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U N C L A S S I F I E D

ISC-760

U N I T E D S T A T E S A T O M I C E N E R G Y C O M M I S S I O N

SEMI-ANNUAL SUMMARY RESEARCH REPORT IN ENGINEERING

For January - June, 1956

by

Ames Laboratory Staff

October 4, 1956

Ames Laboratory
at
Iowa State College
F. H. Spedding, Director
Contract W-7405 eng⁸²

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SEMI-ANNUAL SUMMARY RESEARCH REPORT IN ENGINEERING

For the Period January - June, 1956

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

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ENGINEERING

Under the direction of F. H. Spedding, R. W. Fahien,
R. W. Fisher, G. Murphy and M. Smutz.

Chemical Engineering1. Monazite Processing (Morton Smutz)*

A rather complete literature survey has been made concerning monazite processing methods in an effort to determine the best method of processing monazite sands on an industrial scale. The two methods showing most promise start with either a sulfuric acid digestion or a caustic digestion. The many alternatives are being studied on a laboratory scale.

2. Rare Earth Separation by Solvent Extraction (Morton Smutz)

A continuous multistage mixer-settler extractor has been made from polyethylene sheets. The extractor is of the pump-mix type with extremely sensitive interface control on each stage. A 15-stage unit has operated satisfactorily, although the unit is considered only as a prototype of a pilot plant unit.

Accurate equilibrium data for most of the rare earth nitrates have been obtained for the system rare earth nitrates-tributyl phosphate-water. The separation factor between adjacent lower rare earths is greater than between the adjacent higher rare earths. Yttrium can be made more extractable or less extractable than any of the rare earths by adjusting the total concentration. This explains much of the unexpected behavior frequently encountered when yttrium is present.

A simple algebraic method of determining the operating conditions for a given separation has been demonstrated.

3. Liquid Metal Extraction (E. H. Olson)

This experiment is concerned with the removal of fission products from a metallic uranium reactor fuel by extraction with an immiscible molten metal.

This molten-metal extraction process appears to have several advantages over the liquid-liquid aqueous process currently being used. Briefly, these apparent advantages would be a lower processing cost, a smaller volume of fission products to store, and a much smaller processing plant. The main disadvantage would be

*Names indicate group leaders in charge of work.

a lower decontamination factor than is obtained by the current liquid-liquid aqueous extraction process.

The system chosen for initial investigation was neodymium-uranium with silver as the extractant metal. Neodymium was chosen to represent the rare earth fission products which, as a group, are responsible for approximately 20% of the poisoning effect in uranium fuel. Also, previous experiments at the Ames Laboratory have indicated that neodymium has a high distribution coefficient between silver and uranium. Graphite was used in the construction of the extractor because of its availability, ease of fabrication, and low cost.

An equilibrium melting of silver with uranium containing 2300 p.p.m. of neodymium was carried out at 1270°C. for 15 minutes in a graphite crucible. Analysis of the silver and uranium phases gave neodymium concentrations of 1360 p.p.m. and 110 p.p.m., respectively. The accuracy of the spectrophotometric method of analysis of the uranium phase is questionable, because a very low concentration of neodymium was indicated. In any event, the distribution coefficient is fairly high and probably above 10, which indicates that silver will be quite effective in removing the rare earth poisons from uranium.

A report (ISC-696) entitled "Liquid-Liquid Extraction of Molten Uranium with Silver" by G. H. Beyer and C. W. Watson was distributed.

ABSTRACT

In recent years interest in the United States in the nuclear field has been turning towards power reactors. Among the many problems that must be solved before economical electrical power can be produced from nuclear reactors are the problems connected with the processing of spent reactor fuels. The only reactor fuel processing methods available at present, the aqueous processes, are relatively complex, destroy the metallic character of the fuel, require "long-cooled" fuels necessitating high inventories of fissionable material, and leave huge volumes of radioactive waste solutions to be processed and stored.

For these reasons interest has been turning towards other processing methods, including "pyroprocessing" by liquid-liquid extraction of the molten fuel with silver. The purpose of this study was to develop a part of the equipment and technology needed to study the extraction of uranium with silver and to ascertain some of the problems that might be expected in processing of spent reactor fuels with a silver extraction.

A vacuum system was built in which to carry out uranium-silver contacting; induction heating equipment was set up to heat the materials. Temperature measuring methods and techniques for use in liquid metal extraction work also were studied. Chromel-alumel, platinum-platinum rhodium, and columbium-tungsten thermocouples were investigated. Graphite was selected as the best container material for the molten metals and was used satisfactorily in all of this work.

A number of uranium-silver melts were made in which the phase separation and mass transfer were studied. Phase separation was sharp but occasional macroscopic inclusions were found in both phases. Microscopic phase separation was complete. Uranium was found to crystallize out of the silver phases as they cooled and to settle towards the interfaces. Mass transfer of the uranium to the silver appeared to be fairly rapid.

A graphite extractor for passing silver through a pool of molten uranium was designed and tested. It was found to be very simple to operate, easy to fabricate, and capable of transferring nearly equilibrium amounts of uranium to the silver.

A number of recommendations are presented for improving the equipment and techniques used in this study, along with some proposals for future work in this field. A pyroprocessing scheme, based upon a silver extraction of the fuel in the extractor designed in this study, is proposed as a basis for further study.

4. Zirconium-Hafnium Separation

A report (ISC-682) entitled "Separation of Hafnium from Zirconium Using Tributyl Phosphate" by R. P. Cox and G. H. Beyer was distributed.

ABSTRACT

The separation of hafnium from zirconium using tributyl phosphate offers interesting alternatives to present methods for making reactor-grade zirconium. Feed solution for the extraction step can be prepared from the reaction product of caustic and zircon sand. The purified zirconium can be converted into a variety of compounds, depending on the process chosen for reduction to the metal. It may be that future developments will show tributyl phosphate extraction to have advantages in new plants for the production of low-hafnium zirconium metal.

Zirconium has become an important construction material for nuclear reactors because of its corrosion resistance, structural strength, and low neutron absorption. Approximately 2.5 per cent of hafnium is associated with zirconium in naturally-occurring zirconium ores. Since hafnium has an abnormally high neutron absorption cross-section, it must be removed from zirconium used in nuclear reactors. The close chemical similarity of hafnium and zirconium makes separation by ordinary chemical techniques difficult. This has led to the development of a number of physical separation methods, notably solvent extraction.

As early as 1947, Fisher and Chalybaeus reported the separation of hafnium from zirconium by distribution between two solvents. By 1953 Miller was able to state that "separated, hafnium-free zirconium oxide can be produced for well under \$5 per pound to which should be added the cost of converting to zirconium metal." Although the details of the hafnium removal process are classified, it is stated that zirconium tetrachloride is used as feed and zirconium dioxide is obtained as product. After chlorination this product is reduced with magnesium by the Kroll process.

5. Zirconium Process Development (C. J. Baroch and E. H. Olson)

The purpose of this work is to produce pure, ductile zirconium from zirconium tetrafluoride, using magnesium as the reductant.

The currently used Kroll process produces a non-massive zirconium from zirconium tetrachloride, using magnesium as the reducing metal. This process requires the very careful handling of zirconium tetrachloride to prevent moisture pick up which introduces oxygen and causes the zirconium to be brittle and hard. Also the sponge form of zirconium obtained makes slag removal difficult. The magnesium reduction of zirconium tetrafluoride avoids both of the above mentioned difficulties. Zirconium tetrafluoride is non-hygroscopic and the zirconium metal is obtained in massive form with no slag inclusions.

Previously, zirconium produced from commercial grade zirconium tetrafluoride by magnesium reduction resulted in a non-ductile zirconium, presumably due to the oxygen content. During the period of this report, very pure anhydrous zirconium tetrafluoride was prepared by vacuum drying the hydrated tetrafluoride, followed by vacuum sublimation for further purification. The vacuum sublimation step leaves the non-volatile zirconium oxide as a residue and the sublimed anhydrous zirconium tetrafluoride is presumably oxygen-free.

The sublimed zirconium tetrafluoride was reduced with magnesium and the resulting zirconium gave a Rockwell "A" hardness of 69, which led us to believe that it was non-ductile. A test sample 0.107 inch in thickness was cold rolled to determine whether it could be cold worked. In 20 passes the sample was reduced 88.4% in thickness without failure or severe edge cracking. This ductility was unexpected in view of its hardness value.

More sublimed zirconium tetrafluoride is being prepared and will be reduced with magnesium to determine whether ductile zirconium can be consistently obtained. Also, an attempt will be made to reappraise the relationship between hardness and ductility of zirconium. A more detailed report (ISC-720) covering this work is being distributed.

6. Liquid-Liquid Extraction Studies in a Pulse Column (R. W. Fahien)

The effects of plate spacing, plate hole diameter, per cent free plate area, pulse amplitude, and pulse frequency on the extraction efficiency of a one-inch diameter pulse column were determined. The system hexone-acetic acid-water was employed and the investigation was carried out at a fixed hexone-water flow ratio of 1.3. A statistical design was followed using two levels of each factor. The product of pulse amplitude times pulse frequency was taken as a measure of the amount of agitation supplied to the column by the pulse generator. The values of the amplitude-frequency product used were 6.25, 12.5, 25, and 50 inches/minute. Extraction efficiency decreased with an increase in the amplitude-frequency product, despite the fact that the drop size became smaller and phase dispersion was greatly improved. This decrease in extraction efficiency was believed to be the result of forced recycle or back-mixing which is present in the pulse column and which increases with an increase in the amplitude-frequency product. This back-mixing could be observed visually and predicted mathematically. It represents a deterioration of true counter-current flow through the column.

An increase in the plate spacing from one inch to two inches or an increase in the plate hole diameter from 1/32 inch to 1/16 inch increased the extraction efficiency of the pulse column. The effect of plate spacing decreased with increasing pulsation and no plate spacing effect could be detected at an amplitude-frequency product of 50 inches/minute. Hole diameter produced the greatest effect of any of the factors studied. A change in per cent free plate area from 13% to 25% produced a small change in extraction efficiency which could be detected only at the two high levels of the amplitude-frequency product. Minimum and maximum values of

the height of a transfer unit based on the continuous hexone phase were 3.65 inches and 8.28 inches, with an average standard error of ± 0.25 inches for the experiment.

Engineering Development

1. Slurry Program (G. Murphy)

The investigation of power requirements and associated problems involved in pumping slurries has continued. The concentration range has been extended, and a series of data has been taken using small plastic spheres in water. This gives information on slurries having specific gravities near unity.

Investigations of the phenomenon of coring in vertical flow have continued and it has been noted that coring will not take place in certain concentration ranges which are a function of size and specific gravity of the solid component of the slurry.

At present the investigation is centered on a consideration of why, in certain ranges, the power requirement for pumping a slurry is lower than for pumping the fluid alone at the same average velocity. The identification of these ranges is becoming possible in terms of the Froude number, concentration of solids and specific gravity.

2. Engineering Properties of Reactor Materials (G. Murphy)

Six new high temperature fatigue machines of our own design have been put into operation at temperatures up to 600°C. and are being used in the uranium alloys program. Techniques for coating fatigue specimens have been markedly improved, greatly decreasing the "down" time for specimens.

Data on the tensile and fatigue properties of uranium and selected alloys are being taken over the 25-600°C. temperature range. At present, emphasis is being given to obtaining a better understanding of the marked changes in properties occurring in the 400-450°C. range.

Ceramic Engineering

1. Sintering Mechanism as Applied to Refractory Oxides (D. R. Wilder)

A bibliography (ISC-288 Supp.) entitled "Sintering Mechanism as Applied to Refractory Oxides" by D. R. Wilder, was distributed. This supplement covers the literature published during the period 1952-1955.

Other Engineering Research1. Circulation of Molten Metals (R. W. Fisher)

A "hot loop" containing 10% uranium and 90% bismuth by weight, was operated for approximately 1000 hours at a temperature of 1200°C. before a failure occurred in the inconel sheath, caused by a short circuit in an electrical connection. Enough information was obtained in this experiment, however, to prove the feasibility of operating at this temperature.

At the end of the period, a loop containing 5% uranium, 0.10% manganese, and 94.90% bismuth alloy was in operation at a maximum temperature of 1050°C, and after 1600 hours of operation, shows no indications of failure. This particular alloy has a distinct advantage over the 5% uranium-95% bismuth alloy in that the addition of the manganese depresses the melting point by approximately 175°C. It appears that the addition of manganese does not accelerate the corrosion rate.

APPENDIX I: LIST OF REPORTS FROM THE AMES LABORATORY

1. Reports for Cooperating Laboratories

- ISC-288 D. R. Wilder. Sintering Mechanism as Applied to Refractory (Suppl.) Oxides.
- ISC-638 Hobart Cammack and G. L. Bridger. Recovery of Uranium from Slag from the Electric Furnace Production of Phosphorus.
- ISC-644 Ames Laboratory Staff. Metallurgy. Quarterly Summary Research Report. April, May, June, 1955.
- ISC-647 Loren R. McMurray and D. J. Zaffarano. A Possible Method for Beam Extraction from an Alternating Gradient Synchrotron.
- ISC-648 Lawrence S. Ring, Jr., and D. J. Zaffarano. Beta Spectra of Some Short-Lived Nuclides.
- ISC-652 J. W. Murdock and Glenn Miller. Secondary Electron Emission Due to Positive Ion Bombardment.
- ISC-654 R. W. Laity and F. R. Duke. Measurements and Significance of Transport Number in Fused Salts.
- ISC-655 R. A. Lowry and G. H. Miller. Measurement of the Ionization Yield of Low Energy Atomic Particles in Gases.
- ISC-659 George Pulliam and E. S. Fitzsimmons. Reaction of Lanthanum and Cerium with Ceramic Oxides.
- ISC-662 Martin Welt and Morton Smutz. Thorium and Uranium from Monozite.
- ISC-663 C. Adams, D. W. Barnum, R. Bisque, R. I. Bystroff, F. Debbrecht, J. S. Fritz, D. E. German, P. G. Grimes, M. Johnson, W. Lane, A. Moye, J. W. O'Laughlin, J. L. Spooner, S. Sutton, W. Trask, G. Umbreit, E. Wadsworth, S. S. Yamamura, R. E. Yerick and J. Zimmerman. Survey of Masking Agents Used in Quantitative Analysis.
- ISC-666 R. C. Strittmater and G. C. Danielson. Measurement of Specific Heats by a Pulse Method.
- ISC-667 Max Q. Freeland and J. S. Fritz. I. Direct Titration of Sulfate. II. High Precision Spectrophotometric Analysis.
- ISC-668 J. J. Renier and D. S. Martin. Iodato-Silver Complexing Equilibria.
- ISC-671 F. R. Parchen and F. R. Duke. Kinetics of the Exchange Reaction Between Two Oxidation States of Cerium.

- ISC-672 Richard Hedges and Harrison Shull. The Absolute Infrared Absorption Band Intensities of the Methylene Group Vibrations of Some Methylene Halides.
- ISC-678 W. W. Davis and Morton Smutz. Effect of Phosphate Rock Particle Size on Recovery of Uranium from Superphosphate.
- ISC-679 R. T. Nichols and E. N. Jensen. Decay Schemes of Cl^{34} and Sc^{47} .
- ISC-682 R. P. Cox and G. H. Beyer. Separation of Hafnium from Zirconium Using Tributyl Phosphate.
- ISC-685 R. D. Redin and G. C. Danielson. Measurement of Magnetic Field Gradient by the Hall Effect.
- ISC-686 R. A. Fleming and F. R. Duke. Temperature Coefficient of Electrical Conductivity in the System Potassium Chloride-Zinc Chloride.
- ISC-688 O. N. Carlson, F. A. Schmidt and H. A. Wilhelm. Preparation of Zirconium and Hafnium Metals by Bomb Reduction of their Fluorides.
- ISC-693 R. A. Foos and H. A. Wilhelm. Applications of Liquid-Liquid Extraction for the Separation of Zirconium from Hafnium.
- ISC-694 R. A. Foos and H. A. Wilhelm. Application of Liquid-Liquid Extraction for the Separation of Tantalum from Niobium.
- ISC-696 C. W. Watson and G. H. Beyer. Liquid-Liquid Extraction of Molten Uranium with Silver.
- ISC-699 C. V. Banks and J. P. LaPlante. Annotated Bibliography of α -Furildioxime.
- ISC-701 B. L. Rhodes and Sam Legvold. Magnetic Properties of Holmium and Thulium.
- ISC-702 K. W. Herrmann, A. H. Daane and F. H. Spedding. Some Physical-Metallurgical Properties of Scandium, Yttrium and the Rare Earth Metals.
- ISC-706 Ames Laboratory Staff. Chemistry. Semi-Annual Summary Research Report. July-December, 1955.
- ISC-707 Ames Laboratory Staff. Physics. Semi-Annual Summary Research Report. July-December, 1955.
- ISC-709 Compiled by P. Chiotti and O. N. Carlson. Hanford Slug Program. Semi-Annual Summary Research Report. July-December, 1955.

- ISC-710 Ames Laboratory Staff. Engineering. Semi-Annual Summary Research Report. July-December, 1955.
- ISC-714 C. R. Whitsett and Gordon Danielson. Electrical Properties of Magnesium Silicide and Magnesium Germanide.
- ISC-716 C. R. Whitsett. Inductances of Single Layer Cylindrical Coils for Induction Heating Applications.

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The Preparation of Uranium Metal by the Reduction of Uranium Tetrafluoride with Magnesium. Proceedings of the International Conference on the Peaceful Uses of Atomic Energy, Geneva, 1955, 8, 162-174, 1956.
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The Preparation of Uranium Metal by the Reduction of Uranium Tetrafluoride with Magnesium. Metal Progress 69, No. 3, 81-88 (1956).

APPENDIX II: LIST OF SHIPMENTS

<u>Destination</u>	<u>Item</u>
University of California Los Alamos Scientific Laboratory Los Alamos, New Mexico	20 gm erbium metal 20 gm yttrium metal 20 gm lanthanum metal 66 gm neodymium metal 69 gm samarium metal 72 gm gadolinium metal 77 gm erbium metal
Mr. H. A. Boorse Pupin Physics Bldg. New York, New York	1 cylinder lanthanum metal
Atomics International North Am. Aviation, Inc. Downey, California	250 gm thorium metal
General Electric Company General Engineering Laboratory Schenectady, New York	100 mg dysprosium oxide 100 mg terbium oxide 100 mg holmium oxide 100 mg erbium oxide 100 mg ytterbium oxide 100 mg thulium oxide 100 mg lutetium 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
Dr. Franz R. Metzger Bartol Research Foundation Swarthmore, Pennsylvania	10 gm special N ¹⁴
Carbide & Carbon Chemical Company Union Carbide & Carbon Corp. Oak Ridge, Tennessee	50 gm pure cerium metal
Dr. Richard A. Ogg, Jr. Stanford University Stanford, California	1 gm N ¹⁵

<u>Destination</u>	<u>Item</u>
General Electric Company ANP Department Cincinnati, Ohio	1 gm cerium oxide 1 gm lanthanum 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
General Electric Company Richland, Washington	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
Dow Chemical Company Midland, Michigan	20 lbs. anhydrous thorium
Dr. J. F. McClendon Albert Einstein Medical Center Philadelphia, Pennsylvania	1 gm special hafnium oxide
Brookhaven National Laboratory Upton, Long Island, New York	20 mg holmium oxide
Dr. Thomas C. Hoering Department of Chemistry University of Arkansas Fayetteville, Arkansas	2 samples N ¹⁵
Battelle Memorial Institute Columbus, Ohio	2 gm terbium metal 2 gm erbium metal 2 gm ytterbium metal 2 gm holmium metal 2 gm praseodymium metal

<u>Destination</u>	<u>Item</u>
Research Laboratories of Colorado, Inc. Newtown, Ohio	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 lutetium oxide
Dr. T. J. Bond Department of Chemistry Baylor University Waco, Texas	1 gm neodymium oxide 1 gm dysprosium oxide 1 gm yttrium oxide 1 gm erbium oxide
Dr. Sumner Davis Massachusetts Institute of Technology Cambridge, Massachusetts	1 gm terbium oxide 1 gm holmium oxide 1 gm dysprosium oxide
Yale University New Haven, Connecticut	40 gm gadolinium oxide 10 gm terbium oxide 20 gm holmium oxide 5 gm thulium oxide 20 gm dysprosium oxide 20 gm erbium oxide
The Eagle-Picher Research Laboratories Joplin, Missouri	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

<u>Destination</u>	<u>Item</u>
University of California Scripps Institution of Oceanography La Jolla, California	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
Florida State University Tallahassee, Florida	10 gm praseodymium oxide 10 gm neodymium oxide
Professor Tarhen Huus Institute for Teoretisk Fysik Copenhagen, Denmark	100 mg terbium oxide
Dr. Allen J. VanderWeyden, U.S.A.E.C. Liaison Officer Atomic Energy of Canada, Ltd. Chalk River, Ontario, Canada	2 gm yttrium oxide 2 gm dysprosium oxide 2 gm ytterbium oxide
Dr. Jagdish Shanker Chemistry Division Department of Atomic Energy Government of India Bombay, India	1 gm gadolinium oxide 1 gm dysprosium oxide
U.S.A.E.C. c/o Mallinckrodt Chem. Works Uranium Division St. Louis, Missouri	100 mg holmium oxide 100 mg terbium oxide 100 mg dysprosium oxide 100 mg erbium oxide 100 mg thulium oxide
Argonne National Laboratory Lemont, Illinois	1 sample gadolinium metal 1 sample erbium metal
University of Saskatchewan Betatron Department Saskatoon, Saskatchewan, Canada	25.6394 gm yttrium oxide 50 gm lutetium oxide 50 gm thulium oxide 50 gm terbium oxide 50 gm yttrium oxide 50 gm holmium oxide

<u>Destination</u>	<u>Item</u>
Dr. Fisher c/o Air France Cargo Department New York International Airport Jamaica, Long Island, New York	25 gm neodymium oxide
Anaconda Aluminum Company Columbia Falls, Montana	1 gm hafnium oxide