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MDH-1-4 Ch II

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MANHATTAN DISTRICT HISTORY

BOOK I - GENERAL

VOLUME 4 - AUXILIARY ACTIVITIES

CHAPTER 11 - AMES PROJECT (IOWA STATE COLLEGE)

Department of Energy Document Number	
Determination: [Circle Number(s)]	
1. Classification Permanent	
2. Upgraded/Downgraded To:	
3. Contains No DOE Classified Info	
4. Contains: With	
5. Declassified	
6. Classified Info Preserved	
7. Other (Specify):	9/1/85
8. Review Date: 10/19/2013	
Authority: DC, X, DD, X	
Derived From:	
Declassify On:	
9. Review Date: 10/19/2013	
Authority: T9	

SSA
10/19/2013

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DATE: 9/14/21~~

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FOREWORD

This chapter presents an account of the Ames Project, which was the name given to the activities carried on for and under the Manhattan District at Iowa State College, Ames, Iowa. The chapter was compiled under the direction of Dr. F. E. Spedding, Director of the Project, by his assistant, Dr. E. I. Falmes, from material furnished by Heads of Sections and other members of the Project. It has been incorporated in the Manhattan District History as written, with a negligible amount of editing. The Ames Project was carried on under Contracts Nos. OMI 22-410; OMI 22-423; W-7408 eng-7; and W-7408 eng-82.

The account covers the Ames Project from its inception under OMI 22-410 until 31 December 1946.

December 1947

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MANHATTAN DISTRICT HISTORY
BOOK I, GENERAL - VOLUME A, AUXILIARY ACTIVITIES
CHAPTER 11, AMES PROJECT (IOWA STATE COLLEGE)

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I. INTRODUCTION1. The Role of the Area Project in the Manhattan District Operation

For many years previous to the war, fundamental studies in nuclear chemistry, nuclear physics and related subjects had been conducted in the United States, especially in university laboratories. While this research had progressed far enough to convince scientists that atomic energy might be made available for practical use, and that an atomic bomb might be possible, the financial aid was inadequate, being confined largely to that which the universities could afford over and above their normal teaching and research budgets. Furthermore, the several research programs were not correlated and so, not only did they frequently overlap, but research in important fields, which was urgently needed in order to advance the subject, was entirely neglected.

At the outbreak of the European phase of the war, some aid, through the National Defense Research Council, was given to several university groups to stimulate research in the development of means for releasing atomic energy. However, it was not until December of 1941 that it was decided that the subject warranted an all out effort on the part of the nation. The administration of this intensified effort was placed in the hands of the Office of Scientific Research and Development. Major programs were set up at three centers: Columbia University, the University of California and the University of Chicago.

The programs at Columbia and California were chiefly concerned with the separation of uranium-235 from uranium-238. The program at Chicago had three principal objectives: (1) studies of nuclear properties and

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the possibility of establishing a self-sustaining chain reaction, (2) the development of a basic science required for the development of an atomic bomb and (3) the possibility of the manufacture of plutonium by means of nuclear chain reactions. As a result of the demonstration of a self-reacting atomic energy pile at Chicago on December 2, 1942, the direction of the program was taken over, from the Office of Scientific Research and Development, by the Manhattan District of the Engineer Corps of the United States Army.* Later, large scale installations were built at Oak Ridge, Hanford and Los Alamos. The Oak Ridge project took over the responsibility for building a pilot-plant production pile which resulted subsequently in the construction of large scale piles at Hanford. The Los Alamos laboratory was responsible for the ultimate development of the atomic weapons.

The Iowa State College Project under the Manhattan District, usually referred to as the Ames Project, played a vital role as a supporting laboratory to the Plutonium Project. It was evident that there was a serious lack of basic information in the fields of chemistry and metallurgy, needed in the manufacture of atomic weapons. The Ames Project played a major role in helping to supply this badly needed information.

It was also necessary to develop a large number of chemical and metallurgical processes in order to produce the raw materials to be employed in the atomic weapons. The Ames Project made many important contributions in these fields and was particularly successful in the pioneering stages of such developments. Frequently the need of special processes was outlined at Ames and a method of attack was developed. These problems

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* Actually, the Manhattan District had been activated in August 1942.

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were then turned over to special groups and subsequently set up at other installations for further study.

There was also the problem of obtaining vital raw materials. Frequently, these materials were not made by industry, or the known industrial processes were inadequate for the manufacture of these materials in sufficient quantities at the required purity. The Ames Project was eminently successful in developing processes and supplying many of these materials. A number of these processes were later turned over to industry for large scale production. The research programs in physics, chemistry and metallurgy, at other sites of the Manhattan District, frequently required that materials be fabricated in special forms; methods for producing these objects were worked out at Ames and a large number of them was supplied to other installations. While the bulk of this service was to the other members of the Plutonium Project, considerable material was also sent to the Los Alamos and I-12 Projects.

The Ames Project also did a considerable amount of consulting work with many branches of the Manhattan District and served as a reservoir of trained personnel which were transferred to other installations as the need became critical.

A. Early History

In the early months of 1942, Dr. A. H. Compton began to assemble experts to make definite plans for the program to be centered at the University of Chicago. In February, 1942, he invited Dr. Frank H. Spedding to Chicago to discuss the possibility of setting up and directing the chemical program which would be needed to accompany the program in physics. It was decided that a small research program would be maintained at Iowa

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Iowa State College in order to take advantage of the facilities and research personnel already working in these fields. Dr. Spedding then joined the staff of the University of Chicago, and arrangements were made for him to spend half of each week setting up and directing the chemical research program at the "Metallurgical" laboratory (the code name for the Chicago laboratory) and the remainder of the week at Ames directing the correlated research at Iowa State College. This plan met with the hearty cooperation of President Charles E. Friley of Iowa State College, who placed the administrative details in the hands of Dean Harold V. Gaskill, director of the Industrial Science Research Institute. The first contract was to continue to July 1, 1942.

Dr. H. A. Wilhelm and Dr. I. B. Johns of the Department of Chemistry, Iowa State College, joined the project as associate directors at the request of Dr. Spedding. It was decided that the Ames laboratory would carry the bulk of the metallurgical research and also conduct fundamental studies in the chemistry of other materials which might have to be used in connection with the development of atomic energy machines. The research staff was rapidly expanded by the addition of a number of outstanding chemists to head the various sections. To strengthen the program further, several prominent scientists of Iowa State College were appointed as consultants. The project also acquired the services of a large group of scientific associates and skilled workers. Ultimately, at peak employment, the total number of project personnel exceeded five hundred.

The original contract, from February to July, 1942, with the Office of Scientific Research and Development, was for \$80,000. The success of the project was such that the Ames program was rapidly expanded. When

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the ORE contracts were taken over by the Manhattan District, a prime research contract was set up between Iowa State College and the Manhattan District. The major part of the research program at Ames was continued under this contract, W-7406 eng-82, which operated through Oak Ridge. By December 31, 1945, the face value of the Ames contracts amounted to \$6,907,000. However, the research, metal production and service work were carried out for \$4,000,000. Over two million pounds of uranium-metal billets were produced, thorium metal and other materials and services were supplied, and the entire research program was supported by these funds. When it is considered that the cheapest price per pound of uranium metal, produced by the rival process to that developed at Ames, was twenty-two dollars a pound, it is obvious that the Ames contracts were decidedly profitable even from a direct financial viewpoint.

The history of the developments in the Ames Project is the subject of the following account.

II. METALLURGY

1. Production and Casting of Metals

1.1. Uranium

Among the large number of problems facing the metallurgy group at the time of the initiation of the program, in February of 1942, there were two all-important problems connected with the production of uranium metal. One of these was the development of a method for the production of uranium of high purity and in large amounts in a short time; the other concerned the development of a procedure for large scale casting of the metal. Other problems such as chemical reactivity of pure uranium metal,

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protective coatings for uranium, alloy systems and the preparation of special compounds of uranium also received early study. Later, such problems as the development of processes for the production of pure thorium on large scale, the production of curium and other rare earth metals, the production of beryllium metal and the study of alloy systems involving thorium and beryllium were also investigated as the requirements arose and manpower became available.

The critical metal shortage necessitated that results be obtained quickly with a minimum of experimental work. Very little information was available in developing an adequate process for the production of uranium metal was available in the engineering literature. In general, time, materials and manpower were not available for making thorough investigation of the voluminous data-gathering type in the development work. A careful analysis of each problem, a thorough inspection of the experimental runs for clues to indicate reasons back of results, coupled with degrees of perseverance and inventiveness on the part of the key scientific personnel, led to the development of most of the metallurgical processes during the critical periods.

By early June, 1943, methods, materials and conditions for large scale casting of uranium were worked out at Ames. By early August, 1943, a method for the production of high purity uranium metal, by a low cost process that was capable of ready expansion into large scale production, was also developed in these laboratories.

Using the Iowa State College methods, small scale production of metal and casting into desired shapes for experimental pile testing at other sites were begun at Ames late in the summer of 1943. Production

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increased rapidly and an OSRD sub-contract with the University of Chicago for setting up a pilot plant to produce uranium by the Ames process was granted to the college in October, 1942. While the pilot plant was being set up, the physical chemistry laboratory continued to turn out metal for the Plutonium Project. In November, 1942, a Manhattan District Contract, under Madison Square Area, W-7405 eng-7, was also negotiated with the college for pilot plant production of uranium for the Plutonium Project. This contract and the OSRD sub-contract were concurrent for the same uranium production program during the few weeks preceding January 1, 1943, on which date the sub-contract terminated.

Numerous representatives from the Bureau of Standards, Massachusetts Institute of Technology, Westinghouse Company, Du Pont de Nemours Chemical Company, Mallinckrodt Chemical Company, Electro-Metallurgical Corporation, Battelle Research Foundation, Los Alamos and other project laboratories were shown complete details of the uranium metal process as carried out at Iowa State College. Numerous visits and reports kept these interested in the Ames process abreast of new developments. Research was continued to improve the process by studying factors affecting yield, purity, cost and availability of materials. In addition, members of the Ames technical staff were frequently called on to visit the industrial plants that were set up to produce uranium, using this process, and to assist them by suggesting changes in their procedures to give the same high quality of uranium metal being produced at Ames. The small scale production of metal in the physical chemistry laboratory furnished about two tons of metal for use as heart metal in the first chain reacting pile, which was operated at Chicago early in December, 1942.

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The pilot plant on the campus at Iowa State College was set up in a remodeled one-story wooden building which was referred to locally as the Physical Chemistry Annex. This was to be only a temporary arrangement for development and production until the more elaborate plants being set up at DuPont, Electromet and Halliwell could take over the entire uranium metal production program.

Operations, research, and development connected with uranium production were continued at Iowa State College after January 1, 1943, under the Manhattan Project Contract. The objectives at this point were production of badly needed uranium metal, improvements in the quality of the metal produced, development of better processing techniques, reduction in cost of production and increases in production rate and in overall production capacity of the process.

The main Ames process being used in early January, 1943, for production of uranium metal was based on the chemical reduction of the tetrafluoride by calcium metal. Finely ground uranium tetrafluoride was mixed with an excess of granulated calcium metal and the mixture poured into a refractory-lined iron bomb. A fuse wire buried in the charge was electrically heated to initiate the reaction, which continued throughout the entire charge, giving both uranium metal and calcium fluoride in the molten state. The more dense uranium metal, which collected at the bottom of the bomb, was allowed to solidify and cool to room temperature and was then removed for casting. This metal was cast by charging a graphite crucible with the proper amount, heating the charge in a vacuum to melt the charge and allowing the liquid metal to flow from the crucible into a graphite mold of proper shape.

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Metallurgical calcium was used early in the process for the production of uranium, although experiments in the Ames laboratories had shown that metallic magnesium could be employed as a reductant under special conditions. The relative ease with which calcium functions, and the urgent demand for uranium metal at that time, made it practically essential to operate with calcium until time was available for working out the conditions necessary for the more complicated magnesium process. Magnesium offered the following major advantages over calcium: (1) it was readily available in almost unlimited quantities as required by the project; (2) it was available in greater purity than the calcium; (3) approximately one-half as much magnesium as calcium would produce a certain amount of uranium; (4) the cost of magnesium was only about six per cent that of an equal weight of calcium. The main disadvantage of magnesium, as compared with calcium, was the fact that the heat of the reaction between magnesium and uranium tetrafluoride in the process is not alone adequate to fuse the products and cause a separation of the uranium metal from the slag; the heat produced with calcium is adequate for complete fusion and separation.

In order, then, to employ magnesium in the process, additional heat had to be introduced by means of side reactions, or the charge had to be heated before the reaction was initiated. Investigations of methods for introducing the heat along these lines, separately and in combination, were made before it was finally decided to use only a preheating furnace for the magnesium process. A number of new problems were introduced in using the preheating method with magnesium. Bomb-liner materials and methods of fabrication of liners were the first to receive attention. The magnesium-produced metal also introduced new problems in the casting process. However, a gradual change from calcium to magnesium was initiated

in January, 1943, and the new problems were solved in time to allow a complete change-over to magnesium in early March, 1943, without interfering with the scheduled rate of production which was being stepped up very rapidly from one week to the next.

Since some of the three industrial plants got into production as early as had been planned, the rate of production at Ames was stepped up to meet the increasing requirements of the Manhattan Project. The small pilot plant was expanded so that it reached a production rate of over 100,000 pounds of uranium metal per month by July 1, 1943. This is probably as high a rate, if not a higher one, than was reached by any of the more elaborately planned industrial plants during the war years. By July 1, 1943, the industrial plants had obtained a rate of production that warranted diverting raw materials from the Ames plant; as a sub-task was made in the Ames operations' force at that time. Although the production rate began dropping immediately, continued improvements in the methods brought the potential productive capacity of the Ames plant well above the 100,000 pounds per month to a potential capacity close to the total demands of the entire Manhattan Project.

In April, 1943, the jolting method of forcing the refractory lining in the bomb was developed at Ames. This procedure gave an immediate improvement in the quantity of the metal, since the refractory lining was much stronger and reduced materially the chance of uranium metal contacting the iron wall of the reaction bomb. Early manhole for felt-packing were made of wood, but after proper dimensions were established, with the wooden manhole, the more permanent properly ground and chromium-plated steel manhole were introduced into the operation. Refractory materials for

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forming the liner were not too satisfactory, because of the fact that the commercial materials useable, from the standpoint of mineral content, impurities and availability, usually contained small amounts of water. Specially burned high calcium lime served for some time as the best liner material available. Although some batches of this lime gave exceptionally good results, other batches gave decidedly poor results. Attempts to impress a manufacturer with the importance of maintaining the high quality which was possible with lime were not successful. Therefore, plans were worked out to set up a small kiln at Ames in order to control the burning of the lime to suit the specific needs of the local project.

Electrically fused dolomitic oxide was found to serve quite well as a refractory liner for the reduction hearth. This material could be produced very uniformly from one batch to the next and the analyses showed it to have low water and carbon dioxide contents. Since equipment was set up at an Electrometallurgical Corporation plant for production of the electrically fused dolomitic oxide, it was decided that this plant would furnish this material for the project. The cost of the electrically fused dolomitic oxide to the government was approximately four times the estimated cost of the high burned lime which the Ames Project had planned to produce. Under the urgency of the situation at the time, plans were set aside for producing adequate refractory materials at Ames, and shipments of the electrically fused dolomitic oxide were supplied by the Manhattan District.

One decided improvement worked out locally in the meantime, which applied to all refractory liner materials, electrically fused dolomitic oxide included, was the introduction into the refractory of a material that would react with water in the liner. For example, finely ground

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magnesium metal in the liner tends to react with any water present to give gaseous hydrogen instead of water vapor. When the bomb is heated, water vapor tends to react with the charge and thus considerably reduces the yields. The magnesium "fines" remedied this situation,

The production of virgin uranium metal by the regular Iowa State College process was discontinued at the pilot plant in Ames by January 1, 1945, all production by the process having been turned over completely to Mallinckrodt and Electrocast. The total production of virgin uranium metal at the Ames plant was approximately one and one-half million pounds. Research and development work on special processes for virgin metal continued at Ames until V-J Day.

The operations of extruding the uranium billets into bars and machining the bars into "slugs", to be used in the various piles, resulted in the accumulation of large quantities of waste turnings. A method for the recovery of these turnings was developed at Ames late in 1943. Subsequently, plans were made for setting up a plant at Ames for converting the turnings, from all Manhattan Project sites, into good sound metal ingots. A brick, fire-proof, one-story building was constructed early in 1944 for housing this recovery process and was known locally as Physical Chemistry Annex Number 2.

In this recovery process, the turnings were dumped into an inspection bin where the lumpy turnings were pulled apart. The larger pieces of uranium metal, large foreign objects, and turnings of foreign metals, were removed at this point by hand. The turnings were then passed over a magnetic separator to remove "tramp" iron and other magnetic impurities before delivery to a sifter. The turnings from Hanford were, in general,

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quite free from large pieces of foreign matter, but turnings from some of the projects and early production shops were often highly contaminated with materials such as iron, brass, stainless steel, aluminum, shop tools, etc., and required very careful inspection and hand picking before sending them to the miller.

The turnings were cut, washed, rinsed, dried and again carefully inspected and foreign matter was picked out by hand. They were then repressed over a magnetic separator and pressed into briquettes about one inch in thickness by four and a quarter inches in diameter. These briquettes were sent to the casting room, where they were melted and cast into regular sized ingots. The addition of a small amount of magnesium metal to the briquettes acted as a nitrogen and oxygen "getter" and considerably increased the recovery of sound metal in the casting process. Over 600,000 pounds of uranium metal were recovered by this process and shipped out for fabrication and use in piles, along with the regular production metal. In December, 1945, this recovery process was discontinued at Ames; part of the process was taken over by Metal Hydrides, Inc., Beverly, Massachusetts, and part of it by the metal recovery plant then being set up at Hanford, Washington.

Two variations from the regular Iowa State College process for producing pure uranium metal, which show some promise of reducing cost of production and increasing efficiency in supplying the metal to the pile, were being worked out at Ames late in the war period. One of these variations consisted of casting the molten metal directly from the reduction bomb into a mold of proper shape to give a billet for extrusion. This was

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designed to eliminate the remelting and casting of metal, as ordinarily done by the regular two-step process. Considerable improvement in quality and soundness of the metal had been attained, as compared to early metal produced by the one-step process, when the work was discontinued, because of lack of materials and funds. At that stage in the development of the process the metal was considered as slightly inferior to the metal by the regular two-step process. Further improvements were possible and were planned but were not realized because of curtailment of the research program along such lines at the time.

The other variation which offered considerable promise was the casting of metal in the form of rods which could be machined directly to "slugs" for use in piles. Instead of casting ingots $4\frac{1}{2}$ inches in diameter for extrusion into rods as in the regular process, the castings were made in 30 inch length by $1\frac{1}{2}$ -inch diameter steel molds. Nine of these rods were cast at each pouring, giving enough metal for 27 eight-inch "slugs". The quality of these "slugs" was brought up to equal or surpass that of the regular "slugs" produced by extrusion.

The Ames Project was awarded the Army-Navy "E" flag, with four stars, signifying two and a half years of excellence in production of metallic uranium as a vital war material.

1.2 Cerium

The Ames laboratory was asked, in the early summer of 1944, to supply several other sites with very pure cerium metal. This metal was essential in the fabrication of cerous sulfide crucibles which were very important at that time in the plutonium program. Cerium

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metal of sufficient purity could not be obtained commercially, so it was necessary to have it made on the project.

A method of reducing anhydrous cerous chloride with calcium was rapidly developed at Ames. The reduction was carried out in an iron bomb with a refractory liner; iodine was used as the "booster". The most convenient commercial cerium salt available was hydrated cerous chloride. A method of drying this salt under a reduced pressure (7-8 mm) of hydrogen chloride gas was found which gave an anhydrous product which only needed to be ground before being reduced. A "dry-room" was built in order to carry out this grinding operation so as to prevent the hygroscopic cerous chloride from absorbing water.

Since the cerium metal from the bomb contained some metallic calcium and magnesium as well as occluded slag, it was necessary to reheat the metal to remove these impurities; reheating was also the most convenient method for obtaining the 3/4-inch diameter rods, which was the desired form. The reheating had to be done under vacuum because of the extremely reactive nature of cerium metal. The metal was melted in a magnesium oxide or calcium oxide crucible and heated to 900°C. It was then poured into a graphite mold containing twenty cavities each 3/4-inch in diameter and four inches long.

The first cerium metal was shipped from the Ames laboratory in August, 1944. Increasing amounts were requested almost weekly until the demand for cerous sulfide crucibles declined. Production and casting of cerium was concluded in August, 1945. A total of 437 pounds of metallic cerium was shipped, most of it better than 99 per cent pure.

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1.3 Rare Earths Other than Cerium

The rare earths occur among the fission products of a chain-reacting pile. Some of them are known to have extremely undesirable nuclear properties and must be periodically removed from any production or atomic energy piles. Because of the very limited quantities of pure rare earths available, their nuclear, chemical and physical properties were not well known. The experimental chemists and physicists at other sites desired metal targets of the elements for bombardment in cyclotrons and piles in order to study these properties. Processes were worked out at Ames for producing rare earth metals of high purity. Several lanthanum metal targets were produced and shipped to other sites.

Traces of the rare earth metals in the thorium and uranium metals rendered them unfit for use in piles. The Ames group found it desirable to study the properties of these rare earth metals in order to determine whether they could be removed by metallurgical operations without chemical purification. The rare earth metals also constitute a most favorable group for studies on theories of the metallic state. A program was started, and is still in progress, at Ames, to produce most of the rare earth metals, to be employed in advancing the knowledge of the metallic state.

1.4 Thorium

Soon after the large scale bomb reduction of uranium was in successful operation, similar approaches were made on the reduction of thorium. In August, 1943, attempts were made to reduce a number of thorium compounds in a bomb. The first attempts were unsuccessful

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because of the high melting point of thorium and the great stability of its compounds. Later, small amounts of thorium were produced by reducing ThF_4 with metallic calcium, using calcium as a "booster." The yields were low and the metal was obtained in small pellets which were very difficult to remelt into solid metal. In August, 1944, ThCl_4 was tried as a "booster" and solid biscuits of thorium-calcium alloy were obtained in good yields. Within three months the conditions necessary for good yields had been well enough established to allow expansion of the process to the use of a reduction bomb six inches in diameter. As the demands from other sites increased, the production of thorium was increased to fulfill their needs. The effects of various factors on the yield and purity of the metal were investigated during this small scale production program. By June, 1946, most of the details had been worked out successfully and the bomb reduction of thorium fluoride was ready for expansion to large scale production.

The process, as used in December, 1946, was a metallothermic reduction of thorium fluoride by metallic calcium. Zinc chloride was used to provide additional heat, to give a more fluid slag, and to form a leaching alloy of thorium which could collect in the form of a solid biscuit. The reduction was carried out in an iron bomb 7 inches in diameter and 45 inches long. This bomb was lined with a layer of asbestos which supported into place around a steel mandrel with a pneumatic jolter. The charge was placed in the bomb which was then closed and the reduction started by preheating in a gas-fired furnace. The bomb was allowed to cool after the reaction and the biscuit of thorium-calcium alloy was removed.

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and cleaned. This method produced a deposit of about 50 pounds of thorium-uranium alloy with better than 90 per cent yield of thorium.

The casting of thorium metal presented many difficulties because of its high melting point and its reactive properties. Very little experimental work had been done on casting metals in vacuum at temperatures above 1800°^C, so there was a scarcity of usable literature on the subject.

The use of graphite crucibles was not feasible because of the rapid reaction of thorium with graphite at temperatures above the melting point of thorium and to the lack of temperature control at the melting point of thorium. Several refractory oxides, such as calcium oxide, magnesium oxide and zirconium oxide were tested but were attacked by the molten thorium. Since boron oxide was found to be sufficiently resistant to the high temperature and to chemical attack by thorium metal, it has been used almost exclusively in the Ames process. Thorium oxide crucibles were not satisfactory because of their low strength and the apparent solubility of the oxide in liquid thorium metal.

The accurate measurement of temperatures about 1800°^C, under a vacuum, is difficult, especially in cases in which optical methods are unreliable because of glowing of windows. Since very close and reliable control of the temperature was desired, a thermocouple of molybdenum and tungsten was developed and calibrated. This thermocouple was usable but was replaced by the columbium-tungsten thermocouple which is more reliable and has essentially a constant ratio of electromotive force to temperature.

The early thorium castings at Ames were made by melting the metal and allowing it to run through a beryllia grill which would hold back the oxide skins and slag. The thorium did not flow well through the narrow

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openings of the grill and this method was discarded. Melting the thorium in a crucible the size and shape of the desired ingot proved to be a more reliable method of casting, although it often gave poor separation from the slag and oxide. The thorium-silica alloy produced in the reduction was first heated under vacuum in a graphite crucible to 1300°C to distill off the silica. The silica-free thorium was then placed in the beryllia crucible and heated to 1850 or 1900°C, under a vacuum, by an induction furnace, to give the final ingot.

Late in 1946, a method of casting was worked out for thorium that gave a marked improvement in the quality of the metal. Essentially, the metal was vacuum melted in a beryllia crucible from which the molten thorium was poured into a graphite mold. Castings of up to one hundred and fifty pounds in size were made by this method. By December 31, 1946, over 4500 pounds of thorium had been cast for shipment to other sites. Prior to the war, massive thorium metal was a laboratory curiosity and was listed in Lange's Handbook as selling at three dollars per gram. It is estimated that the metal produced by the Ames process, which was of a considerably higher purity, can now be produced for less than five cents per gram.

1.5 Beryllium

Development of a process for the production of pure beryllium metal was undertaken in December, 1945. This was first accomplished by the reduction of beryllium fluoride with metallic magnesium in an electrically heated open graphite pot. As soon as the information became available, that this process was similar to the then undisclosed process of the

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Brush Beryllium Company, further work on this process was discontinued.

The toxicity of many beryllium compounds made it more desirable to produce beryllium by the closed bomb method similar to the procedure used for uranium, thorium and curium; in a closed bomb reduction the hazard due to toxic dust is reduced to a minimum. A closed bomb reduction of beryllium fluoride with magnesium metal was developed at Ames. The additional heat required to fuse the slag and to bring about the separation of metal and slag was supplied by preheating and by the use of sulfur as a "booster." This process was abandoned because of the high pressures developed by the magnesium sulfide at elevated temperatures, which introduced an explosion hazard.

A highly successful process for producing very pure beryllium metal was developed at Ames. It is a closed bomb process, utilizing the reduction of beryllium fluoride with calcium metal, with the addition of a lead chloride "booster"; it has also considerably reduced the health hazard in the production of beryllium metal. This process has been developed on a laboratory scale and its successful application on a production scale is dependent upon a cheaper source of pure metallic calcium. This latter problem is included in the current Ames program and promising preliminary results indicate an early solution. Experimental castings of beryllium metal were made. The metal was melted and poured in open air, in vacuum and in inert atmosphere. The vacuum-casting process for beryllium gave better quality ingots than did the other casting conditions.

2. Fabrication of Uranium and Thorium

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Aside from casting, research at Ames on other methods of fabrication of metals was rather limited, being confined mainly to such production problems as the cutting and turning of uranium and thorium billets and other shapes. However, some experiments on the cold and hot rolling of uranium and thorium were completed. The effect of cold swaging on the grain size of uranium was studied and reported. In the early days of the project a number of special forms of uranium were fabricated and shipped out. Thorium has been cold-rolled from 1/8-inch thickness to 10 mils.

B. Corrosion Problems

The corrosion of uranium metal and certain uranium compounds by possible pile coolants was among the first problems investigated at the Ames laboratory. Since the postulated coolants were air, water and molten bismuth, studies were made of their effect on materials which might be used in the pile including uranium metal, uranium carbide, and uranium oxide.

In April, 1942, as supporting work for the first air-cooled pile, the reaction of air with uranium metal was shown to be considerable at temperatures well below 400°C. Since such corrosion limited the possible power output of the pile, a number of chemical protective coats were prepared and tested. Uranium oxide coatings prepared by wet and dry methods, sulfide coatings and carbide coatings were all shown to afford little protection against the oxidation of the metal. A sputtered aluminum coating was found to be too porous to provide sufficient resistance to oxidation.

The problem of water corrosion was of paramount importance since the

Large plutonium piles were to be water cooled. The type of materials and the thickness of the protective coatings or envelopes were drastically limited by the nuclear physics and engineering specifications for pile operation. It was shown that uranium is slowly attacked by water at room temperature. The chemical coatings, since they introduced only small amounts of contaminants, were considered as good possibilities for the protection of uranium metal. Uranium oxide, carbide, and sulfide coats were tested; however, they were found to have little or no protective value. The sputtered aluminum coating was also tested, but, as in the case of protection against air oxidation, was found to be too porous and too poorly bonded to the uranium to provide the necessary resistance to corrosion. Corrosion inhibitors such as boric acid and the orthophosphates were tested to find their effect on the reaction of uranium in boiling water. This phase of the project, together with the assisting personnel, was transferred to the Chicago Laboratory in March, 1943, when the large scale testing program was set up there.

The bismuth-cooled piles were designed to run at high power output and consequently at high temperature; this required protective coatings with refractory properties. The first experiments showed that at temperatures slightly above its melting point (271°C), bismuth metal reacted rapidly with uranium metal to form a black flaky powder. The chemical coatings provided little resistance to attack, although the UO_2 , U_2O_3 and BO_3 themselves proved to be quite stable in the presence of molten bismuth and also quite refractory, and so perhaps were suitable for use in this type of pile.

4. Alloy Studies

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4.1 Uranium Alloys

Early in the Ames project several problems arose requiring knowledge concerning the properties and fabrication of various uranium alloys. Since, before the war, uranium metal was relatively scarce, there was little known about its alloy systems. Therefore, considerable time was devoted to the study of alloy systems of uranium and later of thorium, beryllium and the rare earths.

The work on uranium alloys was begun in the spring of 1945, and the uranium-carbon system was first investigated. Before a satisfactory method had been discovered for the production of uranium metal, the Ames group found that uranium carbides could be produced in quantity by the high temperature reduction of uranium oxide with carbon. Because of some desirable properties, such as high purity and high thermal conductivity, these carbides were considered as possible alternatives for uranium metal for use in piles, and this prompted a detailed study of the uranium-carbon system. The method developed at Ames for the production of uranium metal involved melting and casting the metal in a graphite assembly, and this was another reason for the study of the above system. Since calcium and magnesium metals were used as reductants in the Ames process for the production of uranium metal, a study was made of the alloying tendencies of magnesium and calcium with uranium.

The use of uranium in piles to produce plutonium required that the uranium have a protective coating to prevent corrosion by the cooling water of the piles. One of the metals suggested for such a protective coating was copper, and so the uranium-copper system was experimentally determined to establish the type of alloy that would result at a uranium-copper interface, and the corrosion resistance of such an alloy was studied.

Since tin was one of the materials used in soldering aluminum jackets on uranium, the uranium-tin system was examined and the equilibrium diagram was constructed in conjunction with the coating studies.

Experiments carried out at another site had indicated that molybdenum was soluble in uranium metal. It was hoped that this solubility might confer some desirable properties on uranium, such as increased corrosion resistance and increased strength. The Iowa State College group was given the task of determining the uranium-molybdenum system; this work was completed, and the results were published in 1944. In connection with this same type of problem, the phase diagrams of uranium-beryllium and uranium-tantalum systems were constructed. The special interest in beryllium as a pile material prompted studies of the uranium-beryllium alloys.

An unusual method for removing plutonium from uranium metal was developed at Ames, based on the fact that silver and gold are immiscible with uranium in the liquid state. Experiments were conducted in which silver or gold was added to uranium enriched with plutonium, and it was found that over 90 per cent of the plutonium was extracted into the silver or gold layer. The uranium-mercury system was also worked out in an effort to purify uranium and separate plutonium from it. Considerable experimental work was done on a possible all-metallurgical separation of plutonium from uranium and the fission products. This metallurgical process is very attractive in that the fuel metal does not have to be destroyed in order to revitalize the pile. The Ames process showed much promise, but it was evident that a great deal of work would be required before it could be reduced to engineering practice. Because of the war urgency, this method was discontinued, since it was decided to adopt the diamine phosphate process in the Manhattan Project.

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This latter process was chosen because it could be put into successful operation at an earlier date. However, now that the war is over, further research should be done on such alternative processes.

Homogeneous pile studies had indicated that uranium-bismuth alloys might be extremely useful as possible fluid reactants, because of the low cross-section of bismuth for neutrons, and other favorable factors. This system was determined and the solubility of uranium in liquid bismuth was examined quite carefully, since this was the portion of the equilibrium diagram that was of particular interest. The uranium-manganese, uranium-schalt and uranium-thorium systems were also of interest in the homogeneous pile studies and were worked out at Iowa State College.

4.2 Thorium Alloys

An extensive study was made of the thorium-rich end of the thorium-zinc system, since the method developed at Iowa State College for the production of thorium involves the use of a thorium-zinc alloy. In an effort to improve certain properties of thorium, studies were made of alloys of the metal with zirconium, titanium, calcium and vanadium.

Extrusion has been one of the most satisfactory processes in fabricating thorium billets into the desired shapes, and it was found that copper is very helpful in protecting the steel die from alloying with thorium at the temperature of extrusion. Since this is a high temperature operation, the thorium-copper system has been studied extensively and proper extrusion temperature limits have been determined.

The relatively low-melting eutectic discovered in a study of the thorium-beryllium system at Ames was proposed as a possible pile material, and several metallurgical operations involving this eutectic were developed. Other thorium alloy systems which were studied, but not completed, are the thorium-bismuth, -carbon, -tungsten, -chromium, -molybdenum, -manganese, -nickel, -iron, -tin, -mercury, and -copper systems.

4.3 Beryllium Alloys

In the beryllium metal production program at Iowa State College, studies were made of the beryllium-lead, beryllium-bismuth and beryllium-zinc systems. These alloys were formed in the various bomb reduction techniques when metal salt "boosters" were used. The work on the beryllium-thorium and beryllium-uranium systems was mentioned above.

4.4 Rare Earth Alloys

Studies were made at Iowa State College of alloys of rare earths and rare earth mixtures with certain heavy metals, in conjunction with the research program at Los Alamos.

5. Production of Refractory Metals

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The metal production and research carried on at Ames required the forming of many types of refractory bodies of various physical and chemical properties and of many sizes and shapes. It was necessary that these be made of inert refractory materials of high purity for use as casting-molds, liners for reaction chambers, crucibles for melting metals and alloys, radiation shields, thermocouple shields, etc. The refractories used for these applications were principally CaO , MgO , BaO , SrO , ZrO_2 , ThO_2 , alumina and sillimanite.

In the course of the work at other sites a need for refractory crucibles of low porosity, high purity and stability was emphasized. Interest was expressed in the use of refractory crucibles of uranium compounds. In an attempt to meet these requirements, crucibles of uranium, nitride, oxide and carbide were produced. A number of crucibles of these compounds for experimental tests were supplied by Ames. In addition, a number of calcium oxide crucibles, of special properties and shapes, were sent to Los Alamos.

The "Y-12" program also encountered a need for solution cells of a refractory which could withstand severe thermal shock and temperatures of 2000 to 2500°C. In an attempt to meet these requirements, the carbides and nitrides of titanium, zirconium, tantalum and uranium as well as calcium carbide, tungsten carbide, beryllium nitride,

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thorium nitride, and uranium dioxide were investigated. Slits of all these compounds, except beryllium, tantalum and thorium nitrides, were submitted for experimental tests. The nitrides of beryllium and tantalum were found to decompose at approximately 2000°C, and the thorium nitride to be rapidly attacked by atmospheric moisture, and they were therefore unsuitable.

The oxides used in the production of ceramic ware were obtained from commercial sources, which in general were able to supply materials having the necessary purity required by the various processes for which they were used. The nitrides and carbides, however, had to be prepared locally. In general, the carbides were made by heating the powdered oxides or oxides with graphite or lamp black in a graphite crucible to approximately 1800°C; a slightly sintered or loose powder was obtained in each case. The nitrides and carbides of uranium, when in a finely divided state, are quite pyrophoric and will ignite and burn vigorously on exposure to air; this is also true of powdered metal. It was therefore necessary to employ special procedures in their preparation.

These general methods were employed in forming the refractory bodies, namely, slip-casting, tamping or jolting, and die-pressing. The slip-casting method was successfully employed in the fabrication of BeO , UO_2 , and ThO_2 ware. A body formed by slip-casting is permitted to air dry and is then fired to 1800°C or above, thus forming a dense rigid body. The tamping or jolting method consists of compacting the dry powder into a graphite mold by hand tamping or mechanical jolting. In the die-pressing method this is accomplished by the

application of direct pressure to the powder contained in a die. In this case, lubricants such as wax, stearic acid, etc., are often necessary to facilitate transmission of pressure between particles. Difficulty in this respect increases with the size of the object being formed. Therefore, large crucibles or other forms were generally produced by the tamping or jolting method. The refractory bodies so formed were then fired in an induction-type furnace, using a graphite crucible as the heating element. The reactivity of the refractory toward graphite in general determined the final or maximum sintering temperatures to which the compacts were fired.

In the case of small bodies formed by slip-casting or die-pressing, the firing could be carried out without having the refractory in direct contact with the graphite. Shields of various types were sometimes used. However, at temperatures of 1800° or above it was not practicable to shield oxide or nitride refractories from attack by graphite. Any graphite picked up by the ware during the firing operation was later removed by firing the ware overnight in a muffle furnace at approximately 1800°. Crucibles of suitable mechanical strength and porosity were prepared in this manner from the above oxides with the exception of thorium. Thorium crucibles were prepared by slip-casting or die-pressing and were then fired out of direct contact with graphite to approximately 1800°.

The production of nitride and carbide bodies was limited to the die-pressing method. To protect the nitrides from attack by graphite the compacts were fired in a larger container made of the same material or of some other material less reactive toward the nitride in question. Compacts of uranium monocarbide could be fired

to as high as 2400°C in a graphite crucible without any appreciable pickup in carbon content as long as the compacts were kept out of direct contact with the graphite. Pressed compacts of SnI could be fired to as high as 2700°C in a graphite crucible without appreciable increase in carbon content.

It was found that the pure nitride and carbide powders of the previously mentioned metals could not be sufficiently bonded by pressing in steel dies to withstand subsequent handling, except for very small articles. These powders are very hard and abrasive and show little or no tendency toward plastic flow at pressures attainable with hardened tool steel dies. A paraffin wax of fairly high melting point, added to the powder as a CCL_4 solution, served satisfactorily as a temporary bonding agent and as a lubricant to facilitate transmission of pressure between particles. Nitride crucibles and slides were formed in this manner.

The carbides when in a very pure form showed little tendency to sinter at temperatures below 2000°C . Uranium monocarbide compacts had to be fired to 2150°C or above before a sintered body of appreciable mechanical strength could be produced. A pressed compact of pure SnI did not sinter sufficiently to withstand handling when fired to between 2700 and 2800°C . It was found that the addition of sodium silicage to the powder served as a temporary binder and greatly improved the bond of the fired product. The addition of 2 to 3 per cent of iron, either as the powder or a salt in solution, to the SnI powder, gave a sintered product having appreciable mechanical strength when fired to approximately 2250°C . The iron remaining after firing was found to be less than 0.5 per cent.

III. GENERAL CHEMISTRY1. Diffusion Studies and Allied Processes

The Chemistry Division of the Amax Project was started in the spring of 1943. Its main purposes were: (1) to collaborate with the Metallurgical Division; (2) to conduct research in the chemistry of uranium and plutonium; (3) to study the radiochemistry of the fission products; (4) to develop possible processes for the separation of plutonium from uranium and the fission products; (5) to develop analytical methods needed throughout the project for research, control and production; and (6) to study the special chemical problems which arose in connection with the design of a plutonium pile.

Literally, hundreds of problems arose in the design of piles, and many of these were successfully solved at Amax. They ranged from simple problems such as the determination of solubilities, melting points, viscosities - through determination of analytical problems involving rare chemicals, physical chemical properties of various salts - to typical major programs as outlined below.

One important problem attacked by the group (starting in July, 1943) was that of the diffusion of fission products from hot uranium metal and its compounds. It was of great interest and importance in the design of piles to know the extent of the diffusion of the active fission products from the pile fuel into the stream of coolant. Since pile operation at high temperatures was then under consideration, these studies were carried out at 600° and 1000°S. The extent of the diffusion was up to ten per cent of the total amount present at the

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higher temperature for some of the fission products, but much less for others. The information obtained was valuable in contributing to the decision that it would be necessary to anneal the fuel material.

An allied problem was the high temperature technique which was used in the removal of fission products from uranium by metallurgical treatment. In these experiments the metallic uranium was heated to temperatures above the melting point (1800-1800°C) and in contact with various substances, particularly graphite, in order to determine how thoroughly the fission products could be removed without altering the metallic state of the uranium. It was found that most of the fission products were removed in high percentages, while the neptunium and plutonium also present were left with the unchanged uranium metal. In addition to distilling out many of the fission products, the mechanism in many cases was the formation of carbides with the graphite crucibles. The addition of molten slags, immiscible with the molten metal, tended to remove other fission products. Even more complete removal of the fission products was effected by blowing hot gases, such as SOCl_2 and SOCl , through the molten metal, an operation similar to that in the Bessemer furnace. The gases reacted more readily with the bulk of the remaining fission products than with the plutonium and uranium and thus tended to make their compounds volatile or reactive with the slag.

This research led to another type of metallurgical treatment aimed at a separation process for plutonium. It was found that low concentrations of certain metals, when dissolved in molten uranium, would preferentially extract plutonium by formation of compounds. On cooling, a separation of phases based on the formation of U_2Z was

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applied to determine the extent of the enrichment of plutonium in the added metal phase. Information was obtained in this manner on the affinity of metallic plutonium for other metals used in the study. Similar studies were made on the distribution of the fission products.

2. Uranium Chemistry

A general chemistry group conducted pioneering work on the inorganic chemistry of uranium. The importance of uranium hydride was discovered and its chemical and physical properties were extensively studied. The first observation of UH_3 , at Ames, resulted from an attempt, in June, 1948, to cast uranium in a hydrogen atmosphere. Pure UH_3 was prepared in March, 1949, and its very interesting properties suggested a number of applications. Exact studies were made of the equilibrium in the formation and decomposition of the hydride and of the deuteride. Differences in these equilibria suggested a possible H_2 - D_2 separation method. Both the hydride and the finely divided metal, produced on its decomposition, were found to react with mercury. The hydride forms a pseudo-amalgam which is most probably a suspension of hydride particles in mercury, while the metal amalgamates in almost all proportions from any liquid or solid amalgams depending on concentration.

Uranium hydride decomposes readily with heat to give hydrogen gas and a finely powdered metal; this reaction proved to be an excellent one for providing finely divided uranium metal. This very active finely divided metal proved very useful in producing very pure uranium compounds since it reacted readily with many gaseous compounds. A

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number of extremely pure anhydrous uranium salts were prepared in this manner and several of them were shipped to other sites. Among the compounds made in this manner were the tri- and tetrahalides and bromides, the nitrides, iodides, sulfides and other compounds. The reactivity of the uranium hydride-uranium metal mixtures was utilized in purifying a number of laboratory gases. By passing hydrogen over uranium turnings, in a furnace at controlled temperatures, it was possible to obtain extremely pure hydrogen. Any water vapor, nitrogen, carbon dioxide, or hydrocarbon would react with the uranium hydride, in equilibrium with the metal, to produce uranium oxide, nitride, or carbide. Since only the hydrogen is in equilibrium with the metal, only pure hydrogen emerges from the apparatus. If deuterium gas is passed over the powdered metal only deuterium emerges; this process offers an excellent laboratory method for producing pure deuterium, from H_2O , for experimentation.

Starting with powdered uranium metal, prepared from the hydride, advantage can be taken of the UH_3-UH_2 equilibrium, worked out at Ames, to purify nitrogen gas. Similarly, the UCl_3-UCl_2 equilibrium can be used to prepare pure anhydrous chlorine gas. Pure hydrogen chloride gas can be prepared by passing the gas over the uranium hydride-uranium chloride mixture. Also, the rare gases can be purified by first passing them over hot finely divided uranium metal which removes the oxygen, nitrogen and carbon gases and then passing the rare gases over more metal at a lower temperature to remove the hydrogen. All of the above gases frequently have to be purified, in the physical and chemical laboratories, and these Ames processes offer a very simple method for their purification.

Other applications of the uranium hydride techniques include the separation of intermetallic compound phases from uranium alloys mentioned above, etching of metallographic specimens and testing the soundness of the jackets on canned uranium fuel rods.

The uranium halides, prepared by the method just mentioned, were used in studies of the fundamental properties of the anhydrous salts, particularly the vapor pressures of the chlorides and bromides. The first step in the preparation, the reaction of UH_3 with the hydrogen halide, gives the trihalide; treatment of this salt with the free halogen produces the tetra-valent salt. Since these compounds are somewhat volatile at high temperatures, distillation was considered as a method for purifying uranium from the piles. Extensive studies of the vapor pressures were made, using the transport method over a wide temperature range. In addition to the desire for basic scientific facts, there were important applications of this information in the purification of uranium by volatilization, in the use of the halides in the electromagnetic type of isotope separation, and as "stand-ins" for the corresponding plutonium compounds.

Of considerable scientific interest were the production, identification and study of oxyhalides and mixed halides of uranium in its various valence states. Other compounds prepared from the hydride, notably the nitrides, were also studied, from the standpoint of refractoriness as well as from a purely chemical point of view.

The Ames laboratory was requested on a number of occasions to make pure uranium metal from depleted uranium compounds (uranium rich in U^{238})

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but containing very little U^{235}); this material was usually supplied as $U^{238}F_6$. A process was developed in 1944 for the reduction of UF_6 to UF_4 by means of HCl . This process was developed on a pilot-plant scale, at Ames, to produce kilogram quantities, and turned over to the Madison Square Area with a view to employment for large scale development at Oak Ridge.

B. Plutonium Chemistry

One of the major problems facing the Plutonium Project in 1943 and in devising a number of processes for separating plutonium, which it was contemplated to produce in the proposed piles, from the fuel uranium and from the fission products which would be formed during the reaction. These processes had to be simple, capable of being engineered on a large scale and capable of operation under remote control with a minimum of operational maintenance.

The radioactivity of the fission products was expected to be so great that it would be necessary to conduct all operations behind thick concrete shields, and the equipment was expected to be so contaminated at the start of the operation that any subsequent repairs, replacement or maintenance would also have to be done by remote control. Furthermore, because of the great value of the plutonium, its recovery had to be practically a hundred per cent, and it was necessary to obtain the material extremely pure and free from residual radioactivity from the fission products. The problem was made more difficult by the facts that the plutonium was expected to be extremely poisonous in the form of the dust and that large amounts of it could not be accumulated in one place without danger of explosion. The problem was still further complicated by the

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fact that almost no plutonium existed, nor could it be obtained in any quantity until the piles were constructed and the required processes performed.

The only plutonium available at that time consisted of a few micrograms which could be obtained by cyclotron bombardment. Therefore, one of the first problems facing the chemists was to develop chemical and ultra-microchemical techniques which would enable the chemists to determine the physical and chemical properties of the salts, solutions and metal, using such minute quantities. The major emphasis on plutonium chemistry was centered at Chicago. However, California and Ames also contributed to this program.

A number of processes for separating fission-produced plutonium were worked out on the project on a laboratory scale. Finally, one of these processes, the dimethyl phosphate process primarily developed at Chicago, was selected for development to large scale production at Hanford. The alternative processes, although several of them could have been successfully stepped up with further work, were de-emphasized or discontinued at that time in order to advance the selected process. A number of these alternative processes were important as incentives and general backups, in the long run, of being cheaper and more efficient than the one adopted. It seems likely in the future that several of these will be put into operation in connection with the development of peacetime atom energy power piles.

Conferences were held in Chicago almost weekly, at which the information obtained in the several laboratories was correlated. It is accordingly impossible to say that any process was solely developed

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by a single laboratory. In one process, a given laboratory might play a major role while the reverse might be true for another process. The AEC Project took an active part in this program.

In the summer of 1943, the AEC laboratory became interested in a dry process for separating plutonium from the fission products and uranium. The process was based on the preparation of uranium hydride from the metal, followed by conversion to the tetrafluoride and then to the hexafluoride gas. Subsequent reduction of the uranium hexafluoride to the tetrafluoride, after separation from plutonium, was to provide a compound which could be readily reduced to metallic uranium. This process was the outgrowth of an earlier one, developed at Chicago, in which the uranium was converted into UF_6 by reactions producing first U_2F_8 , then UF_5 , UF_4 and finally UF_6 . A different temperature (500-700°C) was required for each step and some of the gases were highly corrosive, for example the $HF-H_2O$ mixtures. The discovery of the hydride suggested an alternative procedure with many advantages. The tetrafluoride could be produced in two steps, hydride formation and hydrofluorination, both at 500°C, with dry HF the only corrosive gas. Studies on this procedure showed it to be feasible, though conversion of large amounts of U to UF_4 was slow. A simultaneous reaction procedure was also tried, in which both gases were allowed to attack the uranium at the same time. This was also shown to be feasible, though again slow because of caking of the reaction products around the uranium metal. During the process, the rare gases of the fission products were liberated, and it was demonstrated that they could be recovered from the other gases for experimental

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use, if desired.

Since the final reaction, conversion of UF_4 to UF_6 , required fluorine gas, methods for producing it were studied. A number of electrolytic cells using $KF \cdot g^{23}F$ as electrolyte and operating at a medium temperature ($75-80^\circ C$) were designed and built. Principal features of the design were a trichloroethylene bath for temperature regulation, a rectangular iron tank as container and cathode, a nickel plate as anode and the highly resistant polytetrafluoroethylene plastic (Teflon) as gasket and insulator material. This design was considerably better than anything then in the literature, although extensive work in another part of the Manhattan District, with which the Ames Laboratory had no contact, had preceded this.

One of the chief problems in the dry fluoride process was the question of the volatility of the higher fluoride of plutonium. Work was done on this problem by the Ames group which contributed to their knowledge, but no clear-cut understanding yet exists as to the volatility, stability and formula of the higher fluorides of plutonium.

In the dry fluoride process the uranium and a few of the fission products were removed from the rest of the fission product impurities and from plutonium by volatilization of their higher fluorides. Thus, a large percentage of the radioactivity could be collected with the plutonium in a highly concentrated form. The uranium and the volatile fluorides of the fission products could then be separated by fractional volatilization or other methods; the uranium could be recovered, in a decontaminated state, in a form which could be directly reduced to the

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metal by the procedure described in the Metallurgical section of this chapter. The specific separation of radioactive columbium was studied at Ames as representative of those volatile fission products whose fluorides could be removed from the plutonium at the temperatures employed. Only preliminary work was completed on the separation of plutonium from non-volatile fluorides by distillation, with subsequent collection of plutonium fluoride. It should be pointed out that this separation could be effected by other means than volatilization. During this period, attention was called to the fact that the dry fluoride process might be adapted, with further development, to the recovery of uranium from the uranium wastes of the bismuth phosphate process.

Considerable work was done at Ames on a metallurgical process for the separation of plutonium which is described in the Metallurgical section of this chapter.

An alternative process, worked on at Ames, was the development of a separation method based on the absorption column procedure developed at another site, but with additional steps using a complexing reagent. The reagent, which proved successful, 5-(2,6-dihydroxyquinoline-3-carboxylic acid, known as Ferron, forms a stable complex with Pu which, in a certain pH range, is not adsorbed by the column of ion-exchange resin which does adsorb most of the fission products.

Since the plutonium and the bulk of the fission products had already been separated from the uranium by the previous process, these materials could be desorbed, the plutonium could then be complexed with the reagent and reabsorbed through the column at the appropriate pH. Under these conditions, the fission products would again be adsorbed but the

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plutonium would pass through without being adsorbed. This process led to a high decontamination of the plutonium. A hot laboratory, capable of handling 5 curies through the adsorption process, was designed and built. The method proved successful in a number of runs. The uranium and plutonium were separated from one another and from the fission products using liq. samples of uranium, from the pile, and which had an activity of 5 curies.

After appreciable amounts of plutonium became available from the piles, it appeared that it would have to be obtained in the metallic state and extremely free from light element impurities, if it was to be used for weapons. An extensive "purity" program was set up in cooperation with the various sites, and the Ames Project took part in this program.

One of the several lines of action, carried out at Ames, was to conduct an exhaustive study into the organic complexes of plutonium. A number of these complexes should be extractable into organic solvents, and it was hoped that one of these would be specific with respect to plutonium or at least highly selective. The most successful reagents were derivatives of dimethylaluminumamine which led to complete extraction of the plutonium. Such a procedure would have been quite effective had not the "purity" program been cancelled six months after its initiation. An attempt was made to use these reagents in a decontamination process, but they did not give sufficient separation from some of the fission products. Some of these complexes offer considerable promise as reagents to be used in the liquid-liquid separation processes for plutonium which are now being developed.

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Usable amounts of neptunium were available much later than in the case of plutonium. Thus, although it was known that plutonium had stable valences of 3, 4 and 6 with a less stable one of 5, similar information was not available for neptunium. Experiments with tracer neptunium were designed to determine whether there was a stable valence lower than 4. Rather surprisingly, it appeared from the tracer experiments that a value intermediate between the lower valence (4) and the higher one (6) was stable in aqueous solution. It was suggested that $Np^{5.5}$ existed, a suggestion which was substantiated elsewhere when larger amounts of neptunium became available.

Since the handling of micro and ultra-micro quantities was a necessity, some thought was given to the design of equipment specifically intended for such use. Chief among the results was a magnetically controlled quartz fibre balance which could be operated externally to the system, weighing being made by means of a potentiometer. Development was also made of a magnetic susceptibility balance, using quartz fibre suspension,

~~A. Thorium Chemistry~~

~~A.1. General Chemistry~~

As soon as the first piles were in operation, and it was generally realized that thorium could be used to extend the available sources of fissionable material, it became necessary to enlarge the then rather meager basic knowledge concerning the chemical, physical and nuclear properties of this element. Practically all of the research programs which had been set up for uranium studies had to be paralleled for thorium. The Arec Project played a major role in this field, specializing in studies on the properties of inorganic compounds of thorium.

Among the results which have been reported in numerous project notes, reports and papers, it might be mentioned that the thorium-hydrogen system was found to consist of two different hydrides.

4.5 Purification of Thorium Compounds
by Liquid-Liquid Extraction

Late in the summer of 1944, when the Ames Project started work on the large scale production of thorium metal, research was initiated for developing a method of preparing thorium salts free from elements having high neutron-capture cross-sections. Thorium salts available commercially were sufficiently pure with respect to all of the undesirable elements except the rare earths. Hence, the problem became one of separating the rare earths from thorium.

Two approaches were considered in solving the problem: one, to find a suitable complexing agent which would facilitate the separation, and the other, to find a suitable extracting agent to employ in a liquid-liquid extraction. The latter seemed the more feasible approach in view of other extraction of uranium and of the work being done on the hexone extraction of plutonium. Major emphasis was soon placed on the extraction method, after preliminary tests indicated very favorable separation factors.

Preliminary tests of the available solvents indicated hexone to be satisfactory and that calcium nitrate was much better as a salting-out agent than the ammonium nitrate being used in Chicago for other elements. In September, 1944, a preliminary design was drawn up for a unit capable of purifying sufficient thorium nitrate to supply the metal production units with their requirements. An all-glass solvent extraction system

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was set up and operated successfully; it also furnished solutions for further investigation and data for improved design. By November, 1944, the design for the large scale unit had been completed, the necessary equipment had been ordered, and work had begun on the column structure. The viscosities and densities were determined for all of the solutions involved in the column process, and a rapid method was developed for analyzing the production solution from a refractometer reading and a specific gravity determination. Since thorium fluoride was required for the production of the metal, it was necessary to work out a procedure for processing the product solution into dry thorium fluoride; the method finally adopted involved amide precipitation, calcination of the amide and hydrofluorination of the oxide. This operation corresponded to the "green salt" (UF_4) manufacturing step in the production of uranium metal.

By June, 1945, the large scale unit had been installed, and operation had begun. By October 1945, sufficient operational data had been obtained to start daily production runs. However, it became evident that hexane was far from being the ideal solvent. The possibility was investigated of diluting, with an inert solvent, some of those which had previously been discarded solely on the basis of unsatisfactory physical properties. Butyl phosphate, diluted with butyl ether, was found to be extremely satisfactory for column operation and was first used in April, 1946. The extraction system finally adopted is capable of producing 25 lbs. of purified thorium per 8-hour day, operating for 20 days a month; this is a monthly production of 1100 lbs. of thorium. If the demand for the metal exceeds the above amount, the production can be greatly increased either by increasing the hours of

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operation of the present unit or by enlarging the column diameter.

B. Radiochemistry

The radiochemistry and fission product groups originated with the men doing the fission product analyses for the diffusion experiments mentioned earlier in this chapter. For this purpose, a method for the analysis of fission products was devised which was unique among project schemes, in that a single sample was used for most of the elements and a systematic method was used similar to that employed in qualitative analysis. The fission products were first separated into groups and subsequently into the individual elements. The development of such a method was required because of the low activities which diffused out in the experiments. Of particular interest were methods for separating the radioactive rare gases produced in fission. Since ordinary solution procedures gave large quantities of gases from which the fission product gases must be separated, methods were studied for removing the gases other than by dissolving the metal in acids. The fine subdivision of the metal on hydride formation was found, surprisingly, not to be effective, but if the hydride was decomposed, the resulting metal dissolved in mercury; on boiling, the gases were removed. Another procedure for dissolving the hydride in aqueous solutions of silver salts also gave the gases free of contaminants.

Various problems in the radiochemistry of the fission products were investigated. Among the isotopes discovered or first found by the Ames group were the 80 yr. Sr^{90} -80 hr. Y^{90} chain; the 17 min. Pr^{144} daughter of 87.5 day Ce^{144} ; the long-lived Sr^{138} , etc. A major contribution

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In this field was in the radiochemistry of elements 44 and 45, Rn and Ra; five new radioisotopes of these elements were discovered, chain relationships were established between some of them, and their mass assignments were made definite. In addition, absorption measurements of the beta-ray and gamma-ray energies were made for all of these isotopes and some others. Chemical methods were also devised and tested for many fission product separations.

L. Ron Rabin

Ever since the beginning of the Manhattan Project, there has been a constant demand for samples of rare earths of exceptional purity in gram amounts or greater. This demand arose for numerous reasons, but mainly because some of the rare earths are found among the fission products from chain-reacting piles. It was highly desirable, therefore, to have a means of preparing pure rare earths so that their nuclear properties could be studied and also to allow a more thorough consideration of their chemical behavior. Their radio-isotopes are less well understood than those of any other group of elements. Previous methods of separating these elements involved the well known, but laborious procedure of fractional crystallization as used by James and further developed in many laboratories. Exceptions are cerium with its tetravalent state and praseodym, europium and their divalent states which do permit a means of separation from the normal trivalent rare earth ions.

During the last half of 1944, groups of workers at the Clinton Laboratories developed a procedure for separating fission products by elution, from a bed of ion exchange resin, using citrate buffers and

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other complexing agents as eluants. This method was shown to be capable of separating cerium and yttrium activities from a carrier-free mixture of rare earths present in fission products.

Experiments were started at Ames in December, 1944, with a view to developing a process for separating large amounts of the rare earths, using the citrate elution technique. After demonstrating that micro-quantities of cerium and yttrium could be separated just as efficiently as the carrier-free activities, the process was applied to a neighboring pair of elements promethium and neodymium. Excellent separations were obtained and sufficient quantities of spectrographically pure salts were soon available.

The separation of the light rare earths (cerium group) was stepped up to pilot-plant scale, using the procedures developed in small scale tests. "Neodymium Carbonate", obtained from the Lindsay Light and Chemical Company, was the source material for this group. A battery consisting of 24 glass columns, each four inches in diameter and ten feet in height, was set up, together with stainless steel mixing tanks and other equipment to handle. Separation of the complex mixtures was aided by the use of other techniques, principally the sodium amalgam separation as developed by Marsh and the amalgamated zinc reduction of cerium as developed by McCoy. Several of this group were separated in amounts of 100 grams or more.

The above procedures were extended to the heavy rare earths (yttrium group), a much more difficult task because of the greater number of members of the group and the greater similarity of their properties. One of the principal problems was the procurement of source materials

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rich in heavy rare earths. The best sources for gadolinium were concentrates from Monazite sands obtained from the Lindsay Light and Chemical Company. For the heavier members of the group, the principal sources were the ores gadolinite, blomstrandine and fischelite. After preliminary tests to determine operational conditions, a pilot plant was set up similar to that employed for the light rare earths. The principal initial separation was that of the group Dy(66) to Lu(71) from Th, which was the major constituent; these concentrates were taken through additional columns for further purification. Sodium amalgam reduction was used to separate the yttrium; this step, in addition to yielding the element in the pure form also reduced the complexity of the mixture. Some of the heavy rare earths were separated in multigram quantities, but the low abundance of many of them prevents their separation in similar amounts.

In studies on liquid-liquid extraction, it was found that cerium (IV) nitrate is readily extracted from aqueous solution by tri-*n*-butyl phosphate, a solvent which is reasonably stable under strong oxidizing conditions. The conditions for extraction are not critical, and favorable extraction is not inhibited by perchlorates or acetates, but sulfates interfere. Evidence was obtained that the cerium (IV) nitrate is extracted from butyl phosphate by virtue of the formation of a new complex. By oxidation of cerium (III) with bromates and exhaustive extraction by butyl phosphate, the cerium may be quantitatively extracted. While this method provided a satisfactory separation of cerium (IV) nitrate from iron (III) nitrate, a little zirconium nitrate and larger amounts of lanthanum and praseodymium nitrates were extracted

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from mixtures of these salts.

IV. ANALYTICAL CHEMISTRY

1. General

Analytical chemistry is highly important in any type of chemical program. This was especially true in the Manhattan District Project since such pioneering work had to be done in new territory. Very little information was available in the literature with reference to many of the rarer elements of major importance in the Manhattan Project. Some of these elements were very frequently present in the various samples which had to be analyzed. Literally, hundreds of analytical procedures had to be developed and even the old established methods had to be modified because of interferences caused by the presence of these elements. The Ames analytical group played a major role in this analytical program.

The chemical analytical research which was carried out at Iowa State College consisted mainly of developing a wide variety of methods for analyzing, by chemical means, mixtures of elements which were of importance throughout the Project. Many methods were developed for the analysis of the raw materials, intermediates, and the final products in the processes for the production of metallic uranium, thorium, beryllium, and curium. A few of the major contributions were: (1) a general method was developed for the analysis of metallic fluorides involving their hydrolysis with superheated steam, followed by titration of the resulting hydrofluoric acid; (2) procedures for the determination of trace amounts of fluoride were critically reviewed, improved and adapted to metal samples; (3) methods were developed for determining the free metal

content of uranium burnings and for the analysis of many alloys of uranium and of thorium; (4) the spectrophotometric method for determining iron was adapted to the analysis of uranium metal and its compounds; (5) titrimetric methods for the determination of thorium were greatly improved and (6) organic analytical reagents were prepared and studied. In addition, the Ames group cooperated with the National Bureau of Standards and other analytical and spectrographic laboratories in the establishment of standard samples of interest to the over-all Project and in the exchange of data for the evaluation of analytical methods.

The routine analytical chemists made no small contribution to the successful completion of the various research programs of the Ames Project. They acted as a service group for other groups of this laboratory as well as for other project sites. To this end, they made well over 47,000 analyses for the Manhattan District Project.

B. Insufficiencies

The high purity requirements for the uranium, thorium, and beryllium used for scientific purposes compelled the imposition of rigorous specifications on the impurity content of these materials. Tolerances have been quoted for almost every element in the periodic table, in some cases as low as tenths or hundredths of a part per million. Spectrographic methods were particularly well suited to many of these required analyses, since by these procedures the impurity concentration of a great many elements could be simultaneously determined with adequate sensitivity and accuracy.

The spectrographic section was organized in the spring of 1943 to act as a service laboratory for the control of the high standards of

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purity required for the uranium that was being produced and to serve as a research laboratory for the development and improvement of spectrographic methods of analysis. During the period the production plant was in operation, approximately 25,000 quantitative analyses were completed. The carrier distillation technique, which was developed at the National Bureau of Standards, was used for the analysis of uranium base materials, while the methods for the analysis of calcium metal, magnesium metal and refractory bomb liners were developed at Ames. Simultaneous with the development of processes for the production of metallic thorium, beryllium and cerium, spectrographic procedures for the analysis of these metals were worked out. Fundamental studies on the carrier distillation technique were also undertaken.

Emission spectra methods were also extensively applied to the analysis of rare earths. These procedures have been very useful in following the ion-exchange fractionation of the rare earths and for the determination of purity of the end fractions.

The Ames Laboratory cooperated with the National Bureau of Standards and other spectrographic laboratories in the establishment of standard samples of interest to the Manhattan Project and in the exchange of data for the evaluation of analytical methods.

A. Mass Spectrometry

In the fall of 1945, a small group was formed in mass spectroscopy to set up instruments to be used in the analysis of isotope content. During 1946, the group was occupied in designing and constructing three mass spectrometers.

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7. X-RAY STRUCTURE

Many of the chemical reactions of interest to the Manhattan Project are carried out in the solid phase. The X-ray diffraction has been an invaluable tool in controlling and analyzing these dry reactions. It rapidly determines whether one or more phases are present, and it frequently furnishes the best means for determining phase composition and the amounts of the various compounds present.

The Ames Project started work on X-ray structure in March, 1942. The early problems were undertaken in cooperation with the metallographic group. Later, the studies were extended to include the field of inorganic chemistry, principally research on uranium, thorium, beryllium and the rare earths.

About 90 compounds or phases were thoroughly examined at Ames to obtain unit cell data; complete structures were worked out for 50 compounds. In many cases, chemical data were insufficient to determine the composition; even the formula of many of these compounds was first determined by X-ray studies. In several important instances, new compounds were discovered in whole or in part by the X-ray group; in many cases, important changes in the solid chemistry of uranium resulted from X-ray studies. A few of the more important contributions of the X-ray section are mentioned below.

All of the compounds of nitrogen with uranium were determined; not one nitride of uranium had been assigned the correct formula before these studies. The first demonstration was made that uranium hydride is a true compound, and its structure was determined; UO and U₂O₅ were discovered and the uranium-oxygen system revised. In cooperation with

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the metallographic group, UC was discovered, and the uranium-carbon system was classified. The monocarbide has a very high melting point, and, during various stages of the project, it was of special interest because of its physical properties and composition. The structure of gamma-uranium was determined. The structure of UF_3 , which is isomorphous with PuF_3 , was determined at a time when the composition of the latter compound and the valence of Pu were in doubt. A theory was developed of electron deficient bonding of importance in interstitial carbides, nitrides and certain hydrides such as UH_3 .

Thorium dihydride and the lower carbides and nitrides of thorium were discovered, and their structures were determined.

VI. PHYSICS

During the war, the Ames laboratory specialized in research in chemistry and metallurgy, and the work of these divisions was closely integrated with the Divisions of Chemistry, Metallurgy and Physics at the University of Chicago and elsewhere. Almost weekly conferences were held and the integration between the Ames group and the physics group was particularly close. As the war drew to a close, conferences became less frequent, and it became desirable to set up a physics Section at Ames, particularly because the Iowa State College physicists were returning from other war projects.

The Ames Physics Section was organized in the latter part of 1945 to further strengthen the fundamental approach to problems in chemistry and metallurgy and to pioneer in phases of nuclear physics of special interest to the Manhattan District. Two subsections were formed, one in

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theoretical physics and the other in experimental physics. The activity of the former group during 1946 was primarily devoted to consulting work within the local project. However, theoretical work was initiated on processes connected with the energy losses of heavy particles in their passage through matter.

The activity of the experimental physics group, during 1946, was largely devoted to the designing, constructing and testing of new equipment. Work was started on a curved crystal X-ray spectrograph to be used in the analysis of X-radiation and soft gamma-radiation accompanying radioactive decay. A major project involved the construction of an iron-free magnetic lens beta-ray spectrometer for precise studies on beta- and gamma-radiation. In this connection, it was found necessary to modify G-M counters and associated circuits. A 70,000,000-volt synchrotron was ordered, on College funds, and plans were made for a building to house the synchrotron, together with adjunct research and control facilities, and work was started on accessory equipment. It is expected that this equipment will be used in connection with Atomic Energy Commission projects.

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APPENDIX A. LIST OF REPORTS FROM THE AMES PROJECT

I. Publications for Cooperating Laboratories

Spedding, F. H., et al

Report for Week Ending April 25, 1942 - Chemistry and Metallurgy	CC-42 (In part)	4/25/42
Supplementary Report for Limited Distribution - Material from Report on Chemistry and Metallurgy	CC-43	4/25/42
Report for Week Ending May 23, 1942	CC-95	5/23/42

Wilhelm, H. A., D. Ahmann

Report for Week Ending June 13, 1942 Chemical Research	CC-123 (Part A)	6/13/42
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Wilhelm, H.A., L. B. Johns, W. H. Keller, E. Nettekoff, E. Rexner

Report for Week Ending June 5, 1942 Study of the Bismuth-Uranium System	CC-132	6/5/42
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Spedding, F. H., et al

Report for Week Ending June 30, 1942 Chemical Research	CC-148 (Part A)	6/30/42
Report of the Ames Chemical and Metallurgical Group for the Week of July 3, 1942	CC-176	7/2/42
Report of the Ames Chemical and Metal- lurgical Group for the Week of July 9, 1942	CC-177	7/9/42
Report of the Ames Chemical and Metallurgical Group for the Week of June 25, 1942	CC-178	6/25/42
Report of the Ames Chemical and Metallurgical Group for the Week Ending July 16, 1942	CC-205	7/16/42
Report for Month Ending August 15, 1942 - Chemical Research	CC-238 (Part E)	8/15/42
Report for Month Ending September 15, 1942 - Chemical Research	CC-259 (Part F)	9/15/42

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Johns, I. B., A. S. Hexton, W. E. Sullivan

The Diffusion of Fission Products
from Polished and Unpolished Metal
at 500°C CC-390

Spedding, F. H., et al

Chemistry of 94	GN-391 (Part C-III)	12/15/42
Technological Research	GT-393 (Part B)	12/15/42
Report for Month Ending December 15, 1942 - Production and Construction	GS-395 (Part C)	12/15/42
Report for Month Ending January 15, 1943 - Technological Research - Sect. II	GT-423 (Part B)	1/15/43
Report for Month Ending January 15, 1943 - Chemical Research - General and Radiochemistry	GC-418 (Part C)	1/15/43
Report for Month Ending January 15, 1943 - Chemistry of 94	GN-419 (Part C- II)	1/15/43
Report for Month Ending January 15, 1943 - Technological Research and Development - Sect. I	GT-422 (Part C)	1/15/43
Report for Month Ending January 15, 1943 - Production and Construction	GS-425 (Part D)	1/15/43
Index to Ames Metallurgical Reports (See also Index to Chemical Reports from Ames GT-438)	GT-431	1/14/43

Essel, V., A. D. Tsvobuch, J. G. Marx

Segregation of Impurities in a Typical In-
got of Cast Uranium In-GC-432 1/22/43

Marx, J. G.

Determination of Carbon in Uranium
and Uranium Compounds GC-433 1/26/43

Johns, I. B., A. S. Hexton, A. F. Veigt, W. E. Sullivan

Preliminary Survey of a Thermal Method for Removing
Fission Products, Developed by the Ames Chemical
Group GN-437

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Spedding, F. H. et al

Index of the Ames Chemical Group through CC-438 1/1/43
January 1, 1943 (See also Index of
Metallurgical Reports OT-431)

Butler, T. A.

Report on Recasting Uranium Metal in Air OT-456

Fussel, V.

Spectrophotometric Determination of CC-480 1/5/43
Iron in the Presence of Uranium

Spedding, F. H. et al

Report for Period Ending February 18, 1943 CC-465 (Part C) 2/18/43
Chemical Research - General and Radio-
chemistry

Report for Period Ending February 18, CC-467 (Part 2/18/43
1943 - Chemistry of 94 C-II)

Report for Period Ending February 20, OT-490 2/20/43
1943 - Ames Metallurgical and Metal-
lographic Groups

Report for the Month Ending March 15, 1943 CC-521 3/15/43
Chemical Research - Radiochemistry

Report for Month Ending March 15, CC-522 (Part C) 3/15/43
1943 - Chemistry of 94

Report for Month Ending March 15, CC-523 (Part C) 3/15/43
1943 - Chemical Research - Analytical

Report for Month Ending March 27, 1943 OT-542 3/27/43
Technological Research - Metallurgy

Report for Month Ending April 15, 1943 CC-578 4/15/43
Chemical Research - Chemistry of 94

Marl, J. C., A. S. Newton, T. Butler, J. A. Ayres, I. B. Johns

Uranium Hydride CC-580 4/15/43

Johns, I. B., A. S. Newton

The Hydride Reaction as a Means of OT-583 4/5/43
Testing the Continuity of Coatings

Spedding, F. H., et al

Report for Month Ending April 19, 1943 - Chemical Research - General and Radiochemical CC-587 4/19/43

Johns, I. B., A. S. Newton, V. H. Sullivan, A. F. Veit

The diffusion of Fission Products from Cast Uranium at 1000°C CC-594 4/43

Wark, J. G.

Spectrophotometric Determination of Calcium to be Adapted to the Determination of Calcium in Uranium Metal CC-607 4/27/43

Spedding, F. H., et al

Part II of Report for Month Ending April 24, 1943 - Technological Research - Metallurgy CC-609 4/24/43

Johns, I. B., A. S. Newton, V. H. Sullivan, A. F. Veit

The diffusion of Fission Products from Uranium at 1185°C CC-616 5/43

Spedding, F. H., et al

Report for Month Ending May 15, 1943 Chemical Research - General and Radiochemical CC-654 5/15/43

Report for Month Ending May 15, 1943 Chemical Research - Chemistry of U4 CN-678 5/15/43

Report for the Month Ending May 15, 1943 Chemical Research - Analytical and General CC-682 5/15/43

Part II of Report for Month Ending May 22, 1943 - Technological Research - Metallurgy CC-686 5/22/43

Newton, A. S.

Production of Deuterium and Uranium Deuteride from Heavy Water CC-695 5/26/43

Johns, I. B., A. S. Newton, W. H. Sullivan, A. E. Valet

The Diffusion of Fission Products from Uranium (Errata) CC-700

Newton, A. S., O. Johnson, A. Kent, E. V. Kottorf

The Preparation of Trivalent and Tetravalent Compounds of Uranium from the Metal CC-705 6/7/43

Spedding, F. H., A. E. Newton, O. Johnson

The Dry Fluoride Process for separating U₄ CE-717 6/17/43

Spedding, F. H., et al

Report for Month Ending June 15, 1943 CC-725 6/15/43
Chemical Research - General and Radiochemical

Report for the Month Ending June 15, 1943 CC-726 6/15/43
Chemical Research - Analytical

Report for Month Ending June 15, 1943 CE-727 6/15/43
Chemical Research - Chemistry of U₄

Part II of Report for Month Ending June 24, 1943 - Technological Research - Metallurgy OT-751-A 6/24/43

Part II of Report for Month Ending June 24, 1943 - Technological Research - Metallurgy OT-751-B 6/24/43

Part II of Report for Month Ending June 24, 1943 - Technological Research - Metallurgy OT-751-C 6/24/43

Nevez, T. B., W. H. Sullivan, C.D. Corvill, A.S. Newton, E.B. Sleight, O. Johnson

The Beta and Gamma Radiations from the Chain T_{1/2} 77 h Fe 2.4 h I (Joint publication of Chicago and Ames) CC-763 5/15/43

Sullivan, W. H.

Chemical Methods for the Separation of the Fission Products CC-765 6/30/43

Spedding, F. H., et al

Report for Month Ending July 15, 1943 Chemical Research - Analytical	CC-794	7/15/43
Report for Month Ending July 15, 1943 Chemical Research - Chemistry of 94	CM-795	7/15/43
Report for Month Ending July 15, 1943 Chemical Research - General	CC-803	7/15/43
Part II of Report for Month Ending July 24, 1943 - Technological Research - Metallurgy	CT-815	7/24/43
Report for Month Ending July 15, 1943 Chemical Research - Radiochemistry	CC-837	7/15/43
Report for Month Ending August 8, 1943 Chemical Research - Chemistry of 94	CM-852	8/8/43
Report for Month Ending August 7, 1943 Chemical Research - General	CC-858	8/7/43
Report for Month Ending August 8, 1943 Chemical Research - Analytical	CC-862	8/8/43
Part II of Report for Month Ending August 21, 1943 - Technological Research - Metallurgy	CT-891	8/21/43
Report for Month Ending September 15, 1943 - Chemical Research - Radio- chemistry	CC-921	9/15/43
Report for Month Ending September 8, 1943 - Chemical Research - Chemistry of 94	CM-925	9/8/43
Part II of Report for Month Ending October 2, 1943 - Technological Research - Metallurgy	CT-954	10/2/43
Part II of Report for Month Ending October 24, 1943 - Technological Research - Metallurgy	CT-1007	10/24/43
Report for Month Ending October 8, 1943 - Chemical Research - Chemistry of 94	CM-1048	10/8/43

Report for Month Ending November 6,
1943 - Chemical Research -
Analytical

CC-1057

11/6/43

Butler, T. A., A. F. Yeist, E. J. Volter, J. A. Ayres, E. E. Hain,
A. D. Sorehaugh, I. E. Johns

Review of the Metallurgical Properties of CM-1058
Tracer Plutonium to October 15, 1943 -
Chemical Research Chemistry of
Plutonium

10/15/43

Spedding, F. H., et al

Report for Month Ending October 9,
1943 - Chemical Research -
Radiochemistry

CC-1059

10/9/43

Report for Month Ending November 6,
1943 - Chemical Research - Chemistry
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	Production of Uranium by Reduction of UF_4	12A & B

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<u>Author</u>	<u>Title</u>	<u>Volume</u>
Keller, W. H.	Small Scale Production of Uranium by Stationary Bomb	12A
Spedding, F. H., H. A. Wilhelm, C. F. Gray, A. Boyt	Developments in Cutting and Machining Uranium at the Iowa State College Laboratory	12B
Spedding, F. H., H. A. Wilhelm, C. F. Gray	Early Methods for Casting Uranium at Iowa State College	12B
Spedding, F. H., H. A. Wilhelm, M. Hull, C. F. Gray	Machining Graphite and Special Operat- ions	12B
Spedding, F. H., H. A. Wilhelm, C. F. Gray, E. Thompson	Present Practices in the Uranium Foundry at Iowa State College	12B
Spedding, F. H., H. A. Wilhelm, W. Calderwood, C. F. Gray	Radiation Shields and their Fabrica- tion	12B
Spedding, F. H., H. A. Wilhelm, C. F. Gray, A. J. Boyt	Recovery of Uranium Turnings, Chips and Borings.	12B
Spedding, F. H., H. A. Wilhelm, C. F. Gray, E. Thompson	Small Scale Casting of Uranium	12B
Ahmann, D., A. I. Snow, A. S. Wilson	The Uranium-Molybdenum Binary System	12B
Wilhelm, M. A., A. H. Daane, J. H. Carter, A. I. Snow	The Uranium-Carbon System	12B
Carlson, O. H., J. H. Carter, H. C. Maensiger	The Uranium-Copper System	12B
Daane, A. H., A. S. Wilson	The Uranium-Chromium System	12B
Treick, D. A., J. H. Carter A. I. Snow, R. R. Baldwin, A. S. Wilson	The Uranium-Tin System	12B
Carlson, O. H., A. H. Daane, A. S. Wilson	The Uranium-Thorium System	12B
Warf, J. C.	Analytical Chemistry of Uranium (Chapter 14 - Div. 8 of MPT5) A.25	12A

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<u>Author</u>	<u>Title</u>	<u>Volume</u>
Warf, J. C.	Analytical Chemistry of Thorium (Chapter 16 - Div. 6 of MPTS)	13A
Fassel, V. A.	Procedures for Analysis of Project Materials Calcium, Magnesium, Lime	13A
Warf, J. C.	Pyrohydrolysis	13A
Tevebaugh, R. D.	The Analysis of Certain Metals for Trace Quantities of Fluorine	13B
Banks, Charles V.	Analysis of Certain Uranium Alloys	13B
Warf, J. C.	Analysis of Metallic Uranium for the Free Element	13B
Erisson, R. P., E. J. Fernefeld	Determination of Iron, Thorium, Beryllium, Cerium, Calcium, Magnesium, and their Compounds	13B
	General Discussion - Production of Uranium, Raw Materials, Purity Specifications, etc.	13
	Solution of Uranium and its In- soluble Compounds	13
	Procedure for the Analysis of Uranium Tetrafluoride for Fluorine by the Ferric Nitrate Method	13
Deane, A., A. Wilson	Note on the Preparation of Uranium- Cadmium Alloys	12
Baldwin, R., H. Baensiger	Note on the Uranium-Zinc System	12
	Precipitation of Uranium with Hy- drogen Peroxide	
Johns, I. B., A. S. Newton, A. W. E. Sullivan, A. F. Veigt, J. A. Ayres, F.A. Butler, E. M. Gladrow, R. E. Hein, A. Kant, R. W. Kottorf, N. E. Sleight, A. D. Tevebaugh, J. C. Warf, F. J. Wolter	A Systematic Chemical Procedure for the Separation of the Fission Products	13B

<u>Author</u>	<u>Title</u>	<u>Volume</u>
Fassel, V. A.	The Direct Spectrophotometric Determination of Iron in Uranium and Its Compounds	13B
Fassel, V. A.	The Spectrographic Analysis of High Purity Magnesium Metal	13B
Voigt, A. F., E. R. Sleight R. E. Hein, J. M. Wright	A Tracer Study of the Valence States of Neptunium	14B
Kant, A., A. F. Voigt, J. M. Wright	The Co-Precipitation of Plutonium with Salts of Organic Acids	14B
Voigt, A. F., A. Kant, E. R. Sleight, R. E. Hein, J. M. Wright, F. J. Volter, H. D. Brown	The Separation of Pu ^{IV} and Pu ^{III}	14B
(Voigt, A. F., editor)	Hydride Intermediate Process	16A
Kant, A., A. F. Voigt	Separation of Plutonium from Rare Earth Fission Products by Precipitation of Salts of Organic Acids	16B
Spedding, F. H., A. S. Newton, O. Johnson, A. D. Tevebaugh, K. Walsh, I. E. Johns, F. Vaslow, R. Fisher, J. A. Ayres, J. Iliff, W. Keller	Some Studies on the Improved Dry Fluoride Process	16B
Brown, H. D., F. J. Volter	Decontamination Studies Involving the Extraction of Organic Complexes of Plutonium	16B
Spedding, F. H., I. E. Johns, A. F. Voigt, T. A. Butler, F. J. Volter, J. A. Ayres, R. E. Hein, A. D. Tevebaugh, E. M. Gladrow, O. Johnson, A. Kant, R. W. Mottorf	Separation Processes Involving Alloy Formations	16B
Spedding, F. H., I. E. Johns, H. A. Wilhelm, A. S. Newton, W. H. Sullivan, A. F. Voigt, J. A. Ayres, T. A. Butler, E. M. Gladrow, R. E. Hein, O. Johnson, A. Kant, G. Moulton, R. W. Mottorf, E. R. Sleight, A. D. Tevebaugh, J. C. Warf, F. J. Volter	The Removal of Fission Products from Uranium and Plutonium by Heat Treatment	16B

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<u>Author</u>	<u>Title</u>	<u>Volume</u>
Wilhelm, E. A., A. S. Newton, A. E. Deane, C. Neher	Thorium Metallurgy	17A
Wolter, F. J., H. D. Brown, J. M. Wright	Studies on the Organic Complexes of Plutonium	19B

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APPENDIX B

Typical Examples of Service Work Done at Ames
for Other Sites of the Manhattan District

The Ames laboratory has been very successful in producing pure chemicals, rare metals, very pure metals, and specially fabricated objects when it has been difficult or impossible to obtain them from ordinary commercial channels for use at other sites. The Ames laboratory does not wish to become a service laboratory, however, it is felt that it is worthwhile, in the interest of the national welfare and safety, to do a certain amount of this type of work providing it does not become so burdensome as to threaten the stability of Ames as a research laboratory. Much of this work requires the services of research men of outstanding ability. While these men are willing to do a certain amount of service work, they are not willing to devote a major part of their time to it. They feel quite rightly that they have made major financial sacrifices in order to stay in the academic atmosphere in which they can do fundamental research. They feel that, if they have to devote the major part of their time to service work, they might better resign and go into industry where the financial rewards are greater.

The nature of these service demands is varied and it is very difficult to say what the demands will be in the future since they usually arise suddenly and are supplied in a relatively short time. The only thing about which one can be certain is that a number of services requests will be received each month. A few typical examples of services rendered, to other sites of the Manhattan District, by the Ames

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Project are listed below.

Supplied about 2,000,000 pounds of uranium metal, largely as billets but also considerable amounts in various specially shaped objects. Some special uranium disks were made from depleted uranium salt (almost pure U^{238}) for use as targets in cyclotron studies.

Supplied about 4500 pounds of pure thorium metal largely in the form of billets; in a number of cases the metal was made in special shapes. A number of machined thorium disks were prepared for use in cyclotron bombardment studies.

Supplied about 450 pounds of highly pure cerium metal in the form of billets.

Supplied a number of specially fabricated objects of other materials such as lanthanum metal, beryllium metal and special alloys. A beryllium ingot was prepared from radioactive beryllium salts.

Ground several hundred pounds of calcium metal and manufactured fluorine gas which was shipped in cylinders.

Fabricated and supplied ceramic bodies such as crucibles, high temperature defining-slots, rods and filaments made of such materials as CaO, MgO, BeO, U_2O , UO_2 , UO, UC, U_2O_3 , UC_2 , UN, TaN, ZrN, IrC, TiN, TiN, W_2C , WC, TaC, TiC, GbC, and MoC.

Supplied samples of extremely pure chemicals such as UBr_4 , UBr_3 , UF_3 , UI_3 , UC, UN, $UOCl_2$, $UOBr_2$, KUF_5 , $KUCl_5$, HbF, CaO, rare earths, anhydrous $TbCl_4$, TbO_2 , TbF_4 , diethyl zinc, dimethyl zinc, dialcylalthylenediamine and α -naphthohydroxamic acid.

B. 2

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Made metallurgical examination and conducted corrosion studies of tantalum filaments, stainless steel sheets and other parts from Y-12 apparatus subjected to corrosion. Studied the depth of penetration of uranium metal into walls and parts of a calutron.

Prepared specially bonded bi-metallic strips of copper and uranium.

Studied the applicability of the multisource unit for the spectrographic determination of boron in calcium nitrate solutions.

Made analytical studies of the effectiveness of the removal of silicon, phosphorus and zirconium from uranium content by hydrofluorination of ore samples supplied by U. S. Geological Survey.

Participated in a number of analytical programs in which standard samples had to be completely analyzed by various methods and the results compared with those of other laboratories to establish best analytical procedure.

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APPENDIX C

List of Scientific Personnel
of the
Ames Project under the Manhattan District

One of the outstanding characteristics of the Ames Project was the wholehearted and unselfish cooperation of the entire group. Without this teamwork the success of the project would have been impossible under the stress and strain of the urgency of the war effort. A list of scientific personnel of the Ames Project, under the Manhattan District, follows:

<u>Name</u>	<u>Title</u>	<u>Appointed</u>	<u>Resigned</u>
Ahmann, Donald H.	Junior Chemist	6/42	
Anderson, Charles H.	Research Assistant	9/42	
Ayers, Augustus Sidney	Junior Chemist	7/42	
Ayers, Euell O.	Associate Chemist	1/42	
Ayers, John A.	Chemist, Section Chief	6/42	5/47
Baensiger, Norman C.	Associate Chemist	12/42	
Baker, Richard D.	Research Associate	9/42	6/43
Baldwin, Robert H.	Associate Chemist	6/42	12/42
Banks, Charles V.	Chemist	2/42	
Basson, John B.	Junior Chemist	12/42	12/44
Beyt, Arthur J.	Research Assistant	3/42	
Breaklander, Laurence	Junior Research Assistant	9/42	4/43
Brown, Herase D.	Junior Chemist	1/44	4/42
Bruse, Clarence V.	Junior Research Assistant	3/42	6/42
Buchner, E. C.	Associate Chemist	7/42	9/42
Butler, Tom A.	Junior Chemist	6/42	
Byrd, Dorwin	Research Assistant	4/42	6/42
Caldewood, Wayne, Jr.	Research Assistant	1/42	6/42
Calkins, Vincent P.	Junior Chemist	7/42	9/44
Carlson, John F.	Physicist	7/42	
Carlson, Oscar H.	Junior Chemist	6/42	
Carter, James Hal	Research Associate, Group Leader	11/42	9/42
Chiotti, Preme	Associate Chemist	2/42	
Oline, Donald	Junior Chemist	1/44	4/42
Coobs, John H.	Research Assistant	9/42	9/44

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<u>Name</u>	<u>Title</u>	<u>Appointed</u>	<u>Resigned</u>
Daane, Adrian	Associate Chemist, Section Chief	2/42	
Doigan, Paul	Research Assistant	9/46	12/46
Donohue, Ruth (Mrs. A. Terebaugh)	Associate Chemist	1/43	12/44
Edwards, Frank O.	Junior Chemist	2/44	
Ericson, Robert P.	Junior Chemist	2/45	3/45
Eving, Richard	Junior Research Assistant	6/45	
Fassel, Velmer A.	Chemist	11/42	
Fassel, Mary	Analyst	9/43	
Feibig, Joseph	Research Assistant	2/45	6/45
Figard, Paul H.	Junior Chemist	3/43	5/45
Fisher, Ray V.	Junior Chemist	1/43	
Fornfeld, Eugene J.	Research Assistant	2/45	9/45
Fornfeld, Elsie M.	Research Assistant	9/44	4/45
Fulmer, Ellis I.	Assistant to the Director	7/45	
Fulmer, Robert	Research Assistant	12/42	1/45
Gladrow, Elroy	Assistant Chemist	6/42	9/47
Gerald, Park S.	Junior Research Assistant	9/42	6/43
Gobush, Michael	Research Assistant	3/43	7/47
Gray, C F	Associate Chemist, Section Chief	4/42	2/45
Hach, Clifford	Junior Chemist	5/44	10/45
Handlin, Louis	Junior Chemist	1/45	6/45
Hayes, Anson W.	Research Assistant	9/45	
Heidel, Robert	Junior Chemist	7/45	
Kein, Richard E.	Junior Chemist	9/42	
Komberg, Robert W.	Research Assistant	3/44	10/44
Hexeng, Raymond	Research Assistant	2/43	
Huke, Frank	Junior Chemist	9/45	
Hunt, Elton	Research Assistant	5/45	
Iliff, James	Junior Research Assistant	6/42	6/45
Jacobsen, Theodore R.	Research Assistant	5/43	9/43
Jensen, Erling	Assistant Physicist	7/45	
Johas, Iral B.	Associate Director	3/42	2/44
Jones, Vernal A.	Junior Research Assistant	6/43	8/44
Johnson, Marlan	Research Assistant	9/45	
Johnson, Oliver	Associate Chemist	1/43	4/45
Kant, Arthur	Junior Chemist	6/42	6/45
Keller, Joseph M.	Physicist, Section Chief	8/45	
Keller, Wayne H.	Senior Chemist	6/42	10/45

<u>Name</u>	<u>Title</u>	<u>Appointed</u>	<u>Resigned</u>
Knipp, Julian	Physicist	7/46	
Koontz, Philip	Physicist	3/48	8/48
Larson, Jane	Junior Research Assistant	6/46	9/47
Leslett, Lawrence J.	Physicist	1/48	
Leashman, Robert	Junior Physicist	6/46	
Lagvold, Sam	Physicist	11/46	
Lipkind, Henry	Associate Chemist	2/46	2/48
Lyon, Ward	Research Assistant	9/42	7/47
McClure, John H.	Research Assistant	12/46	
McDonald, Richard	Junior Research Assistant	12/42	6/46
Martin, Donald	Chemist	9/46	
Maxwell, Elizabeth S.	Junior Research Assistant	2/46	7/43
Mculten, George H.	Junior Chemist	4/42	1/44
Neher, Charles	Associate Chemist	2/46	3/48
Newton, Anne S.	Chemist, Section Chief	6/42	4/48
Netterf, Robert W.	Associate Chemist	3/42	6/46
Noyce, William K.	Associate Chemist	2/44	9/46
Olesen, Mary Lou	Junior Research Assistant	4/44	9/44
Patterson, James H.	Junior Chemist	2/44	
Peterson, David	Research Assistant	12/42	
Petersen, Daniel W.	Research Assistant	8/43	9/44
Pflueger, Willard	Research Assistant	9/46	
Pierick, Edward G.	Research Assistant	9/42	11/47
Porter, Paul E.	Junior Chemist	2/46	
Powell, Jack E.	Junior Chemist	4/43	
Powers, Richard	Research Assistant	7/46	
Pratt, William W.	Research Assistant	9/46	
Raouche, Richard F.	Junior Chemist	2/44	
Reed, Sherman A.	Research Assistant	9/46	
Rundle, Robert	Senior Chemist, Section Chief	5/42	
Sleight, Herman A.	Associate Chemist	6/42	2/46
Smith, Albert Lee	Junior Chemist	4/43	10/47
Smith, Francis C.	Junior Research Assistant	1/44	5/46
Smith, Robert	Research Assistant	2/46	6/46
Snow, Adolf	Junior Chemist	1/43	
Spedding, Frank H.	Director	2/42	
Spencer, Walter	Research Assistant	9/46	
Stokes, Richard H.	Research Assistant	9/46	
Stosick, Arthur J.	Chemist	2/46	8/47
Sullivan, William H.	Chemist	6/42	2/44

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<u>Name</u>	<u>Title</u>	<u>Appointed</u>	<u>Resigned</u>
Svec, Harry	Junior Chemist	5/44	
Tevebaugh, Arthur D.	Associate Chemist	6/42	10/47
Thamer, Burton	Research Assistant	7/45	
Thompson, Richard	Junior Research Assistant	9/42	8/47
Treick, Dalton A.	Junior Chemist	5/43	8/45
Tucker, Walter D.	Associate Chemist	1/45	9/47
Vaslov, Fred	Junior Chemist	3/43	6/45
Voigt, Adolf	Chemist, Section Chief	7/42	
Veter, Roger	Research Assistant	8/45	
Walsh, Kenneth A.	Research Assistant	4/43	
Warf, James C.	Chemist, Section Chief	6/42	9/47
Wilhelm, Harley A.	Associate Director	2/42	
Wilkinson, John A.	Senior Chemist	7/42	9/42
Williams Wayne	Junior Research Assistant	2/45	6/45
Wilson, Archie	Research Assistant	1/43	6/45
Wimmer, Eugenie E.	Junior Research Assistant	1/44	10/44
Wolter, Frederick	Associate Chemist	4/42	6/45
Wright, James M.	Junior Chemist	1/44	