C 4 4 63 a

[redacted]

MANHATTAN DISTRICT HISTORY

BOOK VIII, LOS ALAMOS PROJECT (Y)

VOLUME 3, AUXILIARY ACTIVITIES

CHAPTER 7, BORON (B^{10})

DEPARTMENT OF ENERGY DECLASSIFICATION REVIEW

1st REVIEW-DATE: 9.15.05	DETERMINATION [CIRCLE NUMBER(S)]
AUTHORITY: DD	1. CLASSIFICATION RETAINED
NAME:	2. CLASSIFICATION CHANGED TO:
2nd REVIEW-DATE: 9/13 05	3. CONTAINS NO DOE CLASSIFIED INFO
AUTHORITY:	4. COORDINATE WITH:
NAME: GN Brokhorn	5. CLASSIFICATION CANCELED
	6. CLASSIFIED INFO BRACKETED
	7. OTHER (SPECIFY):

CLASSIFICATION CANCELLED
~~OR CHANGED TO~~
BY AUTHORITY OF DOE/DPC
JOHN E. HARTSOCK 9/4/79
REVIEWED BY DATE

March, 1949

[redacted]

~~RESTRICTED DATA~~
~~Atomic Energy Act 1946~~
~~Specific Restricted Data~~
~~Clearance Required~~

[redacted]

MANHATTAN DISTRICT HISTORY
BOOK VIII, LOS ALAMOS PROJECT (Y)
VOLUME 3, AUXILIARY ACTIVITIES
CHAPTER 7, BORON (B10)

1. General.

a. Early in 1943, the Los Alamos Laboratory indicated a need for quantities of crystalline boron of normal isotopic content and crystalline boron enriched in B10 (see Book VIII, Vol. 2). Production of the materials was initially regarded as extremely important and very essential to the overall Manhattan District activity. While necessity for the materials was somewhat modified at a later date, nevertheless, from the beginning the boron project was given the highest priority and every effort was made to assure that production would be as prompt and as efficient as possible. The original requirements included the utmost secrecy and minimum publicity in all phases of the material production. Compliance with the security restrictions caused many of the negotiations to be conducted through oral discussion and thus limited correspondence and records to bare essentials.

b. The desired materials were obtained through: research and development by Columbia University (SAM Laboratories); development and processing by the Norton Company; supply of raw material by the Harshaw Chemical Company; development, design, plant construction and processing by the Standard Oil Company of Indiana; and development and processing by the American Cyanamid Company. Each of the organizations involved and its respective performance will be referred to herein under a separate heading.

~~RESTRICTED DATA~~
~~Atomic E... Act 1946~~
~~Specific ...ted Data~~
~~Clearance Requir...~~

[redacted]

[redacted]

c. Names of personnel involved in the boron procurement activities may be found in the appendices to this chapter, and in other volumes of the History, to which references will be made hereinafter. Those who were most concerned included the following:

Dr. J. R. Oppenheimer, of Los Alamos Laboratory;

Dr. H. C. Urey and Dr. Martin Kilpatrick, of Columbia University (SAM Laboratories);

Dr. Cyril S. Smith, of the Norton Company;

Mr. E. G. Seubert and Dr. E. W. Thiele, of Standard Oil Company of Indiana;

Maj. Gen. L. R. Groves and Lt.Col. J. R. Ruhoff, of the Manhattan District.

2. <u>Columbia University.</u> (Refer to App. A-1.)

a. In May 1943, the request of the Los Alamos Laboratory, described above, was discussed between representatives of Columbia University and the Madison Square Area. That discussion resulted in arrangements whereby, under Contract W-7405-eng-50, Columbia University was to proceed with the necessary research and development for the production of 500 to 1000 pounds of crystalline boron of normal isotopic content and 50 kilograms of crystalline boron containing not less than 90% B^{10}.

b. The need for this work had been anticipated by personnel at Columbia and four months earlier (in January 1943), under OSRD Contract OEMsr 412, research on the separation of boron isotopes had been started. That research had resulted, in April 1943, in the discovery of a promising method of operation. Such a method of operation was still being investigated when the Madison Square Area arrangements became effective, but the information which had already been obtained was such that development could, and did, proceed with a minimum of delay. The separation procedure, which was proven to be successful by continued investigation and plant operation, was based on a counter-current distillation of a dissociable complex or polymer.

c. In addition to the study for separation of boron isotopes, Columbia initiated a research program concerned with the reduction of boron compounds. It was determined under this program that the reduction of boron trichloride by hydrogen on heated rods would produce best results, and development work for this feature was carried out in co-operation with the Norton Company.

d. The separated product obtained by the method developed at Columbia was dimethyl ether-boron trifluoride complex and it was then necessary to undertake research on the conversion of that compound to boron trichloride. A pilot plant was set up at the Columbia laboratories and development work was carried out in cooperation with the American Cyanamid Company.

3. <u>The Norton Company</u>.

The Norton Company participated in the boron program under two different lines of procedure: first, through cooperation with Columbia University in developing the method of reducing boron trifluoride; and, second, in producing crystalline boron. These activities are referred to in Appendix A-1 for the reduction of boron trifluoride and in Book VII, Vol. 1, for the production of crystalline boron. That

[redacted]

production applied to material of normal isotopic content and was accomplished under Contract W-7405-eng-292, effective 15 January 1944, at a cost of $53,000 (See Book VII, Vol. 1). Government-owned raw material, which had been obtained from the Niagara Smelting Company, was supplied to the Norton Company for their process.

Confirming records and supporting data for the Norton Company participation in the boron project are, of necessity, extremely sketchy, as particular care was exercised to prevent general knowledge of their activity being divulged.

4. The Harshaw Chemical Company.

The Harshaw Chemical Company was selected to supply raw material for the Standard Oil process of the boron project. Contract W-7405-eng-289, effective 15 March 1944, called for a supply of 850 pounds of "synthane" per week for six months, starting 1 May 1944. "Synthane" was the code symbol for boron trifluoride containing not less than 97% of BF_3 of as constant purity as possible. Subsequent supplements to the contract increased the quantity of raw material to a total of 92,450 pounds, at a total contract price of $72,770 (see Book VII, Vol. 1). Raw material delivery was completed 1 March 1946.

5. Standard Oil Company of Indiana.

When research and development at Columbia University had progressed to a stage which permitted design and construction of a production plant for the separation of boron isotopes, contact was made with the Standard Oil Company of Indiana (App. A-2, 3 & 4). That company indicated its

[redacted]

[redacted]

agreement to participate in the project and on 5 January 1944 entered into Contract W-7413-eng-41. The contract was effective as of 2 December 1943, the contemplated work consisting of the design, erection and operation of a suitable production plant for the separation of boron isotopes. In addition to the above work it was understood that the Standard Oil Company would undertake work directed toward: increasing the certainty of the early success of the proposed project; training of technical and non-technical personnel; and, establishing an adequate security system.

A general history of the contractor's activity in meeting contractual requirements is provided in Appendix A-5. That history of the separation project describes: the work objective; selection of the production contractor; plant design features; initial operation; supplementary plant considerations; and the then future possibilities of the process.

Major problems which affected scheduled production resulted from difficulties in obtaining desired construction material (App. A-6), and the inadvertent introduction of mercury into the plant system during the leak test procedure (App. A-5). The latter difficulty was considered to be quite serious in its nature and led to the erroneous conclusion that the initial plant would be unsuitable for production and that replacement would be required. Later on during the contract a corrective measure was discovered and the original plant proved to be operable for production purposes.

During the period of operation, a total of 736 gallons of heavy polymer was produced. Appendix A-7 provides details of that production in regard to lot quantities, percent of heavy polymer for each lot, etc.

[redacted]

[redacted]

Appendix A-7 also includes breakdown figures for the total cost, under Contract W-7418-eng-41, of $2,309,546.88, prior to any adjustment required by final audit.

On 8 March 1946 the Contractor was directed, except for the economical completion of material then being processed, to cease operation of the facilities, effective 4 February 1946 (App. A-8). Supplemental Agreement No. 5, of contract W-7418-eng-41, provided for the period of production performance to end at 11:59 P.M. on 30 June 1946. Also, in accordance with that supplemental agreement, the contractor placed the process equipment in stand-by condition and stored such equipment as was directed by the contracting officer.

6. <u>American Cyanamid Company.</u>

Contract W-7401-eng-91, dated 8 February 1944, with the American Cyanamid Company, required the contractor to process product 890 A to the final product 891 A. Products 890 A and 891 A were the respective code symbols for boron trifluoride dimethyl ether complex and for crystalline boron 10, 96% assay.

The process used in converting the complex to boron was developed under Columbia Contract W-7405-eng-50 (see App. A-1), but the engineering and design work necessary to put the laboratory process on a plant scale was done by the American Cyanamid Company.

It has been mentioned that the initial requirement was the continued production of B^{10} until a quantity of 50 kilograms had been completed. The production schedule called for 1 kilogram of boron as early as possible, a total of 5 kilograms by 15 September 1944, and 5 additional

[redacted]

[redacted]

kilograms every month thereafter until the total of 50 kilograms had been obtained. The startup of the plant was, of course, dependent upon the production of separated complex from the Standard Oil Company of Indiana. The rate of production of the separated complex had been established, by the Chicago Area and the Standard Oil Company, to be 5 gallons every three days. The plant of the American Cyanamid Company was designed to run at twice this rate if necessary, since the Chicago Area had intimated that they might be able to arrange for an increase in the rate of separation. The American Cyanamid Company plant had undergone test runs and was ready to operate by 7 July 1944. Unfortunately, material was not available at that time from the Standard Oil Company. While awaiting material from the Standard Oil Company, work continued at the American Cyanamid Company, to improve their process and to prevent possible losses of material. Upon the start of delivery of the 890 A complex from the Standard Oil Company, the American Cyanamid facilities were in readiness and processing to the final product began.

In accordance with supplementary requirements for contract W-7401-eng-91, production at the American Cyanamid Company continued well into 1946, and 504 pounds of crystalline boron 10, 850 pounds of calcium fluoride-boron trifluoride complex, containing a greater than normal ratio of the B^{10} isotope, and 242 pounds of calcium fluoride-boron trifluoride complex, containing a greater than normal ratio of B^{11}, was obtained (see Book VII, Vol. 1).

[redacted]

COLUMBIA UNIVERSITY, DIVISION OF WAR RESEARCH

~~Project S.A.M.~~

Columbia
Serial No. 10[illegible]-934
File Index 180
[illegible] July 31, 1945

~~SECRET~~
Classification

Distribution
Series A

Copy No.______ ______
(Copy No. Series Letter)

1-10.	District Engineer (Tennessee)
11.	Area Engineer (Col.)
12.	Urey, H. C.
13.	Smyth, H. D.

FINAL REPORT ON CONTRACT W-7405-ENG-50
Short [illegible] Addressee of Letter

Harold C. Urey

W-7405 Eng 50

THIS DOCUMENT [illegible] AFFECTING THE NATIONAL DEFENSE OF [illegible] WITHIN THE MEANING OF THE ESPIONAGE ACT [illegible] ITS TRANSMISSION OR THE REVELATION [illegible] CONTENTS [illegible] ANY MANNER TO AN UNAUTHORIZED [illegible] IS PROHIBITED [illegible]

In general, a classified document shall not be copied or duplicated. If additional copies are necessary they should be requested from the originator. In an emergency the originator may give telegraphic approval for reproductions, specifying series letter and number of copies. The originator must be notified immediately of the distribution of the reproductions. All [illegible] stencils, etc., should be burned.

The Separation of the Boron Isotopes and the
Production of Metallic Boron
Final Report Submitted in Accordance with the Terms
of Contract W-7405 eng-50
Harold C. Urey
July 31,1945

I. Historical Review

In May, 1943, the U.S. Engineer Corps of the War Department requested that the necessary research work be undertaken to determine methods for the production of crystalline boron. In addition to some 500 to 1,000 pounds of crystalline boron of normal isotopic content, a need was indicated for 50 kilograms of crystalline boron containing not less than 90% of B^{10}. Fortunately, the need for this material had been partly anticipated at Columbia University, and research had been started in January, 1943, on the separation of the boron isotopes, under O.S.R.D. Contract OEMsr 412. In April, 1943, a promising method had been discovered at Columbia, and was under investigation.

A pilot plant was set up to accumulate engineering data necessary for the design of a plant which would separate 300 grams of B^{10} per day. In addition, a research program was initiated to study various methods of reducing boron compounds. After a survey, experimental work on the reduction of boron trichloride by hydrogen and of boron trifluoride by sodium, was carried out. The method finally chosen was the reduction of boron trichloride by hydrogen on heated rods, and the development work was carried out in cooperation with the Norton Company. The use of this method involved the production of boron trichloride of high purity. The Niagara Smelting Company undertook this work and produced sufficient trichloride for the production of the required amount of crystalline boron. This work was completed by the Norton Company by the end of May, 1944.

In the meantime, the pilot plant work on the separation had progressed to a stage where the construction of a plant could be undertaken, and in November, 1943, the design, construction and operation of a plant was made the responsibility of the Standard Oil Company of Indiana. As the separated product was to be dimethyl ether-boron trifluoride complex, $(CH_3)_2O \cdot BF_3$, it was necessary to undertake research on the conversion of this compound to boron trichloride before reduction with hydrogen. In addition, further research work was necessary to add refinements to the reduction step. A pilot plant was set up at the Columbia laboratories, and the development work was carried out with the American Cyanamid Company. This company began work in February, 1944.

After some delay due to operational difficulties and accidental introduction of mercury into the plant, production of separated boron as the dimethyl ether-boron trifluoride complex began in January, 1945, and was completed in May, 1945. The American Cyanamid Company plant completed the production of crystalline product of specified purity in June, 1945. Experimental samples of B^{11} were prepared in the pilot plant at Columbia.

2. Classification of Reports

a. Kinds of Reports

Three types of reports have been used for recording progress of the work:

1) "M" reports are communications, usually brief, dealing with [illegible] of immediate interest. Many of the "M" reports were issued bi-weekly, and cover the progress during a fortnight. These reports enabled the associated groups to keep in close touch with the results obtained at Columbia during the progress of work.

2) "A" reports are comprehensive summaries on particular subjects reported in a systematic and logical way, and are essentially final reports of material discussed in the "M" reports.

3) "P" reports are brief summary reports submitted monthly to the Army, which included an estimate of costs and a statement of the progress of the work.

b. Classification of Reports

The reports on this subject can be divided roughly into three main groups:

I. Separation of the boron isotopes
II. Conversion of the [illegible] product (dimethyl ether complex) to metallic boron.
III. Analytical methods.

3. Outline of Work Covered

I. Research on Separation of Boron Isotopes

A. Separation of the boron isotopes by physical methods.

1. Distillation of volatile boron compounds.

2. Steam distillation of boric acid.

3. Thermal diffusion

B. Separation of the boron isotopes by chemical methods.

1. The diethyl ether-boron trifluoride system

2. The dimethyl ether-boron trifluoride system

3. The ethyl bromide-boron trifluoride system

C. Fractionation factor for the ether-boron trifluoride system.

1. Direct determination of the fractionation factor

2. Fractionation factor from enrichment in column packed with Raschig rings.

3. Fractionation factor from enrichment in Stedman packed column

4. Fractionation during production.

5. Value of the fractionation factor.

D. Stability of complexes

1. Decomposition

a. Rate of formation of methyl fluoride
b. Rate of accumulation of methyl borate complex at the top of the column.
c. Manometric method

2. Peroxide formation

3. Effect of water

E. Physical properties

1. Vapor pressure of methyl borate, diethyl ether complex, dimethyl ether complex, and methyl borate complex

2. Heat of vaporization of dimethyl ether complex

3. Density of diethyl and dimethyl ether complexes

4. Viscosity of diethyl and dimethyl ether complexes

5. Specific heat of the dimethyl ether complex

6. Summary of physical properties

7. Solubility of SiF_4

F. Operation of the Stedman column

It was found that isotopes, including boron, could be separated by a countercurrent distillation of a dissociating complex or polymer. This novel method of separating isotopes was tested for the boron isotopes and found to be successful, with an overall one-step efficiency sufficiently large to make the method very attractive from both an engineering and purely scientific point of view. This new method of separation is quite general, and is not limited in any way to the boron isotopes.

This method is now being used for the large scale separation of the boron isotopes. The plant is using the dimethyl ether complex $(CH_3)_2O \cdot BF_3$, which has a simple process fractionation factor of 1.016 ± 0.002. In order to make this method of separating the boron isotopes of practical importance, it was necessary to find a method of recovering quantitatively the BF_3 from the complex, and this was done. The work on a laboratory scale is described in the reports, as well as the large scale development and application to a commercial scale.

Successful application of this method of separation of the isotopes to large scale production required [illegible] the stability of various construction materials to the complex. This [illegible] extensive program on corrosion studies. The results of these studies [illegible] description of the techniques used, many of which are quite new, [illegible] in the reports. Methods of analyzing the complex for [illegible] of [illegible] studying the rate of decomposition, the results [illegible] on the physical properties

[redacted]

...mplex, accurate determination of the α (the simple process fractionation ... are discussed in the reports, as well as data necessary for proper design ... lant.

This work was under the direction of Drs. R. H. Crist and M. Kilpatrick. ...h the research was done by a group working as a whole, for convenience ... of operation, this group was divided into two sections, a chemical ...h section, and an engineering section. The early orienting and investiga... ...rk was done by the chemical section, but upon the formal establishment of ...gineering section, the work was separated into the rough classifications of ...nd engineering research. Actually, however, some of the chemical work was ... by the engineering unit, and vice versa. Towards the end of the research, the chemical group was dissolved and the work, mostly purely engineering, was continued by the engineering group alone. The chemical research section was under the supervision of I. Kirshenbaum, and the engineering section was under P. W. Schutz and N. Sabi. The experimental work was done with the help of the following senior research chemists and engineers: G. Banikiotes, T. Crowell, D. A. McCaulay, W. Visansky, and W. C. Zmachinsky, aided by a staff consisting of T. Blair, R. Bogash, A. Cummin, W. Forsythe, A. Kivnick, M. Kopp, S. Levin, J. Shedlosky, ?. Thomas, and J. Urban.

When the plant for the separation of B^{10} and B^{11} developed difficulties due to the leakage of water into the system, Dr. Schutz and W. Forsythe and D. McCaulay, were employed by the Standard Oil Company of Indiana. With this increase in personnel, the various difficulties of the plant were solved and the plant put into production. N. Sabi and W. C. Zmachinsky also spent some time at the Whiting plant.

The necessity for additional analytical work was taken care of by the transfer of Messrs. Judson and Flynn to the Standard Oil Company of Indiana.

In addition, the problem of aqueous [illegible] boron compounds necessitated further studies on corrosion, [illegible], on analytical control, and on methods for the removal of water. To assist in [illegible] this, an additional group, consisting of E. H. Taylor, F. [illegible], J. R[illegible], H. Schwartz and S. Datz, studied the properties of the boron trifluoride hydrates.

In view of the possibility that the accidental introduction of mercury and the leak of moisture into the plant might [illegible] in the loss of the plant, a new plant was designed and research work was carried out on the possible use of fluorocarbons as heat exchange media. Work was also carried out on substituting a plastic diaphragm for the [illegible] in the pumps. Research work conducted by L. C. Liberatore and R. O. R[illegible] showed that a plastic material which had been developed on the project (known as MFP-1?) was quite satisfactory, and that carbon fluorides could be used as heat exchange media. However, with the success of the original plant, this development was [illegible].

II. Conversion of [illegible] Product to Metallic Form

A. Conversion of $(CH_3)_2O \cdot BF_3$ to BF_3

1. Step I - Conversion of $(CH_3)_2O \cdot BF_3$ to $CaF_2 \cdot BF_3$ complex - small scale

[redacted]

[redacted]

2. Step II - Production of BF_3 from $CaF_2 \cdot BF_3$ complex - small scale

3. Steps I and II - pilot plant

4. Recovery of B

A method for the preparation of pure crystalline boron at rates of the order of 600 grams per day was developed in this laboratory. In order to prepare pure crystalline boron enriched in ^{10}, it is necessary to convert the final product $(CH_3)_2O \cdot BF_3$ of the isotopic separation process to the starting material BCl_3 of the aforementioned process for the preparation of boron. A method for carrying out this conversion was developed, and consists of three steps.

The work done in this laboratory on the first two steps of this conversion process was under the direction of M. Kilpatrick and C. A. Hutchison, and carried out by A. D. Kirshenbaum, J. A. [illegible], J. Shedlesky, J. L. Thomas, R. Reider and R. Schwartz.

B. Conversion of BF_3 to BCl_3 (Step III).

1. Conversion in packed tubes

2. Conversion in stirred vessels

3. Conversion in bomb

4. Recovery of B from residues

A major part of the experimental work on this third step of the conversion process was done by R. M. Eberts and E. R. Glass under the direction of [illegible] and M. Kilpatrick. The following persons also participated in the work at various stages: A. J. Flynn, C. M. Judson, E. Kalichman, A. D. Kirshenbaum, R. C. Schwartz, J. C. Smith, C. D. Turner and G. H. Turner.

C. Production of crystalline boron from its halides.

1. Summary of reduction studies

   a. Small scale reduction in a high voltage spark

   b. Small scale reduction on filaments

   c. Small scale reduction in tubes

   d. Large scale reduction on rods

   e. Plant reduction on rods

2. Recovery of boron

   a. Recovery of BCl_3 from exhaust gases

   b. Recovery of B from tantalum rods

The research done in this phase of the work concerned the development of processes for the production of crystalline boron of high purity and with nearly

[redacted]

complete conversion of the starting boron compound to boron metal. An initial examination of known reactions for the production of boron disclosed that of them all, the reaction $BX_3 + \frac{3}{2} H_2 \rightarrow B + 3HX$, where X is F, Cl, Br or I, would be expected to give the purest product because of the volatility of all reactants and products excepting boron.

The equilibrium constants for the reduction of BF_3 and BCl_3 with H_2 were computed approximately from data available in the literature. The computational results confirmed our experimental studies which showed that BF_3 was not and BCl_3 was reduced by H_2 on, or in the neighborhood of, a surface at 1,300 to 1,500° K. The computations gave for the equilibrium constants at 1,500° K. for BF_3 and BCl_3, 2.4×10^{-11} and 1.3×10^{-4} respectively. The reduction of BBr_3 with H_2 was found to occur as easily as that of BCl_3, but the bromide was less readily available.

Experimental investigations showed that under proper conditions the boron produced by reduction of BCl_3 with H_2 is crystalline and of greater than 99 atom per cent purity. Also it was shown that the unreduced BCl_3 (the yields were always less than 80 per cent) and the B combined with the hot surface could always be recovered as BCl_3 and reused, resulting in very small loss of B. Apparatus was devised for the commercial production of 600 grams or more of B per day. A pilot plant for carrying out this process was constructed and operated.

The experimental work was carried out under the direction of M. Kilpatrick and C. A. Hutchison, by J. S. Smith, R. S. Schwartz, E. Kalichman, J. L. Thomas, and R. Reider.

D. <u>An investigation of alternate methods for reduction of boron compounds</u>

1. The preparation of boron by the reduction of boron trifluoride with sodium.
   a. The batch sodium method and early experiments
   b. Methods involving the simultaneous introduction of sodium and boron trifluoride
2. The preparation of boron by the reduction of sodium fluoborate with sodium.
3. Extraction of sodium fluoride by water from the reaction products
4. Preparation of boron by the reduction of boron trifluoride and sodium fluoborate with aluminum
5. Preparation of boron by the reduction of boric anhydride with active metals
6. The high temperature purification and consolidation of amorphous boron

It was the object of this research program to develop a chemical method for the production of boron of high purity. Initially, the objective was to produce boron either amorphous or otherwise of at least 95 atom per cent purity. Later this was revised to consolidated or crystalline boron of 99 per cent purity. Work was directed toward achieving this goal by two methods: (1) A two-stage process wherein amorphous boron was first prepared by some simple, direct chemical method that was then followed by a purification and/or consolidation treatment at a high temperature. (2) A single-stage process wherein crystalline boron or consolidated boron of high purity was prepared directly by the interaction of free sodium with boron trifluoride at high temperature.

Primary emphasis was placed on the technique of preparing boron rather than on the comprehensive study of the purity of the products obtained or their recovery. This was particularly evident in cases involving the use of sodium, where the handling of the materials and the type of equipment used proved to be the greatest problem.

This investigation did not yield suitable positive results. The difficulties encountered with equipment and handling of materials presented obstacles that would have necessitated a long-term research.

This work was done under the general direction of M. Kilpatrick, J. S. Scevack and A. Kurtz, assisted by H. H. Starke, T. Clarke, G. Cohen, S. Federman, M. Hanig, M. Tetenbaum and R. Ullman.

III. Analytical Methods

A. Spectrometric Analysis

A heavy metal mass spectrometer was used for the first work on the isotopic analysis. This instrument was used to get the necessary fundamental results from which a more simple satisfactory routine machine could be designed.

After the basic analysis method had been found, a special instrument was constructed which was then used for routine analysis of B^{10} and B^{11} compositions. Following the completion of this instrument, a second machine for routine plant control was found necessary and constructed accordingly.

This work was under the general direction of M. G. Inghram and J. S. Smith, assisted by T. G. Schwab, C. Y. Bartholomew and H. Greenberg.

B. Chemical Analysis

Investigations were carried out on the following subjects:

1. Analysis of pure crystalline boron
2. Determination of silicon and iron in boron
3. Analysis of boron trichloride
4. Analysis of boron fluoride-methyl ether complex
5. Constituent analysis of boron fluoride-methyl ether complex
6. Analysis of exit gases from boron trichloride reduction
7. Analysis of constituents and impurities in boron trichlorid
8. Complete analysis of boron

The general analytical problems of this project were investigated by a group composed of C. M. Judson, C. D. Turner, G. H. Turner, A. J. Flynn, J. A. Venn, A. D. Kirshenbaum and M. Kurpka.

4. List of Reports

I. Reports on Separation of Boron Isotopes

A. REPORTS

A-,705	Kirshenbaum, I.	Monthly Report, 5-15-43
A- 734	"	Bibliography, The Chemistry of BF_3, 6-19-43
A- 742	"	Chemical and Physical Properties, 7-2-43
A-1276	Kaplan, I., Cohen, K.P.	The Approach to Equilibrium of a Countercurrent Column or a Square Cascade with Material Whose Mole Fraction Is Not Small, 5-1-44
A-2120	Kirshenbaum, I., Sabi, N., Schutz, P.W.	The Separation of the Boron Isotopes, 9-30-44
A-2192	Taylor, E.H.	Chemistry of $BF_3 \cdot 2H_2O$, 9-1-45

M REPORTS - BI-WEEKLY

2R-229	Kirshenbaum, I.	6-21-43
2R-659	Schutz, P.W.	8-19-43
2R-705	Schutz, P., Kirshenbaum, I.	9- 4-43
2R-731	Schutz, P.W.	9-15-43
M- 400	Kirshenbaum, I., Gilbertson, L.I., Schutz, P.W.	9-24-43
M- 401	Schutz, P.W., Kirshenbaum, I.	10- 4-43
M- 403	Schutz, P.W., Kirshenbaum, I.	10-20-43
M- 406	Schutz, P.W., Kirshenbaum, I.	11- 9-43
M- 408	Schutz, P.W., Kirshenbaum, I.	11-26-43
M- 410	Schutz, P.W., Kirshenbaum, I.	12-20-43
M- 414	Schutz, P.W., Kirshenbaum, I.	1- 8-44
M- 416	Schutz, P.W., Kirshenbaum, I.	1-22-44
M- 418	Schutz, P.W., Kirshenbaum, I.	2- 7-44
M- 420	Schutz, P.W., Kirshenbaum, I.	2-21-44
M-422	Sabi, N.	3- 4-44
M-424	"	3-20-44
M-426	"	4- 7-44
M-428	"	4-19-44
M-430	"	5- 4-44
M-901	"	5-19-44
M-905	"	6- 3-44
M-907	"	6-20-44
M-909	"	7- 5-44
M-917	" and Kirshenbaum, I.	9- 7-44

[redacted]

H. REPORTS

2R- 2	Kirshenbaum, I.	Fractionation Factor for BF_3, 1-?-43
2R- 32	Crist, R.H., et al	Progress Report, 3-?-43
?- 412	Kirshenbaum, I.	Methyl Ether Complex, 12-31-43
M- 427	Sabi, N.	Corrosion of Stedman Packing, 4-11-44
4M-160	Kaplan, I.	Analysis of Data on Vitamin B Separation, 4-17-44
M- 903	Schutz, P. ., Zmachinsky, ?.C.	Liquid Distribution in Stedman Packing, 5-19-44
4M-174	Kaplan, I.	The Design of the Vitamin B Cascade, 6-30-44
M- 910	Schutz, P. ., Zmachinsky W.C.	Liquid Distribution through Stedman Packing No. 3, 7-10-44
M- 911	Sabi, N.	Test of Operating Efficiency, ?-2-44
M- 913	Sabi, N.	Conductivity of Polymer-Water Mixtures. Determination of Water in Polymer, ?-10-44
M- 914	Zmachinsky, W.C.	Vapor Pressure of Methyl Borate Complex, ?-23-44
M- 915	Sabi, N.	Experience in Handling Polymer, 8-29-44
4M-190	Lefkowitz, B.	Computations, 10-17-44
M- 919	Theoretical Group	Time of Approach to Equilibrium in a Column, 10-23-44
M-1183	Zmachinsky, W.C.	Liquid Distribution through Stedman Packing, 10-23-44
4M-192	Kaplan, I.	Equilibrium Time for Vitamin B Cascades, 10-24-44
M-1184	Kirshenbaum, I.	Effect of Water on the Complex, 10-27-44
M-1185	Theoretical Group	Equilibrium Time, 10-30-44
M-1186	Liberatore, L.C.	Diaphragm Pump Investigation, 11-20-44
M-1187	Liberatore, L.C., Rossbach, ?.?.	Distillation Curves for Samples of C-714 and C-616, 12-22-44
M-1188	Liberatore, L.C.	Physical Properties of C-714 and C-616, 12-22-44
M-1401	Kirshenbaum, I.	Stress Corrosion Experiments, 12-2?-44
M-1402	Kirshenbaum, I.	Corrosion of Monel Screen in Samples of Plant Complex, 12-30-44
M-1403	Zmachinsky, W.C.	Efficiency of Stedman Packing, 1-2-45
M-1404	Hutchison, C.A., Thomas, J.L.	Conversion of H_3BO_3 to BF_3, 1-5-45
M-1405	Kirshenbaum, I.	A Study of Some of the Physical and Chemical Properties of Complex, 1-31-45

~~SECRET~~

[redacted]

M-1406	Liberatore, L.C.	Diaphragm Pump Investigation, 1-22-45
M-1407	Sabi, N.	Effect of Water on Fractionation and Decomposition of 890, 1-31-45
M-1408	Zmachinsky, H.C.	Self-Wetting Stedman Packing, 2-14-45
Supplement to M-1407	Sabi, N.	Fractionation at Low Reflux Rates, 2-2[illegible]-45

II. Reports on Conversion of Separated Product to Metallic Boron

A REPORTS

A-1246	Spevack, J.S., Kurtz, A.N.	An Investigation of the Preparation of Boron by Reaction of Boron Compounds with Metals, 5-25-44
A-1247	Starke, H.H.	Attempts to Consolidate Amorphous Boron, 3-10-44
A-1268	Hutchison, C.A., Smith, J.S.	Thermodynamics of Production of Boron Metal by Reduction of Boron Halides, 5-29-44
A-1298	Hutchison, C.A., Smith, J.A.	Research on Methods of Production of Crystalline Boron of High Purity from Boron Halides, 8-10-44
A-2108	Hutchison, C.A., Smith, J.S.	Supplement to A-1268, Thermodynamics of the Reduction of Certain Halides, 8-10-44
A-2121	Kirshenbaum, A.D., Hutchison, C.A.	Conversion of $(CH_3)_2O\cdot BF_3$ to BF_3, Steps I and II) 11-30-44
A-2122	Hutchison, C.A., Roberts, [illegible]	Conversion of BF_3 to BCl_3 (Step III), 1-25-45
A-2123	Hutchison, C.A.	Pilot Plant Conversion of $(CH_3)_2O\cdot BF_3$ to Crystalline Boron, 11-10-44

M REPORTS - BI-WEEKLY

2R-658	Gilbertson, L.I.	8-19-43	M-415	Hutchison, C.A., Spevack, J.S.	1-22-44
2R-691	Gilbertson, L.I.	9- 1-43	M-417	Hutchison, C.A.	2- 7-44
2R-743	Gilbertson, L.I.	9-20-43	M-419	Hutchison, C.A.	2-21-44
M-402	Gilbertson, L.I.	10- 5-43	M-421	Hutchison, C.A.	3- 4-44
M-404	Gilbertson, L.I.	10-21-43	M-423	Hutchison, C.A., Sabi, N.	3-20-44
M-405	Hutchison, C.A., Spevack, J.S.	11- 9-43	M-425	Hutchison, C.A., Sabi, N.	4- 7-44
M-407	Hutchison, C.A., Spevack, J.S., Starke, H. H.	11-26-43	M-429	Hutchison, C.A.	4-19-44
M-409	Hutchison, C.A., Kurtz, A.N., Starke, H.H.	12- 9-43	M-900	Hutchison, C.A.	5-[illegible]-44
M-411	Hutchison, C.A., Spevack, J.S., Starke, H.H.	12-20-43	M-902	Hutchison, C.A.	5-19-44
M-413	Hutchison, C.A., Spevack, J.S. Starke, H.H.	1- 8-44	M-904	Hutchison, C.A.	6- 3-44
			M-906	Hutchison, C.A.	6-20-44
			M-908	Hutchison, C.A.	7- 5-44
			M-912	Hutchison, C.A., et al	8- 2-44
			M-916	Hutchison, C.A.	9- 7-44

SECRET

A REPORTS

A-1400	Hutchison, C.A., Kirshenbaum, A.D.	Preparation of BF_3 from $(CH_3)_2O \cdot BF_3$ to which Water Has Been Added, 12-28-44
A-1409	Hutchison, C.A.	Determination of Isotopic Abundance Ratio in Elemental B

III. Reports on Analytical Methods

A REPORTS

A-1240	Judson, C.M.	Analysis of Pure Crystalline Boron, 2-11-44
A-1266	Judson, C.M.	Determination of Silicon and Iron in Boron, 5-1-44
A-1267	Judson, C.M.	Analysis of Boron Trichloride, 5-1-44
A-1299	Judson, C.M., Turner, C.D.	Analysis of Boron Fluoride-Methyl Ether Complex, 7-27-44
A-2155	Judson, C.M.	Constituent Analysis of Boron Fluoride-Methyl Ether Complex, 11-15-44
A-2156	Judson, C.M.	Analysis of Exit Gases from Boron Trichloride Reduction, 11-30-44
A-2157	Judson, C.M.	Analysis of Constituents and Impurities in Boron Trichloride, 11-30-44
A-2158	Judson, C.M.	Complete Analysis of Boron, 11-30-44
A-3257	Inghram, M.G., Hustad, B.M., Nestor, C.H., Schwab, [illegible].G.	110 Mass Spectrometer, 3-21-45

M REPORTS

M- 918	Smith, J.S., Forstat, H.	Analysis of Gas Sample, Tower 8, 9-18-44

2 4 2 b

1453

INITIAL REPORT - CONTRACT NO. W-7418-eng-41
December 2, 1943

On November 19, 1943, the Standard Oil Company (Indiana) was approached by Brig. Gen. L. R. Groves of the U. S. Army Corps of Engineers as to its willingness to undertake the manufacture of a small amount of a critical chemical. On November 22, 1943 Dr. E. . Thiele of the Standard Oil Company visited the Madison Square Area Office of the Manhattan District to discuss the technical aspects of the proposed project. By letter of November 24, 1943 from Mr. E. G. Seubert to Gen. Groves, Standard Oil Company indicated its agreement to proceed with the design, construction and operation of the necessary facilities, provided a suitable contract were developed to provide for the reimbursement of the cost of construction and operation of the facilities. On December 2, 1943, the U. S. Engineers Office issued a letter of intent covering the proposed project and on December 6, 1943 this letter of intent was accepted by Standard. Subsequently, on January 5, 1944 the parties entered into Contract No. 7418-eng-41 dated December 2, 1943, covering the project.

The objective of the project is the erection and operation of facilities to produce "B" units per day of "X" product (letters "B" and "X" are defined in the "Secret letter" dated December 2, 1943), Hereinafter and in subsequent reports "B" will be considered to be 1.7 gallons (U.S.) and "X" will be called "Heavy Polymer." Heavy polymer is to be fractionated from "Polymer" which is produced by the reaction of "Synthans" with "Monomer."

The steps contemplated for the execution of the contract obligations and the originally expected completion dates for each step are indicated in the following tabulation:

Step	Description	Scheduled Completion Date
1.	Design of Pilot Plant Facilities	Feb. 1, 1944
2.	Preparation of Pilot Plant Site and Installation of Offices and Laboratory	March 1, 1944
3.	Erection of Pilot Plant Facilities	April 30, 1944
4.	Start up of Pilot Plant, Establishment of Equilibrium, and Production of Initial Product	June 2, 1944
5.	Operation of Facilities	-------

A-2

It is to be understood that the above schedule is contingent upon Standard's obtaining delivery at the necessary times of materials and supplies. It is also to be understood that Standard does not guarantee that production of heavy polymer can be started by the indicated date (June 2, 1944); however, Standard will use its best efforts to do so.

In addition to the above work necessary to the execution of the Contract, Standard expects to undertake work directed towards increasing the certainty of the early success of the proposed project. Items of major importance in this category follow:

1. Investigation of performance of high efficiency fractionation columns, particularly Stedman columns.

2. Investigation of the properties of polymer including:

    a. Relative volatility of light and heavy polymer
    b. Corrosion
    c. Decomposition
    d. Inflammability
    e. Toxicity

3. Training of technical personnel

4. Training of non-technical personnel

5. Establishment of an adequate security system from the standpoints of fire protection, espionage, and sabotage.

Work on the above items is of a continuing nature and no schedule is being established in advance.

/s/ J. K. Roberts
J. K. ROBERTS

lf-9-23-47

[redacted]

[redacted] 2 4 2 [illegible]

WAR DEPARTMENT
UNITED STATES ENGINEER OFFICE
MANHATTAN DISTRICT
Chicago Area Office
P. O. BOX 6140-A
CHICAGO 80, ILLINOIS

1233

IDM CGD-1

4 January 1944

Memorandum To: The Files.

Subject: Contract No. W-7418 eng-41 with the Standard Oil Company of Indiana.

1. The work being undertaken is the separation of isotope Boron 10 from Boron 11. The light component is desired at 95% purity. Boron tri-fluoride will be the basic material. This can be procured as a component. Boron tri-fluoride and methyl ether will form a complex which boils at about 200°C. It has been determined that the light complex has a different vapor pressure than the heavy complex and will boil off at a slightly lower temperature. It is believed that the separation can be obtained by distillation. The job is very difficult, however, because the difference of volatility is about one part in a thousand. It is contemplated that fractionating equipment consisting of about a thousand theoretical plates will be used and that the equipment would operate at about a thousand to one reflux ratios. Boron tri-fluoride and methyl ether are both gases but the complex is liquid. The equipment as contemplated will consist of nine columns 35 feet high. The first three will consist of thirteen 6" triangles, the second three of five 6" triangles, and third three of 6" diameter tubes. The chemical engineering job concerned is to get the holdup as low as possible and also prevent decomposition of the complex. This latter will not only ruin production but also cause corrosion of the distillating columns. To decrease corrosion, monel packing will be used. The components will be delivered as gases and it is expected that these will be formed in the complex at the site. This scheme has been worked out at Columbia and no difficulty is expected in effecting the formation ofthe complex.

2. The basic work for this method of separating boron isotopes has presumably been done at Columbia. Drs. Urey and Kilpatrick are the contacts for the Standard Oil people. Technical information for the start of design has been obtained in New York by Dr. Thiele and Mr. Conn from Ruhoff, Kilpatrick, and Urey.

3. Specifications call for the production of 1.7 gallons per day of complex 95% pure. This will produce in the order of 600 grams per day of the desired component. It is planned that the plant will be completed by 30 April and that the start up of operations will be 2 June 1944.

4. Standard Oil will operate under a prime contract calling for the design construction and operation of the pilot plant built to effect this

[redacted]

[redacted]

Subject: Contract No. W-7418 eng-41 with the Standard Oil Company of Indiana.

separation. Standard Oil is in the process of subcontracting the purchase or fabrication of the equipment and erection of the entire plant to the Foster-Wheeler Company. This Company has had experience in the fabrication of Stedman columns which will be used in the pilot plant.

5. At the present moment, the work is organized along the following lines:

- E. G. Seubert - President - Standard Oil Company (Ind.)
  - J. K. Roberts, Director of Research
    - R. C. Gunnes, Project Supervisor
      - Arthur H. Conn - Operations' Supervisor
        - Technical Men (about 7)
        - Operators, etc (about 25)
      - James Mather - Job Engineer
      - E. W. Thiele - Project Consultant
    - Wm. L. Webb - Administrative Assistant

Direct contact with job above this line
Indirect contact below

  - M. G. Paulus - Vice President - Manufacturing
    - S. A. Montgomery - Manager Whiting Refinery
    - D. A. Monro - Chief Engineer (acting)

[redacted] 2 4 2B

Subject: Contract No. W-7418-eng-41 with Standard Oil Company of Indiana

W-7418-eng-41 EIDM CGD-4 4th Wrapper Ind. 1453

U. S. Engineer Office, Chicago Area, Chicago, Illinois. 26 October 1945.
To: The District Engineer, Manhattan District, Oak Ridge, Tennessee Att: Captain W. W. Teeter

1. The information transmitted herewith is furnished in compliance with 3rd Wrapper Ind. This information has been obtained through personal contact with parties interested in the early history of the contract, principally Drs. H. C. Urey and Martin Kilpatrick, and is not substantiated by documents in the files of the Chicago Area Office. It is suggested that information be requested of the Columbia Area Engineer.

2. Dr. H. C. Urey, of Columbia University, was aware of the potential value of the separation of the boron isotopes and undertook a research program to effect such a separation. Dr. Martin Kilpatrick supervised the development program for Dr. Urey. When sufficient data had been accumulated to indicate the feasibility of such a program, and when a requirement for one of the isotopes had been submitted from Los Alamos, the Eastman Kodak Company of Rochester, New York, a firm engaged in the preparation of fine chemicals, was approached with a request to design, construct, and operate a plant capable of producing the desired fractionation. This concern declined to enter into a contract with the government for this purpose.

3. On 19 November 1943, Major General L. R. Groves and Dr. A. H. Compton proposed to Mr. E. G. Subert, President of the Standard Oil Company (Ind.), that his firm undertake an undisclosed project of great importance.

A-1

4. The Standard Oil Company is a firm with a reputation for competency and efficiency in refinery operations, principal of which is that of precise distillation. Certain members of the research staff, particularly Roberts, Gunness, and Thiele, were well-known to the research organization at Columbia University and were considered by this staff to be particularly capable to engage in such work. Mr. Thiele had been previously employed on the DSM Project on a leave of absence granted by The Standard Oil Company. On 22 November 1943, Mr. Thiele was sent to New York to confer with Messrs. Urey and Kilpatrick, and the nature of the proposed activity was disclosed to the desired separation, and on 24 November 1943, Mr. Subert informed General him. He reported to Mr. Subert that the Standard Oil Company could accomplish Groves that the Standard Oil Company (Ind.) was willing to enter into a contract for such services.

5. On 4 December 1943, Major Harold Greenstein presented to the Standard Oil Company (Ind.) a letter-contract dated 2 December 1943. The Chicago Area Office was asked to render administrative services such as priority assistance and certification of deferment requests, for the Standard Oil Company. On 19 January 1944, the Area Engineer, Madison Square Area, transferred

[redacted]

2 a

to the Chicago Area Office, the entire responsibility for the administration of the contract. The original estimate of the cost of the contract was $300,000, which estimate was raised to $600,000 prior to the execution of the instrument. The contract was later supplemented to provide additional funds so that the total value of the contract is now $2,300,000. The contract was negotiated on the basis of cost. The cost is determined as the actual expenditure for this facility, plus operating costs, labor and overhead. The overhead is calculated as 100% of labor, labor being construed to include only operating personnel and not service and maintenance. This calculation is based on the precedent established by a previous contract between the OSRD and the Standard Oil Company (Ind.). The principal item of cost was the design and construction of the facility. This was performed under a subcontract by the Foster Wheeler Corporation of New York, New York. The construction cost is estimated to be $1,225,000.

For the Area Engineer:

T. S. CHAPMAN,
Captain, Corps of Engineers,
Assistant

1 Incl.: N/C

This document consists of 18 pages
No. 2 of 3 copies, series B

[redacted]

HISTORY OF

CONTRACT NO. W-7418 eng-41

STANDARD OIL COMPANY (INDIANA)

1458

1. Objective

Early in the history of the DSM project, Dr. H. C. Urey of Columbia University became aware of the potential value which lay in the fact that the boron isotope of lesser abundance was one of the atoms of greatest cross section known. Under his direction, a program was undertaken at the SAM Laboratories to devise a method of separation of this boron isotope. The result of the study was that the most feasible method of separation was a fractionation by distillation. The principle under which this fractionation is accomplished is still uncertain but the empirical results obtained with the ether complexes of boron trifluoride indicated that a sufficient difference in volatility existed for the successful accomplishment of the separation.

2. Selection of Contractor

The Eastman Kodak Company of Rochester, New York, who have a reputation for the preparation of high-grade chemicals, including a large number which are processed by careful distillation, were considered first as a contractor to design, construct and operate a plant capable of producing the desired fractionation. This company declined to enter into a contract with the government for this purpose.

Since distillation is the foundation of the petroleum refining industry, The Standard Oil Company of Indiana was then approached. The reputation of the Standard Oil Company of Indiana for industry and efficiency in the accomplishment of difficult tasks was well known. Columbia University and others were acquainted with the capabilities of certain members of the research staff, particularly Messrs. Roberts, Gunness, and Thiele. Mr. Thiele had been employed previously by the DSM

[redacted]

A

[redacted]

project on a leave of absence granted by the Standard Oil Company. On 22 November 1943, Mr. Thiele was called into conference with Messrs. Urey and Kilpatrick and the proposed activity was disclosed to him. He agreed that the Standard Oil Company could build a pilot plant which would produce about 600 grams of boron of a concentration of about 95% of the 10 isotope by the method of distillation which had been devised by Urey and co-workers. Direct negotiation between Major General Groves and Mr. J. K. Roberts of Standard Oil Company resulted in the acceptance by the Standard Oil Company of such a contract (No. W-7418 eng-41) on 2 December 1943.

At this time, the Chicago Area Office was assigned by the District Engineer to perform certain functions with respect to the contractor, namely those related to matters of administration such as auditing disbursements, expediting deliveries of materials and equipment, and processing deferment requests. The Technical direction remained under the Madison Square Area until 19 January 1944, when the entire responsibility for the administration of the contract was transferred to the Chicago Area Engineer.

3. <u>Plant Design</u>

After the nature of the problem was disclosed to the contractor, it was necessary to make a very early decision with regard to the method and means which would be used to effect a solution. Immediate decisions were necessary with regard to types of equipment required and materials of construction in order that orders might be placed with some hope of obtaining delivery at a date which would permit the construction of the plant and the production of the isotope in coordination with the delivery schedule of products which it was to supplement. Accordingly, other industrial facilities having experience in certain new and pertinent matters of equipment and material were interviewed, and the SAM

[redacted]

[redacted]

laboratories were requested to undertake a number of tests in order that sound design decisions might be made as soon as possible. In December 1943, the decision was made to perform the fractionation in a plant consisting of six distillation columns of graduated size to operate in cascade. These columns were to be fabricated of monel metal and were to be packed with Stedman packing. Three additional columns were to be erected as stand-by equipment.

After a preliminary review of the factors involved in construction, Standard Oil awarded to the Foster Wheeler Corp. a subcontract for the design and construction of the fractionation equipment. This procedure was selected as the most expeditious for the rapid construction of the desired facilities. In order that design and construction work on the project could proceed forthwith, the available process information was employed for the prompt establishment of the process design basis for the pilot plant. Simultaneous with the mechanical design and construction of the fractionation unit, Standard had undertaken the development of more adequate information on the process requirements of the project and the performance characteristics of high efficiency fractionation columns.

a. Factors Considered

Although the separation of the normal [illegible]ate, hereafter called polymer, would appear to be a relatively simple distillation problem, the operation is complicated by the irreversible decomposition of polymer into liquid and gaseous by-products. This decomposition occurs to an appreciable extent even at the lowest temperatures which are practical for operation. Furthermore, the large number of theoretical plates required and the low pressure drops permissible lead to the use of several columns in series. A careful analysis is required of the factors affecting design in order to permit the selection of the optimum equipment.

[redacted]

The primary factors affecting the process design may be listed as follows:

a. Relative Volatility, Theoretical Plates and Reflux Ratio

b. Decomposition

c. Pressure

d. Holdup

e. Number of Columns.

(1) Relative Volatility

The relative volatility, α, of the two components being separated has been found to lie in the range of 1.010-1.018, which corresponds to a difference in boiling points of only about 0.4 to 018°F. In order to separate two materials boiling so closely together, it is necessary to employ an unusually large number of plates and an unusually high reflux ratio. This is illustrated by the following values, calculated for the production by a single column of a bottoms product containing 95% 10 isotope, and an overhead product containing 90% 11 isotope.

	Minimum Plates	Minimum Reflux Ratio (Reflux/Bottoms)
1.010	518	498
1.012	432	415
1.015	346	333
1.018	289	278

The large change in both the minimum plates and minimum reflux ratio for a small change in   is to be noted. It is to be emphasized that the minimum number of plates and the minimum reflux ratio represent limiting values and that the actual number of plates and actual reflux ratios which must be used are considerably greater.

(2) Decomposition

The irreversible decomposition of polymer into liquid and gaseous by-products is an extremely important factor since the extent of decomposition limits the purity of the product which can be made by direct fractionation. The extent to which decomposition of heavy polymer occurs is determined by the decomposition rate (per cent per day) of the polymer and the holdup of heavy polymer within the unit, hence it is important that both factors be kept to a minimum.

The decomposition rate has been found to be a function of the temperature, and hence the distillation pressure. For example, at a temperature of 195°F and a pressure of 150 mm Hg absolute, the decomposition rate is 0.2%/day, while at 220°F and 290 mm, it is 0.6%/day. Although operation of the columns at the lowest obtainable pressure would assure the minimum decomposition rate, the use of such a low pressure would result in large diameter towers and increased holdup in the system. The resultant effect might be an increased extent of decomposition despite a lower decomposition rate.

(3) Pressure

For the system in question, the available data indicated no effect of pressure on relative volatility, and therefore no effect of pressure upon reflux ratio. Accordingly, with the vapor and reflux duty established by the fractionation requirements of the operation, the required cross sectional area of the column becomes a function of the column pressure. The relationship is such that the required cross sectional area for a given reflux load is approximately proportional to the inverse of the square root of the absolute pressure.

For a given diameter column, the use of a higher pressure will permit a greater reflux capacity which will in turn permit the use of a smaller number of plates to achieve a given separation. With a given diameter column containing a given number of plates, increasing the pressure will make possible the production of a higher purity product, or an increased quantity of a given purity product except as decomposition interferes.

In considering the effect of distillation pressure on the design and operation of the unit the effect of pressure drop through the columns must not be overlooked. This is especially important since it is the operating pressure at the top plus the pressure drop through the column which determines the pressure and temperature in the reboiler, where the largest amount of decomposition occurs. Therefore, for a given type of column, the pressure drop per foot of height will be the factor which determines the maximum permitted height and hence the number of columns required.

(4) Holdup

Since the amount of material decomposed is directly proportional to the total holdup in the system, it is important that holdup be kept to a minimum. This can be accomplished by using as few towers as possible, thus reducing the number of reboilers, and by making the diameters of the towers small. Consideration of the fractionation requirements leads to the conclusion that the reflux ratio can be materially reduced as stripping progresses, without greatly increasing the plate requirements. This makes it possible to reduce successively the sizes of the towers, with the result that the tower containing the largest concentration of heavy polymer has the smallest holdup. Obviously, this is extremely effective in reducing decomposition of the heavy polymer.

Holdup is also an important factor in determining the time required to start up the plant, and in determining the time required for the system to come to equilibrium after a change in operating conditions.

(5) Number of Columns

Since the unit is to be operated essentially as though it were a single distillation column, it is important that the number of columns used be kept to a minimum in order to keep the problem of control as simple as possible. However, the total number of plates required is

[redacted]

so large that [illegible] precludes the construction of the equipment as a single column. In order to have the minimum number of columns, it is desirable to employ a column with a low pressure drop, low holdup of liquid within the column, and a low height equivalent to a theoretical plate.(H.E.T.P.)

(b) <u>Summary of Design Factors</u>

The factors involved in the design may be summarized as follows:

1. The low relative volatility requires the use of a large number of plates and a high reflux ratio.

2. Decomposition is a function of the pressure of operation, the pressure drop through the column, and the holdup. For a given reflux ratio, a decrease in pressure will reduce the decomposition rate, but this is balanced by the increased holdup due to the increased size of the column. It is important that the pressure drop through the columns be kept low.

3. Holdup is also important in determining the time necessary to bring the columns to equilibrium.

4. In order to keep the number of towers to a minimum, it is important that the type of column chosen be one that operates with a low pressure drop, a low holdup, and a low height equivalent to a theoretical plate.(H.E.T.P.)

In selecting the type of column for the project it was recognized that the desired production rate, together with the high reflux ratio, requires a tower larger than twelve inches in diameter at the top of the stripping section and probably not smaller than six inches in diameter at this point. Furthermore, the time schedule to be met requires the selection of columns which are commercially available in this size.

[redacted]

b. Fractionating Columns Considered

The types of fractionating columns considered for this project are tabulated as follows:

(1) Empty Column--
    Wetted Wall
    Concentric tubes

(2) Packed Column--Formed Packing
    "Heligrid"
    Spiral Screen
    Stedman
    Stedman--Weitkamp Modification
    Rotating Column

(3) Packed Column--Dumped Packing
    Berl Saddles
        Solid
        Gauze
        Raschig Rings
        Metal Helices

(4) Plate Column--
    Bubble Cap
    Perforated Plate

Since all of these columns are adequately described in the literature, no attempt will be made to describe them here.

The empty columns were not considered practical for this job in view of the large number of tubes required, resulting in turn in an extremely difficult liquid distribution problem.

Of the packed columns with formed packing, only the Stedman appeared to be worthy of consideration. Although the "Heligrid" packing is available commercially, it is not commonly constructed in sizes over 1". The spiral screen type is not available commercially, and in addition appears limited to columns of about 1" diameter. The rotating column did not appear desirable due to mechanical difficulties which could be anticipated due to moving parts.

Of the packed columns with dumped packing, all of the types except the gauze Berl saddles were available commercially at the time of decision and were considered. Since the construction of the plant, gauze

[redacted]

saddles had become available and had been investigated. They have been found to compare favorably with Stedman packing. However, even at the present time facilities for production of saddles are limited and considerable time is required for their fabrication.

It was found that a bubble cap column of special construction had been developed by the M. W. Kellogg Company in which plate spacings of 1-1/2 to 2 inches were used without excessive entrainment. This had been constructed in diameters as small as 6 inches, and therefore appeared worthy of careful consideration.

Examination of the characteristics of operation of perforated plate columns indicated that they were inapplicable for the job. Although possessing the advantages of low pressure drop and low holdup, their disadvantage is an extremely narrow operating range with the small plate spacing required for this job. For example, it was found that the effect of decreasing the plate spacing was to decrease the operating range between the minimum vapor rate for good efficiency and the point at which entrainment became excessive. Extrapolation to a plate spacing of 2" indicated that this operating range became much too small for practical consideration.

The choice of columns for this project was thus narrowed down to packed columns such as Stedman, Berl Saddles, Raschig rings, and metal helices, and a bubble cap column of the Kellogg type.

A comparison of these types of columns with the exception of Berl Saddles, about which very little was known at the time, showed that Stedman packing was outstanding in all three respects. Metal helices give almost as low an H.E.T.P. as Stedman, but the pressure drop is higher. Raschig rings are definitely inferior in all three respects, while Berl Saddles give several times the holdup and a very high H.E.T.P. It is probable that smaller Berl Saddles would show a lower H.E.T.P.;

 [redacted]

however, the holdup and pressure drop would then undoubtedly be higher. The bubble caps show about twelve times the holdup of Stedman columns, a higher pressure drop, and higher H.E.T.P.

(6) Stedman Column Selected

It appeared evident that Stedman columns were outstandingly superior to all other types of columns which could be used for this project. This conclusion was verified by discussion with men familiar with all phases of high efficiency distillation. The above factors combined with the commercial availability of this type of packing led to the selection of Stedman columns for this project.

Stedman packing is fabricated from wire cloth 60 x 40 meshes per inch, using wire .009 inch in diameter. The cloth is punched, blanked and embossed to form sheets having a regular pattern of raised triangular pyramids, a pattern of holes located between the pyramids, and an upturned lip around the circumference. The sheets are placed one above the other so that the so-called "valleys" of one sheet are in contact with the pyramids on the sheet below, at which points the sheets are spot-welded together. The sheets are welded together in units containing 100 sheets and measuring one foot in height.

The operation of the packing is as follows: The liquid flows along the wire cloth and seals the mesh, while the vapor passes upward through the holes in the sheets. Good liquid distribution is maintained by the repeated division of liquid streams into three portions as they pass down the sides of the triangular pyramids and recombine with other streams in the valleys. Godd vapor contact is assured by the fact that the hole of one sheet is always located directly below the apex of a pyramid in the sheet above.

The packing is fabricated in circular sizes up to 6 inch diameter, and in 6 inch equilateral triangles. Larger sizes are made

[redacted]

by combining triangles into various patterns to form hexagons and other shapes. For example, a 12 inch column is made of six triangular sections and is hexagonal in shape. An 18 inch column is made of thirteen triangular sections in the form of a hexagon having three twelve inch sides and three six inch sides.

In determining the number and sizes of Stedman columns to be used on this project, a large number of combinations were considered and studies were made of each for different values of relative volatility of the two components to be fractionated. It appeared that with a value for the relative volatility of 1.010, accomplishment of the desired separation with six columns is somewhat uncertain due to the relatively high pressure required, whereas with α equal 1.015, fewer than six columns could probably be used. The combination decided upon was to consist of six columns, including two 18" hexagonal, two 12" hexagonal, and two 6" circular columns. This combination is the result of a compromise between theoretical considerations which indicate that six towers of progressively decreasing diameter would be most effective, and practical considerations, which indicate that the towers should all be the same size for interchangeability and maximum flexibility.

In order to offset major process uncertainties which existed at the time the design was established, particularly with respect to α and H.E.T.P., and in order to provide adequate spare facilities in the event of corrosion, leakage, or mechanical difficulties, it was decided to provide an extra column of each size, thus making a total of nine columns. Each column has a complete set of instruments and auxiliary equipment and the piping is so manifolded that any or all of the columns can be used. The wisdom of providing this spare equipment is evidenced by the fact that current operation requires the employment of seven columns instead of the six calculated from the uncertain data

[redacted]

available at the time of design.

Data obtained after the fabrication of the columns had begun indicated a deleterious effect of height on the H.E.T.P. obtained with Stedman packing and hence in the final design of the unit, five redistributor plates were introducted in each column in order to insure that the required H.E.T.P. be obtained. The use of the redistributor plates limited the maximum height of packing between plates to five feet and resulted in a total decrease in packed height of about 10%.

(6) Construction Materials

Since it was necessary to place orders for materials at an early date, the materials of construction for this unit were selected on the basis of a limited amount of data and such other experience as could be brought to bear on the problem. Recent commercial experience with hydrofluoric acid alkylation plants had indicated that the presence of a small amount of moisture caused rapid corrosion of either carbon steel or stainless steel. Since polymer in the presence of moisture might hydrolyze to form hydrofluoric acid, and since moisture might conceivably enter the system through an air, steam, or water leak, it was decided to use monel wherever possible for equipment which might be difficult to replace.

The towers are entirely monel except for carbon steel flanges. It was necessary to make the shell of monel in order to prevent fouling of the packing by scale, to insure maintenance of closely machined tolerances, and to eliminate the possibility of accelerated corrosion rates due to the presence of two dissimilar metals. In the case of the flanges it was felt that the use of monel was not justified since the flanges could not be readily contacted by polymer, and since there was a large thickness of metal present in case the polymer did attack the flanges. Furthermore, a delay might be anticipated in obtaining monel 3" thick.

 [redacted]

[redacted]

The reboilers and condensers are entirely of monel except for the reboiler shells. The use of carbon steel in this instance was based on the fact that any moisture leaking into the system could remain in reboiler for only a negligible amount of time, since the boiling point of water is considerably below that of polymer.

The diaphragms for the pumps are made of monel, while the check valves are of stainless steel. The packless valves have forged steel bodies with monel and copper trim. All other pieces of equipment, including coolers, drums, and piping, could be readily replaced and hence were made of carbon steel. Furthermore, it was felt that any necessary changes in this equipment could be more readily made in the field with carbon steel, due to familiarity with its welding characteristics.

Experimental work at [illegible] Laboratories later showed stainless steel to be as good as monel when tested with polymer alone or with polymer in the presence of small amounts of moisture. However, with polymer in the presence of steam, stainless steel showed the rapid corrosion rate which had been anticipated, whereas monel was only slightly affected. Furthermore, investigation of the welding and machining characteristics of various stainless steels showed them to be inferior to monel, since stainless steels which were readily welded were difficult to machine and those which were easily machined were difficult to weld. Hence it was found desirable to use monel in spite of procurement difficulties which were encountered.

About 18 February 1944, difficulties were encountered by Foster Wheeler Corp. in the procurement of monel from International Nickel and in the placing of orders with fabricators who were experienced in the workings of this material. Expediting assistance was rendered by the Washington Liaison Office, Madison Square Area Office, Chicago Area Office, and the Standard Oil Company with the result that these difficulties were resolved. An important contribution to the early delivery of these essential monel parts was the cooperation of various naval ordnance plants

[redacted]

[redacted]

in the machining of the tower sections.

4. Initial Operations

In order to meet early delivery schedules, an attempt was made to place each unit of the plant in production as fast as its assembly was completed, without the deliberate and extensive testing procedure which would precede the operation of such a novel plant under normal conditions. As might be expected in an attempt to go from incomplete laboratory experimental work to production without intermediate trials on a pilot scale, unforeseen difficulties were encountered. Chief of these difficulties were those related to leaks and an entirely new system of leak detection was developed. By direct comparison at the plant it was found that this leak detection was required to find leaks which could not be detected by the helium mass spectrograph "sniffer", the most exhaustive leak detecting instrument known at present. Another major difficulty was encountered in the unsatisfactory performance of the pumps under operating conditions. The pumps were tested prior to installation but these tests were interrupted and, hence, incomplete, due to the necessity for incorporating the pumps in the operating unit. Standard Oil Company immediately redesigned the pump and the vendor fabricated new pumps in accordance with this design. The new design pumps have given satisfactory service.

During the leak testing procedure, mercury was introduced inadvertently into the system from the pressure relief chambers. These pressure relief chambers had been constructed so that an external pressure greater than atmospheric was required to cause the flow of mercury into the system. However, an extraordinary occurrence of events such as emulsification, loss of mercury by flowing outward, and "rocking" did result in mercury contamination of the operating system. Contrary to published literature and the experience of all concerned, including the operators, the sub-contractor, the research facilities at SAM, and the vendors of monel; the mercury did attack the monel under the peculiar conditions

[redacted]

[redacted]

of operation and brought about structural failure of the component parts of the system. It was later confirmed by laboratory demonstration that under a number of unusual conditions which must be present at the same time, mercury does so attack monel metal.

5. Supplementary Plant Considered

Chiefly because of the structural failure of the first plant but also from consideration of the fact that the first plant had been built without a pilot plant and subsequent experience had shown a large number of possible improvements in design, the contractor proposed that a new plant be designed and constructed in order to insure satisfactory production. It was proposed to construct this new plant of three columns rather than six in order to decrease the number or valves and heat exchangers which appeared to be the foci of leaks and, thus, the chief causes of fundamental difficulties. The reduction in the number of reboilers would also be advantageous in view of the unexpectedly large decomposition which had been experienced in the reboilers. The material of construction was to be carbon steel, which, in spite of its faster corrosion, did not react with mercury under the unusual conditions which might occur again through accident.

After consideration by higher authority, it was decided to proceed with a second, completely new, fractionating plant embodying the new design features. In the meantime, the first plant was to engage in a testing program to determine the best conditions of operation in order that these features might be incorporated in the design of the second plant. The placement of orders for material and other preparations for the new plant were to proceed without delay. In order to save the time necessary for the preparation of a foundation adequate to support the required structure, the Standard Oil Company agreed to the use of the foundation of an existing but abandoned structure, identified as No. 5 Boiler House. It was

[redacted]

necessary to raze the walls of this existing structure to a large extent since they would constitute a hazard to personnel working in the vicinity. Immediate action was taken in the preparation of the site and the placing of orders for new packing and column sections. The packing and column sections were to be identical with the corresponding portions of the existing plant so that they would serve as mutual spare parts in the event of replacement if such became necessary.

While proceeding with the experimentation on the original plant, the contractor discovered (a) that mercury could be removed from the system and the structural failures almost completely stopped, (b) that leaks could be controlled, (c) that, in spite of failure to perform in accordance with predictions from laboratory data and design calculations, the plant was operable, at least in a slow and onerous batch fashion. Accordingly, a quantity of 50% material and later 80% material was prepared by a variety of batch and semi-continuous operations and delivered to the government for further processing and final utilization by the requesting agency.

About 9 January 1945, the government reconsidered and instructed the contractor to proceed no further with the procurement and erection of the second plant, but instead to produce to the best of their ability upon existing facilities and to utilize such equipment as was already procured to repair and maintain the present facility in an operating condition. By testing and adjusting the various units of the plant, the contractor has brought them, one after another, into integrated operation so that successful continuous operation has been attained. Despite the fact that laboratory predictions were not realized and design efficiency was not possible, the skillful operation of the contractor has resulted in the production of a quantity of 80% enriched material, and more recently, in the production of more than 90% material upon a continuous and uninterrupted basis. Operating conditions are still being improved and the desired concentration of 95% is now forthcoming.

6. Future Possibilities

The future operation of the plant is indeterminate. For reasons of security, the exact operation and need underlying this procurement requirement is undisclosed. The contract specifies production at a certain rate for the duration of the war and six months thereafter. In the event that there is a requirement only for a specific quantity of this material, its unique nuclear properties make it useful for other purposes which may justify the operation of the plant for additional quantities. One of the most universal of these additional uses is the employment in neutron detecting instruments. Already, laboratories which have learned of the existence of this material have made request for quantities of the material for such a purpose. A relatively small portion of the production is sufficient to satisfy these needs. However, since the process material is extremely corrosive, it is probable that a prolonged period of idleness would render the plant inoperable. It is doubtful that it will ever be economically feasible to operate this plant or another for the sole purpose of supplying this material for instruments except during the current period of operations.

In addition to potential operations utilizing the process material for which it was designed, the plant represents a completely astounding fractionating instrument. Hence, although its design may render it inefficient for practical production purposes, it is a very valuable instrument upon which to attempt the fractionation of other isotopes by distillation method. If a volatile compound of uranium could be discovered in which the two isotopes had a different relative volatility, the relatively simple distillation process could be used to displace the intricate and cumbersome methods now in use for the separation of the isotopes of this element. Not only would the design features of the present plant be incorrect for practical use in separation of uranium isotopes, but is is likely that the previous use with boron compounds would render the isotopes of uranium produced unsuitable for nuclear purposes. The actual feasibility of the separation process, however, could be demonstrated by means of the powerful tool which exists in the plant. Although the existence of a suitable compound of uranium is not known at this time, it is entirely conceivable that such a compound may be discovered.

Likewise, it is conceivable that needs will arise for isotopes of other elements and that volatile compounds of these elements may exist in which the isotopes differ sufficiently in volatility that they may be separated by distillation. The present facility, in all probability, is not so designed and constructed that an efficient separation might be accomplished but it does represent a very powerful machine which might be employed for experimentation in order to determine the data from which a production facility might be designed and erected.

[redacted]

EIDM CGD-4
S10-18

TSC/VF/lf-9-22-47

29 April 1944

Subject: Vitamin.

To: Dr. J. R. Oppenheimer, P. O. Box 1663, Santa Fe, New Mexico.

1. Confirming telephone conversation between Dr. Oppenheimer and Captain Chapmen of this office of 28 April 1944 and teletype of 29 April 1944, the immediate status of the vitamin project is submitted for your information.

2. The vitamin facility under construction and to be operated by the Standard Oil Company (Indiana) consists of 18 monel metal towers filled with Steadman Packing. In detail, there are 3 - 6" cylindrical towers and 3 - 12" hexagonal towers and 3 - 18" hexagonal towers. The design capacity of the equipment is such as to provide 600 grams of Boron per day in the form of the tri-fluoride dimethyl ether complex and of 95% B^{10} isotopic concentration. Initial delivery date as specified by the contract was 2 June 1944 but due to difficulties in obtaining monel metal, this initial delivery date had been changed to 3 July 1944.

3. Delivery of monel metal was later than in accordance with the established schedule. When it was received, it was found to be in an unsuitable form due to scale and irregular surfaces and further delay was incurred in preparing the plate for fabrication. As a result of these delays, the completion dates of the fabricated sections of the hexagonal towers have been placed at around 60 days from 8 May 1944. There are two authentic but conflicting statements with regard to the date on which the sections can be completed. The exact delay can not be determined at this time. Efforts are being made to place part of the tower machining in other shops in order to expedite final completion date of the entire facility.

4. The cylindrical columns, due to design and size offer smaller machining problems and are expected to be complete not much later than schedule. By rescheduling the hexagonal columns, it is anticipated that at least 2 of the 12" columns can be completed prior to the completion of the 3 -6" columns. Preliminary engineering studies have indicated that with these 5 columns a production rate approximately 75% of the contracted rate and of specification concentration can be attained. The contractor has been instructed to proceed with the fabrication and erection on a schedule which will allow the initial production date to be realized at this reduced rate. Expediting and completion of the entire facility will be continued with no dimunition of intensity.

[redacted]

5. The Chicago Office has been informed informally and unofficially that a redetermination of alpha allows the prediction that this facility will be able to produce the B^{10} complex at a rate greatly in excess of that specified in the contract. It is, therefore, believed that any loss in accumulative production sustained by this reduced operation can be regained in a short period of time after the entire facility is completed and in operation.

6. It is contemplated that delivery of the complex will be initiated at a rate of 450 grams a day based upon B^{10} content on 3 July. This material will be delivered to the Madison Square Area for reduction to the crystalline material in a facility under the supervision of that office.

For the Area Engineer:

T. S. CHAPMAN,
Captain, Corps of Engineers,
Assistant.

Final Report - Contract No. W-7418-eng-41

July, 1947

Following the initiation of the project under Contract No. W-7418-eng-41, dated December 2, 1943, a fractionating unit for the production of heavy polymer was designed, constructed, and successfully operated for a period of 14 months. During the period of operation a total of 726 gallons of heavy polymer was produced. In accordance with instructions received from the U.S. Engineer Office the unit was shut down on February 4, 1946 and was placed in stand-by condition. In accordance with an amendment to the contract, dated February 1, 1946, the unit will be maintained in stand-by condition until January 31, 1948 unless the Engineer Office directs its earlier disposal.

Process design of the fractionating unit was begun on December 6, 1943 and shortly thereafter a sub-contract was entered into with the Foster Wheeler Corporation for the design and erection of the plant. After numerous difficulties were encountered, many of which required extensive modifications of the plant, successful operation was attained about one year from the start of the project. During the last part of the first year, a second unit was designed and plans were made for its installation. However, this unit was abandoned before its erection was started when it became apparent that the first unit could be made to operate properly. Troubles which were encountered in the erection and operation of the first unit included difficulties in procuring and fabricating Monel, mechanical failure of Monel column sections and heat exchangers, and difficulties associated with pumps, valves, and column packings. These difficulties are discussed in detail in Report No. A-2370 issued by the Standard Oil Company under date of July 23, 1946 and entitled "Construction, Operation, and Maintenance of a Polymer Fractionation Unit". This report also contains a number of recommended changes in design which should be considered should a similar unit be constructed in the future. Completion dates for the principal phases of the project work are shown below:

Design of major equipment completed	March 1, 1944
Preparation of pilot plant site and installation of offices and laboratory completed	March 1, 1944
Erection of pilot plant facilities completed and preliminary attempts at operation begun	July 22, 1944
First successful cross-flow operation established	November 28, 1944
Production of 80 per cent heavy polymer attained	January 5, 1945
Production of 95 per cent heavy polymer attained	March 30, 1945
Operations terminated	February 4, 1946

During the period of successful operation, product was withdrawn from the unit more than 98 per cent of the elapsed time. The material produced by the unit consisted of 80 gallons of 80 to 90 per cent heavy polymer, 95 gallons of 90 to 95 per cent heavy polymer, and 551 gallons of material having a heavy polymer concentration of over 95 per cent. More detailed information regarding the material produced is shown in Table I.

SECRET

TABLE I

Material Produced During Term of Contract

Lot No.	Gals.	Mol % Heavy Polymer	Wt. % Iron	Wt. % Water by Karl Fischer	Specific Resistance at 25°C.
1	5	53.2	0.01	0.12	350 ohms
	245 cc*	53.3	-	-	-
2	5	80.5	0.03	0.24	215
3	5	80.6	0.02	0.28	250
4	5	80.7	0.03	0.5	225
5	5	83.0	0.02	0.2	240
6	5	82.9	0.08	0.17	215
7	5	83.6	0.04	0.14	270
8	5	81.6	0.03	0.32	210
9	5	82.5	0.03	0.30	210
10	5	83.7	0.06	0.22	260
11	5	82.6	0.05	0.18	280
12	5	82.0	0.02	0.26	400
13	5	81.3	0.03	0.15	270
14	5	84.6	0.03	0.13	250
15	5	84.4	0.04	0.17	300
16	5	83.3	0.03	0.32	155
17	5	81.8	0.12	0.22	470
18	5	83.0	0.02	0.14	175
19	5	81.4	0.04	0.24	160
20	5	90.7	0.03	0.10	280
21	5	92.3	0.02	0.24	200
22	5	92.2	0.02	0.23	200
23	1	93.9	0.03	0.4	170
24	5	93.9	-	0.15	250
25	5	93.7	0.04	0.14	250
26	5	95.0	0.02	0.04	310
27	5	94.9	0.03	0.08	280
28	5	95.3	0.02	0.14	260
29	5	95.0	0.01	0.14	280
30	5	95.0	0.01	0.11	230
31	5	94.9	0.03	0.07	280
32	5	94.8	0.01	0.09	320
33	5	94.9	0.01	0.08	290
34	5	94.3	0.01	0.06	990
35	5	93.5	0.02	0.04	410
36	5	94.4	0.02	0.04	460
37	5	94.1	0.03	0.09	340
38	5	93.7	0.02	0.14	415
39	5	93.5	0.01	0.05	415
40	5	93.7	0.04	0.11	280
41	5	92.9	0.03	0.16	280
42	5	93.1	0.04	0.11	330
43	5	94.6	0.2	0.08	380
44	5	94.9	0.01	0.09	460
45	5	94.3	0.01	0.09	370

SECRET

*Additional Sample.

SECRET

TABLE I (Cont'd.)

Material Produced During Term of Contract

Lot No.	Gals.	Mol % Heavy Polymer	Wt. % Iron	Wt.% Water by Karl Fischer	Specific Resistance at 25°C.
46	5	94.9	0.01	0.06	700 ohms.
47	5	95.2	0.01	0.08	1340
48	5	95.7	0.01	0.07	850
49	5	95.8	0.01	0.05	1450
50	5	95.6	0.005	0.03	1350
51	5	95.4	0.005	0.05	1900
52	5	95.5	0.005	0.06	1100
53	5	95.3	0.005	0.06	1400
54	5	95.1	0.005	0.09	770
55	5	95.1	0.005	0.08	795
56	5	95.2	0.005	0.06	410
57	5	95.2	0.005	0.06	700
58	5	95.6	0.01	0.08	390
59	5	95.6	0.01	0.08	420
60	5	95.5	0.02	0.06	420
61	5	95.5	0.01	0.10	360
63	5	95.2	0.02	0.07	390
64	5	94.5	0.02	0.15	250
65	[illegible]	96.3	0.02	0.07	360
66	20	95.9	0.01	0.05	640
67	20	95.8	0.00	0.08	680
68	20	96.0	0.005	0.03	432
69	20	96.2	0.005	0.05	526
70	20	95.8	0.005	0.02	1335
71	20	95.8	0.005	0.03	1115
72	20	95.5	0.005	0.04	1018
73	10	95.3	0.005	0.04	1091
74	15	95.4	0.005	0.06	1062
75	5	95.3	0.005	0.08	533
76	10	95.4	0.005	0.03	1683
77	30	96.1	0.005	0.04	1344
78	29	96.4	0.005	0.03	1117
79	30	96.3	0.005	0.02	1140
80	35	96.4	0.005	0.03	1249
81	30	95.9	0.005	0.03	843
82	30	96.0	0.005	0.03	976
83	35	95.8	0.005	0.06	1722
84	15	96.5	0.005	0.04	718
85	3	96.2	0.005	0.04	1492

TABLE II

Reports Issued During Term of Contract

Report No.	Title	Author(s)	Date Issued	Distribution
	A Reports			
A-1975	Design of Pilot Plant for No. 4 Process Laboratory	A.L. Conn J.B. Wolf	5/24/44	Series A Distribution 1. Area Engineer, Chicago 2. " " " 3. " " " 4. " " " 5. Area Engineer, Madison Square, Capt.W.M. Hearon 6. Area Engineer, Columbia, Capt. C.D. Luke 7. H.C. Urey 8. M. Kilpatrick 9. J.K. Roberts 10. " " " 11. R.C. Gunness)Kept at No.4 12. A.L. Conn)Process 13. E.W. Thiele)Laboratory Series B Distribution 14. Area Engineer, Chicago 15. " " "
A-2350	Liquid Distribution in Stedman Packing	W.L. Forsythe,Jr. J.E. Wolf	6/26/45	1. Chicago Area Office 2. " " " 3. " " " 4. " " " 5. " " " 6. " " " 7. J.K. Roberts 8. H.C. Urey 9. L.B. Bragg 10. A.L. Conn 11. " " " 12. " " "

TABLE II (Cont'd)

Reports Issued During Term of Contract

Report No.	Title	Author(s)	Date Issued	Distribution
A-2351	Liquid Feed Preparation Unit	T.G. Stack J.E. Wolf	7/13/45	1. Chicago Area Office 2. " " " 3. " " " 4. " " " 5. " " " 6. " " " 7. J.K. Roberts 8. H.C. Urey 9. A.L. Conn 10. " " " 11. " " " 12. " " "
A-2352	Analysis of Vent Gases from the Fractionation Unit	D.A. McCauley W.L. Rittschof	7/24/45	Same as A-2351
A-2353	The Separation of Polymer from Polymer-Water Mixtures	E.N. Hiebert W.L. Rittschof A.L. Conn	7/31/45	Same as A-2351
A-2354	Investigation of the System Methyl Ether and Methyl Borate-Boron Fluoride Complex	D.A. McCauley W.L. Rittschof	8/2/45	1. Chicago Area Office 2. " " " 3. " " " 4. " " " 5. " " " 6. " " " 7. J.K. Roberts 8. H.C. Urey 9. A.L. Conn 10. " " " 11. " " " 12. Chicago Area Office
A-2355	Dissociation of the Methyl Borate-Boron Fluoride Complex	D.A. McCauley	9/11/45	Same as A-2351

TABLE II (Cont'd.)

Reports Issued During Term of Contract

Report No.	Title	Author(s)	Date Issued	Distribution
A-2356	Operation of a Podbielniak Heligrid-Column	F.L. Rittschof A.L. Conn	10/2/45	1. Chicago Area Office 2. " " " 3. " " " 4. " " " 5. " " " 6. " " " 7. " " " 8. J.K. Roberts 9. A.L. Conn 10. " " " 11. " " " 12. " " "
A-2357	An Interpretation of the Isotopic Separation in the Distillation of Methyl Ether-Boron Fluoride Complex	D.A. McCaulay	10/3/45	Same as A-2356
A-2358	Exchange Method of Preparing Boron Trifluoride Samples for Mass Spectrometer Analysis of Dimethyl Ether-Boron Trifluoride	C.M. Judson	10/23/45	Same as A-2356
A-2359	Analysis of Dimethyl Ether-Boron Trifluoride	C.M. Judson	10/26/45	Same as A-2356
A-2360	Design and Operation of Product Still	H.B. Higgins T. G. Stack J.E. Wolf	10/30/45	Same as A-2356
A-2361	McMahon Packing	W.L. Forsythe, Jr. T.G. Stack J.E. Wolf	11/17/45	Same as A-2356
A-2362	Experience in the Use of Stedman Packing	H.B. Higgins T.G. Stack J.E. Wolf A.L. Conn	1/28/46	1. Chicago Area Office 2. " " " 3. " " " 4. " " " 5. " " " 6. L.B. Brag[illegible] 7. J.K. Roberts

TABLE II (Cont'd.)

Reports Issued During Term of Contract

Report No.	Title	Author(s)	Date Issued	Distribution
A-2363	A Study of the Reactions of Methyl Ether-Boron Fluoride Complex with Water Under Fractionation Conditions	E.N. Hiebert W.L. Rittschof A.L. Conn	10/31/46	Same as A-2356
A-2364	Concerning Some Isotopic Exchange Reactions of Boron Compounds	A.N. Webb W.L. Rittschof	10/30/46	Same as A-2356
A-2365	Reactions of Boric Acid and Boric Oxide with Methyl Ether-Boron Fluoride Complex	A.N. Webb W.L. Rittschof	10/31/46	Same as A-2356
A-2366	Design of New Fractionation Unit	J.E. Wolf A.L. Conn	12/31/46	Same as A-2356
A-2367	Determination of the Boron Isotope of Mass Ten By the Neutron Absorption Counter Method	C.M. Judson	6/14/46	1. Chicago Area Office 2. " " " 3. " " " 4. " " " 5. " " " 6. J.K. Roberts 7. A.L. Conn 8. " " " 9. " " "
A-2368	Performance of Fractionation Unit	J.E. Wolf H.B. Higgins K.W. MacFie J.J. Lukes	6/24/46	Same as A-2367
A-2369	Experience in the Use of Diaphragm Pumps	T.G. Stack J.J. Lukes	7/5/46	Same as A-2367

TABLE II (Cont'd.)

Reports Issued During Term of Contract

Report No.	Title	Author(s)	Date Issued	Distribution
A-2370	Construction, Operation, and Maintenance of a Polymer Fractionation Unit	J.R. Elohns K.G. Carpenter C.J. McManus R. Tuck C. Risher	7/23/46	1. Chicago Area Office 2. " " " 3. " " " 4. " " " 5. " " " 6. J.K. Roberts 7. L.B. Bragg 8. A.L. Conn 9. " " " 10. " " " 11. P.C. Moen
	M Reports			
M-1	Exchange Experiments	W.L. Hittschof	9/28/44	1. Chicago Area Office 2. " " " 3. " " " 4. " " " 5. M. Kilpatrick 6. S.O.Co. 7. Chicago Area Office 8. " " " 9. " " " 10. T.S. Chapman 11. Extra)Kept at No. 4 12. ")Process Laboratory

SECRET

SECRET

TABLE II (Cont'd.)

Reports Issued During Term of Contract

Report No.	Title	Author(s)	Date Issued	Distribution
M-2	Application of Karl Fischer Analysis to Overhead Polymer Samples from the Pilot Plant	W.L. Rittschof	11/18/44	1. Chicago Area Office 2. " " " 3. " " " 4. " " " 5. M. Kilpatrick 6. A. L. Conn 7. Chicago Area Office 8. " " " 9. " " " 10. Extra)Kept at No. 4 11. ")Process 12. ")Laboratory
M-3	McMahon Packing	J. Wolf	12/7/44	1. Chicago Area Office 2. " " " 3. " " " 4. " " " 5. " " " 6. " " " 7. M. Kilpatrick 8. J.K. Roberts 9. A.L. Conn 10. Extra 11. Chicago Area Office 12. " " "
M-4	Determination of the Freezing Points of Mixtures of BF_3-Methyl Borate Complex and BF_3-Methyl Ether Complex	D.A. McCaulay W.L. Rittschof	1/17/45	1. Chicago Area Office 2. " " " 3. " " " 4. " " " 5. " " " 6. " " " 7. J.K. Roberts 8. M. Kilpatrick 9. A.L. Conn 10. Extra)At No. 4 Process Lab.

TABLE II (Cont'd.)

Reports Issued During Term of Contract

Report No.	Title	Author(s)	Date Issued	Distribution
M-5	Formation of Methyl Borate From the Methyl Borate-Boron Fluoride Complex	D.A. McCaulay W.L. Rittschof	9/4/45	1. Chicago Area Office 2. " " " 3. " " " 4. " " " 5. " " " 6. " " " 7. J.K. Roberts 8. H.C. Urey 9. A.L. Conn 10. " " " 11. " " " 12. " " "
M-6	Solubility of Methyl Ether and Boron Fluoride in Methyl Ether-Boron Fluoride Complex	D.A. McCaulay W.L. Rittschof	9/17/45	1. Chicago Area Office 2. " " " 3. " " " 4. " " " 5. " " " 6. " " " 7. " " " 8. J.K. Roberts 9. A.L. Conn 10. " " " 11. " " " 12. " " "
M-7	Detecting and Eliminating Leaks in a Vacuum Unit	T.G. Stack J. Wolf	1/25/46	1. Chicago Area Office 2. " " " 3. " " " 4. " " " 5. " " " 6. J.K. Roberts 7. A.L. Conn 8. " " " 9. " " "

SECRET

TABLE II (Cont'd.)

Reports Issued During Term of Contract

Report No.	Title	Author(s)	Date Issued	Distribution
M-8	Fire Extinguisher Tests on Burning Polymer	K.N. MacPie J.J. Lukes	4/4/46	1. Chicago Area Office 2. " " " 3. " " " 4. " " " 5. " " " 6. J.K. Roberts 7. A.L. Conn 8. " " " 9. " " " 10. J.T. Faust
M-9	Null Method of Analysis	A.M. Webb T.G. Stack J.J. Lukes	4/18/46	Same as M-7
M-10	Proposed Operation of a Polymer Fractionation Unit at Atmospheric Pressure	T.G. Stack J.J. Lukes	7/18/46	Same as M-7

Other Reports

Initial Report and Progress Reports P-1 through P-29	J.K. Roberts	Dec.,1943 through June,1946, inclusive	6 copies Area Engineer 1 copy SOCo Chicago Files 1 " " Whiting " 2 copies SOCo Chicago Duplicates

SECRET

SECRET

TABLE III

Government Personnel Who Visited Project Site*

Andrlik, G. M.	Ferry, J. L.	Karl, C. L.	Oakley, S. H.
Averill, R. P.	Fleury, E. R.	Knepp, D. A.	Oliff, Herschel
Baranowsky, Paul	Flournoy, Copley	Koranda, Hugo	O'Rourke, P. B.
Barbour, J. R.	Flynn, A. J.	Kostka, A. L.	Orrico, R. J.
Bastian, John	Fry, B. M.	Kowal, J. T.	Paulson, H. W.
Bawn, C. V.	Frye, A. H., Jr.	Lancer, O. G.	Peterson, A. V.
Belcher, P. F.	Gilsinan, J. F.	Leslie, W. S., Jr.	Phelps, J. W.
Berg, G. W.	Greig, D. F.	Maass, R. J.	Phillips, H. B.
Bolton, F. H.	Guilfoyle, T. D.	Mahoney, J. H.	Quinlan, R. L.
Campbell, A. M.	Gulberg, G.	Maniere, A. T.	Ream, H. D.
Campbell, C. E.	Handler, Evelyn	Mayer, G. A.	Robertson, W. M., Jr.
Chapman, T. S.	Hans, J. W.	McAdam, R. G.	Russell, G. W.
Cherimpes, George	Hargrave, E. B.	McBain, Bruce	Scott, R. T.
Chezak, A.	Harley, D. J.	McClure, C. R.	Seminara, L. A.
Clarke, C. F., Jr.	Hartley, J. T.	McKibben, J. L.	Smith, A. E.
Cole, S. A.	Henderson, J. B.	McKinley, J. H.	Smith, R. J.
Collins, D. L.	Hennessey, Matthew	Meyerson, Seymour	Sorkin, Gwendolyn
Coy, T. D.	Houska, V. J.	Miller, J. C.	Stowers, D. M.
Delsing, C. G.	Hunsicker, E. A.	Moen, P. C.	Thomas, Robert
Diemer, H. B.	Johannesson, R. E.	Moss, R. A.	Tidd, C. A.
Doyle, N. T.	Johnson, A. V.	Neift, F. A.	Welch, M. F.
Dunham, B. W.	Jones, T. O.	Nemeth, J. J.	Wendt, C. F.
Ellen, M. W.	Jones, T. R.	Nichols, K. D.	Wheaton, G. W.
Farr, K. M.	Kamm, C. H.	Nolan, J. E.	Young, G. A.
Faust, J. T.			

*As determined from monthly visitor reports.

TABLE IV

Personnel Familiar with Objective of Work Under Contract W-7416-eng-41 (Government Personnel Excluded)

- Anderson, K. A.
- Arveson, M. H.
- Baldaste, R. F.
- Berg, W. G.
- Binns, J. E.
- Booth, E. T.
- Bragg, L. B.
- Brown, B. K.
- Brown, W. K.
- Browning, M. K.
- Carpenter, K. G.
- Carpenter, M. T.
- Conn, A. L.
- Daly, J. J.
- Daugherty, C. H.
- DeVries, J. E.
- Diggs, W. C.
- d'Ouville, E. L.
- Eichne, J. R.
- Erickson, C. L.
- Forsythe, W. L.
- Grubb, H. M.
- Gunness, R. C.
- Hathaway, R. M.
- Hoare, P. H.
- Hiebert, E. N.
- Higgins, H. B.
- Hostedler, Dave
- Howard, J. C.
- Howland, W. B.
- Hughes, M. P.
- Hurley, O. L.
- Inghram, M. G.
- Judson, C. M.
- Kay, W. B.
- Kenney, J. E.
- Keranen, G. M.
- Kerr, G. M.
- Kilpatrick, Martin
- Kirn, E. R.
- Kirshenbaum, Isidor
- Koehle, J. E.
- Krauel, P. L.
- Laederach, A. S.
- Lukes, J. J.
- Lutz, W. F.
- MacFie, K. W.
- Mannion, J. V.
- Mason, J. F., Jr.
- Mather, J.
- McCaulay, D. A.
- McGill, W. J.
- McManus, C. J., Jr.
- Miley, Tom
- Monro, D. A.
- Montgomery, S. A.
- Mudgett, F. I.
- Myers, H. G.
- Paulus, M. G.
- Peake, A. W.
- Peterson, H. A.
- Prescott, O. R.
- Ramires, J. R.
- Reeve, B. B.
- Risher, C. B.
- Rittschof, W. L.
- Roberts, J. K.
- Rock, W. A.
- Rusk, A. H.
- Sabi, Nestor
- Schaffer, L. E.
- Schmauch, L. J.
- Schmidt, H. R.
- Schutz, P. W.
- Schwab, W. G.
- Singler, E. R.
- Stack, T. G.
- Stein, F. S.
- Sufak, M. L.
- Sulsbach, A. J.
- Thiele, E. W.
- Townsley, C. E.
- Tuck, R. A.
- Urey, H. C.
- Veerman, M. E.
- Watts, G. W.
- Webb, A. N.
- Webb, R. L.
- Westbay, E. A.
- Wetherald, J. S.
- Wilson, R. E.
- Wolf, J. E.
- Wynkoop, V. C.
- Yant, J. W.
- Zmachinsky, C. W.

TABLE V

Cost Statement

Standard Oil Company Expense

Salaries, wages, and overhead (including taxes)	$ 426,352.28
Materials (including shipping expense)	79,656.54
Shop and service charges	260,103.51
Traveling expense	13,401.95
Telephone and telegraph expense	2,946.61
Utilities	5,944.71
Foster Wheeler subcontract*	1,520,907.78
Miscellaneous	233.50
Grand Total	$2,309,546.88

*Foster Wheeler Expense

Field payroll (including taxes and insurance)	$ 687,425.81
Foster Wheeler shop fabricating charges	365,499.53
Purchased materials (including transportation)	168,402.81
Operators' salaries	46,256.00
Cancellation charges	12,282.00
Contractor's fee	240,940.58
Telephone	101.05
Total Foster Wheeler Subcontract Expense	$1,520,907.78

Above figures subject to final audit.

SECRET

SECRET

SECRET

A number of contributions were made to the fundamental knowledge of the chemistry of the process during a program of research and development undertaken to eliminate difficulties which occurred both prior to and during the period of successful operation. These contributions were concerned with the mechanism of the reaction between polymer and water, the equilibria existing between polymer and its decomposition products, and the isotopic exchange of element between polymer and a number of other compounds.

Chemical engineering information of importance to industry was obtained on the performance of Stedman and McMahon types of column packing. In addition, information of considerable interest was obtained on methods of detecting leaks in process equipment and on the use of diaphragm pumps for handling liquids under difficult operating conditions.

Work of particular importance to the project was that done on the improvement and development of analytical methods for determining the quality of polymer. This included the use of the counter method for determining the concentration of heavy element in polymer and the development of the exchange method for preparing samples for the "111" analytical machine. Complete details of all the research and development contributions made during the work are included in the reports which were issued in connection with the work. These reports are listed in Table II. Included is much information on the construction and performance of the plant equipment, in addition to details of the early design and development work.

The work of the project was carried out by a large number of civilian and government technical and non-technical personnel. All government personnel who visited the project site are listed in Table III. In Table IV are listed the names of those persons, not including government personnel, who were familiar with the objective of the work carried out under this contract. This list includes those who definitely knew the objective of the project as well as those who may have been able to correctly interpret the objective from the information made available to them.

All expenditures involved in the work are presented in detail in Table V. As shown, the total amounted to $2,309,846.38. This amount was the sum spent for all work done up to and including July 31, 1946.

J K Roberts
J. K. ROBERTS

[redacted] [redacted]

and for services in connection with the close-out of operations, all as required or approved by the Contracting Officer. In conformity with current negotiations, it is proposed that you maintain process equipment in No. 4 Process Laboratory in stand-by condition and store Government property on a mutually agreeable fixed-price basis to be covered by a supplemental agreement to this contract.

Receipt is acknowledged of your break-down of proposed fixed-price for stand-by and storage. It is requested that you furnish for approval of the Contracting Officer an estimate of all costs, except for storage and stand-by, that may be incurred in connection with the above directions.

Acknowledgment of receipt of this letter is requested.

Very truly yours,

/s/ A. Tammaro
A. TAMMARO,
Lt. Col., Corps of Engineers,
Contracting Officer.

[redacted]

2 4 1 B

CHS:md:brs:1f-9-26-47

IMMV-320-b
-7418-eng-41

[illegible]

8 March 1946

Standard Oil Company of Indiana,
910 S. Michigan Avenue,
Chicago, Illinois.

Attention: Mr. J. K. Roberts.

Gentlemen:

Reference is made to your Contract No. -7418-eng-41, dated 2 December 1943, for the design, construction, installation and operation of facilities for the production of X Product.

Confirming telegram dated 1 February 1946, you are directed to cease operation of the facilities effective 8 a.m., 4 February 1946, except for final processing of such material then in process which in your judgment is economical. You are further directed as follows:

1. Promptly wash out the plant and place it in stand-by condition.

2. Dispose of product as directed by Contracting Officer.

3. Furnish a report summarizing your activities, findings, and conclusions and such other reports as directed by the Contracting Officer.

4. In conformity with Argicle XII of the contract dispose of all Government property determined by the Contracting Officer to be surplus.

5. Place all other Government property in your No. 3 Boiler House and securely lock such premises as directed by Contracting Officer.

A-8

Reimbursement will not be made to your for expenses incurred after 8 a.m., 4 February 1946, unless subsequently directed to resume production, except for the work above-mentioned and except for such expenses as may be necessary for the protection of Government property