U.S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 4350B

Environmental Radioactivity

Source description	River Sediment
Source identification	4350B
Reference time	September 9,1981

General Comments[1]*

This Standard Reference Material (SRM), which has been developed in cooperation w member laboratories of the International Committee for Radionuclide Metrology, co sists of approximately 85 grams of freeze-dried, pulverized river sediment[2] in polyethylene bottle. The SRM is intended for use in tests of measurements of environmental radioactivity contained in matrices similar to the sample[3].

Working samples of this SRM should be dried in air at 40°C for at least 24 hours prior to weighing.

The material has been tested to a minimum sample size of 5 grams, for which it ha been found to be homogeneous[4].

The $^{239}Pu+^{240}Pu$ are recoverable by normal HNO_3 or HNO_3-HCl leaching procedures as well as by the more vigorous methods of chemical treatment listed below.

Random and systematic uncertainties have been combined in quadrature at a level corresponding to a standard deviation of the mean; the stated overall uncertainti are 3 times this value and are roughly at the 99-percent confidence level.

When additional data become available, it is expected that other radioactivity concentrations will be certified and purchasers will be notified. To aid in thes certifications, users are requested to send their measurement results for uncerti radioactivities together with the methods used to NBS[1].

* See notes

Radionuclide	Activity Concentration (Bq g^{-1} (5))	Total Uncertainty (Percent (6))	Half-life (years)	Method Code (7)
^{60}Co	4.64×10^{-3}	5.0	5.28	1a, 1f, 5a
^{137}Cs	2.90×10^{-2}	6.3	30.7	1b, 1f, 3a, 4f, 5a
^{152}Eu	3.05×10^{-2}	4.0	13.54	3a, 5a
^{154}Eu	3.78×10^{-3}	15	8.48	3a, 5a
^{226}Ra	3.58×10^{-2}	10		2d
^{238}Pu	1.3×10^{-5}	17		1c, 2c, 3c
$^{239}Pu+^{240}Pu$	5.08×10^{-4}	5.8		1c, 2c, 3c
^{241}Am	1.5×10^{-4}	21		1c, 2c, 3c

Mass spectrometry data (8)

This Standard Reference Material was prepared in the Center for Radiation Research, Nuclear Radiation Division, Radioactivity Group, D.D. Hoppes, Group Leader.

Washington, D.C. 20234
September 9,1981

George A. Uriano, Chief
Office of Standard Reference Materials

Notes

(1) For further information call K.G.W. Inn (301) 921-2383 or J.M.R. Hutchinson (301) 921-2396, National Bureau of Standards, Room C114, Building 245, Washington, D.C., 20234.

(2) The soil was pulverized with a "pancake" style air jet mill. The average particle diameter for the resulting powder is 8 μm. More than 99 percent, by weight, of the particles are less than 20 μm in diameter.

(3) See attached sheets: Semi-quantitative emission spectrographic analysis and Gamma-ray spectrum for SRM 4350B.

(4) Summary of homogeneity measurements

A. Ten 100g bottled samples were examined for homogeneities in their gamma-ray-emission rates by counting them in a 5-in NaI(Tl) well detector coupled to a multichannel analyzer. The count rates from each bottle were compared over each of thirteen selected energy regions and also over the total gamma-ray spectrum (0.04-1.85 MeV). The net sample-to-sample inhomogeneities from gamma-ray-emission rates are summarized below:

Energy Region (keV)	Standard deviation of the mean (%)
47 - 107	1.27
109 - 157	1.81
159 - 203	3.15
204 - 271	1.23
273 - 315	2.04
317 - 423	1.51
425 - 540	1.08
542 - 752	0.98
754 - 848	0.94
850 - 1030	0.74
1032 - 1300	0.81
1302 - 1662	0.64
1663 - 1845	3.55
47 - 1845	0.47

B. Inhomogeneities of ^{90}Sr and ^{137}Cs are less than 2 percent for 10g samples.

C. Inhomogeneities of alpha-particle emitting radionuclides, are less than 3 percent.

(5) Certified values are those measured by two or more methods and/or two or more laboratories.

(6) The random and systematic errors have been combined in quadrature at a level corresponding to a standard deviation of the mean; the stated overall uncertainty is 3 times this value.

(7) Analytical Methods (References in parentheses)

1. $HF-HNO_3$ or $HF-HNO_3-HClO_4$ dissolution
2. KF-pyrosulfate fusion (BPH80, MAR79, SHA79)
3. HCl, HNO_3 or $HCl-HNO_3$ leaching (HAR80, LMB75, WNB70)
4. HCl-NaOH leaching (HAR80)
5. Non-destructive analysis
   a. Gamma-ray spectrometry with Ge(Li) detector
   b. Beta-particle counting with thin-window Geiger counter
   c. Alpha-particle spectrometry with surface-barrier detector
   d. Radon emanation counting
   e. Beta-particle scintillation counting with plastic phosphor
   f. X-ray photons or beta-particle counting with gas-flow proportional counter

(8)

Nuclide	Atom Percent	
^{239}Pu	89.91	Analysis at
^{240}Pu	9.43	Savannah River
^{241}Pu	0.318	(Dr. J. Halverson)
^{242}Pu	0.336	

PARTICIPATING IN THE ASSAYS

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STATISTICIAN

Dr. W.S. Liggett Jr., National Bureau of Standards

UNCERTIFIED VALUES

The following activities are uncertified because there are no corroborative measurements with which to compare them.

Radionuclide	Activity Concentration ($Bq\ g^{-1}$)	Laboratory	Method Code
^{40}K	5.6×10^{-1}	RESL	5a
^{55}Fe	1.7×10^{-2}	WHOI	3f
^{90}Sr	5.3×10^{-3}	EML, RESL, WHOI	1b, 2f, 3b, 4e
^{228}Th	3.35×10^{-2}	RESL	2c
^{230}Th	2.95×10^{-2}	RESL	2c
^{232}Th	3.32×10^{-2}	RESL	2c
^{234}U	3.32×10^{-2}	RESL	2c
^{235}U	1.7×10^{-3}	RESL	2c
^{238}U	3.08×10^{-2}	RESL	2c

REFERENCES

BPH 80 R.P. Bernabee, D.R. Percival and F.D. Hindman, Liquid-liquid extraction separation and determination of plutonium and americium, Analytical Chemistry, 52 (14), 2351 (1980).

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LMB 75 H.D. Livingston, D.R. Mann and V.T. Bowen, Analytical procedures for transuranic elements in seawater and marine sediments, Analytical Methods in Oceanography, Advances in Chemistry Series No. 147, T.R.P. Gibb, Jr., ed., American Chemical Society, New York, 124 (1975).

MAR 79 D.B. Martin, Determination of strontum-89 and -90 in soil with total sample decomposition, Analytical Chemistry, 51 (12), 1968 (1979).

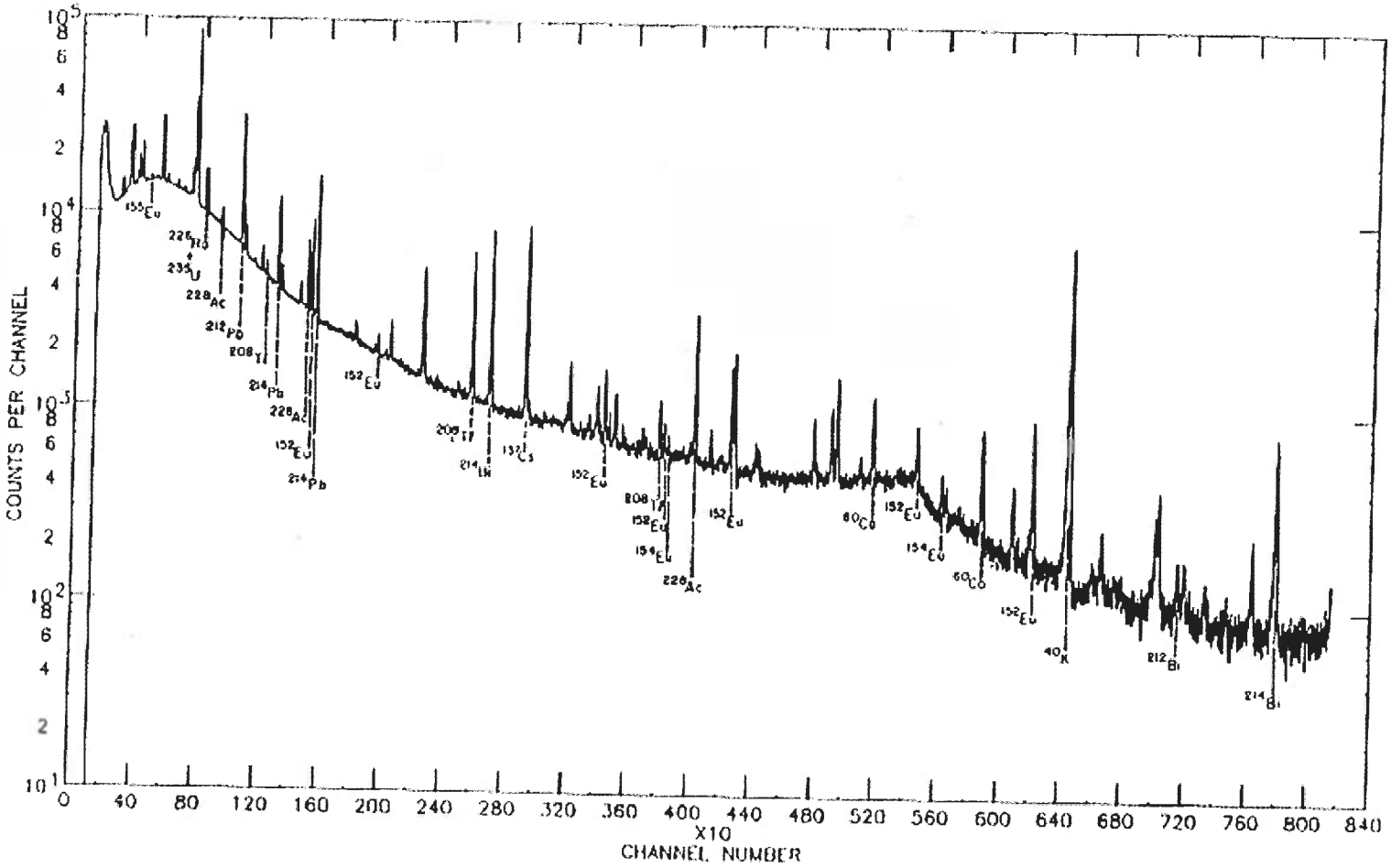
SHA 79 C.W. Sill, F.D. Hindman and J.I. Anderson, Simultaneous determination of alpha-emitting nuclides of radium through californium in large environmental and biological samples, Analytical Chemistry, 51 (8), 1307 (1979).

WNB 70 K.M. Wong, V.E. Noshkin and V.T. Bowen, Radiochemical procedures for the analysis of stontium, antimony, rare earths, caesium, and plutonium in seawater samples, Reference Methods for Marine Radiochemistry Studies, International Atomic Energy Agency Technical Report Series No. 118, International Atomic Energy Agency, Vienna, 119 (1970).

SEMI-QUANTITATIVE EMISSION SPECTROGRAPHIC ANALYSIS FOR SRM-4350B

Si	%	> 34	Gd	ppm	< 15	Sr	ppm	583
Al	%	8.9	Ge	ppm	< 1.5	Ta	ppm	< 460
Fe	%	6.8	Hf	ppm	< 15	Tb	ppm	< 32
Mg	%	1.6	Ho	ppm	< 6.8	Th	ppm	< 22
Ca	%	2.5	In	ppm	< 6.8	Tl	ppm	< 4.6
Na	%	2.2	Ir	ppm	< 15	Tm	ppm	< 4.6
K	%	> 1.5	La	ppm	57	U	ppm	< 320
Ti	%	0.60	Li	ppm	< 68	V	ppm	160
P	%	< 0.068	Lu	ppm	< 15	W	ppm	< 10
Mn	%	0.066	Mn	ppm	660	Y	ppm	33
Ag	ppm	< 0.10	Mo	ppm	< 1.0	Yb	ppm	3.5
As	ppm	< 150	Nb	ppm	29	Zn	ppm	203
Au	ppm	< 10	Nd	ppm	40	Zr	ppm	403
B	ppm	27	Ni	ppm	33			
Ba	ppm	703	Os	ppm	< 22	MAJOR COMPONENTS		
Be	ppm	2.5	Pb	ppm	42	RECALCULATED AS OXIDES		
Bi	ppm	< 10	Pd	ppm	< 1.0	SiO_2	%	> 73
Cd	ppm	< 34	Pr	ppm	< 68	Al_2O_3	%	17
Ce	ppm	90	Pt	ppm	< 4.6	Fe_2O_3	%	9.5
Co	ppm	25	Re	ppm	< 10	MgO	%	2.7
Cr	ppm	58	Rh	ppm	< 2.2	CaO	%	3.5
Cu	ppm	40	Ru	ppm	< 2.2	Na_2O	%	2.9
Dy	ppm	< 22	Sb	ppm	< 32	K_2O	%	> 1.8
Er	ppm	< 10	Sc	ppm	24	TiO_2	%	1.0
Eu	ppm	< 2.2	Sm	ppm	< 10	P_2O_5	%	< 0.16
Ga	ppm	16	Sn	ppm	< 1.8	MnO	%	0.085

The relative uncertainty for each reported concentration is plus 50%, and minus 33%.



Gamma-ray spectrum of SRM 4350B, with 60 cm^3 Ge(Li) detector. Background has not been subtracted and contributes typically 20 percent to the peaks for many natural radioelements.