N00164.AR.000246
NSWC CRANE
5090.3a

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

REPLY TO THE ATTENTION OF:

DRP-8J

September 11, 1996

Mr. Thomas Brent
Environmental Protection Department
Code 5090 Ser 095/6228
Naval Surface Warfare Center
300 Highway 361
Crane, Indiana 47522-5000

RE: Bioremediation Facility
QAP NOD
Naval Surface Warfare Center
Crane, Indiana
IN5 170 023 498

Dear Mr. Brent:

Mr. Allen Debus from the United States Environmental Protection Agency has reviewed the majority of the Quality Assurance Plan for the Bioremediation Facility. We are providing the attached comments to begin on a revision to the document. Allen still has some of the Standard Operating Procedures (SOPs) to review. We felt that by sending you what review comments were gathered to date, we could speed the review process. I will send my review comments on the Operations Plan and the QAP under a separate letter.

There are several important areas that need modification. These are the objective section, the cleanup levels, and the field test methods. Please review the enclosed comments, and we would like to set up a conference call to discuss whether the planned mid-October start-up date still can be met or not, in response to these issues. Please provide a revised document and response to comments as soon as possible to meet the pilot schedule.

Printed on Recycled Paper

If you have any questions regarding this matter, please contact me at (312) 886-6146, or Allen Debus at (312) 886-6186. Allen will be back from vacation on September 16th.

Sincerely,

Carol Witt-Smith
Corrective Action Expert
WMB, IL/IN/MI Section

cc: Steve Downey, MK
Adrienne Wilson, SOUTHDIV
Jim Hunsicker, USN
Chris Freeman, USN
Tom Linson, IDEM
John Manley, IDEM

Comments of the Bioremediation Facility QAP
Dated August 1, 1996
Naval Surface Warfare Center

Allen Debus's review of the following documents: (1) the Operational Plan, (2) the Interim Measures Work Plan, and (3) the Quality Assurance Project Plan (QAPP) with some of the critical SOPs, but not all of them.

A. General Comments

1. The QAPP has been written as a Quality Assurance (QA) plan folded around a sampling plan. It lacks many details, which would ordinarily be presented in a comprehensive QAPP.

2. I have some concerns about the proposed project objectives, which may not address regulatory matters sufficiently.

B. Operational Plan Comments

1. Section 1.2

Apparently, extremely high levels of explosives contaminants have been detected in soils at some of the SWMUs. After excavation has occurred, there exists high potential for high concentrations to remain in the exposed soils. Analysts using proposed methods must be prepared to dilute such samples in order to be able to report meaningful data that is within the range of linear calibration. (This comment applies both to field and laboratory data.) Note that for lab data, the reporting limits of any undetected constituents increase by the factor of dilution.

Also, (especially) at the ABG (see page 3) it seems likely that petroleum contamination may be detected (in more copious amounts than individual constituents). The facility should consider using TPH immunoassay or laboratory confirmation (method 8015B) as a means of assessing "extent" of contamination or "presence" of release. Both diesel range and gasoline range organics (as well as other specific constituents) could be reported for this purpose.

2. Section 1.3.3, p.12

Referring to Table 1-2, the facility should consider what outcome will result if the HMX doesn't degrade in the "pilot" study. (Note that HMX has a considerably higher activation energy than does RDX when subjected to alkaline hydrolysis. Environ. Sci. Technol., 1996, 30, pp.1485-1492))

3. Section 5.1.2, p.22

In the last paragraph on the page, the term "significant" should be quantified?

4. Section 5.2.1, p.24

Here (in the last paragraph) and in other sections, there is repeated mention of a procedure involving addition of truck and rock washings into the compost pile. However, it is possible that the washings may contain minimal concentrations of constituents of concern, which would distort the mass balance of key constituents. (A "closed system" would no longer be maintained.) It would be advisable either to use an established source of water, that is "clean" with respect to key constituents, to adjust the compost pile moisture levels. Alternatively, analyze the constituents in the washings before adding them to the pile at a target level of concern.

5. Section 5.2.5, p.26

In the first line, note that blends #3 through #8 do not contain alfalfa, and these could conceivably be the mixes that define the cleanup level goals. It is premature to mention alfalfa now in the context used here.

6. Section 5.2.7

Although the last sentence in this section reads very affirmatively as if the material will eventually be added to the on-site landfill, what if only low % microbial chemical degradation results, especially for HMX? Have any (minimal) cleanup level goals been defined?

7. Section 6.1.2, pp.29-30

This paragraph mentions CLP guidance. What is the rationale for using a CLP procedure rather than procedures founded in SW-846? Also, CLP's CRDLs should not be used as reporting limits for this project. The term "approved laboratory" at the top of page 30, should refer to the U.S. EPA's approval of the laboratory for this specific project, not imply that the Navy's approval is the only required approval.

8. Section 6.2.1, p.30

See comment B.4.

9. Section 6.3.2, p.32

See comment B.4.

10. Section 7.2.4, p.34

Shouldn't TOC and TKN also be checked both during and at end of lifecycle?

11. Section 7.2.8, p.36

The purpose of the air monitoring data collection should be clarified.

12. Section 8.3, p.39

This section refers to concentrations above the levels established in the pilot scale tests as being the basis for removal (presumably of additional soil from each of the SWMUs). However, we need to have an indication of what these levels might turn out to be (or better yet the measure of "success" expressed as a decision level in concentration units) prior to approving the Work Plan. Very importantly, how do the "cleanup levels" compare to the reporting levels of the field screening methods? The issue pops up several times in the subsequent subsections.

13. Table 8-1, p.41

It is unclear whether the rows marked "windrow compost" refer to pilot scale pile versus the final remediative piles. (This is a universal comment applying to the entire document.) The entry labelled "TO" should be changed to read "TOC". Should provisions for air monitoring be added to the table as well? (See section 7.2.8.)

C. Work Plan for Interim Measures Cleanup

1. Table 3-1

The titles of the Cost and Schedule Engineer and the Contract Administrator are not identified in Figure 3-1. Every individual identified in Figure 3-1 should be discussed in the QAPP. Also, it should be established who will perform independent data validation.

2. Section 4.2.5, p.24

The first sentence reads as if contamination will be intentionally left in the ground, given that the extent will be known but that contamination may still be quantified in the excavation area. This may not be consistent with RCRA policies, and yet it may merely reflect the limitation of removing 60,000 cubic yards of soil. Also, an indication of what the "cleanup levels" will be should be presented, as these are significant project objectives. For instance, are the "cleanup levels" consistent with U.S. EPA's risk assessment approach? (It does not seem as if this is the case.)

3. Section 4.2.6

This paragraph indicates that there are options as to how the decontamination fluids will be managed. However, other sections of the Work and Operational Plans emphasize that the fluids will be added to the bioremediation pile. Some clarification is needed.

4. Section 4.3.6

The addition of large amounts of contaminated wash water may impact the compost mass balance study. Should any effort be made to screen the wash water for compounds of special concern prior to adding wash water to the pile?

5. Section 4.3.7

Should back-filling be allowed to take place prior to generation of laboratory "confirmation" data? (i.e. It could be determined that more soil will be excavated.)

6. Section 4.4.5

Some idea of what the acceptable "cleanup levels" happen to be (levels which should be developed in a manner consistent with Region 5 risk assessment policies) should be added to the QAPP.

7. Section 4.5.4

It should be clarified that only soils from the Rockeye area (possibly changing to one of the Mine Fill Areas) will be added to the compost pile during the pilot phase of testing, and that levels established for Rockeye (or the selected site) will apply to all 4 SWMUs. Therefore, the Rockeye pile ("pilot" scale) should not receive soils/debris or wash water from any of the other 3 SWMUs. The pilot scale test goals should be indicated in this section. Rockeye wash waters should be screened for key compounds of concern prior to adding them to the pile.

8. Section 4.5.5

In order to be able to consider the Mine Fill B objective seriously, some indication of what the cleanup levels will be should be presented. Otherwise, any level that results from the pilot scale tests will be considered acceptable. It would therefore be conceptually impossible to "fail" the pilot scale study.

D. QAPP Comments

1. Title Page

A signature space for a representative of the laboratory(ies) has not been provided on the title page of the QAPP.

2. Section 1 - General

This QAPP appears to be founded on the premise that a pilot scale study will be successful, and that reasonable cleanup levels can be established, even though there is no mechanism for failure of the compost pilot study. However, a critical focus of the QAPP should be on determining the acceptable levels, prior to or even during the excavation of soils from SWMUs.

(The QAPP appears to be a "cart before the horse" type document.) Also, a comprehensive sampling Network table for all SWMUs is needed. This table should represent field and lab parameters. An example of such a table may be found in the Region 5 Model QAPP, (5/23/93).

3. Section 1.2.1, p.3

Referring to the 3rd bullet, what course of action will be taken if suitable cleanup levels cannot be achieved during the pilot study, AND what if the remediation piles cannot meet the cleanup levels attained during the pilot scale study? These are two major deficiencies with the submitted documents.

4. Section 1.4.1, p. 6

The pilot scale compost piles are very important as objective/decision levels will be determined by their performance. Yet, management and performance of the pilot scale piles seem to have been relatively neglected in the submitted documents.

5. Section 1.4.3, p.7

Very high levels of ordnance contamination seem to be present in site soils. Yet is not clear how such levels would compare to the reporting limits and upper calibration range of the proposed immunoassay screening kits. This is significant because the contamination is so high that removal of even large amounts of soil may fail to reach the "uncontaminated" zone. There appears to be TPH contamination associated with the ABG. The Navy should consider the utility of performing GRO and DRO method 8015B analyses. Also, it may be possible to screen some metals using XRF.

Note later in our comments that the immunoassay tests will be replaced with colorimetric tests.

6. Section 1.4.3, p.8, 3rd paragraph

In the second line, the term, "t-xylene" should be changed to read as "total xylenes" (i.e. sum of o + p+ m isomers).

7. Section 1.4.3, p. 8, 5th paragraph

In the next to last sentence, the constituents should not change "as necessary" to meet the program objectives. The purpose of writing the QAPP is so that the proposed sampling and testing program will be designed to meet specified objectives. Changing the scope of the program in mid stream would effectively hamper the Navy's ability to provide the data which is needed by the U.S. EPA in order to base regulatory determinations.

8. Section 1.4.3, p. 9, 2nd paragraph

See comment D.7.

9. Section 1.5.1, p.11

In the second bullet, the document NEESA - 20.2-047B (NEESA, 1988) should be included in the QAPP. (I haven't checked the Appendix to see if it is there yet, but even if it is, it should at least be referenced here.)

10. Section 1.5.2, p.11

In the next to last line on the page, the word "goals" has been spelled incorrectly. Also, the exact meaning of how data will be "compared" to background soil levels, and other values is very unclear, and should be explicitly stated.

11. Table 1-1

The specific "VOCs" should be specifically listed or referenced. Note that the appended methods are capable of reporting many VOCs, yet only a "short list" of VOCs has been proposed in Table 1-2. The relation of the VOCs that are possible versus those which are of greater concern (for some stated reasons) should be clarified. In the references to the "CLP-SOW", note that CRDLs will be reported, which are usually insufficiently sensitive for risk assessment purposes. In the box for Rockeye, second column, "incidental/decon water, if required", should be required. The Pilot compost piles are not distinctively identified apart from the biofacility piles.

12. Table 1-2

Are the Table 1-2 values acceptable for risk assessment, if risk issues are pertinent to this study? Practically speaking, the soil DQL levels are (subjectively) so high as to probably not pose major analytical or risk issues, but their use as "decision levels" may preclude ability to require risk assessment studies later on. The use of the stated levels as "action levels" may not be sensitive to cumulative effects. The relevant field test decision levels which apply to these constituents should be identified. Are the indicated levels also supposed to be levels acceptable for use as backfill? Are these levels the "closure levels" referred to on p. 11 of 22? In the final report, all detected compounds, beyond those listed here should be reported. The Navy should consider adding GRO and DRO analyses to provide further indication of contamination, in at least the ABG area. More explosives can be reported using method 8330 than indicated in this table. Section 4.1 reflects a field decision level of 30 ppm for TNT and RDX. This proposal should be reflected in Table 1-2.

13. Section 2

Figure 2-1 is missing from the QAPP. Under Laboratory Responsibilities, the involvement of Compuchem is poorly represented. It is not clear who will conduct independent data validation. Who will perform field tests and other specific measurement tasks in the field?

14. Section 3

Adding a table comparing the reporting limits for both field and laboratory tests to "decision" (field) and "target" (lab) levels would immensely improve the clarity of the project description and data quality objectives sections. Also, the "laboratory detection limits" referenced in Appendix A are method detection limits, not reporting limits. It is reporting limits, not MDLS, which are pertinent to the project objectives and final assessment of data. A table should be added, reflecting the QC objectives for all the field parameters (analogous to those included reflecting the QA objectives for laboratory measurements).

15. Section 3.1.2, p.1

Composting of explosive samples should be discussed as per recently received (draft) Region 10 guidance. Terms such as "semiquantitative" and "qualitatively" create some alarm in this section. Major decisions will apparently be made on the basis of field (immunoassay) testing. Therefore, it will be important to rely on these tests in as "quantitative" a manner as possible using the full rigor of QC that is possible for these test kits. Also, other field test parameters seem to have been neglected from this section. For instance, are not the tests associated with monitoring of the pilot test pile considered to be field tests?

Also see note on the change in field test kits.

16. Section 3.2.2

Referring to the last phrase in this section, field data holding times should only be a special issue for immunoassay.

17. Section 3.3.3

The laboratory completeness goal should be 95%.

18. Section 3.4.2

The representativeness of explosives samples should be further explored and discussed after the Navy has opportunity to review the recent (draft) Region 10 guidance. Note that this guidance document discusses compositing of samples to improve measurement precision. In the last sentence of this section, it should also be mentioned that adherence to sample preservation will improve representativeness.

19. Section 3.5.1

How does immunoassay data compare between the different types of kits that are available and also to method 8330 (8240 - TCE) data. It was privately mentioned in a conversation with a Navy representative that it would be a goal to compare immunoassay test kits, and this objective has been omitted from the QAPP. Also, since pilot test data will eventually be compared to data resulting from operation of the biofacility, special attention should be devoted to the compost data.

20. Section 3.6

It should be discussed whether field blanks are opened or poured in the field. What is the procedural difference between the field and trip blanks (i.e. only for VOCs). Will a duplicate also be taken for metals to serve as a MSD, or will an alternate procedure be followed. What is the procedure and frequency of collection for equipment rinse blanks?

21. Table 3-1

Which QC target analytes do the QC limits specifically refer to. The completeness goal of 85% should be changed to 95%.

22. Section 4, p.1

Are analytical immunoassay interferences expected for high level contaminants? How many immunoassay tests will be performed? Note that if the geoprobe is used in sandy soil at depth, there could be difficulties. Should the pilot scale compost piles be distinguished from the remediation biofacility piles? We think they should.

23. Sections 4.1 and 4.1.1, p.2

When maximum amounts of soil have been removed, what if the remaining concentrations of contaminants are found to be above the field decision levels (i.e. in the base or sidewalls of the excavation)? Will more soil be excavated until the area has been determined to be "clean"? How does a 30 ppm level compare to the soil DQL/PRG levels for TNT and RDX? In the reference to TCLP metals, is this intended to be an analysis for" total" metals (full digestion) or metals leached into a TC extract?

24. Section 4.2

The sampling of the biofacility is not sufficiently distinguished from sampling of any piles that are regarded to be preliminary or "pilot piles".

25. Section 4.3.1.5

In the second paragraph, note that samples intended for analysis of VOCs should not be split. The approach to using a sample duplicate for matrix spiking purposes is unorthodox and should be fortified by further rationale.

26. Section 4.3.3, p.7

Step 5 can be performed, however, it may not be necessary to follow the proposed procedure in every instance that organic samples are collected. (For instance if the soil is not contaminated with oil.)

27. Section 4.3.4, p.8

Sample tags are needed. (Note that a reference to sample tags has been included in section 5.3. Also, where will be samples be shipped to? (An address is needed.)

28. Table 4-1, p.1

The reference to a TCE field test kit "if required", should be decided upon now, prior to approval of the QAPP. (Personally, my opinion is that better data could be achieved using a field gas chromatograph. Most geoprobe units carry a field GC onboard, with the capability of performing an 8020/8021 VOC scan, which would include TCE and other breakdown products.)

29. Table 4-1, p.2

With respect to the previous comment (C.28), it should be considered whether there is any need for immunoassay TPH and PAH data collection as well (or instead of TCE immunoassay data).

30. Table 4-1, p.11

Referring to the section concerning QA/QC, the presentation is very sketchy. More elaboration of these procedures is needed.

31. Table 4-1, p.1

Table 4-1 indicates TCE will be reported using immunoassay. However, this table also somewhat implies that VOCs can be reported using field GC (see reference to PID/FID), which would be preferred over immunoassay for the ABG.

32. Table 4-2, p.2

In point 6, what might "further action" consist of? Referring to point 7, Table 4-7 indicates that organic TCLP results will also be determined. However, what if TC criteria are exceeded for pesticides or SVOCs?

33. Table 4-2, p.14

a. Referring to point 2, why are field duplicates only being collected at a 1/20 frequency for field explosives, as compared to 1/10 for TCLP-metals?

b. Referring to point 5, add the phrase, "...or if new matrices are identified", to the end of the first sentence.

c. Also, rinsate blanks should be collected at least once per SWMU.

34. Table 4-3, p.15

Under methodology, first paragraph, how is "no further contamination" defined? This deficiency could be addressed in tabular format. Also, if soil is anticipated to be oily, TPH immunoassay data might aid in decision making as to whether the extent of contamination has been identified in the field. This field data could then be confirmed in the laboratory using method 8015B for GRO and DRO. As the goals of the field and laboratory testing are refined, the possibility of using methods sensitive to TPH contamination should at least be considered.

35. Table 4-3, p.17

It may be worthwhile to homogenize soils prior to analysis of explosives constituents, per (draft) Region 10 guidance. Also, referring to the last line on the page, it should be decided now as to whether phosphorous testing fits into the scope of project objectives.

36. Table 4-4, p.20

Under the second box, reference to total metals, will toxicity characteristic metals testing also be required?

37. Table 4-5, p.21

This objective has not been sufficiently developed in the QAPP. If NPDES sampling is thought to be beyond the scope of the RCRA RFI, for simplicity, it should be left out.

38. Table 4-7, p.26

Under "holding time" for explosives, the phrase, "analyze within 40 days after extraction" should be inserted. For VOCs, ("if required") the possible use of methanol preservation should be considered versus the purge and trap technique, if it can be used to address any specific objectives that are significant. This technique will provide more accurate data, but at the expense of higher detection/reporting limits in soil. The purge and trap technique will provide lower reporting limits, but report results which may be biased low by a factor of ten. For metals, it is advisable to dry and homogenize the samples using particle size reduction prior to analysis, per a recent Dave Payne memorandum to improve analytical precision.

39. Table 4-7, p.27

For cyanide (reactivity), note that this a poor analytical test, encumbered by extremely low % recoveries. Under the TCLP box, for VOCs, the phrase, "from preparative extraction to analysis - 14 days" should be added in the holding column; for SVOCs & pesticides, add the phrase, "TCLP extraction to preparative extraction - 7 days, preparative extraction to analysis - 40 days"; for TCLP metals (mercury) add " field collection to TCLP extraction 28 days, from TCLP extraction to analysis 28 days:; for metals add, "180 days to TCLP extraction, 180 days from TCLP extraction to analysis". Referring to the pH test, two days seems excessive for a pH measurement.

40. Table 4-7, p.28

For sulfide, a vague reference to "SW-846 methods" is insufficient. For water analyses, if this is a reference to the NPDES testing, perhaps some of this information should be deleted.

41. Table 4-7, p.30

The reference to the "local laboratory" should be further defined. Note that the final approval, when it is issued, shall only apply to the proposed laboratories which were evaluated for the project. Other laboratories may not be added to the testing program as a form of "corrective action" after the QAPP is approved, without prior approval from the U.S. EPA.

42. Section 5.1.3

Details of the laboratory QA/QC chain of custody were not provided.

43. Section 5.1.4, p.7

Discussion of the use of sample tags should be included.

44. Section 5.3

Prior to disposal of the final evidence file, it should be offered to the U.S. EPA. How long will the final evidence file be retained by Morrison Knudson prior to redistribution?

45. Section 6.1

These procedures should have been tabulated. The presentation adds nothing of substance to the QAPP. (Examples in the Region 5 Model QAPP will help the QAPP writers revise this section.)

46. Section 7

A table indicating both field and laboratory SOPs should have been included in this section. This table should have referred to all analytical instrumentation as well as sample preparation procedures. There is insufficient detail concerning the field procedures.

47. Section 7.2.1

It may be necessary to modify this section based on previous comments. Which detection limits will be reported, PQLs or CRDLs, for the CLP data?

48. Section 7.2.2

Referring to the second sentence, for all VOCs, all hazardous constituents indicated in Table 1-2 should be present in the matrix spiking solution. (i.e. This would be one way of optimizing the target compounds through quality control. Another would be through full calibration for these compounds.)

49. Section 8.2

This presentation adds nothing of substance to the QAPP. Note that much of this information could be tabulated.

50. Section 9.1.1

An item of concern still concerns how the field immunoassay reporting limit data will compare to the DQLs. Here it is stated that a mobile field GC will not be used. However, it may prove more useful to rely on a field GC for TCE and other VOCs data, than to rely on immunoassay data. (There is no TCE immunoassay SOP included in the Appendix.)

51. Section 9.1.2, p.2

Laboratory data reduction for non-CLP methods should also be discussed.

52. Section 9.2.1

Immunoassay data validation could involve more than checking transcription errors.

53. Section 9.2.2

Independent data validation should be performed on 100% of the data, not 10%. Who will perform independent validation of laboratory data?

54. Section 11.1

Refer to the sample table included in the Region 5 Model QAPP (5/23/93 guidance). A tabulation of the preventative maintenance critical spare parts for equipment and frequency of inspection and/or repair should be added to this section.

55. Section 12.1

The word "measured" should be inserted before the words "amount" in the equation for %R.

56. Section 13.3

This section is very general and should be fortified with pertinent details.

57. Section 14

It appears as if QA reports will only be generated following an audit. Wouldn't it be anticipated that more routine reporting would also take place?

58. Figure 1-1

The "pilot" study is not distinctively represented in this table, so it appears as if the pilot study bears no special significance. Screening of liquids prior to adding to compost piles should be reflected here. The inverted symbol indicating "yes" sometimes doesn't flow properly with parts of the diagram. The figure should be clarified and/or revised based on this and previous comments.

E. Nitrogen Field Testing

Although unproposed in the QAPP (biodegradation study - see Table 1-2 for instance), it is conceivable that when compared to suitable background sampling locations, tests for forms of nitrogen in excavated SWMU areas could pose one indicator of the possible presence of explosives compounds (e.g. biodegradation products). However, it is understood that other field tests designed for explosives parameters could address this objective more reliably, directly and readily than laboratory tests for nitrogen containing compounds. The QAPP has proposed nitrogen testing merely as a means of monitoring compost piles in the biofacility.

F. Compuchem Method 8330 data packages

Appendix E to the US Navy Crane biodegradation QAPP. The following comments concern the sample data package for 8330 explosives parameters that was provided.

1. Reporting limits reported in the sample data package (Appendix E) are consistently higher than PQLs expressed in 8330 SOP (Appendix D). Still, these do not exceed the residential soil PRGs (if the latter bear any relevance to this project). Sample extracts did not have to be diluted.

2. It is apparent that for several samples, RDX was present in the form of low level contamination.

3. Surrogate (nitroglycerin) recoveries were consistently near 100% (i.e. 94% to 108%). Matrix spike results (for all 14 target analytes) were consistently near 100% (i.e. 97% to 117%). Precision was generally good.

4. LCS results were not as good as MS data, although consistently within (rather broad) laboratory control limits of 30% to 150%. RDX was at 134% and tetryl was at 35%. For other 12 analytes, % recovery data was consistently in 97% to 116% window.

5. From control chart data that was provided, Compuchem has experienced some particularly poor precision in the past ("blank spikes") for 4-NT, 2,4,6 TNB and 1,3,5 TNB.

6. An inconsistency was noted in the uv wavelength chosen for detection. In Appendix E, "HPLC Parameters for 8330", the uv wavelength is stated to be 210 nm. However, in the 8330 SOP, section 2.0, it is stated that explosives parameters will be determined at 254 nm, which is the recommended wavelength.

7. RDX and tetryl tend to have poorer precision (around or greater than 15% RSD) in establishing initial calibration factors.

8. Inexplicably, some data sheets containing explosives data are marked "pesticide continuing calibration check".

9. On initial calibration chromatograms, 2,4 DNT and 2,6 DNT resolution seems acceptable.

G. Objectives

1. The proposal for sampling the compost pile was only briefly outlined in the Operational Plan. There are no plans for statistically evaluating the performance of the piles or for assessing the composited samples in the means of establishing the final cleanup levels. However, this is also a project objective related matter. In order to determine the clean up levels for explosives compounds, the conceptual approach for statistically assessing the compost data (and establishing that representative samples of the compost piles have been analyzed both infield and in the laboratory) during the pilot study, must be clarified.

2. The contractor needs to tighten up the objectives through adequate presentation.

H. Explosives Sample Preparation and Analytical SOPs

1. Although Table 1-2 of the QAPP has 8 compounds listed, Table 1 of the 8330 analytical SOP has 14 compounds indicated. Data should be reported for all 14 compounds.

2. A quick comparison of the PQL/reporting limits for the method to the Region 9 PRGs for the 14 target analytes indicates that the method should suffice for both soil residential and industrial use levels. Dilution of highly contaminated samples will result in higher reporting limits, which would pose difficulties in cases of determining whether RDX and 1,3,5 TNB were below targeted health assessment levels.

3. Selection of surrogate compounds for the method

   Rationale should be presented fortifying the proposal to use nitroglycerin (which could be encountered as a site contaminant). Also, although it is stated that 3,5 dinitroaniline will be used for " a specific project", is it anticipated that it will be used for this project? Finally, for other EPA approved projects involving the sampling and analysis of soils for explosives target compounds, the compound, 3,4 dinitrotoluene, was proposed as a surrogate. What is the rationale for not using this compounds as a surrogate?

4. Section 6.3.2.2.2, page 10 of the method

   Acceptance criteria for verifying that the initial calibration is still valid should be cited. It would be appropriate to reference section 6.2.3 of the SOP, where the daily calibration criteria of plus or minus 15% is cited as well as corrective action in the event that criteria are exceeded (if this is the case).

5. Section 7.7, page 13 of the SOP

   Referring to analyses of reagent blanks, how is "contamination" defined?

6. Section 6.3.2..2.1, page 10 of the SOP

   The procedures that will be implemented if the % RSD for initial calibration happens to greater than or equal to 20% should be stated.

7. It does not seem as if it has been proposed to analyze either laboratory control spikes or QC checks (prepared from sources independent from standards used for calibration). Unless rationale can be provided for why it is not necessary to analyze such samples, then procedures for adding protocol for preparation and analysis of LCS and QC checks should be considered and proposed.

8. Section 7.2

Where it is stated that MS/MSD sample collection frequency can vary for different monitoring programs, the actual frequency for this project should be 1/20 as stated in section 4.3.1.5 of the QAPP. Also, the composition of the matrix spiking solution and means for its preparation should be stated. This should include all parameters of interest. What will the nature of corrective action be if acceptance criteria for MS/MSD are exceeded.

9. Table 3-2

Table 3-2 of the QAPP summarized QA Objectives for Precision, Accuracy, and Completeness for Ordnance Compounds. This table should be revised to reflect limits expressed in the laboratory SOP, because there are inconsistencies between the acceptance criteria. (Just one example involves the surrogate for soil, which is stated at 50% to 150% in section 7.8 of the analytical SOP, but indicated to be 50% to 140% in the QAPP table.) Also, although the study is intended to emphasize soils (and compost material), Table 3-2 provides data for water matrices. Presumably this information (as well as the salting out analytical procedure for explosives in waters) will only be relevant to analysis of wash waters and equipment rinse blanks.

10. Section 6.3.3

Although section 6.3.3 outlines a procedure for establishing retention time windows, it is not indicated in the QAPP what the anticipated RRT will be for explosives compounds both on primary and confirmation columns.

11. Section 5.4

The initial calibration level range seems to be rather limited in extent (0.2 approx. to 6.0 approx. ug/kg. The units are not indicated in this section, although presumably they are in ug/kg for soils). Other projects which have been approved have had broader ranges (i.e. up to 80 ug/kg). Presumably, this means that samples will be subject to dilution on a more frequent basis in the event that measured concentrations are found to be beyond the upper range of the linear calibration curve.

12. Soil extraction procedure for explosives

The coupling of field explosives testing to the required laboratory tests should be better explained or fortified with rationale. As proposed, the project seems heavily reliant on immunoassay tests, proposed as a basis for when to select samples for laboratory confirmation. Carol Witt-Smith, the project officer for this project, has directed that field colorimetric tests for TNT and RDX be substituted for the immunoassay tests. The objectives for field testing of explosives testing of soil excavation and compost piles should be better defined.

13. Referring to the soil extraction procedure for explosives compounds, it is ancicipated that high TNT and tetryl concentrations will be detected. High TNT concentrations may interfere with 2,4 DNT confirmation unless a procedure can be developed to counter this anticipated difficulty. (See "Development of an Improved confirmation Separation Suitable for Use with SW846 Method 8330", T. Jenkins and S. Golden, June 1993, US Army Corps of Engineers Special Report 93-14.) Also, the eluent expressed in section 6.1.3 does not contain acetonitrile. Addition of acetonitrile may reduce overall retention times for late eluting compounds and improve 2,4 DNT and 2,6 DNT resolution. (An eluent composition of water 65%, MeOH 12%, and ACN 23% using a LC cyanopropyl column proved most effective in one study, notwithstanding tetryl degradation tendencies. See previously cited report by Jenkins and Golden.)

14. Table 4-7 of the QAPP

The explosives holding times for explosives is incompletely indicated. Note that recent studies have indicated that the optimal preservation technique for explosives compounds (especially for nitroaromatics) would be freezing to -15 degrees C for no longer than eight weeks. (See "Experimental Assessment of Analytical Holding Times for Nitroaromatic and Nitramine Explosives in Soil", by C. Grant, T. Jenkins, and S. Golden, US Army Corps of Engineers, June 1993, Special Report 93-11.) It is recommended that this preservation and holding time guideline be followed for this project.

15. Referring to section 2.2 of the soil extraction SOP, (and comment 13 above), where it is stated that methanol is preferred as the extractant when HMX is an important target analyte, for this project acetonitrile should be utilized as the extractant because HMX happens to be only one of several significant target analytes. HMX is known to be present at several site SWMUs in significant quantities, however. Therefore, if field tests indicate its prevalence relative to other explosives constituents, perhaps these samples should be extracted with methanol instead.

16. Should the compound picric acid (and its biodegradation products) be added to the target parameter list? If so, then analytical procedures and specific objectives for assessing the data should be proposed.

I. Metals Analyses

1. An SOP method for mercury analysis was not provided. In order to report mercury as part of the TCLP data package, a complete analytical procedure for measuring mercury in soil matrices must be incorporated into the QAPP.

2. There were no analytical reporting limits provided for the ICP method for soil matrices. Therefore, it is difficult to compare the SOP analytical sensitivity to the decision levels proposed in Table 1-2 of the QAPP. Some assessment as to whether or not the analytical SOP for metals will provide sufficient analytical sensitivity in order to meet specific objectives should be provided in the QAPP.

3. Table 1-2 of the QAPP

   It is apparent that the less rigorous DQL for arsenic in soil of 22,000 ug/kg (nca) has been selected as a "decision level" instead of the more rigorous goal of 320 ug/kg (ca). Will this be sufficient for project purposes? This is partly a toxicological matter which should be resolved after consulting with staff toxicologists.

4. Table 1-2 of the QAPP confuses two terms, "decision levels" with analytical reporting limits. What decisions will be made if metals contaminants are found to either exceed or be less than the decision levels stated in Table 1-2 of the QAPP? The "decision levels" indicated in Table 1-2 are not intended to be regarded as laboratory (arbitrary) reporting limits.

5. Although section 2.1 of the ICP SOP refers to method 3050 for soil digestion, a complete soil digestion procedure was not included in Appendix D. (Note that soil samples should be dried and then homogenized using particle size reduction, prior to sample digestion, per a recent Dave Payne memo, dated July 19, 1996.)

6. The page numbering in the header of the ICP SOP is inconsistent throughout the document (although this does not seem to affect the continuity).

7. Section 5.6.1

   ICV standards should be prepared independently from solutions used for initial calibration. Results should agree to within 5% of "true values", not 10%.

8. Section 5.6.2

   it should be stated that CCV should be the midlevel calibration standard.

9. Initial calibration should consist of 3 levels and a blank. (This is not clear in the SOP.)

10. Section 5.6.8

    This procedure is apparently intended to serve as the analog of an MSD. However, (and this is stated so that there will not be any confusion in terminology), field duplicates should still be collected at a 1/10 frequency. This information should be clarified by submission of a sampling network table.

11. It should be more clearly explained in the QAPP when (and on which samples) TC leach extract metals analysis will be performed versus total metals analysis, involving a soil digestion procedure.

J. Total Organic Carbon SOP

1. Section 6.1

   The homogenization/pulverization process was not adequately described. (Will a blender be used?)

2. Section 7.5

What is the anticipated TOC level? If greater than 2,000 ppm (0.2%), then less sample will be needed, (or dilution of the sample with clean sand or some other suitable matrix will have to be performed). Also, calculations of final results would have to be adjusted accordingly.

3. Section 6.2

The effervescence technique (performed to eliminate inorganic carbon) may have a tendency to volatilize VOCs that would otherwise contribute to the TOC result. It should be discussed whether this outcome would be anticipated to pose difficulties for assessing biodegradation data. If that is the case, total carbon (TC) and inorganic carbon (IC) should be separately determined analytically. (TC - IC = TOC). After adding acid, the samples and standards should be purged using nitrogen for 10 minutes. (See section 7.3 of SW-846, Method 9060.)

4. Section 7.6

Quadruplicate analysis should be performed for this project. As per SW-846 guidance, the average result, and range of results should be reported per sampling event, with respect to each composited sample location. The sampling frequency and proposed reporting scheme should be clarified in the QAPP.

5. Section 8.1

Evidently, there were no specific provisions for MS/MSD TOC analysis in the QAPP. However, the QAPP should be revised to reflect that MS/MSD analyses shall be performed, as proposed in the TOC SOP. A comprehensive sampling network table would clarify this matter.

6. Sections 4.0, 5.12, 5.16, 5.18, and 7.4

Although 4 initial calibration standards are described, and there appears to be a linear working range defined for the instrument, there do not appear to be functional uses for the 25 ppm and 4 ppm standards. This is because there are no acceptance criteria defined for establishing a successful (i.e. linear) initial calibration. Calculations of sample concentration as well as 2% standard deviation acceptance criteria for initial calibration are founded

on the result for the 2,000 ppm standard. How is calibration verified to be "acceptable' through analysis of the 50, 4, and 25 ppm standards (see section 5.16). Besides precision, (i.e. criteria of 2% standard deviation for the 2,000 ppm standard), has the laboratory established minimum response levels in the form of control charts for the IR detector's response to any of the 4 standards. What is the rationale for not also proposing an initial calibration standard intermediate between 50 and 2,000 ppm?

K. TKN, Nitrate/Nitrite, and Ammonia SOPs

1. Table 1-1

Table 1-1 of the QAPP, biofacility section, refers to a TKN analysis, which is based on U.S. EPA method 351.2. However, there is no SOP for TKN appended to the QAPP. Since TKN will be one of the primary analytical tests providing indication of biodegradation feasibility, this SOP should be submitted for review. The proposed TKN procedure should include sample preparation steps that will be necessary, allowing analysis of TKN in a waste matrix at fairly high concentrations ranges (possibly even exceeding the instrumental calibration range).

2. Table 1-1 of the QAPP, biofacility section, refers to nitrate and ammonia analyses (for waters) founded on U.S. EPA methods 353.1 and 350.2, respectively. However, the SOPs for these methods, included in Appendix D, are apparently based on U.S. EPA methods 353.2 and 350.1, respectively. The selection of SOPs should be consistently reflected throughout the QAPP once method selection has been made on the basis of specific project objectives.

3. Certain TKN methods fail to account for certain forms of nitrogen. Some explanation should be provided in the QAPP as to whether the explosive compounds under this facility investigation are intended to be accounted for as TKN. If TKN is insufficiently sensitive to the presence of explosives compounds, as well as other forms of organic nitrogen (plus ammonia), then the purpose of utilizing this test should be clarified. As stated on page 4-75 of Standard Methods for the Examination of Water and Wastewater, 19th ed., 1995, organic nitrogen is a term that includes such natural materials as proteins and peptides, nucleic acids and urea, and "numerous synthetic" organic materials. However TKN can fail to account for certain

nitrogen forms such as nitro and azine groups. As you are aware, various species of nitrobenzenes are known to be site contaminants, and a key target parameter, RDX is also known as hexahydro - 1,2,5-trinitro - sym triazine. It is important to fully consider the purpose of testing prior to selecting methods, and if it has been already determined that TKN should provide the right sort of data for a specific aspect of the compost investigation, then such assurances and rationale for using TKN for this purpose should be incorporated into the QAPP.

L. VOCs

1. Although Table 1-1 of the QAPP suggests that VOCs will be analyzed by CLP-SOW at the ABG, Rockeye, and Biofacility SWMUs, ("if directed" by Project manager), Table 1-2 indicates only 8 VOC analytes will be analyzed at the ABG and a separate list of only 5 VOCs will be analyzed at the Rockeye SWMU. (It isn't clear which constituents are of concern to the biofacility.) All VOCs that are possible to report using the finally approved VOCs method should be reported, if detected. The QC associated with the respective SWMUs should be dependent on the key VOCs of concern with respect to each SWMU. It is requested that VOCs be analyzed at each of the areas indicated in Table 1-1, but that the specific objectives for performing these analyses be further defined.

2. The rationale for collecting VOC data for any purpose should be coupled effectively with the stated purpose for reporting key VOCs in the laboratory. For instance, will samples intended for laboratory VOC analysis be taken in areas where field testing showed nondetectable VOCs levels to prove "absence" of contamination defined at some decision level, or, alternatively, to "confirm" field data in areas where high levels of key (or any) VOCs were detected?

3. The VOC SOPs still need to be evaluated.