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EXPERIMENTS ON THE RELEASE OF FISSION PRODUCTS FROM MOLTEN REACTOR FUELS

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EXPERIMENTS ON THE RELEASE OF FISSION PRODUCTS FROM MOLTEN REACTOR FUELS

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EXPERIMENTS ON THE RELEASE OF FISSION PRODUCTS FROM MOLTEN REACTOR FUELS

ABSTRACT

Experiments in the controlled melting of irradiated fuel specimens, particularly of the APPR, STR, and MTR types, have confirmed that prolonged heating in air at temperatures in excess of the melting point results in the release of a large portion of the radioactivity. On the other hand, a moderate amount of heating in air or steam sufficient only to melt a specimen results mainly in the partial volatilization of the rare gases; the halogens, iodine and bromine; and the alkali metals, cesium and rubidium. In the presence of air or water vapor, strontium and other fission products are not released.

At trace concentration of fission products, slow melting of the APPR plate at 1525°C in air or steam effected the release of 50 per cent of the rare gases, 33 per cent of the iodine, 9 per cent of the cesium and traces of strontium. After 25 per cent burn-up, the cesium value increased to about 60 per cent. Aluminum alloy of the MTR type, also at trace concentration, upon melting at 700°C released up to 2 per cent of the iodine, 10 per cent of the rare gases, and negligible portions of other fission products. Zirconium alloy of the STR type after 15 per cent burn-up, when melted at 1850°C, released up to 95 per cent of the rare gases, 90 per cent of the cesium, 60 per cent of the iodine, and only a trace of strontium.

INTRODUCTION

For general reactor safeguards considerations it has been necessary, lacking precise information, to assume that in the event of a reactor catastrophe all or practically all of the fission products would be released to the atmosphere. Since a nuclear explosion is far from the realm of likelihood, and because of the excessive burden of safety considerations and the liability that this imposes, it is of prime importance to demonstrate to what extent this assumption is true. It has been assumed that the worst disaster short of an explosion would be a complete melt-down in the presence of air and steam, and since it should be possible to simulate such an event on a small scale and to measure readily and precisely the percentage release of each of the fission elements, an investigation of the behavior of several reactor fuels has been conducted.

The principle approach has been based on the assumption that a justifiable condition for a reactor incident might involve loss of coolant with sufficient heating to accomplish melting in approximately two minutes. The period during which a specimen is kept molten has been kept short in relation to the heating period on the basis that in the fluid state it would tend to flow out of the heat zone. The presence of air and/or steam has been assumed and the effect of agitation or purging of the melt has been ignored. When a sample was contained, as in a small crucible, the small fraction of activity adhering to the crucible was considered as retained by the sample.

THEORETICAL CONSIDERATIONS

Fortunately, the widespread interest in the pyrometallurgy[1-5] of uranium has somewhat already pointed out a systematic behavior of the fission elements. From this work and especially from the early theoretical work of Brewer[6] it is possible

to predict that a fission product may be released or retained in the solid residue of a melt according to the following pattern:

Processes Favoring Release to Environment

1. Vaporization of the elemental form of a volatile constituent. This is particularly important for elements such as the rare gases, Xe and Kr, and for I_2 and Br_2, and Cs and Rb.
2. Diffusion into an oxide or slag or skin followed by dispersion or vaporization of the oxide at high temperature. This may occur with Mo.
3. High temperature vaporization of the element in the absence of air, as e.g., Sr and Ba, Se and Te.
4. Formation of chemical compounds between two fission products, particularly with halides.

Processes for Retention by the Melt

1. Occlusion of elemental or combined forms of volatile constituents. The rare gases Xe and Kr are held by molten aluminum unless purged.
2. Conversion to non-volatile carbides, as for example Zr and Nb.
3. Occlusion in crystalline fuel, e.g., UO_2 in a molten stainless steel matrix.
4. Formation of stable alloys with the uranium fuel, e.g., tellurium and ruthenium.
5. Formation of nonvolatile oxides, e.g., BaO, SrO.

In order to evaluate the behavior of individual fission elements it was of value to refer to the thermodynamic stability of the oxides as given by Glassner[7] in Table I. The significance of the free energy of formation is particularly

TABLE I

Free Energy of Formation* of Oxides of Fission Elements**

Compound	1000° K	1500° K	2000° K
La_2O_3	123	112	100
Ce_2O_3	120	107	94
Pr_2O_3	120	107	94
SrO	116	102	80
UO_2	109	99	88
ZrO_2	108	97	86
BaO	111	95	80
PuO_2	100	95	85
Nb_2O_5	76	66	61
MoO_2	46	39	32
TeO_2	16	5	-7.3
Cs_2O	35	3.6	-16
RuO_2	6	-2.7	-10

* Kilogram Calories per mole.

** Glassner, A., ANL-5107.

noteworthy in the low values attributed to the oxides of ruthenium, cesium, tellurium and molybdenum. Since cesium and tellurium are both volatile and both readily dissociated as oxides, one would expect equal release from molten uranium; however, tellurium as well as ruthenium and molybdenum appears to have alloying properties which interfere with their escape.

The vapor pressure data given in Table II[8] is also useful in predicting the behavior of the volatile elements, cesium and rubidium, as well as barium and strontium. The presence of air or steam effectively prevents vaporization of the alkaline earth metals, Ba and Sr, since their oxides do not dissociate readily. Upon melting in a noble gas atmosphere, these two elements should show increased volatility.

EXPERIMENTAL

In order to melt various reactor fuel bearing materials in a controlled atmosphere, a method based on radiant heating of a sample in a silica tube with an exterior graphite induction susceptor ring has been used throughout most of the experiments described. The volatile products of the melting operation have been trapped selectively either on a filter, a chemical adsorbent in the case of iodine, or on activated charcoal for the rare gases. The dispersed solids or condensible vapors were collected in a filter plug of quartz wool at the top of the furnace tube. In the earlier experiments, iodine was also adsorbed on charcoal which was later heated to remove rare gases. The specimen has been loaded and held in various ways, such as in contact with a flat disk of ZrO_2 or by suspension from a tantalum wire or by placing in a small silica crucible which in turn was held by a tantalum wire or quartz rod.

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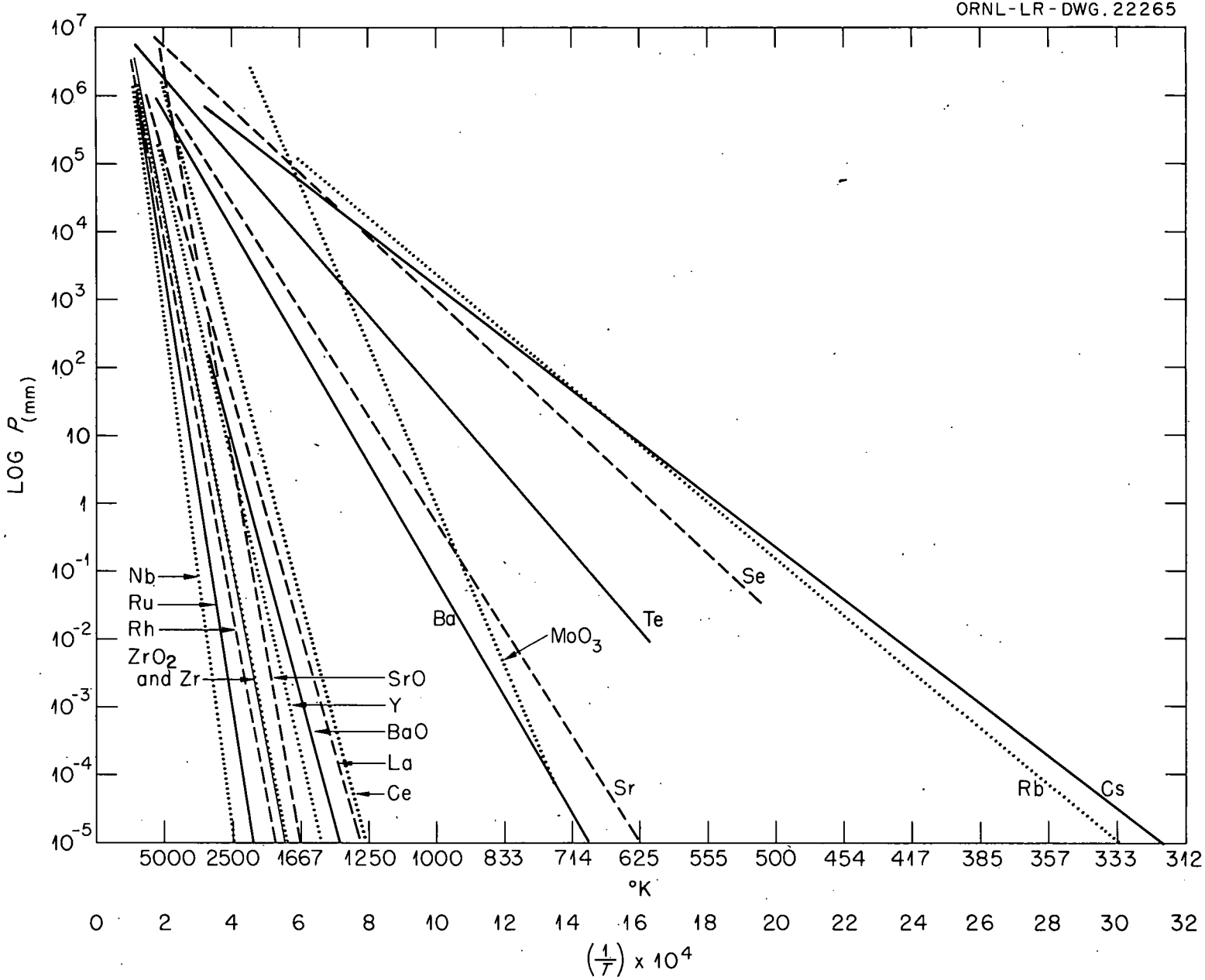

VAPOR PRESSURES OF METALS AND OXIDES

TABLE II

PRELIMINARY RESULTS ON REPEATED MELTING AND SINTERING OF APPR FUEL DISKS

The first exploratory experiments in melting APPR stainless steel fuel plates were performed in the assembly in Fig. 1 for the convenience of dropping into it a short-cooled irradiated punched disk containing about 10 mg of U^{235}. With the arrangement shown, it was not possible to observe the actual temperature attained, and the necessary heating time was only empirically established by the time required to melt a standard such as platinum foil (1720°C).

Since the first concept of a simulated incident was presumed to imply a quite rapid heating cycle, these tests were designed so that the melting temperature was reached in 10 seconds or less. It is likely then, particularly since the temperature standard used was a bright, highly reflective noble metal in the form of a foil, that a considerable over-run above the melting temperature was attained. The melts resulting were sintered and brittle and indicated complete oxidation. The data (Table III) were not entirely inconsistent, although some indicated that the sample when barely melted would probably release much less of a fission product. It was also noted that repeated heating cycles of 10-20 seconds each gave an almost constant percentage yield of the residual activity.

The procedure involved setting up the necessary cold traps and leaving the furnace tube open. The sample was monitored for total initial gamma activity using a vibrating reed electrometer and then was introduced into the furnace tube before the filter and iodine absorber were attached. A suitable gas flow (1 liter/min) was sustained by means of a vacuum line and supply lines if helium or steam was being used and the heating started for the prescribed interval (usually 10 seconds). After heating for one cycle, measurements were made by fixed gamma-ionization chambers on the residue from the melt and on the iodine and rare-gas fractions. These values were converted to percentages by comparing values obtained from a

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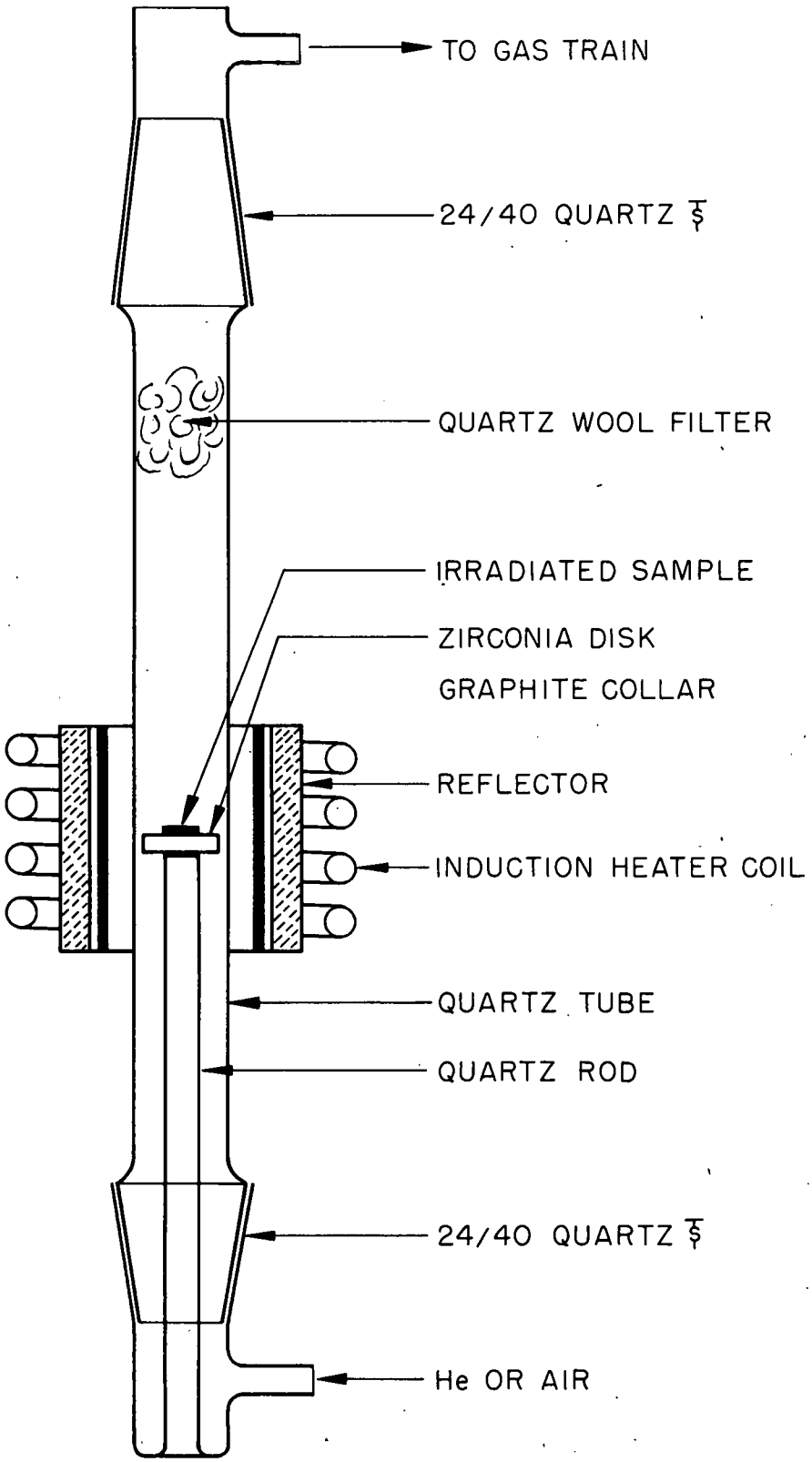

MELTING SYSTEM SHOWING ZIRCONIA DISK

Figure 1

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TABLE III

Volatilization of Fission Products from APPR Disks by High Temperature Sintering

Run No.	Melting and Cooling Cycles	% of Gross γ Activity**						
		Activity in Furnace Tube	Charcoal Trap Degassed (Iodine)	Charcoal Trap (Rare Gases)	Atmosphere During Melting	Pellet Activity Before Melting	Pellet Activity After Melting	Total Activity Loss
1	3				Air	100	47.5	52.5
2	4				Air	100	58.5	41.5
3	3	13.3	6.5	11.6	Air	100	61.7	38.3
4	3	8.8	4.8	9.2	Air	100	72.4	27.6
5	1	1.44	1.79	2.62	Air	100	93.5	6.5
6	1	5.95	0.8	4.00	Air	100	88.55	11.45
7	1	1.3	2.5		Air	100	96.0	4.0
8	1	3.0	1.5	3.0	Air	100	92.6	7.4
9	1	3.6	0.95	2.4	Air	100	93.0	7.0
10	1	2.06	0.04	0.96	He	100	97.0	3.0
11	1	2.77	1.9	2.69	Air	100	92.7	7.3

** Distribution of Gross γ as Measured by High Pressure γ Chamber.

duplicate pellet oxidized in a stream of chlorine gas from which the iodine was collected in a duplicate adsorber. For the rare gas fraction, the percentages were obtained either by dissolving a duplicate sample or by dissolving the residue from the melt. Only one sample was melted in a helium atmosphere and this gave a total activity release value equal to about half the lowest observed in air. In particular the iodine value seemed to be depressed by the helium atmosphere.

Difficulties in correcting for geometric effects in the change of solid angle to the ionization chamber led to the final abandonment of this technique and to the substitution of a complete radiochemical analysis for all non-volatile radio-elements. The rare gases were still estimated by ion-chamber measurements.

MTR ALUMINUM ALLOY WITH SLOW MELTING

In Tables IV and V, a summary of results of melting aluminum alloy of the MTR type shows a pronounced effect of the milder temperature. The behavior of aluminum alloy is of interest in that relatively little of the rare gas activity is released simply upon melting, although in the molten condition it can be purged quantitatively. However, the undisturbed melt shows a "squeeze-effect" upon cooling which accounts for most of the rare gases released. An illustration of this effect is shown in Fig. 2.

APPR MINIATURE PLATES OR CLAD COUPONS

In order to eliminate the possibility of an exposed edge effect from the punched disks as well as to provide samples for extended irradiations, miniature plates containing 100 mg of U^{235} were specially produced for these experiments. The samples measured about 3/4 in. by 1 1/4 in. with a fuel bearing zone being an elongated oval over about 25 per cent of the area. At this time it is doubtful that in melting there is a significant exposed edge effect.

TABLE IV

Fission Product Volatilization from MTR Disks

Run No.	Time In-Pile (Hours)	Time Out-Of-Pile (Hours)	Temp. of Run (° C)	% Total Iodine Released	% of Total γ Released
1	13.5	7.25	700	1.2	0.45
2	13.5	-	700	0.88	0.41
3	60.5	6.33	700	0.27	0.30
4	16	2	700	0.49	0.15
5	60.5	5.5	700	1.8	0.82
Average	-	-		0.93	0.43
6	13.5	7.7	800	5.3	2.26
7	13.5	9.25	800	4.9	2.06
8	16	5.25	800	4.18	1.10
Average	-	-		4.79	1.81

* MTR Pellets Melted in Air and Held at 700 or 800° for 1 Hour.

TABLE V

Release of Rare Gases by Rapid Melting of MTR Disks*

Run No.	Atmosphere During Melting	% Rare Gas Released
1	Air	9.4
2	Air	8.4
3	Air	11.2

* 700°C, 2 Minutes.

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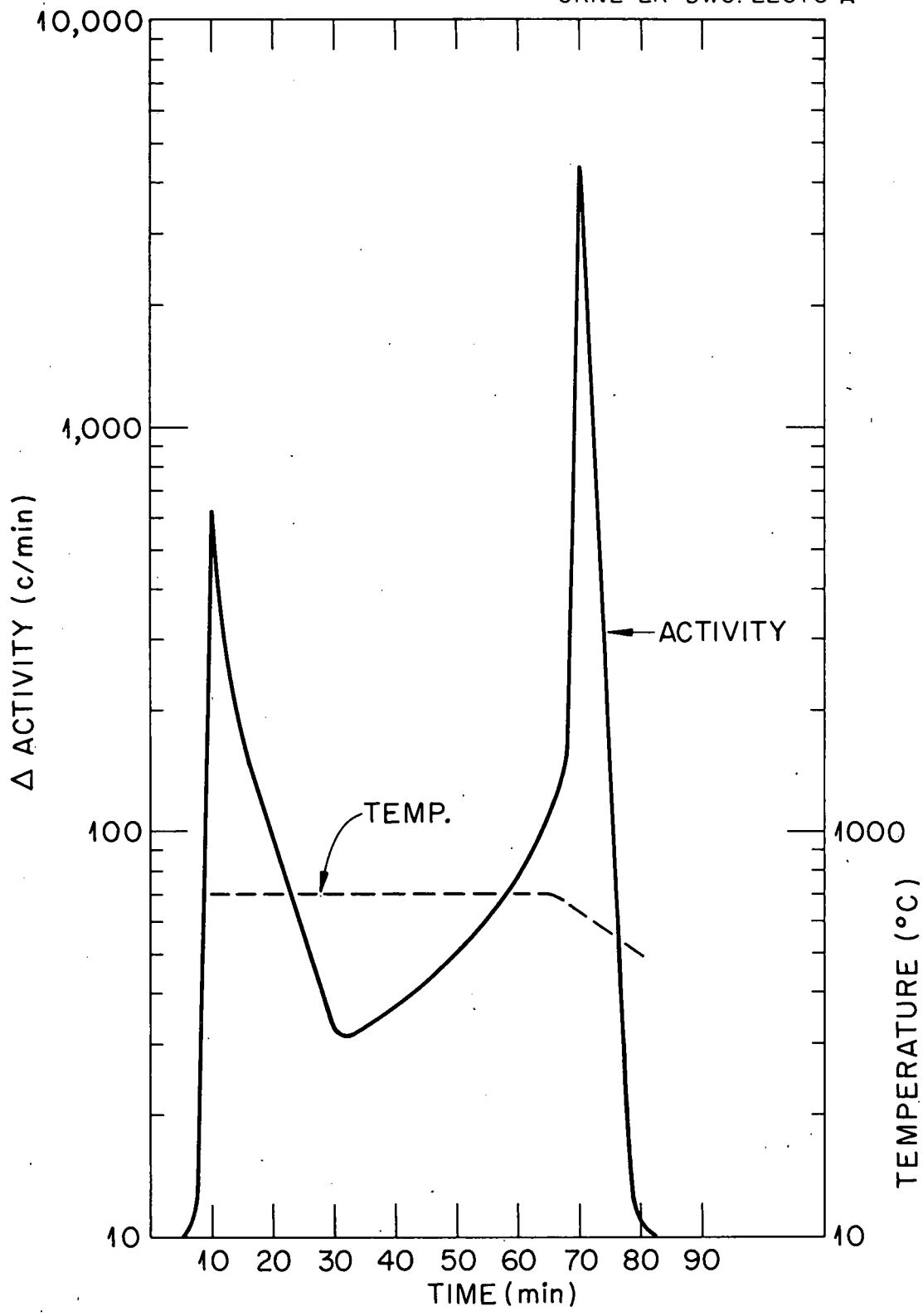

RATE OF RARE GAS RELEASE FROM Al-U PELLET AT 700°C, SHOWING ACTIVITY PEAK ON COOLING

Figure 2

After resorting to several techniques for suspending the fuel specimen in the furnace tube, such as tying to a fine platinum wire; a more convenient method of dropping it into a suspended silica crucible was adopted (Fig. 3). The procedure was otherwise the same. The furance tube and specimen were arranged so that an optical pyrometer could be used to observe the heating and the actual melting temperature. Heating was continued only long enough to accomplish melting which occurred in about 90 seconds. One coupon or miniature plate was melted in a steam atmosphere which resulted in even lower yields of most of the various fission products. This is not considered conclusive however, without further work. The analytical procedure used was the same as described earlier except that the melt residue in the small crucible was dissolved in aqua-regia. All dispersed activity outside of the crucible was included in the washings from the furnace tube. This washing was accomplished by using the iodine absorbent (NaOH pellets) with water as an alkaline wash and was followed by a rinse with nitric acid and ammonium fluoride.

From these samples, the percentage of gross gamma activity released ranged from 1 to 8. The wide spread is probably due to differences in the total time-at-temperature which was difficult to duplicate. The cesium values are in fair agreement, averaging 9 per cent, the iodines average 33 per cent, and the strontium is not clearly different from zero. The effect of the presence of steam was a reduction in volatility as shown in Table Vi.

THE EFFECT OF FISSION PRODUCT BUILD-UP IN APPR AND STR FUEL

A program has been started to irradiate fuel miniatures of APPR type to definite burn-ups in the stages of 15, 30, 45 and 60 per cent. Before these irradiations were complete, it was of interest to test previously irradiated

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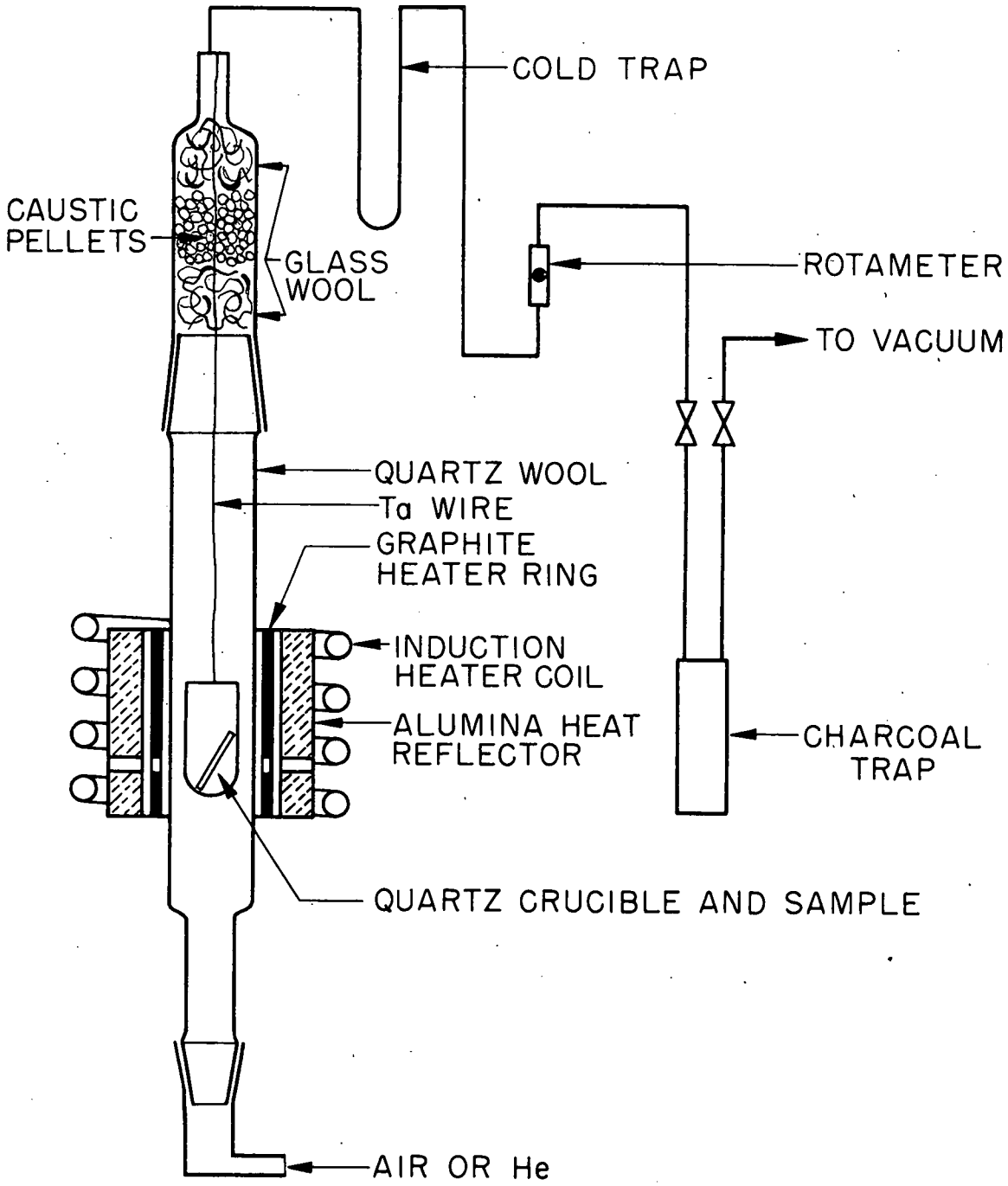

MELTING APPARATUS
SHOWING IODINE TRAP AND GAS TRAIN

Figure 3

TABLE VI

Fission Product Volatilization From

APPR Clad Coupons Melted in Air or Steam

Run No.	Time Out-Of-Pile (Days)	Rare Gases (%)	Gross γ (%)	Iodine (%)	Cs (%)	Sr (%)
1 (Air)	10	45	5.37	49	-	-
2 (Air)	16	59	2.2	25	-	-
3 (Air)	22	38.4	-	-	-	-
4 (Air)	19	47.6	4	15.4	-	-
5 (Air)	25	40	1.5	34	-	-
6 (Air)	7	53.6	8	31	11	0.001
7 (Air)	8	61	6	41	13	0.001
8 (Air)	10	44	-	-	-	-
9 (Air)	17	49.7	4.5	38	-	0.0001
10 (Air)	19	45.7	4.4	35	3.4	0.1
Average		48.4	4.5	33.5	9.1	0.025
11 (Steam)	8	38.5	2.3	11.2	0.3	0.4

* Coupons Irradiated in Graphite Pile for 1 Week.

APPR fuel having an average burn-up of 25 per cent. From sample plates, small disks or pellets about 5/16 inch in diameter were punched with exposed edges and melted about as before. Since these punchings were not reirradiated, no measurements on iodine or rare gases were taken. There was, however, an indicated effect of burn-up in the cesium value which increased to 75 per cent in one case and a significant yield of strontium of 6.8 per cent on only one specimen which was melted in helium. This result should be considered as preliminary, particularly since it was not obtained with clad specimens. It is summarized in Table VII.

The samples of the STR zircalloy fuel obtained had a lesser amount of burn-up and were punched as before. These, however, were reirradiated for 60 hours to obtain the necessary iodine and rare gas activities. For this type of fuel the percentage release of rare gases was extremely high, 95-99 per cent, and the iodine ranged up to about 60 per cent. Since the required temperature, 1850°C, was above the melting point of the silica tube, it was necessary to melt the samples relatively quickly (35 seconds). Again radiochemical results were obtained by dissolving the residues in HNO_3-HF mixtures. The iodine analysis was obtained by dissolving the caustic pellet adsorber and using the solution as a wash for the furnace tube. Radiochemical analyses obtained for Sr, Cs, and I are summarized in Table VIII.

An additional difficulty was encountered with these samples in that, after melting, the silica crucible was always fractured by the cooling of the melt. It is assumed this was caused by a dissolution of ZrO_2 in SiO_2 at high temperature. This resultant scattering of particulate matter might have affected the results. Additional work is indicated.

TABLE VII

Fission Product Volatilization by Melting

APPR Punched Disks, 25% Burn-Up

Run No.	Atmos. During Melting	Max. Temp. (° C)	Time to Melt (Secs.)	% of Total Activity Released				
				Gross γ	Iodine	Cs	Sr	Rare Gases*
1	Air	1575	13	0.4	-	12.0	-	-
2	Air	1650	15	0.2	-	5.8	-	-
3	Air	1575	47	17.3	-	68.3	-	-
4	Steam		45	16.8	-	74.9	0.06	-
5	He	1650	37	26.1	-	99.9	6.8	-

* Decayed thru long-cooling period, not reirradiated.

TABLE VIII

Fission Product Volitilization by Melting

STR - Zircalloy Punched Disks, 15% Burn-Up

Run No.	Atmos. During Melting	Max. Temp. (°C)	Time to Melt (Secs.)	% of Total Activity Released				
				Gross γ	I_2	Cs	Sr	Rare Gases
1	He	1900	33	11.6	40.9	26.3	-	95.6
2	He	2025	37	11.2	38.8	-	0.1	99.4
3	He	-	-	8.0	80.8	65.9	0.4	95.7
4	Air	1925	52	4.5	53.2	4.5	0.2	95.7
5	Steam	1925	-	1.3	53.5	0.6	0.04	98.0

SUSTAINED HEATING OF APPR FUEL BELOW THE MELTING POINT

It was thought than an exploratory experiment to observe the diffusion rate of rare gases through the APPR cladding would be of interest in comparison with the melting data. A special high-temperature, platinum-element furnace was used to heat a coupon in an air stream at 1200-1300°C. The curve in Fig. 4, of rare gas activity release, reveals that the cladding actually completely oxidized and visibly ruptured after about one hour at 1250°C. It was estimated that 90 per cent of the gas was released at this temperature over a period of four hours. A similar experiment in a helium atmosphere indicated no significant release of gases below 1400°C. This experiment will be continued.

CONCLUSIONS

The percentage release of fission products to the atmosphere from molten reactor fuel is apparently proportional to the melting temperature and is affected mainly by the degree of oxidation, the concentration of fission products, and the type of fuel. Lesser factors are the nature of the surrounding atmosphere, size of sample, the degree of subdivision of fuel particles (e.g., UO_2) and time-at-temperature.

No significant effect has been observed to indicate a metal-water reaction for zirconium, aluminum, or stainless steel when melted in steam. The important fission products released* are the rare gases, iodine and cesium.

* See table.

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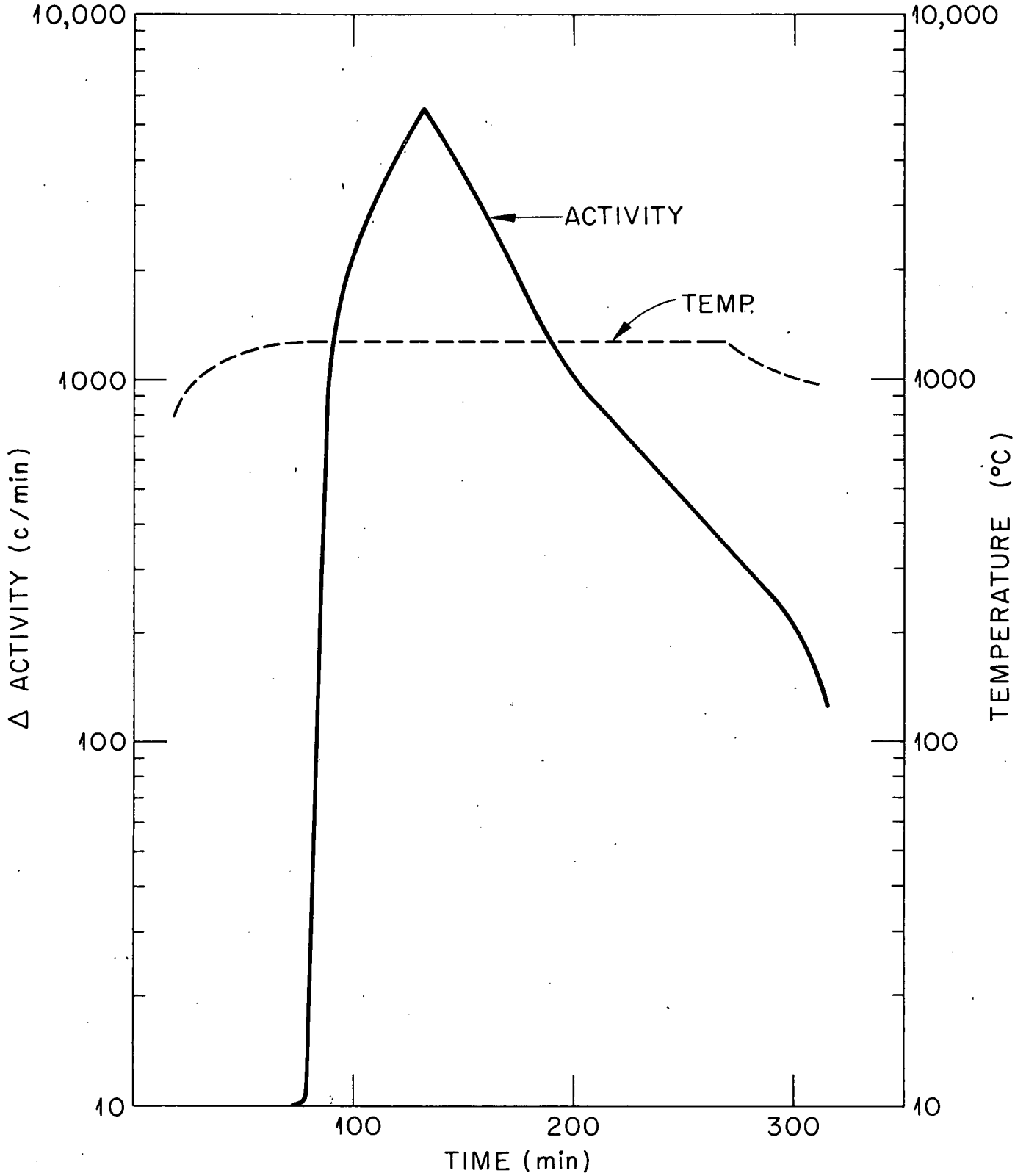

RARE GAS RELEASE FROM IRRADIATED STAINLESS STEEL-URANIUM COUPON HEATED IN AIR STREAM

Figure 4

TABLE IX

CALCULATED BETA ACTIVITY OF THE FISSION PRODUCTS

Per Cent of Total by Element*

IRRADIATION TIME		5 Days				30 Days				MELTING BEHAVIOR AND % RELEASED
Cooling Time		10 Min.	10 Hrs.	10 Days	100 Days	10 Min.	10 Hrs.	10 Days	100 Days	
Group I-A	Xe	6.0	11.0	8.0	0.2	6.0	10.0	4.5	0.1	
	Kr-Rb**	3.4	0.8	0.02	0.06	3.0	0.7	0.02	0.008	
	I-Br	9.0	15.0	8.5	0.1	10.0	13.0	5.7	0.04	High Volatility
	Cs**	3.5	0.001	0.03	0.09	3.0	0.001	0.04	0.24	10-100%
Group I-B	Te-Se	7.0	4.0	4.8	0.05	6.8	4.0	2.2	0.1	Variable
	Mo	14.0	6.2	4.8	0.00	16.0	5.6	2.5	0.0	Moderate Volatility
	Tc	0.3	0.6	0.5	0.02	0.4	0.6	0.3	0.01	1-10%
Group II	Ba	5.0	2.5	10.0	0.15	5.8	5.0	10.5	0.3	Volatile in Vacuum
	Sr	6.0	4.5	3.3	11.0	5.8	4.2	4.5	8.5	Non-Volatile in Air
Group III-A	Y	11.0	10.0	3.3	14.0	10.0	8.0	5.5	14.0	Low Volatility
	La	11.5	3.3	12.0	0.9	6.6	6.0	12.0	0.7	Rare Earths
	Ce	5.0	8.2	8.0	16.0	5.7	8.2	10.2	14.0	Refractory
	Pr	3.6	3.8	11.0	8.0	4.5	6.0	12.0	10.0	<0.1%
	Nd	8.0	1.2	6.0	0.06	1.4	2.5	4.3	0.02	
	Pm	0.6	1.6	1.2	9.0	0.6	1.5	1.7	9.0	
Group III-B	Zr	5.8	13.0	7.0	15.0	5.5	10.0	5.7	14.0	Low Volatility
	Nb	2.9	7.5	1.3	17.0	2.7	5.3	2.6	23.0	Alloying and
	Ru	0.6	0.8	3.0	5.5	1.0	1.7	4.5	6.0	Refractory
	Rh	0.6	2.2	5.5	6.5	1.3	3.3	5.5	6.0	<0.1%

* From Keneshea, F. J., and Saul, A. M.[9]

** Data Extended.

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