MASTER

AMCHITKA BIOENVIRONMENTAL PROGRAM

ANNUAL PROGRESS REPORT

RADIONUCLIDE AND STABLE ELEMENT ANALYSES
OF ENVIRONMENTAL SAMPLES FROM
AMCHITKA ISLAND, ALASKA

by

J. R. Vogt, J. E. Howes, and R. A. Ewing

November, 1968

Prepared for the U. S. Atomic Energy
Commission under Contract No. AT(26-1)-171

~~NOTICE: This document contains information of a
preliminary nature and was prepared primarily for
internal use at the Nevada Operations Office. It
is subject to revision or correction and therefore
does not represent a final report.~~

BATTELLE MEMORIAL INSTITUTE
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

~~DISTRIBUTION OF THIS DOCUMENT IS LIMITED
To Government Agencies and their Contractors~~

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

AMCHITKA BIOENVIRONMENTAL PROGRAM

ANNUAL PROGRESS REPORT

RADIONUCLIDE AND STABLE ELEMENT ANALYSES
OF ENVIRONMENTAL SAMPLES FROM
AMCHITKA ISLAND, ALASKA

by

J. R. Vogt, J. E. Howes, and R. A. Ewing

November, 1968

Prepared for the U. S. Atomic Energy
Commission under Contract No. AT(26-1)-171

~~NOTICE: This document contains information of a preliminary nature and was prepared primarily for internal use at the Nevada Operations Office. It is subject to revision or correction and therefore does not represent a final report.~~

~~NOTICE~~
~~This report contains information of a preliminary nature and was prepared primarily for internal use at the originating installation. It is subject to revision or correction and therefore does not represent a final report. It is passed to the recipient in confidence and should not be abstracted or further disclosed without the approval of the originating installation or DRI Extension, Oak Ridge.~~

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights, or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

BATTELLE MEMORIAL INSTITUTE
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

~~DISTRIBUTION OF THIS DOCUMENT IS LIMITED~~
~~To Government Agencies and their Contractors~~

ABSTRACT

An effort to conduct radionuclide and stable element analyses of environmental samples from Amchitka Island, Alaska was initiated by Battelle-Columbus in order to provide stable element concentrations for use with a specific activity model for calculating the potential internal radiation dose to man, and to provide pre- and post-shot data on radionuclide burdens in the Amchitka Island environment. During FY 68, 230 samples were collected by subcontractors and Battelle for possible radionuclide and stable element analysis. While many of these samples were prepared for analysis, only a few were analyzed prior to the end of FY 68. Inventories of collected and prepared samples, as well as the results of those radionuclide and stable element analyses which were completed are included. Analytical procedures are given in the appendix. As of the date of this report, no additional funding has been allocated for the continuation of sample analyses.

TABLE OF CONTENTS

	<u>Page</u>
INTRODUCTION	1
COORDINATION AND SAMPLE COLLECTION	2
SAMPLE PREPARATION	3
RADIONUCLIDE ANALYSES.	3
STABLE ELEMENT ANALYSES.	4
DISCUSSION AND CONCLUSIONS	4
APPENDIX	A-1

INTRODUCTION

The analytical effort of Battelle's Bioenvironmental Safety Program to assist in determining the feasibility of conducting nuclear tests at Amchitka Island, Alaska, was planned to serve two purposes. The first of these was to provide detailed and accurate data on stable element concentrations in significant food chains for use with a specific activity model for calculating the internal radiation dose to man in case of a venting accident. Many of the stable elements for which concentrations must be known are present in very minute amounts in environmental samples. Because of the sophisticated capability necessary for the analysis of elements such as tellurium, yttrium, cerium, scandium, and tungsten at concentration levels of less than one part per million, there are at the present time little or no data available on their concentrations in the different trophic levels of interest. The second purpose of the analytical effort was to provide accurate pre- and post-shot data on radionuclide burdens in the Amchitka environment. If no venting or slight venting should occur, such data would reassure biologists and conservationists that additional radioactive contamination of the environment was nonexistent or insignificant. In the case of more pronounced venting, the data would define the extent of the contamination and aid in assessing the biological effects. As of the date of this report, no additional funding has been allocated for the continuation of sample analyses.

COORDINATION AND SAMPLE COLLECTION

Three trips were made to Amchitka Island by analytical project personnel for the purposes of reconnaissance, sample collection and coordination of sample collection by subcontractors. On the first trip, from September 10, 1967 to September 17, 1967, general guidelines for the collection and shipping of samples from Amchitka to Battelle were discussed with subcontractors from

the University of Washington, the University of Tennessee, Utah State University, Johns Hopkins University, and the Ohio State University. Subsequently, it proved necessary to revise the shipping guidelines several times because of delays encountered in air freight shipments from Amchitka. These revisions substantially decreased the transit time for perishable shipments, and no further problems were encountered in this area.

The second trip to Amchitka, from February 6, 1968 to February 17, 1968, was made to coordinate sample collection activities during the U.S. Bureau of Commercial Fisheries' cruise, to collect water samples from streams and ponds, and to set up a Millipore filtration system for seawater and freshwater samples. At this time a rain gage was also installed to provide information on the input of radioactive fallout from past nuclear tests to the island ecosystem.

The third trip, from May 21, 1968, to June 22, 1968, was primarily for the purpose of assisting in overpressure experiments on sea otters. However, during this trip a number of stream water and pond water samples were collected, and the equipment which had been used by the project on the island was crated and returned to Battelle. During FY 68, 230 samples were collected by subcontractors and Battelle for possible radionuclide and stable element analysis. Included among the samples were freshwater and marine fish; freshwater and marine plankton; stream, pond and seawater; freshwater and marine invertebrates; birds; plants; sea otters; sea lions; soil; rats; snow; and rainwater. In addition to providing data for a specific activity model, the detailed analysis of these samples would have provided significant basic data on the trace element variations in different trophic levels of the Amchitka ecosystem.

SAMPLE PREPARATION

A list of the samples collected by Battelle and its subcontractors is given in Table 1. Table 2 lists the samples which were prepared for analysis and gives their wet-dry-ash weights where applicable. All perishable samples were frozen as soon after collection as possible and shipped to Battelle packed in dry ice. Following thawing and dissection where applicable, plant and animal specimens were dried at 110°C to constant weight, partially ashed at 300°C for 12-18 hours, and ashed at $400\text{-}420^{\circ}\text{C}$ for approximately 18 hours. Water samples were evaporated to dryness. The weighing of Pacific Ocean Perch bone to determine the wet weight to dry weight ratio is shown in Figure 1. Duplicate bone and muscle samples of Ocean Perch were ashed at both 175°C and 450°C and were then analyzed by spark source mass spectroscopy to determine if significant quantities of any elements were lost by ashing at 450°C . Of the elements for which numerical data were obtained by the mass spectrographic technique, it was concluded that only the volatiles, I, As, Cl, S, F, and Li, were lost in significant amounts by ashing at 450°C .

RADIONUCLIDE ANALYSES

Measurements of the concentrations of ^{137}Cs , ^{90}Sr , ^{40}K were made for a number of prepared samples, and the results are given in Table 3. All of the ^{90}Sr data and most of the ^{137}Cs data were determined using radiochemical separations and low-level beta counting. The analytical procedures are given in the appendix. The ^{40}K data and some of the ^{137}Cs data were determined using low-level gamma-ray spectroscopy.

FIGURE 1. PREPARATION OF PACIFIC OCEAN PERCH BONE FOR
TRACE-ELEMENT AND RADIONUCLIDE ANALYSIS

STABLE ELEMENT ANALYSES

A limited number of samples were analyzed by spark source mass spectroscopy for stable element concentrations, and the results are reported in Table 4. The accuracy of the mass spectroscopy data ranges between $\pm 20\%$ and $\pm$ a factor of three, depending on the element, concentration, and the type of samples involved. Because spark source mass spectroscopy provides quantitative data on a large number of elements in one analysis, it is ideal for applications where data on several elements are required. This technique has rather uniform sensitivity for a wide range of elements down to the parts per million level; however, the sensitivity can be affected by the sample composition. However, below this level the use of pre-concentration techniques or other analytical methods becomes necessary for many elements. Because the concentrations of several of the elements of interest are below the detection limits of spark source mass spectroscopy, it was intended that these elements would be analyzed by neutron activation analysis using the 2-megawatt Battelle Research Reactor (Figure 2). However, no stable element analyses were made by activation analysis during FY 68. The analytical procedures for activation analysis which are ready for use are given in the appendix.

DISCUSSION AND CONCLUSIONS

Because of the funding limitations during FY 68, it became necessary to collect and prepare most of the samples during FY 68, and to leave most of the actual analytical work for FY 69. The adaptation of analytical procedures was also planned for completion in FY 68. For these reasons, many samples were prepared for analysis, but only a few were actually analyzed during FY 68. The adaptation of analytical procedures was terminated in late April in order to devote the remaining time to sample analysis, when it became apparent that funds would not be available for analysis in FY 69. Therefore, not all of the analytical procedure

FIGURE 2. THE 2-MEGAWATT BATTELLE RESEARCH REACTOR SHOWING THE WATER-FILLED REACTOR POOL

development was completed.

As expected, the terrestrial samples contained considerably higher amounts of ^{137}Cs than did the marine samples. The highest radionuclide concentration found in any of the samples was 18.6 picocuries per gram dry weight of ^{137}Cs for a composite sample of Dolly Varden muscle (#1016.1). The ^{137}Cs level in these fish--collected on the western end of Amchitka--was considerably higher than that found in fish from the central part of the island. The results of two radiochemical determinations and gamma-ray spectroscopy for different aliquots of this sample were in good agreement. While no explanation for this high value of ^{137}Cs has been found, it does demonstrate the need for an extensive sampling and analysis effort in work of this type. It is important to know the limits of the environmental variations of radionuclide concentrations in order to prevent misinterpretation of pre- and post-shot data. The ^{137}Cs radioactivity levels found in the Amchitka samples are relatively high compared to ^{137}Cs levels found in similar samples by Battelle in Panama. For example, ^{137}Cs levels in plant samples from Panama are typically an order of magnitude lower than the ^{137}Cs level found in crowberries from Amchitka. This is understandable, because fallout levels are generally highest around 50° north latitude. The results of the ^{137}Cs analyses of a male and a female sea otter indicate a rather low level of fallout contamination in these animals. This should make it easy to detect increases in the radioactive contamination of the sea otter if radionuclides from underground testing are transported to the sea.

As can be seen in Table 4, many of the stable elements of interest in specific activity calculations--scandium, yttrium, ruthenium, cesium and tungsten, for example--were present in amounts too small to be detected by mass spectroscopy and are listed as "less than" values. In the absence of better analytical data,

these "less than" values have some usefulness in estimating specific activities for modeling purposes. It should be recognized, however, that the calculation of specific activities using this approach may result in underestimation of radiation doses. When using the specific activity approach, there is no good substitute for accurate analytical data. The analyses of drilling mud from Site B and stream water samples from the watershed around Site B and from a watershed which is not near a drilling site, clearly indicate stream contamination from operations at Site B. The concentrations of aluminum, phosphorus, scandium, vanadium, manganese, iron, yttrium, zirconium, iodine, barium, the rare earths, and lead are all significantly higher in the Site B stream. Changes in trace element concentrations in the environment around Site B due to drilling mud contamination will further complicate efforts to predict radionuclide transport and radiation dose using the specific activity model.

TABLE 1. INVENTORY OF COLLECTED SAMPLES

SAMPLE NUMBER	SAMPLING LOCATION	DESCRIPTION
<u>FRESHWATER FISH</u>		
1000 (a,c)	529979 (b)	Dolly Varden
1001 (c)	518018	" "
1003 (c)	552947	Pink Salmon
1004 (c)	552947	Dolly Varden
1005 (c)	555946	" "
1006 (c)	636972	" "
1007 (c)	512968	Pink Salmon
1008 (c)	512968	Dolly Varden
1009 (c)	546961	" "
1010 (c)	529979	Sockeye Salmon
1011 (c)	519999	Dolly Varden
1012 (c)	521004	" "
1013 (c)	430066	" "
1014 (c)	325153	" "
1015 (c)	146231	" "
1016 (c)	233185	" "
1017 (c)	234183	" "
1018 (c)	281171	" "
1019 (c)	323149	" "
1020 (c)	281169	" "

TABLE 1. INVENTORY OF COLLECTED SAMPLES (cont'd.)

SAMPLE NUMBER	SAMPLING LOCATION	DESCRIPTION
<u>FRESHWATER PLANKTON</u>		
2000 ^(c)	555946	Mixed Plankton
2001 ^(c)	519999	" "
<u>FRESHWATER</u>		
3000	555946	Lake Water
3001	529979	" "
3002	519999	" "
3003	430066	" "
3004	515962	" "
3005	529979	" "
3006	527994	" "
3007	510967	Stream "
3008	518000	Lake "
3009	372107	Stream "
3010	515007	Lake "
3011	519978	Stream "
3012	519004	" "
3013	513969	" "
3014	514014	" "
3015	515007	Sump "

TABLE 1. INVENTORY OF COLLECTED SAMPLES (cont'd.)

SAMPLE NUMBER	SAMPLING LOCATION	DESCRIPTION
<u>MARINE VERTEBRATES AND INVERTEBRATES</u>		
4000	51° 15' N 179° 10.5' E	King Crab
4001 (c)	51° 15.1' N 179° 10.5' E	Squid
4002 (c)	51° 15' N 179° 10.3' E	Pacific Ocean Perch
4003	51° 15' N 179° 10.5' E	Starfish
4004 (c)	Constantine Hbr.	Greenling
4005 (c)	none given	Amphipods
4006 (c)	Constantine Hbr.	Red Irish Lord
4007	51° 15' N 179° 10.5' E	Sea Cucumber
4010 (c)	none given	Salmon
4011 (c)	551947	Amphipods
4012 (c)	551947	Amphipods
4013 (c)	947551	Snails
4014 (c)	947551	Snails
4015 (c)	none given	Jellyfish
4016 (c)	none given	Jellyfish
4017 (c)	587968	Starfish
4018 (c)	none given	King Crab
4019 (c)	none given	Box Crab

TABLE 1. INVENTORY OF COLLECTED SAMPLES (cont'd.)

SAMPLE NUMBER	SAMPLING LOCATION	DESCRIPTION
<u>SALTWATER PLANKTON</u>		
5000 (c)	51° 17' N 179° 21.6' E	Zooplankton
5001 (c)	" "	" "
5002 (c)	" "	" "
5003 (c)	51° 25.5' N 179° 16.5' E	Phytoplankton
5004 (c)	51° 25.5' N 179° 2.1' E	Zooplankton
5005 (c)	none given	Mixed Plankton
5006 (c)	" "	" "
5007 (c)	" "	" "
5008 (c)	" "	" "
<u>SEAWATER</u>		
6000	51° 16' N 179° 4.3' E	Seawater (surface)
6001	51° 16' N 179° 4.3' E	" " (")
6003	51° 42.5' N 179° 02' E	" " (")
6002	51° 56' N 178° 37.5' E	" " (")
6004	51° 56' N 179° 02' E	" " (bottom)
6005	51° 30' N 178° 33' E	" " (surface)
6006	51° 05' N 179° 04' E	" " (")
6007	51° 05' N 179° 04' E	" " (bottom)
6008	51° 19' N 179° 10' E	" " (surface)
6009	51° 25' N 178° 53' E	" " (")

TABLE 1. INVENTORY OF COLLECTED SAMPLES (cont'd.)

SAMPLE NUMBER	SAMPLING LOCATION	DESCRIPTION
<u>SEAWATER (cont'd.)</u>		
6010	51° 25'N 178° 53' E	Seawater (bottom)
6011	51° 21'N 179° 33' E	" " (surface)
6012	51° 38'N 179° 04' E	" " (")
6013	51° 38'N 179° 04' E	" " (bottom)
6014	51° 27'N 179° 23' E	" " (surface)
6015	51° 27'N 179° 23' E	" " (bottom)
6016	Constantine Hbr	" " (surface)
<u>BIRDS</u>		
7000	536968	Rock Ptarmigan
7001	627964	" "
7002	"	" "
7003	"	" "
7004	"	" "
7005	"	" "
7006	"	" "
7007	510963	" "
7008	517022	" "
7009	"	" "
7010	"	" "

TABLE 1. INVENTORY OF COLLECTED SAMPLES (cont'd.)

SAMPLE NUMBER	SAMPLING LOCATION	DESCRIPTION
<u>BIRDS (cont'd.)</u>		
7011	600980	Pelagic Cormorant
7012	"	" "
7013	"	" "
7014	"	" "
7015	"	" "
7016	"	" "
7017	"	" "
7018	"	" "
7019	"	" "
7020	"	" "
7021	Not given	Red Throated Loon
7022	"	" "
7023	623961	Common Teal
7024	"	" "
7025	623961	" "
7026	515963	" "
7027	"	" "
7028	"	" "
7029	"	" "
7030	"	" "
7031	"	" "
7032	"	" "

TABLE 1. INVENTORY OF COLLECTED SAMPLES (cont'd.)

SAMPLE NUMBER	SAMPLING LOCATION	DESCRIPTION
<u>BIRDS (cont'd.)</u>		
7033	515963	Common Teal
7034	605927	" "
7035	"	" "
7036	"	" "
7037	"	" "
7038 (c)	540986	Lapland Longspurs
7039	613968	Common Eider
7040	680951	" "
7041	"	" "
7042	"	" "
7043	529018	" "
7044	"	" "
7045	"	" "
7046	524017	Herring Gull
7047	"	" "
7048	"	" "
7049	"	" "
7050	617924	" "
7051	511966	" "
7052	"	" "
7053	"	" "
7054	"	" "
7055	"	" "

TABLE 1. INVENTORY OF COLLECTED SAMPLES (cont'd.)

SAMPLE NUMBER	SAMPLING LOCATION	DESCRIPTION
7056	623961	Mallard
7057	515962	"
7058	"	"
7059	"	"
7060	51° 28' N 179° 23' E	Least Auklet
7061	" "	" "
7062	" "	" "
7063	" "	" "
7064	" "	" "
7065	" "	" "
7066	Not given	" "
7067	" "	Rock Ptarmigan
7068	" "	" "
7069	" "	" "
7070	" "	" "
7071	" "	" "
7072	365965	" "
7073	515962	Common Teal
7074	583969	Emperor Goose
7075	"	" "
7076	"	" "
7077	"	" "
7078	585958	" "
7079	598985	" "

TABLE 1. INVENTORY OF COLLECTED SAMPLES (cont'd.)

SAMPLE NUMBER	SAMPLING LOCATION	DESCRIPTION
	<u>BIRDS (cont'd.)</u>	
7080	585958	Emperor Goose
7081 (c)	583969	Aleutian Sand- Pipers (3)
7082	586958	Green Winged Teal
7083	558945	Buffle Head
7084	583956	Herring Gull
7085	974631	" "
7086	"	" "
7087	"	" "
7088	957586	" "
7089	"	Mallard
7090	954583	Green Winged Teal
7091	981597	Emperor Goose
7092	973592	Herring Gull
7093	969583	Emperor Goose
7094	"	" "
7095	956585	Lesser Scaup
7096	"	" "
7097	"	" "
7098	"	" "
7099	965600	Herring Gull
7100	965595	Pelagic Cormorant
7101	965600	" "

TABLE 1. INVENTORY OF COLLECTED SAMPLES (cont'd.)

SAMPLE NUMBER	SAMPLING LOCATION	DESCRIPTION
<u>BIRDS (cont'd.)</u>		
7102	965592	Pelagic Commorant
7103	976601	" "
7104	968608	" "
7105	971592	" "
7106	971611	" "
7107	621986	Oldsquaw
7108	975605	"
7109	962596	"
7110	967606	"
7111	965605	Herring Gull
7112	369142	Rock Ptarmigan
7113	583969	Emperor Goose
7114	"	" "
7115 (d)	549953	Bald Eagle
<u>PLANTS AND SOIL</u>		
8000 (c)	430066	Algae (Green)
8001 (c)	382125	Algae (Filamentous)
8003 (c)	541923	Tundra Lichens
8004 (c)	538922	Algae (Brown)
8005 (c)	"	Fucus
8006 (c)	623943	Crowberries
8007 (c)	629952	Peat

TABLE 1. INVENTORY OF COLLECTED SAMPLES (cont'd.)

SAMPLE NUMBER	SAMPLING LOCATION	DESCRIPTION
<u>PLANTS AND SOIL</u> (cont'd.)		
8008 ^(c)	635965	Peat
8009 ^(c)	622952	Peat
8010 ^(c)	522982	Grass Covered Peat
8011 ^(c)	630934	Peat
8012 ^(c)	379097	Pool Bottom Muck
8013 ^(c)	550950	Fucus
8014 ^(c)	668944	Seagrass
8015 ^(c)	615962	Reedgrass
8016 ^(c)	515007	Peat
8017 ^(c)	515007	Peat
8018 ^(c)	515008	Sump Bottom Muck
<u>MISCELLANEOUS</u>		
9000 ^(c)	352125	Ptarmigan Droppings
9001 ^(c)	375128	Snow (three-gal cans)
9002 ^(c)	586944	Rain & Snow
9003 ^(c)	586944	Rain
9100	unknown	Female Sea Otter
9101	"	Male Sea Otter
9102	584961	Female Sea Otter (viscera)
9103	588972	Male Sea Lion (viscera)
9104	552946	Rat
9105	"	"
9106	550946	"
9107	"	"
9108	519982	Drilling Mud Site B

TABLE 1. INVENTORY OF COLLECTED SAMPLES (cont'd.)

(a) The sample numbering system used by this project is as follows:

Series 1000	=	Freshwater Fish
" 2000	=	Freshwater Plankton
" 3000	=	Freshwater
" 4000	=	Marine Vertebrates and Invertebrates
" 5000	=	Saltwater Plankton
" 6000	=	Seawater
" 7000	=	Birds
" 8000	=	Plants and Soil
" 9000	=	Miscellaneous

When a particular portion of a sample or a composite sample was prepared, a decimal was added; e.g., 7080 = Emperor Goose and 7080.1 = Emperor Goose muscle. There was no uniform system used in the decimal part of the numbering system; e.g., xxxx.1 may be muscle in one case and bone in another. In cases where organs from several samples were combined to make a composite sample, the 4-digit number from one of the samples was used as the main identification number; e.g., 7034.3 is a composite sample of six Common Teal hearts, one of which is from sample 7034.

- (b) UMT grid coordinates.
- (c) Composite sample.
- (d) Found dead by natural causes.

TABLE 2. INVENTORY OF PREPARED SAMPLES

SAMPLE NO.	DESCRIPTION	WEIGHTS, GRAMS		
		WET	DRY	ASH
1003.1 (a,c)*	Pink Salmon, muscle	712.4	157.3	11.8
1003.2 (c)	" " bone	102.7	27.5	8.3
1003.3 (c)	" " skin	72.8	20.7	1.4
1006.1 (c)	Dolly Varden muscle	(not separated)		
1006.2 (c)	" " bone			
1006.4 (c)	" " whole fish	1088.0	273.0	30.5
1007.1 (c)	Pink Salmon muscle	970.7	257.0	18.8
1007.2 (c)	" " bone	168.0	47.0	13.6
1007.3 (c)	" " skin	106.7	30.3	1.8
1008.1 (c)	Dolly Varden muscle	1527.6	451.6	16.6
1008.2 (c)	" " bone	100.0	53.7	14.4
1008.3 (c)	" " eggs	0.8	0.7	0.0333
1008.4 (c)	" " whole fish	287.5	58.2	5.6
1010.1 (c)	Sockeye Salmon muscle	482.6	120.8	8.10
1010.2 (c)	" " bone	64.9	23.2	6.10
1010.3 (c)	" " skin	64.2	24.0	1.81
1011.1 (c)	Dolly Varden muscle	889.3	253.5	6.6
1011.2 (c)	" " bone	117.0	44.3	10.3
1011.3 (c)	" " eggs	34.3	10.4	0.5611
1016.1 (c)	Dolly Varden muscle	207.2	44.0	2.1
1016.2 (c)	" " bone	66.7	20.9	6.0
1016.3 (c)	" " eggs	55.7	14.3	0.60

TABLE 2. INVENTORY OF PREPARED SAMPLES (cont'd.)

SAMPLE NO.	DESCRIPTION	WEIGHTS, GRAMS		
		WET	DRY	ASH
4000.1	King Crab muscle	272.0	44.8	4.5
4000.2	" " shell	161.0	80.0	27.5
4001 (c)	Squid (whole)	979.8	245.6	21.6
4002.1 (c)	Pacific Ocean Perch bone	--	64.9	38.8
4002.2 (c)	" " " muscle	2152	756.8	18.92
4002.3 (c)	" " " skin	297.6	163.0	27.47
4002.4 (c)	" " " stomach contents	25.5	4.07	0.7903
4005 (c)	Amphipods	213.0	41.5	15.6
4011 (c)	Amphipods	110.8	19.0	7.9
4014 (c)	Snails	71.2	37.1	29.9
4018.1 (c)	King Crab muscle	1865.4	671.9	39.3
4018.2 (c)	" " shell	1164.0	427.3	211.2
5000 (c)	Mixed Plankton	--	6.2	3.1125
5003 (c)	Phytoplankton	--	6.0	2.50
5004 (c)	Zooplankton	--	2.8	0.8042
6000	Sea Water	2000	71.0	--
6001.1	Sea Water Organics		0.0846	0.0463
7027.1 (c)	Common Teal muscle	167.3	42.5	2.1
7027.2 (c)	" " bone	83.5	30.0	11.1
7031.1 (c)	Common Teal muscle	182.6	60.3	2.3
7031.2 (c)	" " bone	77.1	40.6	11.7
7034.1	Common Teal muscle	89.0	16.8	0.90
7034.2	" " bone	48.6	21.0	6.2

TABLE 2. INVENTORY OF PREPARED SAMPLES (cont'd.)

SAMPLE NO.	DESCRIPTION			WEIGHTS, GRAMS		
				WET	DRY	ASH
7034.3 (c)	Common Teal	hearts		25.8	7.7	0.80
7034.4 (c)	"	"	livers	56.2	17.0	1.4
7037.1	Common Teal	muscle		87.8	16.3	0.90
7037.2	"	"	bone	37.8	21.0	5.5
7056.1	Mallard	muscle		432.7	112.8	5.1
7056.2	"	bone		98.8	47.0	28.0
7058.1	Mallard	muscle		270.7	68.5	3.6
7058.2	"	bone		71.3	28.5	10.6
7058.3	"	stomach contents		0.8	0.1	0.0274
7059.1	Mallard	muscle		280.1	69.1	3.5
7059.2	"	bone		108.2	40.2	15.0
7059.3	"	stomach contents		0.6	--	0.0020
7074.1	Emperor Goose	muscle		629.8	174.6	19.1
7074.2	"	"	bone	166.4	81.7	31.8
7074.3 (c)	"	"	hearts	93.1	25.1	1.10
7074.4 (c)	"	"	livers	156.2	47.2	2.30
7075.1	Emperor Goose	muscle		622.6	184.3	24.9
7075.2	"	"	bone	157.8	84.8	28.3
7076.1	"	"	muscle	851.4	254.4	20.6
7076.2	"	"	bone	250.3	128.1	42.6
7077.1	"	"	muscle	584.9	176.4	9.4
7077.2	"	"	bone	158.0	74.9	29.0
7078.1	"	"	muscle	685.7	185.1	18.0
7078.2	"	"	bone	133.8	81.2	32.9

TABLE 2. INVENTORY OF PREPARED SAMPLES (cont'd.)

SAMPLE NO.	DESCRIPTION	WEIGHTS, GRAMS		
		WET	DRY	ASH
7080.1	Emperor Goose muscle	760.4	234.5	10.1
7080.2	" " bone	123.8	70.8	31.1
7080.5 (c)	" " esophagus	8.0	0.9	0.2
7080.6 (c)	" " glandular stom.	5.6	0.8	0.3
7080.7 (c)	" " gizzard	243.8	73.2	27.4
7080.8 (c)	" " sm. intestine	222.9	30.4	6.9
7080.9 (c)	" " cecum	47.2	6.7	2.1
7080.11 (c)	" " lg. intestine	28.9	4.0	0.9
7091.1	" " muscle	788.3	222.6	10.0
7091.2	" " bone	180.3	94.3	37.9
7093.1	" " muscle	585.9	150.1	10.7
7093.2	" " bone	122.9	61.8	25.2
7095.1	Lesser Scaup muscle	258.3	72.9	3.6
7095.2	" " bone	86.2	41.7	14.5
7095.3 (c)	" " heart	39.4	11.2	1.4
7095.4 (c)	" " liver	141.7	42.9	3.5
7096.1	" " muscle	211.7	56.7	5.7
7096.2	" " bone	92.1	38.1	13.0
7097.1	" " muscle	260.3	72.7	4.7
7097.2	" " bone	92.8	42.8	15.4
7098.1	" " muscle	274.0	78.0	3.7
7098.2	" " bone	117.3	44.8	16.9
7115.1	Bald Eagle muscle	233.0	48.0	3.2
7115.2	" " bone	67.8	39.3	21.1

TABLE 2. INVENTORY OF PREPARED SAMPLES (cont'd.)

SAMPLE NO.	DESCRIPTION	WEIGHTS, GRAMS		
		WET	DRY	ASH
7115.3	Bald Eagle heart	30.0	4.5	0.2
7115.4	" " liver	36.2	8.9	0.8
8000 (c)	Algae (Green)	56.1	2.8	0.1403
8001 (c)	Algae (Filamentous)	48.7	8.9	4.0
8003 (c)	Lichen-Sedge (Tundra)	962.0	291.5	9.2
8004 (c)	Algae (Brown)	1080	174	32.3
8005 (c)	<u>Eucus</u> sp.	5270	780	104.5
8006 (c)	Crowberries	550.0	44.3	1.5722
9001 (c)	Snow (melted)	Total residue = 3.00 grams		
9002 (c)	Rain Water	Weight of residue = 143.4 mg		
9100.1	Female Sea Otter liver	748.5	254.5	7.4
9100.2	" " lungs	397.1	88.5	4.7
9100.3	" " heart	103.3	24.0	0.9292
9100.4	" " spleen	113.5	25.2	1.3692
9100.5	" " kidneys	246.0	65.7	3.0395
9100.6	" " pancreas	50.9	15.8	0.7947
9100.7	" " muscle	1175.0	392.6	19.0
9100.8	" " bone	--	79.8	67.1
9100.9	" " fur & skin	9.5	--	0.206
9101.1	Male Sea Otter liver	928.3	363.0	11.5
9101.2	" " lungs	578.3	127.5	5.9

TABLE 2. INVENTORY OF PREPARED SAMPLES (cont'd.)

SAMPLE NO.	DESCRIPTION	WEIGHTS, GRAMS		
		WET	DRY	ASH
9101.3	Male Sea Otter heart	134.0	34.3	1.306
9101.4	" " " spleen	89.5	22.0	1.09
9101.5	" " " kidneys	576.7	162.0	6.8
9101.6	" " " pancreas	30.5	10.8	0.413
9101.7	" " " thyroid	3.8	0.8	0.03
9101.8	" " " muscle	979.2	332.0	24.7
9101.9	" " " bone	--	90.1	60.1
9101.11	" " " fur & skin	23.0	--	0.457

* Footnotes listed for Table 1 on page 17a apply also to Table 2.

TABLE 3. RADIONUCLIDE ANALYSES

SAMPLE NUMBER	SAMPLING LOCATION	DESCRIPTION	PICOCURIES PER GRAM, DRY WEIGHT ^(a)		
			¹³⁷ Cs	⁹⁰ Sr	⁴⁰ K
4000.1	51° 15'N 179° 10.5'E	King crab, muscle	<0.04		
4000.2	51° 15'N 179° 10.5'E	King crab, shell	<0.07		
8004	538922	Brown alga	0.012±0.024		
8005	538922	<u>Fucus</u> sp.	0.014±0.027		
4005	unknown	Amphipods, whole	0.11±0.07	0.008±0.04	
4002.1	51° 15'N 179° 10.5'E	Pacific Ocean Perch, bone	0.0067±0.094	0.09±0.06	
4002.2	" "	Pacific Ocean Perch, muscle	0.078±0.01 0.065±0.01 ^(b)	<0.006	5.03±0.25
4002.3	" "	Pacific Ocean Perch, skin	0.031±0.03 0.040±0.03	0.038±0.025	
1016.1	233185	Dolly Varden, muscle	16.02±0.09 17.26±0.08 18.6±1.0 ^(b)		
1016.2	233185	Dolly Varden, bone	11.62±0.14 9.42±0.14		
1011.1	519999	Dolly Varden, muscle	0.96±0.02 0.92±0.02		
1011.2	519999	Dolly Varden, bone	0.81±0.05		
1008.1	512968	Dolly Varden, muscle	0.39±0.02 0.35±0.02 0.37±0.007 ^(b)		9.4±0.15
1008.2	512968	Dolly Varden, bone	0.93±0.05		
1008.4	512968	Dolly Varden, whole	0.98±0.04 0.96±0.05		

TABLE 3. RADIONUCLIDE ANALYSES (cont'd.)

SAMPLE NUMBER	SAMPLING LOCATION	DESCRIPTION	PICOCURIES PER GRAM. DRY WEIGHT (a)		
			¹³⁷ Cs	⁹⁰ Sr	⁴⁰ K
1006.4	636972	Dolly Varden, whole	0.97±0.03 ^(b)		11.0±0.2
7056.1	623961	Mallard muscle	4.43±0.06		
			4.68±0.05		
7056.2	623961	Mallard bone	1.90±0.12		
7058.1	515962	Mallard muscle	1.98±0.05		
			2.04±0.03		
7058.2	515962	Mallard bone	0.74±0.10		
7059.1	515962	Mallard muscle	1.39±0.04		
			1.52±0.03		
7059.2	515962	Mallard bone	0.76±0.10		
6000	51° 16'N 179° 4.3'E	Sea Water	0.28±0.09 ^(b)		6.6±0.4
1007.1	512968	Pink Salmon, muscle	0.124±0.007 ^(b)		12.6±0.1
1010.1	529979	Sockeye Salmon, muscle	0.073±0.01 ^(b)		12.8±0.2
8006	623943	Crowberries	0.74±0.06 ^(b)		4.2±0.6
9100.1	unknown	Female Sea Otter, liver	0.017±0.011		
9100.2	"	" " " , lung	0.008±0.022 0.02±0.02	<0.02	
9100.5	"	" " " , kidney	0.04±0.02	0.0009±0.015	
9100.7	"	" " " , muscle	0.05±0.01		
9100.8	"	" " " , bone	<0.18		

TABLE 3. RADIONUCLIDE ANALYSES (cont'd.)

SAMPLE NUMBER	SAMPLING LOCATION	DESCRIPTION	PICOCURIES PER GRAM, DRY WEIGHT ^(a)		
			¹³⁷ Cs	⁹⁰ Sr	⁴⁰ K
9101.1	unknown	Male Sea Otter, liver	0.03±0.01	<0.016	
9101.2	"	" " " , lung	0.05±0.01	<0.015	
9101.5	"	" " " , kidney	0.02±0.01	0.009±0.014	
9101.8	"	" " " , muscle	0.08±0.02		
9101.9	"	" " " , bone	<0.14		

(a) Error limits are based on counting statistics only.

(b) Cs-137 value obtained by gamma ray spectroscopy.

TABLE 4. STABLE ELEMENT ANALYSES

ELEMENT	STABLE ELEMENT CONCENTRATION (a)						
	Pac. Ocean Perch muscle, #4002.2 ($\mu\text{g/g ash}$)	King Crab muscle, #4018.1 ($\mu\text{g/g ash}$)	Zooplankton #5004 ($\mu\text{g/g ash}$)	Emperor Goose #7074.1 ($\mu\text{g/g ash}$)	Drilling Mud #9108 ($\mu\text{g/ml}$)	Stream Water #3011 ($\mu\text{g/ml}$)	Stream Water #3014 ($\mu\text{g/ml}$)
Li (b)	10.	1.	10.	4.	1.	0.004	0.005
Be	<0.02	<0.006	<0.002	<0.006	0.02	0.000005	<0.000002
B	4.	40.	60.	40.	2.	0.01	0.01
F (b)	40.	6.	2,000.	2.	15.	0.03	0.02
Na	high	major	high	high	high	major	major
Mg	$\approx 2\%$	high	high	high	high	high	high
Al	50.	20.	high	150.	high	high	0.2
Si	300.	300.	high	500.	major	high	high
P	high	high	high	high	high	high	0.03
S (b)	10,000.	3,000.	8,000.	3,000	350.	0.8	1.
Cl (b)	high	high	high	high	high	high	high
K	major	major	7,000.	major	200.	high	high
Ca	20,000.	60,000.	major	10,000.	high	high	high
Sc	<0.2	<1.	<1.	<1.	7.	0.0025	<0.00003
Ti	<20.	<20.	1,000.	<6.	150.	0.15	0.015
V	<1.	<1.	30.	<1.	7.	0.05	0.003
Cr	2.	1.	20.	<0.1	300.	0.001	0.002
Mn	10.	30.	50.	10.	100.	high	0.1
Fe	500.	100.	2,000.	2,000.	$\approx 2,000.$	$\approx 15.$	0.6
Co	0.3	<1.	<3.	<1.	2.	0.001	<0.0003
Ni	3.	5.	5.	10.	<0.07	0.025	0.015
Cu	50.	400.	600.	150.	10.	0.01	0.006
Zn	20.	1,000.	600.	300.	<0.7	0.008	<0.003
Ga	<1.	<2.	<2.	<2.	0.4	<0.0005	<0.0003
Ge	<1.	<1.	<1.	<1.	<0.07	<0.0003	≤ 0.0003
As (b)	<4.	5.	15.	<1.	1.	0.001	0.002
Se	<30.	$\leq 4.$	$\leq 6.$	<0.1	<0.03	≤ 0.001	<0.0002
Br	100.	300.	1,000.	600.	1.	0.03	0.1
Rb	40.	25.	5.	100.	2.	0.003	0.002

TABLE 4. STABLE ELEMENT ANALYSES (cont'd.)

ELEMENT	STABLE ELEMENT CONCENTRATION (a)						
	Pacific Ocean Perch muscle, #4002.2 ($\mu\text{g/g ash}$)	King Crab muscle #4018.1 ($\mu\text{g/g ash}$)	Zooplankton #5004 ($\mu\text{g/g ash}$)	Emperor Goose #7074.1 ($\mu\text{g/g ash}$)	Drilling Mud #9108 ($\mu\text{g/ml}$)	Stream Water #3011 ($\mu\text{g/ml}$)	Stream Water #3014 ($\mu\text{g/ml}$)
Sr	20.	200.	1,000.	2.	15.	0.02	0.01
Y	<0.04	<0.04	<0.04	<0.04	1.	0.002	≤ 0.0006
Zr	<0.1	<0.1	<0.1	<0.1	3.	0.003	≤ 0.0001
Nb	<5.	<2.	<2.	<6.	0.4	0.0002	0.0002
Mo	<0.6	$\leq 2.$	<6.	$\leq 2.$	≤ 0.3	≤ 0.0005	≤ 0.001
Ru	<0.2	<2.	<5.	<0.6	<0.04	<0.0002	<0.0002
Rh	<0.2	<0.2	<0.2	<0.2	<0.1	<0.00002	<0.00006
Pd	<1.	<1.	<8.	<1.	<0.07	<0.00005	<0.0003
Ag	<0.4	<1.	<0.4	<1.	<0.07	<0.0003	<0.001 (T)
Cd	<0.2	<2.	<2.	<0.2	<0.01	<0.00005	<0.00006
In	<0.6	<0.2	<2.	<0.6	<0.04	<0.0002	<0.0002
Sn	$\leq 2.$	<2.	15.	<1.	≤ 0.07	≤ 0.00005	<0.00006
Sb	≤ 0.1	$\leq 1.$	<1.	≤ 0.1	0.1	0.0003	≤ 0.0002
Te	<0.06	<2.	<0.6	<0.06	<0.004	<0.00002	<0.00002
I (b)	<0.2	6.	10.	0.6	<0.1	0.003	0.0006
Cs	<0.6	<0.6	<1.	<0.2	<0.01	<0.00005	<0.0002
Ba	1.	5.	150.	≤ 0.3	5.	0.03	0.0006
La	<0.06	<0.6	$\leq 1.$	<0.2	0.4	0.0003	<0.00002
Ce	<0.06	<0.06	≤ 0.2	<0.1	0.7	0.001	≤ 0.00003
Pr	<0.6	<1.	$\leq 1.$	<0.3	0.1	0.0003	<0.0003
Nd	<0.3	<0.1	<1.	<0.1	0.7	0.001	<0.00003
Sm	<0.2	<0.2	<0.6	<0.3	0.4	0.0005	<0.00003
Eu	<0.2	<0.2	<0.6	<0.2	0.04	0.0001	<0.00006
Gd	<0.1	<0.1	<0.3	<0.3	0.1	0.0001	<0.00003
Tb	<1.	<1.	<0.3	<0.3	0.02	0.00003	<0.00001
Dy	<0.1	<0.1	<0.3	<0.3	0.02	0.0002	<0.00003
Ho	<0.1	<0.03	<0.1	<0.1	0.04	0.00003	<0.00003
Er	<0.1	<0.1	<0.1	<0.1	0.1	0.0002	<0.00003
Tm	<0.03	<0.03	<0.3	<0.3	0.02	0.00005	<0.0001
Yb	<0.1	<0.1	<1.	<0.1	0.2	0.0003	<0.00003
Lu	<0.03	<0.3	<0.3	<0.03	<0.02	<0.00008	<0.00003
Hf	<0.1	<0.3	<0.1	<0.1	<0.7	≤ 0.0001	<0.00003

TABLE 4. STABLE ELEMENT ANALYSES (cont'd.)

STABLE ELEMENT CONCENTRATION ^(a)							
	Pacific Ocean Perch muscle, #4002.2	King Crab muscle, #4018.1	Zooplankton #5004	Emperor Goose #7074.1	Drilling Mud #9108	Stream Water #3011	Stream Water #3014
ELEMENT	(ug/g ash)	(ug/g ash)	(ug/g ash)	(ug/g ash)	(ug/ml)	(ug/ml)	(ug/ml)
Ta	<3.	<3.	<1.	<1.	<0.07	<0.0001	<0.0003
W	<0.1	<0.1	3.	<0.1	2.	<0.0003	<0.0001
Re	<0.06	<0.06	<0.06	<0.06	<0.004	<0.00002	<0.00002
Os	<0.06	<0.06	<0.06	<0.06	<0.004	<0.00002	<0.00002
Ir	<0.06	<0.1	<0.06	<0.1	<0.007	<0.00002	<0.0006
Pt	<1.	<1.	<1.	<1.	<0.007	<0.00003	<0.0003
Au	<0.3	<0.3	<0.3	<0.3	<0.002	<0.00008	<0.0001
Hg	<0.1	<0.1	<0.1	<0.1	<0.007	<0.00003	<0.00003
Pb	1.	<0.1	2.	2.	2.	0.001	<0.0001
Tl	<0.05	<0.05	<0.1	<0.1	<0.007	<0.00002	<0.00002
Bi	<0.1	<0.04	<0.1	<0.04	<0.003	<0.00002	<0.00001
Th	<0.04	<0.04	<0.04	<0.04	0.04	0.00003	<0.00001
U	<0.04	<0.04	<0.04	<0.04	0.01	0.00003	<0.00001

(T) = Trace.

(a) The use of the terms "high" and "major" in reporting the results of spark source mass spectroscopy is nebulous. In general, "high" indicates an abundance between 0.1 and 10%, while "major" indicates an abundance >10%.

(b) Value in doubt for ashed samples due to volatility.

APPENDIX

ANALYTICAL PROCEDURES

Introduction

These analytical procedures were adapted from various literature sources to meet the specific requirements of Battelle's bioenvironmental programs. In many cases the final procedure for a given element is considerably different than the literature reference and involved a great deal of time and effort before the procedure was considered ready for routine use. In other cases only minor modifications were necessary.

Procedure For Determination of Sr-90 In Environmental And Biological Materials

Summary of Method

The direct determination of strontium-90 in biological materials is frequently complicated by the presence of large amounts of calcium. In this procedure, ^{90}Sr is determined by separation and radioassay of the yttrium-90 daughter. An initial extraction with di(2-ethylhexyl)phosphoric acid (HDEHP) is performed which removes the ^{90}Y and contaminants. The ^{90}Y is then allowed to "grow in" for two weeks and then is separated and radioassayed. Radioactive ^{85}Sr is used to determine the yield of ^{90}Sr at the end of the procedure. Recoveries of both ^{90}Sr and ^{90}Y are usually 90 percent. About 15 samples per day can be processed through the first part of the procedure. About the same number can be processed in one day through the final steps.

Reagents

Yttrium carrier (10mg Y/ml) - Dissolve 34.3 grams of $\text{YCl}_3 \cdot 6\text{H}_2\text{O}$ in one liter of 0.08 N HCl. Standardize the carrier in triplicate as follows: Pipette 5.0 ml of carrier into a 40 ml glass centrifuge cone and add 20 ml of distilled water. Heat to boiling and add 20 ml of saturated ammonium oxalate solution with stirring. Centrifuge, decant and discard the supernate. Take up the precipitate in 10 ml of distilled water and filter through Whatman #42 filter paper. Wash precipitate with 10-20 ml of water and transfer to a porcelain crucible. Dry the filter and ignite at 800 C for one hour. Cool and weigh. Repeat ignition and reweighing until a constant weight is obtained (± 0.1 mg). Calculate concentration of the carrier as follows:

$$\text{mg Y/ml} = \frac{(\text{Wt. mg } \text{Y}_2\text{O}_3) (0.7875)}{5}$$

^{85}Sr tracer - Dilute 2.5×10^6 d/m of ^{85}Sr to 500 ml with 1 N HCl. Add 1.2 grams of $\text{Sr}(\text{NO}_3)_2$. Take duplicate 2 ml aliquots to be used as counting standards.

20% HDEHP in toluene - Dissolve 200 ml of HDEHP in 800 ml of toluene.

5% HDEHP in toluene - Dissolve 50 ml of HDEHP on 950 ml of toluene.

0.08N HCl - Dilute 6.7 ml of conc HCl to one liter with distilled water.

3N HNO_3 - Dilute 192 ml of conc HNO_3 to one liter with distilled water.

19M NaOH - Dissolve 760 grams of NaOH pellets in distilled water and dilute to one liter. Cool and store in a polyethylene bottle.

Saturated ammonium oxalate - Dissolve 30 grams of $(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ in one liter of distilled water. Solid ammonium oxalate should remain in the bottom of the container. If it does not, add more ammonium oxalate.

Procedure (1)(2)

1. To a one gram ash sample in a 50 ml beaker, add 1.0 ml of ^{85}Sr spike (~5000 d/m), and 5 ml of 6N HCl. Stir well and evaporate to dryness on a hot plate.
2. Take up residue in 30 ml of 0.08 N HCl. Stir well. If solids are present, transfer to a centrifuge cone, centrifuge, and discard solids.
3. Transfer the supernate to a 125 ml separatory funnel. Add 30 ml of 20% HDEHP and extract for 2 minutes. Discard organic phase.
4. Repeat extraction with an additional 30 ml of 20% HDEHP. Discard organic phase. Record date and exact time of the second extraction.
5. Transfer the aqueous phase to a glass screw cap bottle, add 1 ml (10 mg) of standard yttrium carrier and store for two weeks.
6. After buildup of the ^{90}Y , extract with 30 ml of 5% HDEHP. Record date and time of extraction. Save aqueous for Step 12.
7. Wash organic phase twice with 30 ml aliquots of 0.08N HCl. Discard wash solutions.
8. Extract ^{90}Y from organic with 30 ml of 3N HNO_3 .
9. Neutralize the nitric acid solution with 19M NaOH to precipitate $\text{Y}(\text{OH})_3$.
10. Dissolve $\text{Y}(\text{OH})_3$ in 2 ml of 6N HCl and dilute the volume to 15 ml. Heat to boiling and add 15 ml of saturated $(\text{NH}_4)_2\text{C}_2\text{O}_4$ solution with stirring. Centrifuge and discard supernate. Take up precipitate in 15 ml of water and filter through a glass fiber filter disk. Wash with water and ethanol.
11. Dry at 100 C for about 10 minutes. Cool and weigh. Repeat heating and weighings until a constant weight is obtained (± 0.1 mg).
12. Dilute aqueous phase from Step 6 to 50 ml. Pipette duplicate 2.0 ml

(1) References at end of Appendix.

aliquots into a culture tube and radioassay along with ^{85}Sr standards to obtain yield for strontium.

13. Radioassay ^{90}Y using low-level beta counting. Determine half-life to confirm presence of ^{90}Y .
14. Process at least one blank with each set of samples.

Separation of Cesium From Environmental And Biological Samples

Summary of Method

Cesium is separated from the other alkali metals and other contaminants by precipitation as the silicotungstate. The cesium is finally precipitated as the chloroplatinate for yield determination and radioassay. The method has been used for analysis of ^{137}Cs fallout in environmental and biological samples and for separation of ^{134}Cs in neutron activation analysis of environmental and biological samples. In the latter application, ^{86}Rb is frequently observed as a contaminant in the final precipitate. However, in most cases, the decontamination is sufficient to permit determination of the ^{134}Cs . The time required to perform the analysis is about 1 hour.

Reagents

Cesium carrier (10 mg/ml) - Dissolve 12.5 grams in 1 liter of distilled water. Standardize in triplicate as follows: Pipette 5.0 ml of cesium carrier into a 40 ml glass centrifuge cone and dilute to 10 ml with 6 M HCl. Add 4 ml of 10 percent chloroplatinic acid and stir well. Heat 10 minutes. Filter through a tared fine porosity filter crucible. Wash the precipitate three

times with 5 ml portions on 6 M HCl and three times with 5 ml portions of ethanol. Dry at 100 C for 10 minutes. Cool and weigh. Repeat drying, cooling, and weighing until a constant weight is obtained (± 0.1 mg). Calculate concentration of carrier as follows:

$$\text{mg Cs/ml} = \frac{(\text{mg Cs}_2\text{PtCl}_6) (0.3945)}{5}$$

0.12 M Silicotungstic acid - Dissolve 350 grams of $\text{H}_8\text{SiW}_{12}\text{O}_{42}$ in distilled water and dilute to one liter.

6 M HCl - Dilute 515 ml of conc HCl to one liter with distilled water.

6N NaOH - Dissolve 240 grams of NaOH pellets in distilled water and dilute to one liter

10% Chloroplatinic acid - Dissolve 4.8 grams of platinum in aqua-regia. Evaporate to dryness. Dissolve residue in 17 ml of conc HCl and dilute to 100 ml. with distilled water.

Rubidium carrier (10 mg/ml) - Dissolve 14.1 grams of RbCl in one liter of distilled water.

Procedure (3)

1. To a weighed ash sample (~ 1 g) in a 40 ml centrifuge cone, add 2.0 ml of cesium carrier, 2.0 ml of rubidium carrier^(Note a) and 5 ml conc HCl. Digest in a hot water bath for 15 minutes with occasional stirring. Cool, centrifuge, and transfer supernate to a clean centrifuge cone. Discard residue.
2. Add 1 ml of 0.12 M silicotungstic acid solution and stir thoroughly. Digest in a hot water bath for 5 minutes with occasional stirring. Cool, centrifuge, decant and discard supernate. Wash the precipitate with 5 ml of 6M HCl. Centrifuge, decant, and discard wash solution.

3. Add 1.5 ml of 6M NaOH to the precipitate and heat gently with stirring until solution is clear. Add 10 ml of 6M HCl and heat gently for 5 minutes. Cool the mixture and allow to stand for 5 minutes. Centrifuge and decant the supernate to a clean 40 ml glass centrifuge cone. Wash the precipitate with 5 ml of 6M HCl, centrifuge, decant and combine with supernate. Discard the precipitate.
4. Repeat Steps 2 and 3. Add another 2 ml of rubidium carrier^(Note a) prior to starting Step 2.
5. To final solution add 1 ml of 10 percent chloroplatinic acid and stir well. Allow to stand for 5 minutes. Centrifuge and discard supernate.
6. Add 10 ml of 6M HCl, slurry the precipitate, and filter through a tared fiberglass filter disc. Wash the precipitate-twice with 5 ml portions of 6M HCl and twice with 5 ml portions of ethanol.
7. Dry the precipitate at 100 C for 10 minutes. Cool and weigh. Repeat drying, cooling and weighing until a constant weight^(Note b) is obtained (± 0.1 mg).
8. Radioassay the precipitate for ^{134}Cs by gamma spectrometry and for ^{137}Cs by low level beta counting.

Notes:

- a. Rubidium carrier should not be added when the procedure is being used to separate ^{137}Cs fallout.
- b. The yield is usually about 70 percent.

Separation of Ruthenium From Neutron
Activated Environmental and
Biological Samples

Summary of Method

Ruthenium is oxidized to RuO_4 and extracted into carbon tetrachloride,

providing a high degree of decontamination. The ruthenium is reduced to the metal for yield determination and radioassay. About 45 minutes is required to perform the separation.

Reagents

Ruthenium carrier (10 mg/ml) - Dissolve 2.6 grams of $\text{RuCl}_3 \cdot \text{XH}_2\text{O}$ in 100 ml of 0.1M HCl. Standardize in triplicate as follows: Pipette 5.0 ml of carrier solution into a 125 ml Erlenmeyer flask and add magnesium metal powder slowly until an excess has been added. Dissolve excess Mg by adding conc HCl. Cool, centrifuge, and discard the supernatant liquid. Add 20 ml of 1M HCl, stir, and filter through a medium porosity filter crucible. Wash Ru metal twice with 5 ml of distilled water and twice with 5 ml of ethanol. Dry for 15 minutes at 100 C, cool, and weigh. Repeat drying, cooling, and weighing until a constant weight is obtained (± 0.1 mg). Calculate concentration of carrier as follows:

$$\text{mg Ru/mg} = \frac{\text{Wt Ru Metal, mg}}{5}$$

Potassium hydroxide, pellets

Potassium persulfate, solid

Potassium periodate, solid

Magnesium metal, powder

Carbon tetrachloride

Conc HNO_3

6M HCl - Dilute 515 ml of conc HCl to 1 liter.

Conc HCl

Methanol

Ethanol, absolute

Wetting agent, Aerosol OT[®]; 1% solution

Procedure

1. To a one gram sample of ash in a 40 ml glass centrifuge cone, add 2.0 ml of Ru carrier and 5 ml of 6M HCl. Digest for 15 minutes in a hot water bath with occasional stirring. Cool, centrifuge, and decant supernatant liquid to a clean centrifuge cone. Rinse residue with 5 ml of distilled water. Centrifuge and combine with original supernate. Discard residue.
2. Make solution alkaline by adding KOH pellets and then add four pellets in excess.
3. Add 0.5 grams of potassium persulfate with stirring and dilute volume to 25 ml with distilled water.
4. Heat in a hot water bath for 5 minutes and add one gram of potassium periodate with stirring. Cool to room temperature and transfer to a 125 ml separatory funnel.
5. Add 20 ml of CCl_4 . While mixing phases, add conc HNO_3 dropwise until aqueous phase is just acidic, then add 4 drops in excess. Shake for about 60 seconds to extract RuO_4 .
6. Decant CCl_4 phase to a clean separatory funnel^(Note a). Wash with 15 ml of distilled water. Discard wash. Transfer CCl_4 to a 40 ml centrifuge cone.
7. Add 2 drops of methanol, stir, centrifuge, and discard supernatant liquid.
8. Dissolve precipitate in 5 ml of 6M HCl and heat solution for 5 minutes on a hot water bath to remove CCl_4 .
9. Dilute to a 10 ml and add magnesium metal powder slowly with stirring. Add Mg until an excess is present.

10. Dissolve excess Mg by adding 5 ml of conc HCl slowly and with stirring. Heat to 80-90 C and add 1-2 drops of Aerosol solution.
11. Cool, centrifuge, and discard supernatant liquid. Wash precipitate with 10 ml of water.
12. Filter precipitate using tared fiberglass filter. Wash with 2-5 ml portions of distilled water, and 2-5 ml portions of ethanol.
13. Dry filter and Ru metal for 10 minutes at 100 C and weigh to obtain yield^(Note b). Repeat drying and weighing until a constant weight is obtained (± 0.1 mg).
14. Radioassay for ^{103}Ru by measurement of 0.498 mev gamma ray.

Notes::

- a. Funnel should be washed thoroughly with CCl_4 prior to use to remove grease.
Do not grease stopcock.
- b. Yield is usually 70-80 percent.

Procedure for Separation of Tungsten
From Neutron Activated Environmental
and Biological Samples

Summary of Method

Tungsten is separated by formation of the thiocyanate complex and extraction into ethyl acetate. The complex is decomposed and the tungsten is precipitated as WO_3 for yield determination and radioassay. The time required to perform a single analysis is about one hour. When multiple samples are analyzed, about 10-15 determinations can be performed per day.

Reagents

Tungsten carrier (10 mg/ml) - Dissolve 17.94 grams of $\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$ in distilled water and dilute to 1.0 liter. Standardize the carrier in triplicate, as follows: Pipette 5.0 ml aliquots of carrier into 125 ml erlenmeyer flasks. Add 5 ml of conc HNO_3 and digest on a steam bath for 4 hours. Filter WO_3 precipitate using Whatman #42 filter paper. Ignite for one hour at 850 C in a muffle furnace. Cool in a desiccator and weigh. Repeat the ignition, cooling, and weighing until a constant weight is obtained (± 0.1 mg). Calculate mg W/ml as follows:

$$\text{mg W/ml} = \frac{(\text{Wt mg} \cdot \text{WO}_3) (0.7930)}{5}$$

12M NaOH - Dissolve 480 grams of NaOH in distilled water and dilute to one liter.

6M HCl - Dilute 515 ml of conc HCl to one liter with distilled water.

$\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ - Solid

NH_4SCN - Solid

Ethyl acetate

H_2O_2 - 30%

Procedure⁽⁵⁾

1. To a 0.5-1 gram sample of irradiated ash in a 40 ml centrifuge cone, add 2.0 ml of tungsten carrier and 5 ml of 12 M NaOH. Digest for 15 minutes in a hot water bath.
2. Dilute to 20 ml with distilled water and centrifuge. Transfer supernatant liquid to a 100 ml beaker. Discard residue. Neutralize the solution with conc HCl, then add 4 ml of conc HCl in excess.

3. Add 0.4 grams of solid $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ and 1.0 gram of solid NH_4SCN . Heat on hot plate for 5 minutes to form bright green tungsten complex.
4. Cool solution and transfer to a separatory funnel and extract once with 30 ml of ethyl acetate by shaking for one minute.
5. Decant the organic phase and evaporate to dryness. Caution: Ethyl acetate is flammable.
6. Dissolve residue in 10 ml of 6M HCl with stirring. Add 5 drops of 30% H_2O_2 and 0.5 ml of HNO_3 . Evaporate to near dryness (1-2 ml)
7. Add 10 ml of 6M HCl and stir.
8. Filter yellow WO_3 precipitate through a tared fiberglass filter disc and wash twice with 5 ml aliquots of 6M HCl. Dry by wash with 2-5 ml aliquots of ethanol.
9. Dry filter and precipitate for 10 minutes at 100 C and weigh to obtain yield^(Note a). Repeat drying and weighing until a constant weight is obtained (± 0.1 mg).
10. Radioassay for ^{182}W by measurement of 0.48 mev gamma ray^(Note b).

Notes:

- a. The yield is usually about 30-50%.
- b. No radioactive contaminants were observed in tungsten samples separated from a wide variety of plant, animal and fish ash samples.

Procedure for Separation of Cerium
From Neutron Activated Environmental
And Biological Samples

Summary of Method

Cerium is separated by extraction with di(2-ethylhexyl)phosphoric acid

(HDEHP). After back extraction, the cerium is precipitated as the hydroxide and finally as the oxalate for yield determination and radioassay. About 45 minutes are required to complete a single analysis. Up to about 15 samples have been separated in one day.

Reagents

Cerium carrier (10 mg/ml) - Dissolve 31 grams of cerous nitrate $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ in 1.0 liter of water. Standardize in triplicate using the following procedure. Pipette 5.0 ml of cerium carrier into a 40 ml glass centrifuge cone. Add 20 ml of distilled water, heat to boiling, and add 15 ml of saturated oxalic acid with stirring. Cool in an ice bath for 10 minutes and filter through Whatman #42 filter paper. Wash the precipitate with 5 ml of distilled water and three 5 ml aliquots of ethanol. Transfer the paper and precipitate and ignite at 700 C in a muffle furnace for one hour. Cool the crucible in a desiccator and weigh. Repeat the ignition, cooling and weighing until a constant weight is obtained (± 0.1 mg). Calculate the concentration of the carrier as follows:

$$\text{mg Ce/ml} = \frac{(\text{Mg CeO}_2) (0.8141)}{5}$$

10 M HNO_3 - dilute 650 ml of conc HNO_3 to 1 liter with distilled water.

10M HNO_3 - KBrO_3 solution - Dilute 65 ml of conc HNO_3 to 100 ml with distilled water. Cool and add 16.7 grams of KBrO_3 . Stir to dissolve. This solution must be prepared just prior to use.

10M HNO_3 - H_2O_2 solution - Dilute 65 ml of conc HNO_3 to 100 ml with distilled water. Cool and add 3 ml of 30% H_2O_2 . This solution must be prepared just prior to use.

KBrO₃ - Solid

0.75 M HDEHP - heptane - Dilute 250 ml of HDEHP to 1 liter with n-heptane.

6 w/o Sodium bisulfite solution - Dissolve 6 grams of sodium bisulfite in 100 ml of distilled water.

19 M NaOH - Dissolve 760 grams of NaOH pellets in distilled water and dilute to 1 liter. Cool and store in polyethylene bottle.

6M HCl - Dilute 515 ml of conc HCl to 1 liter.

Ethanol, absolute

Conc Ammonium hydroxide

Saturated oxalic acid solution - Dissolve about 150 grams of oxalic acid in 1 liter of distilled water. Solid oxalic acid should remain in the bottom of the container. If it does not, add more oxalic acid.

30% Hydrogen peroxide

Procedure⁽⁶⁾

1. To a one gram ash sample in a 40 ml glass centrifuge cone, add 2.0 ml of cerium carrier and 20 ml of 10 M HNO₃. Digest on a hot water bath for 15 minutes. Cool, centrifuge, and decant the supernatant liquid into a 60 ml separatory funnel equipped with a stirrer. Discard residue.
2. Add 3.3 grams of solid KBrO₃ and dissolve by stirring. Add 20 ml of HDEHP-heptane and extract for 3 minutes.
3. Withdraw aqueous phase and discard. Wash organic phase twice with 10 ml portions of HNO₃-KBrO₃ solution. Discard wash solutions.
4. Add 20 ml of HNO₃-H₂O₂ solution and extract for 10 minutes.
5. Transfer aqueous phase to a 40 ml glass centrifuge cone. Discard organic phase. Precipitate Ce(OH)₃ by addition of 19 M NaOH solution. Add 1 ml in excess. Cool, centrifuge, and discard supernatant liquid.

6. Dissolve the precipitate in 1 ml of 6 M HCl and 1-2 drops of 30% H_2O_2 . Dilute to 10 ml and add 6 w/o sodium bisulfite until decolorization of the solution occurs. Add an excess of NH_3OH to precipitate the $\text{Ce}(\text{OH})_3$. Centrifuge and discard supernatant liquid.
7. Dissolve the precipitate in 1 ml of 6M HCl and 1-2 drops of 30% H_2O_2 . Dilute to 15 ml, heat to boiling, and add 15 ml of saturated oxalic acid with stirring. Cool in an ice bath.
8. Filter precipitate through a tared fiberglass filter disc. Wash with 10 ml of hot water, 5 ml of cold water, and 3-5 ml portions of ethanol.
9. Dry filter and precipitate for 10 minutes at 100 C and weigh. (Note a) Repeat drying and weighing until a constant weight is obtained (± 0.1 mg).
10. Radioassay for Ce^{141} by measurement of the 0.145 mev gamma ray (Note b).

Notes:

- a. Yield is usually about 80 percent.
- b. No radioactive contaminants were observed in the analysis of a wide variety of plant, animal and fish samples.

References

1. Butler, F. E. "Sr⁹⁰ Monitoring at the Savannah River Plant", DP-473 (1960).
2. Geiger, E. L. "Strontium Trace Method for Determination of Strontium-90", Health Physics 13, 607-12 (1967).
3. Zumwalt, L. R. "Handbook of Radiochemical Techniques", Vol I. Radiochemical Techniques NP-5057 (1953).
4. Arden, J. W. "The Determination of Radioruthenium", AERE-AM-68.
5. Fukai, R., and Meinke, W. W. "Activation Analysis of Vanadium, Arsenic, Molybdenum, Tungsten, Rhenium and Gold in Marine Organisms", Limnology and Oceanology 7, 186-200 (1962).
6. Healy, T. V. "Rapid Solvent Extraction Methods for Fission Product Separation and Analysis", Radiochemical Acta 2, 52-7 (1963).