Semi-annual summary research report in engineering

Ames Laboratory Staff
Iowa State College

F. H. Spedding
Iowa State College

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ENGINNEERING. SEMI-ANNUAL SUMMARY RESEARCH REPORT
JULY-DECEMBER, 1955
UNCLASSIFIED
ISC-710

UNITED STATES ATOMIC ENERGY COMMISSION

SEMI-ANNUAL SUMMARY RESEARCH REPORT IN ENGINEERING

For July - December, 1955

by

Ames Laboratory Staff

February 27, 1956

Ames Laboratory
at
Iowa State College
F. H. Spedding, Director
Contract W-7405 eng 82

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SEMI-ANNUAL SUMMARY RESEARCH REPORT IN ENGINEERING

For the period July - December, 1955

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

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Monazite Processing (M. Smutz)*

Monazite sands have been processed for thorium, uranium and rare earths for more than a half century. Two new processes have been developed as a result of work done for the Atomic Energy Commission by the Battelle Memorial Institute and the Ames Laboratory. The purpose of this investigation was to develop another process that would overcome some of the disadvantages of these processes.

A process was developed that appears economically feasible. The first step in the process is a sulfuric acid digestion of the sands. The digested sands are dissolved in water and decanted from undigested sand and insoluble silica. Oxalic acid is added to the resulting solution to precipitate the thorium and rare earths, leaving the uranium in solution. The thorium, uranium and rare earths are then purified by solvent extraction procedures. The advantages of the process are that the thorium and uranium are separated virtually quantitatively in an early step and only one filtration step is necessary. The only disadvantage is the added cost of oxalic acid.

The investigation also included some work on alternative methods of purifying the uranium and thorium obtained after the oxalation step. It was demonstrated that the uranium could be recovered completely from the sulfate-phosphate liquor by using a solution of 10 percent octyl pyrophosphoric acid in kerosene as the solvent. Additional studies revealed that the uranium could be precipitated from the solvent as the tetrafluoride with 48 percent hydrofluoric acid.

An alternate process for separating thorium from the thorium-rare earth mixture was developed. The cerium was oxidized to the ceric state and was extracted with the thorium. The thorium purification work was coupled with the extraction of over 90 percent of the cerium along with the thorium into undiluted tributyl phosphate. The

*Names indicate group leaders in charge of work.
high extraction of cerium is obtained by calcining the oxalates prior to preparing the rare earth-thorium feed solution. Upon nitration the cerium present in the calcines is converted to basic ceric nitrate which is readily extracted by tributyl phosphate. The advantage in this is that the largest rare earth component is separated, for the most part, from the remaining rare earths, thereby allowing the other rare earths to be separated from one another more easily. The disadvantage is that the thorium must now be separated from the ceric cerium. It was demonstrated that this disadvantage could be overcome by selectively stripping the ceric cerium away from the thorium with a 0.1 molar aqueous solution of sodium nitrite, or that both components could be stripped from the tributyl phosphate with a sodium nitrite-sulfuric acid stripping system, followed by a mesityl oxide thorium extraction for final purity.

2. Uranium from Phosphates (G. L. Bridger)

A report entitled "Recovery of Uranium from Slag from the Electric Furnace Production of Phosphorous" by H. Cammack and G. L. Bridger is being distributed.

ABSTRACT

When phosphate rock is reduced to elemental phosphorus with coke in the electric furnace process, the uranium in the phosphate rock virtually all goes into the by-product slag. It is estimated that approximately 300 tons of uranium is potentially recoverable per year from this use of phosphate rock.

Previous work at Battelle Memorial Institute, Mound Laboratory, and the Tennessee Valley Authority did not result in an economic process for the recovery of uranium from electric furnace slags. The present investigation included thermal reductions with alkaline earth metals, high temperature liquid-liquid extraction with molten extractants, solubilizing fusions, magnetic separations, and leaching with various solutions. As an adjunct to the high temperature liquid-liquid extraction studies, the solubility of uranium in antimony, bismuth, lead, silver and tin were further studied, to corroborate and extend existing data.
Radioassay methods of analysis were developed for the raw furnace slags and residues from the various experimental treatments. A fraction of the slag residues was checked for uranium using a colorimetric procedure. All binary alloys resulting from the uranium solubility study were analyzed with colorimetric procedures developed during the course of the investigation.

The results of the thermal reduction experiments do not indicate a concentration of uranium in either layer of mechanically partitioned slag residues. Bismuth, lead and manganese appear to extract the most uranium from furnace slags by a single contact of the metal with the slag. In all high temperature extraction treatments, about 50 percent of the uranium appeared amenable to extraction. The addition of calcium, magnesium, aluminum and potassium to the melt with iron extractant appeared to improve the uranium extraction. Iron sulfides were found to extract approximately 45 percent of the uranium from the slags by a single contact with the slags.

Contacting one portion of furnace slag with three successive portions of iron-calcium, bismuth or iron sulfide extractant did not improve the total uranium extraction from the slag to above 50 percent. Contacting one portion of these extractants with several portions of furnace slag did not result in a marked increase of uranium concentration in the extractants. Because of the inability to build up a sizeable uranium concentration in an extractant, as shown in the study of uranium distribution between furnace slag and bismuth, the success of a proposed high temperature extraction process seems unlikely.

The only solubilizing fusion treatment which resulted in any appreciable elimination of uranium from the slag was the one with calcium chloride. The uranium was not found concentrated in the magnetic material in the slag. None of the leaching agents tried extracted an appreciable amount of uranium from the slag. Cost calculations based on data obtained indicate that none of the treatments studied would result in an economic recovery process.

The data obtained for the solubility of uranium in antimony agree fairly well with similar data reported by Massachusetts Institute of Technology. The data obtained
for the solubility of uranium in bismuth agree with values interpolated from the established binary phase diagram, but lie below the values reported by Massachusetts Institute of Technology, and above values reported by Brookhaven National Laboratory. The data obtained for the solubility of uranium in lead agree with values reported by the Massachusetts Institute of Technology, but lie below values interpolated from the established binary phase diagram. The data for the solubility of uranium in silver lie close to the reported solid solubility of uranium in silver, but lie well below the values reported by previous investigators. The data obtained for the solubility of uranium in tin agree with values reported by Massachusetts Institute of Technology, but lie below the values interpolated from the established binary phase diagram.

A report (ISC-678) entitled "Effect of Phosphate Rock Particle Size on recovery of Uranium from Superphosphates" by W. W. Davis and M. Smutz is being distributed.

ABSTRACT

The tremendous domestic reserves of phosphate rock together with the large annual production of normal superphosphate from phosphate rock have made the recovery of the small amounts of uranium (0.01 to 0.02 percent) in phosphate rock during the production of superphosphate an important problem. At current superphosphate production rates there exists a uranium potential of approximately 1000 tons per year.

In most phosphate rocks 80 percent or more of the phosphorus content is unavailable to plant life. Normal superphosphate is produced by the reaction of sulfuric acid and phosphate rock to convert the phosphorus to a form available to plant life. The purpose of this investigation was to make a quantitative study of the effect of phosphate rock particle size on the recovery of uranium during the production of normal superphosphate.

Florida land pebble phosphate rock was used in this work. The uranium content of this rock was 0.0184 percent
U₃O₈. Phosphate rock particle sizes of 62, 77, and 92 percent through 200 mesh were studied. Both a 1.81 and a 2.50 acidulation ratio (pounds of 100 percent sulfuric acid per pound of P₂O₅) were studied at each particle size. Industrially, a 1.81 acidulation ratio is used for the production of normal superphosphate; a 2.50 acidulation ratio is used for phosphoric acid production.

The acidulation of the phosphate rock to superphosphate and the extraction of the uranium into the solvent were accomplished simultaneously by adding the sulfuric acid to a slurry of the rock and solvent. The solvent used was a 10 percent di-octyl pyrophosphoric acid solution in normal heptane. A solvent to rock ratio of 0.8 milliliters per gram was used throughout; 60 percent sulfuric acid was also used throughout. The most favorable uranium recovery was 73 percent.

When the 2.50 acidulation ratio was used an additional quantity of rock was added to the reaction products, after the solvent had been removed, to reach an overall 1.81 acidulation ratio. Superphosphate was thus produced. This additional rock was not finely ground as it did not contact the solvent for uranium extraction.

It was found that the uranium recovery obtained in a 30 minute reaction-extraction increased significantly with a decreasing particle size. The process based on an initial over-acidulation ratio (2.50) did not appear to have any advantage over the process based on the normal acidulation ratio (1.81).

3. Rare Earth Separation by Solvent Extraction (M. Smutz and F. H. Spedding)

Accurate equilibrium data for the system tributyl phosphate, rare earth nitrates, water and nitric acid have been obtained. Simulated continuous extraction runs have been made, maintaining the aqueous nitric acid acidity at about 5.0 normal. Results of the run came out as predicted by the calculation method developed verifying the numerous assumptions made. Pilot plant runs with a 36-stage extractor have been delayed because of numerous mechanical difficulties. Although not demonstrated as yet, it seems apparent that solvent extraction
will have a role to play in processing rare earths on a commercial scale. If extremely high purity rare earths are required for a given application, ion exchange processes will probably be used in conjunction with solvent extraction.

4. Zirconium Process Development (G. H. Beyer)

The principal method of producing ductile zirconium is the Kroll Process. Since the calcium reduction of zirconium tetrafluoride has been proven economically competitive, the magnesium reduction of zirconium tetrafluoride will be considerably more economical if ductile zirconium can be obtained.

Numerous reductions of zirconium tetrafluoride with magnesium as a reductant and zinc fluoride as an internal booster yielded good recoveries and excellent metal-slag separation. However, the resulting dezinced zirconium had a Rockwell "A" hardness of 62 or greater. The hardness of the zirconium is probably caused by oxide contamination.

The source of the oxide contamination comes either from the magnesium, zirconium tetrafluoride, bomb liner, or a combination of all three. The determination of the exact causes of the hardness was inconclusive. However, it is believed that by using high purity zirconium tetrafluoride, ductile zirconium will be obtained.

By highly theoretical thermodynamic calculations, the maximum temperature in the bombs was determined. These calculations indicated that maximum temperatures of 1640 and 1900°C can be obtained in the spark-fired and gas-fired magnesium reduction bombs, respectively. The temperatures measured in the spark-fired bomb was about 1200°C and the melting points of the slags were determined to be about 1250°C. The temperature measurements in the gas-fired bombs were impossible due to the melting of the thermocouple wells. It can be concluded that a maximum temperature of 1350 to 1450°C and 1550 to 1650°C can be obtained in the spark-fired and gas-fired bombs, respectively.

The pressure measurements showed that less than 200 psi was obtained in the spark-fired bombs. Less than 400 psi is probably obtained in the gas-fired bombs.

The preparation of zirconium tetrafluoride from zirconium cyanonitride by use of aqueous hydrofluoric acid appears favorable. However, problems arise in the zirconium tetrafluoride
precipitation, and the removal of hafnium and impurities. Tests indicate the possibility of using vacuum distillation to purify and dry zirconium tetrafluoride monohydrate.

The production of zirconium from zirconium tetrafluoride by reduction with magnesium under an inert atmosphere of argon at atmospheric pressure was also demonstrated. The zirconium obtained was not massive but in a porous, finely divided form. The conversion efficiency was approximately 50 percent. Before the process could be economically feasible a satisfactory metal-slag separation process would have to be developed.

5. Extraction with Molten Metals (G. H. Beyer)

Extraction studies on molten metals have been concerned with the development of an extractor for the partial removal of fission products from molten metallic uranium using another molten metal. The extracting system chosen, due to economic and material availability reasons, was silver as the extractant metal with graphite as the material of construction for the extractor. An extractor in which varying amounts of silver could be passed upward through a fixed amount of uranium and then overflow into another graphite collector was made. The tests on this extractor were quite satisfactory. The silver overflow from the latest design analyzed 4.85% uranium. Since the equilibrium solubility value for silver in uranium was found to be between 5% and 8%, the contacting efficiency appears to be good. By controlling the silver flow rate, the contacting efficiency could probably be improved. The present process consists of melting the uranium in the extractor followed by melting the silver in a feed reservoir above the uranium. The molten silver is then directed through drilled holes to the bottom of the extractor and then allowed to bubble up through the uranium. The process is carried out inside of a quartz tube equipped with water cooled brass heads, under a vacuum. The vacuum is obtained through the use of a rotary type vacuum pump. Heating of the extractor is accomplished by an induction coil placed on the outside of the quartz tube. Necessary thermal and radiation insulation is obtained by the use of alundum and molybdenum shielding.

Neodymium was selected as the fission product to be followed in extraction studies because of its higher affinity for silver than uranium. The resulting high distribution
coefficient should enable a better evaluation of operating variables and extractor efficiency than a fission product with a low distribution coefficient.

A "fissium melt" (simulated fuel) of neodymium and uranium was made and the maximum solubility of neodymium in uranium at 1275°C found to be 2185 p.p.m. (± 300 p.p.m.). A recheck on the solubility by remelting a portion of the fissium melt with more neodymium gave 2120 p.p.m.


The purpose of this work is to measure the effects of plate spacing, percent free area (on plates), plate hole diameter, pulse amplitude, and pulse frequency on column efficiency, measured in terms of HTU, (column height required to carry out a theoretical degree of extraction). Data for 80 combinations of the above variables have been collected as part of a statistical design which will eventually involve a total of 96 combinations. Preliminary results indicate that the geometry variables, which have previously received only cursory examination in previous work, are of greater importance than pulse frequency over the ranges studied.

Engineering Development

1. Slurry Program (G. Murphy)

The slurry program is of interest because of the potential use of slurry fuels consisting of fissionable materials suspended in a vehicle which will facilitate handling, fuel processing and heat transfer in comparison with conventional fabricated fuel elements.

During the period of this report problems of vertical flow have been studied. In certain velocity ranges the solids distribution, as observed with glass particles in water, is widely different for upward flow in comparison with downward flow. The phenomenon is being studied from the theoretical viewpoint as well as from the experimental. Theoretical predictions and experimental observations are in agreement in a limited range and efforts will be continued to extend the range.
2. **Engineering Properties of Reactor Materials (G. Murphy)**

A report (ISC-700) entitled, "The Stress-Strain Characteristics of Uranium" by D. A. McCutchan and G. Murphy is being distributed.

**ABSTRACT**

Tests were made in reversed loading and in repeated tensile loading on thirteen specimens of rolled, alpha-uranium at room temperature. Constant strain rates ranging from 0.0003 in./in./min. to 0.0060 in./in./min. were employed.

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1. **Metal-Ceramic Reactions (D. R. Wilder)**

A report (ISC-659) entitled "Reaction of Lanthanum and Cerium with Ceramic Oxides" by G. Pulliam and E. Fitzsimmons is being distributed.

**ABSTRACT**

In this study the surface properties of cerium and lanthanum metals were determined for the metals melted on refractory oxide plaques of alumina, beryllia, thoria, and stabilized zirconia. The reactions occurring at the interface between the metals and the refractories were also determined by metallographic and x-ray techniques. The surface tension, contact angle, and work of adhesion were determined at temperatures of 810, 900, and 1000°C for cerium, and at temperatures of 950, 1000, 1100, and 1200°C for lanthanum. The metallographic examination of the reactions was conducted on polished sections of the interfaces. The temperatures to which the specimens were subjected for this phase of the study were 1100°C for cerium and 1200°C for lanthanum. To aid in the microscopic examination of the reactions between cerium and the oxides, mixtures of metal and ceramic powders were heated to temperatures of 500 and 804°C for cerium. Lanthanum and oxide powders were given a similar treatment to temperatures of 600 and 924°C. X-ray diffraction patterns of the mixtures were then obtained for the identification of the phases produced from the reactions.

The surface properties of the metals melted on the ceramic oxides were established by the sessile drop method of determining surface tension. The characteristic dimensions of the drop were obtained from photographs of the profile of the drop. The equilibrium between the known force of gravity acting on the drop and the unknown surface tension could be determined from the shape assumed by the
drop. The dimensions obtained from the profile were used also to determine the contact angle displayed between the surface of the drop and the ceramic supporting plaque. The work required to remove the metal from the ceramic was then determined from the contact angle and surface tension from a consideration of the surface forces operative at the point of contact between liquid and solid plaque.

The surface tension determined by the sessile drop method is of reasonable accuracy only when the angle of contact of the surface of the drop with the supporting plane, is greater than 90°. For this reason the surface tension of lanthanum was determined from the run on zirconia. At a temperature of 950°C the surface tension of lanthanum was determined to be 710 dyne/cm ± 5%. The surface tension of lanthanum went through values of 693, 648, and 630 dyne/cm as the temperature of measurement became 1000, 1100, and 1200°C. The surface tension of cerium was determined from runs of the metal melted on beryllia and zirconia. At temperatures of 810, 900, and 1000°C the surface tension of cerium was determined on zirconia to be 695, 680 and 666 dyne/cm, respectively. At the same temperatures the surface tension of cerium was determined on beryllia to be 740, 697, and 678 dyne/cm, respectively.

The contact angles of both metals was at their highest values when melted on zirconia. The contact angle of lanthanum on the oxides then decreased in the order: alumina, beryllia, and thoria. Cerium metal showed a very high contact angle with beryllia also, but the contact angle of cerium on thoria was assumed to be zero since the metal completely dissolved in the plaque. The rapid attack of alumina by cerium prevented measurement of the contact angle.

The work of adhesion of the metals to the oxides varied considerably between the metals and with the oxide studied. With both metals the work of adhesion was at a minimum for the melt on zirconia. The work of adhesion of the metals to thoria was by far the highest of all the oxides studied.

The work of adhesion of lanthanum to alumina and beryllia was intermediate between the adhesion to zirconia and thoria. The adhesion of cerium by beryllia, though not as low as the adhesion to zirconia, was lower than the adhesion of lanthanum to any of the oxides.

In general, the interfacial reactions occurring between the metals and the oxides resulted in simple reduction of the oxides. Alumina was reduced by the metals to form the oxides of the molten metals and alloys of aluminum and the metals. Beryllia was reduced in a similar fashion. However,
alloying of the metals with beryllium did not occur. The metals reacted with zirconia to produce both an interfacial layer of the metal oxide and a limited solid solution of metal and zirconia. Both metals produced solid solutions with thoria.

The values determined for the surface tensions of cerium and lanthanum revealed the similarity that was to be expected from the similarity of the other properties of the metals. The value of surface tension decreased linearly with increasing temperature in nearly the same manner for both metals. Generally the work of adhesion of the metals to the oxides increased as the temperature increased. As a result of this, the contact angles decreased with increasing temperatures.

Of the materials studied, beryllia was found to be the best refractory for containing the molten metals. The reactions between the metals and beryllia were less extensive than the other refractories, and the metal did not diffuse into the beryllia plaques. Zirconia was found to be a possible container for the molten metals, though the metal did penetrate the refractory by diffusion. Thoria and alumina were found to be poor container materials for the metals.

Other Engineering Research


The bismuth-uranium alloy molten metal loop experiment was terminated after 5237.5 hours of continuous operation at 950°C. The alloy consisted of 5% U-95% Bi by weight.

This loop experiment has shown that a U-Bi system can be contained in tantalum and the combination appears to be practical from both a design and a pumping standpoint. Data indicate that tantalum corrosion is quite insignificant. It was also found that the addition of 1% manganese to the alloy serves as a temperature depressant and permits the use of 10-12% by weight of uranium in bismuth at temperatures of approximately 900°C. Both static and dynamic tests are being run on a system of this type, which would permit higher fuel concentrations in a reactor.
APPENDIX I: LIST OF REPORTS FROM THE AMES LABORATORY

1. Reports for Cooperating Laboratories


ISC-604 F. H. Spedding, J. E. Powell and E. J. Wheelwright. The Stability of the Rare Earth Complexes with N-Hydroxyethylethilenediaminetriacetic Acid.


ISC-617 J. E. Powell and F. H. Spedding. Basic Principles Involved in the Macroseparation of Adjacent Rare Earths from Each Other by Means of Ion-Exchange.


ISC-621 R. D. Kross and V. A. Fassel. An Infrared Study of Picric Acid Molecular Complexes.
ISC-629  R. T. Nichols and E. N. Jensen. The Decay Scheme of Sc147.
ISC-630  P. Hall and S. Legvold. Remanent Magnetism in Toroids.
ISC-637  E. Wheelwright and F. H. Spedding. The Use of Chelating Agents in the Separating of the Rare Earths Elements by Ion-Exchange Method.
2. Papers Published in Scientific Journals

Corbett, J. D. and R. K. McMullan

Corbett, J. D. and S. von Winbush

Day, M. C., G. W. Eakins and A. F. Voigt

Freeland, M. Q. and J. S. Fritz

Fritz, J. S. and M. Johnson

Fritz, J. S. and S. S. Yamamura

Hansen, R. S.

Happe, J. A. and D. S. Martin, Jr.
Isotope Exchange of Manganese during the Reaction between Manganese(II) and Permanganate. J. Am. Chem. Soc. 77, 4212-17 (August, 1955).

Herwig, L. O., G. H. Miller and N. G. Utterback
Hettel, H. J. and V. A. Fassel

Jones, J. T., Jr. and J. K. Knipp

Kniseley, R. N. and V. A. Fassel

Kross, R. D. and V. A. Fassel

Laslett, L. J. and D. J. Zaffarano
Nuclear Research with High Energy X-Rays at Iowa State College. The Science Counselor (September, 1955).

Nichols, R. T. and E. N. Jensen

Ring, L. S., Jr.

Rundle, R. E.

Simmons, C. R. and R. S. Hansen

Skochdopole, R. E., M. Griffel, and F. H. Spedding

Spedding, F. H. and J. L. Dye

Spedding, F. H. and J. E. Powell
Stone, J. F., D. Kirkham and A. A. Read

Wilhelm, H. A. and B. A. Rogers

APPENDIX II: LIST OF SHIPMENTS

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<td>1 lb. cerium metal</td>
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<td>1 lb. neodymium metal</td>
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<td>1 lb. praseodymium metal</td>
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<td>2 gm samarium oxide</td>
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<tr>
<td></td>
<td>20 gm yttrium oxide</td>
</tr>
<tr>
<td></td>
<td>100 mg lutetium oxide</td>
</tr>
<tr>
<td></td>
<td>1 gm ytterbium oxide</td>
</tr>
<tr>
<td>Research Laboratories of Colorado, Inc.</td>
<td>3 thulium pellets</td>
</tr>
<tr>
<td>Newtown, Ohio</td>
<td></td>
</tr>
</tbody>
</table>
State University of Iowa  
Department of Physics  
Iowa City, Iowa

University of California  
Los Alamos Scientific Laboratory  
Los Alamos, New Mexico

Transportation Officer  
Fort Totten, New York

University of Minnesota  
Minneapolis, Minnesota

Professor C. D. Jefferies  
University of California  
Berkeley, California

Knolls Atomic Power Laboratory  
Schenectady, New York

5 gm \( \text{N}^{14} \) isotope

2 gm pure terbium oxide  
10 gm neodymium metal  
20 gm erbium metal  
20 gm yttrium metal  
66 gm neodymium metal  
69 gm samarium metal  
72 gm gadolinium metal  
77 gm erbium metal  
6 thulium buttons

1 gm lanthanum metal  
1 gm cerium metal  
1 gm praseodymium metal  
1 gm neodymium metal  
1 gm samarium metal  
1 gm gadolinium metal  
1 gm erbium metal  
1 gm yttrium metal  
1 gm dysprosium metal  
1 gm ytterbium metal

200 mg high purity lanthanum oxide

2 gm lanthanum oxide  
10 mg terbium oxide

100 mg neodymium oxide  
100 mg lanthanum oxide  
200 mg erbium oxide  
100 mg samarium oxide  
100 mg praseodymium oxide  
200 mg holmium oxide  
200 mg lutetium oxide  
200 mg ytterbium oxide  
200 mg thulium oxide  
200 mg dysprosium oxide  
200 mg terbium oxide  
100 mg gadolinium oxide  
100 mg cerium oxide  
100 mg yttrium oxide  
1 gm lanthanum oxide  
1 gm cerium oxide  
1 gm praseodymium oxide  
1 gm neodymium oxide  
1 gm samarium oxide  
1 gm gadolinium oxide  
1 gm yttrium oxide
American Metallurgical Products Co.  
New Castle, Pennsylvania

Brookhaven National Laboratory  
Upton, Long Island, New York

Dr. R. B. Price  
Battelle Memorial Institute  
Columbus, Ohio

Army Medical Research Laboratory  
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Department of Physics  
University of British Columbia  
Vancouver, B. C., Canada

Dr. Wm. F. Wagner  
Chemistry Department  
University of Kentucky  
Lexington, Kentucky

Johns Hopkins University  
Baltimore, Maryland

$\frac{1}{2}$ lb praseodymium metal  
$\frac{1}{4}$ lb neodymium metal

5 gm pure thulium oxide  
10 gm yttrium oxide  
25 gm lanthanum metal  
25 gm praseodymium metal  
25 gm samarium metal  
100 gm cerium metal

2 gm thulium metal

8 thulium buttons

2 gm praseodymium oxide  
1 gm neodymium oxide  
1 gm gadolinium oxide  
$\frac{1}{2}$ gm terbium oxide

1 cylinder lanthanum metal

1 thulium pellet

5 gm ytterbium oxide

1 gm lanthanum oxide  
1 gm cerium oxide  
1 gm praseodymium oxide  
1 gm neodymium oxide  
1 gm samarium oxide  
1 gm gadolinium oxide  
1 gm yttrium oxide  
100 mg terbium oxide  
100 mg dysprosium oxide  
100 mg holmium oxide  
100 mg erbium oxide  
100 mg ytterbium oxide  
100 mg thulium oxide  
100 mg lutetium oxide

10 gm terbium oxide