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ISC-69
CHEMISTRY

UNITED STATES ATOMIC ENERGY COMMISSION

QUARTERLY SUMMARY RESEARCH REPORT

For April, May, and June, 1949

Photostat Price \$ 6.30

Microfilm Price \$ 3.00

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

by

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November 28, 1949

Iowa State College

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For The Atomic Energy Commission

J. S. Canale

Chief Declassification Branch *Lu*

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METALLURGY AND CHEMICAL ENGINEERING

Under the direction of H. A. Wilhelm, B. A. Rogers and G. L. Bridger

1. Thorium

Production of thorium ingots has continued as a routine operation with virtually no change in procedure since the previous report (ISC-56). Present needs for the metal are nearly filled.

Several procedures based on conductometric analysis have been tried in an investigation of quick methods of analysis of liquids in the different steps of the purification of thorium nitrate. The results have not been satisfactory as the content of thorium found conductometrically has been from 20 to 90 per cent higher than the values given by the analytical laboratory. This study is being continued.

Microscopic evidence for mutual solid solubility of the monocarbide and dicarbide of thorium at high temperature leads to the supposition that a high temperature form of the dicarbide exists which may be isomorphous with the monocarbide. However, extremely rapid quenching failed to produce a dicarbide with a structure differing from the tetragonal variety characteristic of the slowly cooled compound. Even quenching from the molten state yielded the same result.

The correct melting point of thorium appears to be below 1700° C. Determinations were made with an optical pyrometer sighted on a hole in the side of a bar prepared from pressed powdered metal. Bars with the lowest electrical resistivity, and assumed to be the most nearly pure, gave the lowest values for melting point.

Melting point determinations and thermal curves for the solid metals have been run on a series of thorium-zirconium alloys but the number of reliable points is not sufficient to permit construction of a constitutional diagram of the system. For the zirconium-rich alloys, mixtures of the metals were melted together as an alternative to co-reduction. Thorium was introduced by means of master alloy of zirconium and thorium. Contamination by carbon from the graphite crucible appears not to be serious. An apparatus for quenching specimens under vacuum from temperatures up to 1200° C. has been made and is undergoing preliminary trials.

This report was prepared by W. E. Dreeszen, Administrative Aide to the Director, from material furnished by the group leaders of the Ames Laboratory.

Contract No. W-7405 eng-82.

REF ID: A53110

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Preparation of a series of binary alloys of thorium with iron, nickel, cobalt, manganese, and chromium is partially completed. The alloys are melted in beryllium oxide crucibles under a vacuum and are allowed to solidify in the crucibles. They are to be used for x-ray investigations.

The Chemical Engineering group continued studies on the preparation of pure thorium salts from monazite by liquid-liquid extraction methods, and on recovering residual thorium from the slag resulting from thorium reduction.

The study of conditions affecting the decomposition of monazite with sulfuric acid was completed and the results are presented in detail in a thesis by T. W. Blickwedel, entitled "Decomposition of Monazite", Iowa State College, 1949 (ISC-66). It was found that the optimum concentration of acid is 93% H_2SO_4 . By proper choice of acid proportion, temperature and time of digestion, essentially complete decomposition of monazite can be secured.

Since extensive tests for a single solvent for extraction of thorium from the decomposed monazite solution failed to indicate a suitable solvent, experiments were made with mixed solvents and complexing agents; a detailed account of the search for a single solvent is presented in a thesis by M. L. Feldman entitled "Solvent Extraction of Aqueous Solutions of Rare Earths", Iowa State College, 1949 (ISC-67). Further experiments to find a complexing agent were carried out but no agent was found which would permit good extraction of thorium from the sulfate-phosphate solutions. Experiments were then undertaken to introduce nitrates into the solution from decomposition of monazite with sulfuric acid since nitrate solutions have been found well adapted to solvent extraction in other work. By the addition of calcium nitrate a precipitate of calcium sulfate was obtained which contained less than 1% of the thorium. The phosphate remains in the solution. Experiments are underway on the solvent extraction of this modified monazite solution.

Construction of an 8-foot liquid-liquid extraction column was completed. Tests of the performance of the column were made with the system hexone-water since this system is of considerable interest in other studies at this site. The column was operated at a wide range of flow rates to determine the hold-up of the dispersed phase, the drop size of the dispersed phase and the column capacity. A method of measuring hold-up by a differential manometer was developed. The data were correlated by a modified Reynold's number-friction factor plot. The details of this work are summarized in a thesis by William B. Grandjean entitled "Pressure Drop Studies on a Liquid-Liquid Extraction Spray Column", Iowa State College, 1949 (ISC-63).

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An analytical procedure has been found which is suitable for thorium determination on the slag from thorium reduction. Samples of the slag have been separated into screen fractions and are being analyzed.

2. Calcium

The problem of producing calcium by reduction of the oxide with aluminum has not been resumed as all available time has been spent in setting up a second unit for purifying commercial calcium by distillation. The construction of this unit has been delayed as machinists' time has had to be diverted to other work.

Difficulty with sticking of the charge in the present distillation unit has been overcome mainly by alteration of the distribution of heat to different portions of the retort. These adjustments have caused some lowering of the operating temperature and will be compensated by installation of a large gas burner now on order.

3. Beryllium

The experiments on the best combination of amount of hydrochloric acid and time of leaching for extraction of beryllium from slag demonstrated that the time for preferential neutralization of the beryllium portion was too short for satisfactory separation. In general, the investigation has shown that about 33 per cent of the beryllium can be extracted readily but that the remaining 67 per cent appears to be held in combination, probably with calcium fluoride, and can be released only by a heavy expenditure of acid.

As an alternative, leaching trials have been made with a strong solution of sodium hydroxide. About 33 per cent of the beryllium can be extracted readily as the soluble sodium beryllate but the remaining 67 per cent is not amenable to this simple treatment. New procedures for the extraction of the remaining beryllium are being investigated.

4. Zirconium

Ordinary hydrofluorination of zirconium dioxide, as described in previous reports, has been found to proceed more nearly to completion if the run is interrupted periodically to permit pulverization of the charge. In the investigation of the fluidizing method of hydrofluorination, tests have been made of the effect of particle size of the zirconium dioxide on its fluidizing behavior. Glass apparatus and a current of air were used in the study. The results indicated that particle size had little effect over the range from +28 to -200 Tyler screen size. Monel apparatus was designed on the basis of the experiments and is ready for installation.

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Hydrofluorination of zirconium tetrachloride is under trial. It presents a problem with respect to corrosion of the apparatus but can be performed in graphite containers with a copper shell for catching any vapors that get through the graphite. It does not require a high temperature and gives yields of approximately 90 per cent. Use of the chloride is advantageous in that any unconverted material is reduced during bomb reactions and does not introduce oxygen into the metal.

Experiments on the reduction of zirconium tetrachloride were only moderately successful. Zinc fluoride proved more effective as a booster than zinc chloride. However, no combination gave as good yields as when zirconium tetrafluoride was used. With this compound, zinc chloride was the better booster. Substitution of magnesium for calcium as a reductant was found unsatisfactory, partly because the magnesium combines preferentially with the zinc.

Because values obtained for the vapor pressure of zirconium tetrachloride and hafnium tetrachloride were slightly higher than those reported in the literature, tests were made of the accuracy of the equipment for measuring temperature and on the uniformity of temperature in the bath surrounding the vapor pressure apparatus. No cause for an error as large as 1° C. could be found. Measurements of the sublimation point of zirconium tetrabromide gave a value of 356° C. which is 1° C., below the published value. Difficulty was experienced in work with hafnium tetrabromide, apparently due to the presence of moisture. The apparatus for making bromides is being altered to reduce the possibility of the entrance of water vapor.

The investigation of the zirconium-titanium alloy system is being continued. Of the trials made, 12 yielded products that had been molten and one of the alloys had a promising degree of ductility. Attempts were made to use magnesia and beryllia crucibles despite their unsatisfactory reputation as containers for zirconium but the output of the available oscillator was insufficient to produce melting in a non-conducting crucible. Best results were obtained with graphite crucibles but these contaminated the melt to some extent. A new oscillator with greater output has been

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ordered for this work.

Several methods of attack on the problem of separating hafnium from zirconium are being attempted. Work with high-boiling organic compounds as a distillation solvent for the chlorides has been dropped temporarily in favor of complexing, followed by distillation of the complex in an Oldershaw column. A trial distillation of the chlorides complexed with phosphoryl chloride was made in a 36-inch column packed with glass helices but the results were not promising; consequently the Oldershaw column is being set up for the work. This apparatus is 1 inch in diameter and has 50 plates.

In the expectation that favorable results would be obtained by the distillation of the complex described in the previous paragraph, two preliminary trials were made of the effectiveness of hydrogen fluoride for decomposing the complex. Tests of the products to date indicate that this method may be effective.

5. Uranium

The investigation of the zirconium-rich end of the uranium-zirconium system has been delayed because of lack of means for preparing the alloys. Apparatus is being built for the production of these alloys from the powdered components and a furnace for their subsequent heating at 1600° C. is also under construction. This procedure should give homogeneous specimens.

6. Miscellaneous Metallurgical Investigations

A method for the production of vanadium trifluoride has been developed and experimental amounts of the salt prepared. The procedure involves reduction of vanadium pentoxide to the trioxide with hydrogen and subsequent hydrofluorination of the trioxide to vanadium trifluoride. Bomb reduction of the trifluoride using calcium gave practical yields but attempts to use magnesium as a reductant have not been successful.

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PHYSICAL AND INORGANIC CHEMISTRY GROUP I

1. Separation of Rare Earths by Ion Exchange (Under the direction of F. H. Spedding and E. I. Fulmer).

The ion exchange resin, Amberlite IR-105, was tested on 50-50 mixtures of samarium and neodymium using 0.1% citrate at a pH value of 6.0 and a flow rate of 0.5 cm. per minute. It does not appear that the resin has any properties which would warrant its substitution for either Amberlite IR-100 or Nalcite HCR in pilot plant separations.

A number of resin capacity studies were made to furnish data needed in making a theoretical study of the separation of rare earths by ion exchange. After air drying the resins in their acid form at 40° C., it was found that Amberlite IR-100 had a capacity of 1.75-1.77 and Nalcite HCR a capacity of 4.23 milliequivalents per gram of dry resin.

The large scale rare earth production program is continuing and during this period 50 grams of spectrographically pure Pr_6O_{11} and 200 grams of 98.0% Pr_6O_{11} , containing Ce_2O_3 and La_2O_3 as impurities, were obtained.

The conditions for optimum extraction of ytterbium from its neighboring elements by sodium amalgam were studied. The results of these experiments were found to be in accord with the findings of Moeller and Kremers (Ind. and Eng. Chem. 17, 798-800 (1945)) and approximately 100 grams of pure ytterbium oxide were obtained using this method of purification.

2. Preparation and Properties of Rare Earth Metals (Under the direction of F. H. Spedding and H. A. Wilhelm).

Neodymium and praseodymium have been prepared for experimental use and specimens of both metals sent to other laboratories for studies of magnetic susceptibility and nuclear properties. The metals were prepared by reduction of their chlorides with calcium. Yields above 95 per cent were obtained.

Attempts to prepare yttrium in the same manner were less successful. The reduced product was in a finely divided form that could not be melted and cast into a solid mass. Plans are being made for changes in procedure which may aid in collecting the metal.

Experiments have shown that samarium trifluoride can be prepared in a rather pure state by precipitation of the salt from an aqueous solution and drying the precipitate under anhydrous hydrogen fluoride. The method is being studied as a means of obtaining the pure fluoride free from products of hydrolysis and moisture.

Construction is still proceeding on two sets of equipment to be used for the measurement of vapor pressures of the rare earth metals. Calibration runs have been made on the apparatus for determining heat content but the equipment is not completely reliable as yet.

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4. Zirconium-Hafnium

5. Miscellaneous Investigations

6. Service Analyses

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PHYSICAL AND INORGANIC CHEMISTRY II, III AND IV

Under the direction of
Wm. K. Plucknett, Robert Hansen and Frederick R. Duke

1. Research on Zirconium and Hafnium

A new apparatus for low temperature vapor pressure studies on $ZrCl_4$ has been designed and built. The order for radioactive zirconium was returned with the information that none was available and probably would not be for some time. Sensitive colorimetric methods for zirconium are now being investigated as a substitute for analysis by radioactive tracer.

Work on the preparation of hafnium-free zirconium has continued. It was discovered that hafnium was preferentially adsorbed when a solution of zirconium tetrachloride in acetone was passed through a silica gel column. A 10% recovery of zirconium containing less than 0.05% hafnium was obtained in a single operation. The column can be completely stripped by 1 N anhydrous hydrogen chloride in acetone and the recovered material recycled. By a suitable recycling system it is believed that at least 25% of the zirconium can be recovered containing less than 0.1% hafnium; the process is being scaled up to a small pilot-plant level in order to produce hafnium-free zirconium in pound lots.

Electrolytic separation of zirconium and hafnium from molten salt solution is being investigated. No analyses are as yet available.

Liquid-liquid extraction as a means of separating zirconium and hafnium is being investigated; promising results are being obtained.

2. Preparation of VCl_3

Preparation of pure vanadium trichloride is being attempted. Passage of chlorine gas over hot ferro-vanadium with the subsequent decomposition of the volatile tetrachloride has proved unsatisfactory. Reduction of vanadium pentoxide by sulfur monochloride in a bomb has been tried and appears satisfactory.

3. Theory of Adsorption of Gases

A new type gas adsorption apparatus suitable for the determination of a large number of adsorption isotherms over a range of temperatures has been designed and is under construction. It is felt that if this apparatus approaches the behavior theoretically to be expected it will represent a major improvement in gas adsorption technique.

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4. Mass Spectrometer Construction

Work has continued on the construction of a continuous sampling mass spectrometer to be used for hydrogen-deuterium separation studies. The D.C. amplifier, ion gauge, ionization chamber, and collector unit have been built.

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RADIOCHEMISTRY GROUPS I AND II

Under the direction of D. S. Martin and A. F. Voigt

1. Study of Radiations

Coincidence counting experiments have continued with attention being devoted primarily to a sample of (Nd-61)¹⁴⁷ received for chemical equilibrium studies. Analysis of γ - γ coincidences has been prevented by an effect which may be due to scattering. The effect has been found to be small with the 141 Kev quanta from Ce¹⁴¹; however it is to be studied with Au¹⁹⁸ possessing a 410 Kev γ . Some of the experiments with I¹³¹ have been repeated.

In an old sample of Ce¹⁴¹ the gamma ray component has been found to possess a complex decay period. Absorption measurements also indicate a component with an energy in the region of rare earth x-rays which has not been previously reported. Work has been started to determine by chemical separation if this activity can be definitely assigned to an isotope of Ce.

Several samples of the long lived fission product krypton, Kr⁸⁵, have been separated from uranium slugs removed from the pile about 3 years ago. The samples were collected in glass cells with thin mica windows. An aluminum absorption curve gave an energy for the β 's in agreement with the published value, ~ 0.7 Mev. A sample has been mounted in permanent form for half-life measurements.

A radioactive impurity which was found by the physicists in a sample of p³² originally obtained from Oak Ridge is being studied. It has been separated from phosphorus and seems to have two components, a soft β radiation with energy of about 0.25 Mev and a hard β radiation of about 1.9 Mev. The half-lives of both are long, ~ 150 days. The soft radiation is suspected to be due to Ca⁴⁵. Additional samples have been obtained and the study will be continued.

Studies of the radioactivities in the region of Eu-Gd-Tb are continuing. The ion exchange separation method is being used to purify the activities. A long lived component has been separated from the gadolinium, but its nature has not yet been determined. The coincidence circuit which will be used in the study of these activities has been built, but lack of space precludes its use at the present time.

2. Separation of Rare Earth Tracers

Further separations have been performed with the micro ion exchange column handling milligram samples with the intent of separating tracers in a period of 4-5 hours. Adsorption and elution have been performed with isotopes of Ce, Nd, 61, Gd, Eu and Yb and pairs of these elements. A

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10 ml cell beneath the continuously operated counter which measures activity in the effluent was used to gain the maximum resolution with some loss of sensitivity however. The cell walls, 30 mg/cm² and the solution thickness prevent satisfactory detection of soft beta emitters. Some attention has been directed towards more satisfactory detection of these particular emitters. While good separations have been attained in several instances, the conditions have appeared sensitive to the particular elements involved and considerable development work is still needed to obtain a satisfactory procedure for tracer separations and identification.

3. Fission Product Studies

Further experiments were conducted to gain familiarity and test the standard f.p. analysis procedures. A sample of thorium to be used for this work was purified from U and Ra contamination and daughters for this work. Experiments and computation have indicated that a Po-Be source is too weak for these experiments and pile irradiations are planned for the sample until the kevatron or synchrotron facilities are in operation.

4. Equilibrium Studies

A. Periodic Acid - The results of the determination of the ionization constants of H₅IO₆ performed with the cooperation of the Analytical Chemistry Group has been issued as a special report (ISC-33).

B. Yb+++ oxalate - The radiochemical determination of the equilibria of ytterbium in oxalate solution was prepared as a special report (ISC-58).

C. Nd+++ oxalate - Solubility determination of Nd in oxalate solutions were performed by radiochemical assays. Although the solubility was much lower and conditions nearly marginal, the data would appear to permit a satisfactory description of the equilibria.

D. Ag oxalate - A few solubility determinations of Ag in oxalate solutions were performed. Additional activity is needed for their completion.

E. Starch-iodine - Additional experiments with I¹³¹ tracer have confirmed the fact that reaction between iodine in CCl₄ solution and the amylose fraction of starch proceeds very slowly and satisfactory equilibrium data could not be obtained.

5. Kinetics

This work has been devoted to determination of the rates of reaction of Br₂ with S₂O₃⁼ and H₂SeO₃ when migration across an organic liquid-aqueous interface was involved and comparison with the homogeneous aqueous reaction.

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6. Studies on Hafnium-Zirconium Separation

An extensive literature search into methods for separating zirconium and hafnium gave several promising leads which are being followed. One is a vapor phase reaction between the chlorides and iodides of the two elements followed by hot wire reduction and hence removal of one component. The other involves solution chemistry, in particular the formation of complexes with polyhydric alcohols and related compounds such as sugars. Some peculiar reactions such as gel formation have been observed in these systems and it is hoped that the rather stable complexes formed will be of help in obtaining a separation.

7. G-M Tube Studies

Using the decay of an activity of accurately known half-life, ThB, the dead time of several G-M tubes has been determined. The results point out strongly the fact that in counting at normal rates (0-3000 c/m) a fairly high percentage (up to 5%) of spurious counts are included in the counting rate. At about 5000-10,000 c/m depending on the tube the gain due to spurious counts and the loss due to resolving time about balance each other. These studies were done with tubes filled with tetra-methyl lead which have several advantages over other types, in particular their lower dead time.

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RADIOCHEMISTRY GROUP III

Under the direction of S. Aronoff

1. A Study of the Variabilities in the Chromatography of Lysine

This study has resulted in a recognition that under proper circumstances it is possible to achieve what is equivalent to the separation of the ionic species. It has been shown that the number and position of loci of concentration vary with the hydrogen ion concentration in accordance with expectations from mass law and ionization constants.

2. A Determination of the Degree of Reliability of an Isotope Sugar Degradation Method, by the Use of Labeled Intermediates

Isotopic sugar is degraded by fermentation to lactic acid, which is then oxidized to acetic acid and carbon dioxide. The barium salt of acetic acid is pyrolyzed to barium carbonate and acetone, and the latter is converted by alkali and iodine into iodoform and acetate. By this series of reactions it is possible to determine the distribution of isotope within the lactic acid (and the pairs, 1,6; 2,5; and 3,4 in the sugar). Using glucose-C¹⁴-1, lactic-C¹⁴-acid, and both methyl and carboxyl-labeled acetic acid, it has been shown that the method used for sugar degradation is reliable. It appears that the organism employed to ferment the glucose (*Lactobacillus casei*) possesses the ability to form part of the lactic acid from the 1,6 carbons of sugar (rather than 3,4). Two explanations are offered: a) partial dissociation of a potentially symmetrical molecule (dihydroxyacetone phosphate to dihydroxyacetone - phosphate) or b) fermentation through a 3,4 hexose diphosphate. Either version represents a new step in the fermentation scheme. The details of this work are contained in a special report (ISC-42).

3. Review of Chlorophyll

An extensive review of chlorophyll has been completed. This review covers the analytical methods, the chemistry, optical properties (spectra, photochemical studies, etc.), physiology, industrial, and medical aspects of chlorophyll.

4. Research in Progress

Investigation of the distribution of isotopic carbon in sugars produced by relatively short time photosynthesis is in progress. A new (chemical) method for degradation of isotopic sugar is being developed.

Chromatographic analytical methods are nearing completion. Satisfactory modified methods, based upon known techniques, have been developed for: amino acids; reducing substances, as most sugars; and phosphorylated compounds.

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Investigations are in progress on determination of organic acids by this method. Applications of the above chromatographic techniques to soy bean roots has yielded the following results:

- a) most of the free amino acids present in the roots have been identified.
- b) glucose, fructose, and sucrose are all present, the most prominent being fructose, followed by sucrose. A variety of phosphorylated compounds are present.

$C^{14}O_2$ has been fed to soy bean roots and has been incorporated into stable compounds as a result of reversible reactions. Extracts were subjected to paper chromatography and the sheets radioautographed. Primary activity is found in a single spot of low solubility, possibly a phosphorylated compound. Next in activity was aspartic acid, with other amino acids in decreasing amount. Streaks of high mobility, possibly lipoidal, were present in high specific activity.

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X-RAY CHEMISTRY

Under the direction of R. E. Rundle

1. A New Punched Card Technique for Calculating Structure Factors

With the help of the College I.B.M. office, a method has been developed for calculating structure factors using punched cards and I.B.M. machines. The method uses sorter, tabulator and Model 501 multiplier and hand punch. It differs from the method outlined by Donahue and Schomaker (Am. Mineralogist, 33, 756, 1948) essentially in using terms $\cos 2\pi(hx+ky+lz)$ directly instead of breaking this term into products of sin and cos terms of one variable. It can be shown that our method is faster for certain hexagonal space groups, but it is often slower than the Donahue-Schomaker method for other systems. Generally the fastest I.B.M. method will involve a combination of this method with the Donahue-Schomaker method. This is possible with the same equipment, but a preliminary study of each individual problem will generally be necessary to determine the most rapid procedure.

2. Compounds of Thorium with the Transition Metals of the First Long Period

For some time a study has been in progress on the compounds in the alloy systems of thorium with the transition metals, chromium, manganese, iron, cobalt, nickel and copper. A brief report of progress to date is presented.

(a) Thorium-Chromium System

Sufficient work has been done to establish the fact that there are no intermetallic compounds in this system.

(b) Thorium-Manganese System

ThMn_2 , a hexagonal phase, $a_0 = 5.47$, $c_0 = 8.93\text{kX}$, is probably isomorphous with MgZn_2 . A complete structure investigation is in progress.

ThMn_4 is face-centered cubic, $a_0 = 12.50\text{kX}$. Investigation of structure is almost complete.

One higher compound exists for which neither composition nor x-ray data are yet certain.

(c) Thorium-Iron System

Th_7Fe_3 (See CC-G-3255 for complete structure).

The structure of ThFe is complex, and is as yet unknown.

Some higher compounds, including one at about 90 atomic % Fe, are similar to the higher Mn compound.

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(d) Thorium-Cobalt System

Th_7Co_3 (See CC-G-3255 for complete structure).

The structure of ThCo is complex, similar to ThFe .

ThCo_2 , ThCo_{4-5} and a compound about 90 atomic % Co, are similar to highest Mn compound.

(e) Thorium-Nickel System

Th_7Ni_3 (See CC-G-3255 for complete structure).

ThNi has a complex structure, similar to ThCo and ThFe .

ThNi_2 is hexagonal. $a_0 = 3.955$ $c_0 = 3.83kX$. Structure is probably AlB_2 type but has not yet been thoroughly checked.

ThNi_{4-5} is similar to ThCo_{4-5} . Some structural details known, but incomplete. Continuous change is definitely obtained between ThNi_4 and ThNi_5 .

The composition and structure of $\text{ThNi}_{(12)}$ is uncertain.

(f) Thorium-Copper System

Th_2Cu is tetragonal, body-centered, $z = 4$, $a_0 = 7.28$, $c_0 = 5.74kX$. It is probably C-16 type (CuAl_2) structure with $u = 0.164$.

ThCu_2 is hexagonal, $a_0 = 4.35$, $c_0 = 3.47kX$. Structure is AlB_2 type.

3. Structure of $\text{La}_2(\text{SO}_4)_3 \cdot 9\text{H}_2\text{O}$

The lanthanum sulfate enahydrate structure has been completed except for final refinement. The structure is hexagonal, $a_0 = 10.92A$ $c_0 = 8.13A$ $c = 2.821$; details, including principal distances, etc., will be reported at a later date.

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SPECTROCHEMISTRY

Under the direction of Velmer A. Fassel

1. Quantitative Spectrographic Analysis of Rare Earth Mixtures

The development of emission spectrographic methods for the quantitative analysis of rare earth mixtures and purified rare earths was continued. A method for the determination of samarium in purified yttrium was completed and checked for accuracy and precision. Calibration experiments were completed for the determination of samarium in purified gadolinium and for the determination of praseodymium and neodymium in purified lanthanum.

The broad fluorescent band (peak at 355 mμ) of cerium salts in solution can be used for the determination of cerium down to about 2×10^{-5} per cent in solution. The intensity of the fluorescence follows a relation $I = KC$ up to 1000 times the lower limit of detection.

2. Spectrographic Determination of Hafnium in Zirconium

The spectrographic method for the determination of Hf/Zr ratios in zirconium oxide samples described in the last report can be used for measuring this ratio in samples of the pyrophosphate, chloride, and zirconyl nitrate without prior conversion of these salts to the oxide. Pronounced changes in excitation conditions and in the concentration of extraneous elements such as sodium appear to have no significant effect on the analytical results. It is believed that the unique similarity in the physical and chemical properties of the hafnium and zirconium and the use of a line pair of nearly like excitation potentials combine to provide almost ideal internal standard compensation of excitation variables.

3. Miscellaneous Research

The following investigations are in progress:

(1) Review of the literature on x-ray fluorescence analysis and the construction of an x-radiation detector using a fluorescent screen and photomultiplier.

(2) Design of a bridge circuit and optical system for direct spectral intensity measurements in flames.

(3) Ion-exchange separation of boron from thorium and other materials used in nucleonics.

(4) Application of the porous graphite cup technique to the quantitative analysis of calcium metal.

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4. Service Analyses

A total of 1029 service analyses were completed during this period. The samples consisted primarily of rare earth fractions, zirconium oxide, and calcium metal.

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PHYSICS

Under the direction of W. C. Caldwell, J. F. Carlson, G. C. Danielson, Erling Jensen, J. M. Keller, L. J. Laslett, S. Legvold and G. H. Miller

1. Beta-ray Spectrometer Work

As mentioned in the previous Quarterly Report, the beta-spectra of Sr^{90} and Y^{90} have revealed an unusual form of energy dependence. The work on these spectra has been summarized in a Letter to the Editor by E. N. Jensen and L. J. Laslett, which appeared in the June 15 issue of The Physical Review.

More recently, further information concerning the Sr^{90} spectrum has been obtained. The Sr^{90} spectrum has been evaluated by subtraction of the Y^{90} contribution from the spectrum of our composite source and, in addition, data were obtained with a new and stronger source of separated Sr^{90} . The maximum kinetic energy of the beta particles was found to be 0.536 Mev which is in good agreement with the previous determination of 0.537 Mev.

The modified Fermi plot of Sr^{90} gives a straight line for the high energy part of the spectrum. At the low energy end, the Fermi plot deviates appreciably from a straight line. Further work is planned on the beta spectra of Y^{90} and Sr^{90} .

Recent work done at the California Institute of Technology on the gamma-ray energies of Co^{60} by Hornyak, Lauritsen, and Rasmussen with a magnetic lens spectrometer indicated values slightly higher than those we had reported previously. Since a source of ThB had been obtained after the above results were reported, the spectrometer was recalibrated by means of the conversion lines from ThB and ThC'' . The new calibration was slightly higher than the previous one. The new calibration also verified the linearity of the instrument. The photoelectron lines of Co^{60} were also redetermined. As a result of these determinations, the gamma-ray energies of Co^{60} were found to be 1.168 Mev and 1.330 Mev; consistent with the determinations of Hornyak, Lauritsen and Rasmussen, and with the precision wave length measurements now reported by Lind, Brown and DuMond. From a composite spectrum of Co^{60} and Zn^{65} , the revised value of the gamma-ray of Zn^{65} was found to be 1.118 Mev.

It is believed that, on the basis of the work in this laboratory alone, the foregoing revised values can be regarded as more reliable than those resulting from the early work here, since (i) the recent data were obtained concurrently with the recalibration and (ii) the stability of the spectrometer had been improved since the early measurements were made. A letter reporting this revision of the Co^{60} and Zn^{65} gamma-ray energies, by E. N. Jensen, L. J. Laslett, and W. Pratt, was published in the August 1 issue of The Physical Review.

Work has been in progress in an effort to adapt the beta-ray spectrometer for use in coincidence studies. Special G-M counters for detection of beta and gamma-rays have been designed and the applicability of scintillation counters

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is under investigation. For use with these radiation detectors a coincidence circuit has been built which exhibits a resolving time variable from approximately 0.5 to 3 microseconds and a fast scaler has been completed which is patterned after the Model 200 Pulse Counter circuit described in Rev. Sci. Inst. 18, 706 (1947).

In the hope of improving the transmission of the spectrometer, particularly for application to coincidence work, an active experimental and theoretic investigation of ring-focusing has been in progress. Calculations made by one of the theoretical physics groups were very helpful in locating ring focus in the spectrometer; results of the experimental group confirm these calculations quite satisfactorily. The position of the ring-focus has been found photographically and, by use of a large-window (7/8" diam.) counter in conjunction with various ring-annuli, a study was made of the resolution practically realizable with various transmission coefficients for the spectrometer. It appears that, with sources of the size customarily used, the use of ring-focusing will result in a two-fold increase of intensity without sacrificing the resolution.

Construction of the 180° low energy beta-ray spectrometer has been concentrated on the magnet control circuit. This circuit makes use of a beam from an electron gun which is deflected in the magnetic field and collected by two plates adjacent to each other. A control circuit adjusts the magnetic field until the currents to the two plates are equal. At the present time the magnet supply and magnet control circuit are virtually complete. Experimental work has been done on the R.F. power supply which will be used to accelerate the electron beam.

2. X-ray Spectrograph

Investigation of the radiations from Se^{75} with the bent crystal x-ray spectrograph have been deferred during this period in the interest of an investigation of the radiations from I^{131} , a radioactive material currently being investigated by one of the radiochemistry groups.

For the study of the weak radiations from the rather short-lived (8-day) I^{131} activity, it was planned to use a 20 millicurie source in order to obtain what was hoped to be adequate intensity. Due to last minute difficulties in preparation, however, the source (a silver iodide deposit, 1 cm. square, on one side of a silver foil) had a strength of only 5 millicuries. The 12-inch quartz crystal spectrograph was used for a two week exposure, with a permanent magnet to deflect electrons from the film. No X-rays or low-energy gamma-rays in the region 25-150 Kev were detected in this first exposure, but it is planned to repeat the experiment with a stronger source in the near future. Improved shielding of the Phillips "Searchray-150" unit, used in calibration, has recently been accomplished.

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3. Cloud Chamber Construction

As previously reported, the major components of the 12-inch cloud-chamber under construction at this laboratory have been completed with the exception of the magnetic field coils. The forms for the coils have now been received and winding has begun. The coils are to be constructed of seamless copper tubing ($\frac{1}{4}$ " O.D. and 0.065" wall thickness, covered with double silotex insulation), through which water will be circulated at high pressure. The cloud-chamber, as a whole, without the coils, has appeared to perform satisfactorily in preliminary tests. Facilities are being planned to permit the use of this instrument in connection with the 80 Mev synchrotron which is presently nearing completion.

4. The Decay of Na²²

The decay of a sample of Na²² has been followed, by means of a Lauritsen electroscope, during the past three years. This work has now been terminated and an analysis of the data indicates a half-life of 948 days or 2.60 years. A provisional value of 2.6 years was communicated to Dr. Seaborg during the course of these investigations and has subsequently appeared in the recent table of isotopes by Seaborg and Perlman. A note dealing with this work has been submitted as a Letter to the Editor of The Physical Review.

5. Mass Spectrometer

The mass spectrometer has been mounted in place and connected to the vacuum system. The caps for the tube were not soldered carefully and have had to be repaired several times because of leaks. The Kovar-glass seals have developed leaks and will not tolerate high temperature at present; a fitting has been designed which should remedy this situation.

The General Electric mass spectrometer leak detector has been used to hunt leaks effectively at pressures in the range of 0.01 to 1.0 micron, but at higher pressures the pumping speed through the needle valve to the leak detector is so low that the device becomes insensitive. An ionization gauge using helium as the probe gas is much more effective at pressures above 1.0 micron.

A transformer has been procured and connected to the metal mass tube to heat the tube for outgassing by the conduction of about 400 amperes. Thermistors have been attached to the mass tube to serve as the control elements in a temperature regulator.

A simple ion gun has been built and part of the rubber diaphragm model has been completed.

The results of research for several months have been incorporated into two amplifiers that have been built up in a single container which may be made fairly air tight.

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6. Fission Fragment Experiments

In the electrostatic deflection experiment to determine the effective charge of fission fragments, the collimating slits and film holder have been lined up so that alpha tracks have been observed on the film. Attempts are being made to cover the film with a thin aluminum foil, so that it will not be fogged by light from ionization when the deflecting voltage is applied.

In the experiment to determine the range-energy relationship of fission fragments, a new chamber has been built which should remove some of the objectionable features of the old one. This chamber will be larger and the gas purification will be better. The neutron source will probably be placed inside the chamber. Plans are underway to attempt to make a thin uranium foil by evaporation.

7. Low Energy Proton Experiment

Thin organic films for use in this experiment have been quite thoroughly investigated. These films must be quite thin, since it is desirable that the 0.3 Mev protons do not lose more than 10 Kev energy in passing through the film. So far, Formvar is the only material used. Two techniques have been tried; one involves dropping some Formvar (about 1% solution) on a smooth glass plate, resulting in a film of about 30 micrograms/cm² thickness. The second method consists of dropping Formvar (about 1% solution) on the surface of distilled water which is kept at about 40° C by a constant temperature bath. The films obtained by this method are about 5 micrograms/cm² thick, and will probably be suitable for use in the experiment.

The thickness of films has been checked in a number of ways. The primary method, of course, is the microbalance. However, this has the bad feature of destroying the films, since they must be cut away from the aluminum holder in order to be weighed. Consequently, a study has been made of the optical thickness as a function of weight. The fringe shift produced by the film in a Michelson interferometer has been measured for several groups of films. These films have then been weighed and a curve plotted of fringe shift against weight per unit area, resulting in a smooth curve but not a straight line. It is now felt that one can determine the approximate thickness by making an interferometer measurement and then reading from the curve.

The thickness of films has also been measured by letting 5 Mev alpha particles pass through a group of films in series. Usually about twelve films are used, since the energy loss through one foil would be too small to measure. The alphas are then counted with a photomultiplier and scaler, and curves of counting rate as a function of pressure are made with and without the foils. A comparison of the two curves gives the equivalent air

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thickness. This method warrants further study, as the results seem internally inconsistent.

An electrometer circuit for use in this experiment is now under construction. This circuit should measure very accurately the ratio between the current coming out of the final electrostatic analyzer and the total beam current.

Regulated R.F. power supplies for the deflecting plates of the electrostatic analyzers are under construction. Tests on one of these supplies indicate a short time instability of less than 0.1%; however, the long period drift is rather high.

A surplus electromagnet has been modified so that it will be suitable for the magnetic analyzer. A magnetic field measurement has been made with a rotating coil driven by a synchronous motor. The field thus measured is about 5000 gauss. A regulated magnet supply is now under consideration.

Further work on the electrostatic analyzers awaits completion of machining of the plates and housing.

8. Ferroelectric BaTiO₃

Investigation of the ferroelectric Barkhausen effect in BaTiO₃ using counter techniques is continuing. It has been found that the total number of domains counted at constant electric field strength decreases as $(\theta - T)$ decreases, where θ is the Curie temperature and T is the temperature of the crystal. This means that the domains are oriented in larger sections as the temperature approaches the Curie temperature where the domains are oriented most easily.

Single domain crystals more than 1 mm. square have been cut from large irregular crystals. These have been given electrodes and it is hoped to obtain their dielectric constant, hysteresis loss, piezoelectric constants and other physical properties. Attempts are also being made to determine their crystal structure. To do this, an attempt is being made to use tungsten K α radiation in order to obtain sufficiently high order reflections on the Weissenberg pattern.

9. Crystal Conduction Counters

Several diamonds have now been used in counting conduction pulses resulting from bombardment with alpha particles or gamma rays. Our first attempt to count slow neutrons with a single crystal of boric acid was unsuccessful.

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It has been found that the counting efficiency decreases with time much faster under bombardment with gamma rays than under bombardment with alpha particles. Experimental curves showing the number of counts per second as a function of time have been obtained. The time for the number of counts per second to decrease to one-half of its initial value is only one minute for gamma rays, but is more than two hours for alpha particles. This can be qualitatively understood on the basis of space charge from trapped electrons reducing the effective electric field inside the crystal. In the case of alpha particles, the conduction is largely a surface effect and the conditions are less favorable for the accumulation of space charge.

10. Dislocations in Metals

Progress has been made in (a) growing single crystals of metals, (b) circuit design for forming and observing transient mechanical pulses, and (c) construction of a Bragg dynamic crystal bubble blowing apparatus.

A resistance furnace for growing single crystals of metals is being constructed. The electrical circuits for controlling the temperature of the furnace as well as the variable speed mechanism for lowering metal samples through the furnace have already been designed and constructed. The furnace structure itself is two-thirds complete. Single crystals of tin have been grown by another method and their orientation determined by etching techniques.

A hydraulic cylinder arrangement is being made to replace the thumb-screw for applying pressure on the metal crystal under test. Static forces from 4 to 50 kg. may be applied. A pulsing arrangement has been devised in which a quartz crystal converts an electrical pulse into a mechanical pulse which can be applied to the specimen. The slip can then be more easily controlled, and we hope that the dislocations can then be more readily observed.

A Bragg dynamic crystal bubble blowing apparatus has been completed and operates very well. The dislocations are especially instructive since one can not only see them formed but also follow their rapid motion through the myriads of small bubbles. The analogy is so convincing that one feels certain that similar dislocations must exist inside a metal.

11. Semiconductors

An oscillator and amplifier have been built without vacuum tubes by using transistors. Power output is sufficient to drive a 5-inch speaker. Construction of equipment for measuring the Hall coefficient of metals and semiconductors is underway.

12. General Crystal Studies

A conoscope has been set up for orienting crystals optically. Monoclinic

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selenium crystals have been crystallized from a carbon disulfide solution and an attempt may be made to determine their crystal structure. Beautiful single crystals of sapphire, rutile, and spinel have been obtained from Linde Air Products Company and will be tried as crystal counters. Apparatus is being constructed for growing single crystals of pure boron.

13. Synchrotron*

The design, construction, and testing of the various pieces of auxiliary equipment for the 80 Mev synchrotron can now be considered essentially complete. The control circuits have been layed out in a form enabling the wiring in the accelerator building to proceed this summer in a systematic fashion and the control-desk panels are presently being assembled.

Upon receipt of permission to work in the accelerator building, the various items of heavy equipment required for operation of the synchrotron were moved in and secured in place. Construction of the capacitor rack was begun and the capacitors are presently being installed.

On the advice of Mr. F. I. Boley, currently on leave to work with the synchrotron group at the General Electric Research Laboratory, it is planned to build a new and more powerful R.F. power amplifier, employing two Eimac 4 x 150A tubes, to drive the synchrotron cavity. The Ceramic Engineering Group is continuing developmental work on the fabrication of ceramic vacuum chambers ("donuts").

14. Low Temperature Laboratory*

A. Facilities

The liquid air plant has been substantially completed. Tests of the plant for high pressure leaks and final operation of the plant await the installation of electrical facilities.

In the case of the liquid hydrogen plant, high pressure hydrogen storage facilities and the low pressure tank were installed. Construction of the liquefier was initiated.

The design of the liquid helium plant was completed and materials for its construction were placed on order.

* Although the work described in these sections is not supported by funds from the Atomic Energy Commission, it is believed that this brief progress report may be of interest.

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B. Research Program

Preliminary studies of research projects to be undertaken in the low temperature laboratory have been initiated. It is expected that a considerable amount of effort will be directed toward determining the physical properties of the rare earths at low temperatures.

Among the projects under consideration and for which apparatus may be constructed are: Studies of the specific heats of various substances, studies in solid state phenomena at low temperatures including crystal structure, and the study of nuclear processes using low temperature cryostats.

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ARCHITECTURAL ENGINEERING

Under the direction of T. K. FitzPatrick

1. Investigation of Laboratory Design

Basic research on design of laboratory buildings and equipment has continued. The laboratories listed in the previous report were revisited in part and further comparative data regarding the design of these laboratories and their function was continued.

2. Remote Control for Dry Box

Final construction details and drawings were completed for this equipment in collaboration with the Radiochemistry Groups. These details are now being assembled.

3. Miscellaneous

During this quarter, a quartz ampule tube cutter was developed in collaboration with the Radiochemistry Groups and a search has been made for a material in which to embed the cutter. This equipment is to be used in opening ampoule tubes. Design development was begun on special micro-pipette equipment with micro-control. In addition, the design of special range sink racks for Room 224 Metallurgy Building were completed. Selection of decorative design for main office in the Office and Laboratory Building was made. Special layout for a shop in the Metallurgy Building was completed as well as a lock section for Room 220 Metallurgy Building.

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APPENDIX L LIST OF REPORTS FROM THE AMES LABORATORY

1. Reports for Cooperating Laboratories

- ISC-33 Spectrophotometric Studies of Dilute Aqueous Periodate Solutions, by C. E. Crouthamel, H. V. Meek, D. S. Martin and C. V. Banks.
- ISC-34 The Synthesis of 1,2-Cycloheptanedionedioxime, by R. Vander Haar, R. Voter and C. V. Banks.
- ISC-41 Quarterly Summary Research Report for October, November and December, 1948.
- ISC-42 Degradation of Glucose-1-C¹⁴ and a Possible New Step in Mechanism of Fermentation, by S. Aronoff.
- ISC-43 Progress Report in Radiochemistry, by A. F. Voigt.
- ISC-45 Progress Report in Metallurgy, by B. A. Rogers.
- ISC-46 Progress Report in Physics, by W. C. Caldwell, J. F. Carlson, G. C. Danielson, E. N. Jensen, J. M. Keller, L. J. Laslett, S. Legvold and G. Miller.
- ISC-47 Radiations from Hf¹⁸¹, by E. N. Jensen.
- ISC-49 Purification of Plutonium by a Chromatographic Method, by John A. Ayres.
- ISC-54 The Use of 1,2-Cycloheptanedionedioxime as a Reagent for Nickel, by R. Voter and C. V. Banks.

2. Papers Published in Scientific Journals

- E. N. Jensen, L. Jackson Laslett, "Shape of the Beta-Spectra of Sr⁹⁰ and Y⁹⁰". Phys. Rev. 75, 1949-1950 (1949).
- R. E. Rundle, A. S. Wilson, "The Structure of Uranium Metal". Acta Cryst. 2, 126 (1949).
- R. E. Rundle, A. S. Wilson, "The Structure of Some Metal Compounds of Uranium". Acta Cryst. 2, 148 (1949).

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APPENDIX II. LIST OF SHIPMENTS

<u>Date</u>	<u>Item</u>	<u>Destination</u>
4-2-49	2.30 Kg. Th nitride	Western Reserve University
4-12-49	0.25 g. ytterbium	University of Michigan
4-12-49	3 g. neodymium 3 g. praseodymium 3 g. samarium	University of Chicago
4-14-49	499.51 Kg. thorium	Battelle Memorial Institute Columbus, Ohio
4-19-49	3.306 g. cerium oxide	University of Chicago
5-2-49	462.09 Kg. thorium	Battelle Memorial Institute
5-6-49	55.6 g. neodymium metal	AEC Chicago
5-6-49	64.5 g. didymium metal	AEC Chicago
5-6-49	5 gr. praseodymium	Carbide & Carbon Chemicals Corporation
5-11-49	5 lbs. 10 oz. zirconium	AEC, Homestead, Pa.
5-12-49	20 g. neodymium metal	University of California
5-12-49	5.0 g. neodymium metal	Oak Ridge National Lab.
6-2-49	2.47 Kg. Th fluoride 4.54 Kg. Th dioxide	Brookhaven, Upton, Long Island
6-6-49	115.94 Kg. thorium	Battelle Memorial Institute
6-8-49	429.80 Kg. thorium	Battelle Memorial Institute
6-8-49	25 lbs. calcium	AEC, Scotia, New York

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