Semi-annual summary research report in engineering

Ames Laboratory Staff
Iowa State College

F. H. Spedding
Iowa State College

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SEMI-ANNUAL SUMMARY
RESEARCH REPORT IN ENGINEERING
for
JULY-DECEMBER 1958
by
Ames Laboratory Staff

AMES LABORATORY
RESEARCH AND DEVELOPMENT REPORT
U.S.A.E.C.
UNCLASSIFIED

ISC-1118

Chemistry General, UC-4
TID 4500, Oct. 1, 1958

UNITED STATES ATOMIC ENERGY COMMISSION

Research and Development Report

SEMI-ANNUAL SUMMARY
RESEARCH REPORT IN ENGINEERING
for
JULY-DECEMBER 1958

by

Ames Laboratory Staff

Date of Issuance

April 15, 1959

Ames Laboratory
at
Iowa State College
F. H. Spedding, Director
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For the Period July - December, 1958

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

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Chemical Engineering

1. Chelation of Rare Earths in Solvent Extraction Studies (M. Smutz)*

   Separation of the rare earths by solvent extraction requires a large number of theoretical stages because of the small separation factors between adjacent elements. To increase the separation factors, chelating agents have been added to the rare earth nitrate solutions before extraction with tributyl phosphate. In general, the separation factor increased for the heavy rare earths, elements 64 to 71, and decreased for the light rare earths, elements 57 to 62, when the chelating agent was present. The chelating agent which appears most favorable is tetraammonium ethylenediaminetetraacetate.

2. Mechanisms for Extraction of Rare Earth Nitrates with Tributyl Phosphate (M. Smutz)

   The complex between the rare earth nitrate and the solvent tributyl phosphate that makes possible the separation of the rare earths from one

*Names indicate group leaders in charge of work.
another by liquid-liquid extraction is being studied. The shift of a spectral peak corresponding to the phosphorus oxygen double bond has indicated that the rare earth complexes in that vicinity of the solvent molecule, and an attempt will be made to determine the composition of the complex by spectra methods. Tributyl phosphates were synthesized with different butyl groups and this has been shown to effect the extraction ability of the solvent.

3. Separation of Isotopes by Solvent Extraction (M. Smutz)

A tributyl phosphate-water-nitric acid system is being used for separating $^{15}N$ and $^{14}N$ by solvent extraction. Nitric acid is the source of the nitrogen and contains about 0.365% $^{15}N$ and 99.635% $^{14}N$. Equilibrium data obtained thus far have been determined by using the familiar simulated column technique. Enrichments of $^{15}N$ in the aqueous phase have been small but real.

4. Processing of Monazite Sands (M. Smutz)

A report (ISC-947), "Processing of Monazite Sands" by John J. Barghusen and M. Smutz, was distributed.

For the past ten years, much effort has been expended by several research teams to develop an economic process for recovering thorium, rare earths and uranium from monazite sands. Recently, a process was developed at the
Ames Laboratory which possessed many attractive features. The major disadvantage inherent to the process was the large cost for oxalic acid. The oxalic acid was used to precipitate rare earth and thorium oxalates from a sulfate and phosphate solution of these elements. The object of the oxalate precipitation was to separate the thorium and rare earths from uranium, sulfate and phosphate ions. Since this method for processing monazite sands was so attractive, concerted effort was made to discover a method whereby the oxalic acid could be recycled.

A process was developed whereby the oxalate ions used to precipitate the mixed thorium and rare earth oxalates can be recycled. The mixed oxalates were digested with a 2.5 normal solution of sodium hydroxide for one hour at 95°C. The products of the reaction were the mixed hydroxides and a solution of sodium oxalate. Even though an excess of sodium hydroxide provided greater yields of sodium oxalate, it was found more economical to use a stoichiometric amount of caustic in the digestions. The sodium oxalate was washed from the hydroxide cake with several batches of hot water. This solution was then recycled to precipitate another batch of oxalates. It was found that approximately 95 per cent of the stoichiometric quantity of oxalic acid used to precipitate the mixed oxalates can be recycled in the sodium oxalate solution.
The monazite sulfate solution was diluted with 4.5 parts by volume of water. The pH of the solution was raised to about 1.3 by adding ammonium hydroxide. It was found that a pH of about 1.5 affords the best conditions for the oxalate precipitation. Since some sodium hydroxide was recycled with the sodium oxalate solution, less ammonium hydroxide was needed to raise the pH of the solution to 1.5. The sodium oxalate recycle solution was added to precipitate the mixed oxalates. Sufficient oxalic acid was added to make up the deficiency in oxalate ions and to provide a ten per cent excess. The cake was filtered and washed with an oxalate wash solution.

A small scale run was made using this oxalate recycle technique. The results indicated that the method could be adapted successfully to the monazite process.

The possibility of recovering uranium from the oxalate filtrate by anion exchange was investigated. Uranium was successfully recovered from the monazite sulfate solution using a strong base anion exchange resin--Dowex-1. By providing a residence time of 40 minutes, about 90 per cent of the uranium was adsorbed on the resin. Essentially all of the uranium was eluted from the resin with an eluant 0.7 normal in nitric acid and 0.5 normal in sodium nitrate. Phosphate and oxalate ions did not seem to interfere with the process.
Cost estimates were made for the oxalate recycle process and the previous Ames oxalate process. It was found that the oxalate recycle process was economically superior to the former one. The production costs for producing one pound of thorium were $9.93 and $13.12, respectively. The economic advantage was entirely in the quantity of oxalic acid required for the two processes.

5. **Entrainment from Bubble Cap Trays** (G. Burnet)

Entrainment from bubble cap trays and similar liquid vapor contacting apparatus is frequently a serious problem in the processing of radioactive materials. This project developed a technique for accurately measuring entrainment using radioisotopes as tracers. The experimental system used was an air-water, non-overflow simulation of a distillation system in an 8-inch diameter, 4-plate test column. The I-131 isotope was found to be a satisfactory tracer for detection through the column wall and for simple disposal procedures.

A 400-fold change of entrainment rate was investigated for 48 different operating combinations of the following variables:

1. vapor velocity
2. submergence level
3. slot area
4. tray spacing
Duplication of the operating combinations showed clearly that the entrainment data obtained were reproducible with a high degree of precision.

The entrainment data collected were analyzed statistically and the inter-relationship between the four variables determined. A comparison of the data developed in this work with that previously published in the literature showed that greater precision and reproducibility than heretofore obtainable was evident.

Future work calls for determination of the effect of fluid properties, particularly viscosity, density and surface tension, on entrainment rate. In the first phase of this work, application of dimensional analysis to the data thus far produced and to those reliable data available in the literature has produced a general correlation for predicting entrainment which is applicable to any system.

A second or experimental phase now underway consists of a series of experimental runs in the test column using air to simulate vapor and a three-component liquid system which provides a wide range of fluid properties. The results of this work will be used to test the proposed correlation.
6. A Forced Convection Loop for Circulating Liquid Lead-Bismuth Mixtures (G. Burnet)

Several investigations preliminary to construction of a forced convection loop for circulating the lead-bismuth eutectic are nearly complete. Design of the loop is complete and all components and materials are on hand. The loop is to be used for heat transfer, fluid flow and vapor-liquid equilibria studies. The various preliminary investigations will be discussed individually as follows:

(1) **Dynamic corrosion tests.** From the corrosion tests carried out at 600-900°C to select the best container material for the eutectic, the following generalizations can be made:

(a) of the ferritic steels tested (2 1/4 Cr-1 Mo, 430 and 446) corrosion resistance decreases as the Cr content increases.

(b) Austenitic stainless steels show poor corrosion resistance above 700°C and are subject to preferential solution of nickel.

(c) an oxide film on stainless steel appears to improve corrosion resistance up to 750°C.

(d) metallographic examination has shown preferential solution of metal constituents to be the most common type of attack.

The forced convection loop will be constructed primarily of 446 with certain small parts of 430. The maximum operating temperature will probably be 700°C.
Design and testing of flanged joints. The forced convection loop is to be assembled using flanged joints. Two types of flanged joints were used in a 347 thermal loop operating at 400-600°C for 4,000 hours. These were a stainless steel flange joint with the faces serrated and used with a 20 mil tantalum gasket, and a stainless steel flange joint with a low carbon steel ring-type gasket.

A third type of flange designed to seal with a tongue-and-groove without a gasket has been tested against the eutectic under non-flow conditions at 450°C and 50 psig for 570 hours without leakage. The most important design feature in flanged joints is the use of flange bolts with a coefficient of expansion less than that of the flange.

Design and testing of a cold trap. Where corrosion occurs in a liquid metal system due to a thermal gradient, a trap is necessary to remove material precipitated in the cold leg. A trap which uses change of direction and a packing of metal turnings was operated successfully for 4,000 hours in the thermal loop cited in (2) above.
(4) **Design and testing of packed seals.** Three types of compression seals to be used where a pipe line must pass through the wall of a liquid metal bath have been tested. Powdered silica alumina and firebrick were found to be suitable as packing materials. The seals have shown no leakage after 750 hours at 10-30 psig and 200°C.

7. **Production of Low Oxygen Content Yttrium Fluoride** (G. Burnet and E. Olson)

7.1 **With Fluorine**

Results previously reported have shown that no reduction in oxygen content (compared to that in YF₃ produced by hydrofluorination) occurs when YF₃ is produced by treating with fluorine(1) Y₂O₃, (2) YF₃ produced by hydrofluorination, or(3) blends of the latter two mixtures at atmospheric pressure and temperatures up to 450°C. A series of runs was made with the reactor under 20 psig fluorine and at 600°C that reduced by approximately 20% the oxygen content of the best YF₃ produced by hydrofluorination.

In order to better study the conversion of Y₂O₃ to YF₃ using fluorine the heat of reaction and the free energy change were calculated as function of temperatures. The calculations confirmed the fact that higher temperature favors the conversion to YF₃.
7.2 By Hydrofluorination

A report (ISC-1068), "Preparation of Low Oxygen Content Yttrium Fluoride" by M. Smutz, G. Burnet, J. Walker, R. Tischer and E. Olson, was distributed.

The purpose of this investigation was to determine the optimum conditions for the formation of very low oxygen content yttrium fluoride.

Various methods of preparing a very low oxygen content yttrium fluoride are presented. Since the chemical properties of yttrium are quite similar to those of the rare earths, yttrium was used in these studies with the object in mind that the processing conditions would probably be applicable to the rare earths.

Yttrium fluoride was formed by hydrofluorinating various yttrium compounds with gaseous anhydrous hydrofluoric acid. The compounds used for these hydrofluorinations were yttrium oxide, hydrated yttrium fluoride, yttrium oxalate, yttrium chloride, yttrium nitrate, yttrium sulfate, and yttrium sulfide. The effects of time and temperature, oxalate ignition temperature, particle size, and hydrofluoric acid purity on the conversion of yttrium compounds to yttrium fluoride were also studied.

The yttrium fluoride purification methods studied were molten fluoride hydrofluorination, vacuum distillation and inert gas sparging,
all of which were carried out above the melting point of yttrium fluoride.

The processing methods which were found to give a low oxygen intensity ratio yttrium fluoride are as follows:

(1) Yttrium fluoride with an average oxygen intensity ratio of 0.33 was prepared by drying the hydrate at 600°C in a stream of anhydrous HF. The desirability of this method for large scale production is limited, however, due to the handling problems involved in the preparation of the hydrate.

(2) Yttrium fluorides with oxygen intensity ratios of 0.21 to 0.30 were prepared by hydrofluorinating yttrium oxide in a platinum boat for eight hours at a minimum temperature of 600°C using General Chemical Company anhydrous hydrofluoric acid. This would probably be the best method to scale up for the preparation of larger quantities.

(3) Small quantities of yttrium fluoride with oxygen intensity ratios of 0.18 to 0.33 were produced by melting yttrium fluoride and topping with anhydrous HF for 1.5 hours at 1200°C. The fluoride before topping had an oxygen intensity ratio of 0.38 or less.

(4) Small amounts of yttrium fluoride having oxygen intensity ratios of 0.23 to 0.32 were prepared by vacuum distilling yttrium
fluoride with an oxygen intensity ratio of approximately 0.64 at about 1300°C. This method of purification is limited to small quantities due to the high temperature required and the slow distillation rate.

8. **Recovery of Uranium (G. Burnet)**


A uranium balance was completed across each of two recently constructed wet process phosphoric acid plants of different design. Seventy-five to 80% of the uranium entering in the raw materials was found in a recoverable form in the product acid at both plants. The remainder was found in two waste streams: the gypsum from the filters and the water from the fume or vapor scrubbers. Depending upon the process and type of scrubber used, the latter stream may be a source of economically recoverable uranium. Basic engineering design data were developed for the recovery of the uranium from run-of-the-plant acid using either solvent extraction or ion exchange.


Studies are being made on drop size distribution as a function of flow rates, pulse amplitude, and pulse frequency. A knowledge of
drop size distribution coupled with hold-up information will give an estimate of interfacial area. Photographic methods are being used to determine drop size distribution. Although a complete analysis of the first series of drop size studies has not been completed, the drop size distribution appears to be affected mainly by the pulse frequency and only slightly by the flow rates to the column.

A report (ISC-1095), "Pulse Column Design" by L. E. Burkhart and R. W. Fahien, is being distributed.

By assuming the operation of a pulse column to be a stagewise process a theory was developed which explained some of the data which have been reported for the pulse column and which predicted other phenomena which had not yet been observed and reported. From this theory a design method was derived and illustrated by problems and by experimental data. This represents the first overall theory of the pulse column which has been reported. From this theory a design method was proposed which utilized double operating lines and double stage lines across which stages could be stepped off as in a McCabe - Thiele diagram.

10. Performance of a Scheibel Extractor (L. E. Burkhart)

A technique has been developed to determine the concentration of either stream at any point in the column by means of sample
tubes packed with a material which is preferentially wet by one phase. Glass packing was used to sample the water phase and polyethylene packing was used to sample the organic phase.

11. Two Phase Disperser (E. Olson)

The two phase disperser described in ISC-1051 was modified to prevent possible mixing of the inlet- and exit light phase when the unit is operated as a continuous countercurrent flow extractor. This modification also permitted easy determination of the light phase dispersing rate.

A model of the modified design was constructed of plexiglass and tested with a kerosene-water system. The light phase flow rate was limited by the rate at which it was dispersed by the disperser into the heavy phase. The heavy phase flow rate will have to be matched to the phase dispersing rate since no seal to positively separate the inlet- and exit heavy phase has been provided. Heavy phase flow rates lower than the dispersing rate would result in a recycle of heavy phase through the disperser while higher flow rates would result in part of the heavy phase bypassing the disperser and leaving with the exit heavy phase.

A stainless steel two phase disperser has been built and will be tested as a countercurrent-flow type of extractor using an aqueous-organic system containing a distributable solute to determine extraction efficiencies attainable at various disperser speeds and phase flow rates.
12. **Separation of Cerium from Rare Earth Nitrates** (E. Olson)

A paper (ISC-1069), "Separation of Cerium from Other Rare Earths by Ignition of the Nitrates" by Russell W. Johnson and Edwin H. Olson, was distributed.

Cerium was separated from the light rare earths by ignition of the nitrates whereby the cerous nitrate was decomposed to ceric oxide. Small scale investigations were made using sodium nitrate and magnesium nitrate hexahydrate as fluxes. A cost estimate based on data from the small scale investigations showed the sodium nitrate ignition to be more economical; therefore, bench scale investigations were made using sodium nitrate. With a weight ratio of sodium nitrate to rare earth nitrate hexahydrate of 1:2 and a temperature of approximately 295°C for 12 to 16 hours, a nearly quantitative conversion of cerous nitrate to ceric oxide was obtained. The ceric oxide obtained upon dissolving the soluble sodium nitrate and undecomposed rare earth nitrates and filtering, using "Celite" filtering aid as a precoat, was found to be quite pure. Indications are that an even purer ceric oxide could possibly be obtained if a lower temperature for a longer length of time had been used.
13. Liquid Metal Extraction (E. Olson)

Various promising container materials for the silver-uranium system were tested by melting, cooling to room temperature, then remelting and recooling a silver-uranium charge. Tantalum, yttrium oxide and calcium fluoride crucibles were tested. Tantalum crucibles with 15 mil wall thickness failed by stress cracking during the remelting operation and resulted in leakage of the silver phase. Crucibles with 30 mil walls will be tested to determine whether the thicker wall will avoid this difficulty. Yttrium oxide crucibles were found to be unsatisfactory due to cracking when the cast silver-uranium charge was remelted. Also, it was noted that a thin film of the inner wall was pulled away as the silver contracted upon cooling. Calcium fluoride appears promising in the single crucible tested to date. The walls were not strongly wetted by either phase and the crucible was easily separated from the charge.

A tantalum, single phase disperser for dispersing molten uranium in silver was fabricated and tested. After two stirring runs the disperser appeared to be withstanding the corrosion satisfactorily. Silver and uranium hold-up after a stirring run was found to be approximately 2.2% and 1.6%, respectively.

An economical commercial process has been developed for the recovery of yttrium wastes. This process involves first converting the waste products to an impure yttrium oxide by calcination, and dissolving the oxide in nitric acid. By the addition of ferrocyanides, filter aid, and oxalic acid it is possible to obtain yttrium oxalate of spectrographic purity. This oxalate is further calcined to the oxide which can then be introduced into the regular production process.

It is estimated that the chemical cost of this operation is $1.50 per pound. Details of this process will be included in a special report.

Nuclear Engineering

1. Slurry Program (G. Murphy)

The design of the subcritical nuclear assembly to use an aqueous slurry of uranium oxide has been completed and a prototype constructed. It is proposed to test the hydrodynamics of the prototype thoroughly, using a non-fissionable solid in place of the uranium oxide to check for regions that might cause difficulty through settlement of the solids and to check other possible operational problems.

The analytical work on the hydrodynamics of slurries has continued with particular emphasis on the evaluation of the lift forces.
developed on an individual particle. The contribution of the velocity gradient in the approaching fluid, the "throttling effect" (effect of boundaries), and the roughness of the particle are being isolated through a supplementary series of experiments.

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

Fatigue tests on normal uranium and low-silicon uranium are virtually complete in the range of temperatures between 25°C and 600°C. A pair of equations has been developed for the endurance limit of normal uranium in this temperature range. It is suggested that one equation applies in the range in which twinning is the principal mode of deformation and the other in the range in which slip predominates. The break between the two ranges is well defined and agrees with results obtained in tensile tests.

3. **Molten Metal Corrosion Studies** (R. W. Fisher)

3.1 **Static Tests in Niobium**

Capsule tests of the thorium-magnesium eutectic in niobium metal were run for periods of 1000 hours and 1300 hours at 850°C with no appreciable attack of the niobium. The 1300 hour test was conducted under static conditions. In the 1000-hour test the sample was oscillated about an angle of 156°, permitting the molten metal to travel from one end of the capsule to the other. Aside from a slight wetting inside
the niobium container no other effects were noted. A circulating loop will be constructed on the basis of the results of this test.

3.2 Yttrium Loop

Additional research on the annealing of yttrium metal has shown that the optimum annealing conditions are as follows: The metal is heated in a vacuum of $10^{-4}$ mm Hg and the temperature is raised at a rate of 15° per hour until 1000°C is reached. It is then permitted to soak for one hour at this temperature after which time it is allowed to cool at the rate of 90° per hour. This annealing is done in a sealed quartz tube to prevent contamination by other metals. It was noted at the end of this annealing cycle that the individual crystals appeared to have grown to 1/4 - 1/2" in size. However, this crystal growth did not interfere with the punching or welding operations encountered later in the loop fabrication. It was previously thought that this large crystal growth was responsible for the difficulties in bending the yttrium metal but these later results do not support the previous observation.

When yttrium metal is annealed at 640°C in a vacuum it acquires a black scale on the surface which seriously interferes with further fabrication. When this same metal is again annealed at 1000°C under optimum conditions, the black scale flakes off and leaves a shiny ductile
metal which is quite easily fabricated. All of these tests were run on yttrium metal containing 2000 to 2500 ppm oxygen. Annealing conditions may vary on metal of lower or higher oxygen content. Further work will be conducted on yttrium metal of lower oxygen content.

As a result of these annealing studies an yttrium loop containing 95 w/o uranium-5 w/o chromium alloy was assembled using the conventional pumps and heat transformers described previously.\textsuperscript{1,2}

The yttrium loop was protected from oxidation by enclosing it in a 446 stainless steel sheath. As an additional precaution, a titanium spacer 18 mils thick was introduced between the stainless steel and yttrium to avoid the possibility of intermetallic attack. This loop is equipped with a conventional thief-sampling device so that the circulating alloy can be analyzed during the operation. The loop will be operated at a temperature of 1000°C for a period of 2000 hours or until failure occurs. Since the tubing used in this loop was fabricated by experimental processes, it is quite conceivable that the loop will fail after a short period of time. Additional research is being conducted to improve the tube fabrication techniques.

\textsuperscript{1} ISC-547

\textsuperscript{2} Second Geneva Conference Paper A/Conf. 15/p/1032.
Ceramic Engineering

1. Sintering Investigations (D. Wilder)

A paper (ISC-1109), "Preliminary Sintering Characteristics of Aluminum Oxide" by D. R. Wilder, has been submitted for publication in the Journal of the American Ceramic Society.

The preliminary sintering characteristics of fine aluminum oxide powders were studied in the temperature range of 25°C to 1200°C. The aluminum oxides considered all exhibited the high temperature alpha or corundum structure.

Changes in the surface adsorbed ions were investigated by water slaking characteristics, thermogravimetric analysis, and infrared spectroscopy. The changes consist of a reversible loss of water and hydroxyl groups. This loss is temperature dependent and varies widely for the various types of aluminum oxide considered.

The nature of the sintering phenomena was observed in the temperature range 1000°C to 1200°C, where macro shrinkage becomes apparent. Sintering rates increase with decreasing particle size. The initiation of sintering occurs after, but not necessarily immediately after, the loss of surface adsorbed ions. The mechanisms by which sintering progresses were found to be complex in nature. They are not describable by an Arrhenius equation, nor fully defined by any one
of the possible mechanisms: plastic flow, evaporation and condensation, surface diffusion, or volume diffusion. A combination of the first of these mechanisms and one or more of the remaining could describe the phenomena observed.

2. Refractory Characteristics of MgO-CaF$_2$ System (D. Wilder)

This program concerns the high temperature (800-1500°C) strength characteristics of MgO with 0-6 mol per cent additions of CaF$_2$. Considerable attention has been devoted to establishing a reproducible test specimen. An investigation of the effects of such variables in the forming procedure as moisture content, forming pressure, rate of pressing, and thickness of specimens has resulted in a standardized procedure for the forming of compacts. Further work is in progress to establish the influence of grain size distribution of the CaF$_2$ on the firing behavior.

Work has progressed on the measurement of strength of refractory materials as a function of temperature. A successful technique for firing sample specimens has been developed.

3. New Glass with High UO$_2$ Content (D. Wilder)

A series of glasses containing up to 50 w/o UO$_2$ had been developed previously; however, reheating any of the glasses caused either decomposition or devitrification. During the past six months compositions
were altered to eliminate these faults. Several glasses were produced which were oxidation-stable and relatively resistant to devitrification when reheated to temperatures up to 900°C in air. One such glass consisted of 45 w/o UO₂, 13 w/o SiO₂, 38 w/o P₂O₅, and a trace of molybdenum oxide which was present as an impurity, introduced by the molybdenum crucibles in which the molten glass was contained. Sufficient molybdenum was present in the glass to give it a dark blue color.

4. Protective Film Characteristic of Molybdenum Disilicide (D. Wilder)

A fundamental investigation of the oxidation of MoSi₂ has established that molybdenum disilicide is protected against rapid oxidation at high temperatures by the formation of a surface film. During this report period it was found that MoSi₂ gains weight very slowly when held at elevated temperatures. X-ray diffraction patterns of quenched MoSi₂ samples revealed that MoSi₀.₆₅ was forming in preference to MoO₃ at high temperatures, which would account for the gain in weight. Indications are that the protective film consists of a silicon-molybdenum-oxygen substance which is probably rich in silica at the film-oxygen interface, rich in molybdenum at the film-MoSi₂ interface, and graduated in between.
5. **Protective Coating for Yttrium** (D. Wilder)

During this period work was begun on a porcelain-enamel coating for yttrium which would protect it from oxidation at temperatures up to 1000°C. A number of frits were tried on the metal, but only one, a reasonably high melting glass composed of seven oxides, gave satisfactory results. This glass has a lower thermal neutron cross section than the yttrium metal. The glass alone can be fired on yttrium with success, although the firing range is small and results were poor when the time-temperature combination was not absolutely correct. The addition of CeO in amounts up to 30 w/o to improve the firing range and fit of the coating resulted in some improvement, especially in fit. Protection against oxidation in moving air has been obtained at 800°C. Improvement of the method of application and firing techniques should eliminate pin holes and microcracks which are the paths of oxidation failures.
APPENDIX I: LIST OF REPORTS FROM THE AMES LABORATORY

1. Reports for Cooperating Laboratories

ISC-867 Riley Schaeffer and Hampton Smith. Isotopic Equilibria of Nitrosyl Chloride.


ISC-945 William J. Lane and J. S. Fritz. Metal-Indicator Systems in (Ethylenedinitrilo)tetraacetic Acid Titrations.


ISC-1030 R. G. Barnes. Bibliography of Titles of Articles in the Field of Nuclear Quadrupole Resonance Spectroscopy of Solids.


ISC-1074  Peter H. Vossos and R. E. Rundle. Crystal Structure and Magnetic Properties of LiCuCl$_3$·2H$_2$O.
2. Publications

Amma, E. L. and R. E. Rundle

Anderson, Gerald S. and Sam Legvold

Anderson, G. S., S. Legvold and F. H. Spedding

Arajs, Sigurds and Sam Legvold

Arajs, Sigurds and Sam Legvold

Banks, Charles V., Clara I. Adams and John Richard

Banks, Charles V. and Dennis W. Barnum

Banks, Charles V., Keith E. Burke and Jerome W. O’Laughlin

Banks, Charles V., J. P. LaPlante and John J. Richard

Banks, Charles V., James A. Thompson and Jerome W. O’Laughlin

Barnes, R. G. and R. D. Engardt

Barnes, R. G. and R. A. Hultsch
Barghusen, John, Jr. and Morton Smutz  

Corbett, John D.  

Corbett, John D.  

Dahl, June L. and Frederick R. Duke  

Duke, Frederick R. and James P. Cook  

Duke, F. R. and R. A. Fleming  

Duke, Frederick R. and Marlowe L. Iverson  

Duke, F. R. and Boone Owens  

Duke, Frederick R. and Boone Owens  
Transport Numbers of the Pure Fused Salts, LiNO₃, NaNO₃, KNO₃, and AgNO₃. J. Electrochem. Soc. 105, 548-549 (1958).

Flesch, Gerald D. and Harry J. Svec  

Fritz, James S., Marlene J. Richard and Shirley K. Karraker  

Fritz, James S., Marlene Johnson Richard and William J. Lane  

Fritz, James S. and Gerald R. Umbreit  
Hammer, C. L. and R. H. Good, Jr.

Herman, R. and C. A. Swenson

Jennings, L. D. and C. A. Swenson

Junk, Gregor and Harry J. Svec

Kniseley, Richard N., Velmer A. Fassel, Beverly B. Quinney, Carl Tremmel, William A. Gordon and William J. Hayles

Laslett, L. Jackson

McCarley, Robert E., Don S. Martin, Jr. and Lee T. Cox

McMullan, Richard K. and John D. Corbett
The Lower Oxidation States of Gallium. III. The Constitution of \(\text{Ga}_2\text{Cl}_4\) and Its Analogy with \(\text{Ga}(\text{AlCl}_4)\). J. Am. Chem. Soc. 80, 4761-4764 (1958).

Smith, Harold G. and R. E. Rundle
The Silver Perchlorate-Benzene Complex, \(\text{C}_6\text{H}_5\cdot\text{AgClO}_4\), Crystal Structure and Charge Transfer Energy. J. Am. Chem. Soc. 80, 5075-5080 (1958).

Smith, J. F. and J. R. Ogren
Electrical Properties and Thermal Expansion of the Laves Phases, \(\text{CaMg}_2\) and \(\text{MgCu}_2\). J. Appl. Phys. 29, 1523-1525 (1958).

Spedding, F. H., K. Gschneidner, Jr. and A. H. Daane

Svec, H. J., A. A. Read, D. W. Hilker

Swenson, C. A.

Thoburn, W. C.

Thoburn, W. C., S. Legvold and F. H. Spedding

Worden, D. G. and G. C. Danielson
**APPENDIX II: LIST OF SHIPMENTS**

<table>
<thead>
<tr>
<th>Destination</th>
<th>Item</th>
</tr>
</thead>
<tbody>
<tr>
<td>University of California</td>
<td>6 thulium pellets</td>
</tr>
<tr>
<td>Los Alamos Scientific Laboratory</td>
<td>20 gm thulium metal</td>
</tr>
<tr>
<td>Los Alamos, New Mexico</td>
<td>100 gm thulium oxide</td>
</tr>
<tr>
<td>State University of Iowa</td>
<td>20 gm lutetium metal</td>
</tr>
<tr>
<td>Iowa City, Iowa</td>
<td>100 gm lutetium oxide</td>
</tr>
<tr>
<td>U. S. Bureau of Mines</td>
<td>10 gm lanthanum metal</td>
</tr>
<tr>
<td>Albany, Oregon</td>
<td>10 gm cerium metal</td>
</tr>
<tr>
<td>Ohio State University</td>
<td>10 gm praseodymium metal</td>
</tr>
<tr>
<td>Columbus, Ohio</td>
<td>10 gm neodymium metal</td>
</tr>
<tr>
<td></td>
<td>10 gm samarium metal</td>
</tr>
<tr>
<td></td>
<td>10 gm gadolinium metal</td>
</tr>
<tr>
<td></td>
<td>50 gm lanthanum oxide</td>
</tr>
<tr>
<td></td>
<td>50 gm cerium oxide</td>
</tr>
<tr>
<td></td>
<td>50 gm praseodymium oxide</td>
</tr>
<tr>
<td></td>
<td>50 gm neodymium oxide</td>
</tr>
<tr>
<td></td>
<td>50 gm samarium oxide</td>
</tr>
<tr>
<td></td>
<td>50 gm gadolinium oxide</td>
</tr>
<tr>
<td></td>
<td>50 gm ytterbium oxide</td>
</tr>
<tr>
<td></td>
<td>20 gm terbium oxide</td>
</tr>
<tr>
<td></td>
<td>10 gm thulium oxide</td>
</tr>
<tr>
<td></td>
<td>10 gm lutetium oxide</td>
</tr>
<tr>
<td></td>
<td>150 gm dysprosium metal</td>
</tr>
<tr>
<td>Midwest Microlab, Inc.</td>
<td>100 mg erbia oxide</td>
</tr>
<tr>
<td>Indianapolis, Indiana</td>
<td>100 mg cerium oxide</td>
</tr>
<tr>
<td></td>
<td>100 mg praseodymium oxide</td>
</tr>
<tr>
<td></td>
<td>100 mg neodymium oxide</td>
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<tr>
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<td>100 mg samarium oxide</td>
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<td>100 mg europium oxide</td>
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<td>100 mg gadolinium oxide</td>
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<td>100 mg holmium oxide</td>
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<td>100 mg erbium oxide</td>
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<td>100 mg thulium oxide</td>
</tr>
<tr>
<td></td>
<td>100 mg ytterbium oxide</td>
</tr>
<tr>
<td></td>
<td>100 mg lutetium oxide</td>
</tr>
<tr>
<td>Brown University</td>
<td>1 pkg, organo metallic compound</td>
</tr>
<tr>
<td>Providence, Rhode Island</td>
<td>1 lanthanum cylinder</td>
</tr>
</tbody>
</table>
5-200 mg pellets each of yttrium metal, lanthanum metal, cerium metal, neodymium metal, gadolinium metal, ytterbium metal, dysprosium metal

150 pellets, 100 mg each of yttrium metal, cerium metal, neodymium metal, gadolinium metal, dysprosium metal, ytterbium metal

cylinders of neodymium metal as follows:
6 1" x 2" diam.
6 1/2" x 2" diam.
6 1/4" x 2" diam.
6 1/8" x 2" diam.

50 gm dysprosium metal

5 gm lanthanum metal

200 gm yttrium metal

20 gm neodymium oxide

491 gm calcium