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ISOLATION AND DETERMINATION OF SUBMICROGRAM QUANTITIES  
OF THE HEAVY ELEMENTS FROM GROSS FISSION PRODUCTS

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Project 2228

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PREFACE

The project, designated as Project No. 2228 of the Engineering Research Institute of The University of Michigan, was sponsored by the Air Force Cambridge Research Center, which is part of the Air Research and Development Command of the U.S. Air Force. The project was initiated by the Atomic Warfare Directorate of the Cambridge Center, through Dr. Bernard Manning. Soon after work started, the supervision of the project was shifted to Dr. Arthur Kant of the Cambridge Center. Responsibility for the project was later shifted to the Electronics Research Directorate of the Cambridge Center. Toward the latter stages of the project, Dr. Bernard Rubin became Contract Monitor.

The work of the project was carried on in the Chemistry Building of The University of Michigan under the supervision of Professors Philip J. Elving and Charles L. Rulfs of the Department of Chemistry. Dr. Anil K. De was associated with the project as a full-time research associate from July 1, 1954, to August 31, 1955. Part-time workers on the project included John L. Griffin, Julian Lakritz, Daniel J. Macero, Herman Wissenberg, and Alan L. Woodman, all of whom are graduate students in the Department of Chemistry, and Robert Stenger and Janet L. Kochanny, undergraduate students majoring in chemistry.

## ABSTRACT

The project was concerned with the determination of minute amounts of uranium, using simple equipment and requiring only moderate amounts of time. In particular, attention was focused on the separation and determination of very small amounts of uranium when present in admixture with large amounts of radioactive fission products. In achieving the objective, a number of lines of development were followed.

A bibliography and a critical review of the analytical chemistry of uranium were prepared; all declassified Atomic Energy Commission reports were included. Special attention was given to the various ways in which uranium might be isolated from very small amounts of samples and to technics which lend themselves to automatic or semiautomatic manipulation with the minimal introduction of chemical reagents and solvents.

During the preparation of the above-mentioned review, laboratory investigation was started on the isolation of uranium at the milligram, microgram, and submicrogram levels. Based on the consideration of all factors involved and preliminary studies, emphasis was placed on the separation of uranium by liquid-liquid extraction of the complex compounds formed by uranium with certain types of organic molecules; specifically investigated were oxine (8-quinolinol or 8-hydroxyquinoline) and cupferron (N-nitrosophenylhydroxylamine). The latter in particular seemed to possess a considerable number of possibilities for the efficient separation of uranium, since it formed a stable complex with uranium in one of that element's two common states of oxidation, but not with the other.

Once the uranium had been separated from other constituents, the amount of uranium present, when operating at the milligram uranium level, was measured by colorimetric comparison. When operating with microgram and smaller quantities of uranium, the radioactive uranium-233 isotope was used and the amount of uranium present was measured by counting the alpha particles emitted over a given period of time.

The most feasible way of changing the oxidation state of uranium in solution without introducing reagents was found to be an electrolytic process in which the uranyl ions, containing uranium in an oxidation state of six, are

reduced at a mercury cathode to uranium ions in which uranium has an oxidation state of four.

The actual process finally developed for isolating uranium is relatively simple from the manipulative standpoint, although some moderately complex chemistry is involved. Fundamentally, the process consists of adding to an aqueous solution of the sample an excess of complexing agent. The particular agent selected, cupferron, is one which will not react with uranium(VI) but will react with many other metals, including uranium(IV), to form stable complex species. The aqueous solution is extracted with an organic solvent, ether, which removes the metal complexes from it. The residual aqueous solution, which contains the uranium, is now electrolyzed, while an ether-cupferron solution is added to form a separate upper layer. As the uranium is reduced by electrolysis, it forms a stable chelate species with the cupferron, which is extracted into the ether layer. The other metal ions, which are still present since they were also originally unextracted, are not extracted with the uranium, since either the electrolysis does not reduce them or their lower oxidation states do not form extractable species. A carrier may be added if the uranium in the sample is at the submicrogram level.

The whole process involving the preextraction, the electrolysis, and the uranium extraction is carried out in the same relatively simple piece of glass apparatus.

The uranium then can be determined photometrically, if as much as a milligram is present. In the case of microgram amounts, the uranium, after suitable chemical treatment, is electroplated onto a small platinum disk, whose radioactivity in terms of alpha emission is then measured with a flow counter.

The procedure has been applied to the recovery of as little as  $10^{-7}$  to  $10^{-8}$  grams of uranium-233 from admixture with fission products which themselves have high levels of alpha, beta, and gamma emission.

## OBJECTIVES

The project was primarily concerned with the isolation and determination of microgram and submicrogram quantities of heavy elements, in particular uranium, when present in admixture with fission products. Emphasis was to be placed on the separation of uranium by extraction of the chelate species which it forms with organic molecules. Measurement at microgram and submicrogram levels of uranium was to be made through the use of uranium-233 tracer and alpha counting; at higher uranium levels, photometric measurement was to be utilized. In particular, attention was to be focused on the development of a procedure for the separation and determination of very small amounts of uranium when present in admixture with large amounts of radioactive fission products as well as other chemical elements, which would require simple equipment and only moderate amounts of time. In addition, special emphasis was to be placed on the various ways in which uranium might be isolated from very small amounts of samples and on technics which lend themselves to automatic or semiautomatic manipulation with the minimal introduction of chemical reagents and solvents. Such automatization of analytical processes has obvious advantages and often is an absolute necessity when dealing with such highly radioactive samples as those in which uranium occurs in admixture with fission products.

The manner in which the objectives of the project were met will be discussed in the following sections of the report. The latter divisions are, in some cases, more or less arbitrary, but it is believed that such division into the various lines of activity followed on the project will be helpful in evaluating the work done.

Since all the results obtained as a consequence of work on the project have been or will be described in scientific papers intended for publication in journals and reprint reproduction, only the motivation for the work done, the general plan of attack, the most pertinent results obtained, and the general significance of the work done will be described.

## I. REVIEW OF THE ANALYTICAL CHEMISTRY OF URANIUM

Although uranium has long been known and its analytical chemistry has received considerable attention, research on uranium and its chemistry received tremendous impetus with the initiation of the atomic energy project in the United States of America in 1941. The continuing and actually increasing interest in uranium as a fissionable material and consequent source of energy for both military and nonmilitary purposes has resulted in its being one of the most vigorously investigated of the elements. Naturally, the analytical chemistry of uranium has received tremendous attention. The determination of uranium in the concentrational range from the most minute trace levels to practically 100% pure metal has been and is being studied.

To aid in orienting the project work, the existing literature on the analytical chemistry of uranium and related areas, with emphasis on the work reported during the past decade, has been critically resurveyed with respect to certain of the objectives of the project: (1) the separation and determination of uranium, (2) the isolation and purification of uranium from microgram and submicrogram samples of complex mixtures of fission products, and (3) techniques, such as extraction, which lend themselves to automatic or semi-automatic remote-control manipulation involving a minimal introduction of reagents and solvents.

This literature survey for the work reported since 1947 for the determination of uranium has been based on reports and papers abstracted in Nuclear Science Abstracts and in the antecedent publication, Abstracts of Declassified Documents. About three hundred abstracts were found. The work up to 1947 has been well covered in Rodden's book, Analytical Chemistry of the Manhattan Project, and in other publications resulting from the Manhattan Project.

Cross references to the work on the analytical chemistry of uranium before and since 1947 were obtained from Croxton's comprehensive bibliography on uranium chemistry as well as from the standard reference books dealing with the analytical chemistry of uranium, including those by Fresenius and Jander and Hillebrand, Lundell, Bright, and Hoffman.

All the major areas, with the exception of radiometric techniques, were covered, including gravimetric, titrimetric, electrometric, and photometric methods; the omission was due to the existing specialized literature

available on radiometric determination of uranium.

The review of the analytical chemistry of uranium turned out to be more extensive than originally planned. However, it was felt that a more or less critically developed systematic review of the current status of the analytical chemistry of uranium would fill a definite void in the chemical literature on uranium. A manuscript, based on the literature survey described, is being prepared for submittal for consideration for publication. The manuscript, after clearance for publication by the Air Force Cambridge Research Center and the University of Michigan Engineering Research Institute, will be submitted to Nucleonics or some other journal where it might reach the desired audience.

## II. EXTRACTION OF URANIUM BY 8-QUINOLINOL

In view of the literature references on the separation of uranium as the oxine (8-hydroxyquinoline or 8-quinolinol) compound, it was deemed desirable, as the result of discussion with the Contract Monitor at the Cambridge Center, to make a systematic and critical investigation of the use of 8-quinolinol and its available derivatives. Complete information about 8-quinolinol in this respect was lacking, and no work on its derivatives had been reported.

Consequently, the extraction of uranium at the milligram level over the usable pH range from buffered aqueous solution into chloroform solutions of 8-quinolinol, 5,7-dichloro-8-quinolinol, and 5,7-dibromo-8-quinolinol was investigated in conjunction with the direct photometric determination, by absorption spectrophotometry, of the uranium present in the chloroform extract.

8-quinolinol seems to be a better extracting agent than its dichloro and dibromo derivatives. The pH ranges for 90% or greater extraction are 5.8 to 8.0 for uranyl oxinate, 5.4 to 7.2 for the dichlorooxinate, and 5.6 to 7.3 for the dibromooxinate; maximum extraction occurs at pH 7.0 (98%,  $K_D = 85$ ), 6.0 to 6.6 (92%,  $K_D = 20$ ), and 5.6 to 7.2 (90%,  $K_D = 16$ ), respectively. This is to be compared to the pH range of 7 to 9 previously recommended for uranyl oxinate. There are significant drops in percentage extraction above pH 8. Above pH 12, the aqueous layers become yellow and turbid; the corresponding chloroform layers are colorless and exhibit no absorption.

The results obtained by different workers at different times were generally reproducible to within  $\pm 1\%$ , except for those at pH 2 where the extent of extraction was small and the reproducibility was within  $\pm 5\%$ . The total operations for an oxine extraction required about 15 to 20 minutes.

The chloroform solutions obtained were bright orange in color;



measurements of absorbances, made at 430  $m\mu$  for the oxinate and 420  $m\mu$  for the dihalooximates, followed Beer's law up to 0.03 mg of uranium per ml in a 1-cm cell. As little as 0.003 mg of uranium per ml can be measured. The reproducibility of the absorbance measurement for a given uranium solution was very satisfactory, being about  $\pm 0.001$  for average absorbances of 0.352 and 0.356. A more stringent test of the reproducibility is reflected in the concurrent results obtained by several different individuals working with two different Beckman spectrophotometers.

The extraction of oxine, 5,7-dichlorooxine, and 5,7-dibromooxine (0.004% solution in chloroform) at various pH values from 2 to 12 was followed spectrophotometrically in the ultraviolet region and gave results which correlated satisfactorily with the known pK values of the compounds. The suitable pH ranges for extraction of the oxines themselves are 4 to 8, 3 to 7, and 3 to 7, respectively.

Some attention was given to the effect made on the extraction by such variables as solvent, temperature, and complexing ions in the aqueous phase.

A manuscript was prepared in the form of a scientific paper which summarized the work reported in the earlier submitted rough draft of Scientific Report No. 3 (ERI Report No. 2228-5-T) as well as that indicated in the previous paragraphs. This manuscript, after it had been approved for release for publication by the Air Force Cambridge Research Center and by the University of Michigan Engineering Research Institute, was submitted to the journal, Analytical Chemistry, for consideration for publication. It has since been accepted and should appear in the late fall of 1955.

### III. STUDIES ON THE ELECTRODEPOSITION OF URANIUM

Since radiochemical counting is considered the most sensitive method for the identification and quantitative determination of submicrogram quantities of radioactive substances, recourse would naturally be made to the counting method for the determination of trace amounts ( $10^{-6}$  grams or less) of uranium-233 ( $\alpha$  4.82 mev;  $t_{1/2} = 1.63 \times 10^5$  y) after isolation of the uranium from other elements. Furthermore, since it is possible by electrodeposition onto platinum to obtain very thin films of uranium which are suitable for counting, the separation of uranium as a deposited layer on a small platinum disk or planchet of about one-inch diameter possesses many advantages for isolating milligram, microgram, and submicrogram quantities of uranium.

Previous investigators have plated milligram quantities of uranium on platinum. It was felt that the electrodeposition procedure for microquan-

tities of uranium, which had been developed at the Cambridge Center, could be improved, especially since it might not necessarily apply at the submicrogram level. With this objective in mind, the electrodeposition of uranium from various media under various conditions was undertaken.

The work was initially handicapped in that the workers on the present project had only a verbal account of the final Cambridge Center electrodeposition procedure, which was communicated on a visit to the Cambridge Center at the end of May, 1954. The report of the work done at the Cambridge Center was, unfortunately, not made available to the present project until early August, 1955, about one week before experimental work on the project stopped. However, the report was valuable in indicating that our final procedures would be helpful.

Qualitative studies, prior to the acquisition of uranium-233 which permitted accurate microgram- and submicrogram-level studies, indicated that as little as about  $10^{-10}$  grams of uranium could be electroplated from two previously unused media, citrate and tartrate. Other possibilities, including the electroplating of the uranium on aluminum-zinc disks, and electroplating from solutions of uranium—organic-compound systems, such as uranium cupferrate and oxinate, in organic and aqueous-organic media, were considered and investigated.

The electrical resistance of various organic mixtures (e.g., a solution of ether, ethanol, 3% water, lithium chloride, and cupferron) was examined, attention being focused on such compositions as could be achieved readily, subsequent to the cupferron reduction-extraction procedure described in a later section of this report. Media of moderately good conductance were found, but no electrodeposition of uranium from uranium(IV/III) cupferrate solution in such media could be realized.

Qualitatively, however, a uranium electrodeposition from the aqueous buffer mixture usually used has been observed, when in the presence of excess cupferron decomposition products and abnormally large amounts of nitrate ion.

In order to establish experimental conditions and techniques, orienting studies on the electrodeposition of 1- to 2-milligram quantities of natural uranium were made in which the quantitative nature of the plating was checked by counting alphas, using a windowless flow counter. A method was established for the quantitative deposition of the uranium from ammonium oxalate medium. The oxalate medium is superior to phthalate, citrate, and tartrate media, inasmuch as use of the latter media introduces some difficulty in pH control during electrolysis. Part of the difficulty is due to an apparent polymerization, or other reaction, of the organic matter, which gives rise to a very dark brown solution.

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This procedure has been extrapolated to the plating of quantities of uranium-233 of the order of  $10^{-8}$  grams. Quantitative deposition of such minute amounts of uranium has been obtained. The plates are very smooth, shiny, and adherent, showing bright interference colors.

A manuscript has been prepared in the form of a scientific paper which summarizes the work on and the procedures developed for the electrodeposition of uranium as well as the results reported in the earlier submitted rough draft of Scientific Report No. 1 (ERI Report No. 2228-3-T). This manuscript, after it has been approved for release for publication by the Air Force Cambridge Research Center and the University of Michigan Engineering Research Institute, probably will be submitted to the journal, Analytical Chemistry, or to the Journal of the Electrochemical Society for consideration for publication.

The electrodeposition and counting procedure finally developed is essentially as follows.

The electrolysis cell solution (about 30 ml final volume) contains from  $10^{-7}$  to  $10^{-8}$  grams of uranium-233, to which have been added 20 micrograms of natural uranium as carrier, 5 ml of saturated ammonium oxalate solution, 1 drop of 6M hydrochloric acid, methyl red indicator, and sufficient ammonia solution to turn the indicator yellow. Then, 6M nitric acid is added to turn the solution pink, plus 2 or 3 drops of acid in excess. The cell (Tracerlab model containing a platinum disk as cathode) is immersed in a water bath ( $80^{\circ}$ - $85^{\circ}$ C) and the platinum wire anode is rotated at 550 rpm. The solution is electrolyzed for 30-45 minutes, using an applied potential of about 9 volts with a resulting current flow of 0.8-1.0 ampere; nitric acid and indicator are added as needed to keep the solution acidic. Then, phenolphthalein indicator and 6M ammonia are added until the solution is alkaline, and plating is continued for 60-75 minutes. The electrolyte is then siphoned off as the cell is washed with distilled water. The cell is disassembled and the platinum disk is rinsed with water, followed by acetone, and air-dried. The disk is then ignited for 10 minutes, cooled, and counted in a windowless flow counter using an appropriate scaler.

The foregoing procedure was critically tested in the absence of carrier and with varying amounts of carrier present. Using 20 micrograms of carrier, an average recovery, based on alpha counting, of 94% was obtained for the range of from 0.03 to 0.13 microgram of uranium-233.

## IV. SEPARATION OF URANIUM BY EXTRACTION AS THE CUPFERRATE

From the viewpoint of the present project, extraction procedures are particularly attractive for the problem of isolating submicrogram amounts of substances. Accordingly, the statement made in the literature, that "uranium and antimony are the only elements that will survive a double cupferron separation," became of particular interest. The analytical approach, often used in analyzing uranium minerals, of first extracting with cupferron a solution of the sample containing elements in their higher oxidation states to remove certain elements, and of then reextracting the solution after treatment with a reducing agent to remove uranium(IV), possesses a number of decided advantages. Essentially, cupferron (N-nitrosophenylhydroxylamine) will not react with uranium in its higher oxidation state of six, but will react to form stable complex species with many other metals, including uranium in its lower oxidation state of four.

In the procedure developed, the aqueous solution of the sample is extracted with an organic solvent, such as ether or chloroform, which removes the metal chelate complexes from it. The remaining aqueous solution, which contains the uranium, is now subjected to electrolysis, while the same or a different organic solvent containing the same organic chelating agent previously used is added to form a separate upper layer. As uranium is reduced by the electrolysis to its lower oxidation state, it forms a stable chelate species with the organic chelating agent and is extracted into the organic solvent. The metal ions, originally unextracted, are not now extracted with the uranium(IV); either the electrolysis does not reduce them, or their lower oxidation states do not form species which are extractable.

From the theoretical and manipulative viewpoints, the most important advance in the procedure developed was the introduction of an electrolytic process for changing the oxidation state of uranium in solution without introducing reagents or manual operations. In the electrolytic process, which was based on the fundamental electrochemical behavior of uranium-cupferron solutions as observed by polarographic studies, the uranyl ions, containing uranium in an oxidation state of six, are reduced at a mercury cathode to uranium ions in which uranium has an oxidation state of four.

The actual process finally developed for isolating uranium is relatively simple from the manipulative standpoint, although some rather complex chemistry is involved. Following the necessary orienting polarographic examination of the uranium-cupferron system, an improved procedure was developed which involves the simultaneous electrochemical reduction of uranium(VI) to uranium(IV and/or III) and the extraction of the uranium(IV and/or III) cupferrate into ether. The uranium(IV/III) cupferrate is then reextracted from

the ether solution into  $7M$  nitric acid solution. The nitric acid extract, after decomposition of organic matter with concentrated nitric acid and perchloric acid, is used for measurement of the uranium. A relatively simple one-piece glass apparatus was devised which permits the preextraction, the electrolysis, the uranium extraction by cupferron-ether, and the reextraction of the uranium from the ether to aqueous nitric acid solution to be carried out with a minimum number of operations and can be adapted readily for remote-control operation.

The uranium, isolated as described, can be determined by photometric absorption, if as much as a milligram of uranium is present. In the case of extremely minute amounts of uranium, these, after suitable chemical treatment, are electroplated onto a small platinum disk whose radioactivity in terms of alpha emission is then measured with a flow counter.

Experiments were carried out on milligram quantities of uranium, using the colorimetric ferrocyanide procedure to evaluate the overall uranium recovery. The essentially quantitative character of the extraction-reduction procedure, with a transport of uranium that is at least  $94.0 \pm 1.5\%$  complete, was established. Several runs included the pretreatment of the aqueous uranium(VI) solution with cupferron in chloroform as a check on nonextraction by this step. Recoveries were also checked by the spectrophotometric oxinate procedure described in Section II of this report.

The extrapolation from milligram to microgram and submicrogram levels and the efficiency of separation from other elements were then tested with uranium-233 solutions.

A considerable number of runs were made, involving the complete process for uranium isolation based on simultaneous electrochemical reduction and cupferron extraction of the uranium, followed by reextraction of the uranium, destruction of organic matter, and electrodeposition of the uranium for alpha counting. The runs involved amounts of uranium-233 of the order of from  $10^{-7}$  to  $10^{-8}$  grams initially in 30 ml of solution, to which was added 20 micrograms of natural uranium as carrier. The recovery or yield, based on a comparison of the alpha counts taken of the uranium-233 and of the final plate obtained, was  $85.8 \pm 1.3\%$ . A series of experiments indicated that the recovery in the plating process was 94% or better. The 9% loss, which is consequently involved in the processes of reduction and extraction and of preparation of the extracted uranium for plating, has been traced to a loss of about 5% in the residual aqueous layer after extraction and a loss of from about 3 to 4% in the residual ether layer after extraction with nitric acid.

Considerable difficulties, which were at first encountered in obtaining satisfactory cupferron extraction of amounts of uranium at the microgram level, were overcome by improvements in the procedure. The whole operation at present takes about 4 hours. It is significant to note that an aver-

age recovery of 94% was obtained, using milligram quantities of natural uranium; the recovery was checked by the colorimetric oxine and ferrocyanide methods. Use of milligram additions of carrier uranium cannot be tolerated, due to the self-absorption of alphas that would occur in counting a milligram-size plate of uranium.

One by-product of the investigation of the polarographic behavior of uranyl ion, of cupferron, and of uranyl-cupferron mixtures, which was undertaken to establish optimum conditions for the reduction of uranium(VI) to uranium(IV) in the presence of cupferron, was the establishment of the existence of a uranium(III)-cupferron complex species. This was especially significant and interesting in view of the recent statement that "... little is known about possible complex ions of  $U^{+3}$  in aqueous solution, although the general impression is that little complex-ion formation is to be anticipated." (Quoted from G. T. Seaborg and J. J. Katz, editors, The Actinide Elements, New York, 1954, p. 162.)

A manuscript describing the evidence for the uranium(III) cupferrate, which was approved for release for publication by the Air Force Cambridge Research Center and by the University of Michigan Engineering Research Institute, was submitted to the Journal of the American Chemical Society. It was promptly accepted for publication and is scheduled to appear in a fall, 1955, issue of the Journal.

A manuscript in the form of a scientific paper has been prepared, covering the development and application of the reductive extraction procedure for isolating micro amounts of uranium; this manuscript incorporates the work reported in the earlier submitted rough draft of Scientific Report No. 2 (ERI Report No. 2228-4-T). After approval for release for publication by the Air Force Cambridge Research Center and the University of Michigan Engineering Research Institute, the manuscript will be submitted to the Journal of the Electrochemical Society or to Analytical Chemistry for consideration for publication.

#### V. DETERMINATION OF URANIUM IN FISSION PRODUCT MIXTURES

After the development of the procedures for isolating uranium, described in the previous sections, first by the combined cupferron preextraction-reduction—extraction-reextraction procedure, and then by electrodeposition in a state suitable for counting, a study was made of the isolation and determination of submicrogram quantities of uranium-233 when present in a solution containing mixed fission products. It was considered that the extent to which radiochemical contamination by active nucleides could be removed from the final uranium plate would provide a sensitive measure of the success of

the separation scheme.

The fission products used in testing the procedures were from U.S. Atomic Energy Commission Sample FP-P-1, which is a mixture of fission products present as nitrates in nitric acid solution and prepared by separation from heavy metals which have been exposed for from 40 to 60 days in a reactor and cooled only a short time.

Orientation studies in which the fission products were subjected to the same electroreduction and cupferron extraction, and plating and counting procedures used for uranium, showed that only about 1% of the alpha activity in the mixed fission products (which is logically attributable to the uranium present) is transmitted through the procedure and is finally recovered on the planchet used for counting.

The procedure was then applied to the recovery of uranium-233 when present in admixture with considerable amounts of the fission products mixture, which themselves have high levels of alpha, beta, and gamma emission. A constant amount of uranium-233 (about  $7 \times 10^{-8}$  grams), together with an appropriate quantity of natural-uranium carrier, was mixed with increasing amounts of fission-products mixture; the maximum ratio of the latter to uranium-233 was set at a point beyond which contamination problems would be serious. The regular processes of extraction, reduction, and electroplating were followed.

Radioactivity balance studies were made in which the distribution of the original alpha, beta, and gamma radiation present was followed in order to see where separation was occurring in the various steps of the procedure. The results indicated that uranium-233 can be separated from a fission-product sample.

An average recovery, based on comparison of the alpha counts taken of uranium-233 and of the final plate obtained on analyzing mixed uranium-fission-products samples, was  $85.0 \pm 8.0\%$ . This compares nicely with the yield of  $85.8 \pm 1.3\%$  reported in Section IV of this report. It is thus felt that the separation scheme for uranium, as outlined in this report, is satisfactory for the purpose intended.

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