

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission

A. Makes any warranty or representation, express or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission to the extent that such employee or contractor prepares, handles or distributes, or provides access to, any information pursuant to his employment or contract with the Commission.

SECRET

UNCLASSIFIED

CP-3445
G-C

University of Chicago
Metallurgical Laboratory

Contract No. T-7401-eng-37

Physics and Metallurgy Division

REPORT FOR MONTH OF FEBRUARY 1946

CONFIDENTIAL

...

CLASSIFICATION CANCELLED
DATE MAR 13 1957 *RLT*
For The Atomic Energy Commission
H. R. Canale
Chief, Declassification Branch

Even

A. Crystal Structure Section - W. H. Zachariasen	2	} <i>Delete for Binder</i>
B. Mass Spectroscopy Section - A. J. Dempster	4	
C. Metallurgy Section - F. Foote	8	} <i>Delete for Binder</i>

Photostat Price \$ 1.50
Microfilm Price \$ 1.80

Available from the
Office of Technical Services
Department of Commerce
Washington 25, D. C.

CLASSIFICATION CANCELLED TO
CONFIDENTIAL
DATE 3-13-57
H. R. Canale
Chief, Declassification Branch

27 February 1946

Report Received: February 28, 1946
Issued:

271-1

1946

UNCLASSIFIED

REPORT FOR THE MONTH OF FEBRUARY 1946

Crystal Structure Section

V. H. Zachariasen, Section Chief

* * *

Neptunium Studies (S. Fried)

NpOCl_2 was prepared by heating NpCl_4 in a sealed evacuated capillary to 450°C . Presumably traces of water present on the walls of the capillary were responsible for its formation according to the reaction:



The NpOCl_2 formed in clumps of light yellow needles which could be sublimed at 550°C .

Treatment of NpOCl_2 with hydrogen at 450°C in an attempt to prepare NpOCl failed, NpO_2 being produced. Treatment of NpO_2 with an $\text{H}_2\text{-H}_2\text{O-HCl}$ mixture at 500°C also failed to produce NpOCl .

The Systems KF-UF_4 , KF-ThF_4 , KF-LaF_3 (V. H. Zachariasen)

Crystal structure studies of these systems have been completed.

The following phases have been found:

In the KF-UF_4 system: KUF_6^{25} , KUF_3^{13} , KUF_2^9 , KUF_5 , $\alpha\text{-K}_2\text{UF}_6$, $\beta\text{-K}_2\text{UF}_6$, $\alpha\text{-K}_3\text{UF}_7$, $\beta\text{-K}_3\text{UF}_7$.

In the KF-ThF_4 system: KThF_6^{25} , KThF_2^9 , KThF_5 , $\alpha\text{-K}_2\text{ThF}_6$, $\beta\text{-K}_2\text{ThF}_6$, K_3ThF_9 .

In the KF-LaF_3 system: $\alpha\text{-KLaF}_4$, $\beta\text{-KLaF}_4$.

The detailed results are given in report CC-3426.

SECRET

secret

- 3 -

ThF₂

(SEC-FWEL-168)

The phase reported as β - Th_2F_9 in report OC-3401 has been shown to be ThF_2 .

NpOCl₂

X-ray examination has shown that Dr. Sherman Fried has succeeded in preparing the compound NpOCl_2 . The identity of the compound is established through isomorphism with the corresponding uranium compound.

Cs₂PuCl₆

Mr. H. H. Anderson has prepared Cs_2PuCl_6 and established the formula by direct chemical analysis.

Cs_2PuCl_6 is hexagonal with one molecule per unit cell and lattice dimensions

$$a_1 = 7.45 \pm 0.02 \text{ \AA}, \quad a_3 = 6.05 \pm 0.02 \text{ \AA}.$$

The calculated density is $\rho = 4.05$.

Graphite

Heavily irradiated graphite from Hanford has been examined before and after heat-treatment. The X-ray data indicate that about 35% of the carbon atoms trapped between the layers are not removed after heat-treatment at 1000°C .

In samples examined earlier, it was found that all trapped atoms were removed at 1000°C .

SECRET
DECLASSIFIED FEB 1 - 3

REPORT FOR THE MONTH OF FEBRUARY, 1946

Mass Spectroscopy Section

A. J. Dempster, Section Chief

* * *

1. Mass Assignment of Active Isotopes

a. Fission Products (Wyden, Rall) - There still remain about a dozen active fission products whose mass assignments are uncertain. Most of these will require a new type of source in place of the hot filament previously used. Mr. Wyden has developed a source in which the material is vaporized in a small furnace and ionized by electrons from a filament. Mass spectra have been obtained with cadmium, tin, lead, and mercury, and this source should be applicable to fission product isotopes in germanium, tin, antimony, and tellurium.

Mr. Rall has studied the amount of analyzed palladium that is deposited in the mass spectrograph using a spark source, and found that it is adequate for observing the 19 hour palladium fission product.

b. Heavy Elements (Shaw) - It has been found that uranium nitride gives a high yield of uranium ions at high temperatures. A tungsten crucible heated by electron bombardment has been developed to hold larger amounts of material than can be heated on a tungsten filament. With this it should be possible to find the masses of many weaker active isotopes, as well as some of the new heavy radioactive isotopes.

2. C^{14} Content of Material Produced at "X" (Ingram)

Two samples of the enriched carbon prepared at the Clinton Laboratories were analyzed. The material was in the form of barium

DECLASSIFIED 271-4

carbonate, and carbon dioxide was liberated by heating 20 mg. in a previously exhausted quartz tube. The CO_2^+ ions were observed, and the amount of C^{14} was deduced from the intensity of the peaks at masses 44, 45, 46, 47, and 48.

The percentage of C^{12} , C^{13} , and C^{14} is given in the following

table:

Sample	C^{12}	C^{13}	C^{14}
Normal CO_2	98.92%	1.08%	0.00%
Sample #15	95.604 ± .049	1.042 ± .011	3.353 ± .032
Sample #16	95.730 ± .044	1.045 ± .010	3.227 ± .034

These values will permit the accurate calibration of various detecting instruments, and also give a new determination of the half life of C^{14} .

There was apparently complete evolution of the CO_2 from the barium carbonate near 1500°C , as heating to higher temperatures to collapse the tube gave no more. On first heating, the sample, it was noticed that there was an evolution of CO_2 at 300°C equivalent to about one per cent of the total amount. No more CO_2 was obtained until temperatures near 1000°C were reached. This evolution at 300°C is probably CO_2 from the atmosphere occluded in the barium carbonate. Its carbon 14 content was measured, however, and found to be 1.48 per cent. This probably means that absorbed CO_2 will exchange carbon atoms with the carbon in the barium carbonate. The values given in the table are from measurements on five different amounts of gas set free above 1000°C . There was agreement within the experimental error between these values. Details of the measurements will be given in a separate memorandum by M. G. Inghram.

DECLASSIFIED 71-5

3. Impurities in Beryllium Metal Prepared at M.I.T. (Hall, Dempster)

Samples were examined to determine whether it was possible to measure the oxygen content in different samples of beryllium. The doubly charged oxygen at mass 8 was compared by means of electrometers with the beryllium at mass 9. The observed values fluctuated, however, due to oxidation of the surface in the neighborhood of the spark by residual gases. Special methods will be tried in an effort to eliminate this effect. The presence of many other impurities was observed on mass spectra photographs including Na, K, Cl, N, Mg, Al, Si, and Ni.

4. Abundance of Copper Isotopes (Dempster, Hall)

In connection with the examination of a sample of copper, the normal relative abundance of the copper isotopes was measured with the ion separator. A spark between copper and tantalum was used and the ratio of 63 to 65 was found to be $2.23 \pm .03$. (In a German periodical of 1944 just received, H. Ewald reports the measurement of the ratio with a spark between electrodes of a copper-zinc alloy.) The mass spectra were measured photometrically, using Eiler's values for the zinc isotopes as standards, and give a value for the ratio of $2.35 \pm .02$. The difference is larger than the experimental errors, and further measurements will be made on other samples of metal and alloys.

The importance of careful measurements of abundance ratios by different methods lies in the possibility of detecting unsuspected processes for isotope enrichment. These may have produced differences in the samples, or may give rise to differences during the production of the ions for analysis.

5. Enrichment of Neodymium by Absorption Processes - Further measurements on new samples received from Ames will be made in the near future.

~~SECRET~~

6. The mass spectrograph for the Bureau of Standards (MEO type) has been tested and prepared for shipment.

REPORT FOR MONTH OF FEBRUARY, 1946

METALLURGY SECTION

Frank Foote, Section Chief

1. Extrusion of Thorium-Uranium Alloys (N. P. Pinto)

Copper jacketed ingots of U-Th alloys of various compositions have been extruded to 1/4" rod at Site B. The 25% U-75% Th alloy may be extruded successfully at any temperature between 625°C and 800°C. Optimum temperature for the extrusion of the 50% U-50% Th alloy appears to be near 625°C; attempts to increase or lower the temperature result in decreased rod quality. The alloy of 75% U-25% Th has been extruded successfully at 550°C, but poor results were obtained at 500°, 700°, and 800°C. The pressure required for this work is approximately 50 tons/in².

2. Fabrication of Beryllium and its Alloys

Extrusion (N. P. Pinto, S. H. Paine)

Hexagonal rods, 0.238" across the flats, have been extruded satisfactorily. Composite billets, with 0.070" and 0.100" thick beryllium shells and 10% U-90% Be cores and having ends of pure beryllium, extruded to hex shape show fairly good bonding between the shell and core. A metallographic examination of the cross section revealed a light line between the layers with little interdiffusion; there was no improvement after annealing at 750°C for 2 hours. The minimum thickness of the beryllium shell varied from 12 to 20 mils for the thick wall and 4 to 10 mils for the thin wall. Cross sections of the trailing half revealed a shell of nearly constant thickness; the corners of the alloy core were sharp and conformed closely to the geometry of the die.

Rolling (N. P. Pinto)

The 25% U-75% Be alloy may be rolled without difficulty. A 1-1/8" diameter casting has been rolled to 0.060" sheet at 1000°C without signs of cracking due to rolling.

An order from the Chemistry Section for 5, 10, 20, 50, 100, 200, and 500 mil pure beryllium sheet is half complete.

Casting (Geo. O'Keefe)

A 6-3/4" x 8-3/4" x 6-3/4" high truncated cone of Be-2% Al was cast to complete a mortar and pestle assembly for Argonne.

3. Oxidation Rates of U-Cb-Zr Alloys in Air at 150°C (B. Sawyer)

With the possible use of a non-oxidizing uranium alloy in an air-cooled reflector around a fast pile in mind, a test was begun to measure the weight change rates of all the 19 alloys on hand in the U + 6% alloy corner of the U-Cb-Zr alloy system.

The preparation of the specimens was: a homogenization for approximately one week at 1000°C; then a soak at 850°C and a fast brine quench to preserve as much gamma phase as possible. After this the samples were ground on #250 grit emery paper. Total surface areas were measured, and initial weights taken. Each sample was placed in a watch glass to catch all oxide that might be formed. The set was then placed in a regulated oven at 150°C with slow air circulation, and weight changes were measured regularly.

The last set of weighings was made after a total of 282 hours in oven. It is early to evaluate results, but the following statements can safely be made:

1. Three samples gained weights at fairly constant rates from the beginning of the test. These were: the pure uranium sample, the U + 0.3% Cb, and the U + 1% Zr.
2. All the rest showed insignificant changes until after 50 hours when the U + 0.55% Cb and U + 2% Zr alloys began gaining weight.
3. After 120 hours the 0.9% Cb and the 3, 4, and 6% Zr alloys began gaining weight.
4. All the other alloys have held up for at least 200 hours.

After 282 hours the five most active samples were removed from test and cleaned in 25% HNO₃. Their average weight loss rates for the whole period follow.

Sample:	Pure U	.3% Cb	.55% Cb	1% Zr	2% Zr
Rate: (mg/cm ² /hour)	0.64	0.26	0.25	0.17	0.048

4. Effect of Sodium-Potassium Alloys on Various Materials
(H. Morris and L. Kolman)

A circulating test has been made in which liquid sodium-potassium alloy was pumped for 12 hours at room temperature. The centrifugal pump was run at approximately 1600 R.P.M. which speed produces a linear flow in the system of approximately 100 cm/sec. Approximately 140 pounds of alloy were used and no serious difficulties were encountered in preparing the alloy, getting it into and out of the pump tank, pumping the alloy

for 12 hours, or cleaning the pipes and pump after the run was completed. No leaks occurred in the seven ring seal joints in the external piping system. A nitrogen atmosphere was maintained in the steel housing which encloses the entire system and in the pump tank. The system is now being prepared for corrosion testing at elevated temperatures.

A high resistivity metal which can be used along with low carbon steel in the liquid sodium-potassium alloy circulating system is needed in developing an electromagnetic pump and flowmeter. Several metals are now being tested in static corrosion tests to find a suitable metal for that use.

SECRET

END

DECLASSIFIED

271-10