AP1

GROSS ALPHA AND BETA FOR VARIOUS MATRICES

PART A

PRINCIPLE

Liquids samples are acidified, concentrated, dried in a planchette, and counted in a low-background proportional counter. Solid samples are dried and processed to provide homogeneity and a known quantity is transferred to a planchette and counted in a low-background proportional counter. The activity determined by this method is not indicative of any specific nuclide.

REFERENCES

1. F.B. Johns et al., Radiochemical Analytical Procedures for Analysis of Environmental Samples, EMSL-LV-0539-17, March 1979.

2. Annual Book of ASTM, Standards Vol. 11.02, pp. 296-299 and pp. 304-309.

Certification Record for

PROCEDURE AP1

GROSS ALPHA AND BETA FOR VARIOUS MATRICS

CHECKPOINTS

1.	**JOB HAZARD ANALYSIS (JHA)**	____
2.	**MSDS/HAZARDS DISCUSSED**	____
3.	**VOLUME DETERMINATION**	____
4.	**TRANSFER TO PLANCHETTE**	____
5.	**SAMPLE STABILIZATION**	____
6.	**SAMPLE COUNTING**	____
7	**FINAL CALCULATIONS**	____

ANALYST SIGNATURE:_____

CERTIFIED BY:_____

DATE:_____

ANALYSIS VALUE:_____

KNOWN VALUE: _____

MEASURED/KNOWN RATIO:_____

COMMENTS: _____
_____
_____
_____
_____

PART B

1.0 PURPOSE AND SCOPE

This procedure provides a screening measurement to indicate whether specific chemical analyses are required for water, soil, vegetation, and other solids. Volatile radionuclides (Tc-99, H-3, etc) will not be accurately determined using this procedure.

DISCLAIMER: Gross screening analyses are intended to provide rapid information associated with a particular action level with minimal chemical preparation; therefore, this analysis is not as accurate as specific chemical analyses.

2.0 REAGENTS

All chemicals are hazardous. See MSDS for specific precautions.

Nitric acid, HNO_3, 0.01 M: To 400 mL reagent water, add 0.3 mL 16 M HNO_3. Dilute to 500 mL

Nitric acid, HNO_3, 0.1 M: To 400 mL reagent water, add 3 mL 16 M HNO_3. Dilute to 500 mL

Nitric acid, HNO_3, concentrated, 16 M

Acetone/Super Glue mixture: To 200 mL acetone, add a half of a tube of super glue; mix well

Potassium sulfate, K_2SO_4, 10 mg/ mL: Dissolve 0.5 g of K_2SO_4 in 50 mL of 0.01 M HNO_3

Potassium sulfate, K_2SO_4, 20 mg/ mL: Dissolve 1.0 g of K_2SO_4 in 50 mL of 0.01 M HNO_3

Potassium sulfate, K_2SO_4, 40 mg/ mL: Dissolve 1.0 g of K_2SO_4 in 25 mL of 0.01 M HNO_3

3.0 APPARATUS

Low-background proportional counter
Stainless steel ringed planchettes
Filter paper and apparatus
Hot plate
Beakers
Graduated cylinders
Transfer Pipettes

4.0 PROCEDURE

4.1 General Requirements

Before proceeding, you must be certified as indicated in QCP1 of this manual and Section 3 of the Quality Program Manual. See page two for a copy of the certification record.

4.2 Water Samples

4.2.1 If total activity is desired, the sample should be acidified with nitric acid or hydrochloric acid to a pH of 2. **See step 4.2.1 of AP1 JHA.**

4.2.2 If dissolved and suspended solid activities are desired, the sample should be filtered using a 0.45 micrometer pore size membrane filter. The filter paper should be retained and counted. The filtered sample should be acidified as in 4.2.1. **See step 4.2.2 of AP1 JHA**.

4.2.3 Determine the amount of dissolved solids by drying 5.0 mL of sample on a tared, stainless steel planchette. The planchette may need to be flamed and reweighed. Use the following equation or the GAB program to calculate the volume of sample to analyze.

$$120\ mg \times \frac{5mL}{mg\ of\ residue} = mL\ of\ sample$$

If the value obtained is greater than 250 mL, use 250 mL of sample. The test planchette may be used for sample analysis by subtracting 5.0 mL from the total volume.

4.2.4 Measure an appropriate aliquot of sample (from above) by weight, into a graduated cylinder, or a volumetric container and quantitatively transfer to an appropriate size beaker. **See step 4.2.4 of AP1 JHA.**

4.2.5 Heat until approximately 5-10 mL of the solution remains. **See step 4.2.5 of AP1 JHA.**

4.2.6 Add 10 mL 16 M nitric acid and heat until 5-10 mL remain. Repeat nitric acid addition and heat until 5-10 mL remain. **See step 4.2.6 of AP1 JHA.**

4.2.7 Quantitatively transfer the remaining solution to a tared stainless steel planchette using 0.1 M nitric acid. **See step 4.2.7 of AP1 JHA.**

4.2.8 Slowly evaporate to dryness on a hotplate to avoid spattering. Heat the dried planchette over a low flame Bunsen burner or on a very hot hotplate for two minutes to ensure all nitrate salts are converted to oxide salts. (Note: volatile radionuclides may be lost or partially lost at this step). **Go to step 4.3.5. See step 4.2.8 of AP1 JHA.**

4.3 Solid Samples

4.3.1 Weigh approximately 0.1 g of ashed or dried, previously homogenized, and sieved sample on an analytical balance and transfer to a centrifuge tube. Record the weight as the sample quantity on the data sheet. Samples must be dried and passed through a 35 - 50 mesh sieve. Samples with especially high organic content may require ashing and sieving, prior to analysis. **See step 4.3.1 of AP1 JHA**.

4.3.2 Transfer the sample to a tared planchette by adding water to the sample and transferring using a plastic disposable transfer pipette. **See step 4.3.2 of AP1 JHA.**

4.3.3 Add approximately 2 mL reagent water or 95% ethanol to the planchette using a disposable transfer pipette. Spread the sample uniformly on the planchette. Gently suspend sample in water using the transfer pipette. Slowly evaporate on a hot plate. **See step 4.3.3 of AP1 JHA.**

4.3.4 Add 5-10 drops of the glue/acetone mixture to seal sample in planchette. Allow to dry. **See step 4.3.4 of AP1 JHA.**

4.3.5 Count the sample on a low background proportional counter long enough to met MDC requirements, if possible. **See step 4.3.5 of AP1 JHA**.

4.3.6 Weigh the planchette. Record total planchette and sample weight for counting efficiency calculation on the data sheet. **See step 4.3.6 of AP1 JHA**.

5.0 CALIBRATIONS

5.1 NIST traceable standards of Pu-239 and Cs-137 or Sr-90 are selected to generate the gross alpha and gross beta efficiency/attenuation curves.

5.2 Weigh the planchettes needed for each efficiency/attenuation curve and record the weight either electronically or manually on the assignment sheet. The mass range for the gross alpha and gross beta curves are 0 mg to 120 mg in 10 mg increments.

5.3 Add approximately 1000 pCi of the alpha standard to each alpha planchette and approximately 1000 pCi of the beta standard to each beta planchette.

NOTE: If the standard solution is in HCl, you cannot add directly to planchette. Add the standard solution to a beaker containing 3 to 5 mL of concentrated HNO_3 and reduce the volume to approximately 1 mL. Repeat three times. Do not permit the solution to go to dryness.

5.4 Starting with the 10 mg planchette for each curve and continuing in 10 mg increments until 120 mg is reached, add the appropriate volume of K_2SO_4 solution to yield the desired mg weight on each planchette.

5.5 Take the solution in each planchette to dryness using a hot plate and allow cooling. Weigh record each weight on the assignment sheet.

5.6 Submit the planchettes for counting. Count long enough to achieve the desired counting statistics of less than 1 percent.

5.7 After all planchettes have been counted, the system software will generate the efficiency/attenuation curve.

5.8 The Laboratory Manager or designee must review and approve each curve.

6.0 CALCULATIONS

Critical data values will be documented on approved assignment and calculation forms referenced to the current procedure and revision. See pages 8-10 of this procedure for approved forms. Critical records are maintained as hard copy in the archived site file or electronically. The following equations define the critical data values. All data will be recorded and reduced according to these calculations.

Gross Beta Equations

$$C_\beta = \frac{[(G_\beta - B_\beta) - (G_\alpha - B_\alpha)\cdot \chi_{\alpha\to\beta}]}{E_\beta \cdot Q \cdot T \cdot [1 - \chi_{\beta\to\alpha}\cdot\chi_{\alpha\to\beta}]} = pCi/unit$$

$$2\sigma Error_\beta = \frac{1.96\sqrt{G_\beta + B_\beta + G_\alpha + B_\alpha}}{E_\beta \cdot Q \cdot T} = pCi/unit$$

$$2\sigma\ TPU_\beta = C_\beta \cdot 1.96\sqrt{\frac{G_\beta + B_\beta}{(G_\beta - B_\beta)^2} + RE_\beta^2 + RQ^2 + R\chi_{\alpha\to\beta}^2} = pCi/unit$$

When $G_\beta - B_\beta$=zero, 1 will replace the term $\frac{G_\beta + B_\beta}{(G_\beta - B_\beta)^2}$ in the TPU equation.

$$MDC_\beta = \frac{3 + 4.65\sqrt{B_\beta}}{E_\beta \cdot Q \cdot T} = pCi/unit$$

Gross Alpha Equations

$$C_\alpha = \frac{[(G_\alpha - B_\alpha) - (G_\beta - B_\beta)\cdot \chi_{\beta\to\alpha}]}{E_\alpha \cdot Q \cdot T \cdot [1 - \chi_{\beta\to\alpha}\cdot\chi_{\alpha\to\beta}]} = pCi/unit$$

$$2\sigma\ Error_\alpha = \frac{1.96\sqrt{G_\alpha + B_\alpha + G_\beta + B_\beta}}{E_\alpha \cdot Q \cdot T} = pCi/unit$$

$$2\sigma\ TPU_\alpha = C_\alpha \cdot 1.96\sqrt{\frac{G_\alpha + B_\alpha}{(G_\alpha - B_\alpha)^2} + RE_\alpha^2 + RQ^2 + R\chi_{\beta\to\alpha}^2} = pCi/unit$$

When $G_\alpha - B_\alpha$ = zero, 1 will replace the term $\frac{G_\alpha + B_\alpha}{(G_\alpha - B_\alpha)^2}$ in the TPU equation.

$$MDC_\alpha = \frac{3 + 4.65\sqrt{B_\alpha}}{E_\alpha \cdot Q \cdot T} = pCi/unit$$

where:

B_α	=	alpha background counts
B_β	=	beta background counts
E_α	=	alpha counting efficiency, cpm/pCi
E_β	=	beta counting efficiency, cpm/pCi
G_α	=	alpha gross sample counts
G_β	=	alpha gross sample counts
Q	=	quantity in units Liter (L), sample (S), or gram (g)
T	=	counting time, minutes
C_α	=	alpha concentration of sample in pCi/unit
C_β	=	beta concentration of sample in pCi/unit
$\chi_{\alpha\to\beta}$	=	alpha in beta crosstalk
$\chi_{\beta\to\alpha}$	=	beta in alpha crosstalk
TPU_α	=	alpha total propagated uncertainty
TPU_β	=	beta total propagated uncertainty
MDC_α	=	alpha minimum detectable concentration
MDC_β	=	beta minimum detectable concentration
RE_α	=	1σ relative uncertainty of the alpha efficiency
RE_β	=	1σ relative uncertainty of the beta efficiency
RQ	=	1σ relative uncertainty of the quantity
$R\chi_{\alpha\to\beta}$	=	1σ relative uncertainty of the alpha into beta crosstalk
$R\chi_{\beta\to\alpha}$	=	1σ relative uncertainty of the beta into alpha crosstalk

7.0 RECORDS

7.1 Reference QA Manual for general record requirements.

7.2 The raw count data are saved during the daily incremental backup of the Low Background Alpha/Beta counter to the ORISE network disks. A disk image backup is performed once a month.

7.3 Hard copies of assignment and calculation sheets are maintained in the archived site file. Electronic copies of assignment and calculation sheets are saved during the daily incremental backup of the network system. The following data sheets show the required data and information. These forms or the equivalent should be completed and retained:

- GAB Analysis Assignment Form
- GAB Lab Data Sheet
- GAB Concentration and Uncertainty Report (This report may be generated using approved Excel spreadsheets or from the database, if available.)

AP1(Rev 16) - GAB ANALYSIS ASSIGNMENT FORM

(PAGE 1 OF 2, SEE OTHER SIDE)

Assigned To: _____ **Date:** _____ **Batch:** _____

Task #: _____ **LWR #:** _____ **Activity Lev*:** _____

Sample #'s: _____

_____

_____

QC REQUIRED

BLANK [X]

REPLICATE [ ] **SAMPLE #** _____ **# Replicates** _____

INITIALS

LCS [X] **ALPHA STD #** _____ **QUANTITY** _____ [ ]

UNITS _____

Pipette # _____ Volume _____ Weight _____

BETA STD # _____ **QUANTITY** _____ [ ]

UNITS _____

Pipette # _____ Volume _____ Weight _____

MATRIX SPK [ ] **SAMPLE #** _____

ALPHA STD # _____ **QUANTITY** _____ [ ]

BETA STD # _____ **QUANTITY** _____ [ ]

SPECIAL INSTRUCTIONS: _____

_____

_____

_____

* If Activity Level is indicated Moderate or High, perform area survey.

AP1(Rev 16) - GAB LAB DATA SHEET

(PAGE 2 OF 2, SEE OTHER SIDE)

Carrier #							
Sample #							
Sample Quantity							
Quantity Units							
Smp + Plan Wt.							
Planchette Wt.							
5 ml Sample + Planchette Wt.							
Analysis Volume							
Wt. Units							

Carrier #							
Sample #							
Sample Quantity							
Quantity Units							
Smp + Plan Wt.							
Planchette Wt.							
5 ml Sample + Planchette Wt.							
Analysis Volume							
Wt. Units							

Carrier #							
Sample #							
Sample Quantity							
Quantity Units							
Smp + Plan Wt.							
Planchette Wt.							
5 ml Sample + Planchette Wt.							
Analysis Volume							
Wt. Units							

Carrier #			
Sample #			
Sample Quantity			
Quantity Units			
Smp + Plan Wt.			
Planchette Wt.			
5 ml Sample + Planchette Wt.			
Analysis Volume			
Wt. Units			

AP1(Rev 16) - GROSS ALPHA/BETA ACTIVITY & ERROR REPORT

OPERATOR INITIALS _____ DATE _____ BATCH _____ TASK # _____

DETECTOR # 1

CARRIER TRAY#						
SAMPLE ID						
WT. OF SAMPLE& PLAN. - g						
PLAN. WT.-g						
SAMPLE WT.-mg						
VOLUME OR DRY WT.						
F,G,L OR T						
COUNT TIME(MIN)						
ALPHA TOTAL COUNTS						
ALPHA BKG TTL COUNTS						
BETA TOTAL COUNTS						
BETA BKG TTL COUNTS						
DATE COUNTED						