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DISAPPROVAL OF THE REVISED SITE WIDE QUALITY ASSURANCE PROJECT PLAN

05/20/92

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

MAY 20 1992

3254

REPLY TO THE ATTENTION OF:

HRE-8J

Mr. Jack R. Craig
United States Department of Energy
Feed Materials Production Center
P.O. Box 398705
Cincinnati, Ohio 45239-8705

RE: Disapproval of the Revised Site Wide Quality Assurance Project Plan

Dear Mr. Craig:

The United States Environmental Protection Agency (U.S. EPA) has completed its review of the revised Site Wide Quality Assurance Project Plan (QAPjP). The revised Site Wide QAPjP included laboratory analytical procedures, which were not previously submitted.

Although the United States Department of Energy U.S. DOE has satisfactorily responded to many U.S. EPA comments, several revisions to the QAPjP are necessary. Therefore, U.S. EPA hereby disapproves the revised QAPjP pending incorporation of the attached comments. U.S. EPA requests that U.S. DOE review these comments and that all outstanding issues will be discussed and resolved at the June 2, 1992 QAPjP meeting in Chicago, Illinois.

Please contact me at (312/FTS) 886-0992 if you have any questions.

Sincerely,

James A. Saric
Remedial Project Manager

Enclosure

cc: Graham Mitchell, OEPA-SWDO
Pat Whitfield, U.S. DOE-HDQ
Dennis Carr, WMCO

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FERNALD ENVIRONMENTAL MANAGEMENT PROJECT SITE WIDE CERCLA QUALITY ASSURANCE PROJECT PLAN

[redacted]

GENERAL COMMENTS

Comment No. 1 -- The QAPjP should contain a table that addresses the following for each measurement and matrix: analytical service level (ASL), specific analytical method, detection limit, precision, accuracy, representativeness, comparability, completeness, and sample collection methodologies.

Comment No. 2 -- The text presents the analytical methods for analysis of several different compounds. A table showing the analytical method, the ASL, and the intended use of the data from each sample should be included in the QAPjP. Portions of the QAPjP that restate regulatory guidelines or procedures [for example, Data Validation Guidelines (EPA 1988a, b)], should be deleted, and the text should reference the section in which appropriate general guidance is located.

SPECIFIC COMMENTS

1. **Section 2.2.3.1, page 5 of 27**

   The waste disposed at pit number 4 is estimated at 57,6000 cubic yards. This typographical error should be corrected.

2. **Section 4.0**

   Even though the data quality objectives are provided in the methodology description, it would be helpful to include all the data quality objectives in a separate table for each media.

3. **Section 4.1.1, page 4 of 20**

When defining "duplicate samples," the text should state that these samples will be submitted to the laboratory as blind samples.

4. **Section 4.1.1, page 4 of 20**

The text states that split samples will be used to evaluate the accuracy of the analytical laboratory and field sample handling practices. Accuracy is defined as the degree of agreement between a measurement and true value (Section 14.4 of QAPjP). It is unclear how split samples, shipped to different laboratories, will address this criterium. The relationship between these samples and evaluation of accuracy should be clarified.

5. **Section 5 (all text)**

This section should be combined with Section 6.0 to describe guidelines used to select sampling sites, sampling techniques, decontamination procedures, sample preservative, sample containers to be used, and holding time requirements.

6. **Section 6.1..1, page 2 of 36**

This section should also include the following items for project specific plans (either included in full or by specific reference to the site-wide QAPjP): sample custody procedure, calibration procedures and frequency, analytical procedures, data reduction procedures, data validation and reporting procedures, performance and system audits, preventive maintenance, routine procedures, accuracy and completeness, corrective action, and quality assurance reports.

7. **Section 9, page 1 of 3**

This section refers to the Table 9-1 (Appendix A). This table is not included in Appendix A for review.

8. **Section 13.3, page of 2 of 2**

This section states that preventive maintenance requirements are listed in Table 13-1 (Appendix A). This table is not included in the Appendix A.

9. **Section 15**

This section should include the data acceptability limits beyond which corrective action is required.

10. **Appendix A, Table 2-2, page 65 of 123**

The frequency of split samples, field spike control samples, and material blanks should be discussed in this table.

11. **Appendix A, Table 2-2, page 67 of 123**

Laboratory internal quality control requirements should also include target compound list (TCL) compounds and identification of tentatively identified compounds for organic compounds.

12. **Appendix A, Table 2-3**

The required quantitation limits listed for volatile organics analyses of water and soil are based on *Contract Laboratory Program, Statement of Work*, February 1990 (EPA, 1990a, b), and *Test Methods for Evaluating Solid Wastes*, 3rd edition, (EPA, 1987), Method 8240. However, quantitation limits listed in this appendix are different from those listed for the two analytical methods. The procedures for obtaining

these quantitation limits should be specified. Similarly, the source and rationale for the quantitation limits listed for BTEX; pesticide; 3,3-Dichlorobenzidine; p-Dimethylaminobenzene; 4-Aminobiphenyl; and 3-Nitroaniline should be identified. This table should also list the required detection limits for these analytical methods.

13. **Appendix A, Table 3-2, page 87 of 123**

Chemical Nuclear Laboratory is included in the list of laboratories approved for Fernald Environmental Management Project Chemical Analyses. Furthermore, CLP VOA is included as one of the analyses this laboratory performs. However, this laboratory is listed as non-CLP contractor in the CLP contract column. This discrepancy should be resolved.

14. **Appendix A, Table 4-1, page 94 of 123**

While this table lists some of the environmental training courses that personnel must complete prior to beginning work on site, it does not indicate the frequency, length of training required, or the regulatory requirement pertinent to this training. This table should include this information. This table should also list a 40-hour health and safety training course as one of the requirements

15. **Appendix A, Table 6-1, page 101 of 123**

The table quotes two holding times for semivolatile compounds, organochlorine pesticides, PCBs, herbicides, and organo-phosphorous pesticides. The holding times for these compounds should consistently appear as 7/40 days.

16. **Appendix C, page 10 of 10**

This page is titled "Added to Appendix C, Data Quality Objectives for FEMP." About 100 to 150 additional pages of Data Quality Objective

Summary forms follow this page. The purpose for this inclusion should be clarified.

17. **Appendix D, Section D 6.3.1, page 28 of 110.**

This page indicates that a greater than 0.01 calibration criterium will be used for the analysis of compounds on the nontarget list and the nonhazardous substance list. The rationale and source of this criterium should be stated.

18. **Appendix D, Section D7.3.1, page 46 of 110**

This section states that surrogate samples shall have a relative standard deviation (RSD) less than or equal to 30 percent. However, this limit is higher than 10 percent, which is recommended in *Laboratory Data Validation Functional Guidelines for Evaluating Organic Analysis* (EPA, 1988a). The basis and rationale for selecting this criterium should be stated.

19. **Appendix D, Section D11.2.10, Item 5, page 88 of 100 and Section D11.2.14, page 89 of 110**

This section should read, "if resolution is wider" rather than "greater," since greater resolution implies a narrower width.

20. **Appendix D, Section D11.5.2, Item 5, page 94 of 110**

It may be better to align the criteria in rows rather than in columns, which are easier to misalign. The same problem appears in D11.5.4, item 3; D11.7.2, item 1; and D.11.7.3.

21. **Appendix D, Section D11.6, page 95 of 110**

This section requires that duplicate samples be collected every tenth sample; however, PRC notes that most EPA methods require that duplicate samples be collected every twentieth sample.

22. **Attachment I, FM-ORG-0010, Section 14, page 37 of 37**

This quality control requirements listed for surrogate, spike, matrix spike/matrix spike duplicates, and ASLs B, C, and D refer to the wrong sections under the acceptance range column.

23. **Attachment I, FM-ORG-0040**

This method is used to analyze organophosphorous pesticides by [illegible] (GC). The reference to this method should be SW-846, method 8140.

24. **Attachment I, Method RAD-0010, Section 8.24, page 5 of 12**

This method proposes using 98 percent ethanol. This percent is not commercially available. Absolute (100%) ethanol draws water from the air until it is about 95 percent, same as the commercial azeotrope. The azeotrope [80 percent ethanol (USP)] is probably the intended form. The same reagent appears in Methods RAD-0080, RAD-0100, and RAD-0110.

25. **Attachment I, Method RAD-0050, Section 8.20, page 4 of 14**

This section should be rewritten. If docusate sodium (the generic name of Aerosol OT) is purchased commercially as a 0.1 percent solution, a source for this solution should be given. If the solution must be made in the laboratory, a procedure should be included. The same reagent appears in Method RAD-0060.

26. **Attachment I, Method RAD-0050, Section 8.23, page 4 of 14**

[redacted]

This section should specify what grade of helium is to be used. The same reagent appears in Method RAD-0060.

 [redacted]

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF RESEARCH AND DEVELOPMENT
ENVIRONMENTAL MONITORING SYSTEMS LABORATORY-LAS VEGAS
P.O. BOX 93478
LAS VEGAS, NEVADA 89193-3478
(702/798-2100 - FTS 545-2100)

APR 01 1992

MEMORANDUM:

SUBJECT: Review of Laboratory Analytical Procedures Requested By James A. Saric of Region 5

FROM: Robert Holloway Robert Holloway
Supervisory Chemist, NRA

TO: Ken Brown, Manager
Technical Support Project

Jesse Gerard and I have reviewed the laboratory procedures as requested.

Generally, the SOPs are acceptable. There are several points where we believe changes should be made.

1. For procedure FM-RAD-0010, for isotopic plutonium in water, there is a requirement to correct all sample results for reagent blank contributions. We believe that this type of correction should not be made. Preparation blank values should be used to determine whether or not samples associated with that blank should be reanalyzed. Of course, background counts should always be subtracted from each sample.

2. Procedure FM-RAD-0120 for the analysis of uranium in water by Pulsed-Laser Phosphorimetry needs to address the details of calibration in the SOP. All other procedures have sections on calibration.

3. The procedures need to have specific requirements on quality control rather than relying on project specific QA requirements. These requirements should address acceptance limits for spikes, duplicates and blanks.

4. Low chemical yield can sometimes produce results of poor quality. For those procedures where chemical yield is a factor, a minimum yield should be specified below which results will not be accepted. A 10% minimum is reasonable, although arguments could be made for a higher minimum.

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

REPLY TO THE ATTENTION OF:

MEMORANDUM

SQ-14J

DATE: MAY 08 1992

SUBJECT: Review of the Revision 1 Quality Assurance Project Plan (QAPjP) for the Remedial Investigation/ Feasibility at the Department of Energy Feed Materials Production Center (Fernald, Ohio) Superfund Site

FROM: for George C. Schupp, Chief Denise Boone.
Quality Assurance Section

TO: Kevin Pierard, Chief
Ohio/Minnesota Technical Enforcement Section
RCRA Enforcement Branch

ATTENTION: James Saric

The Quality Assurance Section (QAS) has completed its review of the subject revised QAPjP (QAS SF Log-In # 1711) received on March 10, 1992. The revised QAPjP , while significantly improved, still includes some deficiencies.

Some aspects of the QAPjP have become more generic apparently because of the fact that Westinghouse will not be the engineering/field contractor at Fernald at the end of FY' 92. As a result, a new contractor will need to provide its own specific standard operating procedures for review and approval.

Since the engineering/field contractor may have its personnel trained in its own sampling procedures, chain-of-custody procedures, etc., it may be difficult for the Department of Energy to mandate procedures as written and developed for Westinghouse. What would likely happen is that procedures specified in the QAPjP may not be those actually used in the field.

Since the analytical SOPs are being considered as a statement of work for Department of Energy's contractor laboratories, all comments presented in this memorandum, chemical analysis comments which result from the USEPA Region 5 Central Regional Laboratory's audits or radiochemical analysis comments from the USEPA Region 5 Air & Radiation Division/USEPA-NAREL (Montgomery,

[redacted]

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Alabama) should still be applicable regardless of the engineering/field contractor.

QAS' detailed comments may be found in the attachment to this memorandum. If you should have any questions regarding the attached comments, please contact Kevin Bolger of my staff at 3-7712.

Attachment: QAS' QAPjP Comments

cc: John Kelley, HSRM-6J
Kaushal Khanna, HSRLT-5J
David Payne, SL-10C

USEPA REGION V QUALITY ASSURANCE SECTION COMMENTS ON THE REVISION 1 QUALITY ASSURANCE PROJECT PLAN FOR THE REMEDIAL INVESTIGATION/ FEASIBILITY STUDY AT THE DEPARTMENT OF ENERGY - FEED MATERIALS PRODUCTION CENTER (FERNALD, OHIO) SUPERFUND SITE

GENERAL COMMENT.
The engineering/field contractor should be selected and incorporated into the QAPjP prior to approval. It will be necessary to ensure that the contractor's (and any other subcontractors') procedures are provided for review and approval.

TITLE/SIGNATURE PAGE.
Signature spaces still do not include all project management and quality assurance management entities as described in section 3.0 comments below.

a) Provide a space for the US DOE QA Officer responsible for the Fernald project.
b) Signature spaces for the project managers and QA officers for all engineering firms and contractors must be specified for an approvable QAPjP.
c) Provide spaces for the Ohio EPA project manager and QA officer since Ohio EPA will also review and approve the QAPjP.

TABLE OF CONTENTS.
The Table of Contents will require revision to include changes indicated for comments below on other QAPjP sections, Appendices, Tables, Figures etc.

3.0 PROJECT ORGANIZATION AND RESPONSIBILITIES
This section shall need to specify who the new engineering/field contractor shall be, its specific organization hierarchy as well as any other subcontractors' affiliations, titles and responsibilities. Figures 3-1 through 3-4 will need to reflect the new organization(s).

4.0 QUALITY ASSURANCE OBJECTIVES

a) Revise the title to read "Quality Assurance Objectives for Measurement Data in Terms of Precision, Accuracy, Completeness, Representativeness and Comparability".
b) The information presented in section 4.4 is extraneous to the QA objectives of precision, accuracy, completeness, representativeness and comparability and should be deleted. Document control relative to custody or evidence should be detailed in section 7.0 (Sample Custody).

5.0 FIELD ACTIVITIES
As noted during the January 1992 meeting with US DOE, this section will be allowed to remain intact although the material is best incorporated into the appropriate section on sampling procedures (6.0) if the procedure is relevant to sample collection (i.e. monitoring well development, decontamination of

sampling equipment) or into the Health & Safety Plan (worker screening).

It may be best to reconsider review of this section when the engineering/field contractor is secured since the contractor's (s') corporate procedures may differ from those specified. The present procedures, as stated, are acceptable.

6.0 SAMPLING REQUIREMENTS
The following comments will primarily refer to Appendix K which contains the matrix-specific sampling procedures. Comments relative to the sampling of radiochemistry samples are deferred to the USEPA Region 5 Air & Radiation Division/USEPA-NAREL (Montgomery, Alabama).

Appendix K.3 Responsibilities.
The information in this section should be integrated into QAPjP section 3.0 since this is a more specific description of roles and responsibilities.

Appendix K.4 Aqueous Sample Collection Method.
K.4.3 Surface-Water Sampling.
All surface water samples should be collected as grab samples and not composited. Labile parameters (i.e. volatile organics) may be compromised by compositing samples. Revise wording to indicate that surface waters will not be composited.

K.4.4 Waste Water Sampling.
It is not clear which analytical fractions may be collected for waste waters. Once again, the concerns posed in comment K.4.3 (above) may be valid if labile analytical fractions are composited. Please reconcile.

K.5 Solid Matrix Environmental Samples.
The concerns regarding compositing of samples again must be addressed for solid matrix sampling procedures as well. RCRA requires grab samples instead of composites for land disposal regulations. Compositing may tend to "dilute" samples. This issue must be resolved between USEPA and US DOE.

K.6 Gaseous Matrix Samples
K.6.4 Monitoring for Organic and Inorganic Contaminants in the Field.
These procedures do not appear in Appendix I. Have these procedures been developed and validated? What specific analytical parameters will be included in these field methods? Please reconcile by providing the detailed procedures in the SCQ, an Addendum to the SCQ or provided in the operable unit QAPjPs for review. Indicate which option will be exercised.

K.7 Biological Methods.
These methods are not detailed procedures and should either be provided in this QAPjP, in each operable unit QAPjP or as an Addendum to the Sitewide CERCLA QAPjP (SCQ). Please reconcile.

K.10 Field Storage and Shipment of Samples.
K.10.2 Sample Container Preparation.
The procedure provided is inadequate to ensure that containers are contaminant-free and needs to address separate container-preparation procedures for each analyte fraction. In addition, specific criteria for each container type must be provided (i.e. how clean). An example of a procedure and criteria used for organic and inorganic parameters used for CLP RAS low/medium concentration analytes is "Specifications and Guidance for the Preparation of Contaminant-Free Sample Containers" (USEPA April 1990). In addition, specifications for other sample containers (i.e. radiochemistry) should be detailed.

7.0 SAMPLE CUSTODY
This section still does not provide explicit, stepwise procedures for field custody and laboratory custody. Field custody shall detail the initiation and maintenance of custody from the point of sample generation through field transfers, in-field analyses and/or shipment to an off-site laboratory. All procedures for completing custody documents (tags, labels, forms, logs -), copies of all forms and the chronological sequence must be provided as part of the procedure. It is not clear whether the 2-part lable will meet USEPA NEIC's requirements for sample tags.

Laboratory custody section shall detail the continuation of custody from the point of sample receipt through in-house transfers, sample preparation/analysis and final disposal. All custody forms/logs and associated instructions for complete must be provided in the procedure.

8.0 CALIBRATION PROCEDURES AND FREQUENCY
9.0 ANALYTICAL PROCEDURES
No specific comments relative to any of the chemical analyses (calibration and analytical proicedures) are offered or necessary at this point. The USEPA Region 5 Central Regional Laboratory may provide additional comments relative to any laboratory audits which it may conduct for the project or oversight of the US DOE's lab audit process. These comments or audit results will be provided under separate cover.

Any deficiencies relative to the CRL's audits (if any) must be reconciled prior to QAPjP approval.

11.0 DATA REDUCTION, VALIDATION AND REPORTING
11.1 DATA REDUCTION.
Data reduction procedures for **field** analyses and measurements need to be specifically addressed.

11.2 DATA VALIDATION.
11.3 DATA REPORTING.
The validation procedures appear to be acceptable however there may some further discussion on the appropriateness of particular validation options for a specific operable unit QAPjP (when developed). The defined analytical support levels (i.e. ASLs A through E) have major differences between ASLs C and D since ASL C data does not include raw data while ASL D will include the data. This will directly affect the ability to validate ASL C data.

The ASLs should primarily first consider the amount of quality control that is built within a particular analytical method. Validation should be based upon the level of QC. The complete documentation of the analysis (i.e. complete data deliverables including raw data) should be included as part of both ASLs C and D. ASL C analyses would still require complete documentation for validation.

This issue should be discussed with the US DOE.

12.0 PERFORMANCE AND SYSTEMS AUDITS
Provide the detailed checklists of all items examined and procedures used during internal field and laboratory audits. It seems that the descriptions are still overviews which few procedural details. Please reconcile.

16.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT
a) All summary reports of QA activities should be forwarded to all members of the project management (including the USEPA). This will avoid the necessity of determining what "significantly" (section 16.2) means relative to data produced from the project. There may be differences in concerns between all agencies if the US DOE subjectively defines "significant" QA problems.
b) Identify all project management and QA management personnel who shall receive and review the QA reports.

APPENDICES.

APPENDIX C: DATA QUALITY OBJECTIVES
APPENDIX D: DATA VALIDATION PLAN
Comment relative to Appendices C and D were identified under QAPjP sections 11.2 and 11.3 comments above.

APPENDIX E: ANALYTICAL LABORATORY PERFORMANCE REQUIREMENTS
This Appendix is only an overview of the internal laboratory audit process and does not provide a detailed procedure for evaluating each part of the laboratories as well as the evaluation of each method. If US DOE has a more detailed procedure (i.e. similiar to what HAZWRAP uses), it should be substituted for this Appendix.

APPENDIX J: FIELD ACTIVITY METHODS
Comments relative to this Appendix were identified under QAPjP section 5.0 above.

APPENDIX K: SAMPLING METHODS
Comments relative to this Appendix were identified under QAPjP section 6.0 comments above.

ATTACHMENTS.

ATTACHMENT 1: FEMP LABORATORY ANALYTICAL METHODS MANUAL.
No specific concerns regarding organic and inorganic analytical procedures have been identified at this time. The USEPA Region 5 Central Regional Laboratory may have additional comments in conjunction with their audit of the project laboratories or the oversight of the US DOE audit process.

Comments on radiological parameters are deferred to those identified in the USEPA Region 5 Air & Radiation Division/USEPA-NAREL comments.

Radiation Section Comments On
"Site-Wide CERCLA Quality Assurance Plan,"
Fernald Environmental Management Project
Dated March 1992

Review has been made of the above document. Comments follow.

General Comments

In general, the March 4, 1992 revision of the Site-Wide CERCLA Quality Assurance Project Plan (SCQ) is a significant improvement of the October 31, 1991 version. The SCQ format follows and is consistent with current Agency guidance.

Specific Comments

1. **Section 1.5, page 7 of 11**--This section along with Figure 1-1 in Appendix A summarizes and simplifies the steps involved in implementing the SCQ. The first step in implementing the SCQ is described as being when a problem or a required data collection effort is identified. This section does not address implementation stages for sampling activities currently ongoing at FEMP. Since the scope of the SCQ is to direct all ongoing and future environmental projects at FEMP that implementation planning should include current as well as future projects. If quality assurance specifications have been built on those already existing at the site then this should be discussed.

2. **Section 1.4.2, page 6 of 11**--There are discrepancies within the SCQ as to exactly when a PSP will be generated. Section 1.4.2 states that a PSP shall be generated for each project. Section 1.5 states a PSP will be generated for each sampling activity. This difference between a project and sampling activity should be articulated with respect to the generation of PSP's.

3. **Section 2.2.3.1, page 5 of 27**--The waste material disposed of in waste pit 4 is estimated to be 57,6000 cubic yards. There appears to be an extra zero. Please correct this error.

4. **Section 3.2.2, page 7 of 9**--This section should include a reference that states that "Project Specific Plans (PSP), as part of the 1991 amended Consent Agreement activities, shall be reviewed and approved by USEPA prior to implementation."

5. **Section 4.4.1, page 10 of 20**--This section should be expanded to include training requirements specifically for the SCQ. A orientation to the SCQ should be provided to all personnel involved at the site. This will stress the importance of following SOP's to ensure data quality. This orientation can be included in the 40-hour compliance training program. Specific training needs should be evaluated on a project by project basis. If sampling activities, analytical methods or management systems are affected, specific training should be provided to the appropriate personnel.

[redacted]

6. **Appendix A, Table 4-1, page 93 of 123**--This table is not complete. The regulatory driver, length, and frequency are not provided for all courses.

7. **Appendix A, Table D-1 page 117 of 123**--This table presents correction factors for Ra-226 daughter ingrowth. There should be a reference in the appendix or in the SOQ to explain how these correction factors will be used and how they were derived.

[redacted]

[redacted]

General Comments

1) A section should be included in each procedure that provides the results of performance testing.

2) The acceptance criteria of ±3σ for all QA checks of counting systems (daily performance checks, plateau checks, efficiencies, calibrations, backgrounds, etc.) may be too broad in some cases (see below).

3) It is doubtful that individual alpha-particle energies can be routinely resolved for most alpha emitters. In many cases, the energies differ by only 40-60 KeV and will appear on the alpha spectrum as a single peak. Thus, including a term in the calculations for the probability of alpha emission at a particular energy per decay is unnecessary (see comment on Section 12 below).

4) Each procedure for water analysis mentions noting the presence of undissolved material, but no guidance is suggested if solids are observed.

5) Reagents should be listed in alphabetical order, or at least list the same compounds (e.g., different concentrations of the same acid) successively. This would make reference to them much easier.

Specific Comments

Method: RAD-0010 (Plutonium)

p.4, Step 8.15 - For 9M HCl, dilute 750 mL of concentrated HCl to 1 L with water, not 800 mL.

p.4, Step 8.19 - This step should include the preparation of 1M $(NH_4)_2SO_4$ solution; e.g., dissolve 132.2g of reagent grade $(NH_4)_2SO_4$ in distilled water and dilute to 1 liter.

p.5, Steps 9.3 and 9.4 - The check-source results should not only meet a ±3 sigma requirement, but should also be examined for bias and for the frequent occurrence of daily fluctuations that just do fall within ±3 sigma.

p.6, Note to Step 10.2 - The activities of ^{238}Pu and ^{239}Pu that trigger a change in the tracer from Pu-242 to Pu-236 should be given. Is there an SOP that provides the analyst with this information?

[redacted]

[redacted]

p.7, Step 11.2.8 - It is not necessary to boil the sample to remove the HCl, only heat to fumes.

p.7, Section 11.2 - This is more of a leaching procedure than an acid dissolution procedure, as total dissolution of an insoluble residue is not required.

p.8, Step 11.4.5 - Since the NH_4I solution is referred to in this step as 1 molar, it is suggested that (1M) be added following 145 g/l in Step 8.16.

p.9, Section 11.5 - A detailed figure of the electrodeposition cell would be useful.

p.11, Section 12 - It is questionable that the different alpha-particle energies of the same isotope can be routinely resolved, as they differ by only 40 to 50 KeV. All alpha particles of the same isotope will generally appear in the same peak, thus, Y_t and Y_s should be omitted.

Method: RAD-0040 (Radium-228)

p.11, Section 11.24 - Verifying the activity on the planchet to be Ac-228 by a second decay count is a good practice.

p.11, Section 11.25.2 - Ba-133 emits gamma-rays with energies ranging from about 53 to 384 KeV. It would be useful to identify the gamma-ray peak(s) used to measure the Ba-133.

Method: RAD-0050 (Radium-226)

p.2, Steps 6.1, 6.2, and 6.3 - These figures provide insufficient detail; for example, volume of the radon bubbler and type of stopcocks should be given. In Figure 2, what are the quantities of ascarite and anhydrous magnesium perchlorate, what is the volume and size of the scintillation cell, and how are the various components connected, etc.? Much greater detail is required.

Also, references to Ed Rushing and Henry Lucas who originally developed this procedure and designed the apparatus should be cited in the Reference Section.

p.6, Step 11.1 - Scintillation cells should be purged with helium immediately after counting to reduce decay product build-up on the cell walls. It is unclear from the procedure when this purge is performed.

[redacted]

p.10, Section 12.2 - To be totally clear, change definition of t_3 to read, "decay time of ^{222}Rn from end of de-emanation to start of count."

p.10, Section 12.4 - Units of E, scintillation cell efficiency, should be given (cpm/dpm).

p.11, Section 12.4 - See comment above regarding the definition of t_3.

Method: RAD-0070

p.8, Step 10.1 - The procedure requires commenting on any undissolved material noted, but doesn't tell what action to take if solids are observed. This appears in other procedures for the analysis of water, as well.

p.11, Steps 11.2.11 and 11.2.12 - A statement to mix well should be added to both of these steps.

p.12, Step 11.4.1 - The adhesive, thin plastic tape, film, and collodion solution mentioned in this step are not identified in the Apparatus (Section 6) or Reagents (Section 8) sections.

p.13, Section 12.2 - In the definition of "I," the term $e^{-\lambda_1 t'}$ for Pb-210 is actually 1.00, considering t is about 20 days.

Method: RAD-0080 (Thorium)

p.3, Section 6.15 - A description or a detailed figure of the electrodeposition cell would be helpful.

p.5, Section 8.17 - The silver nitrate solution used to test for chlorides (Section 8.16) is not described. This is usually a 0.1 molar solution.

p.5, Section 8.29 - To be a check source, Po-210 must be supported by Pb-210.

p.8, Section 10.3.5 - The approximate quantity of concentrated HNO_3 required to dissolve the ash should be given.

p.8, Section 10.3.6 - In the last sentence of the note, it should indicate that the evaporations should be to dryness.

p.9, Section 10.4.7 - What concentration of HCl should be used to wash the platinum dish?

p.9, Section 10.4.10 - Wash "solids" rather than "precipitate."

p.10, Sections 10.5.12 and 10.5.13 - It should be stated in each case that the washings are added to the 50 mL volumetric flask.

p.10, Section 10.5.16 - This is confusing as there should be no residue at this point.

p.11, Section 11.1.2 - This separation could be repeated to improve the yield, if necessary.

p.11, Section 11.2.1 - Add here, "and test for chlorides with $AgNO_3$ solution." This may be accomplished in Step 8.16.

p.14, Section 12.2 - I question the ability of the alpha spectrometer to resolve the alpha-particle energies of the individual isotopes (~60 KeV) on a routine basis and, thus, the need for Y_t and Y_s.

p.15, Section 12.2.1 - This will over estimate the Th-228 concentration if it is supported by Th-232, which will often be the case. The Th-232 result should provide information on the equilibrium status of the thorium isotopes in the sample and provide guidance on any necessary decay correction. This should be mentioned.

p.15, Section 12.2.2 - I doubt that it is possible to resolve routinely, or even very often, the individual alpha peaks.

Method: RAD-0100 (Uranium)

p.1, Section 2.0 - Polonium is "adsorbed," not "absorbed" onto the nickel.

p.3, Section 6.12 - A description or a detailed figure of the electrodeposition cell would be helpful.

p.5, Section 8.27 - To be a check source, Po-210 must be supported by Pb-210.

p.8, Section 10.3.5- Give an approximate volume of HNO_3 to dissolve the sample.

p.8, Section 10.3.6 - In the last sentence of the note, change to read "with evaporation to dryness."

p.9, Sections 10.4.12 and 10.4.13 - It should state in both cases to add each wash to the 50 mL volumetric flask.

p.9, Section 10.4.16 - "Dissolve residue" is confusing, because the sample is a spiked solution in the previous step (10.4.15).

p.11, Section 11.1.21 - Add "with stirring" to this step.

p.12, Section 11.3.2 - Actually, all individual uranium isotopes emit alpha-particle energies too similar to resolve on a routine basis. For example, U-238 (ΔE=40 KeV), U-236 (ΔE=50 KeV), and U-232 (ΔE=60 KeV). Thus, in Section 12.1, the terms Y_t and Y_s are unnecessary and should be omitted.

p.14, Section 12.3 - The specific activity of U-236 should be 64.7 pCi/μg rather than 647 pCi/μg, as stated.

p.17, Introduction - Annual decontamination of the U-232 tracer may not be sufficiently frequent for environmental sample analysis. Considering the 1.9 year half-life of Th-228, the activity of Th-228 will be about 31 percent of the U-232 activity after 1 year. Thus, adding 10 dpm of U-232 tracer (Section 10) will contribute about 3.1 dpm of Th-228.

Method: RAD-0130 (Gross Alpha & Beta)

p.8, Sections 10.2.12 and 10.2.13 - In both cases, indicate that washes are added to the 50 mL volumetric flask.

p.8, Section 10.2.16 - An additional step should possibly follow indicating that if the sample density exceeds the limits, 100 mg/cm^2 and 200 mg/cm^2 for alpha and beta counting, respectively, a new planchet would be prepared containing proportionately less sample.

Method: RAD-0140 (Gamma Spectrometry)

p.8, Sections 12.4 and 12.5 - To eliminate any confusion, it might be best to let R in the equation equal the detector plus sample container backgrounds.

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