

#2798

**(This section to be completed by subcontractor requesting document)**

J. Lamb / 1034A  
Requestor Document Center (is requested to provide the following document)

Date of request ~~12/21/95~~ 2/16/96 Expected receipt of document ~~3/15/96~~ 3/16/96

Document number K10-63 Date of document 5/21/48

Title and author (if document is unnumbered)

**(This section to be completed by Document Center)**

Date request received 2/20/96

Date submitted to ADC 2/21/96

Date submitted to HSA Coordinator 2/20/96

**(This section to be completed by HSA Coordinator)**

Date submitted to CICO 2/21/96

Date received from CICO 3/6/96

Date submitted to ChemRisk/Shonka and DOE 3/6/96

**(This section to be completed by ChemRisk/Shonka Research Associates, Inc.)**

Date document received \_\_\_\_\_

Signature \_\_\_\_\_

#2798



027B  
68808

11  
①

~~SECRET~~

This document consists of 17 pages  
No. 1 of 5 copies, Series B

KLO-63

KLO 63 1 B



\*KLO 63 1 B\*

27116

PLANT RECORDS
RECEIPT NO.
P55675

①

ESTABLISHMENT OF A REFERENCE LABORATORY AT K-25

Clifford K. Beck  
May 21, 1948

**UNCLASSIFIED**

Classification changed to: UNCLASSIFIED  
(level and category)

Thomas W. Kelley 2/13/95  
ADD signature (first reviewer) Date

Sey W. Hall 2/21/95  
ADD signature (final reviewer) Date

DISTRIBUTION LIST (Series A)

**K2ERC**  
**NOT TO BE LOANED FROM**  
**PLANT RECORDS**

Clifford K. Beck  
C. E. Center  
A. P. Huber

W. B. Humes  
F. W. Hurd  
S. C. Ling

**CAUTION**

This document contains information affecting the  
National Defense of the United States.  
Its transmission or the disclosure of its contents  
in any manner to an unauthorized person is pro-  
hibited and may result in severe criminal penalties  
under applicable Federal laws.

~~SECRET~~

This document has been approved for release  
to the public by:

John A. Squire  
Technical Information Officer  
Oak Ridge K-25 Site

Date

Carbide and Carbon Chemicals Corporation Operating  
Contractor for the U.S. Atomic Energy Commission.

~~SECRET~~

ESTABLISHMENT OF A REFERENCE LABORATORY AT K-25

Clifford K. Beck  
May 21, 1948

	<u>Page</u>
Abstract-----	1
I. Introduction -----	2
II. A Proposal that an Isotopic and Chemical Analysis Reference Laboratory be Established at K-25 -----	2
III. Laboratory Specifications, and Services to be Rendered ---	3
IV. Methods of Isotopically Analyzing Uranium -----	5
V. Status of K-25 Isotopic Analytical Facilities -----	8
VI. Status of K-25 Chemical Analytical Facilities -----	10
VII. Auxiliary Services Available at K-25 -----	14
VIII. Additional Facilities Required for Establishment of a Reference Laboratory at K-25 -----	15

~~SECRET~~

SECRET

ESTABLISHMENT OF A REFERENCE LABORATORY AT K-25

Clifford K. Beck  
May 21, 1948

ABSTRACT

With increasing emphasis on uranium accountability and an increasing number of commercial and research institutions handling uranium, the need for establishment of an official Isotopic and Chemical Analysis Reference Laboratory is apparent. Several reasons are pointed out which make it desirable, and almost necessary, to establish one laboratory for both isotopic and chemical analyses.

The extensive facilities necessary in order for a laboratory to perform Reference Laboratory services, and the Services a Reference Laboratory should offer, are discussed. The proposal is made that the Isotopic and Chemical Analysis Reference Laboratory be established at K-25.

A number of advantages and unique facilities available to the laboratory at K-25 are listed. It is pointed out that equipment and trained personnel for performing all Reference Laboratory services, including analysis of radioactive samples, are available at K-25, and that all services are already being performed for the K-25 Plant, and to a certain extent, for a small but growing number of outside installations.

② - P. 4

SECRET

~~SECRET~~

ESTABLISHMENT OF A REFERENCE LABORATORY AT K-25

Clifford K. Beck  
May 21, 1948

I. INTRODUCTION

It has become obvious that a Reference Laboratory is needed somewhere in the A.E.C.'s operations where uranium samples may be analyzed for their "official" isotopic and chemical content. The increasing emphasis on and success of uranium control and accountability measures, and the increasing number of laboratories, as well as production facilities concerned with uranium handling, results in a distinct need for establishment of a laboratory equipped with adequate personnel and apparatus for establishing the isotopic and chemical content of uranium samples of assorted chemical forms.

II. A PROPOSAL THAT AN ISOTOPIC AND CHEMICAL REFERENCE LABORATORY BE ESTABLISHED AT K-25

Review of the equipment, personnel, techniques and experience required of any laboratory which attempts to perform Reference Laboratory functions, reveals immediately that few laboratories in the country can meet the requirements. Details of the requirements are pointed out below and the present position of K-25 with respect to these requirements is indicated. In general, it will be noted that the K-25 Laboratory is already in position to perform the services involved and indeed is already doing so for our own plant and, to a certain extent, for outside groups. With little additional equipment, a small amount of additional space and a few extra technicians, the services now being performed could be offered to all installations within the A.E.C.

It is proposed, therefore, that the K-25 Laboratory be designated as the Reference Laboratory for handling isotopic and chemical analysis of uranium samples, and that services the Laboratory is now in position to render be announced and offered to other groups within the Atomic Energy Commission.

Further, it is proposed that the K-25 Laboratory begin immediately to fill in those gaps now existing in equipment, personnel or technique, so that all the duties expected of a Reference Laboratory may be satisfactorily discharged, and that the system of cross-checks of both chemical and isotopic analyses with other installations and institutions be extended to the end that standards of the highest attainable accuracy be maintained.

~~SECRET~~

SECRET

There is one possible objection to the proposal that the K-25 Laboratory become the official Reference Laboratory, namely: it would be desirable that the Reference Laboratory be completely independent of any of the Major handlers of uranium which would be involved in the analysis monitoring of the Laboratory. There are two items which offset this objection: (1) The laboratory at K-25 does operate under the same management as the Process Division which is responsible for handling the uranium inventory at K-25, but operates completely independently of the Process Division. (2) There is within the A.E.C. no other installation equipped to handle the Reference Laboratory duties which is not also a large handler of uranium. The Bureau of Standards might be a logical off-project institution for undertaking these duties, but it is understood that a major accumulation of equipment and personnel would be involved if this were done.

### III. REFERENCE LABORATORY SPECIFICATIONS, AND SERVICES TO BE RENDERED

#### A. Relation of Isotopic and Chemical Reference Analyses

The functions a Reference Laboratory should perform are listed in detail in a later section, but if one considers the broad aspects only, it is immediately apparent that the Reference Laboratory should be capable of superior chemical manipulations as well as isotopic analyses. From the standpoint of a "customer" of the Reference Laboratory, it would be most desirable to be able to ask of a single laboratory the double question: "What is the uranium content of this sample and what is the isotopic composition of the uranium?", rather than two separate questions, accompanying separate samples, directed to two different laboratories.

Furthermore, the Reference Laboratory, in order to purify heterogeneous uranium samples of diverse contaminants and put it into suitable form for isotopic analysis, must possess excellent chemical facilities as well as isotopic. The same applies to the preparation of standards after isotopic analysis for the consumer, who may likely request "Return the sample (or send one) as  $U_3O_8$ , for example, and indicate (a) the total uranium present, (b) the isotopic composition of the uranium, (c) the quantity and identity of contaminants present, and (d) the limit of errors in the measurements".

A large percentage of the samples submitted for chemical and isotopic analysis will undoubtedly be taken from nuclear piles and reactors. These highly radioactive samples, including uranium samples freshly removed from the reactors and heavily contaminated with radioactive fission products, will have to be analyzed and decontaminated (before isotopic analysis) in equipment especially designed for handling "hot" materials. Thus, the Reference Laboratory must possess facilities and travel personnel for handling radioactive materials, as well as superior chemical and isotopic facilities.

SECRET

For those reasons it is recommended that responsibility for handling the Reference Laboratory duties for Isotopic and Chemical analysis be located in the same laboratory.

B. Services to be Rendered by a Reference Laboratory

1. Isotopic Analysis of Submitted Uranium Samples

The sample may be submitted in any chemical form and may contain undetermined contaminants, including radioactive contaminants. It will be purified, converted into suitable form for isotopic analysis, analyzed, and returned to the sender, if desired.

2. Furnish Uranium Standards of Known Isotopic Content

Analyzed uranium, with U-238<sup>5</sup> content anywhere between 0.5 and 93.5 weight percent, as U<sub>3</sub>O<sub>8</sub>, UF<sub>6</sub> or other usual chemical, can be furnished qualified consumers (after proper A.E.C. approval) in gram to kilogram quantities, or even more. The Gaseous Diffusion cascade contains a huge inventory of uranium from "waste" to "product" concentration. The cascade may be tapped at any point, and small quantities at the desired concentration may be withdrawn.

3. Special Isotopic Analyses

Special analytical services as necessary should be rendered for uranium samples containing from 10 ppm to 99.9+% U-235. For example, "primary standards" of uranium could be prepared by mixing known quantities of highly "depleted" U-238 with highly "enriched" U-235 to form a mixture whose isotopic content is known gravimetrically rather than by mass spectrometry.

4. Uranium films with standard Alpha Activity

This duty would not normally be required of a Reference laboratory, but inasmuch as K-25 has performed this related service for some time, it is included. Thin, uniform films of U<sub>3</sub>O<sub>8</sub> containing predetermined quantities of uranium are electroplated onto nickel or platinum discs, thus resulting in "alpha standards" of any desired activity, from 0 to 400,000 counts per minute. The activity is checked before shipment on alpha counters especially designed for this function.

5. Discs or Tubes Coated with Uniform Films of Isotopically Known Uranium

For counter tubes or disc films to be irradiated, uranium coatings enriched or depleted by specified and accurately-known amount in various isotopes, may be desired. Numbers of these have been prepared for customers having many diverse intended uses in mind.

6. Determination of the uranium content of an unknown sample

This service should be rendered, and in addition, the Laboratory should be able to state with considerable confidence the identity and quantity of contaminants included. (K-25 depends now on the spectroscopic services available at Y-12 for some of the impurity determinations and would continue to do so for six months or so until current installations of equipment at K-25 are completed.)

7. Furnish uranium samples in desired chemical form

The Reference Laboratory should be able to furnish samples of uranium up to kilogram quantities, in any usual chemical form the user might desire, as well as state the total uranium and isotopic composition of the sample.

The K-25 Laboratory has, for some time, performed all of the above services for the Gaseous diffusion Plant, and most of the services intermittently for a limited, but increasing, number of outside groups. The nearly constructed Radiochemical Laboratory at K-25 especially constructed for decontamination and analysis of radioactive materials, is available for operations required on radioactive samples.

IV. METHODS OF ISOTOPICALLY ANALYZING URANIUM

There are two general procedures by which the isotopic content of uranium samples may be established. Samples generally encountered are composed of three uranium isotopes: U-234, U-235, and U-238. It is the U-235 content which is of importance in most accountability problems, although U-234 is important also, and U-238 usually must be determined in arriving at the content of the other two. The U-234 in samples generally encountered does not exceed 2%, while U-235 may vary from practically zero to 95% and the U-238 content may range from zero to almost 100%.

A. Mass Spectrometric Method

The mass spectrometer provides at once the most convenient and most accurate method of determining the isotopic content of uranium samples. Several samples (in the same isotopic range) can be analyzed in a day on a single spectrometer, and with special care, the U-235 content can be determined to within 0.1% of the weight percent of U-235 present. Accuracy of 0.1% however, cannot be achieved merely by introducing the sample as a volatile compound into the spectrometer and measuring the relative intensity of the three isotopic beams. This procedure, known as the "absolute method" of spectrometer analysis, may give rise to quite large errors, due to non-linearity of amplifier input resistors, etc., and therefore, is not used where high accuracy is desired.

1. The relative measurement method

The highest accuracy of analysis is obtained with a spectrometer by comparing the relative isotopic beam intensities in the sample with those of a standard sample having known isotopic composition. Comparative measurements made in this fashion permit very accurate analyses, provided the isotopic composition of sample and standard are closely similar. If these two are appreciably different, the non-linearity of amplifier response again introduces errors, and a second error, due to "memory effect", is also introduced. Thus, to use this "Relative Method" for analyzing samples over the entire range of isotopic composition, it is necessary to provide in advance standard samples of known composition at closely spaced intervals over the isotopic range.

2. Effect of memory effect

When a sample of given isotopic composition is introduced into a spectrometer for analysis, some of the sample adsorbs or adsorbs onto the internal surfaces of the instrument. When a subsequent sample is introduced, the molecules of the new sample "exchange" with the molecules already on the surfaces, displacing them into the new gas sample and taking their place on the surfaces. The displaced molecules of the old sample contaminate the new sample and thus influence any analysis of the new sample made before the displacement is complete and the old molecules are swept out of the ionizing chamber.

The "exchange" begins at a rapid rate, which decreases rapidly but may be drawn out over long periods, depending on temperature, etc., before the measurable effect disappears. If the successive samples have quite different isotopic composition, the "memory effect" as it is called, will be quite large and may continue to introduce errors in analysis of the second sample for a long period. If successive samples are nearly alike isotopically, the memory effect will disappear to negligible influence within a short period.

In practice, therefore, it is necessary that samples differing more than a few percent in isotopic composition are not introduced into the same spectrometer. As a result of this requirement, a different mass spectrometer must be available for each range of composition to be analyzed. At least ten instruments should be used over the range from 0.5 to 95% U-235.

C. Preparation of Synthetic Standards

In principle, the preparation of a synthetic standard of known isotopic composition, is quite simple: A calculated amount of uranium of highly enriched (and accurately known) U-235 content is weighed out and mixed with a calculated amount of uranium of highly enriched (and accurately known) U-238 content, to give a "Synthetic" mixture of the calculated isotopic composition.

In practice, numerous difficulties are encountered in preparing a mixture with the accuracy desired. The chief source of errors are: (1) Uncertainty in the absolute isotopic content of the two primary materials, (2) Irreproducibility of the chemical content of uranium compounds (UO<sub>3</sub>, for example, contains various amounts of oxygen, depending on several factors involved in the preparation procedure), (3) Absorption of weighed components prior to mixing, on the walls of containing vessels, (4) Weighing errors.

Considerable success has been achieved in overcoming these difficulties at K-25, and numerous synthetic standards, known to accuracies of 0.1% of the U-235 present, have been prepared. As a result of an investigation and improvement in the techniques developed, plans are now being made to prepare a new set of standards which it is believed will be even more accurate than those now on hand.

D. Alpha, Fission Method of Uranium Analysis

Analysis of uranium samples can be made by means of the radioactive behavior of the three components. The uranium sample is plated out as a thin film of U<sub>2</sub>O<sub>8</sub> on a polished nickel disc and (1) the alpha activity of the film is measured, (2) the fission rate is measured when exposed to a calibrated beam of slow neutrons. From these data, and certain assumptions based on well established knowledge of the properties of the isotopes, and in special cases very rough spectrometer measurements of abundance ratios, the relative abundances of U-234 and U-235, and of the U-238 isotopes can be established. This method enables measurement of the U-235 to within 1.5% (of the weight percentage present).

1. Equipment Required

Use of the counting method of isotopic analysis requires facilities for quantitative electroplating of uniform U<sub>2</sub>O<sub>8</sub> films, for alpha counting, and for fission counting when the film is exposed to a flux of slow neutrons. At K-25 the neutrons are produced by a 5 gm. radium source surrounded by a block of beryllium, which is in turn surrounded by hydrogenous material for slowing the neutrons. Calibration of the beam is accomplished by measurement of the fission produced in films containing known amounts of U-235.

V. STATUS OF K-25 ISOTOPIC ANALYTICAL FACILITIES

In order that an accurate accountability as possible be maintained over the quantities of uranium being processed through the Gaseous Diffusion Plant, it was necessary to install adequate facilities for analyzing the isotopic composition of uranium over wide concentration ranges. Continuous effort has been exerted to increase the accuracy of analysis. As a result of these circumstances, considerable quantities of equipment and experience in specialized techniques have been accumulated. With only minor adjustments, these analytical facilities have adequate capacity to handle a considerably larger number of samples.

It has already been mentioned, but may be repeated, that the analytical facilities at K-25 are a part of the Laboratory Division, which operates completely independently of the Process Division of the Plant.

A. Mass Spectrometers

Thirteen mass spectrometers are presently used in routine analytical determinations over eight concentration ranges (from 0.5% to 99.+ % U-235). Some of these instruments are used as spares during necessary maintenance repairs on alternate instruments, and some are used during peak loads of analytical work.

In addition to the above 13, there are 5 mass spectrometers being used for research investigations on improved analytical methods and other special problems.

1. Analytical Capacity of Mass Spectrometers

On the 13 instruments mentioned above, an average of 60 routine measurements on 15 samples and 140 special measurements on 35 additional samples can be measured each day. The precision attained in these measurements is about 0.1%, and the accuracy is believed to be between 1 and 1.5% (weight percent of the U-235 present). Where higher accuracy is desired, additional effort is required. As much as a month may be spent on one sample if it is of sufficient importance.

B. Alpha, Fission Analytical Facilities

For quantitative electroplating of uranium samples onto metal discs and the subsequent alpha and fission counting, the following equipment is available:

1. Seven plating machines of 4 cells each, now assigned to four concentration ranges.
2. Five alpha counting systems.
3. Two fission counting units, which may contain simultaneously eight samples being irradiated and counted. (only one unit is currently needed for routine analyses, hence one unit is being used for special investigations.)

C. Synthetic Standards and Primary Materials on Hand

Several batches of uranium, of various concentrations, which have been analyzed by the best techniques known, to the highest accuracy now possible, are on hand. These materials may be used as primary ingredients in preparing synthetic mixtures of known composition. A number of carefully analyzed synthetic mixtures are also in stock.

1. 70 grams of  $U_3O_8$  - containing  $93.54 \pm 0.05\%$  U-235.
2. 70 grams of  $U_3O_8$  - containing  $2.42 \pm 0.02\%$  U-234.
3. 51.5 Kg of  $U_3O_8$  - containing  $(0.7113 \pm 0.0006)\%$  U-235.  
 $(0.00544 \pm 0.00011)\%$  U-234.
4. 7.0 Kg of  $U_3O_8$  - containing  $(0.03054 \pm 0.00041)\%$  U-235.
5. 1.0 Kg of  $U_3O_8$  - containing  $(0.00091 \pm 0.00035)\%$  U-235.
6. 60 "gradient" synthetic standards of 20 grams each, known to  $\pm 1.5\%$  (wt. % U-235) ranging from 0.5% to 93.5% U-235.
7. 7 "primary" Synthetic Standards of from 70 grams to 30 kilograms each, known to  $\pm 0.1\%$  (wt. % U-235).

From time to time, as needed, additional samples are withdrawn from the cascade at the concentration desired, or are mixed from primary standards, and after careful analysis, are added to the stock standards on hand.

D. Research Investigation on the Isotopic Content of Natural Uranium

The most important "reference" or "anchor" point in isotopic analysis of uranium is the isotopic content of uranium as it occurs in nature, and particularly the amount of the U-235 isotope in "normal".

(Studies made thus far indicate that uranium from different parts of the world is isotopically identical). The officially accepted "value or normal" U-235 content, measured by Dr. A.O.C. Nier in 1938, is 0.705%. It is now rather certain that this value is incorrect (low).

Over the Past year, two independent groups have reinvestigated the "value of normal" by the method of synthetic standards. These studies are practically complete, and the results obtained will be announced shortly.

E. Isotopic Composition of Highly Enriched U-235 Samples

In enrichment of U-235 by gaseous diffusion, the U-234 isotope is also enriched. Analyses must account for this isotope as well as the U-235 and the U-238. It is impossible to prepare synthetic standards in the highly enriched range which are more accurately known than the highly enriched primary ingredient is known. The highly enriched primary ingredient in turn, is known only to the accuracy achievable (1) by the absolute method of mass spectrometry; admittedly more inaccurate than the target desired, or (2) by measurement of the nuclear radiation characteristics of the isotopes involved.

Investigation reveals that the chief error in absolute isotopic determination in the highly enriched range by method (2) above, is contributed by the uncertainty with which the nuclear properties of U-234 are known. If the half-life of U-234 were known with sufficient accuracy, an independent, absolute method of analyzing highly enriched uranium samples would be available.

Therefore, an investigation of the half-life of U-234 has been launched at K-25. If successful, a considerable improvement in accuracy of isotopic analysis can be achieved.

VI. STATUS OF K-25 CHEMICAL ANALYTICAL FACILITIES

A. Determination of Uranium

Table I, below, summarises the methods of uranium analysis which have been used at K-25 and for which the precision is accurately known. Any of the methods listed could be set up for use on a routine basis and its performance followed by charting daily analyses of standardized control samples. These methods now being used routinely have control samples and control charts already set up.

In addition to the well established methods listed in Table I, other analyses may be carried out by methods whose precisions have not been completely evaluated.

Table I

## METHODS FOR DETERMINATION OF URANIUM

Chemical Form at Start of Analysis	Preliminary Purification Necessary	Method of Determination	Amount of Sample Required	% Precision 95% Confidence Interval Single Detn.	Bias
(a) Essentially Pure Uranium Compounds					
Uranium Hexafluoride	No	Ignition of $UO_2F_2$	250 g.	0.09	$\pm 0.04$
Oxides	Yes	Volumetric Weight Buret	0.25 g.	0.2	none
Oxides	No	Volumetric Chamber Buret	0.20 g.	0.09	none
Oxides	No	Gravimetric $NH_4OH$ pptn.	0.030 g.	0.15	none
(b) Impure Uranium Bearing Materials					
Oxides	Yes	Penta Ether Extraction Gravimetric	0.50 g.	1	$\pm 1\%$
Carbon (Containing Uranium)	Yes	Penta Ether Extraction Gravimetric	10 g.	4	$\pm 1\%$
Water Solns. Containing more than 0.6% U.	Yes	Penta Ether Extraction Gravimetric	100 ml.	1	$\pm 1\%$
Misc. very low conc. materials 0.5-10 ppm. Uranium	Yes	Penta Ether Extraction Visual Fluorometric	50 ml. soln.	6	$\pm 1\%$
Alumina	Yes	Penta Ether Extraction Gravimetric	10 g.	2	$\pm 1\%$
Urine	No	Visual Fluorometric	20 ml.	20	-10%

1. Micro volumetric determinations of 1-3 mg. of U in solutions containing above 50 to 100 ppm. of uranium. The 95% confidence interval on this determination is approximately 0.6 - 0.8%. Further study of this method is being carried out.
2. Ultra micro volumetric determination of 5-200 micrograms of uranium in solutions containing above 50-100 ppm. of Uranium. A negative bias of 2-3% has been found in this method. Evaluation of the precision has not been completed.
3. Polarographic determination of uranium in  $10^{-6}$  to  $10^{-3}$  molar solution of uranium containing various anions, including fluoride, carbonate, phosphate, chloride, sulfate and perchloride. The accuracy of these determinations is + 5%; the evaluation of precision is not complete. A sample of 0.1 ml. is required.
4. Colorimetric determinations of solutions containing above 0.02 mg./10ml., using either peroxide or ferrocyanide. The accuracy is about + 5%.
5. The lower limit of detection of uranium in water solutions is 5-10 parts per billion.

B. Determination of Impurities in Uranium Compounds

Methods have been developed for the analysis of impurities usually found in uranium compounds. Most of the analytical problems encountered fall into two categories: (1) Determination of small amounts of "trace impurities" in quite pure uranium compounds, and (2) Determination (and separation) of one or two gross impurities in uranium compounds. In the paragraphs below, methods are listed for handling the required analyses which have been encountered. Where additional or different analyses are desired, involving materials other than those listed, necessary extension of the analytical methods in general will not involve excessive effort.

1. Spectrochemical Analyses

At present, determination of trace elements in  $U_3O_8$  by optical spectrographic methods is being carried out on a qualitative basis for 25 elements. (Ag, Al, As, Au, Be, Bi, Cd, Co, Cr, Cu, Fe, Ge, In, Mg, Mn, Mo, Ni, P, Pb, Sb, Si, Sn, Tl, V, Zn).

Quantitative analyses by spectrochemical methods depends upon the preparation of suitable standards and the addition of a grating spectrograph (delivery expected shortly). By July 1st, quantitative analysis for 16 trace elements will be routine. (Ag, Al, As, Au, B, Be, Bi, Co, Cr, Cu, Fe, Ge, In, Mg, Mn, Mo, Na, Ni, P, Pb, Sb, Si, Sn, T, V, Zn). These are the elements determined by Scribner and Mullin, J. Res., Nat. Sur. of Stds., 37, 379-89 (1946).

2. Other Analyses Now Performed

- a. Determination of carbon in uranium tetrafluoride. This method will determine from 10 to 1500 parts per million of carbon in uranium tetrafluoride. A 0.5 gram sample is required and the limit of error is about  $\pm 30$  parts per million.
- b. Determination of chromium in uranium hexafluoride or other uranium compounds. This method will determine from 1 to 500 parts per million of chromium with a limit of error of about  $\pm 30\%$ . One gram of sample is required for this determination.
- c. Determination of hydrogen fluoride in uranium hexafluoride. This method will determine from 0 to 0.3 percent hydrogen fluoride. The limit of error for this determination is  $+50\%$  below 0.1% and  $+5\%$  above this value. 2.5 to 4 pounds of sample are required.
- d. Determination of fluorocarbons in uranium hexafluoride. This method will determine from 0.001 to 0.2% fluorocarbons in uranium hexafluoride. 10 grams of sample are required and no estimate of the precision of this method has been made to date.

C. Equipment and Personnel

1. Equipment

a. Spectrographic equipment.

- (1) Bausch and Lomb Littrow Quartz Spectrograph
- (2) Jarrel-Ash 21 foot Wadsworth Mounting Grating Spectrograph is on order and early delivery is expected.
- (3) Spectrograph accessories such as A.C. and D.C. Arc and A.C. Spark Sources, A.R.L. densitometers, and Leeds and Northrup Recording Microphotometer.
- (4) Beckman Flame Photometer.

b. Spectrophotometers and Colorimeters.

- (1) Three Beckman Quartz Spectrophotometers with ultraviolet as well as visible accessories.
- (2) Coleman Model 11 Spectrophotometer.
- (3) One Fisher photoelectric colorimeter.
- (4) Three Lunatron colorimeters. One of these has fluorimeter attachment.
- (5) One Elect-Summerson Photoelectric Colorimeter.
- (6) One Coleman Fluorimeter.

c. Polarographs.

- (1) Sargent Model XII recording polarograph.
- (2) Two Leeds and Northrup Electrochemograph recording polarographs.
- (3) Two Fisher Electropodes Manual polarographs.

d. Miscellaneous

- (1) General Electric X-Ray photometer.
- (2) Infra-red photometer specially built for this project for determination of fluorocarbons in uranium hexafluoride.
- (3) Perkin-Elmer Recording Infra-red Spectrophotometer.
- (4) Raman Spectrograph.
- (5) In addition to these specific instruments, there are also available excellent facilities for the construction and maintenance of general electronic apparatus.

2. Personnel

a. Routine Personnel

At present there are about 40 people carrying on all types of routine uranium analyses in the Works Laboratory Department.

- b. There are 14 people engaged in analytical research in the Chemical Research Department. Up to half of these also carry out special analyses and trouble shooting on routine methods as required.

VII. AUXILLIARY SERVICES AVAILABLE AT K-25.

A. The Cascade Inventory

The gaseous diffusion plant contains a large inventory of uranium in the form of UF<sub>6</sub>, at U-235 isotopic concentrations ranging from well below the normal value (waste) to highly enriched (product). It is possible, to determine the isotopic concentration at any point and to withdraw large quantities, up to many kilograms, if necessary, at the desired concentration. It is a decided advantage, in supplying qualified "customers" (after proper A.E.C. approval) with standards of calibrated isotopic content, to have this inventory available.

B. Statistical Services

There is maintained at K-25 a Statistical Calculations group, headed by an experienced mathematician, whose services are routinely available for analysis of data, determination of limits of error, confidence intervals, etc. The precision of analytical determinations is routinely reported at the 95% confidence interval level, rather than in terms of the less precise Average Deviation, or in other more vague terms sometimes employed.

C. Y-12 Services Routinely Used

The Y-12 spectrographic equipment is more extensive than that at K-25, and has been in use for a longer time. Eventually (6 months to 1 year) the K-25 equipment and technique will provide the same analyses as those at Y-12, but for the present, trace analysis of samples for elements not possible on the K-25 equipment are routinely performed at Y-12.

In addition, it will be of value to continue and extend the cross-checking analyses with Y-12, on chemical methods as well as on spectrographic techniques and procedures.

D. Chemistry and Physics Research Facilities

X-ray, Ramon, Infra-red, ultrasonic, and microwave apparatus and facilities for physical researches into molecular structure and composition are available in the Physics Research Department at K-25 for assistance on any special studies the Chemical or Isotopic Analysis Group might desire.

VIII. ADDITIONAL FACILITIES REQUIRED FOR ESTABLISHMENT OF A REFERENCE LABORATORY AT K-25

As mentioned earlier in this report, the K-25 Laboratory is already performing all services expected of a reference laboratory for the Gaseous Diffusion Plant, and to a limited extent, for outside groups. If officially recognized as a Reference Laboratory, the additional facilities which would have to be added to the existing facilities at K-25 would depend on the amount of additional work requested. At first, only a few extra samples per month would be anticipated. Eventually as the number of samples increased, a proportional increase in facilities required would be expected.

The largest anticipated change at K-25 would result, not from the increased number of samples, but from the increased emphasis on cross-checking with other laboratories and additional research to round out the gaps in analytical techniques so that any problem, however unusual, could be handled.

In the paragraphs below, an estimate is made of the additional facilities the K-25 Laboratory would require by the end of, say, one year, in performing the services of a Reference Laboratory.

A. Space

No additional space would be needed for the work involved in isotopic analysis of the samples. One or two additional rooms, equipped with chemical facilities, say 600 square feet of space, would be required in purification, preparation and chemical analysis of the samples. One room and an office, say 500 square feet, would be needed for receiving, weighing, bookkeeping, calculations, and shipping the samples submitted. (Well-planned and meticulous bookkeeping and control procedures would be required.)

Thus about 1000 square feet, (in addition to the 11,000 now used) would be needed.

**B. Equipment**

The extra chemical space mentioned above would have to be equipped with usual chemical facilities, (hoods, benches, etc.) and in addition, 20 or so fluorinations and transfer systems for gaseous uranium compounds, (i.e., two and perhaps a standby for each concentration range,) would be required.

Eventually, two or three additional alpha counters, and a mass spectrometer, possibly two, if numerous special investigations were required, would be needed.

**C. Personnel**

For isotopic analyses, a number of additional samples could be handled with no increase in personnel. The present analytical load is unevenly scheduled, with capacity requirement during inventory periods and considerably less in between. Scheduling the analysis of reference samples during slack periods would tend to even the load and increase efficiency. If the number of analyses required become too large, additional technicians would be necessary.

For the chemical and control and bookkeeping work involved, three chemists and three clerical workers would be required quite early, and two additional chemists and 4 technicians by the end of one year.

**D. Cross Checks with Other Laboratories**

One part of a limited number of samples, each divided into two equal parts have been sent respectively to Y-12 and K-25 Laboratories for isotopic and chemical analyses. Any discrepancies appearing have been tracked down, until agreement is now essentially complete, within the precision of methods employed. A few samples calibrated by the Bureau of Standards have been obtained and re-analyzed by K-25.

Cross-checking of this kind with these and other laboratories on chemical and Isotopic analyses might be extended to other uranium samples of more diverse chemical composition and other isotopic ranges.

**E. Further Research**

As pointed out in Sections III D and E above, research is now in progress on more accurate determinations of isotopic composition of uranium samples. These and other similar investigations must be continued. The memory effect, ever-present in any equipment previously exposed to uranium compounds, should be further investigated with a view toward reducing its influence on analyses and determining proper corrections to be applied.

~~SECRET~~

A wide variety of investigations could be undertaken to improve the techniques in chemical analysis.

1. Development of consistently reliable methods for purification of uranium prior to quantitative analysis.
2. Development of dependable volumetric methods for uranium determination for 1-10 mg. of U.
3. Development of spectrochemical methods other than the pyroelectric for determination of trace impurities in uranium.
4. Extension of analytical methods to permit identification and determination of many additional elements which may be encountered in uranium compounds but for which methods have not been developed.