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For October, November and December 1950

by

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TABLE OF CONTENTS

Page

METALLURGY

1. Thorium	7
1.2 Metallurgical Research	7
2. Calcium	7
2.2 Metallurgical Research	7
3. Zirconium	8
3.1 Metallurgical Research	8
4. Vanadium	10
4.1 Metallurgical Research	10
5. Uranium.	11
5.1 Metallurgical Research	11
6. High Temperature Alloys	11
6.1 Metallurgical Research	11
7. Equipment	11
8. Rare Earth Metals	12

CHEMISTRYPhysical and Inorganic Chemistry Groups 13

1. Separation of Rare Earths by Ion Exchange . .	13
1.1 The Light Rare Earths (Cerium Group) .	13
1.2 Heavy Rare Earths (Yttrium Group). . .	16
2. Activity Coefficients of the Rare Earth Salts	17
3. Preparation and Properties of the Rare Earth Metals	18
4. Hafnium Recovery	18
5. Slurry Studies	19
6. Corrosion Studies	19
7. Adsorption Studies	19
8. Hydrogen Analysis in Organic Compounds . . .	20
9. Reaction Mechanisms	20
10. Precision Calorimeters	21
11. Single Crystals of MnS	21
12. Hydrogen-Uranium Equilibrium	21
13. Gas Purification	22
14. Oxygen in Metals	22

Page
22

Radiochemistry Groups

1. Research on Ruthenium	22
2. Zirconium Chemistry	23
3. Synchrotron-induced Activities	23
4. Self-diffusion in Nickel	25
5. Photofission Yields of Thorium	25
6. Chemical Equilibrium and Kinetics	25
7. Service Work	26

Spectrochemistry Group

26

1. Spectroscopic Methods for the Analysis of the Rare Earths	26
2. Spectrographic Determination of Residual Impurities in Zirconium Metal	26
3. Miscellaneous Research	27
4. Service Analyses	27
5. Zirconium-Hafnium	27

X-Ray Chemistry Group

28

1. Thorium Metal Compounds-Transition	28
2. Zirconium Alloy Systems	28
3. Electron-Deficient Compounds	28
4. Complexes of Aromatic Molecules	28

Analytical Chemistry Group

29

1. Zirconium	29
2. Miscellaneous Investigation	29
3. Service Analyses	30

PHYSICS

Theoretical Physics

31

1. Ionization Statistics	31
2. Specific Heat of Ce	31
3. Multiple Scattering in a Cloud Chamber	32
4. 2-gamma or 2-electron Decay	32

Experimental Physics

33

1. Beta and Gamma Ray Spectrometry	33
1.1 Beta Spectrum of P^{32}	33
1.2 Radiations from Te^{125}	33
1.3 Radiations from Hf^{181}	33
1.4 Radiations from Sb^{124}	33
1.5 Double Lens Beta Spectrometer	33
1.6 Transmission Characteristics of Thin Foil	34

555-003

2.	Application of Scintillation Counters to β -Ray Spectrometry	34
2.1	Analysis of Instrumental Performance	34
2.2	The Low-Energy Beta-Spectrum of Rb^{86}	34
2.3	The Beta-Spectra of Ag^{112} and Ag^{113}	34
2.4	Other Applications of Scintillation Spectrometry	35
3.	Fission Fragment Experiments	35
3.1	Ionization Defect Experiment	35
4.	Scintillation Pulses from Slow Positive Ions	36
5.	Diamond Counters	36
6.	Piezoelectricity at Low Temperatures	38
7.	Sodium Tungsten Bronze (Na_xWO_3)	38
8.	Work with 12-inch Cloud-Chamber	39
8.1	Examination of Synchrotron Radiation Spectrum	39
9.	Study of Nuclear Processes with Photographic Emulsions	39
10.	Synchrotron Research and Development	40
11.	Scintillation Spectrometer	41
<u>ENGINEERING</u>		
	<u>Chemical Engineering Group</u>	42
1.	Production of Thorium from Monazite	42
2.	Production of Zirconium by Adsorption	42
3.	Production of Hafnium by Adsorption	43
4.	Production of Zirconium by Solvent Extraction	43
5.	Recovery of Spent Gel and Adsorbed Zirconium from Zirconium Production	44
6.	Properties of Slurries	45
	<u>Ceramic Engineering Group</u>	46
	<u>Nuclear Engineering Group</u>	46
1.	Reactor Materials	46
2.	Resistance of Ceramic Materials to Thermal Shock	46
3.	Reactor Slurry	47
	<u>Architectural Engineering Group</u>	48
APPENDIX I: List of Reports from the Ames Laboratory		49
1.	Reports for Cooperating Laboratories	49
2.	Papers Published in Scientific Journals	50
APPENDIX II: List of Shipments		52

QUARTERLY SUMMARY RESEARCH REPORT

For the period October, November and December 1950

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

Previous quarterly research reports in this series are:

ISC-35
ISC 41
ISC 56
ISC 69
ISC 74
ISC 76
ISC 79
ISC 113
ISC 130

555 005

METALLURGY

Under the direction of F. H. Spedding,
H. A. Wilhelm and B. A. Rogers

1. Thorium

1.2 Metallurgical Research

Investigations of several binary alloy systems of thorium have been started. Some of these alloys containing lead, molybdenum and tin have been prepared in the tungsten arc furnace.

Experimental work is also being undertaken in order to collect data on the physical properties of thorium. Few data are available yet, but swaging seems to be a practical method of forming rods. Work done on joining thorium to itself or to other metals with high temperature silver solder indicates that this procedure is impractical. The silver solder seems to take up enough thorium to become excessively brittle.

2. Calcium

2.2 Metallurgical Research

Further work has been done on the reduction of lime with aluminum at 1200°C. By using Alcoa grade 120 aluminum with Marblehead lime the yield of calcium was increased to between

75 - 85 per cent. Other combinations using Baker's aluminum, chunk aluminum and Mississippi lime all gave lower yields. Yields as low as 42 per cent were obtained in some instances.

3. Zirconium

3.1 Metallurgical Research

Approximately 75 pounds of purified zirconium as zirconyl chloride, still containing hafnium, was produced from zirconium tetrachloride. This material is being converted to anhydrous zirconium fluoride to furnish material for experimental reductions.

Calcium oxide liners have been adopted for bomb reductions because of the more consistent results obtained. The yield with sintered calcium oxide is about 85 per cent, that with jolted calcium oxide about 75 to 80 per cent on the small scale reductions. The jolted liners also gave a less massive product which is undesirable.

Variables are being studied in an effort to produce softer and more ductile zirconium. Softer metal was obtained when the calcium used in the reduction was machined in an argon atmosphere, probably because of less oxidation of the calcium. Several additions, including barium chloride, sodium chloride, sodium fluoride, zirconium fluoride, zirconium oxide and several rare earth chlorides, have been made to the charge but in no instance was there a decrease in hardness. Cerium chloride caused the largest increase in hardness which was about 9 points on the Rockwell A scale. Additions of carbon also caused increased hardness although there may be some softening of the metal during vacuum fusion at 2300° to 2400°C because of the carbon. Increasing the length of time of hydrofluorination of the zirconium fluoride has resulted in softer metal. Also, different lots of zirconium fluoride have given metal varying in hardness by as much as 15 points on the Rockwell A scale, but chemical analysis of the fluoride has not as yet explained this variation. By increasing the zinc content to 30 per cent in a zirconium-zinc alloy the hardness has been reduced so that even a poor grade of fluoride gave metal with a hardness of Rockwell A 45 to 50. As a result of these studies it is now possible to reproducibly obtain by the Ames process metallic zirconium which when cast in graphite compares favorably with crystal bar similarly cast.

555 007

Investigations of several binary alloy systems of zirconium including those with bismuth, germanium and antimony have been started recently.

Tentative phase diagrams have been made for the zirconium-carbon, zirconium-nitrogen and zirconium-oxygen systems. There is evidence of a eutectic in the zirconium-oxygen system.

Because of the excellent corrosion resistance obtained by the addition of relatively small percentages of tin, the zirconium-tin system is being studied carefully, especially at the low-tin side. As the result of additional work, the eutectic previously reported at 1400°C and 25 per cent tin has been moved to 1575°C and 21 per cent tin. The boundary of the solid solution region has been established from 1575°C down to 1100°C and an attempt is being made to find its position at lower temperatures. The addition of 10 per cent tin raises the alpha-beta transformation from 865°C to 1100°C. Beyond 10 per cent the temperature of transformation remains at 100°C for considerably larger proportions of tin.

A series of homogeneous zirconium-tin alloys containing from 0 to 5 per cent tin have been prepared by induction melting in graphite for corrosion testing in other laboratories. A number of zirconium-tin alloys have been made by co-reduction. As an example a 2 per cent tin alloy so made had a Rockwell hardness of A-52 and was cold-rolled to 93% reduction.

A complete metallographic study is being made of the zirconium-columbium alloy system. Alloys containing up to 90 per cent columbium have been prepared by an arc melting procedure from Bureau of Mines and Fansteel Metallurgical Corporation columbium. Hardness values have been obtained on the alloys in the as-cast, the annealed, and the quenched conditions. Thermal analysis of the alloys is now in progress. X-ray diffraction studies have shown the existence of two phases in the range between 15 and 40 per cent columbium. One phase is alpha zirconium, the other is a body-centered cubic phase as yet unidentified. The X-ray pattern indicates that the body-centered cubic phase increases in amount as the tin content rises from 15 to 40 per cent. This phase could not be detected in a 12 per cent alloy.

The phase diagram for the zirconium-uranium system has been essentially completed.

Corrosion testing is continuing on a routine basis. Zirconium-tin and zirconium-columbium alloys still appear promising with respect to corrosion resistance. Because some investigators believe that corrosion is more severe in crevices, at joints or in cracks, some tests are being made with samples piled or clamped together. Indications are that crevices do not affect the good corrosion resistance of the zirconium-tin alloys.

Formation of consumable electrodes of zirconium for use in an arc melting furnace has presented some difficulties. In hammer forging at 850°C in steel jackets the jacket ruptured after repeated blows. Usable electrodes have been made but possibly better rods can be formed by rolling or drop forging sponge metal and further work will be done along this line.

Experimental work is being continued on the problem of producing zirconium electrolytically. Use of zirconium iodide has presented some difficulty in the process because of its high volatility and instability. The equipment for this work is being modified.

4. Vanadium

4.1 Metallurgical Research

Metallic vanadium has been made by bomb reduction of several vanadium compounds with calcium. Three oxides: vanadium pentoxide, vanadium trioxide and vanadium dioxide; vanadium trisulfide and vanadium fluoride have all given yields of 80 per cent or more. Metal made from the reduction of vanadium dioxide had sufficient ductility to permit a reduction of 90 per cent in thickness by cold-rolling.

Vanadium for production of vanadium-titanium alloys by the powder method is being made by the electrolysis of vanadium trifluoride as mentioned in ISC-130. A similar process is being used for the production of titanium powder. The apparatus for producing these powders is being enlarged to provide the amounts of these elements needed for the alloy investigation. Also, some alloys have been made by the co-reduction of vanadium pentoxide and titanium dioxide in a bomb with calcium as a reductant. A procedure for determining titanium quantitatively in such alloys is yet to be devised.

5. Uranium

5.1 Metallurgical Research

Alloys of molybdenum and uranium containing from 5 to 25 atomic per cent molybdenum have been prepared and some data collected concerning their physical properties. The alloys containing 20 and 25 per cent molybdenum could be cold-rolled to about 75 per cent reduction in thickness while those containing lower percentages of molybdenum were less ductile.

6. High Temperature Alloys, 2 3

6.1 Metallurgical Research

The molybdenum-rhenium alloy system has been under investigation with a view to obtaining high-temperature thermocouple material. Powder metallurgical methods have been used for preparing the alloys which are then rotary swaged into wires. It appears that as much as 35 per cent rhenium may be dissolved in solid solution in molybdenum. Alloys containing 75 per cent rhenium appear to oxidize during a heating cycle of 24 hours at 1800° to 1900° C whereas those containing 35 per cent appear not to be affected at the same pressure and temperature. Equipment is being built for testing these alloys as thermocouple units.

7. Equipment

The tungsten arc furnace mentioned in previous reports is being used extensively in preparing many of the alloys now being investigated. The use of a Westinghouse rectifier type DC arc welding transformer as the power source for the furnace resulted in improved melting over that previously afforded by a motor-generator set. Construction of a second tungsten arc furnace is now underway which will incorporate several improvements including a more efficient vacuum system and provision for use of a getter to remove traces of contaminants in the furnace atmosphere. Also, several melts may be made without opening the furnace.

Apparatus for testing and calibrating vacuum gauges has recently been assembled and put into operation. The glass vacuum system is capable of calibrating gauges in the pressure range from 1 mm to 10^{-6} mm of mercury. Several vacuum gauges being used in the laboratory have been calibrated.

8. Rare Earth Metals

See page 18.

553 011

CHEMISTRY

Under the direction of F.H. Spedding, C.A. Goetz, C.V. Banks
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Physical and Inorganic Chemistry Groups1. Separation of Rare Earths by Ion Exchange1.1 The Light Rare Earths (Cerium Group)

Production Run 7 was completed during this period. Since this run marked a departure from the usual procedure, a short description of the cascade system, a logical extension of the original method of column separation, is given below.

In the cascade system, identical fractions from several identically loaded primary columns are loaded in a continuous manner on to a single secondary column. The number of secondary columns so loaded is thus the number of original fractions obtained from each primary column. These secondary columns are then eluted and appropriate fractions collected in the usual manner. The net effect of such a system is the separation which would be obtained from two separate column runs without the intermediate labor of precipitation, filtration, ignition, combination, and readsorption. The advantages of the system lie in the time and labor saved in the omission of the steps mentioned above; and in the chemicals saved by the larger amounts of eluant (ammonium citrate solution) which may be recycled and in the reduced amount of oxalic acid required in precipitating the rare earths.

In production run number 7 only 16 of the 20 six-inch columns were used in cascade, the other 4 being employed in further separations of other rare earth mixtures on hand. The column loads and the composition of the fractions employed are given in Table I.

555 012

Table I
Column Loads

Column	Composition of fraction	Load wt.
1 thru 16	Oxide from gadolinite ore having following composition: Y_2O_3 , 50%; Yb_2O_3 , 10%; Er_2O_3 , 7%; Gd_2O_3 , 10%; Dy_2O_3 , 7%; Ho_2O_3 , 1%; Tm_2O_3 , 1%; Pr_6O_{11} , 1%; Nd_2O_3 , 6%; Sm_2O_3 , 7%; oxides of terbium, lutecium and europium, less than 1% each; CeO_2 and La_2O_3 , 5%	313 g. each
17	Gd_2O_3 recovered from metal reduction processes.	296 g.
18	Concentrates containing approximately 30% Tb_6O_{11} .	334 g.
19 and 20	40.4% Gd_2O_3 ; 40.9% Nd_2O_3 ; 18.7% Sm_2O_3 , Dy_2O_3 and Pr_6O_{11}	357 g. each

As was expected, no pure material was obtained from the cascade run but many fractions were obtained in enrichments capable of yielding pure components, after one or two more column processes, of every rare earth. Enough terbium concentrate was obtained from column 18 to make a column load when added to the concentrate already on hand. It is hoped that this column run, scheduled for the near future, will yield very nearly pure terbium. From the remaining columns, 17, 19 and 20, a total of 342.17 grams of oxide was obtained analyzing 95% Gd_2O_3 , 5% Sm_2O_3 . This was delivered to the metallurgy group for subsequent reduction to metal.

The two remaining lots of crude Gd_2O_3 received from Argonne National Laboratory, were processed during this period. The analyses of these two lots, given in the previous report, are repeated in Table II.

555 013

Table II

Lot #	Wt. (g)	% Dy_2O_3	% Gd_2O_3	% Sm_2O_3	% Nd_2O_3	% Pr_6O_{11}	% Y_2O_3
294	1212.0	1.2	14.5	51.1	25.4	1.2	<10.0
295	1741.6	2.4	17.5	47.2	28.4	1.2	<10.0

After sodium amalgam extraction the following fractions were obtained.

Table III

Water phase (as oxide)

From lot #	Wt. (g)	% Dy_2O_3	% Gd_2O_3	% Sm_2O_3	% Nd_2O_3	% Pr_6O_{11}	% Y_2O_3
294	570.6	---	29.0	3.0	52.9	1.3	<4.0
295	887.5	0.8	33.0	3.9	49.8	1.2	<4.0

Table IV

Mercury phase (as oxide)

From Lot #	Wt. (g)	% Gd_2O_3	% Sm_2O_3	% Nd_2O_3
294	648.7	< 1.0	94.5	4.3
295	843.0	< 1.0	93.0	6.9

The water phases from these two lots were set aside to be loaded onto columns in production run 8.

Several samples of ytterbium-rich oxide obtained from columns 1 through 16 from run seven were combined and the ytterbium extracted with sodium amalgam; the results are summarized in Table V.

Table V
Ytterbium extraction

#	Sample	wt.(g)	% Yb ₂ O ₃	H ₂ O phase	Hg phase
1	combined from columns	63.07	>75	29.75 g 66.4% Yb ₂ O ₃	31.75 g 92.2% Yb ₂ O ₃
2	H ₂ O phase from 1	29.75	66.4	8.62 g 5.8% Yb ₂ O ₃	20.25 g 98.7% Yb ₂ O ₃
3	combined from columns	24.55	~70	6.47 g 14.65% Yb ₂ O ₃	22.38 g 92.5% Yb ₂ O ₃
4	combined from columns	35.18	~35	12.61 g 0.95% Yb ₂ O ₃	22.25 g 96.0% Yb ₂ O ₃
5	combined from columns	102.88	>80	12.31 g 28.5% Yb ₂ O ₃	89.35 g 92.2% Yb ₂ O ₃
6	combined from columns	39.37	varying see Σ -10-40	incomplete at this time	
7	H ₂ O phase from 5	12.31	28.5	8.38 g 3.0% Yb ₂ O ₃	3.20 g 89.5% Yb ₂ O ₃

1.2 Heavy Rare Earths (Yttrium Group)

Enriched samples from previous separations were reprocessed on ion-exchange columns yielding the following:

555 015

<u>Wt.</u>	<u>Nature of Sample</u>
139 grams	99.6% Er_2O_3 ; traces of other heavy rare earths
77 grams	98% Er_2O_3 ; 1% Tm_2O_3 ; 1% Ho_2O_3
73 grams	95% Er_2O_3 ; 2% Yb_2O_3 ; 3% Tm_2O_3
43 grams	88% Er_2O_3 ; 12% Ho_2O_3 ; Dy_2O_3 and Y_2O_3
66 grams	99% Yb_2O_3 ; 1% other heavy rare earths
35 grams	88% Tm_2O_3 ; 2% Er_2O_3 ; 10% Yb_2O_3
38 grams	75% Tm_2O_3 ; 25% other heavy rare earths
36 grams	90% Ho_2O_3 ; 9% Dy_2O_3 ; 1% Er_2O_3
25 grams	90% Dy_2O_3 ; 10% Ho_2O_3

Amalgam extractions are being performed on other samples obtained from the above separation. These should yield some pure Yb_2O_3 and Lu_2O_3 .

1200 grams of 90% Y_2O_3 and 10% Dy_2O_3 were eluted with 5% ammonium citrate, according to the method described in the previous report. This separation yielded 217 grams of 97% Y_2O_3 , 222 grams of 98.7% Y_2O_3 and 159 grams of 99% Y_2O_3 ; the main contaminants in these samples are Gd_2O_3 and Sm_2O_3 . Further separation of the gadolinium and samarium is to be made in order to obtain spectrographically pure yttrium.

2. Activity Coefficients of the Rare Earth Salts

The activity coefficients for the chlorides of neodymium, cerium, samarium, europium, and ytterbium have been measured utilizing concentration cells with transference. The Debye-Huckel equation was obeyed over the range studied (from 0.001 to 0.035 molal) for all of these salts. The data of Robinson and Mason, as brought up to date by Robinson and Stokes*, were fitted to the data obtained in these

*Robinson and Stokes, Trans. Faraday Soc., 45, 612, (1949).

laboratories to give activity coefficients for neodymium chloride, cerium chloride, samarium chloride and europium chloride from 0 to 2.0 molal.

3. Preparation and Properties of the Rare Earth Metals

Using the tantalum crucible technique, samples of lanthanum, praseodymium and gadolinium metal have been prepared in very high yields. Since samarium chloride is not reduced to metal by this process, residual amounts of samarium which are usually present in gadolinium salts remain in the slag in a gadolinium reduction; this effects a more complete separation of samarium from gadolinium than is possible by conventional separation methods.

The vapor pressures of lanthanum and praseodymium have been determined. Preliminary measurements have been made of the heat of solution of cerous chloride, using solution calorimeters which have been constructed during this period.

4. Hafnium Recovery

Research was continued on the preparation of pure hafnium from high hafnium strip by-products in the silica gel adsorption purification of hafnium. Conditions permitting the recovery of approximately one quarter of the hafnium in a mixture of oxides containing a hafnium/zirconium weight ratio of 1 as a hafnium product with hafnium-zirconium weight ratio of 25 and higher by means of ion-exchange resins were established. In this work (based on work reported by Gluekauf at Harwell) the metals are adsorbed from aqueous solution of the oxychlorides, the zirconium preferentially stripped with dilute sulfuric acid, and the hafnium rich residue stripped with dilute oxalic acid. Variation of stripping agents and other adsorbents are currently under study. A number of ideas for extracting zirconium and hafnium from 7N sulfuric acid solution (silica gel adsorption process strip solution) with simultaneous concentration, so as to reduce hafnium recovery to laboratory scale and at the same time recover sulfuric acid for re-use, were investigated. These methods included the use of complexing agents such as cupferron, salicylic acid, lactic acid and mandelic acid in various organic solvents.

5. Slurry Studies

Research on the problem of preparing uranium slurries suitable for use in homogeneous reactors was initiated. A preliminary theoretical analysis, coupled with experimental investigation of wetting characteristics of various uranium compounds, was made and a preliminary goal set at the preparation of an aqueous uranium oxide slurry with oxide particles having diameters of 50 μ or less. Methods for monitoring particle size in this range were already available; methods for monitoring particle size in the range 200 μ - 10 μ were developed. Procedures for attempting the preparation of uranium oxide of the desired particle size were outlined and are under investigation. These include:

- (1) Combustion of active uranium prepared by decomposition of uranium hydride.
- (2) Synthesis of volatile organo-uranium compounds, and their combustion.
- (3) Atomization of molten uranium compounds, followed by thermal decomposition.
- (4) Atomization of solutions of uranium compounds, followed by combustion or thermal decomposition of the resultant fog.

6. Corrosion Studies

A basic research program on the corrosion of metals was initiated, and a fairly extensive literature search conducted. Apparatus components and materials for investigation were ordered and will be assembled in the near future. An initial goal is the development of methods for greatly reducing the time required to estimate the probable corrosion of slowly corroding metals such as zirconium.

7. Adsorption Studies

Work on the pendent drop surface tension apparatus was continued; extensive modifications of the high vacuum system used for very high surface tension materials were made. Further modifications in the optical system are in progress.

555 018

Temperature control apparatus, sensitive to 0.01°C , was developed and adapted for low temperature gas adsorption work, together with shielding techniques to reduce radiation heat gains in the gravimetric method. A second case of inversion of adsorption in aqueous solutions (adsorption from aqueous solutions of caproic acid by carbon black) has been discovered.

8. Hydrogen Analysis in Organic Compounds

Trial was made of a method for analyzing organic compounds for hydrogen by decomposition of the compound with hot active uranium with subsequent measurement of the pressure and volume of the liberated hydrogen. Several attempts with standard compounds yielded consistently low results indicating incomplete decomposition. Further attempts with slightly different conditions will be made.

9. Reaction Mechanisms

Interpretation of the results of the ferric-stannous reaction shows that $\text{Fe}^{+3} + \text{Sn}^{+2} + 3\text{Cl}^{-} \longrightarrow$ fast reaction, $\text{Fe}^{+3} + \text{Sn}^{+2} + 4\text{Cl}^{-} \longrightarrow$ very fast reaction, $\text{Fe}^{+3} + \text{Sn}^{+2} + 5\text{Cl}^{-} \longrightarrow$ fast reaction, and $\text{Fe}^{+3} + \text{Sn}^{+2} + 6\text{Cl}^{-} \longrightarrow$ very fast reaction.

The cupric-cyanide reactor was found to involve one cupric and four cyanide ions. The slow step is $\text{Cu}(\text{CN})_4^{=2} \longrightarrow \text{Cu}(\text{CN})_3^{=} + \text{CN}^0$. The formation of the complex is rapid. No cupric ion with less than four cyanides attached gives a reaction.

Studies on the mechanism of tetraphenyl arsonium permanganate precipitation demonstrated that probably only one permanganate ion is involved in the crystal precursor. No results on the number of tetraphenyl-arsonium ions involved have been obtained as yet.

In the ceric-chloride reaction, chloride was found to be oxidized by the intermediate formation of complexes such as CeCl^{+3} , CeCl_2^{+2} , etc. Data are still insufficient to sort out the specific effects of each complex on the rate of reaction.

The titanous-perchlorate reaction was found to be first order in each Ti^{+3} and ClO_4^- and second order in H^+ . Preliminary indications are that a $TiClO_4^{+2}$ complex is involved as intermediate in this reaction.

10. Precision Calorimeters

Machining of the parts for the construction of a precision calorimeter for the range 15° to $300^\circ K$ was completed and assembly was started. Thermocouples were made and the platinum resistance thermometer received. The electrical circuits were tested and checked to be operating correctly with the exception of the interval timer which was returned to the vendor for repair. Design of a precision calorimeter for the range $1^\circ K$ to $15^\circ K$ has been continued.

11. Single Crystals of MnS

An initial effort was made to grow single crystals of MnS by a sublimation technique. In this procedure powdered MnS was heated to $1400^\circ C$ in a quartz tube under a pressure of 1 atm of H_2S . Although the contents sintered, only microscopic crystals were formed; it is hoped to continue this work at higher temperatures.

12. Hydrogen-Uranium Equilibrium

The work with the hydrogen-uranium equilibrium system is slow but progresses steadily. Pressure-composition isotherms being observed are similar to those already published. The isotherm at $350^\circ C$ has been completed. In conjunction with this isotherm work an interesting observation has been made which supercedes all other reported observations concerning the initial lag in the reaction between H_2 and uranium metal. Burke and Stanley* describe the effect of hydrogen impurities and temperature upon this lag. It has been found that scrupulous attention to keeping the uranium metal clean eliminates the incubation period of the reaction. No observable lag is evident at $250^\circ C$ even with uranium turnings, provided the metal is clean. Small amounts of impurities in the hydrogen seem to have no effect. No further study of this phenomenon is contemplated at present.

* J. Am. Chem. Soc.

13. Gas Purification

The study to date of gas purification methods has led to the conclusion that uranium turnings even at 800°C will not purify rare gases of nitrogen, carbon dioxide or methane. A single pass of the gas over a bed of hot uranium metal in a tube 1 inch in diameter by 15 inches long will reduce the residual nitrogen content in argon by about 30%; the CO_2 by about 50% and the methane by about 30%. An artificially impure gas containing about 2.5% total impurities as N_2 , O_2 , H_2O , CO_2 and CH_4 was used in these experiments. At temperatures above 500°C , O_2 and H_2O are completely taken out of the gas stream. A study of the effects of flow rates is being made at present but no conclusions are immediately forthcoming.

14. Oxygen in Metals

The problem of determining oxygen-in-metals is in its initial stages of investigation. An isotope dilution technique using O^{18} is the essential feature of the attack upon the problem. Calcium metal is the first metal being examined; it is hoped that the principles can be applied to Th and Zr.

The intermediate-mass-range mass spectrometer in this Laboratory is undergoing changes to meet the more exacting requirements of the oxygen-in-metals problem. New amplifiers with greater stability and decreased drift are being constructed. A liquid sample leak has also been added to the mass spectrometer. When all changes are complete the instrument will be capable of being used as a general purpose analytical tool in the 1-90 mass range using magnetic scanning in addition to being used as an isotope ratio machine.

Radiochemistry Groups

1. Research on Ruthenium

Research on the complexes formed between thiourea and Ru(IV) has continued. Previous indication was that two complexes of formulas RuTh and RuTh_3 were formed. Magnetic

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susceptibility measurements on the second show that it is paramagnetic and hence that the ruthenium is in the trivalent state. Ru(IV) is apparently reduced by the thiourea to Ru(III) which forms the complex. The complex does not migrate in an electric field and, consequently appears to be neutral. The formation constants for the two complexes are not large and their measurement is consequently difficult but work on this point is proceeding.

2. Zirconium Chemistry

The nature of the aqueous zirconium ion in acid solution is being studied using ion exchange on resins as a method of study. The extent of exchange or adsorption of the zirconium as a function of pH, zirconium concentration, concentration of anions etc. is under investigation. It is hoped that information on the nature of the hydrolyzed zirconium ion, the hydrogen ion concentration at which it is formed and similar items of importance in aqueous zirconium chemistry will be obtained.

3. Synchrotron-induced Activities

In connection with the program of investigation of radioactivities produced by synchrotron bombardment, methods of concentrating these activities are of considerable importance. Various recoil processes resulting from γ, n reactions have been studied. Among these are the recoil of chlorine atoms from carbon tetrachloride in which a large enrichment of the 33-minute Cl^{34} was obtained. Another system studied was phosphorous, the 2.5 minute P^{30} was enriched by recoil from triphenyl phosphate. Other systems amenable to recoil enrichment are being investigated.

In photon irradiations of tungsten by the synchrotron beam followed by chemical separation of the tantalum radioactivities, the radioactivities listed in the table have been obtained.

Isotope Produced	Process	Half-life	$E_{\text{Max.}}$ (Mev)	Relative yield compared to Cl^{11} by (γ, n) at 65 Mev.
Ta^{180}	(γ, np)	8.0h	0.6 - 0.7	3.4
Ta^{183} *	(γ, p)	6.0d	0.5 - 0.6	2.3
Ta^{185}	(γ, p)	48m	1.6 and 0.15	1.4

* Most probable assignment

The Ta^{180} isotope was identified by the use of a separated W^{182} isotope target, and the processes involved were established by their relative probabilities at 30 and 68 Mev. Energies were obtained from absorption data, and the relative yields have been given on an isotopic basis neglecting reactions involving a loss of three or more particles.

The two electron groups in Ta^{185} were apparent from the absorption curve. Coincidence counting techniques indicated that the low energy group did not consist of conversion electrons following the higher energy electrons in a time interval of less than 5 μ sec. It probably corresponds to a second beta group. A 2 - 4 minute radioactivity has also been found in W produced by the irradiation of W metal and W_2C . An attempt will be made to establish its chemical and isotopic identity.

Radioactivities have been found in the mercury and gold fractions of Tl irradiated in the synchrotron but the identification is not yet complete.

The study of the formation of Co^{58} and Co^{58m} by a (γ, n) process was continued. The Szilard-Chalmers type of decomposition of the complex salt $\text{K}_3\text{Co}(\text{C}_2\text{O}_4)_3 \cdot 3 \text{H}_2\text{O}$ has been utilized to obtain the required specific activities. Before accurate yield data can be obtained for these isomers it has been found necessary to determine precisely and minimize the thermal and photochemical decomposition of the complex in the various chemical reactions. Factors affecting the separation of Co^{++} from the complex oxalate ion have been established using Co^{60} tracer. By suitable control of pH the effective separation can be maintained at better than 95% by an ion exchange method.

A concentration of activities has been obtained using a similar technique with a salt of the platinum complex $\text{Pt}(\text{NH}_3)_4^{++}$. The chemical features of this reaction will be studied further. An attempt to utilize these techniques with $\text{Hg}\phi_2$ and ϕHgCl failed apparently because of rapid exchange in the target material.

The p^{32} radioactive standard prepared by the Bureau of Standards has been compared against Co^{60} standards calibrated by coincidence techniques in this Laboratory. Agreement of the measured disintegration rate with the calibration was within 3%.

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From an irradiation of sulfur in the synchrotron a phosphorous activity has been removed which appears to be the same as the weak, long half-life component found by others in the Laboratory to be present to a small extent in pile produced P^{32} .

4. Self-diffusion in nickel

In exploratory experiments it was found essential to test the homogeneity of nickel deposits by radioautograph techniques. Consequently preparation of a new set of samples has been started. An absorption curve of electroplated nickel deposits for the soft electrons of Ni^{63} (.06 Mev.) over deposits of the radioactive Ni have been obtained. Definite changes in intensity have followed prolonged heating but it was not possible to demonstrate that these changes were due to volume diffusion in the original experiments.

5. Photofission yields of Thorium

Peak to valley yields for the fission of thorium induced by the 65 Mev. synchrotron beam have been obtained from a comparison of the strontium to silver assays. Preliminary results show a value of about 10:1. Since Ba separations carried the Ra activities with some Pb descendents, the Ba yield could not be found by conventional fission assays. A procedure for determining zirconium and niobium fission yields in thorium has been worked out and is to be utilized in the near future.

6. Chemical Equilibrium and Kinetics

A number of features of the mechanism for the oxidation of Mn^{++} to MnO_4^- by periodic acid have been worked out which explain the observed kinetics. These results have indicated that an iodate complex of Mn^{III} is an important intermediate, and the possibility of demonstrating and actually measuring the disproportionation constant for this complex is being investigated.

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7. Service Work

The group continues to prepare and mount samples of various kinds for the experimental physics people, to make separations of radioactive material and to develop such separation methods. The latter frequently involves minor research projects, since published separation methods are often designed for different conditions and adaptation of these methods is not always easy.

Spectrochemistry Group

1. Spectroscopic Methods for the Analysis of the Rare Earths

The chemical properties of the rare earths are so strikingly similar that mixtures of these elements defy analysis by the usual methods of analytical chemistry. Although most of the rare earths can be determined by means of the characteristic sharp absorption bands of aqueous solutions of their salts, these bands lack sufficient intensity for measuring rare earth concentrations below several per cent. To provide a more sensitive test, spectroscopic methods have recently been completed for the quantitative determination of those rare earths commonly associated with purified lanthanum, cerium, praseodymium, and neodymium, either during the large scale fractionations being conducted in these Laboratories, or in the separated "pure" rare earths. Experimental work is continuing to extend these methods to the remainder of the rare earth series.

Research on the possible analytical applications of diatomic rare earth oxide band emission spectra and fluorescent spectra of aqueous solutions of the rare earths is continuing.

2. Spectrographic Determination of Residual Impurities in Zirconium Metal

The development of a spectrographic method for the simultaneous quantitative determination of magnesium, aluminum, calcium, titanium, iron, nickel, copper and

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hafnium is practically completed. This method will be described in more detail in the next quarterly report.

3. Miscellaneous Research

Other spectrochemical research problems currently under investigation are:

(a) Direct spectral line intensity measurements using photomultiplier tubes and associated electronic circuits.

(b) Infrared study of hydrogen bonding in dioxime reagents and in their Ni and Pd complexes.

(c) Application of X-ray fluorescence spectra to various analytical problems.

4. Service Analyses

A total of 1766 service spectrographic analyses were completed during this period. The samples consisted primarily of rare earths and zirconium.

5. Zirconium-Hafnium

A paper^{*} entitled "Quantitative Spectrographic Analysis of Zirconium-Hafnium Mixtures" was presented at the annual meeting of the Optical Society of America, Cleveland. An abstract of this paper follows:

The great similarity in the chemical properties of zirconium and hafnium precludes the use of chemical methods for analysing mixtures of these elements. The active interest of the AEC in hafnium-free zirconium and zirconium-hafnium separations created the need for an analytical method. This paper describes a spectrographic method for the analysis of hafnium-zirconium mixtures covering the complete hafnium/zirconium concentration ratio range from 0.001 to 100. The samples are excited as conducting pellets prepared from mixtures of the combined oxides with powdered flake graphite. An overdamped condenser discharge is used for excitation. Precision determinations showed a standard deviation of 1.5 to 2.0 per cent.

^{*}J. Opt. Soc. Am., 40, 742 (1950).

X-Ray Chemistry

1. Thorium Metal Compounds-Transition

During the period of this report considerable effort has been devoted to determining the structure of thorium-transition metal alloys. We have found that thorium, like uranium, forms no intermetallic compounds with chromium; compounds start with manganese and increase in number to nickel, and thereupon fall off. The structure of a considerable number of thorium-transition metal compounds have been determined.

2. Zirconium Alloy Systems

An investigation into the structure of zirconium alloy systems has been initiated. CuZr_2 has the CaC_2 structure, and ZrFe_2 is hexagonal with the Cl_5 type structure.

3. Electron-Deficient Compounds

The structure of dimethylberyllium, recently published indicated that beryllium forms four bonds, even in dimethylberyllium. Beryllium chloride was examined to see if an analogous type of bonding occurred in this compound.

The chloride has a structure similar to that of dimethylberyllium, with chains

angles show that when chlorine forms bridge bonds, two electron pairs are used in the bridge, the first time this has been demonstrated.

Problems in this series are of interest in understanding chemical valence and the theory of metals. Apparently delocalized bonds in metals can be related to delocalized bonds in simpler systems.

4. Complexes of Aromatic Molecules

The structure of the silver perchlorate benzene complex has been completed. Other complexes of this type are under study to see if the length of the silver-to-aromatic

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bond is related to the electron-donor ability of the aromatic molecule. This work is of interest in determining the nature of certain Lewis acid-base reactions, and may have a bearing on reaction mechanisms.

Analytical Chemistry Group

1. Zirconium

The dry hydrochloric acid-sublimation method of determining zirconium dioxide in zirconium metal has been studied. The presence of carbon has been found to give high results, presumably because zirconium carbide is not attacked by dry hydrochloric acid and remains with the zirconium dioxide residue. Colorimetric methods of determining zirconium are being studied in order to improve the above method.

The following procedure has been found to be satisfactory for determining tin in zirconium-tin alloys: dissolve in hydrofluoric acid-sulfuric acid mixture, fume, reduce tin in carbon dioxide atmosphere with metallic lead, titrate at 10°C with sulfatoceric acid using ferroin as the indicator. ✓

Methods are being developed for analyzing zirconium-antimony alloys and zirconium-columbium mixtures.

2. Miscellaneous Investigations

A magnetic susceptibility apparatus is now in operation and is being used to develop new analytical methods and to study the structure of compounds of interest to the analytical chemist. Magnetic measurements are also being made for other research groups.

Infrared studies of the nickel and palladium inner-complex compounds with the vic-dioximes have yielded convincing evidence that the remaining oxime hydrogens are hydrogen bonded.

The reaction between iron (III) and 1,2-cyclohexanedionedioxime in acid solution is being studied.

The experimental work on the method of determining iron and aluminum on the same sample of calcium metal has been completed.

A combustion method for determining carbon in calcium metal is being developed. Highly ignited zirconia seems to make a satisfactory lining for the combustion boats.

Several improvements over the previously reported method (ISC-26) for preparing 1-(o-arsonophenylazo)-2-naphthol-3,6-disulfonic acid ("Thoron") have been made.

Work is continuing on the spectrophotometric determination of thorium with "Thoron".

Methods are being developed for determining the usual impurities (Fe, Mn, N, etc.) in vanadium metal and vanadium compounds.

3. Service Analyses

A total of 1800 service analyses were made during the period of this report. The service analyses included: Calcium metal for iron, carbon, nitrogen, and manganese; thorium metal for iron, manganese, nitrogen and carbon; zirconium metal for iron, zirconium oxide, carbon, zinc and nitrogen; zirconium fluoride for nitrogen, iron, manganese, chlorine, fluorine, zirconium and water; tin-zirconium alloys for tin, carbon and zirconium; zirconium slag for magnesium, calcium, zinc and zirconium; rare earth mixtures for individual rare earths; silica gel for water; thorium fluoride for fluorine; stabilized zirconia for iron and calcium; zirconium-bismuth alloys for bismuth; sodium-tungsten bronze for sodium; lanthanum metal for free metal content; vanadium metal for free metal content; europium chloride for chlorine; thorium-rare earth mixtures for thorium and total rare earth oxides; copper-zirconium alloys for copper and nitrogen; monazite sands for uranium and silica; zirconium-uranium alloys for carbon, zirconium, iron, nitrogen and uranium; zirconium dioxide for carbon; zirconium tetrachloride for zirconium and chlorine; zirconium nitride for nitrogen; hydrogen peroxide for nitrogen; uranium metal and uranium compounds for uranium (SF accountability); thorium metal and thorium compounds for thorium (SF accountability).

PHYSICS

Under the direction of G. W. Fox, W. C. Caldwell,
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J. M. Keller, J. K. Knipp, L. J. Laslett,
S. Legvold, G. H. Miller and
D. J. Zaffarano

Theoretical Physics1. Ionization Statistics

During the period of this report the problem of ionization statistics has been studied theoretically. The probability that a particle of definite energy will produce a certain amount of excitation or ionization is described by the Smoluchowski-Kolmogoroff equation for the composition of probabilities. The fundamental process is the single atomic or molecular collision, for which there are certain probabilities of excitation, ionization, and elastic energy transfer. First and second moments as calculated from the basic equations give integral equations for the average amount of excitation, average ionization, and mean square fluctuations for the excitation and ionization. These are of the form of continuity equations in which the probabilities for the fundamental process enter as coefficients.

This approach is being applied first to the excitation and ionization by electrons stopped in a monatomic gas. The ionization by heavy particles stopped in a gas is also being studied, as well as other applications of considerable practical interest.

2. Specific Heat of Ce

Study of the specific heat of cerium, discussed in the previous report, was continued. The potential in the neighborhood of an ion in a crystal can be expanded into spherical harmonics according to its angular dependence. The magnitude of the Y_2^0 part of the potential in a hexagonal close packed crystal is under investigation. A calculation, in which the contributions of successive neighbors were computed and added, failed to converge with sufficient speed

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to put reasonable bounds on the magnitude of the potential. Upon recomputation, using a modification of Madelung's calculation of energy in a crystal, a result was obtained that was definite, but fell outside of the uncertainty of the successive neighbor calculation. A Madelung-type calculation on a cubic crystal made it clear that the method - at least in the form used here - would converge to the correct value only for an ionic crystal.

3. Multiple Scattering in a Cloud Chamber

The current problem connected with this program is the distribution in curvature, in a magnetic field, of tracks of large angle of arc. The best work available* seems to be based on approximations valid only for tracks of small angle of arc. A differential equation, free of these objections, was written but has not been solved.

4. 2-gamma or 2-electron Decay

Dr. Elliot of Chalk River suggested the possibility that low energy electrons found by E. N. Jensen of this Laboratory in connection with the decay of P^{32} arose from an excited state of the daughter nucleus S^{32} . If both ground and excited states had zero angular momentum but opposite parity, the excited state could decay only by emission of two quanta, two atomic electrons (internal conversion) or one electron and one quantum. According to the calculations of Sachs,** the emission of two quanta is so much more probable than the emission of any electrons that this does not seem a possible explanation for Jensen's low energy electrons.

*Scott, Phys. Rev. 76, 212 (1949).

**Phys. Rev. 57, 194 (1940).

Experimental Physics

1. Beta and Gamma Ray Spectrometry

1.1 Beta-Spectrum of P^{32}

Work is continuing on the beta-spectrum of P^{32} in order to identify the low energy beta group which has been observed. The samples used previously were prepared by an $S^{32}(n,p)P^{32}$ reaction. A sample of P^{32} , prepared from P_2O_5 irradiated with neutrons, was obtained from Oak Ridge. This sample was followed in the spectrometer, but its specific activity was too low to obtain definitive data. The low energy group was present in the sample, but it could not be definitely established that its half-life was the same as that observed in the sulfur irradiated samples.

1.2 Radiations from Te^{125}

The internal conversion spectrum of Te^{125} has been determined with the thin lens spectrometer. A search for gamma-rays will be made at a later date when a stronger source is available.

1.3 Radiations from Hf^{181}

The beta-spectrum of Hf^{181} has been redetermined; the gamma-ray spectrum had been determined previously. These two spectra will be determined again at a later date to check on the relative intensities of the various gamma-ray lines. Recent information indicates that one of the gamma-rays attributed to Hf^{181} may be due to Hf^{175} .

1.4 Radiations from Sb^{124}

The beta-ray spectrum of Sb^{124} has been determined. This spectrum will be analyzed for various components. The gamma-ray spectrum, using a uranium radiator, is being determined. The low energy end of the gamma-ray spectrum has also been determined with a silver radiator. One additional gamma-ray has been observed that has not been reported in the literature.

1.5 Double Lens Beta Ray Spectrometer

The main components of the new double lens spectrometer have been completed, including the winding of the coils. The

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main task now is to complete construction of minor parts of the spectrometer, assemble the spectrometer and make adjustments for optimum operating conditions.

1.6 Transmission Characteristics of Thin Foils

Progress has been made on the apparatus for determination of the electron transmission characteristics of thin foils, such as those used for low energy Geiger counters. The vacuum chamber, electron gun, foil holder, and high voltage supply should be completed in the near future.

2. Application of Scintillation Counters to β -Ray Spectrometry

2.1 Analysis of Instrumental Performance

As indicated in the preceding summary report, attention has been devoted to the means whereby an approximate correction can be made for the imperfect instrumental resolution of the scintillation spectrometer when applied to continuous beta distributions. Further work on the examination of the transmission function of the instrument is in progress, facilitated by the use of mono-kinetic electrons from $\text{Pt}^{196\text{m}}$ prepared by synchrotron-irradiation of a platinum complex. The line-shapes obtained under various conditions with such mono-kinetic sources can serve as a guide to improvement of the instrument.

2.2 The Low-Energy Beta-Spectrum of Rb^{86}

The low-energy component in the beta-spectrum of Rb^{86} was examined with a coincidence scintillation spectrometer and led to an upper-limit of approximately 0.67 Mev, with limits of error estimated as 15 per cent. A measurement of the beta-ray from a (gamma-free) Sr^{90} source resulted, under similar geometrical conditions, in an end-point of 0.562 Mev, in excellent agreement with previously reported magnetic-lens spectrometer values. The shapes of the Sr^{90} and the lower-energy Rb^{86} beta-spectra appeared closely similar, but it is believed that the precision of this work does not permit a definitive comparison of spectral form.

2.3 The Beta-Spectra of Ag^{112} and Ag^{113}

The radioactivities of Ag^{112} and Ag^{113} , produced by synchrotron irradiation of cadmium, were examined with the

scintillation spectrometer and led to upper-limits for the beta-spectra of 4.2 and 2.0 Mev, with half-lives of approximately 3.2 and 5.3 hr., respectively. From the observed yields of these activities, and on the supposition that they are essentially produced solely by (γ ,p) reactions, the cross-section for the $\text{Cd}^{113}(\gamma, p) \text{Ag}^{112}$ process was estimated as about 1.5 times that for the $\text{Cd}^{114}(\gamma, p) \text{Ag}^{113}$ reaction. The Ag^{111} , Ag^{112} , and Ag^{113} activities not only were found to result from synchrotron irradiation of cadmium but also, in lower intensity, from the irradiation of indium.

2.4 Other Applications of Scintillation Spectrometry

Use of scintillation spectrometry is considered particularly suitable for the investigation of synchrotron-produced radionuclides and further work of this type is in progress, together with continued efforts to improve the resolution and hence the usefulness of the equipment. Apparatus is being assembled to permit, with the aid of a high-speed pneumatic tube apparatus, the examination of short-lived radioactivities produced by synchrotron irradiation.

3. Fission Fragment Experiments

3.1 Ionization Defect Experiment

Work has started on the ionization defect experiment outlined in the previous report. In this experiment the pulse height distribution due to fission fragments in an ionization chamber is measured for a light gas and for a heavy gas. These heights are normalized by comparing with the pulses of alphas of known energy in the two gases. The ionization defect should be different in the two gases, and therefore the distributions should be different when normalized in each case.

The electronic equipment for this experiment has been carefully checked and some revisions made. The present scheme uses a Los Alamos Model 100 amplifier followed by a Model 1000 to drive the oscilloscope. A d.c. power supply has been constructed to supply the filaments of the first stages of the model 100, replacing the storage batteries. A Bell-Jordan amplifier has been used to trigger the oscilloscope sweep. The chamber itself has been revised by increasing the plate separation. A frisch grid has been

constructed and placed between the source and collector. This grid removes variations in pulse height due to obliquity of the particle track, since the pulse arises only from the passage of electrons between the grid and collector. Thus if the ionizing particle is stopped in the space between the source and grid, all of the electrons travel the complete distance from grid to collector. A rather novel feature has been made possible by the addition of the grid. In addition to the pulse from the collector, the source electrode can also be made to give a pulse. The pulse from the source precedes that from the collector by several microseconds so that it may be used to trigger the sweep before the pulse which is of interest is started. A collimator has been constructed and placed in front of the source. The purpose of the collimator is to remove particles which would go outside of the region in which the field is constant and also particles which have lost too much energy in the source due to their oblique path.

Thus far, some pictures have been taken of the alpha particles coming from the uranium source within the chamber. There are mainly two energies present, namely about 4.2 and 4.8 Mev. These energies have been resolved quite well in argon and the ratio of the heights is in very good agreement with the energy ratio. A few fission pictures have been taken, and although the pulses appear to be quite satisfactory, the statistics are not yet good enough to draw any conclusions.

Work is continuing on the construction of a proportional counter and a magnetic analyzer for use in fission fragment experiments.

4. Scintillation Pulses from Slow Positive Ions

An investigation of the pulse shapes from various phosphors due to slow ion bombardment has been undertaken. The equipment required for this experiment in addition to a source of ions, consists of various types of phosphors, a photomultiplier, a fast sweep, and a fast amplifier. The amplifier has been constructed and the sweep is now being built.

5. Diamond Counters

The ultraviolet and infrared absorption spectra of more than thirty counting diamonds have been taken. On the basis

of the results it appears that the ultraviolet absorption spectra is not a useful criterion in selecting counting diamonds and the absence of large absorption peaks in the infrared is a necessary but not a sufficient condition for a diamond to be a good counter.

Transmission photographs of many of these diamonds using ultraviolet light of 2600 Å show that most diamonds are mixtures of type I, which is opaque to this radiation, and of type II, which is transparent to this radiation. Hence it is not surprising that the ultraviolet absorption criteria for classifying diamonds would not be useful in selecting counting diamonds even though type II diamonds were superior counters. The classification as type I or type II gives no information about some trapping levels which can reduce the counting efficiency to zero.

It is now believed that there are at least four levels of shallow traps for electrons at 0.06, 0.15, 0.30, and 0.40 ev below the conduction band and one level of deep traps at 0.9 ev below the conduction band. One must have information about the density of all these traps in order to predict the counting characteristics of a diamond. Type II diamonds usually have few traps at 0.15 ev but the other traps may be present. Infrared absorption spectra can indicate the density of the shallow traps only.

Using Co^{60} gamma source the effect of temperature upon the counting rate has been observed for several diamonds. The counting rate is unchanged as the temperature is decreased from 25°C to -78°C; the counting rate is decreased to almost zero as the temperature is increased from 25°C to 150°C.

Using a Po-Be neutron source emitting 10^4 neutrons per second it has not been possible to distinguish between counts caused by knocked-on atoms and counts caused by gamma rays from neutron capture in the surrounding metal. It may be possible to make this distinction with the newly-obtained source which emits 10^6 neutrons per second.

A preamplifier for use with a chain of six wideband amplifiers has been designed and constructed. When the sweep circuit is completed it is hoped to be able to measure the rise time of the pulse from a diamond counter.

6. Piezoelectricity at Low Temperatures

Pitt and McKinley* reported a sharp decrease in the piezoelectric response of quartz at temperatures below 6°K. Their experiments were repeated in this Laboratory with some additional precautions and have failed to find any effect at 4°K. The experiment will be repeated with a different crystal and if no effect is observed the project will be dropped.

7. Sodium Tungsten Bronze (Na_xWO_3)

The first part of this project has been successfully completed. Measurements of conductivity and temperature coefficient of conductivity show that the crystals exhibit a conduction behavior similar to that of metals in contrast to that of semiconductors or ionic crystals.

The Hall coefficient of sodium tungsten bronze at 30°C has been determined. The temperature coefficient of the Hall coefficient in the range -80°C to 30°C is small, decreasing in absolute magnitude about 0.2 per cent per degree centigrade. The negative sign for the Hall coefficient indicates the conduction is by electrons, not by holes. Owing to the small value of the Hall voltage when using single crystals numerous spurious effects are possible. Confidence is gained in the alternating current method used here, however, from measurements of the Hall coefficients of copper and magnesium which agree well with previously published results.

From the value of Hall coefficient the electron density is calculated to be 1.18×10^{22} electrons per cubic centimeter. From a quantitative chemical determination, the sodium density is found to be 1.20×10^{22} sodium atoms per cubic centimeter--corresponding to $x = 0.68$ in the formula Na_xWO_3 . Hence it may be concluded that each sodium atom added to the crystal WO_3 donates its valence electron to the conduction band.

Having experimental values for the conductivity and Hall coefficient, other electronic properties, including mobility, mean free path and thermal velocity of electrons, magnetic susceptibility, thermal conductivity and electronic specific heat can be calculated.

* Can. Journ. Res. 14, Sec. A-B (1936).

8. Work with 12-inch Cloud-Chamber

8.1 Examination of Synchrotron Radiation Spectrum

As indicated in the preceding summary report, the 12-inch cloud-chamber has been applied to check, through the measurement of electron pairs, the spectral distribution of the X-rays produced by the synchrotron. Pairs produced in the chamber gas (air and alcohol) have appeared preferable for this purpose to those produced in a foil introduced near the edge of the chamber. At the present time a sufficient number of stereoscopic photographs of suitable tracks are believed to have been taken to permit the measurement of close to one thousand electron pairs; the measurements are now in progress.

In the course of the work described above, occasional tracks of pairs produced in the field of an electron (triplets) have been observed. Nuclear disintegrations, in which one or more heavy-charged particles are emitted, have also been seen. As data of these types accumulate a statistical analysis of the phenomena will be of interest.

Consideration has been given to the possible applicability of the cloud-chamber to a study of a synchrotron-induced radioactivity in argon. The character of the supposed activity is currently being studied, by way of preparation, with a Geiger-Mueller counter situated near the synchrotron and to which the argon may be quickly transferred following an irradiation.

9. Study of Nuclear Processes with Photographic Emulsions

In addition to work on the angular distribution of fission-fragments from natural uranium subjected to synchrotron irradiation, preparations are continuing for an investigation of photodisintegration of the deuteron. Some steps have also been taken to investigate the applicability of nuclear photographic emulsions to the study of certain other X-ray induced nuclear processes in which charged particles are emitted.

Some interest has been devoted to the techniques involved in the use of electron-sensitive emulsions to record the tracks of beta-particles. It is felt that this technique may be of value in special problems where the intensity

or questions concerning correlation make the continuously sensitive feature of the photographic medium particularly attractive.

10. Synchrotron Research and Development*

During the period covered by the present report, the 70 Mev synchrotron has been operative and at the disposal of the Ames Laboratory. A number of irradiations have been made for members of the Radiochemistry Groups. Irradiation of a germanium sample is in progress to permit a determination of the possible effect of protracted synchrotron irradiation upon the semi-conductive properties of this material.

In addition to the exploratory radiographical work, referred to above, consideration has been given to the applicability of the synchrotron as a service tool in certain problems of activation analysis.

A device for the alternative radiation of two samples in the synchrotron beam has been constructed. Use of this apparatus for the measurement of excitation functions is planned for the near future.

Some success was obtained in an effort to develop a technique for the radiography of thorium billets three-inches (and perhaps, eventually, larger) in diameter, using the synchrotron.

A replacement oscillator, using a pair of 24-G tubes, has been put into operation and a new oscillator, employing two UT-127A's completed. An extended program to test the performance of the beam-monitoring ionization chamber has been in progress and, in particular, reasonably good linearity found to exist with the chamber in its present form when checked by activation of copper samples at various beam intensities.

*The 70 Mev synchrotron was erected with College funds as a facility of the Iowa State College Institute for Atomic Research. The synchrotron research program is currently supported in part by A.E.C. funds.

The use of a recording meter in connection with the ionization chamber has been found of considerable value for monitoring irradiations and it is expected that the value of the equipment will be further enhanced by the incorporation of a standard cell for definitively checking the amplification.

The construction of gun structures and the assembly of ceramic sections for the synchrotron vacuum chamber is continuing in an encouraging manner. Sections of fused silica have been acquired to serve as the R. F. cavity and at present are being cut, coated, and tuned for operation at the correct frequency.

11. Scintillation Spectrometer

For the determination of nuclear energy levels in nuclides formed by X-ray irradiation with the synchrotron, it is desirable to use devices which yield the spectra of the radiations emitted. For this purpose, scintillation spectrometer and proportional counter techniques are being developed. To date, the scintillation spectrometer has been used to investigate the beta-spectra of strontium-yttrium 90, iron 53, and other isotopes. A photographic recording technique is being employed which will permit the observation of spectra from activities with half lives shorter than 1 minute. The large proportional counter has been calibrated with internal conversion beta-rays from tellurium 125 and cesium 137.

ENGINEERING

Under the direction of T. K. FitzPatrick,
G. L. Bridger, G. H. Beyer, W. R. Millard,
W. E. Hauth and G. Murphy

Chemical Engineering Group

1. Production of Thorium from Monazite (G. Bridger, M. Whatley
K. C. Shaw, J. H. Bochinski)

Additional fractional precipitation studies were made of solutions prepared by sulfuric acid digestion of monazite. Soda ash, caustic soda, and ammonium hydroxide were used as precipitants; it was tentatively decided that soda ash is the best precipitant on the basis of its lower cost and ease of handling.

It was found possible to extract thorium nitrate with tributyl phosphate directly from the sulfuric acid-monazite solution if sufficient strong nitric acid is added to the solution before extraction, thus eliminating the need for fractional precipitation. However, the large amount of nitric acid required would greatly increase the required size of extraction and acid recovery equipment. Further studies are being carried out to determine the practicability of this scheme.

A summary report (ISC-134) was prepared describing the proposed process for production of pure thorium and uranium nitrates from monazite.

2. Production of Zirconium by Adsorption (G. H. Beyer, R. H. Giffen, J. B. West, N. Barson, R. B. Edwards)

The removal of used silica gel from the continuous column through an opening at the bottom was complicated by the removal of comparatively large amounts of feed solution at the same time. This condition made accurate control of the net feed upward through the column difficult. By having the gel leaving the column displace its own volume of feed, it seems likely that much better control can be achieved. Current experimentation is centered on having the gel leaving the column fall, not into the room, but into a

reservoir filled with feed. In this way the total liquid feed passing upward through the column can be accurately controlled.

In addition to removing the gel conveniently, it is, of course, highly desirable to load the gel with as much hafnium as possible. It is proposed to run the column both with and without a slowly-moving stirrer which gently turns over the gel particles without displacing them vertically to any appreciable extent. If the stirring action produces an attractive improvement in the degree of loading of the gel, its use may be justifiable.

3. Production of Hafnium by Adsorption (G. H. Beyer, A. Jacobs, R. Masteller)

The differential stripping of the used gel from the bottom of the zirconium column allows concentration to 50% Hf in one process, compared to 2% concentration in the feed and 15% Hf on the used gel. Whether it is possible to produce highly enriched Hf of 80-90% is being tested by successive re-adsorptions of hafnium concentrates on new gel, followed by differential stripping.

4. Production of Zirconium by Solvent Extraction (G. H. Beyer, W. R. Millard, H. Peterson, R. P. Cox)

Since the solvent butyl phosphate is preferential for zirconium, the purification of zirconium is being experimentally investigated in a Scheibel column of 14 stages. Results to date show that specification zirconium can perhaps be more conveniently achieved by solvent extraction. Present equipment is vulnerable to corrosion by chloride ion, so that conversion of the raw chloride to nitrate is necessary before operating the column. It is planned to put in operation a small drum filter unit for this purpose. With appreciable quantities of nitrate available it is planned to determine the optimum flow rates of solvent, feed, and scrub solution (water).

Use of the solvent extraction method for large scale operation depends heavily on efficient solvent recovery, and it is proposed to also strip the solvent of its zirconium in a Scheibel column.

555 042

The anticipated arrival of a second Scheibel column should permit investigation of systems containing chloride ion, to see if these systems give a markedly improved separation factor. Using the chloride directly would eliminate the conversion to nitrate, but greatly increases problems of materials of construction.

5. Recovery of Spent Gel and Adsorbed Zirconium from Zirconium Production (W. R. Millard, R. H. Maitland, R. P. Cox)

Batch tests show that the spent silica gel from the process for the adsorption separation of hafnium from zirconium may be stripped of Hf and Zr compounds, reactivated, and reused in the separation process with apparently the same activity as new gel. No continuous column runs have been made on reactivated gel at this time.

The spent gel may be stripped completely of adsorbed metals using 6-7 N H_2SO_4 , or differentially stripped using first a 1.8-2.0 N H_2SO_4 strip. The HCl-methanol strip will remove up to 97 per cent of the adsorbed Zr with a Hf/Zr ratio approximately the same or slightly higher than the initial feed solution, but the resulting solution is only about one per cent ZrCl_4 as compared with 20 per cent for the feed solution. The excess HCl may be neutralized with NH_3 and the solution built up to feed concentration with additional ZrCl_4 . Batch tests using feed made up by this procedure behaved essentially the same as the present feed solutions. A number of aqueous acids have been investigated as possible stripping agents, but none of these has indicated any preference for either Zr or Hf. With differential stripping, the 7N H_2SO_4 strip will give a hafnium concentrate containing up to 50 per cent HfO_2 . This may be a satisfactory feed material for Hf production.

Zirconium losses by adsorption on the silica gel are somewhat higher than those originally anticipated, probably running as high as 40 per cent of the Zr fed to the columns. This loss would seem to indicate the desirability of a recovery process. Preliminary cost estimates for the differential stripping procedure show that the chemical cost for recovery of the Zr and silica gel are approximately equal to the value of the Zr and gel lost without recovery. Operating costs and amortization would, of course, increase the stripping cost to the point where recovery would be uneconomical at the present cost of ZrCl_4 . Estimates indicate, however,

that a 7N H_2SO_4 strip to recover the spent silica gel would probably be economically feasible, even though no Zr recovery is obtained. Careful cost studies of the various alternatives are being made.

6. Properties of Slurries (W. R. Millard, F. M. Jacobsen)

Work has been started on determining the properties of aqueous fuel slurries for proposed slurry reactor experiments. The first phase of this research will deal with the stability of uranium oxide slurries of varying oxide particle size. A method of preparing approximately 1μ U_3O_8 particles has been furnished by one of the Physical and Inorganic Chemistry Groups, and sedimentation rates for slurries made up from this material will be compared with those containing larger oxide particles. Particular emphasis will be placed on sedimentation rates at reactor temperatures (about $250^\circ C$) where the slurry viscosity will be small and the sedimentation rates correspondingly high. Stability tests will be performed on smaller particles as methods for their preparation are developed.

Further work planned under this project will include:

1. Study of possible particle flocculation or agglomeration.
2. Fluid dynamic properties of slurries
 - (a) Power requirements for pumping slurry
 - (b) Effect of flow rate on sedimentation
 - (c) Variation of apparent viscosity with solid concentration and particle size
3. Corrosion and abrasion properties of slurries
4. Preparation of fuel slurry on semi-plant scale
 - (a) Preparation of solid component
 - (b) Reduction of agglomerates and dispersion of solid
5. Heat transfer coefficients for forced convection for the fuel slurry.

Ceramic Engineering Group

Design of the apparatus for the determination of thermal conductivities at high temperatures was completed, and construction of the equipment was begun.

A preliminary investigation of a method to correlate rupture of ceramic materials with applied stresses was initiated. The stresses are to be applied in combination, e.g. bending and torsion. Fundamental strength data concerning these materials, obtained in this manner, will permit a more accurate evaluation of their properties and uses.

Equipment is being constructed which will give this group a greater number of methods for fabrication of refractory crucibles and shapes. Included in this will be:

1. hydrostatic pressing equipment
2. dry-pressing molds
3. hot-pressing equipment.

Nuclear Engineering Group

1. Reactor Materials

The tabulation of properties of materials of potential importance in reactor construction has been continued. Nuclear, thermal and mechanical properties as reported in the literature are being included.

2. Resistance of Ceramic Materials to Thermal Shock

The desirability of investigating a basic approach to the evaluation of the resistance of ceramic materials to thermal shock has been suggested to the Ceramic Engineering Group. In evaluating the resistance of metals and certain other materials to failure under biaxial stresses a general "law" of failure may be developed by loading the materials, using various ratios of principal stresses, to failure. The ratios of the two critical stresses to the strengths in

555-645

axial loading are plotted as x and y coordinates to give a general relationship which may be compared with the conventional "theories" of failure, such as the maximum normal stress theory, the maximum normal strain theory, the maximum shearing stress theory, the strain-energy theory and the Hencky-von Mises theory.* Once the failure curve, or law, is established, the critical loads may be evaluated for a wide variety of shapes by conventional calculations. It would be expected that the failure curve for a given ceramic material would be temperature sensitive. A test program has been outlined, and test specimens and loading device designed for the Ceramic Engineering Group.

It is expected that the tests will be conducted by the Ceramic Engineering Group, but the basic analysis of results will be conducted by Nuclear Engineering. To the latter end, equations have been developed for the evaluation of stresses and strains in specimens under thermal loading.

3. Reactor Slurry

For the present, work on two phases of the investigation of reactor slurries, namely pumping characteristics and heat transfer properties, are under consideration. The development of engineering equipment, including the reactor shell, heat exchanger, pumps and valves, will follow later after the properties of the slurry are known.

A literature survey was made on the subject of slurries and experiments planned to obtain information of value for the HRE. Because of the complexity of the circulation within the proposed sphere, a glass model was constructed to aid in establishing critical areas within the sphere. To date, only qualitative studies have been made, using (1) sand slurries of different concentrations and different particle sizes, and (2) an Fe_2O_3 slurry. All tests have been conducted with the top of the system open to the atmosphere.

The following observations have been made:

1. The critical region appears to be the entire inside surface of the sphere, within the boundary layer,

*
Ref: Murphy, Glenn "Advanced Mechanics of Materials"
McGraw-Hill, New York.

where velocities are low, becoming zero at the wall. Concentration of particles occurs on the walls and, within the limits investigated, increases with decrease in particle size.

2. If particles are sufficiently large to extend through the boundary layer, segregation does not occur.
3. Consideration should be given to the modification of the shape to a prolate spheroid to eliminate the tendency for sedimentation in regions where the slope of the inner surface is less than the angle of repose of the sediment.
4. Precession of the central vortex occurs, and is more pronounced in amplitude when inhalation does not occur.

Modifications of the equipment are being made to permit taking observations of rates of flow, and loss in head. Analyses have been made to assist in utilization of the results for predicting the behavior of the prototype.

Architectural Engineering Group

Further design studies were carried on for a remote-control device to handle radioactive samples for balance weighing. Description of operational necessities were explained in a previous report (ISC-113).

Design of Slit #2 in the control mechanism for the multiplier phototube to be used in connection with spectrographic work, has been completed and detail drawings have been finished. Housing details have been started and are nearly complete.

Assembly drawings have been completed on the device which will exhaust the toxic fumes from the primary area of an arc emitter. Details have been completed, including method of housing.

The second phase of the ampule cutter mechanism was fabricated and tested. Corrections were made and final tests showed operation to be completely successful.

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APPENDIX I: LIST OF REPORTS FROM THE AMES LABORATORY

1. Reports for Cooperating Laboratories

- ISC-91 "Synthesis of New Organic Analytical Reagents" by Roy Henry Kinsey (Thesis). (UNCLASSIFIED).
- ISC-94 "Preparation of Zirconium Metal" by Kenneth Albert Walsh (Thesis). (SECRET).
- ISC-95 "Radioactivities Produced in Europium and Gadolinium" Richard Earl Hein. (Thesis). (UNCLASSIFIED).
- ISC-97 "The Use of Ammonium Bifluoride in the Preparation of Fluorides from Oxides" by John Long. (Thesis). (SECRET).
- ISC-98 "On the Ionization Yields of Fission Fragments" by R. B. Leachman. (UNCLASSIFIED).
- ISC-103 "Thorium-Carbon System" by Premo Chiotti (Thesis). (SECRET).
- ISC-104 "Progress Report in Physics, October 1, 1949 to June 30, 1950" by G. W. Fox et. al. (UNCLASSIFIED).
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- ISC-124 "A Polarographic Investigation of Iron-Sulfosalicylate Complex Ions" by Charles V. Banks and James H. Patterson. (UNCLASSIFIED).
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555 048

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555 048

- ISC-130 "Quarterly Summary Research Report, July 1 to September 30, 1950." by W. E. Dreeszen. (SECRET).
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555 050