



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

N00164.AR.000265
NSWC CRANE
5090.3a

REPLY TO THE ATTENTION OF:

December 16, 1996

DRP-8J

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

RE: Quality Assurance Plan
Bioremediation Facility
Naval Surface Warfare Center
Crane, Indiana
IN5 170 023 498

Dear Mr. Brent:

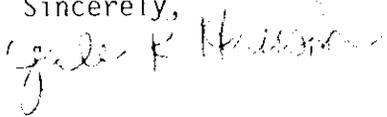
The purpose of this letter is to transmit our technical comments on the Quality Assurance Plan for the Bioremediation Facility, dated November 1, 1996, including the Method 8330 SOPs. Our specific comments are included in Attachment I. Although the majority of the document has improved greatly, a final revision addressing the comments is in order prior to approval. However, do not be alarmed by the number of individual comments. Some of these comments could potentially be addressed by the Navy clarifying some of their reasoning or procedures.

The major remaining deficiency is that the fixed-laboratory (Southwest) has not been represented in this document. Obviously, some means of gaining an appropriate level of comfort with the projected laboratory performance must be achieved prior to approving the QAPP. At a minimum, we would hope that any submitted SOPs would only be submitted after the Navy discusses the specific points noted as deficiencies in the review of CompuChem's SOPs, (last August) for the original QAPP.

Also included in this letter is information to clarify our request for an audit of the explosives test method prior to the start-up of the pilot-scale. The outline for the audit demonstration is included in Attachment II.

Please have your contractor create a response to comments document along with the revisions in order to speed the final review. If you would like to have a conference call or meeting to discuss any of these issues, please call me or Allen Debus to arrange a time. If you have any questions regarding this matter, please contact me at (312) 886-6146.

Sincerely,



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

cc: Jim Hunsicker, NSWC
Steve Downey, MK at NSWC
Adrienne Wilson, SOUTHDIV
Tom Linson, IDEM

ATTACHMENT I

Comments on the Quality Assurance Plan for the Bioremediation Facility
Dated November 1, 1996
Naval Surface Warfare Center
Crane, Indiana

A. THE 8330 SOP FROM SOUTHWEST LABS

1. Section 1.A

This SOP can be used to report a compound, tetryl, which is not on the QAPP target parameter list, Table 1-4. The SOP is missing two target parameters which appear on the QAPP Table 1-4, 1,2 dinitrobenzene and 1,4 dinitrobenzene. The SOP and QAPP do not address picric acid. Would there be any intended data use for this compound?

2. Section III.B

In Table 1-2 of the QAPP it is apparent that relatively high concentrations of tetryl are anticipated. If it is an objective to report reliable and representative data for tetryl, perhaps the anticipated interference issue should be considered to further degree.

3. Sections V.D & V.E

The nature of the Working Standards intended for analysis of soil samples should be more fully defined. Referring to V.D.2, what is considered to be the appropriate solvent for soil samples? (Shouldn't it be a 50% acetonitrile/50% aqueous solution of 0.5% calcium chloride?)

4. Sections V.H and VIII.E

The recommendation to use 3,4 DNT is a valid proposal, as it has been successfully used in other RCRA projects outside of the Region.

5. Section V.I & VII.B

It is not evident which eluent will be used.

QAP and SOP Comments Continued....

6. Section VI.B

One conclusion of CRREL report # 93-11 (June 1993) was that samples collected for analysis of explosives parameters should be kept frozen, especially when TNT happens to be a key parameter, as is the case with this project. Therefore, it is recommended that until extraction, samples intended for TNT analysis be kept frozen, (not simply chilled to 4 degrees C).

7. Section VI.C & VII.A.2.a

With respect to the concern raised in the previous comment, rationale should be provided explaining why soil samples should be air dried prior to extraction.. For homogenized samples, CRREL report # 93-11 recommends use of a vortex mixer for one minute, instead of the mortar & pestle apparatus that are proposed.

8. Section VII.A.2.a & VII.A.2.b

Note that if undried samples are subjected to analysis, then to mitigate sample heterogeneities, a 5.0 gram subsample should be analyzed instead, (provided the sample isn't suspected to contain high concentrations of analyte).

9. Section VII.C

In the equation for %RSD appearing at the top of p. 8, shouldn't the denominator instead refer to the mean calibration factor?

10. Section VII.C.5

Although there is a reference to PQLs in this section, both Table I and section I.C refer instead to EQLs.

11. Section VIII.A

The meaning of the term, "contamination" should be further defined.

12. Sections VIII.A.2 & VIII.C

Is there a suitable Crane source for the "clean soil" method blank, which will reflect the actual sort of matrix to be encountered in contaminated areas? Will the soil method blank be used for this project? Is "clean soil" to be used for the LCS too?

QAP and SOP Comments Continued....

13. Table 6

The control limits should be generated for all explosives parameters listed in QAPP Table 1-4.

14. Section X.A

The term "PQL" is used in this section. See comment #10 above.

15. Appendix G

The data package does not alleviate several concerns pertaining to the matter of how data will be documented and reported. No HPLC chromatograms were provided, and the data package does not generally correspond to what would constitute a "CLP-like" data deliverable package. The data package submitted was for a set of water analyses, yet the proposed project is for soil matrices. It would be more meaningful to submit a more complete data package for a recently completed soil study. In the submitted data package, it is apparent that 3 calibration levels were chosen, when for the Crane project, there should be 5 initial calibration levels and a method blank. Although this Appendix is claimed to be an explosives data package, there is more pesticides, herbicides and metals data included than explosives data. (The title of Appendix G is a misnomer.)

B. QAP

1. Please note that this review does not cover possible Superfund agenda, or address specific objectives that may be pertinent to Superfund. This is intended to solely comprise a RCRA review.

2. Section 1

In the fifth bullet, it should be clarified whether or not process goals will be founded on risk/health data, or other criteria.

3. Section 1.3.4, page 8

In the first paragraph, would the presence of bituminous solvent contribute to a PAH problem? Should PAHs be added as a field or lab parameter for any particular objective associated with the pilot study?

4. Section 1.4.1, page 10

At the top of the page, if PID readings were instead not taken, would adversity result in adequately characterizing the site?

QAP and SOP Comments Continued....

5. Section 1.4.1, page 10

Under data collection Task 3, if NPDES limits can't be met, will water be considered for windrow addition? What criteria will be used to allow windrow addition?

6. Section 1.4.1, page 11

Referring to the 4th paragraph, is there any anticipated need to perform risk assessment in addition to directly comparing values?

7. Section 1.4.1, page 12

Should VOCs also be included in the sampling scope for Data Collection Task 5?

8. Section 1.4.1, page 12

In Data Collection Task 6, will retreatment require a workplan and QAPP modification? Should LDR criteria be more fully represented in this QAPP, wherever applicable?

9. Section 1.4.2, page 12 of 29

Are these intended uses for laboratory data only, or also for field data?

10. Section, 1.4.2.2, page 13

The 1st and 2nd full paragraphs on this page create confusion over rationale for determining an indicator of pilot test success. Will bench scale testing be performed prior to Task #6? (This could involve recomposting and. new pilot scale tests.)

11. Section 1.4.2.2

In the third full paragraph, then why not simply use 1,400 and 680 ppm, respectively? What is the basis of the 5 ppm level?

12. Section 1.4.2.2

On page 14, I am under an impression that although this < 30 ppm level sounds rationally founded on the premise of risk criteria, in reality, the original basis for it was a field reporting limit for an RDX test kit.

QAP and SOP Comments Continued....

13. Section 1.4.2.3

How does a "performance goal" relate to a "process goal", or even an "interim remedial level"? What is the relative significance of these 3 types of definitions?

14. Section 1.4.3

At the top of page 15, this is presumably a reference to Task #1. Maybe the paragraph could be modified to reflect this fact.

15. Section 1, Table 1-3

Referring to Task 1, what observations and criteria specifically would dictate a need for VOCs testing.

16. Table 1-3, Task 1/Data Use

Given that soil contamination heterogeneity might result with respect to explosives, "confirming" previous test results may be tricky. Clarify the purpose of delineating excavation limits. (Also see the second bullet on page 9 of 29.)

17. Table 1-3, Task 1, Data Collection

Are the VOCs indicated here being tested to compare to TCLP levels? (See page 10 of 29, last sentence of section 1.4.1, Task #1.) The QAPP should more fully distinguish between the meaning of "TCLP" and "RCRA" metals.

18. Table 1-3, Task 2, Data Collection

What are the "decision" associated with results of the equipment wash, water decontamination sampling?

19. Table 1-3

Is soils data needed for hexavalent chromium? (i.e. RCRA metals group).

20. Table 1-3, Task 4, Data Use

The field process monitoring measurements associated with the compost pile monitoring should also be mentioned as part of this Task #4 entry.

QAP and SOP Comments Continued....

21. Table 1-3, Task 5

Is Task 5 deferred to the Full Scale Operations QAPP? (This is a little confusing.) What is the difference between "post excavation sampling" and "confirmation sampling" mentioned in Task #5.

22. Table 1-3, Task 5, Data Use

Shouldn't metals also be mentioned? (See page 12 of 29.) Also, should VOCs be mentioned here?

23. Table 1-3, Task 6, Data Quality Use

Are LDR criteria relevant to this task? If so, perhaps a specific parameter list (more detailed than mentioned either in this Table or in Table 1-5), should be indicated.

24. Table 1-4

Note that CLP's CRQLs and CRDLs should not be relied on for reporting data. Does the term "Acceptable Reporting Limit" reflect the laboratory's actual capability? Under VOCs, to the extent possible, the term "if required", should be eliminated prior to QAPP approval. Note that there are no TC levels established in 40 CFR Part 261 for some of these VOCs. (See note appended to Task #1, Table 1-3. Why choose two separate methods, 8015A and 8015B. Why not rely on a single SOP?

25. Table 1-5, Task 1, Analytical Method

To match the third column, a line space is needed after the "RCRA metals" line.

26. Table 1-5, Parameters to be Reported

Additional VOCs should be reported by method 8240. Determining hazardousness of soil with respect to VOCs may be a problem because only a short list of VOCs has been proposed, and only a few of these have associated TC criteria.

27. Table 1-5, Task 6

This task, as presented, doesn't reflect the extent to which LDR criteria may be pertinent. Should references be made to the full organic scan of the TC organics group. In the third column, it is necessary to know what the "full list" is comprised of.

QAP and SOP Comments Continued....

28. Section 2.2, page 1

In the introductory paragraph, it should be indicated that the RCRA Project Coordinator will also take responsibility for approving project objectives.

29. Section 2.3, page 3

The term "RQAM" should be changed to "QAPP Coordinator".

30. Section 2.4, page 4

Does the Laboratory Operations Manager perform in-house data validation? If not, then who does?

31. Section 2.4, page 4

How independent of the laboratory is the Laboratory Quality Assurance Officer? It should be stated that this person will perform independent data validation, if this is the case.

32. Section 3.6, page 5, last paragraph on page

The frequency for field duplicates should be 1 per 10. For pilot study, there should be one per field sampling event, per parameter group (yet, no less than 10).

33. Tables 3-1 through 3-3, pages 6 to 8

It should be stated which VOC compounds this criteria specifically applies to. What are the surrogate compounds for all organics methods indicated in this table? What are the matrix spiking compounds for all respective organics methods? More details are needed concerning determination of the parameter, "naphtha". Why is this a relevant parameter? Thus far, there are no assurances in this QAPP that naphtha can be confidently and reliably reported.

34. Table 3-4, page 9

Note that additional metals may be reported using ICP.

35. Table 3

Information pertinent to proposed field tests should be tabulated in an analogous fashion to how data has been presented for laboratory parameters.

QAP and SOP Comments Continued....

36. Section 4, page 1

Referring to the last paragraph in the introduction, will this really be a "temporary" backfilling, even if the outcome of testing produces an unfavorable result? How much flexibility will there be to direct further excavation, and remove the backfill, even if the area has been temporarily backfilled?

37. Section 4.1.1, page 2

Rationale for the 20' by 20' grid should be presented. Composites should only be collected for explosives samples. Special precautions required for collecting explosives samples if the levels of contamination are thought to be exceedingly high should be appropriately addressed in the Health and Safety Plan. What special precautions will be taken to minimize VOCs losses from soil during sample collection?

38. Section 4.1.1, page 3

It should be stated in this paragraph that this activity will be performed with respect to the explosives (& possibly VOCs) parameters only.

39. Section 4.1.2, page 3

Will the staging area be lined? By use of the term, "RCRA metals", is "Total" metals (full digestion) implied. Dave Payne's memo of July, 1996 is relevant to these samples.

40. Section 4.4.2, page 6

The purpose(s) of these additional field process monitoring measurements is not mentioned in Section #1.

41. Section 4.7.3, page 8

In the last sentence of this section, at a minimum, Type A and Type B should each be 1/20.

42. Section 4.7.4, page 8

The frequency of field duplicates should be 1/10.

43. Section 4.8.2, page 9: Explain the rationale for step #5 of the sample equipment decontamination process. Can the use of pesticide-grade hexane or isopropanol be eliminated? In most cases, can steam cleaning be performed instead to minimize field use of solvents?

QAP and SOP Comments Continued....

44. Table 4-1, page 14

Referring to the "QA/QC" section, note that there should be one field duplicate collected to adequately represent each parameter group. The field duplicate for VOCs should be collected in such a manner to minimize atmospheric losses.

45. Table 4-2, page 15

Referring to item 2, when or how will it be determined whether VOCs are required? Use of a nonperistaltic, slow rate pump is preferred.

46. Table 4-2, page 16

Will VOCs samples be preserved with HCl? (See Table 4-9.)

47. Table 4-3, page 17

When it will be decided if VOCs are required? Can it be decided now, or can some more specific criteria be proposed for conditionally requiring circumstances under which VOCs samples should be collected? Use of a bailer for collecting aqueous samples for VOCs analysis is not recommended.

48. Table 4-5, page 21

Does this refer to each grid spacing, or an area surrounding all sampled grid spaces?

49. Table 4-6, page 22

Many parameter groups are potentially involved here. More specific information pertaining to them is required.

50. Table 4-8

Under task #1, a field MS/MSD pair should be added for explosives. Under task #2, the POTW and NPDES parameter groups seem too broadly defined. Under task 4, the terminology presented for the field duplicate column is rather confusing.

QAP and SOP Comments Continued....

51. Table 4-9, pages 29, and 31

For VOCs, method 8015A, what is the rationale for a 30 day holding time? Why not 14 days instead? For method 8015B, why is the test parameter "naphtha" required. A custom SOP will be needed for this parameter. For PCBs/pesticides, will an SOP founded on method 8080 or 8081 be proposed? For PCBs/pesticides as well as herbicides, how many days later, following extraction, will the extract be analyzed for the respective parameter groups? For VOCs and SVOCs, will it be CLP or SW-846? These details should be decided now. For explosives samples, special sample preservation techniques outlined in the first notice of deficiency letter should be followed to avoid decomposition of target analytes. ("Ice to 4 degrees C", may be insufficient, according to recent research studies.)

52. Section 5.1.2, page 3

The type of preservative should also be added to the information included on the sample label.

53. Section 5.1.2, page 4

To clarify, it would help to know which of these codes will be applicable to each of the 6 tasks. (Also see Table 4-8.)

54. Section 5.1.3, page 7

Discussion of sample tags is absent from this discussion, yet briefly mentioned on page 9 of 10. In the last paragraph on the page, the laboratory QA/QC processing sequence (i.e. chain of custody) should be attached as an appendix.

55. Section 5.2, pages 8 to 9

Presently, Appendix D is blank. On page 9, for how long will the evidence file be maintained by MK?

56. Section 6.1

References should also be made to the Ensys explosives field test.

57. Section 6.2

A table is needed indicating acceptance criteria for each method per the Model QAPP guidance.

QAP and SOP Comments Continued....

58. Table 7-1, page 3

Obviously, until this QAPP has been fortified with the laboratory SOPs, there is a glaring deficiency in this section. Also, it would help to have information reflecting selection of field methods tabulated. (Note that the RDX method by Ensys, proposed by the Navy for use, based on SW-846 method 8510, has not yet been validated by the U.S. EPA. Consequently, this method, in particular, will require special review.)

59. Section 8.1

Appendix C should also be referenced.

60. Section 8.2

The bullets should be limited to QC checks that are internal to the laboratory. Field duplicates, field/trip blanks, and MS/MSD could be eliminated, as these are commonly regarded as field QC samples. The final paragraph in this section can be deleted, as it adds little substantive information. A table should be provided indicating specifically where the procedures for internal QC checks are located in each respective SOP. Then, for each type of QC check, the nature of the sample should be defined, the procedure for collecting or preparing the sample should be stated, the frequency of sample collection and analysis should be stated, the associated control limits should be defined, and the corrective action in the event that control limits are exceeded should be indicated.

61. Table 8-1, page 3

Raw data should be included, if requested, for initial calibrations. The proposed approach may suffice for RCRA reporting provided that we would have access to this information, if needed. This table should appear in section 9 of the QAPP instead.

62. Section 9.11.2, page 2

Referring to the first full paragraph, other equations will also be used, as defined in specific SOPs. In the last paragraph of this section, who will proof the data logging process? Will "qualified" data be logged into the project data base? What is the procedure for proofing the data logging process? Shouldn't data logging be restricted until after an independent data validation has been performed?

QAP and SOP Comments Continued....

63. Section 9.2.2, page 3

Referring to the final paragraph in this section, instead of validating only 10% of the confirmation samples, why not perform validation on most or all of the final and initial results and associated QC data. In other words, rationale may exist for deemphasizing validation review of samples thought to somehow less critical than others. (Note that Superfund's protocol might require a 100% validation of all samples.) Who will perform data validation of the proposed 10%?

64. Section 9.3.2, page 4

Information contained in this section could be combined with information presented in Table 8.

65. Section 10.2.1, page 2

The MK Project QC manager is not specifically identified in section 2 of the QAPP. In the third paragraph on the page, note that inclusion of an audit checklist would increase understanding of how audits will be performed.

66. Section 10.3.1, page 3

Referring to the next to last paragraph, shouldn't all this be done prior to analysis of samples by the laboratory? Otherwise, this sounds quite a bit like "validation".

67. Section 11.1, page 1

Why is TCE mentioned here as a field target parameter? This hasn't been defined previously. What is the test objective associated with TCE?

68. Section 12.1, page 1

Specifically, which kind of samples does this strategy (i.e. control charts) pertain to? (LCS, MS, blank spike, surrogate...etc?).

69. Section 13.3, page 2

The Laboratory Coordinator isn't specifically mentioned in section 2 of the QAPP. The Laboratory QA Manager is not specifically mentioned in section 2 of the QAPP.

QAP and SOP Comments Continued....

70. Section 13.4

Referring to the first paragraph, it should be stated that if the project objectives are not met, then resampling must occur. Referring to the second paragraph, which "project manager" is being referred to? This individual should correspond to a role already identified in section 2. (It shall not be a accepted course of corrective action to substitute a laboratory for the one which is eventually approved in conjunction with this QAPP.)

71. Section 14

Why are only "postaudits" (apparently) performed? Will the corrective action report mentioned in the next to last paragraph be included in the QA Report?

72. Appendix H, section 1.3

Note that a Level 3 would not be equivalent to CLP. Therefore, it would generally be regarded as insufficient for purposes of RCRA data documentation. Also, Superfund DQO levels are irrelevant to RCRA.

73. Appendix H, Table 1.1

Note that 10% field duplicates are generally recommended in the collection of RCRA samples. What is the rationale for proposing less than 10%?

74. Appendix H, Section 1.3.1

Note that standard performance evaluation samples may not closely correspond to site specific issues.

75. Appendix H, section 7.1

In the first paragraph, perhaps the compound dibutyl chlorendate is being referred to. In the case of PCBs analyses, this surrogate is generally not recommended for RCRA analyses. (A combination of 2,4,5,6 tetrachlorometaxylene, and decachlorobiphenyl is recommended.)

76. Appendix H, Table 7.6

Chromatograms and mass spectra for initial calibration may be requested by the U.S. EPA for final data validation.

QAP and SOP Comments Continued....

77. Appendix H, page 64

At the top of the page, note that 5 point initial calibrations (not 3 or 4 point) should be performed for RCRA work.

78. Appendix H, page 64

All key compounds of concern should be reported through full initial calibration. These should not be reported as TICs. References to the TCL create alarm because generally the CLP TCL is a "short list". The "full list" referred to in this QAPP, should be fleshed out through the addition of calibration standards to more fully represent the concerns of RCRA. Also, the QC criteria associated with each SOP should correspond to SW-846 QC.

79. Appendix H, page 65 item 3

Explosives compounds are not included on the TCL. How will data for explosives be validated?

80. Appendix H, page 66

The final sentence on the page is incomplete.

81. Appendix H, section 8.3

The reference to Table 7.7 should really refer to Table 7.6 instead. Also, the deliverables referred to should be comparable to a CLP-like data package.

C. ADDITIONAL CHANGES

1. In the QAPP revision insert specific language for homogenizing and splitting samples from the soil excavation area into section 4.1.1 of the QAPP.. (See pages 7 and 10 of the Region 10 report.)
2. Add the special provisions for homogenizing the compost samples as stated on the bottom of p. 7 of the Region 10 report into section 4.4 of the QAPP.

QAP and SOP Comments Continued....

D. Appendix C: Comments Concerning the Field Test Kit for the TNT Parameter

1. Page 2

Although there are noted difficulties associated with an air drying step, especially if the time involved for drying to < 10 % moisture will take longer than about 2 hours, the rationale and associated measurements that will be used to determine when air drying would be considered necessary should be provided. (Note that in SW-846, Method 8515, section 7.2, it is stated that soil samples should be air dried.)

2. Page 4

The wavelength used to measure TNT absorbance (540 nm) should be recorded in the field operating log, prior to each batch of measurements.

3. Page 7

What is the basis for the four times factor? In Talanta, vol. 39, no.4, p.423, 1992, "Development of Field Screening Methods For TNT, 2,4-DNT, and RDX in Soil", by Jenkins and Walsh, note that a factor of 2 is suggested instead. Also, if TNT soil concentrations are > 30 ppm, and dilution of the extract is necessary, wouldn't the four times factor have to be adjusted for the dilution factor (because the concentration of soil humic matter is being diluted as well)?

4. Page 8

The daily TNT control sample is required for the Crane project. (It is not optional.) What is the certified concentration of the TNT control standard?

5. Page 9

Note that the test operating temperature must be within the range of 40 to 100 degrees F. There should be a note added to the field operating log indicating whether or not the test kits have been adequately stored (with respect to shelf life and storage temperature), prior to using any TNT test kits. The expiration dates of all test kits used must be recorded in the log book. Add this protocol to the QAPP.

QAP and SOP Comments Continued....

6. Page 10

At a minimum, a three point daily initial calibration must be performed that will encompass the linear working range of 1 to 30 ppm. Additionally, the suggested QA/QC procedures must be employed. Appropriate sections of the QAPP should be revised to describe specific procedures for implementing adequate QA/AC. At a minimum, a method (acetone) blank, MS/MSD, continuing calibration standard, and field duplicates should be used. The results should be confirmed at an adequate level of confidence using method 8330 results which have been proposed. (Under these circumstances samples should be split according to procedures outlined in the Audit Demonstration approval.) For each type of QC sample, procedures defining how the sample will be collected, the frequency of collection, the acceptance criteria after analysis, and relevant corrective action procedures should be inserted into appropriate sections of the QAPP.

7. General

Provisions for performing replicate TNT field analyses when initial results are < 1 ppm should be added to the QAPP. What is the sample size to be field collected? If the sample size is larger than the amount used for extraction, prior to analysis, samples should be field mixed and homogenized so that the analytical measurement will adequately represent the contents of the sample jar and the area subject to testing, according to procedures defined in Draft Version 6, May 28, 1996, EPA Federal Facilities Forum issue, "On-Site Analytical methods and Field Sampling For Explosives in Soil", by Crockett, Crain, Jenkins and Sisk.

E. Appendix C: Comments Concerning the Field Test Kit for the RDX Parameter

1. Pages 2 to 4

It should be stated whether the same extracts will be used for both RDX and TNT field parameter tests. How will extracts be labelled, stored, etc., in between testing. Testing of TNT extracts should take place before RDX testing.

2. Page 2

Provisions for air drying samples intended for RDX field analyses to < 10% moisture should be added to the QAPP.

QAP and SOP Comments Continued....

3. General

Only high purity acetone should be used for extraction.

4. Pages 5 & 9

What does the .05 threshold absorbance level at which "presence" of nitrate/nitrite will be defined correspond to in terms of concentration units. It would be preferred if all extracts are subjected to anion cartridge extraction.

5. Page 6

Can the absorbance setting be fine tuned to 507 nm instead of 510 nm? The absorbance setting used in the measurement of RDX should be recorded in the field operating log prior to each batch of measurements.

6. Page 7

What does the .014 absorbance value correspond to?

7. Page 8

Testing of the RDX control sample should be performed daily, not on an optional basis. It can be used as a continuing calibration standard, reanalyzed with each batch of samples. What is the certified concentration of the RDX control sample?

8. Page 10

Note that the operating range of the RDX test kit is 40 to 100 degrees F.

9. Page 11

QA/QC procedures mentioned in this section are not optional and must be employed. Appropriate sections of the QAPP should be revised to describe specific procedures for implementing adequate QA/QC. At a minimum, a three point daily initial calibration must be performed that will encompass the linear working range of 0.8 to 30 ppm. Additionally, the suggested QA/QC procedures must be employed. Appropriate sections of the QAPP should be revised to describe specific procedures for implementing adequate QA/QC. At a minimum, a method (acetone) blank, MS/MSD, and field duplicates should be used. The results should be confirmed at an adequate level of confidence using method 8330 results which have been proposed. (Under these circumstances, samples should be split

QAP and SOP Comments Continued....

according to procedures outlined in the Audit Demonstration approval.) For each type of QC sample, procedures defining how the sample will be collected, the frequency of its collection, the acceptance criteria after analysis and relevant corrective action procedures should be inserted into appropriate sections of the QAPP.

10. General

Provisions for performing replicate RDX field analyses when initial results are < 0.8 ppm should be added to the QAPP. What is the sample size to be field collected? If the sample size is larger than the aliquot to be subjected to measurement, then the sample should be field mixed and homogenized prior to analysis as defined in the previously referenced EPA Federal Facilities Forum Issue document. On each operating day, for both RDX and TNT testing, an item of known mass should be weighed on the field balance to determine whether or not the balance is functioning properly. should be tested with an item of known mass.

11. General/Contents of Evidence File

The expiration dates of all test kits used must be recorded in the field log book.

ATTACHMENT II

Outline for the Audit Demonstration for RDX & TNT Test Kits

1. Select 12 sampling locations, providing an anticipated range of RDX soil contamination. The range should be distributed as follows:
 - a. 100 to 1,000 dilution factor needed. (Two samples)
 - b. Near but within the upper calibration limit of the field test kit. (One sample)
 - c. Within the linear calibration range of field instrument. (Four samples)
 - d. Just above detection limit of field instrument. (Three samples)
 - e. Clean soil. (Two samples)
2. The Quality Control samples should include the following:
 - a. Three (composited & split/collocated) field duplicates (Both for colorometric test kits and 8330 method) These should be correspond to:
 - (1) A sample location thought to be contain levels of contamination within the linear range of calibration,
 - (2) A sample location thought to be just above the field detection limit, and
 - (3) Clean soil.
 - b. Two MS/MSD per method (field test kits and method 8330) For TNT & RDX, spike at 8 to 10 ppm, wet weight. These samples should correspond to samples thought to be just above the detection limit of the respective field methods.
 - c. Will a "standard soil" be used?
 - d. Internal QC checks should include calibration verification, surrogate recovery data, LCS, and second column confirmation for 8330.

Audit Comments Continued...

- e. Two equipment rinse blanks.
 - f. Reagent blanks for RDX and TNT (acetone) - field test kits.
 - g. Multiple (composited) split sample analysis. (See item # 5 below).
 - h. Method blanks for field and lab methods.
3. Each soil sample should be homogenized at each location prior to splitting. Follow the procedure suggested by U.S. EPA Region 10. Soil cores (0 to 15 cm, 5.6 cm in diameter) should be placed in resealable plastic bags, and vegetation should be removed. The soil sample should be placed into 23 cm aluminum pie pans. The soil should be broken up using gloved hands and large rocks should be removed. (Sieving may also be applied). A second pie pan should be used to cover the sample, which should then be shaken and swirled vigorously to disperse and homogenize the soil. The sample should then be coned and quartered, and subsamples about 5 gm in weight should be removed from each quarter and composited to form the 20 gm sample for analysis. Sufficient sample should be composited and homogenized in this way to support both the RDX, and TNT field tests as well as the 8330 confirmation analyses.
 4. Preservation of samples for 8330 samples should involve freezing. (A 4 degree C temperature is not sufficient for the TNT and nitroaromatics.) Also, the TNT field samples should be analyzed within two hours of sample collection.
 5. The sample splitting procedure should be performed following homogenization, according to Region 10 guidance. Split the sample in triplicate. One sample of sufficient quantity will go to the lab. The other two will be analyzed in the field (for TNT and RDX). Split 3 soil samples, corresponding to contamination levels thought to be within the calibration range of the field test kits, in this manner again so that the splits can be analyzed multiple times by each method (field and lab).
 6. A five point initial calibration curve shall be used for all method 8330 explosives compounds. All explosives compounds should be reported.
 7. Provide all calibration HPLC printouts (field and lab), results for MS/MSD, blanks, surrogates, duplicates, LCS (internal QC checks).
 8. Appropriate correlations should be established between 8330 results and field test kit data.

Audit Comments Continued....

9. Confirm all 8330 detects using second column confirmation. Reporting limits for 8330 and test kit data should be less than the soil DQL values for respective target compounds in clean soil.
10. Use high purity acetone for field RDX tests. Histology or commercial grade is not acceptable.
11. Follow criteria from CRREL Report 90-38 for two times background subtraction for TNT.
12. Cleanest areas should be sampled first. Referring to item #1, the order of sampling should be E through A.
13. The deficiency comments generated as a result of the U.S. EPA's review of the "QAPP for Pilot Scale Operations at the Biofacility", dated 11/1/96, Appendix C should be factored into the protocol for both RDX and TNT testing prior to implementation of this Audit Demonstration Plan.

References:

1. EPA Federal facilities Forum Issue: "On-site analytical methods and Field Sampling For Explosives In Soil". (Draft Version, May 6, 1996, by A. B. Crockett, H. D. Craig, F. T. Jenkins, and W. E. Sisk.
2. "Development of Field Screening Methods for TNT, 2,4 DNT and RDX In Soil", in Talanta, Vol.39, no.4, pp.419-428, 1992
3. "Experimental Assessment of Analytical Holding Times for Nitroaromatic and Nitramine Explosives in Soil", by Clarence Grant, Thomas Jenkins, and Susan Golden, CRREL Special Report # 93-11, June 1993

Audit Comments Continued....

Sample Network and Design:

Soil Sample Type	Method	No. Split Samples*	No. Field Duplicates	No. MS/MSD	Equipment Rinse blanks (water matrix - final rinse)
A.) 100 to 1,000 dilution factor needed	RDX	2	0	0	0
	TNT	2	0	0	0
	8330	2	0	0	1
B.) Upper Range of Test Kit Calibration Ranges	RDX	1	0	0	0
	TNT	1	0	0	0
	8330	1	0	0	0
C.) Within Linear Calibration range of field instruments	RDX	4**	1	0	0
	TNT	4**	1	0	0
	8330	4**	1	0	1
D.) Just above detection limit of field instruments	RDX	3	1	2	0
	TNT	3	1	2	0
	8330	3	1	2	0
E.) Clean soil	RDX	2	1	0	0
	TNT	2	1	0	0
	8330	2	1	0	0

* If criteria for sampling RDX and TNT do not correspond, additional samples may be required for 8330 analyses to establish correspondence.

** Note that one of these split samples is to be tested further as a replicate. (See item 5 of this Audit Demonstration Plan).