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

ISC-904

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, 1957

by

Ames Laboratory Staff

September 11, 1957

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 January - June, 1957

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, G. Burnet, R. W. Fahien, R. W. Fisher, G. Murphy, E. H. Olson, M. Smutz and D. R. Wilder.

Chemical Engineering

1. Uranium Recovery During the Manufacture of Wet Process Phosphoric Acid (G. Burnet)*

Samples of raw material, process and waste streams from a commercial fertilizer plant using the Prayon process were analyzed for uranium. A standard fluorometric method was employed. The Prayon process uses a low-temperature digestion of the rock-acid mixture, which tends to cause the uranium from the phosphate rock to appear in the product acid. This was confirmed by the analytical results which showed nearly 85% of the available U in the product acid.

A similar set of samples from a plant using the Dorr-Oliver process are now being analyzed. This process employs a high-temperature digestion and larger amounts of U are expected in the various waste streams which will be investigated for economical U recovery.

2. Mass Transfer in Gaseous Streams (R. W. Fahien)

Total diffusivities have been calculated from concentration and velocity data for the transfer of carbon dioxide in air in a 4-inch cylindrical tube at Reynolds numbers of 1700, 4500 and 7600. Point values of these diffusivities were found to vary with radial position and longitudinal position within the tube. Although molecular diffusion was found to control near the wall of the tube, total diffusivities near the center were found to be several times the molecular values. Average diffusivities were found to decrease with longitudinal position, the decrease being less pronounced at the higher Reynolds numbers. It is believed that these average values are influenced by entrance effects at short diffusional distances. The results of this work indicate that non-molecular diffusional processes are of greater importance in most process equipment than is commonly assumed to be the case.

*Names indicate group leaders in charge of work.

A report (ISC-908) entitled "Mass Transfer in Low Velocity Gas Streams" by John E. Frandolig and R. W. Fahien is being distributed.

ABSTRACT

Experimental studies of the mixing of coaxial streams of carbon dioxide and air at ambient temperature and atmospheric pressure were made at Reynolds numbers of 1695, 4510, and 7605.

The test section consisted of a vertical, 4-inch galvanized iron pipe. Carbon dioxide was introduced into the center of an air stream free of moisture and carbon dioxide by means of an injection tube which extended the full length of a calming section 14 feet 4 inches long. Radial concentration profiles of carbon dioxide were measured at three positions downstream. Gas samples were withdrawn by a pitot-type probe and analyzed by a thermal conductivity cell. Velocity distributions were determined by means of a five-loop, circular hot-wire anemometer.

Total diffusivities of carbon dioxide in air for the entire radius of the pipe were computed by a semi-numerical method. The total diffusivities were found to vary significantly with position in the pipe and to be rather complicated functions of local flow conditions.

An approximate analytical solution of the general mass transfer equations describing the system was employed to compute total diffusivity values representative of the overall mass transfer process. This solution was derived assuming the total diffusivity and velocity to be independent of radial position.

3. Heat Transfer in Gaseous Streams (R. W. Fahien)

The accurate determination of thermal and momentum diffusivities in turbulent gas streams requires the simultaneous measurement of velocity and temperature. A single-element sensing device was constructed to carry out such measurements at five radial positions in a cylindrical four-inch tube. The device consisted of five concentric circular hot-wire anemometers. The current-voltage relationship was measured while current was continuously varied. Equations were developed to permit the calculation of temperature and velocity from current, voltage, and the rate of change of current with voltage. Calibration data at three levels of gas temperature and velocity and three levels of wire resistance indicated that modifications are necessary in order to make the measurements less cumbersome.

A report (ISC-909) entitled "Simultaneous Single Element Air Temperature and Velocity Measurements" by James E. Benjamin and R. W. Fahien is being distributed.

ABSTRACT

Equations were derived for measuring gas velocity and temperature simultaneously, using a single heated platinum wire as the sensing element. These equations contained four unknown coefficients which were considered constant. These constants were to be determined by calibrating at two known levels of temperature and velocity.

The method of measuring required numerical values for the current and voltage drop in the wire and the derivative of the voltage with respect to the current. These were achieved by plotting curves of current versus voltage with an X-Y plotter and taking slopes from the resultant curves.

Calibration was attempted on an instrument consisting of concentric platinum wire loops mounted in a vertical 4-inch pipe which has heated air flowing upward through it. Equipment and procedure of the calibration are given in detail.

From the values obtained for the four calibration "constants" found at three levels of gas temperature and velocity and three levels of resistance, it was concluded that the theory was oversimplified in assuming constancy of these coefficients. However it was felt that determination of the relationships between the four coefficients and the system parameters would make this means of measurement possible though complicated.

A vivid graphic portrayal of the degree of turbulence in "laminar" and turbulent flow was also demonstrated.

4. Bastnasite Processing (E. H. Olson)

The purpose of this project is to develop an economical processing method for the recovery of pure rare earths from bastnasite ore or concentrate.

The two major deposits of bastnasite ore in the United States are located in California and New Mexico. The bastnasite investigated in this project was obtained from the California deposit.

Although hydrochloric, nitric, and sulfuric acids will all open the bastnasite ore or concentrate, preliminary cost estimates

indicated that nitric acid would result in a more economical process, provided the excess nitric acid required could be recovered and re-used in the process. It was demonstrated that 95 to 99 per cent of the excess nitric acid used to open the ore could be recovered by fractional distillation from a sulfuric acid-nitric acid leach liquor solution from which the calcium and rare earths had been removed.

From the investigations completed to date, the most economical processing method would consist of the following operations:

- a. Grinding the ore.
- b. Igniting the ore to reduce the carbonates to oxides.
- c. Leaching the ore to dissolve the rare earths with nitric acid.
- d. Filtering the insoluble residue from the leach solution.
- e. Solvent extracting the rare earths from the filtrate with tributyl phosphate.
- f. Precipitating the calcium from the raffinate solution with sulfuric acid.
- g. Filtering the calcium sulfate from the raffinate solution.
- h. Fractionally distilling the nitric acid from the filtrate for recycle to the leaching step (c).

From small-scale tests of the process, the overall recovery of rare earths was greater than 95 per cent.

Scale-up of the process and preparation of a cost estimate are in progress.

5. Oxidation of Cerium(III) to Cerium(IV) (E. H. Olson)

The purpose of this project is to determine the amount of trivalent cerium (cerium(III)) that can be converted to tetravalent cerium (cerium(IV)) in a nitric acid solution. Since cerium(IV) is nearly quantitatively extractable from a nitric acid solution into tributyl phosphate, the separation of cerium from a mixture of rare earths would be much easier if the cerium could be oxidized to the tetravalent state before extraction.

The cerium is oxidized by heating the cerium nitrate in a strong nitric acid solution at temperatures from 100 to 120°C and sparging with an inert gas to remove the nitrogen-oxygen decomposition products formed. Theoretically, if the gaseous decomposition products formed during the oxidation of cerium are removed, the trivalent cerium should be quantitatively converted to the tetravalent state. However, the highest conversion obtained has been approximately 35 per cent. Possible reasons that a higher conversion has not been realized are (1) incomplete sparging of the decomposition products or (2) tetravalent cerium nitrate is

unstable at the temperatures used in these investigations. Both of these possibilities are being investigated.

Another possible method of separating cerium from a mixture of rare earths is to carry out a fusion of a magnesium nitrate-rare earth nitrate mixture at approximately 250°C. The cerium is preferentially oxidized to the water insoluble ceric oxide while the other rare earths are left unchanged as soluble nitrates.

It has been found that the cerium can be completely oxidized by fusion of a 1:1 magnesium nitrate-rare earth nitrate mixture for one hour at 275°C. The ceric oxide was noted to be very pure and practically free of other rare earths present in the original mixtures.

The major difficulty with this fusion process has been in separating the finely divided ceric oxide from the other rare earth nitrates. At present it appears that a filtration of an aqueous solution of the fused material containing Celite filter aid will be the most feasible method.

A preliminary test using sodium nitrate in place of the magnesium nitrate was not very promising. A 2:1 weight ratio of sodium nitrate to rare earth nitrates was fused for 16 hours at 275°C. The cerium oxidation was incomplete.

Future studies on this project will include the determination of the optimum fusion temperature and the temperature at which the first of the other rare earth nitrates starts to decompose to the oxide. The possibility of substituting sodium or potassium nitrate for the magnesium nitrate will also be investigated.

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

The purpose of this project is to find an economical method for the removal of fission products from uranium by liquid metal extraction.

The investigations during this period consisted of a series of silver extraction runs whereby radio-tracer neodymium was extracted from a uranium-neodymium alloy containing 1500 ppm neodymium. In the eight extraction runs made, a graphite extractor was used in which successive equal-sized drops of silver could be passed upward through a cylindrical column of molten uranium. The following variables were investigated:

- a. Temperature - 1250 and 1290°C.
- b. Size of silver drops - 2.4 and 1.5 gms/drop.
- c. Rate of feed of silver - 3.65 to 15.10 gms/min.

The size of the silver drops was found to be the most important factor affecting the removal of neodymium. Contrary to expectations, the large drops resulted in the removal of greater amounts of neodymium with a smaller amount of total silver. Normally one would expect the smaller silver drops with a higher surface area/volume ratio to extract more neodymium. Part of the answer may be due to the fact that the diameter of the large drops was very close to the diameter of the column of uranium through which they were passing. This could mean that, although the surface area/volume ratio was smaller, the time of rise through the uranium phase of the large silver drops was much greater due to wall effects, thereby giving increased time for extraction of neodymium.

Decontamination factors (concentration of Nd in uranium phase before extraction/concentration of Nd in the uranium phase after extraction) ranged from 3.43 to 2.46 for the large silver drops. From one run in which no silver was used, it was noted that a considerable amount of neodymium was removed with the carbide scale which forms on the crucible wall. It appears that in this silver extraction process, neodymium removal occurs by a combination of carbide formation and silver extraction.

Results of these first extraction runs are quite encouraging, since a 70 to 90 per cent removal of fission product poisons from a uranium fuel would probably be satisfactory if the process equipment and remote handling operations were not too expensive.

The effects of agitation on neodymium removal will be investigated.

Engineering Development

1. Slurry Program (Glenn Murphy)

Additional experimental data have been obtained on the mechanical performance of slurries consisting of small plastic spheres in water. Curves for predicting loss of head and pumping power have been developed. The observation made previously that less power is required to pump certain heavy slurries than is required for the liquid phase alone has been confirmed. This important finding has been further studied and it appears to result from the extension of laminar flow to Reynolds numbers which would result in turbulent flow in the fluid alone.

An analysis of the coring phenomenon has been made and, on the basis of the analysis, predictions were made on the coring in slurries containing particles having a specific gravity less than that of the liquid phase. The predictions were then checked experimentally.

A description of a photographic method for the study of flow in slurries and the results obtained are given in detail in ISC-874, which is being distributed.

Ceramic Engineeinging

1. Effect of Calcium Fluoride Additions on the Sintering of Magnesium Oxide (D. R. Wilder)

The first phase of this work has been largely completed and will be reported in detail in ISC-910. The general conclusions drawn from the work are that in highly compacted (dry pressed) magnesium oxide powders, up to four mol-per cent additions of calcium fluoride aid sintering at all sintering temperatures from 1300°C to 1900°C. Greater amounts appear to cause bloating and general loss of desired characteristics. No compound formation nor solid solution was found to exist. The mechanism involved is thought to be formation of a liquid phase above a binary eutectic temperature, rather than a solid state reaction. With a lower degree of cold compaction, such as encountered in the jolt-pack technique, no optimum content of calcium fluoride was found to occur.

It should be noted that the increase in sintering rate, as indicated by shrinkage, densification, and loss of porosity, does not necessarily indicate an improvement in either cold or hot load strength. Work is being directed toward investigation of the relation between calcium fluoride additions and hot and cold strength properties.

Other Research

1. Hot Loop Experiment (R. W. Fisher)

Plans for a double pass uranium-bismuth to sodium loop were completed and the various components are under construction.

Preliminary tests were required prior to the combining of these loops to ascertain whether or not sodium could be contained in an Inconel system. A loop of Schedule 40 Inconel pipe was charged with metallic sodium under an inert atmosphere of helium and operated for 600 hours at 600°C before a leak developed in one of the exterior welds.

An examination of the system after shutdown indicated the leak was not caused by the corrosive action of the sodium. Consequently, a second similar loop was constructed and it has been operating at 800°C for 750 hours with no signs of damage. This experiment will be continued for 2000 hours.

APPENDIX I: LIST OF REPORTS FROM THE AMES LABORATORY

1. Reports for Cooperating Laboratories

- ISC-687 John Gibson and J. M. Keller. Multi-Band Electrical Conduction.
- ISC-700 D. A. McCutchan and Glenn Murphy. The Stress-Strain Characteristics of Uranium.
- ISC-726 R. M. Henry and D. S. Martin. Comparative Yields of $\text{Fe}^{54}(\gamma, \text{pn})\text{Mn}^{52}$ and $\text{Fe}^{54}(\gamma, 2\text{n})\text{Fe}^{52}$.
- ISC-729 Thomas Hendrickson and J. M. Keller. Ferromagnetic-Antiferromagnetic Phase Transitions.
- ISC-746 Joseph I. Genco, M. Griffel, F. R. Duke and L. D. Jennings. The (C-H) Bond Dissociation Energy in the Methyl Group of Toluene.
- ISC-757 Ames Laboratory Staff. Chemistry. Semi-Annual Summary Research Report. January-June, 1956.
- ISC-758 Ames Laboratory Staff. Physics. Semi-Annual Summary Research Report. January-June, 1956.
- ISC-759 Ames Laboratory Staff. Metallurgy. Semi-Annual Summary Research Report. January-June, 1956.
- ISC-777 Charles Apel and H. J. Svec. Kinetics of the Reaction Between Calcium and Water Vapor.
- ISC-779 Dale Gibbs and H. J. Svec. Kinetics of the Reaction Between Magnesium and Water Vapor.
- ISC-802 Ernest L. Koerner, Jr., Morton Smutz and H. A. Wilhelm. Separation of Niobium and Tantalum by Liquid Extraction.
- ISC-804 G. W. Eakins, B. J. Loupee, W. A. Rhinehart, G. Schupp and E. N. Jensen. A Method for Reducing Time-Jitter in the Output of a Single Channel Analyzer.
- ISC-812 K. A. Gschneidner, D. J. Beerntsen, R. W. Vest, J. A. Kingston and J. F. Smith. Tabulation, Bibliography, and Structure of Binary Intermetallic Compounds. II. Compounds of Beryllium, Magnesium and Calcium.
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APPENDIX II: LIST OF SHIPMENTS

<u>Destination</u>	<u>Item</u>
Dr. Gilbert Eggert Sandia Corp. Albuquerque, New Mexico	650 gm zinc-zirconium alloy
University of Minnesota Minneapolis, Minnesota	1 gm hafnium metal
University of Kansas Lawrence, Kansas	25 gm cerium metal
State University of Iowa Iowa City, Iowa	5 gm terbium oxide 2 gm special N ¹⁵ sample
Union Carbide Nuclear Company Oak Ridge, Tennessee	100 gm yttrium oxide 10 gm lanthanum oxide 10 gm neodymium oxide 10 gm samarium oxide 10 gm gadolinium oxide 10 gm dysprosium oxide 5 gm praseodymium oxide 1 gm ytterbium oxide 500 mg holmium oxide 500 mg erbium oxide 250 mg terbium oxide 250 mg thulium oxide 100 mg lutetium oxide 2 kgm dysprosium oxide 1 kgm ytterbium oxide 20 gm samarium metal 20 gm gadolinium metal
General Electric Research Laboratory Schenectady, New York	5 gm holmium metal
Brookhaven National Laboratory Upton, Long Island, New York	1 gm holmium oxide 1 gm thulium oxide 1 gm terbium oxide 1 gm lutetium oxide 4 pieces erbium metal 100 mg thulium metal
Princeton University Palmer Physical Laboratory Princeton, New Jersey	3 gm hafnium oxide
Lansen-Naeve Corp. New York 4, New York	10 gm ytterbium metal

<u>Destination</u>	<u>Item</u>
Oak Ridge Institute of Nuclear Studies Oak Ridge, Tennessee	1 gm holmium oxide 1 gm lutetium oxide
University of Kentucky Lexington, Kentucky	2 gm samarium oxide 2 gm neodymium oxide
Dr. John Owen The Clarendon Laboratory Oxford, England	2 specimens of gadolinium-yttrium alloy
Heavy Minerals Company Chattanooga, Tennessee	1 gm lanthanum oxide 1 gm cerium oxide 1 gm praseodymium oxide 1 gm neodymium oxide 1 gm samarium oxide 1 gm yttrium oxide 1 gm gadolinium 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
University of California Los Alamos Scientific Laboratory Los Alamos, New Mexico	20 gm yttrium oxide 20 gm lanthanum oxide 20 gm praseodymium oxide 20 gm neodymium oxide 20 gm samarium oxide 20 gm gadolinium oxide
	5 gm erbium oxide 5 gm ytterbium oxide 3 gm terbium oxide 3 gm dysprosium oxide 3 gm holmium oxide 3 gm thulium oxide 3 gm lutetium oxide
Institut für Physikalische Chemie an der Technischen Hochschule Vienna, Austria	10 gm hafnium metal
Dr. Frank Benner National Research Corp. Cambridge, Massachusetts	5 samples yttrium metal

<u>Destination</u>	<u>Item</u>
Purdue University Lafayette, Indiana	10 gm cerium oxide
Massachusetts Institute of Technology Cambridge, Massachusetts	2 gm yttrium oxide 2 gm lanthanum oxide 2 gm erbium oxide 3 gm dysprosium metal 1 gm gadolinium metal
Columbia-Southern Chemical Corp. Barberton, Ohio	50 gm xenotime sand
Physikalisches Institut der Technischen Hochschule Munich, Germany	1 gm terbium metal 1 gm holmium metal 1 gm thulium metal
National Bureau of Standards Washington 25, D. C.	2/10 gm each of the following oxides: cerium dysprosium holmium thulium ytterbium thorium
Universitetet Institute for Teoretisk Fysik Copenhagen, Denmark	1 gm dysprosium oxide 1 gm erbium oxide
Dr. Joseph Graca Iowa State College Ames, Iowa	9.7 gm yttrium oxide 13.45 gm erbium oxide 11.17 gm dysprosium oxide 200 ml each of the following 5% solutions: holmium ethylenediamine- tetraacetate yttrium chloride thulium chloride ytterbium chloride terbium chloride holmium chloride cerium chloride dysprosium chloride samarium chloride gadolinium chloride erbium chloride