9 Sieve – 30 mesh

8.4 Preparation of Material:
8.4.1 Glassware will be washed in a phosphate-free detergent with tap water, followed by tap water rinse, and then a final deionized water rinse. Glassware used with organic solvents will be rinsed with a suitable organic solvent, followed by acetone, followed by tap water and then receive the above listed minimum wash. Prior to use all glassware will be rinsed three times with the solvent in use.

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9.0 REAGENTS

9.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.

9.1.1 Methanol (CH3OH), HPLC grade
9.1.2 Tetrabutylammonium Hydroxide (TBA) – HPLC grade, 1.0M in methanol
9.1.3 Organic-free reagent water. All references to water in this method refer to organic-free reagent water, as defined in SW846, Chapter One.

9.2 Stock Standard Solutions
9.2.1 Due to the rarity of some organic colorants, the standards may not be available from commercial sources, and therefore bulk lot samples can be used as standards,
9.2.2 Dry each solid analyte standard to constant weight in a vacuum desiccator in the dark. Place 0.0100 g (weighed to 0.0001 g) of a single analyte into an amber 100 mL volumetric flask and dilute to volume with methanol. Invert flask several times and sonicate for twenty minutes until dissolved. Store in refrigerator at 4 °C in the dark. Calculate the concentration of the stock solutions from the actual weight used. Stock solutions may be used for up to one year.

9.3 Working Standards:
9.3.1 Calibration standards at a minimum of five concentration levels should be prepared for the initial calibration through dilution of the stock standard solutions. Calibration standards should contain multiple target analytes. It is recommended the analytes be grouped to aid in integration. Recommended groupings are as follows: Group 1 – AB45, AY23, AY73, AO10, BY2, DV1, SY33, and DB14; Group 2 – AR64, AB9, BV10, 2AQ, S03, 1AQ, SY14, and S07; Group 3 – SY3, BZ, AN, and SR1; Group 4 – DR9, SY2, SG3, and SR24. An example of the dyes concentration

ranges can be found in Table 5. These solutions must be refrigerated and stored in the dark. Prepare the working calibration standards as follows:

Standard	Source Solution	Amount Added (ml)	Final Volume (mL)
Standard 5	A	10	100
Standard 4	B	50	100
Standard 3	C	50	100
Standard 2	D	25	100
Standard 1	D	5	10

Source Solution
A = Stock Standard (Each Dye)
B = Standard 5
C = Standard 4
D = Standard 3

9.3.2 For continuing calibration, run a midpoint standard of all dyes at the beginning of the day, and run one group of midpoint standards every 10 samples.

9.4 Surrogate Spiking Solutions

9.4.1 The analyst should monitor the performance of the extraction and analytical system as well as the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard and reagent water blank with two surrogates (i.e., analyte not expected to be present in the sample). The surrogates selected for this method are benzanthrone and anthracene. Both are very strong UV absorbing analyte, yet are somewhat colorless.

9.5 Matrix Spiking Solutions

9.5.1 Prepare matrix-spiking solutions in methanol such that the concentration in the sample is ten times the MDL (See Tables 3 & 4). The matrix spiking solution will be made from the stock standard of the individual analytes chosen.

9.6 HPLC Mobile Phase

9.6.1 To prepare 1 L of 0.005M TBA mobile phase, dilute 5ml of 1.0M TBA in methanol to 1 L with methanol in a volumetric flask

10.0 SAMPLE COLLECTION, PRESERVATION, HANDLING AND RECEIPT

10.1 Upon sample receipt, the laboratory's sample custodian examines each cooler's custody seals to verify that they are intact and that the integrity of the environmental samples has been maintained. The sample custodian signs the COC report. The sample custodian then opens the cooler and measures its internal temperature. The temperature reading is noted on the

accompanying COC report. The sample custodian then examines the contents of the cooler. Sample container breakages or discrepancies between the COC report and sample label documentation are recorded. All problems or discrepancies noted during this process are to be promptly related to the field team leader. Inter-laboratory COC procedures and specific procedures for sample handling, storage, disbursement for analysis, and remnant disposal will be followed as specified in the CR4052-PD-0500.

10.2 Samples are collected in amber glass containers with Teflon-lined caps. All samples and sample extracts are stored at 4" C ± 2° C. All samples and sample extracts must be stored in the dark until analysis to avoid decomposition of the dyes. Based on guidelines listed in SW-846 for the extraction of non-volatile compounds, aqueous samples must be extracted within 7 days of collection and solid samples must be extracted within 14 days of collection. All extracts must be analyzed within 40 days of sample preparation.

11.0 PROCEDURE

11.1 Sample Preparation

11.1.1 Aqueous Samples: Solid-phase extraction

11.1.1.1 Using a 1 L volumetric flask, add 5 mL of tetrabutylammonium hydroxide (in methanol) by pipette.

11.1.1.2 Dilute to volume with aqueous sample. Mix the contents well before extraction.

11.1.1.3 If the sample contains sediment, pour the contents of the 1 L volumetric flask into a 1 L beaker and allow the sediment to settle to the bottom. Pour the water portion onto the solid phase extraction disk first, then add the sediment after the water has been pulled through but prior to extraction with methanol.

11.1.1.4 Pull sample through the Cl8 solid phase extraction disk using the vacuum manifold apparatus. A flow rate of 10 to 25 mL/min through the SPE disk is optimal for target analyte recovery. Record the flow rate through the extraction disk in the laboratory notebook.

11.1.1.5 Dry the disk using the vacuum manifold apparatus.

11.1.1.6 Switch the stopcock to the receiving vessel and pull through a minimum of 30 mL of methanol.

11.1.1.7 Evaporate to 1 mL using a rotary evaporator.

11.1.1.8 Transfer extract to a 10 mL volumetric flask. Rinse the round bottom flask with methanol and transfer

rinse to the 10 mL volumetric flask. Dilute with methanol to volume.

11.1.2 Soil and Sediment Samples

11.1.2.1 Dry the soil samples in air at room temperature (or colder) to a constant weight, being careful not to expose the samples to direct sunlight. Grind and homogenize the dried sample thoroughly in an acetonitrile-rinsed mortar to pass a 30-mesh sieve.

11.1.2.2 Place a 10.0-g sub-sample of each soil sample in a -- 250-mL volumetric flask. Add 5.0 mL of tetrabutylammonium hydroxide (in methanol) and 5.0 mL organic-free water. Place the stopper on the flask, shake for 1 minute, then place in a cooled ultrasonic bath for 0.5 hour.

11.1.2.3 After sonication, shake sample for 1 minute, then add 50.0 mL of methanol. Shake for 1 minute then place in a cooled ultrasonic bath for 1.0 hour.

11.1.2.4 Transfer approximately 4 mL of the slurry in a disposable syringe and filter it through a 0.45-micron nylon filter. Discard the first 1 mL and retain the remainder in a Teflon-capped vial for HPLC analysis.

11.2 Chromatographic Conditions: Short Gradient

11.2.1 Recommended Column: Waters Xterra MS, Cl8 column, 4.6mm x 100mm, 3.5um particle size

11.2.2 Mobile Phase: High Purity Water/0.005 M Tetrabutylammonium Hydroxide (TBA) in Methanol

11.2.3 Dual Gradient: Run time = 35 min

11.2.3.1 Initial Parameters:

75:25 Water:TBA
1.0 mL/min
Column Temp = 30°C

11.2.3.2 Final Parameters:

0: 100 Water:TBA
1.0 mL/min
Column Temp = 30°C

11.2.4 Mobile phase flow and composition are varied on a linear gradient throughout the analytical run. The instrument is allowed to come to equilibrium after returning to the initial parameters for 5 min prior to the next run.

11.2.5 Detector Conditions: PDA detector is set to scan from 21 lnm to 700nm. To maximize sensitivity, extract and integrate a separate

chromatogram for each dye at the appropriate, most sensitive, analytical wavelength according to the following chart:

Water Soluble Dyes

Dye Name	Abbr.	Analytical Wavelength (nm)
Acid Blue 1	AB1	635
Acid Blue 9	AB9	628
Acid Blue 45	AB45	633
Acid Orange 10	AO10	485
Acid Red 64	AR64	524
Acid Yellow 3	AY3	383
Acid Yellow 23	AY23	404
Acid Yellow 73	AY73	493
Basic Violet 10	BV10	549
Basic Yellow 2	BY2	378

Organic Soluble Dyes

Dye Name	Abbr.	Analytical Wavelength (nm)
Anthracene	AN	251
Benzanthrone	BZ	399
Disperse Blue 14	DB14	638
Disperse Red 9	DR9	513
Disperse Violet 1	DV1	588
Solvent Green 3	SG3	635
Solvent Orange 3	SO3	408
Solvent Orange 7	SO7	499
Solvent Red 1	SR1	504
Solvent Red 24	SR24	519
Solvent Yellow 2	SY2	416
Solvent Yellow 3	SY3	385
Solvent Yellow 14	SY14	486
Solvent Yellow 33	SY33	381
1-Aminoanthraquinone	1AQ	481
2-Aminoanthraquinone	2AQ	299

Example UVNIS spectra of each dye and its approximate retention time can be found in Attachment 1

11.3 Chromatographic Conditions: Long Gradient

11.3.1 Recommended Column: Waters Xterra MS, Cl8 column, 4.6mm x 100mm, 3.5um particle size

11.3.2 Mobile Phase: High Purity Water/0.005 M Tetrabutylammonium Hydroxide (TBA) in Methanol

11.3.3 Dual Gradient: Run time ≈ 65 min

11.3.3.1 Initial Parameters:

75:25 Water:TBA
0.7 mL/min
Column Temp = 30°C

11.3.3.2 Final Parameters:

0:100 Water:TBA
1.0 mL/min
Column Temp = 30°C

11.3.4 Mobile phase flow and composition are varied on a linear gradient throughout the analytical run. The instrument is allowed to come to equilibrium after returning to the initial parameters for 5 min prior to the next run.

11.3.5 Detector Conditions: Same as those described in Section 11.25

11.4 Calibration of HPLC:

The entire HLPC system is allowed to warm up for a minimum of 30 minutes. During this time the mobile phase, at the initial gradient conditions, should be pumped through the column.

11.4.1 Initial Calibration

11.4.1.1 Injections of each calibration standard over the concentration range of interest are preformed. Peak heights and peak areas are obtained for each analyte at the appropriate wavelength.

11.4.2 Daily Calibration

11.4.2.1 Analyze the midpoint calibration standards along with an instrument blank at the beginning of each day and after each set of 10 samples or less. Obtain the response factor for each analyte from the mean peak heights or peak areas and compare it with the response factor obtained for the initial calibration. The mean response factor for the daily calibration must agree within ±25% of the response factor of the initial calibration. The same criterion is required for subsequent standard responses. If this criterion is not met, a new initial calibration must be obtained. Note exemption in Section 2.8.

11.5 HPLC Analysis

11.5.1 Analytical Sequence

All acceptable samples must be analyzed within a valid analysis sequence as given below:

Initial Calibration:

Injection Number	Material Injected
1	Instrument Blank
2-21	Five Initial Calibration Standards
22	Instrument Blank at end of initial calibration

Daily Analysis Sequence:

Time	Injection Number	Material Injected
0hr	1	Instrument blank
	2 – 5	Midpoint standard of all dyes (ICV)
	6	Instrument Blank
	7	LCS
	8 – 18	Sample Analyses
~12 hr	19	Instrument Blank
	20	Mid-level CCV of one group of dyes
	21	LCS
	22 – 32	Sample Analyses
~12 hr	33	Instrument Blank
	34	Mid-level CCV of one group of dyes

After the initial calibration, the analysis sequence may continue as long as acceptable instrument blanks and continuing calibration verifications are analyzed at the required frequency. This analysis sequence shows only the minimum required blanks and standards. More blanks and standards may be run at the discretion of the analysts; these must also satisfy the criteria presented in Section 12 in order to continue the run sequence.

An analysis sequence must include all required matrix spike/matrix spike duplicate and method blank analyses. The analysts may decide at what point in the sequence they are to be analyzed.

11.5.2 Laboratory Control Sample (LCS)

11.5.2.1 The LCS (reagent blank spike) should be prepared separately from the standards and include at least 6 of the dyes, 3 water soluble and 3 organic soluble. The concentration of the dyes should be approximately 500 µg/L.

11.5.3 Follow the guidelines found in SW846, Method 8000 for the following parameters.

11.5.3.1 Retention Windows

11.5.3.1.1 Retention time windows are established by performing 7 replicate injections over a 72 hour period. Retention time windows are set at 3 times the standard deviation of the replicate injections.

11.5.3.2 Any modifications to the RTW

11.5.3.2.1 If the retention time of any analyte in the standards at the beginning of the analytical shift does not fall within the ±3 standard deviation window, then a new initial calibration is necessary unless system maintenance corrects the problem.

11.5.3.3 Initial Calibration

11.5.3.3.1 The initial calibration involves the analysis of standards containing each one of the analytes at five concentrations over the working range of the instrument.

11.5.3.4 Initial Calibration Verification

11.5.3.4.1 The initial calibration is verified at the beginning of each 12-hour analytical shift. The process involves the analysis of the mid-point standard for each group of dyes. If the response for the analyte is within ±25% of the response obtained during the initial calibration, then the initial calibration is considered still valid. Note exemption in Section 2.8.

11.5.3.5 Instrument Blank

11.5.3.5.1 An instrument blank is analyzed prior to injection of the standards, after the analysis of all standards, and after every 10 samples.

11.5.3.6 Continuing Calibration Verification

11.5.3.6.1 The continuing calibration verification process involves the analysis of the mid-point standard for one group of dyes. If the response for the analyte is within ±25% of the response obtained during the initial calibration, then the initial calibration is considered still valid. The CCV is analyzed every 10 samples. Note exemption in Section 2.8

11.5.3.7 Sample Analysis

11.5.3.7.1 An autosampler is used to inject 10 to 100 µL of a sample. Samples are analyzed according to the sequence in 11.5.1.

11.5.4 Compound Identification

11.5.4.1 Compound identification is achieved through spectral matching with known standards of the dyes.

11.5.4.2 In the event of co-elution problems, the analyst will attempt to separate the co-eluting compounds using either the short or the long gradient as appropriate. Re-analysis of the sample to address co-elution problems will occur only if a target dye is positively identified (by retention time and spectra) and is co-eluting with another positively identified target dye. All samples must be analyzed within a valid analysis sequence as defined in Section 11.5.

11.5.5 Compound Quantification

11.5.5.1 Data Acquisition, Calculations and Data Reduction

11.5.5.1.1 All data acquisition, chromatogram integration, calibration curve calculation and unknown concentration calculation is preformed utilizing the Waters Millennium 32 chromatography software.

11.5.5.1.2 Manual calculations such as standard deviation and percent recovery will be performed utilizing a spreadsheet.

11.5.6 Data Management and Records Management

11.5.6.1 All documents are managed according to CR4052-PD-0100 and CR4052-PD-0150. Raw data is stored on using the Millennium 32 chromatography software. The data is backed-up monthly.

12 QUALITY CONTROL AND QUALITY'ASSURANCE

12.1 Initial Calibration

12.1.1 Criteria: Analyze standards at five concentrations over working range of instrument. Linearity is determined by an RSD $\leq$ 30% or a coefficient of determination greater or equal to 0.99

12.1.2 Corrective Action: If the initial calibration does not meet the criteria for linearity, reduce/increase the concentration of the upper/lower standards until the criteria is meet.

12.2 Initial Calibration Verification

12.2.1 Criteria: The initial calibration is verified at the beginning of each 12-hour analytical shift. A mid-point standard for each group of dyes is analyzed. If the response for the analyte is within ±25% of the response obtained during the initial calibration, then the initial calibration is considered still valid. Up to 5 dyes are allowed to be outside this tolerance for the initial calibration to be considered still valid. Note exemption, in Section 2.8.

12.2.2 Corrective Action: If the initial calibration is no longer valid, a new initial calibration must be performed unless system maintenance corrects the problem.

12.3 Continuing Calibration Verification

12.3.1 Criteria: The continuing calibration verification process involves the analysis of a mid-point standard for one group of dyes. If the response for the analyte is within ±25% of the response obtained during the initial calibration, then the initial calibration is considered still valid. Up to 2 dyes are allowed to be outside this tolerance for the initial calibration to be considered still valid. The CCV is analyzed every 10 samples. Note exemption in Section 2.8.

12.3.2 Corrective Action: If the initial calibration is no longer valid, a new initial calibration must be performed unless system maintenance corrects the problem.

12.4 Instrument Blank

12.4.1 Criteria: An instrument blank is analyzed prior to injection of the standards, after the analysis of all standards, and after every 10 samples. The results of the instrument blank should be less then the IDL.

12.4.2 Corrective Action: If the instrument blank does not meet the acceptance criteria, then the source of the contamination will be located and reduced. The samples associated with the contaminated instrument blank will be re-analyzed.

12.5 Method Blank

12.5.1 Criteria: A clean sample will be extracted using the appropriate procedure and analyzed. The results of the method blank should be less then the MDL or less than 5% of the sample result for the same analyte. A method blank will be prepared for each group of 20 samples.

12.5.2 Corrective Action: If the method blank does not meet the acceptance criteria, then the source of the contamination will be located and reduced. The samples associated with the contaminated method blank will be re-extracted and re-analyzed.

12.6 Laboratory Control Sample

12.6.1 Criteria: A clean matrix similar to the sample matrix and of the same weight or volume will be spiked with the same analytes at the same concentrations as the matrix spike, extracted using the same method, and analyzed. When the results of the matrix spike analysis indicates a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

12.6.2 Corrective Action: If the LCS does not meet the acceptance criteria, the source of the problem will be located and corrected.

12.7 Matrix Spike

12.7.1 Criteria: A sample matrix will be spiked with a minimum of 2 water-soluble. dyes, 2 organic-soluble dyes, and the surrogate standards, extracted using the same method, and analyzed. Percent recovery is calculated.

12.7.2 Corrective Action: If the matrix spike indicates there may be interference from the matrix, then the LCS will be used to verify that the laboratory can perform the analysis in a clean matrix.

12.8 Matrix Spike Duplicate

12.8.1 Criteria: The same matrix used for the matrix spike will be spiked with the same analytes at the same concentrations as the matrix spike. Percent recovery is calculated

12.8.2 Corrective Action: If the matrix spike indicates there may be interference from the matrix, then the LCS will be used to verify that the laboratory can perform the analysis in a clean matrix.

12.9 Surrogate Standards

12.9.1 Criteria: A solution of benzanthrone and anthracene will be spiked on the sample matrix. Percent recovery is calculated.

13 REPORTS

13.1 Electronic Deliverables: Final results will be provided in a Microsoft Excel format. The following information will be included in the Excel sheet: sample number, laboratory identification, laboratory name, associated blank, quality control type, sample date, receipt date, extraction date, analysis date, run number, parameter, CAS number, method, laboratory results, instrument detection limits, method detection limits, percent moisture, percent recovery of surrogates, and comments. Another Excel sheet will report RSD and coefficients of determination for the initial calibration curves, and tabulated results of the daily and continuing calibration standards.

13.2 Full data package: In addition to the electronic deliverables, the full data package will include chromatograms, HPLC concentration results, and UV-Vis spectra for the initial calibration, daily calibration, continuing calibration, instrument blanks, method blanks, and each sample. Full package will be provided when there is a positive detection of a target analyte.

13.3 Abbreviated data package: In addition to the electronic delieverables, the field screening data package will include chromatograms and HPLC concentration results for the initial calibration, daily calibration, and

continuing calibration. Chromatograms for the samples will be provided to prove the absence of detectable dyes. The abbreviated **data** package will be provided when no target analyte is detected in the sample.

13.4 If an analyte is noted on the chromatogram **but** can not be confirmed by its UV-Vis spectra, then "NSC" (no spectra confirmation) will be noted in the comments section of the Electronic Deliverables

14 REFERENCES

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14.1 U.S. Environmental Protection Agency, Method SW-846-8330.

14.8 U.S. Environmental Protection Agency, Method SW-846-8000.

14.9 U.S. Environmental Protection Agency, Method SW-846-3500.

14.10 U.S. Environmental Protection Agency, Method SW-846-3550.

15 REVISION BLOCK

REV	DATE	REVISION	AUTHOR
1	5/09/01	Changed holding times from stable organic compound requirements to non-volatile compound requirements. Added Section 11.5.4.2	JJK

TABLE 1
Quality Control Limits
Matrix Spike/Matrix Spike Duplicate Samples and Surrogate Spikes
Dye Analyses by the Short Gradient
Naval Surface Warfare Center
Crane, Indiana

Chemical	Solid Matrix	
	Accuracy (%R)	Precision (RPD)
Acid Blue 9	3-9	35
Acid Blue 45	46-75	35
Acid Orange 10	191-213	35
Acid Red 64	61-69	35
Acid Yellow 23	55-74	35
Acid Yellow 73	61-68	35
1-Aminoanthraquinone	73-94	35
2-Aminoanthraquinone	42-46	35
Basic Violet 10	94-105	35
Disperse Blue 14	34-73	35
Disperse Red 9	40-48	35
Disperse Violet 1	45-68	35
Solvent Green 3	25-72	35
Solvent Orange 3	51-54	35
Solvent Orange 7	58-94	35
Solvent Red 1	25-88	35
Solvent Red 24	TBD	TBD
Solvent Yellow 2	41-47	35
Solvent Yellow 3	210-225	35
Solvent Yellow 14	41-44	35
Solvent Yellow 33	35-41	35
Anthracene	42-57	NA
Benzanthrone	99-113	NA

NA : Not applicable

QC limits are based on the seven low spike samples analyzed during method performance/validation. Matrix spike QC limits are the same as laboratory control sample QC limits. Limits are not presented for aqueous samples since they will be quantitated using the long gradient.

TABLE 2
Quality Control Limits
Matrix Spike/Matrix Spike Duplicate Samples and Surrogate Spikes
Dye Analyses by the Long Gradient
Naval Surface Warfare Center
Crane, Indiana

Chemical	Aqueous Matrix (1)	
	Accuracy (%R)	Precision (RPD) (2)
Acid Blue 9	16-113	37
Acid Blue 45	0-45	53
Acid Orange 10	50-114	20
Acid Red 64	43-109	22
Acid Yellow 23	48-111	20
Acid Yellow 73	47-110	20
1-Aminoanthraquinone	41-96	20
2-Aminoanthraqumone	36-87	21
Basic Violet 10	46-111	21
Disperse Blue 14	35-109	26
Disperse Red 9	46-115	22
Disperse Violet 1	46-100	20
Solvent Green 3	2-84	47
Solvent Orange 3	41-92	20
Solvent Orange 7	37-65	20
Solvent Red 1	38-74	20
Solvent Red 24	34-102	25
Solvent Yellow 2	47-104	20
Solvent Yellow 3	49-116	20
Solvent Yellow 14	42-88	20
Solvent Yellow 33	46-106	20
Anthracene	0-106	NA
Benzanthrone	32-120	NA

NA: Not Applicable
Solid samples will be quantitated by the short gradient.

1. QC limits based on 11 low spike DI water samples analyzed during Rounds 2 and 3 of the Method Validation Study. New limits for groundwater will be developed when sufficient data has been generated.

2. RPD limits of 20% for aqueous and 35% for solids were substituted for the calculated value in instances where the calculated RPD was less than the 20% or 35% limit.

TABLE 3
Analytical Methods and Limits of Detection
For the Short Gradient
Naval Surface Warfare Center
Crane, Indiana

Chemical	Laboratory MDL		Laboratory RL		Risk-Based Target Level	
	Aqueous (ug/L)	Solid (ug/kg)	Aqueous (ug/L)	Solid (ug/Kg)	Aqueous (ug/L)	Solid (ug/kg)
Acid Blue 1	55.3	558.9	26.63	15978	450	10000
Acid Blue 9	27.5	141.8	22.63	13578	230000	1630000
Acid Blue 45	79.1	791.6	23.25	13950		
Acid Orange 10	39.3	522.7	25.13	15078	100	6460
Acid Red 64	17.1	240.7	62.5	37500		
Acid Yellow 3	104.3	149.0	22.88	13728		
Acid Yellow 23	92.7	403.7	25.13	15078		
Acid Yellow 73	19.7	179.9	22.63	13578		
1-Amino-anthraquinone	18.7	425.6	16.06	9636		
2-Amino-anthraquinone	10.9	138.7	23.36	14016	2	14700
Basic Violet 10	20.8	177.2	19.88	11928	570	12000
Basic Yellow 2	384.6	1350.6	23.13	13878	60	1300
Disperse Blue 14	60.7	912.5	18.69	11214		
Disperse Red 9	20.7	213.1	16.51	9906		
Disperse Violet 1	35.5	467.8	16.26	9756		
Solvent Green 3	45.2	1156.9	18.38	11028		
Solvent Orange 3	28.7	65.3	15.99	9594	600	129000
Solvent Orange7	97.4	799.3	18.00	10800	730	52000
Solvent Red 1	84.6	2304.2	28.99	17394		
Solvent Red 24	27.9	0	7.1	4260		
Solvent Yellow 2	23.7	192.5	19.95	11970	0.01	110
Solvent Yellow 3	54.1	391.2	18.88	11328	0.02	130
Solvent Yellow 14	33.7	80.7	17.70	10620	460	3230
Solvent Yellow 33	20.5	136.5	17.48	10488		
Anthracene	115.9	331.9	25.96	15576		
Benzanthrone	30.4	176.2	15.2	9120		

The risk-based screening levels were developed by USEPA Region 5 specifically for this project

TABLE 4
Analytical Methods and Limits of Detection
For the Long Gradient
Naval Surface Warfare Center
Crane, Indiana

Chemical	Laboratory MDL		Laboratory RL		Risk-Based Target Level	
	Aqueous (ug/L)	Solid (ug/kg)	Aqueous (ug/L)	Solid (ug/Kg)	Aqueous (ug/L)	Solid (ug/kg)
Acid Blue 1	43.8 (1)	523.4	26.63	15978	450	10000
Acid Blue 9	25.1	89.5	22.63	13578	230000	1630000
Acid Blue 45	26.7	407.5	23.25	13950		
Acid Orange 10	13.0	262.0	25.13	15078	100	6460
Acid Red 64	12.1	325.8	62.5	37500		
Acid Yellow 3	24.6 (1)	2029.5	22.88	13728		
Acid Yellow 23	12.8	512.8	25.13	15078		
Acid Yellow 73	13.5	169.0	22.63	13578		
1-Amino-anthraquinone	12.2	242.5	16.06	9636		
Z-Amino-anthraquinone	12.6	230.0	23.36	14016	2	14700
Basic Violet 10	15.0	146.0	19.88	11928	570	12000
Basic Yellow 2	170	1334.1	23.13	13878	60	1300
Disperse Blue 14	16.7	390.9	18.69	11214		
Disperse Red 9	14.8	176.7	16.51	9906		
Disperse Violet 1	11.1	950.1	16.26	9756		
Solvent Green 3	18.5	697.9	18.38	11028		
Solvent Orange 3	11.7	56.7	15.99	9594	600	129000
Solvent Orange 7	6.1	176.2	18.00	10800	730	52000
Solvent Red 1	7.9	2295.6	28.99	17394		
Solvent Red 24	2.9		7.1	4260		
Solvent Yellow 2	11.8	221.0	19.95	11970	0.01	110
Solvent Yellow 3	16.2	194.6	18.88	11328	0.02	130
Solvent Yellow 14	13.5	94.7	17.70	10620	460	3230
Solvent Yellow 33	13.7	137.4	17.48	10488		
Anthracene	18.4	236.6	25.96	15576		
Benzanthrone	13.1	83.1	15.2	9120		

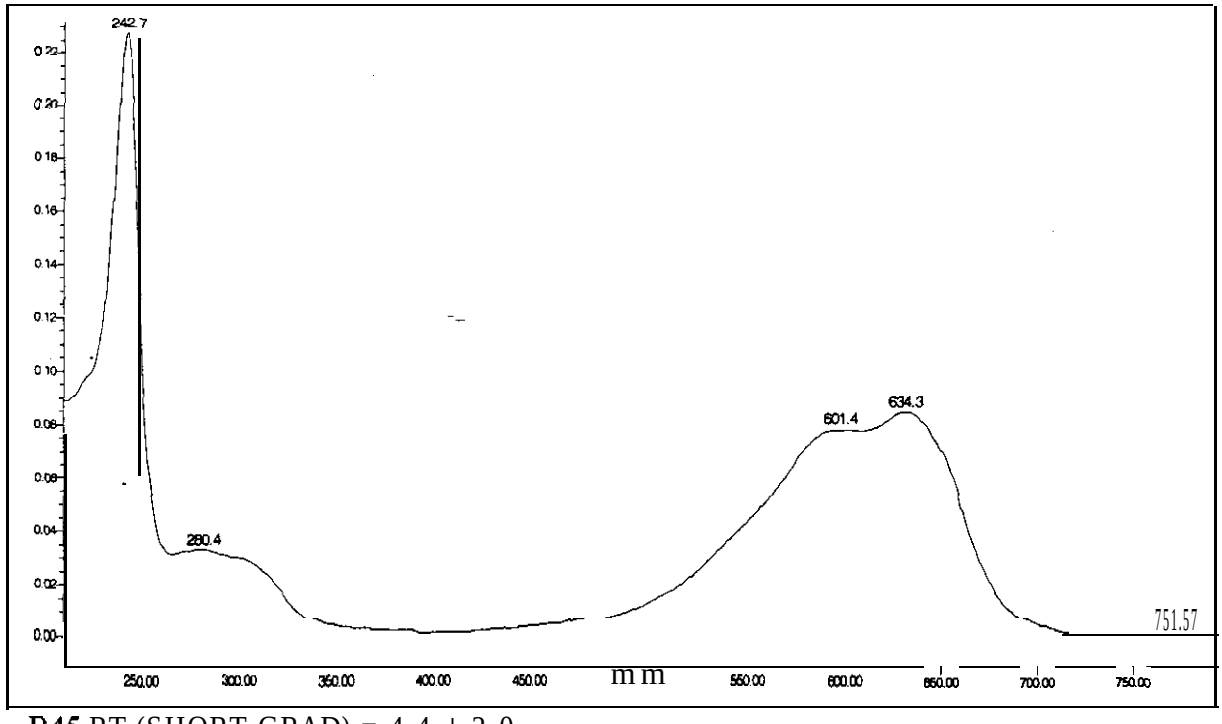
The risk-based screening levels were developed by USEPA Region 5 specifically for this project.

1. Values determined during Round 1. All other values determined during Rounds 2 and 3 of the Method Validation **Study.**

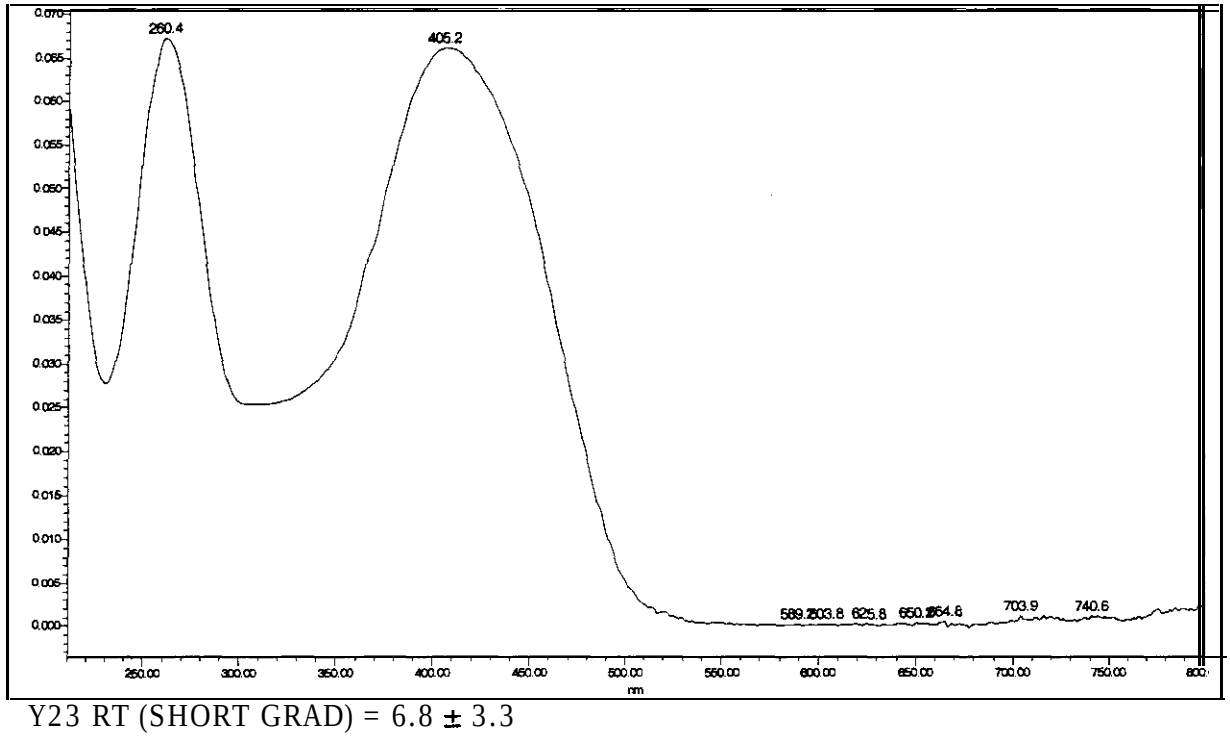
Table 5
Example Standard Concentrations for the Initial Calibration

Dye	Level 5 (ug/mL)	Level 4 (ug/mL)	Level 3 (ug/mL)	Level 2 (ug/mL)	Level 1 (ug/mL)
Acid Blue 45	37.200	18.600	9.300	4.650	2.325
Acid Yellow 23	40.200	20.100	10.050	5.025	2.513
Acid Yellow 73	36.200	18.100	9.050	4.525	2.263
Acid Orange 10	40.200	20.100	10.050	5.025	2.513
Acid Blue 1	42.600	21.300	10.650	5.325	2.663
Basic Yellow 2	37.000	18.500	9.250	4.625	2.313
Disperse Violet 1	26.020	13.010	6.505	3.253	1.626
Solvent Yellow 33	27.960	13.980	6.990	3.495	1.748
Disperse Blue 14	29.900	14.950	7.475	3.738	1.869
Acid Red 64	50.800	40.640	25.400	12.700	6.250
Acid Blue 9	36.200	18.100	9.050	4.525	2.263
Basic Violet 10	31.800	15.900	7.950	3.975	1.988
2-Aminoanthraquinone	37.380	18.690	9.345	4.673	2.336
Solvent Orange 3	25.580	12.790	6.395	3.198	1.599
1-Aminoanthraquinone	25.700	12.850	6.425	3.213	1.606
Solvent Yellow 14	28.320	14.160	7.080	1.540	1.770
Solvent Orange 7	28.800	14.400	7.200	3.600	1.800
Acid Yellow 3	36.600	18.300	9.150	4.575	2.288
Solvent Yellow 3	30.200	15.100	7.550	3.775	1.888
Benzanthrone	24.320	12.160	6.080	3.040	1.520
Anthracene	41.540	20.770	10.385	5.193	2.596
Solvent Red 1	46.380	23.190	11.595	5.798	2.899
Disperse Red 9	26.420	13.211	6.605	3.303	1.651
Solvent Yellow 2	31.920	15.960	7.980	3.990	1.995
Solvent Green 3	29.400	14.700	7.350	3.675	1.838
Solvent Red 24	5.680	2.840	1.420	1.136	0.710

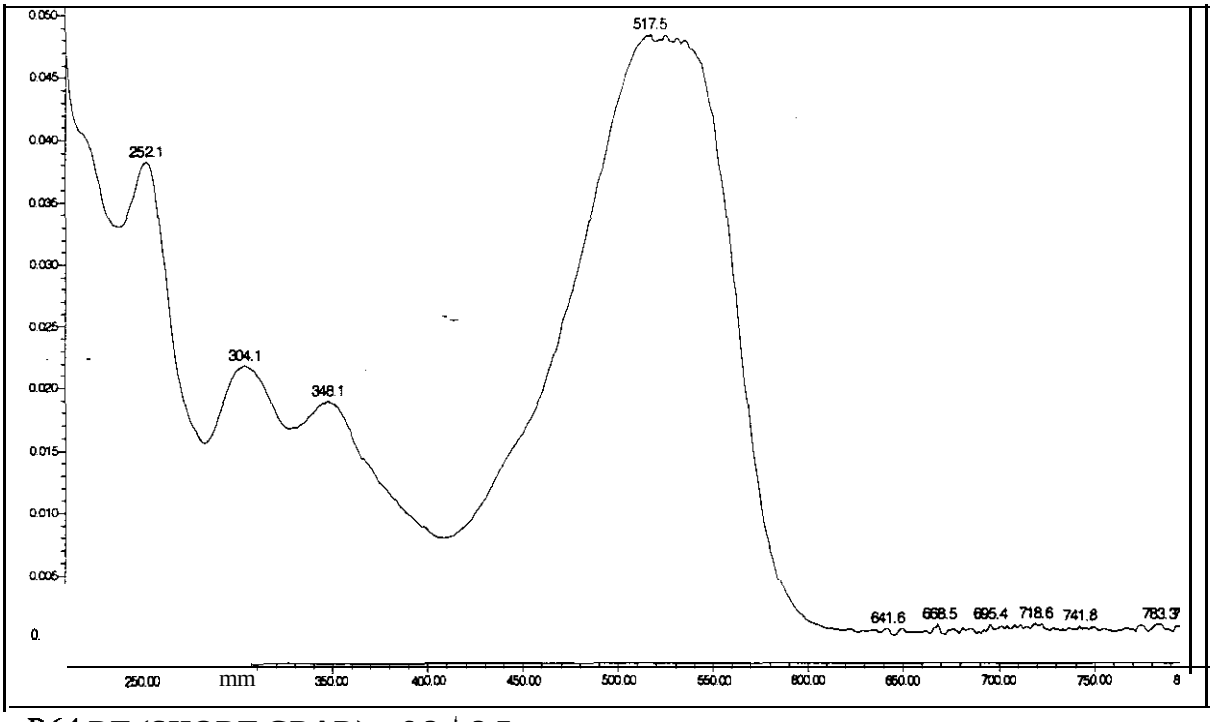
ATTACHMENT 1: SPECTRA AND APPROXIMATE RETENTION TIMES



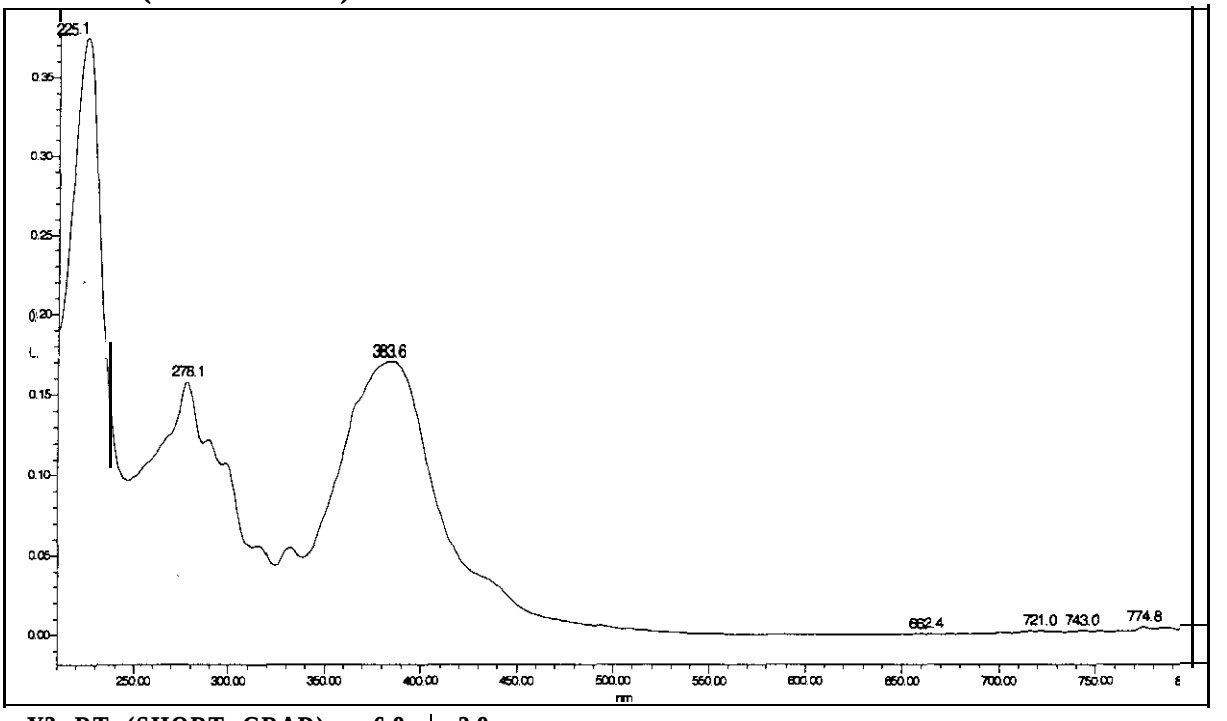
.B45 RT (SHORT GRAD) = 4.4 ± 2.0
AB45 RT (LONG GRAD) = 6.8 ± 10.6



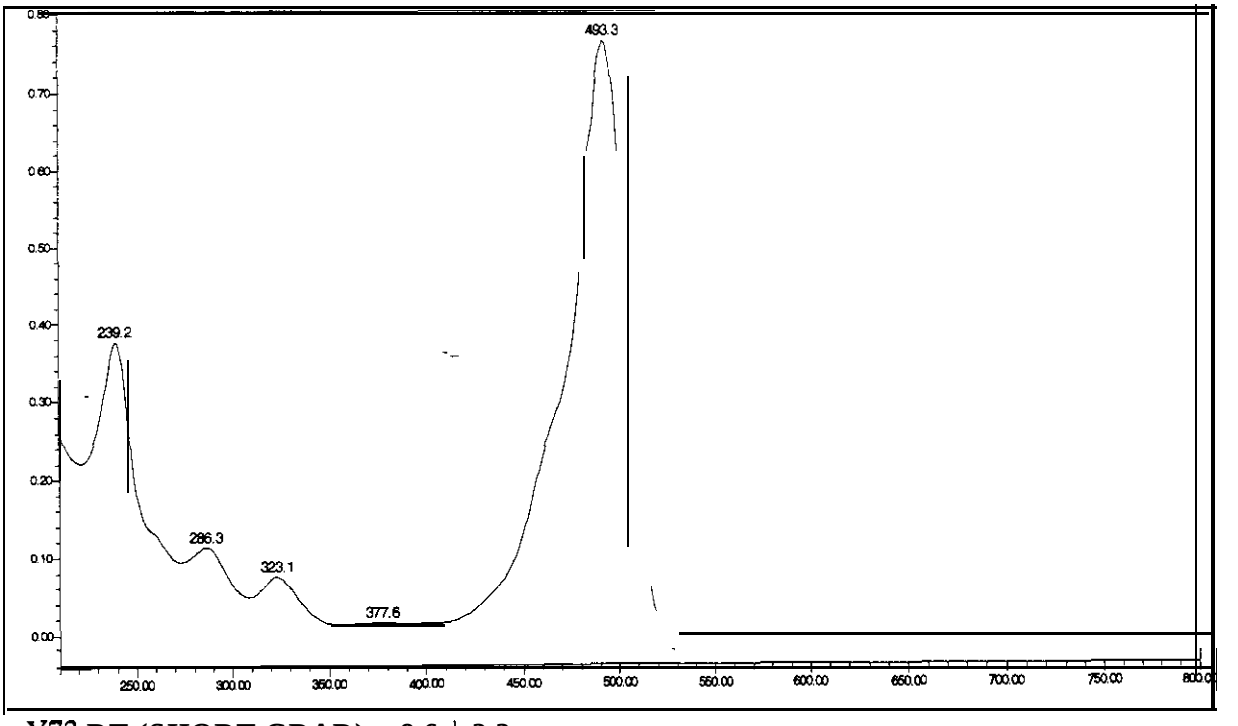
Y23 RT (SHORT GRAD) = 6.8 ± 3.3
AY23 RT (LONG GRAD) = 14.1 ± 5.2



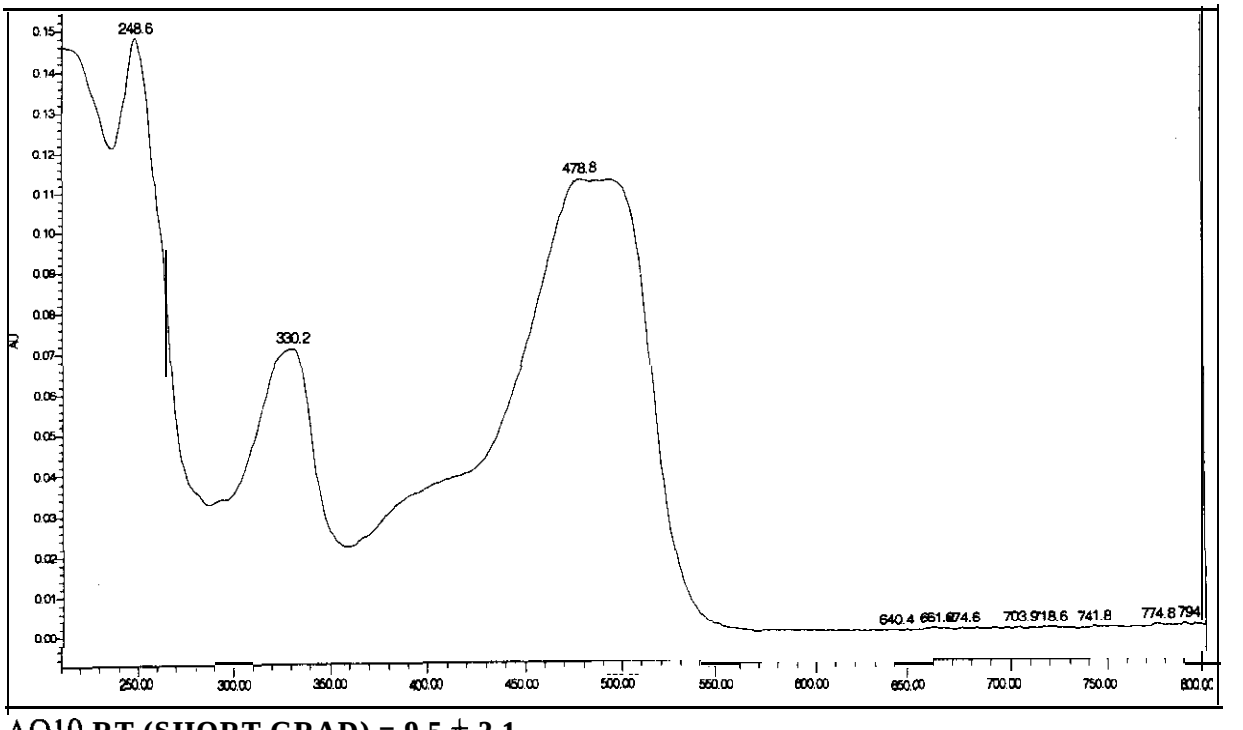
R64 RT (SHORT GRAD) = 6.2 ± 2.7
R64 RT (LONG GRAD) = 13.1 ± 3.0



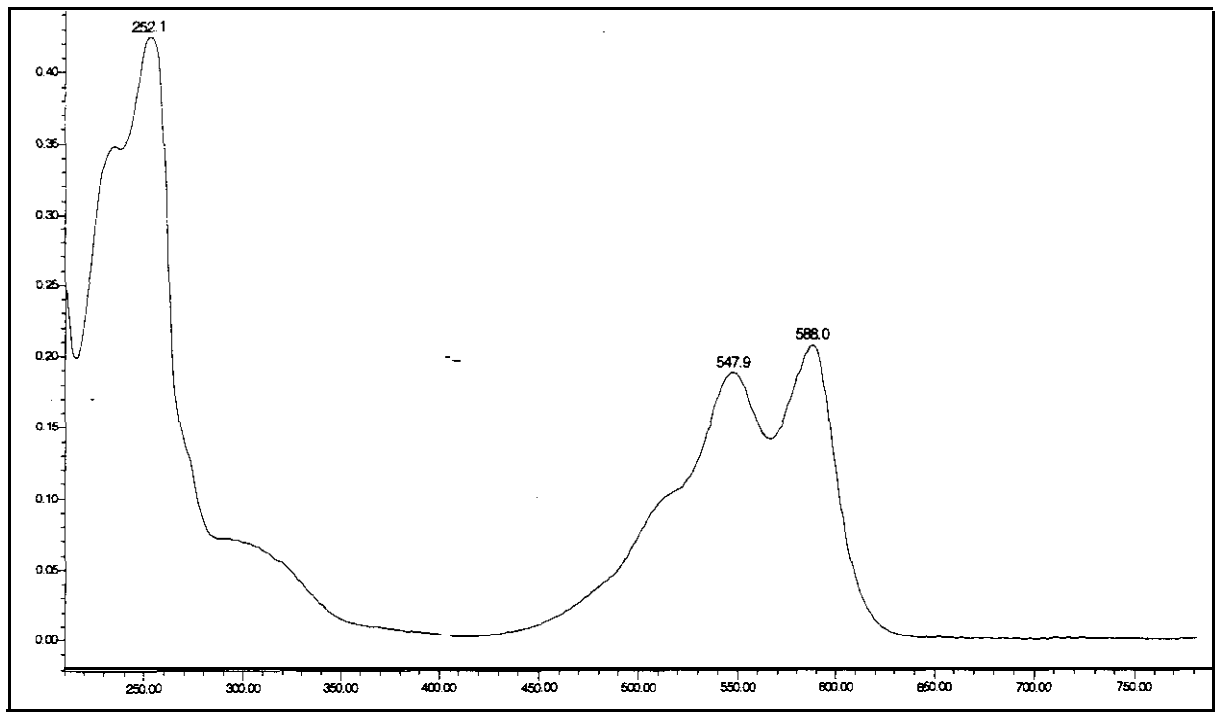
Y3 RT (SHORT GRAD) = 6.8 ± 2.9
AY3 RT (LONG GRAD) = 13.7 ± 3.4



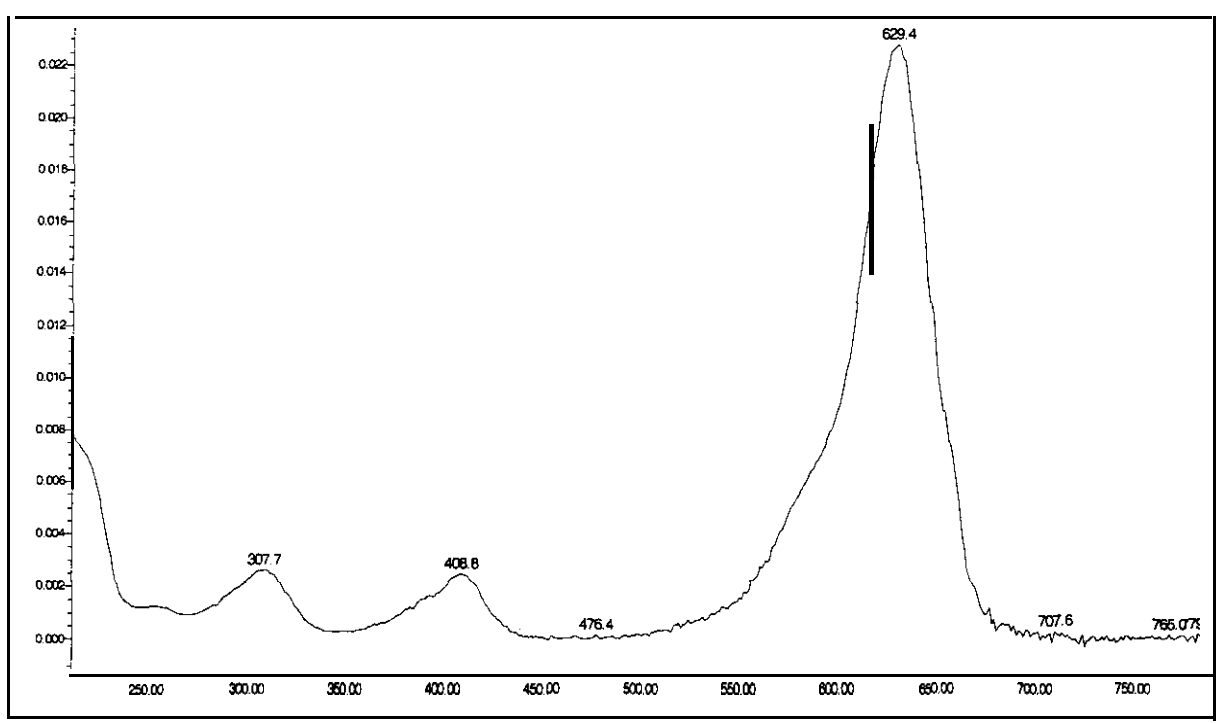
AY73 RT (SHORT GRAD) = 8.6 ± 2.3
AY73 RT (LONG GRAD) = 17.0 ± 4.0



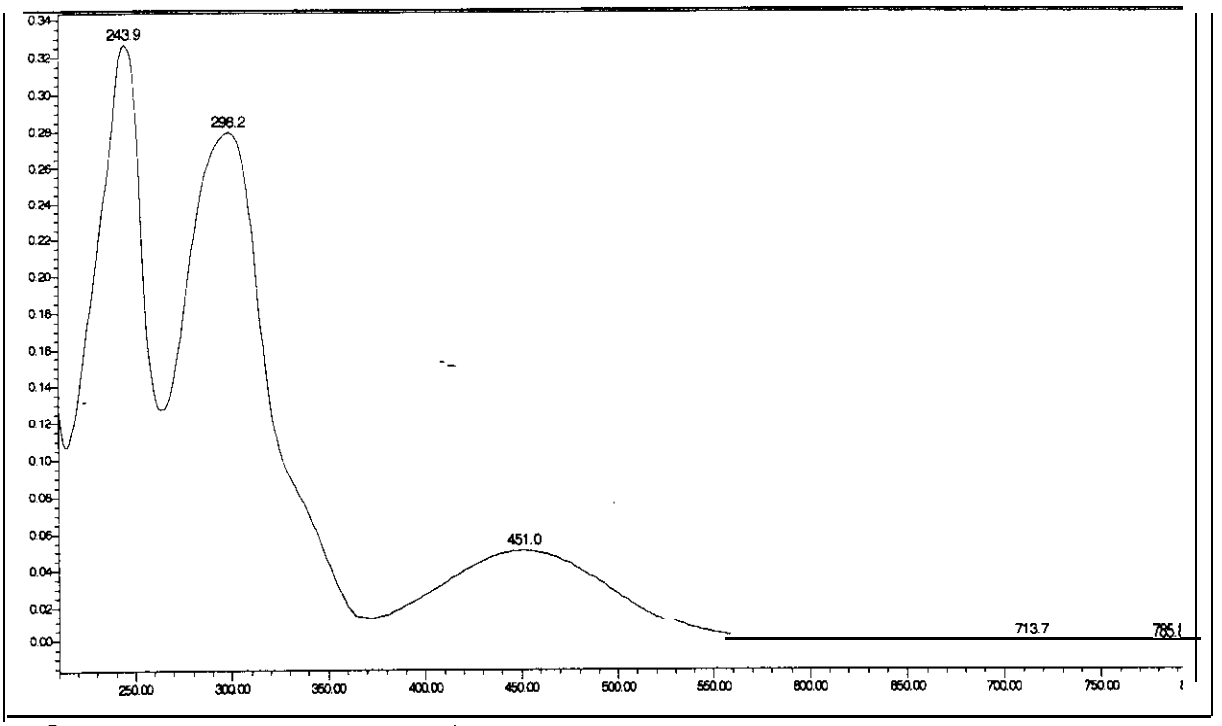
A010 RT (SHORT GRAD) = 9.5 ± 2.1
A010 RT (LONG GRAD) = 18.4 ± 3.6



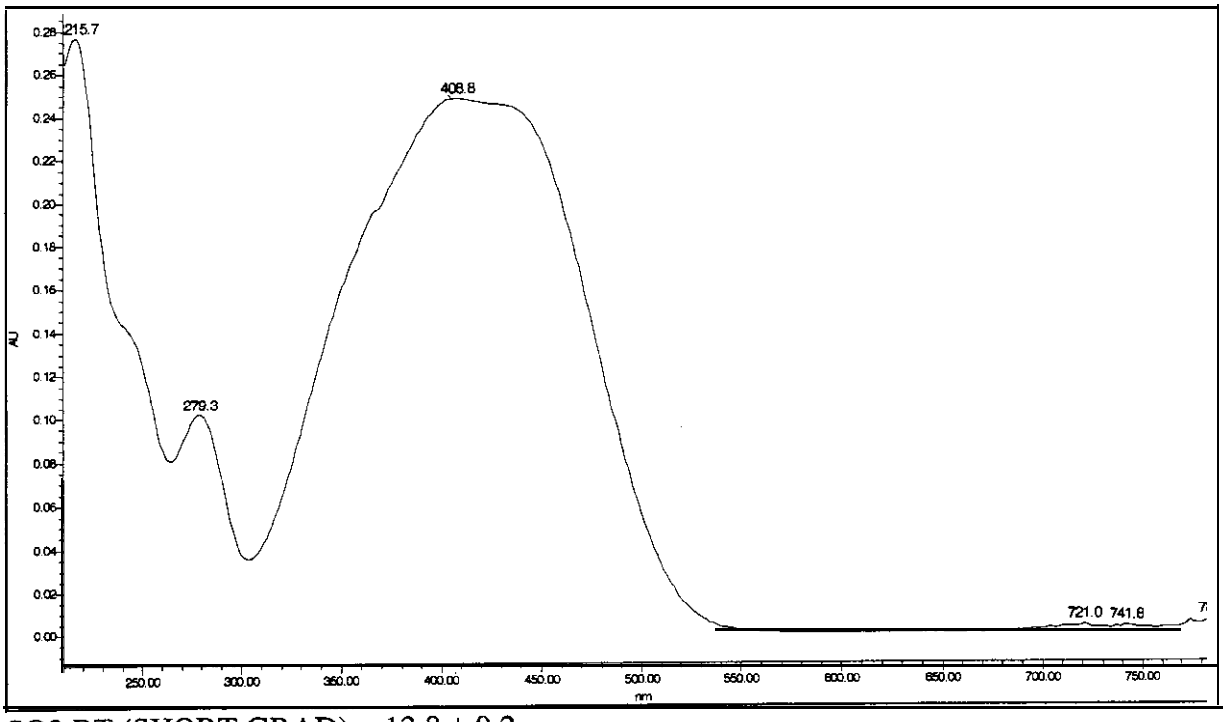
DVI RT (SHORT GRAD) = 12.7 ± 0.2
DV1 RT (LONG GRAD) = 22.8 ± 0.2



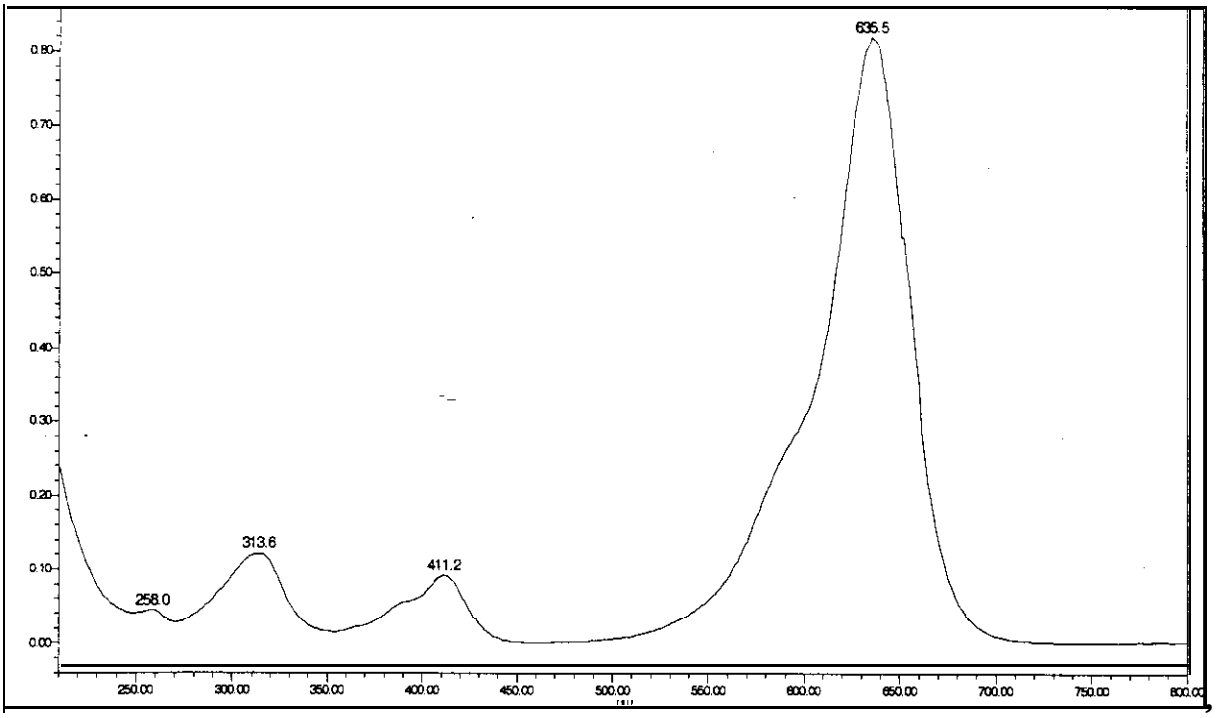
B9 RT (SHORT GRAD) = 11.3 ± 1.4
AB9 RT (LONG GRAD) = 21.8 ± 2.6



2AQ RT (SHORT GRAD) = 13.2 ± 0.5
2AQ RT (LONG GRAD) = 23.8 ± 0.5

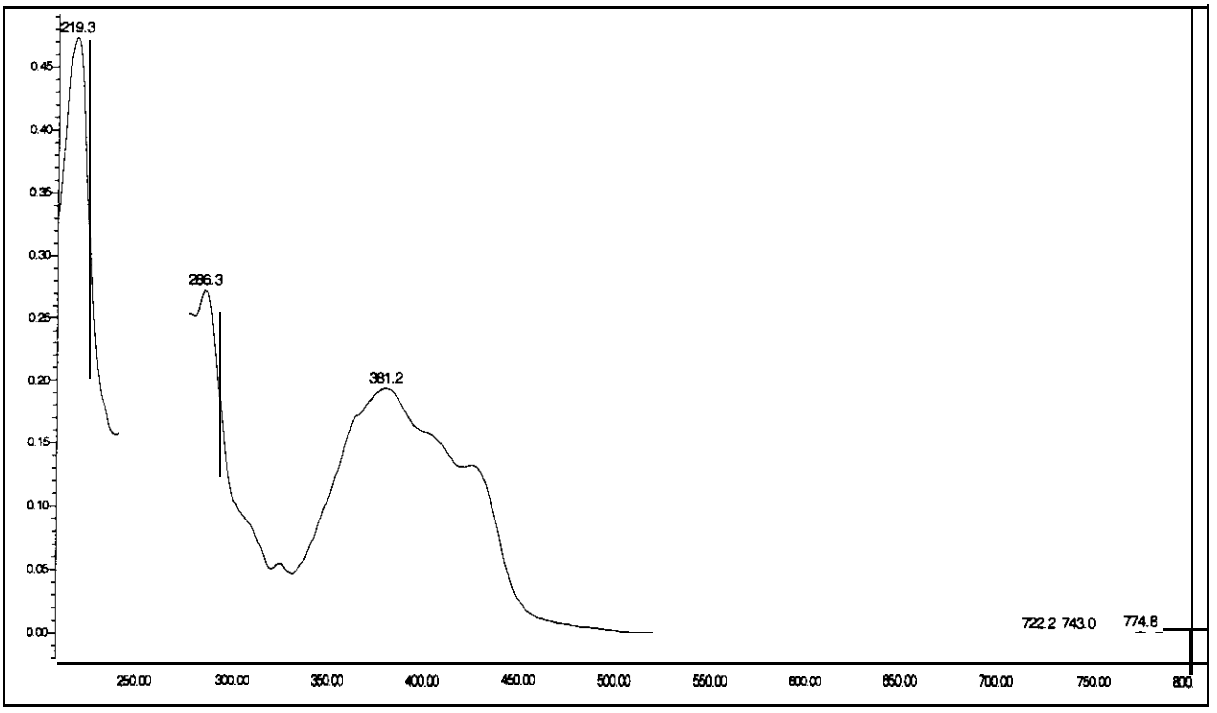


SO3 RT (SHORT GRAD) = 13.8 ± 0.2
SO3 RT (LONG GRAD) = 24.8 ± 0.2



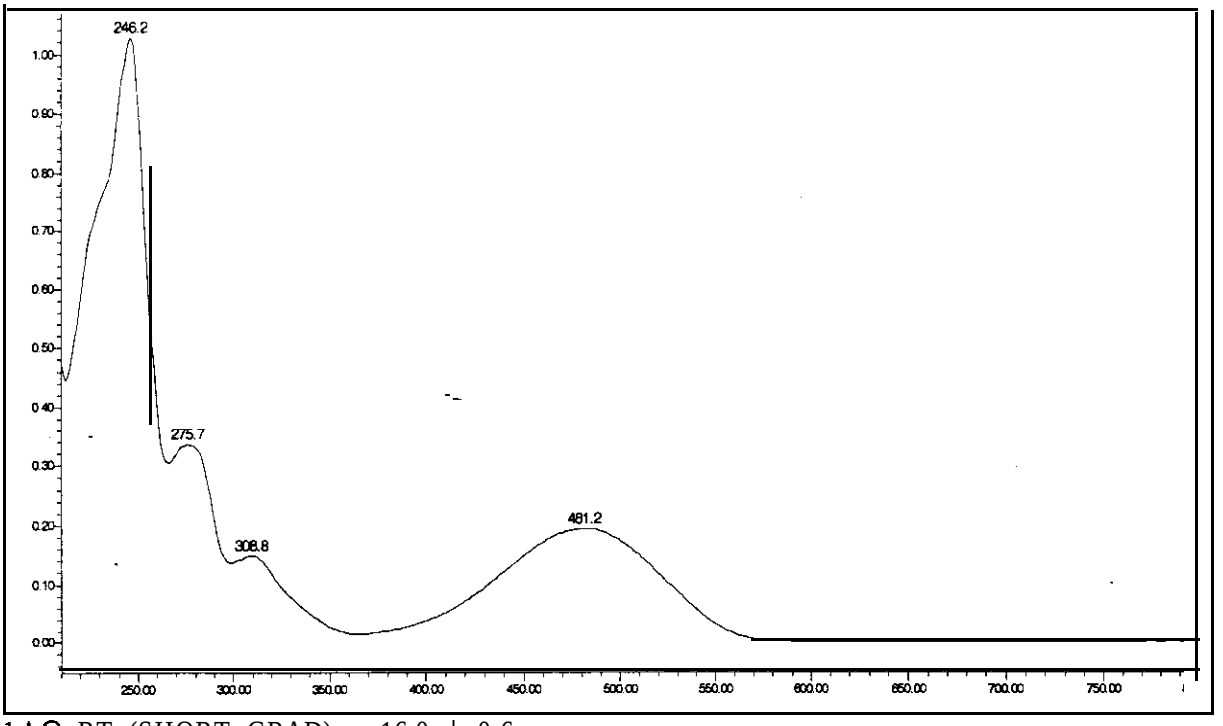
B 1 RT (SHORT GRAD) = 12.4 ± 1.5

AB1 RT (LONG GRAD) = 23.5 ± 2.3

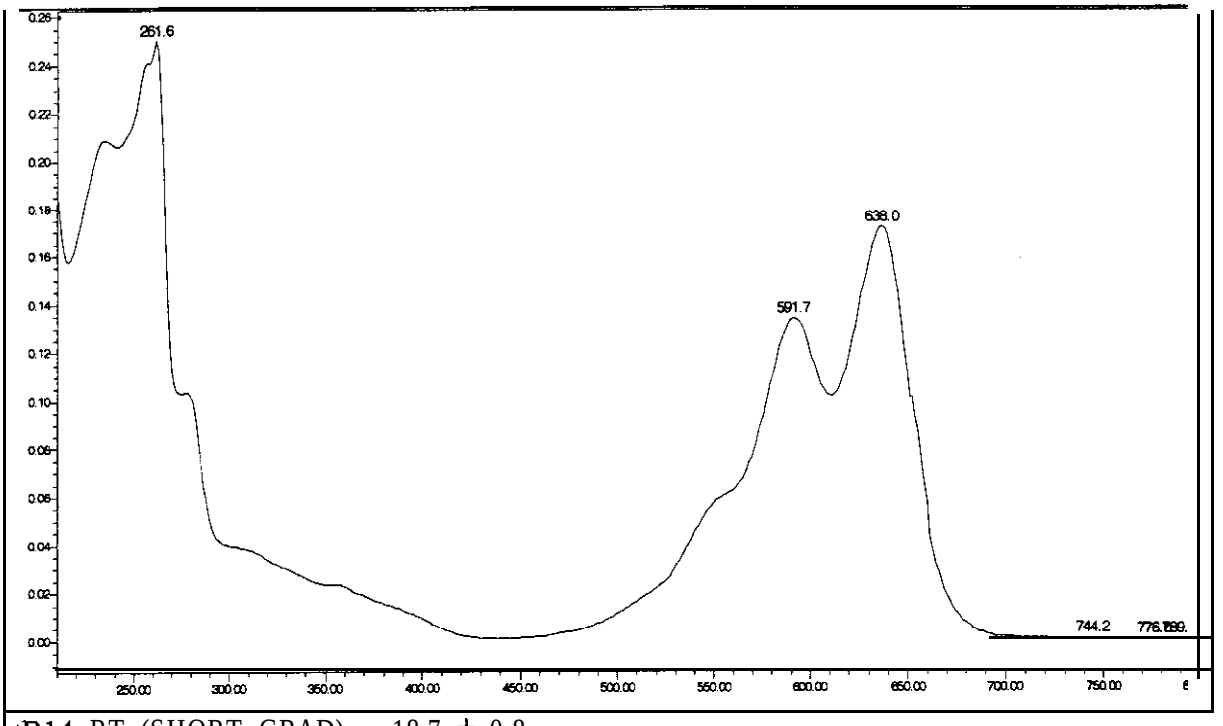


Y33 RT (SHORT GRAD) = 15.3 ± 0.8

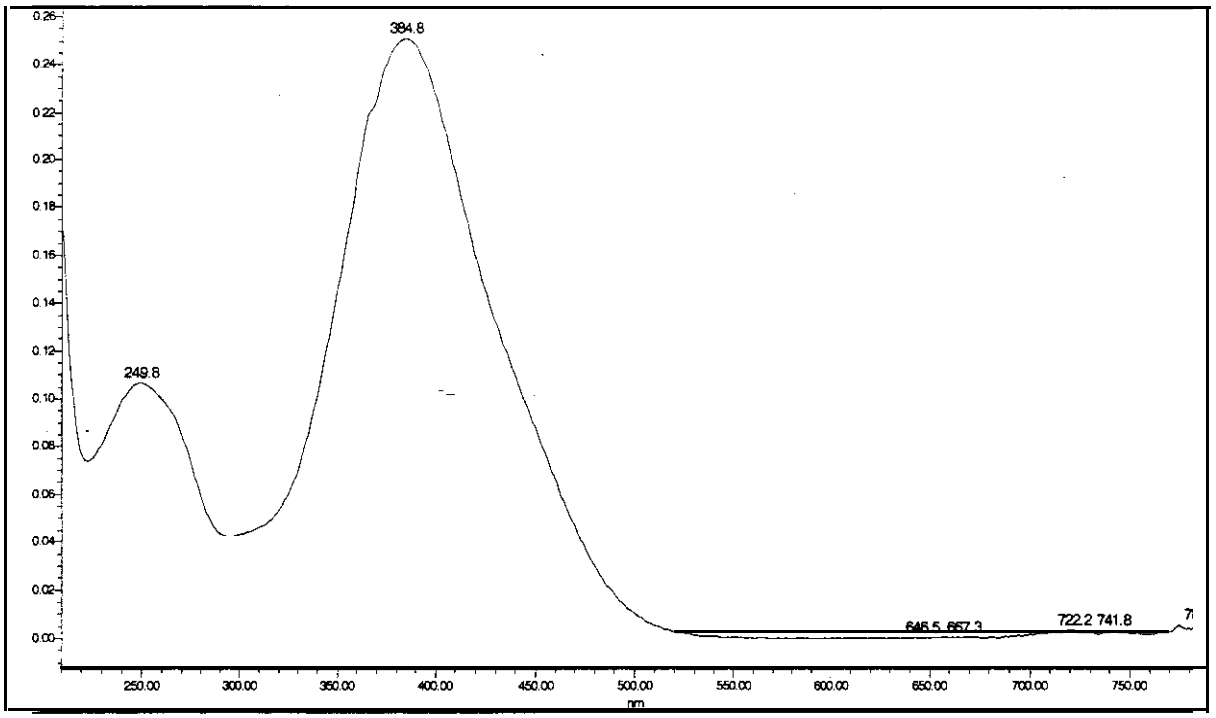
SY33 RT (LONG GRAD) = 28.1 ± 1.2



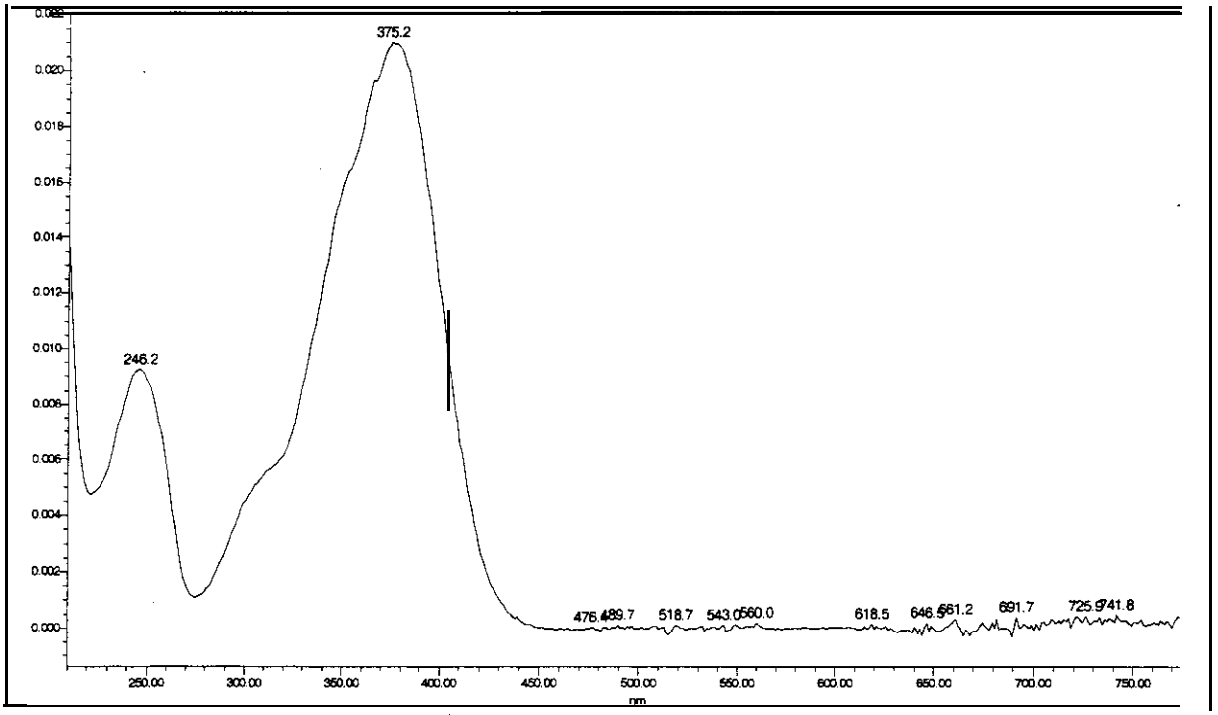
1AQ RT (SHORT GRAD) = 16.0 ± 0.6
1AQ RT (LONG GRAD) = 28.9 ± 0.9



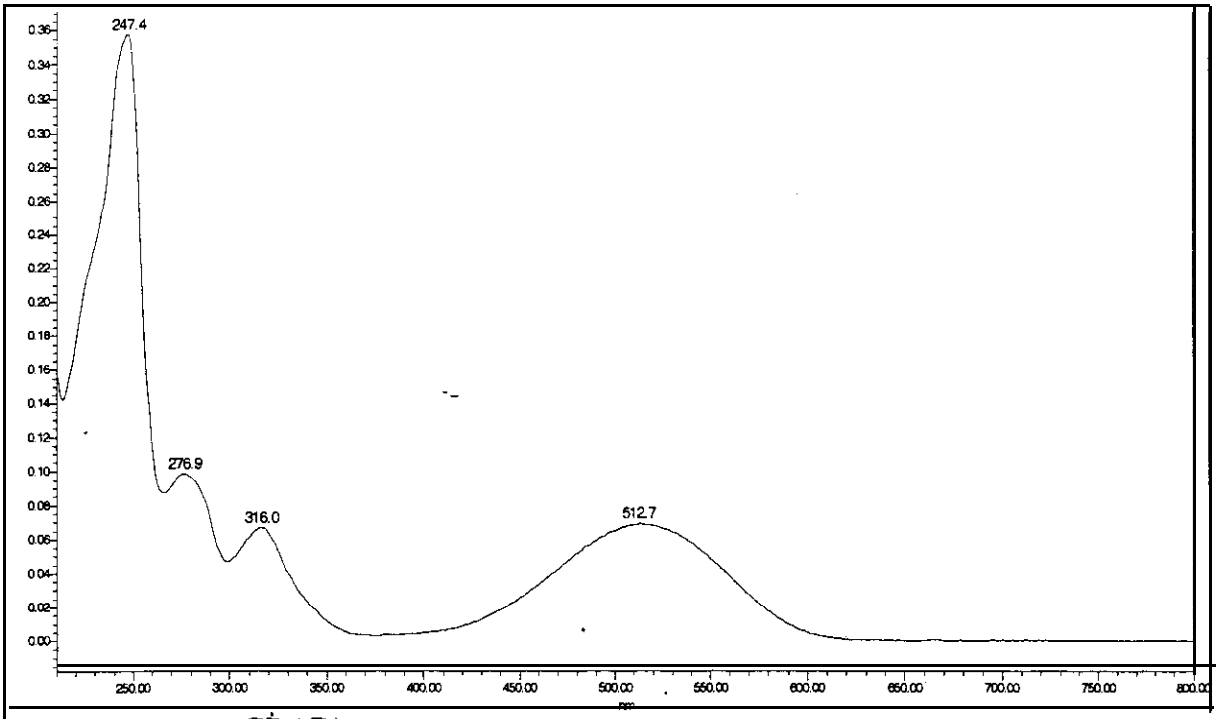
DB14 RT (SHORT GRAD) = 18.7 ± 0.8
DB14 RT (LONG GRAD) = 33.9 ± 1.0



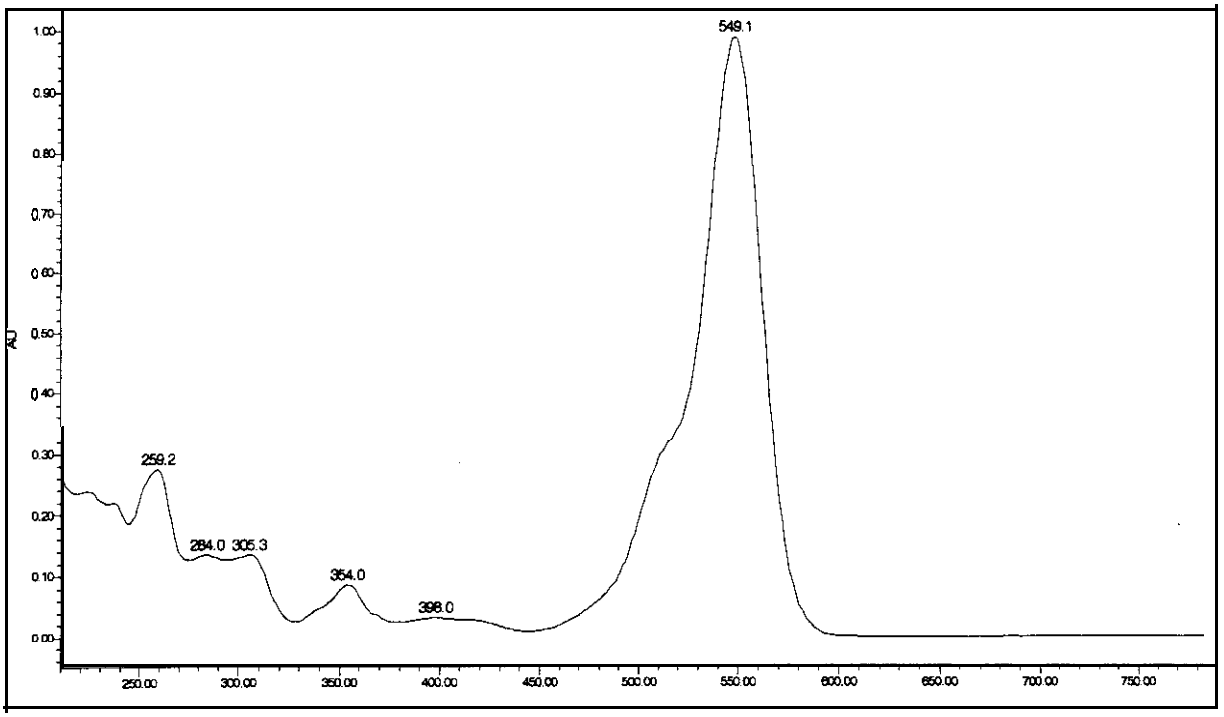
Y3 RT (SHORT GRAD) = 19.8 ± 0.7
SY3 RT (LONG GRAD) = 36.1 ± 1.0



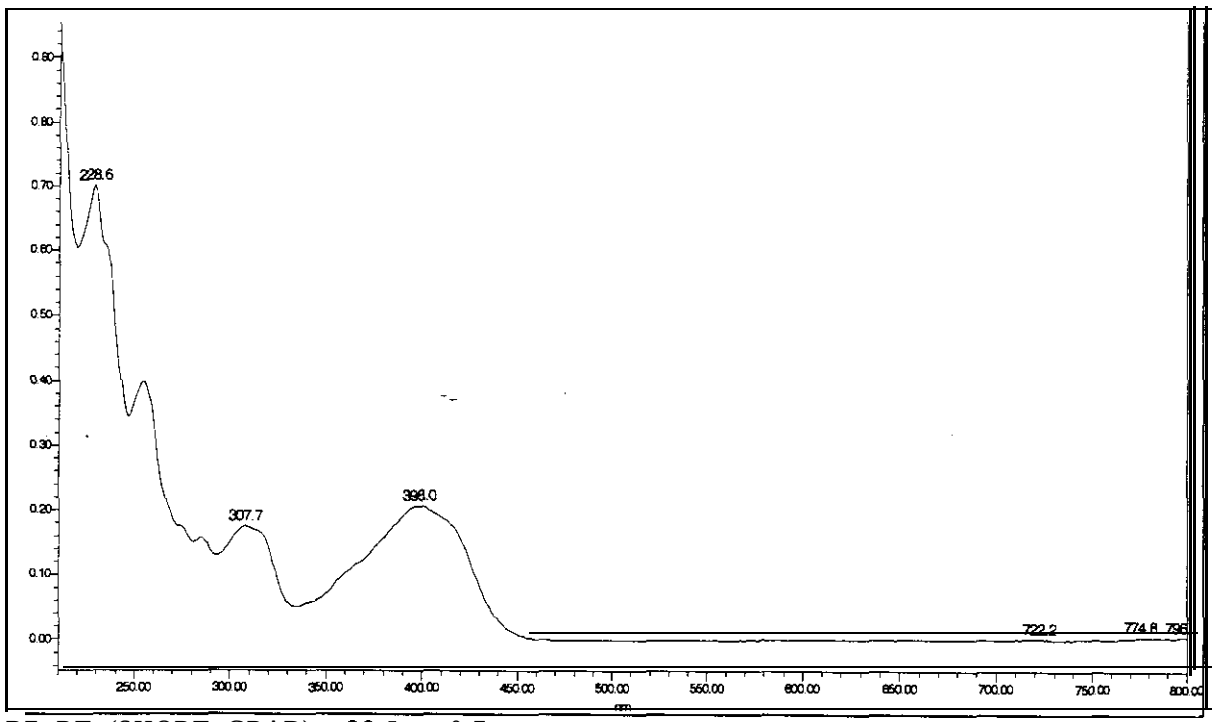
Y2 RT (SHORT GRAD) = 19.3 ± 0.9
BY2 RT (LONG GRAD) = 35.2 ± 1.0



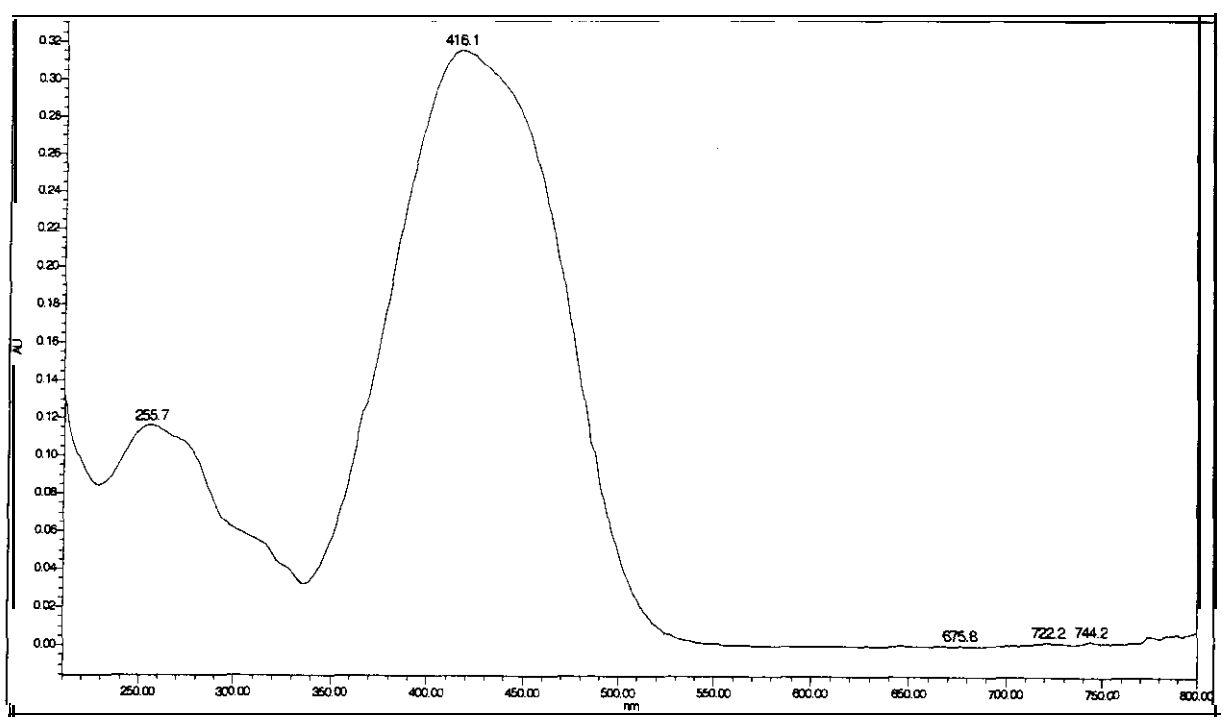
R9 RT (SHORT GRAD) = 20.2 ± 1.0
DR9 RT (LONG GRAD) = 36.5 ± 1.5



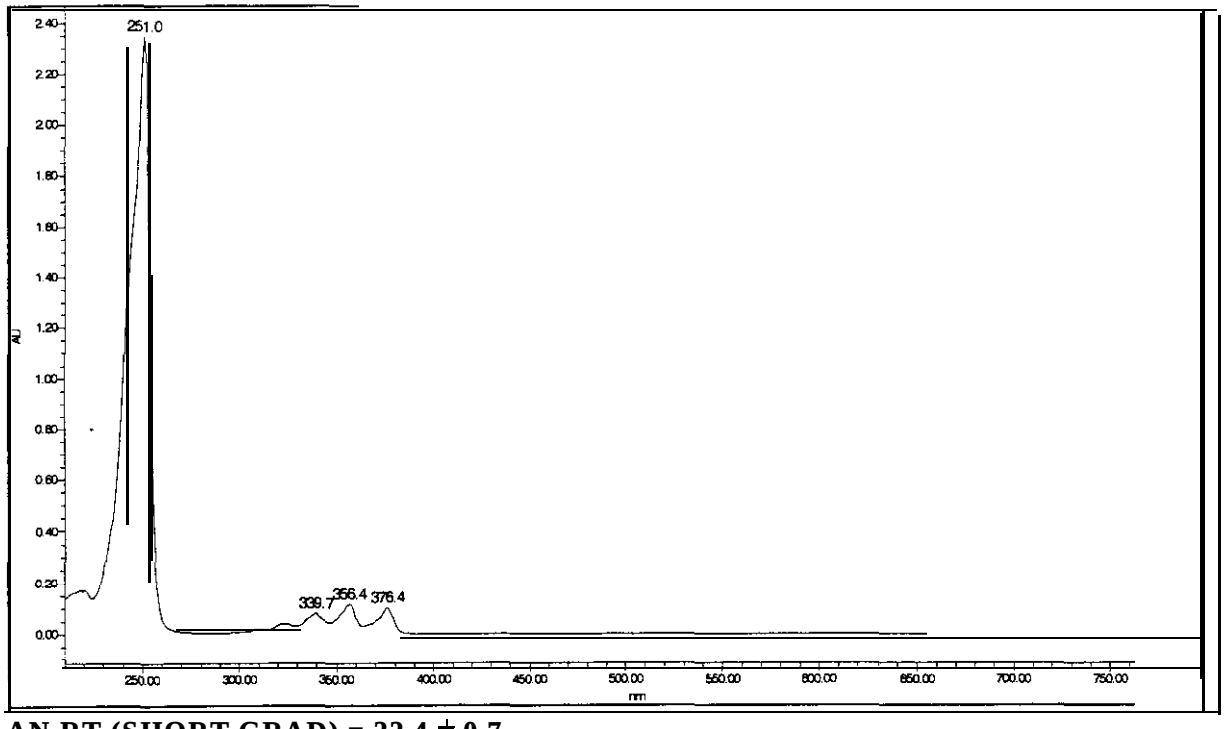
BV10 RT (SHORT GRAD) = 19.6 ± 0.7
BV10 RT (LONG GRAD) = 36.0 ± 0.8



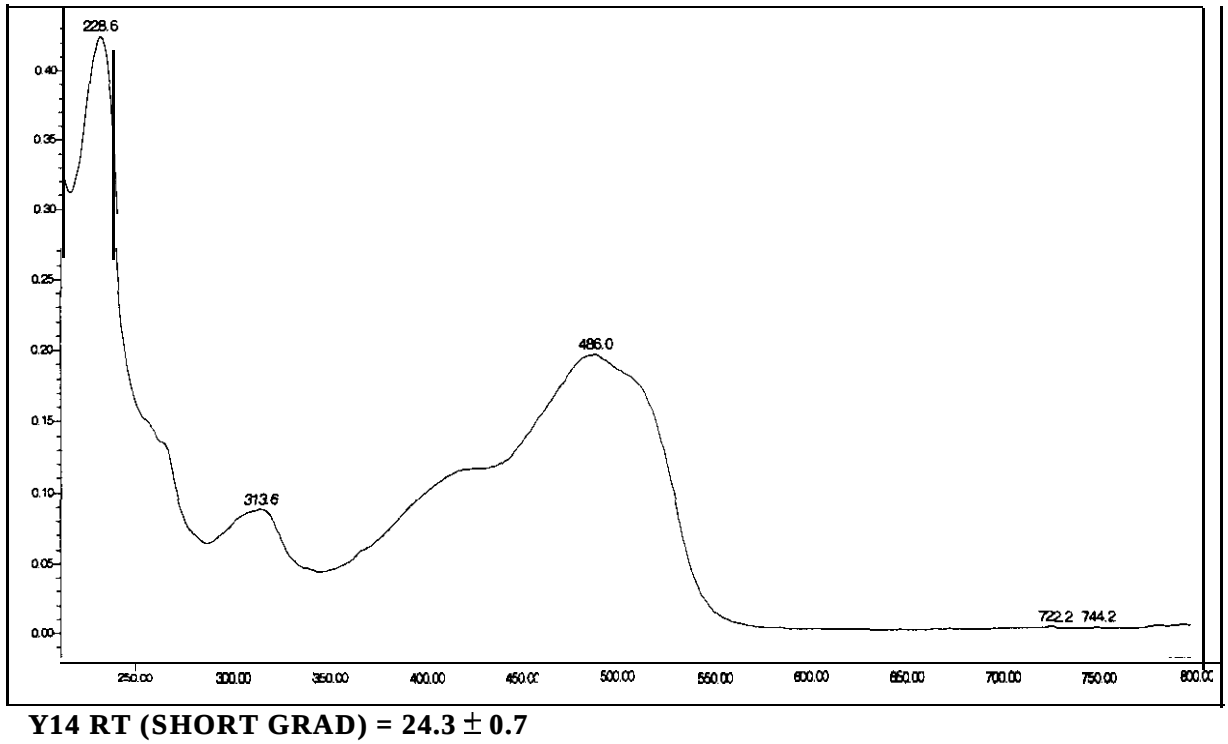
BZ RT (SHORT GRAD) =20.3 ± 0.7
BZ RT (LONG GRAD) = 36.8 ± 0.9



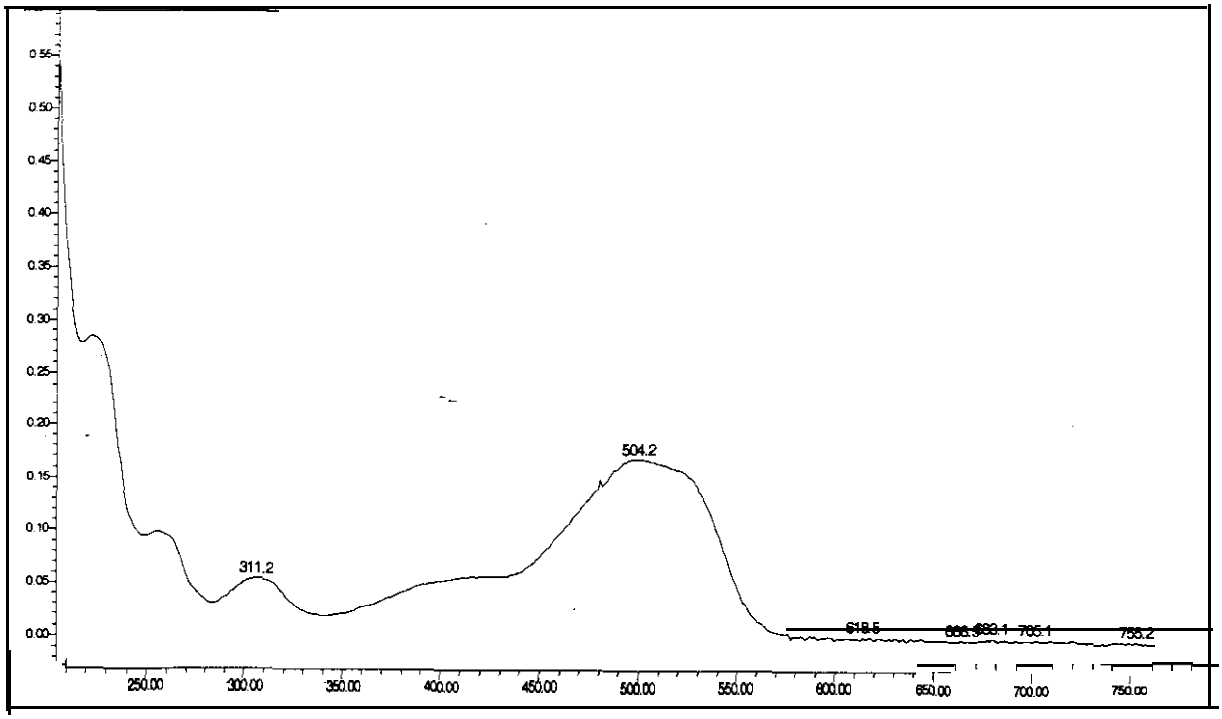
SY2 RT (SHORT GRAD) = 22.1 ± 0.9
SY2 RT (LONG GRAD) = 40.2 ± 1.4



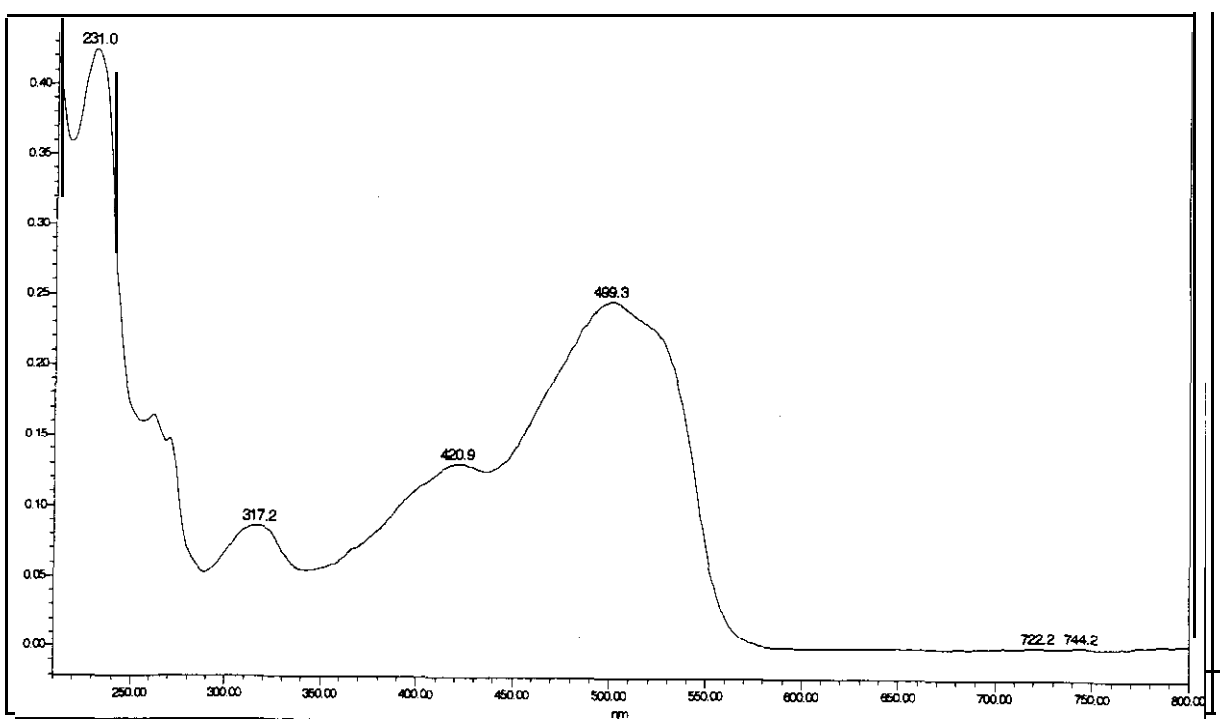
AN RT (SHORT GRAD) = 22.4 ± 0.7
AN RT (LONG GRAD) = 40.7 ± 1.1



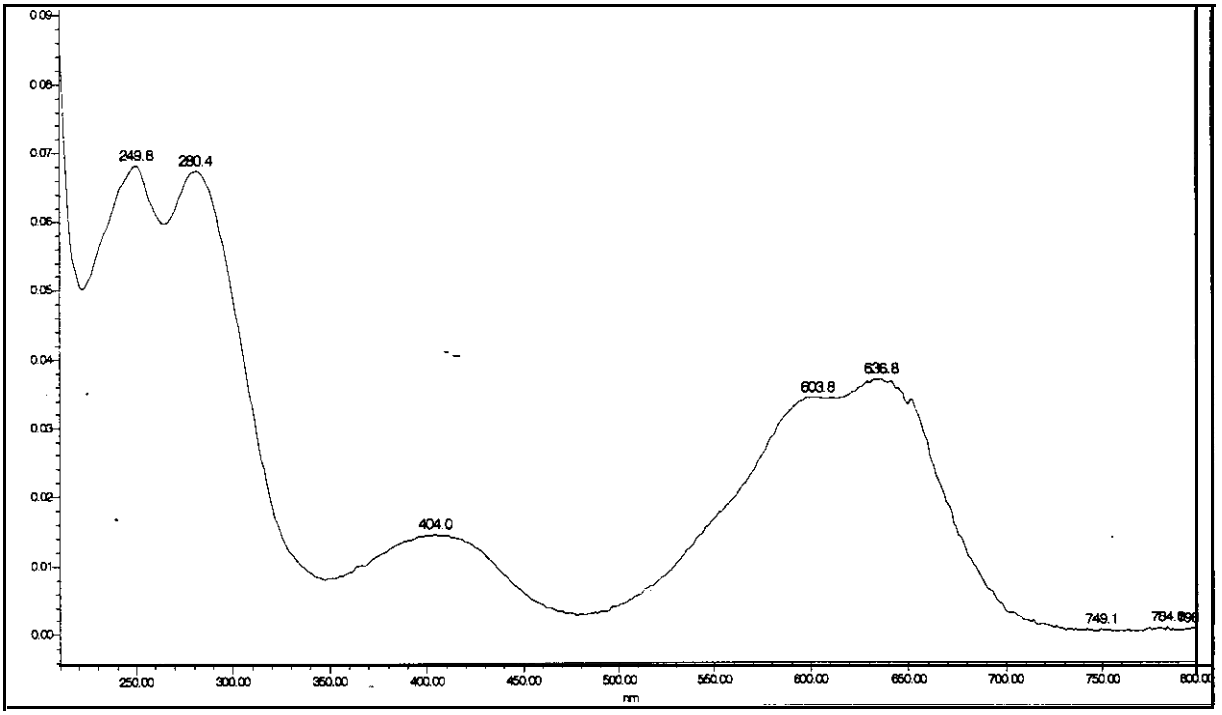
Y14 RT (SHORT GRAD) = 24.3 ± 0.7
SY14 RT (LONG GRAD) = 44.4 ± 1.3



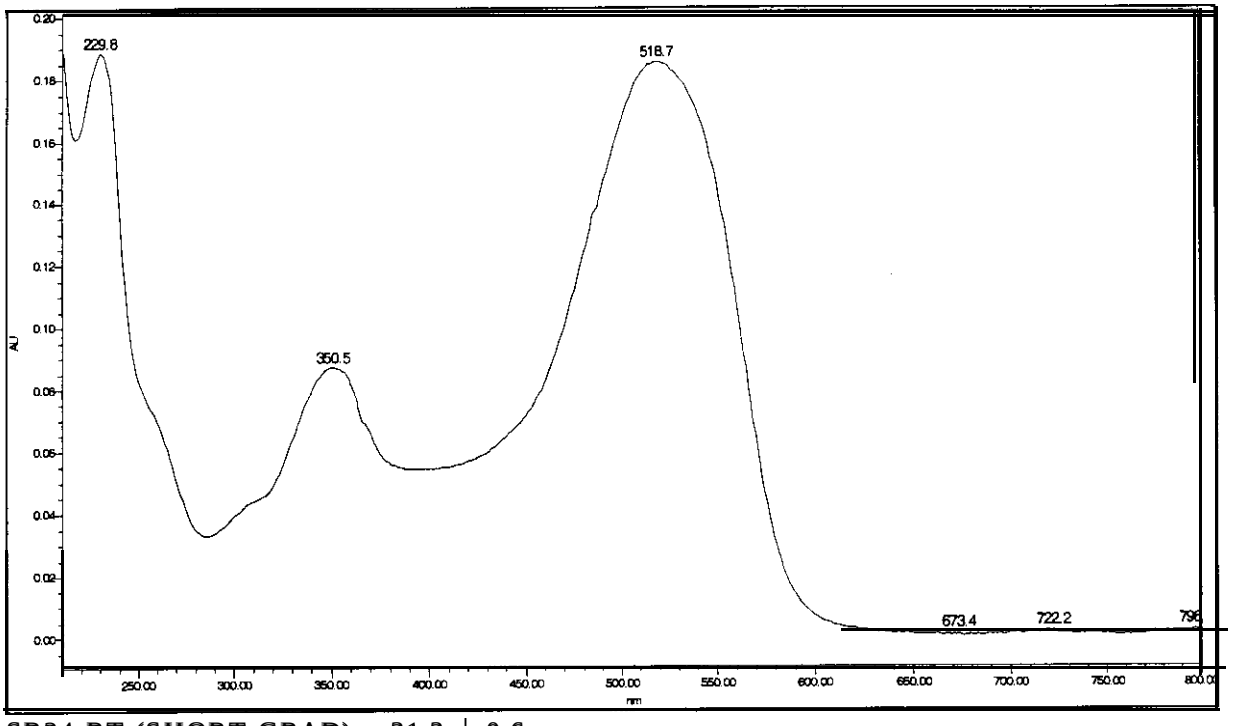
SR1 RT (SHORT GRAD) = 24.5 ± 0.8
SR1 RT (LONG GRAD) = 44.9 ± 1.1



07 RT (SHORT GRAD) = 27.7 ± 0.6
SO7 RT (LONG GRAD) = 50.9 ± 1.2



SG3 RT (SHORT GRAD) = 31.0 ± 0.7
SG3 RT (LONG GRAD) = 57.8 ± 1.3



SR24 RT (SHORT GRAD) = 31.3 ± 0.6
SR24 RT (LONG GRAD) = 58.2 ± 1.0

ATTACHMENT XIII

SAMPLE CALCULATIONS

Page No.

This is an example calculation to show how we calculated "Corrected" HPLC results, % Recovery, and MDL. We'll use the results for Acid Yellow 23, Sample: W1H00LHS; Long Gradient

$$\underbrace{281.2\,\frac{\mu g}{mL}}_{\text{AY23 Std used to spike water}} \times \frac{1\,mL}{1000\,\mu L} \times \underbrace{500.0\,\mu L}_{\text{Spike amt}} = 140.6\,\mu g\ AY23$$

$$\frac{140.6\,\mu g\ AY23}{\underset{\text{total volume water}}{1\,L}} = 140.6\,\frac{\mu g}{L} = \text{Total Spike Concentration}$$

HPLC Results

$$\underbrace{27.837\,\frac{\mu g}{mL}}_{\text{HPLC Result}} \Big/ \underset{\text{Correction factor for injection volume difference between std and sample}}{2} = 13.9185\,\frac{\mu g}{mL} \times \frac{\overset{\text{total volume after rotary evaporation}}{10.00\,mL}}{\underset{\text{total amt sample}}{1\,L}} = 139.19\,\frac{\mu g}{L} = \text{Corrected HPLC Result}$$

std = 10 µL
sample = 20 µL

$$\%\ \text{Recovery} = \frac{\text{Corrected HPLC Result}}{\text{Total Spike Conc.}} \times 100 \Rightarrow \frac{139.19}{140.6} \times 100 = 99.0\%$$

10-2-03

To Page No. 14

From Page No. 47

MDL Calculation : Reference 40CFR Chp 1 (7-1-92 Edition) pg 136

$$S^2 = \frac{1}{N-1}\left[\sum_{i=1}^{n} X_i^2 - \frac{\left(\sum_{i=1}^{n} X_i\right)^2}{N}\right]$$

$$S = (S^2)^{1/2}$$

$$MDL = t\,(S)$$

t = students t value

$X_i^2 = (139.19)^2 = 19372.5$ I got 19373.9 JEJ 10-2-00

$\sum X_i^2 = 19372.5 + 20890.4 + 27357.2 + 30171.7 + 29539.3 + 22675$
$= 150006.9$ ✓ JEJ

$(\sum X_i)^2 = (139.19 + 144.54 + 165.40 + 173.70 + 171.87 + 150.59)^2$
$= 893573.18$ ✓ JEJ

$$S^2 = \frac{1}{(6-1)} * \left[150006.9 - \left(\frac{893573.18}{6}\right)\right]$$

$S^2 = 216.5$ I got 215.6 JEJ 10-2-00

$S = \sqrt{216.5} = 14.7$ ✓ JEJ

$MDL = 3.365\,(14.7)$
$MDL = 49.5$ ✓ JEJ

To Page N

Witnessed & Understood by me, | Date | Invented by | Date

Page No.

This is an example calculation to show how we calculated "Corrected" HPLC Results, % Recovery, and MDL for soil results. Will use the results for Acid Yellow 23; Sample: S001LS; Long Hom

$281.2 \frac{\mu g}{mL} \times \frac{1 mL}{1000 \mu L} \times 100 \mu L = 28.1 \mu g$ ✓ JEJ

Acid Yellow 23 std. Conc. to spike 10 g soil

I get 2783.66 ✓ JEJ

$\frac{28.1 \mu g}{0.0100946 kg \text{ soil}} = 2783.66 \frac{\mu g}{kg}$ = Total spike concentration

total amt soil in kg

HPLC Result

$1.030 \frac{\mu g}{mL} / 2 = 1.515 \frac{\mu g}{mL} \times \frac{10.00 mL}{0.0100946 kg} = 1500.8 \frac{\mu g}{kg}$ = Corrected HPLC Result ✓ JEJ

HPLC Result

Correction factor for injection volume difference between std and sample
std = 10 µL
sample = 20 µL

% Recovery = $\frac{\text{Corrected HPLC Result}}{\text{Total Spike Conc}} \times 100 = \frac{1500.8}{2783.66} \times 100 = 53.9\%$ ✓ JEJ 10-2-00

MDL Calculation

$$S^2 = \frac{1}{N-1}\left[\sum_{i=1}^{N} X_i^2 - \frac{\left(\sum_{i=1}^{N} X_i\right)^2}{N}\right]$$

$$S = (S^2)^{1/2}$$

$$MDL = t(S)$$

t — Students t value

$X_i^2 = (1500.8)^2 = 2252407.9$ I got 2252400.6 TEJ 10-2-00

$\Sigma X_i^2 = 2252407.9 + 2742673.2 + 2273910.8 + 2446332.7 + 221948.4 + 2075926.8 + 3682032.8$

$\Sigma X_i^2 = 1.769 \times 10^7$ I got 1.3827×10^7 TEJ 10-2-00

After inserting the missing digits, the correct value of 1.769×10^7 is obtained

$(\Sigma X_i)^2 = (1500.80 + 1656.10 + 1507.95 + 1564.08 + 1490.62 + 1440.63 + 1918.86)^2$

$(\Sigma X_i)^2 = 1.227 \times 10^8$ ✓ TEJ 10-2-00

Note: Missing digits were obtained from the raw data supplied electronically from the Crane laboratory. TEJ 10-2-00

$$S^2 = \frac{1}{7-1}\left[1.769 \times 10^7 - \left(\frac{1.227 \times 10^8}{7}\right)\right]$$

$S^2 = 266616.0$ I got 269074.8 using Crane numbers

$S = \sqrt{266616.0} = 163.1$ I got 164.0 using Crane numbers

$MDL = 3.143(163.1)$

$MDL = 512.8 \frac{ug}{L}$ = 515.5 using Crane numbers rounded to nearest 10.

~~Using TEJ numbers: $S^2 = \frac{1}{7-1}\left[1.3827 \times 10^7 - \left(\frac{1.227 \times 10^8}{7}\right)\right]$~~

~~$= -616928.6$~~

~~$S = \sqrt{-616928.6}$ = imaginary no.~~ TEJ 10-2-0

ATTACHMENT XIV

MEETING NOTES

and

GUIDANCE DOCUMENTS

ATTACHMENT XIV – MEETING NOTES

Copies of the following documents are included. These documents formed the basis for development and *guidance* of the analytical method development effort at the Crane Laboratory.

Memorandum: NSWC Crane SWMU 2 – Dye Burial Ground Outstanding Issues Paper. May 3.1999

Minutes Pre-Pre-QAPP Meeting – May 13. 1999

Agenda NSWC Crane Laboratory Dye Method Development – September 10.1999

Conference Call Minutes: NSWC Crane Laboratory Method Development for Dyes at SWMU 2 – Dye Burial Ground-September 10, 1999

Proposed Agenda: Pre-QAPP Meeting SWMU 2 - Dye Burial Ground NSWC Crane, Indiana, 02/28/99

Minutes Pre-QAPP Meeting – March 9.2000

Guidance for Methods Development and Method Validation for the RCRA Program - http://www.epa.gov/epaoswer/hazwaste/test/methdev.htm

MEMORANDUM

To: Carol Witt-Smith and Allen Debus, U.S. EPA Region 5. Chicago. IL
From: Karen Smecker, Tetra Tech NUS, Inc. and Terrie Baranek, ECT.CON INC.
Date: May 3. 1999

Subject: NSWC Crane SWMU 2 – Dye Burial Ground Outstanding Issues Paper

BACKGROUND: RCRA Facility Investigations (RFIs), risk assessments, and a Corrective Measures Study (CMS) need to be completed for SWMU 2. the Dye Burial Grounds at Naval Surface Warfare Center (NSWC) Crane, Indiana. Aithough several phases of RFIs have been conducted at the site, the nature and extent of contamination has not been defined. One field sampling effort is planned to address the needs of the RFIs, risk assessments, and CMS. Planning documents (Work Plan, Field Sampling Plan, Quality Assurance Plan (QAPP), and Health and Safety Plan) are being developed for the investigations by Tetra Tech NUS. Inc. ECT.CON, Inc. is providing Tetra Tech NUS with QAPP support for the project. A pre-pre-QAPP meeting will be held on May 13, 1999 to discuss general issues for the project.

A brief summary of activities conducted at the site is provided in this paragraph. From the 1940s until 1960. approximately 50 tons of dyes were placed into three open trenches at SWMU 2. The trenches were approximately 10 feet wide. 6 feet deep and 50 feet long. The trenches were filled with soil in 1972. A RCRA cap was constructed at the site as an interim measure. During clearing activities for the RCRA cap, soil mounds were observed around the dye burial site. These soil mounds were excavated and placed within the limits of the RCRA cap.

ISSUES **FOR DISCUSSION:** The following issues are to be discussed during the pre-pre-QAPP meeting. Resolution and consensus on the issues are required prior to further development of the planning documents.

1. Overall Project Objectivea

The overall objective for this project is the completion of RFI, risk assessment, and CMS activities. The following issues (a through e) pertain to the project objectives.

a) **Collect the necessary samples needed to complete human health and ecological risk assessments and CMS.**

Soil, ground water, surface water, and sediment samples will be collected from SWMU 2 to fulfill the project objectives. In general, all samples collected from the site will be analyzed for dyes, inorganics, nitrate/nitrite, and sulfate. Some miscellaneous parameters (such as TOC. pH, etc.) will be obtained to provide infonation on migration for the risk assessments and CMS. A brief summary of samples to be collected per media is provided below:

Ground Water: Samples will be collected from upgradient locations and locations impacted by site activities. In the Phase III RFI for the site (U.S. ACE WES, July 1996). well cluster 02C10 was identified as an upgradient "background" location.

Surface Water and Sediment: Collocated samples will be collected from locations in the various drainageways. springs, and tributaries/creeks present at or near the site (to evaluate ground water discharge and migration away from the site).

Soil: Limited soil sampling will be conducted at the site since it is believed that all the contaminated soil present at the site has been removed and placed under the RCRA cap.

Soil samples will also be collected from locations where disturbed soil was previously excavated and placed under the RCRA cap (discussed in as Issue Number 2).

b) Collect coil samples to confirm that all dye-contaminated soil from disturbed area at the site was excavated and placed under the RCRA cap.

Under the assumption that all contaminated soil present at the site has been excavated and placed under the RCRA cap, it is anticipated that there will be no complete exposure pathway for soil (i.e., no risks for exposure to soil). To confirm that all contaminated soil was removed from the disturbed areas, soil sample(s) will be collected from each disturbed area at the interface of backfilled soil and virgin soil. Sample(s) will be analyzed via laboratory screening techniques for dyes.

If dyes are not determined to be present in the sample(s), the area will be identified as "clean", potential risks from exposure to soil will be regarded as minimal ('acceptable"), and no further testing of the sample will be performed at that location. If dyes are determined to be present using the dye screening techniques, the sample(s) will either be (1) analyzed for dyes via a quantitative (HPLC) analytical method, in addition to typical Appendix IX constituents. to determine residual dye concentrations or (2) soil will be excavated until the area is considered to be "clean" using the dye screening techniques. In addition. additional samples will be collected to determine the vertical and horizontal extent of contamination. If the excavation approach is not used, additional subsurface soil samples from the location will be needed to determine the extent of contamination. If the excavation approach is used, soil will be removed until the area is "clean" and no further analysis (analytical or risk assessment) is needed.

The results of the screening techniques for the dyes will be needed on a quick-turn basis. The field crew will be waiting on the results of the analyses to either (1) confirm that the area is "clean" or (2) to determine if subsurface soil samples need to be collected to determine the extant of contamination and residual risks.

The NSWC Crane Laboratory is developing quantitative and qualitative analytical methods for dye constituents and will be performing the dye analyses. Additional analyses for Appendix IX constituents (i.e.. VOCs, SVOCs, pesticides/PCBs/ herbicides, and inorganics) will be conducted by Laucks Testing Laboratories.

c) Analysis of representative ground water, surface water, and sediment samples for dye constituents.

Representative ground water, surface water and sediment samples will be collected from the site and screened for dyes. If the screening indicates that dyes are present in the sample, the sample will be analyzed via the HPLC method for dyes and for Appendix IX constituents. If the screening indicates that dyes are not present, the sample will be analyzed only for Appendix IX constituents.

d) Conduct a human health and ecological risk assessment

Only analytical data from this risk assessment/CM sampling event will be used to perform human health and ecological risk assessments. Historical data for the site will not be used because of the various uncertainties associated with data quality.

As mentioned previously, it is assumed that the interim corrective measure (i.e., RCRA cap) is effective and that all dye-contaminated soil at the site was excavated and placed under the RCRA cap. Under this scenario, there is no complete exposure to contaminated soil (i.e., there is no contaminated soil outside the capped area). Risks will not be quantitated for the soil pathway if dyes are not detected in the previously disturbed soil areas using the

screening techniques. If, however, dye-contaminated soil is found in these areas, the potential for a complete soil exposure pathway exists. If contaminated soil is not excavated in these areas, the potential risks via soil exposure will need to be quantified and presented in the risk assessment.

Risks associated with ground water, surface water and sediment will be characterized quantitatively in the risk assessment.

e) Conduct a CMS.

Based on the results of the risk assessment, a CMS will be conducted for the site. If the risk characterization indicates that "acceptable risk" is posed cumulatively by the soil (if applicable), ground water, surface water, and sediment, the CMS will state that the interim corrective measure (i.e., cap) is suffiiient for the SWMU and no further action is warranted. If an "unacceptable risk" is calculated, appropriate remedial alternatives for the media of concern will be evaluated in the CMS to mitigate potential risks.

2. Development of Toxicity Parameters

For many of the dyes, no USEPA-approved toxicity criteria (e.g., reference dose (RfD) and cancer slope factor (CSF)) are published. For this project, a determination will need to be made as to who will develop the RfDs/CSFs for the dyes. The RfDs and CSFs are needed to quantify potential risks and to calculate risk-based screening levels,

Typically, for chemicals that do not have published toxicity criteria, the USEPA's Environmental Criteria Assessment Office (ECAO) develops the criteria for use in the risk assessment. TtNUS has conducted on behalf of the Navy and NSWC Crane a literature search for published toxicity information on the dyes that are thought to be present at the site. The attached table presents a summary of the information that was obtained from the search. As shown in the table, toxicity information was available for only a small number of the dyes. TtNUS using the literature information and USEPA guidance on toxicity development, can develop RfDs/CSFs that could be used to assess risk posed by exposure to these dyes. However, toxicity criteria not developed by the ECAO will be subject to scrutiny. If the toxicity criteria developed by Tetra Tech NUS, Inc. are scrutinized and determined to be inadequate, some aspects of the project (method development or risk assessment) may need to be reworked, which could result in potentially signffiiant delays in the project schedule.

3. Development of Analytical Method for Dves

There are no USEPA-approved analytical methods for the dyes that may be present at the site. Currently, NSWC Crane Laboratory is in the process of developing screening techniques for the dyes and quantitative (HPLC) analytical methods for both aqueous and solid matrices.

The method development is being conducted in accordance with the guidance provided in SW-846 entitled "Guidance for Methods Development and Methods Validation for the RCRA Program". The quality assurance/quality control (QA/QC) for the methods will follow the guidance provided in SW646 Method 8000 series (analysis by chromatography). The Standard Operating Procedures (SOPs) for the methods will be developed in accordance the guidelines established by USEPA Region 5 in the April 1998 OAPP Policy.

Throughout the development of the methods, it is anticipated that questions will arise, which will need to be directed to the USEPA for consensus/resolution. To focus the development of the methods and proceed forward, the following questions need to be addressed at this time:

a) To streamline the method development process, can the list of dyes on which to focus be limited to those that were detected in the soil samples collected from the site during the RCRA cap clearing activities?

b) As shown on the attached table, standards and toxicity information are not available for all the dyes. Can the list of dyes be narrowed to include only those for which standards and toxicity information are readily available?

c) Who is the USEPA contact for the dye analyses?

MINUTES

PRE-PRE-QAPP MEETING
SWMU 2 – DYE BURIAL GROUND
NSWC CRANE, INDIANA
MAY 13, 1999

ATTENDEES:

Bill Gates – Navy
Tom Brent – NSWC Crane
Jackie Vorhauer – NSWC Crane Lab
Carol Witt-Smith – EPA
Allen Debus – EPA
Mario Mangino – EPA
Dan Mazur – EPA
George Bollweg – EPA
Meagan Smith – EPA
Karen Smecker – TtNUS, Inc
David Stair – TtNUS, Inc.
Daneen Resnick – TtNUS, Inc.
Linda Karsonovich – TtNUS. Inc.
Terrie Baranek – ECT.CON. Inc.

BACKGROUND: Because of inherent complexities associated with SWMU 2 – Dye Burial Ground, a pre-pre-QAPP meeting was held to discuss the significant issues (project objectives, toxicity, method development) related to the completion of risk assessment, RFI, and CMS activities at the site. On behalf of the Navy and NSWC Crane, TtNUS, Inc. developed an issue paper for SWMU 2, dated May 3. 1999, for discussion purposes during the pre-pre-QAPP meeting.

MEETING MINUTES: Site *Background* – The history of site operations and summary of investigations completed at the site were reviewed. The brief summary of the site background provided in the aforementioned issue paper was expanded upon (by the EPA) to provide the attendees with a clearer understanding of the issues to be discussed during the pre-pre-QAPP meeting. The following items summarize the site background:

- From 1940s to 1960 military smoke dyes were buried in three main trenches at site.
- In 1972 the trenches were backfilled with soil.
- There is no record of what specific dyes were buried at the site.
- RFI activities have been initiated at the site. Groundwater and seep samples have been collected; no soil samples have been collected. No specific analyses were conducted for dye constituents. Metals contamination was *noted* in the groundwater samples collected from locations within the known disposal area.
- The waste and groundwater table were determined to be in contact with each other (i.e., the groundwater table is saturated).
- As an interim measure, a RCRA cap was designed to eliminate direct contact with the waste and the infiltration of precipitation (leaching of contaminants to groundwater).
- Prior to the completion of the RCRA cap, a geophysical study was completed to delineate the extent of waste disposal. Several disturbed areas of soil (characterized by “fingering” trenches, small

drums, etc.) were found to be located outside of the previously defined waste disposal area. Two trenches were dug at locations south-southwest of the known waste disposal areas. Soil was excavated from the two trenches until it was determined that the soil was "visually clean". The trenches were backfilled with clean soil. The excavated soil was placed within !he planned limits of the RCRA cap. Several types of dyes (identified based on color) were found at the site. Soil samples visually contaminated with dyes were collected from the site; no chemical analyses have been conducted on these samples.

. A RCRA cap has been constructed over the site.

Overall Proiecf *Objectives* – Soil, groundwater, surface water. and sediment samples will be collected at the site to fulfill the objective of the completion of risk assessment (human health and ecological), RFI, and CMS activities. The following general RFI objectives were identified for sample collection activities:

- Soil - Sample soil from the know waste disposal areas, located outside the limits of the RCRA cap where previous excavation activities occurred, to confirm whether residual contamination exists at these locations. If contamination is found to be present, delineate extent of contamination. Soil samples should be collected from the interface between backfilled soil and virgin soil.
- Groundwater - Determine extent of metals groundwater plume. tdentify whether dyes are present in the groundwater and delineate groundwater plume.
- Surface Water and Sediment - Evaluate the potential for the migration of contaminants away from the site via groundwater discharge (drainage and seeps).

From a human health and ecological risk perspective, the objective is to evaluate the incremental risks associated with exposure to contaminated media in absence of further remedial action. For the CMS, the objective is to evaluate whether further remedial actions are warranted to mitigate risks (i.e., whether the RCRA cap can serve as the final remedy). Attendees acknowledged that based *on* the toxicological and waste disposal uncertainties the least likely outcome of the CMS would be the removal of the waste from the site, unless there was a driving risk issue that warranted removal.

Analytical Program – In general, samples collected from the site will be analyzed for a limited list of constituents based on an evaluation of the historical site data. A reduced list of Appendix IX constituents will be proposed in the draft planning documents and rationale will be provided to support this proposal.

Background, Upgradient Well identification – During the meeting, questions were raised as to whether the previously defined background well cluster (02C10, as identified in the RFI Phase Ill Groundwater Characterization study) was still regarded as an upgradient well cluster or whether additional upgradient wells were needed. Well 02C10, which is north-northwest of the main disposal area of the site. may still

be upgradient of the site (i.e., not impacted by site activities). The rough draft Interim Measures Report (Morrison Knudsen) should be reviewed to make this determination. Tom Brent will send TtNUS a copy of the report so that TtNUS can evaluate the information and provide the Navy and NSWC Crane with a recommendation to use 02C10 as a background well cluster or to install a new upgradient monitoring well(s).

Laboratory Screening Methods for *Dyes* – The use of laboratory screening methods for the soil sampling (in the disturbed soil areas outside the limits of the RCRA cap) was discussed. HPLC screening methods are being developed by the NSWC Crane Laboratory. EPA emphasized that the screening method would be a useful tool; that the QA/QC associated with the screening method would be critical to the decision-making process; and that a sensitive screening level (limit of detection) with a specified confidence level would be needed. Attendees recognized that toxicity of the dyes would be an important factor in the method development and the development of an action level for the dyes. Depending on the confidence associated with the screening methods, EPA suggested that the accuracy of the screening level methods be confirmed by quantitative analysis.

Screening methods will be used to characterize the extent of residual contamination at the disturbed soil areas. If dyes are detected at the interface between backfilled soil and virgin soil, additional soil samples will be collected by stepping out from the locations sampled. The sampling approach will be defined in the associated planning documents.

Excavation of *Soil* vs. Characterization of *Extent* of *Residual* Contamination – Attendees briefly discussed whether quantitative dye analyses were needed if the screening methods identified the presence of residual contamination at the disturbed areas of soil. It was determined that quantitative analyses would be required to determine the incremental risks associated with the residual dye contamination. EPA doesn't feel that additional excavation of soil is likely because of the uncertainties associated with waste disposal. Regardless, the waste would have to be characterized prior to disposal.

Type of Risk Assessments to be Performed – Attendees briefly discussed the type of risk assessments to be conducted. Risk assessments (human health and ecological) will be conducted to evaluate the incremental risks associated with the site under current and future site conditions. Since the risk assessment leads to the CMS, assumptions regarding the limiting of exposure must be clearly defined in the assessment. EPA stressed the use of the term "incremental risks".

The human health risk assessment will be an exposure-driven scenario evaluation. A screening level ecological risk assessment will be performed. For the ecological risk assessment, EPA indicated that the

additive risks associated with the Ammunitions Burning Ground (a nearby site) be considered in the assessment because of the likelihood of exposure to both sites.

Toxicity/Toxicological Parameters *for Dyes* - Attendees recognized that limited to no USEPA-approved toxicity criteria (Reference Doses and Cancer Slope Factors) may be available for the dye contaminants. The attendees discussed the approach of developing toxicity parameters for a limited list of dyes with known chemical structures and potentially using the toxicity parameters as surrogates for other dyes whose structures are not known. EPA indicated that the EPA Office of Research and Development (ORD) typically develops toxicity criteria when none are published. George Bollweg was identified as the EPA lead for the development of toxicity factors for the dyes. Some toxicity information for me dyes has been pulled together by TtNUS; TtNUS will submit a summary of the information to the EPA (in Lotus format).

Dan Mazur indicated that toxicity tests may be necessary to determine the toxicity of dyes in relation to ecological receptors. TtNUS indicated that this would be considered, but that the ecological risk assessment to be performed was designed to be a screening-level ecological risk assessment, which typically do not include toxicity testing. Dan also questioned whether the EPA had any information on the chemical and physical properties of dyes. Carol Witt-Smith indicated that the team would be relying on information available from NSWC Crane.

Analytical Methods for Dyes - Allen Debus was identified as the EPA contact for method development. Because of the previous analytical work performed by the NSWC Crane Lab (for the frac tank water analyses), the EPA emphasized the importance of Nancey Maegerlein's (NSWC Crane Lab Manager) involvement during method development. The Lab indicated that the methods previously used for the water analyses were being built upon for this project. The EPA indicated that the proper QA/QC should be included during method development.

For the qualitative screening method, the EPA indicated that, at a minimum, information on the following should be included: detection limits, spike recoveries, calibrations using different scenarios [individual vs. a mix of dye constituents], and a method detection limit study.

For the quantitative analytical methods. the EPA questioned whether mass spectroscopy (MS) could be used in addition lo high performance liquid chromatography (HPLC). The NSWC Crane Lab did not feel this was a viable option since most of the dyes are not volatile.

Approaches to limiting the method development for the quantitative analyses were discussed by the attendees. Most attendees agreed that the soil samples collected from the site could be used to narrow

the list of dyes. However, Carol Witt-Smith commented that samples were not collected from all know disposal areas. The EPA suggested that groundwater samples be collected at the site to further narrow the list of dyes for groundwater. surface water, and sediment. NSWC Crane suggested that samples be collected from 3-4 monitoring wells that are closest to the main waste disposal area, which previously exhibited high concentrations of inorganics. A specific approach was not decided upon by the attendees; an approach will be developed by the Navy and presented in associated planning documents.

From a risk assessment perspective, George Boilweg expressed concerns about limiting the list of dyes to be analyzed. However, other EPA attendees indicated that a surrogate approach to risk assessment would probably be necessary. TtNUS indicated that this uncertainty with the assessment would be discussed in the uncertainty section of the risk assessment.

Schedule – The project schedule was briefly discussed by the attendees. The development of analytical methods for the dyes were identified as being critical to the project schedule. The methods may take between 3- 6 months to develop. TtNUS indicated that the planning documents were currently being prepared.

Miscellaneous – The EPA indicated that the safely concerns associated with sampling and analysis for the dyes should be addressed in the associated planning documents.

Mario Mangino questioned whether there were any records of volume of dyes disposed at the site. NSWC Crane indicated that specific records are not available. Mario also suggested that the volume be estimated based on the dimensions of the trenches and the results of the geophysical study. Tom Brent agreed to do some research to see if any records are available lo provide a general idea of how much waste was disposed.

AGENDA
NSWC CRANE LAB DYE METHOD DEVELOPMENT

Attendees:

EPA Region V	Peter Ramanauskas, Allen Debus
NSWC Crane:	Tom Brent, Forrest Hawman, Nancey Maegerlien, Sarah Payline
Navy SouthDiv:	Bill Gates
TetraTech NUS:	Linda Karsonovich, Daneen Resnick, Karen Smecker, David Stair
ECT.CON INC.	Terrie Baranek

Date: September 10, 1999

Subject: NSWC Crane Lab Dye Method Development

Discussion Items:

. Brief Site Background

From the 1940's until 1960. approximately 50 tons of dyes were placed into three open trenches at SWMU 2. There is no record of the specific dyes that were burled in these trenches. The trenches were approximately 10 feet wide, 6 feet deep and 50 feet long. In 1972, the trenches were backfilled with soil. RFI activities have been initiated at the site. Ground water and seep samples have been collected and analyzed for VOCs, SVOCs, pesticides, PCBs, metals. and miscellaneous inorganic parameters. No soil samples have been collected at the site. As an interim measure, a multi-layered cap system was proposed for the site. To initiate this process, a geophysical survey was conducted to delineate the boundaries of the site. Based on these results, a cap was designed. During installation of the cap, disturbed soil/waste areas outside the boundaries of the cap were identified. Work stopped and a field investigation was initiated. NSCW Crane and USEPA Region 5 identified these soil/waste disturbed areas and provided oversight during excavation of visually contaminated material and placement of the material into the dye burial trenches. These areas were excavated until no dyes were visually observed in the soil. After completion of these excavations, the installation of the multi-layered cap system was completed. Since some of the work during the cap installation was conducted in the winter months, precipitation in the trench areas was accumulated. This material was placed in frac tanks. The aqueous waste was qualitatively analyzed for dyes. These analyses were performed by the NSWC Crane Lab with oversight from USEPA Region 5. This is the only dye analysis performed at SWMU 2.

. Project Objectives

The overall objective of this project is to conduct a RFI, Risk Assessment and Corrective Measures Study. To achieve this goal, soil, ground water, surface water and sediment samples will be collected. The following general sampling and analysis objectives were identified:

- Soil – Sample soil from the disturbed soil/waste areas that were located outside the cap boundaries and excavated prior to cap installation. This sampling will be to confirm that all dye contamination was excavated from these areas. Soil samples will be collected from the interface between the backfilled soil and virgin soil. These Samples will be screened for dye contamination. If dyes are found, the samples will be quantitatively analyzed for dyes.

- Ground water – Confirm the presence of metals contamination in the ground water and determine the extent of metals ground water plume. Identify whether dyes are present in the ground water. If they are present delineate plume These samples will be screened for dye contamination. If dyes are found, the samples will be quantitatively analyzed for dyes,
- Surface water and sediment – Evaluate the potential for the migration of contaminants away from the site via ground water discharge (seeps and drainage ways). These samples will be screened for dye contamination. If dyes are found, the samples will be quantitatively analyzed for dyes.
- All samples will be analyzed for metals and miscellaneous inorganic parameters.

. Dye Analytical Method Summaries

- Screening Method (Thin Laver Chromatography).

Background:

Thin-layer chromatography is a separation method based on adsorption of the sample to the stationary phase. The adsorbent layer (stationary phase) is usually formed on a 20x20 cm glass plate and consists of silica gel. The sample mixture is applied to the lower edge of the plate. The plate is then placed in a tank with a suitable developing solvent(s). The individual substances from the sample mixture are transported upwards at differing rates. Adsorption, partition, or ion-exchange processas effect the separation. When the solvent has traveled sufficiently up the silica plate, the plate is removed and dried. The separated substances are detected visually. by UV lamp. or modified with a staining reagent The substances are identified by the ratio: rate of migration from the starting point to the distance the solvent traveled from the starting point. This value is called the R_F value.

$$R_F = \frac{\text{distance of substance from start}}{\text{distance of solvent from start}}$$

Experimental:

To date the focus has been on the organic dyes and trying to find a suitable solvent that can separate the dye standards. Solvents which have been tested are ethyl acetate/hexanes, cyclohexane/ethyl acetate THF/xylenes, THF/p-xylene, hexanes/p-xylene. These solvents have bean used in differing ratios. The results indicate that the best separation is achieved for THF/p-xylene.

Work has also been done on varying the amount of sample on the TLC plate. Problems such as bleeding and poor resolution can occur if the TLC plate is overloaded.

Scanning the plates into the computer allows for enhancement of dye color separation and preservation/documentation of the findings.

Quantitative Method- High-Performance Liauid Chromatography (HPLC)

Dye quantitation will be accomplished with HPLC utilizing a Photo Diode Array (PDA) detector. To date, the work has focused in several areas:
(1) Analytical/Literature identification of the dyes that ware used in the production process at the Base;

(2) Identification of an appropriate HPLC mobile phase and column that will enhance separation of the dyes; and
(3) Development of a spectral library based on the standards that have been obtained. This will be used to identify dyes based on absorption spectral pattern recognition.

The use of the PDA will enable chromatograms to be **extracted** at multiple wavelengths from a single analytical run. as well as the **extraction** of an absorption spectrum for each dye. The absorption spectra library may be used to compare field sample absorption spectra with absorption spectra obtained from standards for dye identification.

The dyes in question range from very polar acid and base dyes to nonpolar dyes. With such a broad range of dyes it is doubtful that a single HPLC method will be sufficient although recent procurements may change this. Currently, the dyes have been divided into two classes consisting of water-soluble dyes and nonwater-soluble dyes. Separation methods for these two classes are being developed separately.

At the present time we are still unable to separate several of the dyes for quantitation. Recently. afler consulting with a column manufacturer, several different columns have been ordered to assist in the separation. Our lab has also procured a new state-of-the art HPLC system, in addition to our current system, which will be operational within the next week.

Several publications are being referenced for HPLC mobile phase and column selection, although most do not attempt to separate such **a large quantity of dyes. The** only publication found which analyzed a multitude of dyes was reference 9 (See Attachment 2). The method proposed by Wegener has produced the best results in our tests. Initial results indite that this may be the best method for both the water-soluble and nonwater-soluble **dyes.** We have attempted to reproduce this method exactly, although we did not have the exact same column available at the time. if has since been ordered. It is hoped that when this column is received our separations can be further improved.

. Method Development Objective and Action Timeline

- See Attached Abbreviated Activity Timeline (Attachment 1)
- See Attached List of References (Attachment 2)
- Update on Method Development Status
  - **Standards (See** Attachment 3)
    - The analytical dye method development (according to RCRA Guidance for Method Development) will be performed for the dyes for which standards could be obtained.
    - Many of the dye standards were developed specifically for this project and are not off-the shelf products.
  - **Co-elution** Issues (Further refined upon receipt and implementation of the new HPLC system and column)
  - **Unknown Issues**
    - Dyes for which no standards could be obtained but were identified in the RFI report or were known to be used in the production process at the Base.
    - Dyes that were detected in the soil samples from the site but were not in the RFI report or known to be used in the production process at the Base.

- Utilization of various methods (e.g., NMR. MS, etc.) to identify unknown dyes that were in the RFI report or ware known to be used in the production process at the Base.
- Method Development will not be executed for the unknown dyes.

- **QC Issues**
  - Anthracene was analyzed for in previous work conducted at the site. It was analyzed as a SVOCs via SW-846 8270 (GUMS).
  - Anthracene was not identified as a parameter of interest for the site.
  - Anthracene is a precurser to making dyes and does not exhibit color.
  - Anthracene may be used as a surrogate spiking compound for the method.
- Preliminary Method Detection Limits
  - TLC To Be Determined
  - HPLC To Be Determined

. Summary of Toxicological Information

- See Attached Toxicity Table (Attachment 4)
- The toxicological information will be used to develop risk-based screening criteria. These criteria will drive the level of detection required for the methods.

ATTACHMENT 1

ABBREVIATED ACTIVITY TIMELINE

ABBREVIATED ACTIVITY TIMELINE

Timeline	Activity Completed	Activitv
On-going Jan-99 thru Present		1. Continue literature research on potential analytical methods alreadv developed (e.g., SW646 Method 8321A). Read articles to assist in identifying viable extraction procedures, system requirements. interferences, and corrective actions. See Attachment 1 Item 1 for additional articles/contact for the research. **Required Documentation (See Attachment 1)** • Copies of articles used to develop analytical and/or extraction methods - Copies of telephone contact logs • Compilation and summary of potential analytical methods, extraction procedures and interferences from the articles.
Jun-99		2. Accurately identify dyes. Group all dyes that could be present at the site according to similar chemical structures. **Required Documentation (See Attachment 2)** • Copies of documentation to support identification of dyes presently listed • Documentation of attempts to identify unknown dyes by mass spectral matching with NIST or HSDB
Jun-1-99 Thru Sept-l-99		3. Obtain known standards for dves (or at least one for each chemical group) Contact Pfaltz & Bauer – Request dye standards and chromatograms. Standards obtained See attached list. Talked to chemists regarding their experience with dye. analysis. Summary of pertinent information is attached. Contact Aldrich Contact Aakash Chemicals and Dyestuffs Contact Crompton and Knowles Contact Sandoz Chemicals **Required Documentation (See Attachment 3)** • Final list of standards per supplier and timeframe for obtaining the supplies. • Copies of telephone logs, faxes, etc., documenting contacts with chemical suppliers
Sept -1-99 Thru Oct -1- 99		4a. Perform Calibration Study Determine high and low end of calibration range Determine linear portion of curve Establish reporting limit Establish spiking level Identify and resolve co-elution problems - Identify and resolve chemical interference problems [NOTE: Initial calibration linearity (%RSD and correlation coefficient) are to be determined using procedures outlined in SW-646 Method 8000.] **Required Documentation (See Attachment 4)** • Tabular compilation of the various analytical methods • Evaluation of pros and cons of the various methods. Identification of necessary modifications to the referenced methods based on availability of equipment, applicabitii of solvent systems, etc. - Selection of methods to test - Tabular compilation of test results for each compound and method - Evaluation of methods tested. Identification of the need for further testing and the systematic alterations of variables to obtain the greatest response, least interference, greatest repeatibility, etc. Identify stock standard(s) and concentrations and surrogates, internal standards, and matrix spike/blank spike mixtures per method

ABBREVIATED ACTIVITY TIMELINE

Timeline	Activity Completed	Activity
Sept-1-99 Thru act-1 -99		4b. Develop TLC Screening Method for Oraanic Dye Standards • Find suitable solvent(s) for separating organic dyes • Protocol for staining dyes that cannot be visually seen or detected with a UV lamp • Prepare method for extracting sample from TLC plates • Determine detection limits via HPLC • Order more standard dyes that were not ordered earlier
Oct -1-99 Thru Nov -1-99		5a. Develop and/or Confirm Extraction Procedures Analyze seven replicate per matrices appropriate for project at two concentrations for each extraction method lo be tested. **Required Documentation (See Attachment 5)** • Tabular compilation of aqueous and solid extraction methods • Evaluation of methods tested. Identification of modifications to referenced methods based on availability of equipment, applicability of solvent systems, etc. • Select methods for testing • Tabular comparison of method blank performance, bias. precision, MDL. and RQL for each extraction method. (NOTE: MDURQL are determined from the low concentration only.) • Evaluation of methods tested. Identification of the need for further testing and the systematic alterations of variables to obtain the greatest response, least interference, greatest repeatability. etc. 5b. Develop TLC Screening Method for Water-Soluble Dves • Find suitable solvent(s) for water-soluble standard dyes • Develop protocol for staining samples that cannot be visually seen or detected with a UV lamp • Prepare method for extraction of sample from TLC plates • Determine detection limit via HPLC
Nov -1-99 thru Nov -16-99		6a. Method Optimization/Determine Exact Analytical Method to Use Method optimization will commence with the following parameters: List initial conditions (Based on analyst's best judgement) Based on the information obtained in Steps 4 and 5, identify variables that need to be 'tweaked" in order to optimize the best possible field screening method and best laboratory method. NOTES: a) Analytical methods already developed for dyes (e.g.. methods described in the articles reviewed and SW-846 8321A) should be a great help in this step. For this reason, this step should be relatively short in duration (approx. 2 weeks) b) The following QC items are included in these runs: Surrogates Internal Standards Method Blanks Laboratory Control Samples (i.e., blank spikes) **Required Documentation (See Attachment 6)** • Tabular compilation of test conditions and results Describe the systematic alterations of variables to optimize extraction and analysis conditions. Selection of optimal field screening and laboratory methods

ABBREVIATED ACTIVITY TIMELINE

Timeline	Activity Completed	Activity
Nov -1-99 thru Nov -16-99		6b. Method Optimization of TLC Analysis for Dves Based on the information obtained in Steps 4b end 5b, identify variables that need to be 'tweaked' in order to optimize the best possible field screening method and best laboratory method.
Nov-17-99 Thru Dac-1-99		7. Validate Analytical Method (Evaluate accuracy. precision, and repeatability in a clean matrix) Initial determination of these QC factors will be done in a spiked clean matrix similar to the environmental media to be analyzed but free of interferences (i.e., certified clean soil and reagent water) Values will be obtained using 7 replicates at both high (50 x low standard) and low (5 x tow standard) spike concentrations. The following QC items will be used to evaluate accuracy, precision and repeatability: Surrogates Method Blanks Internal Standards Laboratory Control Samples (i.e.. blank spikes) Corrective actions will be developed and documented to ensure that QC requirements are maintained, **Required Documentation (See Attachment 7)** Compile bias, precision, MDL. and RQL data for both the field screening and laboratory methods for each "clean' matrix Compile method blank, surrogate. LCS, and IS data for both the field screening and laboratory methods for each "clean' matrix. Calculate relative percent difference and acceptance ranges based on procedures located in SW-846 Method 8000.
Dec -1-99 Thru Dec -7-99		a. Effect of Interferences Mlethod interferences will be determined in a spiked clean matrix. Both positive and negative interferences will be documented and addressed. **Required Documentation (See Attachment 8)** Based on the possible interferences identified during your research and interferences encountered during method development, identify variables in the "optimal" method which may be altered to minimize or eliminate the interference for each matrix. Design an experiment or experiments to systematically alter the variables and test the effect on target compound recovery. identification, and quantitation. Discuss the results of the experiment and present percent recovery data, chromatograms, or other related material to support the adoption and/or rejection of the modification of the optimal method. Documentation of interferences are attached and include the following: Co-alution of or with target analytes Enhancement or suppression of target analyte signals caused by interference Necessary and optional cleanup procedures to minimize the effect of interferences Matrix-specific difficulties

		(NOTE: The items listed above are typical interference issues. These must be addressed as well as any other interference issues that arises during the method development.)
Timeline	**Activity Completed**	**Activity**
Dec -7-99 thru Dec -14-99		9. Analyze Site-Specific Soil and Groundwater Samples/Matrix Suitability Using the information obtained in steps 1 through 8, apply the methodology to site-specific samples. Ground water, surface water, sediment and soil are potential field samples for the field program. Therefore, these matrices will be analyzed using the method developed. In conjunction with the analyses of these samples, QC samples including the following will be incorporated in the analytical sequence: Initial calibration (if the current one is older than 6 months) Daily calibration at the low, midpoint, and high end of the calibration range at the beginning of the day; a midpoint sample after each sample group; and a midpoint sample after the last sample of the day. (NOTE: The three-point calibration must be done every 12 hours if the system runs for over 12 consecutive hours.) Each sample is spiked with a surrogate(s) and internal standard Method Blank Matrix Spike/Matrix Spike Duplicate sample extracted the frequency of 1 pair per 20 samples Laboratory Control Sample extracted at the frequency of 1 per 20 samples If QC items are not meet, corrective action will be implemented as described in Attachment 7. **Required Documentation (See Attachment 9)** - Compile the results of the replicate analyses for each site-specific matrix (ground water, surface water, sediment, soil) in the format given previously in section 3 of Steps 4 and 5. This includes method blank performance, matrix, bias, precision, and quantitation limits. - Present data in a CLP-like format (minus the electronic deliverable) as described in the December 16.1996 Memorandum. Typical deliverable requirements for organic environmental analyses by chromatography include the following documentation: • Copy of applicable extraction and instrument logbook pages • Chromatogram, quantitation report, and Form 1 for each sample, method blank, matrix spike, and blank spike • Initial calibration data including: concentration, %RSD, correlation coefficient. chromatograms, and quantitation reports • Continuing calibration data including: percent difference from initial calibration, chromatograms. and quantitation reports • Summary of surrogate recoveries and comparison to acceptance range calculated in Step 7. • Summary of matrix spike recoveries and RPDs and comparison to acceptance ranges calculated in Step 7. • Summary of blank spike recoveries and comparison to acceptance ranges calculated in Step 7. • Summary of internal standard areas and comparison to internal standard areas of continuing calibrations. - Compile matrix spike/matrix spike duplicate recovery data, surrogate recovery data, and blank spike recovery data for each site-specific matrix

		for both the field screening and laboratory methods. Calculate relative percent difference and acceptance ranges based on procedures located in SW-646 Method 6000.
Timeline	**Activity Completed**	**Activity**
Dac -14-99 thru Dec -21-99		10. Method Detection Limits (MDLs) and Reliable Quantitation Limits (RQLs) The limits determined in clean matrices indicate the limits of the acceptable performance for the method. These will be tabulated as the method detection limits. Matrix effects may affect the achievable quantitation limits on site-specific samples. Therefore, as noted in step 9, reliable quantitation limits based on the site-specific samples will be determined. Both MDLs and RQLs will be tabulated and presented as part of the method. Because MDLs are generated from a statistically valid population data set, information used in the development of the MDLs and RQLs will be gathered from steps 3 through 9. At a minimum, the MDL must be calculated from the low concentration standard deviation (s) determined from 7 replicate measurements par matrix (NOTE: Blanks will be run with these 7 samples.) MDL = 3.143 * s The RQL is calculated as follows: RQL = 4 * MDL **Required Documentation (See Attechmant 10)** Compare/contrast MDLs and RQLs for each matrix in both "clean" and site-specific matrices. Discuss effects of site-specific matrix interferences on the MDLs/RQLs for both the field screening and laboratory methods. Document alterations of the RQL based on the information obtained in step 9.
Jan -3-2000 thru Jan -17-2000		11. Evaluate False Positives and False Negatives for the Field Screaning Method A candidate screening method should produce no more than 10% false positives and should not produce false negatives. To evaluate the method, 35 samples par matrix will be spiked at one half the RQL tested to establish the percentage of false positives. 35 samples per matrix will be spiked at twice the RQL and tested to establish the percentage of false negatives. A sufficient volume of each spiked sample will be prepared to complete each test with one lot of material, The effect of non-target interference will be addressed. A candidate method should produce no more than 10% false positives for a set of 20 samples that contain a 100-fold excess of interference. Positive interferences to test should be taken from step 6.

ABBREVIATED ACTIVITY TIMELINE

Timeline	Activity Completed	Activity
		Rewired Documentation (See Attachment 11) - 'Compile results for false negative/false positive test. Calculate percentage of false negatives/false positiies. - Compile results of non-target interference test Identify positive and negative interferences. C&late percentage of false positives.
Jan -17-2000 thru Jan -24-2000		12. Prepare SOP Prepare SOP according to example provided previously.

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7 . Dr. Robert E. Keyes. Chemist, Abbott Laboratories, in communication.
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6. Dr. George Baughman. University of Georgia, in communication.
-Published multiple articles on dyes in the environment (USEPA Research Lab Athens, retired)

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9. Wegener, J.W. Determination of Organic Colorants in Cosmetic Products bv High-Performance Liquid Chromatooraphv. Chromatographia, Vol. 24 1967

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21. SW-846 Third Ed. Method 3500. September 1986.

22. SW-046 Third Ed. Method 8310, September 1986.

23. George L. Baughman, Eric J. Weber, Rebecca L. Adams, and Mary Sue Brewer.*Fate* of *Colored Smoke Dyes.* U.S. Dept. of Commerce, January 1992.

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ATTACHMENT 3

STANDARDS

STANDARDS ORGANIC DYES

Lab #	Source	CI Name	Other Name	CAS#	CI#	Type
Std(D0003)	Aldrich	Disperse Blue 14	1,4-bis(methylamino)anthraquinone	2475-44-7	61500	Anthraquinone
Std(D0045)	Aldrich	Disperse Red 9	1-methylaminoanthraquinone	82-38-2	60505	Anthraquinone
**	Aldrich	Disperse Violet 1	1,4-diaminoanthraquinone	128-95-0	61100	Anthraquinone
Std	ACROS	Anthracene		120-12-7	N/A	Anthracene
Std	ACROS	Azoic Diazo Component 36	1-aminoanthraquinone	82-45-1	37275	Anthraquinone
Std	ACROS	Benzanthrone		82-05-3	N/A	Anthraquinone
Std	ACROS	Smoke Dye (orange)	Quinizarin	81-64-1	58050	Anthraquinone
Std	ACROS	Solvent Green 3	Quinizarine Green, SS	128-80-3	61565	Anthraquinone
Std	ACROS	Solvent Orange	Sudan II	3118-97-6	12140	Monoazao
**	Not found	Solvent Orange 3		495-54-5	11270B	Monoazao
**	ACROS	Solvent Yellow 3		97-56-3	11160	Monoazao
**	Aldrich	Solvent Yellow 14	Sudan I	842-07-9	12055	Monoazao
Std(D0050)	ACROS	Solvent Yellow 33	Quinoline Yellow. spirit soluble	8003-22-3	47000	Quinoline
**	Pfaltz & Bauer	Solvent Yellow 2	Butter Yellow	60-11-7	11020	Monoazao
**	Pfaltz & Bauer	Solvent Violet 13	Alizurine Purple SS/Quinizarin Blue	91-49-1	60725	Anthraquinone
	TCI	Solvent Red 1	Sudan R	1229-55-6		Monoazao
Std	ACROS	Solvent Red 24	Sudan IV	85-83-6	26105	Disazo
	TCI	Indanthrene Yellow		129-09-9		Anthraquinone
	Pfaltz and Bauer	Pigment Red 1		10/2/10		Monoazao
**	Aldrich	Vat Blue 1	Indigo	482-89-3	73000	Indigoid
Std (D0005)	Not found	Vat Yellow 4		128-66-5	59100	Anthraquinone
	Pfaltz and Bauer	Anthrafurlin		117-12-4		Anthraquinone
Std (D0002)	Pfaltz and Bauer	1,4-Diamino-2,3 dihydmanthraquinone		81-63-0	N/A	Anthraquinone
**	Aldrich	2-Aminoanthraquinone		117-79-3	N/A	Anthraquinone

** Dyes on RFI that Tetra Tech did not know about

ATTACHMENT 4
TOXICITY TABLE

TOXICITY TABLE - ORGANIC DYES

CI Name	Other Name	CASS	CIS	Type	
Anthracene		120-12-7	N/A	Anthracene	This chemical is on the Hazardous Substance List
					IARC- Not Classifiable as to Its Carcinogenicity to Humans (Group 3)
					ECDIN-LD50 mouse, oral, >17g/kg
					NTP- Ames Test (+ and - results)
					HSDB- Ecotox (LC50's, fish,frog, crustacean), acute dermal, oral studies, bioconcentration,soil concentrations
Disperse Blue 14	1,4-bis(methylamino)anthraquinone	2475-44-7	61500	Anthraquinone	This chemical is in the EPA Inventory under TSCA
					ECDIN: LD50, Mouse, Intravenous, 180 mg/kg
Disperse Red 9	1-methylaminoanthraquinone	82-38-2	60505	Anthraquinone	No toxicity data available
Disperse Violet 1	1,4-diaminoanthraquinone	128-95-0	61100	Anthraquinone	ECDIN- LDLo rat, oral 4920 mg/kg/ Chromosome aberration
Azoic Diazo Component 36	1-aminoanthraquinone	82-45-1	37275	Anthraquinone	ECDIN- LD50 rat, Intraperitoneal 1500 mg/kg
Benzanthrone		82-05-3	N/A	Anthraquinone	ECDIN- LD50 rat, Intraperitoneal 1500 mg/kg,LD50 mouse, intraperitoneal, 290 mg/kg
					CITI- Ecotox, Bioaccumulation, Biodegradation
					HSDB- Environmental Fate/ Exposure Summary
					CCRIS- Ames Test w/wo metabolic activation (+ and - results)
					MSDB- Not listed by ACGIH, IARC, NIOSH, NTP or OSHA as Carcinogenic
Smoke Dye (orange)	Quinizarin	81-64-1	58050	Anthraquinone	CCRIS- Ames test w/wo metabolic activation (+ and - results)
Solvent Green 3	Quinizarine Green, SS	128-80-3	61565	Anthraquinone	ECDIN- LD50 rat, oral 3060 mg/kg
Solvent Violet 13	Alizurine Purple SS/Quinizarin Blue	81-48-1	60725	Anthraquinone	CCRIS- Carcinogenicity, mouse, dermal
					ECDIN- LDLo mouse, intraperitoneal, 512 mg/kg
					NTP- Ames Test (- result)
Indanthrene Yellow		129-09-9		Anthraquinone	CCRIS- Ames test w/wo metabolic activation (- results)
Vat Yellow 4		128-66-5	59100	Anthraquinone	IRAC: Not classifiable as a human carcinogen (Group 3); Animal Limited Evidence
					CCRIS: Carcinogenicity, mouse, rat, oral/ and mutagenicity studies
					NTP- 90 Day,rat, mouse, oral/ Chronic (2 year) carcinogenicity rat, mouse, oral
					Ames test (- results)
Anthrafurin		117-12-4		Anthraquinone	CCRIS- Ames test w/wo metabolic activation (+ and - results)
Solvent Violet 47	1,4-Diamino-2,3 dihydroanthraquinone	81-63-0	N/A	Anthraquinone	CCRIS- Ames test w/wo metabolic activation (+ and - results)
	2-Aminoanthraquinone	117-79-3	N/A	Anthraquinone	NTP- LD50 rat, Intraperitoneal, 1500 mg/kg, 14 Day and 13 week, rat, mouse, oral/
					Chronic (2 year) carcinogenicity, rat, mouse, oral/ Ames Test (+ results)
					IARC- Not Classifiable as a Human Carcinogen (Group 3)
					CCRIS- Carcinogenicity, mouse, oral/ Ames Test w/wo metabolic activation (+ and - results)
					HSDB- Environmental Fate/Exposure Summary
					CEPA - Inhalation 3.3E-02; Oral 3.3E-02 1/(mg/kg-day)
Solvent Red 24	Sudan IV	85-83-6	26100	Unsub.	CITI- Ecotox, Bioaccumulation
					IARC-Not Classifiable As to Their Carcinogenicity to Humans (Group 3)
					ECDIN- Ames Test w/wo metabolic activation (+ and - results)
Vat Blue 1	Indigo	482-89-3	73000	Indigoid	NTP- Ames Test positive
					No other toxicity data available
Solvent Orange 7	Sudan II	3118-97-6	12140	Monoazao	IARC- Not Classifiable as a Human Carcinogen (Group 3)
					ECDIN- Ames Test w/metabolic activation (+ result)
					CCRI- Ames Test w/wo metabolic activation (+ and - results)
Solvent Orange 3		495-54-5	112700	Monoazao	IARC- Not Classifiable as a Human Carcinogen (Group 3)
					ECDIN- LDLo mouse, oral 1875 mg/kg
					NTP- Ames Test (+ results)
					HSDB- Environmental Fate/Exposure Summary
					CCRIS- Carcinogenicity study mouse, oral (+ result)
Solvent Yellow 3		97-56-3	11160	Monoazao	NTP- LDLo mouse, oral 800 mg/kg/ LDLo rat, oral 1500 mg/kg
					IARC- Possibly Carcinogenic to Humans (Group 2B)
					CCRIS-Ames Test w/wo metabolic activation (+ and - results)/ Carcinogenicity mouse, oral (+ result)
					HSDB- Environmental Fate/ Exposure Summary
					CEPA - Inhalation 3.8E+00; Oral 3.8E+00 1/(mg/kg-day)
Solvent Yellow 14	Sudan I	842-07-9	12055	Monoazao	IARC- Not Classifiable as a Human Carcinogen (Group 3)
					NTP- 14 Day and 13 Week, rat, mouse, oral/Chronic (2 Year) rat, mouse, oral
					CCRIS- Carcinogenicity study mouse, subcutaneous, (+ result)/ Ames Test w/wo metabolic activation (+ and - results)
Solvent Yellow 2	Butter Yellow	60-11-7	11020	Monoazao	IARC- Possibly Carcinogenic to Humans (Group 2B)
					NTP- LD50 rat, oral 200 mg/kg/ LD50 mouse, oral 300mg/kg
					ECDIN- LD50 rat, Intraperitoneal 500 mg/kg/ Aquatic Toxicity
					CCRIS-Ames Test w/wo metabolic activation (+ and - results)
					HSDB- Environmental Fate/Exposure Summary
					CEPA - Inhalation 4.6E+00; Oral 4.6E+00 1/(mg/kg-day)
Solvent Red 1	Sudan R	1229-55-6		Monoazao	CCRIS- Ames Test w/wo metabolic activation (+ and - results)
					HSDB - Environmental Fate/Exposure Summary
Pigment Red 4		6410-10-2		Monoazao	No toxicity data available
Solvent Yellow 33	Quinoline Yellow, spirit soluble	8003-22-3	47000	Quinoline	CCRIS- Chronic rat, oral, Ames Test, w/wo metabolic activation (+ and - results)
					HSDB-LD50, mouse, oral, 4 gm/kg./ LD50 rat, oral >13650 mg/kg
					Listed on the TSCA Inventory
					NTP- Chronic (2 year) study, rat, oral,/ 90 Day and 14 Day, rat and mouse, oral

NTP - National Toxic Program
CCRIS - Chemical Carcinogenesis Research Information System
HSDB - Hazardous Substances Data Base
CITI - Chemicals Inspection and Testing Service, Japan
IARC - International Agency for Research on Cancer
MSDS - Material Safety Data Sheets
ECDIN - Environmental Chemicals Data and Information Network
CEPA - California Environmental Protection Agency Criteria for Carcinogens

CONFERENCE CALL MINUTES

SOUTHDIV CLEAN – NSWC Crane
September 10, 1999; 10am (Pittsburgh, PA/Charleston, SC) and 9am (Crane, IN)
Recorded by TtNUS

Subject: NSWC Crane Lab Method Development for Dyes at SWMU 2 - Dye Burial Grounds

Attendees: EPA Region 5 - Peter Ramanauskas and Allen Debus; SOUTHDIV - Bill Gates; NSWC Crane (environmental division) - Tom Brent; NSWC Crane Lab - Forrest Hawman, Sarah Paylien, Nancey Maegerlein; TtNUS - Karen Smecker, Daneen Resnick, Linda Karsonovich, David Stair; and ECT.CON, Inc (TtNUS subcontractor) – Terrie Baranek

Background: RFI, CMS, and risk assessment activities are to be completed at SWMU 2. TtNUS is currently preparing planning documents for these activities, which includes environmental sampling. The NSWC Crane Laboratory is developing analytical methods for dyes. Two possible analytical methodologies have been identlied for use in sample analysis -- Thin Layer Chromatography (TLC) and High Performance Liquid Chromatography/Photo Diode Array (HPLC/PDA). The purpose of today's conference call was to apprise the EPA of the analytical methodologies, to obtain preliminary approval of the use of the analytical methods, and to receive input regarding the path forward for method development.

Summary of Conference Call: Based on research performed by the Crane Lab, TLC appears to be the best method for the field screening (screening-level) analysis and HPLC/PDA appears to be the best method for the quantitative analysis. The method development work will continue to concentrate on the dyes suspected to be present at the site and for which known standards can be purchased. Dyes that are considered to be "unknowns" will be evaluated using a similar approach to Tentatively Identified Compound (TIC) analyses. The quantitative analytical methods (for HPLC/PDA) will be developed using anthracene as a surrogate spiking compound. EPA's toxicological support will be brought into the process so that they can develop toxicity criteria (Reference Doses/Cancer Slope Factors [RfDs/CSFs]) for dyes.

The following items summarize the conversation that took place between the attendees:

1. ***Site History*** - Terrie summarized the history of the site and emphasized that dye analyses were not conducted during historical site investigations. Tom clarified that initially the site was thought to consist of 3 trenches where dyes were disposed, however, during historical site investigations additional areas (trenches, fingers of trenches, etc.) of disposal were discovered.

   Terrie stated that soil, groundwater, surface water, and sediment samples are to be collected at the site to complete RFI, CMS, and risk assessment activities. Groundwater, surface water, and sediment samples will be collected to evaluate the migration of dyes from the site. Soil sampling will be limited to areas surrounding the RCRA cap, which were previously excavated using visual observations (i.e., the additional areas of disposal discussed in the previous paragraph). Soil from these areas were placed under the RCRA cap prior to its construction. However, no confirmation sampling was conducted to determine that the are was "clean". The objective of the soil sampling will be to confirm that areas previously excavated are in fact free of dye contamination.

   Terrie indicated that two types of analytical methods for dye analysis were being developed by the Crane Laboratory --- one for use as a screening method, TLC, and one for use as a quantitative analysis, HPLCIPDA.

2. *TLC Method* Development ▪ Sarah and Forrest summarized their work on the screening method. Originally HPLC had been proposed as the screening method. However, based on an extensive literature search and contact with technical experts in the field of dye analysis, TLC was identified as the most viable method. The advantages of TLC include speed of sample preparation, ease of analysis, and applicability to various types of dyes. (The Lab is in contact with George Boggman, who previously worked for the EPA in the Athens, GA Laboratory. Mr. Boggman is believed to be the expert in dye analyses.)

The Lab indicated that the method development work done to date (for TLC and HPLC/PDA) has concentrated on organic soluble dyes only. The Lab began with the nine organic soluble dyes for which they currently had standards and were able to see separation of all nine compounds. The Lab indicated that they had begun with the organic soluble dyes since they were harder to work with than the water soluble dyes. Based on previous experience, the Lab did not anticipate problems with the water soluble dyes.

Allen indicated that the EPA, in general, viewed TLC as a viable method and approved of the use of screening methods. Allen indicated that a second methodology might be needed in the TLC procedure did not work of all the dyes. Terrie stated that a quantitative analytical method was also being developed for dyes.

3. *HPLC/PDA Method Development* ▪ Forrest gave a summary of progress on HPLC method development. Forrest indicated that the Lab had found a literature reference to a method capable of separating 120 dyes. Forrest tried the method and was able to get good separation of a variety of dyes. Forrest was also able to identify the manufacturer of the referenced analytical column and placed an order for the same column. The Lab was hopeful that it would be possible to use one analytical method for all dyes, rather than the multiple methods, which were originally thought to be needed.

4. *Sensitivity of Analytical Methods & Toxicological Support from EPA* ▪ Allen questioned the sensitivity of the analytical methods being developed. The Crane Lab responded that they did not have preliminary detection limits as they were still optimizing the solvent systems and waking for the arrival of staining compounds. The Lab indicated that it would be approximately two months before they had preliminary detection limits.

Karen indicated that a risk-based screening level for dyes was needed in order to evaluate the sensitivity of the analytical methods. Terrie emphasized that the required reporting levels were an integral part of optimizing the proposed analytical method. TtNUS indicated that they have been gathering information on the toxicity, chemical structure, and physical characteristics of dyes and have summarized that information in Attachment 4 of the conference call agenda. Karen stated that a limited number of published RfDs and CSFs are available for dyes and suggested that the EPA toxicologist be brought into the process so that the EPA could develop toxicity criteria (and risk-based screening levels) for the dyes. Karen reminded the attendees that during the pre-pre-QAPP meeting held for the site (on May 13, 1999). the EPA had agreed to develop toxicity criteria for the dyes. Bill indicated that during the pre-pre-QAPP meeting George Bollweg was identified as the EPA lead for developing toxicity criteria for this project. Peter stated that he would discuss the issue with George and provide him with the conference call agenda (and attachments) and meeting minutes.

5. *Focusing the Analytical Development* on Known *Dyes/Dealing with* Unknown *Dyes* ▪ Forrest emphasized that method development would proceed using the dyes for which standards had been obtained (see Attachment 3 of the conference call agenda). Terrie indicated that the original dye target compound list had contained compounds listed in the RFI, dyes present in the Crane Lab leftover from previous dye operations at the Base, and dyes found in soil samples collected from SWMU 2. Standards were obtained for many of these dyes. However, some dyes could not be identified and therefore are considered to be “unknowns”.

The Lab has attempted to identify these 'unknowns" using techniques such as Nuclear Magnetic Resonance (NMR) and Gas Chromatography/Mass Spectroscopy (GC/MS). However, the Lab has bean unable to identify these dyes. Karen indicated that due to the number and complexity of the dyes, unknowns are likely to exist even after quantitation by HPLC and that an approach will have to be developed to handle these compounds in a manner similar to TICs (for GC/MS methods). Allen agreed but also indicated that any additional dye standards found during the method development phase should be incorporated into the analytical methods. The Lab indicated that they would do this.

6. *Support Documentation & QA/QC* - Allen asked about Standard Operating Procedures (SOPs) and the level of support documentation, which would be available. The Lab indicated that SOPs would be generated and adequate support documentation would be available to aid in the review of the methods. Terrie indicated that the support documentation was being compiled as the method development proceeds.

   Allen questioned the level of QA/QC to be performed (i.e., evaluation of false positives, etc.). Linda and Terrie indicated that the Lab was going to be conducting QA/QC procedures that are typically performed for all SW-646 methods. Attachment 1 of the conference call agenda provides the "Abbreviated Activity Timeline" for method development; a summary of the QA/QC procedures to be performed are presented in the timeline.

7. *Anthracene* as a *Surrogate* - Forrest proposed the use of anthracene as a surrogate spike compound for the HPLC analysis. Anthracene is a precursor for many dyes and can be reasonably expected to give an indication of extraction efficiency and method performance. Terrie indicated that anthracene had not been detected in the environmental samples previously collected at the site. Allen suggested using an isotopically labeled form of this compound. The Lab indicated that was an excellent idea and would incorporate this into the method development.

6. *Revisions to Timeline* – Forrest indicated that the "Abbreviated Activity Timeline" for the method development (Attachment 1 of the conference call) would be updated to specify activities that apply to the TLC and/or HPLC/PDA methods. The revised timeline would be submitted to all attendees, when available.

9. *EPA* Concurrence -The EPA indicated that method development efforts were proceeding in the right direction and wished the Lab luck in their continued efforts.

PROPOSED AGENDA

PRE-QAPP MEETING
SWMU 2 – DYE BURIAL GROUND
NSWC CRANE, INDIANA

INTRODUCTION: RCRA Facility Investigation (RFI), risk assessment, and Corrective Measures Study (CMS) activities need to be completed for Solid Waste Management Unit (SWMU) 2, the Dye Burial Ground at Naval Surface Warfare Center (NSWC) Crane, Indiana. Although several phases of RFIs have been conducted at the site, the nature and extent of contamination has not been defined. One field sampling effort is planned to address the needs of the RFI, risk assessment, and CMS. Tetra Tech NUS, Inc. (TtNUS) is currently preparing the Internal Draft Planning Documents for SWMU 2. The NSWC Crane Explosives Science Laboratory is currently developing analytical methods for dye constituents.

A pre-Quality Assurance Project Plan (pre-QAPP) meeting is scheduled to be held for SWMU 2 on March 9, at 9 A.M. Central Time (CT), 10 A.M. Eastern Standard Time (EST). The phone number for the conference call is l-800-860-2442, ask for the TtNUS. Karen Smecker conference call. Representatives from SOUTHDIV, NSWC Crane, EPA, IDEM, and TtNUS are expected to attend the pre-QAPP meeting. The presence of EPA risk assessors (human health and ecological) and QAPP personnel are requested at the meeting.

Because of inherent complexities associated with SWMU 2, two meetings with EPA were held previously to discuss significant issues such as project objectives, dye toxicity data, and dye analytical method development. A pre-pre-QAPP meeting was held on May 13. 1999 and a meeting to specifically discuss the dye method development was held on September 10, 1999. To facilitate pre-QAPP meeting discussions, a copy of the meeting minutes from these meetings is provided as Attachment A.

PROPOSED AGENDA: The following agenda is proposed for the pre-QAPP meeting:

TIME (A. M. C. T.)	*AGENDA ITEM*	*DESIRED OUTCOME*
9:00 – 9:15	Background	Provide a brief overview of the site and historical work performed.
9:15 – 9:45	Overall Project Objectives	Review of project objectives, the conceptual site model, and the proposed sampling plan. Obtain regulatory concurrence on objectives.
9:45 – 10:45	Analytical Program	Review work completed on analytical method development for dyes. Summarize proposed analytical methods for other chemical analyses. Obtain regulatory concurrence on proposed analytical methods.
10:45 – 11:15	Toxicity Data for Dyes and Risk Assessment	Discuss toxicity data for dyes and risk assessment approach. Confirm that EPA will develop toxicity criteria for dyes.
11:15 – 11:30	Current Schedule	Review project schedule.

This agenda is considered tentative and is provided so that there is a focus to the pre-QAPP meeting. To facilitate discussions at pre-QAPP meeting, a summary of information to be discussed during the meeting is provided in the remainder of this memorandum.

BACKGROUND: The background of SWMU 2 – Dye Burial Ground was discussed during the pre-pre-QAPP meeting. Refer to Attachment A for the minutes from this meeting.

OVERALL PROJECT OBJECTIVES: The ultimate objective of the field sampling program for this project is to gather sufficient to complete RFI risk assessment, and CMS activities. The risk assessment is conducted to determine whether hazardous constituents released into the environment by past SWMU operations pose an "unacceptable" or 'acceptable' risk to potential human and/or ecological receptors. Figure B-l provided in Attachment B presents a decision rule flowchart, which is designed to meet the project objectives.

Conceptual Site Model

To initiate development of the site sampling plan, a conceptual site model (CSM) was developed for the site. The CSM is presented as Figure B-2 in Attachment B.

Proposed Samolina Plan

Based on the CSM for SWMU 2, the proposed sampling plan summarized in Table B-l in Attachment B was developed.

ANALYTICAL PROGRAM: Tables B-2 (dyes) and B-3 (other parameters) presented in Attachment B provide the list of the analytical methods and parameters to be included in the analytical program for SWMU 2. TtNUS has worked closely with the subcontracted analytical laboratory to select and optimize analytical methods in an effort to attain, to the greatest extent possible, laboratory reporting limits (RLs) at concentrations less than or equal to the risk-based target levels.

Dve Analysis

The NSWC Crane Lab is developing analytical methods for dye analysis. A meeting was held on September 10, 1999 with USEPA Region V to discuss the approach and progress of the method development. The two methods being developed are thin-layer chromatography (TLC) and high performance liquid chromatography (HPLC). TLC is the proposed screening method', which is a semi-quantitative method. HPLC is the quantitative analytical method.

Preliminary standard operating procedures (SOPs) for the TLC and HPLC methods are provided in Attachment C. Preliminary instrument detection limits and method detection limits for aqueous and solid matrices for the dye analytical methods are summarized in Table D-2. Preliminary risk-based target levels for dyes are also included in Table D-2. These values were calculated by TtNUS using available toxicity data compiled as a result of a literature search. When available, toxicity data was used to calculated a reference dose using methodology presented in risk assessment guidance. Target levels were then calculated using EPA Region IX methodology for determining preliminary remediation goals (PRGs).

Other Analysis

Inorganics will be analyzed via SW-646 Method 6020 (Inductively Coupled Plasma/Mass Spectrometry [ICP/MS]) in place of SW646 Method 6010B (ICP/Atomic Emission Spectroscopy) for the Appendix IX metals to achieve lower RLs. It should be noted for the site that there is some uncertainty in linking the presence of metals to ground water activities. During previous site investigations, anomalously tow pH readings from reported for ground water samples collected from several existing monitoring wells. Samples collected from these wells were found to contain elevated concentrations of inorganics. Low pH sandstone is probably a natural phenomenon and may be responsible for the high concentrations of metals. Additionally, the distribution of metals within the aquifers is not necessarily coincident with the proximity of the wells to the dye burial trenches.

As shown on Table B-3. the RLs for some metals/cyanide are greater than the screening criteria. The IDLs for these inorganics, however, are less than the screening criteria. As noted above, the presence of inorganics as a result of past operational activities is questionable.

TOXICITY DATA FOR DYES AND RISK ASSESSMENT: Risk assessments (human health and ecological) will be conducted to evaluate the incremental risks associated with the site under current and future site conditions. A screening level ecological risk assessment will be performed. In the pre-pre-QAPP meeting, it was acknowledged that there are limited toxicity criteria for dyes. As noted during the meeting, the EPA Office of Research and Development typically develops toxicity criteria when none are published. George Bolfweg at EPA Region V was identified as the lead for development of toxicity factors for dyes.

As mentioned previously, TtNUS on behalf of the Navy and NSWC Crane has done a thorough Internet search for available toxicity information for the dyes applicable to SWMU 2. Table D-2 provided in

Attachment D contains a summary of chemical information for the dyes to be analyzed. Table D-2 presents a summary of the Internet sites containing toxicity information for the dyes. Information on toxicity and the calculation of preliminary target levels for dyes can be provided to the regulatory agencies, at their request. Because of the amount of information is extensive, this information was not attached to this memorandum.

SCHEDULE: The current project schedule is provided in Attachment E.

CONFERENCE CALL MINUTES

SOUTHDIV CLEAN – NSWC Crane
September 10, 1999; 10am (Pittsburgh, PA/Charleston, SC) and 9am (Crane, IN)
Recorded by TtNUS

Subject: NSWC Crane Lab Method Development for Dyes at SWMU 2 - Dye Burial Grounds

Attendees: EPA Region 5 - Peter Ramanauskas and Allen Debus; SOUTHDIV - Bill Gates; NSWC Crane (environmental division) - Tom Brent; NSWC Crane Lab - Forrest Hawman, Sarah Poehlein, Nancey Maegerlein; TtNUS - Karen Smecker, Daneen Resnick, Linda Karsonovich, David Stair; and ECT.CON, Inc (TtNUS subcontractor) – Terrie Baranek

Background: RFI, CMS, and risk assessment activities are to be completed at SWMU 2. TtNUS is currently preparing planning documents for these activities, which includes environmental sampling. The NSWC Crane Laboratory is developing analytical methods for dyes. Two possible analytical methodologies have been identified for use in sample analysis – Thin Layer Chromatography (TLC) and High Performance Liquid Chromatography/Photo Diode Array (HPLC/PDA). The purpose of today's conference call was to apprise the EPA of the analytical methodologies, to obtain preliminary approval of the use of the analytical methods, and to receive input regarding the path forward for method development.

Summary Of Conference Call: Based on research performed by the Crane Lab, TLC appears to be the best method for the field screening (screening-level) analysts and HPLC/PDA appears to be the best method for the quantitative analysis. The method development work will continue to concentrate on the dyes suspected to be present at the site and for which known standards can be purchased. Dyes that are considered to be 'unknowns" will be evaluated using a similar approach to Tentatively Identified Compound (TIC) analyses. The quantitative analytical methods (for HPLC/PDA) will be developed using anthracene as a surrogate spiking compound. EPA's toxicological support will be brought into the process so that they can develop toxicity criteria (Reference Doses/Cancer Slope Factors [RfDs/CSFs]) for dyes.

The following items summarize the conversation that took place between the attendees:

1. *Site History* - Terrie summarized the history of the site and emphasized that dye analyses were not conducted during historical site investigations. Tom clarified that initially the site was thought to consist of 3 trenches where dyes were disposed, however, during historical site investigations additional areas (trenches, fingers of trenches, etc.) of disposal were discovered.

   Terrie stated that soil, groundwater. surface water, and sediment samples are to be collected at the site to complete RFI, CMS, and risk assessment activities. Groundwater. surface water, and sediment samples will be collected to evaluate the migration of dyes from the site. Soil sampling will be limited to areas surrounding the RCRA cap, which were previously excavated using visual observations (i.e., the additional areas of disposal discussed in the pievious paragraph). Soil from these areas were placed under the RCRA cap prior to its construction. However, no confination sampling was conducted to determine that the are was "clean". The objective of the soil sampling will be to confirm that areas previously excavated are in fact free of dye contamination.

   Terrie indicated that two types of analytical methods for dye analysis were being developed by the Crane Laboratory --- one for use as a screening method. TLC, and one for use as a quantitative analysis, HPLC/PDA.

2. *TLC Method Development* - Sarah and Forrest summarized their work on the screening method. Originally HPLC had been proposed as the screening method. However, based on an extensive

literature search and contact with technical experts in the field of dye analysis, TLC was identified as the most viable method. The advantages of TLC include speed of sample preparation, ease of analysis, and applicability to various types of dyes. (The Lab is in contact with George Boggman. who previously worked for the EPA in the Athens, GA Laboratory. Mr. Boggman is believed to be the expert in dye analyses.)

The Lab indicated that the method development work done to date (for TLC and HPLC/PDA) has concentrated on organic soluble dyes only. The Lab began with the nine organic soluble dyes for which they currently had standards and were able to see separation of all nine compounds. The Lab indicated that they had begun with the organic soluble dyes since they were harder to work with than the water soluble dyes. Based on previous experience, the Lab did not anticipate problems wfth *the* water soluble dyes.

Allen indicated that the EPA, in general, viewed TLC as a viable method and approved of the use of screening methods. Allen indicated that a second methodology might be needed in the TLC procedure did not work of all the dyes. Terrie stated that a quantitative analytical method was also being developed for dyes.

3. *HPLC/PDA Method Development* - Forrest gave a summary of progress on HPLC method development. Forrest indicated that the Lab had found a literature reference to a method capable of separating 120 dyes. Forrest tried the method and was able to get good separation of a variety of dyes. Forrest was also able to identify the manufacturer of the referenced analytical column and placed an order for the same column. The Lab was hopeful that it would be possible to use one analytical method for all dyes, rather than the multiple methods, which were originally thought to be needed.

4. *Sensitivity of Analytical Methods & Toxicological Support* from *EPA* - Allen questioned the sensitivity of the analytical methods being developed. The Crane Lab responded that they did not have preliminary detection limits as they were still optimizing the solvent systems and waiting for the arrival of staining compounds. The Lab indicated that it would be approximately two months before they had preliminary detection limits.

   Karen indicated that a risk-based screening level for dyes was needed in order to evaluate the sensitivity of the analytical methods. Terrie emphasized that the required reporting levels were an integral part of optimizing the proposed analytical method. TtNUS indicated that they have been gathering information on the toxicity, chemical structure, and physical characteristics of dyes and have summarized that information in Attachment 4 of the conference call agenda. Karen stated that a limited number of published RfDs and CSFs are available for dyes and suggested that the EPA toxicologist be brought into the process so that the EPA could develop toxicity criteria (and risk-based screening levels) for the dyes. Karen reminded the attendees that during the pre-pre-QAPP meeting held for the site (on May 13, 1999). the EPA had agreed to develop toxicity criteria for the dyes. Bill indicated that during the pre-pre-QAPP meeting George Bollweg was identified as the EPA lead for developing toxicity criteria for this project. Peter stated that he would discuss the issue with George and provide him with the conference call agenda (and attachments) and meeting minutes.

5. *Focusing the Analytical Development on Known Dyes/Dealing with Unknown Dyes* - Forrest emphasized that method development would proceed using the dyes for which standards had been obtained (see Attachment 3 of the conference call agenda). Terrie indicated that the original dye target compound list had contained compounds listed in the RFI, dyes present in the Crane Lab leftover from previous dye operations at the Base, and dyes found in soil samples collected from SWMU 2. Standards *were* obtained for many of these dyes. However, some dyes could not be identified and therefore are considered to be “unknowns”.

   The Lab has attempted to identify these “unknowns” using techniques such as Nuclear Magnetic Resonance (NMR) and Gas Chromatography/Mass Spectroscopy (GC/MS). However, the Lab has been unable to identify these dyes. Karen indicated that due to the number and complexity of the

dyes, unknowns are likely to exist even after quantitation by HPLC and that an approach will have to be developed to handle these compounds in a manner similar to TICs (for GC/MS methods). Allen agreed but also indicated that any additional dye standards found during the method development phase should be incorporated into the analytical methods. The Lab indicated that they would do this.

6. ***Support*** Documentation ***& QA/QC*** - Allen asked about Standard Operating Procedures (SOPs) and the level of support documentation, which would be available. The Lab indicated that SOPs would be generated and adequate support documentation would be available to aid in the review of the methods. Terrie indicated that the support documentation was being compiled as the method development proceeds.

   Allen questioned the level of QA/QC to be performed (i.e., evaluation of false positives, etc.). Linda and Terrie indicated that the Lab was going to be conducting QA/QC procedures that are typically performed for all SW-646 methods. Attachment 1 of the conference call agenda provides the "Abbreviated Activity Timeline" for method development; a summary of the QA/QC procedures to be performed are presented in the timeline.

7. ***Anthracene as a Surrogate*** - Forrest proposed the use of anthracene as a surrogate spike compound for the HPLC analysis. Anthracene is a precursor for many dyes and can be reasonably expected to give an indication of extraction efficiency and method performance. Terrie indicated that anthracene had not been detected in the environmental samples previously collected at the site. Allen suggested using an isotopically labeled form of this compound. The Lab indicated that was an excellent idea and would incorporate this into the method development.

8. ***Revisions to Timeline*** -- Forrest indicated that the "Abbreviated Activity Timeline' for the method development (Attachment 1 of the conference call) would be updated to specify activities that apply to the TLC and/or HPLC/PDA methods. The revised timeline would be submitted to all attendees, when available.

9. ***EPA Concurrence*** -The EPA indicated that method development efforts were proceeding in the right direction and wished the Lab luck in their continued efforts.

TABLE B-I

SUMMARY OF PROPOSED SAMPLING PLAN
SWMU 2 -DYE BURIAL GROUND
NSWC CRANE, INDIANA

Sample Matrix	Objective	Sample Locations	Analysis	Evaluations
Surface and Subsurface Soil	Confirm presence or absence of residual contamination et previously excavated areas located outside the limits of me cap.	A minimum of 33 locations will be sampled using a phased approach[1]. Number of locations to be sampled will depend on whether dyes are detected. Samples will be collected at previously excavated areas identified using historical report information (site maps, geophysical survey data, etc.)[2].	Qualitative dye screening analysis (using TLC method) will be performed. Quantitative dye analysis (using HPLC method) will be conducted on select samples. TAL inorganics will also be analyzed for these select samples. 30% of samples to be analyzed for miscellaneous parameters to fulfill CMS needs[3].	Evaluate nature and extent of residual contamination at vertical and horizontal soil horizons for previously excavated areas. Evaluate incremental risks[4]) associated with residual contamination. Evaluate interim action and additional remedial options.
Ground water	Confirm presence or absence of contamination for migration pathways (infiltration/percolation).	First round of samples (8 samples) will be collected from existing wells located near known waste disposal areas (i.e.. trenches. previously excavated areas. etc.) plus an upgradient location. If dyes are detected or if inorganic concentrations exceed upgradient concentrations, a second round of samples will be collected from 17 additional wells currently located at the Site. An additional well cluster may be installed in the Pennsylvania aquifer northeast of the site, it ground water is found to Row In this direction.	Quantitative dye analysis (using HPLC method) will be conducted. TAL inorganics, natural attenuation parameters. and other miscellaneous water quality parameters will also be analyzed.	Evaluate nature and extent of contamination. Evaluate incremental risks[4] associated with contamination.. Evaluate *interim action and additional* remedial options.
Surface Water	Confirm presence or absence of contamination for migration pathways (surface water runoff, ground water discharge. etc.).	samples will be collected from drainageways, tributaries of Little Sulphur Creek. and seeps/springs located near the site. which may be impacted by sit* activities. Approximately 10 locations will be sampled[5].	Quantitative dye analysis ("sing HPLC method) will be conducted. TAL inorganics will also be analyzed. 30% of samples to be analyzed for miscellaneous parameters to fulfill CMS needs[3].	Evaluate nature and extent of contamination. Evaluate incremental risks[4] associated with contamination. Evaluate interim action and additional remedial options.
Sediment	Confirm presence or absence of contamination for migration pathways (surface water runoff, ground water discharge. etc.).	Samples will be collected from drainageways, tributaries of Little Sulphur Creek. and seeps/springs located near the site, which may be impacted by site activities. Approximately 10 locations will be sampled[5].	Quantitative dye analysis (using HPLC method) will be conducted. TAL inorganics will also be analyzed. 30% of samples to be analyzed for miscellaneous parameters to fulfill CMS needs[3].	Evaluate nature and extent of contamination. Evaluate incremental risks" associated with contamination. Evaluate interim action and additional remedial canons.

CMS Corrective Measure Study HPLC High Performance Liquid Chromatography TAL Target Analyte List TLC Thin Layer Chromatography

1 Although a phased approach is proposed, one field event is planned. Re-mobilization to the site will not be required because of the relatively quick turn-around time anticipated for qualitative dye screening analysis (TLC method).

2 Locations to be confirmed during the field investigation by observing the prevailing geologic conditions (i.e., identifying the presence of natural soil horizons [fragipan] in contrast to soil used to backfill the excavation).

02/28/00
for Pre-QAPP Meeting
TtNUS, Inc.

TABLE B-I

SUMMARY OF PROPOSED SAMPLING PLAN
SWMU 2 -DYE BURIAL GROUND
NSWC CRANE, INDIANA

3 Soil samples for miscellaneous parameters will be randomly assigned during the field event.
4 Human health and ecological incremental risks will be evaluated.
5 Exact number of samples to be collected will be determined during a site visit (field survey). Surface water and sediment samples will be collocated.

TABLE B-2

ANALYTICAL METHODS AND DETECTION LIMITS
SWMU 2 - DYE BURIAL GROUND
NSWC CRANE, INDIANA

Dye Name	TLC Method - High Level Analysis			HPLC Method - Low-level Analysis				Preliminary Risk-Based Target Levels - Human Health[2]	
	IDL (ug/L)	MDL Aqueous Sample Matrix (ug/L)	MDL Solid Sample Matrix (ug/kg)	Analytical Wavelength (nm)	IDL (ug/L)	MDL Aqueous Sample Matrix[1] (ug/L)	MDL Solid Sample Matrix[1] (ug/kg)	Aqueous Sample Matrix (ug/L)	Solid Sample Matrix (ug/kg)
Water Soluble Dyes									
Acid Blue 1	663	663	TBD[3]	635	55	19	1,381	9,000	15,000,000
Acid Blue 9	3,200	3,200	TBD	628	54	18	1,343	230,000	230,000,000
Acid Blue 45	1,800	1,800	TBD	633	30	10	743	---[4]	---
Acid Red 64	4,500	4,500	TBD	524	75	25	1,868	---	---
Acid Yellow 3	4,600	4,600	TBD	383	77	26	1,920	---	---
Acid Yellow 23	3,600	3,600	TBD	404	61	20	1,513	---	---
Acid Yellow 73	2,700	2,700	TBD	493	45	15	1,113	---	---
Acid Orange 10	2,100	2,100	TBD	485	35	12	871	300	500,000
Basic Violet 10	300	300	TBD	549	50	17	1,253	600	1,000,000
Basic Yellow 2	2,100	2,100	TBD	378	36	12	894	180	300,000
Organic Soluble Dyes									
1-Aminoanthraquinone	3,500	3,500	TBD	481	59	20	1,477	---	---
2-Aminoanthraquinone	3,100	3,100	TBD	299	52	17	1,295	2[5]	14700[5]
Disperse Blue 14	2,500	2,500	TBD	638	42	14	1,062	---	---
Disperse Red 9	1,900	1,900	TBD	513	32	11	796	---	---
Disperse Violet 1	238	238	TBD	588	40	1	991	---	---
Pigment Red 1	8,800	8,800	TBD	495	147	49	3,667	---	---
Smoke Dye	224	224	TBD	547	37	13	935	---	---
Solvent Green 3	414	414	TBD	635	35	12	863	---	---
Solvent Orange 3	232	232	TBD	408	39	13	968	12,000	20,000,000
Solvent Orange 7	2,400	2,400	TBD	499	41	14	1,017	750	1,200,000
Solvent Red 1	381	381	TBD	504	32	11	794	---	---
Solvent Red 24	768	768	TBD	519	13	4	320	---	---
Solvent Yellow 2	2,200	2,200	TBD	416	37	12	917	180	310,000
Solvent Yellow 3	2,000	2,000	TBD	385	33	11	823	---	---
Solvent Yellow 14	199	199	TBD	486	33	11	831	460	760,000
Solvent Yellow 33	2,600	2,600	TBD	399	43	14	1,065	---	---
Internal Standards and Surrogate Spikes									
Anthracene	NA[6]	NA	NA	251	35	12	881	1,800	22,000
Benzanthrone	NA	NA	NA	399	43	14	1,073	30	50,000

1 Theoretical MDLs based on a single extraction. An MDL study will be performed.
2 Values calculated using USEPA Region IX Preliminary Remediation Goal (PRG) methodology and a calculated reference doses (RfD).
3 To be determined.
4 A value was not calculated because of the lack of adequate toxicity information.
5 Value calculated using an available cancer slope factor (CSF) (Cal-EPA).
6 Not applicable.

TABLE B-3

ANALYTICAL METHODS AND LIMITS OF DETECTION
SWMU 2 - DYE BURIAL GROUND
NSWC CRANE, INDIANA

Chemical	Aqueous Matrix			Solid Matrix			
	Laboratory MDL/IDL[1] (ug/L)	Laboratory RL[1] (ug/L)	Lowest Risk-Based Target Level[2] (ug/L)	Laboratory MDL/IDL[1] (mg/kg)	Laboratory RL[1] (mg/kg)	Lowest Risk-Based Soil Target Level[2] (mg/kg)	Lowest Risk-Based Sediment Target Level[2] (mg/kg)
APPENDIX 9 METALS (SW-846 METHOD 6020 ICP/MS, UNLESS OTHERWISE NOTED)							
Antimony	0.05	1.0	6	0.025	1*	0.1423	--
Arsenic	0.23	1.0*	0.045	0.1	1*	0.38	0.59
Barium	0.05	1.0	2000	0.022	1	1.04	--
Beryllium	0.09	1.0*	0.016	0.04	1*	0.1	--
Cadmium	0.09	1.0*	0.66	0.039	1*	0.18095	0.596
Chromium (total)	0.06	5.0	11	0.03	1*	0.4	26
Cobalt	0.04	3.0	5	0.018	1*	0.14033	50
Copper	0.1	2.0	5	0.045	1*	0.3132	16
Lead	0.05	1.0	1.3	0.021	1*	0.45053	31
Mercury (SW-846 Method 7470A/7471A)	0.14	0.2*[3]	0.0013	0.07	0.2*	0.0079	0.174
Nickel	0.13	10	29	0.06	1	7	16
Selenium	0.4	1.0	5	0.18	1*	0.02765	--
Silver	0.01	3.0*	1	0.004	1*	2	0.0005
Thallium	0.03	1.0*	0.56	0.015	1*	0.04	--
Tin	0.076	10	73	0.034	5	7.62	--
Vanadium	0.06	2	19	0.027	1	1.59	--
Zinc	1.2	10	58.9	0.53	1[3]	6.62	120
MISCELLANEOUS METALS (SW-846 METHOD 6010B)[4]							
Aluminum	76	200	--	6.8	20	--	--
Calcium	26	5000	--	2.3	500	--	--
Iron	7.7	100	--	0.69	10	--	--
Magnesium	37	5000	--	3.3	500	--	--
Manganese	1.7	15	--	0.15	1.5	--	--
Potassium	136	5000	--	12	500	--	--
Sodium	29	5000	--	2.6	500	--	--
MISCELLANEOUS PARAMETERS (UNITS AS IDENTIFIED)							
Cyanide (SW-846 Method 9012A) (ppm)	2.4	10*	5.2	0.2	1*	1.33	0.0001
Nitrite/Nitrate (U.S. EPA 353.2) (ug/L)	2	10	10000 (nitrate)	ANR	ANR[5]	--	--
Sulfate (SW-846 Method 9056) (ug/L)	74.2	1000	500000	ANR	ANR	--	--
Chloride (SW-846 Method 9056) (ug/L)	80	1000	250000	ANR	ANR	--	--
Hardness (Standard Method 2340B) (mg/L)	NA[6]	1	--	ANR	ANR	--	--
Total Suspended Solids (U.S. EPA 160.1) (mg/L)	800	1000*[4]	--	ANR	ANR	--	--
Total Organic Carbon (TOC) (SW 846 9060) (ppm)	306	1000	--	306	1000	--	--
pH (SW-846 9045C) (S.U.)	ANR	ANR	6.5 - 9	NA	1	--	--
CEC (SW-846 9081) (mS/cm)	ANR	ANR	--	NA	1	--	--

ug/L micrograms per liter; mg/kg-milligrams per kilogram

* Asterisks indicate those chemicals for which the laboratory RL exceeds the risk-based target level for the project.

1 Method detection limits (MDLs) (all parameters except metals), instrument detection limits (IDLs) (metals only), and reporting limits (RLs) as provided by Laucks Testing Laboratories, Inc. and NSWC Crane Lab (dyes only). These values may change prior to the SWMU investigations as laboratory MDLs and IDLs are updated.

2 Value is based on the lowest human health or ecological risk-based criterion as presented in Appendix D of the Work Plan.

3 Laucks Testing Laboratories is confident that it can reliably report to this PQL, even though this value is less than two times the MDL.

4 These are not Appendix 9 metals. They are being analyzed for general ground water quality information. They are not to be included in the screening process; therefore, no criteria are listed.

5 Analysis Not Required

6 Not applicable.

NSWC CRANE LABORATORY
Crane, Indiana

SOP # XxXxX

Screening of Organic Colorants via Thin Layer Chromatography

Revision: 0

Revised by: _____ Date: _____

Approved by: _____ Date: _____
XXX QA Officer

Approved by: _____ Date: _____
XXX Laboratory Director

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1.0 SCOPE AND APPLICABILITY

This method is intended for the screening of dyes in the aqueous-soluble and organic-soluble family by thin layer chromatography (TLC) using a TLC autospotter and scanning software. This method is used to determine the presence of the following dyes in aqueous, soil or sediment matrices:

List of Organic Colorants

Aqueous Soluble			Organic Soluble		
Compound Name	Abbr.	CAS #	Compound Name	Abbr.	CAS #
Acid Blue 1	AB1	129-17-9	Disperse Blue 14	DB14	2475-44-7
Acid Blue 9	AB9	3844-45-9	Solvent Yellow 2	SY2	60-11-7
Acid Blue 45	AB45	2861-02-1	Solvent Yellow 3	SY3	97-56-3
Acid Yellow 3	AY3	8004-92-0	Solvent Yellow 14	SY14	842-07-9
Acid Yellow 23	AY23	1934-21-0	Solvent Yellow 33	SY33	8003-22-3
Acid Yellow 73	AY73	518-47-8	Disperse Violet 1	DV1	128-95-0
Basic Yellow 2	BY2	2465-27-2	Pigment Red 1	PR1	6410-10-2
Basic Violet 10	BV10	81-88-9	Solvent Red 1	SR1	1229-55-6
Acid Red 64	AR64	NA	Disperse Red 9	DR9	82-38-2
			Solvent Red 24	SR24	85-83-6
			Solvent Green 3	SG3	128-80-3
			Solvent Orange 3	SO3	495-54-5
			Solvent Orange 7	SO7	3118-97-6
			Smoke Dye	SD	81-64-1
			1-Aminoanthraquinone	1-AQ	82-45-1
			2-Aminoanthraquinone	2-AQ	117-79-3

1.1 This method provides a XXX extraction procedure for low concentration of dyes in surface or ground water. Direct injection of diluted and filtered aqueous samples can be used for samples of higher concentration. (See Table XXX -Estimated Quantitation Limits)

1.2 These dyes may be dangerous. When handling samples and stock solutions, follow safety precautions listed in Section 5.0.

1.3 The low and high estimated quantitation limits for solid and aqueous matrices are listed in Table XXX.

2.0 SUMMARY OF THE METHOD

Thin-layer chromatography is a separation method based on adsorption of the sample to the stationary phase. The adsorbent layer (stationary phase) is usually

formed on a 20x20-cm glass plate and consists of silica gel (polar material). The sample mixture is applied to the lower edge of the plate. The plate is then placed in a tank with a suitable developing solvent(s). The individual substances from the sample mixture are transported upwards at differing rates. The separation is effected by adsorption, partition, or ion-exchange processes. When the solvent has traveled sufficiently up the silica plate. the plate is removed and dried. The separated substances are detected visually, W lamp, or computer enhanced. The substances are identified by the ratio: rate of migration from the starting point to the distance the solvent traveled from the start. This value is called the R_F value.

$$R_F = \frac{\text{distance of substance from start}}{\text{distance of solvent from start}}$$

The key step in performing TLC analysis is finding the suitable solvent for the separation. The solvent make-up depends on the type of TLC plate but more importantly the polarity of your analyte. The solvent attempts to break up the interaction between the analyte and the TLC plate. In most cases, the stationary phase is made of silica gel. The silica gel comprises of silanol groups (Si-OH) that interact with the analyte. Therefore, the more polar an analyte is the more it will interact with the silica gel. This strong interaction can only be broken up with a more polar solvent. Basically, the better the affinity for the solvent the more the analyte moves up with the solvent. If the analyte has no preference for the solvent. then the analyte will not move up on the TLC plate.

2.1 This method provides TLC conditions for the detection of ppm levels of dyes in the aqueous-soluble and organic-soluble family in aqueous and solid matrices. Prior to using this method, appropriate sample preparation must be used.

2.2 Low-level Aqueous Direct Screening Method: TO BE ADDED

2.3 High-level Aqueous Direct Screening Method:

2.3.1 Turn on the TLC autospotter and confirm the settings arc correct. A preconcentrated silica gel plate is taken out of the dessicator and placed on the TLC autospotter. This is done first to thoroughly heat the plate.

2.3.2 Approximately 3 ml of the aqueous sample is filtered through a Whatman Anatop filter (0.2 µm) into an amber vial. The aqueous sample is drawn up into a 100 µl TLC autospotter syringe. Care is taken into drawing up the sample, making sure that there are no air bubbles in the syringe barrel.

2.3.3 Carefully, place the syringe in its holder and place the bar over the syringe. Be sure the tip of the needle is just above the silica gel plate. The closer the tip is to the plate, the smaller the sample spot.

2.3.4 Start the TLC autospotter when ready.

2.3.5 After the 100 µl sample has been spotted onto the plate, remove it from the autospotter and place the plate into the tank prepared in accordance with Section 11.2.

2.3.6 Remove the plate from the tank after the solvent front has moved approximately 10 cm.

2.3.7 Immediately mark the solvent front with a pencil. Allow the plate to dry thoroughly.

2.3.8 Once the plate has dried, place the plate on an Acer flatbed scanner.

2.3.9 Using PhotoSuite II scanning software, Scan the plate by selecting Get Photo and choosing source as Scanner. Set the following scan parameters:

Original: Reflective
Image Type: Color
Scan Mode: High Quality
Resolution: 200
Scaling: 100%
Filter: No Filter
Descreen: No Descreen
Color Wizard: None

2.3.10 Once the image has been scanned, select Touch Up & Transform followed by Touch Up. From the list, select Auto Enhance. You must Apply this to the image.

2.3.11 Save the image and then print it. Be sure the printer is set for high quality photo paper.

2.3.12 Once the image has been printed, use a ruler to measure the solvent front and then measure the spot height. Obtain an Rf value by taking the ratio of the spot height and the solvent front.

2.4 Soil and Sediment Sample Extraction: TO BE ADDED

3.0 DEFINITIONS

This section defines the terms and acronyms as used in this SOP.

TLC	Thin Layer Chromatography
Rf	Relative to the solvent front. This value is the migration distance of the solute relative to the migration of the solvent.
MP	Mobile phase
µl	microliter ($1x10^{-6}$ L)
W	Ultraviolet

4.0 HEALTH AND SAFETY WARNINGS

4.1 Standard laboratory protective clothing, gloves. and eye covering is required during the execution of this method.

5.0 CAUTIONS

5.1 Reagent working standards must be prepared fresh on the day of analysis.

5.2 Toxicity of constituents-TO BE ADDED

5.3 Waste Disposal

5.3.1 All spent organic solvents are stored in glass bottles and labeled as "TLC Spent Solvents", along with the appropriate solvent names.

5.3.2 When the bottles are tilled to capacity, the spent solvents are handled and disposed of in accordance with the Crane Division Hazardous Waste Management Plan, NAVSURPWARCENDIV INSTRUCTION 5090.2B.

5.4 Routine and Preventative Maintenance

Table 2: PREVENTIVE MAINTENANCE FOR TLC ANALYSIS

Instrument	Preventive Maintenance	Maintenance Frequency
TLC Tank	Clean and Wipe Dry	After each trial run
TLC lid	Grease the top edges of tank to ensure tight seal	As needed
TLC Plate	Store in dark until ready for use	
Analtech autospotter	Clean Syringes with acetone	After each use

6.0 INTERFERENCES

6.1 Solvent, reagents, glassware and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences.

6.2 Several dye compounds decompose rapidly when exposed to light. Standards must be kept in the dark until used. Samples and sample extracts must be stored in the dark until analyzed.

7.0 PERSONNEL QUALIFICATIONS

This method is restricted to use by or under the supervision of analysts experienced in TLC, skilled in the interpretation of Rf values, and experienced in handling dye materials. The analysts should be familiar with the scanning software. Each analyst must demonstrate the ability to generate acceptable results with this method. The acceptable criteria is based on the person's ability to successfully obtain Rf values that are within two standard deviations of the average Rf values for that specific dye.

8.0 APPARATUS AND MATERIAL

8.1 TLC System

- TLC Preconcentrated Silica Gel Plates
  TLC tanks with glass lids
- TLC Analtech Autospotter
- 100 µL autospotter syringes
- High-volume particulate filter paper

8.1.1 Mobile Phase: For organic-soluble dyes, use 95% toluene / 5% methanol. For aqueous-soluble dyes, use butanol / water / acetic acid in 4: 1: 1 composition.

8.2 Detection Equipment

8.2.1 UV lamp
8.2.2 Acer Prisa Scanner
8.2.3 MGI Photo Suite II Scanning Software

8.3 Other Equipment

8.3.1 Balance ± 0.0001g
8.3.2 Dessicator with nitrogen purge
8.3.3 Oven- temperature set at 150 °C

8.4 Material

8.4.1 Disposable cartridge filters – 0.45 um teflon filter
8.4.2 Pipets – Class A, glass, appropriate sixes
8.4.3 Pasteur pipets
8.4.4 Vials – 15 mL, glass, teflon-lined cap
8.4.5 Vials – 40 mL, glass teflon-lined cap
8.4.6 Disposable syringes – plastipak, 3 mL and 10 mL or equivalent
8.4.7 Volumetric flasks – appropriate sizes with ground glass stoppers, Class A

NOTE. the 100 mL and 1 L volumetric flasks used for magnetic stirrer extraction must be round.

8.4.8 Mortar and pestle-steel
8.4.9 Graduated cylinders – appropriate sizes

8.5 Preparation of Materials

8.5.1 Glassware will be washed in a phosphate-free detergent with tap water, followed by tap water rinse, and then a final deionized water rinse. Glassware used with organic solvents will be rinsed with a suitable organic solvent, followed by acetone, followed by tap water and then receive the above listed minimum wash. All glassware prior to use will be rinsed three times with the solvent in use.

9.0 REAGENTS

9.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.

9.1.1 Methanol, CH_3OH – HPLC grade
9.1.2 n-butanol. $C_4H_{13}OH$ – HPLC grade
9.1.3 Glacial Acetic Acid
9.1.4 Toluene, $C_6H_5CH_3$ – HPLC grade

9.2 Organic-free reagent grade water – All references to water in this method refer to organic-free reagent water.

9.3 Stock Standard Solutions

9.3.1 Due to the rarity of some organic coloranta, the standards may not be available from commercial sources, and therefore bulk lot samples can be used as standards.

9.3.2 Dry each solid analyte standard to constant weight in a vacuum dessicator in the dark. Place 0.0100 g (weighed to 0.0001 g) of a single analyte into an amber 50 mL volumetric flask and dilute to volume with methanol. Invert flask several times and sonicate for twenty minutes until dissolved. Store in refrigerator at 4 °C in the dark. Calculate the concentration of the stock solutions from the actual weight used. Stock solutions may be used for up to one year.

9.4 Intermediate Standard Solutions for Disperse Violet 1, Solvent Green 3, Disperse Blue 14, Acid Red 64, Acid Blue 9 and Basic Violet 10

9.4.1 Prepare a 1:10 dilution of the stock solution of each dye. Pipette 10.00 ml of stock solution into a 100 ml amber volumetric flask. Dilute to the mark with methanol. Invert several times to ensure. thorough mixing. Store intermediate standards in the refrigerator at 4 °C in the dark.

9.5 Working Standards

9.5.1 Prepare a 1: 10 dilution of the intermediate standard solution of each dye. Pipette 10.00 ml of the intermediate standard solution into a 100 ml amber volumetric flask. Dilute to the mark with methanol and invert several times. Store. working standards in the refrigerator at 4 °C in the dark.

9.5.2 These six working standards are prepared on the day of calibration. The calibration is performed to ensure that the Rf values for these six dye standards are within two standard deviations of the initial calibration. This is also to confirm that the mobile phase and environment for the TLC development are correct,

9 . 6 Surrogate Spiking Solutions

9.6.1 The analyst should monitor the performance of the extraction and analytical system as well as the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard and reagent water blank with one surrogate (i.e., analyte not expected to be present in the sample). The surrogate selected is benzanthrone. It is a very strong W absorbing analyte, yet it is somewhat colorless.

9.6.2 Prepare a stock solution of benzanthrone at 100 mg/L in methanol. Weigh approximately 0.0100 g of benzanthrone in an amber 100 ml volumetric flask. Record the actual weight to the nearest ± 0.0001g. Dilute to the mark with methanol. Invert several times and sonicate for twenty minutes. Store in the refrigerator at 4 °C in the dark.

9.6.3 Pipette 20.00 ml of benzanthrone stock solution into a 2 L volumetric flask. Dilute to the mark with methanol. Invert several times. Store in the refrigerator at 4 °C in the dark.

9.1 Matrix Spiking Solutions

9.7.1 Prepare matrix-spiking solutions in methanol such that the concentration in the sample is five times the EQL (See Table XXX). The solutions are to be prepared so that the mixture of water-soluble dyes can be separated and identified. The aqueous-soluble dye matrix spiking solutions will comprise of two-aqueous soluble dyes (BV 10; AR 64) and two organic-soluble dyes (DV 1; SR 1).

9.8 TLC Mobile Phase

9.8.1 To prepare. mobile phase for aqueous-soluble dyes, pipette 200 ml of n-butanol, 50 ml of deionized water, and 50 ml of glacial acetic acid into a 500 ml volumetric flask. This solution can be used up to 1 week.

9.8.2 To prepare mobile phase for organic-soluble dyes, pipette 50.00 ml of methanol into a 1 L volumetric flask and dilute to the mark with toluene. Store this mobile phase in the refrigerator, when not in use.

10.0 SAMPLE COLLECTION, PRESERVATION, HANDLING AND RECEIPT

10.1 When samples are received by the laboratory, the laboratory's sample custodian examines each cooler's custody seats to verify that they are intact and that the integrity of the environmental samples has been maintained. The sample custodian signs the COC report. The sample custodian then opens the cooler and measures its internal temperature. The temperature reading is noted on the accompanying COC report. The sample custodian then examines the contents of the cooler. Sample container breakages or discrepancies between the COC report and sample label documentation are recorded. All problems or discrepancies noted during this process are to be promptly related to the Field Team Leader. Inter-laboratory COC procedures and specific procedures for sample handling, storage, disbursement for analysis, and remnant disposal will be. followed as specified in the laboratory's SOP and/or QA Plan.

10.2 Samples are collected in amber glass containers with Teflon-lined caps. All samples and sample extracts are stored at 4° C ± 2" C. All samples and sample extracts must be stored in the dark until analysis to avoid decomposition of the dyes. Based on guidelines listed in SW-846 for the extraction of stable organic compounds (e.g., dioxins), aqueous and solid samples must be extracted within 30 days of collection. All extracts must be analyzed within 45 days of sample preparation. Since aqueous samples analyzed with the high level method are not extracted, they must be analyzed within 30 days.

11.0 PROCEDURE

11.1 Sample Preparation

11.1.1 Aqueous Samples

11.1.1.1 Low-level Method

11.1.1.2 High-level Method

11.1.1.3 Soil and Sediment Samples

11.2 Chromatographic Conditions

Plate: Preconcentrated Silica Gel (60F-254 nm) 20x20 cm, glass-backed. Place silica gel plates on the TLC carrier and heat for 20 minutes at 150 °C. After heating, store carrier in nitrogen-purged dessicator. Remove silica gel plates, as needed.

Mobile Phase: Aqueous-soluble dye analysis use n-butanol, water, and acetic acid in a 4:1:1 composition.
Organic-soluble dye analysis use 95% toluene, 5% methanol solution.

Tank: Between each run, the TLC tank and filter paper must be dried. Place 50 ml of fresh mobile phase, prepared in accordance with Section 9.8. Line the front and backsides of the tank with filter paper that is saturated with the mobile phase in the tank.

TLC Autospotter: Temperature is set at 2 (51 "C)
Speed is set at 1

12.0 QUALITY CONTROL AND QUALITY ASSURANCE

12.1 Analysis Sequence

All acceptable samples must be analyzed within a valid analysis sequence as given below.

Time Frame	Injection #	Material Injected
Prior to project initiation and every 6 months minimum		Organic and Aqueous soluble dyes for Initial Rf Determination
Daily	1	Daily Rf value confirmation standards
	2	Method Blank
	3	Sample Analysis

12.2 Rf Value Determination

12.2.1 Initial Determination of Rf Values

Spot 100 µl each of the 17 organic-soluble dyes with the TLC autospotter syringes onto the Silica Gel plate. Place the plate into the tank and allow the solvent front to move at least 10 cm before removing from the tank. After removing the plate, immediately mark the solvent front with a pencil and let dry. Repeat six times to obtain an average Rf value for each standard dye. This procedure is repeated for the aqueous-soluble dyes.

12.2.2 Daily Rf Value Confirmation

Spot 100 µl of Basic Violet 10, Acid Red 64, and Acid Blue 9 (1:100 dilution) on a silica gel plate. Place plate in tank and elute. Check the Rf value for this dye and confirm the Rf value. Repeat this procedure for Disperse Violet 1, Solvent Green 3, and Disperse Blue 14.

12.3 Initial Determination

12.3.1 Criteria: Presence of visually detectable spot after TLC run and software manipulation. Calculate Rf values and standard deviation of the seven trial runs for aqueous-soluble dyes and organic-soluble dyes. Repeat initial determination every 6 months.

12.4 Daily Determination

12.4.1 Criteria: Presence of visually detectable spot after TLC run and software manipulation. Calculated Rf values and standard deviation of the three dyes for both aqueous-soluble dyes and organic-soluble dyes must be within 2 standard deviations of initial determination.

12.4.2 Corrective Action: Stop work immediately and clean the TLC tanks, prepare fresh mobile phase.

12.5 Method Blank

125.1 Criteria: The surrogate spike (benzanthrone) must be detected by a UV lamp and no other extraneous spots should be present.

12.5.2 Corrective Action: Stop work immediately and clean the TLC tanks, prepare fresh mobile phase, and check W lamp.

12.6 Matrix Spike

12.6.1 Criteria: The Rf values for the aqueous-soluble dyes (BV 10; AR 64) and organic soluble dyes (DV1; SR1) should be within ± 2 standard deviations of the average Rf values.

12.6.2 Corrective Action: Stop work immediately and clean the TLC tanks, and prepare fresh mobile phase.

12.7 System Monitoring Compound

12.7.1 Criteria: The system monitoring compound (benzanthrone) must be detected by a W lamp and be within +/- 2 standard deviations of the initial determination.

12.7.2 Corrective Action: Stop work immediately and clean the TLC tanks, prepare fresh mobile phase, and check W lamp.

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TABLE 1
ESTIMATED QUANTITAION LIMITS

Compounds	Aqueous (ug/L)		Soil (mg/kg)
	Low-level	High-level	
Basic Violet 10			
Acid Blue 45			
Acid Yellow 3			
Acid Red 64			
Basic Yellow 2			
Acid Yellow 23			
Acid Yellow 73			
Acid Blue 1			
Acid Blue 9			
Acid Orange 10			
Disperse Blue 14			
Solvent Yellow 2			
Solvent Yellow 3			
Solvent Yellow 14			
Solvent Yellow 33			
Disperse Violet 1			
Pigment Red 1			
Solvent Red 1			
Disperse Red 9			
Solvent Red 24			
Solvent Green 3			
Solvent Orange 3			
Solvent Orange 7			
Smoke Dye			
1-Aminoanthraquinone			
2-Aminoanthraquinone			

TABLE 2
RF VALUES

Compounds	High Rf	Low Rf	Average Rf	Standard Deviation
Acid Blue 1	0.41	0.34	0.37	0.02
Acid Blue 9	0.42	0.35	0.38	0.02
Acid Blue 45	NA	NA	NA	NA
Acid Yellow 3	0.35	0.28	0.32	0.02
Acid Yellow 23	0.15	0.07	0.12	0.03
Acid Yellow 73	0.85	0.78	0.82	0.03
Basic Yellow 2	0.59	0.56	0.58	0.01
Basic Violet 10	0.60	0.57	0.58	0.01
Acid Red 64	0.09	0.07	0.08	0.01
Disperse Blue 14	0.55	0.45	0.50	0.04
Solvent Yellow 2	0.72	0.68	0.70	0.01
Solvent Yellow 3	0.50	0.45	0.48	0.02
Solvent Yellow 14	0.70	0.65	0.68	0.02
Solvent Yellow 33	0.43	0.25	0.33	0.06
Disperse Violet 1	0.23	0.16	0.20	0.03
Pigment Red 1	0.69	0.61	0.64	0.03
Solvent Red 1	0.59	0.48	0.54	0.04
Disperse Red 9	0.66	0.62	0.65	0.02
Solvent Red 24	0.71	0.73	0.69	0.02
Solvent Green 3	0.77		0.75	0.01
Solvent Orange 3	0.22	0.16	0.19	0.02
Solvent Orange 7	0.70	0.65	0.68	0.02
Smoke Dye	0.68	0.66	0.67	0.01
1-Aminoanthraquinone	0.54	0.49	0.52	0.02
2-Aminoanthraquinone	0.28	0.22	0.25	0.02

NAVAL SURFACE WARFARE CENTER CRANE DIVISION
EXPLOSIVE SCIENCES BRANCH
Code 4052
Crane. Indiana

SOP # XXXXX

Determination of Organic Colorants in Environmental Matrices by High-Performance Liquid Chromatography

Revision: 0

Revised by: _____ Date: _____

Approved by: _____ Date: _____
XXX QA Officer

Approved by: _____ Date: _____
XXX Laboratory Director

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1.0 SCOPE AND APPLICABILITY

This method is intended for the analysis of organic colorants by high performance liquid chromatography (HPLC) using a Photodiode Array Detector (PDA). This method is used to determine the concentration of the following dyes in aqueous and solid matrices:

Aqueous Soluble			Organic Soluble		
Compound Name	Abbr.	CAS #	Compound Name	Abbr.	CAS #
Acid Blue 1	AB1	129-17-9	Disperse Blue 14	DB14	2475-44-7
Acid Blue 9	AB9	3844-45-9	Solvent Yellow 2	SY2	60-11-7
Acid Blue 45	AB45	2861-02-1	Solvent Yellow 3	SY3	97-56-3
Acid Yellow 3	AY3	8004-92-0	Solvent Yellow 14	SY14	842-07-9
Acid Yellow 23	AY23	1934-21-0	Solvent Yellow 33	SY33	8003-22-3
Acid Yellow 73	AY73	518-47-8	Disperse Violet 1	DV1	128-95-0
Basic Yellow 2	BY2	2465-27-2	Pigment Red 1	PR1	6410-10-2
Basic Violet 10	BV10	81-88-9	Solvent Red 1	SR1	1229-55-6
Acid Red 64	AR64	NA	Disperse Red 9	DR9	82-38-2
			Solvent Red 24	SR24	85-83-6
			Solvent Green 3	SG3	128-80-3
			Solvent Orange 3	SO3	495-54-5
			Solvent Orange 7	SO7	3118-97-6
			Smoke Dye	SD	81-64-1
			1-Aminoanthraquinone	1-AQ	8245-1
			2-Amino; mthraquinone	2-AQ	117-79-3

1.1 This method provides a salting-out extraction procedure for low concentration of dyes in surface or ground water. Direct injection of diluted and filtered aqueous samples can be used for samples of higher concentration. (See Table 1 -Estimated Quantitation Limits)

1.2 These dyes may be dangerous. When handling samples and stock solutions, follow safety precautions listed in Section 5.0.

1.3 The low and high estimated quantitation limits for solid and aqueous matrices are listed in Table 1.

2.0 SUMMARY OF THE METHOD

2.1 This method provides HPLC conditions for the detection of ppb and low ppm levels of dyes in aqueous and solid matrices. Prior to using this method, appropriate sample preparation must be. used.

2.2 This method utilizes a dual gradient, where flow and mobile phase composition are varied over time, to achieve sufficient separation of the

dyes. Due to retention time variations inherent in utilizing a dual gradient system, a marker compound, anthracene, will be added to all solvents utilized for this project. Relative retention times with respect to anthracene will be used to monitor system performance.

2.3 Low-level Aqueous Sample Extraction: This method is an adaptation of a salting-out procedure for nonvolatile organics described in EPA Method SW-846-8330. Aqueous samples of low concentration are extracted using a salting-out extraction procedure with acetonitrile and sodium chloride. The small volume of acetonitrile that remains undissolved above the salt water is drawn off and transferred to a smaller volumetric flask. It is back-extracted by vigorous stirring with a specific volume of salt water. After equilibration, the phases are allowed to separate, and the small volume of acetonitrile residing in the narrow neck of the volumetric flask is removed using a Pasteur pipette. This aliquot is then blown to dryness with a gentle stream of nitrogen.

2.4 High-level Aqueous Sample Direct Injection Method: For aqueous samples in which the dye content is visually detectable, it is permissible to directly inject the sample after filtration with a 0.45um syringe filter.

2.5 Soil and Sediment Sample Extraction: Solid samples are extracted with a 60:40 v/v mixture of acetonitrile:water, sonicated, centrifuged and filtered prior to analysis.

3.0 DEFINITIONS

This section defines the terms and acronyms as used in this SOP.

PDA	Photodiode Array Detector
c c v	Continuing Calibration Verification - Standard that is injected at some prescribed frequency during the analytical run sequence to determine if the instrument is still calibrated.
RT	Retention Time - The time (in minutes) at which a target analyte elutes from the LC column.
RT Window	Retention Time Window The + value which is applied to the ICV to establish the time range used to make tentative compound identifications.
R_a	Retention Factor – Ratio of the analyte retention time compared to the retention time of anthracene.

UV/VIS Ultraviolet/Visible absorption spectra

4.0 HEALTH AND SAFETY WARNINGS

4.1 Standard laooratory protective clothing and eye covering is required during the execution of this method.

4.2 Any other lab specific requirements – TO BE ADDED

5.0 CAUTIONS

5.1 Reagent standards must be prepared fresh on the day of analysis.

5.2 Refer to the instrument manufacturer's manual for routine instrument precautions. This includes an awareness of moving parts and the potential for electrical shock.

5.3 Toxicity of constituents -TO BE ADDED

5.4 Waste Disposal – All waste will be disposed of according to federal, state, and local regulations.

5.5 Routine and Preventative Maintenance

PREVENTIVE MAINTENANCE FOR ANALYTICAL INSTRUMENTS

Instrument	Preventive Maintenance	Maintenance Frequency
HPLC	Change filter frit in mixer.	As needed (when pressure builds)
	Change column pre-filter.	As needed (2-3 months)
	Rinse water pump with methanol, filter water, sonicate water intake filter frit.	Approximately weekly.
	Change pump seals.	As needed.

6.0 INTERFERENCES

6.1 Solvent, reagents, glassware and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation

of the chromatograms. All of these materials must be demonstrated to be free from interferences.

6.2 Several dye compounds decompose rapidly when exposed to light. Standards must be kept in the dark until used. Samples and sample extracts must be stored in the dark until analyzed.

7.0 PERSONNEL QUALIFICATIONS

This method is restricted to use by or under the supervision of analysts experienced in the use of HPLC, skilled in the interpretation of chromatograms, and experienced in handling dye materials. Each analyst must demonstrate the ability to generate acceptable results with this method.

8.0 APPARATUS AND MATERIALS

8.1 HPLC System

- Waters 2690 Separations Module (auto-sampler, gradient pump)
- Waters 996 Photodiode Array Detector
- Waters Temperature Control Module
- 30 uL minimum volume sample loop
- Waters Millennium[32] V3.05.01 Chromatography Software

The system is equipped with a Waters 2690 Separation Module. The pump is capable of achieving 5000 psi. For the low concentration option, the detector must be. capable of a stable baseline at 0.001 absorbance units full scale.

8.2 Other Equipment

8.2.1 Temperature controlled ultrasonic bath
8.2.2 Balance ± 0.0001 g
8.2.3 Magnetic stirrer with stirring pellets
8.2.4 Oven -forced air, without heating
8.2.5 Centrifuge

8.3 Materials

8.3.1 Disposable cartridge filters – 0.45 um teflon filter
8.3.2 Pipettes -Class A, glass, appropriate sizes
8.3.3 Pasteur pipettes
8.3.4 Scintillation vials – 20 mL, glass
8.3.5 Vials – 15 mL, glass, Teflon-lined cap
8.3.6 Vials – 40 mL, glass Teflon-lined cap
8.3.7 Disposable syringes – plastipak, 5 cc or equivalent
8.3.8 Volumetric flasks – appropriate sizes with ground glass stoppers, Class A. NOTE: the 100 mL and 1 L volumetric flasks used for magnetic stirrer extraction must be round.
8.3.9 Vacuum desiccator-glass

8.3.10 Mortar and pestle – ceramic
8.3.11 Sieve – 30 mesh
8.3.12 Graduated cylinders -appropriate sizes

8.4 Preparation of Material:

8.4.1 Glassware will be washed in a phosphate-free detergent with tap water, followed by tap water rinse, and then a final deionized water rinse. Glassware used with organic solvents will be rinsed with a suitable organic solvent, followed by acetone, followed by tap water and then receive the above listed minimum wash. Prior to use all glassware will be rinsed three times with the solvent in use.

9.0 REAGENTS

9.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.

9.1.1 Methanol, CH_3OH – HPLC grade
9.1.2 Sodium chloride, NaCl – Reagent grade – Prepare an aqueous solution
9.1.3 Acetonitrile (CH_3CN), HPLC grade.
9.1.4 Tetrabutylammonium Hydroxide (TBA) – HPLC grade., 1.0M in methanol
9.1.5 Methanol (CH_3OH), HPLC grade.
9.1.6 Calcium chloride ($CaCl_2$), reagent grade. Prepare. an aqueous solution of 5 g/L.
9.1.7 Sodium chloride (NaCl, shipped in glass bottles), reagent grade.
9.1.8 Organic-free reagent water. All references to water in this method refer to organic-free reagent water, as defined in SW846, Chapter One.

9.2 Stock Standard Solutions

9.2.1 Due to the rarity of some organic colorants, the standards may not be available from commercial sources, and therefore bulk lot samples can be used as standards,

9.2.2 Dry each solid analyte standard to constant weight in a vacuum desiccator in the dark. Place 0.0100 g (weighed to 0.0001 g) of a single analyte into an amber 100 mL volumetric flask and dilute to volume with methanol. Invert flask several times and sonicate for twenty minutes until dissolved. NOTE: PR1 is only soluble to Xmg/L. Store in refrigerator at 4 °C in the dark. Calculate the concentration of the stock solutions from the actual weight used. Stock solutions may be used for up to one year.

9.3 Intermediate Standard Solutions:

9.3.1 Prepare a 1:10 dilution of the stock solution of each dye. Pipette 10.00 ml of stock solution into a 100 ml amber volumetric flask. Dilute to the mark with methanol. Invert several times to ensure thorough mixing. Store intermediate standards in the refrigerator at 4" C in the dark.

9.4 Working Standards:

9.4.1 Calibration standards at a minimum of five concentration levels should be prepared for the initial calibration through dilution of the intermediate standards solutions. Multi-standards can be broken into groups to aid in integration. These solutions must be refrigerated and stored in the dark, and they must be prepared fresh on the day of calibration.

9.4.2 For continuing calibration, run a midpoint standard of all dyes at the beginning of the day, and run one group of midpoint standards every 10 samples.

9.5 Surrogate Spiking Solutions

9.5.1 The analyst should monitor the performance of the extraction and analytical system as well as the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard and reagent water blank with one surrogate (i.e., analyte not expected to be present in the sample). The surrogate selected for this method is benzanthrone. It is a very strong UV absorbing analyte, yet it is somewhat colorless.

9.6 Working Standards

9.6.1 Calibration standards at a minimum of five concentration levels should be prepared through dilution of the intermediate standards solutions. These solutions must be refrigerated and stored in the dark and prepared fresh on the day of calibration. Prepare the working calibration standards as follows:

Standard	Source Solution	Amount Added (mL)	Final Volume (mL)	Final Concentration (ug/mL)
Standard 5	A B			
Standard4	A			
Standard 3	C			
Standard 2	D			
Standard 1	E			

Source Solution	Concentration (ug/mL)

A = XX Component Mix	
B = Surrogate Stock Solution	
C = Standard 5	
D = Standard 4	
E = Standard 2	

9.7 Matrix Spiking Solutions

9.7.1 Prepare matrix-spiking solutions in methanol such that the concentration in the sample is five times the EQL (See Table 1). All target analytes are included in the spiking material.

9.8 HPLC Mobile Phase

9.8.1 To prepare 1 L of 0.005M TBA mobile phase, dilute 5ml of 0.5M TBA in methanol to 1 L with methanol in a volumetric flask

9.9 Anthracene marker

9.9.1 TO BE ADDED

10.0 SAMPLE COLLECTION, PRESERVATION, HANDLING AND RECEIPT

10.1 Upon sample receipt, the laboratory's sample custodian examines each cooler's custody seals to verify that they are intact and that the integrity of the environmental samples has been maintained. The sample custodian signs the COC report. The sample custodian then opens the cooler and measures its internal temperature. The temperature reading is noted on the accompanying COC report. The sample custodian then examines the contents of the cooler. Sample container breakages or discrepancies between the COC report and sample label documentation are recorded. All problems or discrepancies noted during this process are to be promptly related to the field team leader. Inter-laboratory COC procedures and specific procedures for sample handling, storage, disbursement for analysis, and remnant disposal will be followed as specified in the laboratory's SOP and/or QA Plan.

10.2 Samples are collected in amber glass containers with Teflon-lined caps. All samples and sample extracts are stored at 4" C ± 2" C. All samples and sample extracts must be stored in the dark until analysis to avoid decomposition of the dyes. Based on guidelines listed in SW-846 for the extraction of stable organic compounds (e.g., dioxins), aqueous and solid samples must be extracted within 30 days of collection. All extracts must be analyzed within 45 days of sample preparation. Since aqueous samples analyzed with the high level method are not extracted, they must be analyzed within 30 days.

11.0 PROCEDURE

11.1 Sample Preparation

For aqueous samples, it is highly recommended that the samples be screened with the high-level method to determine if the low-level method (1 to 50 µg/L) is required; the high-level method will not be necessary in most cases.

11.1.1 Aqueous Samples, Low-Level Method: Salting-out extraction (still undergoing testing)

11.1.1.1 Place 251.3 g of sodium chloride into a 1-L round volumetric flask.

11.1.1.2 Using a 1-L graduated cylinder, measure out 770 mL of a aqueous sample and transfer it to the volumetric flask containing the sodium chloride. Add a stir bar and mix the contents at maximum speed on a magnetic stirrer until the salt is completely dissolved.

11.1.1.3 Add 164 mL of acetonitrile (measured with a 250-mL graduated cylinder) while the solution is being stirred, and stir for an additional 15 minutes. Turn off the stirrer and allow the phases to separate for 10 minutes.

11.1.1.4 Remove the acetonitrile (upper) layer with a Pasteur pipette, approximately 8 mL, and transfer it to a 100-mL round volumetric flask.

11.1.1.5 Add 10 mL of fresh acetonittile to the aqueous sample in the 1-L flask. Again, stir the contents of the flask for 15 minutes followed by 10 minutes for phase separation.

11.1.1.6 Combine the second acetonittile portion with the initial extract in the 100-mL flask. The inclusion of a few drops of salt water at this point is unimportant.

11.1.1.7 Add 84 mL of salt water (325 g NaCl per 1,000 mL of reagent water) to the acetonitrile extract in the 100-mL volumetric flask. Add a stir bar and stir the contents on a magnetic stirrer for 10 minutes, followed by 10 minutes of phase separation.

11.1.1.8 Carefully transfer the acetonitrile phase to a 10-mL graduated cylinder using a Pasteur pipette. At this stage, the amount of water transferred with the acetonitrile must be minimized..

11.1.1.9 Add an additional 1.0 mL of acetonitrile to the 100 - mL volumetric flask. Once again, stir the contents of the flask for 15 minutes, followed by 10 minutes of phase separation.

11.1.1.10 Combine the second acetonitrile portion with the initial extract in the 10-mL graduated cylinder

(transfer to a 25-mL graduated cylinder if the volume exceeds 5 mL). Record the total volume of acetonitrile extract to the neatest 0.1 mL; use this as the volume of total extract [V_t] in the calculation of concentration after converting it to • L.

11.1.2 Aqueous Samples, High-level Method

11.1.2.1 Filter sample through a 0.45• m Teflon filter cartridge and inject directly into the HPLC

11.1.3 Soil and Sediment Samples

11.1.3.1 Dry the soil samples in air at room temperature (or colder) to a constant weight, being careful not to expose the samples to direct sunlight. Grind and homogenize the dried sample thoroughly in an acetonitrile-rinsed mortar to pass a 30-mesh sieve.

11.1.3.2 Place a 2.0-g sub-sample of each soil sample in a 15-mL glass vial. Add 10.0 mL of 60:40 acetonitrile:water, cap with a Teflon-lined cap, shake for 1 minute, and place in a cooled ultrasonic bath for 18 hours.

11.1.3.3 After sonication, allow the sample to settle for 30 minutes. Remove 5.0 mL of supematant.

11.1.3.4 Place supematant in a disposable syringe and filter it through a 0.45-microm Teflon filter. Discard the first 3 mL and retain the remainder in a Teflon-capped vial for HPLC analysis.

11.2 Chromatographic Conditions

11.2.1 Recommended Column: Varian Chrompack, Microsphere Cl8 column, 4.6mm x 100mm, 3um particle size

11.2.2 Mobile Phase: High Purity Water/O.005 M Tetrabutyl-onium Hydroxide (TBA) in Methanol

11.2.3 Dual Gradient: Run time = 65 mm

11.2.3.1 Initial Parameters:

75/25 Water/TBA
0.1 mL/min
Column Temp = 27°C

11.2.3.2 Final Parameters:

0/100 Water/TBA
1.0 mL/min
Column Temp = 27°C

11.2.4 Mobile phase flow and composition are varied on a linear gradient throughout the analytical run. The instrument is allowed to come

to equilibrium after returning to the initial parameters for 10 min prior to the next run.

11.2.5 Detector Conditions: PDA detector is set to scan from 211nm to 700nm. To maximize sensitivity, extract and integrate a separate chromatogram for each dye at the appropriate, most sensitive, analytical wavelength according to the following chart:

Water Soluble Dyes

Dye Name	Crane Acronym	Analytical Wavelength (nm)	Retention Time (min)
acid blue 45	AB45	633	11.58
acid yellow 23	AY23	404	14.53
acid red 64	AR64	524	14.98
acid yellow 3	AY3	383	15
acid yellow 73	AY73	493	18.17
acid orange 10	AO10	485	19.61
acid blue 9	AB9	628	22.7
acid blue 1	AB1	635	25.01
basic yellow 2	BY2	378	36.75
basic violet 10	BV10	549	37.43

Organic Soluble Dyes

Dye Name	Crane Acronym	Analytical Wavelength (nm)	Retention Time (min)
disperse violet 1	DV1	588	21.64
2-aminoanthraquinone	2-AQ	299	23.42
smoke dye	SD	547	23.93
solvent orange 3	SO3	408	24.22
solvent yellow 33	SY33	399	28.17
1-aminoanthraquinone	1-AQ	481	28.4
disperse blue 14	DB14	368	34.28
solvent yellow 3	SY3	385	36.21
disperse red 9	DR9	513	36.97
benzanthrone	BZ	399	37.47
pigment red 1	PR1	495	39.97
solvent yellow 2	SY2	416	40.76
anthracene	AN	251	40.85
solvent yellow 14	SY14	486	44.72
solvent red 1	SR1	504	45.36
solvent orange 7	SO7	499	51.68
solvent green 3	SG3	635	56.51
solvent red 24	SR24	519	56.66

Example UV/VIS spectra of each dye and its approximate retention time can be found in attachment XX.

11.3 Calibration of HPLC:

The entire HLPC system is allowed to warm up for a minimum of 30 minutes. **During** this time the mobile phase, at the initial gradient conditions, should be pumped through the column.

11.3.1 Initial Calibration

11.3.1.1 Injections of each calibration standard over the concentration range of interest are preformed. Peak heights and peak areas are obtained for each analyte at the appropriate wavelength.

11.3.2 Daily Calibration

11.3.2.1 Analyze the midpoint calibration standards along with an instrument blank at the beginning of each day and after each set of 10 samples or less. Obtain the response factor for each analyte from the mean peak heights or peak areas and compare it with the response factor obtained for the initial calibration. The mean response factor for the daily calibration must agree within **±XX%** of the response factor of the initial calibration. The same criterion is required for subsequent standard responses compared to the mean response of the triplicate standards at the beginning of the day. If this criterion is not met, a new initial calibration must be obtained.

11.4 **HPLC** Analysis

11.4.1 Analytical Sequence

All acceptable samples must be analyzed within a valid analysis sequence as given below:

Initial Calibration:

Injection Number	Material Injected
1	Instrument Blank
2 - 6	Five Initial Calibration Standards
7	Instrument Blank at end of initial calibration

Daily Analysis Sequence:

Time	Injection Number	Material Injected
0hr	1	Instrument blank
	2 - 4	Midpoint standard of all dyes

	5	Instrument Blank
	6	LCS
	I- 17	Sample Analyses
-12 hr	18	Instrument Blank
	19	Mid-level CCV of one group of dyes
	20	LCS
	21-31	Sample Analyses
-12 hr	32	Instrument Blank
	33	Mid-level CCV of one group of dyes
	etc	

After the initial calibration, the analysis sequence may continue as long as acceptable instrument blanks and continuing calibration verifications are analyzed at the required frequency. This analysis sequence show only the minimum required blanks and standards. More blanks and standards may be run at the discretion of the analysts; these must also satisfy the criteria presented in Section 12 in order to continue the run sequence.

An analysis sequence must include all required matrix spike/matrix spike duplicate and method blank analyses. The analysts may decide at what point in the sequence they are to be analyzed.

11.4.2 Laboratory Control Sample (LCS)

11.4.2.1 The LCS (reagent blank spike) should be prepared separately from the standards and include at least 6 of the dyes, 3 water soluble and 3 organic soluble. The concentration of the dyes should be approximately 500 µg/L.

11.4.3 Follow the guidelines found in SW846, Method 8000 for the following parameters.

11.4.3.1 Retention Windows

11.4.3.1.1 Retention time windows are established by performing 7 replicate injections over a 72 hour period. Retention time windows are then set at 3 times the standard deviation of the replicate injections.

11.4.3.2 Any modifications to the RTW

11.4.3.3 Initial Calibration

11.4.3.4 Initial Calibration Verification

11.4.3.5 Instrument Blank

11.4.3.6 Continuing Calibration Verification

11.4.3.7 Sample Analysis

11.4.4 Compound Identification

11.4.4.1 Compound identification is achieved through spectral matching with known standards of the dyes.

11.4.5 Compound Quantification

11.4.5.1 Data Acquisition, Calculations and Data Reduction

11.4.5.1.1.1 All data acquisition, chromatogram integration, calibration curve calculation and unknown concentration calculation is preformed utillg the Waters Millennium chromatography software.

11.4.5.1.1.2 Manual calculations such as standard deviation and percent recovery will be performed utilizing a spreadsheet.

11.4.5.1.1.3

11.4.6 Data Management and Records Management

12 QUALITY CONTROL AND QUALITY ASSURANCE

12.1 Initial Calibration

12.1.1 Criteria
12.1.2 Corrective Action

12.2 Initial Calibration Verification
12.2.1 Criteria
12.2.2 Corrective Action

12.3 Continuing Calibration Verification
12.3.1 Criteria
12.3.2 Corrective Action

12.4 Instrument Blank
12.4.1 Criteria
12.4.2 Corrective Action

12.5 Method Blank
12.5.1 Criteria
12.5.2 Corrective Action

12.6 Laboratory Control Sample
12.6.1 Criteria
12.6.2 Corrective Action

12.7 Matrix Spike
12.7.1 Criteria
12.7.2 Corrective Action

12.8 Matrix Spike Duplicate
12.8.1 Criteria
12.8.2 Corrective Action

12.9 System Monitoring Compounds
12.9.1 Criteria
12.9.2 Corrective Action

12.10 Internal Standards
12.10.1 Criteria
12.10.2 Corrective Action

13 METHOD PERFORMANCE

13.1 Method Detection Limit Study

13.2 Method Validation

14 REPORTS

Information to be included in the reports for independent validation is to be listed. This would include the QA/QC information, analytical summaries, etc. -TO BE ADDED

15 REFERENCES

15.2 Wegener, J.W., J.C. Klamer, H. Govers, and U.A. Th. Brinkman (1987) Determination of Organic Colorants in Cosmetic Products by High Performance Liquid Chromatography.

15.3 Chromatographia, 24: 865-875.Garrison, A.W., G.L. Baughman, E.J. Weber, R.L. Adams, and M.S. Brewer (1992) Fate of Colored Smoke Dyes. Army Project Order 88PP8863.

15.4 Alcantara-Licudine, J.P., M.K. Kawate, and Q.X. Li (1997) Method for the Analysis of Phloxine B, Uranine, and Related Xanthene Dyes in Soil Using Supercritical Fluid Extraction and High Performance Liquid Chromatography. Journal of Agricultural Food Chemistry, 45: 766-773.

15.5 Gagliardi, L., G. Cavazzutti, A. Amato, A. Basili, and D. Tonelli (1987) Identification of Cosmetic Dyes by Ion Pair Reversed Phase High Performance Liquid C hromatography. Journal of Chromatography, 394: 345-352.

15.6 Eakes, W., J. Heath, M. Cavit, K. Rimm, W. Powers, C. Connelly, J. Anderson, M. Johnson (1983) Initial Assessment Study of Naval Weapons Support Center Crane, Indiana. NEESA 13-003.

15.7 U.S. Environmental Protection Agency, Method SW-846-8330.

15.8 U.S. Environmental Protection Agency, Method SW-846-8000.

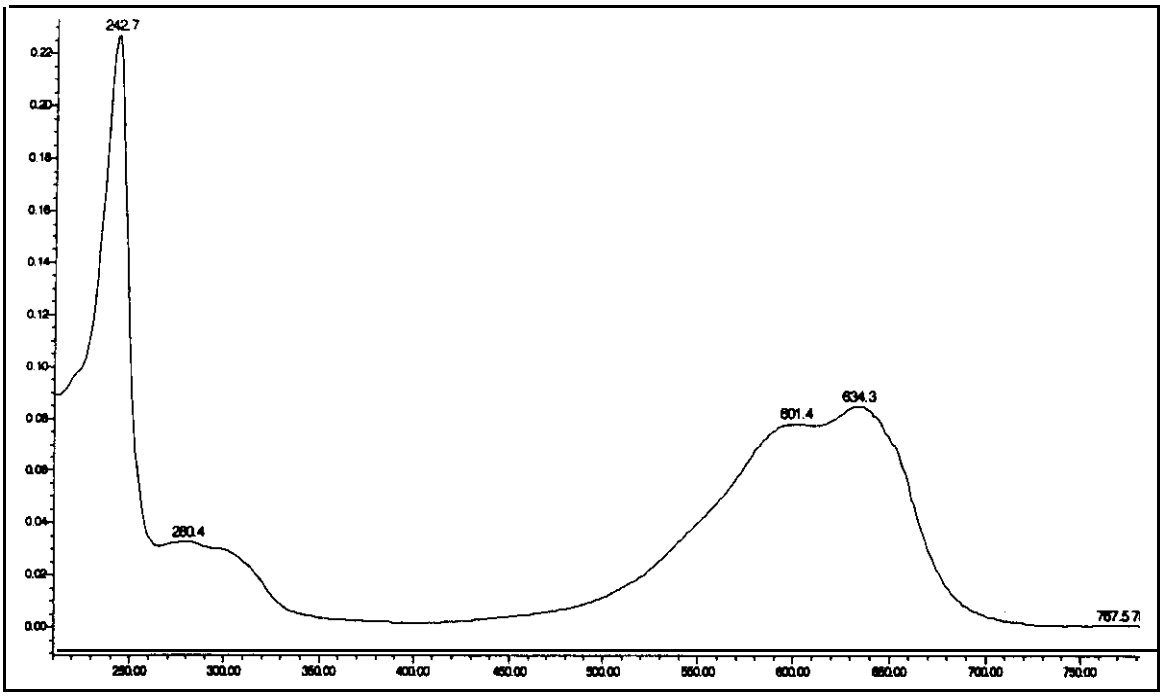
15.9 U.S. Environmental Protection Agency, Method SW-846-3500.

15.10 U.S. Environmental Protection Agency, Method SW-846-3550.

TABLE 1
ESTIMATED QUANTITAION LIMITS

Compounds	Aqueous (ug/L)		Soil (mg/kg)
	Low-level	High-level	
Basic Violet 10			

SPECTRA AND APPROXIMATE RETENTION TIMES



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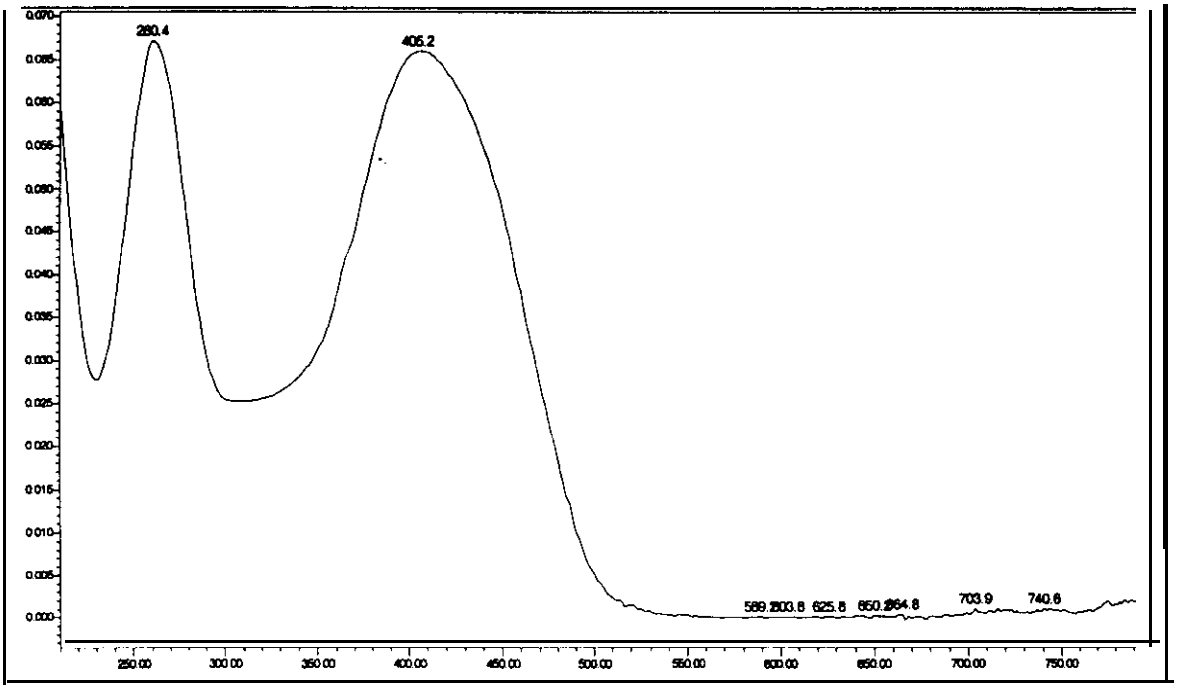

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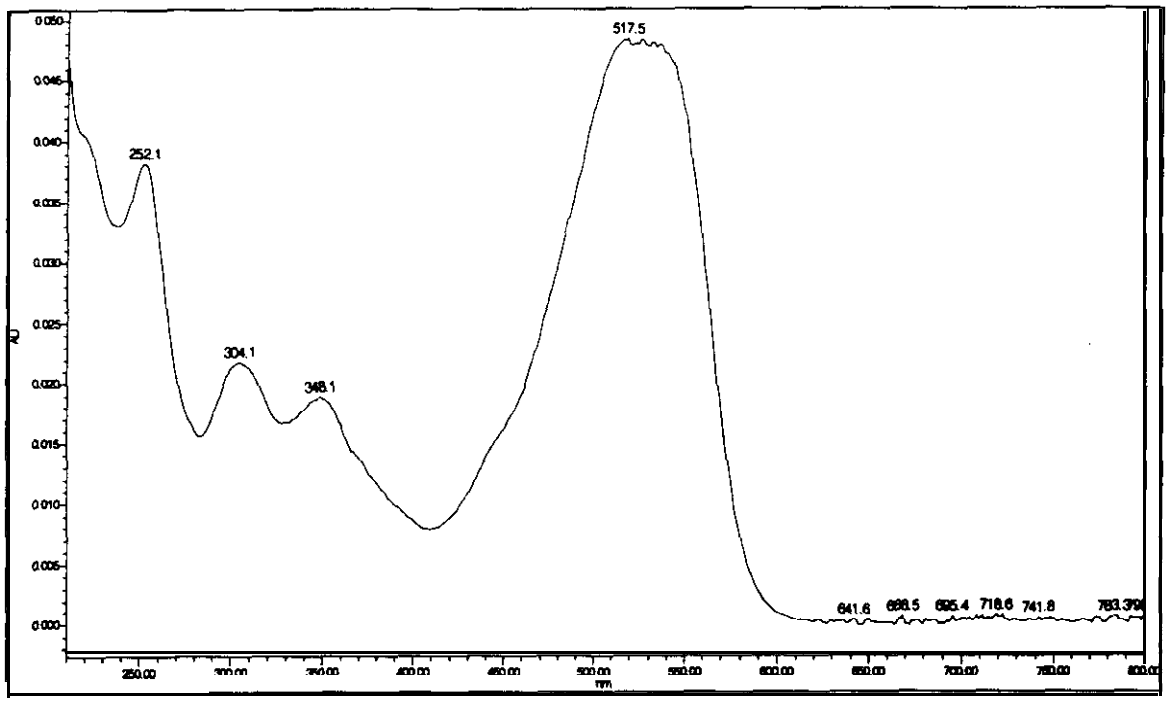

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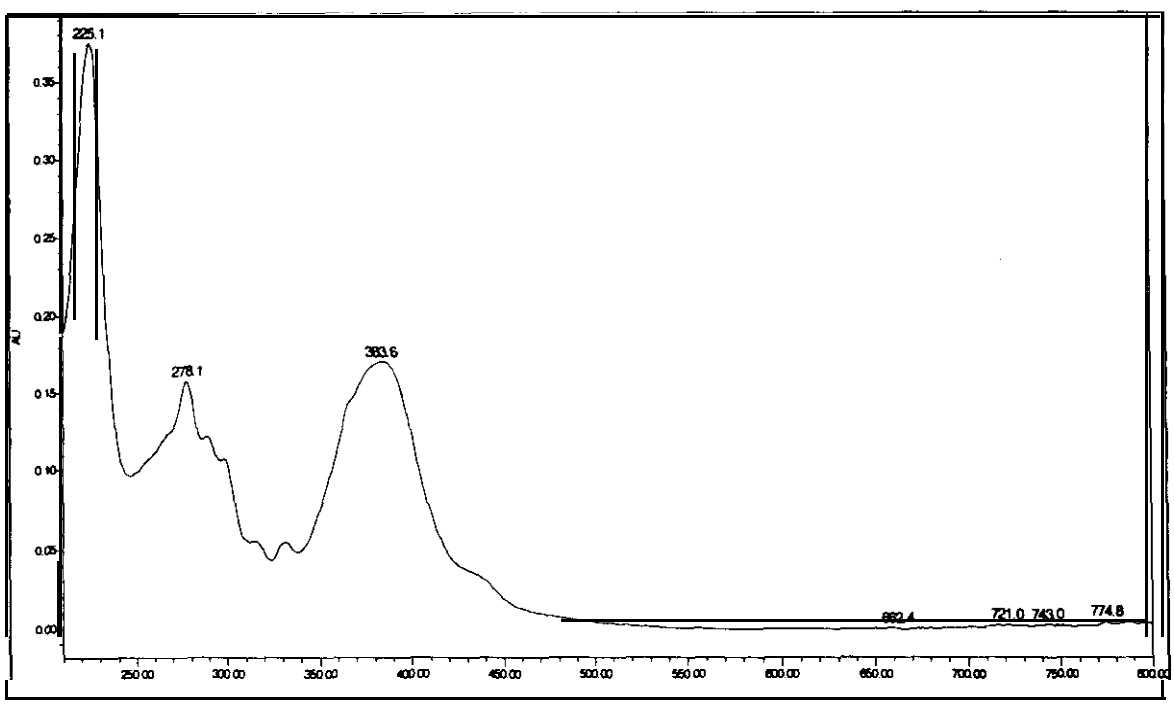

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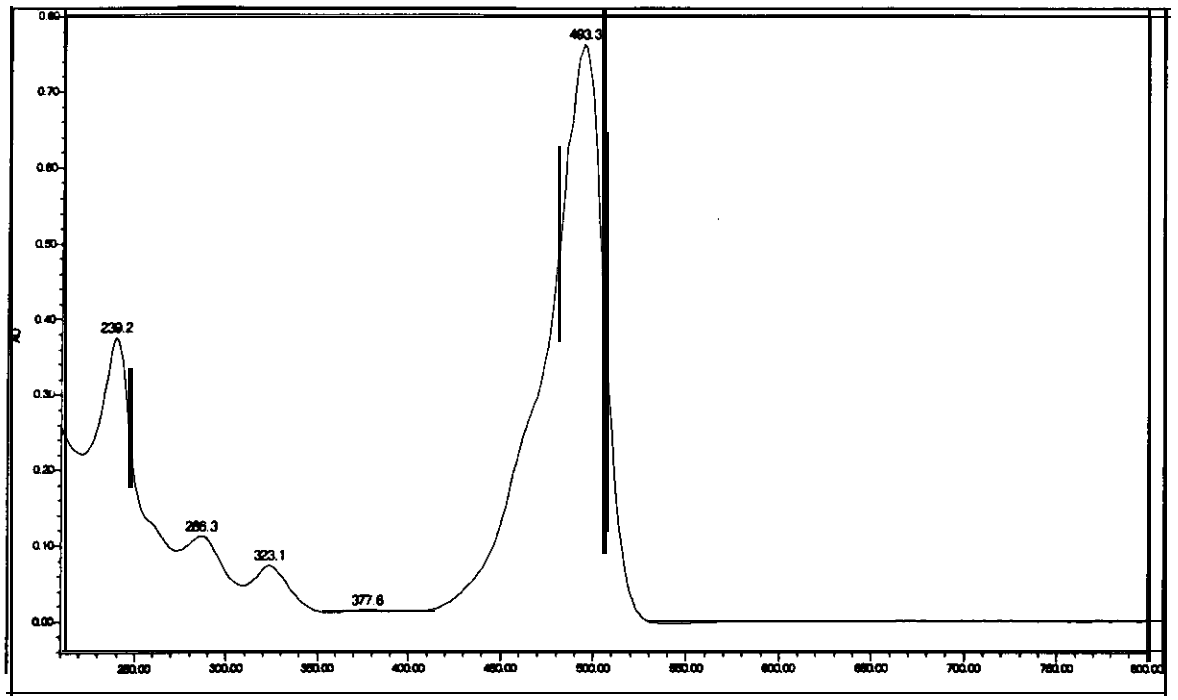

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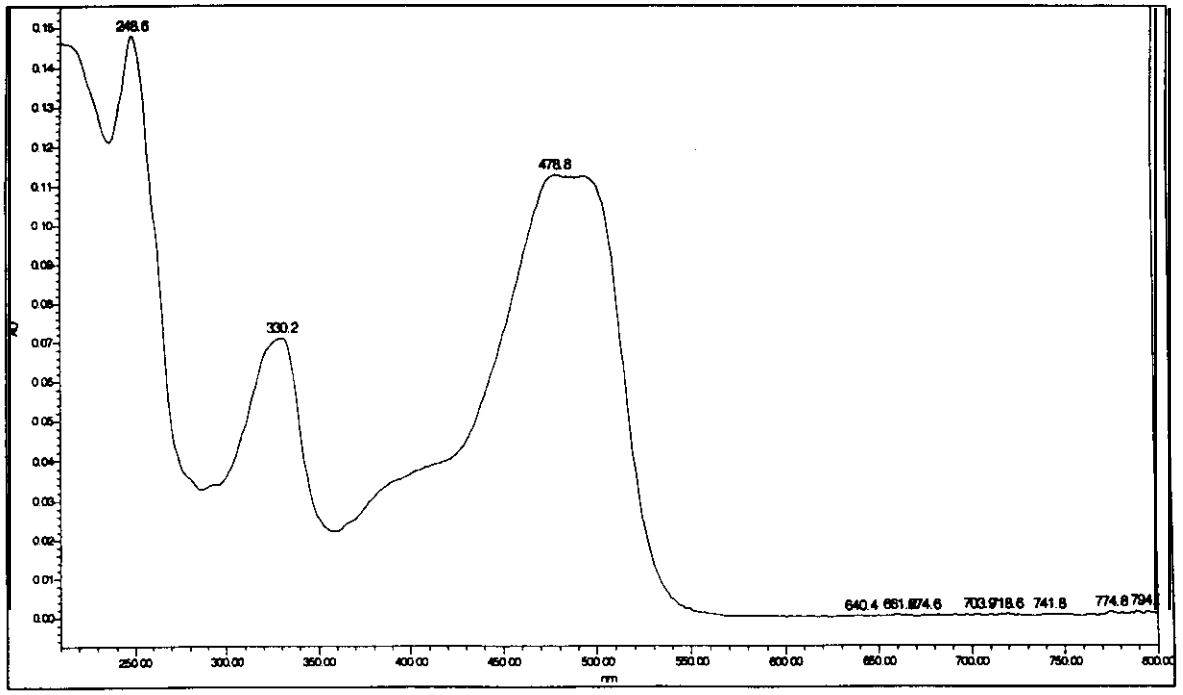

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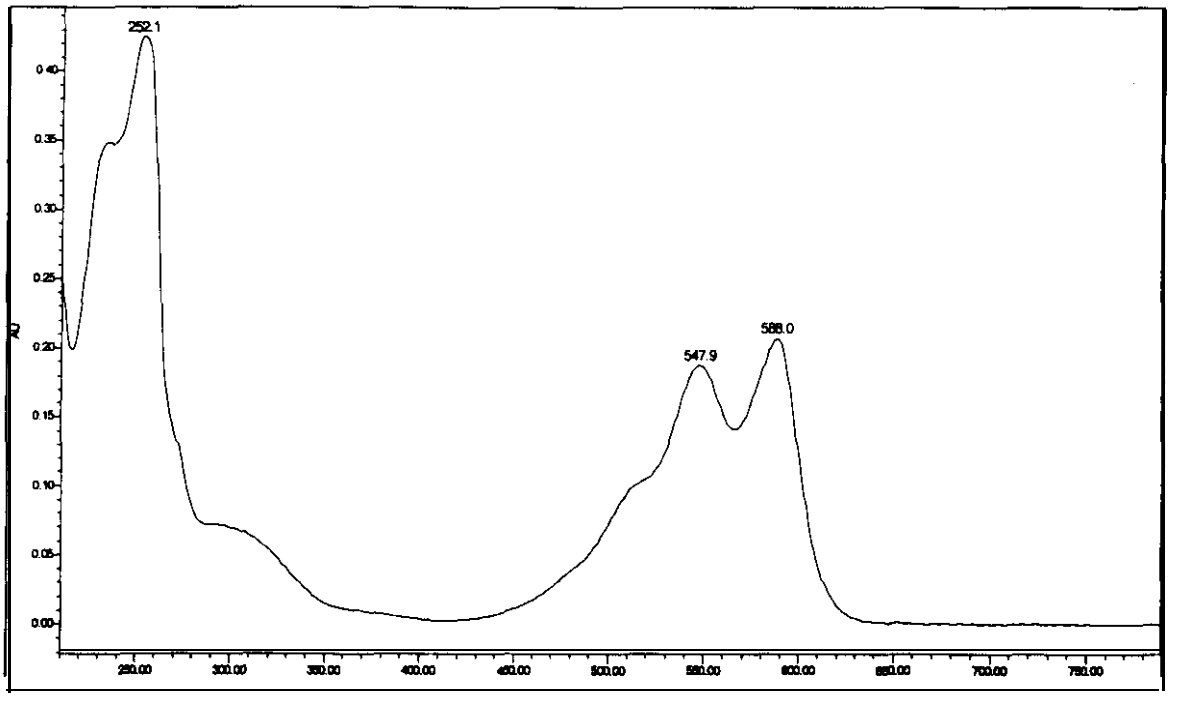

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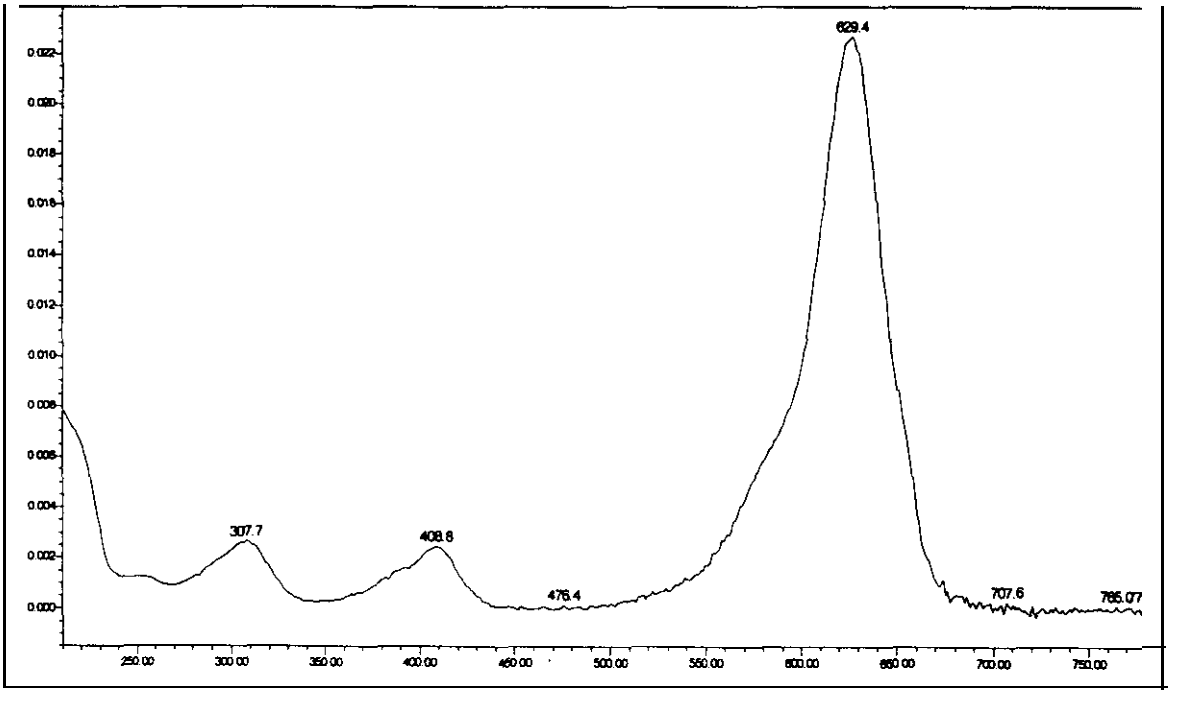

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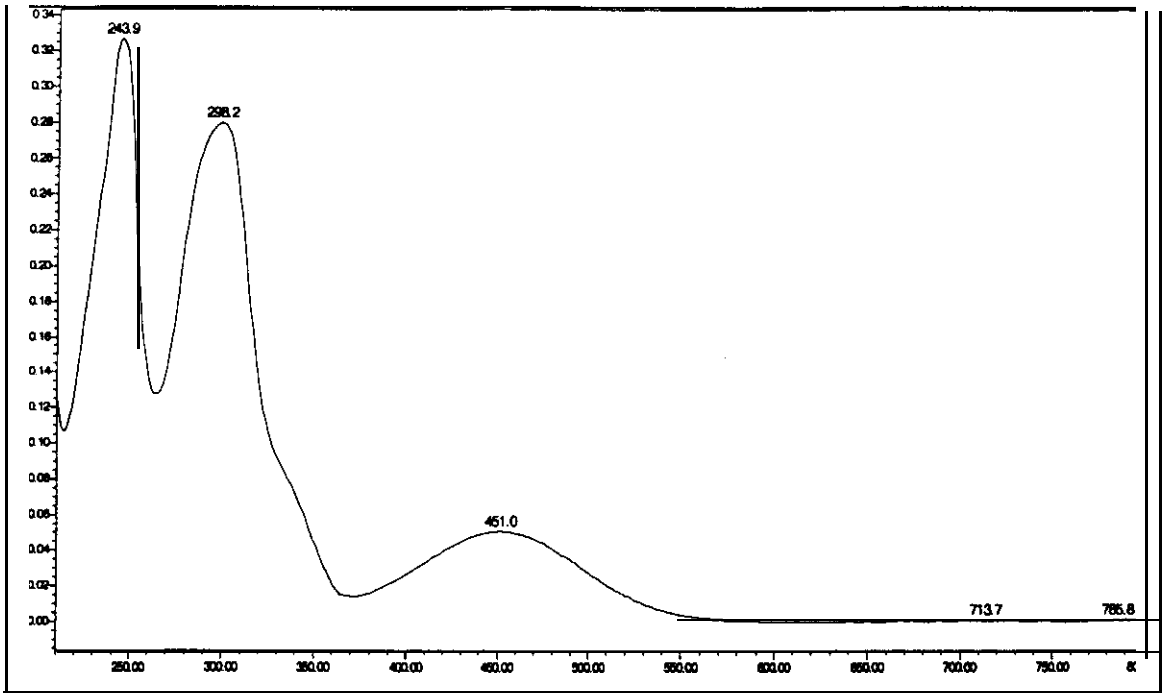

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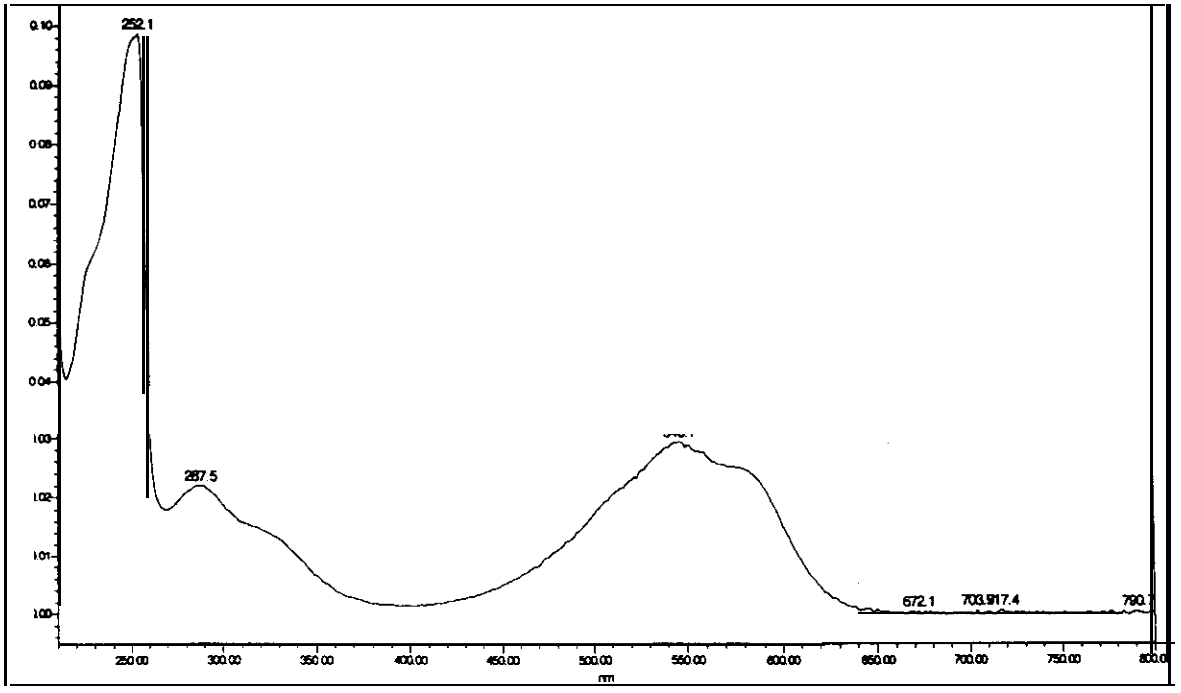

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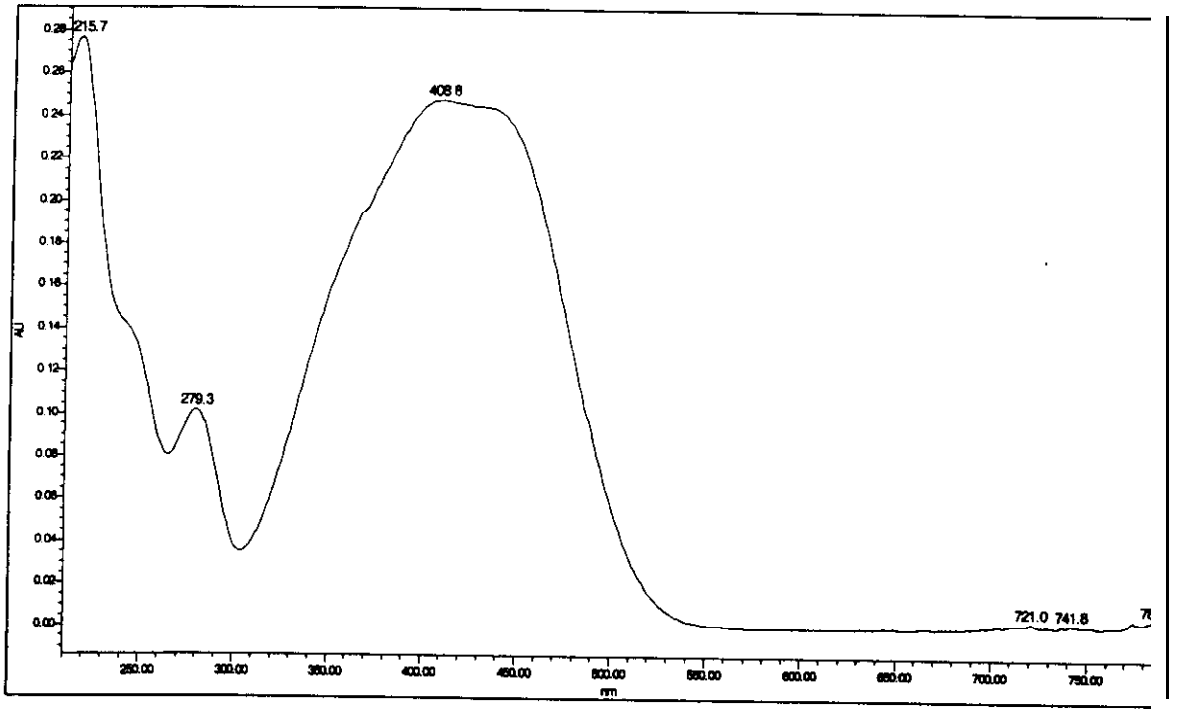

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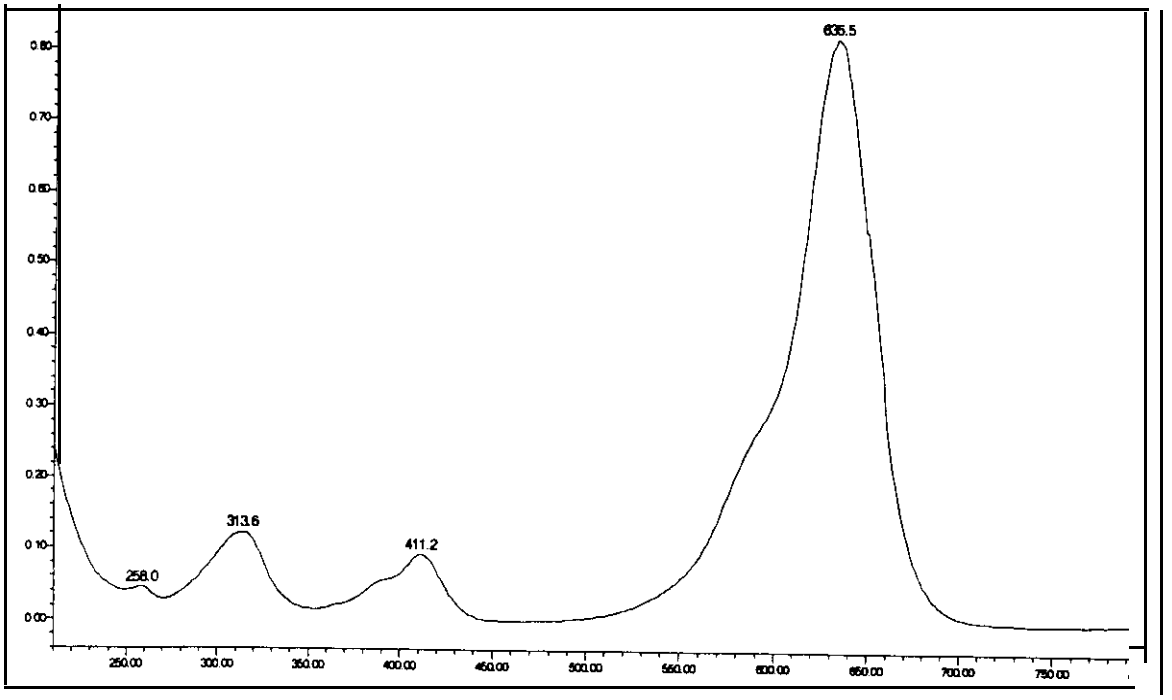

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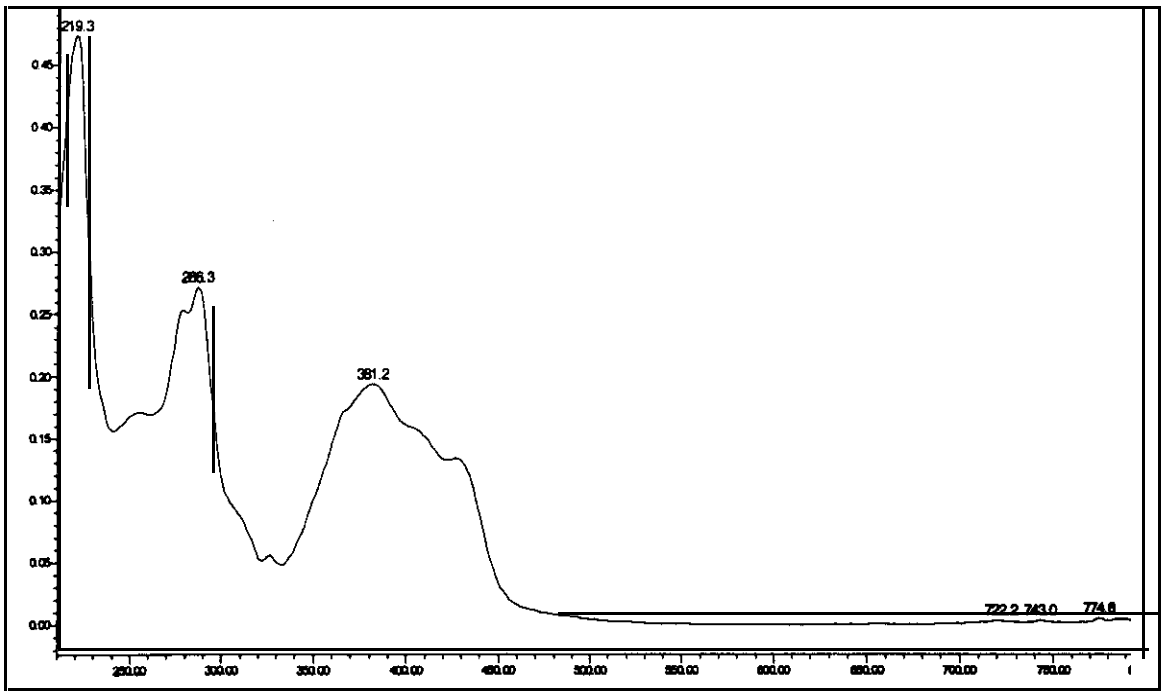

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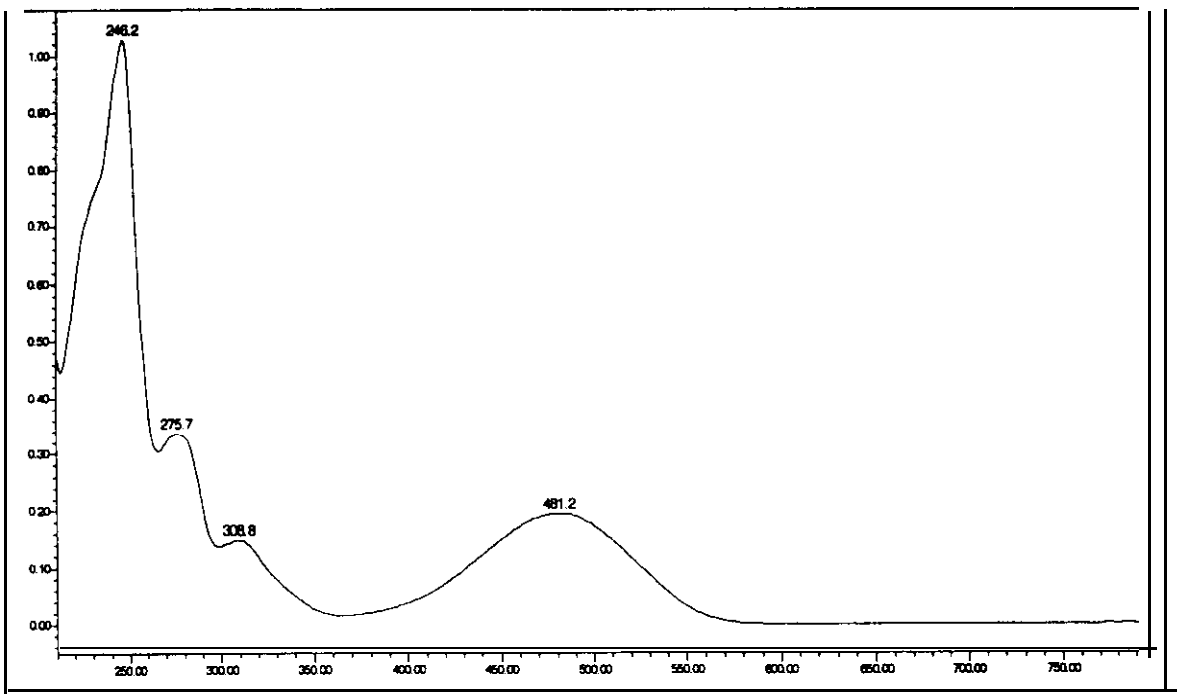

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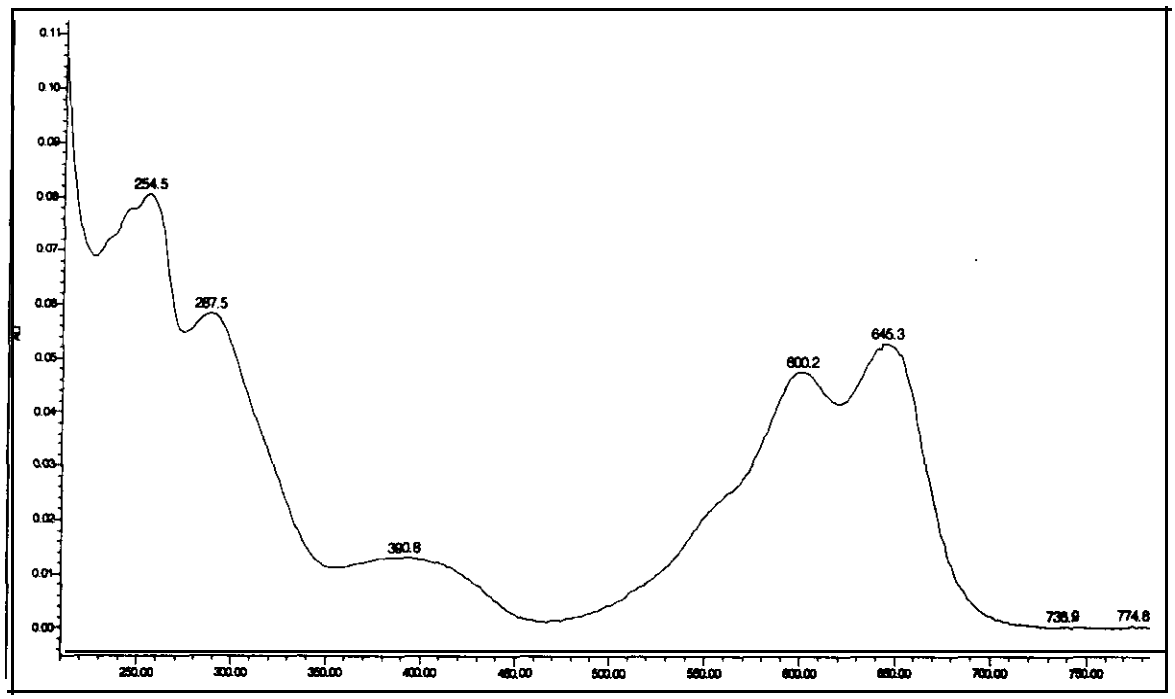

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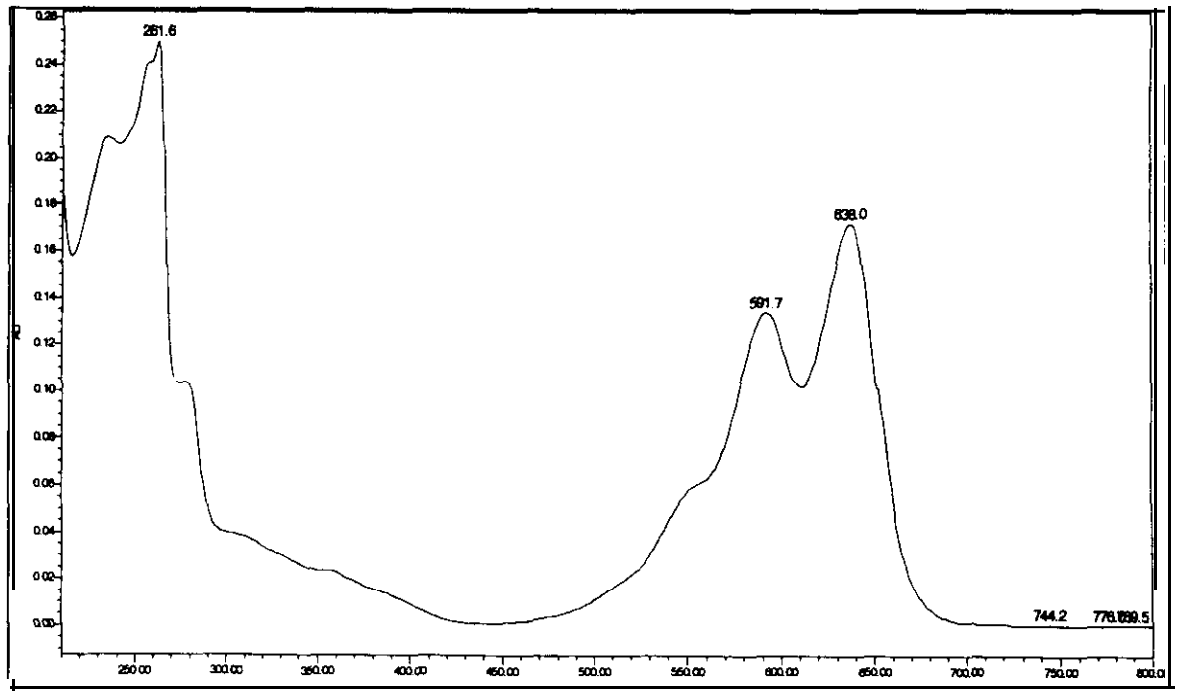

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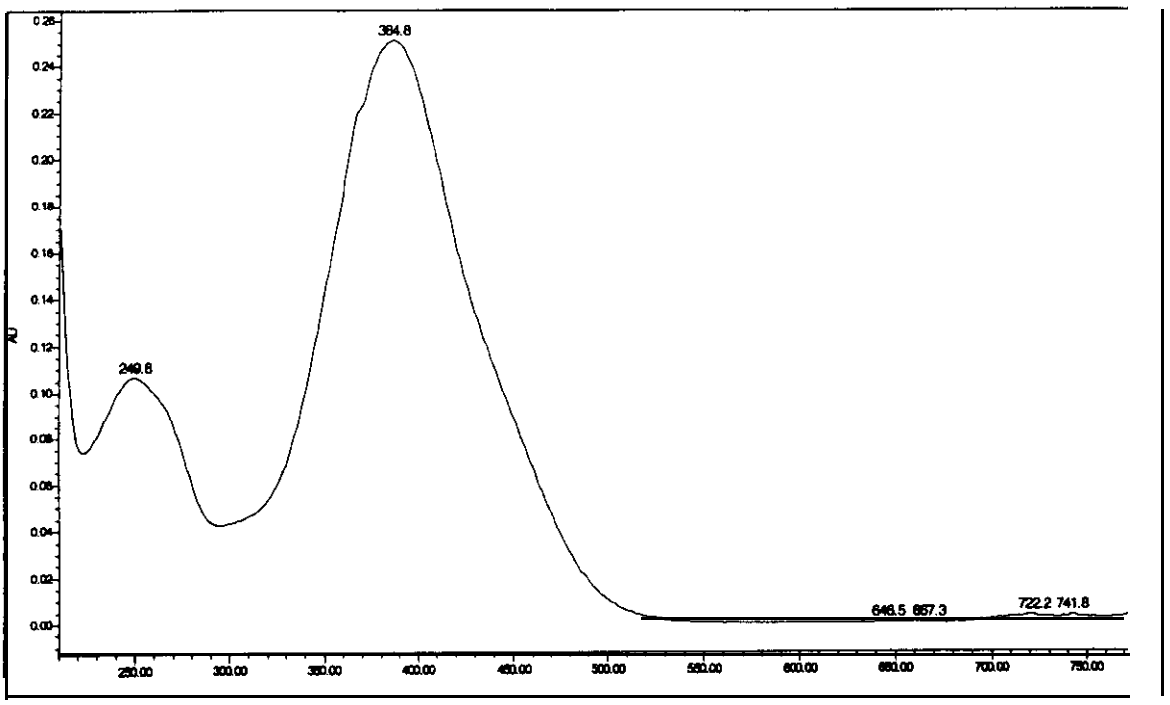

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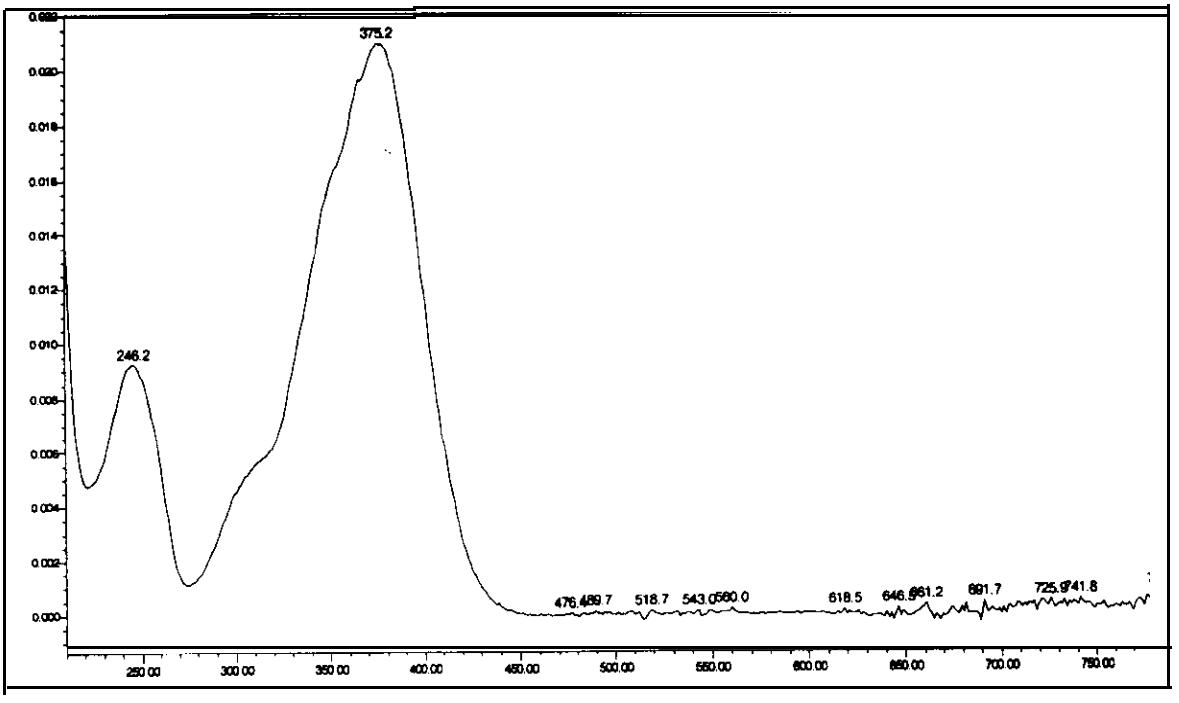

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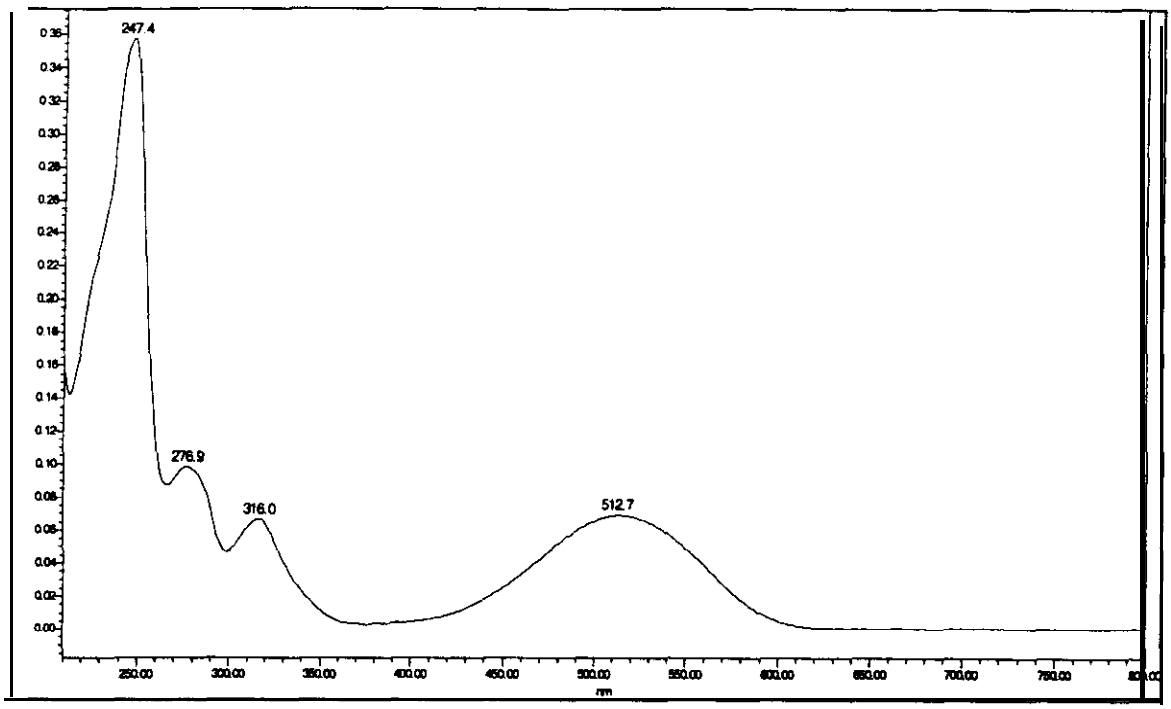

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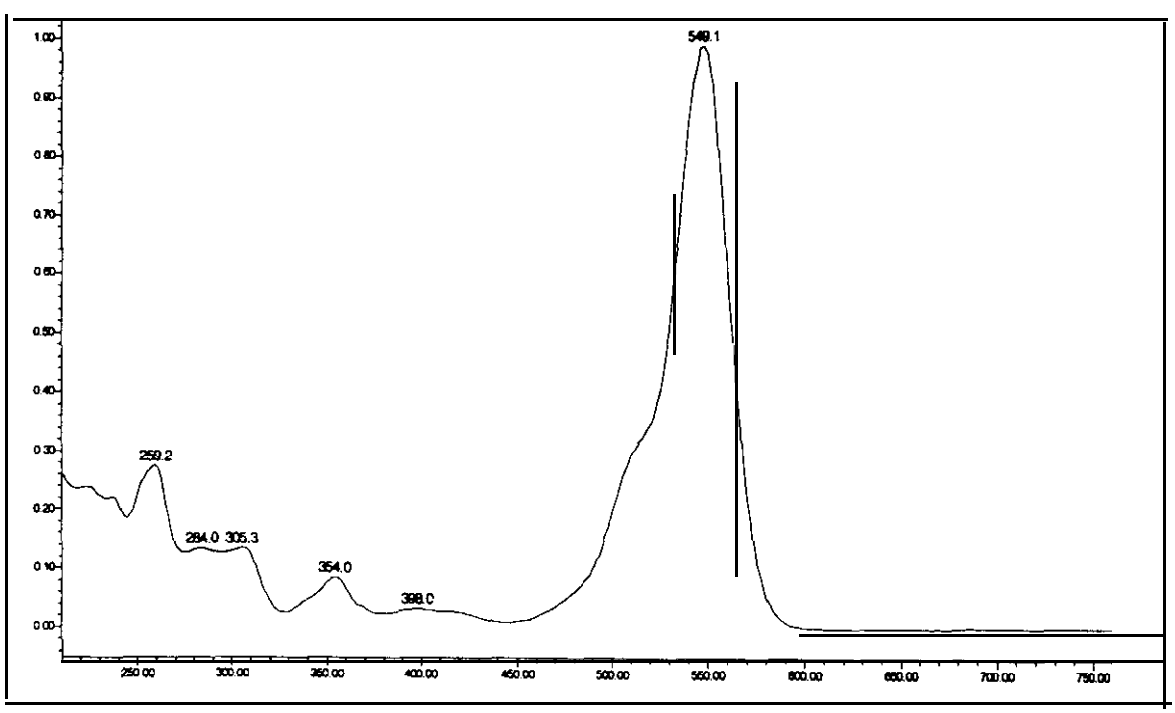

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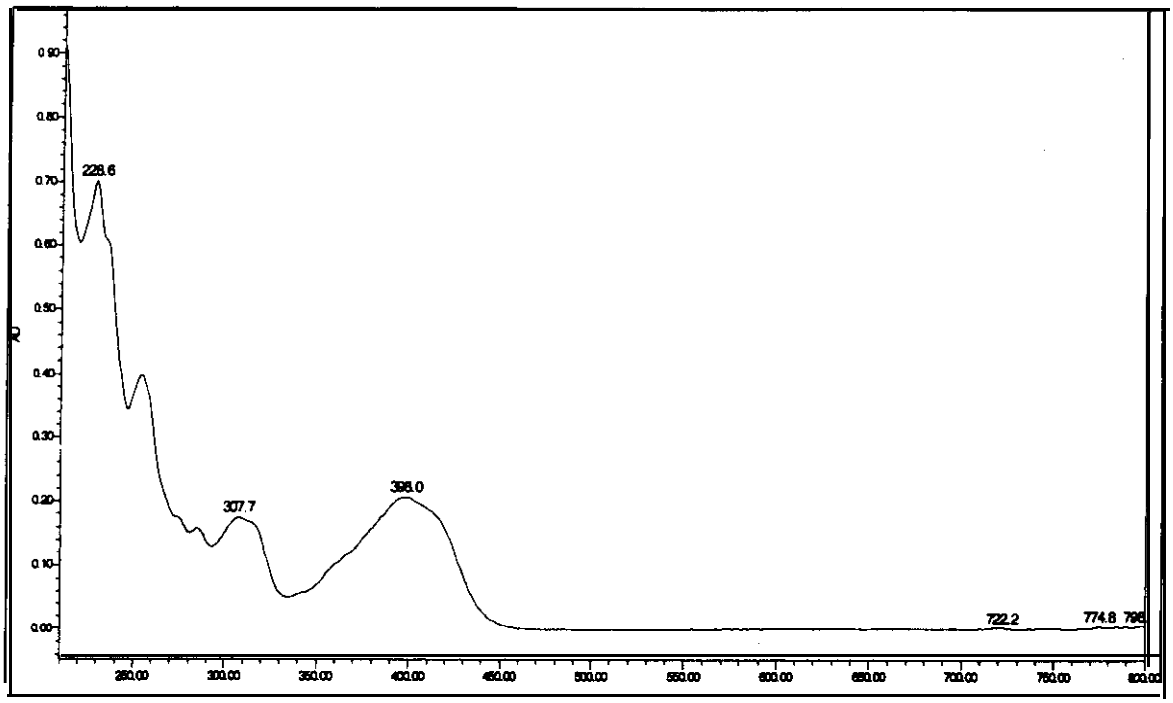

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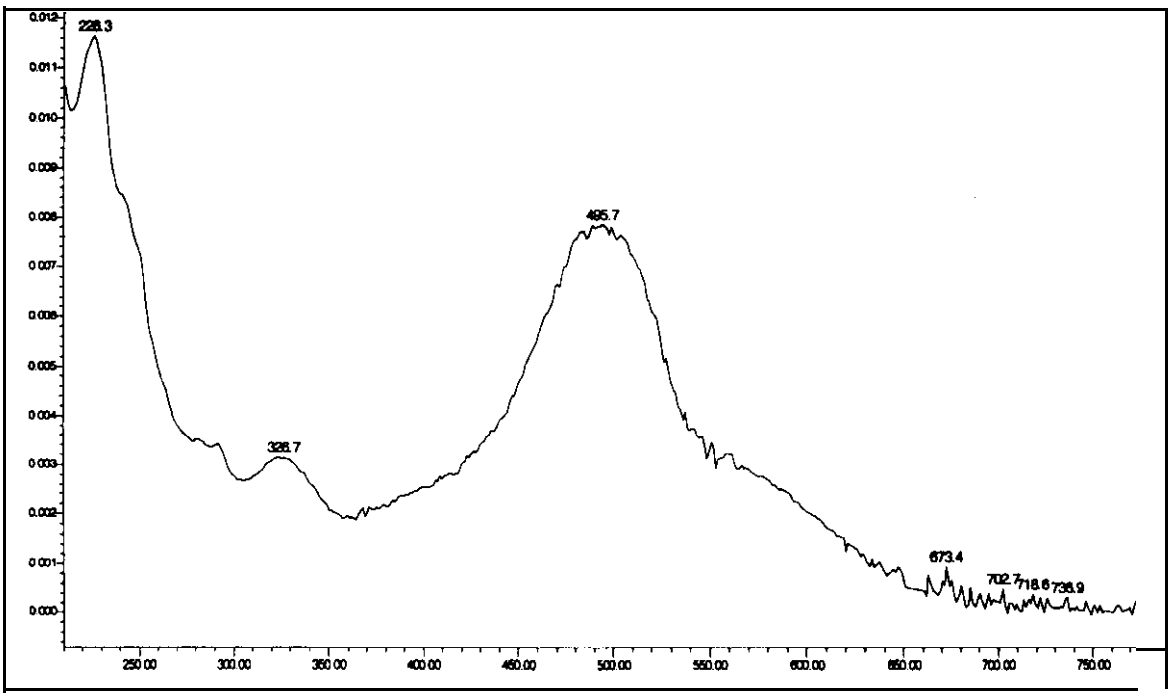

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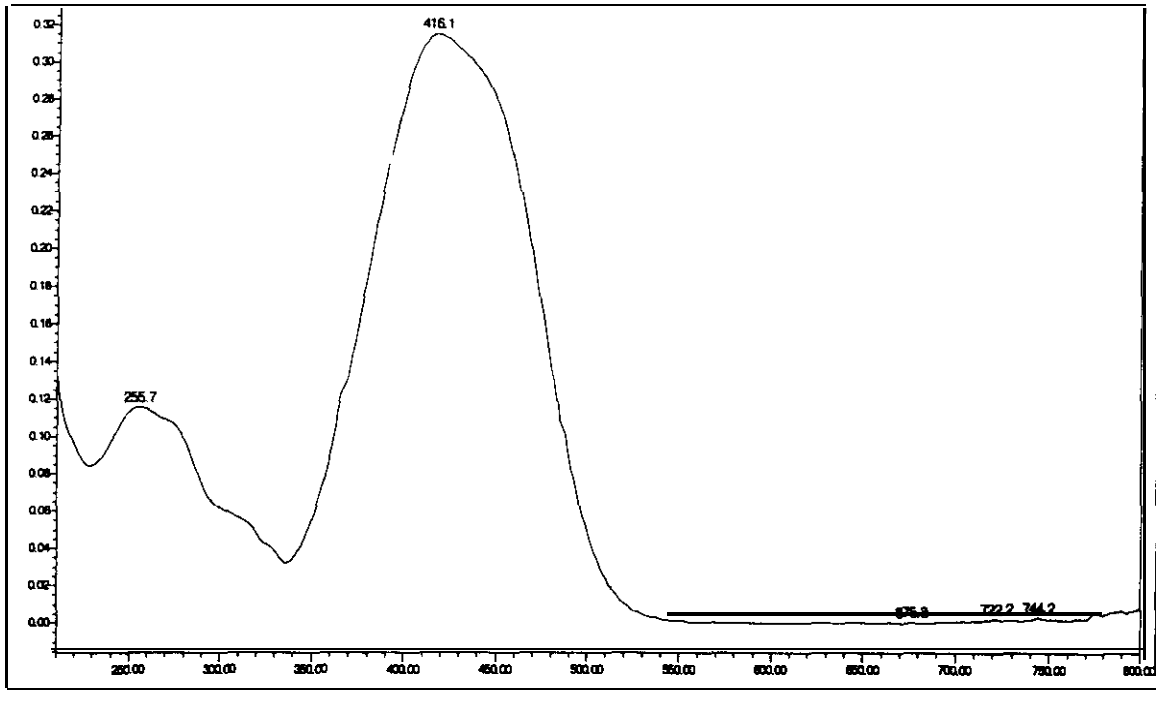

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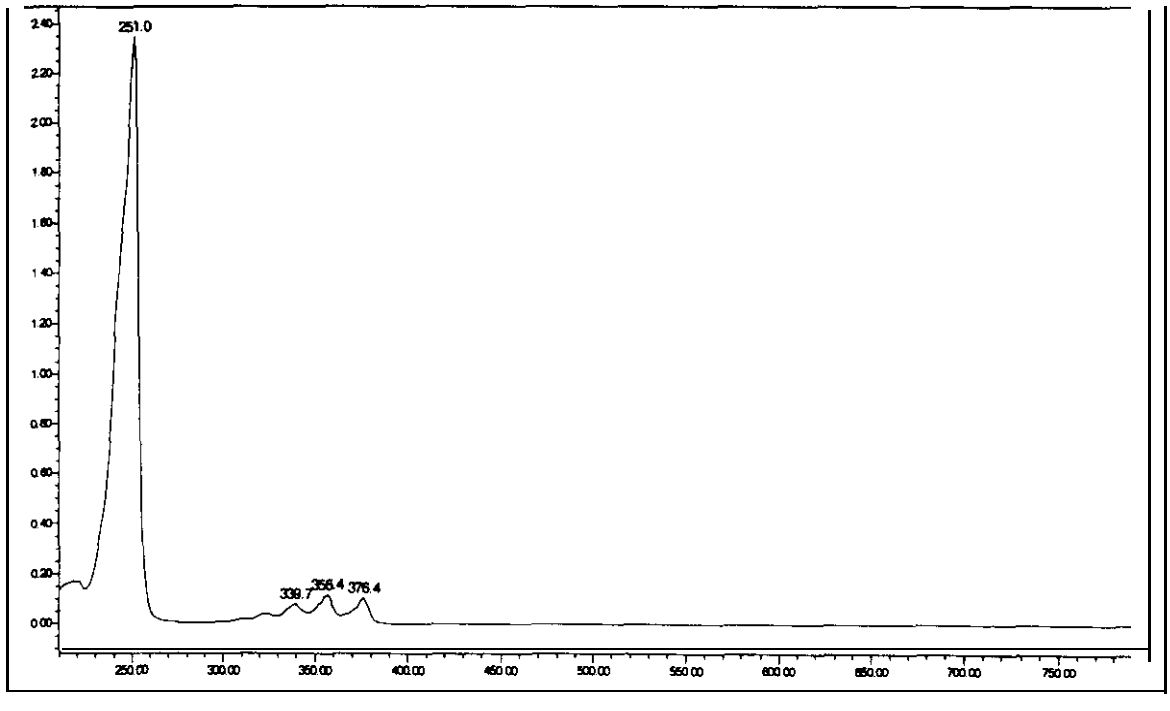

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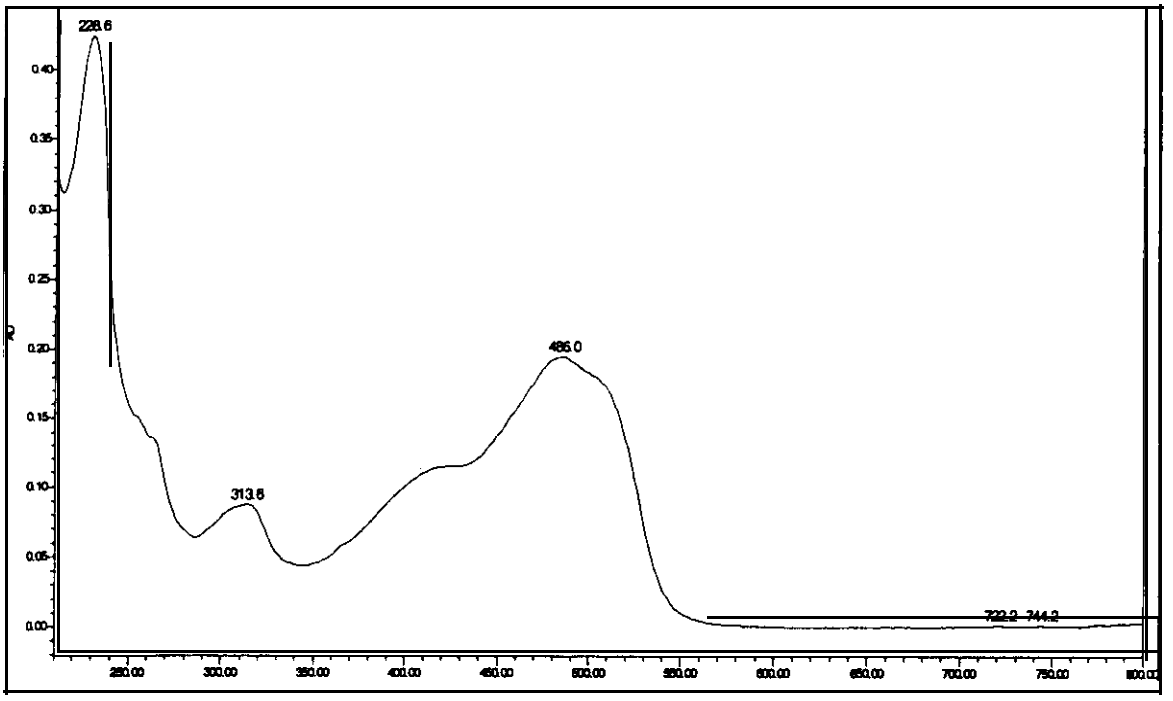

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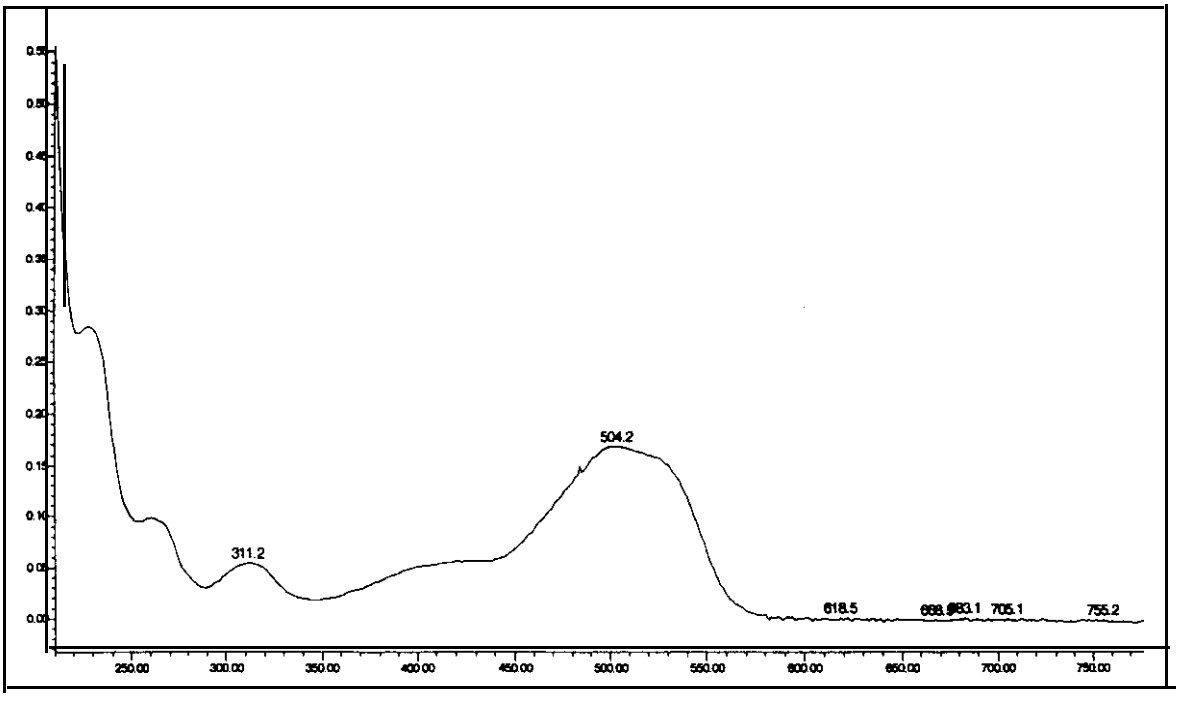
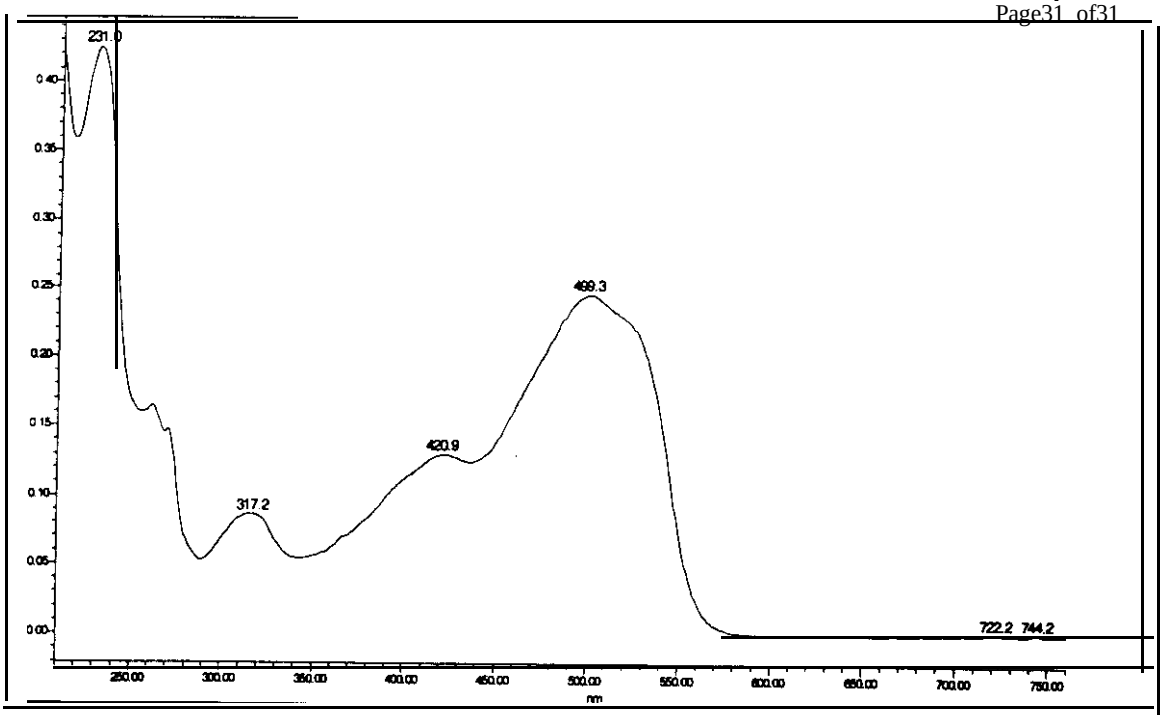
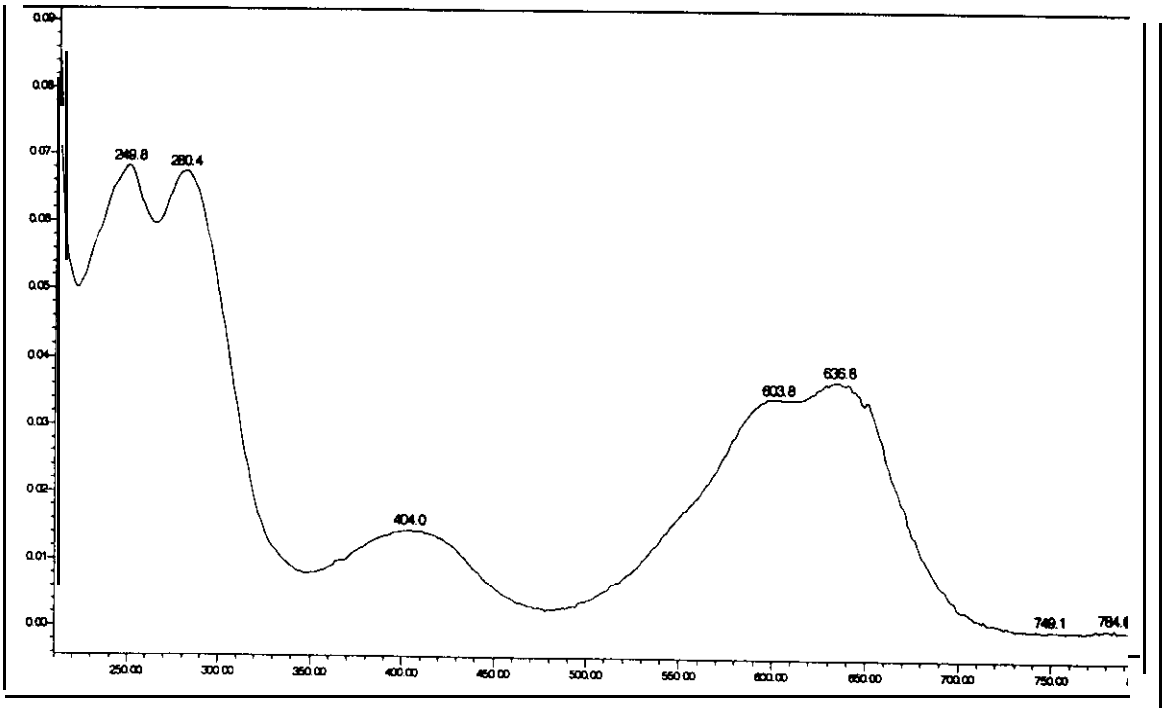


SR1 RT=45.358



SO7 RT=51.68



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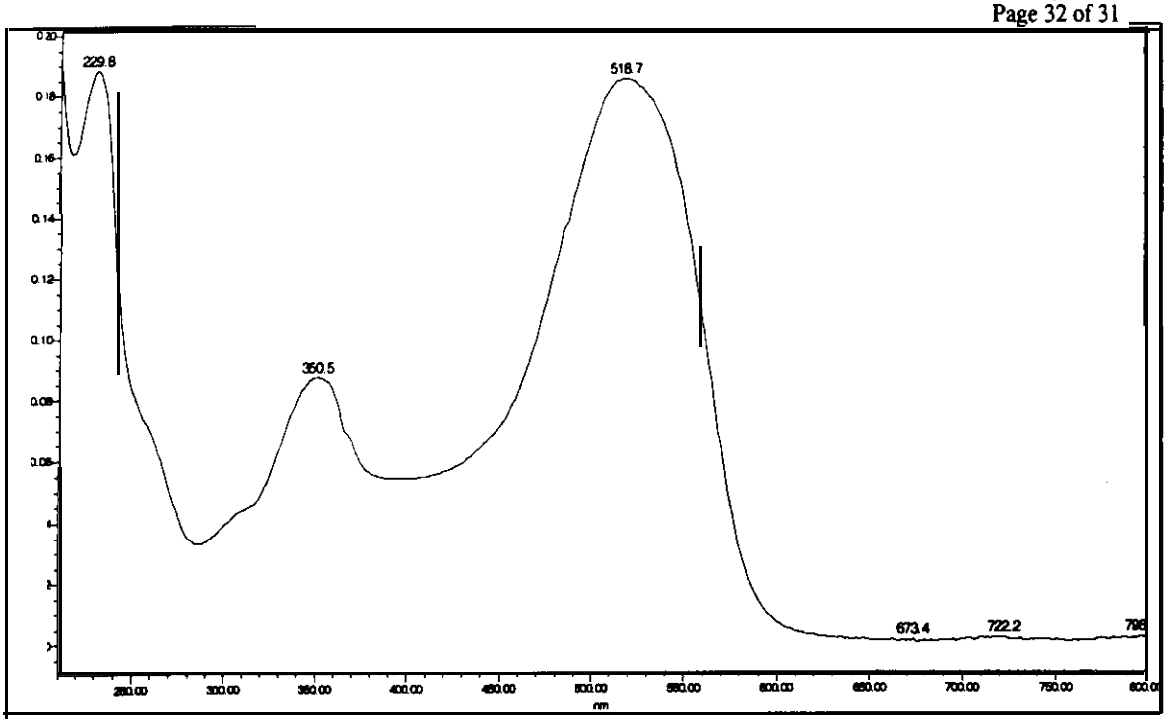

SR24 **RT=58.86**

TABLE D-1

SUMMARY OF CHEMICAL INFORMATION FOR DYES
SWMU 2 • DYE BURIAL GROUND
NSWC CRANE, INDIANA

Dye Name	CAS #	Crane Acronym	Color	Color Index #	Dye Classification	Trade/Chemical Name	Formula	Lab Standard Source
Water Soluble Dyes								
Acid Blue 1	12417-Q	AB1	blue	42045	Triarylmethane	Alphazurine 2G	$C_{27}H_{31}N_2NaO_8S_2$	ACROS
Acid Blue 9	3844-45-Q	AB9		42090	Triarylmethane	Alphazurine FG	$C_{37}H_{34}N_2Na_2O_9S_3$	Abbey Color
Acid Blue 45	286 102-I	AS45	blue	63010	Anthraquinone		$C_{14}H_8N_2Na_2O_{10}S_2$	Aldrich
Acid Red 64		AR64	red					ACROS
Acid Yellow 3	8004-92-0	AY3	yellow	47005	Quinoline	Quinoline yellow	$C_{18}H_8NNa_3O_{11}S_3$	ACROS
Acid Yellow 23	1934-21-o	AY23	yellow	19140	Monoazo	Tartrazine	$C_{16}H_9N_4Na_3O_9S_2$	ACROS
Acid Yellow 73	51 S-47-8	AY73	yellow(F)	45350	Xanthene	Fluorescien	$C_{20}H_{10}Na_2O_5$	ACROS
Acid Orange 10	1936-15-8	AO10	orange	16230	Monoazo	Orange G	$C_{16}H_{10}N_2Na_2O_7S_2$	ACROS
Basic Yellow 2	2465-27-2	BY2	yellow	41000	Diphenylmethane	Auramine hydrochloride	$C_{17}H_{22}ClN_3$	ACROS
Basic Violet 10	81-88-Q	BV10	pink(F)	46170	Xanthene	Rhodamine B	$C_{28}H_{31}ClN_2O_3$	ACROS
Organic Soluble Dyes								
1-Aminoanthraquinone	82-45-1	1-AQ	orange	37275	Anthraquinone	1-aminoanthraquinone	$C_{14}H_9NO_2$	ACROS
2-Aminoanthraquinone	117-79-3	2-AQ	orange		Anthraquinone	2-aminoanthraquinone	$C_{14}H_9NO_2$	Aldrich
Disperse Blue 14	2475-44-7	DB14	blue	61500	Anthraquinone	Celliton Fast Blue B	$C_{16}H_{14}N_2O_2$	Aldrich
Disperse Red 9	82-38-2	DR9	red	60505	Anthraquinone	1-(methylamino)anthraquinone	$C_{15}H_{11}NO_2$	Aldrich
Disperse Violet 1	128-95-0	DV1	purple	61100	Anthraquinone	1,4-diaminoanthraquinone	$C_{14}H_{10}N_2O_2$	Aldrich
Pigment Red 1	6410-10-2	PR1	orange	12070	Monoazo	1-(4-nitrophenylazo)-2-naphthol	$C_{16}H_{11}N_3O_3$	Fisher Scientific
Smoke Dye	81-64-1	SD	red streak	58050	Anthraquinone	Quinizarine	$C_{14}H_8O_4$	ACROS
Solvent Green 3	128-80-3	SG3	blue	61565	Anthraquinone	Quinizarine green	$C_{28}H_{22}N_2O_2$	ACROS
Solvent Orange 3	495-54-5	SO3	orange		Monoazo	Chrysoidine Y base	$C_{12}H_{12}N_4$	Abbey Color
Solvent Orange 7	3118-97-6	SO7	red/orange	12140	Monoazo	Calco Oil Scarlet (Sudan II)	$C_{18}H_{14}N_2O$	ACROS
Solvent Red 1	1229-55-6	SR1	red	12150	Monoazo	1-(2-methoxyphenylazo)-2-naphthol	$C_{17}H_{14}N_2O_2$	TCI
Solvent Red 24	85-83-6	SR24	red	26105	Disazo	Oil Red (Sudan IV)	$C_{24}H_{20}N_4O$	ACROS
Solvent Yellow 2	60-11-7	SY2	yellow	11020	Monoazo	Dimethylaminoazobenzene	$C_{14}H_{15}N_3$	Phaltz & Bauer
Solvent Yellow 3	97-56-3	SY3	yellow	11160	Monoazo	Fast Garnet gbc base	$C_{14}H_{15}N_3$	ACROS
Solvent Yellow 14	84207-S	SY14	orange	12055	Monoazo	Sudan I	$C_{16}H_{12}N_2O$	Aldrich
Solvent Yellow 33	8003-22-3	SY33	yellow	47000	Quinoline	Quinoline yellow	$C_{18}H_{11}NO_2$	ACROS

TABLE D-2

SUMMARY OF AVAILABLE TOXICITY INFORMATION
SWMU 2- DYE BURIAL GROUND
NSWC CRANE, INDIANA

Dye Name	Sources of Information												
	NTP C&S	NTP-PUB	OSHA	CAL-EPA	EPA-FACT	SCORECARD	ECDIN	JAPAN	IRAC	IRAC MONO.	RETECS	HSDB	CCRIS
Water Soluble Dyes													
Acid Blue 1	X	X				X	X		G3	X	BP6830000	X	X
Acid Blue 9								X	G3		BQ4725000		
Acid Blue 45													
Acid Red 64													
Acid Yellow 3							X				GC5796000		
Acid Yellow 23						X	X	X			UQ6400000		X
Acid Yellow 73	X	X					X				LM5425000		X
Acid Orange 10	X	X					X		G3	X	GJ6500000	X	X
Basic Violet 10			X	P-65		X	X	X	G3	X	BP3675000	X	X
Basic Yellow 2	X	X			X	X	X	X	G2B		BY3675000	X	X
Organic Soluble Dyes													
1-Aminoanthraquinone							X				CB5075000		
2-Aminoanthraquinone	X	X		P-65/CRICAR	X	X	X	X	G3	X	CB5120000	X	X
Disperse Blue 14							X						
Disperse Red 9							X			X			
Disperse Violet 1							X				CB6300000		X
Pigment Red 1							X				QL4510000		
Smoke Dye											CB6600000		
Solvent Green 3							X				CB5775000		
Solvent Orange 3													
Solvent Orange 7						X	X		G3	X	QL5850000	X	X
Solvent Red 1							X					X	X
Solvent Red 24							X	X	G3	X	QL5775000		X
Solvent Yellow 2	X	X	X	P-65/CRICAR	X	X	X		G2B	X	BX7350000	X	X
Solvent Yellow 3	X	X		P-65/CRICAR		X	X		G2B	X	XU8800000	X	X
Solvent Yellow 14	X	X	X	P-65		X	X		G3	X	QL4900000	X	X
Solvent Yellow 33	X	X					X				GE5925000		X

*NTP C&S & PUB	http://ntp-server.niehs.nih.gov/
*OSHA	http://www.osha.gov/
CAL-EPA	CRIC http://www.oehha.org/docs/covrttrb.htm P-65 http://www.oehha.org/prop65/
EPA FACT	http://www.mail.odsnet.com/TRIFacts/ - TOXIB45
SCORECARD	http://scorecard.org/ - TOXIB47
*ECDIN	http://www.ecdin.etomep.net/
JAPAN	http://www.citi.or.jp/
IRAC	http://193.51.164.11/ - TOXIB53
*TTN-WEB	http://www.epa.gov/ttn/
*HSDB & CCRIS	http://toxnet.nlm.nih.gov/ - TOXIB58
*Chemfinder	http://www.chemfinder.com
*NIST Chembook	http://webbook.nist.gov/chemistry/
*SW-846 On-line	http://www.epa.gov/epaoswer/hazwaste/test/main.htm
*SW-846 Update 4A	http://www.epa.gov/epaoswer/hazwaste/test/up4a.htm
Internet Grateful Med	http://130.14.32.45/cgi-bin/

* Sites which contain physical/chemical/analytical information

Project Schedule

MINUTES
PRE-QAPP MEETING
SWMU 2 – DYE BURIAL GROUND
NSWC CRANE, INDIANA
MARCH 9, 2000

ATTENDEES:

Bill Gates – Navy
Tom Brent – NSWC Crane
Jackie Vorhauer – NSWC Crane Lab
Nancey Maegerlien – NSWC Crane Lab
Forrest Hawman – NSWC Crane Lab
Sara Poehlein – NSWC Crane Lab
Allen Debus - EPA
Dan Mazer - EPA
Marion Mangino - EPA
Peter Ramanakus - EPA
Karen Smecker – TtNUS. Inc
Linda Karsonovich – TtNUS,Inc.
Terrie Baranek – ECT.CON, Inc.

INTRODUCTION: RCRA Facility Investigation (RFI), risk assessment, and Corrective Measures Study (CMS) activities need to be completed for Solid Waste Management Unit (SWMU) 2. the Dye Burial Ground at Naval Surface Warfare Center (NSWC) Crane, Indiana. Although several phases of RFIs have been conducted at the site, the nature and extent of contamination has not been defined. One field sampling effort is planned to address the needs of the RFI, risk assessment, and CMS. Tetra Tech NUS, Inc. (TtNUS) is currently preparing the Internal Draft Planning Documents for SWMU 2. The NSWC Crane Explosives Science Laboratory is currently developing analytical methods for dye constituents.

Because of inherent complexities associated with SWMU 2. two meetings with EPA were held previously to discuss significant issues such as project objectives, dye toxicity data, and dye analytical method development. A pre-pre-QAPP meeting was held on May 13, 1999 and a meeting to specifically discuss the dye method development was held on September 10, 1999.

MEETING MINUTES:

Decision Rule Flowchart - A decision rule flowchart was presented (Figure B-l) which was designed to meet the project objectives.

- EPA proposed the addition of another step in the flow chart to eliminate specific chemicals from consideration in addition to the step eliminating individual SWMUs from consideration if no "unacceptable" risks are found at the SWMU. Dan Mayer also inquired if the same decision flowchart was to be used for ecological receptors. Karen Smecker answered affirmatively.

Proposed Sampling Plan . A conceptual site model (CSM) was developed for the site (Figure B-2). Based on the CSM the proposed sampling plan was summarized in Table B-l.

- EPA comments included a request that the objective for inorganic sampling be added into the table since historically the objective for inorganic sampling is different. Also that TtNUS check U.S. EPA

Region 5 guidelines for use of historical data in the RA. Karen Smecker indicated that TtNUS was not recommending that the historical data be used in the RA since the quality of the data was not known. These data would be used qualitatively to support conclusions drawn by current data. EPA also inquired what would be done if dyes were not found during the screening analysis. Karen indicated that according to the decision rule flowchart dyes would be considered as not present (minimal levels) and no further action would be taken. Screening will be done only for soil samples.

. EPA also questioned turnaround lime and the need for more than one mobilization. Karen replied that the screening methods were developed with the need for a two-day turnaround as an objective since only one mobilization was scheduled.

Analytical_Program - Tables B-2 (dyes) and B-3 (other parameters) provided the list of the analytical methods and parameters lo be included in the analytical program for SWMU 2. TtNUS has worked closely with the subcontracted analytical laboratory to select and optimize methods in an effort to attain, to the greatest extent possible, laboratory reporting limits (RLs) at concentrations less than or equal to the risk-based target levels. Preliminary instrument detection limits and method detection limits for aqueous and solid matrices for the due analytical methods are summarized in Table B-2. Preliminary risk-based target levels for dyes are also included in Table B-2.

Thin-Layer Chromatography (TLC) - TLC is the proposed screening method for dyes. A preliminary standard operating procedure (SOP) was provided in Attachment C.

. EPA questioned the basis for the initial/continuing calibration standards. Sarah replied that the initial calibration would include all compounds; however, the continuing calibration would contain only six compounds. The six compounds selected were characteristic of both groups (water soluble and organic soluble dyes) and would provide adequate proof of continued method performance. EPA also questioned how method detection limits (MDLs) were developed. Sara replied that MDLs were developed based on the ability to see the dyes either visually or by enhancement (i.e., fluorescence/computer manipulation). EPA asked Sara to include a explanation of the concept for developing detection limits in the SOP.

High Performance Liquid Chromatography (HPLC) - HPLC is the proposed quantitative method. A preliminary SOP was provided in Attachment C.

. EPA inquired if the laboratory had achieved extraction of the dyes from soil and water matrices. Forrest replied that the lab had developed aqueous extraction procedures but were having trouble developing procedures for extraction from solid matrices. Several of the dyes (i.e., acid blue 1 and 9) bond preferentially to the soils and are extremely difficult to get into solution. Forrest also pointed out

that this meant that the dyes would be unlikely to migrate into the groundwater. Forrest also indicated that the lab was in the process of evaluating the use of Solid Phase Extraction (SPE) techniques for solid matrices and that this would impact the reporting levels presented in Table B-2.

. EPA inquired as to the use of second column confirmation for the presence of dyes. Forrest indicated that they were not considering the use of a second column due to the difficulty in finding columns that could separate all the dyes. Forrest also indicated that the use of the photo-diode array detector (PDA) allowed for the use of the sample absorption spectra as confirmation.

- EPA asked ii the laboratory had used this technique using individual standards and known standard mixtures. Forrest replied that they had run both individual standards and mixtures of all the compounds in both clean matrices and in matrices taken from the site. EPA asked about interferences. Forrest indicated that they have not had a problem with interferences even in samples taken from the site. Forrest also pointed out the dyes are one of the few organic compounds which absorb above 500 nm and that this is a good criteria for screening out interferences.
- EPA indicated that they would like to see a sample package containing actual samples/quantitation/calibration/MDL information for both the HPLG and TLC.
- Forrest raised a question concerning whether the MDL should be based on the chromatogram or on the spectra. Several dyes do not have spectra at the lowest point (i.e., a peak can be seen on the chromatograph but the detector cannot generate a spectra). EPA indicated that the issue would need to be addressed and asked TtNUS to develop a data qualifier proposal. EPA also indicated that they would like to see example spectrum for each case. It was tentatively agreed that if the MDL was below the screening criteria, the MDL would be used. However, ii the MDL was above the screening criteria, the IDL would be used with a qualifier.

. EPA also questioned the use of surrogates. Forrest indicated that benzanthrone would be used as a surrogate and that he intended to use anthracene as a retention time marker due to the extended length of the analytical run (60 minutes).

Toxicity Data for Dves and *Risk Assessment* - Preliminary risk-based target levels for dyes were included in Table E-2. These values were calculated by TtNUS using available toxicity data complied as a result of a literature search. When available, toxicity data were used to calculate a reference dose using methodology presented in risk assessment guidance. Target levels were then calculated using EPA Region IX methodology for determining preliminary remediation goals (PRGs).

. EPA indicated that they would like to see the TtNUS methodology for developing the risk-based target levels. EPA also questioned how the data had been searched. Linda indicated that the data had been searched using the CAS number, since many dyes had similar commercial and/or user names. Karen indicated that TtNUS would send the PRG information ASAP since the target levels would impact the need for continued analytical development. The EPA also inquired ii the literature search

had indicated any dyes were used as cosmetic or food additives. Jackie indicated that yes several of the dyes were but did not know the specific dyes.

. EPA also requested that TtNUS develop preliminary ecological numbers and provide this information to EPA.

GUIDANCE FOR

METHODS DEVELOPMENT

AND

METHODS VALIDATION

FOR THE

RCRA PROGRAM

PREFACE AND OVERVIEW

Test Methods for Evaluating Solid Waste, or SW-846, is the compendium of analytical and test methods approved by EPA's Office of Solid Waste (OSW) for use in determining regulatory compliance under the Resource Conservation and Recovery Act (RCRA). SW-846 functions primarily as a guidance document setting forth acceptable, although not required, methods to be implemented by the user, as appropriate, in responding to RCRA-related sampling and analysis requirements.

There seems to be an impression among methods developers and the regulated community that there is some esoteric or mystical process that must be followed in order to get an analytical method "approved" by regulatory agencies like the USEPA. In this document., OSW would like to dispel these misconceptions, identify some basic principles, and present a logical approach to methods development that is currently followed by OSW in developing methods for SW-846. This approach is based on sound scientific principles, and methods developed according to this process should be acceptable for use in other Agency programs as well as OSW.

Two levels of methods development are covered in this guidance document, initial "proof of concept" and a formal validation. This guidance is applicable to both new methods submitted for potential inclusion in SW-846 or for adapting existing SW-g46 methods for additional applications. When measurements for RCRA applications are required for which no validated methods exist, e.g., from unusual matrices or below the quantitation limits of conventional SW-846 or other appropriate methods, qualified analysts can serve as "in-house" methods developers to modify existing methods to meet these regulatory needs following the guidelines delineated in Elements 1 through 9.

The RCRA method development approach utilizes three basic principles for either demonstrating "proof of concept" or for use in a formal validation. These basic scientific principles are:

1) Identify the scope and application of the proposed method, (What is this method supposed to accomplish?)

2) Develop a procedure that will generate data that are consistent with the intended scope and application of the method, and

3) Establish appropriate quality control procedures which will ensure that when the proposed procedure is followed, the method will generate the appropriate data from Step 2 that will meet the criteria established in Step 1.

In some cases, such as a variation of an existing SW-846 method using new equipment or a modified procedure, it is sufficient only to demonstrate validation to the "proof of concept" stage. For new technologies to be considered for inclusion in SW-846, it is necessary for the developer to perform the formal validation procedure including multilaboratory validation.

DEVELOPMENT AND

VALIDATION OF

SW-846 METHODS

PHASE 1:

PRELIMINARY VALIDATION

OR

DEMONSTRATION OF

"PROOF OF CONCEPT"

DEVELOPMENT AND VALIDATION OF SW-846 METHODS
PHASE I: PRELIMINARY VALIDATION OR "PROOF OF CONCEPT"

The two documents included in this section are letters that were requested by potential methods developers on bow to begin a methods development project for submission to OSW for inclusion in SW-846. One letter (dated April 6, 1992) addresses a preliminary validation or "proof of concept" for new sample preparation methods, while the other (dated April 23, 1992) addresses a preliminary validation or "proof of concept" for new screening methods.

A shortened list of target analytes, representative of typical RCRA analyte classes for volatile organics, semivolatile organics, pesticides, and metals were included. These target analyte lists provide a range of target analyte performance within existing methods. The idea in this preliminary stage was that if the potential new method could successfully analyze the target analyte lists included in these documents, it had potential applicability to successfully analyze the compounds on the extended RCRA target analyte lists. These guidance documents also recommend using multiple matrices, e.g., groundwater, TCLP leachate, and wastewater for aqueous matrices, sand, loam, and clay for soil matrices, and multiple spiking concentrations to demonstrate appropriate method performance.

If a new technique performs adequately in this "proof of concept" phase, it is ready for the formal validation process. In this way, by doing a preliminary method development screening, the developer can easily determine if it is worth continuing to invest in a project without a large outlay of time or money.

In some cases, e.g., development of alternative equipment for an existing method, this preliminary validation may be all that is necessary to demonstrate adequate method performance for the intended method application.

April 6, 1992

Dear Colleague:

The Methods Team of the Office of Solid Waste is responsible for the promulgation of rugged and reliable analytical techniques in support of the Resource Conservation and Recovery Act (RCRA) program. The methods published in Test Methods for Evaluating Solid Waste, SW-846, are used to measure the concentration of specific pollutants or to establish whether a waste stream demonstrates a hazardous characteristic (e.g. ignitability, corrosivity, reactivity or toxicity).

A number of sources have developed new methods for preparing samples that could have application for the RCRA program. The U.S. EPA is eager to adopt any new techniques that provide high quality data in a reliable, reproducible and cost-effective manner. This letter provides developers with a description of the type of performance data that is required for an effective initial evaluation of new SW-846 methods. If a developer's data supports adoption of a new method, the Methods Team will work through the SW-846 Work Group process to promulgate it. This letter does not supersede or replace the more rigorous requirements described in Test Method Equivalency Petitions, EPA/530-SW-87-008, OSWER Policy Directive No. 9433.00-2 (2/87). That document provides the requirements for a method equivalency petition which may be used to promulgate a method outside of the Work Group process.

In order to evaluate the performance of sample preparation procedures, data should be submitted for samples prepared using the proposed technique, and analyzed using approved SW-846 quantitative methods. Widely used, multi-analyte procedures such as Method 8270 (GUMS for semi-volatiles), Method 8081 (GC/ECD for organochlorine insecticides), Method 8260 (GC/MS for volatiles), or Method 6010 (ICP/AES for metals) should generally be employed. New sample preparation techniques need not be validated for the entire target list of a multi-analyte method, but a representative selection of targets should he measured. Examples of representative target analytes and some of the rationale for selecting them are provided in the attachments.

Developers should analyze three different types of matrices. Samples that are analyzed should either he characterized reference 'materials or spiked matrices containing known amounts of target analytes. In either case, bulk samples should be carefully homogenized to reduce sub-sampling errors. The sample matrices should be selected to represent what is regulated under RCRA (e.g. soil, oily waste or waste waters), not to provide the best performance data. Blanks should be analyzed with each set of samples.

Method performance is established by analyzing seven replicate aliquots of three different sample matrices spiked at two concentrations. Suggested spiking levels are 5 times the lower quantitation limit for the preparation/analysis method and 50 times the low concentration. As an alternative, specific concentrations for selected target analytes in soil are

provided in the attachments to this letter. The low values are normal reporting limits for routine analyses and the high value is 50 times the low. Recovery, precision, and matrix method detection level must he calculated. [redacted]y) is determined at both concentrations by calculating the [redacted] of the spiked (or characterized) analytes for the seven replicates.

[redacted]

Method precision is determined by calculating the [redacted] of the spiked analyte recoveries for the seven replicates [redacted] each concentration.

[redacted]

Tbe U.S. EPA requires methods that provide reliable measurements at or below regulatory action levels. The lowest level at which accurate quantitation can be attained must be determined for each matrix. To determine the Reliable Quantitation Limit (RQL), the Method Detection Limit (MDL) is first calculated from the I [redacted] termined from seven replicate measurements (six degrees of freedom):

[redacted]

The RQL is calculated using the following equation:

[redacted]

To summarize, the Methods Team does not require an unreasonable body of data for the initial evaluation of new techniques. With the additional requirement of one blank per matrix, 45 samples will have to be prepared and analyzed to complete the table below.

Type of sample	bias	precision	MDL	RQL
Matrix 1, low				
Matrix 1, high				
Matrix 2, low				
Matrix 2, high				
Matrix 3, low				
Matrix 3, high				

It is our belief that completion of this table represents only a small fraction of a developer's effort in developing a new method. If your technique will improve the quality of EPA monitoring programs, submitting these data should expedite the SW-846 approval process.

I look forward to working with you on this important activity.

Sincerely,

Barry Lesnik
Organic Methods Program Manager
USEPA Office of Solid Waste
Methods Team (5307W)

attachments

REPRESENTATIVE ORGANOCHLORINE PESTICIDES

The following chlorinated pesticides from the Best Demonstrated Available Technology (BDAT) list are representative of analytes for Method 8081. All should be sufficiently resolved to ensure good quantitation using two columns such as the DB-608 or DB-1701 (or equivalents). Suggested low and high concentrations should be appropriate for relatively uncontaminated solid matrices such as soil. Higher concentrations may be required for highly contaminated samples.

COMPOUND	LOW (µg/kg)	HIGH (µg/kg)
Aldrin	5	250
β-BHC	5	250
δ-BHC	5	250
y-BHC (Lindane)	5	250
α-Chlordane	5	250
y-Chlordane	5	250
4,4'-DDD	10	500
4,4'-DDE	5	250
4,4'-DDT	5	250
Dieldrin	5	250
Endosulfan I	5	250
Endosulfan II	5	250
Endrin	10	500
Endrin aldebyde	5	250
Heptachlor	5	250
Heptachlor epoxide	5	250

REPRESENTATIVE SEMIVOLATILE COMPOUNDS

The following compounds are representative of analytes for Method 8270. Although many of these compounds are considered difficult, all can he extracted from waste matrices using conventional techniques. Suggested low and high concentrations should be appropriate for relatively uncontaminated solid matrices such as soil. Higher concentrations may be required for highly contaminated samples. Phthalate esters are not spiked hut should be reported as a measure of contamination.

COMPOUND	LOW (µg/kg)	(µg/kg)
Phenol	250	12,500
o-Cresol	250	12,500
2-Methyl phenol	250	12,500
2,4,6-Trichlorophenol	250	12,500
Pentachlorophenol	250	12,500
1,2-Dichlorobenzene	250	12,500
Naphthalene	250	1290
2-Chloronaphthalene	250	12,500
Anthracene	250	12,500
Chrysene	250	12,500
Benzo(a)anthracene	250	12,500
Benzo(a)pyrene	250	1290
Fktoranthene	250	12,500
Indeno(1,2,3-cd)pyrene	250	12,500
Benzo(g,h,i)perylene	250	12,500
o-Toluidine	250	12,500
p-Nitrotoluene	250	12,500
2,6-Dinitrotoluene	250	12,500
2-Nitroaniline	250	12,500
Di-n-propylnitrosamhte	250	12,500
4-Bromophenyl phenyl ether	250	12,500
3,3'-Dichlorobenzidine	250	12,500

In addition, surrogates should he added such that 50 µg or 100 µg would be present in final extracts assuming 100% recovery.

Nitrobenzene-d_5	50
2-Fluorobiphenyl	50
p-Terphenyl-d,,	50
Phenol-d,	100
2-Fluorophenol	100
2,4,6-Tribromophenol	100

REPRESENTATIVE VOLATILE COMPOUNDS

The following compounds are representative of analytes for Method 8260; many are TCLP analytes. All can be purged from waste matrices using Methods 5030 or 5035. Suggested low and high concentrations should be appropriate for relatively uncontaminated solid matrices such as soil. Higher concentrations may be required for highly contaminated samples.

COMPOUND	LOW (µg/kg)	HIGH (µg/kg)
Vinyl chloride	5	250
Dibromochloromethane	5	250
1,1-dichloroethane	5	250
Chloroform	5	250
Carbon tetrachlorlde	5	250
Trichloroethylene	5	250
1,1,1-Trichloroethane	5	250
Benzene	5	250
Toluene	5	250
Ethylbenzene	5	250
Chorobenzene	5	250
Nitrobenxene	5	250
Methyl ethyl ketone	5	250
Carbon disulfide	5	250

The following compounds are representative of non-purgeable volatiles which currently require azeotropic distillation. Suggested low and high concentrations should be appropriate for relatively uncontaminated solid matrices such as soil. Higher concentrations may be required for highly contaminated samples.

COMPOUND	LOW (µg/kg)	HIGH (µg/kg)
1,4-Dioxane	10	500
n-Butanol	10	500
iso-butanol	10	500
Ethyl acetate	10	500
Pyridine	10	500

REPRESENTATIVE METALS

The following metals include the TCLP analytes except mercury. Suggested low and high concentrations are based on ICP analysis and should be appropriate for relatively uncontaminated solid matrices such as soil. Higher concentrations may be required for highly contaminated samples.

ELEMENT	LOW (µg/kg)	HIGH (µg/kg)
Arsenic	2500	75,000
Barium	100	5,000
Cadmium	200	10,000
Chromium	350	17,500
Copper	300	15,000
Lead	2ooo	100,000
Silver	350	17,500
zinc	100	5,000

Mercury is generally analyzed by cold vapor AA.

Mercury	100	5,000

April 23, 1992

Dear Colleague:

The Methods Team of the Office of Solid Waste is responsible for the promulgation of rugged and reliable analytical techniques in support of the Resource Conservation and Recovery Act (RCRA) program. The methods published in Test Methods for Evaluating Solid Waste, SW-846, are used to measure the concentration of specific pollutants or to establish whether a waste stream demonstrates a hazardous characteristic (e.g. ignitability, corrosivity, reactivity or toxicity).

SW-g46 currently provides reliable and sensitive laboratory methods for the analysis of Appendix VIII anaiytes. However, some of these methods may be too costly or require too much analysis time for some applications. The Methods Team also recognizes the savings that could be achieved by sending only contaminated samples to analytical laboratories for quantitative analysis. Therefore, the Methods Team has recognized the need for more rapid, less expensive field screening procedures.

A number of sources have developed reliable, reproducible and cost-effective field or screening procedures with potential application for the RCRA program. Thii letter provides developers with a description of the type of Performance data that is required for an effective initial evaluation of screening or field procedures. If a developer's data supports adoption of a new method, the Methods Team will work through the SW-g46 Work Group process to promulgate it. This letter does not supersede or replace the more rigorous requirements described in Test Method Eauivalencv Petitions, EPA/530-SW-87-008, OSWER Policy Directive No. 9433.00-2 (2/87). That document provides the requirements for a method equivalency petition which may be used to promulgate a method outside of the Work Group process.

While screening procedures need not be fully quantitative, they should measure the presence or absence of target analytes at or below regulatory action levels. Therefore, initial demonstration of method performance involves measuring the percentage of false negatives and false positives generated using the procedure for a single sample matrix. Data should be submitted for split samples analyzed using the developer's technique and an approved SW-g46 quantitative method. A candidate procedure should ideally produce no false negatives. Detinition of a false negative is a negative response for a sample that contains the target analyte(s) at or above the stated action level of the method. A candidate procedure should produce no more than 10% false positives. Definition of a false positive is a positive response for a sample that contains the target analyte(s) below the stated action level of the method. Between 20 and 50 samples spiked at one half the detection level should be tested to establish the percentage of false positives. Between 20 and 50 samples spiked at twice the detection level should be tested to establish the percentage of false negatives. It is recommended that a sufficient volume of each spiked sample be prepared to complete each test with one lot of material. Sufficient randomly selected aliquots of each spiked matrix should be analyzed by

appropriate SW-846 methods to demonstrate sample homogeneity and to characterize the sample in terms of target analytes and potential interferences.

A separate study should also be conducted to establish the effect of non-target interferences. A screening procedure should produce no more than 10% false positives for a set of 20 samples that contains a 100 fold excess of interferences. Positive interferences should be selected that are chemically related to the target analytes and are environmentally relevant. Negative interferences (i.e. masking agents) should also be investigated whenever they are suspected.

Developers should also analyze three different types of samples to provide matrix-specific performance data. These samples should either be characterized reference materials or spiked matrices containing known amounts of target analytes. In either case, bulk samples should be carefully homogenized to reduce sub-sampling errors. The sample matrices should be selected to represent what is regulated under RCRA (e.g. soil, oily waste or waste waters), not to provide the best performance data. Blanks should be analyzed with each set of samples.

Matrix-specific performance data, including detection limits and dynamic range, are gathered by analyzing ten replicate aliquots of three different sample matrices spiked at two concentrations. If spiked samples are used, suggested spiking levels are the matrix-specific detection limit and 50 times the detection limit. Positive or negative results should be reported for the low concentration samples. Results for high concentration samples should be reported as either semi-quantitative results or as positive/negative with the dilution factor used for the samples. As an alternative to establishing matrix-specific detection limits, specific spiking concentrations are provided for selected target analytes in the attachments to this letter. The low values are normal reporting limits for routine analyses and the high value is 50 times the low concentrations. The Methods Team recognizes that it may not be appropriate to spike all of the target analytes listed within a chemical class.

If the developer has field data, the Methods Team would welcome the opportunity to compare the results obtained using the screening procedure with sample concentrations determined in a laboratory using SW-846 methods.

To summarize, the Methods Team does not require an unreasonable body of data for the initial evaluation of new techniques. Data will need to be submitted on the percentage of false negatives, percentage of false positives, sensitivity to method interferences, and matrix-specific performance data in order to complete the table below. In addition to these data, the developer should also provide a description of the procedure and a copy of any instructions provided with the test kits.

Type of sample	Number of samples	number of samples greater than the detection limit	number of samples less than the detection limit	semi-quantit-ative results/ dilution factor
False positive	20-50			
False negative	20-50			
Interference	20			
Matrix 1, low	10			
Matrix 1, high	10			
Matrix 2, low	10			
Matrix 2, high	10			
Matrix 3, low	10			
Matrix 3, high	10			

It is our belief that completion of this table represents only a small fraction of a developer's effort in developing a new method. If your technique will improve the quality of EPA monitoring programs, submitting these data should expedite the SW-846 approval process.

I look forward to working with you on this important activity.

Sincerely,

Barry Lesnik
Organic Methods Program Manager
USEPA Office of Solid Waste
Methods Team (5307W)

attachments

REPRESENTATIVE ORGANOCHLORINE PESTICIDES

The following chlorinated pesticides from the Best Demonstrated Available Technology (BDAT) list are representative of analytes for Method 8081. All should be sufficiently resolved to ensure good quantitation using two columns such as the DB-608 or DB-1701 (or equivalents). Suggested low and high concentrations should be appropriate for relatively uncontaminated solid matrices such as soil. Higher concentrations may be required for highly contaminated samples.

COMPOUND	LOW (µg/kg)	HIGH (µg/kg)
Aldrin	5	250
β-BHC	5	250
δ-BHC	5	250
γ-BHC (Lindane)	5	250
a-Chlordane	5	250
y-Chlordane	5	250
4,4'-DDD	10	500
4,4'-DDE	5	250
4,4'-DDT	5	250
Dieldrin	5	250
Endosulfan I	5	250
Endosulfan II	5	250
Endrin	10	500
Endrin aldehyde	5	250
Heptachlor	5	250
Heptachlor epoxide	5	250

REPRESENTATIVE SEMIVOLATILE COMPOUNDS

The following compounds are representative of analytes for Method 8270. Although many of these compounds are considered difficult, all can be extracted from waste matrices using conventional techniques. Suggested low and high concentrations should be appropriate for relatively uncontaminated solid matrices such as soil. Higher concentrations may be required for highly contaminated samples. Phthalate esters are not spiked but should be reported as a measure of contamination.

COMPOUND	LOW (µg/kg)	HIGH (µg/kg)
Phenol	250	12,500
o-Cresol	250	12,500
2-Methyl phenol	250	12,500
2,4,6-Trichlorophenol	250	12,500
Pentachlorophenol	250	12,500
1,2-Dichlorobenzene	250	12,500
Naphthalene	250	12,500
2-Chloronaphthalene	250	12,500
Anthracene	250	12,500
Chrysene	250	12,500
Benzo(a)anthracene	250	12,500
Benzo(a)pyrene	250	12,500
Fluoranthene	250	12,500
Indeno(1,2,3-cd)pyrene	250	12,500
Benzo(g,h,i)perylene	250	12,500
o-Toluidine	250	12,500
p-Nitrotoluene	250	12,500
2,6-Dinitrotoluene	250	12,500
2-Nitroaniline	250	12,500
Di-n-propylnitrosamine	250	12,500
4-Bromophenyl phenyl ether	250	12,500
3,3'-Dichlorobenzidine	250	12,500

In addition, surrogates should be added such that 50 µg or 108 µg would be present in final extracts assuming 100% recovery.

Nitrobenzene-d_5	50
2-Fluorobiphenyl	50
p-Terphenyl-d,,	50
Phenol-d,	100
2-Fluorophenol	100
2,4,6-Tribromophenol	100

REPRESENTATIVE VOLATILE COMPOUNDS

The following compounds are representative of analytes for Method 8260; many are TCLP analytes. All can be purged from waste matrices using Methods 5030 or 5035. Suggested low and high concentrations should be appropriate for relatively uncontaminated solid matrices such as soil. Higher concentrations may be required for highly contaminated samples.

COMPOUND	LOW (µg/kg)	HJGH (µg/kg)
Vinyl chloride	5	250
Dibromochloromethane	5	250
l,l-dichloroethane	5	250
Chloroform	5	250
Carbon tetrachloride	5	250
Trichloroethylene	5	250
1,1,1-Trichloroethane	5	250
Benzene	5	250
Toluene	5	250
Ethylbenzene	5	250
Chlorobenzene	5	250
Nitrobenzene	5	250
Methyl ethyl ketone	5	250
Carbon disulfide	5	250

Thefollowingcompoundsarerepresentativeof non-purgeablevolatileswhichcurrently require azeotropic distillation. Suggested low and high concentrations should be appropriate for relatively uncontaminated solid matrices such as soil. Higher concentrations may be required for highly contaminated samples.

COMPOUND	LOW (µg/kg)	HIGH (µg/kg)
1,4-Dioxane	10	500
n-Butanol	10	500
iso-butanol	10	500
Ethyl acetate	10	500
Pyridine	10	500

REPRESENTATIVE METALS

The following metals include the TCLP analytes except mercury. Suggested low and high concentrations are based on ICP analysis and should be appropriate for relatively uncontaminated solid matrices such as soil. Higher concentrations may be required for highly contaminated samples.

ELEMENT	LOW (µg/kg)	HIGH (µg/kg)
Arsenic	2500	75,000
Barium	100	5,000
Cadmium	200	10,000
Chromium	350	17,500
Copper	300	15,000
Lead	2ooo	100,000
Silver	350	17500
z i i	100	5,000

Mercury is generally analyzed by cold vapor AA.

Mercury	100	5,000

DEVELOPMENT AND

VALIDATION OF

SW-846 METHODS

PHASE 2:

FORMAL VALIDATION

DEVELOPMENT AND VALIDATION OF SW-846 METHODS
PHASE 2: FORMAL VALIDATION

If a methods developer successfully demonstrates that the method appears to be applicable for its intended application(s) through the "proof of concept" stage, as described in the previous section of this document, OSW will then consider incorporating the proposed method into SW-846 after it has completed the formal validation process. The document included in this section describes OSW's formal validation process and documentation requirements for new methods submissions. Multilaboratory validation data is necessary for a method to be considered for inclusion in SW-846.

The document included in this section delineates OSW's basic approach to developing and validating analytical methods for the RCRA Program. It is based on the performance-based approach to RCRA regulatory policy, i.e., the generator must demonstrate the ability to measure the analytes of concern in the matrices of concern at the regulatory limits. It involves tbe same elements for method development that RCRA requires for a demonstration of analyst and method proficiency in the standard Quality Assurance Project Plans (QAPjP) described in Chapter One and the technique-specific methods, e.g., Method 8000 in SW-846.

Tbe RCRA method development approach utilizes three basic principles:

1) Identify the scope and application of the proposed method, (What is this method supposed to accomplish?)

2) Develop a procedure that will generate data that are consistent with the intended scope and application of the method, and

3) Establish appropriate quality control procedures which will ensure that when the proposed procedure is followed, the method will generate the appropriate data from Step 2 that will meet the criteria established in Step 1.

A developer must also meet two other specific criteria before a method will be considered for inclusion in SW-846 are: 1) Is there either an existing or anticipated RCRA regulatory need for this method, and 2) Is it significantly different in principle or approach from existing SW-846 methods?

OSW has identified eleven key elements essential to a sound method development and validation effort These are listed in Table 1 on the next page.

These principles should also be followed when an analyst uses the built-in flexibility of SW-846 to modify methods for particular applications. Qualified analysts should be considered as "in-house" methods developers (Elements 1 to 9) when measurements are to be made from unusual matrices or below the quantitation limits provided with conventional SW-846 or other appropriate methods.

Table 1: Key Elements for Regulatory Methods Development

Element 1:	**Identification of Scope and Application and Regulatory Need**
Element 2:	**QA/QC Requirements**
Element 3:	**Analytical Approach**
Element 4:	**Method/Instrument Sensitivity**
Element 5:	**Method Optimization and Ruggedness Testing**
Element 6:	**Accuracy, Precision and Repeatability (Clean Matrix)**
Element 7:	**Effect of Interferences**
Element 8:	**Matrix Suitability**
Element 9:	**Quantitation and Detection Limits**
Element 10:	**Laboratory Reproducibility (Multiple Operators and Multiple Laboratories)**
Element 11:	**Document Submission and Workgroup Evaluation**

METHODS DEVELOPMENT AND VALIDATION FOR THE RCRA PROGRAM

Introduction

There seems to he an impression among methods developers and the regulated community that there is some esoteric or mystical process that must be followed in order to get an analytical method "approved" by regulatory agencies like the USEPA. In this document, OSW would like to dispel these misconceptions, identify some basic principles, and present a logical approach to methods development that is currently followed by the Office of Solid Waste (OSW) in developing methods for SW-846. This approach is based on sound scientific principles, and methods developed according to this process should be acceptable for use in other Agency programs as well as OSW.

Basic Principles

The basic principles involved in analytical methods development are very simple adaptations of the scientific method and can be summarized in the following three steps:

1) Identify the scope and application of the proposed method, (What is this method supposed to accomplish?)

2) Develop a procedure that will generate data that are consistent with the intended scope- and application of the method, and

3) Establish appropriate quality control procedures which will ensure that when the proposed procedure is followed, the method will generate the appropriate data from Step 2 that will meet the criteria established in Step 1.

Two other specific criteria that a developer must also meet before a method will be considered for inclusion in SW-846 as a separate method are: 1) Is there either an existing or anticipated RCRA regulatory need for this method, and 2) Is it significantly different in principle or approach from existing SW-846 methods? The rest of this document will provide more detail, including eleven key elements for methods development, and two examples of how this approach has been used successfully in the development of methods for SW-846.

The Nature of Regulatory Analysis

Regulatory analysts cover a wide variety of applications from regulatory compliance to informational data gathering. Some analyses are used to satisfy legal requirements, while others are used for monitoring of internal waste streams within a generator's facility.

Analyses used to determine the extent of contamination of a grossly contaminated site in the high ppm to low % range do not need to be performed with the same degree of precision and accuracy as analyses performed to demonstrate compliance with corrective action cleanup

levels in the low ppm to ppb range. Other factors to be considered in performing regulatory analyses include intended use of the data generated (i.e., project data quality objectives), method ruggedness and sensitivity, analyte/matrix interactions, laboratory performance, availability of equipment, and cost. All of these factors and more are taken into consideration when developing methods for regulatory purposes.

Kev Elements for Regulatory Methods Development

Methods developers should address the following eleven elements, which are listed in Table 1, when developing methods for OSW and other regulatory programs:

1. Identification of Scone and Application and Regulatory Need

The key factor that a developer must establish before proceeding with a method development project is a clearly defined scope and application for the proposed method. Factors to be considered should include type of method, (i.e., screening or assay), applicable target analytes, appropriate matrices, sensitivity, bias and precision, availability of equipment, and cost. It is advisable to limit the scope of most potential new methods, because a method that works well for a few specific applications is usually much more useful (and marketable) than one which is marginal for a large number of applications. Also, remember that it is usually much easier to develop and market a method that is designed around commercially available equipment. Cost is also a significant factor in determining whether a method has potential utility and marketability. Very few laboratories are prepared to buy expensive equipment today when comparable methods will do an adequate job at a much more affordable cost.

When establishing the scope and application for a potential new method, it is essential that the method developer identify that there is a regulatory need for the method. This can be a current or anticipated program office regulatory requirement determined by the methods staff, a Regional need, or a need for a method improvement identified by calls to Agency information services such as the MJCE line. A method may be scientifically elegant, but it has very little value if there is no application for its use in the regulatory program for which It is intended. For example, tissue samples and nutrients, e.g., ammonia and nitrate, are not usually of concern to the RCRA program. Therefore, methods addressing these matrices and analytes would be of low priority to OSW, although they may be of great interest to other Agency programs.

2. Ouality Control Rwuirements

When developing a method, the developer needs to identify the appropriate quality control procedures that must be performed to unequivocally demonstrate that the data generated by the method will meet the objectives defined in the scope and intended application(s). General QC procedures for RCRA analyses can be found in Chapter One of SW-846. Technique-specific QC procedures are included in the overview methods in SW-846,

e.g., Method 8000 (Chromatography), Method 3500 (Extraction), Method 3600 (Sample Preparation for Volatile Organics), Method 4000 (Immunoassay) and Method 7000 (Atomic Absorption). Method-specific QC procedures are included in the Quality Control section of the individual methods. When an apparent contradiction between QC criteria occurs in SW-846, the order of precedence is as follows: method-specific QC criteria have precedence over technique-specific QC criteria which have precedence over Chapter One QC criteria.

Examples of QC factors include appropriate calibration or tuning criteria, the need for replicate analyses, appropriate surrogates, blanks and spikes. QC criteria specific to the particular method should be well-documented and included in the QC section of the method as well as the method development project report. General and technique-specific QC requirements should be included in the Quality Assurance Project Plan (QAPjP) during the planning stage and in the project report (Section 11).

3. Analytical Aaoroach

In developing an analytical approach, the developer should keep in mind that the ultimate goal of the project is to develop a method that will be published for general use by the analytical community. Therefore, it is essential that any analytical instrumentation or equipment used in the development of the method needs to be commercially available to potential users at the time of the publication of the method. OSW encourages the use of either conventional or innovative technology, provided that it is demonstrated to be appropriate for the intended method application and provides data of sufficient quality to satisfy the criteria delineated in the scope of the method. OSW has been leading the effort to introduce new innovative analytical technologies to EPA's regulatory programs. Examples of new technologies first introduced in SW-846 include microwave digestion, potentiometric voltammetry, HPLC/MS, Immunoassay, Supercritical Fluid Extraction (SFE), and Accelerated Solvent Extraction (ASE). Those who criticize regulatory methods because they do not include innovative technologies are not aware of the level of review required for method promulgation.

In order to be considered for inclusion is SW-846, a method must be practical, i.e., has the potential for general use in the environmental analytical community, address a RCRA regulatory need, and be significantly different from existing SW-846 methods. Neutron Activation is a technique which has been used for analysis of metals for RCRA compliance. However, it is not an appropriate candidate for inclusion in SW-846 as a general-use method, since relatively few analytical laboratories have ready access to a nuclear reactor.

4. Method/Instrument Sensitivity

The method sensitivity requirements for a proposed new method are influenced by several factors. These include the instrument detection limits, method quantitation limits, and the regulatory requirements for the proposed applications. RCRA regulations basically require that an analyst demonstrate the ability to measure the analytes of regulatory concern in the matrices of concern at the regulatory levels. Therefore, a method must exhibit analytical

sensitivity appropriate for its intended application, as delineated in the scope of the method, before it will be considered for inclusion in SW-846. Many applications in the RCRA and other EPA programs do not require the use of methods at their extreme limits of instrument or method sensitivity. Pertinent performance information submitted in the methods package should include the instrument or method detection and quantitation limits, i.e., the minimum mass of analyte which can be quantitated (or detected, in the case of screening methods), the instrument or method calibration range for all target analytes, and information as to whether the calibrations are linear or non-linear (per the criteria in Method 8000). At this stage in the methods development process, the analyst should demonstrate the appropriate analytical parameters and procedure on clean standards of known concentration.

5. Method Optimization and Rueeedness Testing

After determining that the chosen analytical approach should work for its intended application with appropriate sensitivity, the method developer should begin to optimize the method and determine whether it possesses sufficient ruggedness to be considered for inclusion in SW-846. This is also accomplished using known standards.

The initial parameters should be chosen according to the analyst's beat judgment. These are varied systematically (usually using Youden pairs as described in J. K. Taylor, Quality Assurance in Analytical Measurements) to obtain the greatest response, least interference, greatest repeatability, etc. Developers must determine those variables which should not be changed without adversely affecting method performance. Potential operator-sensitive steps, e.g., color development time in colorimetric methods or other timed reactions, also need to be identified at this stage.

6. Accuracy (Bias). Precision. Repeatability (or Lone-term Precision) in a Clean Matrix

Accuracy, or in most cases method bias, is defined as nearness to the true value. Precision is defined as the dispersion of results around the mean value. Repeatability (or long-term precision) is defined as the ability to reproduce a measurement from one week to the next.

Bias is measured by determination of % recovery of target analytes spiked into the matrix of concern. An acceptable spike recovery range for most method development applications is from 80% to 120%. Precision is measured as relative % difference of target analyte concentration(s) between duplicates or duplicate spikes, and should usually be <20%. Repeatability is measured as long-term, e.g., weekly, precision, when the instrument is calibrated using comparable standards, and on a different day should not vary by more than 15%.

These are key method performance factors which determine how a method can be used in real world situations. The initial determination of bias, precision and repeatability should be made in a spiked clean matrix which is similar to a real environmental matrix, but free from interferences, e.g., reagent water, sand, soil (for inorganic methods), vermiculite or ash. These

values should he obtained using multiple replicates at both high and low spike concentrations. Guidance on bias and precision determinations and preliminary method evaluation for new sample preparation and screening methods is available from the Offtce of Solid Waste.

7. Effect of Interferences

The determination of method interferences, both positive and negative is a key factor in method development. It is a critical element in methods development for the developer to determine the effects of potential analytical interferences and to develop techniques to minimize or eliminate these interferences. In chromatographic methods, interferences include coeluting peaks and/or analyte degradation due to interaction with either the injector port, transfer line or column. In spectroscopic methods, interferences can result from overlapping spectral lines causing either positive or negative signal enhancement. In immunoassay methods, interferences include cross-reacting compounds.

Method interferences should be determined in a spiked clean matrix. Developers should determine the effects of interferences in a potential new method between target analytes and other compounds reasonably expected to be present in waste matrices.

False negative rates, i.e., the percentage that a method generates a negative result when the sample contains the target analytes at or above the action level and false positive rates, i.e., the percentage that a method generates a positive result when the sample contains the target analytes below the actlon level, are critical factors which will determine the utility of a potential method for its intended application.

Documentation of interferences should include any coelution of or with target analytes, any enhancement or suppression of target analyte signals caused by interferences, any necessary or optional cleanup procedures to minimize the effect of interferences, and any matrix-specific difficulties.

8. Matrix Suitahilitv

The previous elements of the methods development process involved the use of either known standards or target analytes spiked into clean matrices, designed to indicate potential method performance in real RCRA matrices. Once the potential new method has passed all of the preliminary tests, it is now ready for the most important demonstration in the entire methods development process, i.e., how it will perform in the real world matrices for which it is intended to be used.

The method should be suitable for a variety of matrix types. Therefore, the developer should choose appropriate RCRA matrices for the demonstration of method performance. By matrix types, OSW refers to different matrices within a particular medium, e.g. water, soil and ash. Appropriate RCRA water matrices include groundwater, TCLP leachate and wastewater, while appropriate RCRA soil matrices include sand, loam and clay. Appropriate ash matrices

include bottom ash, fly ash and/or combined ash. The method should perform adequately in a variety of spiked matrices and then in a variety of well-characterized natural samples or standard reference materials (SRMs) when SRMs are available. Performance data including matrix, precision, bias, quantitation limits (see next section), and any other pertinent data should be documented in appropriate tables. A summary of the single-laboratory performance data should be included in tabular format in the method while the detailed performance data, including QC, should be included in the supporting document (Section 11).

9. Method Detection and Ouantitation Limits

In a new method submission, OSW is most concerned about the performance of the method in the RCRA matrices of concern. The developer should generate estimated method quantitation (EQL) and method detection limits (MDL) for the analytes of concern in the matrices of concern following the guidelines established in Chapter One of SW-846. The practice of generating MDLs based on reagent water is not usually a very useful parameter for most RCRA methods.

Method detection and quantitation limits for a determinativemethod are usually based on a specific sample size and a specific sample preparation scheme. The limits determined in clean matrices indicate the limits of the acceptable performance for the method. Matrix effects may affect the achievable quantitation limits on real world samples. However, the method quantitation limits for the target analytes in the target matrices must meet the analytical requirements of the intended application, as defined in the scope of the method, before it can be considered for inclusion in SW-846. Quantitation limits for the target analytes in representative matrices should be included in summary tables in the methods, while the rationale for and details of the MDL and EQL concentrations should be included in the supporting document (Section 11).

10. Laboratorv Rearoducibilitv (Multiple Operators and Multiple Laboratories)

The final stage in the method development process, prior to the submission of the method for Agency review, is the determination of laboratory reproducibility. By reproducibility, OSW means that multiple operators and multiple laboratories should be able to obtain comparable performance data on split samples using the method. Since all of the previous elements involved single operators or single laboratories, it is necessary to demonstrate that satisfactory method performance is not limited to the individual operator or laboratory that developed the method.

The minimum number of laboratories that are needed to participate in a multilaboratory method validation is three, with preferably more. In previous years, when the Agency budget permitted, multilaboratory validations of new methods were funded by the Agency. Large numbers of laboratories were paid to participate in these studies with EPA doing the "data-crunching". Agency funding can no longer support these types of studies, so developers of new methods today need to do a limited multilaboratory evaluation and provide

the individual laboratory and summary performance data in the method submission. Developers are encouraged to consult with the appropriate regulatory agency when planning a multilaboratory study.

In order to minimize the number of variables involved in method validation, the developer needs to follow a few simple guidelines to demonstrate appropriate multilaboratory method performance. When validating a sample preparation method, the participating laboratories should only perform the sample preparation procedure. The collected samples should then he sent to one laboratory for analysis. The analysis should be done by a single operator on a single instrument in a single hatch to minimize analytical variability inherent to the determinative method. Conversely, if a determinative method is to be validated, the developer should have a single operator perform all of the sample preparation operations in order to minimize operator and laboratory variability inherent to the sample preparative procedures. The sample extracts should then be split and sent to the laboratories participating in the validation study for the analytical determination.

11. Document Submission and Workeroun Evaluation

When the method project is completed, the developer must assemble a package of documents describing the project, and submit it to the Agency for review and evaluation. This documentation package should include 1) draft copies, both hard and electronic, of the method in an approprlate Agency format; 2) a supporting document describing the rationale behind the methodsdevelopment effort and how the key elements of the methods development project as described in this document were addressed; 3) a data package containing both the raw and summarized single laboratory and multilaboratory data; 4) any specific equipment diagrams and chromatograms, spectra, etc. pertinent to the demonstration of appropriate performance for the intended application of the method; 5) copies of any referencea listed in the method; and 6) any method-specific quality control criteria.

The OSW Methods Team reviews the methods submission package for completeness and quality, and then decides whether the method is ready to be sent to the SW-g46 Workgroup for review or back to the developer for additional work. OSW has several standing SW-g46 Methods Workgroups which meet formally each July to review the methods packages submitted by developers for potential inclusion in SW-846. Workgroup members are Agency scientists from the EPA Program Offices, Regional Laboratories, Office of Research and Development (ORD), and the National Enforcement Investigations Center (NEIC) who evaluate the procedures and performance data submitted by the methods developers. Workgroup members provide the benefit of their own experience in performing and evaluating analyses. Workgroup comments address both the written method and the completeness of the performance data, other method documentation, and interpretation of the data. Methods which the Workgroups accept for inclusion in SW-g46 are edited based on Workgroup comments. After editing, the OSW-Methods Team distributes them as draft methods until the SW-846 Update package of which they are a part is ready to be submitted for Agency approval through the official regulatory "notice and comment" process. The

supporting documentation for each method is then put into the RCRA Docket for public review immediately before the initial proposal of the new SW-846 Update package.

Conclusion

OSW believes that the information provided here clarifies the RCRA method development process. Qualified analysts should be considered as "in-house" methods developers (Elements 1 to 9) when measurements are to be made from unusual matrices or below the quantitation limits provided with conventional SW-846 or other appropriate methods. The need for such a demonstration of analyst/laboratory proficiency has been implicit in SW-846 Chapter One and Method 8000. These implicit requirements have been clarified and made explicit in Method 8000B, which is included in Update 3.

Practical Examples of Methods Developed Using the RCRA Validation Process

This section of the document provides two examples of metbods developed for and included in Update 3 of SW-846. Example Number One is of a sample preparation method developed for use with existing SW-846 determinative methods. Example Number Two is of a screening method for determining the range of concentrations of PAHs in soils.

Example 1: Method 3545 - Accelerated Solvent Extraction (ASE)

Tbe Accelerated Solvent Extraction Method (Method 3545) was developed by Dionex as a rigorous, rapid sample preparation method for extractable organic analytes in solid matrices using small quantities of solvent. The following paragraphs provide a brief overview of how Method 3545 was developed using the analytical approach discussed in this document:

1) ASE was developed as a general-purpose, rigorous extraction procedure for RCRA extractable analytes amenable to the Soxhlet technique, either manual or automated, and with comparable performance. It also uses much less extraction solvent than the existing general-purpose techniques, thus helping to promote the Agency's policy of pollution prevention in analytical methods.

2) No method-specific QC procedures are necessary.

3) The analytical approach uses conventional solvents as theextraction fluid rather than supercritical CO_2, while utilizing the basic operations of supercritical fluid extraction (SFE), e.g., elevated temperature, elevated pressure, and minimum void volume. Dionex has demonstrated that these conditions provide a rigorous extraction of the target analytes from a solid matrix in minutes instead of hours.

4) The method sensitivity using standard determinative methods, i.e, Methods 8270, 8081, 8082, 8141, and 8151, is comparable to Soxhlet using the same sample size.

5) The method was optimized using about a lo-minute extraction with the appropriate extraction solvents for the target analytes, e.g., methylene chloride, hexane:acetone, etc., at a temperature of about 100°C and a pressure of about 2000 psi (a relatively low pressure for SFE). Sample size can be varied without changing the extraction conditions by varying the size of the extraction vessels used.

6) Recoveries (bias) in a sandy soil, a loam, and a clay were comparable to Soxhlet extraction, while precision (%RSD) was a little better than standard Soxhlet and about the same as Automated Soxhlet. Repeatability was not a problem.

7) ASE did not appear to generate any additional interferences other than those commonly encountered in Soxhlet procedures.

8) Method 3545 was validated in a variety of soil matrices ranging from sand to clay and demonstrated performance comparable to that of automated Soxhlet extraction. Due to time and cost factors, the method was not evaluated in other RCRA matrices. However, OSW believes that it should he appropriate for all RCRA matrices for which Soxhlet extraction is currently used, based on the Performance data submitted with the method.

9) Quantitation and detection limits based on Method 3545 recovery data from soils for the standard determinative methods were comparable to thoseobtained using automated Soxhlet extraction.

10) Tbe method was performed in three laboratories, all of which obtained acceptable results.

11) Method 3545 was reviewed by the SW-846 Organic Methods Workgroup in July, 1994, and was accepted for inclusion in Update 3 of SW-846. Dionex submitted a second documentation package containing performance data for additional analytes in November, 1994. Both reports are included in the RCRA Docket supporting Update 3.

Example 2: Method 4035 - Screening Method for PAHs in Soil bv Immunoassay

The PAH immunoassay method was developed by EnSys as a screening method for PAHs in soils at cleanup levels. It was the first method included in RCRA's second group of immunoassay methods accepted for inclusion in Update 3 of SW-846. The following paragraphs provide a brief overview of how Method 4035 was developed using the analytical approach discussed in this document:

1) Method 4035 was developed as a screening method for PAHs containing 3- and 4-ringed PAHs at the nominal RCRA/CERCLA cleanup action level of 1 ppm. It was also one of a series of methods which addressed the Agency's need for low-cost, rapid, effective screening methods which could be used either on-site or in a fixed laboratory. The target false negative rate for screening methods is 0%, while the target false positive rate is 10%.

2) Standard QC requirements apply to immunoassay testing. However, there are some specific QC criteria for immunoassay methods which must be followed. These include 1) do not use test kits past their expiration date, 2) do not use tubes or reagents designated for use with other kits; and 3) use the test kits within the specified storage temperature and operating temperature limits.

3) The analytical approach used was an inverse colorimetric method utilizing a competitive antibody technique sensitive to the target PAHs, i.e., color was discharged when the target PAHs were present at or above the action level.

4) The sensitivity of the test is influenced by the binding of the target analyte to the antibodies used in the kit. The commercial PAH kit used for evaluation of this method is most sensitive to the three- (i.e., phenanthrene, anthracene, fluorene) and four- (i.e. benzo(a)anthracene, chrysene, fluoranthene, pyrene) ring PAH compounds listed in Method 8310, and also recognizes most of the five- and six-ring compounds listed. The method can be used for screening the target PAHs at various predetermined action levels down to 1 ppm.

5) Method 4035 (EnSys) was optimized for PAHs containing 3- and 4-ringed PAHs in a variety of soil matrices.

6) Screening data correlated >95% with the reference method used (Method 3540/8270) for the target analytes.

7) Interferences for immunoassay methods are referred to as cross-reactivity. The target compound for the EnSys PAH kit with a cross-reactivity of 1 was phenanthrene. The other 3- and 4-ringed compounds had similar responses. All of the 16 RCRA target compounds had a cross-reactivity within one order of magnitude of phenantbrene, except for naphthalene (200), dibenzo(a,h)anthracene and benzo(g,h,i)perylene (both >200). The kit was optimized to respond to three and four ring PAHs. The sensitivity of the test to individual PAHs is highly variable. Naphtbalene, dibenzo(a,h)anthracene, and benzo(g,h,i)perylene have 0.5 percent or less than the reactivity of phenanthrene with the enzyme conjugate. The alkyl-substituted PAHs, chlorinated aromatic compounds, and other aromatic hydrocarbons, such as dibenzofuran, have been demonstrated to be cross-reactive with the immobilized anti-PAH antibody. The presence of these compounds in the sample may contribute to false

positives.

8) The EnSys PAH kit was designed for use in soil matrices. It was demonstrated to be applicable to a cross-section of soil types from sand to loam to clay.

9) Method 4035 is designed to act as a screening method at a predetermined action level (usually 1 ppm). Using the test kit from which this method was developed, $\geq$95% of samples confirmed to have concentrations of PAHs below detection limits will produce a negative result in the 1 ppm test configuration.

10) Multiple field trials involving split samples were conducted at a power plant site and on a variety of well-characterized samples from Region 10 ranging in concentration from <0.1 ppm to >200 ppm. Several operators and laboratories participated in the studies. Actual false negative rates were <5%, while false positive rates ranged between 10 and 15%.

11) Method 4035 was reviewed by the SW-846 Organic Methods Workgroup in July, 1993, and was accepted for inclusion in Update 3 of SW-846. The method has been available in draft form from OSW since early in 1994. The original data submission package and the Region 10 study results are included in the RCRA Rocket supporting Update 3.

ATTACHMENT XV

SUPPORT DOCUMENTATION

ATTACHMENT XV – SUPPORT DOCUMENTATION

Volume I contains summarized information. tables, standard operating procedures, and guidance documents. Volumes II through XI contain all the raw chromatograms and associated data generated by the Crane Laboratory in the course of the method validation study. The following table summarizes the volumes and their contents.

VOLUME #	TITLE	CONTENTS
Volume I	Analytical Method Development Summary	Summarized information. tables, standard operating procedures, and guidance documents
Volume II	Solid Waste Management Unit – 02/1 1 NSWC Crane, Crane, Indiana Long Gradient Initial Calibration, Continuing Calibration, and Instrument Blanks Full Data Package	ICAL, CCV, and instrument blanks for Long Gradient
Volume III	Solid Waste Management Unit – 02/1 1 NSWC Crane, Crane, Indiana Short Gradient Initial Calibration and Continuing Calibration Full Data Package	ICAL and CCV for Short Gradient
Volume IV	Solid Waste Management Unit – 02/1 1 NSWC Crane, Crane, Indiana Instrument Blanks Short Gradient Full Data Package	Instrument blanks analyzed via Short Gradient
Volume V	Solid Waste Management Unit – 02/1 1 NSWC Crane, Crane, Indiana Water – Low Method, High Spike Short and Long Gradients Full Data Package	Long and short gradient analyses of high spike water samples by low-level extraction method
Volume VI	Solid Waste Management Unit – 02/1 1 NSWC Crane, Crane, Indiana Water – Low Method, Low Spike Short and Long Gradients Full Data Package	Long and short gradient analyses of low spike water samples by low-level extraction method
Volume VII	Solid Waste Management Unit – 02/1 1 NSWC Crane, Crane, Indiana Water- High Method, High Spike Short and Long Gradients Full Data Packaae	Long and short gradient analyses of high spike water samples by direct injection
Volume VIII	Solid Waste Management Unit – 02/1 1 NSWC Crane, Crane, Indiana Water – High Method, Low Spike Short and Long Gradients Full Data Package	Long and shod gradient analyses of low spike water samples by direct injection
Volume IX	Solid Waste Management Unit – 02/1 1 NSWC Crane, Crane, Indiana Soil -High Spike Short and Long Gradients Full Data Package	Long and short gradient analyses of high spike soil samples
Volume X	Solid Waste Management Unit – 02/1 1 NSWC Crane, Crane, Indiana Soil-Low Spike Short and Long Gradients Full Data Packaae	Long and short gradient analyses of low spike soil samples
Volume XI	Solid Waste Management Unit – 02/1 1 Screening Level Data Packaae	Example of information provided in a screening level data package