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SEP 25 1992



92-RPB-212

Mr. A. W. Conklin, Head
Air Emissions & Defense Waste Section
Division of Radiation Protection
State of Washington Department of Health
Post Office Box 47827, Mail Stop LE-13
Olympia, Washington 99352

Dear Mr. Conklin:

TRANSMITTAL OF 222-S LABORATORY LIQUID STANDARDS

The following enclosure is provided per the request of Ms. Marina Silverstone, Mr. George Hilton, and Ms. Kathy Fox-Williams during their July 20, 1992, surveillance of the 222-S Laboratory. This letter is intended to document the transmittal of the following 222-S Laboratory procedures: LR-958-103, Alpha/Beta ABliq Standard, and LR-958-104, Environmental Alpha/Beta Standard.

If we can be of further assistance to you, please contact Mr. S. D. Stites of my staff on (509) 376-8566.

Sincerely,

James D. Bauer, Acting Program Manager
Office of Environmental Assurance,
Permits, and Policy

RPB:SDS

Enclosure:
Procedures

cc: R. E. Lerch, WHC

| Issued By: | Title |
|---|---|
| L. H. Taylor<br>Manager,<br>Analytical Laboratory | ALPHA/BETA LIQUID ABLIQ STANDARD |
| T. G. Ibsen | Impact Level 4 |


## SUMMARY

This Reference Material Specification (RMS) provides the specifications and instruction for preparation of the Alpha/Beta Liquid ABLIQ standard.


## SPECIFICATIONS

Standard: ABLIQ
Standard Units: µCi/L
Samples Analyzed: Effluent Water Samples

| ITEM | *TYPICAL RANGE | ACCEPTABLE ITEM UNCERTAINTY |
|---|---|---|
| $^{60}Co$ | (1.5-45) x $10^{-5}$ µCi/L | ± 5% |
| $^{241}Am$ | (2.5-75) x $10^{-5}$ µCi/L | ± 5% |

*The range may vary depending on the type of samples being simulated. See the controlled laboratory notebook for the current makeup values.

## LIMITATIONS

This standard is used in checking analytical methodology involving samples with alpha and beta radioactivity. The values for the alpha and beta activities will vary from standard to standard to cover the current range to assure measurement capability at the Derived Concentration Guide (DCG) value.

No inferences are to be made for materials with alpha or beta activities higher than twice the Derived Concentration Guide (DCG) value. The values for the standard are established by available instrumentation within the analytical laboratories, not always under control of Laboratory and Data Quality.

## SAFETY

The equipment and reagents identified in this procedure do not have hazards beyond those normally found in an analytical laboratory. User should review the equipment and reagent lists and assure familiarity with applicable safety precautions. Follow applicable laboratory safety precautions for handling radioactive materials, hazardous chemicals, and hazardous wastes.

REAGENTS

Nitric Acid ($HNO_3$), Concentrated (MSDS #1384)

Reagent grade concentrated $HNO_3$.

Nitric Acid ($HNO_3$), 1-2 M (MSDS #1384)

Dilute sufficient concentrated $HNO_3$ with deionized water to make 1 L at 1-2 M concentration.

Cobalt-60 ($^{60}Co$), 25-300 µCi/L (MSDS #NA)

Characterized for $^{60}Co$ concentration in 1-2 M $HNO_3$.

Americium-241 ($^{241}Am$), 35-500 µCi/L (MSDS #NA)

Characterized for $^{241}Am$ concentration in 1-2 M $HNO_3$.

EQUIPMENT

Magnetic Stirrer

50 mL calibrated volumetric flask

Pipets - calibrated, assorted sizes

2000 mL volumetric flask

Pipet control, dental cotton, tweezers

## PROCEDURE STEPS

### Stock Solution Preparation

1. Calculate the volume of each reagent required to make up the desired standard stock in the concentration range given in Appendix A. See CALCULATIONS, Step 1.

2. Pour 10-20 mL of 1-2 M $HNO_3$ into the calibrated 50 mL volumetric flask. Record the flask volume in a controlled laboratory notebook.

3. Accurately pipet a reagent solution into the flask and rinse the pipet three times with 1-2 M $HNO_3$. Record the volume of the pipet in the controlled laboratory notebook. (Stir each reagent solution before use.)

4. Repeat Steps 2 and 3 for each reagent needed to make up the particular stock solution.

5. Fill the flask to the index mark with 1-2 M $HNO_3$.

6. Add a stir bar and stir at least 20 minutes. Pour into a suitable storage bottle.

7. Calculate the concentration of each specified item in the stock solution. See CALCULATIONS, Step 2. Record the calculation.

### Standard Preparation

1. Refer to Appendix B and determine the pipet volume of stock solution to be used.

2. Stir the appropriate stock solution with a magnetic stirrer.

3. Pour 1.5 L of Q-water into a 2 L calibrated volumetric flask.

CAUTION: Use appropriate safety equipment when handling concentrated acid.

4. Add 65 mL of reagent grade concentrated $HNO_3$.

5. Accurately pipet the desired amount of the stock solution into the flask. Rinse the pipet three times with 1-2 M $HNO_3$. Record the volume of the pipet and the stock solution used in the controlled laboratory notebook.

6. Fill the flask to the calibration mark with Q-water.

7. Stir at least 3-4 hours.

PROCEDURE STEPS (Continued)

8. Pour the standard into a 2 L plastic bottle and label as per Procedure LO-120-001.

9. Calculate the value of each item in the final standard solution. See CALCULATIONS, Step 3.

VERIFICATION

1. The stock solution will be verified by two technicians making two sets of four mounts each. The first set is sent to the counting facility for alpha analysis and the second is sent for beta analysis. Use Procedures LA-548-101 and LA-548-103 for instructions on mounting techniques.

2. The data for the two sets of data will be tested by the F-test. See Appendix C for a typical calculator program for this. If the calculated F-ratio is less then or equal to the appropriate critical value of F, pool all the data for a stock and calculate the standard deviation of the mean at the 95% confidence level. If the F-test is failed or the confidence interval does not include the calculated value of the stock, consult the scientist in charge.

3. If the calculated and measured values agree, the standard is issued with the calculated value. Otherwise, the assigned scientist or manager will determine whether the material must be remeasured or discarded.

PACKAGING

1. The standard is left in the 2 L plastic bottle.

2. Label according to Procedure LO-120-001.

3. The expiration date is 4 months after makeup.

## CALCULATIONS

1. To calculate the amount of each reagent to use in preparing stock solutions:

$$\text{mL wanted} = \frac{C_d \times V_d}{C_r}$$

Where: $C_d$ = Concentration in μCi/L of desired stock.

$V_d$ = Volume in mL of stock to be made.

$C_r$ = Concentration in μCi/L of the reagent solution used (decayed, if necessary).

2. To calculate the concentration of any radioactive item in a stock solution.

$$C_{IS} = \frac{V_r \times C_r}{V_d}$$

Where: $C_{IS}$ = Concentration of an item in the stock solution.

$V_r$ = Volume in mL of reagent solution used.

$C_r$ = Concentration in μCi/L of the reagent solution used (decayed, if necessary).

$V_d$ = Volume in mL of the stock.

3. To calculate the concentration of any radioactive item in the final standard solution:

$$C_{IF} = \frac{C_{IS} \times V_s}{V_f}$$

Where: $C_{IF}$ = Concentration of an item in the final standard solution in μCi/L.

$C_{IS}$ = Concentration of an item in the stock solution in μCi/L.

$V_s$ = Volume of the stock used in mL.

$V_f$ = The final volume of the standard in mL.

## REFERENCES


1. LA-548-101, "Preparation of Aqueous Mounts for Total Alpha."
2. LA-548-103, "Gross Beta by Evaporation - Direct Mount."
3. LO-120-001, "Labeling of Standards and Reagents."

APPENDIX A

GUIDELINES FOR ABLIQ STOCK SOLUTIONS

| NUCLIDE | Concentrations (μCi/L) ± 10% | |
|---|---|---|
| | Stock A | Stock B |
| $^{60}Co$ | 0.15 | 0.45 |
| $^{241}Am$ | 0.75 | 0.25 |

APPENDIX B

RANDOM DILUTIONS OF STOCK ABLIQ

After each dilution number, a pipetted volume and stock is specified. These are to be used consecutively with the next dilution number chosen for each new ABLIQ standard. After dilution number 20, the next dilution will be dilution number 1.

| Dilution Number | Pipetted Volume (λ) | Stock | Dilution Number | Pipet Size (λ) | Stock |
|---|---|---|---|---|---|
| 1 | 400 | B | 11 | 200 | A |
| 2 | 400 | A | 12 | 200 | B |
| 3 | 200 | B | 13 | 200 | B |
| 4 | 2000 | A | 14 | 400 | B |
| 5 | 400 | B | 15 | 400 | A |
| 6 | 400 | B | 16 | 200 | B |
| 7 | 200 | A | 17 | 2000 | B |
| 8 | 400 | B | 18 | 200 | A |
| 9 | 200 | A | 19 | 400 | B |
| 10 | 400 | B | 20 | 400 | A |

APPENDIX C

F-TEST USER INSTRUCTIONS

ANOVA I

<1 f t.val 2>

START : Enter X : $\bar{X}$,$RS_m$ : Correct. : F crit.

| STEP | INSTRUCTIONS | KEYS | OUTPUT |
|---|---|---|---|
| 1 | Load both sides of card. | | |
| 2 | Initialize program. | A | |
| 3 | Enter values for a data set. | B | |
| 4 | After all points of a data set have been entered, calculate mean and relative standard deviation of the mean. | C | $\bar{X}$<br>$RS_m$ |
| 5 | Repeat 3 and 4 of all data sets. | | |
| 6 | Enter critical F value from table 1 below. | E | $\bar{X}$ overall<br>F crit.<br>F ratio<br>n |
| 7 | Based on "n", enter the appropriate value for t at the 95% confidence level. | f e | %Sm @ 95% |

Table 1

CRITICAL VALUES OF F

| Total # of measurements made by each technologists | # of data sets: 2 | 3 | 4 |
|---|---|---|---|
| 3 | 7.71 | 5.14 | 4.07 |
| 4 | 5.99 | 4.26 | 3.49 |
| 6 | 4.96 | 3.63 | 3.10 |

Issued By: T. G. Ibsen
Manager,
Analytical Laboratory

B. A. Crawford

Title: ENVIRONMENTAL ALPHA/BETA STANDARD (ENVAB)



## SUMMARY

This Reference Material Specification provides the specifications and instructions for preparation of the ENVAB (Environmental Alpha Beta) standard.

## SPECIFICATIONS

Standard: ENVAB
Standard Units: μCi/L
Samples Analyzed: Effluent water samples

| ITEM | *TYPICAL RANGE | ** ACCEPTABLE ITEM UNCERTAINTY | STOCK RANGE |
|---|---|---|---|
| $^{60}Co$ | $(4 - 8) \times 10^{-3}$ μCi/L | ± 5% | 20 - 40 μCi/L |
| $^{241}$ $^{239}Pu$ | $(4 - 8) \times 10^{-4}$ μCi/L | ± 10% | 2 - 4 μCi/L |
| $HNO_3$ | 0.1 to 1.0 M | ± 10% | |

*Range may vary depending on application. See the controlled laboratory notebook for the current makeup value.

**Uncertainty of ± 10% in $^{239}Pu$ measurements are acceptable when stock solutions have been previously characterized on alpha proportional counters and verified on total alpha detectors. Otherwise, uncertainties of ± 5% should be obtainable.

## APPLICATIONS

This specification is applicable in making any reference material, standard, or calibration standard. The range listed in the Specification Section is a guideline for producing the ENVAB standard. Other concentration ranges may be requested by the assigned scientist.

## LIMITATIONS

This standard is used in checking analytical methodology involving samples with alpha and beta radioactivity. Before any other use is undertaken, the chemical standards laboratory should be consulted as to the suitability of this material. The value for this standard is established based on purchased, certified material and counting room values and is, therefore, not completely under the control of the chemical standards laboratory.

## IMPACT LEVEL - 4

The reagents used are small quantities of common laboratory chemicals with a limited potential for environmental or personnel safety. Certification of prepared reference materials does not require QA verification; therefore, Impact Level 4 is assigned.

## SAFETY

Prior to application of this procedure, The user should review the equipment and reagents lists and assure familiarity with applicable MSDSs and safety precautions. Follow applicable laboratory safety precautions for handling radioactive materials, hazardous chemicals, and hazardous wastes.

## REAGENTS

Nitric Acid ($HNO_3$), (MSDS #1384)

Concentrated, reagent grade.

Nitric Acid ($HNO_3$), 1-2 M (MSDS #1384)

Dilute sufficient concentrated $HNO_3$ to make 1-2 M $HNO_3$.

Cobalt ($^{60}Co$) (MSDS #16301)

Certified material.

Plutonium ($^{239}Pu$) (MSDS# N/A)

Certified material.

## EQUIPMENT

Magnetic stirrer

50-mL calibrated volumetric flask, cleaned with detergent, Q-water, 1:1 $H_2O$:$HNO_3$, and 1:1 $H_2O$:HCl (MSDS #1250)

Pipets: calibrated, assorted sizes

4000-mL volumetric flask

Pipet control

Dental cotton

Tweezers

50-mL glass storage bottle with poly-lined cap, cleaned with detergent, Q-water, 1:1 $H_2O$:$HNO_3$, and 1:1 $H_2O$:HCl

Parafilm

4-L plastic storage bottle: cleaned, new bottle or reuse previous storage bottle

## PROCEDURE STEPS

### Stock Solution Preparation

1. Calculate the volume of each certified material required to make up the stock solution in the concentration ranges given. See Calculations Section, Step 1. Depending on the activity, an intermediate dilution may be required between the certified solution and the stock solution.

2. Pour 20-30 mL of 1-2 M $HNO_3$ into the calibrated 50-mL volumetric flask. Record the calculated volume of the flask volume in a controlled laboratory notebook.

3. Accurately pipet a certified material into the flask. Record the volume of the pipet in the controlled laboratory notebook.

4. Repeat Step 2 for the other certified material.

5. Fill the flask to the index mark with 1-2 M $HNO_3$.

6. Add a stir bar and stir at least 20 minutes. Pour into a suitable storage bottle, cap, and seal with parafilm.

7. Calculate the concentration of each specified item in the stock solution. See Calculations Section, Step 2. Record the calculation in a controlled laboratory notebook.

### Standard Preparation

1. Pour approximately 3.5 liters of Q-water into a 4-liter calibrated flask.

2. Add 125 mL of reagent grade concentrated $HNO_3$ to the flask, slowly.

3. Accurately pipet 0.800 mL of the stock solution into the flask. Rinse the pipet three times with 1-2 M $HNO_3$.

4. Fill the flask to the calibration mark with Q-water.

5. Stir at least 3-4 hours.

6. Pour the standard into a 4-liter, plastic bottle and label properly.

7. Calculate the value of each item in the final standard solution. See Calculations Section, Step 3.

## VERIFICATION

1. The reagent stock material is verified by two technicians preparing at least four mounts each and sending them to the counting facility for analysis. Use Procedure LA-548-122 for instructions on mounting techniques.

2. If the stock material and measured value agree, the final standard solution is issued with the calculated value. Otherwise the manager or scientist must decide to remeasure or discard the material.

3. In the event that the material does not meet specifications, the two sets of data will be tested by the F-test. If the calculated F-ratio is less than or equal to the appropriate critical value of F, pool all the data for a stock and calculate the standard deviation of the mean at the 95% confidence level. If the F-test is failed or the confidence interval does not include the calculated value of the stock, consult the scientist in charge. See Appendix A for an example of a typical calculator program.

## PACKAGING

1. The standard is left in the 4-liter, plastic bottle.

2. Label each standard according to Procedure LO-120-001.

3. Expiration date is one year after makeup.

4. Dispense on request.

## CALCULATIONS

1. To calculate the amount of each certified material needed in preparing the stock solution:

$$\text{mL of certified material needed} = \frac{C_s \times V_s}{C_{cm}}$$

Where:

$C_s$ = Concentration in $\mu$Ci/L of desired stock.

$V_s$ = Volume in mL of stock to be made.

$C_{cm}$ = Concentration in $\mu$Ci/L of the certified material used (decayed, if necessary).

## CALCULATIONS (Continued)

2. To calculate the concentration of any radioactive item in the stock solution:

$$C_s = \frac{V_{cm} \times C_{cm}}{V_s}$$

Where:

$C_s$ = Concentration of an item in the stock solution

$V_{cm}$ = Volume in mL of the certified material used

$C_{cm}$ = Concentration in $\mu$Ci/L of the certified material used (decayed, if necessary)

$V_s$ = Volume in mL of the stock

3. To calculate the concentration of any radioactive item in the final standard solution:

$$C_f = \frac{C_s \times V_s}{V_f}$$

Where:

$C_f$ = Concentration of an item in the final standard solution in $\mu$Ci/L

$C_s$ = Concentration of an item in the stock solution in $\mu$Ci/L

$V_s$ = Volume of the stock used in mL

$V_f$ = The final volume of the standard in mL

APPENDIX A

F-TEST USER INSTRUCTIONS

ANOVA I

<1 f t.val 2>

START : Enter X : $\bar{X}$,$RS_m$ : Correct. : F crit.

| STEP | INSTRUCTIONS | KEYS | OUTPUT |
|---|---|---|---|
| 1 | Load both sides of card. | | |
| 2 | Initialize program. | A | |
| 3 | Enter values for a data set. | B | |
| 4 | After all points of a data set have been entered, calculate mean and relative standard deviation of the mean. | C | $\bar{X}$<br>$RS_m$ |
| 5 | Repeat 3 and 4 of all data sets. | | |
| 6 | Enter critical F value from table 1 below. | E | $\bar{X}$ overall<br>F crit.<br>F ratio<br>n |
| 7 | Based on "n", enter the appropriate value for t at the 95% confidence level. | f e | %Sm @ 95% |

Table 1

CRITICAL VALUES OF F

| Total # of measurements made by each technologists | # of data sets: 2 | 3 | 4 |
|---|---|---|---|
| 3 | 7.71 | 5.14 | 4.07 |
| 4 | 5.99 | 4.26 | 3.49 |
| 6 | 4.96 | 3.63 | 3.10 |

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Subject: TRANSMITTAL OF 222-S LABORATORY LIQUID STANDARDS

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