U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 4353

Environmental Radioactivity

Source description	Rocky Flats Soil Number 1
Source identification	4353-
Reference time	December 15, 1980

General Comments(1)*

This Standard Reference Material (SRM), which has been developed in cooperation with member laboratories of the International Committee for Radionuclide Metrology, consists of approximately 90 grams of air-dried, pulverized soil(2) in a polyethylene bottle. The sample was collected from Rocky Flats, Colorado. This SRM is intended for use in tests of measurements of environmental radioactivity contained in matrices similar to the sample(3,4,5).

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 extensively for homogeneity and the results are summarized in(6). Based on over 70 plutonium measurements, the sample contains typically one or two "hot" particles in each bottle. As described in Note (5), by judicious handling of user data, the effect of the "hot" particles upon estimates of deduced user analytical error can be made negligible for most applications.

Concentrations and uncertainties, EXCLUDING HOT PARTICLES, are quoted in the following table.

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

* See notes

Radionuclide	Activity Concentration (Bq g^{-1} (7))	Total Uncertainty (Percent) (8)	Method Code Comments (9)
^{40}K	7.23×10^{-1}	9.6	5a
^{90}Sr	7.63×10^{-3}	10.2	1b, 2f, 3b, 4e
^{137}Cs	1.76×10^{-2}	4.5	1b, 1f, 3a, 4f, 5a
^{226}Ra	4.30×10^{-2}	6.6	2d
^{228}Ac	6.98×10^{-2}	5.1	5a
^{228}Th	7.08×10^{-2}	5.1	2c
^{230}Th	4.43×10^{-2}	5.1	2c
^{232}Th	6.93×10^{-2}	5.1	2c
^{234}U	3.91×10^{-2}	3.6	2c
^{238}U	3.89×10^{-2}	5.1	2c
^{238}Pu	1.66×10^{-4}	11.0	1c, 2c, 3c EXCLUDING
$^{239}Pu+^{240}Pu$	8.03×10^{-3}	7.5	1c, 2c, 3c HOT
^{241}Am	1.25×10^{-3}	7.3	1c, 2c, 3c PARTICLES

Mass spectrometry data (10)

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
April, 1981

George A. Uriano, Chief
Office of Standard Reference Materials

Notes

(1) For further information contact 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 size for the resulting powder is 8 μm. More than 99 percent, by weight, of the particles are less than 20 μm in diameter (VFI80).

(3) Semi-quantitative mineralogical composition by x-ray diffraction measurements (performed by Dr. H. Tourtelot, U.S. Geological Survey, Denver, CO):

Mineral	Percent by Weight
Quartz	55 - 60
Clays	25 - 30
Alkali Feldspars	5 - 10
Plagioclase	5

(4) See attached sheets: Semi-quantitative emission spectrographic analysis and Gamma-ray spectrum for SRM 4353.

(5) Suggested handling of data to obtain estimate of user analytical error for plutonium 239+240 measurements.

A. For each sample measurement, consider the difference between it and the certified $^{239+240}Pu$ concentration of 8.03×10^{-3} Bq g^{-1}. Classify each measurement according to whether the difference is too negative to be attributed to measurement error (outside the sum of the user and NBS uncertainties at the 3s estimated standard deviation level), too positive to be attributed to measurement error, or in between. The user uncertainty for a single measurement should be taken to be larger than 10 percent (1s), the uncertainty of the certifying laboratories. This 10 percent may reflect some inhomogeneity in the material.

B. If all sample measurements are in between take no action.

If any sample measurement is too low, the user's measurements process is suspect.

If no measurements are too low but k out of a total of n sample measurements are too high, calculate the probability of k or more being caused by hot particles and decide accordingly. The probability of at least one hot particle in a sample is given by $p = 1 - e^{-\alpha w}$, where $\alpha = 0.02$ with a 95 percent confidence interval of $0.004 < \alpha < 0.047$, and w is the sample weight in grams. Thus, the probability of k or more samples with hot particles is given by

$$\sum_{i=k}^{n} \frac{n!}{(n-i)!\, i!} p^i (1-p)^{n-i}$$

This probability has been calculated for a few typical examples and is given on the next page.

Samples Size(g)	Probability that k samples out of n contain hot particles									
	One Sample	Two Samples		Three Samples			Four Samples			
	n=1 k=1	n=2 k=1	n=2 k=2	n=3 k=1	n=3 k=2	n=3 =3	n=4 k=1	n=4 k=2	n=4 k=3	n=4 k=4
1	0.02	0.04	0.00	0.06	0.00	0.00	0.08	0.00	0.00	0.00
2	0.04	0.08	0.00	0.11	0.00	0.00	0.15	0.01	0.00	0.00
5	0.10	0.18	0.01	0.26	0.03	0.00	0.33	0.05	0.00	0.00
10	0.18	0.33	0.03	0.45	0.09	0.01	0.55	0.15	0.02	0.00
20	0.33	0.55	0.11	0.70	0.25	0.04	0.80	0.40	0.11	0.01

n = number of samples measured

k = number of samples with hot particle(s)

(6) Summary of homogeneity measurements

A. Fourteen 100g bottled samples were examined for inhomogeneities 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 twelve selected energy regions and also over the total gamma-ray spectrum (0.04-2.05 MeV). The net sample-to-sample inhomogeneities in the gamma-ray-emission rates are summarized below:

Energy Region (MeV)	Standard deviation of the mean (%)
0.04 - 0.11	0.70
0.11 - 0.16	1.81
0.16 - 0.19	0.57
0.19 - 0.27	0.59
0.27 - 0.31	2.21
0.31 - 0.45	1.37
0.45 - 0.79	0.76
0.79 - 1.03	0.73
1.03 - 1.28	1.06
1.28 - 1.62	0.58
1.62 - 1.95	1.05
1.95 - 2.05	4.08
0.04 - 2.05	0.35

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

C. Inhomogeneities of alpha-particle emitting radionuclides, excluding "hot" particles, are less than 3 percent.

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

(8) The random and systematic uncertainties have been combined in quadrature at a level corresponding to a standard deviation of the mean. The stated overall uncertainties are three times this value.

(9) 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)

continued next page

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

Approximately 8 percent of the contained $^{239}Pu+^{240}Pu$ resist HNO_3, HCl and HNO_3-HCl leaching.

(10) Mass spectrometry measurement

Nuclide	Atom Percent	
^{239}Pu	94.57	
^{240}Pu	5.23	Savannah River
^{241}Pu	0.178	(Dr. J. Halverson)
^{242}Pu	0.023	

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UNCERTIFIED VALUES

The following activities are uncertified because there are no corroborative measurements with which to compare them, or because, as is the case for $^{239}Pu+^{240}Pu$ the value has been measured directly by only one laboratory (EML). The quoted $^{239}Pu+^{240}Pu$ value, below, is deduced from measurements on smaller samples and agrees, within estimated uncertainties with the EML value. A 90 g sample contains an average of 1.8 hot particles, each with an average activity of about 0.04 Bq.

Radionuclide	Activity concentration (Bq g^{-1})	Laboratory	Method Code
^{55}Fe	2.49×10^{-3}	WHOI	3f
^{235}U	1.9×10^{-3}	RESL	2c
$^{239}Pu+^{240}Pu$ (total in a large sample)	8.8×10^{-3}	Refer to certification page	

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HAR 80 Environmental Measurements Laboratory Procedures Manual, HASL 300 with 8 supplements, J.H. Harley, ed., New York (1980).

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 strontium-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).

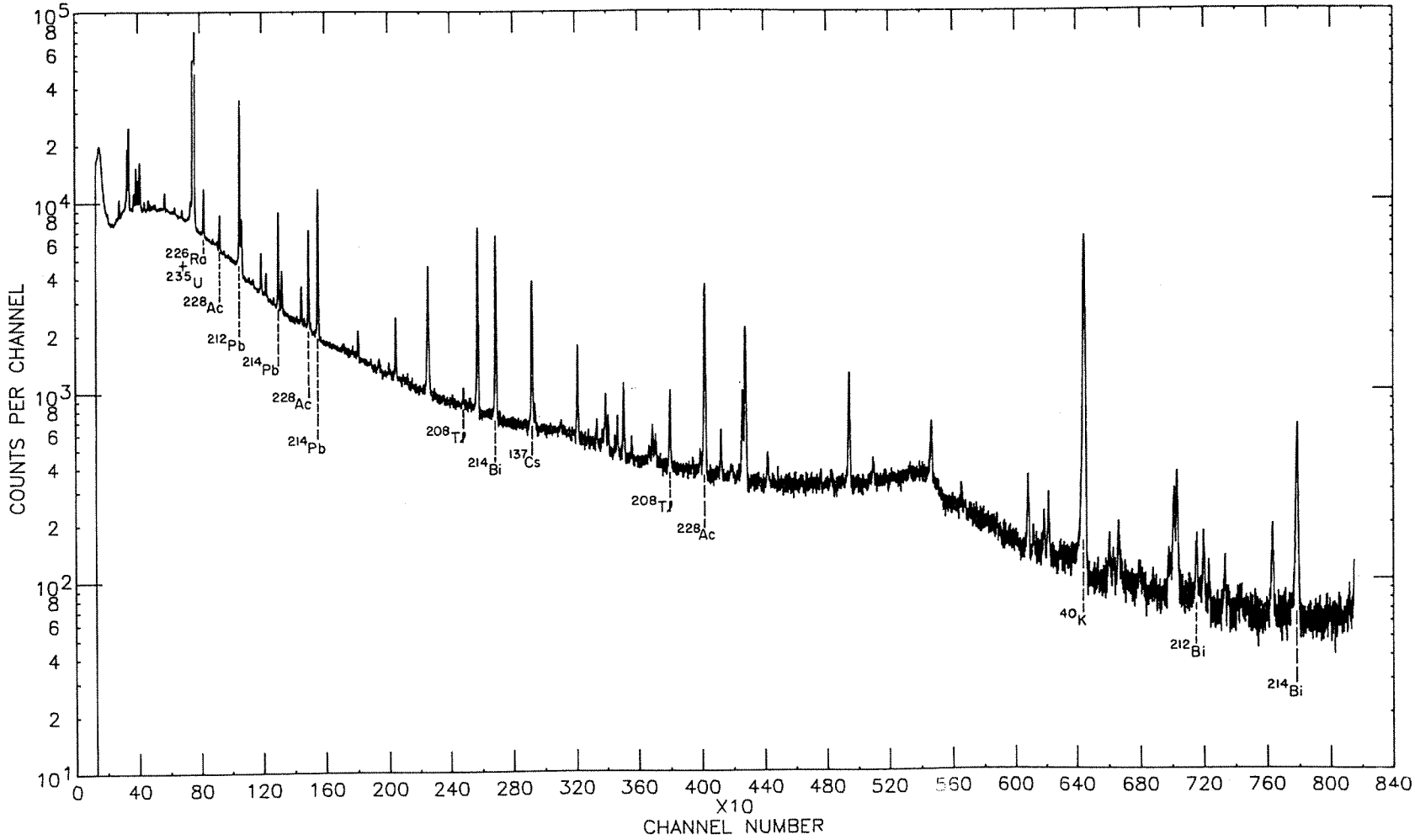
VFI 80 H.L. Volchok, M.S. Feiner, K.G.W. Inn and J.F. McInroy, Development of some natural matrix standards - progress report, Environmental International, 3, 395 (1980).

WNB 70 K.M. Wong, V.E. Noshkin and V.T. Bowen, Radiochemical procedures for the analysis of strontium, 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-4353

Si	%	> 34	Gd	ppm	< 15	Sr	ppm	220
Al	%	6.5	Ge	ppm	< 1.5	Ta	ppm	< 460
Fe	%	4.0	Hf	ppm	< 15	Tb	ppm	< 32
Mg	%	0.30	Ho	ppm	< 6.8	Th	ppm	< 22
Ca	%	0.65	In	ppm	< 6.8	Tl	ppm	< 4.6
Na	%	0.77	Ir	ppm	< 15	Tm	ppm	< 4.6
K	%	> 1.5	La	ppm	53	U	ppm	< 320
Ti	%	0.20	Li	ppm	< 68	V	ppm	68
P	%	0.088	Lu	ppm	< 15	W	ppm	< 10
Mn	%	0.055	Mn	ppm	550	Y	ppm	22
Ag	ppm	< 0.10	Mo	ppm	5.3	Yb	ppm	3.0
As	ppm	< 150	Nb	ppm	16	Zn	ppm	35
Au	ppm	< 10	Nd	ppm	74	Zr	ppm	330
B	ppm	22	Ni	ppm	290			
Ba	ppm	583	Os	ppm	< 22	MAJOR COMPONENTS		
Be	ppm	2.2	Pb	ppm	42	RECALCULATED AS OXIDES		
Bi	ppm	< 10	Pd	ppm	< 1.0	SiO_2	%	> 73
Cd	ppm	< 32	Pr	ppm	< 68	Al_2O_3	%	12
Ce	ppm	72	Pt	ppm	< 4.6	Fe_2O_3	%	5.7
Co	ppm	16	Re	ppm	< 10	MgO	%	0.49
Cr	ppm	533	Rh	ppm	< 2.2	CaO	%	0.92
Cu	ppm	33	Ru	ppm	< 2.2	Na_2O	%	1.1
Dy	ppm	< 22	Sb	ppm	< 32	K_2O	%	> 1.8
Er	ppm	< 10	Sc	ppm	8.8	TiO_2	%	0.33
Eu	ppm	< 2.2	Sm	ppm	< 10	P_2O_5	%	< 0.19
Ga	ppm	11	Sn	ppm	< 1.5	MnO	%	0.072

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



Gamma-ray spectum of SRM 4353, with 60 cm^3 Ge(Li) detector. Background has not been subtracted and contributes typically 20 percent to the peaks for many natural radioelements.