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METALLURGY AND CERAMICS

UNITED STATES ATOMIC ENERGY COMMISSION

QUARTERLY SUMMARY RESEARCH REPORT IN METALLURGY

For October, November, December, 1954

by

Ames Laboratory Staff

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QUARTERLY SUMMARY RESEARCH REPORT IN METALLURGY

For the period October, November, December, 1954

This report is prepared from material
submitted by the group leaders of
the Laboratory

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METALLURGY

Under the direction of F. H. Spedding, H. A. Wilhelm,
B. A. Rogers, O. N. Carlson, P. Chiotti,
D. Peterson, and J. F. Smith

1. Purification and Separation Studies

1.1 Some Extractions with Primene-JMT Solutions (1)*

Some low grade uranium ore was treated with a sulfuric acid solution and the acid aqueous solution then extracted with a solution consisting of 25 volume per cent Primene-JMT in n-heptane. The uranium and iron both transferred to the organic phase. However, when a reducing agent was first added to the aqueous phase only the uranium transferred to the organic phase. It was found that the transfer of uranium to the organic is greater as the aqueous solution is made less acid.

A stock solution was made up of the sulfates of aluminum, bismuth, cadmium, copper, chromium, iron, lanthanum, magnesium, manganese, neodymium, nickel, tin, thorium, uranium, vanadium, yttrium and zinc. Of these bismuth, iron, thorium and uranium showed preferential distribution into the organic phase.

1.2 The Separation of Niobium from Titanium (1)

The preparation of niobium spectrographically free of titanium, for use as a base material in setting up a spectrographic method of analysis for titanium in niobium has been given some study.

Previously, as reported, niobium spectrographically free of titanium was obtained by liquid-liquid extraction of niobium from a strong hydrofluoric acid solution of niobium and titanium. The extraction was done with isophorone, and this was followed by scrubbing of the isophorone solution with dilute hydrofluoric acid. The hydrated oxide was precipitated from the isophorone solution and washed with a solution of ammonium hydroxide and acetone. However, even this procedure did not give consistent results and traces of titanium were found in some samples and not in others. Since porcelain crucibles were being used in which to ignite the samples, they were considered as a possible source of contamination, and so were analyzed for titanium. The porcelain crucibles were found to contain considerable amounts of titanium and so were replaced by platinum crucibles. Using the technique above and the platinum crucibles consistently good results were obtained and 80 g of niobium oxide spectrographically free of tantalum and

*Numbers indicate group leaders in charge of work. See Appendix III.

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titanium were prepared. The niobium was sent to the spectrographic laboratory where it will be used as standard material in setting up a method of analysis for small amounts of titanium (<2.5%) in niobium.

1.3 Studies on the Separation of Hafnium from Zirconium (1)

Work has continued on separations of these elements by liquid-liquid extraction systems. The studies involve generally three systems. These are the nitric acid aqueous-tributyl phosphate organic, the sulfuric acid (or hydrochloric acid) aqueous-hexone (or other organic) with thiocyanate, and the sulfuric acid aqueous-Primene-JMT organic solution. The last system named does not follow ordinary liquid-liquid distribution behavior.

1.4 Separation of Magnesium from Calcium Metal by Sublimation (2)

Results of trials attempted during the previous quarter indicated favorable separation of magnesium from calcium was achieved using a vacuum sublimation process. Starting with calcium metal containing about 3000 ppm magnesium a series of sublimation experiments was run varying time of the experiment and particle size of the impure calcium. The temperature of the retort was held at 600°C in each run, the condenser temperature at 100°C and a vacuum of less than 10^{-3} mm Hg was maintained. Analyses indicated that a finely ground charge plus a long sublimation time give the greatest magnesium removal. A 150 g charge ground to 20 mesh and sublimed for 8 hours contained less than 100 ppm magnesium following sublimation.

As a result of the success of these small scale experiments an attempt was made to purify 100 pounds of calcium metal using some of the large scale vacuum distillation equipment designed for use in the large scale redistillation of calcium. After 16 hours of sublimation at 600°C the calcium had shown only a small decrease in the magnesium content.

1.5 Separation of Mo, Zr, Nd, and Ru from Uranium-Chromium Eutectic by Liquid-Metal Extraction (6)

Since uranium-chromium eutectic (5.18 w/o chromium) has been considered of interest as a possible fuel material for power reactors, a program has been initiated to study the possibility of removing fission products from this alloy by liquid-metal extraction. The general procedure has been to prepare uranium-chromium eutectic containing 760 ppm Mo, 3440 ppm Nd, 650 ppm Ru and 1190 ppm Zr and then to attempt to extract the Mo, Nd, Ru and Zr with various liquid-metal extractants at temperatures above the melting point of the U-Cr eutectic and that of the extractant.

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A number of extractants have been tried including pure magnesium, and the eutectics Mg-16.3 w/o Ca, Mg-52 w/o Zn, Mg-36.4 w/o Sn, Mg-33.2 w/o Pb, and Mg-59.5 w/o Bi. Approximately 100 grams of U-Cr eutectic containing the above additions and an equal volume of extractant are enclosed inside 15 mil, one inch diameter, tantalum crucibles, these crucibles are then enclosed inside #309 stainless steel jackets. Welding of the covers on the tantalum crucible and stainless steel jacket are carried out under an argon or helium atmosphere. This assembly is then placed inside a "Kanthal" wound resistance furnace and heated at 900 to 1000°C for two to three hours. The furnace is constructed to rotate back and forth through a 360 degree arc once each minute to provide mixing of the charge. After two to three hours the rotation of the furnace is stopped and the charge permitted to settle for approximately one hour at temperature. The crucible is then removed from the furnace and quenched in water. The quenched charge is then removed from the crucible, sectioned, and analysed.

Details of the rotating resistance furnace are shown in Figure 1. The furnace is fourteen inches long with an outside diameter of nine inches and an inside diameter of one and one half inches. The maximum temperature which can be attained inside the furnace is approximately 1200°C. One end of the furnace is closed and the other end can be opened to introduce the sample. Refractory spacers are used to position the sample near the center of the furnace; the spacers and sample are held in place by a cover plate (A). The furnace is supported within an angle iron cage (C) which is fastened to a steel drive shaft (E) supported by brass bearings (D). The drive shaft is fastened directly to the cage by collars (F) on opposite sides of the furnace. A flexible coupling (G) connects one end of this drive shaft to a reversible motor. The direction of rotation is reversed by means of a toggle switch which is tripped by a bar (B) fastened to the drive shaft.

Chemical analyses have not been completed. However, enough data have been obtained to indicate that the neodymium is almost completely removed by the various extractants tried while only very small amounts of the Mo, Zr, and Ru are removed. It has also been observed from microscopic examination of the U-Cr and extractant layers, that the various magnesium eutectic extractants used do not appreciably alter the U-Cr eutectic.

2. Preparation of Pure Compounds

2.1 Preparation of Thorium Tetrachloride (5)

The difficulties encountered in the simultaneous chlorination-sublimation apparatus for preparing thorium tetrachloride led to a decision to discard this method. Sublimation of

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Fig. 1 - Rotating resistance furnace

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thorium tetrachloride at atmospheric pressure required high temperatures which were troublesome and the purity and recovery of the product were unsatisfactory. Development was begun of a lower-temperature chlorination method in which the thorium oxide and carbon would be reacted with chlorine below the sublimation temperature of thorium tetrachloride. Chlorination was quite rapid at 600°C and the exothermic nature of the reaction caused excessively high temperatures in the reaction charge. These higher temperatures resulted in material losses by sublimation and in fusing of the partially reacted charge. Reducing the chlorine flow rate eliminated overheating. The thorium oxide and carbon was formed into pellets which reduced entrainment of the charge. The method seems to be capable of producing thorium tetrachloride suitable for vacuum sublimation purification.

A number of runs were made purifying thorium tetrachloride by sublimation under vacuum. The rate of sublimation increased with temperature. Below 700°C, sublimation was very slow and above 800°C the purity of the product was reduced by corrosion and entrainment of thorium oxide and carbon. The rate of sublimation decreases with time, probably due to formation of a layer of thorium oxide through which the chloride must diffuse. Sublimation of a 500 gram charge under 50 microns pressure was almost complete after 6 hours at 780°C. The product was a dense crystalline mass which assayed above 99 per cent thorium tetrachloride. Analyses for metallic impurities are not yet available.

2.2 Preparation of Niobium Pentachloride (2)

Niobium pentachloride was prepared by the chlorination of niobium carbide. Niobium pentoxide was mixed with carbon and heated to 1600°C in order to prepare the carbide and this was then chlorinated at 400°C in an anhydrous chlorine stream. The resulting niobium pentachloride was collected on an air-cooled condenser and stored under an inert atmosphere.

3. Metal Preparation Studies

3.1 Preparation of Vanadium Metal (5)

A batch of vanadium metal was prepared by reducing vanadium tetrachloride with magnesium at 800°C. The vanadium tetrachloride was prepared by chlorinating vanadium carbide and it was then purified by distillation through a fractionating column under 100 microns pressure. The mixture of vanadium powder, magnesium chloride, and magnesium from the reduction operation was divided into four samples. The vanadium metal was separated from one of these samples by acid leaching to remove magnesium and magnesium chloride. The vanadium powder was dried, pressed into a bar and arc-melted. The magnesium and magnesium chloride were removed from the other samples by heating under vacuum and the vanadium sponge arc-melted. The hardness and analysis of these samples are given in Table I.

Table I
Hardness and Analysis of Vanadium Metal

Sample	Hardness RA	Carbon	Nitrogen	Iron	Oxygen
Acid Leached	56	290 ppm	190 ppm	2%	2000 ppm
Vacuum Heated	68	310 ppm	415 ppm	---	2500 ppm
Vacuum Heated	53	270 ppm	430 ppm	0.1%	1690 ppm
Vacuum Heated	57	130 ppm	880 ppm	0.01%	3500 ppm

The vacuum heating procedure has been improved so that it yields vanadium metal of essentially the same purity as the acid leaching procedure. Iron contamination from the reduction vessel is still excessive and the oxygen content of the metal is rather high. If these impurities can be reduced this method will be capable of producing good purity vanadium metal.

3.2 Studies on the Preparation of Tantalum and Niobium Metals (1,2)

In an attempt to employ the carbon reduction of Nb_2O_5 to give soft metal without the high temperature sintering near the melting point, experiments were performed in which stoichiometric amounts of carbon and the oxide were approached.

One mixture of Nb_2O_5 and carbon with less than stoichiometric carbon and another mixture containing slightly greater than stoichiometric carbon were heated separately at about 1400°C under vacuum. The reacted mixtures were then analyzed and then combined in a ratio to give stoichiometric residual oxygen to carbon. This combination was then mixed and heated to 1800°C in a vacuum. The resulting metal had a Rockwell hardness of A-50 and could be cold rolled.

A mixture of one mole of T_2O_5 and five moles carbon was heated under vacuum to a temperature of 2400°C , then arc-melted. The resulting metal had a hardness of Rockwell A-40. This product could also be cold rolled.

A fine metallic powder believed to be niobium metal, has been prepared by mixing niobium pentachloride with magnesium chips and sealing the mixture in a stainless steel reaction furnace. The

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mixture was heated to 500°C and the temperature was slowly increased to 800°C. After cooling, a very fine black powder was collected and washed with water and dilute HCl. This powder has the characteristic of burning rapidly in air at low ignition temperatures.

3.3 Preparation of Thorium Metal (5)

Thorium metal of good purity was prepared by reducing purified thorium tetrachloride with magnesium. Magnesium chloride and excess magnesium were removed by heating under vacuum and the resulting thorium sponge was arc-melted. The reduction was carried out by heating a mixture of magnesium and thorium tetrachloride to 825° - 950°C. An excess of magnesium was used to form a thorium-magnesium alloy which collected as a solid biscuit. The reduction vessel was changed from a closed bomb to a stainless steel tube with an open magnesium oxide crucible to contain the reduction mixture. This eliminated the leakage of air which occurred with the closed bombs. An atmosphere of argon was maintained in the stainless steel tube to prevent oxidation of the thorium metal. Use of a 100 per cent excess of magnesium and a reduction temperature of 900°C gave a compact biscuit, well separated from slag, and a high yield of thorium metal.

The magnesium chloride and excess magnesium were removed from the reduction product by heating under vacuum at 925°C for one hour. The thorium sponge could be arc-melted without splattering or fuming due to volatile materials. The hardness of the thorium after arc-melting and annealing at 900°C ranged from RE 36 to 70. The oxygen content varied from 0.1 to 0.4 per cent, carbon from 100 to 250 ppm and nitrogen from 80 to 400 ppm. Most of the oxygen and carbon is believed to come from the thorium tetrachloride and could be reduced by better purification of the thorium tetrachloride.

4. Alloy Studies

4.1 Uranium-Zinc System (6)

Metallographic and thermal analyses of uranium-zinc alloys containing 80, 85, 90 and 97 w/o uranium show a new feature not indicated in the tentative phase diagram previously reported (ISC-532). This is a monotectic horizontal at 1030°C extending from about 70 w/o uranium to 100 w/o uranium, the monotectic being at about 93 w/o uranium. At 1030°C the liquid immiscibility gap extends from 70 to 93 w/o uranium. Further details will appear in ISC-576.

4.2 Thorium-Zirconium System (2)

During this report period work was continued on the high temperature x-ray examination of alloys of thorium-zirconium. Alloys

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of the following compositions, Th-25 w/o Zr, Th-40 w/o Zr and Th-60 w/o Zr were used in the studies undertaken. They were prepared by arc-melting together crystal bar zirconium and Ames thorium sponge. Specimens approximately 1 cm x 2.5 cm x 2 mm were sectioned from the arc melted buttons for use in the high temperature x-ray studies. Diffraction patterns were run on each specimen at room temperature, at 750°C and at 950°C. At room temperature alpha thorium and alpha zirconium were identified in the higher composition alloys. However, at the higher temperatures, particularly at 950°C, the room temperature phases disappear and lines corresponding to an unidentified high temperature phase appear. Further work on the nature of this phase is in progress.

4.3 Thorium-Hafnium System (2)

The investigation of the thorium-hafnium system has been continued. Alloys were prepared by arc melting zirconium-free crystal bar hafnium or bomb reduced hafnium sponge together with Ames reduced thorium sponge. They were arc-melted into bar form approximately 10 cm in length and with a uniform cross-section of 0.2 to 0.3 centimeter.

Electrical resistivity vs temperature data were obtained for alloys containing 5.2, 9.9, 22.1, 29.9, 39.9, 47.8, 65.4, 75.3, 77.6, 94.7 and pure hafnium. These measurements show clearly the presence of eutectoid type horizontal extending across the system at 1290°C.

There appears to be a minimum in the solidus occurring at approximately 1400°C although additional melting point data are needed to confirm this.

The temperature of the transformation in hafnium was studied by electrical resistivity vs temperature measurements on a bar of arc-melted hafnium sponge metal prepared by the bomb reduction of HfF_4 with calcium. A break in the resistivity-temperature curve was observed at 1950°C which is considerably higher than values usually reported for the hafnium transformation temperature.

4.4 Thorium-Carbon Alloy Studies (6)

The effect of carbon on the electrical resistivity of thorium at room temperature has been measured. The results obtained are shown in Figures 2 and 3. As shown in Figure 2 the extrapolated value for the resistivity of thorium of zero carbon content is 15.0 micro-ohm-cm. The resistivity rises rapidly with increase in carbon content. The extension of the curve to higher carbon contents is shown in Figure 3. This plot indicates that the limit of solubility of carbon in thorium at room temperature is 0.35 w/o. This value is in good agreement with the value obtained

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Figure 2 - Effect of carbon on the room temperature electrical resistivity of thorium.

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Figure 3 - Effect of carbon on the room temperature electrical resistivity of thorium.

by Peterson and Michaelson from metallographic data, ISC-463. The solubility limit associated with the ThC phase could not be determined from the resistivity measurements made since no well defined break was indicated. This is believed to be due to the very high resistivity of the monocarbide phase which reaches a maximum of the order of 245 micro-ohm-cm at about 4.3 w/o carbon, and partly due to the fact that the alloys in this composition range were prepared by powder metallurgy techniques and were quite porous. Resistivity measurements were also made at 800°C and above but the plots of resistance versus composition showed a much greater degree of curvature for thorium-rich alloys. The data also showed more scatter with increasing carbon content. Therefore, it was not possible to determine the solubility limits by this method. High temperature x-ray diffraction data appear more promising in this respect.

The temperature dependence of the electrical resistivity of crystal bar thorium is shown in Figure 4 and Table II. Chemical analysis of this metal showed it to contain 50 ppm nitrogen and 200 ppm carbon. A very well defined break is obtained at the transformation temperature.

Table II

Electrical Resistivity of Crystal-Bar Thorium

Temperature °C	Resistivity Micro-ohm-cm	Temperature °C	Resistivity Micro-ohm-cm
0	16.8*	1000	68.0
25	18.1	1100	71.5
100	22.8	1200	74.8
200	28.7	1300	77.2
300	34.6	1370	79.0
400	40.6	1400	86.1
500	46.1	1500	87.5
600	50.2	1600	88.5
700	55.9	1700	89.1
800	60.3	1750**	89.1
900	64.2		

*Extrapolated from room temperature.

**Melting point.

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Figure 4 - Temperature-dependence of the electrical resistivity of crystal-bar thorium.

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4.5 Uranium-Niobium System (3)

Additional x-ray studies have shown that the upper boundary line of the two-phase region of decomposed solid solution rises above 925°C instead of reaching only to 900°C as suggested in last quarter's report. The horizontal extent of this region appears to have been stated correctly.

An attempt was made to determine the boundary of the niobium-rich region at 500°C. Powder specimens of the 25, 30, 40 and 50 per cent niobium alloys were taken to 1000°C for 24 hours, then cooled to 500°C and held at that temperature for 300 hours after which they were cooled as rapidly as possible. The boundary lies below 50 per cent niobium but the lattice parameter values obtained on the other alloys, all of which showed two phases, varied slightly. A period of 300 hours appears to be insufficient to bring about equilibrium at 500°C.

Dilatometric studies of the uranium-rich alloys show that the eutectoid horizontal at 645°C can be traced down to one per cent niobium. A 0.5 per cent niobium alloy gave no evidence of this line but showed a pronounced anomaly at 665°C, presumably, indicating passage into the beta plus gamma condition.

New melting point determinations on the 5 and 10 per cent niobium alloys gave slightly higher values than earlier measurements indicated.

4.6 Tantalum-Zirconium System (3)

Work on the tantalum-zirconium alloy diagram continued during the quarter. Part of the investigation was concerned with the position of the solvus line that bounds the region of tantalum-rich solid solution. This line begins at the tantalum end of the eutectic horizontal, which has the coordinates 1830°C and 50 per cent tantalum, and follows a curving downward path that terminates near the tantalum side of the diagram. X-ray patterns of the 60, 70, 80, and 90 per cent tantalum alloys cooled rapidly from 1000°C indicate that the solvus line crosses the 1000°C horizontal beyond 80 per cent tantalum. Electrical resistance-temperature measurements indicate that at 800°C, the curve lies well beyond 80 per cent tantalum and certainly below 98 per cent tantalum. Tests on the 90 and 95 per cent alloys are yet to be made. Curves obtained from resistance-temperature measurements on the 60 per cent tantalum alloy in the 1500° to 1750°C temperature interval show a slight change in direction at 1700°C to 1725°C but the connection between this faint anomaly and the crossing of the solvus line is not established.

Attempts to determine the composition of the eutectic alloy by metallographic methods still fail to reveal any structure of eutectic

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character. Redetermination of the melting point of the 3 per cent tantalum agrees with previous measurements that the eutectic horizontal lies at 1830°C and that it approaches very near to the zirconium side of the diagram.

Resistance-temperature measurements and dilatometric data agree in placing the eutectoid horizontal near 800°C.

4.7 Zirconium-Zinc System (6)

Since it has not been possible to obtain dense homogeneous Zr-Zn alloys for compositions extending from about 5 to 50 w/o zinc, attempts are being made to determine the phase boundaries for this system from vapor pressure measurements. A dew-point method is being used for this purpose. Effort during this report period has been directed toward improving the experimental procedure so that more closely reproducible results may be obtained.

4.8 Tantalum-Vanadium System (2)

The investigation of the tantalum-vanadium system has been resumed. It has previously been shown that complete solid solubility exists at elevated temperatures and that at lower temperatures there exists an intermediate phase, believed to be similar to sigma, in iron-chromium, in the composition range of 60 to 75 w/o tantalum. This phase is stable up to 1300°C. Further data regarding the formula and structure of this phase are now being obtained. Series of alloys in the composition region of 60 to 75 w/o tantalum have been annealed for 100 hours at 1200°C, and for 423 hours each at 1100, 1000 and 900°C for metallographic examination. Alloy bars are now being prepared for use in determining the solidus curve.

5. Metal Coatings and Corrosion

5.1 Corrosion of Refractory Metals by Liquid Metals (5)

The corrosion of tantalum by uranium-chromium eutectic alloy to which 0.3 per cent by weight of silicon had been added was measured in the agitated capsule test. A tantalum tube partially filled with the alloy and enclosed in a stainless steel tube was mounted between two muffle furnaces maintained at 950° and 1050°C. The tube was tilted back and forth every seven minutes, causing the liquid alloy to flow from one end of the tube to the other. The most rapid corrosion occurred on the end cap at the hot end of the tube. The corrosion rate was 0.3 mils per day. Uranium-chromium eutectic alloy without the silicon addition tested in a similar manner at 900°C and 1000°C gave a corrosion rate of 0.7 mils per day.

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5.2 Corrosion of Coated Tantalum by Liquid Metals (3,5)

The carbide-coated tantalum crucible mentioned in the previous report as being in a 720 hour test of its ability to contain uranium-chromium eutectic at 1000°C was still holding up at the completion of the run. However, it was in very poor condition upon removal. A similar crucible was tested in the same way for 744 hours with about the same final result.

As the coatings of these crucibles were not more than 0.0001 inch thick, production of thicker coatings appeared desirable. The coatings had been formed with naphthalene as a source of carbon but additional trials with this substance failed to give increased thickness. Results obtained with toluene as a source of carbon also were unsatisfactory. Trial was then made of lamp black. The crucible was packed in the lamp black and heated at 2400°C for several minutes. This procedure yielded reasonably uniform coatings about 0.001 inch thick.

Tests of the more heavily coated crucibles filled with uranium-chromium eutectic were run at 1100°C. After periods of 336 hours and 768 hours, the coatings had not broken through and appeared to be capable of holding up for a considerably greater period.

A test of 120 hours duration at 1000°C was made of a crucible with a tantalum sulfide coating made by heating the crucible in the vapor of elemental sulfur. The crucible showed serious deterioration. Furthermore, the uranium-chromium eutectic showed a pronounced tendency to creep upward and over the rim of the crucible. Tests with sulfide coatings are being deferred in favor of the more promising carbide.

The effect of tantalum carbide coating on the corrosion of tantalum by liquid uranium metal was also studied under static conditions. The carbide coating was formed by heating tantalum crucibles to 1600°C in naphthalene for a few minutes. The resulting coating was less than 0.1 mil thick. This coating reduced the corrosion rate of tantalum by uranium at 1200°C to about one-half that of uncoated tantalum.

5.3 Corrosion of Carbides by Molten Uranium (2)

Work was continued on the investigation to ascertain the corrosion resistance of certain high-melting compounds to liquid uranium.

The tests were conducted by melting uranium metal in a crucible of the refractory being tested at a temperature of 1200 to 1300°C for one to two hours. Crucibles were prepared from the monocarbides of molybdenum, niobium, vanadium, tantalum and zirconium. All of the carbides were prepared by heating a mixture of the metal powder or oxide and carbon to approximately 2000°C in air or argon

atmosphere. The resultant powders were then pressed into crucibles under a pressure of 15,000 psi and fired to above 2000°C. Small amounts of paraffin wax were added to aid bonding. The appearance of the crucibles was generally good and, with the exception of TaC, indicated little porosity following the final firing.

The carbides of molybdenum, vanadium and niobium exhibited excessive penetration of the crucible wall by the uranium. After 1 hour at 1200°C only zirconium and tantalum carbides showed any promise of corrosion resistance under these conditions. Chemical samples were taken from the uranium specimen upon cooling and were analyzed spectrographically for the appropriate metallic element. The analytical results are shown in Table III.

Table III

Crucible Contamination of Uranium at 1200°C for 1 Hour

Crucible	Analysis of Uranium
MoC	0.15 w/o Mo
NbC	complete penetration of crucible by uranium
VC	0.1 to 1.0 w/o V
TaC	Not detectable (< 300 ppm Ta)
ZrC	225 ppm Zr

A crucible of ZrC containing uranium metal was tested in vacuo at 1300°C for 3 hours to determine the effect of still higher temperatures on its resistance to attack by molten uranium. Examination of the cross-section revealed complete penetration of the crucible wall by the uranium.

Several attempts were made to combine the two more favorable carbides into the reportedly high melting mixed carbide $4\text{TaC} \cdot \text{ZrC}$. However, the crucibles prepared from such a mixture were extremely brittle and cracked extensively before testing could be attempted.

5.4 Metal Coating (6)

Aluminum can be readily wet with pure lead which is well bonded to the aluminum; however, the lead coat is quite rapidly separated from the aluminum by boiling distilled water in two to 24 hours. The addition of zinc to the lead bath retards the separation of the coat but does not prevent separation of the coat. Consequently, lead does not appear to be a feasible solder for a bonded uranium fuel element, but it might still be of interest as a protective coat for an unbonded slug, or as a diffusion barrier between a zinc-coated slug and the aluminum can.

An attempt has been made to improve the ductility of Cu-9.7 w/o Mg dip coats which have been successfully applied to uranium. Heat treatment of the Cu-9.7 w/o Mg alloy in a lead or bismuth bath at 400°C effectively removes the magnesium from the alloy to give a soft two phase Cu-Pb or Cu-Bi alloy. The copper-lead alloy formed has good corrosion resistance to 178°C water. The results obtained show that the magnesium can be removed from a 1 to 2 mil coat of Cu-9.7 w/o Mg in 15 to 30 seconds. (For further details see ISC-576.)

5.5 Corrosion of U-Zr-Nb Alloys (2)

Investigation of the ternary U-Zr-Nb alloys was continued. The high temperature tensile strength of the U-3 w/o Zr-3Nb, U-2.5 Zr-5Nb and U-5Zr-2Nb was measured at temperatures up to 600°C. In general the ternary alloys have higher tensile strength at 400°C than uranium has at room temperature.

Corrosion tests were made on several gamma quenched alloys which had been isothermally aged. Corrosion rates and electrode potentials were determined for a series of alloys annealed for 1000 hours at 375°C and for a series annealed at 400°C.

This work has been reported in detail in ISC-576.

5.6 Casting Uranium into Zirconium Containers (2)

Work on the casting of uranium into zirconium containers has been continued with emphasis on the capping of the canned slug. A new die for forming caps and an improved inert gas arc-welding chamber have been constructed. It is anticipated that by eliminating flaws in the caps and by improving the quality of the welds, a corrosion-resistant closure can be made. A glass autoclave has been constructed and tested with water up to 150°C.

This work has been reported in detail in ISC-576.

6. Solid State Investigations

6.1 Physical Constants of Metals from Ultrasonic Measurements (4)

Measurement of the elastic constants of metal single crystals by ultrasonic velocity measurements has continued. A single crystal of lead was cut with a jeweler's saw so that pulses could be propagated in the $\langle 100 \rangle$ direction. Values of $C_{11} = 4.84 \times 10^{11}$ dyne/cm² and $C_{44} = 1.48 \times 10^{11}$ dyne/cm² were obtained. Corresponding values quoted by Jones* are: $C_{11} = 4.76 \times 10^{11}$ dyne/cm² and $C_{44} = 1.44 \times 10^{11}$ dynes/cm².

Low temperature apparatus has been constructed which is capable of yielding data down to liquid nitrogen temperatures. Some difficulty has been encountered in finding a suitable cement to couple the quartz driver-crystal to the sample crystal. A rubber-vaseline mixture has been found to work best at low temperature.

6.2 Calcium Allotropes (4)

X-ray diffraction patterns and electrical resistivity measurements were taken on calcium at various temperatures. Three grades of calcium were used in the investigation. Analytical data are shown in Table IV, with the exception of the nitrogen analysis which was obtained by a micro-Kjeldahl method, the analytical results were obtained by emission spectroscopy.

X-ray diffraction results were reported for calcium A in the previous report. The results were duplicated on powdered metal and on massive samples. In the intermediate purity calcium B the h.c.p. phase was not present as a stable phase. In the high purity calcium A only two stable phases were found: f.c.c. from room temperature to 464°C and b.c.c. from 464°C to the melting point. A comparison of the results of the diffraction studies with the results of Graf** is shown in Table V.

Electrical resistance measurements were made to check the transition points. Graphs of resistance versus temperature for calcium A and calcium C are shown in Figures 5 and 6. The correspondence between the x-ray data and the resistivity data is quite satisfactory.

The results indicate that high-purity calcium metal exists in only two allotropic modifications. The high-temperature phase is, contrary to existing literature reports, b.c.c. rather than h.c.p. The sensitivity of structure to small amounts of contamination is quite striking and was unexpected.

*H. Jones, Physics, 15, 13 (1949).

**L. Graf, Phys. Z., 35, 551 (1934).

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Table IV

Analysis of Calcium Metal

Impurity	Calcium A Analysis in w/o	Calcium B Analysis in w/o	Calcium C Analysis in w/o
Mg	0.300	0.110	0.010
N	0.025	0.022	0.011
Fe	0.006	0.010	0.010
Al	0.001	0.001	0.001
Mn	0.004	0.005	0.005
Net Ca	99.66	99.85	99.96

Table V

Comparison of Observed Transitions in Calcium

Temp.	Graf*	Calcium A 99.66%	Calcium B 99.85%	Calcium C 99.96%
100°C	f.c.c.	f.c.c. $a=5.612 \pm 0.012 \text{ \AA}$	f.c.c.	f.c.c. $a=5.582 \pm 0.004 \text{ \AA}$
200°C				
300°C	300°C	300°C	300°C	
400°C	Complex 450°C	Complex 375°C	Complex 450°C	464°C
500°C	h.c.p. $c/a = 1.64$	h.c.p. $c/a = 1.63$ 500°C		
600°C		b.c.c. $a=4.474 \pm 0.004 \text{ \AA}$		b.c.c. $a=4.477 \pm 0.007 \text{ \AA}$
700°C				
800°C				

*Data represent Graf's best Ca

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Fig. 5 - Temperature dependence of resistivity of calcium
Sample A - Heating cycle

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Fig. 6 - Temperature dependence of resistivity of calcium
Sample C - Heating cycle

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6.3 Structure of ZrGe₂ (2,4)

The structure of the compound ZrGe₂ was investigated by x-ray diffraction. Single crystals were used, and diffraction patterns were taken on a precession camera. The crystals were shown to belong in the orthorhombic system with the space group symmetry D_{2h}^{17} -Cmcm. The lattice constants were determined as:

$a = 3.764\text{\AA}$, $b = 14.97\text{\AA}$, and $c = 3.796\text{\AA}$. From these values and density data it was calculated that there are four formula weights per unit cell.

Intensity data were acquired by visual comparison of diffraction spots on photographic films using timed exposures. Patterson and Fourier syntheses were run with these intensity data. The best fit for the intensity data was obtained by using three sets of fourfold positions of the type: $0, y, 1/4$; $0, -y, 3/4$; $1/2, 1/2 + y, 1/4$; $1/2, 1/2 - y, 3/4$. The values obtained for the parameters were: $y = 0.106$ for the Zr atoms, $y = 0.750$ for one Ge set, and $y = 0.445$ for the second Ge set. Further refinement of the data may change these parameters slightly, but any changes are expected to be minor.

A powder pattern of a Zr-42 w/o Ge alloy was indexed on the basis of the above mentioned knowledge and the phase ZrGe₂ was identified in this alloy. An additional 70 lines of the powder pattern could not be accounted for and indicate the presence of an unknown compound in this region. An attempt was made to obtain single crystals of this phase for x-ray studies. The crystal habit of alloys in this region is such as to produce tiny bundles of aligned columnar crystals. A rotation pattern of one such bundle was obtained and from the layer line spacing one lattice dimension was calculated to be about $5.36\text{\AA}$.

6.4 Study of the Zirconium-Uranium Epsilon Phase (5)

Powder x-ray diffraction patterns from the epsilon phase in the uranium-zirconium system have been obtained and indexed. This phase was found to be body centered cubic with a lattice constant of $10.67\text{\AA}$. The space group is at least T_d^3 and may be one of higher symmetry. A single crystal of this phase was obtained from a 60 weight per cent zirconium alloy. Rotation and Weissenberg pictures from this crystal confirmed the choice of unit cell. This structure appears to be similar to the gamma brass structure. It is derived from 27 unit cells of the body centered solid solution with a shift in atomic positions or an ordering process producing additional diffraction lines. The phase appears to exist over composition range from 40 to 60 weight per cent zirconium.

7. Other Investigations7.1 Reclamation of Zirconium Chips (2)(a) H₂SO₄ Pickling Treatment

The effect of the sulfuric acid pickling of zirconium and zircaloy scrap upon the corrosion resistance of the final metal has been studied. Corrosion specimens approximately two millimeters thick and one centimeter square were prepared from the following:

(1) Small bar specimens which were found in the scrap and which were assumed to be samples representative of the original stock.
(2) Zirconium lathe turnings and machine chips both as-arc-melted and as-melted in graphite. (3) Zircaloy^{#2} turnings and chips both as-arc-melted and melted in graphite. The samples to be tested were sealed in a water-filled steel bomb with a pyrex test tube as a liner. The liner was inserted to discourage any galvanic action between the zirconium and the steel. The bombs were then placed in a furnace and held at 300°C for 300 hours. When the bombs were opened the pyrex test tubes were found disintegrated into layers of quartz-like shells. All except one of the samples had sustained weight gains. Two of the samples had relatively large weight gains; noticeable structural flaws had developed in these particular samples. The data secured is summarized in Table VI. Because of the questionable results this experiment

Table VI

Metal Tested	Weight Change in mg/cm ² /day
Pieces of original bar stock, Zr	2.96
Zirconium chips, arc melted	0.88
Zirconium chips, graphite cast	20.0
Zirconium turnings, arc melted	31.2
Zirconium turnings, graphite cast	3.00
Zircaloy chips, arc melted	3.11
Zircaloy chips, graphite cast	4.36
Zircaloy turnings, arc melted	2.425
Zircaloy turnings, graphite cast	3.191

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is being repeated without the use of the pyrex liners in the steel bomb.

(b) Zinc Used in Treatment of Scrap Metal

The intention of this investigation is to alloy the zirconium or zircaloy metal scrap with zinc followed by a removal of the zinc by vacuum distillation in hopes of obtaining some purification which would result in ductile metal.

As previously reported, attempts have been made to alloy the zirconium and zinc by heating in a sealed crucible; however, these attempts have failed to produce massive alloys due probably to zinc vaporization at elevated temperatures. In order to prepare the zirconium-zinc alloy the scrap material was mixed with calcium and zinc fluoride; this mixture was packed in a sintered calcium fluoride lined bomb, and the bomb charge was fired. This resulted in a massive, well formed alloy containing 30 to 35 w/o zinc. This material was then melted in a graphite crucible in vacuo during which the zinc was distilled off. Metal thus treated had a hardness of Rockwell A-48 as compared with a hardness of A-57 for the original untreated material similarly melted. The quality of the final metal seems to be somewhat dependent on the quality of the calcium used to reduce the zinc fluoride.

7.2 Vacuum Fusion Analysis of Metals (5)

Vacuum fusion analysis of lanthanum for oxygen and nitrogen has so far been unsuccessful. An iron bath has been used at temperatures from 1500° to 1700°C. In all cases the gas was evolved from the bath very slowly and recovery of the oxygen and nitrogen was incomplete. Temperatures up to 1850°C will be tried and if the evolution of gas from the iron bath is still incomplete, other metals will be investigated as bath materials.

7.3 Steel Mold Dressings in Uranium Casting (1)

In the casting of uranium rods in steel molds a number of mold dressing treatments have been tested. The steel molds have a 3/4 inch wall thickness since they are made from 3 inch O.D. steel tubing having a 1.5 inch bore. Treatment of the molds with a zirconia slip made from commercial stabilized zirconia gave fairly good coverage if the molds were warmed previous to treatment so that fairly rapid drying of the slip in position could be accomplished. However, equally good if not better, results were obtained when the molds were given a benzene smoke treatment instead. So for a number of castings of some special uranium alloy rods, the benzene smoke treatment was employed.

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The treatment with benzene smoke was that adopted a number of years ago on the basis of only a few exploratory tests. In this treatment the warm steel mold, 30 inches in length, serves as a chimney as a benzene soaked paper is burned at its base. The mold is reversed at about half way through the smoking operation. After the smoking the loose feathers of soot are blown out of the mold and the mold is unplugged at one end and set in position.

7.4 Melting Uranium in Oxide-Painted Graphite Crucibles (1)

On the basis of previous experiments a paint made from commercial stabilized zirconia suspended in a solution containing magnesium nitrate was applied to the inner surface of the graphite crucibles for melting uranium metal.

The crucible was then heated in air to above 400°C to decompose the magnesium nitrate which served as a binder to give a more durable coating of zirconia on the crucible walls. Uranium rods cast from melts made in these treated melting crucibles contained less than 200 ppm carbon.

7.5 Low Melting Fuel Element Studies (1)

A quaternary alloy made by mixing the low-melting eutectic from aluminum-uranium with the low-melting eutectic from magnesium-thorium gave a lower melting alloy which gave a eutectic type break in the cooling curve at 460°C. Results of the experiment have not been interpreted but it is suspected that the 460°C temperature is associated with aluminum and magnesium solution and may not involve solutions of uranium and thorium.

7.6 Hot Pressing of Calcium Fluoride (5)

Calcium fluoride has interesting properties as a potential refractory crucible material. It is one of the cheapest and most stable of the non-oxide refractories. For experimental work completely non-porous shapes and crucibles would be desirable. An investigation of hot pressing as a rapid and convenient method of fabricating such shapes was initiated. Cylindrical samples were formed in a heated graphite die at various temperatures and pressures. At 1200°C and 850 psi the resulting cylinders were dense and strong. The porosity was less than one per cent and dye absorption was negligible. Work will be continued to find the best temperature and pressure for hot forming.

7.7 High Temperature Furnace for Growing Single Crystals (2)

Work was started on the problem of preparing single crystals of thorium as well as other high melting metals. The Bridgeman technique of growth from the melt under a steep temperature

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gradient was adopted. The two major difficulties in this method are the construction of a suitable furnace and control mechanism and the selection of sufficiently unreactive materials for containing the metals.

A furnace was designed and constructed which satisfactorily meets the specifications for crystal growing at temperatures up to about 2000°C. It consists of a graphite split-tube core with a uniform 1 inch inside diameter and a varying outside diameter. A high current is passed through this tapered core, producing temperatures along the core which correspond to the power being generated at any point. By suitable selection of thermal insulation and core cross-sections a uniform temperature gradient of over 100°C per axial inch was obtained. The furnace is operated in an evacuated chamber although provision was made for possible use of an inert atmosphere.

The temperature is controlled by two motor driven variable autotransformers. One of these transformers controls the total input voltage to a fixed ratio, high current step-down transformer which is directly coupled to the furnace. The second transformer provides a variable primary voltage to several filament type transformers whose secondaries are in series with the line voltage applied to the first autotransformer. Thus a coarse and a fine temperature control is achieved.

The container problem was apparently solved for the two metals, thorium and nickel, by using a fine grade of high fired BeO for the crucibles. The crucibles are fabricated so that the inside tapers to a point at the bottom. However, with the one crystal growth attempt with thorium, the metal was initially too impure to detect the presence of possible additional oxide inclusions from the crucible.

Up to this time only one run using thorium metal and a crucible test run with nickel have been conducted. The thorium sample was polycrystalline although a distinct segregation of the oxide dendrites and inclusions occurred. The upper quarter of the sample contained many clusters of the oxide while in the lower portion the oxide appeared in the form of widely separated tree-like dendrites. No reaction was detected between molten nickel and BeO in the preliminary test.

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APPENDIX I: LIST OF REPORTS FROM THE AMES LABORATORY

1. Reports for Cooperating Laboratories

- ISC-380 W. C. Shaw, D. E. Hudson and G. C. Danielson. Electrical Properties of Crystalline Boron. UNCLASSIFIED.
- ISC-458 H. A. Wilhelm and R. Foos. A Countercurrent Liquid-Liquid Extractor. CONFIDENTIAL.
- ISC-469 W. A. Hunt and D. J. Zaffarano. Radioactive Disintegration Spectra of Some Short-Lived Nuclides. UNCLASSIFIED.
- ISC-497 J. Dooley and H. Shull. Scintillation Counters for Slow Neutrons. UNCLASSIFIED.
- ISC-498 L. O. Herwig and G. H. Miller. Ionization Yields of Fission Fragments in Gases. UNCLASSIFIED.
- ISC-499 A. W. Snyder and D. J. Zaffarano. Device for Detection and Identification of Charged Particles from Photonuclear Reactions. UNCLASSIFIED.
- ISC-507 Compiled by P. Chiotti and O. N. Carlson. Hanford Slug Program. Quarterly Summary Research Report. April, May and June, 1954. SECRET.
- ISC-510 R. M. Kline and D. J. Zaffarano. Decay Characteristics of Some Short-Lived Nuclides of Low Atomic Number. UNCLASSIFIED.
- ISC-515 J. T. Jones, Jr. and E. N. Jensen. Radiations from Cerium¹⁴¹. UNCLASSIFIED.
- ISC-517 W. G. Borduin and G. S. Hammond. Substituent Effects on the Spectra and Ionization Constants of Diaroyl-methanes. UNCLASSIFIED.
- ISC-518 P. O. Davey and G. C. Danielson. Thermal Conductivity of Nickel. UNCLASSIFIED.
- ISC-520 J. J. Ford and J. S. Fritz. A Titrimetric Determination of Thorium. UNCLASSIFIED.
- ISC-521 F. J. Hughes and D. S. Martin, Jr. Kinetics of the Hydrogen Peroxide Oxidation of Selenious Acid. UNCLASSIFIED.

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- ISC-522 A. V. Pohm, R. C. Waddell, J. P. Powers and E. N. Jensen. The Beta Spectrum of C^{14} . UNCLASSIFIED.
- ISC-524 C. V. Banks and R. J. Davis. Analytical Applications of the Reaction of Thorium with Benzenephosphonic Acid. UNCLASSIFIED.
- ISC-528 N. Barson and M. Smutz. Pilot Plant Production of Thorium Fluoride. SECRET.
- ISC-539 M. Q. Freeland and J. S. Fritz. High Precision Micro Spectrophotometric Analysis with Application to Vanadium-Aluminum Alloys. UNCLASSIFIED.
- ISC-540 J. S. Fritz and S. S. Yamamura. Rapid Microtitration of Sulfate. UNCLASSIFIED.
- ISC-543 F. R. Duke and R. W. Laity. The Measurement of Transport Numbers in Pure Fused Salts. UNCLASSIFIED.
- ISC-547 G. R. Winders and R. W. Fisher. An Electro-Magnetic Pump Heating Transformer for High Temperature Liquid Metals. UNCLASSIFIED.
- ISC-548 G. H. Beyer and F. M. Jacobsen. Operating Characteristics of a Centrifugal Extractor. UNCLASSIFIED.
- ISC-550 G. R. Pulliam and E. S. Fitzsimmons. A Study of the Reaction of Thorium with Some Pure Refractory Compounds. UNCLASSIFIED.
- ISC-551 D. R. Wilder. High Temperature Laboratory Furnaces. UNCLASSIFIED.

2. Papers Published in Scientific Journals

- Brehm, R. K. and V. A. Fassel
Direct Spectrochemical Analysis with a Rapid Scanning Spectrometer. Spectrochim. Acta 6, 341-372, (December, 1954).
- Bureau, A. J., H. A. Austrheim, and D. J. Zaffarano
An Electron Injector for a 70 Mev Synchrotron. Rev. Sci. Instr. 25, 1129-1130, (November, 1954).

- Chiotti, P.
High Temperature Crystal Structure of Thorium. J. Electrochem. Soc. 101, 567-570, (November, 1954).
- Fisher, R. W. and G. R. Winders
Lab for Radiochemistry, Metallurgy Tests. Nucleonics 12, 44, (November, 1954).
- Fritz, J. S.
Titration of Bismuth with Ethylenediaminetetraacetic Acid. Anal. Chem. 26, 1978-1980, (December, 1954).
- Fritz, J. S. and M. Q. Freeland
Direct Titrimetric Determination of Sulfate. Anal. Chem. 26, 1593-1595, (October, 1954).
- Hansen, R. S. and B. H. Clampitt
The Apparent Adsorption of Some Aliphatic Compounds from Aqueous Solutions as Inferred from Hydrogen Over-voltage Measurements. J. Phys. Chem. 58, 908-911, (October, 1954).
- Kline, R. M. and D. J. Zaffarano
Decay Characteristics of Some Short-Lived Nuclides of Low Atomic Number. Phys. Rev. 96, 1620, (December, 1954).
- Johansson, Sven A. E.
The Decay of UX_1 , UX_2 and UZ . Phys. Rev. 96, 1075-1080, (November, 1954).
- Spedding, F. H. and J. E. Powell
Methods for Separating Rare Earth Elements in Quantity as Developed at Iowa State College. Transactions, AIME 200, 1131-1135, (October, 1954).
- Spedding, F. H. and J. E. Powell
A Practical Separation of Yttrium Group Rare Earths from Gadolinite by Ion-Exchange. Chem. Eng. Prog. Symposium Series, No. 14 50, 7-15, (1954).
- Winders, G. R., E. H. Dewell, R. G. Clark, M. O. Voss and K. L. Malaby.
Equipment for Processing Reactor Fuels. Nucleonics 12, 50-51, (November, 1954).

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APPENDIX II: LIST OF SHIPMENTS

<u>Destination</u>	<u>Item</u>
Astrophysical Lab, Vatican Observ. Attn: The Rev. Joseph Junkes S. J. Castel Gondolfo, Rome, Italy	2 g lutetium oxide
Carbide and Carbon Chemical Company Union Carbide and Carbon Corporation P. O. Box P Oak Ridge, Tennessee	2 lb anhydrous thorium tetrachloride
Consulate General of Denmark Commercial Section 17 Battery Place New York 4, New York	1 g lanthanum oxide 1 g cerium oxide 1 g praseodymium oxide 1 g neodymium oxide 1 g samarium oxide 1 g erbium oxide 1 g ytterbium oxide 1 g yttrium oxide 100 mg holmium oxide 100 mg dysprosium oxide 100 mg thulium oxide 100 mg lutetium oxide
Dow Chemical Company Midland, Michigan	10 g samarium metal
Dow Chemical Company Midland, Michigan	50 mg holmium oxide 50 mg thulium oxide 50 mg lutetium oxide 50 mg terbium, 90%
Dow Chemical Company Dr. Daniel R. Stull Midland, Michigan	sample of uranium alloy
E. I. du Pont de Nemours and Co. Jackson, South Carolina	10 g lanthanum oxide 1 g cerium oxide 1 g samarium oxide 1 g gadolinium oxide 1 g erbium oxide 1 g yttrium oxide 1 g dysprosium oxide 1 g ytterbium oxide 100 mg holmium oxide 100 mg thulium oxide 100 mg lutetium oxide

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<u>Destination</u>	<u>Item</u>
Eldorado Mining and Refining, Ltd. Refining Division Port Hope, Ontario, Canada Attn: B. C. Smart	1 g lanthanum oxide 1 g cerium oxide 1 g praseodymium oxide 1 g neodymium oxide 1 g samarium oxide 1 g gadolinium oxide 1 g erbium oxide 1 g yttrium oxide 1 g ytterbium oxide 100 mg dysprosium oxide 100 mg thulium oxide 100 mg lutetium oxide
Geophysical Measurement Corp. 804 Wright Building Tulsa, Oklahoma Attn: S. A. Scherbatskoy	5 g samarium oxide
Dr. Jules Gueron C. E. A. (DCC360) 69 rue de Varenne Paris 7e, France.	2 g lutetium metal 2 g ytterbium metal 2 g thulium metal 2 g erbium metal 2 g holmium metal 2 g dysprosium metal 2 g yttrium metal 10 g praseodymium metal
Los Alamos Scientific Laboratory Los Alamos, New Mexico	10 g terbium oxide, 90%
University of California Barrows Lane and Bancroft Way Berkeley 4, California	1 g lanthanum oxide 1 g cerium oxide 1 g praseodymium oxide 1 g neodymium oxide 50-100 mg of the remaining rare earths (oxides) except terbium, europium, and promethium
University of California Radiation Laboratory, H-92606 Cyclotron Site East End of Hearst Ave. Berkeley 4, California	2 g lutetium oxide
Vanderbilt University Physics Department 4B-Garland Hall Nashville, Tennessee	200 mg thulium metal

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<u>Destination</u>	<u>Item</u>
Washington University Department of Chemistry Louderman Hall St. Louis 5, Missouri	1 g ytterbium oxide

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APPENDIX III

- (1) H. A. Wilhelm
- (2) O. N. Carlson
- (3) B. A. Rogers
- (4) J. F. Smith
- (5) D. Peterson
- (6) P. Chiotti

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