



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

REPLY TO THE ATTENTION OF

December 2, 1998

DW-8J

Mr. Thomas Brent
Environmental Protection Department
Code 095
Naval Surface Warfare Center
300 Highway 361
Crane, Indiana 47522

RE: Notice of Deficiency
Ground Water Quality Assurance Plan
ABG, Demo, ORR, OJT
Naval Surface Warfare Center
Crane, Indiana

Dear Mr. Brent:

The United States Environmental Protection Agency (U.S. EPA) has reviewed the Quality Assurance Plan for Ground Water Monitoring at the Ammunition Burning Ground (ABG), Old Rifle Range (ORR), Demolition Range (Demo), and Old Jeep Trail, dated May 1998. Our comments on the plan are attached. These comments include the reviews by Allen Debus, our Quality Assurance Plan Coordinator, on the Standard Operating Procedures (SOPs). Most of these comments were given to you previously in draft form.

It appears that the lab is very close to becoming approved. We still need to see how the performance sample audit performs for final approval. There is only one Appendix IX constituent that we found that is missing from the requirement for metal analysis, that is Tin. This constituent and its SOP must be included in the plan. Modifications must be made addressing the attached comments, with a response to comments document.

In order to keep on the intended approval of the ground water plans by December 30th, we would encourage working closely back and forth to get this plan finalized. If you have any questions regarding this matter, please contact me at (312) 886-6146.

Sincerely,



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

Enclosure

Filename: NODGWQAP.USN

cc: Core Team Members: Bill Gates, SOUTHDIV
Christine Freeman, NSWC
Phil Keith, NSWC
Doug Johnson, CAAA
E.P. Johns, SOUTHDIV
Michelle Timmerman, IDEM

Project Team Members: Allen Debus, U.S.EPA
Ralph Basinski, Tetrattech
James May, ACOE-WES
Noel Krothe, IU
Cheryl Frischkom, IDEM

Management Team Members: Tom Linson, IDEM
Hak Cho, U.S. EPA
Jim Hunsicker, NSWC
Jim Ferro, SOUTHDIV

Notice of **Deficiency**
Quality Assurance Plan for Ground Water Monitoring
ABG, ORR, Demo, and OJT

1. Page 1-1, Section 1.0
 - a. At the end of paragraph 1 add the following: “The objectives of the ground water monitoring programs also include compliance with RCRA Section 3004(u) and (v) Corrective Action requirements at all three units, and 40 CFR Part 265 closure requirements for land disposal units at the **ABG**.”
 - b. In paragraph 3, “Mode” should be “Model.” Also cite the April 1998 version which is being used for reviews. For the DQL levels cite also the 1998 version The levels have been updated until the QAP is approved.
2. Change all references to the Ground Water Monitoring Plan to the approved plan instead of a dated one.
3. Section 1.1

Add a period at the end of the second sentence. Refer to the “approved **GWMP**” not a dated version. At the end of the paragraph add: “For the purposes of this plan, springs are also considered as “well locations” for compliance.”
4. Section 1.1.1
 - a. In the second sentence, revise the objective statement to match the April 1998 Model QAP.
 - b. After “alternative risk-based criteria” add “(alternate concentration limits (**ACLs**))” since this is the regulatory term that should be used.
 - c. The table isn’t a data summary but a summary of which data was evaluated
5. Section 1.1.2
 - a. Add “(which includes surface water sampling)” after “RCRA ground water monitoring program.”
 - b. Add “(**ABG**, ORR, and **DR**)” after “three operating units.”
 - c. Change “closure units associated with the operating units” to “closing units (waste pile and surface impoundments) associated with the past operations.”

- d. Add “/corrective action” after “detection or compliance.”
 - e. Add at the end of the first paragraph: “For the purpose of this ground water program, the U.S. EPA has designated the “operating unit” or “Facility” as the area circumscribing many individual burning or detonating individual units or structures. Points of compliance are established based on this.”
 - f. In paragraph two, add “and surface water” after “ground water” in the second sentence. Add “springs and surface water locations” after “monitoring wells” in the fourth sentence. Add “, and amended to this plan” at the end of the paragraph
 - g. In the third paragraph, add “and compliance” at the end of the first sentence.
6. Section 1.1.3, refer to the April 1998 version also.
7. Section 1.3.1.2
- a. Check with Doug and Phil that there are only 3 burning pits.
 - b. Add that “Between ___ and ___ in bum pans on top of clay and synthetic lined depressions.”
 - c. Page 1-19, explain what “along with other material” is.
8. Section 1.3.2.1, Page 1-20
- At the end add “, to establish background and plume constituents.”
9. Section 1.3.2.2.
- At the end add “, to establish background and plume constituents.”
10. Section 1.3.2, Page 1-21
- At the end add “, to establish background and **confirm** the absence of a plume.”
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- a. Make “piles” to “pile” there was only one
 - b. Sentence 4, they had clay liners for a period. Clarify this. Closure began when?

The units were physically removed when?

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12. Section 1.4.1, Page 1-23
In paragraph 2, sentence 3, add “and custom” **after** “SW-846.”
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In the second from last sentence, add “to establish background.”
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 - a. In point 2, add at the end “and DR and **OJT.**”
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 - a. The first paragraph may need to be changed based, on the Field Sampling **Plan**.:
c o m m e n t s :
 - b. Dioxins could be associated with the past burning of explosive contaminated solvents or plastics, so the last sentence is not correct.
 - c. In the fourth paragraph, add “the **OB/OD** units at” before “NSWC Crane.”
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 - a. Add “or background” at the end of the second from last sentence
 - b. Add “,including background” at the end of point two.
 - c. Add “and background” at the end of point five.
 - d. In Point 9, change “exceed” to “be lower than.”

17. Section 1.5

Delete “also” in the last sentence.

18. Section 1.5.2

The Field Sampling Plan need to have the sampling locations shown better for this reference.

19. Page 1-37, Section 1.6

Change “outlines” to “refers to” and add “in the Field Sampling Plan” at the end.

20. Page 1-3 1, Section 1.4.2.2

- a. Refer to the approved FSP, not dated versions.
- b. Make the last sentence the same as in the FSP comments from U.S.EPA.
- c. Should ethanes be included to the TCE degradation list?

21. Page 1-32

- a. In the first paragraph, if TETRYL. is identified at ABG, picric and picramic may be added since they are degradation products that should be looked for.
- b. Add at the end of the first paragraph, “If any of these parameters are found in the unit’s plume, they will be added to the compliance list.”
- c. In the second paragraph, change “, are essentially the lowest limits of detection required to determine potential human and/or ecological effects.” to “, these limits are based on 1998 human and ecological risk values.”
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22. Table 1-1 (see hard copy)

23. Table 1-3

- a. Tin is missing from the list and must be added since it is an Appendix IX constituent. An SOP for Tin must also be added.

- b. On page 1-27, **Dibenzo(a,h)anthracene** by SIM is misspelled. It should be **Dibenz(a,h)anthracene**.
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24. Section 2.0
- The Field Sampling Plan does not address the U.S. EPA does external audits, and the plans do not address who does independent data validation and data assessment. This needs to be clarified.
25. Section 3.6, Page 3-26
- It is not clear what the ambient blank will be tested for. Were ambient blanks discussed in the FSP? Cross-reference. In paragraph 3 explain where the ambient blank is opened.
26. Table 3-11, Page 3-17
- Add **hexachlorophane** to the Herbicide list
27. Page 4-1, Section 4.0
- This section may need modification based on FSP changes, Adding which SOP is used would help.
28. Page 6-1, Section 6.2
- Paragraph 3, last two sentences. Add “, unless they are identified and become a part of the compliance monitoring program.” after “natural attenuation.” Delete “None of these compounds are associated with compliance monitoring.”
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In the third and fifth sentences, this is not clearly coordinated with the FSP. Either logbooks or log sheets are used in the FSP. But the logbook is not shown to have detailed sampling data that repeats the log sheet, which it should.

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- a. "Copes" should be "Copies."
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COMMENTS FROM ALLEN DEBUS:

Page 1: Allen

Date: 9/25/98 - (DRAFT & INCOMPLETE)

Subject Explosives Testing SOPs for U.S. Navy Crane Groundwater RFI project

From: Allen A. Debus, IL/IN/MI Section

To: Carol Witt-Smith, IL/IN/MI Section

Although I have not had the time to comprehensively review much of the revised QAPP and attachments including revised SOPs from Lauck's Testing Laboratories (& other lab facilities), as well as method performance data for the explosives analysis SOPs, I do have some preliminary comments, outlined below. The comments which are specifically relevant to Lauck's' explosives SOPs should be submitted to U.S. Navy Crane as deficiency comments at the earliest opportunity.

A. QAPP Comments:

1. Pane 1-1 of Section 1.0: In the final paragraph, the April 1998 RCRA Region 5 QAPP Policy should also be utilized as a reference and noted here.
2. Page 1-2. Section 1. 1. 1: In the first paragraph, it would be relevant to perhaps revise the objective statement per the April 1998 QAPP Policy. (Note references to DQLs and "alternative risk-based criteria".)
3. Table 1-1. Page 1-1 1: In the **fifth** footnote, please note that the chlorinated degradation products for PCE and TCE **have not** been correctly identified. (Some are missing and should be added to the list of key project parameters.) Also, PCE cannot be a breakdown product of TCE.
4. Page 1-13, Section 1. 1. 1 : The 'summary' that is listed isn't really a "data summary", but a summary of which data sets were evaluated.
5. Pane 1-3 1. Section 1. 4. 2. 2: In the last paragraph, note that the PCE and TCE degradation products haven't been properly listed.
6. Page 1-32. Section: In the second paragraph, the sentence beginning with the phrase, "The risk-based target levels presented...." may be misconstrued. This sentence should be clarified or revised.
7. Page 1-35: In the final "bullet", the word "exceed" should be changed. (Hopefully the RLs will not exceed the target levels.)

8. Pane 2-1. Section 2.0: Referring to p. 8 of 13 in the FSP, note that it is the U.S. EPA which performs the “external” audits. Also, it should be explained who performs independent data validation and data assessment.
9. Table 3-1 1. page 17 of 27: For risk assessment purposes, should hexachlorophene should be added to this list. (Also see Table I-3.)
10. Pane 3-26. Section 3.: In the first paragraph, how does a ‘source’ water blank differ from a regular investigational sample? What is the procedure for collection of an “ambient blank”? What will it be tested for (VOCs)? (Perhaps the answers to these questions are given elsewhere in this section.) On page 27 of 27, second par., last sentence, the phrase beginning with the words, “.. .or if new wells are installed,…” is confusing.

B. Lauck’s Method # LTL-3077

1. Referring to section 3.3.4, note that if this sample preparation/extraction method will be utilized for the three sets of explosives analyses, (8330, “additional breakdown products”, and **NG/PETN**), then those compounds must also be represented in matrix spiking procedures. The extraction & sample preparation procedures for each of these methods must be clearly identified.
2. Section 6.1. Page 4 of 4: Note that SW-846, method 8330 recommends a 1: 1 dilution with organic free reagent water, with **pH**< 3 for tetryl. Also the extract should be **filtered if** turbid to prevent clogging of the LC columns. Why aren’t these procedures reflected. Since tetryl is a target analyte, the **pH** of all 8330 explosives samples ‘should be ‘acidified. How will this be done? Also, since tetryl is light sensitive, special precautions must be taken to prevent uv photodegradation of this analyte in all containers and vials (and standard vials) used for its analysis.

C. Lauck’s SOP # LTL-8303

1. The primary concern **with this** technique and the other techniques proposed for explosives is coelution with interferents. The structural chemistry of the target compounds in question is all very similar. The method performance data was generated using clean water samples, in which potential interferents were not present, Analytical runs of a series of picric & picramic acid standards should be performed reflecting a “worst case” scenario, in which all the compounds proposed for analysis by Lauck’s method # LTL-8330 are present in known concentrations. Quantitation should be attempted using the techniques outlined in this SOP for picric & picramic acids with other potentially interfering analytes present in the same sample.

2. Pane 7 of 3 1. Section 2.1: Note that the method of standard additions (MSA) approach to peak **confirmation** will not be capable of resolving interference contributions due to coelution. Therefore, it is important to have interference data concerning other probable target **analytes** available before investigative samples are analyzed.
3. Pane 10 of 3 1. Section 4.1.1: Please explain the identification of the column as a "**PAH-C18**". (This is not a PAH analysis. Can the same column be used? Please clarify.)
4. In section 4.4.3 of Method LTL-8303, will the retention time shift be founded on the latest CCV sample's measured surrogate absolute RT, provided the surrogate's RT is within RT window specifications?
5. Referring to section **4.5.1** of LTL-8303, please note that the low standard used for initial calibration should approximate the reporting limit values indicated in Table 1-3 of the QAPP for **picric** and picramic acids set for this project, whenever data is reduced to reporting limits in real groundwater samples. (In other words does the low standard conform to the level of the reporting limit value indicated in the QAPP table. I haven't had the time to check this, but sample calculations should be provided.) Also see section 4.10.3.1.1 of this SOP.
6. In section 4.10.2.1 of this SOP, 'Compound Identification', it is explained that a post-spike is added to the sample extract. (Evidently a confirmatory **column** is not used.) Method 8090 of **SW-846** recommends the use of a confirmatory column for chromatography methods. A post-spike may only augment the response of a coeluting compound (i.e. within the same RT window). Why can't this measurement system be supplemented with a confirmatory column?
7. Please demonstrate through an example calculation that the **MDL** values of 0.5 **ug/mL** expressed in Appendix I of this SOP, p.23 of 3 1, correspond closely to the levels expressed in Table 1-3 of the QAPP (when converted to sample concentrations from extract measurements).
8. The ICV standard for the **picric/picramic** acids analysis should be obtained from a source independent from the source of the initial and continuing calibration standards.

D. Lauck's method LTL-8330

1. The standard SW-846 guidance method 8330 recommends two calibration mixes, to avoid potential coelution difficulties. Ordinarily if all 14 standard method 8330 target analytes are mixed together coelution of certain peaks will occur. The complexities would be amplified if the other "Occasionally Required Additional compounds" are present in site samples. In this case, however, it is not clear whether calibration for the 14 standard analytes shall be performed using two separate mixes, or if the gradient elution program referred to in section 1.2.1 is intended to resolve each of the 14 standard components, even if mixed in the same standard (or samples). (However, I do think a 14 component mix is being used based on instructions presented in section 2.2.4 of this SOP.) It should be clarified as to whether or not separate initial calibration standards shall be used. Secondly, if a single calibration mix is to be used, then **Laucks** should submit a set of initial calibration chromatograms for the set of fourteen 8330 analytes, including the surrogate compound.
2. Because real samples might pose as yet undetermined coelution difficulties, a sample containing known quantities of each of the compounds listed in section 1.1.2 of this SOP should be prepared for analysis. Then this sample should be run using all three techniques outlined in this SOP, in attempting to quantify first the standard 14 method 8330 compounds, then the "Occasionally Required Additional Compounds", and finally NG and PETN detected at 210 nm. The purpose of this exercise would be to determine the impact of target analytes as potential interferences. Chromatograms of all three tests should be submitted.
3. Section 1.2.1. bullet 3: How is the stability documented and controlled with respect to tetra analysis?
4. Section 1.2.1. bullet 5. page 6 of 39: "NBE" should perhaps be "NB (nitrobenzene). These sections offer no indication as to whether there are coelution problems associated either within the "occasionally required group", or with the standard 8330 analytes if present in an investigative sample.
5. Page 7 of 39. section 1.4.1: Both the ICV and QC check standards should be prepared from a source independent from sources used for calibration purposes. Can these standards be one and the same?
6. Page 7 of 39. section 1.3.2: Can the eight week holding time for frozen samples be applied to compost samples (or should a shorter time be imposed)?

7. Pane 11 of 39. Section 2.2.4.1: Note that there should also be a set of instructions for preparing the calibration standard set for analysis of the “Occasionally Required” group. Note that this is referred to as a “separate analysis” in section 1.1.2.
8. Section 2.2.4.1. page 11 of 39: Is the low calibration standard consistent with the projected reporting limits for each of the key compounds of concern? A low standard concentration should be selected for each of the target analytes which, when converted to reporting limit values for real samples, is similar or lower to the action levels for each compound to be reported.
9. Pane 12 of 39. Section 2.2.4.2: At the top **of the** page, in the fifth column, should the compound DNB refer to 1,2 DNB, which is the surrogate compound?
10. Pane 14 of 39: At the top of the page, the column **labelled** as “mL/min” appears to be inappropriate for the units appearing below.
11. Pane 14 of 39,: Under “Gradient Profile (**HPLC**), do these conditions pertain to NG and PETN too?
12. Pane 15 of 39. Section 4.1.3: Is the term, “additional compounds” intended to refer only to the “Occasionally Required Additional Compounds”, or to NG and PETN as well? Which analytes are linked to which operating conditions expressed in these sections?
13. Panel7 of 39. Section 4.4.3: Shouldn't there be two if not 3 calibration mixes used to avoid coelution problems on the **primary column**? (Mix A, Mix **B**, including surrogates) Is retention time “shift” based on the surrogate RT **of the** CCV, provided the CCV surrogate RT is within RT windows? (Also see section 6.2.1.3.)
14. Page 17 of 39. Section 4.5.1: The 50 ppb standard isn't referred to here. Also, how is it known that the low standard selected for initial calibration is appropriate for anticipated reporting limits and intended health based “target levels”? (See comment D.8 above.)
15. Pane 17 of 39. Section 4.6.3: This section seems to contrast with previous discussion. Aren't the retention time windows determined statistically? (See section 4.4.) I think the discussion presented here is consistent with my interpretation as indicated in comment D. 13 mentioned above, but it could be clarified.

16. Page 18 of 39, Section 4.10.2.1: Discussion in the middle of this paragraph should be tied to data assessment issues. It should be anticipated how flagged data will be used and interpreted by the writer of the **RFI** Final Report?
17. Pane 19 of 39, Section 4.10.2.1: Will the **RT's** be “administratively set” in the case of the Crane project RFI? Due to the potential for analyte coelution, the ramifications of compound identification on the basis of determined retention times should be anticipated now.
18. Pane 19 of 39, Section 4.10.3.2: It should be demonstrated using this equation that the proposed low standard is an appropriate standard, on the basis of individual “action levels” targeted for risk assessment and regulatory decision-making purposes, (Also, see previous comments D.8 and D.14.)
19. Pane 23 of 39, Section 6.3.2.1: Are the “associated samples” the method or reagent blanks? Please clarify.
20. Pane 25 of 39, Section 6.8.1: The **MS/MSD** pair should not be chosen at random. These should be preselected samples and this fact should also be reflected in the QAPP and FSP. (Please make sure this is the case.)
21. Pane 27 of 39, Section 6.10.2.1: Can the logic of the second sentence be either explained or clarified and modified?
22. Auoendix IV: Referring to the **MS/MSD**, why is it that two target analytes can be outside of the control limits? (I would suggest that none should be.)
23. Auoendix V: What are the approximate retention times for each of the listed **analytes**? How do the retention times of the 6 additional target analytes compare to the standard 14? (Refer to comments D.1 and D.2.)

E. Method Performance Data:

1. Generally, it would have been helpful to have the 5 initial calibration standard data plotted, (i.e. response versus retention time).
2. It is noted that as indicated in the QC Summary Forms for the “8330 extras **MDL**” in attachment A, recoveries for some target analytes were rather low, (50% range).
3. What are the unidentified peaks evident in the “Occasional Required Additional **Analytes**” data package which come in at approximately 19.7 min., 22 min. and 26 min.? What is the large unidentified peak at RT 9.4 minutes in the NG/PETN scans on the C18 column (See Attachment 7)?

4. In the “Additional **Analytes**” group, chromatogram # STD 1 **MA5-47-05**, injected April 24, 1998, at **13:07:24**, **C18** column, why is the chromatography poor relative to the **MDL** study?
5. Some **TNX** contamination was noted in the **IBLK**, April 24, 1998, injected at **18:47:16**.
6. Note that 3 compounds are missing from the **MDL** database for the confirmatory column. (Only data for **TNX**, **MNX** and **3,5** dinitroaniline has been provided.) Yet this is unexplained.
7. Data for the **picric** and picramic acids feasibility study indicates some additional unidentified peaks in the initial calibration standards of lesser concentration. What might these peaks be? (Structural isomers?)
8. It is noted that there was some picramic acid contamination in the **IBLK**. Is there an explanation for **why** this happened? Also, there was picramic and **picric** acids contamination in the method blank.
9. Referring to Attachment 7, evidence of contamination was noted in the **IBLK C18** column (**1/6/98 - 22:25:06** injection) for the **PETN/NG** technique. What, most likely, were these compounds?
10. In Attachment 7, for the **PETN/NGanalysis**, an unidentified peak was noted in the **C18** method blank (**1/7/98 - 7:58:40** injection). What is the likely nature of this compound? Would this “detect” have triggered a need for corrective action if it had been detected during a sample run?
11. In Attachment 8, for initial calibration standards analyzed on the **C18** column, there is an unidentified peak evident at (approximately) 9.7 minutes. What is the likely nature of this compound? Would this detect have triggered a need for corrective action if it had been detected during a sample run?

Page 8: Allen

From: ALLEN DEBUS
To: WITT-SMITH-CAROL
Date: 9/28/98 2:23pm
Subject: NG/PETN method

Correction:

The TCE & PCE should bio-decompose into **trans** or cis DCE. So, is there a possibility of encountering DCA in the investigative samples, even if it doesn't form through biodegradation of TCE?

(Please neglect the last part of the comment regarding DCA interferences for the NG/PETN analysis, Sorry for that contusion.)

Allen

CC: IN:"basinskir@ttnus.com"
From: ALLEN DEBUS
To: WITT-SMITH-CAROL
Date: 9/28/98 8:30am
Subject: NG/PETN testing

Carol:

I've done a bit of **further** digging & discovered a copy of the **CRREL** method for NG/PETN analysis. In this HPLC method (**dated August 1989**) it is stated that, "**Nitrobenzene** and dichloroethane will coelute with NG**However**, these **analytes** can be separated from NG on an LC-CN column **eluted** with 1/1 v/v methanol-water at a 1 .S mL/min flow rate. Retention times are 4.2, 6.9 and 9.4 minutes for NB, NG and DCA, respectively. Thus LC-CN can be used for second column chromatography if a suspect peak is found on the primary column at the proper retention time."

These chromatography conditions may be those which have been proposed in the submitted SOPs for analysis of NG and PETN However, through **Laucks**, Crane should make a statement concerning the possible interferences caused by NB and DCA. (It is noted that the 1,1 DCA isomer is a breakdown product of TCE. The latter is a contaminant of concern for this project.)

Allen

Page 9: Allen

CC: IN:"Basinskir@ttnus.com"
From: ALLEN DEBUS
To: WITT-SMITH-CAROL
Date: 10/14/98 8:57am
Subject: Method 8015 SOP comments for Crane

Carol:

Here are my comments concerning Lauck's Method 8015 SOP, proposed for the U.S. Navy Crane groundwater study:

1. Method LTL-8019, p. 5 of 39: A correlation **coefficient** of .990 isn't great. it would be better to impose a more stringent criterion of .995 instead.
2. Method LTL-8019, p.16 of 30: In section 4.3, the definition for Method Reporting Limits is **identical** to that provided for Method Detection limit study in section 4.2. What is the difference, and how is a method reporting limit truly defined?
3. Method LTL-8019, p. 18 of 39: In section 4.8.4, note that although this subsection isn't relevant to the Crane groundwater study, soil samples intended for **VOCs** analysis should be sampled (and analyzed) in a manner consistent with current U.S. EPA policy. (This Region 5 policy should be applied to method 8015 as well as other analytical methods for **VOCs** analyses.)
4. Method LTL-8019, p. 19 of 39: In section 4.8.9.1, for ordinary method reporting purposes, it should be noted that the term, "As" corresponds to primary column data. ..
5. Method LTL-8019, p. 21 of 39: How does the PQL mentioned in section 5.4 compare to the method reporting limit?
6. Method LTL-8019, p.33 of 39: To relieve a matter of potential confusion, shouldn't it be clarified that nearly all the standards are "multiple component standards", (i.e. including the continuing calibration and linearity standards)?
7. Referring to Attachment 6 of the method development study, note that the CCV precision for p-dioxane was out of control limits on several occasions. Also, acetonitrile was out of control limits on one occasion. Please discuss these circumstances
8. On p. 3067 of Attachment A, there appears to be a great deal of blank contamination. Please discuss this circumstance.

Allen

Page 10: Allen

***** NEW COMMENTS NOT PREVIOUSLY E-MAILED *****

From: Allen A. Debus, IL/TN/MI Section

To: Carol Witt-Smith, IL/TN/MI Section

Subject: Lauck's Testing Laboratory **SOPs** for analysis of explosives constituents proposed for U.S. Navy Crane Groundwater **RFI**

Date: November 18, 1998

SUMMARY:

Laucks Testing laboratories has made a positive effort to address special concerns raised by the U.S. EPA in its October 1, 1998 notice of deficiency letter. The laboratory **SOPs** in question have been revised and additional data has been generated to supplement the on-going method development studies. In particular, Laucks has added a **confirmatory** column for the **picric & picramic acids** analytical procedure and supplied us with chromatograms indicating the "worst case" scenario "sample" into which 19 explosives compounds had been spiked. However, coelution difficulties still exist with method LTL-8330. However, in the case of the standard 14 method 8330 explosives compounds (which will all be calibrated from a single mix, as opposed to two mixes as is usually done), the coelution **difficulties** are relegated to the confirmatory column.

SPECIFIC CONCERNS:

Project specific **DQOs** and associated data assessment should be anticipated now in the event that "detects" will have to be confirmed as a coeluting spike (e.g. 2-, 3-, and 4- nitrotoluenes; or 2,4 and 2,6 dinitrotoluenes; and 1,3,5-trinitrobenzene and 1,3 dinitrobenzene). In other words, U.S. Navy Crane should propose how certain data that is reported as "flagged data", might be utilized in meeting specific targeted objectives such as those involving human health risk assessment.

Another chromatogram, Appendix D-2 in the submittal, indicated the possibility of coelutions (i.e. HMX and 2,6 diamino 4-nitrotoluene; and 3,5 dinitroaniline and tetryl) on the primary **column** used for quantitation. Thus, if samples are contaminated with these compounds it will be impossible to report them using the proposed method and cited chromatographic conditions. U.S. Navy Crane must propose a scheme for dealing with these consequences because they could occur. How will "flagged data" be utilized in meeting targeted objectives?

Laucks should revise section 1.2.1 of method LTL-8330 to reflect the fact that there is potential for HMX and **2,6-diamino-4-nitrotoluene** as well.

Laucks also affirmed that the compounds, picric and picramic acids would not be detected using their method LTL-8330. This is interesting in light of what we recently learned in the case of another laboratory where trace **amounts** of picric acid were identified in samples. While I am not contesting Laucks' claim (because it is likely that other chromatographic conditions were used), it would be helpful if they could submit one sample chromatogram representing a sample in which picric and picramic **acids had** been spiked. (There might be no calibration data for this run, which is counter to the case cited previously in which a full **5** point initial calibration had been performed along with the other fourteen 8330 explosives compounds.)

There was insufficient information available for determining why fairly significant retention time **shifts** occurred when comparing the test run using all 14 method 8330 analytes, with the run using this same mix but with the addition of **5** additional explosives breakdown analytes. (Here I am referring to comment D.2.) When I overlaid the two chromatograms such that the surrogate peaks were aligned, it is apparent that retention time differences ranging from 30 seconds to a minute resulted for several target parameters. Were **all** compounds within retention time limits during the run which included the five additional compounds? (I presume the chromatographic conditions cited in LTL-8330 for the standard set of 14 compounds were used.)

Laucks should also continue to validate their refined use of the confirmatory column for use in the picric & picramic acid study. I would also like to know whether any of the 22 **LTL-8330** parameters would be detected on either the primary or confirmatory columns cited in the **LTL-8303** SOP.

Otherwise, Laucks has effectively addressed all other concerns. We await the results of the performance evaluation audit samples to make a final determination concerning Laucks' **adequacy** for the U.S. Navy Crane project with respect to the explosives parameter group. Thus far all indications appear favorable.