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GROSS BETA ACTIVITY

Analyte: Beta-Emitting Nuclides	**Method No:** R130
Matrix: Urine, Water	**Accuracy and Precision:** 70% ± 5% (mean ± 1 sigma) at the 10-pCi level
Contact Person: William D. Moss	
Minimum Detectable Activity: 25 pCi/L	**Acceptable Minimum Detectable Activity:** 25 pCi/L
Procedure: Oxalate Coprecipitation and Beta Counting	**Effective Date:** 01/01/74 to 01/01/89

1. Principle of Method

1.1. Beta-emitting material is coprecipitated with calcium from a urine specimen as calcium oxalate, using ammonium oxalate in an acid medium.

1.2. The washed precipitate is vacuum-filtered through a glass fiber filter and the beta activity counted on a low-background, methane flow proportional counter.

2. Sensitivity

2.1. The detection limit is usually 1 dis/s/1000 cm^3 of urine. High background levels in the Beckman Wide Beta Counter may raise the levels to 2 dis/s.

3. Accuracy and Precision

3.1. Samples spiked with ^{90}Sr at a level of 20 dis/s gave a mean recovery of 70% ± 5% at the 10-pCi concentration level.

4. Collection and Storage of Samples

4.1. Equivalent 24-hour samples are collected in glass specimen bottles.

4.2. Refrigerate samples pending analysis.

5. Apparatus

5.1. Graduated cylinders: 1000-cm^3.

5.2. Centrifuge tubes: 90-cm^3, round bottom, Pyrex.

5.3. Aluminum heating block: capable of holding sixteen 90-cm^3 centrifuge tubes.

5.4. Hot plate: to hold aluminum heating block.

5.5. Glass fiber filter paper: 5.08-cm-diameter circles, Type E, Gelman Instrument Co., Ann Arbor, Michigan.

5.6. Millipore filter assembly: Millipore Corp., Bedford, Mass.

5.7. Beckman Wide Beta II Counter.

6. Reagents

6.1. Ammonium hydroxide (concentrated 28%, specific gravity 0.90).

6.2. Ammonium hydroxide (0.05%). Add 1 cm^3 concentrated NH_4OH to 500 cm^3 distilled water and mix.

6.3. Ammonium oxalate.

6.4. Glacial acetic acid (97%, specific gravity 1.049).

6.5. Methane (Phillips Pure Grade, 99% Mol Minimum): Phillips Petroleum Co., Special Products Division, Bartlesville, Oklahoma.

6.6. 1-Octanol (Eastman, practical-grade).

6.7. Oxalic acid.

6.8. Sulkowitch reagent. Place 25.0 g oxalic acid and 25.0 g ammonium oxalate in a 1000-cm^3, glass-stoppered, graduated cylinder. Add 50 cm^3 glacial acetic acid and 500 cm^3 distilled water. Stir until dissolved. Cool and dilute to 750 cm^3 with distilled water. Mix well and filter. (This is double the strength of standard Sulkowitch reagent to minimize the volume of liquid in the analysis.)

7. Calibration and Standards

7.1. The primary ^{90}Sr standard may be purchased from New England Nuclear, 549 Albany St., Boston, Massachusetts 02118.

7.2. The primary solution is made by diluting a 1-mCi standard solution of ^{90}Sr to 1000 cm^3 with distilled water. This solution is standardized in a calibrated beta counter by counting disks containing 0.1 cm^3 of the stock solution.

7.3. If the primary solution is greater than 1000 pCi/cm^3, a dilution of this solution should be made to keep the concentration less than 1000 pCi/cm^3.

7.4. Urine standards are prepared by adding the required amount of standard solution to blank urine samples.

7.5. Standards and reagent blanks are processed through the entire analytical procedure.

8. Procedure

8.1. Pool each urine sample in a 1000-cm^3 graduated cylinder.

8.2. Record the temperature and specific gravity.

8.3. Place 75 cm^3 of the pooled sample in a 90-cm^3, round bottom centrifuge tube.

8.4. Heat at 70 to 80°C in an aluminum block for 15 to 20 minutes. Do not add boiling chips.

8.5. Add dropwise, with vigorous stirring, 10 cm^3 of Sulkowitch reagent. Add a drop of octyl alcohol if the urine should effervesce uncontrollably.

8.6. Digest the sample for 60 min or until the precipitate is no longer gelatinous.

8.7. Cool and centrifuge for 20 min at 1500 rpm.

8.8. Place a 5.08-cm^3-diameter glass fiber filter on the base of a millipore filter funnel and turn on the vacuum.

8.9. Decant and discard the supernate.

8.10. Wash the precipitate onto the filter with 25 cm^3 of 0.05% NH_4OH by forcibly squirting the reagent in a fine stream from a wash bottle to stir up the precipitate. Transfer the precipitate to the filter. Repeat the washing until the transfer is completed.

8.11. Wash down the walls of the funnel with a few drops of 0.05% NH_4OH.

8.12. Remove the funnel with the vacuum still on.

8.13. Release the vacuum. Carefully transfer the filter to a drying oven with a flat tweezer and dry for several hours at 100°C.

8.14. Place the filter on the counting planchets and submit for measurement of beta activity.

9. Operation of the Beta Counter

9.1. Low-level beta emissions are counted on a methane flow proportional counter.

9.2. The gross counting data must be corrected for counter background and efficiency.

9.2.1. Counter background is determined by counting a blank plate before and after each sample.

9.2.2. Efficiency is determined daily from a New England Nuclear ^{210}Bi standard of known activity.

9.3. Blanks, standards, and samples are placed in counting planchets.

9.4. Sample area and mass must be reproducible. Holders for samples, blanks, and standards must be identical.

9.5. Refer to the operating manual for the Wide Beta II gas flow proportional counter for complete instructions.

10. Determination of the High-Voltage Operating Point of the Beta Counter

10.1. Adjust the gas flow and turn on the counter.

CAUTION: Never operate the counter unless adequate gas is flowing through the chamber. No count is valid if pressure in the methane tank is less than 500 lbs.

10.2. Position the standard plate in the counting chamber.

10.3. Adjust the high-voltage control back to a point at which the voltage is too low for the counter to function.

10.4. Raise the voltage slowly until the scaler registers approximately 100 counts/minute.

10.5. Raise the voltage in increments of 50 volts, recording the counts/minute at each stop.

10.6. Plot counts per minute versus high voltage.

10.7. Set the high voltage to operate at a point just above the knee of the plateau.

11. Determination of Counter Efficiency

11.1. Replace the standard plate with the blank plate.

11.2. Flush the chamber by counting the newly inserted plate for 2 minutes. Do not record the count.

NOTE: Always flush the chamber for 2 minutes after changing plates.

11.3. Count the blank for 10 minutes. Calculate the counts/minute.

11.4. Repeat the 10-minute blank counts until 3 successive counts agree within ± 0.5 counts/minutes.

11.5. Count the standard for 10 minutes. Calculate the counts/minute.

11.6. Count the background plate again. Average the before and after background counts/minute. Subtract this average count from the standard counts/minute to obtain the net standard counts/minute.

11.7. Divide the net standard counts/minute by the known count of the standard to obtain the counter efficiency, which should be 0.50.

11.8. The average chemical recovery of the control samples is 0.8.

12. Calculations

12.1. The beta activity is determined using the following formula.

$$pCi/L = \frac{C_s - C_b \times 1000}{T \times V \times E \times R \times K}$$

where C_s = sample counts
C_b = background counts
T = time in minutes (50)
V = volume in cm^3 of aliquot analyzed
E = efficiency of the counter
R = average chemical recovery of control samples (0.8)
K = 2.22 (to convert dis/min to pCi)

13. Source Materials

13.1. C. A. Mawson and I. Fischer, "The Estimation of Radioactive Strontium and Other Fission Products in Urine and Water," National Research Council of Canada, Research Division, Chalk River, Ontario, Canada, report CRM-455 (1950).

13.2. Manual of Standard Procedures, Health and Safety Laboratory USAEC, New York Operations Office report NYO-4700 (March 1957).

GROSS ALPHA AND BETA ACTIVITY IN ENVIRONMENTAL MATRICES — GAS-FLOW PROPORTIONAL COUNTING

Analyte: Alpha- and beta-emitting nuclides	**Method No.:** ER100
Matrix: Soil, water, and air particulates	**Minimum Detectable Activity:** Soil, water ~1.2 pCi (alpha, beta) Filters 0.3 pCi (alpha) Filters 0.6 pCi (beta)
Procedure: Gas-flow proportional counting	**Accuracy and Precision:** Filters 94% ± 5% Water 105% ± 11%
Effective Date: 01/01/75	**Authors:** Richard J. Peters George H. Brooks

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 7. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment.

1. Principle of Method

1.1. Samples are mounted on stainless steel planchets following leaching of soil samples and evaporation of water samples.

1.2. Samples are counted for 100 min on a low-background, gas-flow proportional counter.

2. Sensitivity

2.1. Sensitivity will vary greatly for alpha activity and to a lesser degree for beta activity due to self-absorption by the residual sample mass.

2.2. The minimum detection limit for both alpha and beta is approximately 3 pCi total activity for a representative soil or water having 0.1 g of residue.

2.3. For a representative air filter, the minimum detection limit is 0.5 pCi total alpha activity and 1 pCi total beta activity.

3. Accuracy and Precision

3.1. The efficiency and crosstalk between alpha and beta counts in a gas-flow proportional counter may vary greatly depending on the energy of the alpha or beta emitter. Plutonium-239 and $^{90}Sr/^{90}Y$ are used as the basis for all crosstalk and relative efficiency determinations. The accuracy of a given measurement will depend on the actual energies of the unknown nuclides.

3.2. The following accuracy and precision measurements were made in 1988 on air filter and water proficiency samples provided by the Environmental Monitoring Systems Laboratory-Las Vegas quality assurance program.

	Filter Samples	Water Samples
alpha	93% ± 6%	107% ± 13%
beta	94% ± 4%	103% ± 10%

4. Interferences

4.1. The crosstalk correction method may not adequately protect from incorrect identification of radiation from samples containing very low-energy alpha or high-energy beta activities.

4.2. Moderate to high levels of gamma radiation can be misinterpreted as alpha or beta activity by gas-flow proportional counters.

4.3. The addition of a thin mylar sheet covering the planchet results in increased self-absorption in samples containing low-energy alpha and beta activities and may result in an underestimation of the actual concentrations.

5. Collection and Storage of Samples

5.1. There are no special collection and storage requirements.

6. Apparatus

6.1. Low-background alpha/beta proportional counter: equipped with a 2.25-in. ultrathin counting window (80 $\mu g/cm^2$) and a 50-sample automatic changing capability. Tennelec Model No. LB-5100, Tennelec Inc., Oak Ridge, Tennessee.

6.2. P-10 (argon/methane) gas cylinder.

6.3. Planchets: stainless steel, 2.0- by 0.25-in., Atomic Products Corp., Center Moriches, NY.

6.4. Mylar sheet: 0.00025-in. thick.

6.5. Beakers: 50-, 600-, and 1000-mL, pyrex.

6.6. Tubes: 40- and 50-mL, centrifuge.

6.7. Heat lamp.

7. Reagents

7.1. Nitric acid (concentrated, reagent-grade).

7.2. Hydrofluoric acid (concentrated, reagent-grade).

7.3. Hydrochloric acid (concentrated, reagent-grade).

7.4. Hydrogen peroxide (30%, reagent-grade).

7.5. Nitric acid (7.2 M). Dilute 460 mL of concentrated HNO_3 to 1000 mL with distilled water.

8. Calibration and Standards

8.1. Generate detector efficiencies for the determination of alpha and beta activities from curves that plot the absolute detector efficiency as a function of the sample mass residue.

8.1.1. Use National Institute of Standards and Technology (NIST) standards 5516 and 4919-D.

8.1.2. Prepare a group of 10 standards with the same amount of spike and vary the amount of residue (0.05 - 1.50 g) to generate the curve.

8.1.3. Plot efficiency as a function of the sample mass residue.

8.1.4. Derive an equation to describe the curves for both alpha and beta energies. Efficiencies can then be interpolated from these curves for a sample of any mass.

8.2. Count plated ^{239}Pu and $^{90}Sr/^{90}Y$ standards for 10 min with each set of samples. Use the data to determine two factors for gas-flow proportional counting: 1) the alpha/beta and beta/alpha crosstalk and 2) the system's relative performance based on the derived efficiencies.

8.3. Count two mylar-covered empty planchets along with the sample set for 100 min to determine alpha and beta backgrounds.

9. Procedure

9.1. Air filters.

9.1.1. Directly mount air filters, without chemical processing, face up on planchets.

9.2. Soils.

9.2.1. Place 2 g of dried soil into a 50-mL beaker.

9.2.2. Add 10 mL of concentrated HNO_3 and 5-10 drops of concentrated HCl to the beaker. Swirl carefully and allow to leach overnight at room temperature.

9.2.3. Heat the sample until the yellow-orange NO_2 fumes no longer appear.

9.2.4. Transfer the sample to a 40-mL glass centrifuge tube. Rinse the beaker with 5 to 10 mL of 7.2 M HNO_3 and add the rinse to the centrifuge tube. Centrifuge for 5 min.

9.2.5. Write the sample number on the bottom of a planchet and weigh to the nearest mg. Record the sample number and planchet weight in a Laboratory notebook.

9.2.6. Carefully decant the acid mixture from the centrifuge tube to the preweighed planchet.

9.2.7. Rinse the centrifuge tube with approximately 5 mL of 7.2 M HNO_3 and decant the wash to the planchet.

9.2.8. Evaporate the solution on the planchet to dryness under a heat lamp. Control the heating rate so that the sample does not spatter.

9.2.9. Allow the planchet to cool, reweigh, and record the weight.

9.2.10. Cover the sample tightly with mylar film.

9.3. Sewage or sludge.

9.3.1. Weigh a 600-mL glass beaker and record the weight in a Laboratory notebook.

9.3.2. Pour approximately 100 mL of sample into the beaker, reweigh, and record the weight.

Wet weight = (beaker + sample) - beaker

9.3.3. Evaporate the sample to dryness. Reweigh and record the weight.

Dry weight = (beaker + sample) - beaker

9.3.4. Heat the sample on a hot plate and treat with concentrated HNO_3 and H_2O_2 until all organic material has been oxidized.

NOTE: The sample will not have a black tarry consistency, but may still have a grayish or reddish appearance.

9.3.5. Add approximately 20 mL of concentrated HNO_3 and 10 mL of HCl and boil until all HCl has been driven off (the NO_2 fumes will no longer appear).

9.3.6. Continue to heat until approximately 3 mL of solution remains.

9.3.7. Add 10-15 mL of 7.2 M HNO_3 to the sample.

9.3.8. Pour the sample into a 50-mL glass centrifuge tube. Rinse the beaker with 3-5 mL of 7.2 M HNO_3 and add the rinse to the centrifuge tube. Centrifuge for 5 min.

9.3.9. Proceed with Steps 9.2.5 through 9.2.10.

9.4. Water.

9.4.1. Place 900 mL of water and 5 mL of concentrated HNO_3 into a 1-L glass beaker. Evaporate the solution to dryness overnight on a hot plate, medium setting.

9.4.2. Dissolve the residue in approximately 10 mL of concentrated HNO_3. Wash down the sides of the beaker with HNO_3 and 6 drops of HF.

CAUTION: HF is extremely corrosive.

The amount of HNO_3 used is not as critical, but the sides of the beaker must be washed down thoroughly.

9.4.3. Boil the sample to dryness to volatilize any silica.

9.4.4. Dissolve the residue in approximately 10 mL of concentrated HNO_3 and treat the solution with 30% H_2O_2 until all organic material is destroyed

and the sample residue is white or pale yellow and dry. Carefully add H_2O_2 to avoid sample spatter. Usually 1-2 mL of H_2O_2 are required.

9.4.5. Add 10-15 mL of concentrated HNO_3 and 5-10 mL of HCl to the sample. Heat until the evolution of NO_2 ceases and the sample is clear or pale yellow. Continue heating until 2-3 mL remain.

NOTE: Do not let the sample go to dryness.

9.4.6. Dissolve the residue in approximately 10 mL of 7.2 M HNO_3, washing down the sides of the beaker.

9.4.7. Proceed with Steps 9.2.5 through 9.2.10.

9.5. Loading the proportional counter.

9.5.1. Place the plated standards above program indicator A in the sample holding rack.

9.5.2. Place the quality assurance samples above program indicator B, along with the two blank planchets.

9.5.3. Place the samples into planchet holders and record their position on the alpha-beta sample sheet (Fig. 1), using program indicator C for the sample set.

9.5.4. Place the aluminum block on top of the sample stack.

10. Operation of Instrument

10.1. Turn the key switch on the front panel of the LB-5100 proportional counter from **RUN** to **INITIALIZE**, then back to **RUN**. This initializes the microprocessor and queries the operator for the proper information concerning the sample set.

10.2. Check that detector gas flow is 0.15 square cubic feet per hour (SCFH) and that the high voltage is turned to the proper setting (1323 V).

NOTE: Flows higher than 0.25 SCFH can damage the counter.

10.3. Answer the questions pertinent to the sample set (Fig. 2) and the counter will automatically begin the counting sequence. Consult the count room manager if problems arise.

10.4. Refer to the operating manual (Source Material 12.1) for further information on instrument use.

10.5. When data collection is complete, staple all hard copy output (Fig. 3) to the alpha-beta sample sheet for use in data reduction.

11. Calculations

11.1. Use the following formulas to determine detector efficiencies for soil and water (these equation lines were generated from the absolute efficiency versus sample mass residue curves):

for residual mass (M) < 0.2, use

$$Ea = 0.085 \times e^{-4.9 \times M}$$
$$Eb = 0.36 \times e^{-1.5 \times M} \quad ;$$

for residual mass (M) > 0.2, use

$$Ea = (0.08 \times e^{-5.0 \times M}) + 0.01$$
$$Eb = (-0.0695 \times M) + 0.268 \quad ;$$

where Ea = alpha efficiency,
Eb = beta efficiency, and
M = residual mass (g).

11.2. Use the following efficiency factors for air filter samples.

Ea = 0.25
Eb = 0.44

Since sample mass residue is assumed to be constant, the efficiency values shown are a result of standard efficiency calculations (cpm/dpm) using a NIST standard.

11.3. Alpha activities are calculated using the following equation:

$$Aia = \frac{Ac - Ab - Ax}{Ea \times Q \times 2.22} \quad ,$$

where Aia = alpha activity (pCi),
Ac = alpha activity from the LB-5100 output (cpm),
Ab = alpha background (cpm),
Ax = crosstalk (see below for calculation),

Ea = alpha efficiency,
Q = sample quantity (g or L), and
2.22 = factor to convert dpm to pCi.

Ax is derived using the following equation:

$$Ax = Xba \times \left(\frac{Bt}{T} - Bb\right) ,$$

where Ax = crosstalk,
Xba = beta to alpha crosstalk,
Bt = total beta counts,
T = time of count (min), and
Bb = beta background (cpm).

11.4. Beta activities are calculated using the following equation:

$$Aib = \frac{(Bc - Bb - Bx)}{Eb \times Q \times 2.22} ,$$

where Aib = beta activity (pCi),
Bc = beta activity from the LB-5100 output (cpm),
Bb = beta background (cpm),
Bx = crosstalk (see below for calculation),
Eb = beta efficiency,
Q = sample quantity (g or L), and
2.22 = factor to convert dpm to pCi.

Bx is derived using the following equation:

$$Bx = Xba \times \left(\frac{At}{T} - Ab\right) ,$$

where Bx = crosstalk,
Xba = alpha to beta crosstalk,
At = total alpha counts,
T = time of count (min), and
Ab = alpha background (cpm).

11.5. Uncertainties are generated using the following formula:

$$Sax = |Aia| \sqrt{((T_1/T_2) + T_3)\ (T_3 + SQUAN)\ (SQUAN)}\ ,$$

where Sax = standard deviation of the alpha activity,
Aia = alpha activity (pCi),
T1 = (Ac + Ab + Ax) T (where Ac, Ab, Ax, and T are defined as in Step 11.3).
T2 = Ac - Ab - Ax (where Ac, Ab, and Ax are defined as in Step 11.3).
T3 = Ast/Ea (where Ast equals the standard deviation of Ea, the alpha efficiency), and
SQUAN = standard deviation of the sample quantity.

For the calculation of beta uncertainties, replace alpha factors with beta factors in the above expression.

12. Source Materials

12.1. Instruction Manual for LB-5100, Rev. 4.2, Tennelec, Inc., Oak Ridge, Tennessee (1978).

12.2. Environmental Protection Agency, "Tentative Reference Method for the Measurement of Gross Alpha and Gross Beta Radioactivities in Environmental Waters," Quality Assurance Branch, Technical Support Laboratory, National Environmental Research Center, Las Vegas, Nevada, ROAP Number 22 ACW, EPA-680/4-75-005 (1975).

12.3. *EML Procedures Manual*, 26th ed., H. L. Volchok and G. de Planque, Eds. (Environmental Measurements Laboratory, US Department of Energy, New York, New York, HASL-300, 1986).

Revision or additions to the procedure are marked (▒▒▒▒). Where a section heading is marked, the entire section has been revised.

ALPHA-BETA COUNTING SEQUENCE

COUNT STARTED: _____ SAMPLE TYPE: _____ COUNT LENGTH (min): _____

SAMP. NO.	REQUEST NUMBER	OWN	VOL/MASS QUANT.	UNITS	SAMP. NO.	REQUEST NUMBER	OWN	VOL/MASS QUANT.	UNITS
1					20				
2					21				
3					22				
4					23				
5					24				
6					25				
7					26				
8					27				
9					28				
10					29				
11					30				
12					31				
13					32				
14					33				
15					34				
16					35				
17					36				
18					37				
19					38				

Fig. 1. Alpha-beta sample sheet.

TENNELEC LB5100 REV. 4.0 - POWER ON
MODE
1-SINGLE SAMPLE
2-SINGLE CYCLE
3-SINGLE CYCLE AND RESTACK
4-MULTI-CYCLE
WHICH MODE DO YOU WANT (1,2,3,4)? 3 (3 = Single Cycle and Restack).

DO YOU WANT TO AVERAGE (Y/N)? N (Y will average 2 or more counts).

GROUP A (Group "A" is first group in initialization).

PLEASE ENTER SAMPLE ID (UP TO
12 CHARACTERS) AIR SAMPLE

ENTER TIME PS(XXXXX.XX MIN) 100	Requests Time Preset in hundreths of min. User enters a Time Preset of 1.00 min (max = 99,999.9).
ENTER A PS (CNTS) 999999	Requests Channel A Preset in Counts. User enters 999,999 (max = 9,999,999).
ENTER B PS(CNTS) 0	Requests Channel B Preset in Counts. User enters 0 which means system will not preset on B counts (max = 9,999,999).
ENTER BKG A(XXXXX.XX CPM) 14	Requests alpha bkg cpm be subtracted from gross alpha cpm. User input represents 0.14 cpm bkg subtract (default "0" indicates no subtract).
ENTER BKG B(XXXXX.XX CPM) 95X 94	Requests beta bkg cpm be subtracted from gross beta cpm. User enters wrong value, abort by entering "X" after value. User inputs 0.94 cpm bkg subtract.
ENTER Ka(XXXX.XXX) 333	Requests denominator for A/Ka column. User enters .333 (max = 9,999.999).
ENTER Kb(XXXX.XXX) 550	Requests denominator for B/Kb column. User enters .550 (max = 9,999.999).
ARE ALL THE GROUP INPUTS CORRECT (Y/N)? Y	If "N", the program again asks the user for the correct information.
DO YOU WANT TO DEFINE ANOTHER GROUP (Y/N)? N	If "Y", the program asks for the new group and the above question/answer format is repeated.

When the above question is answered N, the system will print a summary of the groups' parameters, as shown below.

#	IDENTIFICATION	TIME	PST A	PST B	BKG A	BKG B	Ka	Kb
A	AIR SAMPLE	1.00	999999	0	0.14	0.94	0.333	0.550
B		0.00	0	0	0.00	0.00	0.000	0.000
C		0.00	0	0	0.00	0.00	0.000	0.000

Fig. 2. Tennelec LB-5100 user interface session.

#	IDENTIFICATION	TIME	PST A	PST B	BKG A	BKG B	KA	KB
A	STD/S	10.00	0	0	0.00	0.00	0.000	0.000
B	QA/S + SAMPL	100.00	0	0	0.00	0.00	0.000	0.000
C		0.00	0	0	0.00	0.00	0.000	0.000
D		0.00	0	0	0.00	0.00	0.000	0.000
E		0.00	0	0	0.00	0.00	0.000	0.000

TUE MAR 6,1990
GROUP A STD/S

SN	TIME	COUNTS A	COUNTS B	CPM A	CPM B	A/0000.000	B/0000.000	2sig A
B	HR:MIN:SEC							
1	10.00	881	85	88.10	8.50			6.73%
%	08:48:04							
2	10.00	10	45125	1.00	4512.50			63.24%
%	08:58:17							

TUE MAR 6,1990
GROUP B QA/S + SAMPL

SN	TIME	COUNTS A	COUNTS B	CPM A	CPM B	A/0000.000	B/0000.000	2sig A
B	HR:MIN:SEC							
3	100.00	39	195	0.39	1.95			32.02%
%	10:38:41							
4	100.00	144	824	1.44	8.24			16.66%
%	12:18:57							
5	100.00	156	798	1.56	7.98			16.01%
%	13:59:12							
6	100.00	140	800	1.40	8.00			16.90%
%	15:39:28							
7	100.00	77	967	0.77	9.67			22.79%
%	17:19:43							
8	100.00	18	424	0.18	4.24			47.14%
%	18:59:59							
9	100.00	41	305	0.41	3.05			31.23%
%	20:40:14							
10	100.00	19	374	0.19	3.74			45.88%
%	22:20:30							
11	100.00	18	779	0.18	7.79			47.14%
%	00:00:45							

#	IDENTIFICATION	TIME	PST A	PST B	BKG A	BKG B	KA	KB
A	STD/S	10.00	0	0	0.00	0.00	0.000	0.000
B	QA/S + SAMPL	100.00	0	0	0.00	0.00	0.000	0.000
C		0.00	0	0	0.00	0.00	0.000	0.000
D		0.00	0	0	0.00	0.00	0.000	0.000
E		0.00	0	0	0.00	0.00	0.000	0.000

Fig. 3. Tennelec LB-5100 data output sheet.

GROSS GAMMA IN ENVIRONMENTAL MATRICES

Analyte: All gamma-ray activities	**Method No.:** ER150
Matrix: Soil, biologicals, and water	**Minimum Detectable Activity:** Water 600 pCi/L Soil 3 pCi/g
Procedure: Instrumental gamma-ray NaI counting	**Accuracy and Precision:** Water 108% ± 53% Soil 104% ± 3%
Effective Date: 11/24/87	**Authors:** Ernest S. Gladney George H. Brooks

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 7. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment.

1. Principle of Method

1.1. Samples are packaged in 500-mL (16-oz Nalgene) plastic bottles and are counted in a NaI[Tl] well-type scintillation detector for 5 min without chemical processing.

1.2. The sample size is 500 mL for liquids and 100 g for solids.

2. Sensitivity

2.1. The limits of detection are 600 pCi/L for aqueous solutions and 3 pCi/g for solids at the 661-keV ^{137}Cs line.

3. Accuracy and Precision

3.1. This method is intended as a qualitative assessment of gross gamma activity, but the accuracy and precision have been assessed using ^{137}Cs standards. For 0.5-L water samples the accuracy at 1400 pCi/L is 108% ± 53%. For 100-g soil samples at 220 pCi/g the accuracy is 104% ± 3%.

3.2. Samples showing activity levels significantly above background may be subjected to further analysis to determine the exact source of the activity.

4. Interferences

4.1. Although the method is intended to measure gamma-emitting isotopes, the wall thickness of the detector is such that high-energy beta particles from the sample will also be counted.

5. Collection and Storage of Samples

5.1. There are no special collection and storage requirements.

6. Apparatus

6.1. Gamma counter: Model No. 550 EG&G Ortec, equipped with a 4-in. diam × 6-in. NaI[Tl] well-type scintillation detector, with a 3-in. diam × 5.5-in. well and a single-channel analyzer. The system window is adjusted to observe 0- to 2-MeV gamma rays.

6.2. Amplifier: Model No. 572, EG&G Ortec.

6.3. Timer/counter: Model No. 871, EG&G Ortec.

6.4. Bias supply: Model No. 459 EG&G Ortec.

6.5. Bottles: 500-mL, polyethylene, Nalgene, catalog no. 20002-0016.

6.6. Analytical balance: 300-g minimum capacity.

6.7. Bags: Low-density polyethylene (LDPE) clear bags (0.002-in.-thick) sized to fit 500-mL bottles. The bags protect the detector from surface contamination on the bottles.

6.8. Funnel: polyethylene.

7. Reagents

7.1. No reagents are required.

8. Calibration and Standards

8.1. Prepare all standards from National Bureau of Standards (NBS) Standard Reference Material (SRM) 4233-B, ^{137}Cs Burn-up standard.

8.1.1. Prepare the liquid standard by diluting an aliquot of the stock solution to 500 mL with distilled water. Place the standard in a 500-mL polyethylene bottle.

8.1.2. Prepare the soil standard by evaporating an aliquot of the stock solution on 100 g of blank soil under an infrared lamp. The solution must not contact the walls or bottom of the container. Thoroughly blend the dry standard with a stainless steel spatula and transfer it to a polyethylene bottle with the aid of a polyethylene powder funnel and a small brush.

8.2. Count an appropriate quality control material with each sample set to record detector system performance.

8.3. The liquid blank is 500 mL of distilled water and the soil blank is 100 g of washed deep-well sand.

8.3.1. Count the appropriate blank with each sample set. Use the data to calculate the background count of the counter.

9. Procedure

9.1. Soils.

9.1.1. Weigh 100 g of a dried soil sample into a polyethylene bottle.

9.1.2. Tightly cap the bottle and clean off any exterior contamination.

9.1.3. Place the sample into a plastic bag and insert it into the NaI[Tl] detector well.

9.1.4. Count the sample for 5 min.

9.1.5. Record the total counts and count time in minutes on the gross gamma analysis sheet accompanying the sample from the radiochemistry laboratory.

9.1.6. Count a soil quality control sample, a soil blank, and a soil standard for 5 min each. Record the counts of the quality control sample, blank sample, and soil standard on the gross gamma analysis sheet (Fig. 1).

9.2. Water.

9.2.1. Place 500 mL of a filtered water sample into a polyethylene bottle.

9.2.2. Proceed with Steps 9.1.2 through 9.1.5.

9.2.3. Count a water quality control sample, a water blank, and a water standard for 5 min each. Record the counts of the quality control sample, the blank sample, and the water standard on the gross gamma analysis sheet (Fig. 1).

10. Operation of Equipment

10.1. Check the EG&G Ortec Model No. 871 Timer/Counter parameters before each counting session. The timer display should read **5.0**, and the **Time Base** select light should read **Min**. Push the appropriate switches to modify the parameters to the correct setting. The counter display should read **Counter**. Use the Counter/Timer select switch to adjust to the correct setting.

10.2. The EG&G Ortec Model No. 459 Bias Supply should be set at 1000 V. If the bias supply is not at the correct setting, adjust the potentiometer to provide the correct voltage.

NOTE: If the voltage is incorrectly set, adjust to the correct voltage with increments no larger than 100 V/s.

11. Calculations

11.1. Calculations of radiometric counting data for determinations are made without a radiotracer. Most data values are assumed to have Poisson distributions and all time-measurement errors are assumed to be insignificant.

11.2. Counting efficiency.

11.2.1. The counting efficiency of the counter for a single standard run is calculated using the following equation:

$$E = \frac{\left(\frac{C_s}{T_s} - \overline{B}\right)}{A_s} ,$$

where E = efficiency (counts per disintegration),
C_s = gross counts for the standards,
T_s = counting time for standard (min),
$\overline{B}$ = average background count rate, and
A_s = activity of the standard (dpm).

11.2.2. The average counter efficiency is calculated from a running average of the last n values of individual E determinations using the following equation:

$$\overline{E} = \frac{\sum_{i=1}^{n} E_i}{n} ,$$

where $\overline{E}$ = the running average of the counting efficiency,
E_i = the individual counting efficiencies from Step 11.2.1, and
n = the number of measurements used for calculation.

11.2.3. The standard deviation of $\overline{E}$ is calculated as follows:

$$SD(\overline{E}) = \overline{E} \times \sqrt{\frac{\sum_{i=1}^{n} (E_i - \overline{E})^2}{(n-1)\, n\, \overline{E}^2} + \frac{(\sigma_{A_s})^2}{A_s^2}} ,$$

where $SD(\overline{E})$ = standard deviation of the average efficiency,
$\overline{E}$ = average efficiency (counts per disintegration) from Step 11.2.2,
E_i = individual efficiencies from Step 11.2.1,
n = number of values of E used,
A_s = activity of the standard (dpm), and
σA_s = stated standard deviation associated with the known value of A_s.

11.2.4. The standard deviation of an individual E is calculated as follows:

$$SD(E) = E \times \sqrt{\frac{\frac{C_s}{T_s^2} + \frac{C_b}{T_b^2}}{\left(\frac{C_s}{T_s} - \frac{C_b}{T_b}\right)^2} + \frac{(\sigma_{A_s})^2}{A_s^2}} ,$$

where $SD(E)$ = standard deviation of E (counts per disintegration),
E = counting efficiency from Step 11.2.1,
C_s = gross counts for the standard,
T_s = counting time for the standard,
C_b = gross counts for the background,
T_b = counting time for the background (min),
A_s = activity of the standard (dpm), and
σA_s = standard deviation of standard activity.

11.3. Background.

11.3.1. The background count rate is calculated as follows:

$$B = \frac{C_b}{T_b} ,$$

where B = background count rate (cpm),
C_b = gross counts of background, and
T_b = counting time of the background (min).

11.3.2. A running average of the background count rate is calculated using the following equation:

$$\overline{B} = \frac{\sum_{i=1}^{n} B}{n} ,$$

where $\overline{B}$ = average background count rate,
B = background count rate, and
n = number of values used.

11.3.3. The standard deviation of the average background count rate is calculated using the following equation:

$$SD(\overline{B}) = \overline{B} \times \sqrt{\frac{\sum_{i=1}^{n} (B - \overline{B})^2}{(n-1) \times n \times \overline{B}^2}} ,$$

where $SD(\overline{B})$ = standard deviation of the running average of the background,
$\overline{B}$ = average background from Step 11.3.2,
B = individual background count rate, and
n = number of values used.

11.4. Sample activity.

11.4.1. The activity in the samples is calculated as follows:

$$A_x = \frac{\left(\frac{C_x}{T_x} - \overline{B}\right)}{\overline{E}} ,$$

where A_x = sample activity (dpm),
C_x = gross counts for the sample,
T_x = counting time for the sample (min),
$\overline{B}$ = average background count rate (cpm), and
$\overline{E}$ = average counting efficiency.

11.4.2. The standard deviation of the activity in the sample is calculated as follows:

$$SD\ (A_x) = |A_x| \times \sqrt{\frac{\frac{C_x}{T_x^2}}{\left(\frac{C_x}{T_x} - \overline{B}\right)^2}} ,$$

where $SD\ (A_x)$ = standard deviation of sample activity (dpm),
$|A_x|$ = absolute value of A_x from Step 11.4.1,
C_x = gross counts of sample,
T_x = sample counting time, and
$\overline{B}$ = average background count rate.

11.4.3. The minimum detectable activity for high-background instruments is determined to be three times the standard deviation of the background.

$$MDA = \frac{3 \times SD\ (B)}{K} ,$$

where MDA = minimum detectable activity (dpm),
SD (B) = standard deviation of the background count rate (cpm), and
K = constant in sample units.

11.5. Calculation to put the radiometric measurement into the required form for final reporting.

11.5.1. The reported activity is calculated as follows:

$$R_x = \frac{A_x}{K \times Q} ,$$

where R_x = sample activity in correct units for reporting (pCi/L),
A_x = sample activity (dpm),
K = conversion factor (2.22 pCi/dpm), and
Q = sample quantity analyzed (0.5 L or 100 g).

11.5.2. The propagated uncertainty of the reported activity is calculated using the following equation:

$$U_x = |R_x| \times \sqrt{SD\,(A_x)^2 + SD\,(\overline{B})^2 + SD\,(\overline{E})^2 + \frac{SD\,(Q)^2}{Q^2}} ,$$

where U_x = propagated uncertainty of the reported activity,
$|R_x|$ = absolute value of the reported activity,
$SD(A_x)$ = standard deviation of sample activity,
$SD(\overline{B})$ = standard deviation of the average background,
$SD(\overline{E})$ = standard deviation of the average efficiency,
$SD(Q)$ = standard deviation of the sample quantity aliquoted, and
Q = sample quantity aliquoted in reporting units.

12. Source Materials

12.1. M. A. Gautier, E. S. Gladney, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1985," Los Alamos National Laboratory report LA-10813-MS (1986).

12.2. "American National Standard for Performance Criteria for Radiobioassay," draft, American National Standards Institute, ANSI N13.30 (1989).

12.3. L. A. Currie, "Limits for Qualitative Detection and Quantitative Determination," *Analytical Chemistry* **40**, 586-593 (1968).

ANALYSIS _____ ANALYST _____ DATE _____

SAMPLE NO.	REQUEST NO.	OWNER	WEIGHT OR VOLUME	RESIDUE WEIGHT	SAMPLE TYPE	GROSS COUNTS

Fig. 1. Gross gamma analysis data sheet.