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CHARACTERIZATION OF ACTINIDE PHYSICS SPECIMENS FOR THE US/UK JOINT EXPERIMENT IN THE DOUNREAY PROTOTYPE FAST REACTOR

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ABSTRACT

The United States and the United Kingdom are engaged in a joint research program in which samples of the higher actinides are irradiated in the Dounreay Prototype Fast Reactor in Scotland. The purpose of the program is (1) to study the materials behavior of selected higher actinide "fuels" and (2) to determine the integral cross sections of a wide variety of the higher actinide isotopes. Samples of the actinides are incorporated in fuel pins inserted in the core. For the fuel study, the actinides selected are Am-241 and Cm-244 in the form of Am_2O_3, Cm_2O_3, and $Am_6Cm(RE)_7O_{21}$, where (RE) represents a mixture of lanthanides. For the cross-section determinations, the samples are milligram quantities of actinide oxides of Cm-248, Cm-246, Cm-244, Cm-243, Am-243, Am-241, Pu-244, Pu-242, Pu-241, Pu-240, Pu-239, Pu-238, Np-237, U-238, U-236, U-235, U-234, U-233, Th-232, Th-230, and Pa-231 encapsulated in vanadium. Coincident with the irradiations, neutron flux and energy spectral measurements are made with vanadium-encapsulated dosimeter materials located within the same fuel pins. The development and application of the technology for preparing the actinide samples have been described in a previous report by T. C. Quinby et al.[1] This report characterizes the starting materials used in the samples for the cross-section determinations (denoted as physics specimens) and in the dosimeters. The isotopic composition and concentrations in the starting materials were determined by mass spectrometry and/or radiometric methods, and the impurities contained in the materials were determined by spark source mass spectrometry.

I. INTRODUCTION

Following earlier discussions, Mr. C. W. Blumfield (Director, Dounreay Experimental Reactor Establishment, Thurso, Caithness, Scotland) wrote in November 1977 to Dr. G. W. Cunningham (then Acting Program Director for Nuclear Energy, Department of Energy, Washington, D. C.) proposing a joint experiment on the irradiation of higher actinides in the Dounreay Prototype Fast Reactor (PFR) at Dounreay, Scotland (United Kingdom). Dr. Cunningham agreed to set up a technical meeting to draft a detailed proposal. This meeting was held at the Department of Energy (DOE), Headquarters, Germantown, Maryland, in March 1978 and was attended by K. M. Swanson and A. L. Mills (UK, Dounreay); P. B. Hemming, J. Lewellen, S. Whetstone, and R. D. Walton (DOE); S. Raman, J. E. Bigelow, and M. L. Williams (Oak Ridge National Laboratory); E. T. Weber and W. Little (Hanford Engineering Development Laboratory); S. Beamans and C. Cowan (General Electric Company); W. Maeck and W. Emel (Idaho National Engineering Laboratory); and C. Johnson (Argonne National Laboratory). At this meeting a proposal for an experiment was drafted that was subsequently the basis of the "Higher Actinides Agreement" signed in May 1979 as part of the long-term cooperation in the field of Liquid Metal Fast Breeder Reactor (LMFBR) technology between the United States and the United Kingdom under the LMFBR agreement of 1976.

The original proposal from the UK (presented by Mills and Swanson) was primarily aimed at a preliminary evaluation of the materials behavior of higher actinides present in the fuel pins of an operating reactor. Gram quantities of the selected actinides were to be prepared in the form of fuel pellets[1] and incorporated in special fuel pins for irradiation in the PFR core. During the March 1978 meeting, Raman (ORNL) proposed an additional experiment in which milligram quantities of several other actinides (physics specimens) would be irradiated simultaneously with the actinide fuel samples in order to improve our knowledge of basic nuclear cross sections and reaction rates. Both experiments were subsequently incorporated into the Higher Actinides Agreement.

Major responsibilities for implementing this agreement were assigned to HEDL (E. T. Weber) and ORNL (S. Raman) by Dr. P. B. Hemmig, Chief, Physics Section, DOE Office of Reactor Research and Technology. ORNL was responsible for selecting and providing the isotopes, for fabricating the fuel pellets, for fabricating the ORNL and HEDL dosimeters and ORNL physics samples, for loading and seal-welding the pin segments (with HEDL assistance) and for pre- and post-irradiation analysis of the physics specimens and dosimeter materials. HEDL was responsible for test pin design, pin fabrication, final assembly of pin sections, quality assurance, shipping to and from the UK, and post-irradiation analysis of HEDL dosimeters.

The US/UK Higher Actinide Irradiation Test consists of four fuel pins (FP) to be irradiated in the Dounreay PFR. The fuel pin configuration is shown in Fig. 1. The tentative irradiation scheme based on full power days is as shown in Table 1. Pins FP1 and FP2 are identical; pins FP3 and FP 4 are modified versions of FP1 and FP2 which were designed to permit extended irradiation periods, provided several other conditions are also met (for example, safety, reactor space availability, irradiation rig availability, etc.). The engineering test plan and fuel pin designs are contained in References 2 and 3. This report characterizes the starting materials used for the physics specimens and for the dosimeters. The starting materials used in the actinide fuel samples have been described earlier.[1]

Table 1. Tentative Irradiation Scheme for US/UK Higher Actinide Irradiation Test

Pin	Bottom Section	Top Section	Irradiation Time
FP1	Physics	Fuel	90 full power days
FP2	Physics	Fuel	180 full power days
FP3		Fuel	>180 full power days
FP4	Physics		To be determined

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HIGHER ACTINIDES PIN FOR PFR IRRADIATION

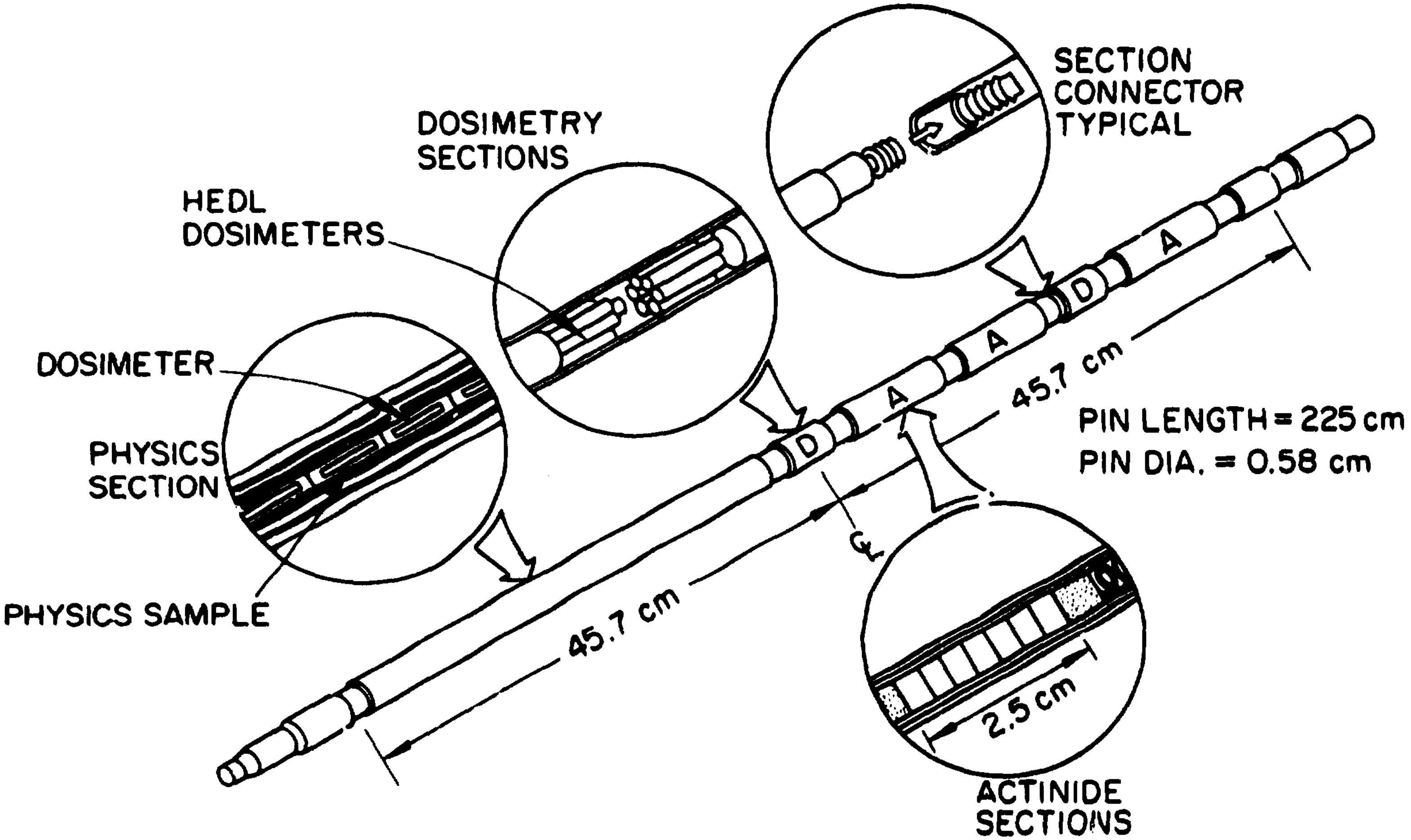

Fig. 1 Higher Actinides Pin for PFR Irradiation

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II. PHYSICS SPECIMEN CHARACTERIZATION

A. Selection of Actinide Materials

A total of 21 actinide materials were selected for use as physics specimen materials for the US/UK actinide experiment. Criteria used in selecting the materials included availability, isotopic enrichment, and chemical purity. The physics specimen materials chosen are shown in Table 2.

Table 2. Physics Specimen Materials Selected for US/UK Higher Actinide Experiment

Isotope	Batch Number	Position in Fuel Pins 1, 2, 4	Enrichment (Wt. %)
Cm-248	1117	4	92.15
Cm-246	C59SHIP	5,6	66.53
Np-237	24HP	7	99.99
Cm-244	C57CM46	8,9	92.35
Cm-243	1011	10	57.67
U-238	Q1	11	99.955
Am-243	HIP1018	12,13	99.987
Am-241	79AMB4	14,15	99.995
Pu-242	290A	16	97.96
Pu-244	297-C	20	87.69
Pu-240	HIP1068	21,22	99.86
Pu-239	453-BO	23	99.10
Pu-241	307-A	24	96.77
Th-232	4151	25	100.00
U-236	201DMR	26	88.96
U-234	M9	27	99.774
U-235	264C	28	99.89
Pa-231	Pa-F-1	29	100.00
Pu-238	06HP014	30	99.38
Th-230	256A	31	89.39
U-233	240A	32	99.886

B. Selection of Dosimeter Materials

Accurate characterization of the irradiated physics specimens requires that the neutron energy spectra, flux and fluence to which the physics specimens are exposed be accurately determined. The six neutron dosimeter materials used for this purpose are shown in Table 3.

Table 3. Dosimeter Materials Selected for Use With Physics Specimens in US/UK Higher Actinide Experiment

Isotope	Batch Number	Position in Fuel Pins 1, 2, 4	Isotope Enrichment (Wt. %)
U-235	264C	1,17,33	99.89
U-238	ESZ	1,17,33	99.999
Pu-239	453-B0	2,18,34	99.10
Np-237	24HP	2,18,34	99.99
Cu	N	3,19,35	Normal
Co	Co-1	3,19,35	Normal

C. Physics Specimens and Dosimeter Encapsulation and Loading

1. Encapsulation

All physics and dosimeter materials were encapsulated in vanadium as described in the report by Quinby.[1] The dot code system for the capsules is used for identification after irradiation.

2. Loading

The physics specimens and dosimeters were loaded into three fuel pins according to the arrangement shown in Table 4. The vanadium-encapsulated materials were loaded into each fuel pin in five sections as shown in Fig. 2.

D. Characterization of Physics Specimen and Dosimeter Materials

All encapsulated isotope materials have been characterized by mass spectrometry and/or radiometry for isotopic composition and concentration analyses. Impurities in these materials have been determined by

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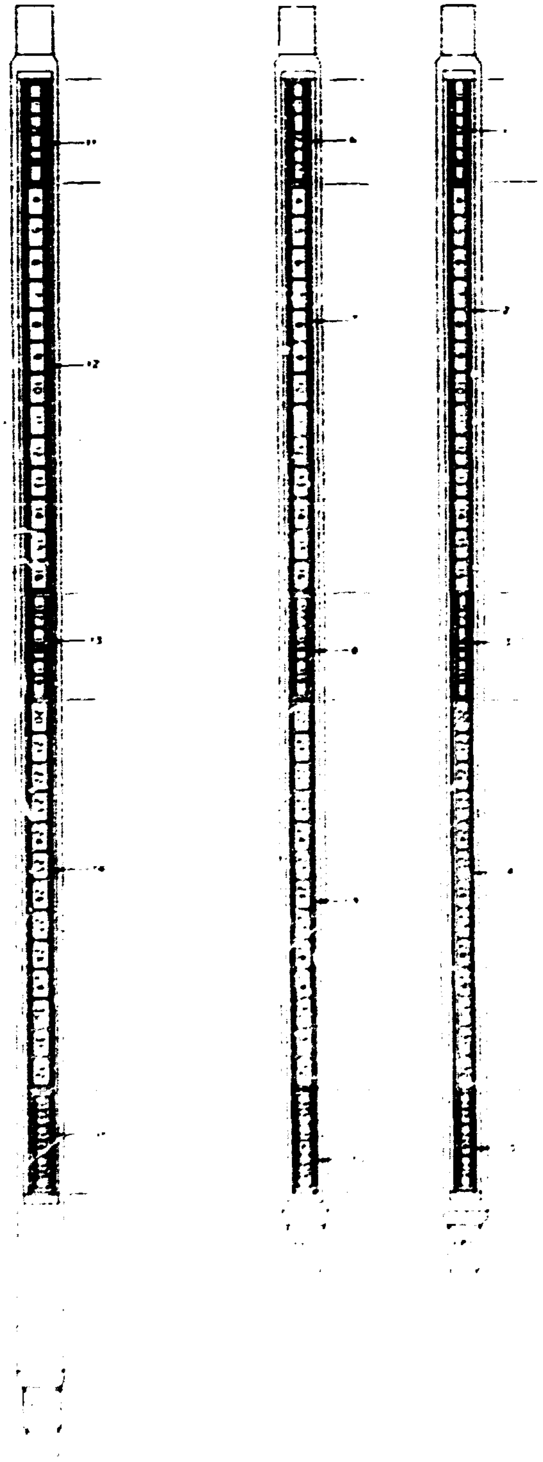

Fig. 2 Sample Locations for Physics Specimens and Dosimeter Capsules in Fuel Pins

Table 4. Vertical Ordering of Physics Specimens and Dosimeter Capsules for FP1, FP2, and FP4

Position	Isotope
1	Dos (U-235, U-238)
2	Dos (Pu-239, Np-237)
3	Dos (Cu, Co)
4	Cm-248
5	Cm-246
6	Cm-246
7	Np-237
8	Cm-244
9	Cm-244
10	Cm-243
11	U-238
12	Am-243
13	Am-243
14	Am-241
15	Am-241
16	Pu-242
17	Dos (U-235, U-238)
18	Dos (Pu-239, Np-237)
19	Dos (Cu, Co)
20	Pu-244
21	Pu-240
22	Pu-240
23	Pu-239
24	Pu-241
25	Th-232
26	U-236
27	U-234
28	U-235
29	Pa-231
30	Pu-238
31	Th-230
32	U-233
33	Dos (U-235, U-238)
34	Dos (Pu-239, Np-237)
35	Dos (Cu, Co)

spark source mass spectrometry (SSMS). These analyses are necessary in interpreting the analytical data to be obtained upon post-irradiation examination. The quantities of material encapsulated in each physics specimen and in each dosimeter, together with the isotopic and concentration analyses, are shown in Table 5 for capsules loaded into fuel pins 1, 2, and 4 (FP-1, FP-2, and FP-4). Complete analytical data for all physics specimens and dosimeters are given in Appendix 1 and Appendix 2, respectively.

Table 5. Analytical Data for Physics Specimens and Dosimeter Materials Used in US/UK Experiment

Fuel Pin Position	Dot Code	Capsule Material	FP-1 Compound Wt. (mg)	FP-1 Isotope Wt. (mg)	FP-2 Compound Wt. (mg)	FP-2 Isotope Wt. (mg)	FP-4 Compound Wt. (mg)	FP-4 Isotope Wt. (mg)
1	.	Dos(U-235)	1.245	1.094	1.296	1.139	1.339	1.177
		Dos(U-238)	1.154	1.013	1.314	1.153	1.345	1.180
2	..	Dos(Pu-239)	1.451	1.243	1.406	1.204	1.396	1.196
		Dos(Np-237)	1.300	1.136	1.229	1.074	1.194	1.043
3	:	Dos(Cu)	1.231	1.231	1.405	1.405	1.353	1.353
		Dos(Co)	1.300	0.00160	1.309	0.00161	1.221	0.00150
4	˙.	Cm-248	2.051	1.705	2.120	1.762	2.124	1.765
5	.˙	Cm-246	11.488	6.657	11.748	6.808	11.547	6.691
6	:	Cm-246	11.284	6.539	11.317	6.558	11.730	6.797
7	˙.	Np-237	13.674	11.950	13.903	12.150	13.979	12.216
8	.˙	Cm-244	11.108	8.154	11.249	8.258	11.337	8.322
9	:˙	Cm-244	11.303	8.297	11.442	8.399	11.239	8.250

Table 5 - Continued

Fuel Pin Position	Dot Code	Capsule Material	FP-1 Compound Wt. (mg)	FP-1 Isotope Wt. (mg)	FP-2 Compound Wt. (mg)	FP-2 Isotope Wt. (mg)	FP-4 Compound Wt. (mg)	FP-4 Isotope Wt. (mg)
10	⠙	Cm-243	1.010	0.336	1.135	0.377	1.180	0.392
11	⠓	U-238	12.108	9.859	12.692	10.334	12.048	9.810
12	⠚	Am-243	11.121	9.804	11.491	10.130	11.152	9.831
13	⠇	Am-243	11.275	9.940	11.347	10.003	11.118	9.801
14	⠣	Am-241	12.046	10.383	11.252	9.699	10.931	9.422
15	⠱	Am-241	11.081	9.551	10.892	9.388	11.051	9.525
16	⠥	Pu-242	2.470	2.088	2.470	2.088	2.421	2.046
17	⠍	Dos(U-235)	1.193	1.048	1.338	1.176	1.259	1.106
		Dos(U-238)	1.286	1.128	1.234	1.083	1.119	0.982
18	⠩	Dos(Pu-239)	1.387	1.188	1.201	1.029	1.331	1.406
		Dos(Np-237)	1.286	1.124	1.027	0.898	1.286	1.124
19	⠬	Dos(Cu)	1.056	1.056	1.388	1.388	1.406	1.406
		Dos(Co)	1.378	0.00169	1.249	0.00154	1.279	0.00157

Table 5 - Continued

Fuel Pin Position	Dot Code	Capsule Material	FP-1		FP-2		FP-4	
			Compound Wt. (mg)	Isotope Wt. (mg)	Compound Wt. (mg)	Isotope Wt. (mg)	Compound Wt. (mg)	Isotope Wt. (mg)
20	⠕	Pu-244	2.716	2.089	2.784	2.141	2.741	2.108
21	⠪	Pu-240	13.906	10.782	13.652	10.585	13.980	10.839
22	⠜	Pu-240	13.590	10.537	13.738	10.651	13.951	10.816
23	⠎	Pu-239	9.330	7.990	9.251	7.923	9.890	8.470
24	⠶	Pu-241	5.705	4.096	5.621	4.036	5.920	4.251
25	⠭	Th-232	20.252	17.771	20.382	17.885	21.516	18.880
26	⠫	U-236	12.173	7.906	12.195	7.921	12.258	7.962
27	⠝	U-234	4.792	3.442	4.558	3.274	4.873	3.500
28	⠮	U-235	9.708	8.531	9.232	8.112	9.528	8.373
29	⠵	Pa-231	3.615	2.885	3.249	2.593	3.148	2.512
30	⠗	Pu-238	3.504	2.912	3.939	3.273	3.740	3.108

Table 5 - Continued

Fuel Pin Position	Dot Code	Capsule Material	FP-1 Compound Wt. (mg)	FP-1 Isotope Wt. (mg)	FP-2 Compound Wt. (mg)	FP-2 Isotope Wt. (mg)	FP-4 Compound Wt. (mg)	FP-4 Isotope Wt. (mg)
31	⠺	Th-230	3.973	2.921	3.986	2.931	3.921	2.883
32	⠳	U-233	9.939	7.920	9.759	7.777	9.602	7.652
33	⠞	Dos(U-235)	1.218	1.070	1.316	1.156	1.182	1.039
		Dos(U-238)	1.149	1.008	1.103	0.968	1.224	1.074
34	⠹	Dos(Pu-239)	1.407	1.205	1.179	1.010	1.383	1.184
		Dos(Np-237)	1.290	1.127	1.100	0.961	1.355	1.184
35	⠏	Dos(Cu)	1.358	1.358	1.247	1.247	1.433	1.433
		Dos(Co)	1.337	0.00164	1.169	0.00144	1.159	0.00143

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III. METHODS USED FOR ANALYSIS OF PHYSICS SPECIMENS FOR US/UK EXPERIMENT

A. Introduction

Because of the limited amounts of material available for the irradiation, very sensitive methods for determining the various nuclides were used. A combination of high sensitivity mass spectrometry and radiometric methods meet these analytical requirements. Isotope dilution mass spectrometry (IDMS) techniques were used to measure the elemental concentrations when practical and when high-purity isotope spikes were available. Isotopic compositions of all specimens, other than mononuclidic, were measured using the high sensitivity instruments in the Analytical Chemistry Division's Mass Spectrometry Section. Radiometric analyses were required to determine elemental concentrations of specimens containing Pa, Np, Am, Cm, Pu-238, and U-232. These measurements were made in the Analytical Chemistry Division's Transuranium Laboratory.

B. Description of Mass Spectrometer Used for Actinide Analysis

A double magnetic deflection mass spectrometer with ion counting was used for isotopic and concentration analyses of actinides.[4] Ionization is achieved by the thermal emission method using zone-refined rhenium for the filament material. This instrument was built primarily for high alpha-emitting actinides. Precautions to prevent contamination are required because of the radioactivity. The electronic console and the spectrometer are shown in Figs. 3 and 4. A schematic drawing of the electronic system is given in Fig. 5. The room where the instrument is located is at negative pressure with respect to the adjacent room which houses the electronic consoles to minimize contamination in the event of an accident.

The mass spectrometer is the "KAPL" type instrument since it was originally described by workers at the Knolls Atomic Power Laboratory.[5] The ions are magnetically deflected through 90° on a 30-cm radius. Ions are detected with an electron multiplier, amplified, and stored as pulses in a PDP-11/10 computer. A slit midway between the

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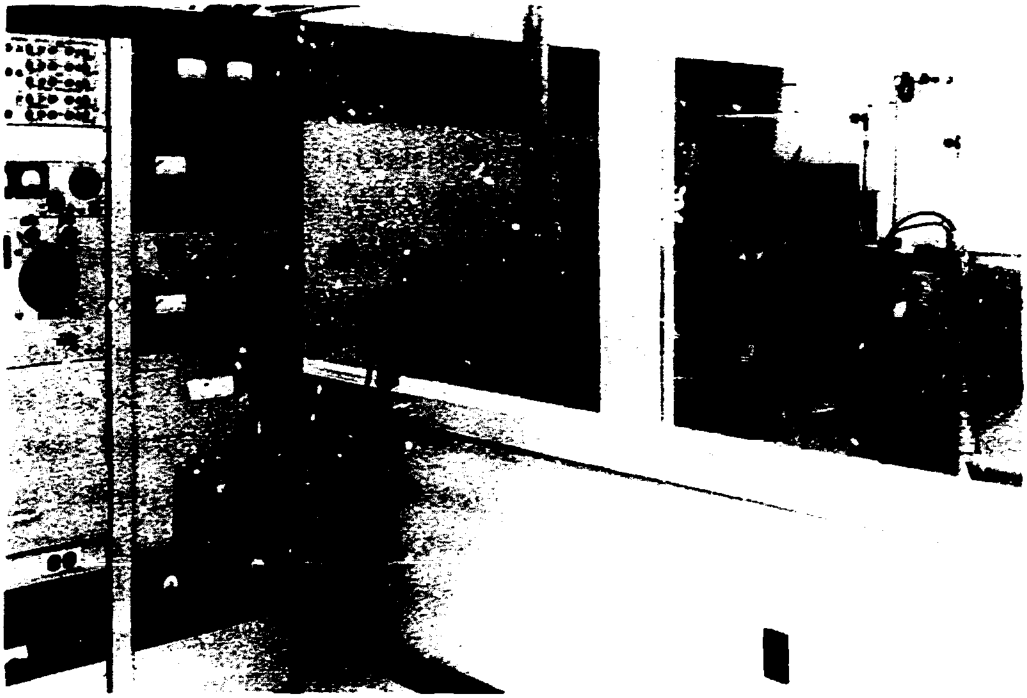

Fig. 3 Mass Spectrometer for Analysis of Actinides

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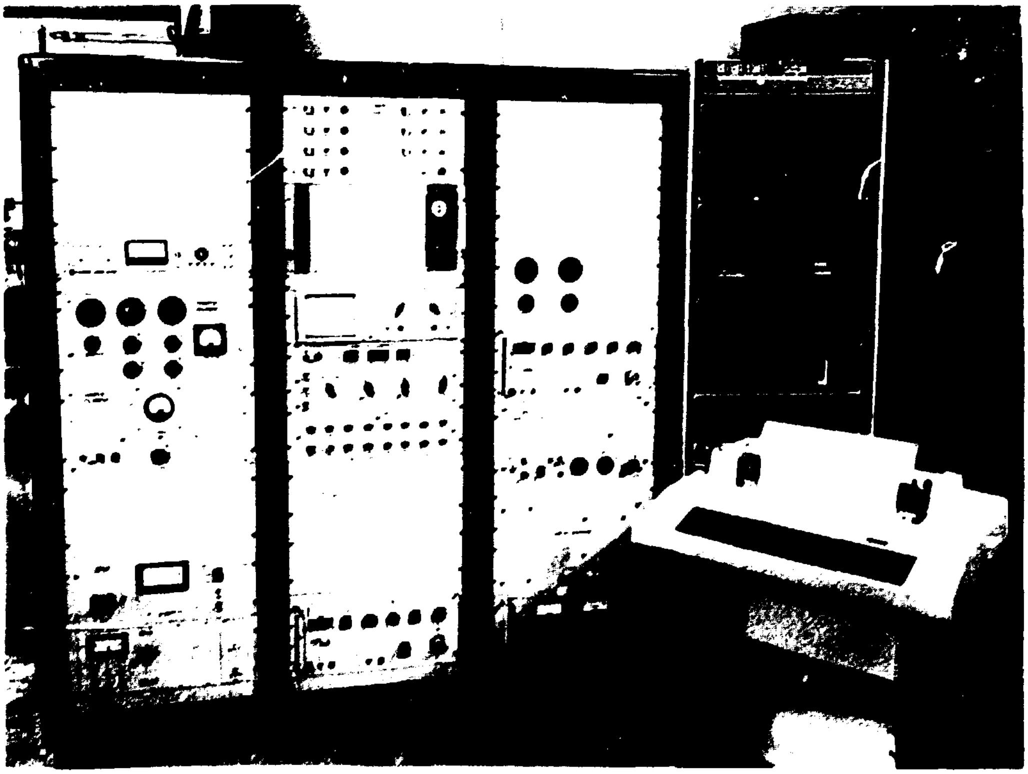

Fig. 4 Electronic Console for Mass Spectrometer

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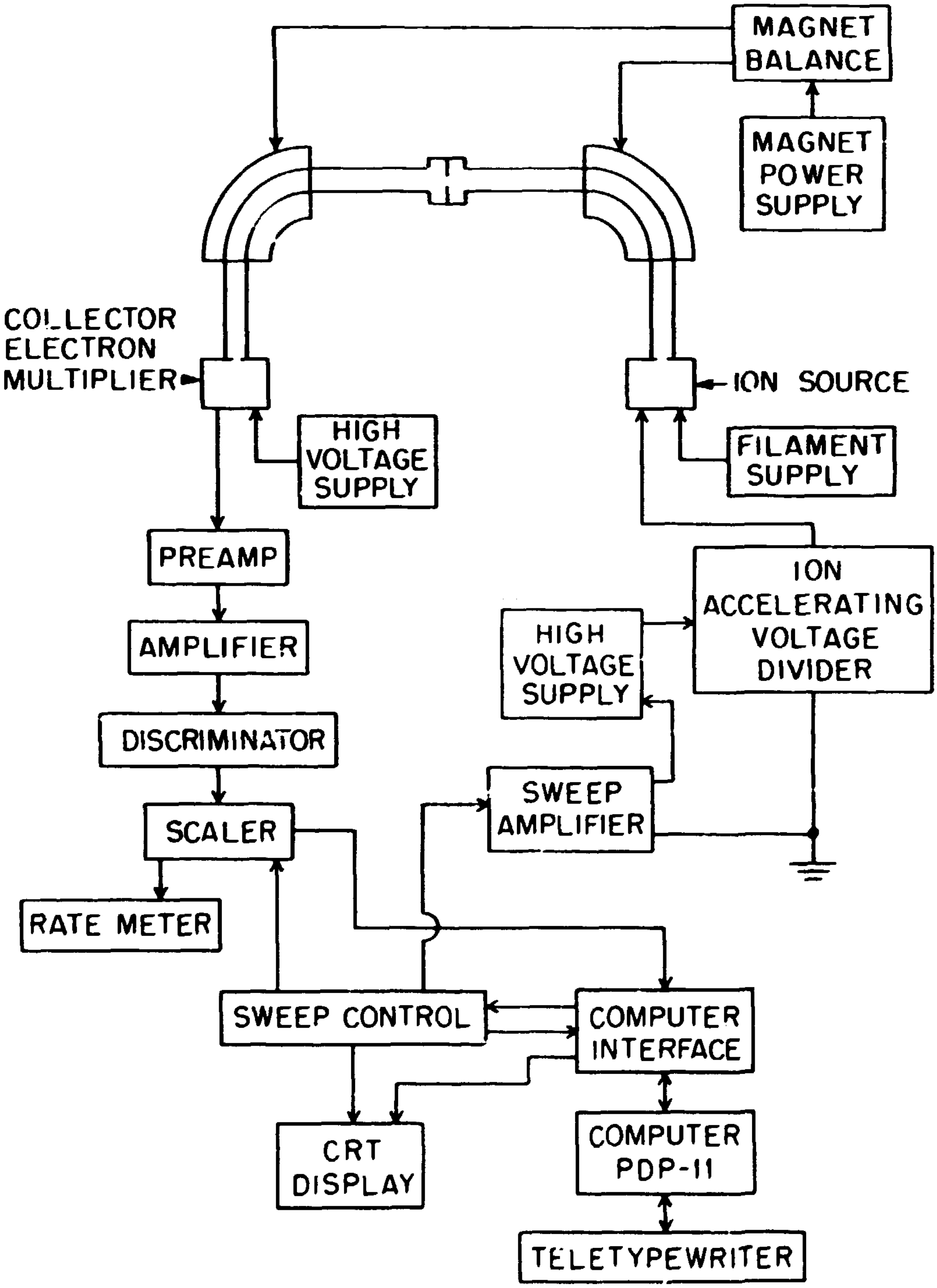

Fig. 5 Block Diagram of Electronic Equipment

magnets allows only a single mass to be admitted to the second stage. This results in a spectrum at high mass which is very clean and free from scattered ions. The magnetic field is approximately 8000 gauss across a gap of 14.5 mm. The accelerating voltage for this field and the mass region for the actinides is approximately 8 kV. The vacuum system is all metal except for the glass dome on the source and is evacuated with ion pumps.

The preparation of samples for loading into the instrument is done using the resin-bead technique.[6,7] For elements which adsorb on anion resin (Th, Pu, and U), a solution of the element, adjusted to 8 <u>M</u> in HNO_3, is contacted with a small number of resin beads. These are washed with 8 <u>M</u> HNO_3 and a single resin bead is isolated and transferred to the single, zone-refined rhenium filament for isotopic analysis. Tri-valent elements which do not adsorb on anion resin under these conditions are purified by solvent extraction and loaded from a weak acid solution onto cation beads (these include Am and Cm). There are several advantages of the resin bead loading technique: 1) better control of radioactivity in the mass spectrometer and sample introduction area, 2) improved ion optics from the point source, and 3) greater ionization efficiency which allows for more stable ion signals and, hence, better precision of measurement.

Mass analysis procedures using the resin bead loading technique and the instrument described above for the specimen are very similar. For any actinide, spectrum scanning and data recording consist of taking the horizontal sweep voltage from a sweep controller and applying it in series with the accelerating voltage. The sweep voltage is a stair-step function which successively gates the memory channels of the sweep controller. The voltage is thus swept in synchronism with the channel gating so that counts occurring at a given voltage are stored in the same channel. Any mass in the spectrum can be scanned any predetermined number of times for each traversal of the entire mass range. This allows the scanning scheme to be optimized for the requirements of a given actinide. A scanning scheme is chosen for a given analysis so that the time required for one run is 80-90 seconds. This length of time is a compromise between the long period that the best precision would require and the realities of sample through-put requirements. A typical scanning scheme used for uranium is shown in Table 6.

Table 6. A Typical Mass Scanning Scheme for Uranium Analysis

Subgroup	1	2	3	4	5	6	7	8
Mass	231.5	233	234	235	236	236.5	238	239
No. of sweeps	4	1	4	2	4	4	1	1

The half mass positions are swept to obtain an estimate of the background noise in the system. The number of counts in these positions is subtracted from the counts in all other subgroups. This correction is normally quite small (<1 ppm) since the electronic noise in the counting system is normally a few counts per minute. For an analysis, ten runs are recorded and computer calculations made with software written in the Mass Spectrometry Section.

For any particular analysis, the counting rate for the major isotope is kept at 2×10^5 to 3×10^5 sec^{-1} to avoid a high "count loss" correction. A bias correction is also required because the accelerating voltage is swept and fractionation effects are inherent in thermal ionization methods. The count loss or system dead-time correction and bias correction are determined by measuring the composition of NBS SRM U500 standard. From the observed ratios are calculated the bias correction required per mass unit and the system dead time. Since the elements being measured do not differ drastically in mass from uranium, the corrections are assumed to apply. After the corrections are established, analyses of SRM's are carried out for quality control purposes.

C. Description of Radiometric Methods Used for Actinide Analysis

1. Plutonium-238

Plutonium-238, with a half-life of 87 y, comprises a very small fraction of the material in these samples and cannot usually be analyzed by mass spectrometry. A weighed portion of the sample is dissolved in

8 M HNO_3-0.05 M HF under reflux and is diluted to a specific volume. Aliquots of this solution are diluted to 1 M HNO_3, and the plutonium valence is adjusted to the tetravalent state with hydroxylamine hydrochloride and sodium nitrite. Residual fluoride is complexed with aluminum. Pu^{+4} is extracted into 0.5 M thenoyltrifluoroacetone (TTA) in xylene. Aliquots of the organic phase are evaporated onto polished stainless steel planchets for counting the alpha activity on a gas flow proportional (2-π geometry) counter. The alpha pulse-height distribution on these plates is measured on a surface barrier detector coupled with a multichannel analyzer. Enough counts are collected to give at least 1000 counts in the 5.49-MeV peak of Pu-238. The total alpha count, percent of Pu-238 alpha, sample weight, and dilution factor are used to calculate the Pu-238 concentration.

2. Americium and Curium

A weighed aliquot of sample is dissolved with heating in 8 M HNO_3 and diluted to a specific volume. Aliquots of this solution (or of appropriate dilutions) are evaporated onto stainless steel planchets, and the gross alpha activity is measured by gas flow proportional counting. Alpha pulse-height counting gives the ratios of Am-243 (5.27 MeV), Am-241 (5.48 MeV), and Cm-244 (5.81 MeV). The amount of each nuclide can be calculated from the gross alpha activity and their respective specific activities. Measurement of the 60-keV gamma from Am-241 on a Ge(Li) detector coupled with a multichannel analyzer gives a further check on the Am-241 content.

3. Neptunium-237

Aliquots of the dissolved sample are spiked with known amounts of Np-239 tracer and are treated with $K_2Cr_2O_7$ to oxidize neptunium (and plutonium) to the hexavalent state. The sample is adjusted to 2 M $Al(NO_3)_3$, and the neptunium is extracted (along with plutonium and uranium) into hexone. The neptunium and plutonium are stripped from the organic into a reducing solution containing $FeCl_2$ in 1 M HCl.

The reduced neptunium is then extracted into 0.5 M TTA while the Pu^{+3} remains in the aqueous. Aliquots of the organic are plated and counted for alpha activity as previously described. Alpha pulse-height analysis is again used to determine the amount of 4.78-MeV Np-237, as well as any plutonium carryover. To determine the efficiency of neptunium recovery, an aliquot of the TTA phase is counted for Np-239 gamma. The alpha activity of Np-237 is corrected for the recovery.

4. Protactinium-231

Samples for protactinium analysis must be kept in at least 4 M HCl solutions. An aliquot of this solution is adjusted to 6 M HCl and contacted with di-isobutyl carbinol (DIBC) to extract the protactinium. The organic phase is scrubbed with 6 M HCl, and aliquots of the DIBC are evaporated onto tantalum planchets and counted for alpha activity. Purity of the protactinium fraction (5.01 MeV) is checked by pulse-height analysis. Since the gamma tracer, Pa-233, is not readily available, recovery estimates are based on analysis of protactinium solutions of known concentration.

5. Uranium-232

Uranium is separated from other radioelements by anion exchange. Uranium in 6 M HCl is adsorbed on the resin and washed with HCl to remove Th, Am, Cm, Pu, and Np. Elution with dilute (0.01 M) HCl gives a purified uranium solution for alpha pulse-height analysis. The 5.32-MeV U-232 and 4.82-MeV U-233 alpha peaks are integrated, and the ratio of U-232 to U-233 is determined. Usually U-232 is reported as parts-per-billion in U-233.

D. Description of Spark Source Mass Spectrometer Used for Impurity Analyses of Physics Specimens and Dosimeter Materials

A spark source mass spectrometer, modified with an alpha-containment glove box around the source region, was used to determine the elemental impurities in all physics specimens and dosimeter materials for the US/UK experiment. The instrument is an Associated Electrical Industries (AEI) Model MS-702. The basic design of the instrument involves rf spark ion source and Mattauch-Herzog double focusing geometry.[8]

Ionization of the sample is achieved by an rf spark generated between two sample electrodes. Rf spark discharge is used so that material from both electrodes is included in the plasma, where temperatures are so high that all elements are vaporized and ionized with essentially equal efficiency. Ions formed in the spark are accelerated through the ion source into the analyzer section.

The electrostatic analyzer is a cylindrical condenser made of two gold plates mounted on quartz insulators. A fixed voltage is applied to these plates (±1000 V, for 20 kV accelerating potential). The electrostatic analyzer acts as an energy filter with a band pass of about 600 volts. Energy focusing is required because of the very high energy spread of ions formed in the rf spark. Ions leaving the electrostatic analyzer are essentially monoenergetic and are still homogeneous in mass-to-charge ratio.

The beam monitor (Fig. 6) is a series of plates that provides readout of the total ion current in the mass spectrometer. The plate of the monitor both defines the physical size of the ion beam and intercepts a constant fraction of it. The instantaneous ion current is measured with an electrometer whose output is integrated to register the total accumulated ion charge.

Ions that pass through the monitor slit enter the magnetic analyzer. The magnetic field and accelerating voltage are both constant so that ions entering the field are deflected in curved paths whose radii are dependent only on mass-to-charge ratio. Ions are brought to focus in a plane (photographic plate in Fig. 6), as opposed to a point as in

ORNL-DWG. 73-3117

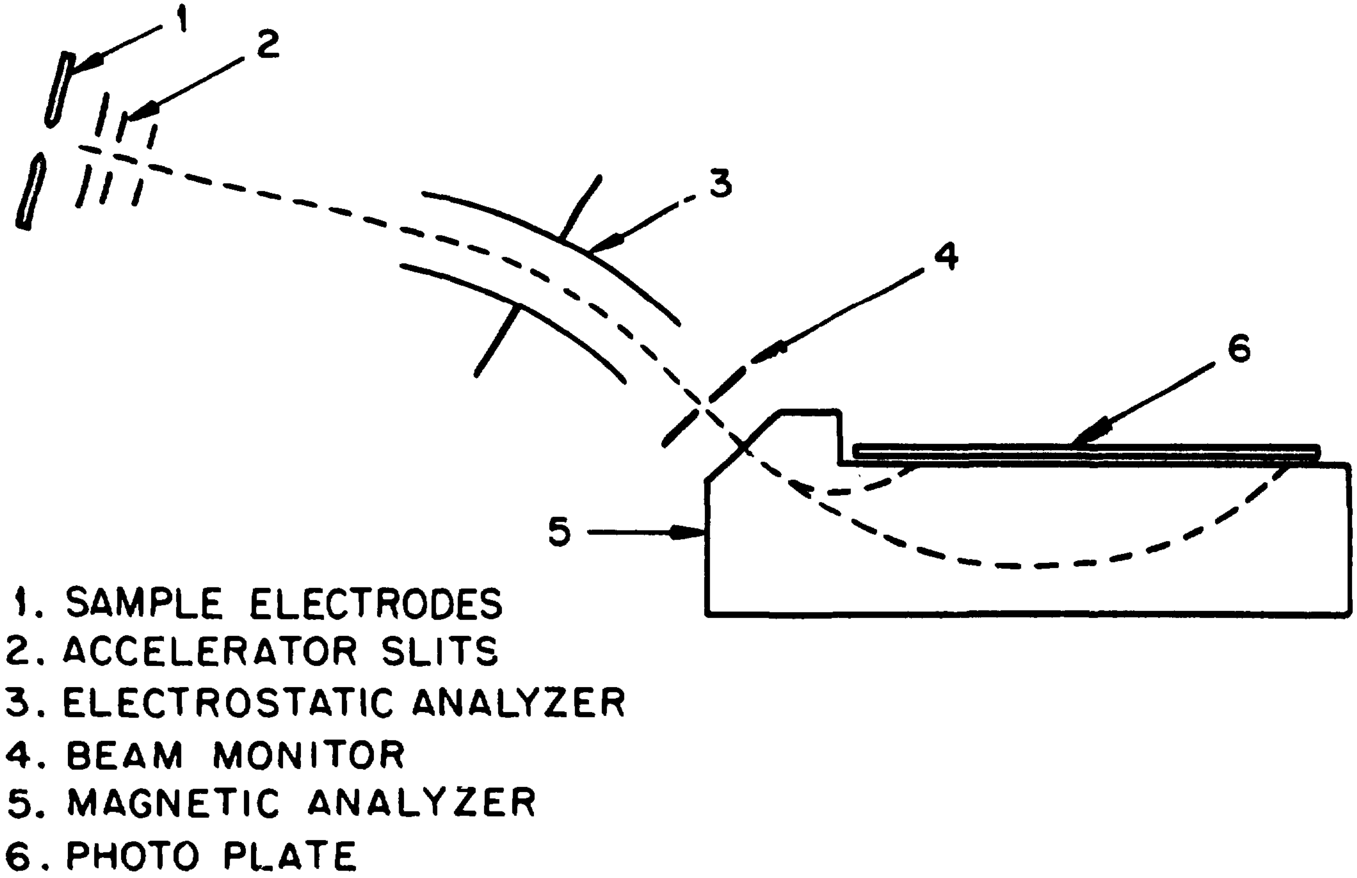

Fig. 6 Schematic Diagram of Ion Optics of AEI MS-7 Spark Source Mass Spectrometer

the Nier-Johnson configuration. A photographic plate is placed in the focal plane so that a series of ion images is formed, each image representing a single mass-to-charge ratio.

The vacuum system is arranged so that the photographic plate magazine and/or the ion source may be vented to atmosphere without disturbing the vacuum in the rest of the instrument. The system is differentially pumped to allow ion source and magazine pressures during operation to be as much as 10^4 times higher than the analyzer pressure. The ion source will produce ions at any pressure up to 10^{-1} Pa, but the analyzer system must be below 5 x 10^{-6} Pa to prevent fogging of the detector plate and to minimize charge exchange species.

The MS-702 has a glove box around the ion source housing for containment of radioactive (α emitting) samples. Figure 7 is a photograph of the mass spectrometer with the glove box in place. This system has been used for analysis of α emitters of up to 10^{10} disintegrations per minute, but is limited to 200 mr/hr or less for β and γ emitters. Samples of U, Pu, Np, Am, Cm, Cf and Es have been analyzed without release of α activity to the laboratory environment.

For satisfactory operation of the spark, the sample must be an electrically semi-conducting or conducting material. Alloys and pure metals may be analyzed by making or cutting an electrode of the sample material. Typical electrodes are cylinders 1.3 cm long, with a cross-sectional area of 0.02 cm^2. Powder samples must be pressed into the correct size and shape. The sample is mixed with high purity graphite or silver powder (99.999+% Ag) before pressing. The sample is isostatically pressed in a low density polyethylene mold for about one minute.

Aqueous solutions are prepared for analysis by adding an element or enriched isotope as an internal standard and drying the mixture of sample and standard on high purity graphite or silver electrodes.

ORNL PHOTO 97137

Fig. 7 Alpha-Containment Glove Box Around the Source Region of a Spark Source Mass Spectrometer

Spark source mass spectrometric data are obtained on a photographic plate (usually Ilford QII emulsion, although Kodak SWR and Ionomet evaporated silver halide plates are occasionally used). A photographic emulsion is a non-linear integrating detector and therefore requires calibration of its response to the number of collected ions. In normal operation, a series of graded exposures covering the range of 1 x 10^{-13} to 1 x 10^{-7} coulombs is used for calibration of the plate. This 10^6-fold dynamic range, in combination with isotopic abundance variations, can yield elemental concentration data over a range of 10^9.

There are several methods of calibrating the emulsion, such as Seidel plots, log probability plots, the so-called Hull equation, and the Churchill two-line method.[9] The Churchill method is presently in use. Each new emulsion requires determination of a new calibration curve. A data system has been designed to collect data from the photoplate, calibrate the emulsions, and determine ion intensity from spark source data. This system consists of a microphotometer, an interface, and a Digital Equipment Corporation PDP-8 computer with DEC tape bulk storage and teletype output. The microphotometer used for plate reading was originally a double beam instrument, but has been modified so that it is now a single beam photometer. It is scanned across the desired lines of the mass spectrum, and its output is digitized and stored in the core of the PDP-8 as percent transmittance. When the desired lines have been scanned, the computer is used to convert percent transmittance into ion intensity by comparison with a previously determined calibration curve. Values of ion intensities are read and stored on a disc for calculation of elemental concentrations.

A complete summary of the analytical results for each physics specimen and dosimeter material is given in Appendixes 1 and 2. These data sheets contain isotopic composition, concentration of actinide, and elemental impurities.

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APPENDIX 1

Analytical Data for Actinide Physics Specimen Materials

Explanation of Headings on Data Sheets:

Isotopic:	Method is Thermal Emission Mass Spectrometry
Impurities:	Wt. ppm equal to μg/g oxide

Isotope: Cm-248

Batch: 1117 Material: CmO_2 Position: 4

Concentration: 910 ± 45 mg Cm/g oxide

Date: 18 Jan. 82 Method: Alpha Spectrometry

Isotopic: Date of analysis: 11 Feb. 81

	244	245	246	247	248
Atom %	3.231	0.102	4.516	0.086	92.065
Std. Dev.	0.015	0.002	0.020	0.002	0.030

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: 15 Feb. 82

Element	wt ppm	Element	wt ppm
Al	30	Ni	3
B	>300	P	2
Ba	3	Pb	1
Ca	10	S	200
Co	≤1	Sb	≤3
Cr	10	Si	200
Cu	2	Sn	≤5
F	30	Ta	100
Fe	70	Th	5
K	7	Ti	≤3
La	≤2	V	3
Mg	70	W	50
Mn	3	Zn	200
Mo	10	RE's	2000
Na	10	Pu-240	2000

Isotope: Cm-246

Batch: C59SHIP Material: CmO_2 Position: 5 and 6

Concentration: 871 ± 45 mg Cm/g oxide

Date: 1 Oct. 80 Method: Alpha Spectrometry

Isotopic: Date of analysis: 5 Aug. 80

Cm	243	244	245	246	247	248
Atom %	0.043	18.43	0.234	66.51	1.84	12.95
Std. Dev.	0.004	0.05	0.002	0.10	0.02	0.04

Am	241	242	243
Atom %	2.28	0.02	97.70
Std. Dev.	0.10	0.01	0.10

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: 4 Feb. 82

Element	wt ppm	Element	wt ppm
Al	3	Ni	10
B	20	P	5
Ba	≤2	Pb	3
Ca	10	S	100
Cr	6	Si	90
Cu	3	Ta	100
F	1000	Zr	30
Fe	700	Pu-238*	2.8
K	1	Pu-240*	470
Mn	1	Am-243*	540
Na	30	Cf-252*	≤0.5

*Actinide impurities were analyzed by alpha spectrometry on 22 July 80.

Isotope: Np-237

Batch: 24HP Material: NpO_2 Position: 7

Concentration: 874 ± 26 mg Np/g oxide

Date: 29 Mar. 76 Method: Alpha Spectrometry

Isotopic: Date of analysis: 11 Aug. 72

	236	237	238	239
Atom %	<0.0005	≥ 99.99	≤ 0.003	≤ 0.004
Std. Dev.	-	-	-	-

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: Feb. 75

Element	wt ppm	Element	wt ppm
Al	2	Ni	1
As	<1	P	<1
B	<0.5	Si	3
Ba	1	Sn	2
Ca	2	S	4
Co	<1	Tc	<1
Cr	<1	V	<1
Cu	2	Zn	<1
Fe	4	Zr	<1
K	1	U-235	<1
Mg	<1	U-238	10
Mn	<1	U-239	<1
Na	<1	Cm-244	<1

Isotope: Cm-244

Batch: C57CM46 Material: CmO_2 Position: 8 and 9

Concentration: 794.9 ± 24 mg Cm/g oxide
65.1 ± 3.0 mg Pu/g oxide

Date: 26 May 81 Method: Alpha Spectrometry

Isotopic: Date of analysis: 16 June 81

Cm	243	244	245	246	247	248
Atom %	0.016*	92.40	1.162	6.26	0.101	0.057
Std. Dev.	0.002	0.03	0.004	0.03	0.002	0.001

* Analyzed by gamma-ray spectrometry.

Pu	238	239	240	241	242
Atom %	0.002	0.048	99.922	0.004	0.024
Std. Dev.	0.0005	0.001	0.010	0.001	0.001

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: 16 June 81

Element	wt ppm	Element	wt ppm
Al	20	Na	5
B	0.1	Ni	40
Ca	50	P	2
Cd	0.7	S	2
Cl	2	Si	5
Co	0.03	Zn	0.1
Cr	2	Zr	5
Cu	1	Ba	0.07
F	20	Ce	20
Fe	10	La	0.3
K	0.05	Pr	0.3
Mg	3	Sr	0.1
Mn	0.05	RE's	900
Na	5		

Isotope: Cm-243

Batch: 1011 Material: CmO_2 Position: 10

Concentration: 576 ± 17 mg Cm/g oxide

Date: 26 Jan 82 Method: Alpha Spectrometry

Isotopic: Date of analysis: 18 July 80

	243	244	245	246	247	248
Atom %	57.78	40.26	0.96	0.98	≤0.02	≤0.01
Std. Dev.	0.15	0.15	0.02	0.02	-	-

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: 10 Feb. 82

Element	wt ppm
Al	300
B	100
Fe	500
Nd	20,000
Ce	2,000
La	2,000
Pu-239	20,000
Pu-240	23,000
Pu-241	13,000

Isotope: U-238

Batch: Q-1 Material: (U_3O_8) Position: 11

Concentration: 814.6 ± 7.3 mg U/g oxide

Date: 20 July 81 Method: Isotope Dilution Mass Spectrometry

Isotopic: Date of analysis: 20 July 81

	233	234	235	236	238
Atom %	<0.001	0.0015	0.044	<0.001	99.955
Std. Dev.	-	0.0002	0.0005	-	0.001

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: 20 Apr. 81

Element	wt ppm	Element	wt ppm
B	4	Na	25
Ca	3	Ni	2
Co	0.3	P	8
Cr	0.4	S	25
Cu	1	Si	≤17
Fe	34	Ti	4
K	0.4	V	8
Mg	≤0.7	Zr	1
Mn	0.4	Zn	0.4
Mo	5		

Isotope: Am-243

Batch: HIP1018 Material: AmO_2 Position: 12 and 13

Concentration: 881.7 ± 18 mg Am/g oxide

Date: 22 Feb. 79 Method: Alpha Spectrometry

Isotopic: Date: 19 Dec. 77

	241	242	243
Atom %	0.012	<0.001	99.987
Std. Dev.	0.001	-	0.001

Impurities: Method: Spark Source Mass Spectrometry

a. Chemical Date of analysis: 17 Aug. 78

Element	wt ppm	Element	wt ppm
Al	300	P	0.1
Ca	10	S	50
Cu	5	Si	200
Fe	20	Te	≤10
K	700	Zn	≤5
Mg	20		

b. Radiochemical Date of analysis: 24 July 78

Nuclide	μCi/mg ^{243}Am
Np-239	194
Pu-238	0.036
Pu-239+240	0.542

Note: A typical amount of Cm-244 contamination, 0.01 alpha % abundance was measured by alpha spectrometry on 22 Feb. 79.

Isotope: Am-241

Batch: 79AmB4 Material: AmO_2 Position: 14 and 15

Concentration: 862 ± 17 mg Am/g oxide

Date: 9 Apr. 81 Method: Alpha Spectrometry

Isotopic: Date: 21 Mar. 81

	241	242	243
Atom %	99.995	≤0.001	0.004
Std. Dev.	0.001	-	0.001

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: 27 March 81

Element	wt ppm	Element	wt ppm
Al	700	P	500
B	30	Pb	100
Ca	100	S	300
Co	10	Si	100
Cr	400	Th	20
Cu	40	Ti	50
Fe	1000	Zn	30
K	5	RE	110
La	3	Np-237	2000
Mg	10	Pu-240	60
Mn	50	Cm-244	1000
Mo	7	Cm-245	10
Na	100	Cm-246	40
Ni	800		

Isotope: Pu-242

Batch: 290A Material: PuO_2 Position: 16

Concentration: 862.9 ± 8.6 mg Pu/g oxide

Date: 30 June 82 Method: Isotope Dilution Mass Spectrometry

Isotopic: Date: 30 June 82

	238	239	240	241	242	244
Atom %	0.124	0.383	1.023	0.093	97.950	0.427
Std. Dev.	0.001	0.002	0.003	0.001	0.010	0.002

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: 1 Aug. 80

Element	wt ppm	Element	wt ppm
Al	0.7	Na	1
B	0.3	Ni	0.2
Ba	0.2	S	50
Ca	2	Si	1
Ce	0.2	Sr	0.5
Cl	1	Th-232	100
Fe	3	Zr	0.2
La	0.2		

Isotope: Pu-244

Batch: 297C Material: PuO_2 Position: 20

Concentration: 877 ± 13 mg Pu/g oxide

Date: 16 July 81 Method: By Difference*

Isotopic: Date of analysis: 30 July 81

	238	239	240	241	242	244
Atom %	0.013	0.040	2.796	0.540	9.036	87.575
Std. Dev.	0.002	0.002	0.010	0.005	0.015	0.045

Impurities: Method : Spark Source Mass Spectrometry

Date of analysis: 16 Apr. 81

Element	wt ppm	Element	wt ppm
Al	26	Na	26
B	3	Ni	4
Ca	26	S	175
Cr	175	Si	88
Cu	200	Th	70
Fe	88	Zn	18
Mg	3	Am-241	4510
K	9	U-234	177

*The portion taken for concentration analysis was lost in hot cell; when discovered no more of Batch 297 was available. Hence, the concentration is estimated by taking the difference from theoretical after subtracting the weight for impurities.

Isotope: Pu-240

Batch: HIP1068 Material: PuO_2 Position: 21 and 22

Concentration: 776.4 ± 8.0 mg/g oxide

Date: 30 June 82 Method: Isotope Dilution Mass Spectrometry

Isotopic: Date of analysis: 30 June 82

	238	239	240	241	242	244
Atom %	0.092	0.019	99.86	0.003	0.020	<0.001
Std. Dev.	0.001	0.001	0.01	0.001	0.001	-

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: 1 Aug. 81

Element	wt ppm	Element	wt ppm
As	3	Mg	9
B	620	Mn	62
Ba	70	Na	440
Ca	25	Ni	35
Ce	0.2	P	2
Cl	265	Pb	9
Cr	5	S	6
Cu	7	Sb	7
Fe	6200	Ta	350
I	35	Te	35
K	35	U-236	175

Isotope: Pu-239

Batch: 453B0 Material: PuO_2 Position: 23

Concentration: 864.2 ± 8.6 mg Pu/g oxide

Date: 18 Nov. 75 Method: Coulometry

Isotopic: Date of analysis: 11 Aug. 72

	238	239	240	241	242	244
Atom %	<0.002	99.107	0.877	0.011	0.005	<0.005
Std. Dev.	-	0.01	0.002	0.001	0.001	-

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: 21 Aug. 72

Element	wt ppm	Element	wt ppm
Al	15	Na	1
As	1	Ni	1
B	0.2	P	<1
Ba	<1	Pb	<1
Ca	8	S	1000
Co	<1	Si	18
Cu	6	Sn	1
Cr	<1	U-233	1
Fe	10	U-238	5
K	10	Zn	2
Mg	1	Zr	<1
Mn	<1		

Isotope: Pu-241

Batch: 307A Material: PuO_2 Position: 24

Concentration: 742 ± 4 mg Pu/g oxide

Date: 23 Apr. 82 Method: Isotope Dilution Mass Spectrometry

Isotopic: Date of analysis: 23 Apr. 82

	238	239	240	241	242
Atom %	0.011	1.177	1.268	96.759	0.785
Std. Dev.	0.001	0.010	0.002	0.012	0.004

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: 16 Apr. 81

Element	wt ppm	Element	wt ppm
Al	6	Na	88
Ca	9	Ni	4
Cr	4	P	0.3
Cu	9	S	88
Fe	26	Si	44
K	9	V	0.4
Mg	1	Zn	9
Mn	0.4	Np-237	880

- Date of Americium-241 separation = 15 Apr. 74.

Isotope: Th-232

Batch: 4151 Material: ThO_2 Position: 25

Concentration: 877.5 ± 4.0 mg Th/g oxide

Date: 30 June 81 Method: Isotope Dilution Mass Spectrometry

Isotopic: Date of analysis: 30 June 81

	232
Atom %	100.0
Std. Dev.	-

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: 14 Feb. 79

Element	wt ppm	Element	wt ppm
Al	10	Mg	10
B	3	Mo	3
Ba	10	Na	0.3
Ca	10	Ni	10
Co	0.3	P	5
Cr	30	Si	10
Cu	3	V	3
Fe	100	Zn	3
K	1	Zr	0.3

Isotope: U-236

Batch: 201DMR Material: U_3O_8 Position: 26

Concentration: 730.1 ± 4.1 mg U/g oxide

Date: 20 July 81 Method: Isotope Dilution Mass Spectrometry

Isotopic: Date of analysis: 20 July 81

	233	234	235	236	238
Atom %	0.207	0.122	9.352	88.929	1.390
Std. Dev.	0.042	0.001	0.001	0.052	0.010

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: 10 Mar. 82

Element	wt ppm	Element	wt ppm
B	845	Ni	250
Ca	170	P	7
Co	85	S	85
Cr	85	Si	8500
Cu	845	Te	425
Fe	Major	Ti	85
K	85	V	17
Mg	85	Zn	70
Mn	25	Zr	35
Na	425	Np-237	20,000

Isotope: U-234

Batch: M-9 Material: U_3O_8 Position: 27

Concentration: 720 ± 7 mg U/g oxide

Date 5 Mar. 82 Method: Isotope Dilution Mass Spectrometry

Isotopic: Date: 5 Mar. 82

	233	234	235	236	238
Atom %	0.0022	99.766	0.168	0.040	0.024
Std. Dev.	0.0005	0.002	0.001	0.001	0.001

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: 10 Mar. 82

Element	wt ppm	Element	wt ppm
B	17	Ni	1
Ca	8	P	1
Cr	1	S	8
Co	3	Sb	3
Cu	3	Si	25
Fe	40	Zn	3
K	5	Zr	3
Mg	8	Np-237	170
Ga	2	Th-230	17
Na	40	Th-232	17

Isotope: U-235

Batch: 264C Material: UO_2 Position: 28

Concentration: 879.7 ± 9.0 mg U/g oxide

Date: 19 June 75 Method: Coulometry

Isotopic: Date: 11 Aug. 72

	233	234	235	236	238
Atom %	<0.0005	0.034	99.89	0.025	0.053
Std. Dev.	-	0.001	0.01	0.001	0.002

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: 21 Aug. 72

Element	wt ppm	Element	wt ppm
Al	<1	Na	<1
As	<1	Ni	<1
B	<0.5	P	1
Ba	<1	Pb	1
Ca	2	Si	<3
Co	<1	S	4
Cr	<1	Ti	10
Cu	5	V	100
Fe	8	W	<1
K	<1	Zn	<1
Mg	<2	Zr	1
Mn	<1		

Isotope: Pa-231

Batch: Pa-F-1 Material: PaO_2 Position: 29

Concentration: 798.0 ± 24 mg Pa/g oxide

Date: 1 Sept. 81 Method: Alpha Spectrometry

Isotopic: Date: 1 Sept. 81 Method: Alpha Spectrometry

	231
Atom %	100.0
Std. Dev.	-

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: 1 Sept. 81

Element	wt ppm	Element	wt ppm
Au	10	Na	1000
B	30	Ni	200
Ba	1000	Pb	200
Bi	50	La	1
Ca	500	Y	1
Ce	5	Pt	100
Cd	30	Rb	3
Co	5	Sr	30
Cr	40	Ti	10
Cu	100	V	1
F	40	Zn	100
Fe	1000	Zr	10
K	100	Ac-227	200
Mg	200	Th-232	700
Mn	70		

Isotope: Pu-238

Batch: 06HP014 Material: PuO_2 Position: 30

Concentration: 836.1 ± 17 mg Pu/g oxide

Date: 26 June 72 Method: Alpha Spectrometry

Isotopic: Date: 20 June 72

	238	239	240	241	242
Atom %	99.39	0.279	0.319	<0.01	0.018
Std. Dev.	0.04	0.01	0.01	-	0.002

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: 15 Feb. 82

Element	wt ppm	Element	wt ppm
Al	100	Mg	2000
B	5	Mn	5
Ba	3	Na	50
Ca	1000	Zn	4000
Cr	20	S	3000
Cu	1	Si	3000
Fe	200	Ti	500
K	50	Th-232	5000

Isotope: Th-230

Batch: 256A Material: ThO_2 Position: 31

Concentration: 822.5 ± 2.3 mg Th/g oxide

Date: 5 Mar. 82 Method: Isotope Dilution Mass Spectrometry

Isotopic: Date: 5 Mar. 82

	230	232
Atom %	89.47	10.53
Std. Dev.	0.13	0.13

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: 15 Feb. 82

Element	wt ppm	Element	wt ppm
B	50	Si	40
Ba	10	Np-237	15
Ca	30	U	550
Co	1	Pu	125
Cu	2	U-235	200
Fe	10	U-236	200
K	40	U238	150
Mn	0.3	Pu-239	80
Na	100	Pu-240	20
Rb	0.1	Pu-241	12
S	20	Pu-242	12
Sr	0.2		

Isotope: U-233

Batch: 240A Material: U_3O_8 Position: 32

Concentration: 797.8 ± 3.3 mg U/g oxide

Date: 5 Mar. 82 Method: Isotope Dilution Mass Spectrometry

Isotopic: Date: 5 Mar. 82

	233	234	235	236	238
Atom %	99.887	0.052	0.0032	0.0018	0.056
Std. Dev.	0.002	0.001	0.0005	0.0002	0.002

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: 23 Apr. 81

Element	wt ppm	Element	wt ppm
B	2	Ni	3
Ba	2	P	4
Ca	17	S	60
Cr	1	Si	17
Cu	4	Np-237	25
Fe	25	Th-229	25
K	8	Pu-239	25
Mg	2	Pu-240	10
Mn	1	Am+Pu-241	17
Na	17	Pu-242	25

APPENDIX 2

Analytical Data for Dosimeter Materials

Explanation of Headings on Data Sheets:

- Isotopic: Method is Thermal Emission Mass Spectrometry
- Impurities: Wt. ppm equal to μg/g oxide

Isotope: U-235

Batch: 264C Material: UO_2 Position: 1, 17, and 33

Concentration: 879.7 ± 9.0 mg U/g oxide

Date: 19 June 75 Method: Coulometry

Isotopic: Date: 11 Aug. 72

	233	234	235	236	238
Atom %	<0.0005	0.034	99.89	0.025	0.053
Std. Dev.	-	0.001	0.01	0.001	0.002

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: 21 Aug. 72

Element	wt. ppm	Element	wt. ppm
Al	<1	Na	<1
As	<1	Ni	<1
B	<0.5	P	1
Ba	<1	Pb	1
Ca	2	Si	<3
Co	<1	S	4
Cr	<1	Ti	10
Cu	5	V	100
Fe	8	W	<1
K	<1	Zn	<1
Mg	<2	Zr	1

Isotope: U-238

Batch: ESZ Material: UO_2 Position: 1, 17, and 33

Concentration: 877.5 ± 8.8 mg U/g oxide

Date: 19 June 75 Method: Coulometry

Isotopic: Date: 11 Aug. 72

	233	234	235	236	238
Atom %	<0.0001	<0.0001	0.0012	<0.0001	99.999
Std. Dev.	-	-	0.0001	-	0.001

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: 21 Aug. 72

Element	wt. ppm	Element	wt. ppm
Al	5	Ni	2
As	1	P	10
B	2	Si	3
Ca	15	Sn	5
Ce	1	S	10
Cu	2	Ti	10
Fe	25	V	100
Mg	2	Pu-239	10

Isotope: Pu-239

Batch: 453-BO Material: PuO_2 Position: 2, 18, and 34

Concentration: 864.2 ± 8.6 mg Pu/g oxide

Date: 18 Nov. 75 Method: Coulometry

Isotopic: Date of analysis: 11 Aug. 72

	238	239	240	241	242	244
Atom %	<0.002	99.107	0.877	0.011	0.005	<0.0005
Std. Dev.	-	0.010	0.002	0.001	0.001	-

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: 21 Aug. 72

Element	wt. ppm	Element	wt. ppm	Element	wt. ppm
Al	15	Fe	10	Si	18
As	1	K	10	Sn	1
B	0.2	Mg	1	S	1000
Ba	<1	Mn	<1	Ti	<1
Ca	8	Na	1	U-238	5
Co	<1	Ni	1	U-233	1
Cr	<1	P	<1	Zn	2
Cu	6	Pb	<1	Zr	<1

Isotope: Np-237

Batch: 24HP Material: NpO_2 Position: 2, 18, and 34

Concentration: 874 ± 26 mg Np/g oxide

Date: 29 Mar. 76 Method: Alpha Spectrometry

Isotopic: Date of analysis: 11 Aug. 72

	236	237	238	239
Atom %	<0.0005	≥99.99	≤0.003	≤0.004

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: 21 Aug. 72

Element	wt. ppm	Element	wt. ppm
Al	2	Ni	1
As	<1	P	<1
B	<0.5	Si	3
Ba	<1	Sn	2
Ca	2	S	4
Co	<1	Tc	<1
Cr	<1	V	<1
Cu	2	Zn	<1
Fe	4	Zr	<1
K	1	U-235	<1
Mg	<1	U-238	10
Mn	<1	Pu-239	<1
Na	<1	Cm-244	<1

Element: Cu

Batch: N Material: Cu Position: 3, 19, and 35

Concentration: ≥999 mg/g Cu of normal composition

Date: 30 Apr. 81 Method: Spark Source Mass Spectrometry

Impurities: Method: Spark Source Mass Spectrometry

Date of analysis: 30 Apr. 81

Element	wt. ppm	Element	wt. ppm
Ag	2	K	2
Al	0.3	Mn	0.1
As	0.3	Na	20
B	0.02	Ni	1
Ca	2	P	0.7
Cl	30	Sb	0.7
Cr	0.2	Se	2
Fe	6	Si	5

Element: Co

Batch: Co-1 Material: CoO-MgO Position: 3, 19, and 35

Concentration: 1.23 ± 0.02 mg Co/g mixed oxide

Date: 12 Apr. 77 Method: Neutron Activation

Impurities: Method: Emission Spectrometry

Date of analysis: 11 Oct. 76

Element	wt. ppm	Element	wt. ppm
Ag	≤1	Mg	Major
Al	500	Mn	7
B	100	Mo	7
Ca	50	Na	150
Co	1200	Ni	150
Cr	100	Pb	50
Cu	100	Si	400
Fe	100	Ti	10
K	20	Zr	50

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