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June 3, 1970

Memorandum to File

MANHATTAN DISTRICT HISTORY, BOOK VII, FEED MATERIALS, SPECIAL  
PROCUREMENT, AND GEOGRAPHICAL EXPLORATION

In April, 1970, I requested the Division of Classification to  
review Book VII, Volume I of the Manhattan District History to  
see whether it would be practical to prepare an unclassified  
version.

The deletions necessary for declassification were listed in a  
memorandum, Charles F. Knesel to R. G. Hewlett, May 27, 1970,  
Copy 1 of 4A, 4 pages, SECRET RESTRICTED DATA.

Copies of this memorandum are on file in the Division of Classi-  
fication and the Office of the Secretary.

Richard G. Hewlett  
Chief Historian

Department of Energy Declassification Review	
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Copy No. 2 of 4 Series A

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FEE (SIGNED) J. G. ... XVII-582A  
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**MANHATTAN DISTRICT HISTORY**

**BOOK VII, FINE MATERIALS, SPECIAL PROCUREMENT,  
AND GEOGRAPHICAL INFORMATION**

**VOLUME 1 - FINE MATERIALS AND SPECIAL PROCUREMENT**

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FOREWORD

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This document contains information which is classified as Restricted Data in the Atomic Energy Act of 1954 or the Atomic Energy Act of 1946 and the disclosure of its contents to an unauthorized person is prohibited.

Volume 1 of Book VII of the Manhattan District History deals with the feed materials and special procurement program from its inception through 31 December 1946. To make the history complete, references are made to work sponsored by the Office of Scientific Research and Development (OSRD) prior to June 1942. This volume is divided into three parts and 11 appendices: Part A outlines the general features concerning the program as a whole; Part B relates to the procurement of raw materials, which constitutes the first of two operational phases of the program; Part C describes the refining and treatment of the raw materials, which comprise the second operational phase of the program; the first 6 appendices contain: a Glossary, Organization Charts, Flow Diagrams, Graphs, References, and Contract Data, respectively; and the other appendices cover the procurement of special chemicals or other materials for various parts of the Manhattan Project.

The feed materials program is a particularly intriguing and interesting subject, dealing as it does chiefly with the material uranium, which had little commercial usage previously, and whose refining and industrial treatment had not been performed on any significant commercial scale. Its scarcity and source control by national government and other major international organizations, the extreme secrecy with which all operations had to be continued, the magnitude of the quantities involved, and the speed with which manufacturing plants had to be constructed and the products obtained, as well as the personalities and organizations involved, made the program one of the highest importance and interest.

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During the period that the District Office was located in New York City, August 1942 to August 1943, the program was handled by the Materials Section as its primary responsibility. With transfer of the District Office to Oak Ridge, Tennessee, in August 1943, the Madison Square Area was established primarily for the continuation of the supervision of the feed materials program, although this office, as the Materials Section had done previously, also handled the procurement of certain special operating materials for the processing plants, which activities are discussed in the histories of those plants and briefly covered in Appendices to this volume.

This book has been prepared by the Madison Square Area Office on the basis of the personal knowledge of the staff at that office concerned in the program, and the records of that office. References are made in the text to pertinent documents of record, which are in the Madison Square Area files, and other sources of information, and a list of such documents is appended. Copies of War Department contracts referred to in the text are on file in the Contract Section of the Manhattan District in Oak Ridge.

A summary is included ahead of the text to enable the reader to get a condensed but comprehensive picture of the subject matter without reading the entire book. Paragraph numbers and titles in the summary correspond to section numbers and titles in the text. The asterisk (\*) has been used to indicate those words which have been defined in the Glossary.

The story of the studies of geological literature, field explorations, and inspections and surveys to locate new sources of



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uranium ore or to evaluate and investigate existing known deposits  
will be found in Volume 2 of this book, "Geographical Exploration".

June 12, 1947

MANHATTAN DISTRICT HISTORY

BOOK VII

VOLUME 1 - FEED MATERIALS AND SPECIAL PROCUREMENT

TABLE OF CONTENTS

Par. No. Page No.

FOREWORD

SUMMARY

Part A - General Features

SECTION 1 - INTRODUCTION

1-1	Objective	1.1
1-2	Scope	1.1
1-3	Authorizations	1.2
	a. General	1.2
	b. Specific	1.2
1-4	Occurrence, Uses and Properties of Uranium	1.3
	a. Occurrence	1.3
	b. Commercial Uses	1.4
	c. Properties	1.4
	(1) Physical	1.4
	(2) Chemical	1.5
	(3) Radioactivity	1.5
	(4) Relationship to Radium	1.6
1-5	Development of Program	1.7
1-6	Procurement	1.8
	a. Uranium-containing Materials	1.8
	b. Miscellaneous Materials	1.9
1-7	Raw Material Prices	1.9
1-8	Refining and Treatment	1.10
1-9	Research and Development	1.12
1-10	Recovery Operations and By-Products	1.13
1-11	Results	1.14
1-12	Future Considerations	1.14
1-13	Costs	1.15
1-14	Organization and Personnel	1.15

Part B - Procurement

SECTION 2 - AFRICAN SOURCES

2-1	Operations	2.1
2-2	Contract Negotiations	2.3

~~TOP SECRET~~

<u>Par. No.</u>		<u>Page No.</u>
2-3	Transportation of Ore from Africa	2.5
2-4	Storage	2.5
2-5	Weighing, Sampling and Assaying	2.7
SECTION 3 - CANADIAN SOURCES		
3-1	Operations	3.1
3-2	Contract Negotiations	3.2
SECTION 4 - AMERICAN SOURCES		
4-1	Operations	4.1
4-2	Contract Negotiations	4.3
SECTION 5 - MARKET AND MISCELLANEOUS PROCUREMENT		
5-1	Operations	5.1
5-2	Contract Negotiations	5.1
5-3	Miscellaneous	5.2
SECTION 6 - PROCUREMENT OF OTHER RADIOACTIVE MATERIALS		
6-1	Operations	6.1
	a. Radium	6.1
	b. Radioactive Lead	6.1
6-2	Contract Negotiations	6.1
	a. Radium	6.1
	b. Radioactive Lead	6.3
<u>Part C - Refining and Treatment</u>		
SECTION 7 - REFINING OF RAW ORES TO BLACK OXIDE AND SODA SALT		
7-1	Operations	7.1
7-2	Refining by Eldorado Mining and Refining	7.2
	a. Operations	7.2
	b. Contractual Arrangements	7.3
7-3	Refining by Vitro Manufacturing Company	7.4
	a. Operations	7.4
	b. Contractual Arrangements	7.5
7-4	Refining by Linde Air Products Company	7.5
7-5	Concentrating of American Ores by U. S. Vanadium Corporation	7.8
7-6	Concentrating of American Ores by Vanadium Corporation of America	7.13

~~TOP SECRET~~

SECTION 8 - PRODUCTION OF BROWN OXIDE  
AND ORANGE OXIDE

8-1	Operations	8.1
8-2	Mallinckrodt Brown Oxide Plant	8.2
8-3	du Pont Brown Oxide Plant	8.4
8-4	Linde Brown Oxide Plant	8.5
8-5	du Pont Scrap Recovery Plant	8.7

SECTION 9 - PRODUCTION OF GREEN SALT  
AND HEXAFLUORIDE

9-1	Operations	9.1
9-2	Mallinckrodt Green Salt Plant	9.1
9-3	du Pont Green Salt Plant	9.2
9-4	Harshaw Green Salt Plant	9.4
9-5	Linde Green Salt Plant	9.6
9-6	Harshaw Hexafluoride Plant	9.6

SECTION 10 - PRODUCTION OF METAL

10-1	Operations	10.1
10-2	Mallinckrodt Metal Plant	10.2
10-3	Electro Metallurgical Company Metal Plant	10.3
10-4	du Pont Metal Plant	10.5
10-5	Iowa State College Metal Plant	10.5
10-6	Metal Hydrides Metal Plant	10.7
10-7	Westinghouse Metal Plant	10.9
10-8	Brush Laboratories Metal Plant	10.9

SECTION 11 - THORIUM

11-1	Thorium Procurement	11.1
------	---------------------	------

SECTION 12 - QUALITY CONTROL

Part I - Uranium

12-1	Operations	12.1
12-2	University of Chicago	12.2
12-3	Princeton University	12.3
12-4	Massachusetts Institute of Technology	12.3
12-5	National Bureau of Standards	12.4

Part II - Thorium

12-6	National Bureau of Standards	12.5
12-7	Iowa State College	12.5



SECTION 13 - ACCOUNTABILITY

13-1

Program

13.1

APPENDIX "A" - GLOSSARY

APPENDIX "B" - ORGANIZATION CHARTS

APPENDIX "C" - FLOW DIAGRAMS

APPENDIX "D" - GRAPHS

APPENDIX "E" - REFERENCES

APPENDIX "F" - CONTRACT DATA

APPENDIX "G" - SPECIAL CHEMICALS  
for K-25

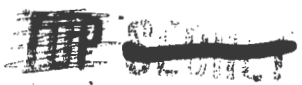
APPENDIX "H" - MISCELLANEOUS MATERIALS  
for P-9

APPENDIX "I" - MISCELLANEOUS MATERIALS  
for X-10

APPENDIX "J" - PROCUREMENT FOR SITE Y

APPENDIX "K" - BERYLLIUM PROCUREMENT

INDEX



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SUMMARY

Part A - General Features

1. Introduction - The feed materials program had as its objective the procurement of the basic raw materials containing uranium and their conversion to feed materials for the processing plants. The program involved the locating and processing of raw materials and their refining and treatment to obtain feed materials, in the particular forms and of the highest purity, for utilization in the processing plants within the stringent time limitations which had been established. At the outset of the program, the objective was to procure approximately 1,700 tons of black oxide ( $U_3O_8$ ), or its equivalent in ore concentrates by the middle of 1944, for conversion to feed materials, while the present objective is to procure sufficient  $U_3O_8$  to permit operation of production and research facilities as required.

The occurrence of the element uranium was recognized in 1789 by W. H. Klaproth in the mineral which he called Uranite. Uranium is found primarily in pitchblende and carnotite ores, and to a lesser extent in other ores such as torbernite. The principal ore deposits are located in the Belgian Congo of Africa, the Great Bear Lake region of Canada, and the Colorado plateau region of western United States, and lesser deposits are known to exist in many other countries throughout the world. Although there was no particular demand for pure uranium metal, as such, prior to the start of the project, there were some commercial uses for compounds of uranium, i.e., for the oxide in the ceramic industry and for the nitrate in the photographic industry. The properties of uranium are extremely interesting; physically, uranium atoms are of three kinds (each referred to as an isotope) and are among the heaviest

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known; while chemically, uranium is considered to be a relatively active element. It is also radioactive; that is, its atoms are unstable, and they undergo spontaneous and uncontrollable transmutation into simpler, or lighter weight, elements. Uranium and radium, in their natural occurrence in ore, bear a definite relationship to each other, radium occurring in uranium ores in the ratio of 1 part to every 3 $\frac{1}{2}$  million parts of black oxide. The primary demands for radium are for medical purposes, luminous paint manufacture, and industrial radiography.

In the original development of the program, it was envisioned that one contractor would perform all of the necessary work under one prime contract. However, as the program was enlarged, it readily became apparent that it would be both desirable and essential to engage the services of as many leading industrial concerns as possible in order to distribute the many complex tasks among the best personnel available in industry. In general, operations consisted essentially of locating adequate sources of raw materials, procuring the raw materials, refining them, and finally converting them in a series of treatment operations to obtain feed for the processing plants.

Initial procurement of black oxide was begun in 1941 by the OSRD. In July 1942, the first order for black oxide was placed by Stone & Webster for the Manhattan District, and in the latter part of 1942, all procurement was assumed directly by the Manhattan District. As part of the procurement program, certain other items, such as radium, radium-neutron sources, and radioactive lead, were procured for other project installations. In addition, Madison Square Area acted as a special procurement agency for Los Alamos, and the K-25 program. In the case of the former, many items of a highly specialized nature were procured, and

they will not be detailed. However, a cost summary of the work done is given in Appendix J. The procurement for K-25 consisted in contracting for the design, construction, and operation of plants for special and unique fluorocarbons, and the details are outlined in Appendix G. Raw material prices varied according to the grade and type of ore, and the resultant difference in subsequent cost of refining to recover the uranium content. The sequence of operations and the type of processes to be used in refining and treatment operations, and the requirements for new and expanded plant facilities for large-scale manufacture, were established in the fall of 1942, at which time the construction program was initiated and carried forward at an accelerated rate, with the result that, by the middle of 1943, practically all of the plants which were required were in full operation.

Throughout all stages of the program, it was necessary that varied and extensive research and development work be undertaken since enormous quantities of uranium, in special form and of the highest purity (since even small concentrations of impurities reduce the efficiency of the X-10 process), had to be produced on a commercial scale never before thought possible. Recovery operations were instituted to salvage scrap and by-product materials, and the uranium content which was recovered was reintroduced into the processing circuit, where it was eventually converted to usable feed materials. The results of the program have been the attainment of both the procurement and production objectives. The project has obtained title to approximately 10,000 tons of  $U_3O_8$  in ore concentrates of which 3,625 tons were obtained through the Washington office. There have been produced as feed materials for the processing plants approximately 6,600 tons of high purity uranium or its equivalent, includ-



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ing recast metal. At no time were deliveries behind the forecast schedule of requirements. Among the future considerations which will be brought about by the completion of the program are problems with foreign interests, problems regarding distribution of ore residues and disposition of the processing plants.

The cost of the program, to 1 January 1947, has been approximately \$90,268,490, of which \$27,592,360 was for procurement of raw materials, \$58,622,360 for refining and treatment operations, and \$3,357,690 for research, development, and quality control activities; \$88,400 for the procurement of radioactive lead; \$547,160 for radium, exclusive of that procured for Site Y; and \$60,520 for thorium salts. The direction and supervision of the program was handled essentially by officer personnel on the staff of the District Engineer. The organization originally established in 1942 to administer the program in its early stages was the Materials Section of the Manhattan District, at that time located in New York, N.Y. Since August 1943, at which time the District Office was moved to Oak Ridge, Tenn., the program has been administered by the Madison Square Area in New York, N.Y. Acknowledgement is made of the untiring efforts of those who contributed to the success of the program; and all military and civilian personnel, both in the War Department and the contractors' organizations, deserve the highest praise for the results which they have helped achieve.

#### Part B - Procurement

2. African Sources - The richest source of uranium-bearing raw materials for the project came from the Shinkolobwe mine, located in the southeastern corner of the Belgian Congo. The mine was discovered in 1915, but operations were not begun by its owner--Union Miniere du Haut Katanga,

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a Belgian Company - until after World War I. The mine operated intermittently from its opening until 1937, during which time mining and refining operations were conducted principally for the recovery of radium. The extreme richness of the African ore, assaying as high as 65% to 75%  $U_3O_8$ , has enabled Union Miniere du Haut Katanga to dominate completely the radium and uranium market. By 1937, sufficient ore had been mined and stock-piled to satisfy the future normal radium and uranium market for approximately 30 years. Therefore, all mining and refining operations were stopped in 1937.

In order to procure raw materials from this rich African source, the Manhattan District entered into negotiations with the African Metals Corporation of New York, N.Y. This company was the sole agent for the sale of Union Miniere's products in the United States. As of 1 January 1947, 3,839 tons of  $U_3O_8$  contained in ore had been procured by contract from African Metals at a cost of approximately \$9,113,800. Of the total 3,839 tons, 767 tons of  $U_3O_8$  were obtained from ore which was already in this country, and the remainder was obtained from the stockpile at the Shinkolobwe mine in Africa. In addition to this material obtained by contract, 3,144 tons of  $U_3O_8$  in ore had been obtained by the Washington office as of 1 January 1947, at a cost of approximately \$10,267,800. The transportation of ore from Africa presented a difficult task, since enemy submarines were active during most of the period of shipment. However, only two shipments, totaling about 200 tons of  $U_3O_8$  in ore, were lost at sea. One of these shipments was lost through enemy action, and the other was lost through a marine accident.

Since shipments from Africa arrived faster than refining opera-

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tions in this country could be carried out, it was necessary to obtain storage facilities in the United States for these ores. Ores arriving from Africa were stored originally at the Seneca Ordnance Depot, and later, at the Clinton Engineer Works. They are currently being shipped to a warehouse in Middlesex, New Jersey, and are stored there until they can be shipped to the refineries. The weighing, sampling, and assaying of African ores are also conducted at the Middlesex Warehouse.

3. Canadian Sources - The second richest source of raw materials for the project was the Eldorado mine, located on the southeast shore of Great Bear Lake in Canada. Mining and refining operations at the Eldorado mine, owned by Eldorado Mining and Refining<sup>Co.</sup> began in 1933 and continued until 1940, during which time, the ore was mined and refined for the recovery of radium, uranium, and silver. The mine was closed in 1940, since sufficient ore had been mined and stock-piled to satisfy the normal future commercial market for approximately 5 years. However, in 1941, the Office of Scientific Research and Development placed an order for ore which necessitated the reopening of the mine. The mine was reopened and started operations late in 1942, and mining and refining operations have continued since that date in order to fill the subsequent requirements of the Manhattan District.

There had been contracted for, to 1 January 1947, approximately 4,149 tons of ore, at a cost of approximately \$5,082,300, to be delivered as 1,137 tons of black oxide. To 1 January 1947, 921 tons of U<sub>3</sub>O<sub>8</sub> have been delivered. The early contracts for the purchase of black oxide were written directly with Eldorado, or with Boris Pregel, president of the Canadian Radium and Uranium Corporation of New York City, the sales agency for Eldorado's products in the United States. Since September 1943,

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Manhattan District contracts, for security reasons, have been written in the name of Carl French or Gilbert A. LaBine, who were, respectively, treasurer and president of Eldorado and acted as agents for that company. Eldorado had its own refinery at Port Hope, Ontario, where ore concentrates were refined to produce black oxide. Therefore, it was possible to purchase black oxide directly from Eldorado, and there was no need for the storage, the weighing, sampling, and assaying, and the preliminary refining steps, as in the case of the African ores.

4. American Sources - The third important source of uranium-bearing raw materials was the carnotite ores of the Colorado Plateau region in the United States. Operations in this area had begun about 1911, and, between 1911 and 1923, high-grade carnotite ores had been selectively mined, principally for the radium content and to a minor degree for uranium and vanadium. The high-grade carnotite ores contained a maximum of only 1.25%  $U_3O_8$ , and, operations on these ores declined rapidly after 1923 because of the unfavorable position which carnotites then had in the radium field with respect to radium derived from the newly discovered rich ores from the Belgian Congo. However, the great demand for vanadium brought about by World War II stimulated the development and exploitation of domestic sources, and carnotites have been mined since 1937 principally for their vanadium content.

As a result of the mining and refining of carnotite ores, principally for the recovery of radium and vanadium, during the past 30-35 years, there were accumulated tremendous stockpiles of tailings which contained varying low, but economically recoverable, percentages of uranium. The District entered into contract negotiations with the three major companies in the vanadium field to procure the stock-piled tailings from the former

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operations as well as the products of the current operations. To 1 January 1947, the U. S. Vanadium Corporation, the Vanadium Corporation of America, the Metals Reserve Corporation, and various other small producers had contracted for the delivery to the project of approximately 1,349 tons of  $U_3O_8$ , contained in 379,671 tons of ore tailings, at a cost of \$2,072,330.

5. Market Procurement - Prior to the start of World War II, approximately 150 tons of uranium compounds were being consumed annually in the manufacture of ceramic colors. The need for conservation and control of uranium supplies was recognized in December 1942, at which time the War Production Board was requested by the District to take appropriate action, and, in accordance with that request, Conservation Order M-285 was issued on 26 January 1943 and further amended in August 1943, prohibiting the sale or purchase of uranium compounds for any use other than a few vital military and industrial applications. As a result of these operations, the stocks of refined uranium compounds held by various manufacturers and distributors were made available to the project.

As of 1 January 1947, contract negotiations with the Vitro Manufacturing Company, African Metals, and several other companies resulted in the procurement of approximately 270 tons of  $U_3O_8$  contained in various refined uranium salts, at a cost of approximately \$1,056,130. The Washington office also obtained approximately 481 tons of  $U_3O_8$  in the form of miscellaneous compounds, mostly impure sodium salts. These materials were found by our armed forces in the European Theatre of Operations.

6. Procurement of Other Radioactive Materials - In addition to the procurement of uranium as a raw material for the project, it also became necessary to procure certain other substances, i.e., radium and radium-neutron sources and radioactive lead. Inasmuch as these materials occur

in nature in conjunction with uranium, their procurement was handled as a part of the general procurement program. The varied operations of the District made necessary the procurement, to 1 January 1947, of 73 grams of radium, at a cost of \$679,399, and 85 tons of lead oxide, as a source of radioactive lead, at a cost of \$88,400. Negotiations to date have procured all the radium and radioactive lead required by the Manhattan District.

Part C - Refining and Treatment

7. Refining of Raw Ores to Black Oxide and Soda Salt - The processing operations for refining raw ores and ore concentrates were performed by Eldorado Mining and Refining<sup>Co.</sup> at Port Hope, Ontario, Canada, and the Linde Air Products Co. at Tonawanda, New York, both of whom produced black oxide, and by the Vitro Manufacturing Co. at Cannonsburg, Pa., who produced soda salt ( $\text{Na}_2\text{U}_2\text{O}_7$ ). Both products required substantially the same subsequent treatment.

All Canadian ores were processed by Eldorado, whereas African ore concentrates were processed at all three of the refineries mentioned above, the high grade concentrates being processed by Eldorado and Vitro and the low grade concentrates by Linde. The majority of American ore concentrates were processed by Linde, and relatively small quantities by Vitro. The Linde plant was placed in stand-by in July 1946 because of insufficient supplies of suitable raw materials. In May of 1945, construction of a high-grade ore refinery was started at the Mallinckrodt Chemical Works in St. Louis. This plant was completed in May of 1946, and at this writing was just beginning to reach quantity operations.

The American ore concentrates which were refined by Linde were given a preliminary processing by the U.S. Vanadium Corporation at Grand

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Junction, Colorado. The ores and tailings obtained from U. S. Vanadium Corporation at Uravan and Durango, Colorado, and other smaller mines in that area were concentrated in mills at Uravan and Durango. Concentrates and tailings were also obtained from the Vanadium Corporation of America, whose mills were located at Monticello, Utah, and Naturita, Colorado.

8. Production of Brown Oxide and Orange Oxide - The production of brown oxide (uranium dioxide,  $UO_2$ ) is considered the first step in each of the three parallel chains of treatment operations involved in the purification and treatment of materials to produce uranium metal. Operations in this step were carried out primarily to produce the brown oxide, but, in addition, there were withdrawn at an intermediate point the quantities of orange oxide (uranium trioxide,  $UO_3$ ) required for the Y-12 process. The withdrawal for the Y-12 process was a preliminary measure since later arrangements were made to utilize the end products of the K-25 and S-50 processes as feed materials for Y-12; and withdrawal of orange oxide ceased in the spring of 1945. The Mallinckrodt Chemical Works, at St. Louis, Mo., E.I. du Pont de Nemours & Co., Inc., at Deepwater, New Jersey, and the Linde Air Products Company at Tonawanda, New York, were the three organizations engaged in the production of brown oxide. All of the orange oxide requirements were supplied by Mallinckrodt.

In the spring of 1944, the combination of a shortage of raw materials, with subsequent excess brown oxide production capacity, and a need for certain equipment of the type used in the brown oxide process, for use in the production of nickel compounds for the K-25 process, allowed the Linde brown oxide plant to be placed in standby condition and enabled part of its equipment to be used on the K-25 "nickel work", which was under the jurisdiction of the New York Area. Continued excess capacity in this process

step has made it unnecessary to start brown oxide production again in the Linde plant after completion of the "nickel work".

Total production of brown oxide to 1 January 1947, has been 6,626 tons, and of orange oxide 341 tons, at an operating cost, in this step, of \$7,928,030. The average processing cost of production from black oxide and soda salt has been \$0.55 per pound, and the present cost is \$0.45 per pound. The average processing cost of brown oxide produced from high grade ore (Mallinckrodt Refinery) has been \$0.82 per pound.

The du Pont scrap recovery plant was constructed and put into operation in September 1943 in order to recover the uranium content of scrap materials, residues, dust, etc., produced in the processing operations. The plant is able to process nearly all types of economically recoverable scrap materials and produces from such materials a wet uranium peroxide sludge which is charged directly into the brown oxide process just as if it were black oxide. 5,486 tons of scrap material have been processed to 1 January 1947 to produce about 982 tons of equivalent black oxide, at an operating cost of \$2,294,150, or an average processing cost of \$1.17 per pound of  $U_3O_8$ .

9. Production of Green Salt and Hexafluoride - The second step in the parallel chain of treatment plants is the production of green salt (uranium tetrafluoride,  $UF_4$ ). Operations in this step were necessary in order to convert the brown oxide produced in the previous purification step into a form in which it could be reacted with magnesium in the succeeding step to produce uranium metal. The process involved essentially the treatment of the brown oxide at a high temperature with hydrofluoric acid, which converts the brown oxide to the green salt. Mallinckrodt, du Pont, and Linde each had a plant for the production of green salt. In addition,



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Harshaw Chemical Company at Cleveland, Ohio, had a green salt plant, and the material produced by them was used until the summer of 1944 in metal production operations and, after that time, was converted by Harshaw, in an additional step, into the hexafluoride (gaseous uranium hexafluoride,  $UF_6$ ), which was the feed material for the K-25 and S-50 processes and subsequent to Sept., 1945 for the K-25 process alone. Operations at the du Pont green salt plant were discontinued in the summer of 1944, and the plant was put in stand-by condition after a review of the performance of all plants from the quality, yield, and cost standpoint. For reason of economy, the Linde green operations were terminated in July 1946.

The total production of green salt for use in the manufacture of metal, to 1 January 1947, has been 7,342 tons, at an operating cost, in this step, of \$6,605,320, and an average processing cost of \$0.45 per pound. Hexafluoride production to date has reached 1,622 tons, at an operating cost of \$2,221,470 for this operation, and an average processing cost of approximately \$0.68 per pound.

10. Production of Metal. - In the third step in the chain of treatment operations, the green salt is converted to uranium metal by reaction with magnesium at high temperatures. The metal agglomerate obtained in this reaction is melted in an induction-heated vacuum furnace and pured into graphite molds. In addition to this main process, which was carried out by Mallinckrodt, du Pont, Electro Metallurgical Co., in Niagara Falls, New York, and Iowa State College in Ames, Iowa, two other processes were used in the early stages of operation but were later discarded because of lower quality metal obtained and higher cost. One of these processes, used by Metal Hydrides, Inc., in Beverly, Massachusetts, involved the reduction of brown oxide with calcium hydride and the subsequent recasting of the metal powder obtained. The second process, used by Westinghouse Electric & Mfg. Co.,

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in Bloomfield, New Jersey, employed the electrolysis of green salt in a molten calcium chloride bath. Another process, never used on a production basis, was also developed by the Brush Laboratory Co., in Cleveland, Ohio.

To 1 January 1947, 3,900 tons of virgin metal have been produced at an operating cost, for this step, of \$10,120,020; and 1,410 tons of re-cast metal have been produced at an operating cost of \$1,219,600 (exclusive of the cost of Westinghouse production in the early stage of the program).

11. Thorium. - Early in 1946, the Madison Square Area entered into the procurement of thorium salt for the research project at Iowa State College. To January 1/1947, all of this material procured had been obtained from the Lindsay Light and Chemical Co. under Contract W-17-028 eng-33 and W-12-028 eng-35 at a cost of \$60,520.

12. Quality Control. - From the beginning of production of uranium metal by the Manhattan District, it was recognized that the consideration of quality was of equal importance to that of quantity in setting up a requirement program and that the attainment of the high quality required would necessitate a much more comprehensive program of testing, both chemical and physical, than is ordinarily carried out in controlling the quality of material manufactured to usual commercial standards. Accordingly, the program of operations provided for: routine process control testing in the laboratories at each of the individual production facilities; a system of central control laboratories for the analysis of composite samples from each producer and for research on methods of analysis; and certain physical tests on brown oxide and on metal shipped to the X-10 process.

The magnitude of the analytical program, involving the determination of some sixty elements to a low limit of detection of a few parts or fractions of a part per million, was such as to prevent the possibility

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of carrying it out at each of the producers' laboratories. Therefore, it was decided to limit the testing at each producer's laboratory to routine control analyses and to have more comprehensive analyses carried on at four central control laboratories, at the University of Chicago Metallurgical Laboratory in Chicago, Illinois, Princeton University in Princeton, New Jersey, Massachusetts Institute of Technology in Cambridge, Massachusetts, and the National Bureau of Standards in Washington, D.C. The functions of the central control laboratories may be summarized as follows: to perform analyses as indicated in the quality control program; to improve existing methods and develop new methods of analysis; to act as a "fire brigade", that is, to supply special services needed in times of emergency to supplement the manufacturers' personnel and facilities; to analyze special materials not covered in the quality control program; and to give advice and guidance on the entire analytical program. From experience gained in three years of operations, it was found that adequate control could be maintained by using the contractors' organizations and employing only the National Bureau of Standards in the "fire brigade" capacity.

The cost of the quality control program has been approximately \$826,930 to 1 January 1947.

13. Accountability. - The enormous quantities of valuable Government owned uranium-containing materials which were being processed and transferred from one plant to another necessitated that an accountability program be established to enable close control to be exercised on all materials both for accounting purposes and for purposes of preserving the security of the program. To this end, a detailed system of accounting records, procedures, and controls was established, and continued surveys and studies of the material handling and processing operations were undertaken, in close cooperation

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with the various contractors who processed material for the Government.

Procurement for K-25 - To 1 January 1947, procurement for K-25 totaled \$32,497,560. Of this amount, \$11,725,750 covered construction.

(App. G)

Procurement for P-9 (Heavy Water Process) - Procurement of catalysts for this process by the Madison Square Area involved the expenditure of \$289,600 (App. H).

Procurement of Miscellaneous Materials for X-10 - Procurement of helium for the Hanford Engineer Works was handled by the Madison Square Area for some time. The helium was procured from the Bureau of Mines by Government Transfer of Funds. An "off-gas" recovery research program was carried out under Contract W-7412 eng-151 with E.I. du Pont de Nemours & Co. (App. I)

Procurement for Site Y - To 1 January 1947, \$6,340,051 had been expended for the procurement of materials for Site Y. Of this amount, \$132,236 covered the rental or purchase of radium and is included in the total cost of radium procurement given in paragraph 6 above (App. J).

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PART A

GENERAL FEATURES

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MANHATTAN DISTRICT HISTORY

BOOK VII - FEED MATERIALS

SECTION 1 - INTRODUCTION

1-1. Objective. - The objective of the feed materials program was the procurement of uranium-containing compounds and their conversion, in a series of refining and treatment operations, into feed materials for the processing plants at the Clinton Engineer Works and the Hanford Engineer Works.

1-2. Scope. - The program involved the delivery to 1 January 1947 of about 10,000 tons of uranium oxide ( $U_3O_8$ ) in ore concentrates (more than the total quantity produced in the world prior to the start of the project), with final delivery as varied feed materials to the several processing plants: i.e., uranium trioxide ( $UO_3$ ), "orange oxide" for the Y-12 plant, electromagnetic process; uranium hexafluoride ( $UF_6$ ) for the K-25 plant, gas diffusion process, and the S-50 plant, thermal diffusion process; and uranium metal for the K-10 plant, pile process. Shipments of orange oxide to the Y-12 plant were discontinued in the spring of 1945 when the K-25 plant commenced operations. Shipments of hexafluoride to the S-50 plant ceased in the fall of 1945 when this plant's operation was discontinued. At the present time, feed materials are delivered to the K-25 and K-10 plants only. The quantities of feed materials and other radioactive materials required by the processing plants necessitated the fullest exploitation of every feasible source of material, and the time limitations required that the program be carried out with the utmost speed to meet the anticipated needs of the processing plant. The initial objective

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in the early days of the project was to procure about 1,700 tons of black oxide in ore concentrates by the middle of 1944. Deliveries, in refined state, of the first quantities of material procured under the program were required for experimental purposes in December 1942, and the stockpiling of refined metal for the X-10 process was scheduled to begin in February 1943. The feed materials program alone, on the required scale, necessitated the establishment during wartime of what amounted to a relatively new industry on a scale of operations of roughly 25 million dollars per year.

1-3. Authorizations.

a. General. - The Manhattan District project was authorized by the President of the United States pursuant to authority conferred on him by Public Law No. 580, 77th Congress and Public Law No. 354 (First War Powers Act), 77th Congress, as more fully described in Book I, Volume 1.

b. Specific. - In a report to the President, dated 13 June 1942, signed by J. B. Conant, Chairman, National Defense Research Council and V. Bush, Director, Office of Scientific Research & Development (OSRD), and approved by the Chief of Staff, the Secretary of War, and the Vice President of the United States, it was recommended that research and development be continued and that design and construction of production plants for material for atomic fission bombs be started at the earliest possible date. This report was transmitted on 17 June 1942 by Dr. Bush to the President, who approved it.

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the tailings. Certain companies did some experimental work in attempting to reduce the cost of the uranium refining operations, but no work was done towards the refining of radium from these ores because the quantities contained were so infinitesimal. In addition to the above-mentioned sources of uranium, there are known to be other deposits in Czechoslovakia, Portugal, England, Madagascar, and other countries throughout the world, containing varying percentages of uranium.

b. Commercial Uses. - Prior to the start of the project, there had been no particular demand for pure uranium metal as such, but there was a small demand for the oxide in the ceramic industry and for the nitrate in the photographic industry. Consequently, the ores were refined primarily for the recovery of radium, the uranium compounds, such as uranium oxide, uranyl nitrate, and sodium diuranate, being recovered as by-products. The common commercial forms of uranium were black uranium oxide, sodium diuranate, sodium uranyl carbonate, and uranyl nitrate. The first three were used primarily in the ceramics industry for coloring glassware and pottery, imparting a beautiful yellow color to the articles to which they were added; whereas the nitrate was used for tinting photographic film.

c. Properties. - There is included below a brief general description of some of the more interesting properties of the element uranium, and further details concerning the properties of this element may be found in standard texts such as "Treatise on Inorganic Chemistry", by Mellor, "Applied Nuclear Physics" by Pollard and Davidson, and similar reference works.

(1) Physical. - As found in nature, uranium atoms are of three kinds, indistinguishable chemically, but of different mass.

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An atom of the heavy kind weighs 238 times, one of the intermediate kind 235 times, and one of the lighter kind 234 times, as much as an atom of hydrogen, which is the lightest known. These so-called isotopes of uranium are always found in the same proportion: 99.3% of the heavy variety, 0.7% of the intermediate variety, and a very minute percentage of the light variety; i.e., about seven out of every thousand uranium atoms are of the intermediate-weight 235 isotope. The discovery in 1939 that  $U_{235}$  atoms can be made to yield tremendous quantities of energy brought the element uranium from its position of relative obscurity to one of transcending importance.

(2) Chemical. - Uranium is fairly active chemically, which property is altogether separate and distinct from its radioactivity. Consequently, uranium is generally found in nature as an oxide, just as iron is most often found in nature as an oxide. As a matter of fact, pitchblende ore is essentially uranium oxide.

(3) Radioactivity. - Uranium is radioactive; that is, its atoms are unstable. The instability is similar to that of radium, and consists of spontaneous and uncontrollable transmutation at a definite rate into simpler (lighter) elements, with the simultaneous ejection of extremely small electrically charged particles (alpha rays). The lighter elements so produced are, in their turn, radioactive and undergo similar transmutations. Thus, a whole series of elements is continuously produced, each breaking down at its own characteristic rate, the quantity of each present depending upon the rate at which its parent generates it and the rate at which it itself breaks down. The uranium series ends with an isotope of lead which is not radioactive. The rate of breakdown, a distinctive property of radioactive elements, is measured as the "half-

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life", that is, the time required for one half of a given quantity of the element to disintegrate. For the isotopes of uranium, the average half-life is of the order of two thousand million years; for radium, sixteen hundred years. Radium is a member of the uranium series which begins with the 238 isotope. It can thus be seen why radium (and all the other members of the series) is always found associated with uranium in nature.

(4) Relationship to Radium. - As pointed out previously, prior to the start of the project the primary object of exploiting the uranium-bearing deposits had been the recovery of radium; and uranium was in the category of a by-product. In 1941, the price of radium in large quantities was about \$20 per milligram (1/1,000 of a gram) whereas black oxide ( $U_3O_8$ ) was sold on the market in large quantities for about \$2.05 per pound. As there are about 454 grams in a pound and since radium occurs in uranium ore in the ratio of only one part in every three and one half million parts of black oxide, it can be seen that for each pound of black oxide worth \$2.05, the ore contains about \$2.60 worth of radium.

The primary demands for radium were for medical purposes, luminous paint, and industrial radiography (for example, in detecting internal flaws in metal castings). The material used in luminous paints was mixed with other constituents and applied to a great many articles and, hence, was lost permanently, the material used for medical purposes and radiography was contained in capsules and because of its long half-life, remained essentially unaltered in form and quantity from year to year.

To produce the annual prewar requirements of radium (approximately 35-40 grams), there resulted as a by-product about 160 tons of

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refined uranium compounds such as black oxide and soda salt ( $\text{Na}_2\text{U}_2\text{O}_7$ ) which the radium producers were just able to dispose of to the ceramics industry and other customers. In fact, it appears from limited information available concerning this industry, that only during the last few years prior to the war did anything like an even balance exist between radium sales and uranium sales.

1-5. Development of Program. - At the outset, it was planned that all operations in connection with the project would be performed by one prime contractor, the Stone & Webster Engineering Corporation. However, as the program developed, it became apparent that it would be desirable to engage directly the services of other industrial organizations, in order to spread the load of many complex and perplexing problems among the best technical and operating personnel available in American industry. During the latter part of 1942, when the feed materials program began to take shape, it was recognized that the program fell into several general phases; i.e., location of raw material sources, procurement of raw materials, and refining and treatment of the raw materials to develop the proper feeds for the processing plants. Distinct lines of separation between these phases often could not be drawn since, frequently, operations in one phase included of necessity operations in another phase. For example, additional supplies of Belgian Congo ores were revealed while negotiations were being conducted for procurement of previously disclosed supplies; uranium from Canadian sources was bought as refined black oxide and not as raw ore; while uranium from American sources, in certain instances, was purchased as refined sodium salt rather than as raw ore. Essentially, however, the plan of operations was: (1) the location of adequate quantities of raw materials, (2) the procurement of materials

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in raw, semi-refined, or refined states, (3) the refining of raw materials, and (4) their treatment to obtain feed materials, in the necessary quantities and high degree of purity, required for the processing plants (App. E-70).

In general, procurement of ores from producers was handled under unit price contracts (See Part B, Procurement). The construction and operation of the various refining and treatment plants were handled, for the most part, under cost-plus-fixed-fee contracts and administered, successively, by the Manhattan District Materials Section and the Madison Square Area (See Part C, Refining and Treatment; and flow diagram, App. C1).<sup>A & B</sup>

1-6. Procurement.

a. Uranium-containing Materials. - It was realized from the start that, of the three available principal deposits containing uranium compounds (mentioned above in Par. 1-4a), the most important from the standpoint of both quantity and richness of ore was the Shinkolobwe mine in the Belgian Congo, and it was apparent that the bulk of the ore required for the project would have to come from the Belgian Congo. Accordingly, every effort was made to procure the maximum ore possible from this rich source, in addition to all that could be obtained from the Canadian and domestic sources (App. E1).

Initial procurement of black oxide was begun in 1941 by the OSRD, who handled research work prior to the formation of the Manhattan District, and provided the source of uranium for the very early work in connection with the project. In July 1942, the first order for black oxide was placed by Stone & Webster for the account of the Manhattan District. With the rapid development of the scope of the project, the raw material procurement activities, in the latter part of 1942, were relinquished by Stone & Webster and assumed, up to August 1943, by the

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Manhattan District Materials Section, and, since August 1943, by the Madison Square Area.

b. Miscellaneous Materials. - In addition to uranium-containing materials, which were of primary interest in the feed materials program, other auxiliary substances, such as radium and radium-neutron\* sources and radioactive lead, were procured for other project installations and programs (See Section 6).

1- 7. Raw Material Prices. - The extensive feed material requirements made desirable the procurement of all available uranium in the world which could possibly be acquired. Thus, ores varying widely in grade, and products at various stages of refining, had to be procured, and, consequently, no uniform price could be set for these uranium-bearing materials.

Material procured from Africa was obtained from the African Metals Corporation, which was by far the largest supplier of ore concentrates for the project, and the concentrates obtained from them were purchased at prices ranging from \$1.00 per pound to \$1.90 per pound of contained  $U_3O_8$ . All of the African ores required subsequent refining. However, a minor portion of the materials procured from this source was refined salts, which had been produced prior to the inception of the project, and the current market prices were paid for these materials.

In the case of the Canadian source, the material was paid for on the basis of delivery of refined black oxide at a price of \$1.95 per pound of black oxide produced, or approximately \$2.01 per pound of contained  $U_3O_8$ . Because of increasing costs in mining and refining operations a later contract for black uranium oxide from Canadian ore was made at a cost of approximately \$6.00 per pound.

In the case of materials procured from American sources, the bulk of supplies was derived from the purchase of low-grade tailings

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(from vanadium operations), which were obtained at prices ranging from \$0.20 per pound of recoverable  $U_3O_8$  in low-grade tailings to \$1.50 per pound of contained  $U_3O_8$  in more highly refined uranium-bearing sludges (See App. F3). All of the materials obtained from domestic sources required subsequent refining.

It should be noted, however, that a true comparison of costs cannot be made on the basis of the price of the contained  $U_3O_8$  in ore alone, because of the wide variation in grades and types of material, with the resultant difference in the cost of refining to recover the uranium content.

The bulk of the materials purchased on the open market was obtained at the current commercial market prices paid by the ceramics trade, the principal commercial user of various refined uranium compounds; i.e., \$2.05 per pound of black oxide, \$1.55 per pound of soda salt, \$0.75 per pound of sodium uranyl carbonate, \$2.36 per pound of uranium nitrate, etc.

1-8. Refining and Treatment. - At the beginning of the program, the only plants available for refining raw ores to black oxide were those of Eldorado Mining and Refining, and Vitro Manufacturing Company, who had been processing uranium-containing ores to commercial grade black oxide or soda salt (App. F7). It was soon apparent that these plants did not have sufficient capacity to process the quantities of raw material which would be required. Late in 1942, the Eldorado facilities were enlarged, and it was also decided to have Linde Air Products Company construct and operate a plant to refine low-grade pitchblende ores and the carnotite (App. A) concentrates which would be obtained by the preliminary treatment in Colorado of tailings from vanadium operations in the Colorado plateau region.

The nature and sequence of the black oxide treatment operations (to convert refined black oxide, in a series of steps to feed materials) was established in the late fall of 1942, and construction of manufacturing facilities was pushed ahead with all possible speed.

The first brown oxide plant, at Mallinckrodt Chemical Works, had been in operation since May 1942 under a contract with OSRD. Plans were made to enlarge this plant and to construct additional facilities at Linde and E. I. du Pont de Nemours & Company. The Mallinckrodt expansion was completed early in the spring of 1943, the du Pont plant started operations in June 1943, and the Linde plant in August 1943.

Harshaw Chemical Company had operated a small-scale green salt\* plant (~~App. A~~) under a contract with OSRD, as had du Pont at the Jackson Laboratory, and arrangements were made in the summer of 1942 for the Manhattan District to take over these plants. The Harshaw plant was expanded during the early part of 1943, and a new plant was constructed at the du Pont Chambers Works, which started operating in February 1943. Two other green salt plants were built, one at Mallinckrodt, which started operations in the spring of 1943, and the other at Linde, which came into production in October 1943.

Metal Hydrides, Incorporated, and Westinghouse Electric and Manufacturing Company were operating plants for the manufacture of metal, (~~App. A~~) under contracts with OSRD, at the time the Manhattan District came into being. In addition, research on new methods for the manufacture of metal was being carried on at Iowa State College and Brush Laboratories. In October 1942, it was decided to concentrate on the Iowa State College process, and contracts were entered into with Mallinckrodt, Electro-Metallurgical Company, and du Pont for the construction of the necessary



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plants. In addition, plans were made to expand the Iowa State College pilot plant, and this expansion was completed in March 1943, a month prior to the startup of production at Electro Metallurgical Company. The du Pont plant began operation in the spring of 1943, while the Mallinckrodt plant started production in July 1943. In the fall of 1943, the Westinghouse and Metal Hydrides processes for the manufacture of metal were discarded because of higher costs and lower yields than the Iowa State College process. Metal Hydrides, however, continued work for the project and recast scrap from the X-10 fabrication operation (See Part C).

1-9. Research and Development. - In view of the fact that prior demands for uranium had been relatively small compared to the large quantities required for the project, and since the contemplated use of the material involved obtaining highly purified forms never before developed on a commercial scale, it was necessary to devise new methods and procedures throughout all stages of the refining and treatment operations. The critical need for uranium demanded that wastes from the refining and treatment operations be held to an absolute minimum and that such wastes be recovered, if possible, and reintroduced into the system. In addition, the time schedules required that all operations be conducted with the utmost speed. Inasmuch as previously existing production facilities had been geared to a very small demand, extensive research and development work was necessary to combine efficiency and speed of operations. Accordingly, several contractors conducted careful studies and research programs in conjunction with specific operations (See Part C) to devise ways and means of performing operations which had never been performed before, to develop alternate methods, and to improve current processes. The cost of research and development activities to date had been about \$2,530,760.

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1-10. Recovery Operations and By-Products. - Throughout the refining and treatment operations, elaborate measures were taken to salvage by-product materials from the various processing operations and reintroduce those materials as feed for recovery operations. For example, the turnings resulting from the operation of machining the slugs (metal rods\* used in the pile process) to final size were cleaned and remelted to cylindrical-shaped billets\* and reintroduced as feed for the extrusion and machining plants. Likewise, the sheared-off ends of extruded metal rods were recast to billets. In addition, the by-products of the metal refining and treatment operations were re-treated in the du Pont recovery plant, and the recovered uranium content was introduced as feed for the brown oxide plants. A total of four plants was involved primarily in this reclamation procedure: the du Pont recovery plant; the Hooker Electrochemical Company slag plant, which concentrated certain slag for delivery to the du Pont plant; the Metal Hydrides recasting plant, which recast billets from scrap ends of the extruded bars and other solid metal scrap; and the Iowa State College turnings preparation and recasting plant, which reprocessed the turnings from the slug machining into metal billets (App. C1). This latter plant ceased operations in the winter of 1945 at which time the Hanford Engineer Works took over the turnings preparation operation and shipped the pressed briquettes to Metal Hydrides for recasting to billets.

Although the uranium-containing by-products were reintroduced as feeds whenever possible, those by-products whose reprocessing was not economical were normally stored at their point of origin, for security reasons, and in order that the uranium content would be retained for possible future recovery. Certain other by-products, such as recovered hydrofluoric acid and potassium fluoride, which do not contain uranium and

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which could be disposed of without jeopardizing security, were disposed of to commercial manufacturers, in accordance with standard War Department policies as expressed in Procurement Regulations paragraph 363 and Technical Manual 14-910.

1-11. Results. - To date, both the procurement and production objectives of the program have been successfully achieved. As to the procurement goal: the project has procured a total of about 10,000 tons of  $U_3O_8$  in ore concentrates. Of the raw material delivered to 1 January 1947, 72% has come from the Belgian Congo, 9% from Canada, 14% from the Colorado plateau region, and 5% from miscellaneous sources. (App. E73.)

Concerning the production goal: research and development work, construction of large-scale plants located throughout the country, solution of numerous and highly complex technical problems, and operation of the plants to produce the varied feed materials never before made on a commercial scale have all been accomplished within the time schedules which were established. The initial requirements of metal for experimental work at the University of Chicago had been filled by June 1943, and accumulation of metal for Clinton Laboratories and the Hanford X-10 plant was begun just prior to that time. In October 1943, the initial shipments of orange oxide to the Y-12 plant were begun; and in July 1944, deliveries of hexafluoride for the K-25 and S-50 plants began to reach significant quantities. To 1 January 1947 approximately 6,600 tons of uranium or its equivalent, including recast metal, were produced to meet the requirements of the several processing plants; and it may be noted that at no time were deliveries to any processing plants behind the forecast requirement schedules (App. D1, D2, D3).

1-12. Future Considerations. - The completion of the program will bring about a number of problems with certain foreign interests, problems

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in connection with the disposition of the residues from the raw ores as well as by-products of processing operations, and, finally, the disposition of the several refining and treatment plants themselves. As described more fully in Part B, the procurement arrangements made with African Metals Corporation provide for the purchase of only the uranium content of the ores by the Government; the storage and preservation of the remaining components of the ores, including the highly valuable radium content; and the return of these remaining components to the supplier. In addition, large stocks of vanadium pentoxide, which is commercially valuable for producing vanadium steel, have been accumulated as by-products of the refining operations. These products have been turned over to Army Navy Munitions Board for their metal stockpile.

1-13. Costs. - The estimated cost of the feed materials program, exclusive of O.S.R.D. costs, to 1 January 1947, was \$90,268,490, which represents \$27,592,360 for procurement of raw materials delivered, \$58,622,360 for refining and treating the raw materials to produce high purity feed materials in the required forms, and \$3,357,690 for research and development, and quality control activities (App. E69), \$88,400 for the procurement of radioactive lead; \$547,160 for radium, exclusive of that procured for Site Y; \$60,520 for thorium salt. Of the refining and treatment cost of \$58,622,360 (shown above), \$12,237,850 was for construction of government-owned facilities, \$46,384,510 was for operation of all facilities, including those previously existing or privately owned (App. F6 and E72).

The total cost of the feed materials program may be allocated to the processing plants approximately as follows: \$67,913,330 to the X-10 process, \$15,881,530 to the K-25 process, \$1,964,280 to the S-50 process (including the Navy Yard Pilot Plant), \$1,013,500 to the Y-12 process, and \$3,495,790 to miscellaneous uses.

1-14. Organization and Personnel. - The direction and supervision of the procurement and refining and treatment phases of the feed materials

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program were handled, for the most part, by officers on the staff of the District Engineer. At the outset, when the scope was relatively small and it was planned that the design, construction, and operation of the program would be performed by Stone & Webster Engineering Corporation, that company made the preliminary arrangements for the initial procurement of the raw materials. However, when the scope of the project began to expand and it was decided to confine the activities of Stone & Webster to the design and construction of the Y-12 plant and supplementary facilities, the initial procurement of ores and arrangements for refining and treatment were handled by the District Engineer, Col. J.C. Marshall, and his Deputy, Col. F.D. Nichols, who subsequently became District Engineer in August 1943. In October 1942, the Materials Section of the District office was organized under the supervision of Lt. Col. T.F. Greshaw for the administration of the feed materials program and remained under his direction until July 1943. He was succeeded by Lt. Col. J.R. Ruhoff, who had been his assistant. Lt. Col. Ruhoff continued directing the work of the Materials Section and, later on, became Area Engineer of the Madison Square Area, which was organized to continue the work of the District Materials Section when the District office was moved from New York to Oak Ridge, Tennessee, in August 1943. In October 1944, Lt. Col. Ruhoff was succeeded as Area Engineer by Lt. Col. W.E. Kelley, who was in turn succeeded by Colonel G.W. Beeler in April 1946.

B5 & B6

There are attached (App. B1, B2, B3) charts showing the organizations involved in the procurement and refining and treatment of raw materials, as of January 1/ 1943, 1944, 1945, 1946 and 1947. In addition, a chart (App. B4) shows the names of key contractors' organizations engaged in procurement and refining and treatment operations, and the general relationship of these organizations to Madison Square Area and

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the sub-areas, as of January 1, 1946 and 1 January 1947.

The key personnel concerned with the feed materials program were as follows:

Madison Square Area

Col. G.W. Beeler	-	Area Engineer, Madison Square Area, April 1946 to January 1947.
Lt. Col. T.T. Crenshaw	-	Head, Materials Section, October 1942 to July 1943.
Lt. Col. J.R. Ruhoff	-	St. Louis Area, July 1942 to October 1942; Assistant to Head, Materials Section, October 1942 to August 1943; Area Engineer, Madison Square Area, August 1943 to October 1944.
Lt. Col. W.E. Kelley	-	Area Engineer, Madison Square Area, October 1944 to April 1946. Deputy Area Engineer, Madison Square Area, April 1946 to January 1947.
Lt. Col. J.E. Vance	-	Head, Research and Development Sub-Section, November 1943 to April 1945; Area Executive Officer, April 1945 to August 14, 1946.
Lt. Col. A.W. Oberbeck	-	Executive Officer for Operations, April 1946 to January 1947.
Lt. Col. R.J. Walsh, Jr.	-	Executive Officer for Administration, July 1946 to January 1947.
Major G.W. Russell	-	Area Executive Officer, April 1945 to November 1944.
Major C. Hadlock	-	Materials Section, September 1942 to August 1943; Technical Officer, August 1943 to October 1944; Area Executive Officer, November 1944 to April 1945.
Major P.L. Merritt	-	Materials Section, October 1942 to August 1943; Head, Raw Materials Section, (now Raw Materials Division) August 1943 to January 1947.
Major O.H. Greager	-	Technical Officer, May 1945 to March 1946.

- Major E.A. Brinkman - Administrative Officer, August 1943 to November 1944.
- Major W.G. Akeley - Raw Materials Section, January 1944 to November 1944; Administrative Officer November 1944 to March 1946.
- Major W.W. Stag - Labor Relations and Safety Officer, August 1943 to November 1944.
- Major D.G. Sturges - Materials Section, October 1942 to February 1943; Head, M. Production Section, February 1943 to October 1944; Assistant to Technical Officer, October 1944 to August 1946.
- Capt. R.D. Morse - M. Production Section, March 1943 to April 1946.
- Capt. L.C. Burman - Materials Section, November 1942 to August 1943; Special Materials Section, August 1943 to July 1944; Raw Materials Section, July 1944 to June 1946.
- Capt. W.M. Hearon - Special Materials Section, January 1944 to March 1944; Head, Special Materials Section, March 1944 to June 1946.
- Capt. M.L. Hecker - Assistant to Area Executive Officer, September 1943 to June 1944; Head Control Section, June 1944 to May 1946.
- Capt. R. McKenzie - Assistant Administrative Officer, September 1944 to February 1946.
- Capt. L.G. Bassett - Analytical and Reports Sub-Section, April 1943 to November 1945.
- Capt. C.H. Bunker - Property Officer, January 1944 to March 1946.
- Capt. B.W. Menke - Intelligence Officer, January 1943 to September 1944.
- Capt. J.L. Davies - Intelligence Officer, July 1944 to June 1945.
- H.J. Sentiff - Accountability Section, August 1943 to February 1946.



F.M. Belmore - M. Production Section, February 1943 to October 1944; Head, M. Production Section (now Production and Accountability Division), October 1944 to January 1947.

G.C. Selfridge - Raw Materials Division, October 1946 to January 1947.

F. Zeitlin - Head, Special Projects Section, July 1943 to October 1945.

G.E. Winters - St. Louis Area, April 1943 to August 1943; Madison Square Area Research and Development Section, August 1943 to November 1944.

E.E. Chipman - Iowa Area, January 1943 to April 1943; M. Production Section, July 1943 to December 1944.

S. Sturges - Materials Section, December 1942 to August 1943; M. Production Section, August 1943 to January 1944. Special Projects Section, January 1944 to September 1946.

A.A. Levin - District Legal & Contracts Section, October 1942 to August 1943; Head, Madison Square Area Legal and Contracts Section, August 1943 to October 1945.


M.S. Lokietz - Head, Madison Square Area Legal and Contracts Section, March 1946 to January 1947.

S.P. Sullivan - District Audit Section, December 1942 to August 1943; Head, Madison Square Area Audit and Cost Section, August 1943 to August 1946.

J.F. McKee - Head, Madison Square Area, Audit and Cost Section, August 1946 to January 1947.

L.J. Cotton, Jr. - Administrative Division, November 1944 to February 1946.

J.S. Quider - Administrative Division, April 1946 to January 1947.





Tonawanda Area

- Major E.L. Van Horn - Area Engineer, November 1942 to August 1946.
- Capt. W. Thomas - Operations Officer, November 1943 to March 1946.

Wilmington Area

- Major W.L. Sapper - Area Engineer, December 1942 to November 1944.
- Major Dewey M. Stowers - Area Executive Officer, March 1943 to November 1944; Area Engineer, November 1944 to October 1945.
- Major C.W. Swartout - Production Officer, January 1943 to November 1944.
- Capt. O. Bergelin - Production Section, July 1943 to November 1944; Production Officer, November 1944 to October 1945; Area Engineer, November 1945 to February 1946.
- Capt. G.L. Ryan - Area Engineer, February 1946 to January 1947.

Colorado Area

- Major P.C. Leahy - Area Engineer, March 1943 to August 1946.
- R. Alexander - Tonawanda Area, February 1943 to December 1943; Technical Officer, Colorado Area, December 1943 to December 1945.

Cleveland Area

- Major H.S. Benbow - Area Engineer, February 1944 to June 1944.
- Capt. Wm. E. Dalton - Assistant to Area Engineer, February 1944 to June 1944; Area Engineer, June 1944 to November 1944, and June 1945 to December 1945; Assistant Administrative Officer, Madison Square Area, November 1944 to June 1945 and December 1945 to March 1946.

St. Louis Area and Iowa Area

- Major J.H. McKinley - Area Engineer, November 1942 to November 1943.
- Major H.A. Savigny - Area Engineer, November 1943 to August 1944.

- Major P.S. Finn - Area Engineer, August 1944 to November 1944.
- Capt. E.M. Velten - Assistant, November 1943 to November 1944; Area Engineer, November 1944 to January 1947.

(Iowa Area established in July 1943)

Beverly Area

- Capt. D. Duffey - Area Engineer, January 1943 to January 1944, M. Production Section, Madison Square Area, January 1944 to January 1947.

Mallinckrodt (Brown oxide, green salt, metal)

- Mr. J. Fistere, Jr., Secretary
- Mr. H.V. Farr, Vice Pres.
- Mr. H.E. Thayer, Project Manager

Harshaw (Green salt, hexafluoride)

- Mr. W.J. Harshaw, President
- Mr. K.E. Long, Director of Research

Linde (Black Oxide, brown oxide, green salt)

- Mr. J.A. Holladay, Vice Pres., Electro Metallurgical Co.
- Mr. T.J. Coleman, Plant Supt.
- Mr. A.R. Holmes, Plant Supt.

ElectroMet (Metal)

- Mr. J.A. Holladay, Vice Pres., ElectroMetallurgical Co.
- Mr. E.C. Forbes, Supt.
- Mr. R.M. Brinney, Asst. to Research Director

du Pont (Brown oxide, green salt, metal, scrap recovery)

- Mr. S.W. McCune, Jr., Mgr., War Products Div., Orchem Dept.
- Dr. H.W. Elley, Director of Research, Orchem Dept.
- Mr. E.W. Fielding, Area Supervisor
- Dr. J.M. Clark, Asst. Area Supervisor

Metal Hydrides (Metal, scrap metal recasting)

- Dr. P.P. Alexander, President
- Dr. R.J. Anicetti, Plant Supt.

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Westinghouse (Metal)

Dr. J.W. Marden, Asst. Director of Research  
Mr. A. Frankel, Business Mgr.

Iowa State College (Metal, turnings recovery)

Dean H.V. Gaskill, Dean of Science  
Dr. F.H. Spedding, Project Mgr.

U. S. Vanadium Corp. (Concentrating ores and tailings)

Mr. B. Burwell, Vice Pres.  
Mr. J.L. Robinson, Supt., Colorado Operations

Eldorado (Refining of ores)

Mr. G. LaBine, President  
Mr. A. Ross, Plant Supt.

Vanadium Corp. of America (concentrating ores and tailings)

Mr. E.D. Bransome, President  
Mr. D. Viles, Plant Supt.

Vitro (Refining of ores to soda salt)

Mr. E.M. Fleck, President  
Mr. A.J. Strod, Vice President

Princeton University (Quality control, Analytical)

Dr. M.N. Furman, Prof. of Analytical Chemistry

Mass. Inst. of Technology (Quality control, Analytical)

Dr. G.R. Harrison, Dean of Science  
Mr. R. Kent, 3d., Project Manager

Yale University (Research on ore extraction and refining)

Dr. A. Hill, Chairman of Chemistry  
Dr. H.S. Harned, Prof. of Physical Chemistry

National Bureau of Standards (Quality control, Analytical)

Dr. C.J. Rodden, Project Manager  
Mr. J.G. Thompson, Chief, Metallurgical Section  
Mr. B.F. Scribner, Chief, Spectroscopy Section

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Part B

PROCUREMENT

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SECTION 2 - AFRICAN SOURCES

2-1. Operations. - The Shinkolobwe mine, located in the southeastern corner of the Belgian Congo, has provided the Manhattan District with the richest and most abundant source of uranium-bearing raw materials (App. E2). Union Miniere du Haut Katanga, a Belgian company, owned the controlling interest in the Shinkolobwe mine and all sales in the United States were handled by the African Metals Corporation of New York, New York. The extreme richness of the mine's ore, assaying as high as 65% to 75%  $U_3O_8$ , has enabled Union Miniere du Haut Katanga and its agent, the African Metals Corporation, to dominate completely the radium and uranium market ever since the beginning of the mine's production in about 1928.

The Shinkolobwe mine was discovered in 1915, but production did not begin until after World War I. Prior to 1931, the Shinkolobwe mine operated intermittently as the market for radium required, and, in 1931 it was reopened after being closed for several years. In 1938, an ore treatment plant was constructed, primarily for the recovery of precious metals occurring in the ore. However, because of poor worldwide economic conditions, sales of radium and uranium were not commensurate with mining and recovery of precious metals. As a consequence, large stocks of ore containing radium and uranium were accumulated. Enough radium and uranium in ore were contained in stock piles to satisfy the ordinary commercial market for approximately 30 years. Therefore, the mining and recovery operations were stopped in 1937.

Prior to the Manhattan District project, mining operations at Shinkolobwe had been conducted primarily for the recovery of radium in

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subsequent refining operations at Union Miniere's refinery at Oolan, Belgium. The uranium and other precious metals contained in the ore were considered as by-products of radium production. Because of the extremely high value of the radium and other precious components of the ore, exclusive of uranium, it was undesirable for the United States Government to become involved, over an extended period of time, in disposing of the radium and other by-products to the commercial markets of the world; and because Union Miniere was not desirous of relinquishing its control over the radium market, an arrangement was made whereby only the uranium content of the ores was to be purchased. This plan was followed in every contract for the procurement of African ore, with one exception, African Metals Contract W-7405 eng-279 (App. E20).

Except for the purchase of approximately <sup>181</sup>186 tons of  $U_3O_8$  in ore which was mined from an open cut at the Shinkolobwe mine (African Metals W-7405 eng-280), all ore purchased was obtained from accumulated stock piles (App. E3). Approximately 767 tons of  $U_3O_8$  contained in 1,200 tons of 65% ore was obtained from a stock pile which had been shipped by African Metals from the Belgian Congo to the United States during the latter half of 1940 and stored at the Archer-Daniels-Midland Company Warehouse at Port Richmond, Staten Island, New York (App. E4). Previously, all refining of ores had been conducted at Union Miniere's refinery at Oolan, Belgium. However, in view of the seriousness of the European situation in 1940, when the Axis forces were in complete domination of the continent of Europe and were moving rapidly across North Africa, this ore had been shipped to the United States both for the purpose of preventing its falling into the hands of the enemy and for

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possible use in this country during the war. The fact that this material was in this country was well known in trade circles, as there was no secret regarding the importation of uranium ore at that time. The ready availability of this stock pile made possible the early experiments and trials in refining processes; and the initial procurement of ore was made from this stock pile, with the option to purchase the remainder of the material stock-piled in the United States as well as the additional quantities stock-piled at the Shinkolobwe mine.

As of 1 January 1947, approximately 5,839 tons of  $U_3O_8$ , contained in about 29,734 tons of ore, varying from about 3% to 66%  $U_3O_8$  content, had been contracted for from African sources at a total cost of approximately \$9,113,800. In addition to this material obtained by contract, title to approximately 3,144 tons of  $U_3O_8$  in ore had been procured by the Washington office as of 1 January 1947 at a total cost of approximately \$10,267,800.

2-2. Contract Negotiations. - African ores were purchased by the Manhattan District on the basis of the recoverable  $U_3O_8$  content. When the early contracts were negotiated, sufficient data as to the amount of  $U_3O_8$  recoverable from the ore were not available. As a result, the calculation of the percentage of  $U_3O_8$  contained in the ore to be paid for by the Manhattan District was of necessity based on the available information concerning the recoveries at the Union Miniere's refinery in Belgium and also on the percentage recovery which would be guaranteed by the two largest refining companies in North America, Eldorado Mining and Refining (see par. 3-1) and the Vitro Manufacturing Company (App. E5). At that time, Eldorado, which of these two companies had the greater refining knowledge and experience, would not guarantee a recovery of more than

80% of the  $U_3O_8$  contained in the ore. Therefore, in the first procurement contract, the Government agreed to pay for only 80% of the  $U_3O_8$  contained in the ore.

A price of \$1.00 per pound for an 80%  $U_3O_8$  recovery was agreed upon for the original lot of ore processed. This price was arrived at by taking the current wholesale market price for black oxide, less the normal commission on sales to wholesalers, less the cost of refining the ore. The wholesale price of black oxide was \$2.05 per pound, commissions on sales to wholesalers were approximately \$0.45 per pound, and the cost of refining as established by Eldorado was \$0.60 per pound.

During subsequent negotiations for the procurement of additional lots of ore, African Metals stated that this initial price of \$1.00 per pound of recoverable  $U_3O_8$  was considerably lower than the actual value of the ore to them. African Metals based the value of the ore to them on the sale price of refined black oxide minus the refining cost in Belgium and freight rates on the black oxide from Belgium to the United States. In 1941 and 1942, African Metals sold black oxide in the United States for \$2.05 per pound. African Metals estimated the refining cost of ore to black oxide in Belgium to be \$0.20 per pound of black oxide (as compared to the Eldorado refining cost of \$0.60 per pound). The approximate freight charge on black oxide from Belgium to United States prior to the war was \$0.05 per pound. Thus, on this basis, the sales value to African Metals of  $U_3O_8$  in ore was about \$1.80 per pound of recoverable  $U_3O_8$  content, f.o.b. Belgium. This premise was not accepted by the Government, as indicated by the



fact that the average cost per pound of recoverable  $U_3O_8$  in ore bought to date is \$1.12. The price rise from the original figure of \$1.00 per pound for 80% of the contained  $U_3O_8$  to an average of \$1.12 per pound was based upon the fact that subsequent refining operations at Eldorado proved that they could recover considerably more than the original estimated 80%.

The procurement of African ores was accomplished under the contracts and purchase orders shown in App. F1 (also see App. B5).

2-3. Transportation of Ore from Africa. - The transportation during wartime of large shipments of vitally needed material necessitated the use of extreme care in scheduling operations and shipments. Inasmuch as the submarine menace continued during much of the period of shipment, arrangements were made whereby the ores were shipped by fast motor vessels traveling out of convoy. These arrangements were made with the shipper after consulting with the Transportation Corps, U. S. Army, as to the safest method of shipment. Two shipments were lost at sea; one late in 1942 and one early in 1943. The first shipment was lost through enemy action and the second through a marine accident. A total of approximately 200 tons of  $U_3O_8$  in ore was lost through sinking. Inasmuch as all purchases were made c.i.f.\* New York, all losses at sea were the responsibility of African Metals.

2-4. Storage. - Since shipments from Africa arrived faster than refining operations in this country could be carried out, it was necessary to store the ores temporarily in the United States before shipping them to the refineries. The original storage facilities were in the Seneca Ordnance Depot, Romulus, New York. These facilities were

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utilized because they were the only ones at that time readily available near the point where the ore would subsequently be processed. A little later in the program, incoming ore was stored in a warehouse at the Clinton Engineer Works, which had been constructed primarily for the purpose of storing radium-bearing sludges derived from the ores (see below). In November 1945, a warehouse was leased at Middlesex, New Jersey, (Perry Warehouse, W-7407 eng-133, and W-42-069 eng-7 App. F9), in order that storage and sampling facilities would be available near the Port of New York, thus, avoiding the unnecessarily long shipments by rail (App. B7). The uranium ores were received at the Port of New York in bags and drums and were then shipped directly to Middlesex for storage and sampling, after which the sampled ores were shipped to refiners as needed.

It was also necessary to store the valuable sludges remaining after the uranium had been extracted from the ores. In accordance with the original arrangements, these sludges remained the property of African Metals and were to be returned to them after the war for recovery of radium and precious metals. Special warehouses were constructed at the Clinton Engineer Works for the purpose of storing radium-bearing sludges which were derived from the ores. The radium-bearing sludges derived from grades of ore of greater than 10%  $U_3O_8$  content were packed in wooden barrels after completion of the refining operations and then shipped to the Clinton Engineer Works, and to the Middlesex Warehouse for storage. The radium residues from low-grade ores containing 10% and less  $U_3O_8$  were stored in a bulk pit at the Lake Ontario Ordnance Works, Modeltown, New York, near the plant of the Linde Air Products

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Company, where these low-grade ores were refined. A small quantity of these sludges from low-grade ores was also stored at the Middlesex Warehouse. During 1946, the approximately 1,024 wet tons of radium sludges from high-grade ores in storage at the Clinton Engineer Works <sup>WERE</sup> shipped to the Middlesex Warehouse for storage. As of 1 January 1947, there were approximately 20,209 wet tons of radium sludges from low-grade ores in storage at the Lake Ontario Ordnance Works and 21 wet tons of similar material at the Middlesex Warehouse. As of 1 January 1947, approximately 1,645 wet tons of radium sludge from high-grade ores had been returned to African Metals in accordance with contractual arrangements, and approximately 761 wet tons of similar material remained in storage at the Middlesex Warehouse.

2-5. Weighing, Sampling and Assaying. - A program of weighing, sampling, and assaying was necessary in order to establish the actual amount of uranium content in the African ores for which payment was to be made, since in most cases, only the uranium content of the ore was purchased. Also, it was necessary to establish with the refineries the actual amount of uranium content in the raw materials for which they were to be charged.

This weighing, sampling, and assaying program was necessary only for the African-source materials. In the case of materials purchased from Eldorado, weighing and sampling of the ores were not required, since the ores were refined by Eldorado itself, and the product was purchased directly by the Manhattan District. Likewise, in the case of the procurement of uranium products from American producers, the supplies were purchased on the basis of refined materials, and no

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weighing, sampling, and assaying program was necessary for the crude ores or tailings procured.

At the beginning of the program, African ores were weighed, sampled, and assayed at the refinery to which they were delivered for processing, and the results of these weighings and samplings were accepted by the vendor and the processor, as well as by the Manhattan District. In order to limit the knowledge which the vendor might gain through this procedure, a separate sampling program was initiated at the Middlesex Warehouse in November 1943, and, after this date, all weighing and sampling results from Middlesex were accepted by the refineries who subsequently received the ore. Prior to the establishment of the warehouse, sampling was carried out in the presence of a representative of the vendor and a representative of the processor, and, as long as agreement could be reached by these two, the results were acceptable to the Manhattan District. After the establishment of the Middlesex Warehouse, Contract W-7421 eng-16, dated 11 May 1944, was written with the firm of Lucius Pitkin, Inc., of New York, to serve as the sampling representative and consultant for the Manhattan District in weighing and sampling matters. This contract was terminated on 12 January 1945, because the amount of raw materials then entering the Middlesex Warehouse was not sufficient to warrant full-scale weighing and sampling services. On 5 August 1945, Contract W-35-058 eng-9 was written with Lucius Pitkin, Inc., to continue weighing and sampling services, and this contract was active as of 1 January 1947. African Metals was represented in all sampling operations by Ledoux and Company of New York. Samples were taken in accordance with recommendations made by the sampling representatives, and all procedures followed were those most adequate and

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adaptable to the numerous problems presented. All procedures were approved by competent authorities, including the National Bureau of Standards, Ledoux and Company, Lucius Pitkin, and the contractors of the Manhattan District who subsequently refined the ore.

Assays were made both by African Metals and by the Government. When materials were forwarded for refining, the refinery was supplied with a sample taken and sealed at Middlesex, and the refinery carried out its own assay on this portion of the ore. African Metals employed the firm of Ledoux and Company of New York for their assaying purposes, and the Government used the services of the Prick Chemical Laboratories at Princeton University Contract W-7408 eng-81 in New Jersey. In the Spring of 1948, the contract with Princeton University was completed and Contract W-35-068 eng-10 was negotiated with the firm of Lucius Pitkin to perform the Government assay work. The National Bureau of Standards was used as an umpire for assaying when the results of the original two assays were not within 0.30%  $U_3O_8$ .

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SECTION 3 - CANADIAN SOURCES

3-1. Operations. - As mentioned in Part A, Par. 1-4a, an important source of uranium-bearing ores was the Eldorado mine, located on the south-east shore of Great Bear Lake in Canada. This mine was originally owned by the Eldorado Gold Mines, Ltd. On 3 June 1943, the name was changed to Eldorado Mining and Refining, Ltd., and on 28 January 1944, the mine was expropriated by the British Crown, and the name was changed to Eldorado Mining and Refining (1944) Ltd. (App. E8). The mining and refining operations commenced in 1933 and were directed toward the recovery of radium, uranium, and silver. The mine is ice-bound from November through June, and, though mining operations are continued during that period, it is not possible to ship ores to the refinery at Port Hope, Ontario. Operations were geared so as to provide year-round production at the mine, and shipments made from the mine during the months of July to October consisted of the entire ore output of the mine for the previous year and served as feed materials for the refineries for the succeeding year. Operations at the Eldorado mine were carried on from 1933 until the summer of 1940, at which time sufficient stocks of ore had been accumulated at the mine to satisfy the commercial market for approximately five years. However, in 1941, the OSRD placed an order for ore which necessitated the opening of the mine. Therefore, in the spring of 1942, equipment and supplies were procured and mining, as well as refining operations at Port Hope, have continued since that date in order to fill the subsequent requirements of the Manhattan District. The early contracts for the purchase of black oxide were written directly with Eldorado, or with Boris Pregal, president of the Canadian Radium and Uranium Corporation of New York City, the sales agency for Eldorado's products in the United States. Since September 1943,

Manhattan District contracts for the purchase of uranium ores, for security reasons, have been written in the name of Carl French or Gilbert A. LaBine, who were, respectively, treasurer and president of Eldorado, and acted as agents for that company.

The ores of the Eldorado mine have assayed approximately 1%  $U_3O_8$ , and, because this ore is of considerably lower assay than that of the Shinkolobwe mine, the refining costs to produce radium and uranium are considerably higher than similar refining costs of the higher grade ore. Consequently, Eldorado has never been able to challenge Union Miniere's domination of the radium and uranium market.

A total of approximately 4,149 tons of ore concentrates to be delivered as 1,137 tons of  $U_3O_8$  in the form of black oxide, had been contracted for from Canadian sources as of 1 January 1947. Late in 1942, approximately 140 tons of  $U_3O_8$  in the form of black oxide, requiring 172 tons of  $U_3O_8$  in ore concentrates, were delivered under a purchase order issued by the Stone & Webster Engineering Corporation for the account of the Manhattan District. Since that date, the Manhattan District has contracted for 3,659 tons of ore concentrates to be delivered as 997 tons of  $U_3O_8$  in the form of black oxide. The total cost of the procurement of Canadian ore to 1 January 1947 was approximately \$5,082,300 (See App. F2).

3-2. Contract Negotiations. - Inasmuch as Eldorado had its own refinery at Port Hope, Ontario, it was possible for the Manhattan District to purchase black oxide directly as such, and it was not necessary to enter into arrangements for the storage or disposition of the by-products of refining processes, since these by-products remained with Eldorado. Consequently, the procurement contracts with Eldorado were based only on the delivery of black oxide. The prices for this black oxide were based

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primarily on the market prices for large orders of black oxide at the outset of the procurement program in 1942. The wholesale price of black oxide for the ceramic trade at that time was \$2.05 per pound. Contracts were negotiated on the basis of a 5% reduction in price due to the volume of the purchases, making an average price of approximately \$1.95 per pound. This price of \$1.95 per pound probably did not reflect the true cost to Eldorado; however, Eldorado was forced to sell their product at this price in order to compete in the open market with the lower cost producer, Union Miniere. The last contract with Eldorado, which was negotiated with the Canadian Government, set a top price of \$4.20 per pound of black oxide, with credits to be given later when actual costs had been determined and when the value of the contained radium had been determined. As of 1 January 1947 negotiations were nearing completion for increasing the top price to \$6.17 per pound of black oxide. This contract is a no-profit contract and will probably reflect the true cost of operating at Eldorado (App. E9).

The procurement of black oxide from Canadian ores has been conducted under the Stone & Webster Purchase Order No. 135 and Contracts W-7405 eng-145 with Boris Pregel, and W-7405 eng-252 and W-26-021 eng-6 with Gilbert A. LaBine, Agent for Eldorado. Contract W-7405 eng-145 was terminated for security reasons, and the black oxide to be produced under this contract was added to that to be produced under Contract W-7405 eng-252. These contracts and the purchase order are listed in App. F2.

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SECTION 4 - AMERICAN SOURCES

4-1. Operations.- As mentioned in Part A, Par. 1-4a, the third important source of uranium-bearing materials was the Colorado Plateau region in the United States. Carnotite, the most important type of uranium-bearing ore in the United States, was discovered in this area in 1899, but large-scale mining and refining operations did not begin until about ten years later. Between 1911 and 1923, high-grade carnotite ores were selectively mined principally for the radium content and, to a minor degree, for the by-products, vanadium and uranium. The minimum grade of ores mined during this period is reported to have had a  $U_3O_8$  content of approximately 1.25%. After 1923, production declined rapidly because of the unfavorable position which carnotites had in the radium field, with respect to radium derived from the rich ores mined in the Belgian Congo. During the period of exploitation of carnotites for radium, it was reported that a total of approximately 200 grams of radium, equivalent to approximately 900 tons of  $U_3O_8$  in ore, was produced.

Since 1937, carnotites have been mined principally for their vanadium content. The ores, as mined, averaged approximately 1.75% vanadium pentoxide, ( $V_2O_5$ ) and approximately 0.25%  $U_3O_8$ . The high demand for vanadium, brought about by World War II, stimulated vanadium production throughout the region. The Metals Reserve Corporation, a Government agency, attempted to increase production by offering increased prices for the vanadium and by offering loans to small independent operations<sup>ORS</sup>, in order to increase their production. U. S. Vanadium Corporation, Vanadium Corporation of America, and the Metals Reserve Corporation were the most active organizations in the production of vanadium during this period.

Early in 1944, the critical period in vanadium requirements was passed and Metals Reserve ceased its stimulation of production. U.S.V. and several small producers accordingly decreased their production commensurate with commercial requirements, while V.C.A. continued its normal production.

As a result of the mining and refining of carnotite ores, principally for the recovery of radium and vanadium, during the past 30-35 years, there were accumulated tremendous stock-piles of tailings which contained varying low, but economically recoverable, percentages of uranium. Investigations conducted late in 1942 showed that these valuable stock-piled tailings, as well as the tailings from the current vanadium operations, could be obtained from the U.S.V. plant at Uravan, Colorado, the V.C.A. plant at Naturita, Colorado, and from Metals Reserve, which owned a plant operated by U.S.V. at Durango, Colorado, and a plant operated by V.C.A. at Monticello, Utah. In the case of the Metals Reserve Monticello plant, recovery of uranium was possible only from the current operations tailings, since the stock-piled tailings of former vanadium operations were too low-grade to be economically treated (App. C1).

In addition to the tailings procured from these three major companies, small lots of tailings were purchased in scattered localities and processed in Manhattan District-owned facilities, if the delivered cost of such tailings was not excessive.

As of 1 January 1947, a total of approximately 1,349 tons of  $U_3O_8$ , contained in 379,671 tons of ore tailings (App. F3) had been contracted for. Of the total  $U_3O_8$  content contracted for, approximately 891 tons have been secured from U.S.V., 230 tons from V.C.A., 135 tons

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from Metals Reserve, 26 tons from the Vitro Manufacturing Company and 67 tons from other miscellaneous sources. All of the  $U_3O_8$  purchased was contained either in low-grade tailings assaying approximately 0.25%  $U_3O_8$ , or in uranium sludges assaying from 10% to 50%  $U_3O_8$ . The total cost of the procurement of American-source materials to 1 January 1947 was approximately \$3,072,330.

4-2. Contract Negotiations. - In order to obtain the uranium contained in the carnotite ores of the Colorado Plateau region, as well as that contained in the tailings of current and past vanadium operations, negotiations were conducted with the three major companies in the vanadium industry, namely, U.S.V., V.C.A., and Metals Reserve.

From the strict viewpoint of the procurement of raw materials these negotiations were merely for the purchase of tailings and sludges, to be further refined to black oxide by other processors. However, it seems advisable at this time to mention generally how these tailings and sludges were obtained, even though the details of these operations are discussed in Part G. U.S.V. and V.C.A., the largest two producers in the vanadium field, had each developed and wished to use a different process for the recovery of uranium from domestic sources. The U.S.V. process consisted of the treatment of the tailings which were left from their current and past vanadium operations, to produce a uranium sludge suitable for further refining to black oxide. The V.C.A. process consisted of the direct treatment of the carnotite ores to produce a combined uranium-vanadium sludge, which would then be sold to the Government for further treatment to remove the vanadium and produce black oxide. Previous to the entrance of the Manhattan District into the field, V.C.A. did not further process the tailings from the uranium-vanadium sludge

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extraction, since these tailings did not contain enough uranium or vanadium to make further processing commercially economical. However, the urgent requirements of the Manhattan District for uranium-bearing feed materials made the treatment of such tailings desirable. U.S.V. was reluctant to allow V.C.A. the use of their process for the recovery of uranium through the treatment of tailings, and V.C.A. was reluctant to allow U.S.V. the right to construct and operate tailings-treatment plants on the sites of the V.C.A.-operated vanadium plants (App. E10). Therefore, since it was necessary for the Manhattan District to obtain all the uranium available from domestic ores, arrangements were made to procure both types of raw materials: (1) a uranium sludge produced by U.S.V. from treatment of vanadium-operations tailings, and (2) a combined uranium-vanadium sludge produced by V.C.A. from the direct treatment of carnotite ores. The tailings from the current and past uranium-vanadium extraction at V.C.A. were to be bought by the Government and sent to the U.S.V. plants for further processing. (Vides Case 100)

In general, the following arrangements were made to procure uranium-bearing raw materials from domestic sources for further processing to black oxide (App. E11). The details of these operations are discussed in Part C, Paragraphs 7-5 and 7-6.

a. U.S.V. agreed to:

- (1) Construct and operate, at their own expense, a uranium sludge plant on the site of the U.S.V. plant at Uravan, Colorado. The feed for this plant was to be derived from the existing vanadium tailings stockpile at Uravan, supplemented by the tailings of current

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vanadium operations at Uravan. The output of uranium sludge was purchased by the Manhattan District on a unit price basis and was then further refined to produce black oxide.

- (2) Construct at Government expense, and operate for the Government, a uranium-vanadium sludge plant on the site of the vanadium plant at Uravan, Colorado, on a cost-plus-fixed-fee basis. The feed for this plant was to be the stock-piled tailings existing at Uravan, as well as various tailings procured by the Manhattan District from V.C.A.'s stock-piles at Naturita. The tailings were purchased by the Manhattan District at a unit price based upon the recoverable  $U_3O_8$  and  $V_2O_5$  obtained in the central refinery at Grand Junction, Colorado.
- (3) Construct at Government expense, and operate for the Government, a uranium-vanadium sludge plant on the site of the Durango, Colorado, plant which was being operated for Metals Reserve. This construction and operation was done on a cost-plus-fixed-fee basis. The feed for the plant was to be the vanadium tailings stock-piled at Durango and also tailings derived from current vanadium production at Durango, both of which were owned by Metals Reserve. The tailings were purchased by the Manhattan District at a unit price based upon the recoverable  $U_3O_8$  and  $V_2O_5$  obtained in the central refinery at Grand Junction.
- (4) Construct at Government expense, and operate for the Government, a central refinery at Grand Junction, Colorado,

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on a cost-plus-fixed-fee basis. The object of the central refinery was to remove the vanadium contained in the uranium-vanadium sludge produced at Uravan and Durango. This uranium sludge was then suitable for further refining to black oxide.

b. V.C.A. agreed to:

- (1) Produce uranium-vanadium sludge from current vanadium operations at their Naturita, Colorado, plant. These sludges were then purchased by the Manhattan District on a unit-price basis. Most of the tailings resulting from the sludge-producing operation were of too low-grade for further treatment.
- (2) Sell to the Manhattan District stock-piles of vanadium tailings from the past vanadium operations and high-content tailings from current production at Naturita. These tailings were to be further treated in the Manhattan District-owned facilities at Uravan and Grand Junction.

c. Metals Reserve agreed to:

- (1) Sell to the Manhattan District stock-piles of vanadium tailings from past operations and current vanadium tailings resulting from the operation of the vanadium plant at Durango, Colorado, operated by U.S.V. for Metals Reserve. The tailings were purchased at a unit price based upon the recovery of  $U_3O_8$  and  $V_2O_5$  in the Manhattan District-owned facilities at Grand Junction, Colorado.

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(2) Produce uranium-vanadium sludge from the current vanadium operations at the Monticello, Utah, plant which was operated by V.C.A. for Metals Reserve. The sludge was purchased on a unit price basis by the Manhattan District from Metals Reserve. After the Monticello plant was closed by Metals Reserve in February 1944, it was reopened by V.C.A. and the output of uranium-vanadium sludge was then purchased directly from V.C.A. (App. E13).

Procurement of uranium-bearing materials from U.S.V. was made under three contracts, W-7405 eng-201, W-7405 eng-250, and W-26-021 eng-1. These materials were procured at prices varying from \$0.30 per pound of contained  $U_3O_8$  and \$0.30 per pound of  $V_2O_5$  to \$1.10 per pound of  $U_3O_8$  and \$0.31 per pound of  $V_2O_5$  (App. E13). A total of approximately 891 tons of contained  $U_3O_8$  had been contracted for as of 1 January 1947 from U.S.V. at a cost of approximately \$941,800. These contracts are listed in App. F3.

Six contracts were entered into with V.C.A. for the procurement of uranium-bearing materials. These contracts were W-7405 eng-12, W-7405 eng-144, W-7405 eng-256, W-7405 eng-257, W-7405 eng-267, and W-26-021 eng-12. These materials were procured at prices which varied from \$0.25 per pound of contained  $U_3O_8$  and \$0.55 per pound of  $V_2O_5$  to \$1.50 per pound of  $U_3O_8$  and \$0.90 per pound of  $V_2O_5$ . A total of approximately 230 tons of  $U_3O_8$  had been contracted for as of 1 January 1947, at a cost of approximately \$692,350. The contracts are listed in App. F3.

Three contracts were entered into with Metals Reserve for the procurement of raw materials. These contracts were W-7405 eng-260,

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W-7405 eng-282, and W-7405 eng-287. The cost of the materials varied from \$1.00 per ton for tailings containing about 0.25%  $U_3O_8$  (which amounts to approximately \$0.20 per pound of  $U_3O_8$ ) to \$1.10 per pound of  $U_3O_8$  and \$0.90 per pound of  $V_2O_5$  in sludges assaying 50%  $U_3O_8$  and 25%  $V_2O_5$ . A total of approximately 135 tons of  $U_3O_8$  had been contracted for as of 1 January 1947 from this company, at a cost of approximately \$216,300. The contracts are listed in App. F3.

In March of 1946, a contract, W-26-021 eng-24, was entered into with the Vitro Manufacturing Company for the purchase of this contractor's stockpile of high grade carnotite ore, containing approximately 26 tons of  $U_3O_8$ , at a lump sum of \$71,880. (App. F3.) Contract W-26-021 eng-24, also covered the processing of the carnotite ore to soda salt. This phase is covered in Section 7.

A number of purchase orders were written with various vanadium producers throughout the country for the procurement of uranium-bearing sands and tailings. A total of approximately 67 tons of  $U_3O_8$  had been contracted for as of 1 January 1947, at a cost of approximately \$150,000 (App. F3).

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SECTION 5 - MARKET AND MISCELLANEOUS PROCUREMENT

5-1. Operations. - Prior to the Manhattan District program, approximately 150 tons of uranium compounds were being consumed annually in the manufacture of ceramic colors. The need for conservation and control of uranium supplies was recognized in December 1942, at which time the War Production Board was requested to take appropriate action. In accordance with that request, Conservation Order M-286 was issued by WPB on 26 January 1943, prohibiting the further sale or purchase of uranium compounds for use in the manufacture or decoration of ceramic products (App. E14). In August 1943, the purchase or sale of uranium compounds for photographic use was also restricted and certain stocks and transaction reports were required to be sent to the War Production Board, in order to control more closely the permitted use of uranium compounds for military and essential industrial applications (App E15). These reports were made available to the Manhattan District and, based upon the information contained in them, a purchasing program was established, with the objective of securing as much as possible of the available stocks of various refined uranium salts for the Manhattan District (App. E16).

A total of approximately 270 tons of  $U_3O_8$ , contained in various refined uranium salts, had been obtained from available stocks as of 1 January 1947. These materials were procured at a cost of \$1,056,130 (See App. F4).

5-2. Contract Negotiations. - Some of the available uranium stocks were held by such companies as the Vitro Manufacturing Company, the Harshaw Chemical Company, African Metals, and the Canadian Radium and Uranium Corporation, who were already under contract to the Manhattan District for other phases of the work. In the case of these companies, direct

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contracts and purchase orders were written to obtain their uranium stocks. However, because security regulations made it inadvisable for the Manhattan District to contact the many other commercial concerns who had small stocks of uranium compounds, the Vitro Manufacturing Company was authorized to act as an agent of the Government in order to obtain the stocks of uranium compounds available throughout the United States. Vitro was selected to conduct this procurement program since they were already active in the Manhattan District work and also were well known in the trade as a commercial processor, seller, and consumer of uranium compounds.

Most of the market procurements were made at the current commercial market prices paid by the ceramics trade, the principal commercial user of various refined uranium compounds; i.e., \$2.05 per pound of black oxide, \$1.55 per pound of sodium uranate, \$0.75 per pound of uranyl carbonate, \$2.36 per pound of uranium nitrate, etc. (App. E21).

The market procurements of uranium salts were made under the contracts and purchase orders listed in App. F4.

5-3. Miscellaneous. - The Washington Office also obtained approximately 481 tons of  $U_3O_8$  in the form of miscellaneous compounds, mostly impure sodium salts. These materials were found by our armed forces in the European Theatre of Operations.

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SECTION 6 - PROCUREMENT OF OTHER RADIOACTIVE MATERIALS

6-1. Operations. - In addition to the procurement of uranium as a raw material for the Manhattan District project, it also became necessary to procure certain other radioactive substances, i.e., radium and radium-neutron sources and radioactive lead. Inasmuch as these materials occur in nature in conjunction with uranium, their procurement was handled as a part of the general procurement program.

a. Radium. - The need for radium and radium-neutron sources increased steadily until, as of 1 January 1947, a total of 73 grams of radium had been procured for the various installations of the Manhattan District. Of this total, about 36½ grams of radium were purchased and about 36½ grams rented at a total cost to 1 January 1947 of \$679,399 (App.F6).

b. Radioactive Lead. - In September 1943, it became necessary to obtain substantial quantities of radioactive lead (App. E17). Fortunately, radioactive lead was one of the by-products recovered in refining uranium ore to black oxide, and it was possible to recover enough radioactive lead from this source to fill all requirements (App. E18). As of 1 January 1947, approximately 85 tons of lead oxide had been procured, which amount fulfills the present known requirements of the project. The total cost of the procurement of radioactive lead was approximately \$68,400 (App. F5).

6-2. Contract Negotiations.

a. Radium. - During 1943, the market price for radium ranged from \$23.00 per milligram for quantities of less than 25 milligrams to \$18.00 per milligram for quantities greater than 100 milligrams. As the Manhattan District's requirements for radium increased, an arrangement

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was made with the two main suppliers, the Radium Chemical Company and the Canadian Radium and Uranium Corporation, whereby the Government would get the benefit of a reduction in price according to the following schedule:

Radium Chemical Company

Less than 25 milligrams .....	\$23.00 per milligram
25 to 100 milligrams .....	\$20.00 per milligram
100 milligrams and over .....	\$15.00 per milligram

The above schedule applies to both plain radium and to radium-neutron sources.

Canadian Radium and Uranium Corporation

Plain radium, any amount.....	\$16.00 per milligram
Radium-neutron sources.....	\$17.00 per milligram

The increased price charged by the Canadian Radium and Uranium Corporation for radium-neutron sources was due to the greater hazard in preparing such items.

As of January 1, 1947, 18 purchase orders and 6 contracts had been issued to Joseph A. Kelly, Agent for the Radium Chemical Company, and 15 purchase orders and 6 contracts had been issued to Boris Pregel of the Canadian Radium and Uranium Corporation for the purchase of radium.

In some cases, it was desirable to rent radium and radium-neutron sources, which were leased from both of the previously mentioned companies at the rate of \$250.00 per gram per month. This price is equivalent to 20% of the value of the radium per year. As of 1 January 1947, 7 purchase orders and 4 contracts had been issued to Joseph A. Kelly, Agent, for the Radium Chemical Company, and 2 purchase orders and 3

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contracts had been issued to Boris Pregel of the Canadian Radium and Uranium Corporation for the rental of radium. However, as of January 1, 1947, there remained in effect only 1 purchase order for 100 milligrams with Mr. Kelly and 1 contract for 200 milligrams with Mr. Pregel at this increased price.

Three contracts and one purchase order have been issued to the Radium Chemical Company for the rental of radium.

One contract with the Eldorado Mining and Refining Company was issued in May 1946 to cover the rental of radium from that company.

These contracts and purchase orders for the purchase and rental of radium are listed in App. F6.

b. Radioactive Lead. - Radioactive lead was procured from two main sources: lead produced as a by-product in the Canadian refining operations (App. E19), and lead produced as a by-product in refining sludges derived from African ores. The Canadian by-product was the largest source of supply and was purchased at a price of \$0.636 per pound of lead element. That derived from the second source was procured at a price of \$1.00 per pound of lead element. A summary of the contracts for the purchase of radioactive lead is listed in App. F5.

Part C

REFINING AND TREATMENT

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SECTION 7 - REFINING OF RAW ORES TO BLACK OXIDE  
AND SODA SALT

7-1. Operations. - In general, the refining operations (App. C2) consisted of a mechanical crushing and grinding of the raw ores to a fine sandy material, the treatment of this sandy material with acid to extract the uranium content, the partial treatment of the extract with caustic and other chemicals to precipitate the majority of impurities; a further treatment with caustic and other chemicals to precipitate the uranium; and a final roasting or drying operation to produce the refined material as either black oxide (uranium oxide,  $U_3O_8$ ) or as soda salt (sodium uranate,  $Na_2U_2O_7$ ).

The process developed and used by the Vitro Manufacturing Company for their commercial business, and subsequently used in their contracts with the Manhattan District, had been designed to produce soda salt. The equipment and process were such that this plant operated most efficiently and economically on relatively high grade ores, that is, 50% or greater  $U_3O_8$  content. The Vitro refinery is designed to handle approximately 40 tons per month of gross ore input.

The process used by Eldorado Mining and Refining at its Port Hope plant was also designed for its commercial business and produced black oxide. This process and equipment allowed efficient and economical operations on ore concentrates containing 20% or higher  $U_3O_8$  content. The Eldorado refinery is designed to handle a gross ore input of approximately 225 tons per month and, in addition, has associated facilities for the refining of radium from the uranium ores.

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To fulfill the need for a plant capable of economically and efficiently processing ores or concentrates containing less than 20%  $U_3O_8$ , the Linde Air Products Company refinery at Tonawanda, New York, was originally built and designed to handle American ore concentrates at an input of approximately 640 tons per month, but through subsequent process improvements it has been possible to process both American and African ore concentrates at much higher rates (150-220% designed capacity). Consideration of the type of ore, percent  $U_3O_8$  and other factors have been necessary in determining in which refinery the several ore concentrates should be refined.

The American ores were in reality tailings from previous vanadium refinery operations and were of such low uranium content that it was necessary to concentrate them in successive stages at or near the mine where they were produced, because of the excessive transportation charges which would otherwise have been required in transporting very low-grade ores to Linde's refinery at Tonawanda, New York. Accordingly, it was necessary for the Government to provide preliminary concentrating plants at Uravan and Durango, Colorado, and a central refinery to remove vanadium at Grand Junction, Colorado (App. CIA and D4). The U. S. Vanadium Corporation owned one plant at Uravan, Colorado, which was also utilized in the preliminary concentration step.

#### 7-2. Refining by Eldorado Mining and Refining.

a. Operations. - The first material refined by Eldorado at Port Hope, Ontario, Canada, consisted of the initial 100 tons of 65% ( $U_3O_8$ ) ore procured from African Metals Corporation, as a trial lot to



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determine the efficiency of refining operations. These ores were delivered to Eldorado in November 1942 and refining was started a month later. At the time of the delivery of the ores, the plant was small and did not have a production capacity of more than 30 tons of black oxide per month. However, the plant was expanded at the expense of the contractor to a point where he was able to deliver 150 tons of black oxide per month when working on 65% African ore. Expansion was started late in 1942 and by February 1943 the capacity had been raised to 100 tons of black oxide per month. Refining operations have continued to date both on African ores supplied under refining contracts and on Canadian ores from which the black oxide produced was sold to the Government.

To 1 January 1947, Eldorado produced, from African ore, approximately 1,832 tons of  $U_3O_8$ . The total cost of the work performed under eight contracts was \$2,823,310, the cost of refining  $U_3O_8$  was \$2,528,560, and the average processing cost was approximately \$0.69 per pound of  $U_3O_8$  in black oxide. In addition to the African ores processed, approximately 847 tons of black oxide have been produced to date from Canadian ores. (See App. F7.)

b. Contractual Arrangements. - As explained in Part B, Section 3, no separate arrangements were necessary with regard to Canadian ores, inasmuch as the uranium was procured from Eldorado in the form of refined black oxide. Thus, as far as contractual arrangements were concerned, the refining of Canadian ores was integrated with the procurement contract. In the case of treatment of African ores, however, the refining contracts only were negotiated with

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Eldorado, who, at the outset of the refining operations on African ores, did not wish to be held accountable and responsible for U. S. Government property in their refinery. Consequently, under Contract W-7405 eng-6 and W-7405 eng-17, the uranium content of the ores was sold to Eldorado but the contracts required the contractor to resell the total quantity of recovered uranium content back to the Government at an increased price which included the refining charges. Under subsequent refining contracts, namely, Contracts W-7505 eng-264, W-7405 eng-281, W-7405 eng-318, W-26-021 eng-21, and W-26-021 eng-26, the ore was accepted for refining by Eldorado with a stipulation that all the uranium content recovered would be returned to the Government, and in each contract a minimum recovery guarantee was stipulated. Because of the importance of the material, close checks were maintained on operations in order to preserve the required secrecy of the work, to protect the heavy financial interest which the U. S. Government had in the materials involved, and to make certain that all materials recovered were returned to the U. S. Government. In addition to refining African and Canadian ores, Eldorado refined crude soda salts to black oxide under Contract W-7405 eng-20. Appendix F7 shows data concerning the refining contracts.

7-3. Refining by Vitro Manufacturing Company.

a. Operations. - The Vitro Manufacturing Company in its Cannonsburg, Pennsylvania, refinery, which was constructed originally by the Standard Chemical Company for processing carnotite ores to recover the radium-vanadium and uranium content, refined from African ores a total of approximately 623 tons of soda salt containing the equivalent

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of 600 tons of black oxide. The refining operations commenced in December 1943 and were conducted under Contracts W-7405 eng-21, W-7406 eng-22 and W-26-021 eng-16. In addition to its work on African ore, Vitro has carried out refining of uranium-vanadium sludges (purchased from Vanadium Corporation of America) under Contracts W-7405 eng-54 and W-26-021 eng-7. The high grade carnotite ore purchased from the Vitro Manufacturing Co., under contract W-26-021 eng-24, was also processed by the Vitro Company under that contract.

b. Contractual Arrangements - A total of six refining contracts were entered into with Vitro, and under these contracts 768 tons of  $U_3O_8$  as soda salt was refined to January 1, 1947, at an average  $U_3O_8$  recovery of approximately 97%. The total cost of the work performed by Vitro was approximately \$1,203,490 or an average processing cost of about \$0.78 per pound of  $U_3O_8$ , produced in the form of soda salt. Data concerning refining contracts is shown in Appendix F7.

7-4. Refining by Linde Air Products Company - Prior to the development of the project, Linde Air Products Company had been engaged in the small-scale production of commercially pure black oxide. The OSRD, through the University of Chicago and Stone & Webster Engineering Corporation, had made arrangements with Linde for the procurement of small quantities of black oxide and had also discussed the production of specially purified materials by other processes. Linde was selected to be one of the principal processing plants because of its existing association with the project, its know-how in processing uranium, and the inter-relationship between its parent company, the Union Carbide &

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Carbon Corporation, and both the U. S. Vanadium Corporation (one of the suppliers of American ore concentrates of uranium) and the Electro Metallurgical Company (whose experience in metallurgical fields appeared to be valuable to the project).

The Linde black oxide plant located at Tonawanda, New York, was originally designed and built for the processing of uranium-containing concentrates from American ore. These concentrates contained approximately 10% black oxide and 2% vanadium pentoxide ( $V_2O_5$ ). In the Fall of 1943 alterations were made to the plant to permit the processing of African ore concentrates containing from 6% to 10%  $U_3O_8$ . The product from this plant was black oxide of a grade somewhat superior to the normal commercial black oxide.

In the process used by Linde, the ore concentrate was slurried with sulfuric acid and water for about two hours. Soda ash was added to the acid slurry to precipitate most of the impurities. The slurry was filtered. The filter cake was washed with hot dilute sodium carbonate solution and discarded. The filtrate was treated with ferric and ferrous sulfate to precipitate the residual vanadium and phosphorus. The resultant slurry was again filtered. The cake was discarded. Sodium hydroxide was added to the carbonate liquors to precipitate the uranium as sodium uranate. The sodium uranate was leached in a weak acid solution with ammonium sulfate to form ammonium uranate, which was removed from the slurry by filtering and calcined to form the purified black oxide.

Under Contract No. W-7401 eng-14, Linde contracted to build and operate a plant for refining African and American ore concentrates to produce black oxide in a first step, for the further processing of the black oxide to produce brown oxide in a second step, and for the conversion of brown oxide into green salt in a third step. The brown oxide and green salt phases are discussed separately in Sections 8 and 9. The total construction cost was \$3,040,230 for all three steps, of which \$1,759,940 was for the refining step. The contract was negotiated on a cost-plus-a-fixed-fee basis, with the fixed fee to apply to operations only. Design of the Linde refining plant was based upon previous pilot plant studies and development work done by Linde. Construction of this plant was completed in July 1943 and the plant started into operation processing American ore concentrates (App. E28, E29). After certain starting-up troubles, the refining step operated at approximately 110% of its designed capacity of 52 tons of black oxide per month until December 1943. In December 1943, operations were changed to consume 10% and 6% African ore. During the period December 1943 through November 1944, consuming African ore the plant operated at 162% of its designed capacity of 52 tons of black oxide per month. In the Spring of 1944, operations were at a rate of 225% of designed capacity. After November 1944, at the request of Madison Square Area, Linde made alterations in the plant and changes in the method of processing in order to increase the extraction of the uranium content from the ore. These improvements resulted in an increase of extraction from 93% to 98% of the ore content. Because of the increase in chemical consumption and the additional operations necessary to secure this

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higher extraction, the unit operating cost in this step increased from approximately \$.80 to \$.85 per pound of black oxide. However, this unit cost would have increased considerably more if other developments in plant operations had not enabled large increases in output and efficiency. In December 1944, operations were changed back to American ore concentrates, and because of developments made during the period of operation on African ore concentrate, operating rates of approximately 150% of the original designed capacity were maintained through January 1946 when all of the available American ore concentrates were consumed. In February 1946 the Contractor began processing 3% African ore. Approximately 10,250 tons of ore were processed during the period February 1946 to (July 18) 1946 at approximately 110% of the rated capacity of the plant. The plant was shut down 18 July 1946 <sup>because of</sup> ~~due to~~ insufficient suitable raw material and has been maintained in stand-by since then. Total production to 1 July 1946 was 2,428 tons of black oxide, at a total operating expenditure of approximately \$5,074,260, including material furnished by the Government.

Construction of a high-grade ore refinery, at Mallinckrodt Chemical Works in St. Louis, was started in May 1945 and completed in May 1946. At the end of the year this plant was just beginning to reach quantity production.

7-5. Concentrating of American Ores by U. S. Vanadium Corporation.

The sources of uranium within the United States consisted of carnotite ores, ordinarily containing less than about 2% of uranium, and sand tailings from previous operations for the production of vanadium. Most of the uranium available was in the form of sand tailings which contained from one-quarter to one-half of 1% of black oxide and approximately the same quantities of vanadium. Since it would have been very expensive to transport such a low grade material from its source in the

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Colorado, New Mexico and Utah area to the refineries near Buffalo, New York, it was obviously desirable to make arrangements for the preliminary processing of these materials in plants near the sources. With this in mind, initial arrangements were made with the U. S. Vanadium Corporation (a subsidiary of Union Carbide & Carbon Corporation) to produce in a U. S. V. plant in Uravan, Colorado, a concentrate containing approximately 15% black oxide from a large quantity of sand tailings which were the property of U.S.V. The output of this plant, totaling 150 tons of  $U_3O_8$  in concentrates, was delivered directly to Linde (App. C1).

Concurrently with this processing, arrangements were made with U.S.V. to build and operate plants at Durango, Colorado, and Uravan, Colorado, for the preliminary concentration of the uranium content of the sands\* by a process involving leaching with sulphuric acid and subsequent precipitation with caustic, filtration, and drying of a concentrate called green sludge\*, containing 5% to 10% black oxide. Since the acid leaching process also removed the residual vanadium content from the sand, it was necessary to process further the concentrate from the Durango and Uravan plants, to remove the majority of the vanadium content and recover it in usable form as a by-product. The plant for this operation was located at Grand Junction, Colorado, to which point the Durango and Uravan green sludge was brought by truck, and from which point a concentrate known as yellow sludge\*, containing 10% to 15%  $U_3O_8$  and 2%  $V_2O_5$ , was shipped by rail to the Linde refinery at Tonawanda, New York. The Grand Junction process involved, essentially,

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roasting of the green sludge with soda ash to make the vanadium soluble in water, leaching of the roasted materials with water to remove the vanadium, and, finally, the drying of the leached sludge to produce the final product, yellow sludge. In accessory operations, the vanadium dissolved from the green sludge was recovered as red cake,\* which was then converted into a salable form of fused vanadium pentoxide ( $V_2O_5$ ).

Construction and operation of the Government plants at Durango, Uravan, and Grand Junction <sup>WERE</sup> started in March 1943 by the U. S. Vanadium Corporation under cost-plus-fixed-fee contract W-7405 eng-32. Construction cost of these plants was approximately \$1,591,580 and operating cost was approximately \$203,000 per month, with a fixed fee of \$6,000 per month, starting from the beginning of operations in September 1943. The designed capacity of these plants were as follows: Uravan, 300 tons of sand tailings per day; Durango, 100 tons of sand tailings per day; Grand Junction, capacity sufficient to handle the concentrates from 600 tons of sand tailings per day.

Contract W-7405 eng-201, which covered the production by U.S.V. of the ore concentrate from its own mill at Uravan, Colorado, had been established with certain unit prices, namely, \$1.10 per pound of contained  $U_3O_8$  and \$0.30 per pound of contained  $V_2O_5$  in the product. However, after the contract had been in operation for a sufficient length of time to produce approximately 150 tons of  $U_3O_8$  in the concentrates, it was stated by U.S.V. that because of a decrease in the country's need for vanadium, continued production of large quantities of vanadium pentoxide would not be warranted. The U. S. Vanadium



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Corporation requested suspension of Contract W-7405 eng-201 in order to avoid a financial loss, but they were willing to enter into a cost-no-fee contract for the production of a uranium product only, from sand tailings. This was to the advantage of the Government since it provided Government supervision and an increased recovery of uranium from available domestic sources. The alternative would have been for the Government to hold U.S.V. to Contract W-7405 eng-201, in which case U.S.V. would have been required to operate in such a way as to minimize their financial loss. This alternative would have resulted in a depletion of supplies and a serious reduction in the useful life of the Government-owned Uravan plant because of lack of raw materials to be processed. It was also found that this method of operation would lose irretrievably about 95 tons of  $U_3O_8$  which would be recovered if the U.S.V. Uravan plant operations continued under conditions of maximum recovery rather than of minimum cost. With those considerations in view, it was decided that it would be in the best interests of the Government to suspend operation of Contract W-7405 eng-201 and, by supplementary agreement to cost-plus-fixed-fee Contract W-7405 eng-32, to take up the operation of the U.S.V. Uravan plant on a cost-no-fee basis, and have it operated to secure maximum recoveries of uranium. This change-over was made effective January 15, 1944 by supplemental agreement to both contracts (App. E22-E27).

From the beginning of operations to <sup>^</sup>January 1, 1947, a total of 831 tons of  $U_3O_8$  in yellow sludge was produced by the U.S. Vanadium Corporation, at an operating expenditure of approximately \$4,871,140.

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There is not included in the above cost the credit value of 924 tons of vanadium pentoxide ( $V_2O_5$ ), produced as a by-product at the Grand Junction plant. This material has been transferred to the Treasury Department to be retained as a reserve of a critical material.

Operations of the plants in the Colorado Area are summarized by the graphs in Appendix D4. It will be noted that the unit costs of the Durango operation and the U.S.V. Uravan operation have risen rather than decreased. In the case of the Durango plant, the reason for this rise has been threefold: (a) decreased rate of availability of feed stocks, (b) decreased percentages of uranium in the feed stocks, and (c) increased chemical consumption which was caused by changes in the nature of the feed stocks.

In the case of the U.S.V. Uravan operations, the reason for the higher costs after the shutdown has been the change from unit price operation to cost-plus-fixed-fee operation under such conditions as to secure maximum recovery of uranium contained in the feed materials, as discussed above. The variation in cost from the beginning of operations in mid 1944 to the latter part of 1945 results largely from changes in rate of plant operations, under conditions where a large part of the total costs are indirect and do not vary with production.

In connection with operations in the Colorado Area, a considerable quantity of research and development work was done, some of which resulted in improvements in processes and reduction in operating costs. Processes were developed and engineering estimates and designs have been made which demonstrate that, if future operations are undertaken on uranium-containing materials of the type found in

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the Colorado Area, new plants using improved processes can be constructed and operated at reduced costs.

Credits for the majority of the ideas studied, upon which developments were based, is due various members and associate research laboratories of the Army groups. The research and development of these ideas ~~was~~ <sup>were</sup> carried out by the U.S.V. laboratories in the Colorado Area, in collaboration with Government-operated laboratories in the same area.

7-6. Concentrating of American Ores by Vanadium Corporation of America -

The Vanadium Corporation of America, under supply contracts (See Part B, Section 4), processed in its own refineries in the Colorado area at Monticello and Haturita various ores from which were produced black oxide concentrates, containing approximately 45% black oxide and 25% vanadium pentoxide, which were then delivered to the Vitro Manufacturing Company for further refining.

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SECTION 8 - PRODUCTION OF BROWN OXIDE AND  
ORANGE OXIDE

8-1. Operations - In the summer of 1948 the Mallinckrodt Chemical Works, in cooperation with the OSRD and the University of Chicago, had developed a process for the manufacture of uranium compounds of high purity. This process was based upon the extraction, with ether, of the uranium from its impure solution in nitric acid, under such conditions that the impurities remained in the solution. The purified uranium nitrate was then boiled down and de-nitrated by roasting to produce the orange oxide (uranium oxide,  $UO_3$ ), which was then reduced to the brown oxide (uranium dioxide,  $UO_2$ ) by heating in an atmosphere of hydrogen (App. C3). This process had never been used on a commercial scale and facilities for its use did not exist. Since it was the only known process which would produce material of the required purity, it became necessary to construct and put into operation, at the fastest possible rate, adequate facilities for the utilization of the process, to produce the required quantities of purified uranium compounds. Following the basic plan of providing three parallel chains of operation with certain accessory units, arrangements were made and negotiations were begun for the construction and operation of the required facilities. Since the Mallinckrodt Chemical Works had developed the process and had the know-how, they were chosen as a unit of one of the three chains. Linde Air Products Company, with know-how in the refining of uranium, and already associated with the project and well separated geographically from the Mallinckrodt plant, was chosen as a unit of the second chain. E. I. du Pont de Nemours & Company, with a plant located at Deepwater, N. J., across the Delaware River from Wilmington,

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Delaware, was associated with the project, through the OSRD, in the step of manufacturing the green salt, detailed in Section 9 hereof. Since this company had wide experience in the chemical field and the geographical location was satisfactory from the security standpoint, du Pont was selected as a unit of the third chain of operations. Processes at all plants were based upon the original Mallinckrodt design with only minor variations (App. 03).

8-2. Mallinckrodt Brown Oxide Plant.- The Mallinckrodt plant for the production of brown oxide is located at St. Louis, Mo. This plant, using either commercial black oxide or soda salt, produced a highly purified brown oxide by the use of an ether extraction process developed by Mallinckrodt, and described above, in paragraph <sup>8</sup> 8-1. In addition to the brown oxide, this plant also produced orange oxide and uranium nitrate hexahydrate ( $UO_2(NO_3)_2 \cdot 6H_2O$ ) as intermediate compounds.

Negotiations with Mallinckrodt in the fall of 1942 and early 1943 resulted in a decision whereby they would construct, as their own property and at their own expense, a plant designed to process 52 tons of black oxide or soda salt per month into brown oxide or the other compounds mentioned above.

The basic Contract W-7405 eng-1 called for the production of 150 tons of brown oxide or equivalent, at a unit price of \$1.56 per pound (App. 150). The rate of delivery was to be one ton per day, six days a week. This contract was made effective 20 July 1943, in order to include that material produced under letter contract with the Stone & Webster Engineering Corporation acting for the OSRD. Operations in the expanded plant were started early in 1943.

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Through various improvements in the process, the majority of which were developed by Mallinckrodt, the capacity of their plant has been increased from the designed rate of 52 tons of black oxide per month to a final capacity of approximately 165 tons per month, or more than 200% increase. At the same time, the improvements in operation and resultant economies permitted a reduction in unit price, from the basic contract rate of \$0.38 per pound on termination of Contract W-7405 eng-1 (App. D7).

After completion of deliveries called for under Supplement #2 of the contract, Mallinckrodt, in reviewing its costs, found that through various improvements put into effect since the quotation for this supplement was furnished, costs had been so much reduced that the price for the 400 tons of brown oxide covered by that supplement could be reduced from \$1.11 to \$0.70 per pound; and Mallinckrodt therefore made a voluntary refund for this quantity, totaling \$332,000 (App. E31).

Mallinckrodt continued to operate the Brown Oxide plant until May 1946, when operations under Contract No. W-7405 eng-1 were terminated and the plant was dismantled. From the start of operations until May 1946 Mallinckrodt produced approximately 4,190 tons of orange and brown oxides at a total processing cost of \$4,745,250 (App. F-8). In these operations, handling large quantities of uranium oxide, Mallinckrodt has accounted for 98.8% of the materials furnished to them.

As mentioned above, Mallinckrodt engaged in considerable research in developing and improving the other extraction process, which resulted in large increases in efficiency of operations, reduction in costs, and increases in the capacity of equipment. They also engaged in the development, with the

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Yale University Laboratory, of processes suitable for the direct and continuing extraction of high grade ores.

On the basis of this work, it was decided about May 1945 to construct a new continuous extraction plant to produce 200 tons per month of Brown oxide from high-grade pitchblende ores. A plant was designed by Mallinckrodt and construction was started by E. B. Badger & Sons Company on 15 June 1945 and completed on 15 June 1946 at a total cost of \$3,698,360. The cost of constructing this plant was paid for by the Government. Mallinckrodt has operated this plant since May 1946 for the Government on a unit price basis under Contract No. W-14-198 eng-8. The Refinery has operated below its rated capacity through most of the year as a result of certain operating difficulties and changes which were made in the plant. However, the rate of operations increased continuously since the start of operations until it reached 100 per month in December 1946. Since the start of operations through 31 December 1946, 507 tons of Brown and Orange oxide have been produced at an average cost of \$0.82 per pound.

8-3. du Pont Brown Oxide Plant. - The du Pont brown oxide plant is located at Deepwater Plant, New Jersey. At this plant are also located the du Pont Chambers Works activities which produce organic chemicals, dye stuffs, etc. The du Pont plant manufactures brown oxide, by the standard ether extraction process previously outlined, from uranium peroxide sludge, which is the product of the de Pont recovery plant (See paragraph 8-5), from black oxide and soda salt, and from small quantities of clean uranium oxide which is produced as dross in certain of the metal fabrication operations.

The du Pont brown oxide plant was constructed on a cost-plus-fixed-fee basis under Contract W-7412 eng-3, which includes also the plants for the

production of green salt and uranium metal. The total construction cost was \$1,050,000 (See App. FB), the majority of which is chargeable to the brown oxide portion of the equipment, which includes considerable special stainless steel equipment for the handling of nitric acid solutions.

The du Pont brown oxide plant, together with the additional steps mentioned, is operated on a cost-plus-fixed-fee basis. Operations of the brown oxide step commenced in early June 1943 and have proceeded without serious difficulty since that time. du Pont's total production of brown oxide has been 1,970 tons, at a cost of \$1,891,050 or an average processing cost of \$0.48 per pound for this step. The present cost of \$0.45 per pound is a distinct reduction, under earlier costs, which can be attributed to the development of improved operating methods and techniques on the part of the operators (App. <sup>and D7</sup> BB). Of the total quantity of brown oxide produced by du Pont approximately 1,370 tons, or 64% have been produced from various scrap and by-product materials which were fed either to the previous step (Recovery Plant) or directly to the brown oxide plant, depending upon the quality of the feed. It will be seen from this figure that the recovery operations were of major importance in assuring the meeting of project requirements with a minimum of new materials.

As a result of improved efficiencies in all operations, the quantities of scrap material which are recirculated to the du Pont plant have been significantly reduced.

8-4. Linde Brown Oxide Plant.— The Linde Air Products Company, located at Tonawanda, New York, constructed and operated a plant for the production, in three consecutive steps, of black oxide, brown oxide, and green salt. The process used followed in its main points that of the Mallinckrodt



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plant, involving ether extraction, de-nitration and reduction of the orange oxide with hydrogen to form brown oxide. In the hydrogen reduction step this plant's process differed slightly from the Mallinckrodt and du Pont plants in that continuous rotary kilns were used rather than the batch process at the other plants.

The Linde brown oxide plant was constructed as a part of contract W-7401 eng-14, which has been referred to in connection with Linde's ore refining operation in Section 7. The operation of this plant under Contract W-7401 eng-14 was on a cost-plus-fixed-fee basis. The fee for the operation of all steps was \$12,500 per month and the operating cost of the brown oxide step was \$456,470 from the time it was started in August 1943 until the time the plant was shut down and placed in standby condition in the spring of 1944. During this period, a total of approximately 300 tons of brown oxide was produced by Linde, at an average processing cost of \$0.75 per pound for this step. Significant reductions in operating costs were made so that at the time of shutdown the unit cost had been reduced to approximately \$0.40 per pound (App. D<sup>6</sup>).

A combination of circumstances resulted in the decision early in 1944 to place the Linde brown oxide plant in a standby condition. Reduced supplies of black oxide to all operations had resulted in an excess of brown oxide production capacity over supplies available as input to the brown oxide step, and a review was made of all operations to determine which one could be shut down. Mallinckrodt was processing approximately 110 tons of brown oxide per month compared with total brown oxide requirements of approximately 160 tons per month. If the Mallinckrodt operation were shut down, inadequate capacities would remain in the other two plants to supply requirements and to consume available supplies.

The duPont operations were ~~integrated~~ with their recovery plant operations which were producing a wet uranium peroxide sludge for feed to the duPont brown oxide plant, and considerable difficulty and expense would have been involved in preparing the peroxide in such form that it could be shipped to other brown oxide plants for further processing. In addition, the Union Carbide and Carbon Corporation, in connection with its work on production of nickel compounds for the K-25 project, had need for certain facilities similar to a portion of those in the Linde brown oxide plant (App. E32).

In view of the foregoing considerations, the decision was made to place the Linde brown oxide plant in stand-by condition and to use a part of its facilities in the K-25 work during that period. Since the completion of the work on nickel compounds described above, the balance of supply versus requirements and capacities has been such that there has been no need to reopen the Linde brown oxide plant. However, in order to be prepared for any emergency or disaster which might interrupt operations in other brown oxide plants, the Linde plant has been maintained in stand-by condition.

8-5. duPont Scrap Recovery Plant. - In the majority of chemical processing plants and metal fabricating plants, there is a production of by-products and scrap materials. This holds true also in uranium processing; in each of the various steps there are certain waste or scrap materials produced which contain uranium in significant quantities. The majority of this scrap comes from the metal production and fabrication operations required for the X-10 process (See Sec. 10). In the metal production operation, approximately 2% of the metal charged appears in the by-product slag from the magnesium reduction process and approximately 4% appears in drosses from the recasting process. In the extrusion process, in addition to the solid metal scrap,

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metal oxide, constituting approximately 1% of the metal extruded, is produced as scrap. In other operations of the uranium-processing chain, the scrap consists of residues, materials recovered from effluents, sumps and drainage systems, dust recovered from ventilating systems, and floor sweepings (App. E34).

In order to recover the uranium content of the various scrap materials and put it back into process, du Pont, at the request of the Manhattan District, developed a method for handling these scrap materials. The du Pont recovery plant at Deepwater Point, New Jersey, is adjacent to their brown oxide processing plant. The plant is adapted to the processing of nearly all types of scrap materials and produces from them a wet uranium peroxide ( $UO_4 \cdot 2H_2O$ ) sludge, which is suitable for charging directly into the brown oxide process in the same manner as if it were black oxide (App. C4).

The construction and operation of the plant for this recovery process was undertaken by du Pont under cost-plus-fixed-fee Contract W-7413 eng-28. The construction cost of this plant was approximately \$842,280. (See App. F-8).

The designed capacity of the plant was originally 130 tons of low value slag and scrap materials per month and 14.5 tons of high value dress material per month. The original process used involved the grinding and roasting of the scrap materials, then a treatment with sulphuric acid in a high temperature retort to drive off hydrofluoric acid from the fluoride contained in the scrap, followed by a treatment with lime, filtration to remove the bulk of the slag impurities as "gyp cake", and finally a precipitation of the uranium content of the solution by addition of hydrogen peroxide.

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The precipitate of uranium peroxide was removed by centrifuging, producing a wet cake which was charged to the brown oxide process.

Operation of the recovery plant began late in September 1943, some difficulty was encountered in obtaining a product of satisfactory low fluoride content (which is essential in the material charged to the brown oxide process) and in reaching designed consumption rates. Furthermore, it was found that the cost of operating the hydrofluoric acid removal step was considerably more than the value of the hydrofluoric acid produced (App. E33). During this period, a large backlog of scrap materials in dross had been accumulating, and, in May 1944, du Pont was requested to make the necessary changes and additions to provide for increasing the processing rate to 210 tons of low value materials per month and 36 tons of high value dross material per month, with such changes in methods of processing as would be necessary to take care of the fluoride situation. Before du Pont had developed a satisfactory method for fluoride removal, Yale University, acting under direction of Madison Square Area, developed a method, involving the use of aluminum sulphate, to prevent the fluoride from contaminating the precipitated peroxide. This method was adopted in the revised recovery plant and it was found possible to dispense entirely with the hydrofluoric acid removal system, which eliminated certain bottlenecks in the plant and allowed the production of high quality peroxide. The adoption of this method of operation in the recovery plant made it possible to increase consumption of dross and slag to a total of 250 tons per month, with practically no additional equipment except for a steam dryer to handle certain wet scrap material. The plant has operated satisfactorily since this period and has rapidly depleted the backlog of scrap material.

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A total of 5,486 tons of scrap material has been consumed by the recovery plant from the beginning of operations to 1 January 1947, and from these materials, approximately 982 tons of equivalent black oxide have been produced, at a total operating cost of approximately \$2,394,150, or an average processing cost of \$1.17 per pound for this operation. Since the direct cost of this operation is influenced primarily by the consumption of scrap, rather than by the output of peroxide and since, as the backlog of scrap materials has been consumed, the uranium content of available feeds has been progressively decreasing, the monthly unit cost based upon the peroxide output has recently been trending upward. Actually, however, the monthly total cost has shown a slight trend downward as operating methods and techniques have improved. The cost graph (App. D<sup>5</sup>) shows the effects of the various operating factors discussed above.

Through the use of this recovery plant, more than 98% of the uranium content of the scrap materials, which otherwise could not have been reprocessed, has been removed and put back into the processing circuit, where it will eventually be converted to usable feed materials.

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SECTION 9 - PRODUCTION OF GREEN SALT AND HEXAFLUORIDE

9-1. Operations. - At the time the Manhattan District took over the procurement and production of uranium-containing materials, the OSRD had made arrangements with duPont and Harshaw Chemical Company for the development of processes and the production of small quantities of green salt (uranium tetrafluoride,  $UF_4$ ). Processes previously available were long and complicated, expensive to operate and involved various wet operations. duPont and Harshaw had each put into small-scale operation dry processes (differing only from each other in detail) which involved heating brown oxide to a high temperature in an atmosphere of hydrofluoric acid, which converted the brown oxide to green salt in one simple dry operation, and the only additional step necessary was grinding the cake of green salt into a fine powder (App. C5). The process was therefore adopted for all plants required for the production of green salt. However, Harshaw has found it unnecessary to grind the green salt for conversion to the hexafluoride. Operations are summarized in the production and cost graph (App. D9 and 10).

The large-scale plant for manufacturing hexafluoride (uranium hexafluoride,  $UF_6$ ), which is the feed material for the K-25 process and for the S-50 process until it was shutdown in the fall of 1945, was operated by Harshaw. The process consists essentially of reacting green salt with fluorine to obtain the hexafluoride (App. C6). A summary of production and prices is shown in App. D11.

9-2. Mallinckrodt Green Salt Plant. - Mallinckrodt, in a building adjacent to its property at St. Louis, Mo., operated a plant for the production of green salt from brown oxide produced in previous Mallinckrodt operations, and used a process similar to that used by duPont.

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Graphite boxes with graphite shelves were used to hold the brown oxide while it was fluorinated within steel retorts at high temperature. The green salt produced is ground sufficiently fine for use in the next operation, that of processing green salt to metal. Mallinckrodt undertook construction of the plant under lump sum Contract W-7405 eng-13, which also covers construction of a plant for the manufacture of uranium metal. In order to accomplish this construction, additional building space was rented, from the St. Louis Sash and Door Works, adjacent to the Mallinckrodt factory. The plant was built at a total cost of \$632,400 for both the green salt and metal steps. Operations in the green salt plant were begun in April 1943 under Contract W-7405 eng-29, which provided also for the operation of the metal plant. Operations under both of these plants were on a unit price basis (See App. F8). Under the basic contract, production of 150 tons of green salt, at a unit price of \$0.97, was contracted for, with the brown oxide raw material to be furnished by the Government. Through several supplemental agreements, additional quantities of green salt have been ordered, at generally decreasing rates, the cost under the latest supplement being \$0.28 per pound. The total production to 1 January 1947 has been 2,926 tons of green salt, at a total processing cost of \$2,591,120, or an average processing cost of \$0.44 per pound for this step (App. D-10).

The large reductions in price obtained from Mallinckrodt in this operation are due to the adoption and installation of various improvements in the plant. During the whole period of operation, the Mallinckrodt green salt plant has produced uniformly high quality product, with but insignificant interruption in production of material.

9-3. duPont Green Salt Plant. - The manufacture of green salt had

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been carried out initially by duPont under OSRD auspices. The Manhattan District took over this work and incorporated it into Contract W-7412 eng-5, which was effective 29 July 1942 (See App. F8). Operation under this contract was on a small-scale production basis at first but, after other larger plants became available, continued operations under this contract were for experimental and developmental purposes. The contract was established on a unit price basis and under it approximately 108 tons of green salt were produced; the first 31 tons at a unit price of \$2.00 per pound and the balance at \$1.25 per pound, for a total cost of about \$320,000. Since all of the major green salt plants were operating satisfactorily in the fall of 1943 and since no major research work was in progress or contemplated which could then be carried out in the pilot plant, operation of this unit was suspended as of 19 October 1943.

As mentioned under the section on brown oxide, duPont, as an additional step in the chain of operations for the production of uranium metal, had incorporated under Contract W-7412 eng-3 a plant for large-scale manufacture of green salt from brown oxide produced in its initial operation. This plant was in the same building as the brown oxide and the metal plants, and was constructed under Contract W-7412 eng-3 on a cost-plus-fixed-fee basis. Production of green salt was begun about the middle of February 1943 and operations were designed to handle the full output of the previous step, that is, approximately 47 tons of brown oxide per month.

In the early summer of 1944, when the supply of raw materials began to be insufficient to satisfy the capacity of all plants, a study of quality, yield, and comparative operating costs at the several plants producing green salt indicated the advisability of suspending operations in the duPont green salt plant. The plant was therefore placed in stand-by condition and was so maintained in order to satisfy any emergency requirements.

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From the beginning of operations until the time of shutdown, the total cost of producing 608 tons of green salt was \$869,170, or an average processing cost of \$0.71 per pound in this step. Just previous to the suspension of operations, the average unit cost was slightly higher than unit prices at other green salt plants (App. D9).

9-4. Harshaw Green Salt Plant. - Prior to the formation of the Manhattan District the Harshaw Chemical Company at Cleveland, Ohio, under arrangements made with the OSRD and its agent Stone & Webster, was producing green salt on a small-scale basis (App. E35). When the Manhattan District undertook the work of uranium production in the latter part of 1942, arrangements were made with Harshaw for the continued production of green salt, using a process similar in principle to that used by the other manufacturers. The details of equipment in this process differed from the others in that the raw material, brown oxide, was placed in magnesium trays which were in turn placed inside of steel tubes lined with magnesium. Later nickel trays and steel tubes were adopted and this type is in use at the present time. The tubes are externally heated electrically and hydrofluoric acid is passed through the tubes to produce the green salt (App. E36). The production of green salt was carried out under Contract W-7405 eng-2 on a unit price basis, effective 1 September 1942 (App. F8). Expansion of the green salt plant was completed in early 1943 by Harshaw at its own expense and the expanded plant had a capacity of 25 tons of green salt per month. The total quantity of green salt produced, from the beginning of the contract until its completion on 21 July 1944, was approximately 469 tons at a cost of \$634,630. The processing cost for the first quantity produced was \$0.92 per pound for this step and this figure was gradually reduced to \$0.48 per pound on the last quantity produced (App. D10).

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Contract W-7405 eng-2 was allowed to expire as of 1 October 1944, in order that the facilities might be used as the first step of a two step process for production of uranium hexafluoride for the K-25 and S-50 processes and subsequent to September 1945 for K-25 alone.

The contract under which the green salt was made for this process was W-7405 eng-276, effective 5 January 1944. The present capacity of the facilities on 1 January 1947 is 60.5 tons per month. However, the manufacturing facilities for green salt were being expanded at contractor's expense as of 1 January 1947 to a capacity of approximately 81 tons per month. Construction was scheduled for completion and operations at the higher rate to commence on or about 15 January 1947. The unit price for the first quantity of material produced under W-7405 eng-276 was \$0.40 per pound. This has been gradually reduced to the current price per pound of \$0.33. 1,171 tons of green salt had been produced under this contract to 1 January 1947.

On 18 October 1944 an urgent request was received from Y-12 for the production of 40 tons of uranium tetrachloride ( $UCl_4$ ) at the earliest possible date, by a process which had been used in the laboratory but had never been used commercially. Since Harshaw had produced  $UCl_4$  on a laboratory scale, it was believed that the maximum speed could be secured in fulfilling the request by having Harshaw undertake the work. By 7 November 1944, only three weeks later, Harshaw began large-scale production of this material under Contract W-26-021 eng-4, and the 40 ton order was completed on approximately 1 January 1945 at an overall unit price of \$0.64 per pound. An additional 32 tons of uranium tetrachloride for Y-12 was produced from residues under Supplement No. 1 to this contract at a unit price of \$0.285. Total cost under this contract amounted to \$68,800.

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9-5. Linde Green Salt Plant. - Linde, in the third step of its chain of operations, produced green salt from the brown oxide produced in its second step. The plant for this work was constructed under Contract W-7401 eng-14 on a cost-plus-fixed-fee basis, and was operated under the same contract (App. F8). The construction cost of the green salt step was approximately \$518,180. The process used in the operation was almost identical to that used by Harshaw, with the exception that arrangements were made for the recovery of excess hydrofluoric acid and separation into high grade anhydrous hydrofluoric acid and low grade hydrous hydrofluoric acid. The high grade acid was recycled through the process, reducing the net requirement, and the low grade acid was sold to commercial users, thereby reducing the operating cost. The green salt operations began in October 1945, approximately two months behind the scheduled date. Early operating difficulties were few and operations quickly attained the necessary rate. Although the plant was designed to produce 52 tons of green salt per month, experience indicated that approximately 30% more than this quantity could be obtained. Operation of the Linde green salt step was concluded on July 1, 1946. The cost of continuing this operation would have been excessively high since this operation would have had to bear the total overhead and administrative costs of the refinery and brown oxide steps with which the green salt step was integrally associated. The refinery and brown oxide steps had been terminated under conditions and for reasons mentioned previously. From the beginning of operations to 1 July 1946, 2,060 tons of green salt have been produced, at a total processing cost of \$1,394,670, of which \$167,600 covered Government-furnished material, (App. B<sup>9</sup>7).

9-6. Harshaw Hexafluoride Plant. - The plant for manufacturing uranium

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hexafluoride, which was the process gas feed material for the K-25 and S-50 projects and subsequent to September 1945 for the K-25 project only is operated by Harshaw on their property known as the Brooklyn plant in Cleveland, Ohio, the same plant in which the green salt was manufactured. This plant produces hexafluoride by the reaction between green salt and elemental fluorine. The process consists of passing fluorine over trays containing green salt; the resultant hexafluoride volatilizes, is swept out of the reaction vessel, and is caught in cold traps. The crude material is purified by distillation to remove hydrofluoric acid (HF) and inert gases. It is then placed in suitable containers for shipment (App. C6).

Research work on the hexafluoride had been carried out by duPont under OSRD program and, when the Manhattan District assumed responsibility for production of the process gas, the work at duPont, which had resulted in development of a satisfactory method of production, was continued under Contract W-7412 eng-10 (later incorporated into Contract W-7412 eng-151) effective 12 February 1943 with duPont (See App. F8). In addition, it was decided to have Harshaw carry out further development work, which was done under Contract W-7405 eng-43, effective 18 March 1943, since they possessed an exceptional background of experience in processing inorganic fluorides and had done work on similar materials for the OSRD (App. E37, E38, E39).

The plant for full-scale production was constructed after extensive research and development work which included not only the design of the reactor equipment but also the design of a suitable fluorine cell.

The contract for the production of hexafluoride was awarded to Harshaw under Contract W-7405 eng-276, effective 5 January 1944, after competitive bids were received from Harshaw and duPont. Harshaw submitted

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a lower bid than duPont for the construction of the plant and installation of equipment (App. E40-E44).

Contract W-7405 eng-276 covers construction and operation of the plant, which was equipped at a cost of \$480,000 (the building for the plant was constructed at Harshaw's expense). Subsequent to the construction of the original plant, additional equipment was purchased and partially installed, at a cost of \$322,000, in anticipation of increased requirements for the K-25 and S-50 plants. Since this increase did not materialize, the expansion was cancelled when partially completed. However, in September 1946, it was decided that the plant would be expanded and on 1 January 1947 the work involved was well under way.

The contractor, Harshaw Chemical Company, offered a bid to do the necessary work at a cost of \$123,860 under Supplement 19, effective 26 September 1946. Production at the increased rate was scheduled for 1 February 1947. Production of hexafluoride was carried out on a unit price basis and the contract initially called for a plant capacity of 1.65 tons per day, to produce 87.5 tons of product at a rate of 5.5 tons per week during 1944; the cost of production from brown oxide was to be \$1.35 per pound, and from green salt \$0.95 per pound. In a supplement to the contract, dated 6 November 1944, plant capacity was increased to 2.25 tons per day, and in Supplement 19 to 3 tons per day.

To 1 January 1947, the plant produced a total of 1,615 tons of hexafluoride; current contract price per pound of producing hexafluoride from green salt is \$0.60. A summary of the production rates and unit prices for processing in this operation is shown in Appendix E11.

During the course of the operations, Harshaw, at the request of the Manhattan District, in the same equipment used for the hexafluoride,

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manufactured a special lot of about 2.5 tons of uranium oxyfluoride ( $UO_2F_8$ ) by fluorination of the orange oxide. Because of its physical activity and properties, this material was particularly difficult and unpleasant to handle; however, Harshaw processed it at the same cost in effect at that time for its regular green salt operation, that is \$0.48 per pound, under Contract W-7405 eng-2, at a total cost of \$2,270.

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SECTION 10 - PRODUCTION OF METAL

10-1. Operations - At the beginning of the Manhattan District metal manufacturing program, three processes were evaluated on a production basis. Iowa State College at Ames, Iowa, was using a process involving the reduction of green salt with calcium at high temperature. Metal Hydrides, Incorporated, at Beverly, Massachusetts, was using calcium hydride to reduce brown oxide; and Westinghouse Electric and Manufacturing Co. at Bloomfield, New Jersey, was dissolving green salt in calcium chloride and electrolyzing the solution to the metal. In each case, the metal obtained by the above described operations was later recast in induction-heated vacuum furnaces. Further development at Iowa State College demonstrated the feasibility of substituting magnesium for calcium in the reduction of green salt and this substitution greatly reduced the cost of preparing the metal by the reduction of green salt. This process was adopted in the major metal manufacturing plants then under construction (App. C7).

According to the original three-chain plan of operations, metal producing operations were located at the plants of Mallinckrodt du Pont, and Electro Metallurgical Company, the latter as part of the Linde chain. Iowa State College, which had begun research and development work on metal production under the OSRD, was intended to be used as a small-scale production and development plant. However, because the urgent need for metal, the demonstrated ability of the Iowa State College plant to produce, and the fact that construction of the major plants was not yet completed, expanded large-scale production was

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begun at Iowa State College in the Spring of 1943 and continued until the Fall of 1944.

In addition to the production of virgin metal, some of the plants making metal were also used to recast scrap metal returned from the various fabricators. Metal in the form of accurately turned slugs is required for the X-10 project. A process for recasting the metal turnings obtained from these machining operations was developed by Iowa State College in the Winter of 1944; recasting of current turnings continued at this installation until December 1945. Recasting of solid scrap has been, and is currently being, carried out at Metal Hydrides, Incorporated.

Production and cost data for the metal plants are shown in Appendix D9.

10-2. Mallinckrodt Metal Plant - The Mallinckrodt metal plant in St. Louis, Missouri, occupies the second floor of a building, the first floor of which is occupied by the green salt plant. The manufacturing process used in this plant is similar to that employed at the du Pont, Electromet, and Iowa State College metal plants, namely, the reduction of green salt with magnesium at elevated temperatures in a steel bomb (App. C7) lined with lime or dolomite. The fused metal ingot resulting from this reaction is cleaned and remelted in an induction-heated vacuum furnace and cast in the form of billets in graphite molds. The Mallinckrodt metal plant was constructed along with their green salt plant under Contract W-7405 eng-13. (App. F-8)

Operations have been carried out under Contract W-7405 eng-29 on a unit price basis. Metal operations under this contract were begun



in July 1943 and 1,364 tons of metal had been produced, to 1 January 1947, at a cost of \$2,773,750, and an average processing price of \$1.02 per pound for the metal step. The unit price as of 1 January 1947 was \$0.71 per pound (App. D12). The unit price agreed on at the time the original contract was signed was \$4.17 per pound for the first 90 tons of metal. Improvements in the process made by Iowa State College and Electromet during the period the plant was under construction, and improved operating efficiencies obtained by Mallinckrodt during the production of the first 90 tons, resulted in production of this amount at a lower cost than that originally calculated. Accordingly, Mallinckrodt made a voluntary refund to the Government of \$2.20 per pound, resulting in a net cost to the Government of \$1.97 per pound for the first 90 tons of metal processed in this step.

10-3. Electro Metallurgical Company Metal Plant. - The metal plant of the Electro Metallurgical Company is located at Niagara Falls, New York, on their plant property. The manufacturing process was similar to that previously described for Mallinckrodt. The plant was constructed and operated under cost-plus-fixed-fee Contract W-7405 eng-14 (App. E48).

The construction cost was approximately \$234,300 and the operating cost to 9 August 1946, the date when production operations were terminated approximately \$2,167,000, inclusive of research. Since August 1946 the plant was placed in stand-by condition at a cost of approximately \$6,500 and is being maintained in stand-by at a cost of approximately \$4,640 per month. During the period of plant scale operations from April 1943 to July 1946, 1,538 tons of virgin metal were produced at an average processing cost in this step of \$0.67 per

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pound. Operations at the Electro-Metallurgical metal plant were very satisfactory and the quality of the metal produced in this plant was equal to or better than that produced by any other processor. (App. E12.) Production operations were terminated at Electromet even though their bid for operation on a unit cost basis was \$0.70 per pound as compared with Mallinckrodt Chemical Works' bid of \$0.71 per pound. This decision was reached based on the following facts:

a. Electromet had insufficient capacity to produce the total metal requirements. This would have necessitated keeping the Mallinckrodt Chemical Works metal plant in operation and splitting the required production between the two plants. Under optimum distribution of production the overall metal costs would have been more than operating Mallinckrodt Chemical Works alone at their slightly higher unit cost.

b. The Mallinckrodt Chemical Works green salt plant was the only source of green salt (the Linde and du Pont plants were in stand-by for reasons stated previously) and the overall cost of producing green salt and metal was even more favorable for keeping only the Mallinckrodt Chemical Works plant in operation since under the existing setup the administration and technical supervision of the Mallinckrodt Chemical Works green salt and metal plants are the same, as the plants are located in the same building. Consequently, any decrease in the operating rate of the Mallinckrodt Chemical Works metal plant tended to increase the unit cost of the green salt. Consideration was given to taking either the Linde or the du Pont green salt operations out of stand-by for use with the Electromet metal operation but the net result was even more unfavorable on the basis of cost comparison with the course of action taken.

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10-4. du Pont Metal Plant. - The du Pont metal plant is located at Deepwater Point, New Jersey, on the property of the Chambers Works plant and is housed in the so-called "Blue Products Area", in a building which also houses the brown oxide and green salt plants. Construction and operation of all three of these plants were carried out under cost-plus-fixed-fee Contract W-7412 eng-3. The total cost of construction was \$1,050,000 (App. F8). Some difficulty was encountered in the operation of the du Pont metal plant; the yields obtained were never so high as those of Mallinckrodt, Electromet or Iowa State College and the average cost per pound of metal produced was also higher than the prices paid to the other producers who used the same process. Thus, when operating efficiencies and improved yields over those originally contemplated made it apparent that some of the metal production facilities could be shut down, it was decided in the summer of 1944 to suspend operations in the du Pont metal plant, which was done in August 1944 (App. E49). During the period of operations, 232 tons of metal were produced at an average processing cost of \$1.72 per pound for this step.

10-5. Iowa State College Metal Plant. - The Iowa State College metal plant is located on the Iowa State College campus in Ames, Iowa. The plant is housed in a frame building, which was available at the time work was begun and to which several additions have been made as operations progressed and work increased. The installation was originally intended for use in research and development on the metal manufacturing step, and work of this type was already in progress under OSRD when the Manhattan District took over operations under Contract W-7405 eng-7

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(See App. F8). Since Iowa State College is a non-profit institution, this contract contains a clause providing for an audit at the completion of the contract and the refund of any profit to the Government. Iowa State College was a pioneer in the development of the metal process and they did the original work on the thermite process, using calcium as the reducing agent. Subsequent developments proved that magnesium, which was much less expensive than calcium, could be satisfactorily used and this process was adopted for use in all of the metal making plants. Because of the demands for metal prior to the completion of the metal plants at Mallinckrodt, du Pont, and Electromet, operations at Iowa State College, which were started in the pilot plant in the fall of 1942, were expanded to a large-scale production basis in March 1943 and production was continued at a rate similar to that employed in the three other metal plants until November 1944, after which experimental research and development operations on the green salt reduction process were continued until 1 April 1945 (App. E5D). During the period of operations at this plant, 653 tons of metal were produced at an average processing cost of approximately \$1.55 per pound for this step, inclusive of the cost of research and development carried on prior to and simultaneously with production.

In September 1943, Iowa State College was requested to develop a process for casting metal turnings. The sum of thirty-five thousand dollars was allocated in the fall of 1943 for the design and construction of a building and the necessary equipment for this operation. The process which was developed included the degreasing and cleaning of the turnings, separation of fine particles of oxide and

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metal, drying, pressing into briquets and melting and casting in an induction-heated vacuum furnace to form billets (App. E61-E63). Casting of turnings was begun early in 1944 on a pilot plant scale and on 26 April 1944 the process was put into full-scale operation, and continued until 31 December 1945, at which time the contract was allowed to expire. Up to 1 January 1946, 319 tons of finished metal had been recast from turnings, at a cost of approximately \$0.80 per pound, including cost of research and development work. It will be noted from the graph (App. D<sup>13</sup>) that the cost of recasting metal turnings increased slightly in the early part of 1945. This is not an increase in actual operating costs but results from the increased proportion of overhead costs charged to this operation after large-scale virgin metal production was suspended.

10-6. Metal Hydrides Metal Plant - The Metal Hydrides metal plant is located at Beverly, Massachusetts. Production of metal was begun in 1941 under a contract with the National Bureau of Standards and an OSRD sub-contract with Columbia University. This was followed by another OSRD contract (OEM sr-335) for the production of metal on a cost-plus-fixed-fee basis, which was supplanted by the Manhattan District, cost-plus-fixed-fee Contract W-7405 eng-8 in November 1942 (App. F8). The process used at Metal Hydrides involved the reduction of brown oxide with calcium hydride. This reaction produced a metal powder which was subsequently recast in induction-heated vacuum furnaces to form billets. The metal powder was found to have a strong tendency to be spontaneously inflammable and this tendency constituted a hazard to operations and resulted in the loss of considerable metal

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when fires occurred. This process was more expensive than the thermite reduction process, using magnesium, and the quality of metal produced was also below the standard of that produced by the thermite process, because of the difficulty of preparing calcium hydride of the requisite purity. Accordingly, after extensive research was unable to make this process as promising as the thermite process, the metal making operations were discontinued on 31 August 1943 (App. E54, E55). During the course of virgin metal making operations, at Metal Hydrides, 41 tons of metal were produced, at an average processing cost for this operation of \$8.20 per pound.

In the meantime, a necessity had arisen for recasting scrap material derived from the fabrication of the billets into final form for the X-10 process. Additional casting capacity was available at Metal Hydrides and recasting of this scrap, along with small metal ingots produced at Westinghouse, was started in the Spring of 1943 (App. E56, E57). Recasting of Westinghouse material was discontinued in the fall of 1943, after the Westinghouse contract had been suspended. Recasting of scrap at Metal Hydrides was continued, and is presently continuing, at a rate of 28 tons of scrap consumed per month. 1,090 tons of finished metal had been manufactured by recasting as of 1 January 1947, at an average cost in this operation of approximately \$0.33 per pound. The current unit price for recasting at Metal Hydrides is \$0.29 to \$0.47 per pound, depending upon the type of scrap (App. D<sup>13</sup><sub>9</sub>). All Metal Hydrides operations for the Manhattan District have been carried out under Contract W-7405 eng-8, which was changed from the original cost-plus-fixed-fee basis to the unit price basis for recasting of scrap.

10-7. Westinghouse Metal Plant - The Westinghouse metal plant was located at Bloomfield, New Jersey. Metal had been produced at the Westinghouse plant in experimental quantities for a number of years previously, and research in small-scale operations was being carried out under OSRD contract (OEM sr-619) before the formation of the Manhattan District. The OSRD contract was superseded by the Manhattan District unit price Contract W-7407 eng-2 as of 1 August 1942 (See App. F8), which called for an increase in production from 135 pounds per day to 380 pounds per day by the end of 1942 (App. E58, E59). The Westinghouse process involved the solution of green salt (and later a mixture of green salt and brown oxide) in molten calcium chloride, followed by electrolysis to produce finely divided metal, which was pressed into briquets and recast into small ingots in induction-heated vacuum furnaces. Despite improvements made in this process, the quality of production and yields were relatively poor, losses of material were high, and costs were higher than those of competing producers. Accordingly, the Westinghouse contract was terminated as of 15 October 1943 (App. E60). During the period of operations, Westinghouse produced approximately 69 tons of metal, at a total processing cost of \$1,599,200 including the cost of research and development work, and additional plant facilities.

10-8. Brush Laboratories Metal Plant - While they were never a producing unit, the facilities of Brush Laboratories, Cleveland, Ohio, a subsidiary of Brush Beryllium Company, were used in research and development of a process for the production of metal from molten green salt by reduction with magnesium under a blanket of inert gas at

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atmospheric pressure (App. 845-847). This process was similar to that used by the Brush Beryllium Company in their manufacture of beryllium. Original work was started under an OSRD contract in August 1942, which was superseded by the Manhattan District cost-plus-fixed-fee contract #7405 eng-3 in October 1942 (App. 88). Work was stopped at the expiration date stated in the contract, 31 July 1943, since the quality of metal produced was not satisfactory and the yields were low. The total cost of this work was approximately \$85,000.



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SECTION 11 - THORIUM

11-1. Thorium Procurement. - In order to provide quantities of thorium salts to the research project at Iowa State College, the Madison Square Area entered into Contract No. 2-17-028 eng-33 on 21 January 1946 with the Lindsay Light and Chemical Company, West Chicago, Illinois, for the purchase of 9,120 pounds of thorium nitrate tetrahydrate (mantle grade) at a cost of \$1.80 per pound. The Lindsay Light and Chemical Company was chosen for this work because of its long experience with thorium and rare earth chemistry in the preparation of gas mantles and because it was the only source of thorium which was cleared by Army Security at the time. Deliveries were completed under this contract on 25 January 1946 and the contract was closed.

On 2 July 1946, the Madison Square Area was requested by the Research Division at Oak Ridge to procure more thorium nitrate for the research at Iowa State College. Contract No. W-17-028 eng-35 was drawn up with Lindsay Light and Chemical Company for the supply of 3,610 pounds of thorium nitrate tetrahydrate at \$1.80 per pound. Deliveries were completed under this basic contract by 15 August 1946. By the end of December 1946, five supplemental agreements had been added to the basic contract whereby Lindsay would provide additional quantities of thorium nitrate (mantle grade) at \$1.80 per pound, small quantities of thorium nitrate (atomic weight grade) at \$4.00 per pound, small quantities of thorium carbonate at \$7.00 per pound and small quantities of thorium oxide at \$9.00 per pound.

The function of the M.S.A. through all this period was merely that of a procurement office which had nothing to do with the research being done in thorium anywhere in the project.

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SECTION 12 - QUALITY CONTROL  
PART A - URANIUM

12-1. Operations. - At the time the feed materials program was initiated, it was recognized that the consideration of quality was equally as important as that of quantity in setting up a program. The goal toward which all efforts were directed was the production of the highest quality metal attainable, i.e., metal having what was known as a "total danger summation" of 0.1% or less. The "total danger summation" is a measurement of the weighted effect of all impurities on the efficiency of utilization of uranium metal in the X-10 process. It was recognized that to reach the established goal would require a much more comprehensive program of testing, both chemical and physical, than is ordinarily carried out in controlling the quality of material manufactured to the usual commercial standards. Accordingly, steps were taken to correlate the testing activities of the facilities taken over from OSRD and Stone & Webster and to expand those activities as demanded by the enlarged scope of the program.

The extensive analytical program which was organized (App. E61) provides for: routine process control testing in the laboratories at each of the individual production facilities; a system of central control laboratories for the analysis of composite samples of product from each producer and for research on methods of analysis; and an over-all physical test on brown oxide and a final physical test on finished metal for the X-10 process, to be conducted at the University of Chicago.

The magnitude of the analytical program, involving the determination of some 60 elements to a lower limit of detection of a

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few parts or fractions of a part per million was such as to preclude the possibility of carrying <sup>tests</sup> % out at each of the producers' laboratories. Therefore, it was decided to limit testing at the producer's laboratories to routine control analyses and to have more comprehensive analyses carried on at four central control laboratories located as follows: the chemical section of the Metallurgical Laboratory, University of Chicago; Princeton University, Princeton, N.J.; Massachusetts Institute of Technology, Cambridge, Mass.; and the National Bureau of Standards, Washington, D.C.

The functions of this central control laboratory group may be summarized as follows: (a) to carry out analyses as indicated in the Madison Square Area quality control program; (b) to improve existing methods and develop new methods of analysis; (c) to act as a "fire brigade", that is, to supply the special services needed in times of emergency: supplying, if need be, manpower and facilities to supplement the manufacturers' personnel and facilities; (d) to analyze special materials not covered in the quality control program; (e) to give advice and guidance on the analytical program in general (App. E62, E63).

Expenditures under the quality control program have totaled about \$826,930 to 1 January 1947.

12-2. University of Chicago. - There was no direct contract between the Madison Square Area office and the University of Chicago group for administrative reasons, and, on 1 June 1943 the latter asked to be relieved from routine duties in connection with the program because of the pressure of other work (App. E66). The group agreed, however, to continue to perform the routine physical testing. Arrangements were made for the group to cease routine chemical analyses as soon as the laboratory at Iowa State College was in a position to take care of their routine control

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testing. This was accomplished about the middle of July 1943, and, subsequent to this date, the activities of the Chicago group in the quality control program were limited to the physical testing mentioned above.

The physical test on brown oxide was transferred to the National Bureau of Standards on 9 October 1946 leaving the physical test on metal the only activity of the Chicago group in this program at this time (E-102).

12-3. Princeton University. - Analytical work on uranium and its compounds had been carried out by Princeton University under an OSRD contract as early as 1 February 1942. On 13 April 1943, the services of this small group were acquired by the Manhattan District under a lump sum Contract W-7405 eng-81 (See App. F8). Steps were immediately taken to expand facilities and personnel to cope with the demands which continued to be made on this group. In addition to carrying a heavy load of routine analysis on weekly composite samples, this group has carried out fundamental research both on analytical methods and on the physical chemistry of uranium and its compounds. Of particular interest are the development of an electrolytic-polarographic method of analysis and the preparation of a series of papers on the separation of uranium from impurities by ether extraction. Contract W-7405 eng-81 was completed on 15 April 1946 (E 103). The official ore assay at this time was taken over by the Lucius Pitkin Company under contract number W-35-058 eng-9, and the National Bureau of Standards assumed the remainder of the analytical program duties.

12-4. Massachusetts Institute of Technology. - In the fall of 1942, the group at M.I.T. was making spectrographic analyses under a sub-contract with Metal Hydrides. The group was organized as a central

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laboratory operating under Contract W-7405 eng-40, dated 1 April 1943. The work of this group was largely a spectrographic nature, and the unusually complete spectrographic equipment at M.I.T. was put to extensive use, not only in the routine control analysis of weekly composite samples from the producers, but also in the development of new procedures. Contract W-7405 eng-40 was completed on 30 April 1945, but M.I.T. continued with the spectrographic analysis of recast metal on a purchase order basis until 1 November 1946. This work was transferred to the Hanford Engineer Works on that date.

12-5. National Bureau of Standards. - Since early 1942, both Princeton University and National Bureau of Standards had been engaged, through OSRD, on analytical problems concerning uranium. Many of the early methods of analysis, both chemical and spectrographic, had been improved or developed in the Bureau's laboratory, and it was entirely fitting, therefore, that National Bureau of Standards should be included in the central control group.

The work at the National Bureau of Standards has been of both a chemical and spectrographic nature, and, in addition to performing routine control analyses, the burden of which has been carried at this location, the National Bureau of Standards has been active in the preparation of suitable primary standards for the use of the project as a whole. The Bureau has also been the principal  $U_3O_8$  assay laboratory, and improved analytical methods for these determinations have been developed at the Bureau and are widely used throughout the project (App. E64, E65). In addition to work of an analytical nature, including radium assays, this group has been active in research on methods of processing ores and concentrates, especially for the Colorado operations.

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PART II - THORIUM

12-6. National Bureau of Standards. - The only work done by Madison Square Area in regard to thorium quality control has been some development work on raw material assay by the National Bureau of Standards. They have also prepared a standard series of monazite samples.

12-7. Iowa State College. - The major portion of the analytical development work done up to 1 January 1947 has been done at Iowa State College in connection with their research program.

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SECTION 13 - ACCOUNTABILITY

13.1. Program. - The need for conserving and accounting for the extremely large tonnage of uranium-bearing materials owned by the Government was recognized early in the development of the feed materials program, both for maintaining close control on operations and for purposes of security. The development of a sound accounting program was therefore tied in directly with the control of operations.

The procedure used by the contractors in reporting to OSRD on uranium-bearing materials was superseded in January 1943 by the "Weekly Production Report" (App. E72) submitted by each major contractor who processed materials. This report contained detailed data as to inventories, receipts, consumption, production, and shipments of raw, in-process, finished, and by-product uranium-bearing materials and was used by Madison Square Area in planning, scheduling, and coordinating over-all operations at the contractors' plants.

In order to supplement the Weekly Production Report, a "Monthly Material Balance", instituted in May 1943, was submitted each month by all major contractors (App. E68). The Monthly Material Balance gave additional data required for accountability purposes, inasmuch as it took into account, in addition to the net "as is" weights shown on the Weekly Production Reports, the uranium content of the materials and over-all operational data concerning good material produced, scrap produced, and process losses, which latter are inherent in the complex chemical processing work being undertaken.

The contractors were advised, in July 1943, of standard procedures for the handling of by-product materials which would eventually

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be consumed in recovery operations. Prior to that time, in November 1942, they had been advised that no material of any kind was to be discarded without the approval of Madison Square Area (App. E67).

Periodic inspections of the contractors' plants were made by technical personnel to investigate sources of loss and, wherever possible, to make recommendations for their reduction or elimination. Standard procedures were also set up to establish policies for accurate weighing methods (to eliminate variations in reported weights of material shipped by one contractor and received by another), methods of assaying uranium content, and checking of contractors' assays at the central control laboratories. In addition, contractors were requested to take elaborate precautions in handling, processing, and protecting uranium-bearing materials in all forms, and they have been impressed continually with the need for maintaining accurate records to account for materials in their possession.

In September 1943, a standard shipping memorandum procedure was instituted (App. E71) which provided for the forwarding to Madison Square Area of copies of both shipping and receiving memoranda on all transfers of government-owned uranium-bearing materials into and out of plants under the jurisdiction of Madison Square Area and its sub-areas. The contractors' material balances have been carefully cross-checked against the shipping memoranda to see that all the materials shipped by each consignor have been received by the various consignees; and, in November 1944, detailed audits were undertaken at the contractors' plants, to scrutinize carefully procedures and accuracy of records, and to make a complete audit of their uranium-bearing material transactions. Thus, by means of the procedures and educational programs outlined



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above, it was possible to maintain a satisfactory control over the movements of uranium-bearing materials.

In February 1946, program of accountability was established for thorium and thorium-containing materials in Madison Square Area. This program was analogous to that maintained for uranium, but was augmented to include voluntary submission of material balances by the prime supplier of thorium chemicals, in anticipation of ultimate licensing under the Atomic Energy Act of 1946.

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MANHATTAN DISTRICT HISTORY

BOOK VII - FEED MATERIALS

APPENDIX "A"

GLOSSARY

Billets	Uranium metal cast into cylinders 4.25 inches in diameter and 13 to 21 inches long.
Carnotite	A vanadium-uranium mineral of canary yellow color occurring in the Colorado region.
C. I. F.	Cost, Insurance and Freight.
F. A. S.	Free alongside ship.
Green Salt	Green uranium tetrafluoride, $UF_4$ .
Green Sludge	A concentrate, obtained from carnotite ores, containing 5-10% black oxide, and 10-20% vanadium pentoxide.
Metal	Uranium metal.
Radium-neutron	A mixture of radium and beryllium to produce a neutron source.
Red Cake	Vanadium precipitate, recovered from ore refining operations, which is converted to a salable form of fused vanadium pentoxide $V_2O_5$ .
Rods	Uranium rods, obtained by extrusion or rolling of billets, 1.5 inches in diameter and 8 to 13 feet in length.
Sands	Tailings containing relatively low percentages of uranium oxide, $U_3O_8$ , obtained as by-products from vanadium refining operations.
Yellow Sludge	A concentrate obtained from carnotite ores containing 10-15% black oxide and 2% vanadium pentoxide.

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MANHATTAN DISTRICT HISTORY  
BOOK VII - FEED MATERIALS

APPENDIX "B"  
ORGANIZATION CHARTS

<u>No.</u>	<u>Description</u>
1	Materials section organization, 1 January 1943
2	Madison Square Area organization, 1 January 1944
3	Madison Square Area organization, 1 January 1945
4	Relationship between key contractors and Madison Square Area, 1 January 1945
5	Madison Square Area organization, 1 January 1946
6	Madison Square Area organization, 12 January 1947
7	Relationship between key contractors and Madison Square Area, 1 January 1947

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DISTRICT ENGINEER  
Col. J. C. Marshall

Lt. Col. K. Nichols, Deputy  
Maj. R. Blair, Exec. Off.

MATERIALS SECTION  
Major T. F. Crenshaw  
Major J. R. Ruhoff, Asst.

PROCUREMENT  
Capt. P. L. Merritt

PRODUCTION  
Capt. C. Hadlock

SPECIAL MATERIALS  
Lt. L. C. Burman

ADMINISTRATIVE SECTIONS  
Col. J. Harmon  
Maj. C. Vanden Bulck, Asst.

ORGANIZATION CHART  
(Showing Names of Section Heads)

MATERIALS SECTION

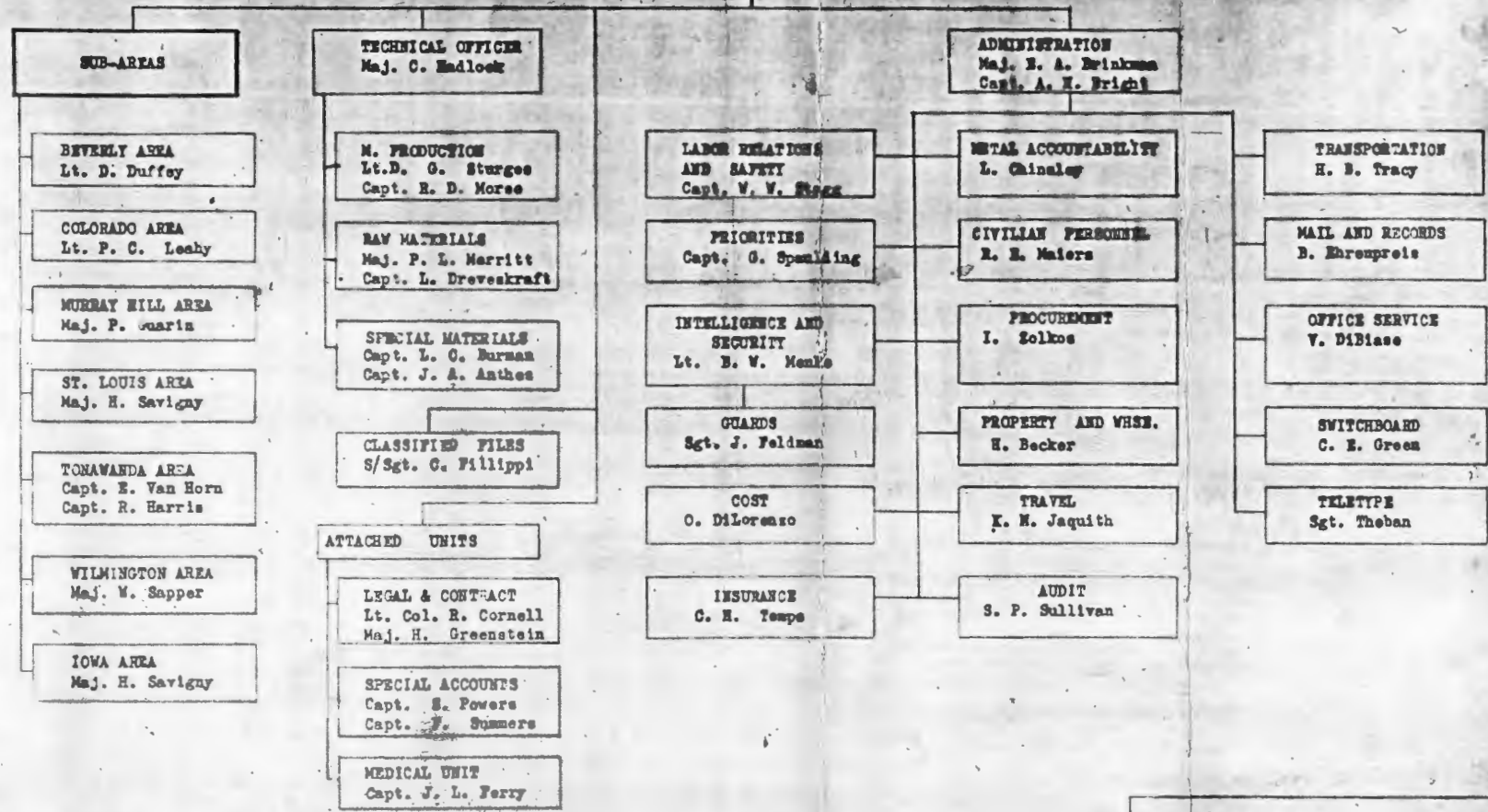
1 January 1943

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No. B 2

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AREA ENGINEER  
Lt. Col. J. R. Ruboff  
Maj. G. W. Russell (Exec. off.)



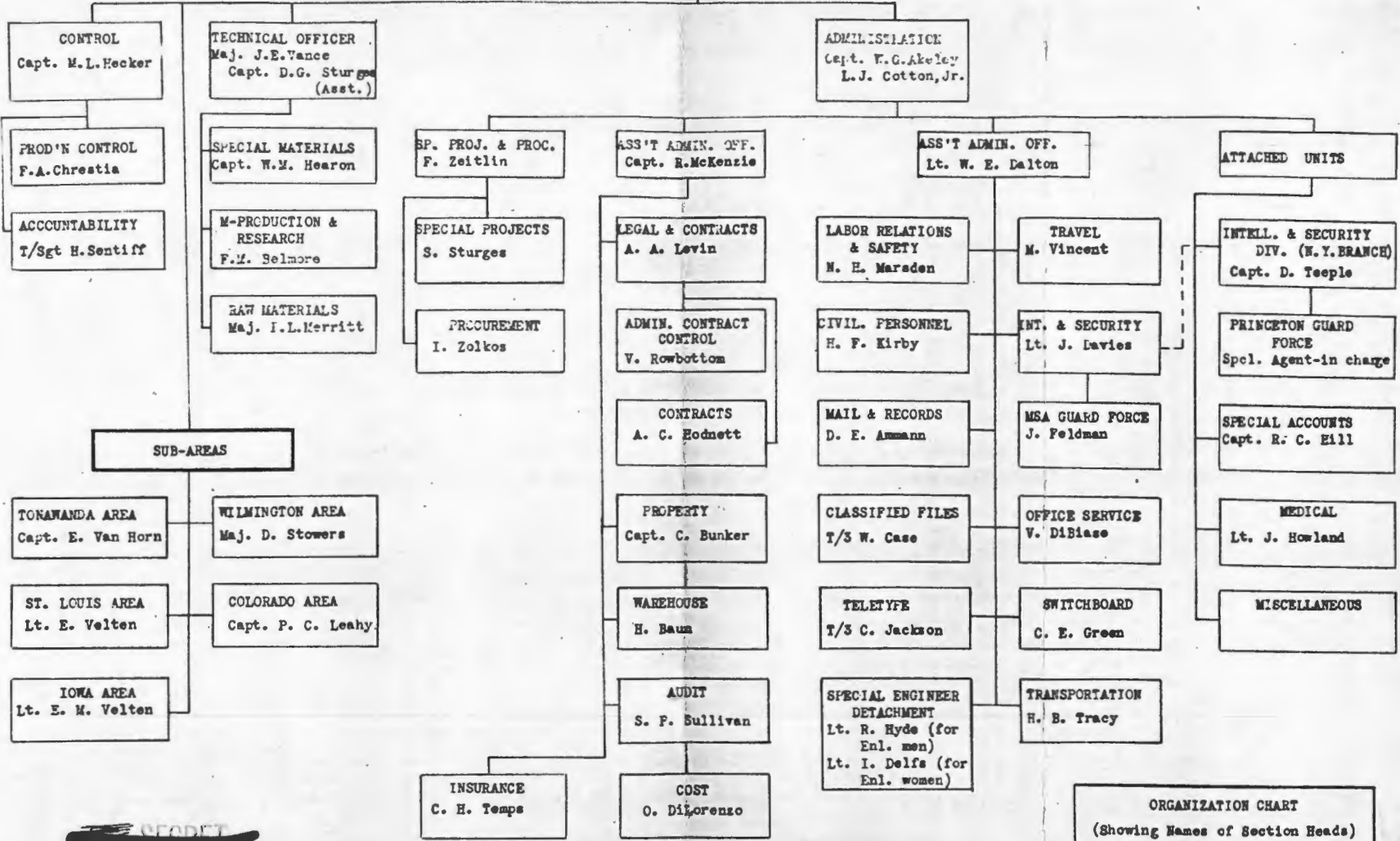
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ORGANIZATION CHART  
(Showing Names of Section Heads)  
MADISON SQUARE AREA  
1 January 1944

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AREA ENGINEER  
Major W. E. Kelley  
Maj. C. Hadlock, (Exec. Off.)

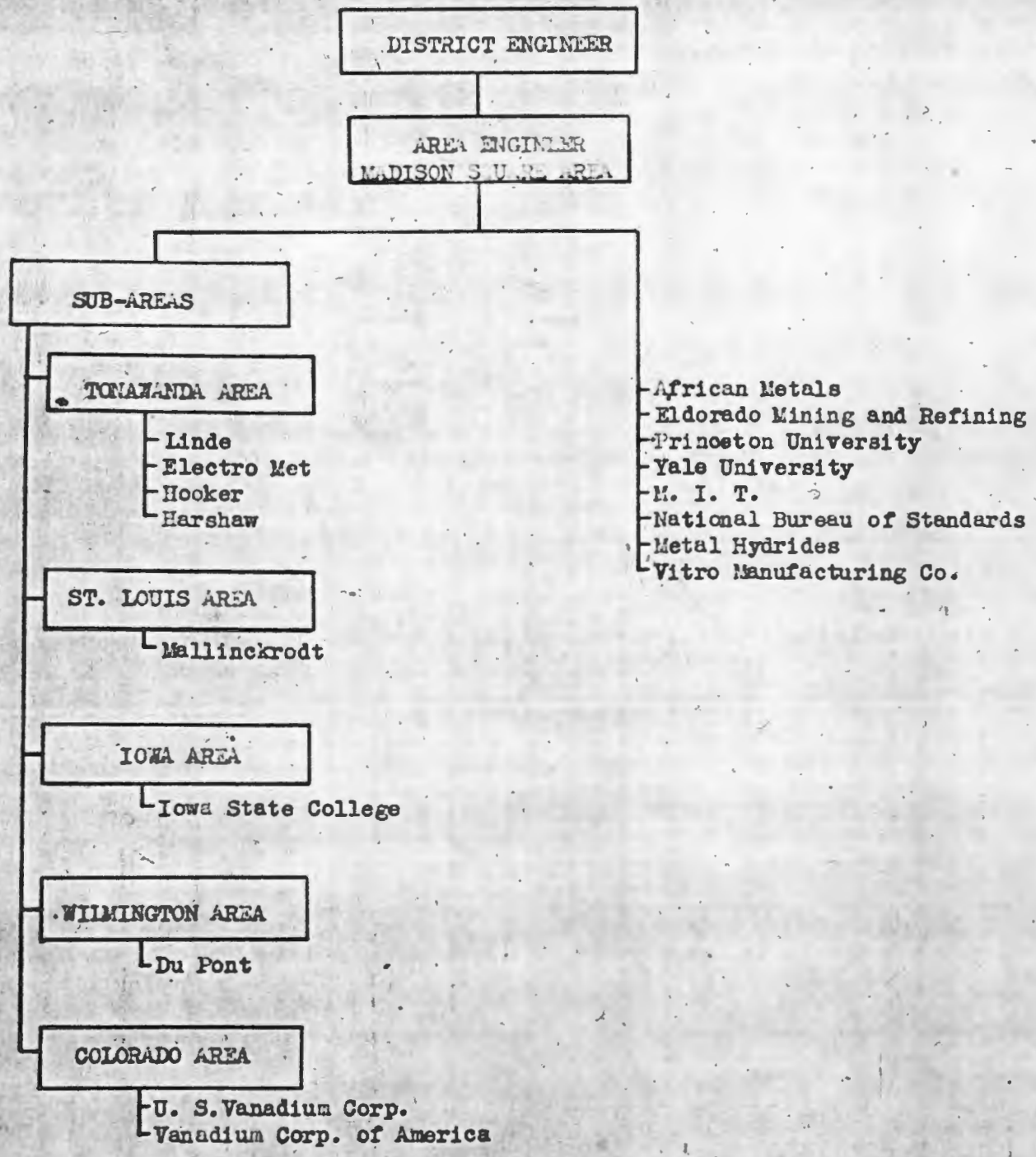


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ORGANIZATION CHART  
(Showing Names of Section Heads)  
MADISON SQUARE AREA  
1 January 1945

Relationship Between Key Contractors and  
Madison Square Area and Sub-Areas

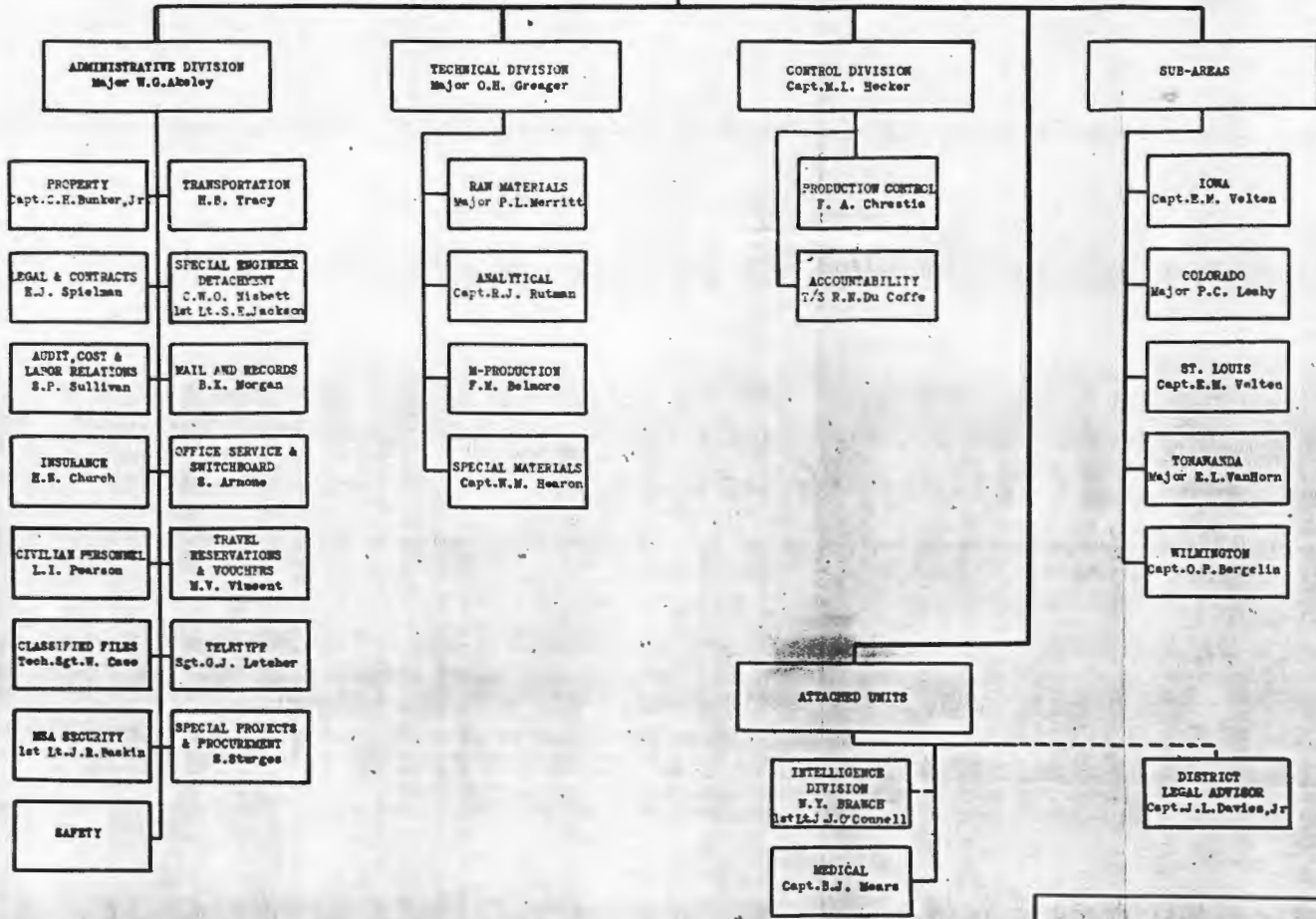
As of 1 January 1945



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Lt. Col. W.E. Melley  
Area Engineer  
Lt. Col. J.E. Vance  
Executive Officer

~~SECRET~~



ORGANIZATION CHART  
MANHATTAN DISTRICT  
MADISON SQUARE AREA  
January 1, 1946

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Colonel G.W. Deeler  
Acting Manager  
Lt. V.E. Lolley  
Deputy Manager

Lt. Col. R.J. Walsh, Jr.  
Executive Officer for  
Administration

Lt. Col. A.W. Oberbeck  
Executive Officer for  
Operations

U.S.C.S. Liaison  
(Washington)  
Lt. Col. G.B. Page

ADMINISTRATIVE DIVISION  
J.S. Spider

RAW MATERIALS DIVISION  
P.L. Merritt

PRODUCTION & ACCOUNTABILITY  
DIVISION  
F.M. Belmore

RESEARCH DIVISION  
J.H. Hayner

INTELLIGENCE & SECURITY  
DIVISION  
P.W. Kirkman

AUDIT BRANCH  
J.F. McKee

TRAVEL BRANCH  
M. V. Vincent

EXPLORATION BRANCH  
G.C. Selfridge

RECORDS & MISC.  
SOURCE MAT'L'S BR.  
B.V. Pietyga

URANIUM-THORIUM  
BRANCH  
E.M. Yelton

SPECIAL MATERIALS  
BRANCH  
DeK. Hunter

RESEARCH BRANCH  
G.L. Koenig

PUBLICATIONS BRANCH  
C. Glasser

NSA GUARD FORCE  
D.J. Moses  
(New York, N.Y.)

MIDDLESEX  
PATROL FORCE  
C. Montross  
(Middlesex, N.J.)

CORRES. BRANCH  
V.D. Boschetti

SAFETY BRANCH  
R.A.N. Turner, Jr.

MIDDLESEX BRANCH  
F.J. Giaccolo

PLUTONIUM BRANCH  
S.B. Boboff

ANALYSIS & DEVEL-  
OPMENT BRANCH  
G.W. Toelken

SPECIAL PROJECTS  
BRANCH  
H. Anderson

GEN'L. ADMINISTRA-  
TIVE BRANCH  
R.A. Joseph

PROPERTY BRANCH  
F.B. Conley

LEGAL BRANCH  
M.S. Lokietz

REDISTRIBUTION  
SALVAGE & CON-  
TRACT CLEARANCE  
F. Bertino

CIVILIAN  
PERSONNEL BRANCH  
L.I. Pearson

SPECIAL ASSIG-  
NMENTS BRANCH  
1st Lt. J. Pedenius

PROCUREMENT BRANCH  
I.Z. Cornell

SUB-AREAS

SUB-OFFICES

ST. LOUIS  
D. Duffey

BROOKHAVEN  
E. L. Van Horn

BOSTON

ROCHESTER  
R.S. Pearson

CLEVELAND  
W.A. Traussig

TOHAMANDA  
F. J. Epp

WILMINGTON  
G. L. Ryan

COLORADO  
G.W. Hunter

ATTACHED UNITS

MEDICAL DIVISION  
Capt. R.S. Wolf

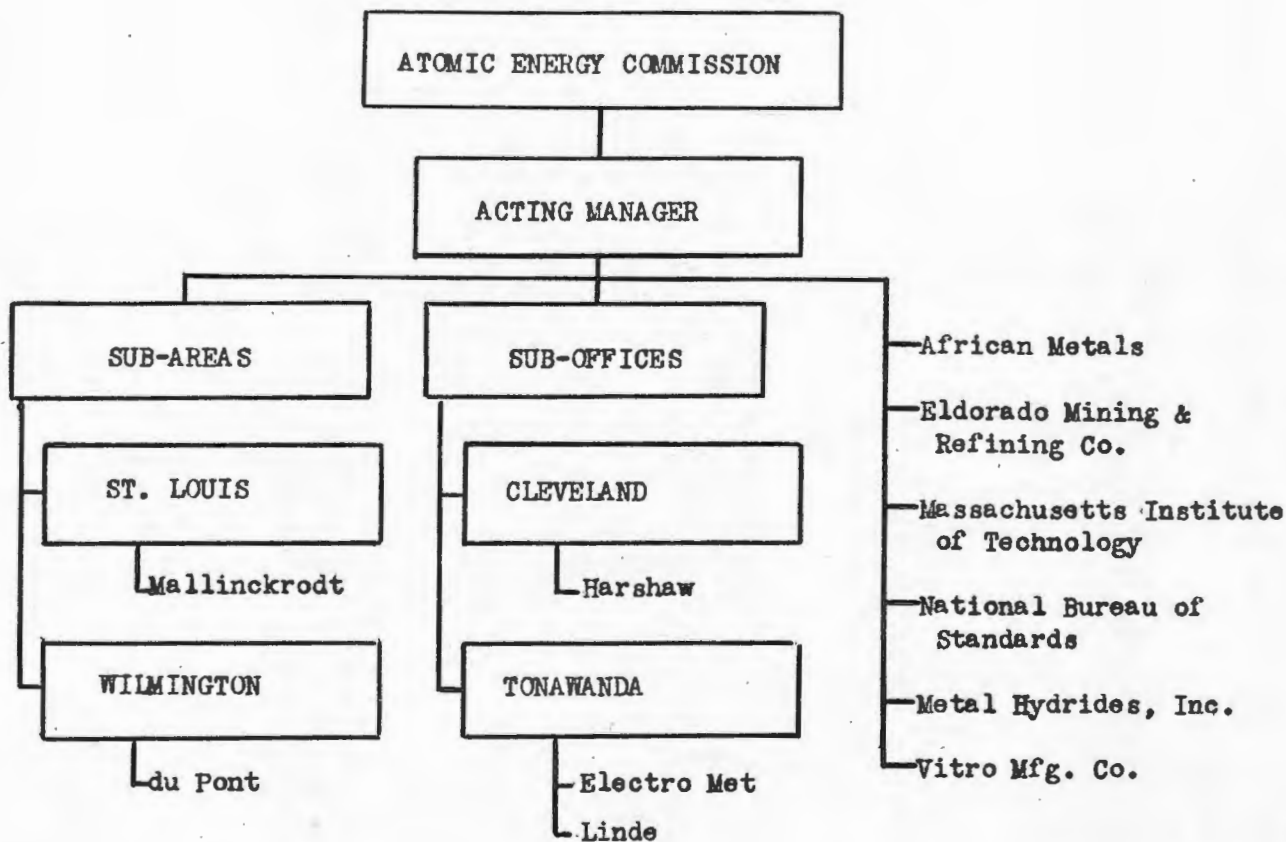
PATENT DIVISION

ORGANIZATION CHART  
ATOMIC ENERGY COMMISSION  
MADISON SQUARE AREA  
JANUARY 12, 1947

~~SECRET~~

RELATIONSHIP BETWEEN KEY CONTRACTORS AND  
MADISON SQUARE AREA, SUB-AREAS, AND SUB-OFFICES

AS OF JANUARY 1, 1947



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MANHATTAN DISTRICT HISTORY

BOOK VII - FEED MATERIALS

APPENDIX "C"

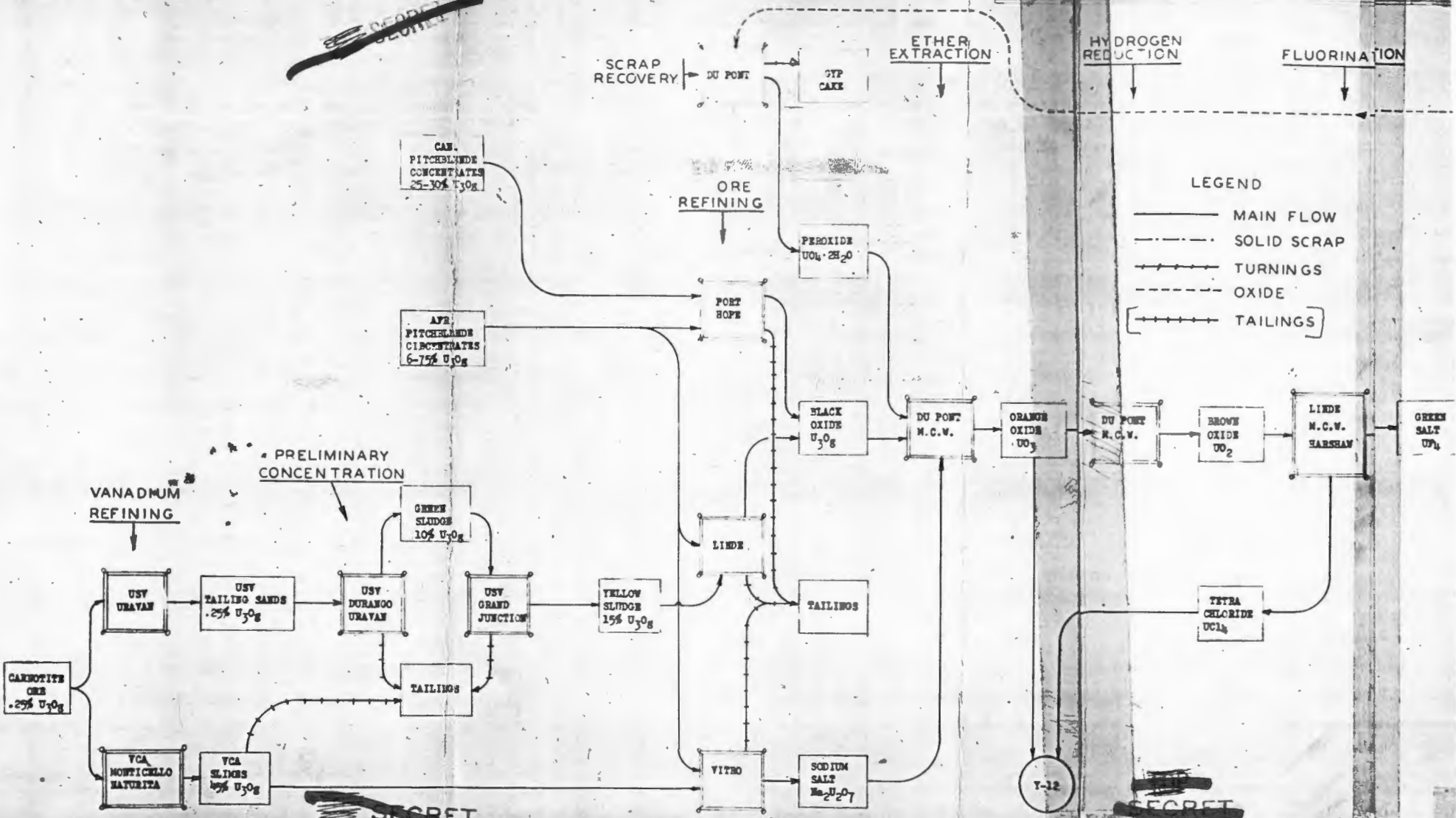
FLOW DIAGRAMS

<u>No.</u>	<u>Description</u>
1 A	Uranium production (1 January 1945)
11 B	Uranium production (1 January 1947)
2	Black oxide operation
3	Brown oxide operation
4	Recovery operation
5	Green salt operation
6	Hexafluoride operation
7	Metal operation

~~TOP SECRET~~

# URANIUM PRODUCTION FLOW DIAGRAM

~~SECRET~~



- LEGEND**
- MAIN FLOW
  - - - - - SOLID SCRAP
  - TURNINGS
  - - - - - OXIDE
  - [—————] TAILINGS

~~SECRET~~

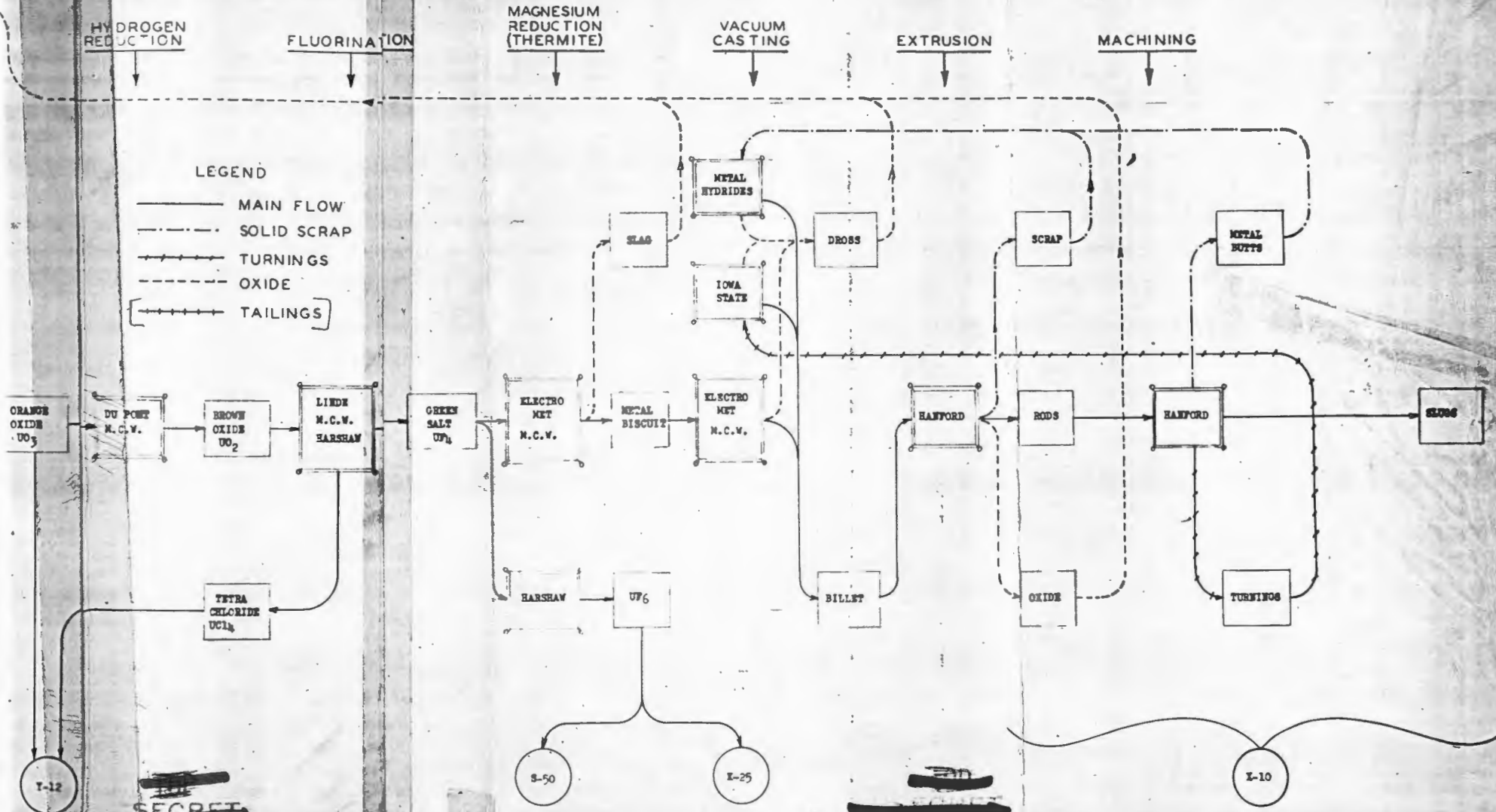
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Y-12

~~SECRET~~

~~SECRET~~

# URANIUM PRODUCTION FLOW DIAGRAM (1 JAN. 1945)



T-12

S-50

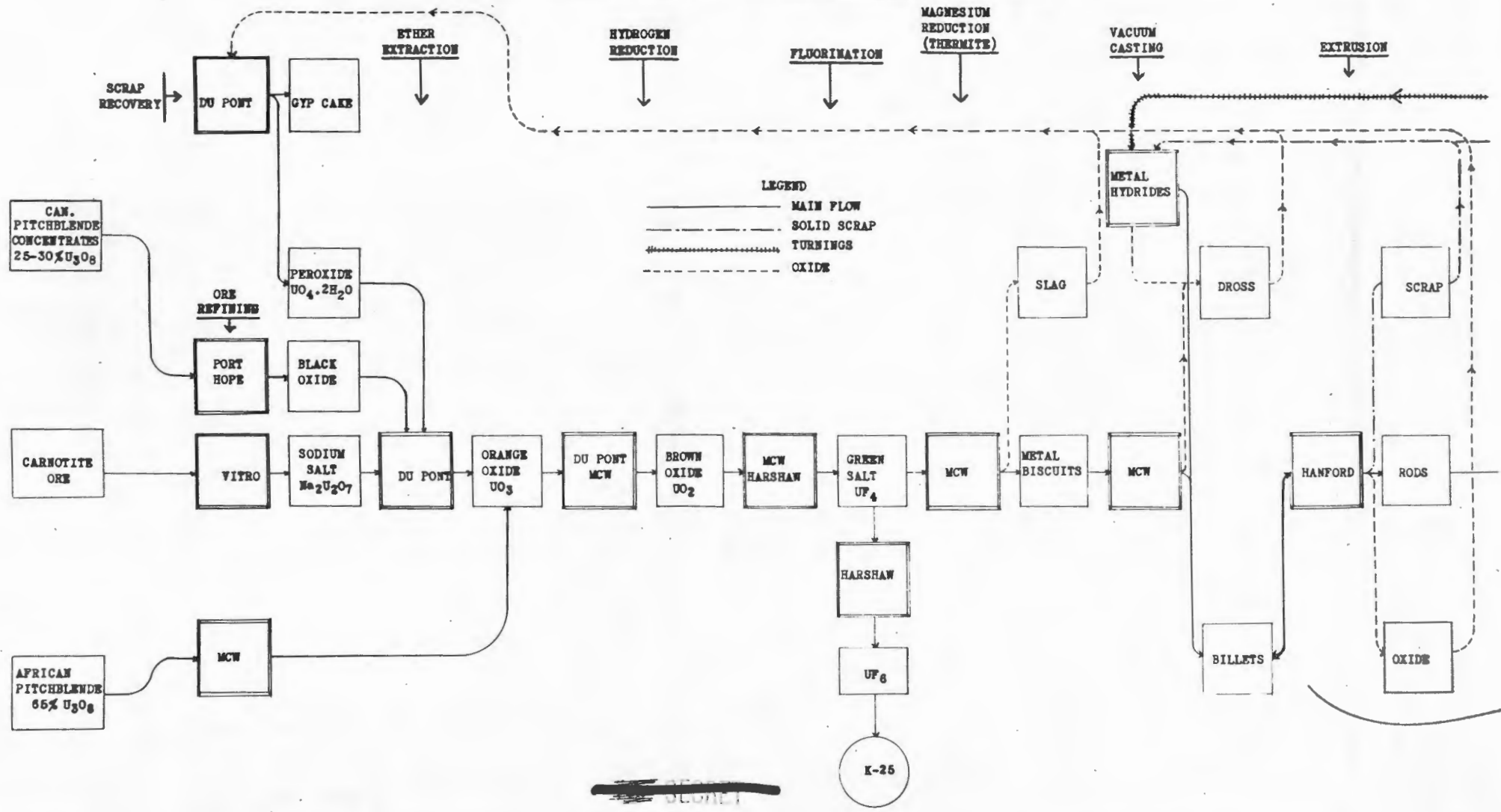
K-25

X-10

~~SECRET~~

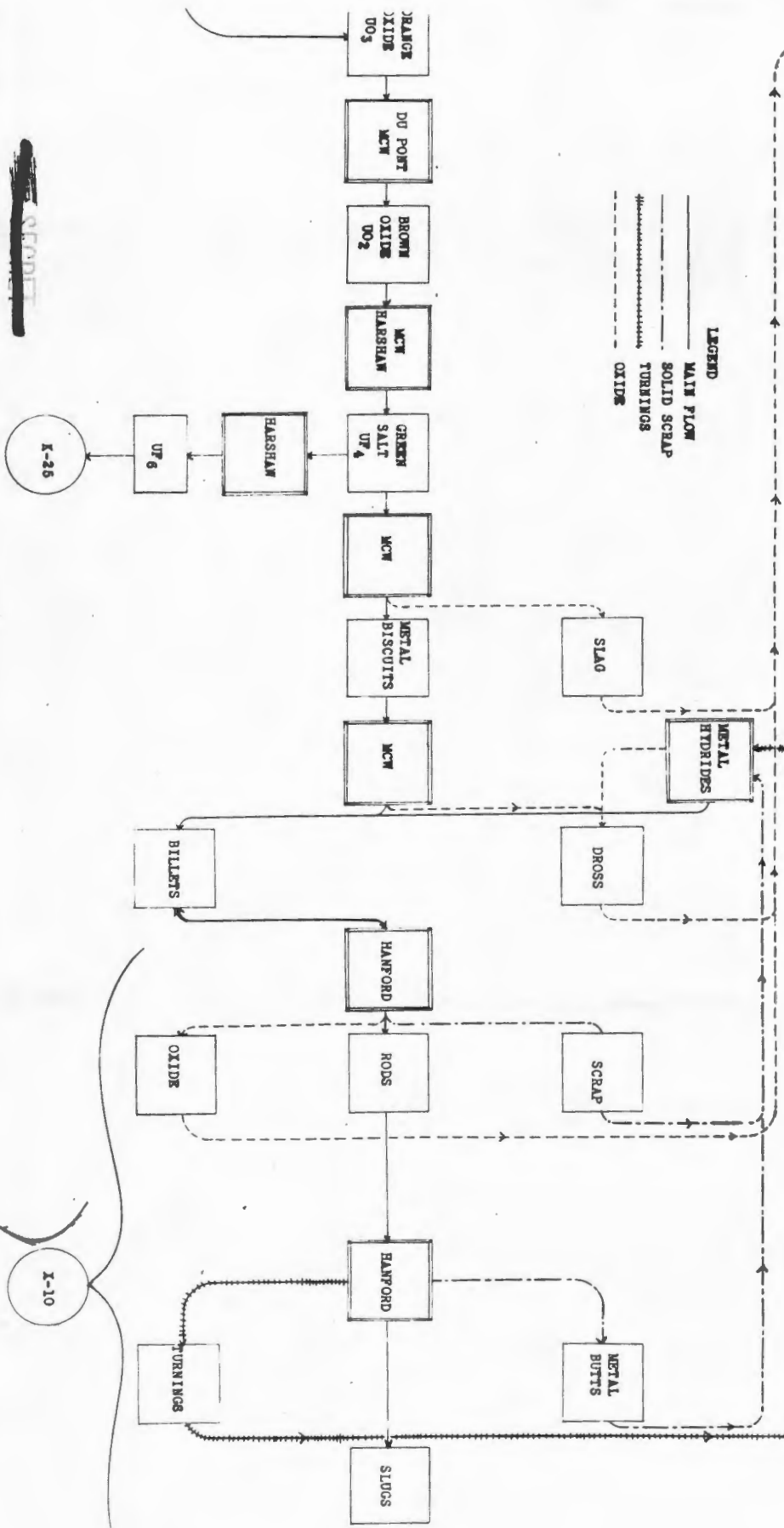
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~~SECRET~~  
**URANIUM PRODUCTION FLOW DIAGRAM (1 JANUARY 1947)**





~~SECRET~~  
 HIGH PRODUCTION FLOW DIAGRAM (1 JANUARY 1947)



~~SECRET~~

K-25

K-10

No. 10

~~SECRET~~

No. 2

ORE CONCENTRATE REFINING PROCESS

ORE CONCENTRATE

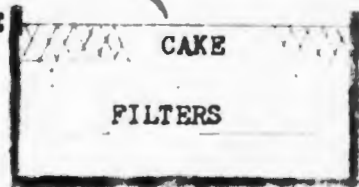
SULPHURIC ACID,  
THEN CARBONATE  
OF SODA



DIGEST  
TANKS

STEAM

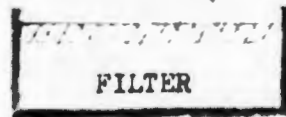
FILTER CAKE  
TO DUMP



CAUSTIC SODA,  
THEN AMMONIUM  
SULFATE

LIQUOR

PRECIPITATION  
TANKS



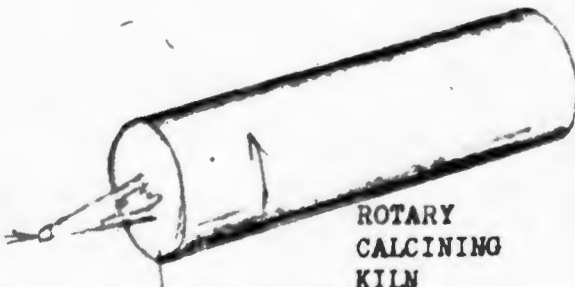
FILTER

AMMONIUM  
SULFATE FUMES

FILTER  
CAKE,  
AMMONIA  
SALT

LIQUOR  
TO  
SEWER

GAS  
BURNER



ROTARY  
CALCINING  
KILN

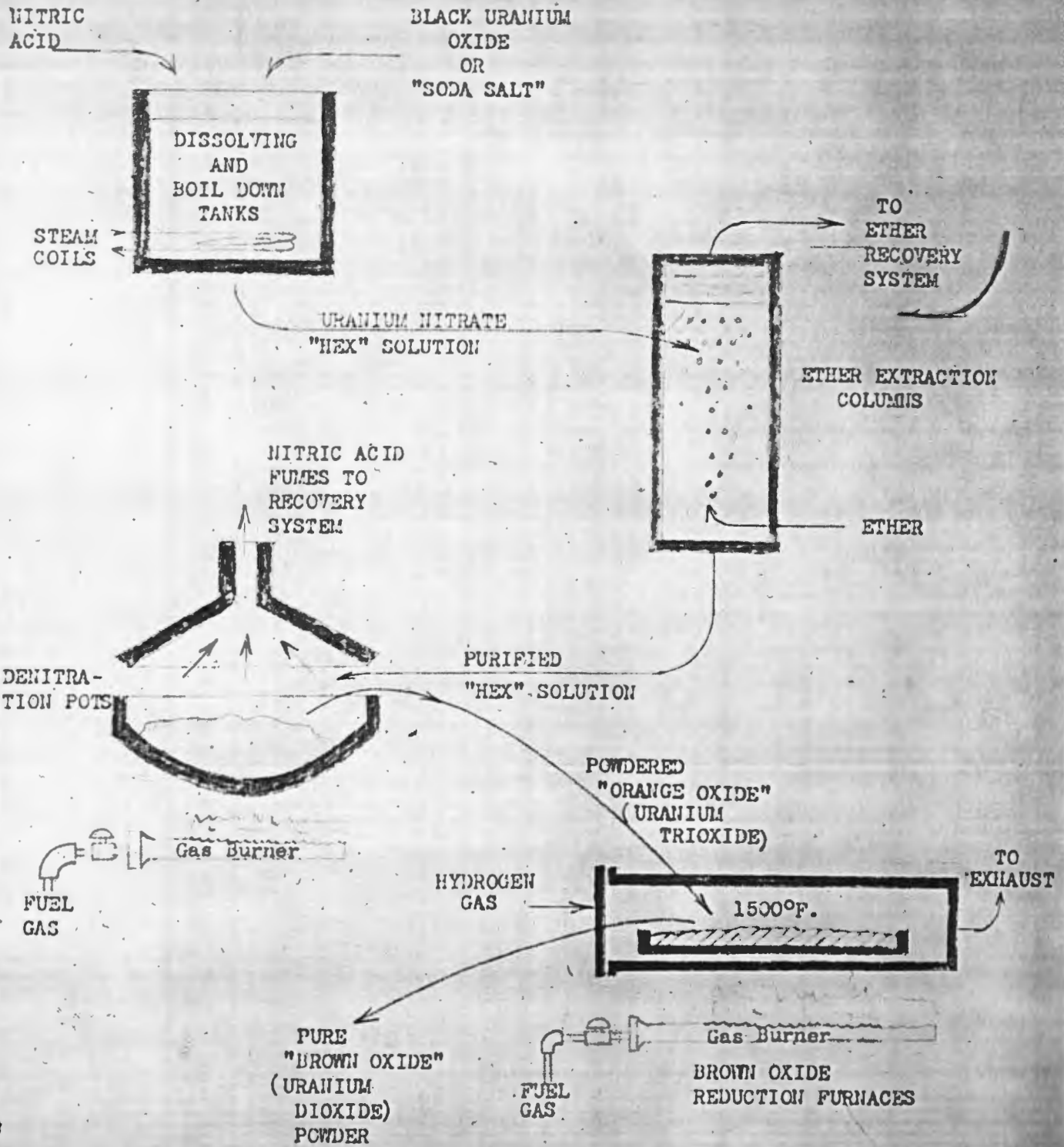


FINISHED  
BLACK  
OXIDE,  $U_3O_8$

~~SECRET~~

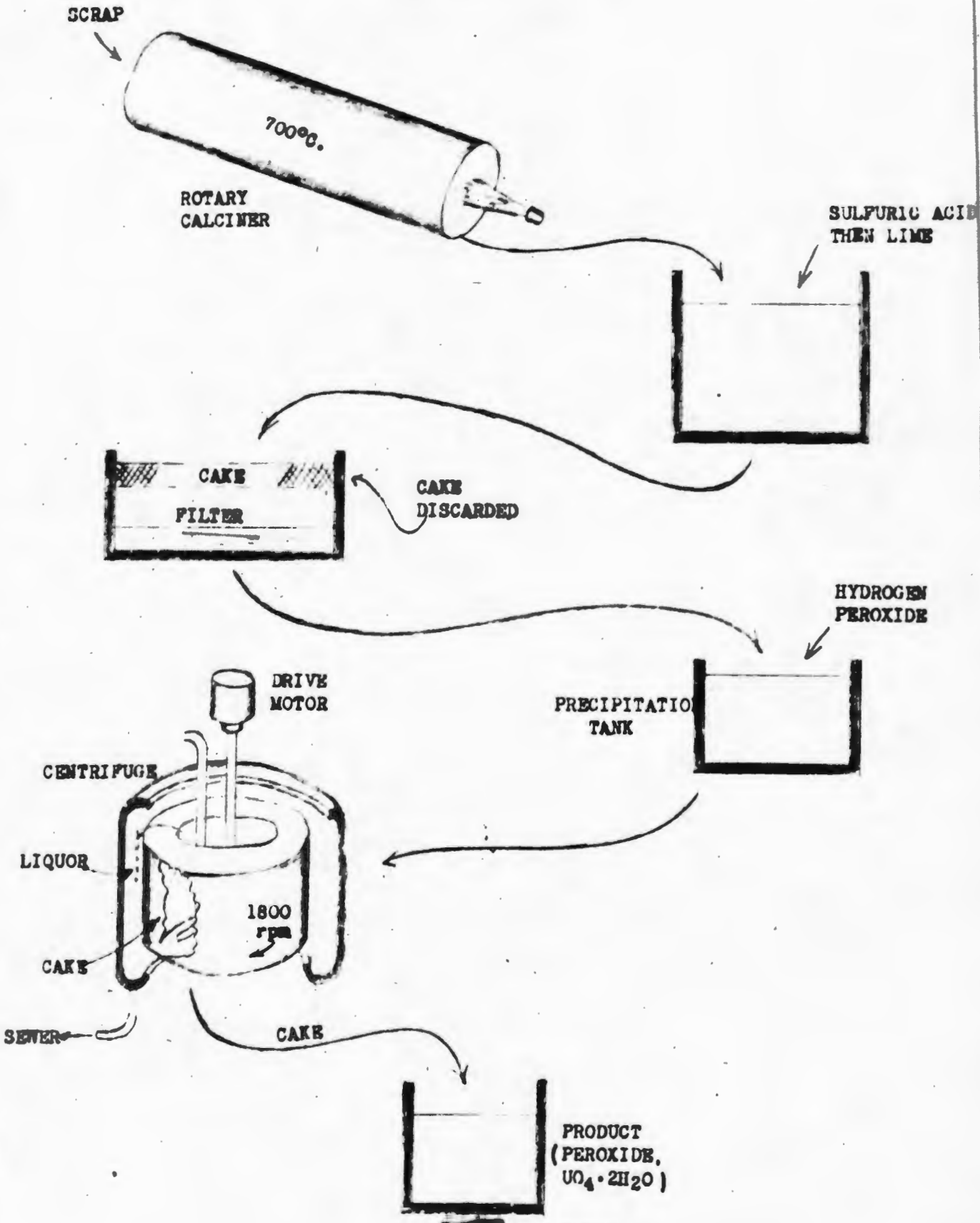


"BROWN OXIDE" PROCESS



RECOVERY PROCESS

~~SECRET~~ No. 4



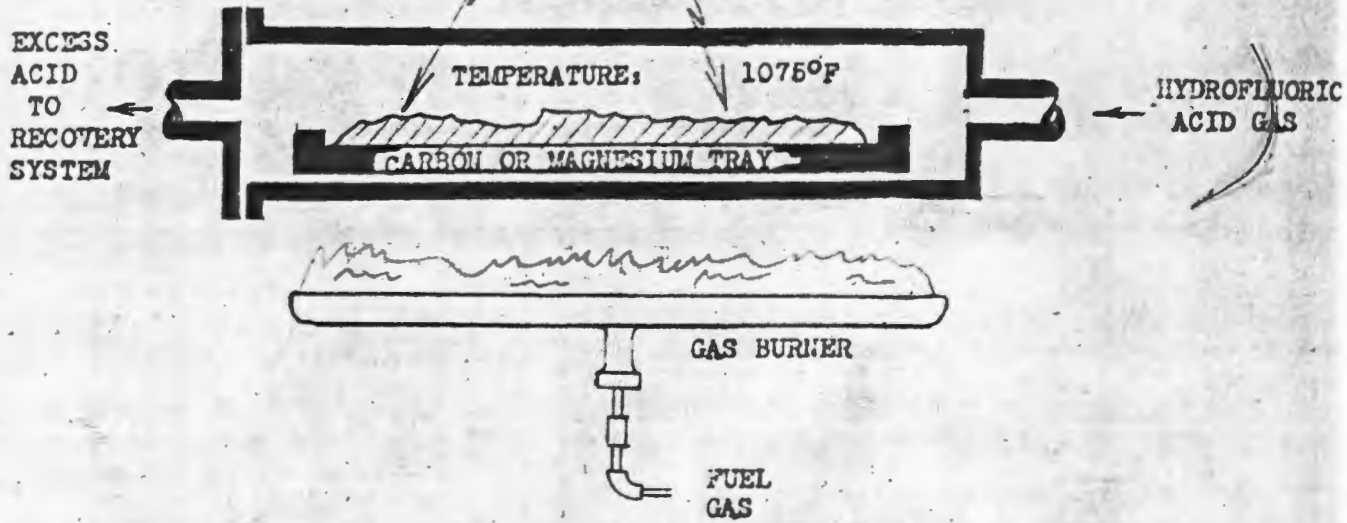
~~SECRET~~

~~SECRET~~

No. 5

"GREEN SALT PROCESS"

"BROWN OXIDE" (URANIUM DIOXIDE)  
ON TRAY IS  
CONVERTED BY ACID TO  
GREEN URANIUM TETRAFLUORIDE

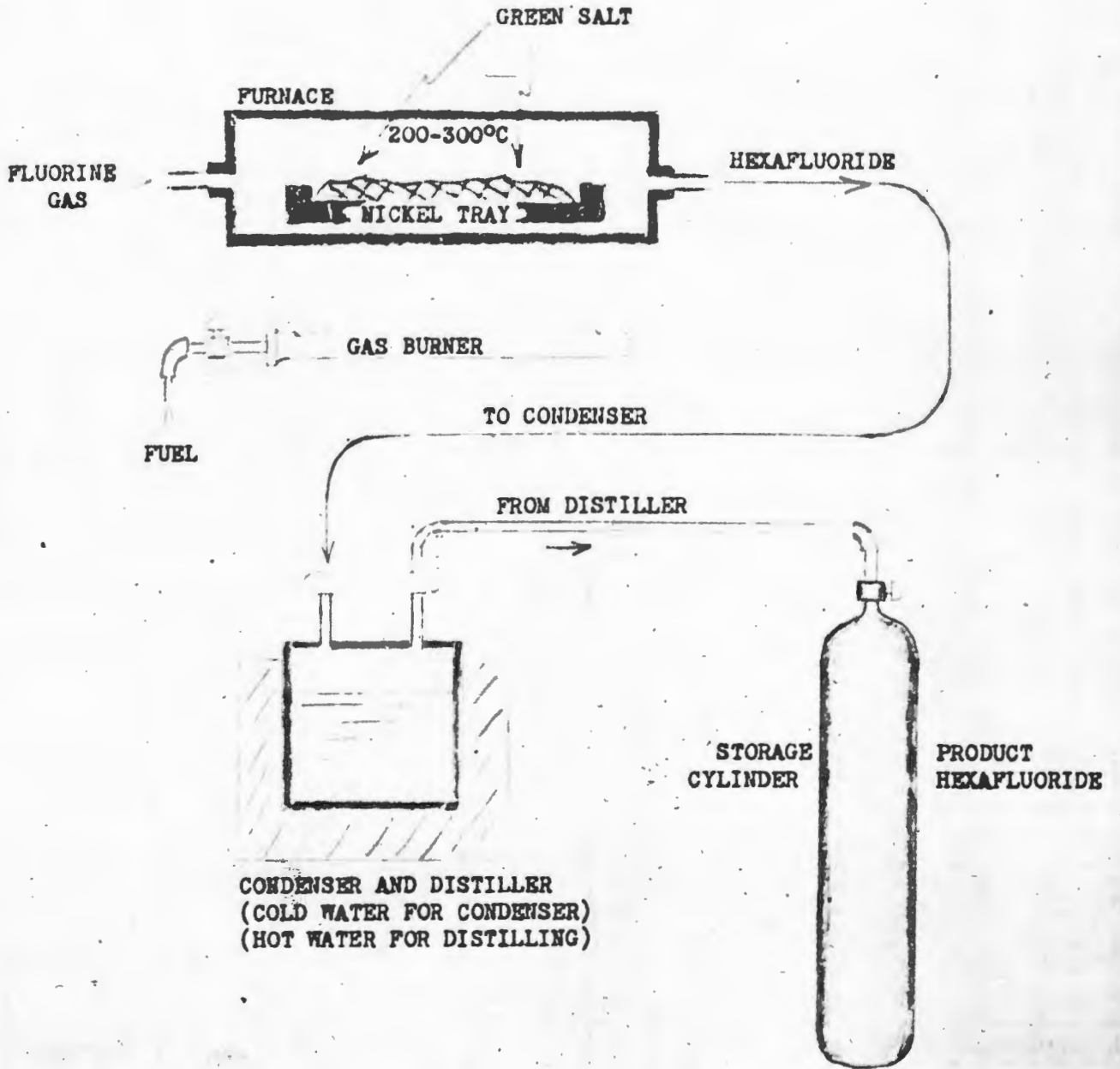


~~SECRET~~

~~SECRET~~

No. 6

HEXAFLUORIDE PROCESS

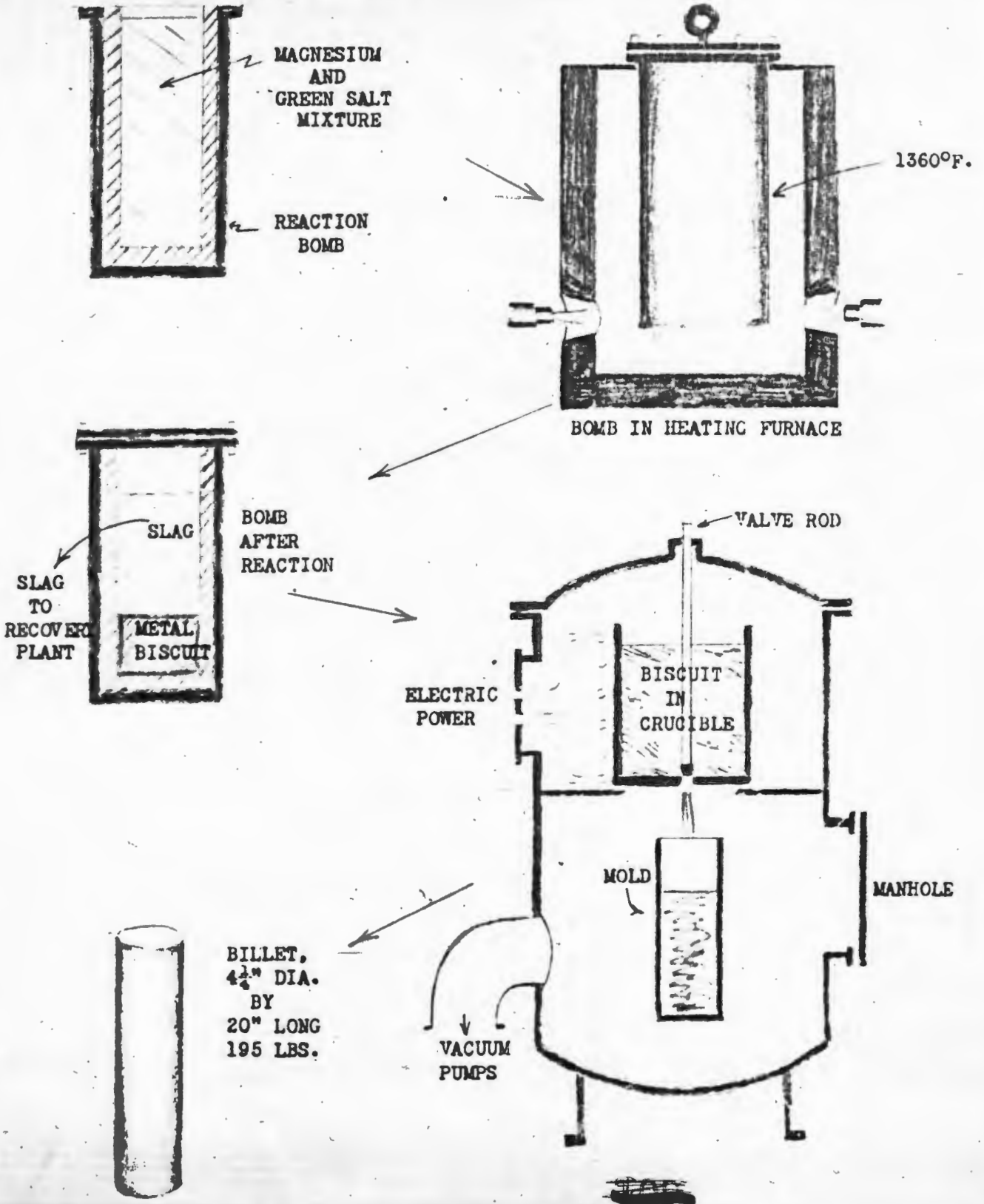


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~~SECRET~~

No. 7

METAL PROCESS



~~SECRET~~

~~SECRET~~  
MANHATTAN DISTRICT HISTORY

BOOK VII - FEED MATERIALS

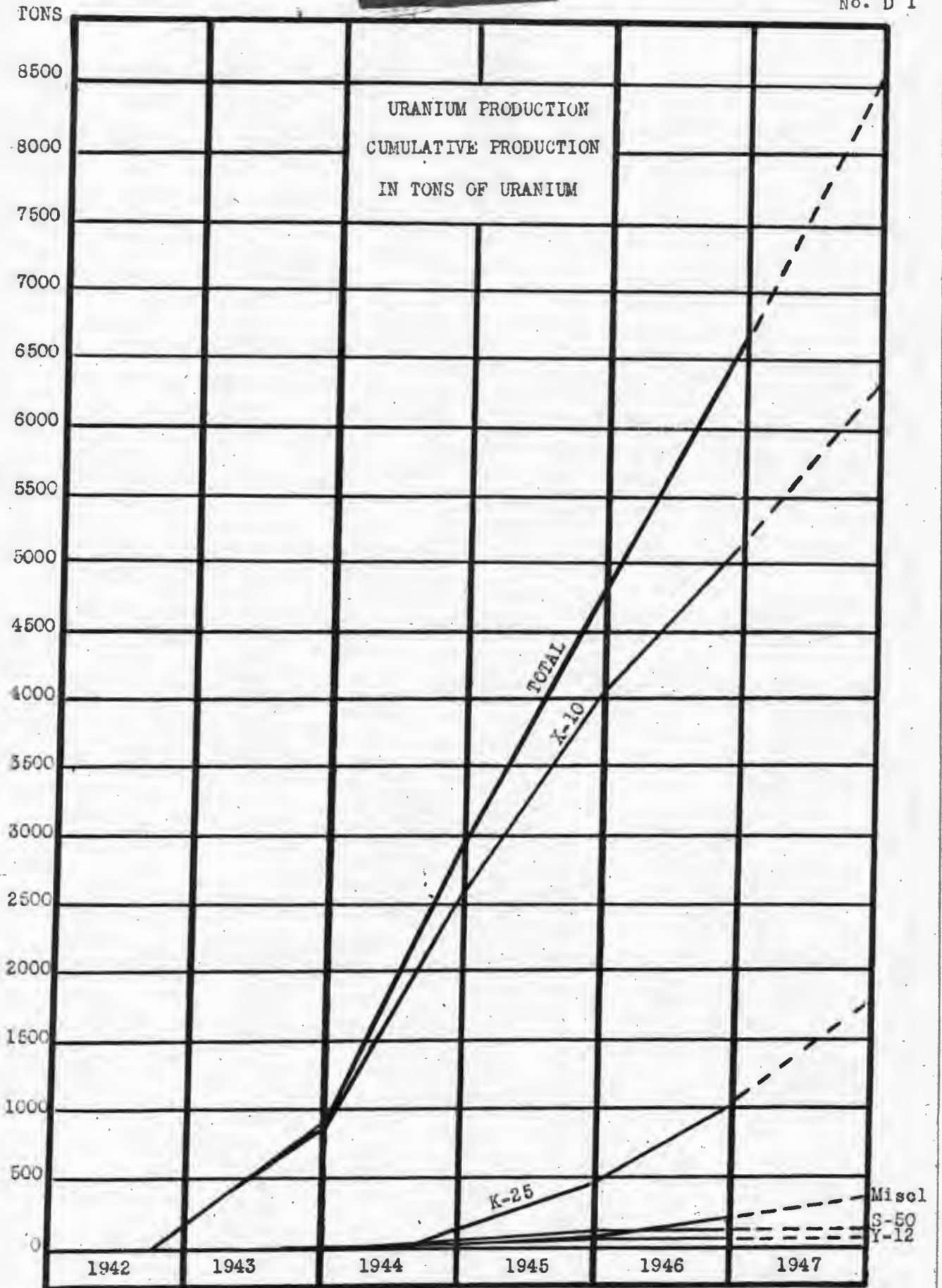
APPENDIX "D"

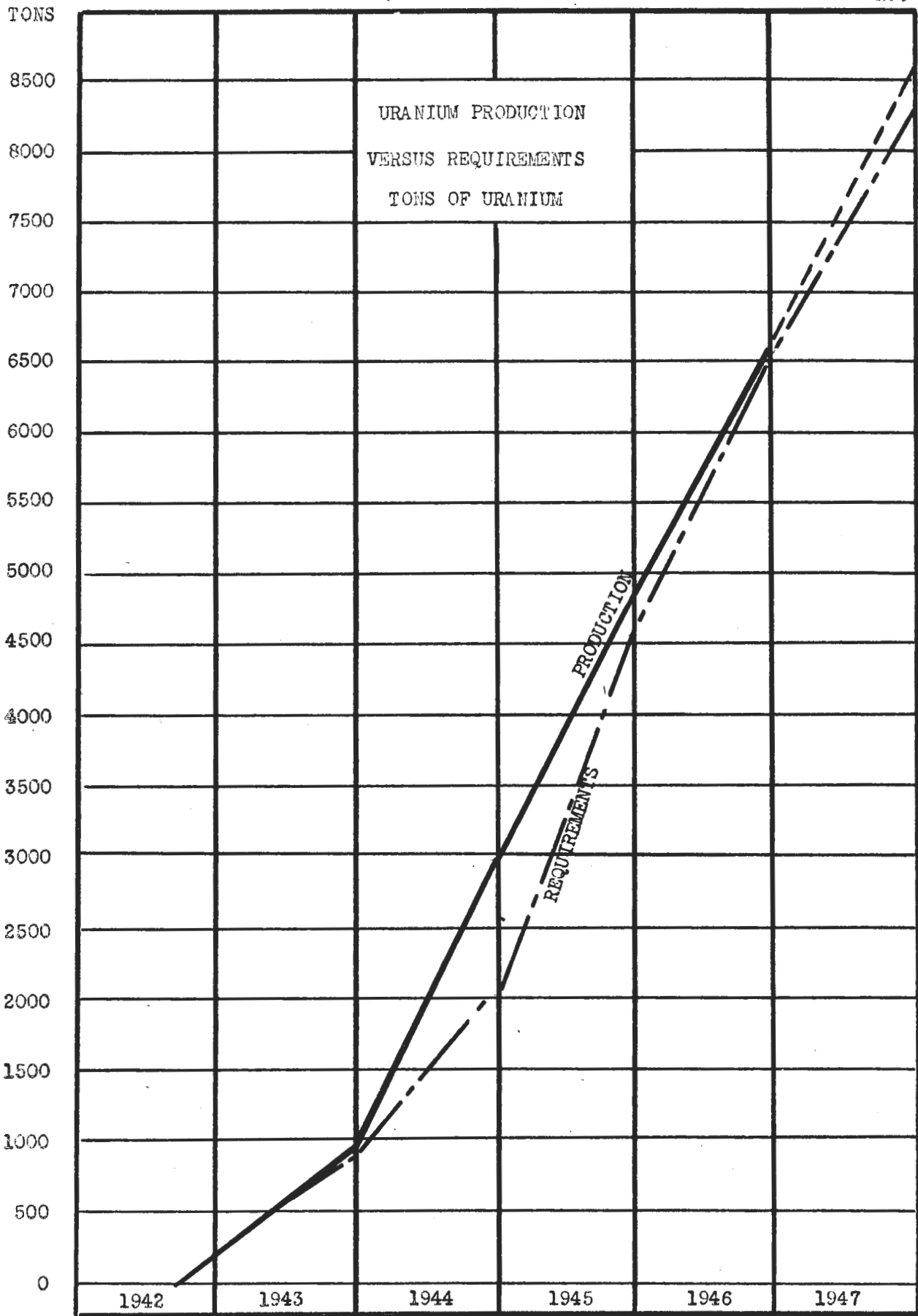
GRAPHS

<u>No.</u>	<u>Description</u>
1	Uranium production
2	Uranium production versus requirements
3	Receipt of $U_3O_8$ in ore concentrates
4	Ore concentration operations - production and cost data
5	Recovery operation - production and cost data
6	Brown Oxide operations - production and cost data
8	Linde
7	Mallinckrodt and du Pont
8	Mallinckrodt Refinery
9	Green Salt operations - production and cost data
8	du Pont and Linde
10	Mallinckrodt and Harshaw
11	Hexafluoride operation - production and cost data
12	Metal operations - production and cost data
13	Recast metal operations - production and cost data

~~SECRET~~









TONS

11000

10000

9000

8000

7000

6000

5000

4000

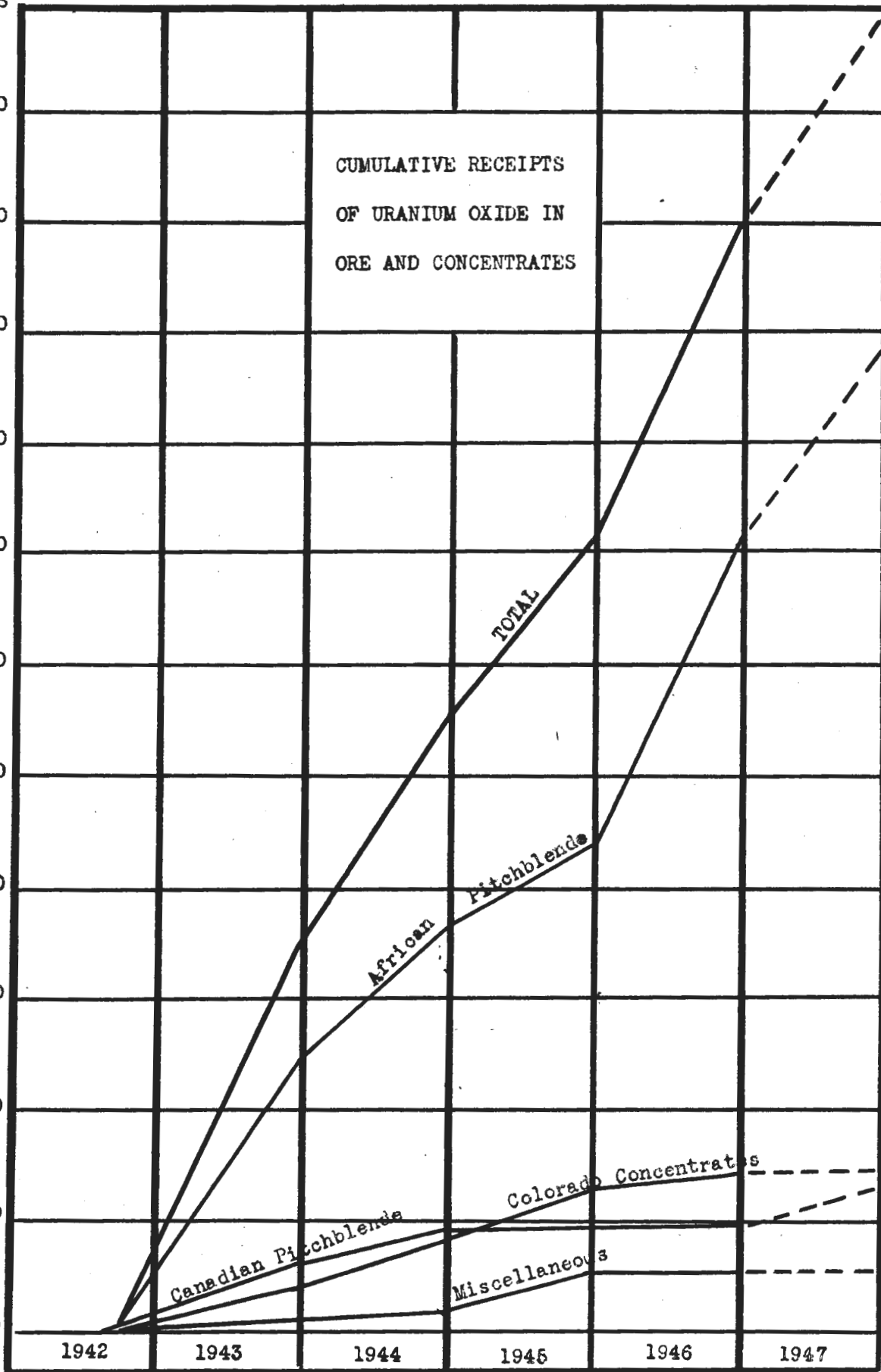
3000

2000

1000

0

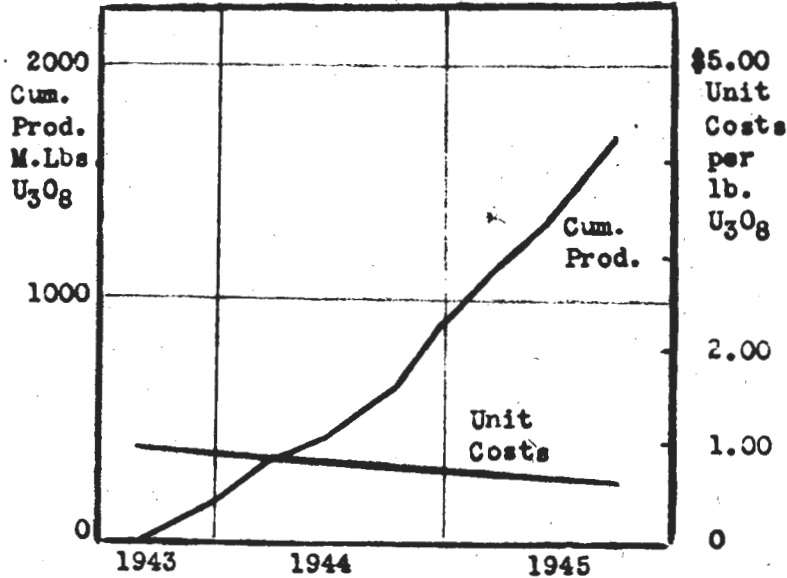
CUMULATIVE RECEIPTS  
OF URANIUM OXIDE IN  
ORE AND CONCENTRATES



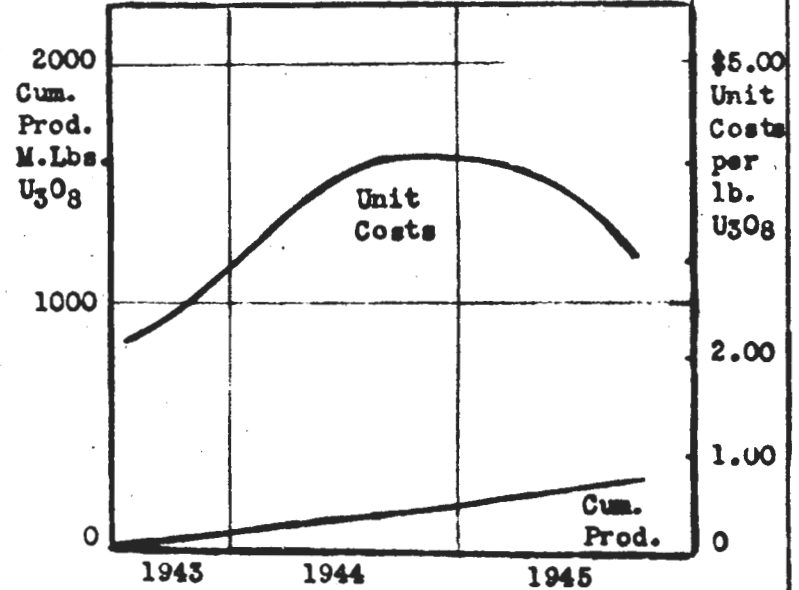
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~~SECRET~~

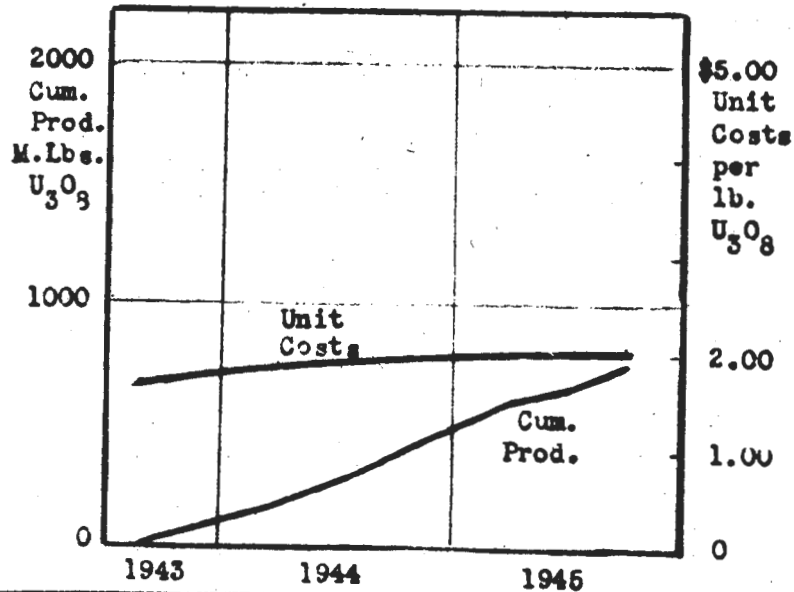
GRAND JUNCTION



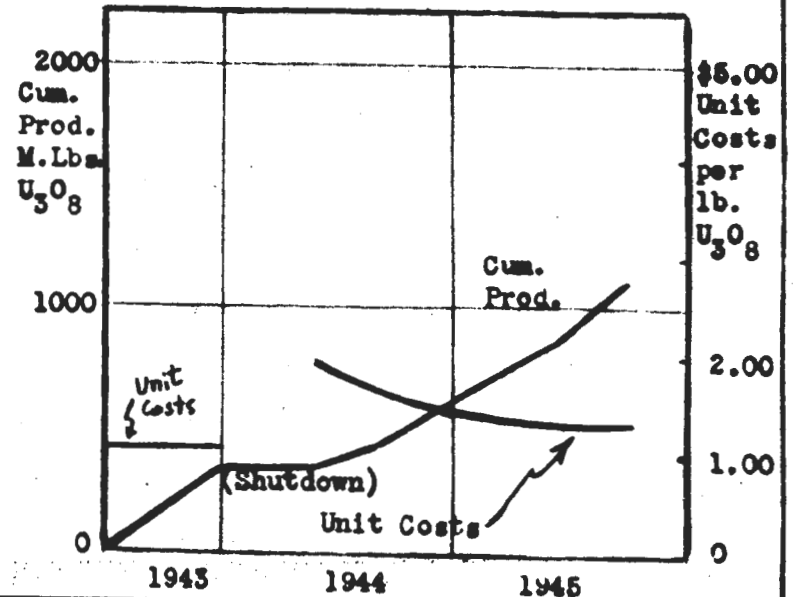
USEO DURANGO



USEO URAVAN



USV URAVAN



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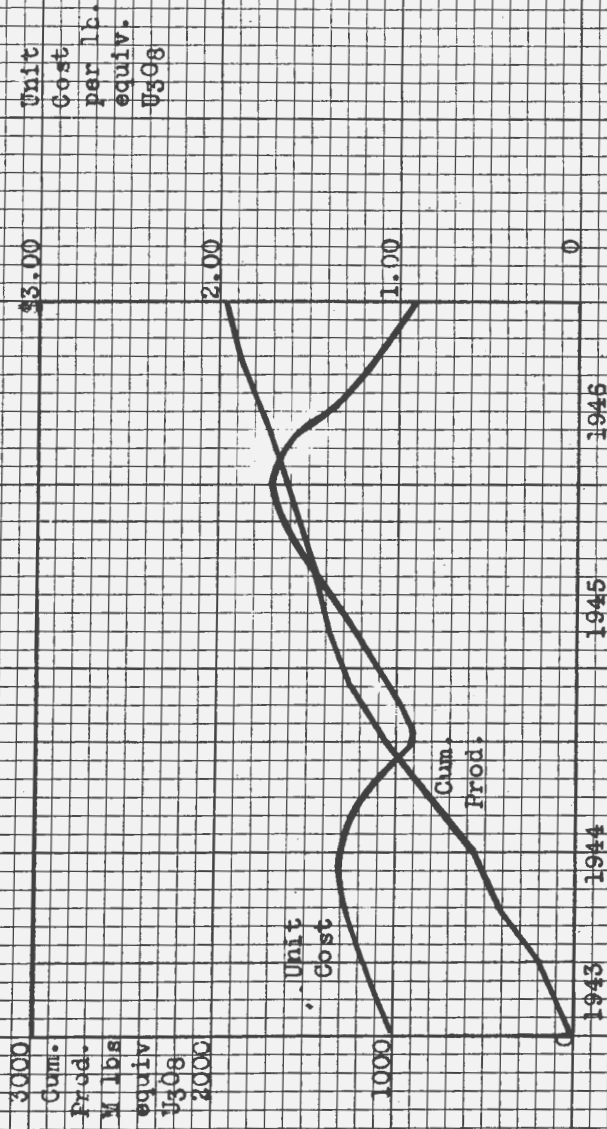
ORE CONCENTRATION OPERATIONS

~~SECRET~~

No. D 4

RECOVERY OPERATION

DUPONT



BROWN OXIDE OPERATION

LINDE

Unit  
Cost  
per lb.  
UO<sub>2</sub>

\$2.25

1.50

0.75

0

4500  
Cum.  
Prod.  
Tons  
UO<sub>2</sub>

3000

1500

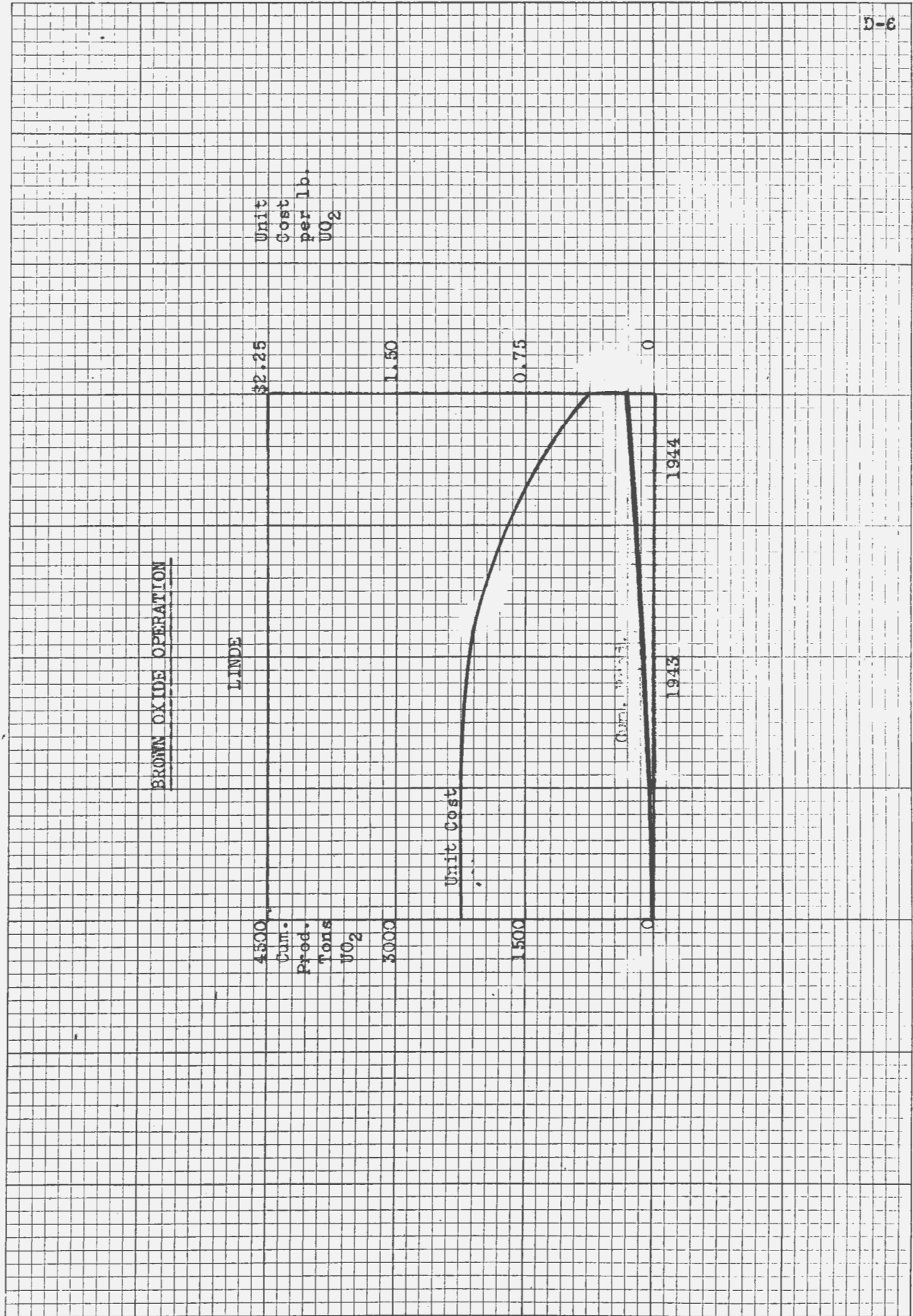
0

Unit Cost

Cum. Prod.

1943

1944

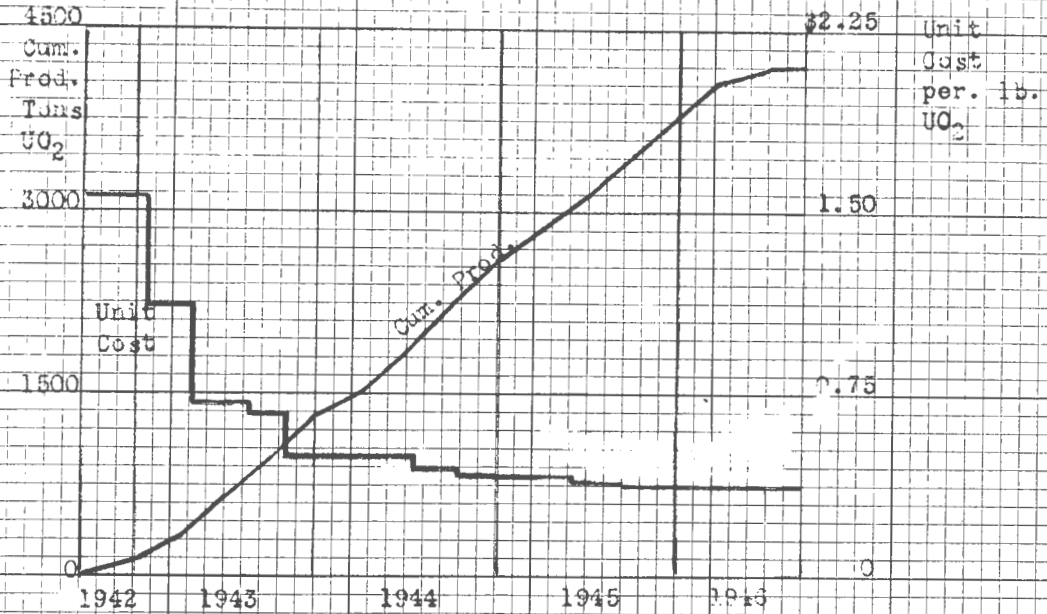


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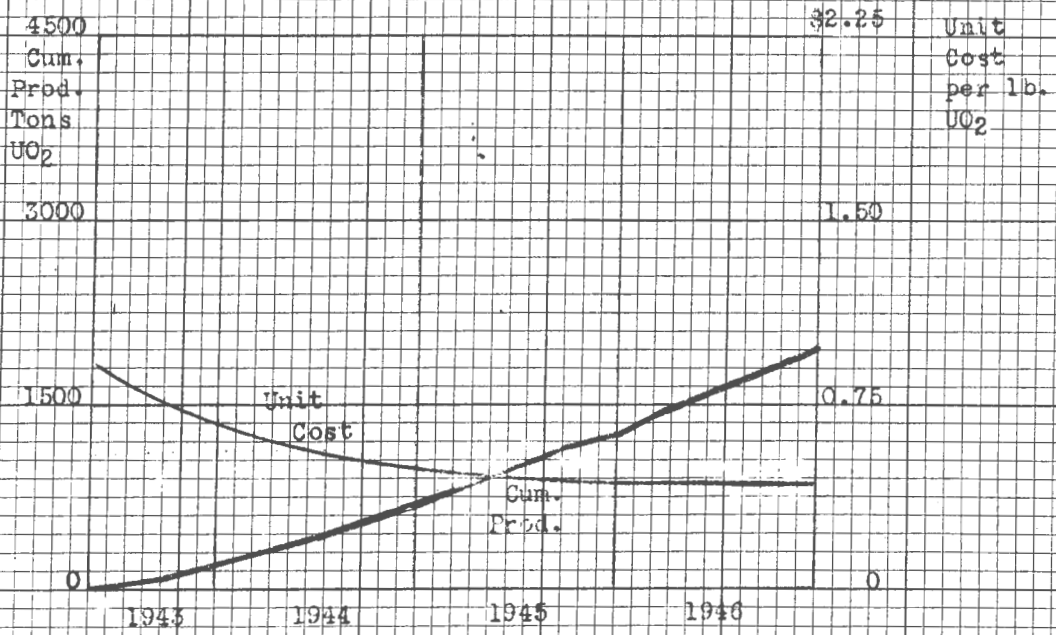
BROWN OXIDE OPERATIONS

D-7

MALLINCKRODT



DU PONT



KEUFFEL & ESSER CO., N. Y. NO. 358 5  
10 X 10 to the Inch.  
MADE IN U.S.A.

~~TOP SECRET~~

KEUFFEL & ESSER CO., N. Y. NO. 358 5  
10 X 10 to the Inch.  
MADE IN U. S. A.

BROWN OXIDE OPERATION

MALLERBROOK

Unit Cost per lb. UO<sub>2</sub>

4500 Cum. Prod. Tons UO<sub>2</sub>

3000

Unit Cost

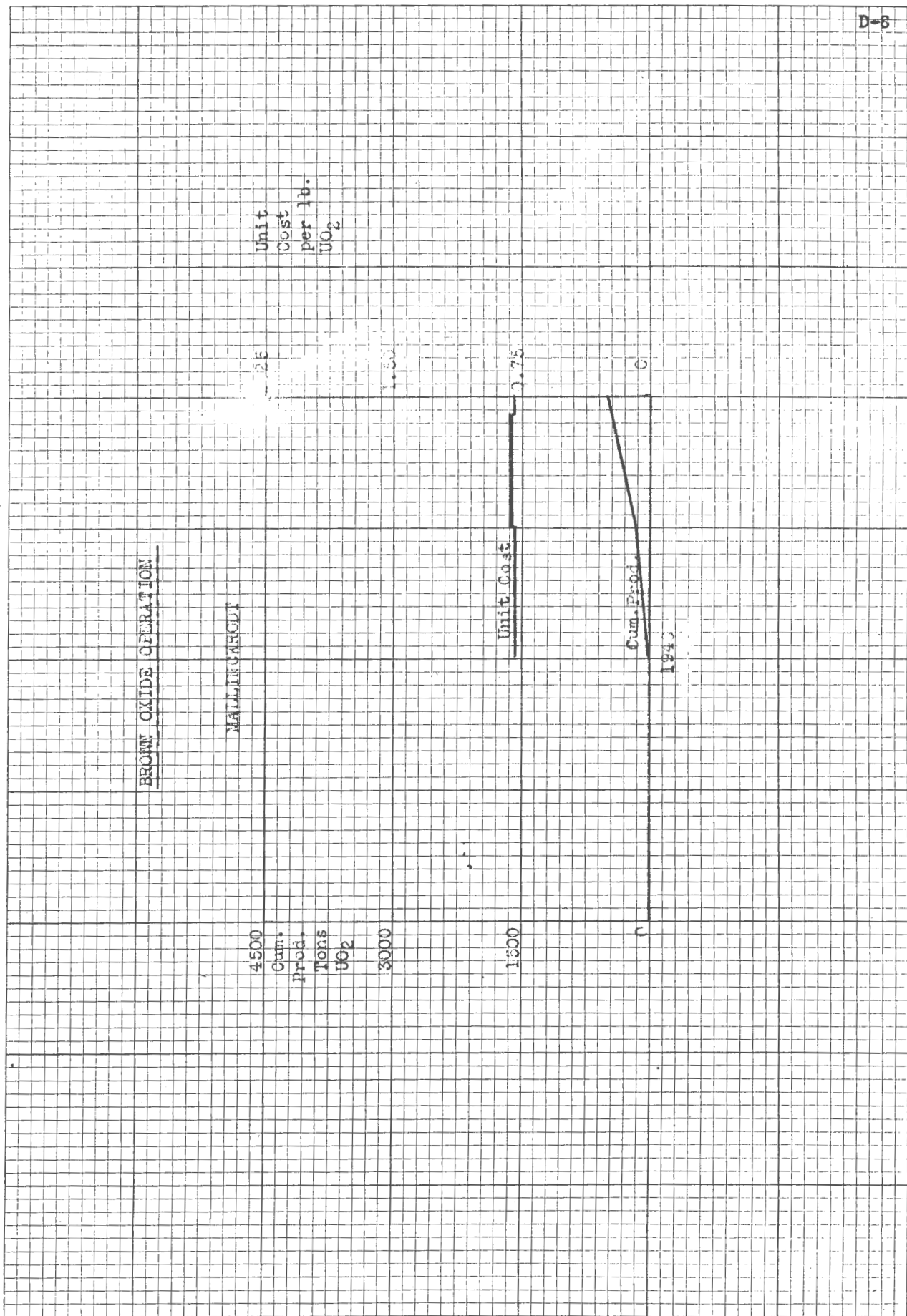
1500

Cum. Prod.

1940

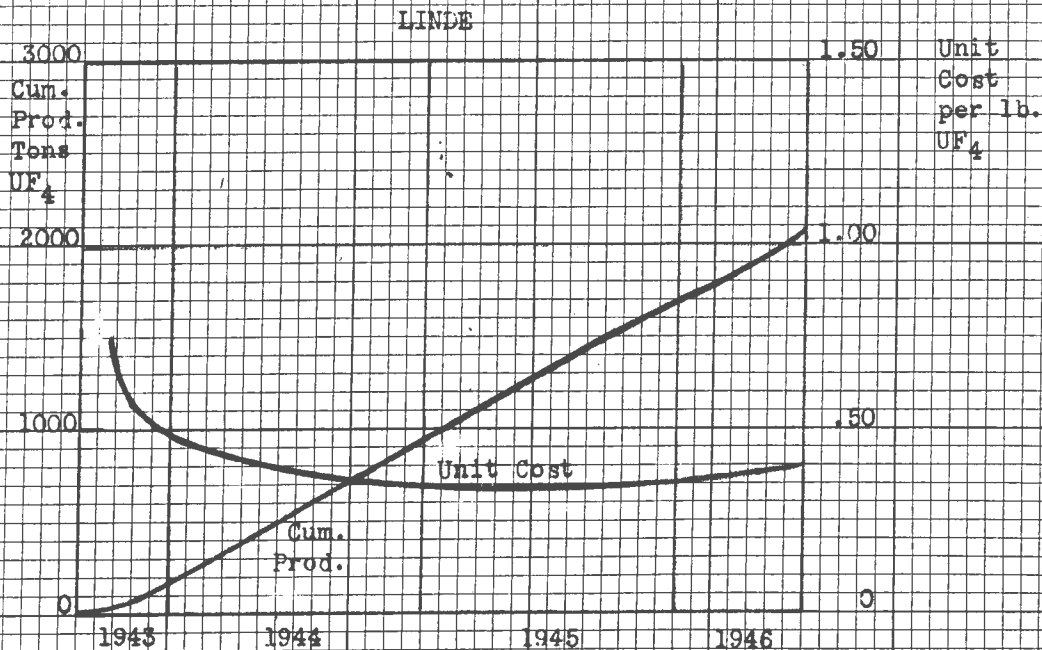
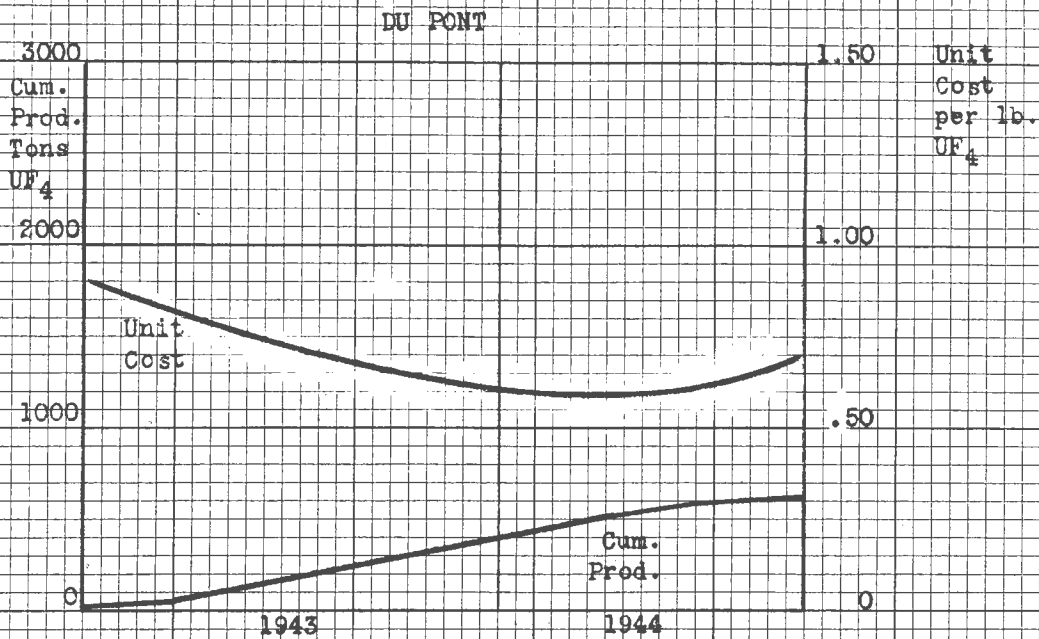
0.75

0



~~TOP SECRET~~  
GREEN SALT OPERATIONS

D-9

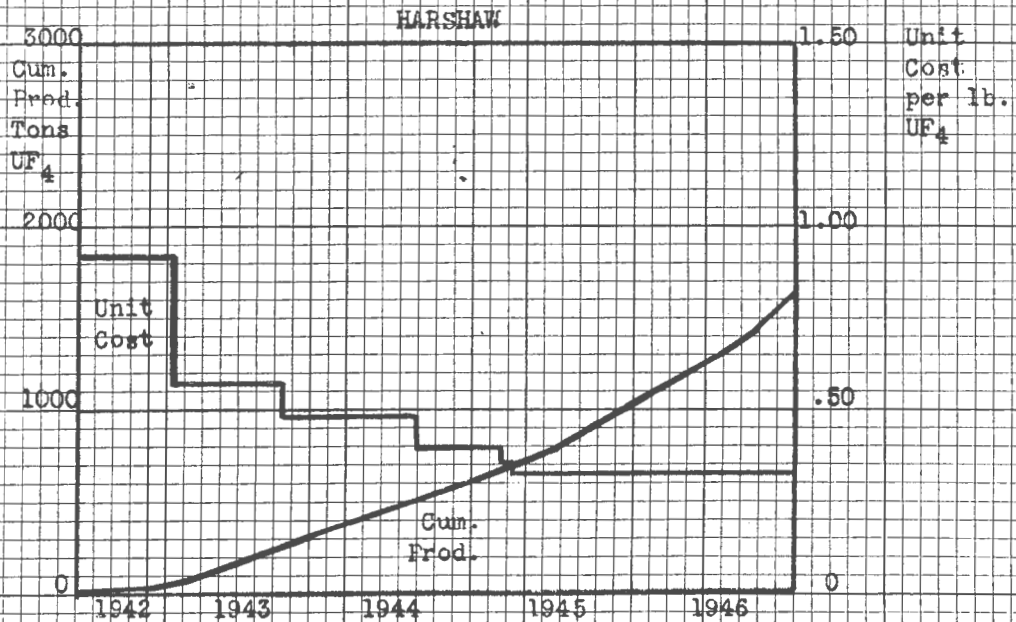
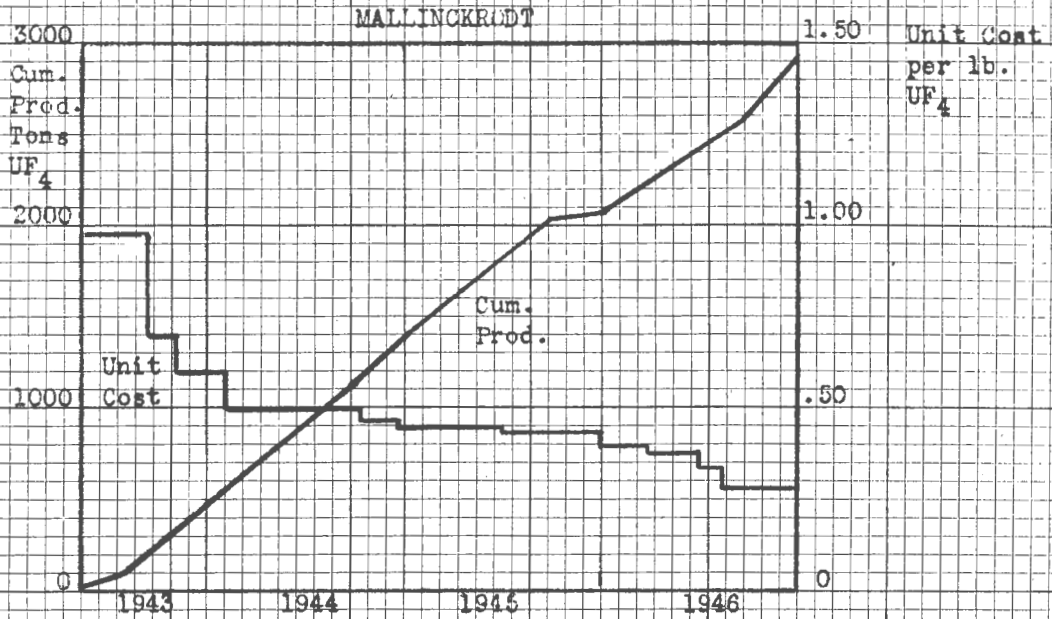




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GREEN SALT OPERATIONS

D-1C



KEUFFEL & ESSER CO., N. Y. NO. 388-S  
10 X 10 to the Inch,  
MADE IN U.S.A.

~~TOP SECRET~~



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D-11

HEXAFLUORIDE OPERATION

HARSHAW

Unit Cost per lb. UF<sub>6</sub>

\$2.25

1.50

0.75

0

1944

1945

1946

4500  
Cum. Prod. M. Lbs. UF<sub>6</sub>

3000

1500

0

Unit Cost

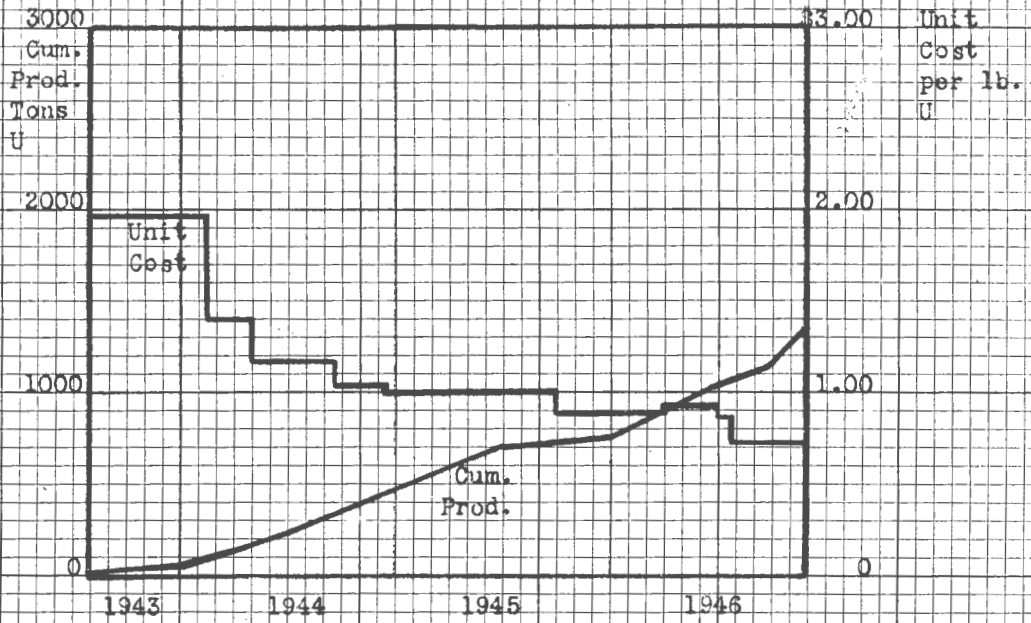
Cum. Prod.

~~TOP SECRET~~

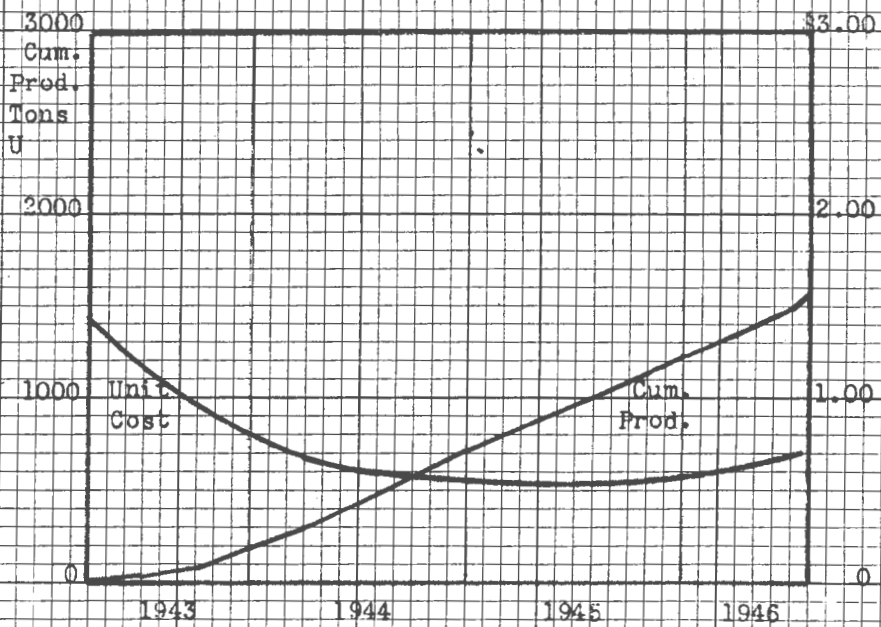
METAL OPERATIONS

D-12

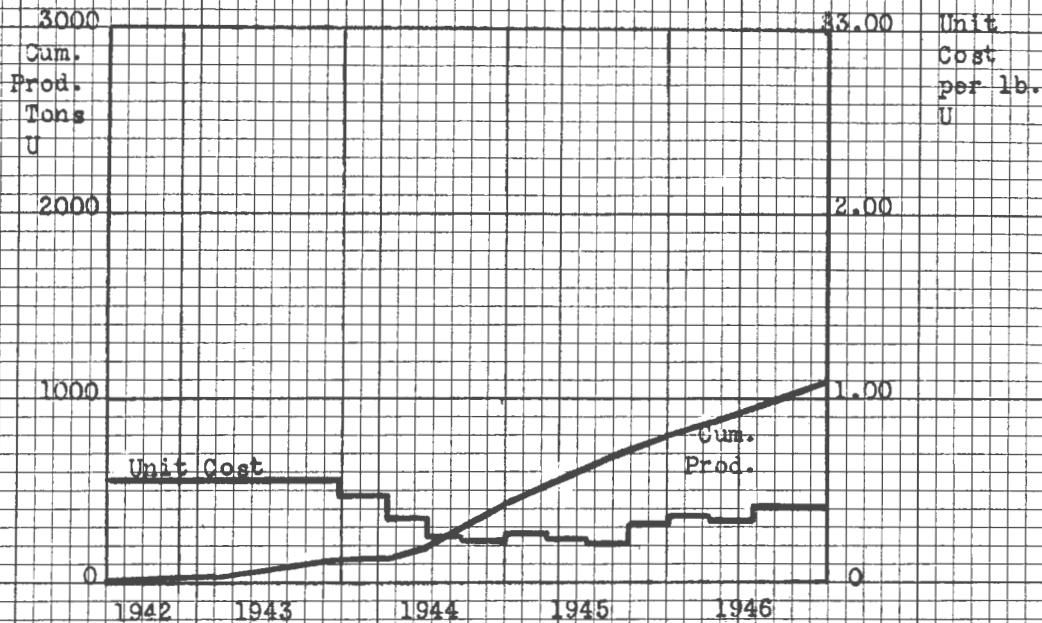
MALLINCKRODT



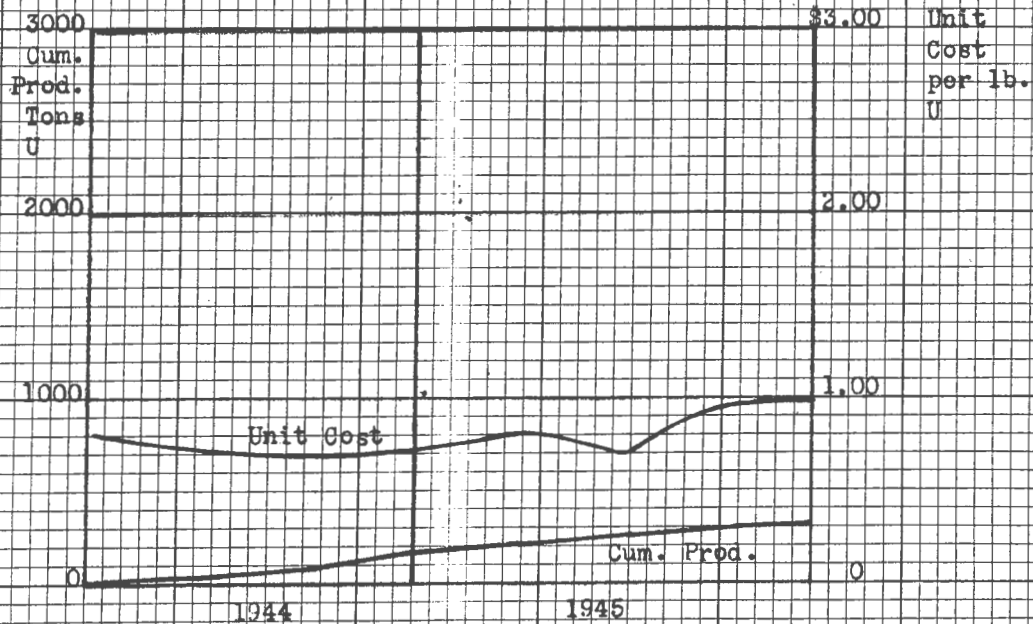
ELECTRO-MET



METAL HYDRIDES



IOWA STATE COLLEGE



~~TOP SECRET~~

MANHATTAN DISTRICT HISTORY

BOOK VII - FEED MATERIALS

APPENDIX "E"

REFERENCES

(All documents are located in the MSA files of the Atomic Energy Commission, Office of New York Directed Operations.)

- | <u>No.</u> | <u>Description</u>  |
|------------|---|
| 1          | Memorandum to the Files by Capt. P. L. Merritt, dated 21 December 1942, Subject: Present and Prospective Supplies of Uranium-bearing Material.  |
| 2          | Memorandum to Brigadier General L. R. Groves by Lt. Col. J. R. Ruhoff, dated 23 November 1943, Subject: Position of Belgian Congo with regards to Uranium and other Metal Production.   |
| 3          | Letter from Mr. Julian Leroy, African Metals Corporation, to Lt. Col. J. R. Ruhoff, dated 27 October 1943, stating that the Shinkolobwe Mine could not be re-opened.  |
| 4          | Memorandum, dated 17 September 1942, concerning a meeting in New York between Capt. J. R. Ruhoff, Dr. R. L. Geddes of Stone & Webster, Mr. E. Sengier of African Metals and Mr. Prigel of Canadian Radium and Uranium Corporation.              |
| 5          | Memorandum to Major General L. R. Groves by Col. K. D. Nichols, dated 18 April 1944, Subject: Ore Prices.   |
| 6          | Memorandum to Brigadier General L. R. Groves by Lt. Col. J. R. Ruhoff, dated 15 February 1944, Subject: Summary of Ore Contracts.   |
| 7          | Memorandum to Col. K. D. Nichols by Lt. Col. J. R. Ruhoff, dated 26 November 1943, Subject: Plans for the handling of African Ore.  |
| 8          | Letter from Mr. Carl French, Eldorado, to Major P. L. Merritt, dated 11 October 1944, enclosing the Order of the Governor General in Council of Canada which appropriated all the issued capital stock of Eldorado Mining and Refining Limited. |

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~~TOP SECRET~~

APPENDIX "E"

REFERENCES (Contd.)

- 9 Letter from Mr. Lesslie R. Thomson, Special Liaison Officer, to Col. K. D. Nichols, dated 16 April 1945, enclosing a Memorandum showing the cost of producing black oxide at Eldorado.
- 10 Memorandum to the Files by Capt. P. L. Merritt, dated 29 January 1943, Subject: Conferences Regarding the Recovery of  $U_3O_8$  From Carnotite Tailings.
- 11 Memorandum to the Files by Capt. P. L. Merritt, dated 18 January 1943, Subject: Treatment of Carnotite Tailings from Vanadium Plants in Colorado and Utah.
- 12 Letter from Lt. Col. J. R. Ruhoff to Mr. E. D. Bransome, V.C.A., dated 20 January 1944, concerning the re-opening of the Metals Reserve Monticello plant by V. C. A.
- 13 Memorandum to the Files by Major J. R. Ruhoff, dated 31 January 1943, Subject: Cost of Vanadium Sands.
- 14 War Production Board Order M-285, dated 26 January 1943.
- 15 Amended War Production Board Order M-285, dated 15 August 1944.
- 16 Letter from Lt. Col. J. R. Ruhoff to Mr. R. J. Lund, W.P.B., dated 27 November 1943, concerning the procurement of available commercial uranium stocks by Vitro Manufacturing Company.
- 17 Memorandum to the District Engineer by Lt. Col. J. R. Ruhoff, dated 4 November 1943, Subject: Polonium.
- 18 Memorandum by Dr. J. H. Lum and Dr. W. C. Fernelius, dated 23 December 1943, concerning a meeting at Eldorado to discuss lead residues and polonium recovery.
- ~~TOP SECRET~~

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APPENDIX "E"

REFERENCES (Contd.)

- 19 Memorandum to Lt. Col. J. R. Ruhoff by Brigadier General L. R. Groves, dated 27 October 1943, Subject: Transmittal of Letters.
- 20 Memorandum to the Files by Major P. L. Merritt, dated 6 December 1943, Subject: Purchase of Tailings.
- 21 Memorandum to the Area Engineer, Madison Square Area, Attention: Major G. W. Russell by Col. K. D. Nichols, dated 6 October 1944, Subject: Authorization for Purchase of Uranium Compounds.
- 22 Memorandum to the Files by Capt. J. A. Bergants, dated 27 January 1944, Subject: Operation of U. S. V. Uravan acid leach plant.
- 23 Letter from Mr. J. R. Van Fleet, U.S.V. to Col. K. D. Nichols, Dated 9 May 1944, regarding discontinuing operations at the U.S.V. Uravan plant.
- 24 Letter from Mr. Blair Burwell, U.S.V., to Lt. Col. J. R. Ruhoff, dated 1 May 1944, giving estimates of costs of leaching operations at Uravan and refining operations at Grand Junction.
- 25 Memorandum to the Files by Major J. E. Vance, dated 18 April 1944, Subject: Estimated Cost of Operation of the U.S.V. Uravan Mill for Army Account.
- 26 Letter from Mr. Blair Burwell, U.S.V., to Lt. Col. J. R. Ruhoff, dated 13 June 1944, discusses the cancellation of Contract W-7405 eng-201 and the resumption of acid leach at our Uravan Plant by the Army Contract Account.
- 27 Memorandum to the Files by Major J. E. Vance, dated 27 June 1944, Subject: Operation of the U. S. V. Uravan Plant under Supplemental Agreement No. 1 to Contract W-7405 eng-32.
- ~~SECRET~~

~~TOP SECRET~~

APPENDIX "E"

REFERENCES (Contd.)

- 28 Letter from Mr. J. A. Holladay, Union Carbide and Carbon Corporation, to Lt. Col. J. R. Ruhoff, dated 13 August 1943, giving an estimated regarding the average monthly output of Step I of the Linde Ceramics Plant.
- 29 Letter from Mr. J. A. Holladay, Union Carbide and Carbon Corporation, to Lt. Col. T. T. Crenshaw, dated 5 June 1943, giving status of construction and/or operations of Steps Nos. 1, 2, 3 and 4 of the project.
- 30 Letter from Mr. S. W. Coleman, Mallinckrodt Chemical Works, to Major C. Vanden Bulck, dated 13 November 1942, giving the cost of the manufacture of Refined Tube Alloy Dioxide for 300,000 lbs. at \$1.56 per lb.
- 31 Letter from Col. K. D. Nichols to Mr. Edward Mallinckrodt, Jr., Mallinckrodt Chemical Works, dated 8 May 1944, acknowledging the voluntary and unsolicited return of the savings realized as a result of operating techniques.
- 32 Letter to the Area Engineer, Tonawanda Area by Major C. Hadlock, dated 2 February 1944, Subject: Release of Linde Step 2 Equipment for Other Work.
- 33 Letter from Mr. Louis Spiegler, du Pont, to Lt. Col. J. R. Ruhoff, dated 18 March 1944, regarding economic survey on the manufacture of C-112 by various methods.
- 34 Letter from Major C. Hadlock to Mr. J. A. Holladay, Union Carbide & Electro Metallurgical Research Lab., dated 26 July 1943, giving standard procedure for handling by-product materials scheduled for processing in the du Pont slag recovery plant.
- 35 Letter from Mr. A. C. Klein, Stone & Webster Engineering Corp., to the Harshaw Chemical Co., dated 26 September 1942, regarding tetrafluoride production.
- ~~TOP SECRET~~

APPENDIX "E"

REFERENCES (Contd.)

- 36 Summary of process entitled "Production of Tetrafluoride" written by Mr. K. E. Long, of the Harshaw Chemical Co., dated 8 April 1943.
- 37 Letter from Mr. M. K. Richards, du Pont to Dr. H. W. Elley, Director Chemical Div., dated 20 April 1943, regarding requirements of 2000 lbs/day of Product C-616 for U. S. Engineers commencing 1 May 1944.
- 38 Letter from Mr. K. E. Long, Harshaw, to Lt. Col. J. R. Ruhoff, dated 24 August 1943, discusses the methods of preparation of Product C-616.
- 39 Letter from Mr. M. K. Richards, du Pont, to Dr. H. W. Elley, dated 11 September 1943, regarding location and cylinder study of Product C-616.
- 40 Letter from Mr. W. J. Harshaw, to Lt. Col. J. R. Ruhoff, dated 5 November 1943, discusses erection of building for manufacture of Product C-616.
- 41 Letter from Mr. S. W. McCune, du Pont to Lt. Col. J. R. Ruhoff, dated 19 November 1943, regarding facilities for manufacture of 2,200 lbs/day of Product C-616.
- 42 Letter from Mr. S. W. McCune, du Pont, to Lt. Col. J. R. Ruhoff, dated 4 December 1943 regarding manufacturing cost of Product C-616.
- 43 Letter from Mr. W. J. Harshaw, Harshaw, to Lt. Col. J. R. Ruhoff, dated 23 December 1943, giving revised cost calculations covering the production of Product C-616.
- 44 Letter from Major E. A. Brinkman to Mr. C. S. Parke, Harshaw, dated 22 August 1944, authorizing construction of a plant for production of Product C-616.



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APPENDIX "E"

REFERENCES (Contd.)

- 45 Letter from Mr. R. L. Doan, Metallurgical Lab., to Dr. C. B. Sawyer, dated 15 September 1942, regarding the production of metal by the magnesium reduction process.
- 46 Letter from Mr. R. L. Doan, Metallurgical Lab. to Dr. C. B. Sawyer, Brush Labs., dated 6 October 1942, regarding the undertaking on a small scale of production of metal by the magnesium reduction process.
- 47 Letter from Dr. C. B. Sawyer, Brush Labs., to Major T. T. Crenshaw, dated 12 November 1942, confirming production of metal.
- 48 Letter from Mr. J. A. Holladay, Union Carbide and Carbon Corporation, dated 17 September 1943 to Lt. Col. J. R. Ruhoff, discussing capacity of plant to produce finished billets and eggs.
- 49 Letter from Major W. L. Sapper to Mr. McCune, du Pont, dated 7 August 1944, authorizing continuation for Product C-103 and discontinuing operation of plants for Products C-104 and C-105.
- 50 Letter from Major C. Hadlock to Dr. Frank H. Spedding, Iowa State College, dated 20 September 1943, discusses the future status of supplies of Slo-Set.
- 51 Memorandum to the Files by Capt. R. D. Morse, dated 1 February 1944, Subject: Ames Experimental Program for Turnings Recasting.
- 52 Letter to the Area Engineer, Madison Square Area, Attention, Capt. R. D. Morse from Frank Huke, dated 31 May 1944, Subject: Report on Recasting of Turnings.
- 53 Memorandum to the Files by E. E. Chipman, dated 30 October 1944, Subject: Development Program at Iowa State College.
- ~~XXXXXXXXXX~~

~~SECRET~~

APPENDIX "Z"

REFERENCES (Contd.)

- 54 Letter from Lt. Dick Duffey to Dr. P. P. Alexander, Metal Hydrides, dated 26 June 1943, authorizing continuation of the production and recasting of metal under Contract W-7406 eng-8 through 31 August 1943.
- 55 Letter to Lt. Col. J. R. Ruhoff from Lt. Duffey, dated 29 July 1943, Subject: Cost of Cast Metal at Metal Hydrides, Inc.
- 56 Letter from Lt. Col. T. T. Grenshaw to Dr. P. P. Alexander, Metal Hydrides, dated 5 March 1943, requesting the recasting of 500-1000 lbs/day of trialloy.
- 57 Letter to the Area Engineer, Beverly Area, from Lt. Col. J. R. Ruhoff, dated 21 April 1943, Subject: Recasting of "Heels" from Mr. F. A. Shinn.
- 58 Letter from A. C. Klein, Stone & Webster Engineering Corporation, to Westinghouse Electric & Manufacturing Co., dated 6 October 1942, regarding extension of facilities.
- 59 Letter from Major T. T. Grenshaw to Mr. A. Frankel, Westinghouse, dated 30 December 1942, confirming production of 900 lbs/week of 1" cubes until 20,000 lbs. have been produced.
- 60 Letter from Major E. A. Brinkman to Mr. A. Frankel, Westinghouse, dated 13 November 1943, cancelling contract No. W-7407 eng-2 as of 15 October 1943.
- 61 Memorandum from Major J. R. Ruhoff dated 18 February 1943, Subject: Proposed Quality Control Program and Organization of Central Laboratories.
- 62 Memorandum to the Materials File by Lt. D. G. Sturges, dated 15 April 1943, Subject: Conference in Chicago on February 26, 1943, for Discussion of Central Control Group.
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APPENDIX "E"

REFERENCES (Contd.)

- 63 Letter from Major C. Hadlock to Mr. N. McL. Sage, MIT, dated 18 March 1944, regarding sending two members of the MIT staff on "fire brigade" duty at the District Office.
- 64 Letter from Major J. R. Ruhoff to Dr. Clement J. Rodden, NBS, dated 11 March 1943, regarding samples being sent for analyses with a schedule attached.
- 65 Memorandum to the Files by Lt. D. G. Sturges, dated 17 November 1943, Subject: Ore Assay Schedule.
- 66 Memorandum to the Files, by Lt. D. G. Sturges, dated 1 June 1943, Subject: Analytical Conference at Chicago on May 28, 1943.
- 67 Letter from Major T. T. Crenshaw to Mr. A. Frankel, Westinghouse, dated 18 November 1942, requesting that wastage and by-products of certain tuballoy compounds not be thrown away.
- 68 Letter from Major C. Hadlock to Mr. J. A. Holladay, UCC, dated 11 September 1943, regarding the accounting for, and safeguarding of, special metal or its compounds.
- 69 Letter (and tabulation) from Capt. M. L. Hecker, to District Engineer, Attention: Capt. B. G. Seitz, dated 27 March 1946, Subject: Quarterly Budget Review.
- 70 Memorandum to Files by Capt. D. G. Sturges, dated 3 November 1944, Subject: Survey of future operations of Metal and Metal-intermediate plants (with attachments dated 1 June 1944 and 26 September 1944).
- 71 Letter from Major C. Hadlock to Dr. Spedding, Iowa State College, dated 20 Sept. 1943, establishing a standard shipping memorandum procedure.
- 72 Letter from Lt. Col. Crenshaw to Mr. K. E. Long, Harshaw Chemical Co., dated 15 January 1943, establishing "Weekly Production Report" procedure.
- 73 Memorandum from Mr. W. E. Kelley, Manager New York Directed Operations, dated 22 October 1947, Subject: Manhattan District History, Book VII, Feed Materials and Special Procurement, with Exhibits Nos. 1, 2 and 3 attached.

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~~SECRET~~AFRICAN RAW MATERIAL PROCUREMENT SUMMARY

<u>Contract or Purchase Order Number</u>	<u>Date of Contract</u>	<u>Delivery Period</u>	<u>Tons of Ore Contracted For</u>	<u>% U<sub>3</sub>O<sub>8</sub> in Ore (Approx.)</u>	<u>Tons of U<sub>3</sub>O<sub>8</sub> in Ore</u>	<u>% U<sub>3</sub>O<sub>8</sub> Content Paid for</u>	<u>Contract Price per pound of U<sub>3</sub>O<sub>8</sub> (Approx.)</u>	<u>TOTAL COST</u>
African Metals Corporation								
W-7405 eng-4	October 19, 1942	October 1942	100	85	75	80	\$1.00	\$ 118,200
W-7405 eng-24	October 15, 1942	October 1942 - August 1943	2,050	65	1,333	85	1.30 )	3,106,000
W-7405 eng-24	October 15, 1942	February - July 1943	1,000	20	169	85	1.30 )	
W-7405 eng-30	November 19, 1942	November 1942	250	65	157	85	1.08	183,000*
W-7405 eng-94	May 27, 1943	October 1943 - June 1944	530	65	352	98	1.25 )	3,748,500
W-7405 eng-94	May 27, 1943	August 1943 - June 1944	1,075	20-26	199	90	1.25 )	
W-7405 eng-94	May 27, 1943	October 1943 - July 1944	10,250	10	967	90	1.25 )	
W-7405 eng-94	May 27, 1943	August - September 1944	2,000	8	114	90	1.25 )	
W-7405 eng-259	October 15, 1943	October 1943	20	10	2.3	100	1.13	12,100
W-7405 eng-279	December 5, 1943	March 1944 - February 1946	12,000	3	328	100	1.13	1,518,000**
W-7405 eng-280	December 5, 1943	September 1944 - May 1946	181	50-60	113	100	1.40 )	423,700
W-7405 eng-280	December 5, 1943	November 1944 - June 1945	275	20	50	100	1.35 )	
P.O. 371	November 25, 1942	November 1942	1	65	0.4	100	1.13	1,800**
P.O. 3044	April 12, 1943	February 1943	0.5	20	0.1	100	1.13	500*
P.O. 8215	October 12, 1943	August 1943	0.3	10	0.03	100	1.13	200**
P.O. 28635	December 4, 1943	December 1943	1	65	0.8	100	1.17	4,500**
P.O. 29707	December 1, 1944	December 1944	0.5	50	0.3	100	1.17	1,300**
T O T A L			29,734		3,839		\$1.12 (Average)	\$9,113,800
Washington Office Procurement								
TAB-1				65	1,866		1.45	\$5,411,400
TAB-2				65	1,278		1.90	4,856,400
T O T A L					3,144			\$10,267,800

\* Cost of ore after credits have been deducted for the radium and other precious residues returned to the contractor.  
 \*\* Includes the cost of radium contained in the ore and purchased by the Government.

6433

1.30

13,217,600

CANADIAN RAW MATERIAL PROCUREMENT SUMMARY

<u>Contract or Purchase Order Number</u>	<u>Date of Contract</u>	<u>Delivery Period</u>	<u>Tons of Ore Contracted For</u>	<u>Tons of Black Oxide to be Delivered (as U<sub>3</sub>O<sub>8</sub>)</u>	<u>Estimated Cost of Ore Contracted For*</u>	<u>Total Cost of Black Oxide Contracted For</u>
<b>Stone and Webster</b>						
P. O. 135	July 15, 1942	Aug-Dec. 1942	490	140 1.54	\$ 430,600.	\$ 668,200
<b>Boris Fregel</b>						
W-7405 eng-145	May 22, 1943	Jan.-June 1943	868	--	554,100**	--
<b>Gilbert A. LaBine, Agt.</b>						
W-7405 eng-252	Sept. 11, 1943	June 1943 to date	1621	687 97	1,332,400	2,161,760
W-26-021 eng-6	Dec. 1, 1944	May 1943 to date	1170	310 446	2,765,200 †	3,825,400
			<b>T O T A L</b>	<b>4149</b>	<b>\$5,082,300</b>	<b>\$6,655,360</b>
				<del>1137</del>		

\*Contract costs include the sale and refining of ore to black oxide.  
The costs in this column are calculated.

\*\*This contract was terminated and the requirements of black oxide were  
transferred to W-7405 eng-252. The cost shown is for the ore obtained  
before termination of the contract.

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DOMESTIC RAW MATERIAL PROCUREMENT SUMMARY

<u>Contract or Purchase Order Number</u>	<u>Date of Contract</u>	<u>Total Tons of Sands or Tailings Obtained</u>	<u>% U<sub>3</sub>O<sub>8</sub> Content (Average)</u>	<u>Tons of U<sub>3</sub>O<sub>8</sub> Content</u>	<u>Contract Cost per Pound of Recovered U<sub>3</sub>O<sub>8</sub></u>	<u>TOTAL COST</u>
<b>U. S. Vanadium Corp.</b>						
W-7405 eng-201	June 1, 1943	694	12	85	\$1.10 ✓	\$ 194,300
W-7405 eng-250	July 10, 1943	253,689	0.3	758	0.30	653,500
W-26-021 eng-1	Oct. 1, 1944	25,529	0.2	48	0.30	94,000
<b>Vanadium Corp. of America</b>						
W-7405 eng-12 ✓	Nov. 13, 1942	7.1	39	2.7	0.75	6,000
W-7405 eng-144 ✓	July 29, 1943	229	48	111	1.10	388,800
W-7405 eng-255 ✓	Aug. 7, 1943	5.8	48	2.8	1.10	8,400
W-7405 eng-257 ✓	Oct. 5, 1943	3.9	48	1.9	1.10	5,500
W-7405 eng-267 ✓	Dec. 1, 1943	48,566	0.14	68	0.25	157,300
W-26-021 eng-12 ✓	Feb. 15, 1945	85	52	44	1.50	165,550
<b>Vitro Manufacturing Co.</b>						
W-26-021 eng-24	1946	--	--	26	1.50	71,650
<b>Metal Reserve Corp.</b>						
W-7405 eng-260	July 23, 1943	45	50	22	1.10	66,800
W-7405 eng-282	July 1, 1943	46,165	0.3	103	0.30	146,500
W-7405 eng-287	Feb. 4, 1944	3,852	0.3	10	0.20	3,800
Miscellaneous Purchase Orders		820	--	67	--	180,000*
<b>TOTAL</b>		<b>379,671</b>		<b>1,349</b>		<b>\$2,072,530**</b>

\*Estimate

\*\*Includes a total estimated cost of \$600,000. for the vanadium content of the sands and tailings purchased by the Government.

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<u>Contract or Purchase Order Number</u>	<u>Date of Contract</u>	<u>Delivery Period</u>	<u>Type of Material</u>	<u>Tons of Material Purchased</u>	<u>Tons of U<sub>3</sub>O<sub>8</sub> Content</u>	<u>Cost per pound of U<sub>3</sub>O<sub>8</sub> Content</u>	<u>TOTAL COST</u>
<b>African Metals Corp.</b>							
W-7405 eng-18	Nov. 11, 1942	Nov. 1942	Soda Salt	106	21	86	\$ 326,000
W-7405 eng-47	Mar. 29, 1943	June 1943	Orange Oxide	44	7 1/2	35	133,600
W-7405 eng-47	Mar. 29, 1943	June 1943	Black Oxide	39	97	38	159,700
W-7405 eng-47	Mar. 29, 1943	June 1943	Uranyl carbonate	11	48.5	8	16,000
<b>Vitre Manufacturing Co.</b>							
K-7405 eng-9	Oct. 29, 1942	Nov. 1942	Soda salt	14	26	12	43,500
W-7405 eng-9	Oct. 29, 1942	Nov. 1942	Uranyl carbonate	9	30 1/2	5	14,700
W-7405 eng-52	Mar. 27, 1943	May 1943 to date	Various refined uranium salts, e.g. black oxide, soda salt, carbonate, nitrate, etc.	84	39	35	128,500
P. O. 2789	Apr. 5, 1943	Apr. 1943	Various refined uranium salts	2	70	0.4	1,400
P. O. 3343	June 22, 1943	June 1943	Brown oxide	0.04	100	0.04	200
P. O. 43672	May 20, 1943	May 23, 1943	Soda salt	0.03	100	0.03	130
<b>Harshaw Chemical Co.</b>							
W-7405 eng-45	Mar. 27, 1943	March 1943	Black oxide	0.8	85	0.7	3,100
W-7405 eng-45	Mar. 27, 1943	June 1943	Soda salt	4.3	84	3.6	13,800
P. O. 3220	Mar. 26, 1943	April 1943	Uranyl nitrate	0.5	60	0.3	2,700
<b>Boris Pregel</b>							
W-7405 eng-44	Mar. 27, 1943	May 1943	Black oxide	2.9	97	2.8	11,200
W-7405 eng-44	Mar. 27, 1943	May 1943	Soda salt	3.3	84	2.7	10,200
W-7405 eng-44	Mar. 27, 1943	May 1943	Uranyl nitrate	2.0	55	1.1	8,200
<b>General Chemical Co.</b>							
P. O. 42615	Sept. 7, 1943	Oct. 1943	Uranyl nitrate	0.2	50	0.1	900
<b>Linds</b>							
W-7401 eng-15	Oct. 17, 1942	Dec. 1942-July 1943	Black oxide	43	85	41	176,400
W-17-028 eng-29	May 4, 1943	Aug. 1943	Ore concentrates	23	12	2.3	5,800
<b>TOTAL</b>				<b>339</b>	<b>270</b>		<b>\$1,056,130</b>
<b>WASHINGTON OFFICE PROCUREMENT - MISCELLANEOUS</b>				<b>Soda Salt</b>		<b>481 (capture)</b>	<b>(Net available)</b>



~~SECRET~~RADIOACTIVE LEAD PROCUREMENT SUMMARY

<u>Contract Number</u>	<u>Date of Contract</u>	<u>Tons of Lead Oxide</u>	<u>% Lead Content</u>	<u>Cost per Pound of Elemental Lead</u>	<u>TOTAL COST</u>
Boris Pregel					
W-7405 eng-51	June 1, 1943	3.5	80	\$1.00	\$ 7,000.
W-7405 eng-266*	May 10, 1944	9.5	67	1.00	19,000.
Dept. of Munitions and Supplies					
W-44-153 eng-1	Apr. 5, 1945	33	80	0.54	28,100.
Gilbert A. LaBine, Agt.					
W-26-021 eng-14	May 1, 1945	39	82	0.54	54,300*
		—			—
	TOTAL	85			\$88,400

\*Now included in African Metals Contract W-7405 eng-24

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RADIUM AND RADIUM-NEUTRON SOURCE PROCUREMENT SUMMARY

<u>CONTRACT NUMBER</u>	<u>DATE OF CONTRACT</u>	<u>TYPE OF CONTRACT</u>	<u>MILLIGRAMS OF RADIUM</u>	<u>COST TO 12/31/46</u>
<b>Boris Pregel</b>				
W-7405 eng-33	28 Jan. 1943	Rental	3,021	\$ 25,625
W-7405 eng-297	9 May 1944	Rental	1,000	4,549
W-7405 eng-313	1 Sept. 1944	Rental	5,595	51,718
W-7405 eng-35	24 Feb. 1943	Purchase	4,941	83,997
W-7405 eng-91	1 June 1943	Purchase	2,021	34,857
W-7405 eng-148	22 July 1943	Purchase	1,001	17,018
W-7405 eng-229	1 Nov. 1943	Purchase	2,002	34,036
W-7405 eng-286	14 Feb. 1944	Purchase	2,638	44,852
W-7405 eng-291	30 Mar. 1944	Purchase	2,472	42,015
P. O. A-25006	5 Feb. 1942	Rental	1,000	7,900
P. O. A-29228	30 Mar. 1942	Rental	180	100
15 Miscellaneous Purchase Orders 1942-1943-1944			721	10,800
<b>Eldorado Mining &amp; Refining Co.</b>				
W-26-021 eng-30	29 May 1946	Rental	2,584	2,077
<b>Joseph A. Kelly, Agent</b>				
W-7412 eng-155	27 Nov. 1943	Purchase	2,002	30,031
W-7412 eng-157	28 Mar. 1944	Purchase	57	1,496
W-7412 eng-159	12 Apr. 1944	Purchase	1,004	15,308
W-7412 eng-160	10 May 1944	Purchase	10,078	152,949
W-38-094- eng-16	23 Oct. 1944	Purchase	1,126	16,891
W-38-094 eng-18	20 Feb. 1945	Purchase	5,848	87,720
W-38-094 eng-19	20 May 1945	Rental	5,000	7,125
W-38-094 eng-22	20 June 1945	Rental	5,000	6,750
W-38-094 eng-23	29 June 1945	Rental	5,000	6,375
W-38-094 eng-24	7 Aug. 1945	Rental	200	2,400
P. O. A-25594	12 Feb. 1942	Rental	202	400
P. O. #8313	15 Dec. 1943	Purchase	50	1,259
P. O. 8814	15 Dec. 1943	Purchase	10	257
P. O. 12659	10 May 1944	Purchase	1	26
P. O. 12490	1 May 1944	Purchase	51	1,358
P. O. 12732	15 May 1944	Purchase	4	174
P. O. 27228	17 June 1944	Purchase	2	187
P. O. 24441	30 June 1944	Purchase	5	120
P. O. 27695	19 July 1944	Purchase	20	465
P. O. 11807	15 Feb. 1944	Rental	100	100
P. O. 28422	6 Sept. 1944	Purchase	101	2,051
P. O. 28421	6 Sept. 1944	Purchase	6	198
P. O. 28420	6 Sept. 1944	Purchase	17	445
P. O. 29099	30 Oct. 1944	Rental	250	50
P. O. 29628	30 Nov. 1944	Rental		50

<u>CONTRACT NUMBER</u>	<u>DATE OF CONTRACT</u>	<u>TYPE OF CONTRACT</u>	<u>MILLIGRAMS OF RADIUM</u>	<u>COST TO 12/31/46</u>
Joseph A. Kelly, Agent (continued)				
P. O. 29386	17 Nov. 1944	Purchase	20	\$ 488
P. O. 29100	30 Oct. 1944	Purchase	11	271
P. O. 28141	21 Aug. 1944	Rental	-	100
P. O. 35079	28 Jan. 1945	Purchase	12	281
P. O. 29979	18 Jan. 1945	Purchase	11	372
P. O. 35232	5 May 1945	Purchase	1	33
P. O. 35432	1 June 1945	Purchase	40	925
P. O. 35737	21 Mar. 1945	Rental	-	100
P. O. 35620	25 June 1945	Purchase	100	1,994
P. O. 42535	23 Aug. 1945	Rental	-	100
Radium Chemical Co.				
W-38-094 eng-25	29 Jan. 1946	Rental	200	135
W-38-094 eng-28	15 Aug. 1946	Rental	1,000	500
W-38-094 eng-30	1 Oct. 1946	Rental	6,211	1,090
P. O. 7662	11 Aug. 1945	Rental	100	100
TOTAL RENTALS			56,613	97,092
TOTAL PURCHASES			58,375	582,307
GRAND TOTAL			72,988	\$679,399

REFINING OF ORES AND CONCENTRATES

To 1 January 194<sup>7</sup><sub>6</sub>

<u>Contract Number</u>	<u>Date of Contract</u>	<u>% U<sub>3</sub>O<sub>8</sub> in Ore (Approx.)</u>	<u>Total Tons U<sub>3</sub>O<sub>8</sub> in Ore (Approx.)</u>	<u>Tons U<sub>3</sub>O<sub>8</sub> Recovered To Date</u>	<u>U<sub>3</sub>O<sub>8</sub> Refining Cost To Date</u>	<u>Other Refining Costs</u>	<u>TOTAL</u>	
<b>ELDORADO CONTRACTS:</b>								
W-7405 eng-6	10/21/42	8	73	68	\$ 83,900	\$ --	\$ 83,900	
W-7405 eng-17	10/17/42	25	1,251	1,164	1,418,600	--	1,418,600	
W-7405 eng-20	11/24/42	82	48	48	37,500	--	37,500	
W-7405 eng-264	10/25/43	20	168	160	392,090	231,170	623,260	
W-7405 eng-281	1/15/44	20	54	52	127,450	--	127,450	
W-7405 eng-281	1/15/44	50	111	105	126,820	18,800	145,620	
W-7405 eng-318	9/1/44	20	76	71	116,210	--	116,210	
W-7405 eng-318	9/1/44	26	74	69	106,210	34,220	140,430	
W-26-021 eng-21	7/30/45	20	29	27	44,320	10,560	54,880	
W-26-021 eng-26		65	75	70	75,460	--	75,460	
African Ore			1,969	1,832	\$2,528,560	\$ 294,750	\$2,823,310	
W-7405 eng-145	5/22/43	30	222	687	\$ 829,360	\$ --	\$ 829,360	
W-7405 eng-252	9/11/43	30	532					
W-26-021 eng-6*		30	344	20	40,000	--	40,000	
Purchase Order 135		7/15/42	30	172	140	237,600	--	237,600
Canadian Ore			1,270	862 847	\$1,106,960	--	\$1,106,960	
ELDORADO TOTAL			3,229	2,895 2,619	\$3,635,520	\$294,750	\$3,930,270	
<b>VITRO CONTRACTS:</b>								
W-7405 eng-21	12/24/42	65	66	63	\$ 83,500	\$ --	\$ 83,500	
W-7405 eng-251	9/1/43	65	393	379	498,900	--	498,900	
W-26-021 eng-16	5/15/45	65	164	158	199,500	--	199,500	
African Ore			623	600	\$ 781,900	\$ --	\$ 781,900	
W-7405 eng-54	4/20/43	38	3	3	4,500	--	4,500	
W-26-021 eng-7	1/1/45	50	144	141	218,900	--	218,900	
W-26-021 eng-24			26	24	198,130	--	198,130	
Western Ore			173	168	\$ 421,530	\$ --	\$ 421,530	
VITRO TOTAL			796	768	\$1,203,430	\$ --	\$1,203,430	
<b>U. S. VANADIUM CONTRACT:</b>								
W-7405 eng-32	1/25/43		675	(831A)	\$4,871,140**	\$238,390**	\$5,109,530**	

SUMMARY

REFINING OF ORES AND CONCENTRATES

TO 1 JANUARY 1946<sup>7</sup>

(Continued)

<u>Contract Number</u>	<u>Date of Contract</u>	<u>% U<sub>3</sub>O<sub>8</sub> in Ore (Approx.)</u>	<u>Total Tons U<sub>3</sub>O<sub>8</sub> in Ore (Approx.)</u>	<u>Tons U<sub>3</sub>O<sub>8</sub> Recovered To Date</u>	<u>U<sub>3</sub>O<sub>8</sub> Refining Cost To Date</u>	<u>Other Refining Cost</u>	<u>TOTAL</u>
LINDE CONTRACT: W-7401 eng-14 African Ore Western Ore Reprocessing Residues	11/16/42 11/16/42		1,433	1,338 ) 964 ; 128 )	\$5,074,260**	\$ -	\$5,074,260**
			6,333	5,891 5,875	\$14,784,350	\$533,140	\$15,317,490

\*Deliveries of refined product not complete

\*\*Includes cost of Government-furnished materials

(A) Western ore was concentrated under this contract and delivered to Linde for refining

## REFINING AND TREATMENT CONTRACT DATA

TO JANUARY 1, 1947

<u>Contract</u>	<u>Operation</u>	<u>Effective Date</u>	<u>Construction Cost</u>	<u>Operating Cost*</u>
Badger, E.B. & Sons W-44-153 eng-7	Construction of Mallinckrodt Refinery	May 28, 1945	\$5,698,360	\$ --
Brush Laboratories W-7405 eng-3	Research - Metal	October 14, 1942	--	85,000
E.I. du Pont de Nemours & Co. W-7412 eng-3	Brown Oxide Green Salt Metal	November 20, 1942 ) : )	1,050,000	1,891,050 869,170 800,150
W-7412 eng-5	Standby & Eqpt. storage	July 29, 1942	--	19,650
W-7412 eng-151	Green Salt Hexafluoride Research and Production**	September 5, 1944	20,130	320,000 38,760
W-7412 eng-22	Research - C-105 series Research - Scrap Recovery Scrap Recovery	December 30, 1942	-- -- 842,280	59,440 139,410 2,294,150
Eldorado Mining & Refining Co. W-7405 eng-6	Refining	October 21, 1942	--	83,900
W-7405 eng-17	Refining	November 17, 1942	--	1,418,600
W-7405 eng-20	Refining	November 24, 1942	--	37,500
W-26-021 eng-6	Refining	December 1, 1944	--	40,000
W-26-021 eng-26	Refining	January 15, 1946	--	75,460
Electro Metallurgical Co. W-7405 eng-14	Metal Research and Development Standby	November 4, 1942	234,500	2,132,900 34,100 26,250
W-7405 eng-227	Plant extension	March 1, 1943	10,400	--
W-7405 eng-255	Plant design and engineering installation	March 1, 1943	2,600	--
Evans, Robley D W-7409 eng-20	Analytical services	December 27, 1943	--	1,800
W-22-075 eng-13	Ra assay	May 15, 1946	--	480
General Chemical Co. W-7405 eng-315	Anhydrous hydrofluoric Acid	September 1, 1944	--	42,900

\*Includes Research and Development and Quality Control Costs

\*\*Includes costs incurred under W-7412 eng-10 and OE MSR 409 which were incorporated into W-7412 eng-151

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REFINING AND TREATMENT CONTRACT DATA

<u>Contract</u>	<u>Operation</u>	<u>Effective Date</u>	<u>Construction Cost</u>	<u>Operating Cost*</u>
Grove, Shepherd, Wilson & Kruge W-14-108 eng-17	Chemical Storage tank for Mallinckrodt Refinery	March 5, 1946	\$ 46,000	\$ --
Harshaw Chemical Co. W-7405 eng-2	Green Salt	September 1, 1942	--	634,630
	Oxyfluoride		--	2,270
W-7405 eng-43	Hexafluoride	March 18, 1943	--	77,300
W-7405 eng-37	Tetra chloride	February 25, 1943	--	27,300
W-7405 eng-276	Green Salt	January 5, 1944	--	795,730
	Hexa fluoride		802,000	2,105,410
W-26-021 eng-4	Tetrachloride	October 18, 1944	--	68,800
Hooker Electrochemical Co. W-7405 eng-28	Slag Recovery	January 4, 1943	42,000	28,400
Iowa State College W-7405 eng-7	Metal ) Recast Metal )	November 9, 1942	35,000	2,524,240
Kinetic Chemicals W-7405 eng-27	Hydrofluoric Acid	November 16, 1943	--	65,000
LaBine, G.A. Agent W-7405 eng-252	Refining	September 11, 1943	--	829,360
W-7405 eng-264	Refining	October 25, 1943	--	623,260
W-7405 eng-281	Refining	January 15, 1944	--	273,070
W-7405 eng-318	Refining	September 1, 1944	--	255,640
W-26-021 eng-21	Refining	July 30, 1945	--	54,880
Linde Air Products Co. W-7401 eng-14	Refining Brown Oxide Green Salt Standby	November 16, 1942	1,759,940 782,160 518,130 --	5,074,260 455,470 1,394,670 368,110
LeDoux and Company W-14-108 eng-18	Sample sludge	April 1, 1946	--	7,720
Mallinckrodt Chemical Works W-7405 eng-13	Plant for green and metal	November 23, 1942	632,400	
W-7405 eng-29	Green Salt	November 23, 1942	--	2,591,120
	Metal		--	2,775,750
W-7405 eng-1	Brown and orange oxide	November 24, 1942	--	4,745,250
W-7405 eng-8	Brown and orange oxide	May 12, 1943	--	835,260
	Research		--	155,000

\*Includes Research and Development and Quality Control Costs

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REFINING AND CONTRACT TREATMENT DATA

<u>Contract</u>	<u>Operation</u>	<u>Effective Date</u>	<u>Construction Cost</u>	<u>Operating Cost*</u>
<b>Massachusetts Institute of Technology</b>				
W-7405 eng-40	Analytical services	April 1, 1943	--	\$ 110,000
W-7405 eng-55	Research	November 1, 1942	--	40,900
W-7405 eng-85	Research	August 12, 1944	--	330,000
<b>Metal Hydrides</b>				
W-7405 eng-8	Metal Recast Metal	November 1, 1942	35,000	670,800 719,660
<b>New England Lime Co.</b>				
W-7415 eng-23	Mg. Stockpile	December 1, 1943	--	9,200
<b>Pitkin, Lucius</b>				
W-7421 eng-16	Sample Ore	May 11, 1944	--	7,900
W-35-058 eng-9	Sample Ore	August 15, 1946	--	7,620
W-35-058 eng-10	Assay Ore	February 1, 1946	--	1,930
<b>Pregel, Boris</b>				
W-7405 eng-31	Process radium-bearing sludge	June 1, 1943	--	104,600
<b>Princeton University</b>				
W-7405 eng-81	Analytical Research	April 15, 1943	--	201,000
<b>Penn Salt Mfg. Co.</b>				
W-7405 eng-80	Hydrofluoric Acid	March 27, 1943	--	471,900
<b>U.S. Vanadium Corp.</b>				
W-7405 eng-32	Yellow Sludge V <sub>2</sub> O <sub>5</sub> Standby	January 25, 1943	1,591,580	4,871,140 238,390 191,250
<b>Vitro Manufacturing Co.</b>				
W-7405 eng-21	Refining	November 24, 1942	--	83,500
W-7405 eng-54	Refining	April 20, 1943	--	4,500
W-7405 eng-251	Refining	September 1, 1943	--	498,900
W-26-021 eng-7	Refining	January 1, 1945	--	218,900
W-26-021 eng-16	Refining	May 15, 1945	--	199,800
W-26-021 eng-24	Refining	February 1, 1945	--	198,150
<b>Westinghouse</b>				
W-7407 eng-2	Metal	August 1, 1942	--	1,599,200
W-7409 eng-31	Thorium Metal	August 7, 1944	--	16,000
W-7407 eng-132	Equipment	December 18, 1943	--	8,200
<b>Yale University</b>				
W-7415 eng-22	Research	December 1, 1943	--	68,490
<b>T O T A L</b>			<b>\$12,082,280</b>	<b>\$48,048,130</b>
<b>TRANSFER OF GOVERNMENT FUNDS</b>				
National Bureau of Standards Analytical Services				514,000
National Bureau of Standards Research				266,000
<b>T O T A L</b>				<b>780,000</b>
<b>GRAND TOTAL</b>				<b>\$48,828,130</b>

\*Includes Research and Development and Quality Control Costs



MISCELLANEOUS CONTRACT DATA

Services and Supplies Incidental to Refining and Treatment

<u>Contract</u>	<u>Operation</u>	<u>Effective Date</u>	<u>Construction Cost</u>	<u>Operating Cost</u>
Alward, Henry W W-23-094 eng-5	Ore storage facilities	May 16, 1945	\$ 116,600	\$ --
Agruss, Meyer S W-44-153 eng-8	Protactinium	June 5, 1945	--	3,500
Baker & Co. W-7401 eng-1 W-7407 eng-4	Platinum-Lined pots Platinum-Lined pots	October 1, 1942 November 1, 1942	-- --	7,000 13,000
Defense Plant Corp. W-22-075 eng-7	Steel drums - Colorado	October 1, 1944	--	12,300
Dunellen Electric Co. W-17-028 eng-31	Electrical Installation Middlesex Warehouse	July 1, 1945	11,400	--
Erie City Iron Works W-7401 eng-178	Boilers for Colorado Sludge Plants	December 2, 1943	10,200	--
General American Transportation Co. W-7401 eng-89 W-7401 eng-84	Rental of tank cars Rental of tank cars	January 3, 1944 September 17, 1943	-- --	100 2,500
Inland Steel Container Co. W-7407 eng-54	Steel drums - Colorado	January 12, 1944	--	5,800
Mechanics Overall Co. W-44-153 eng-4	Supplies - Middlesex Whse.	April 15, 1945	--	2,040
Pan American Engineering Co. W-26-021 eng-13	Consultant	June 1, 1945	--	3,000
Perry Warehouse W-7407 eng-133 W-42-069 eng-7	Warehouse ore - Middlesex Warehouse ore - Middlesex	April 10, 1944 July 1, 1945	-- --	115,400 218,240
Rheem Manufacturing Co. W-7409 eng-25	Steel drums - Colorado	July 10, 1944	--	52,300
Roadway Express W-7412 eng-30	Trucking service	June 1, 1944	--	12,000

MISCELLANEOUS CONTRACT DATA

<u>Contract</u>	<u>Operation</u>	<u>Effective Date</u>	<u>Construction Cost</u>	<u>Operating Cost</u>
Stearns-Rogers W-7407 eng-52 W-35-058 eng-5	Boilers - Colorado Design and Engineering Services	January 27, 1944 October 1, 1944	\$ 16,500 15,200	\$ -- --
Southampton Hauling Co. W-28-094 eng-11	Hauling by-products	April 27, 1945	--	13,100
Transportation Equipment Co. W-7423 eng-20	Trailers for hexafluoride	May 15, 1944	--	4,200
Treecoob - Federal Truck W-7423 eng-21	Trailers for hexafluoride	June 1, 1944	--	11,700
Union Bag & Paper Co. W-7423 eng-17 W-7423 eng-25	Supplies - Middlesex Supplies - Middlesex	April 15, 1944 June 28, 1944	-- --	9,400 13,500
Vanadium Corp. of America W-7405 eng-268 W-26-021 eng-9	Haul tailings Trucking services	April 22, 1944 January 2, 1945	-- --	6,600 40,700
Woods Mercantile, Hugh M W-7423 eng-5	Asphalt felt-covered sheet iron	November 1, 1945	5,800	--
Miscellaneous Service Contracts (Telephone, Gas, Electric, etc.)				349,780
		<b>T O T A L</b>	<b>\$ 175,700</b>	<b>\$ 893,940</b>

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<u>Contract</u>	<u>Material</u>	<u>Date of Contract</u>	<u>Lbs. Delivered</u>	<u>Contract Cost per lb.</u>	<u>TOTAL COST</u>
Lindsay Light & Chemical Co.	Thorium Nitrate	January 21, 1946	9,120	\$1.80	\$16,420
W-17-028 eng-33	Thorium Nitrate, Mantle grade	July 10, 1946	24,130	1.80	43,340
W-17-028 eng-35	Thorium Nitrate, chemically pure		190	4.00	760
		T O T A L	33,440		\$60,520

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APPENDIX G

SPECIAL CHEMICALS FOR K-25

INTRODUCTION

Considerable quantities of fluorinated chemicals were required for the operation of the K-25 plant. In the manufacture of these chemicals, HF and F<sub>2</sub> had to be obtained in considerable quantities. In obtaining elementary fluorine in the amounts required, it was necessary to develop procedures for its generation.

The process gas (C-616) is uranium hexafluoride which is, as the name implies, the working material used in the K-25 process. Since the Manhattan District became responsible for the project, no other material has shown such possibilities that it could be seriously considered as a substitute for C-616 in the process. (This process is covered in detail in Section 9 of this volume).

Details and supplementary information relating to the development and procurement of special chemicals for K-25 may be found in the "Completion Report on the K-25 Gas Diffusion Plant", by the Kellogg Corporation, 1 January 1946 (Reference: Book II, Volume 3), pages 843-869. That report covers most of the activities described in this Appendix, and, in addition, explains more fully the complex reasons for the changes and revisions which affected them. As stated in the Kellogg Report (on page 844): "Special chemicals requirements fluctuated with the evolution of the design of the main cascade and of the process equipment. This complicated an already complex development problem. Fortunately, however, the particular chemicals contemplated for one use were frequently suitable for a number of other services, and so, in general, changes were

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reflected in the quantities required, and occasionally in the specifications. Thus the work which had gone into their development was not wasted."

The fluorocarbons had to include materials which would be suitable for a "sealant", a "dummy gas", a heat transfer medium or coolant, a lubricant or vacuum pump oil and a fluorine resistant grease. The most outstanding attribute that these compounds have to possess is inertness to attack by uranium hexafluoride and elementary fluorine gas. It was apparent that only completely fluorinated hydrocarbon derivatives would possess this inertness and such compounds were accordingly developed for the above uses. Two methods of attack were used; the fluorination of hydrocarbons with silver and cobalt fluorides both in the liquid and vapor phase, and the polymerization of fluorinated olefins. In the latter case, a final treatment with cobalt trifluoride was necessary to obtain a product of sufficient chemical inertness.

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C-216	-	Fluorine
C-2144	-	Fluorinated lube oil
C-2144S	-	Chlorofluorolube oil (from terphenyls)
C-714	-	Mixture of tetradecafluoromethylcyclohexane and tetradecafluoroethylcyclopentane
C-715CL	-	Chloro-pentadecafluoro-heptane
C-716	-	n-perfluoroheptane
C-816	-	Perfluorodimethylcyclohexane
C-816CL	-	Monochloro-pentadeca dimethylcyclohexane
FL	-	Fluorolube
FLS	-	Fluorinated lubricant solvent or fluorinated kerosene
Freon 113	-	Trichloro-trifluoro-ethane
HF	-	Hydrofluoric acid
MFL	-	Polymerized and further fluorinated trifluorochloro-ethylene (liquid)
MFI	-	Polymerized and further fluorinated trifluorochloro-ethylene (grease)
OG	-	Fluorine
P-45	-	Hexafluoroxylenes
P-45CL	-	Monochlor P-45
P-45CL <sub>2</sub>	-	Dichlor P-45
P-539	-	Trifluorochloroethylene

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n-perfluoroheptane (C-716)

C-716 was proposed for specific uses. The first was as a "sealant" in the diffusion plant. This chemical was a liquid which was intended for use in packinands and similar parts under pressure in order to exclude atmosphere which might otherwise leak into the evacuated system containing process gas. This idea of using a fluorocarbon as a "set" was abandoned later in favor of nitrogen which was much cheaper and had only the disadvantage of a lower molecular weight. The second use for C-716 was for a "dummy gas" to fill units which had been aired and were to be placed again in production and for testing the performance of diffusion units. Furthermore, since C-816 production was not expected to be ready for use as a coolant on the test floor, so coolant grade C-716 was manufactured for this requirement. Nevertheless, almost the entire program for the manufacture of C-716 was based on the premise that it would be used as a "sealant".

The essential properties for sealant were the same as those given for the coolant under the section devoted to C-816, except that the volatility of the material has to be more carefully controlled in the case of the sealant. As a result of this carefully controlled vapor pressure specification no organic liquid but normal perfluoroheptane was seriously considered as being capable of filling this sealant requirement. The reason for the more stringent specification for the

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sealant was that this grade material was expected to be bled slowly into the process gas stream through packing glands while the coolant material would not get into the process gas except through leakage. The manufacture of C-716 consisted of fluorination of normal heptane in a vapor phase by passing it over heated cobalt trifluoride. The process for the manufacture of this material had to be developed on the basis of research results at Johns Hopkins and du Pont and the material was manufactured in a plant especially constructed for this purpose. The OSRD had already developed a workable process for the production of C-716 at the time the Manhattan District took over this project, and an OSRD order for 20,000 pounds with the du Pont Company was taken over by the Manhattan District in November 1942 (Ref. 1). The C-716 was procured from the du Pont Company in Wilmington, Delaware, under the cost-plus-fixed-fee contract No. W-7412 eng-2 which was effective on November 17, 1942 and covered construction and operation of a plant for manufacturing this material. This contract was awarded to the du Pont Company since they were at that time the only company in the country which had previous experience in the production of completely fluorinated hydrocarbons of this sort and in addition they had done considerable work on the initial development of this process.

In December 1942, specifications were given to du Pont covering the stability of C-716 process gas (Ref. 2 and 3). It was planned that 20,000 pounds of the material would be produced in two grades;

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3,400 pounds of sealant grade and 16,600 pounds coolant grade for use in place of C-816 on the test floor. It should be noted that the term C-716-1 which appears frequently throughout correspondence and documents refers to this initial order of 20,000 pounds.

In early 1943, it was believed by those working on the program, that the use of synthetic heptane was necessary to obtain a grade of C-716 suitable for use in the plant (Ref. 4), and further work along those lines bore out these opinions. The initial lots of C-716 were showing production costs in excess of \$40 per pound and, as a result, it was necessary to increase the amount of money provided for the manufacture of this product.

In June 1943, it had been definitely determined that the previously considered C-816 was not satisfactory as a sealant material for the large plant and it was, therefore, necessary that the entire sealant requirements would have to be made of C-716. Supplements to the Contract No. W-7412 eng-2 were issued for additional amounts of the C-716 and at the same time additional specifications which were incorporated in the contract were set on the material. Throughout the literature and the documents, this additional order is referred to as C-716-2 and the specifications as C-716-2 specifications. A new Stedman Column had to be installed in order to take care of the C-714 (mixture of tetradecafluoromethylcyclohexane and tetradecafluoroethylcyclopentane) specification limit in the C-716-2 and the cost of improving the process for this reason amounted to \$58,000.

Cost at the conclusion of the production of the initial 20,000 pounds of C-716 was \$700,000 exclusive of the fee. However, du Pont

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emphasized the fact that the price reflected an enormous amount of further development work not only in the process itself but in the generation and handling of fluorine, the preparation of the cobalt trifluoride catalyst and general fluorocarbon process development. Those latter costs would normally have been covered under a research contract. In February 1944 information was received stating that enough C-816 would be available to permit its use as replacement for the C-716 as coolant for the test floor, this resulting in a saving of \$250,000. At this time, it was decided to redistill the C-716-1 of coolant grade which was still held by the du Pont Company through the Stedman Column in order to provide for the recovery of a considerable amount of C-716-2, the sealant grade specification material. This processing of the coolant grade C-716-1 to C-716-2 proceeded so satisfactorily that production of 716 exceeded scheduled requirements and there was no further difficulty connected with the procurement of C-716.

Because of the fluctuating requirements caused by changes in conditions, the production schedules of C-716 were changed at various times throughout the contract. However, under the Contract 20,280 pounds of C-716-1 and 76,755 pounds of C-716-2 were produced. 9,936 pounds of the C-716-2 were produced from the coolant grade C-716-1 which became available when it was not needed for use on the test floor. Production of C-716 ceased on June 25, 1945. Cost of construction under W-7412 eng-2, which covered production of both C-716 and C-2144 (fluorolube) amounted to \$737,290. Under this contract, 20,280 pounds of C-716-1 were produced at a cost of \$656,290. 76,752 pounds of C-716-2 were

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produced, 66,816 pounds at a cost of \$1,492,680, and 9,936 pounds (from C-716-1) at a cost of \$7,800.

It should be mentioned that the 715CL (chloro-pentadecafluoroheptane) process developed at Purdue under W-7405 eng-74 by Professor E. T. McBee could be operated to produce a sufficient amount of C-716 of satisfactory quality to meet the sealant requirement as a by-product of the 715CL coolant. This was an attractive feature of the 715CL process. Undoubtedly this process would have been used had it been possible to develop it in time since the cost of the material as a by-product would have been relatively low in any case and the process was particularly attractive on account of its very low requirements for elementary fluorine.

Perfluorodimethylcyclohexane (C-816) and Hexafluoroxylene (P-45)

C-816 was developed for use as a coolant material in the diffusion plant. The physical properties and an inertness to process gas required for this coolant restricted the choice of practical compounds to fluorinated hydrocarbon derivatives having 7, 8 or 9 carbon atoms (Ref. 6). Of these C-816 proved to be the most satisfactory and the least expensive to manufacture.

When the coolant requirements for the large plant were presented in December 1942 (Ref. 7), it was obvious that the greater proportion of the coolant for the large plant should be manufactured as a cheaper material with lower requirements for fluorine, since the cost of using C-716 (n-perfluoroheptane) would be almost prohibitive in the amounts that would be necessary. C-816 was chosen since considerable

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work had been done on the manufacture of this material at Johns Hopkins and Purdue Universities and at the Hooker Electrochemical Company and the du Pont Company on a semi-plant scale. The process involved the treatment of P-45 (hexafluoroxylyene) with cobalt trifluoride in the vapor phase. C-816 had been the subject of a long report from the Kellogg Company stating that its properties made it satisfactory for use as coolant in the large plant. Accordingly, Contract No. W-7412 eng-6 effective as of December 31, 1942 was executed with the du Pont Company for its manufacture. du Pont was the logical choice for this contract for several reasons. They had had more experience in the field of organic fluorine chemistry than any other company in the country. They were engaged in exclusive work on fluorination for the OSRD before the Manhattan District took over this work. They were engaged on the work for the manufacture of C-716, and in connection with C-716 they had developed considerable fluorine generating capacity and experience in fluorine manufacture. Furthermore, they had done much work on the C-816 process itself. Obviously, the choice of the du Pont Company for this manufacturing program was indicated. The raw material hexafluoroxylyene (P-45) was furnished by the Hooker Electrochemical Company under W-7405 eng-28. Since the du Pont plant would not start to produce until late 1943, there was still a requirement for coolant for the Kellogg pilot plant which it was expected would be manufactured as C-761 since the pilot plant was expected to start operating in the summer of 1943.

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In February 1943, it was decided to use C-716 as a sealant in the large plant because the volatility of C-816 was too low and, although it had been hoped that the cheaper C-816 could be used, with this decision, no further consideration of C-816 as a sealant material was made (Ref. 8). Later in February 1944, it was found that C-816 could be substituted for C-716 as coolant for the test floor, and this resulted in savings of approximately \$250,000 (Ref. 11). During practically all of the period of production of this material requirements varied considerably, (Ref. 8, 9, 10 and 12). These variations in requirements were not conducive to smooth economical operations since it was necessary at times to shut down certain portions of the plant and then start up these same portions again when it was understood that requirements would be increased again.

Early in June 1944, the question of the C-716<sup>4</sup> content of C-816 was raised by the Kellogg Corporation as it had been with the C-716, and Kellogg desired to know the cost of reducing the C-714 content in the C-816 to "not more than 0.5%." du Pont supplied the information that the cost of producing C-816 of such quality would increase the cost by \$257,000 (Ref. 13). The Kellogg Company then formally requested this change in specification to be made and stated that C-816 of the old specification grade already made could be used (Ref. 14). In July 1944, the new specification was issued and the du Pont Company was informed that the change should be made.

In August 1944, the C-2144 (fluorolube) situation was so far behind schedule that two reactors were taken off the C-816 production and used for C-2144 production. Accordingly, the delivery schedules

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for C-816 were set back considerably. As stated previously, requirement schedules were changed almost daily and especially in the latter part of 1944 and early 1945 these changes varied as work proceeded with the construction of various plants which required this material. Early in March 1945, instructions were given for reducing the plant capacity of C-816 in which about 75% of the facilities were to be shut down and placed in standby condition. Shortly afterwards, however, in the latter part of the same month, additional requirements were received and it was necessary to ask du Pont to increase production rates. In early April 1945, it was requested that C-816 be made at the rate of 150,000 pounds per month and at the same time it was thought that additional C-2144 would be necessary. However, requirements changed so that the C-2144 process was shut down entirely a little more than a month later. In general, increased production of C-816 seemed to be necessary as the picture appeared in April. However, by the middle of the year production requirements were again lowered somewhat. In September, a letter of intent was sent to du Pont with the information that production of C-816 would be stopped as of the 25th day of December 1945 and certain facilities would be placed in standby with the excess declared surplus. It was planned to place enough equipment in standby condition to produce 140,000 pounds per month of C-816 and 1,000 pounds per month of C-2144.

Production was completed, as planned, on December 25, 1945. Cost of construction under W-7412 eng-6, which covered production of C-2144 as well as C-816, amounted to \$9,101,260. 3,888,219 pounds of C-816 were produced under this contract; 371,290 pounds at an average cost

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of \$4.94 per pound, and the remainder of 3,516,929 pounds at \$1.80 per pound. In addition there was also produced at an average price of \$1.82 per pound 286,461 pounds of C-816CL (monochloro-pentadeca dimethylcyclohexane). Operating costs for the C-816 process totaled \$8,673,830. At this time the portion of the facilities as stated above were placed in standby and procedures were instituted to dispose of the excess and after 25 March 1946 disposal of the entire plant was begun.

du Pont made an offer for the entire plant and this offer was ultimately raised to \$186,000 in early November 1946. This offer took into account estimated dismantlement costs of \$500,000 which du Pont would absorb if the offer were accepted. However, du Pont withdrew their offer entirely as of 31 December 1946 because of changing business and economic conditions and the plant is now being disposed of through normal channels.

Hexafluoroxylene (P-45), the raw material from which C-816 is made, was produced by the Hooker Electrochemical Company under Contract No. W-7405 eng-28 effective 4 January 1943. A plant was constructed for its manufacture which consisted of the chlorination of xylene to hexachloroxylene followed by the replacement of the chlorine by fluorine on treatment with hydrogen fluoride under pressure. The fact that the compound could be manufactured without the use of elementary fluorine was an important factor resulting in the selection of C-816 as a coolant. Considerable work had been done on P-45 at Hooker under OSRD Contract No. OEMsr-811 and this work was continued under the Manhattan District with Contract No. W-7405 eng-76, effective 1 April 1943, for research on fluorocarbons.

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When the C-816 process was adopted for the coolant for the large plant in December 1942, it was agreed that the intermediate P-45 should be obtained from Hooker. This was logical since all development work on this process had been done by Hooker and they had moreover a great deal of experience with the manufacture of this type of product, particularly the intermediate chloro compounds. Considerable work was done in the spring of 1943 in the development of the P-45 process and tentative specifications for the material were developed at a meeting at du Pont in March 1943 (Ref. 15). The delivery schedule increased very considerably during the early months, 400,000 pounds on the original order being later increased to 1,620,000 pounds to be delivered by November 1944 (Ref. 16, 17, 18).

In August 1943, du Pont changed the specifications for the P-45 (Ref. 19). At this time, conferences were held in which the difficulty with the P-45 quality were discussed and the question was raised of the affect of the quality of the xylene raw material upon the difficulties Hooker was having in meeting the P-45 specifications. As a result of research it was decided to try better xylene and a new grade was ordered from the Donner, Hanner Coke Company, who reported that this xylene could be furnished in the necessary quantity with appropriate priorities assistance. At this time, the initial P-45 requirements were set back from November to December 1943 (Ref. 20). Also the details of the xylene costs under the new specifications were submitted since this new grade of xylene satisfactorily took care of the troubles with the P-45 specification (Ref. 21).



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In August 1944, Hooker was asked to reduce the rate of production of P-45 (Ref. 22). This was done since the P-45 production program was far ahead of schedule and far ahead of the requirements of the du Pont Company. This action enabled the Hooker Company to devote their energies to the production of the experimental order of MFL. During the manufacturing process, there were considerable amounts of residues which were set aside and stored. As tests indicated that some chlorine could be tolerated in the molecule, it was requested that Hooker recover P-45CL (Monochlor P-45) and P-45CL<sub>2</sub> (Dichlor P-45) from these residues. P-45CL was recovered from the residues from the P-45 production and shipped during the life of the contract. The residues from the P-45CL recovery were again stored. Regular production of P-45 ended in October 1945 and at the end of this production all P-45CL and P-45CL<sub>2</sub> was recovered from the remaining residues and was shipped in November. The entire plant was then placed in stand<sub>A</sub>by for six months. Earlier, in March 1945, the plant had been placed in stand<sub>A</sub>by but less than a month later more production was found to be necessary and it was started up again in April, and production then continued until the final shutdown in October 1945, as noted above.

Cost of construction of the plant for the manufacture of P-45 under W-7405 eng-29 amounted to \$1,533,000. This plant was also used for the production of MFL and MFI and for the unrelated function of slag recovery from metal operations of the Madison Square Area (which necessitated an additional \$42,000 of construction costs). During the life of this contract, 2,729,846 pounds of P-45 were produced at an

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operating cost of \$3,324,860, or \$1.22 per pound. In addition, 168,350 pounds of P-45CL were processed at a cost of \$30,490, or \$0.18 per pound, and 69,790 pounds of P-45CL-2 at an operating cost of \$9,480, or \$0.14 per pound. Total operating cost for the P-45 process amounted to \$3,364,830.

A certain amount of the equipment under Contract No. W-7405 eng-28 had been set aside for further MPL production and under a supplement to this contract, standby and further construction has been arranged. However, Hooker has submitted a bid to the North Atlantic Division for the remainder of the plant under this contract and negotiations are now in process for the acceptance of Hooker's bid for the plant.

Fluorolube - G-2144, FL, PLS, MPL, MPI and G-2144B

As mentioned previously, a fluorinated lubricant had to be developed to meet the requirements for a lubricant with vapor pressure and viscosity characteristics such as to make it satisfactory as a vacuum pump oil. It was necessary to have a satisfactory stability to process gas as indicated by failure to form a residue with this material, nor should this lubricant be too rapidly decomposed by fluorine in concentrations up to 20%. Such properties as these are possessed only by fluorocarbons containing about 20 carbon atoms per molecule. The code name G-2144 applied to the fluorolube was derived from the fact that it was assumed a satisfactory product would consist of molecules having an average composition of 21 carbon atoms and 44 fluorine atoms. This material as well as other materials for this purpose were frequently referred to as "FL" for fluorolube. It was later found that a certain amount of chlorine can

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be tolerated in the molecule and that within certain limits such a chlorine content does not adversely affect the requirements for stability towards process gas and fluorine.

No fluorinated material with these characteristics had ever been prepared even in the laboratory when the Manhattan District started work on this problem. Research on the preparation of such a material had been carried out under OSRD contracts at Columbia and Johns Hopkins Universities and at the du Pont Company's Jackson Laboratory before the Manhattan District took over the work. However, none of the processes investigated had really worked satisfactorily at that time. The requirements for fluorolube were slow in being worked out since it was known from the research work that the material would be extremely hard to make and very expensive. Since requirements had to be kept at a minimum, the Kellogg Company did considerable work on obtaining pumps which would have a minimum requirement for the oil and they were attempting to design the plant in such a way as to use a minimum number of such pumps. For these reasons, the requirements listed up to December 1942 gave no quantitative estimates, but stated only that there would be such requirements at a future date.

On the basis of these statements, it was provided in Contract No. W-7412 eng-6, effective 31 December 1942, with the du Pont Company that they would build a plant using a process developed by them. This process involved the interaction of elementary fluorine

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and lubricating oil in the vapor phase in the presence of a silver plated copper gauze catalyst. du Pont was chosen for this work since they had done all the basic research work on this process which was the most promising to date. Obviously, there could be no other choice for this contract. However, it may be stated here that this process was never satisfactorily worked out since explosions and burn outs in the plant were a constant source of difficulty.

The first requirements were received in June 1943 for the Kellogg Test Floor and for the pump manufacturer's testing program (Ref. 23). At the time these requirements were received the production of the lubricant on a large scale had not yet been worked out. In June 1943, a meeting was held at du Pont discussing the problems involved. The only operating units for FL preparation were these at the Jackson Laboratory using the vapor phase process. This small plant had a capacity of 4.3 gallons per month of FL and 4.3 gallons per month of the so-called fluorinated lubricant solvent (FLS), the FLS being a fluorinated hydrocarbon in the kerosene range necessary for dissolving FL in the manufacturing process. There was also a small pilot plant at Johns Hopkins which had been set up under Contract No. W-7401 eng-43 with a capacity of 3 gallons per month of FL and 3 gallons per month of FLS. This plant was not in operation. To meet the Kellogg requirements, it was decided at the meeting to do the following: (a) Discontinue the vapor phase process work under Contract No. W-7412 eng-6 and construct instead a plant with a capacity of 170 pounds per day using a liquid phase process which had been developed at du Pont involving

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the reaction of the oil with silver difluoride in a stirred reactor, (b) double the capacity at Johns Hopkins University. This material was to be sent to Jackson Laboratory in a crude state where it was to be fractionated under Contract No. W-7412 eng-7 (later incorporated into W-7412 eng-151), (c) put in a process under Contract No. W-7412 eng-2 at du Pont for the manufacture of 35 gallons per month FL by a liquid phase process similar to the one manufactured above, the plant to be ready by October 1943, and (d) continue the Jackson Laboratory operations at the present capacity.

du Pont stated they felt that the arrangements above would enable them to meet requirements satisfactorily. Shortly after the middle of June, Kellogg furnished the specifications which had been decided upon for the fluorinated lubricant (Ref. 24).

Production of the FL failed to meet expectations. The liquid phase program gave a great deal of difficulty since the FL obtained by the process as it had been worked out at that time did not meet the stability specifications. Also there was a great deal of unexpected trouble due to the fact that numerous reactors used in the liquid phase process burned out in an unpredictable manner, thereby causing very great delays in production. Only the pilot plants at Johns Hopkins and the Jackson Laboratory could be counted on for any production. In addition to this, in late September, new requirements were received from Kellogg. They were 67 gallons (1120 pounds) by February 1944 for the Test Floor and other test purposes and 640 gallons (10,700 pounds) by February 15, 1945, for the large plant.

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As compared to these requirements during the month of October, 36.2 pounds of FL and 192.6 pounds of FLS were produced, making a total production up to November 1, 1943 of only 93.1 pounds of FL and 357.4 pounds of FLS. It is obvious that at this date the situation with respect to fluorelube was most critical since no large scale process had been put into operation which could be expected to handle the 640 gallons requirement. However, research was continuing constantly on these processes in the hopes of developing satisfactory procedures and at this time, du Pont was doing considerable work on the use of a new raw material, polychloroterphenyl, which was hoped would be a more satisfactory raw material than the hydrocarbon lubricant oil then in use. In late December 1943, Dr. Rosen stated that the Test Floor requirements might be reduced slightly if absolutely necessary but that the requirements for the large plant could not be reduced. After a series of meetings, new contractual commitments were made with du Pont to provide for (a) a plant with a capacity of 45 pounds per day using the vapor phase process, (b) a plant with 22 pounds per day capacity using a liquid phase process where the catalyst was regenerated in a separate vessel in the hopes that burn outs could be minimized or eliminated, (c) a plant similar to (b) with a capacity of 44 pounds per day.

The Johns Hopkins Laboratory program was to continue at its present rate and as of this date they had produced 280 pounds of fluorelube. Expenditures under Contract W-7405 eng-43 with Johns Hopkins University for this work and certain other studies in fluorine aggregated \$628,000.

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About the middle of January 1944, a revised requirement schedule was received reducing the requirement somewhat, but it was agreed that even with these requirements it was still a matter of necessity to attempt to use the du Pont vapor phase process again. Meetings were held almost constantly in attempting to lay down requirements and develop means of increasing production. In early February, FL requirements for the large plant were increased (Ref. 25). Accordingly, arrangements were made to use two of the C-816 vapor phase reactors units for the production of FL by the vapor phase process. The method used for this production was essentially that using cobalt trifluoride which had been satisfactorily used on the pilot plant scale at Johns Hopkins.

In March 1944, a first description of a new fluorelube process which had been developed by Dr. Miller of Columbia was made (Ref. 26). It was this process and material which was ultimately used by the Hooker Electrochemical Company and is covered hereafter.

On <sup>15</sup> March 15, 1944, the du Pont vapor phase process under W-7412 eng-6 was finally dropped on account of extreme operating difficulties. In early April, there was an ample supply of FLS on hand and Johns Hopkins was instructed to modify the pilot plant process to produce as much FL as possible which would then be distilled at the Jackson Laboratory (Ref. 27). In August 1944, (Ref. 28 and 30), Dr. Rosen stated that C-2144S was satisfactory for use as a substitute for C-2144 but that it should not be mixed with C-2144 to an extent of more than 2%. C-2144S was the material prepared from polychloroterphenyl.

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Meanwhile work was proceeding satisfactorily on the production of MFL at Hooker, and initial figures indicated MFL to be cheaper than the other fluorolubes on the basis of Hooker estimates and du Pont production costs (Ref. 29). By early October, Hooker had produced some 1500 pounds of MFL without undue difficulty. This raised the total FL production almost to requirements, this FL production being both C-2144 and MFL combined figures. It was now possible to discontinue the uneconomical operations at Johns Hopkins which had been necessary during the period of critical emergency in the FL production program. Such production was finally discontinued as of November 22, 1944. Also it was decided to return the two C-816 vapor phase reactors to the C-816 production program at du Pont.

During the latter part of 1944 and early 1945, requirements on C-2144 changed considerably in that commitments were not firm and the requirements would fluctuate almost from day to day. However, production of the liquid phase C-2144 was continuously carried out. Due in part to changes in requirements and to the satisfactory progress being made with MFL it was decided in the middle of May that no C-2144 would be made after July 1, 1945. However, total FL production was in excess of requirements and as a result of further conferences, a letter was written on <sup>21</sup>May 21, 1945 stopping the crude feed to the C-2144 process with the instructions that only the crude then in process would be made into finished material. Also, instructions were given to place the facilities in standby condition. The overall production of C-2144 by du Pont under all its contracts was 30,513 pounds.



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Under Contract W-7412 eng-2 with E. I. du Pont de Nemours & Co. 8,216 pounds of C-2144 were produced at a cost of \$783,810, or an average cost of \$95.40 per pound. Under Contract W-7412 eng-6, 15,447 pounds were produced at a cost of \$893,530, or \$57.83 per pound. Under Contract W-7412 eng-151 with the du Pont Company, 6,850 pounds of C-2144 and 728 pounds of DXZ (silver difluoride) were produced at a cost of \$211,070.

In March 1944, the first description of a new fluorolube process which had been developed by Dr. Miller at Columbia was referred to (Ref. 26). The process consisted of polymerization of P-539 (trifluorochloroethylene) (see Paragraph on P-539), page G.28 using a peroxide catalyst. The polymer was fractionated and the lubricating oil fraction stabilized by further treatment in the liquid phase. The special fractions were carried out at Distillation Products, Inc., Rochester, New York. By the end of July 1944, Dr. Rosen had determined and so stated that the oil developed by Dr. Miller's research would be satisfactory for use as a substitute or alternate for C-2144 (Ref. 31). Also at this time, it was recommended that the production of this material, MFL, be carried out at the Hooker Electrochemical Company. du Pont was not interested in taking on any more work since they were completely occupied with the C-2144 program. Hooker was chosen for this program because they had the equipment available, the supervision and the manpower to do the work, were already under contract for a variety of other work, had considerable "know-how" in the general program and there would be no new security problems. Therefore, in

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August 1944, a supplement to Hooker Contract W-7405 eng-75 was initiated calling for the production of polymerized fluorolube (MFL) at a cost of \$125,000, which figure included the cost of research and development. At this time, the production of C-2144 at du Pont was considerably behind schedule. Shortly after the initiation of this supplement with Hooker, official approval of MFL as a substitute for C-2144 was received.

In October 1944, Hooker informed this office that they were willing to undertake the production of an additional quantity of MFL under Contract No. W-7405 Eng-28 (Ref. 32). Since firm requirements for the large plant had not been received, the quantity contracted for was large in order to have a factor of safety. In the latter part of October 1944, a survey was made of the various fluorolube processes with the object of attempting to decide what would be the most economical one to use for the requirements. At this time, the cost estimates for the various processes were not sufficiently firm to make any final decision, but the cost shown for MFL was low enough in comparison with the others that it seemed obvious that this process would be much less expensive. The cost actually shown was very much below the C-2144 cost but it was believed that these would be subject to revision as production proceeded. Moreover, on the basis of information available at this time the quality of MFL was shown to be at least equal to that of C-2144 and probably superior to it.

Early in 1945, the production of MFL was extended (Ref. 33), and production continued without trouble. All during the time MFL was being

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produced considerable research was in progress on recovery of MFL from the residues by cracking, and on the preparation of MFI, a heavy vaseline-like grease obtained from the residues (Ref. 34). A considerable amount of acceptable materials was thus obtained.

In a series of conferences, it was shown that overall production of all types of fluorolubes was exceeding schedules and the fact was brought out that it appeared very probable that consumption would not be as high as primary figures indicated because of improved operation and recovery by the users. Accordingly, MFL production was stopped as of <sup>31</sup>May 31, 1945 (Ref. 35 and 36), and only the material in process was finished. The MFL from a considerable amount of residues was recovered as well as some more MFI after the cessation of regular operation. The plant was then placed in standby.

The MFL plant was maintained in standby with the P-45 portion of the plant and this standby period expired on <sup>30</sup>April 30, 1945. At this time enough equipment for future MFL production thought necessary was segregated and placed in standby while the remaining equipment for all products manufactured under Contract No. W-7405 eng-28 was declared surplus (Ref. 37). It was planned to terminate W-7405 eng-28 as of April 30, 1946 and to execute a new contract to take care of the MFL standby start-up and production after that date. This new contract was prepared but later <sup>because of</sup> ~~due to~~ the changeover from the Corps of Engineers to the Atomic Energy Commission it was decided that it would be more suitable to supplement W-7405 eng-28 rather than issue a new contract, and a new supplement was issued accordingly. Originally, start-up of

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these MFL standby facilities was planned for July 1947, but later consumption of MFL indicated that the plant should remain in standby until July 1948 and then start production. The supplement was issued to cover these revised requirement dates.

The entire remainder of the plant operated under W-7405 eng-28 is in the process of disposal. Hooker, having entered a bid on the facilities, is now negotiating with the proper authorities.

Cost of research on MFL under Contract No. W-7405 eng-75 with the Hooker Electrochemical Company totaled \$121,010. Operating costs from the MFL and MFI process under W-7405 eng-28 totaled \$670,680. 21,710 pounds of MFL and 559 pounds of MFI were produced under this contract. Converting MFI to MFL (559 pounds multiplied by a conversion factor of 2.5) gives 1,398 pounds as MFL, and this amount added to the 21,710 pounds above yields a total production as MFL of  $23,108$  ~~23,269~~ pounds. Thus the unit price per pound of MFL produced was  $\$29.02$  ~~\$30.11~~.

Fluorine (G-216, OG) and Hydrofluoric Acid (HF)

The bulk of this gas was used by Madison Square Area contractors engaged in the manufacture of special materials and at the diffusion plant site for conditioning purposes. These requirements were filled by electrolytic generation at the point of consumption. However, a certain amount of packaged fluorine was supplied by the Madison Square Area to off-site users in nickel cylinders under 400 pounds pressure. This gas was used for research and development purposes and for conditioning operations at locations where certain equipment was being fabricated.

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When development work on the project was begun by the OSRD, elementary fluorine was a scientific curiosity which had been prepared only on a laboratory scale. A good deal of work was done in developing satisfactory electrolytic cells for the generation of fluorine by electrolysis of potassium fluoride, hydrofluoric acid electrolyte. This development work was carried out cooperatively at the du Pont Company, W-7405 eng-151, at the Hooker Electrochemical Company, W-7405 eng-76, at Johns Hopkins University, W-7401 eng-43, and at the Harshaw Chemical Company, W-7405 eng-43. Frequent conferences were held to coordinate the results of research at all installations. By the time the U. S. Engineer Department took over this work, the du Pont Company had already progressed well in the development of a cell capable of producing fluorine on a large scale. However, this cell had two outstanding disadvantages which were (1) the yield of fluorine was less than 70% of the theoretical based on the electrical current consumed, and (2) there was a very great consumption of nickel anodes, which were the type being used in an amount corresponding to 1/6th of a pound of nickel per pound of fluorine gas produced. This work was carried out at du Pont under W-7412 eng-151.

Since the Hooker Electrochemical Company had developed an outstanding chlorine generation process and was known to have in its employ a staff with outstanding knowledge of electrolytic processes, it was felt desirable to get the benefit of their experience in the work for the project. Contract No. W-7405 eng-76, effective <sup>A</sup>(April 1), 1943, was signed with the Hooker Company calling for research and development

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work on the production of fluorine on a large scale. This work was a continuation of work done under OSRD. Work on this project at Hooker was very successful in that they developed a production cell which gave a yield of fluorine based on electrical current consumption of better than 90% and these cells used carbon anodes, thereby eliminating the loss of nickel in the anodes which was obtained in the du Pont cell.

Harshaw's development work on cells followed greatly along the line of Hooker's experience. Harshaw found that the addition of a small amount of lithium fluoride to the electrolyte eliminated a great deal of the trouble with polarization and lack of wetting of the carbon anodes.

Large scale generation of fluorine was carried out at the above companies under the various contracts involving its use. The preferred cell used was the carbon anode cell, and the nickel anode cells originally installed at du Pont were gradually changed over to the preferred type.

In addition to the work done at these industrial concerns, laboratory-scale work was done at Johns Hopkins University under W-7401 eng-43 and at Massachusetts Institute of Technology under W-7401 eng-288.

It is interesting to note that under this program, in a comparatively short time, the generation of fluorine developed from that of a curiosity to large scale industrial production. Also the first cells constructed gave only a comparatively few hours of cell life while, at the present, it is estimated the life of cells with specially-hardened carbon anodes will run to almost 5,000,000 hours.

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Work on packaging elementary fluorine in cylinders was carried out at du Pont and Hooker. It was found at du Pont that the most satisfactory method of packaging fluorine at 400 pounds per square inch pressure was by liquefaction of the fluorine gas with liquid nitrogen and evaporation into the nickel cylinder at desired pressure. For lower pressures, a satisfactory diaphragm pump was developed at Hooker. Procurement of the packaged fluorine at 400 pounds pressure was covered by the du Pont Contract W-7412 eng-151 under which 1,797 pounds of C-216 was obtained.

HF is one of the most vital chemicals to the Manhattan District operations as they are set up. It is necessary for the production of metal, of the process gas C-616 and the various fluorinated organic chemicals, all of which are necessary for the operation of the K-25 process. The quantity of HF required by the District has been very large and in 1944 about 6500 tons or almost one-third of the country's entire output of HF was consumed by the District. Though the consumption of this material is not so large at present, its importance has not decreased.

HF was supplied to the contractors on the following three bases:

- (1) the Government contracted with HF producers for shipments of the material which were supplied to the consuming contractor without expense to him;
- (2) the Contractor acquired HF on his own purchase orders;
- (3) the Contractor manufactured all of the HF needed for his operations.

In the fluorine cell generation problem it was believed that a purer grade of HF was required than the commercially available





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scale in the years immediately before the war by polymerization of tetrafluoroethylene, which was derived from a Freon. Since this was particularly a du Pont product, arrangements were made to meet requirements by purchase of the material from du Pont.

Preliminary requirements indicated that very large amounts would be necessary and it was intended that a plant would be built by the Government in which du Pont could produce the required amounts. However, further research indicated that this material was not as satisfactory as originally expected and the quantity needed would therefore be very much smaller. All material was subsequently obtained on normal purchase orders. Also arrangements were made with du Pont to provide for the priority of the Manhattan District orders and for suitable security arrangements with respect to the use of this material by the District.

Trifluorochloroethylene (P-539) - This is the material which Hooker used for the manufacture of MFL. P-539 is manufactured from Freon 113 (trichloro-trifluoro-ethane). Since Freon 113 is a du Pont product and since the production of P-539 from it was in the experimental stage and was a du Pont development, the contract for this material was awarded to du Pont. P-539 was purchased on a unit price basis under Contract W-7412 eng-161, effective May 20, 1944.

The original price quoted by du Pont was \$9.00 per pound. This figure was later found to be excessive and they reduced

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the price to \$4.50 per pound, at which price the first 2,500 pounds were purchased. Three subsequent reductions in price were made; 8,400 pounds being purchased at \$2.90, 2,000 pounds at \$2.50, and the balance, 66,950 pounds, at \$2.25. Total cost for the 79,850 pounds purchased under W-7412 eng-161 was \$191,250, or an average cost of \$2.935 per pound.

#### Research Contracts

A number of research contracts were written with a number of industrial concerns by this office. Very complete information relative to the work done by the various contractors working on research contracts is to be found in regularly submitted reports under all of these contracts except W-7412 eng-161 with du Pont. With the exception of this contract, each contractor submitted a monthly technical report describing the work during the month in detail. Also each contractor submitted a monthly progress report with a summary of the work accomplished during the month and the monthly financial statement. du Pont under W-7412 eng-161 submitted a great number of special project reports covering the large number of research projects carried out under this contract. All of these contracts with the exception of W-7405 eng-288 with Massachusetts Institute of Technology grew out of OSRD contracts.

American Cyanamid - Contract W-7401 eng-53, effective <sup>5</sup> June 5, 1943 was a combined research and service contract which provided mainly for analytical services of special types. The main development under this

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contract was the Infra-Red Gas Analyzer. This apparatus was developed by Dr. V. Z. Williams as a simpler and cheaper method than the mass spectrograph for analyzing fluorocarbons. Four were constructed at American Cyanamid under this contract and furnished to various agencies under the U. S. Engineer Department. In addition, a great deal of analytical work was done by Dr. Williams using the various specialized physical methods which he had developed. Since Dr. Williams is an outstanding authority in the field of optical analysis, it was not believed that this work could have been done as well elsewhere.

du Pont Contract W-7412 eng-151 - This contract, effective <sup>5</sup> September 5, 1944, not only originated in a large number of OSRD contracts but Contracts eng-9, 10, 47 and 156 with the du Pont Company were all combined in it. This was done at du Pont's request. The contract therefore incorporated all research and development work connected with the very extensive program carried out for the Madison Square Area at du Pont as well as a small amount of production which was carried out as part of the development work. Since du Pont is the largest chemical company in the U. S. and its experience with fluorine compounds was almost unparalleled, it was inevitable that a very considerable part of the work for the U. S. Engineer Department should fall to this company. All the research work for the fluorocarbons, fluorine generation and packaging and the metal and metal recovery process was done under W-7412 eng-151.

Hooker - W-7405 eng-75 - This contract covered the research on the development of methods of manufacture of organic fluorine derivatives and particularly of hexafluorocyclohexene, P-45. Also the research

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work done by Hooker on the manufacture of chlorofluoroheptane (P-46) which is the intermediate in the Purdue 715CL alternate coolant process, the research work on MPL and MFI plus the initial 100 gallon production lot was made under this contract.

Hooker - W-7405 eng-76 - This contract was for the research and development on the generation and compression for packaging of fluorine. Hooker was chosen to carry on this work because (1) it is one of the oldest and best known manufacturers of chlorine gas in this country, (2) the cells developed for the electrolytic generation of chlorine at Hooker have been extremely successful, and (3) it was believed that the same talent which had brought this cell to such a high state of perfection would be equally successful for the generation of fluorine gas. That this has proved to be the case is evident from the fact that the Hooker carbon anode cell exceeded in efficiency the nickel anode cell already in use at du Pont by 50% and that steps were taken because of this efficiency and the long cell life to substitute the Hooker cell for all du Pont nickel anode cells. The fluorine generation experience accumulated by Hooker was of great importance to the District since they were under contract to install fluorine generating equipment at the site for the conditioning of the units there. Development of mechanical equipment for low pressure packaging of fluorine was also developed and financed under this contract.

Johns Hopkins University W-7401 eng-43 was for research and development work on fluorocarbons. Most of the work under this contract was devoted to the development of a high temperature fluorine generation cell and the use of metal salts as fluorinating agents for organic chemicals in the vapor phase. However, Johns Hopkins conducted a great

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variety of small research programs at the request of Madison Square Area under this contract. In a seven-month period in 1944 when the fluorinated lubricant program had fallen greatly behind schedule, production of fluorinated lubricant was carried out in order to relieve the critical state of affairs.

Purdue University - W-7405 eng-74. This contract covered work on research and development relative to fluorocarbons. Considerable general research experience in this field had previously been developed at Purdue and it was felt that Madison Square Area should avail itself of this exceptional background of experience. The main work has dealt with the problem of developing an alternate coolant to take the place of C-816 which was part of a more general program of developing methods of manufacture of fluorocarbons without the use of elementary fluorine, because fluorine is the most expensive item used as well as the most difficult to handle. Dr. E. F. McBea who carried out this program at Purdue achieved considerable success and brought the process for developing 715CL (chloro-pentadecafluoroheptane) to a high degree of perfection during this work. Unfortunately by the time the process had been perfected the operating contractors had invested so much in the C-816 process that it was not feasible to put the C-715CL process in operation. In addition, considerable work was done on a variety of associated projects as requested by Madison Square Area. The Purdue University Group was particularly active in developing analytical methods which were applicable to fluorocarbons, and also functioned as a service organization by

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carrying out many analyses for the U. S. Engineer Department. In November 1944, the resources available at Purdue under this contract were turned over to Lt. Colonel Mahoff at Oak Ridge who had urgent need of them for his work.

Massachusetts Institute of Technology - W-7405 eng-288 - This contract was written to provide for certain research work on fluorine, which it was decided at a general conference should be instituted at a university, <sup>since this was a type of work</sup> ~~being of a type~~, for which the universities were particularly adapted. The contract was arranged with MIT since Professor Schumb of that school had had many years of experience with fluorine and fluorides in general and was an outstanding authority on this subject. By the latter part of 1944 work on fluorine had progressed under all the contracts covering this type of work to such a point that it was felt that there was no further need for research on this subject. Accordingly, research work at MIT was discontinued in November 1944.

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SUMMARY OF PROCUREMENT FOR K-25

CONTRACT DATA

<u>Contract</u>	<u>Operation</u>	<u>Effective Date</u>	<u>Construction Cost</u>	<u>Operating Cost*</u>
Massachusetts Institute of Technology W-7405 eng-288	Research C-216	February 15, 1944	\$ --	\$ 14,600
Penn Salt Manufacturing Co. W-7405 eng-80	Hydrofluoric Acid	January 1, 1944	--	458,700
Purdue University W-7405 eng-74	Research - C-715 CL, P-45 , other	May 1, 1943	--	209,000
			<hr/>	<hr/>
			\$11,725,750	\$20,771,800

\*Includes research and development costs

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SUMMARY OF PROCUREMENT FOR K-25

<u>Contract</u>	<u>Operation</u>	<u>CONTRACT DATA</u>		<u>Construction Cost</u>	<u>Operating Cost*</u>
			<u>Effective Date</u>		
American Cyanamid Co. W-7401 eng-53	Research, Analytical Services		April 15, 1943	\$ --	\$ 32,000
Canadian Industries, Ltd. W-44-153 eng-9	Liquid Chlorine		July 20, 1945	--	5,600
E.I. du Pont de Nemours & Co. W-7412 eng-2	C-716		November 17, 1942 )	737,290	2,156,770
	C-2144		)		783,800
W-7412 eng-6	C-816		December 31, 1942	9,101,260	8,673,830
	C-2144				893,530
	OS-12-116 (cobalt tri-fluoride)				66,370
W-7412 eng-7	D-29		April 22, 1943		92,700
W-7412 eng-8	Construction of HF Plant		December 31, 1942	100,000	--
W-7412 eng-151	Research C-716**		September 5, 1944	--	76,180
	Research C-816			--	147,870
	Research and Production C-2144***			--	557,860
	Research and Production C-216 (1)			184,200	449,700
W-7412 eng-161	Product 539		July 1, 1944		191,250
General Chemical Co. W-7405 eng-315	Anhydrous hydrofluoric acid		September 1, 1944	--	42,900
Harshaw Chemical Co. W-7405 eng-43	Research C-216		March 18, 1943		8,100
Hooker Electrochemical Co. W-7405 eng-28	P-45 Production		January 4, 1943 )	1,533,000	3,364,850
W-7405 eng-75	MPL & MFI		)		670,680
	Research - MPL		April 1, 1943		121,010
	Research - alternate coolants				118,680
W-7405 eng-76	Research C-216		April 1, 1943	20,000	232,840
Johns Hopkins University W-7401 eng-43	Research C-2144 and C-216		April 1, 1943	50,000	578,000
Kinetic Chemical, Inc. W-7405 eng-27	Hydrofluoric Acid		November 16, 1943		850,000

- (1) Includes costs incurred under W-7412 eng-47 and OEMsr 809 and 652 which were incorporated into W-7412 eng-151  
 \*\*\* Includes costs incurred under W-7412 eng-9 and OEMsr 414 and 664 which were incorporated into W-7412 eng-151  
 \*\* Includes costs incurred under OEMsr 551 which were incorporated into W-7412 eng-151  
 \* Includes Research and Development Costs

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Special Chemicals for K-25

References (All located in the MSA files of the Atomic Energy Commission, Office of New York Directed Operations.)

<u>No.</u>	<u>Description</u>
1	Letter from Lt. Col. Crenshaw to Dr. Stewart of the OSRD, dated Nov. 9, 1942.
2	Letter from Dr. Benedict of Kellogg to Dr. H. W. Elley, E. I. du Pont de Nemours & Co., Dec. 8, 1942.
3	Letter from Dr. Elley of du Pont to S. W. McCune, Jr., du Pont, Dec. 22, 1942.
4	Letter from Dr. R. Rosen to Lt. Col. Crenshaw, Feb. 16, 1943.
5	Letter from Dr. R. Rosen to the New York Area, Feb. 15, 1944.
6	Letter from Dr. R. Rosen, Kellogg, to Lt. Col. Crenshaw May 1, 1943.
7	Letter from Mr. Arnold, Kellogg, to Lt. L. C. Burman, Nov. 28, 1942.
8	Letter from Lt. Col. Crenshaw to S. W. McCune, Jr., du Pont, Feb. 22, 1943.
9	Letter from Lt. Col. Crenshaw to S. W. McCune, Jr., du Pont, March 27, 1943.
10	Letter from Dr. Rosen to Lt. Col. Crenshaw, May 1, 1943.
11	Letter from Dr. Rosen, Feb. 15, 1944.
12	Letter to Madison Square Area, May 15, 1944.
13	Letter from S. W. McCune, Jr., du Pont, to Capt. W. M. Hearon, 14 June 1944.
14	Letter from Mr. Arnold, Kellogg Co., June 24, 1944.
15	Letter from Mr. Babcock to L. C. Burman, March 17, 1943.
16	Letter from Mr. Fielding, du Pont, to Lt. Col. Crenshaw, April 22, 1943.

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References

<u>No.</u>	<u>Description</u>
17	Letter from Lt. Col. Crenshaw to Murray, Hooker Electrochemical Co., June 8, 1943.
18	Letter from Clark, du Pont, to Lt. Col. Ruhoff, July 19, 1943.
19	Letter from Benning, du Pont, to Capt. Anthes, Aug. 25, 1943.
20	Letter from Capt. Russell to Murray, Hooker Electrochemical Co., Nov. 2, 1943.
21	Letter from Meek, Hooker Electrochemical Co. to Capt. Russell, Nov. 11, 1943.
22	Letter from Capt. Russell to Bartlett, Hooker Electrochemical Co., Aug. 22, 1944.
23	Letter from Dr. Rosen, June 4, 1943.
24	Letter from Dr. Rosen to Lt. Col. Crenshaw, June 19, 1943.
25	Letter from Major Moxen to Lt. Col. Ruhoff, Feb. 7, 1944.
26	Memo by Capt. Anthes, Mar. 4, 1944.
27	Letter from Maj. Hadlock to Mr. Clark, du Pont, Apr. 6, 1944.
28	Letter from Dr. Rosen to Capt. Beckwith, Aug. 4, 1944.
29	Memo by Capt. Anthes, Aug. 25, 1944.
30	Letter from Captain Swartout to Mr. Clark, du Pont, Sept. 4, 1944.
31	Letter from Dr. Rosen to Dr. Currie, Columbia, July 25, 1944.
32	Letter from Mr. Bartlett, Hooker Electrochemical Co., to Maj. Russell, Oct. 3, 1944.
33	Letter from Mr. Bartlett, Hooker Electrochemical Co., to Maj. W. E. Kelley, Jan. 23, 1945.
34	Letter from Mr. Bartlett, Hooker Electrochemical Co., to Maj. W. E. Kelley, Mar. 16, 1945.
35	Memo by W. M. Haaron, May 28, 1945.
36	Letter from W. E. Kelley to Mr. Bartlett, Hooker Electrochemical Co., June 4, 1945.

**References**

<u>No.</u>	<u>Description</u>
37	Letter from W. E. Kelley to Mr. Bartlett, Hooker Electrochemical Co., July 23, 1945.
38	Letter from W. E. Kelley to Mr. Bartlett, Hooker Electrochemical Co., Mar. 27, 1945.

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APPENDIX H

MISCELLANEOUS MATERIALS FOR P-9  
(Heavy Water Process)

Baker & Co., Inc., Contract W-7407 eng-6, provided for supplying platinum-impregnated carbon catalyst for the P-9 Project. The process for manufacturing this catalyst had been worked out by the Baker Company in collaboration with Professor Taylor of Princeton University under OSRD contract OEMsr-412. When the USED took over this project, contract W-7407 eng-6 was written with the Baker Company to provide for continuing to supply this product. This was a unit price contract providing for supplying of platinum, charcoal, and making the catalyst, by properly combining the two. A total of 31,000 lbs. of Product 43 (platinum-impregnated carbon catalyst) was supplied under this contract at a cost of \$197,400 or \$6.37 per lb.

Harshaw Chemical Co., contract W-7405 eng-16, was developed for the purpose of providing a nickel-chrome catalyst for use at the P-9 project. This catalyst was an alternate to the platinum charcoal catalyst which was to be furnished by Baker. Difficulties <sup>which</sup> arose in the manufacture of this catalyst <sup>were</sup> largely due to inability to get proper equipment and the contract was terminated as of May 6, 1943. Costs under this contract totalled \$80,000; \$22,000 of which covered purchase and installation of equipment. The remainder of \$58,000 covered research and development and the production of Product 80 (nickel-chrome catalyst) and intermediates. 4,509 lbs. of Product 80 were supplied under this contract at a unit price of \$2.42 per lb.; 4,366 lbs. of intermediate No. 1 at \$1.1899 per lb.; and 19,313 lbs. of intermediate No. 2 at \$1.1989 per lb.

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Contract No. W-26-021 eng-10 with the Harshaw Chemical Co. was negotiated in order that material manufactured under W-7405 eng-16 over and above quantities set up as termination inventory in the termination settlement of that contract and not previously paid for under W-7405 eng-16 might be acquired by the Government without re-opening the termination agreement on Contract W-7405 eng-16.

Because of security considerations, it was essential that all quantities of Catalyst 80 and intermediates be obtained from the contractor and placed where it would not come into the hands of the public.

12,200 lbs. of Product 80 and intermediates were procured under this contract at a unit price of \$1.00 per lb.

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APPENDIX I

MISCELLANEOUS MATERIALS FOR X-10

Helium

The Madison Square Area received various requests to supply Helium. The TNX Division of the Explosives Department at duPont desired helium for the Hanford Engineer Works. For this request arrangements were made with the Bureau of Mines of the Dept. of Interior to supply the helium to Hanford. The business between this office and the Bureau of Mines was handled by transfers of funds from the War Department to the Department of Interior at a cost of approximately \$30,000. As the work at Hanford progressed, the requirements tapered off greatly and later the schedule was withdrawn by the Area Engineer at Hanford Engineer Works and shipments were then made on the basis of individual requests from Hanford Engineer Works. When the requirements loomed large, 7 helium tank cars were purchased from the General American Transportation Company under Contract No. W-7401 eng-41 at a cost of \$258,700. For security reasons these cars were numbered with Navy tank cars numbers and were operated on a joint basis with the Navy in order to use the total number of tank cars available in the most efficient manner.

Off-Gas

The term "off-gas" refers to process gas which has been run through the K-25 separation process and has been depleted in valuable material to such an extent that it is no longer useful in such processes. However, the depletion of the valuable material was not sufficient so that it<sup>did not</sup> have

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value in the X-10 process and for this reason it was desirable to transform this off-gas into metal for X-10 use. Also the storage of off-gas is inconvenient <sup>because of</sup> ~~due to~~ the extreme volume, and transformation to some material more easily stored was sought.

DuPont was requested to assume the responsibility for studying the chemical treatment of the off-gas under W-7412 eng-10 which contract was later incorporated into W-7412 eng-151. The program was to include methods for the conversion of process gas to metal or some intermediate product and the design and construction of a plant to utilize such a process. The importance of providing for the recovery of any C-716 or C-816 in the off-gas was stressed and it was suggested that it probably would be <sup>more</sup> ~~the most~~ desirable to convert the "off-gas" to the tetrafluoride.

DuPont developed a satisfactory method for the recovery of off-gas involving reduction of the gas with sodium hydrosulfite. Provision was made for the recovery of the fluorocarbons and process had been developed through the pilot <sup>plant</sup> stage. It had been assumed that the duPont Co. would construct and operate the off-gas plant. However, duPont was not willing to construct such a plant nor was this office able to change their decision. The reasons given for this reluctance were that it did not fit in administratively with the duPont program and duPont was reluctant to assume additional war work because of the large number of projects on which they had already assumed responsibility.

Because of this reluctance, it was decided to attempt to interest the Hooker Electro Chemical Company in this project. Hooker was chosen because of their familiarity with other Manhattan District Work. Negotiations

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were satisfactorily completed with Hooker and these negotiations eventually resulted in Contract W-7405 eng-319. However, Hooker stated that they were not willing to do any research on this program, but were only willing to construct and operate a plant according to designs and directions furnished by the Madison Square Area.

About this time word was received that Dr. Spedding at Iowa State College had developed a large-scale process for producing tetrafluoride directly from process gas by a method involving pyrolysis with dry hydrochloric acid. This process was successfully tested by duPont. Because of the advantages of this process it was immediately decided to substitute this for the duPont sodium hydrosulfite process.

<sup>With</sup>~~Due to~~ the cessation of hostilities, plans were changed and the design work was not completed. However, Hooker was reimbursed for the amount expended and the final draft of Contract W-7405 eng-319 covered this reimbursement which totaled \$2,200 and also the provision that Hooker would be willing to assume this work again at any time up to the end of the war and six months thereafter.

The cost of research in the "off-gas" program at duPont under W-7411 eng-151 totaled \$59,970.



APPENDIX J

Revised Summary of Contractual Procurement for Site X (as of January 1, 1947).

<u>CONTRACTOR</u>	<u>CONTRACT NO.</u>	<u>DATE OF CONTRACT</u>	<u>ITEM</u>	<u>CONSTRUCTION COST</u>	<u>OPERATING COST*</u>
Allis Chalmers Company	W-26-021 eng-3	4 Oct. 1944	Betatron.	\$ --	\$ 50,300*
American Cyanamid Company	W-7401 eng-91	8 Feb. 1944	504 lbs. crystalline boren 10**	140,000	324,400
American Phenolic Corp.	W-38-094 eng-17	10 Jan. 1945	1,100,000 ft. coaxial cable.	--	61,600*
" " "	W-38-094 eng-20	18 May 1945	1,900,000 " " "	--	92,900*
American Truck & Body Co.	W-7423 eng-19	10 Apr. 1944	2 custom built trailers.	--	6,300*
American Smelting & Refining Company	W-7401 eng-75	30 Aug. 1943	50 tons (sh) lead	--	8,200*
" " "	W-7401 eng-180	14 Sept. 1944	11,000 lbs. lead bismuth alloy.	--	8,600*
" " "	W-14-108 eng-10	12 June 1945	15,000 lbs. bismuth tin alloy.	--	15,100*
Arnold Engineering Co.	W-7407 eng-57	1 Feb. 1944	Electric current (night load) 1 Feb. to 31 March 1944.	--	7,600*
Babcock & Wilcox Co.	W-7421 eng-19	20 Dec. 1944	1 hydraulic accumulator.	--	144,400*
" " "	W-31-109 eng-3	6 March 1945	2 " " w/spare parts.	--	14,300*
Baker and Company	W-7407 eng-15	12 June 1943	1 circular gold disc (780 os.)	--	58,200*
" " "	W-7407 eng-22	22 July 1943	1 circular platinum disc (886 os.)	--	
" " "	W-7407 eng-22	22 July 1943	200 os. iridium	--	33,000*
Baldwin Locomotive Works	W-31-109 eng-2	10 Dec. 1944	3 testing machines.	--	400*
Belmont Smelting & Refining Works	W-7401 eng-76	31 Aug. 1944	19,800 lbs. bismuth metal	--	25,100*
" " "	W-14-108 eng-5	30 Jan. 1945	3 tons cerrottru.	--	9,300*
" " "	W-14-108 eng-6	17 March 1945	75,000 lbs. cerrottru.	--	82,500*
" " "	W-14-108 eng-3-1	21 Dec. 1944	Sale of 8166½ lbs. bismuth metal bars.	--	6,900*
Blaw-Knox Company	W-14-108 eng-7	30 March 1945	1 tower.	--	6,300*
Bowser, Inc.	W-28-094 eng-1	12 Dec. 1944	1 Bowser unit.	--	17,600*
Brewster Aeronautic Corp.	W-7409 eng-28	7 July 1944	1 turret lathe w/accessories.	--	5,200*
Brush Beryllium Co.	W-7401 eng-78	15 Sept. 1943	Powdered beryllium oxide.	--	45,600
" " "	W-22-075 eng-10	9 July 1945	Beryllium metal, lump & pebble form.	33,000	366,000
" " "	W-7401 eng-60	18 Aug. 1943	Beryllium metal & fluoride flux.	--	137,800*
Bendix Aviation Eclipse Pioneer Division	W-35-058 eng-12	14 Feb. 1946	15 inverters.	--	5,000*
Buffalo Foundry & Machine Co.	W-7407 eng-56	21 Feb. 1944	3 steam jacketed kettles with tilting mountings.	--	6,700*
Callite Tungsten Co.	W-7409 eng-29	10 May 1944	152.005 kgs. Puretest fabricated into various forms.	--	6,300*
" " "	W-14-108 eng-1	21 Sept. 1944	Preparation of 100 lin. ft. 4" & 100 lin. ft. 6" Puretest.	--	1,400*
Carboley Company, Inc.	W-7401 eng-81	10 Oct. 1943	2000 lbs. tungsten carbide powder	--	15,000*
" " "	W-7401 eng-98	9 June 1944	15 " " " "	--	9,500*
" " "	W-22-075 eng-2	30 Oct. 1944	1500 lbs. " " " "	--	8,200*
" " "	W-22-075 eng-3	21 Nov. 1944	Research & production of 1 tungsten carbide sphere.	--	5,100*

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<u>CONTRACTOR</u>	<u>CONTRACT NO.</u>	<u>DATE OF CONTRACT</u>	<u>ITEM</u>	<u>CONSTRUCTION COST</u>	<u>OPERATING COST*</u>
Carboloy Company, Inc.	W-22-075 eng-4	7 Dec. 1945	12,000 lbs. tungsten carbide powder & (3%, 9%, 6% cobalt).	---	59,500*
" " "	W-22-075 eng-8	12 Apr. 1945	7500 lbs. tungsten carbide.	---	40,600*
" " "	W-22-075 eng-9	6 June 1945	4000 lbs. tungsten carbide, 6% cobalt.	---	21,700*
" " "	W-22-075 eng-14	1 Nov. 1946	25,000 lbs. tungsten carbide powder.	---	129,200*
Carnegie Illinois Steel Co. F. H. Crawford	W-31-109 eng-6 W-7418 eng-55	15 March 1945 8 June 1944	Lease of freight car. 1 rolling mill, 1 V belt motor drive, 1 20 h.p. motor & 1 magnetic starter.	---	500* 7,700*
Eastman Kodak Company	W-7405 eng-300	22 June 1944	9 cameras.	---	6,200*
" " "	W-26-021 eng-2	22 Sept. 1944	8 "	---	6,700*
" " "	W-26-021 eng-18	21 May 1945	1050 rolls 35 mm panchromatic film.	---	19,300*
East Coast Service Co.	W-31-109 eng-12	11 June 1946	90 shipping cases (for "X" Units).	---	6,950*
Kimer and Amend Co.	W-44-154 eng-2	2 Apr. 1945	2809 gms. gallium metal.	---	16,825*
Electronic Mechanics, Inc.	W-28-094 eng-12	31 Oct. 1946	220 mikroy rings.	41,150	22,890
Electro Metallurgical Sales Corporation	W-26-021 eng-19	22 May 1945	2000 lbs. redistilled calcium.	---	8,400*
Fansteel Metallurgical Co.	W-7425 eng-27	1 Feb. 1944	720 beryllia bricks.	---	44,200*
" " "	W-7425 eng-29	24 May 1944	359 "A Bars" 1 1/2" to 10 1/2" long.	---	23,600*
" " "	W-17-028 eng-30	21 June 1945	30 ft. tantalum rod.	---	4,500*
Federal Telephone & Radio Corporation	W-38-094 eng-21	18 May 1945	100,000 ft. coaxial cable.	---	5,100*
Garwood Industries, Inc.	W-31-109 eng-7	28 March 1945	Attachments for Caterpillar E-7 tractor & LeTourneau AF-2 crane.	---	7,800*
General Motors Corp., AC Spark Plug Division	W-7409 eng-24	28 June 1944	Beryllium oxide bricks.	---	6,300*
Globe Industries	W-7409 eng-30	22 July 1944	1 vibration test machine	---	5,500*
Hendy & Harmon	W-7405 eng-147	20 July 1943	500 troy os. gold.	---	17,800*
" " "	W-7405 eng-269	10 June 1944	5 sheets gold.	---	7,300*
" " "	W-7405 eng-299	12 June 1944	11 sheets gold.	---	5,400*
" " "	W-7405 eng-309	26 June 1944	120 ft. gold wire.	---	6,300*
" " "	W-7405 eng-317	12 Sept. 1944	10 sheets gold.	---	10,600*
" " "	W-26-021 eng-8	29 Nov. 1944	50 sheets gold.	---	12,600*
" " "	W-26-021 eng-15	14 March 1945	38 gold sheets, 25 ft. gold wire.	---	11,700*
" " "	W-7405 eng-289	6 June 1944	92,450 lbs. boron trifluoride.	---	72,770*
Harshaw Chemical Company	W-14-108 eng-14	19 Dec. 1945	5 wire recorders.	---	26,620*
Illinois Institute of Technology	W-44-153 eng-5	1 Nov. 1945	Services & equipment to perform calculations.	---	23,320*
International Business Machine Co.	W-7407 eng-23	22 July 1943	350 os. iridium powder	---	57,800*

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<u>CONTRACTOR</u>	<u>CONTRACT NO.</u>	<u>DATE OF CONTRACT</u>	<u>ITEM</u>	<u>CONSTRUCTION COST</u>	<u>OPERATING COST*</u>
Johnson Matthey & Co.	W-7407 eng-24	22 July 1943	200 oz. iridium	\$ --	\$ 31,000*
"	W-7407 eng-35	7 Sept. 1943	100 oz. osmium	--	5,000*
LAB Corporation	W-28-094 eng-2	26 Dec. 1944	1 500 lb. shake table.	--	6,000*
Laboratory Associates	W-31-109 eng-8	7 June 1945	4 seismographs.	--	8,000*
Le Tourneau	W-17-026 eng-26	8 Jan. 1945	4 cranes.	--	7,300*
Massachusetts Institute of Technology	W-26-021 eng-22	20 Aug. 1945	Research & development of "X" Unit.	--	20,500*
Monarch Machine Tool Co.	W-7423 eng-22	16 June 1944	4 lathes w/accessories.	--	18,900*
Monsanto Chemical Co.	W-7407 eng-134	16 Aug. 1944	200 plastic tubes, 400 plastic discs.	--	10,000*
"	W-7407 eng-146	11 Sept. 1944	6000 sq. ft. plastic sheet.	--	46,900*
"	W-35-058 eng-1	25 Oct. 1944	5000 " " " "	--	29,400*
National Carbon Company	W-7401 eng-77	10 Sept. 1943	40 tons graphite AGNT or AOOT	--	28,800*
"	W-7401 eng-179	13 Sept. 1944	72 pieces graphite rod.	--	7,800*
"	W-22-075 eng-6	18 Dec. 1944	69 " " " "	--	9,900*
"	W-44-108 eng-8-4	7 Jan. 1946	Sale of 40,000 lbs. scrap graphite.	--	300*
Northam Warren Corporation	W-28-094 eng-6	2 July 1945	Electrical equipment.	--	8,200*
Horton Company	W-7405 eng-292	15 July 1944	Crystalline boron.	--	53,000*
"	W-28-094 eng-7	1 Feb. 1946	593 magnesia crucibles, type S-1, 1226 " " " B-2.	22,000	68,600*
Picker X-Ray	W-7412 eng-162	21 Aug. 1944	1 X-Ray unit w/mobile chassis & tube stand.	--	6,900*
Pough, Fred H.	W-22-075 eng-1	30 Sept. 1944	Consultant services regarding tourmaline & procurement of same.	--	3,900*
Racine, Wm. A.	W-31-109 eng-10	15 April 1946	1 portable radiograph timer.	--	37,000*
Raytheon Mfg. Co.	W-14-108 eng-3	28 Oct. 1944	"X" Units: 45 M-1, 280 M-2, 125 M-3	--	2,422,500
"	W-14-108 eng-9	15 May 1945	25 other (experimental), 12 M-2-60. Splices for coaxial cable; 500 sets coaxial cable; empty cases & covers for eng-3.	--	135,200*
"	W-14-108 eng-12	8 Aug. 1945	Components for "X" Unit M-2.	--	54,200*
"	W-14-108 eng-13	9 Jan. 1946	5 "X" Units, M-3.	--	80,000*
"	W-14-108 eng-19	11 Oct. 1946	1550 spark gaps.***	--	90,000
Sickles Company	W-42-069 eng-8	1 July 1945	230 delay lines.	--	5,500*
Stauffer Chemical Co.	W-7407 eng-39	5 Nov. 1943	1,131,525 lbs. boron trichloride	--	63,000*
Sperry Gyroscopes, Inc.	W-28-094 eng-8	21 Feb. 1946	1 portable radiograph timer.	--	47,000
Sprague Electric Co.	W-31-109 eng-4	20 March 1945	Condensers.	--	19,900*
Union Switch & Signal Co.	W-42-069 eng-4	16 Apr. 1944	Electrical equipment.	--	8,200*
"	W-42-069 eng-6	18 May 1945	Electrical equipment.	--	31,000*
University of Illinois	W-44-153 eng-3	31 March 1945	4 vacuum tubes.	--	2,000*

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<u>CONTRACTOR</u>	<u>CONTRACT NO.</u>	<u>DATE OF CONTRACT</u>	<u>ITEM</u>	<u>CONSTRUCTION COST</u>	<u>OPERATING COST*</u>
Vascoloy-Ramet Corporation	W-14-108 eng-S-3	18 Oct. 1945	Sale of 1875 lbs. tungsten carbide powder (6% cobalt).	\$ --	\$ 12,800*
" " "	W-7409 eng-27	5 July 1944	Tungsten carbide bricks.	--	36,000*
" " "	W-7409 eng-33	2 Sept. 1944	3000 lbs. tungsten carbide powder.	--	20,600*
" " "	W-17-028 eng-27	1 Feb. 1945	1500 lbs. tungsten carbide powder.	--	10,300*
" " "	W-17-028 eng-28	9 April 1945	2500 lbs. tungsten carbide.	--	17,100*
Western Electric Company	W-7405 eng-298	8 Aug. 1944	75 delay lines.	--	3,900*
Westinghouse Electric & Manufacturing Co.	W-7407 eng-131	24 Feb. 1944	2000 inches tungsten bar.	--	10,000*
" " "	W-7405 eng-312	15 Dec. 1944	Research & development regarding X-ray tubes.	--	51,400*
" " "	W-35-058 eng-3	27 Nov. 1944	544.8 kgs. tungsten powder.	--	5,700*
Westvaco Chlorine Prod.	W-28-094 eng-4	26 March 1945	100 T. barium nitrate.	--	35,400*
" " "	W-28-094 eng-15	25 Nov. 1946	60,000 lbs. barium nitrate.	--	6,900*
Yale & Towne Mfg. Co.	W-22-094 eng-3	4 Jan. 1945	Chain hoisting equipment.	--	10,700*
			Sub-total	\$236,150	\$5,826,775

- \* Financially completed.
- \*\* Contract later supplemented adding the following:
  1. 850 lbs. calcium fluoride-boron trifluoride complex in which the ratio of B-10 isotope to B-11 isotope is greater than normal.
  2. 242 lbs. calcium fluoride-boron trifluoride complex in which the ratio of B-11 isotope to B-10 isotope is greater than normal.
- \*\*\* Contract originally called for delivery of 3050 spark gaps; however only 1550 delivered and remainder cancelled. Contract not as yet executed pending the Government's acceptance of Contractor's claim; no payments have been made. The operating cost noted constitutes the amount of the claim submitted by Contractor.

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The following contracts covering radium rental and procurement, also listed under F-6, were for Site Y:

<u>CONTRACTOR</u>	<u>CONTRACT NO.</u>	<u>DATE OF CONTRACT</u>	<u>TYPE OF CONTRACT</u>	<u>MILLIGRAMS OF RADIUM</u>	<u>TOTAL COST TO 12/31/46</u>
Boris Pregel	W-7405 eng-313	1 Sept. 1944	R	5,595	\$ 31,718
" "	W-7405 eng-91	1 June 1943	P	2,021	34,357
" "	W-7405 eng-291	30 March 1944	P	2,471	42,015
Joseph A. Kelly	W-7412 eng-157	28 March 1944	P	57	1,496
" " "	W-38-094 eng-19	20 May 1945	R	5,000	7,125
" " "	W-38-094 eng-22	20 June 1945	R	5,000	6,750
" " "	W-38-094 eng-23	29 June 1945	R	5,000	6,375
" " "	W-38-094 eng-24	7 August 1945	R	200	2,400
Sub-total Radium Procurement				25,344	\$132,236
Total Contractual Procurement					\$6,195,161

Procurement for Site Y on Purchase Orders, totaled:

	<u>No. of Purchase Orders</u>	<u>Total</u>
1944	383	\$278,815
1945	319	250,645
1946	<u>74</u>	<u>51,700</u>
Total Purchase Orders	776	\$581,160

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APPENDIX K  
BERYLLIUM PROCUREMENT

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By Authority of the U. S. Atomic Energy Commission,  
Per: [Signature] Date: 11/24/54  
Document No. [Number] 12A

Introduction

Almost from the inception of the Manhattan Engineer District, the element beryllium was of considerable interest to scientists and engineers because of its many desirable nuclear properties. Because of its low atomic weight (9.02), beryllium could possibly be a highly successful moderating material for thermal energy neutrons, and the fact that measurements indicated that beryllium had a comparatively small cross section to thermal neutrons (0.03, based on uranium as 1) led many investigators to believe that beryllium could be employed in nuclear reactors with great success. In addition to its excellent moderating and neutron absorption characteristics, beryllium appeared to have several advantages over other moderating materials because of the higher temperature levels to which beryllium and beryllium oxide could be subjected. Other properties of beryllium also added to its interest. Because of its low atomic weight and low thermal neutron absorption characteristics, beryllium was of potential importance as a reflector of neutrons. In addition, the nuclear reaction  $Be(n,2n)$  led many investigators to believe that the usefulness of beryllium in atomic reactors could be greatly enhanced by this reaction.

Although interest in beryllium was high in the early days of the Manhattan Project, the availability of beryllium was extremely limited. Only small quantities could be obtained from commercial sources

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and at prices which made the large scale use of beryllium prohibitive. Furthermore, the material was of a quality completely inferior to that required even for most Project purposes.

Prior to the Manhattan Project's interest in beryllium, very little use had been found for the metal as such, most of it being used in the preparation of alloys with aluminum, nickel and copper. Because methods had been found by which many of these alloys could be made directly from beryllium oxide, it was not necessary to produce large quantities of beryllium metal in order to produce large quantities of beryllium alloys. As a result of the increased demand for the metal, however, within the Manhattan Project itself, the Madison Square Area of the Manhattan Engineer District undertook, in 1943, the responsibility of procuring the various requests for beryllium metal and beryllium compounds which originated at many of the Manhattan Project installations. Since these requests for beryllium were comparatively small and were received in a highly sporadic manner, the procurement of beryllium and beryllium compounds was handled by the Special Projects Group of the Madison Square Area. This arrangement endured from 1943 through the Summer of 1945. However, in July, 1945, as a result of discussions between Colonel K. D. Nichols and Major W. E. Kelley, it was decided that the entire technical and procurement position of beryllium should be completely investigated and recommendations made concerning a continuous production program for beryllium and its compounds. This decision was an outgrowth of an increasing demand for beryllium metal and beryllium oxide, not only in the raw metal and powdered forms but as special fabricated shapes

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suitable for use in the development of new atomic reactors and for the development of special military weapons.

On July 31, 1945, Major Kelley requested 1st Lt. S. B. Roboff to carry out an investigation of the status of beryllium production and fabrication throughout the United States and to recommend action to be undertaken by the Madison Square Area Office to initiate a large scale program for the production and fabrication of beryllium and its compounds.

During the month of August, 1945, a comprehensive study of the existing beryllium companies, as well as Manhattan District metallurgical laboratories involved in the development and fabrication of beryllium was made. Based on the findings of Lt. Roboff's survey, a report entitled The Production and Fabrication of Beryllium was issued to Major Kelley on September 6, 1945. This report outlined the current sources of beryllium, methods of metal production, methods of beryllium fabrication known at that time, a description of the research and development work being performed on beryllium at the time and a summary of the experimental results obtained as of that date, statements of methods of increasing the production of the crude metal, statements concerning the provision of facilities for the fabrication of the metal, and discussions of the various health factors known at that time which were involved in the production and use of beryllium. In addition, the report listed recommendations to be followed by the Madison Square Area in initiating a production program for beryllium and its compounds necessary to meet the increasing quantities and varieties required by the Manhattan District.



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Soon after Lt. Roboff's report on beryllium was issued, a Beryllium Section was formed to operate within the scope of the M-Production Group of the Madison Square Area. Lt. Roboff was placed in charge of the Beryllium Section and was assigned the responsibility of organizing a beryllium production program capable of meeting the quantity and quality requirements of the various Manhattan District installations. In addition, because of the nebulous state of knowledge concerning the state of fabrication and production of high quality beryllium, Lt. Roboff was also charged with organizing the necessary development projects to enable beryllium to be produced and fabricated in accordance with Manhattan District requirements. Lt. Roboff was to report directly to Mr. F. M. Belmore, then Chief of the M-Production Group, who supervised and coordinated the work of the Beryllium Section in accordance with the overall policies of the M-Production Group.

Since prior to the formation of a separate beryllium production section within the Madison Square Area, the procurement of beryllium production was handled by the Special Projects Group, the procurement of beryllium prior to September, 1945 is described in the section of the History dealing with the activities of this group. However, in many instances in the descriptions that follow herewith, reference will be made to contractual actions and production data occurring prior to September, 1945 in order that a more complete continuity of text may be provided. In the main, however, the discussions of beryllium procurement and development presented will cover the period

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from the formation of the Beryllium Section in September, 1945 through December, 1946, which latter month was the last month of active operation of the Manhattan Engineer District.

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ORGANIZATION OF BERYLLIUM SECTION

The Beryllium Section was organized in September, 1945 as an operating unit within the M-Production Group of the Madison Square Area of Manhattan Engineer District. At the time of organization, Mr. F. M. Belmore was Chief of M-Production and Lt. S.B. Roboff was assigned as head of the Beryllium Section, reporting directly to Mr. Belmore. Corporal R. E. Morie acted as assistant to Lt. Roboff. By the Spring of 1946, the technical and administrative work of the Section had increased to such an extent that a position was established to enable a man to set up and handle the hundreds of small requests for beryllium materials received, and to set up a production procurement control system for not only the small requests but to cover the large scale production then being carried out by the Beryllium Section. This position was filled in May, 1946 by Mr. Bernard Engel, who reported directly to Lt. Roboff.

During the Summer and early Fall of 1946 the production and development programs within the Beryllium Section had expanded to such a point that it became necessary to obtain the services of an additional engineer. Hence, a new position was established in October, 1946 for an engineer to specifically supervise the technical administration of the development work then being carried out by the Beryllium Branch. To fill this position, Lt. H. P. Walter, Corps of Engineers, was assigned.

By the end of 1946 the organization of the Beryllium Section was as shown on the organization chart in Addendum No. 1.

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RAW MATERIALS

As found in nature, beryllium occurs chiefly in the minerals beryl ( $3 \text{ BeO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2$ ) and phenacite ( $2 \text{ BeO} \cdot \text{SiO}_2$ ). Beryl is by far the more common and large deposits of this mineral are found in Brazil, Argentina, India and in the Black Hills of South Dakota. Lesser deposits of beryllium-containing minerals exist in all parts of the world and in this country can be found extensively in New Hampshire, Massachusetts, Connecticut, New York, Maine, and Pennsylvania. In 1945 the total quantity of beryllium in the earth's crust was deemed to be equal to the total combined quantity of copper, lead, and zinc.

Because it is by far the most prevalent ore for beryllium, beryl has been used almost exclusively as the raw material for the manufacture of beryllium metal, beryllium oxide and beryllium alloys. The chief commercial sources of beryl have been Brazil and Argentina with Brazilian ores being used almost exclusively from the years 1944 thru 1945. Occasional shipments of beryl from India were received in the United States and small amounts were mined in South Dakota for commercial use. Because of wartime restrictions existing in 1945, however, control of all beryllium-containing minerals, foreign and domestic, which were to be used in the United States was under the control of the Metals Reserve Corporation, and it was necessary that all purchases of beryllium-containing ores be made through and with the consent of this corporation.

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During the period 1945-1946, the price of beryl ore delivered in this country ranged from \$9.00 to \$12.00 per ton unit.\* This price was considerably more than that charged at the onset of World War II, but since the cost of beryllium metal does not vary considerably with fluctuations in price of the ore, the relatively high price of beryl ore at the end of 1946 did not materially raise the cost of beryllium metal at that time.

Although the Manhattan District was interested in the supply of beryllium ore to the producers, the Manhattan District did not enter directly into a procurement program for beryl ore. This was for two reasons: First of all, the producers of beryllium metal were able to obtain, on the open market, sufficient quantities of beryl ore to more than meet the total production requirements of the Manhattan District during this period, and hence it was felt that no active participation in a beryllium ore procurement program was required by the Manhattan District. Secondly, during this period, beryllium ore procurement was being undertaken by the Metals Reserve Corporation, which organization was building a sizeable stockpile of beryllium ore. This stockpile was available to the Manhattan Engineer District should the need for the ore become necessary.

Although the Manhattan District did not actively engage in the procurement of beryllium ore, a study was undertaken by the Raw

\* A ton unit is defined as that quantity of beryllium ore containing 26 pounds of beryllium oxide content.

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Materials Group of the Madison Square Area to determine the future status of the beryllium mineral supply and to lay all necessary ground work for a Manhattan District program which could be put into effect should the sources for beryllium ore no longer be available to the beryllium companies for Manhattan District production. (Ref. 111) There were indications, even in 1945, that the availability of beryl ore may be subject to a sharp reduction, and plans were made by the Raw Materials Group to initiate a special ore production program for the Manhattan District in the event a tight situation became prevalent in the procurement of beryl ore. Up through the end of 1946, however, beryl ore was available in sufficient quantity to supply the producers of beryllium so that no concrete action by the Manhattan District was necessary in the ore procurement field.

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PRODUCTION

General

Because the requirements for beryllium metal, beryllium oxide, and fabricated shapes of both metal and oxide were received in the office of the Madison Square Area in large numbers, in various proportions, and for various types and shapes of materials, it is virtually impossible to describe the detailed production of these requirements as part of this discussion. Therefore, for the convenience of the reader, a complete compilation of all production and procurement requirements received by the Beryllium Section from its inception through December 1946 have been grouped, together with related information, in Addendum No. 3. Addendum No. 3 lists all requirements, both large scale production and small procurement type, upon which action was taken during this period by the Beryllium Section. The requirements are grouped under the name of the installation for which the material was to be procured, and included in this compilation is the type of material required, the type of material produced, the quantity produced, the source of production or delivery, the contract number or purchase order number under which the procurement was effected, and the date upon which final shipment of the requirement was made. A study of Addendum No. 3 will give the reader a thorough insight as to the wide range of materials and types of production and fabrication which were required.

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In discussing the production and fabrication of beryllium and its compounds, in view of the detailed listings given in Addendum No. 3, it is well to break down the types of production and fabrication into five main groups, namely, production of beryllium metal, fabrication of beryllium metal, production of beryllium oxide, fabrication of beryllium oxide, and the production of beryllium nitride. It was the aim of the Beryllium Section to establish as far in advance as possible the requirements for production and fabrication of beryllium in order that a steady rate of production and fabrication could be planned and executed. As can be seen from the listings in Addendum No. 3, the production requirements for beryllium and its compounds were received in an extremely sporadic manner and normally called for a highly erratic rate of production in order to meet the requirements. To more evenly balance the rates of production, a continual check was made by the Beryllium Section of all Manhattan Project installations to obtain insofar as possible the future requirements of these installations. Based on the future requirements, it was possible to set up the production of beryllium and its compounds on an average rate basis. Such production, together with a beryllium stockpile established at Middlesex, New Jersey, enabled deliveries of most requirements within a reasonable period of time, and still allowed an efficient and steady rate of beryllium production. Hence when requirements were low, it was possible for the Beryllium Section to accumulate in the Middlesex stockpile considerable quantities of beryllium metal and beryllium oxide, which stockpiled materials were

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shipped to users at such times as delivery requirements overbalanced the production rate. Hence, by using the Middlesex stockpile as a buffer to meet requirements, an overall effect of comparatively steady production was realized at the producers' plants.

For those readers who are interested in the actual processes used by each of the three beryllium producers in the manufacture of beryllium and beryllium compounds for the Manhattan Engineer District, complete process descriptions as well as process flow sheets are presented in Addendum No. 2. (Also see Ref. 149).

Production of Beryllium Metal

As was mentioned previously in the introduction, there was an early interest throughout the Manhattan Project in the procurement of beryllium metal, and as early as the Summer of 1943, a requirement for 2,000 pounds of beryllium metal was received in the Madison Square Area Offices for shipment to New Mexico. In addition, there was considerable interest in the use of beryllium oxide (Ref. 7, 9). As a result of the New Mexico requirement for 2,000 pounds of beryllium metal, authorization was received from Lt. Col. Cornell, dated August 9, for the Madison Square Area to enter into a unit price contract for the supply of 2,000 pounds of beryllium metal (Ref. 8).

In 1943, the only domestic commercial producer of beryllium was the Brush Beryllium Company of Cleveland and Lorain, Ohio. The chief business of the Brush Beryllium Company up until this time had been in the production of beryllium copper alloy, but the Brush Company had available a small size plant which Brush claimed at the

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time, was capable of producing 354 to 500 pounds of beryllium metal per month (Ref. 9). Hence, Contract No. W-7401 eng-60 dated August 18, 1943, was entered into with the Brush Beryllium Company for the manufacture of 2,000 pounds of beryllium metal and 100 to 200 pounds of beryllium fluoride, which, incidently, had also been requested by New Mexico. The total consideration of this contract was \$89,000, and the Brush Beryllium Company was given authorization under this contract to employ Government-owned equipment which had been used by the Brush Laboratories in connection with an earlier development project for manufacture of uranium metal.

At about this time, as the result of Manhattan Project interest in the Brush Company, Brush was given authority by the War Production Board to increase its production capacity of beryllium metal to 600 pounds (Ref. 10). The Brush estimates as to their existing production capacity had been greatly over-estimated, and by February 1944 approximately only 400 pounds total of beryllium metal had been produced under this contract (Ref. 11). Part of the difficulty with the Brush Beryllium Company was occasioned by the lack of ability to meet the specifications covering part of the material to be produced. In addition, it was found by Brush that the equipment which had been on hand and which had been thought capable of producing upwards of 350 to 500 pounds of metal per month could only produce somewhat under 100 pounds per month of beryllium metal as required by the Manhattan Project. As the result of technical assistance given by the New York Office and assistance

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provided through the War Production board, production of the beryllium was effected at a slightly greater rate throughout 1944, although Brush never reached the 600 pounds per month output which had been desired.

The type of beryllium metal produced at that time, and which was, incidentally, <sup>at</sup> the best available, was produced with an assay range from 85% to 96%, the remaining percentage consisting chiefly of beryllium fluoride - magnesium fluoride slag. Furthermore, in spite of the fairly liberal specifications then placed on the material, the beryllium also was high in aluminum, iron, boron and silica content, and much of Brush's production could not be used because of the prevalence of these impurities. It was possible to reduce the slag content of the metal by a remelting operation at Brush, and in some cases it was necessary to obtain remelt metal in order to meet the physical and chemical specifications on the metal. The price of the beryllium procured during this period was \$45.00 a pound for raw lump metal and \$55.00 a pound for recast or high assay metal (Ref. 7, 17).

During the latter part of 1944 and during early 1945, the requests for production of beryllium metal began to grow in magnitude, and it was necessary for several evaluations to be made by the Special Projects Branch to advise the various Manhattan Project installations of the possibilities of receiving beryllium metal in the quantities desired. (Ref. 13, 15, 18). By March 1945, the projected requirements for the Manhattan Project had reached such a stage that the production

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rate then in effect, namely 125-200 pounds per month, would fall far short of meeting the overall requirements (Ref. 16, 18). On the basis of the new requirements, negotiations were entered into with the Brush Beryllium Company for an expansion of the metal production plant at this organization. Inasmuch as the Brush Beryllium Company's Lorain Plant was then capable of producing 15,000 pounds of beryllium oxide per month (equivalent of approximately 5,000 pounds of beryllium metal), it was not necessary to increase the facilities for the production of oxide. The bottleneck therefore existed only in the conversion of beryllium oxide to beryllium metal, and it was necessary that additional equipment be procured and installed at the Lorain Plant to increase the output of the Brush metal plant to a maximum of 1,500 pounds per month (Ref. 19). As a result of the negotiations with Brush Beryllium Company, a new contract, No. W-22-075 eng-10, was entered into with this Company on July 9, 1945, which provided for the additional plant capacity at Brush Beryllium Company (cost to Government approximately \$26,000), and for the manufacture of 4,390 pounds of beryllium metal. The total value of the contract was \$193,000.

Shortly thereafter, the Beryllium Section of the Madison Square Area was created and the responsibility for the production and development of beryllium and its compounds was undertaken by this new Section. As a result of increased requirements, Supplement No. 1 to the Eng-10 contract was issued on October 17, 1945 which raised the total beryllium to be manufactured under this contract to

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5,570 pounds and resulted in an increase in the contractual consideration of \$45,000. By the end of 1945 and by virtue of the increased production capacity, Brush Beryllium Company was producing metal at a rate of approximately 1,000 pounds per month and shipments were being made to fill the existing requirements.

There was no improvement, however, at this time in the quality of beryllium metal manufactured over that which had been made by Brush Beryllium Company in the previous two years. However, since a large proportion of the metal produced was to be used for metallurgical studies at various Manhattan Project installations, the assay of the metal could be improved by remelting operations either at Brush or at the user's site, and a comparatively large quantity of other impurities did not appear too important at the time since nuclear properties of the beryllium were not a dominant factor.

By the Spring of 1946 as a result of process improvements and small equipment additions to the Brush plant, the capacity of this plant for the production of raw beryllium metal rose to approximately 1,500 pounds per month, although claims were made by the Brush Beryllium Company plant at this time of a total production output of 2,000 pounds per month (Ref. 4). By late Spring 1946, practically all metal required had been delivered under the original contract and Supplement No. 1, but additional requirements had been received in the Beryllium Section for approximately 700 pounds of remelted beryllium metal. This was provided for under Supplement No. 4 to the Eng-10 contract dated April 17, 1946. For the remainder of 1946 most

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of the beryllium metal output of the Brush plant was used directly in the fabrication of special shapes required by New Mexico and Chicago, as discussed under the subheading "Fabrication of Beryllium Metal."

By the end of 1946, a stockpile of several hundred pounds of beryllium metal had been accumulated at the Middlesex Warehouse and was being held in reserve to meet new requirements for delivery early in 1947. The beryllium metal stockpile was planned for use, together with full scale Brush output, early in 1947 and by the end of 1946 negotiations had been started with the Brush Company for increased output of beryllium metal to meet new large scale beryllium requirements. (Ref. 1 through 6).

As a result of the increase of the potential requirements in the year 1947 it appeared that the production schedule of Brush, which was anticipated between 1,500 and 2,000 pounds per month, would not be sufficient to meet the requirements of the Project in 1947 which were to be upwards of 2,500 to 3,000 pounds per month. (Ref. 101, 103, 104). Hence, late in 1946 discussions were entered into with the Beryllium Corporation of Reading, Pennsylvania, and preliminary contractual arrangements were made for the production of approximately 1,000 pounds per month of high purity beryllium metal. However, no permanent agreement had been reached with this Company by the end of 1946 and hence no contract had been issued by this time. (Ref. 91, 92, 93, 94).

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It must be mentioned that early in 1944 Clifton Products, Inc., Painesville, Ohio, had supplied the Project with approximately 100 pounds of beryllium metal in flake form. (Ref. 150, 151, 159, 160). However, this material was used only for small scale experimental metallurgical purposes and it was not required thereafter for any Project purposes.

#### Fabrication of Beryllium Metal

Although a considerable portion of the beryllium metal produced up through 1946 was in the form of raw lump beryllium metal for use in metallurgical studies by various Project installations, in 1946 fairly large scale requirements were received for special fabricated shapes of beryllium. The large scale requirements for fabricated shapes were chiefly for beryllium metal in the form of beryllium blocks measuring up to 2" x 2" x 8" and for beryllium rods measuring up to 3" in diameter. In addition to the multitude of small individual requests, there were three large scale production-type fabrication requirements received during 1946. Two of these requests were received from Los Alamos, one request being approximately 2,000 pounds of beryllium rods ranging from 5/16" in diameter to 3" in diameter, the other being a series of beryllium blocks ranging from 1" cube up to approximately 4" cube, and weighing a total of approximately 1,750 pounds. The third large requirement received in 1946 was from Chicago and involved the fabrication of approximately 3,000 pounds of beryllium metal blocks

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2" x 2" x 8" for use by Dr. Zinn in the study of neutron diffusion and life measurements in beryllium, which measurements were necessary before the design of nuclear reactors employed <sup>ing</sup> ~~in~~ beryllium could be completed.

By the early Summer of 1940, it was apparent that there were two processes by which the three orders could be completed, the first being the standardized extrusion process as developed at Massachusetts Institute of Technology; the second being a newly developed powder metallurgy-sintering process developed at the Brush Beryllium Company. Inasmuch as the requirements had not listed specifically how the fabrication of the various shapes should be made, sample shapes made by both the Brush sintering method and the extrusion method were sent to Los Alamos and Chicago for a comparison and evaluation, together with the request that, as soon as the comparisons were completed, Madison Square Area be notified as to which fabrication process should be used in making the various final pieces.

Within two or three months after the various shapes had been forwarded to Los Alamos and Chicago, instructions were received to fabricate all of the Chicago blocks, as well as all the blocks required at Los Alamos, by the sintering process. However, probably because of the high corrosion resistance of the extruded material, it was requested that the rods required for Los Alamos be manufactured by the extrusion process.



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As a result of these instructions, arrangements were made with Massachusetts Institute of Technology late in 1946 to have sufficient raw metal produced to enable the casting of required extrusion ingots and the subsequent extrusion of these ingots to rods of the desired diameter at the Revere Copper and Brass Company located at Detroit, Michigan. Since Massachusetts Institute of Technology had made it a regular practice to carry on extrusions one or two days per month at the Revere press, it was felt that they could include each month a sufficient number of extrusions for Los Alamos in the regular monthly schedule at Revere to enable the fabrication of the rod order for Los Alamos over a period of several months. No attempt was made by the Madison Square Area Office to contact Revere Copper and Brass Company directly, inasmuch as the number of extrusions to be performed in fulfilling the Los Alamos rod requirement was comparatively small and it was felt that considerable expense could be saved by having Massachusetts Institute of Technology perform the work at Revere on the same days that they normally rent the press for use on their own extrusion work (Ref. 79).

In order to provide for the production of the Los Alamos bricks as well as the bricks for Chicago, Contract No. W-22-075 eng-12 was issued to the Brush Beryllium Company effective May 2, 1946, to provide for the production, at prices varying from \$100 to \$140 per pound, of all these shapes (Ref. 61, 64, 67, 120, 128). Furthermore, this contract was so written that any additional requirements for sintered beryllium shapes could be made by simply supplementing the

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contract (Ref. 62, 63). The first shipment of blocks to Chicago under this contract was made on August 2, 1946, and by the end of December 1946 this requirement was more than 50% completed. Since it had been determined that the shipments for Chicago were of higher priority than the blocks for Los Alamos, full effort was placed in 1946 on the fabrication of the Chicago blocks, and hence no shipments against the Los Alamos order had been made by the end of the year. (Ref. 61 through 74).

It should be pointed out that in addition to these three large scale requirements received in 1946 a great many other fabrication requirements were received and met during this year. These requirements involved small but important shapes in the form of foils, discs, flats, plates, spheres, hemispheres and various oddly shaped pieces. Since the Metallurgical Laboratory at Massachusetts Institute of Technology was exceedingly well equipped, it was possible for this laboratory to produce these small scale, and in many cases, difficult orders within their laboratory. Hence most of the smaller orders, which in many cases involved a research program to develop special techniques, were fabricated at Massachusetts Institute of Technology. A comprehensive listing of the requirements for various fabricated pieces can be obtained by a review of Addendum No. 3.

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Production of Beryllium Oxide

The Project's interest in beryllium oxide was evident as early as the Summer of 1943, at which time the Madison Square Area was requested to arrange for the production of 4,000 pounds of 00 beryllium oxide at the Brush Beryllium Company (Ref. 21). At that time, the Brush Beryllium Company was capable of manufacturing approximately two to three hundred pounds of 00 grade beryllium oxide per week (Ref. 22). Because of the strictness of Project Security, it was impossible to determine the exact final use for the order for Los Alamos. At that time, Los Alamos and cooperating laboratories at Chicago and Battelle Memorial Institute were not completely definite as to the specific form in which the beryllium oxide was to be delivered, that is, whether or not it should be high-fired or low-fired oxide, or whether the mesh size should be predominantly in the higher range or the lower range (Ref. 23). However, by the end of November sufficient work had been performed by the users of the oxide to indicate that the 4,000 pounds should be of the high-fired type, and hence Brush was instructed that the oxide should be high-fired, re-ground, and acid washed to remove impurities (Ref. 24, 25). It was found by Brush, however, that in high-firing the oxide considerable unanticipated losses occurred, and in order that Brush would not lose money, revised costs were allowed to cover the increased manufacturing cost of the high-fired beryllium oxide (Ref. 26, 27). By May 1943 it was possible to complete deliveries on the 4,000 pounds of beryllium oxide, and from May 1944 through July 1945, a series of

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supplements were issued to Contract Eng-78 to provide for the production and delivery of beryllium oxide, in many cases sintered into special shapes, to various Manhattan Project installations as required. (Ref. 28 through 34).

In October 1945, requests had been received in the Madison Square Area Office for beryllium oxide which actually had been fused and then reduced in size to -60 mesh. Facilities at the Brush Beryllium Company would not enable this Company to completely fuse beryllium oxide and hence the Brush Company, through a subcontract with the Norton Company of Worcester, Massachusetts, was able to arrange for the fusion of approximately 2,000 pounds of raw beryllium oxide (Ref. 35). It was felt that this 2,000 pounds after grinding would give approximately 1,500 pounds of -60 mesh beryllium oxide, the quantity required by the Manhattan Project. Hence on November 8, 1945, Supplement No. 8, which was later modified by Supplement No. 10 to Contract W-7401 eng-78, was issued to Brush to provide for the manufacture of approximately 1,500 pounds of Norton fused beryllium oxide for delivery to the Manhattan Project (Ref. 36). The manufacture of this material was completed in early February 1946 (Ref. 37).

In early 1946, the design of the Daniels nuclear reactor was well under way and considerable discussions were held between members of the Beryllium Section and the reactor design group at Chicago pertaining to the possibility of the manufacture of the

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entire moderating structure of this pile from beryllium oxide (Ref. 99, 100). As anticipated by the reactor design group, the beryllium oxide would be fabricated in the form of hollow hexagonal bricks but no concrete information was available as to the density and special physical characteristics of such bricks. Thus, in order to determine the various characteristics imparted to such bricks by the use of varying kinds of beryllium oxide and various methods of oxide fabrication, arrangements were made for the procurement of 5,000 pounds each of Brush SP grade beryllium oxide and 5,000 pounds of Brush GC grade beryllium oxide to be fabricated into the hexagonal bricks for use in development work on the Daniels Pile. The 5,000 pounds of SP grade beryllium oxide was to be sent to the A C Spark Plug Division of the General Motors Corporation in Flint, Michigan, where it would be fabricated into bricks by the use of a cold press followed by a sintering operation method. On the other hand, the 5,000 pounds of Brush GC grade oxide was to be shipped to the Norton Company for fabrication into the hexagonal bricks by the hot press process which had been specially developed at the Norton Company. In order to provide the oxide for these special fabrication orders, Supplements Nos. 11 and 12 to Contract W-7401 eng-75 were issued in late 1946 to Brush to provide for the production of the 5,000 pounds each of the GC grade beryllium oxide and the SP grade beryllium oxide. (Ref. 39, 40, 41, 42, 105).

In addition to the beryllium oxide ordered from Brush, it was decided by the reactor development group to attempt the fabrication

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of fluorescent grade beryllium oxide as manufactured by Clifton Products, Inc. Hence Contract No. W-31-109 eng-19 effective November 4, 1946 was awarded to Clifton Products, Inc. to provide for the production of 500 pounds of refractory grade beryllium oxide for the Manhattan Project. This material, which was of primary interest to the Norton Company for use in their hot pressing method, was to be delivered to that Company for test. (Ref. 50, 51, 52).

Fabrication of Beryllium Oxide

Until the interest of the Daniels Group in fabricated forms of beryllium oxide, most beryllium oxide manufactured for the Manhattan Project was delivered to the ultimate user as the unfabricated powder. There were occasions from time to time as far back as 1943, when requests for fabricated material were received. Thus, in order to supply the requirement for 600 bricks 3" x 3" x 6" (made from Brush GC beryllium oxide), Contract No. W-7425 eng-27 dated December 23, 1943 was issued by the Madison Square Area to the Fansteel Metallurgical Corporation of North Chicago, Illinois (Ref. 116). This contract provided for the manufacture of the 600 bricks which were delivered to Los Alamos. Other than this fabrication contract, little fabrication of beryllium oxide was done on a commercial scale until the fabrication discussions were started, in 1946, with the Daniels Pile Group. During this early period, considerable beryllium oxide was manufactured into crucibles, but this

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work was performed chiefly at the user's site, such as Massachusetts Institute of Technology, Chicago, Battelle Memorial Institute, and Iowa State College. Occasionally each of these organizations placed orders for oddly shaped crucibles or crucible stopper rods with such companies as the Norton Company or the McDaniel Refractory Company, but no large scale commercial program was initiated.

However, late in 1946 discussions and negotiations were undertaken by the Madison Square Area with both the Norton Company and the A O Spark Plug Division of General Motors concerning the fabrication of 5,000 pounds of Brush beryllium oxide to be sent to each of the two companies. Special tests were being made late in 1946 by each company to develop the most efficient and practical means for the employment of their own process in the fabrication of beryllium oxide. However, the fabrication techniques had not developed to such an extent nor had negotiations proceeded far enough to enable definitive fabrication contracts to be issued to each of these organizations by the end of 1946.

#### Production of Beryllium Nitride

In the Summer of 1946, considerable interest was expressed both at Chicago and Oak Ridge in the possibility of manufacturing beryllium nitride for use in nuclear reactors for the manufacture of Carbon 14. Since specific requests for over 200 pounds of beryllium nitride had been received in the Madison Square Area Office, steps were undertaken to provide production methods for the manufacture of this material. Since no known production method for the

manufacture of beryllium nitride was available, requests were issued to both Brush Beryllium Company and Clifton Products, Inc., asking that suitable methods be investigated for the production of beryllium nitride (Ref. 119). To assist both Brush and Clifton, Professor W. G. Schumb of Massachusetts Institute of Technology, operating through Contract W-7405 eng-175, acted as a consultant to both Brush and Clifton in providing actual operating data on the conversion of beryllium metal to beryllium nitride by the reaction of hot ammonia or nitrogen gas directly on the metal (Ref. 83). By June 1946, it was definitely established, as a result of a study of samples submitted by Brush and Clifton respectively, that the nitride as manufactured by the Clifton process was far superior in quality to that manufactured by Brush, and hence in July production contract No. W-31-109 eng-18 dated July 10, 1946, was issued to Clifton for the manufacture of 100 pounds of beryllium nitride (Ref. 44, 46). This contract was again supplemented on October 9, 1946 to provide for further production of an additional 120 pounds of beryllium nitride. All beryllium nitride was made by Clifton Products through the use of beryllium metal flake, owned and furnished by the Government (Ref. 45). By the end of 1946, a total of 135 pounds of beryllium nitride had been delivered and it was anticipated that during January 1947 the production of the entire required amount would be completed.



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DEVELOPMENT

As of September, 1945 the research and development work on beryllium had been performed chiefly at four project installations, e.g., University of Chicago, Massachusetts Institute of Technology, Iowa State College and Battelle Memorial Institute. Of the four, Iowa State had been interested mainly in methods of procuring crude metal while Chicago, M.I.T. and Battelle concerned themselves primarily with methods of fabricating the metal. Since the work on development of methods for producing crude beryllium metal had not been performed on a priority basis at Iowa, the work could be done only when time was available from higher priority projects. The work on the production of beryllium metal at Ames during 1945 and 1946 never was placed on a high priority project basis and by the end of 1946 the Ames method for producing beryllium metal from the fluoride, although successful, apparently was not capable of competing with the methods then in use or contemplated for use at the Brush Beryllium Company or The Beryllium Corporation. The work that Ames performed resulted in a good method for producing beryllium metal from the fluoride which was based on reacting powder magnesium with powdered fluoride and allowed the reaction to take place in an open bomb resulting in the production of beryllium metal which was interspersed with a considerable amount of slag. Ames had also done considerable work on production of beryllium fluoride by passing hydrogen fluoride gas over beryllium oxide and/or beryllium hydroxide

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at elevated temperatures. However, by the end of 1946, neither the Ames method for producing metal nor the Ames method for producing beryllium fluoride had yet been developed to a point where the process as a whole was commercially feasible. Plans were under way by the end of 1946, however, for the continuation of this work; and based on the scale of development assigned to this project it was expected that at least another year to a year and a half might be spent on development before conclusive and efficient processes could be evolved.

The fabrication development work which was being performed at Chicago, M.I.T. and Battelle was continued, at the request of Madison Square Area, and in many cases increased in scope and expenditure of effort. Actually, the fabrication and development work at each of these installations was not under the direct jurisdiction of the Madison Square Area since the work being performed at Chicago was under the jurisdiction of the Chicago Area, while the Battelle and M.I.T. laboratories were supervised directly from Oak Ridge (Reference Nos. 75, 76, 77, 78). The Madison Square Area Office though, through the cooperation of both Chicago and Oak Ridge, as well as the individual laboratories themselves, set up coordinated programs at each of the three institutions, each program aimed at developing several means of producing fabricated shapes of the types which, it was anticipated, would be required by other Manhattan District installations.

As the work at M.I.T. progressed, greater and greater emphasis was placed on the fabrication of beryllium, until perhaps three-fourths

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of all effort being expended at the M.I.T. Laboratory in 1946 was on investigation of beryllium metallurgical and fabrication problems. As a result, early in 1946 the Beryllium Section acquired technical responsibility for supervision over the work being performed at the M.I.T. laboratory under contract W-7405 Eng-175, while administrative supervision of this contract remained the responsibility of Oak Ridge. This arrangement, however, soon proved to be highly inefficient, inasmuch as it was virtually impossible in many cases to determine where the line between technical and administrative supervision was to be drawn. Hence, during the Summer of 1946, administrative control of the M.I.T. project was gradually turned over to the Madison Square Area and by the end of Fall of 1946 all administration, both technical and administrative was the responsibility of the Madison Square Area. (Ref. 75 thru 89, 147)

A summary of major developments on beryllium fabrication techniques performed at the Manhattan District's metallurgical laboratories during the years 1945 and 1946 is as follows:

- (a) Development of a successful method of hot rolling beryllium by the use of iron jackets.
- (b) Development of methods of vacuum casting beryllium metal into various shapes and ingots suitable for extrusion.
- (c) Development of air casting techniques for beryllium metal.
- (d) Development of a technique for the extrusion of beryllium metal by use of an iron jacket cladding a beryllium ingot.
- (e) Techniques for machining both cast and extruded beryllium metal.

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(f) Methods of welding beryllium metal.

(g) Development of preliminary methods of forged beryllium metal.

As has been mentioned previously, the ~~quality of~~ beryllium metal supplied by the Brush Beryllium Company under contracts W-7401 Eng-60 and W-22-075 Eng-10 was of a quality which could not be used for large scale nuclear application inasmuch as the impurities contained therein were of such magnitude as to increase the effective neutron capture cross section of beryllium beyond allowable limits. During 1945 a study of future long range requirements indicated that the use of beryllium metal in actual nuclear reactors was a possibility within the next two or three years (Ref. 54) and, hence, plans were started with the Brush Beryllium Company for a special program of research and development to provide a new production process for the manufacture of extreme high purity beryllium metal. Hence, in November, 1946, contract W-22-075 Eng-11 was entered into with the Brush Beryllium Company to enable that company to perform development work for the Manhattan District on a new process for the conversion of beryllium oxide to beryllium metal, which process would be designed to produce beryllium metal of such purity that the effective total danger summation of impurities would not be greater than 30 millibarns. (Ref. 53, 55, 56, 57) Briefly, the development work was to be carried along the lines of converting beryllium oxide to ammonium beryllium fluoride in the standard way, the decomposition of the ammonium beryllium fluoride in the feed end of a sublimation retort, and the subsequent

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sublimation and condensation of the resulting beryllium fluoride to produce a beryllium fluoride of extremely high purity. Investigation was also to be undertaken as to the possibilities of effecting a reaction between beryllium fluoride and magnesium in the vapor state, which if proved to be successful would produce a simple one or two step process for the manufacture of high purity beryllium metal from beryllium oxide. In addition, Brush was to undertake the study of other means of the beneficiation of beryllium-containing ores directly to the fluoride without passing through the oxide step. Thus it was hoped that a reduction of the cost of beryllium metal could be effected by the development of an overall process for the direct conversion of beryllium in ore to beryllium fluoride, the subsequent purification of the fluoride by sublimation, and the reduction of such fluoride to beryllium metal with magnesium.

By the end of 1946, considerable success had been obtained on a laboratory scale to indicate that the process being developed under the Brush program was successful, and plans had been started for the construction of a pilot plant which would incorporate the developments on a semi-plant scale and would give sufficient data on the operation of the new process to design full scale plant revisions to incorporate the new process.

As a result of experimental work being performed on Clifton flake metal at M.I.T., it appeared that the flake metal, when used in certain fabrication processes, such as extrusion and rolling, imparted a greater strength as well as other desirable properties to the resultant.

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fabricated piece. (Ref. 95) Furthermore, it appeared that an additional method of producing experimental high purity metal could be evolved by further development work on the electrolytic process for fabrication of flake metal. Therefore, with the approval of the Research Division at Oak Ridge, Contract W-31-109 Eng-19 was issued to Clifton Products, Inc. on November 19, 1946 in the amount of \$51,000 to enable this company to establish a development program for the development of a revised process for producing experimental beryllium metal flake by the electrolysis of beryllium chloride. (Ref. 50, 51, 52, 157, 158) This program was set up to redesign existing processes for the chlorination of pure beryllium oxide such that the chlorination of the oxide resulted in the reduction of the impurity content of the resulting beryllium chloride. Furthermore, the program aimed at the establishment of revised techniques for the conversion of the extremely pure beryllium chloride to pure beryllium metal flake without the contamination of the flake usually found in the electrolytic process. Since this contract was issued late in 1946, the results obtained by the end of 1946 were not sufficiently comprehensive to enable any conclusions to be drawn.

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HEALTH FACTORS

Even before beryllium began to be manufactured in various forms on a large scale for the Manhattan District, it was known that many compounds of beryllium were hazardous, and for this reason special precautions had to be taken to protect workers who were closely associated with the production and handling of these materials. It was known, for example, that the compounds  $\text{BeSO}_4$ ,  $\text{BeCl}_2$  and  $\text{BeF}_2$  were considerably hygroscopic, forming sulfuric, hydrochloric and hydrofluoric acids upon even contacts with small quantities of moisture such as are found on the skin. Thus, painful and injurious burns could be caused by prolonged contact with these injurious salts. It was further known that the inhalation of beryllium compounds had, in many cases, a severe but almost unknown effect on the lungs of workers. Little was known, however, of the nature of the disease caused by the inhalation of beryllium fumes nor was information available as to the tolerance levels below which exposure to dust and fumes of beryllium compounds could be tolerated.

Effort was made at the various beryllium companies to protect workers by the installation throughout the plant of what was then believed to be normal, adequate ventilation of various areas of the production plant which appeared to give high concentrations of beryllium fumes and dusts. However, even with the precautions which were then followed many cases of "chemical pneumonia" appeared to occur in workers, especially after exposure to a high concentration of beryllium

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fumes. Most cases were not reported to be severe, and general rest over a period of two to three months seemed to be sufficient to effect a complete cure. Occasionally, however, deaths resulted from such exposures.

During 1946, because of the increased interest of the Manhattan District in beryllium, the Medical Division of the Madison Square Area, under the supervision of Captain B. S. Wolf, began a thorough investigation of the beryllium health factors. Captain Wolf's group, working in cooperation with other interested groups such as the Massachusetts General Hospital, the Ohio State Board of Health and the Cleveland Clinic, began to uncover evidence which showed that, in addition to the obvious superficial types of beryllium toxicity such as the ability to cause burns and rashes upon the skin and to cause a chemical pneumonia from exposure to large doses of fumes, there existed a more insidious form of the disease, which form did not manifest itself until three to six years after exposure to beryllium. Furthermore, studies showed that the delayed form of the disease was far more serious than the overexposure type and resulted almost invariably in the death of the patient. Even worse, it appeared that the tolerance levels at which susceptibility of the disease was evident were of a very low order and it seemed that in many cases prolonged exposure to very minute quantities of beryllium fumes could result, several years later, in the contraction of the delayed form of beryllium disease. By the end of 1946 there was sufficient information on hand to enable a distinction to be made between the two types of beryllium



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diseases and tentative names were applied to each form. "Acute beryllium pneumonitis" was the name applied to the disease resulting from a short exposure to a large concentration of beryllium fumes which disease resulted usually, within a period of two to three days, in the contraction of respiratory disorders of varying intensity. To the long range type of disease, obtained from a prolonged exposure to low concentrations of beryllium fumes, which resulted several years later in the contraction of serious and almost inevitably fatal lung disorders, the name "chronic beryllium pneumonitis" was given.

By the end of 1946 an investigation into the cause and nature of the disease was being undertaken by the Medical Group and studies were being made as to means of determining the exact exposure tolerances of beryllium for the prevention of such diseases. (Ref. 108) In addition, work had been started on the means of treatment of such diseases and a study had been started on improved means of ventilation and preventive measures which could be installed at the beryllium producers' plants and which would prevent the contact of the workers with beryllium fumes and dust.

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SECURITY

Prior to the dropping of the first atomic bomb on August 6, 1945 all activities of the Manhattan Engineer District were, of course, classified, and hence all interests of the Manhattan District in beryllium were afforded Secret classifications. All contracts written for the procurement of beryllium and beryllium compounds, together with the specifications and quantities of the materials were classified Secret and the fact that the Manhattan District was purchasing beryllium compounds was classified Secret.

Subsequent to the dropping of the first atomic bomb on August 6, 1945 and the immediate release of the Smyth Report, the fact that the Manhattan District was interested in beryllium as a moderator became known and hence this portion of security was lifted. Furthermore, in spite of the fact that a good portion of the facilities at the Brush Beryllium Company and Clifton Products was used to manufacture beryllium for the Manhattan District, the general plant processes used for the manufacture of this beryllium and beryllium products were not considered classified, inasmuch as the processes used by each of these companies had, with minor exceptions, been widely published in technical journals.

In the latter part of 1945 and in 1946, therefore, no attempt was made to conceal the fact that the Manhattan District was producing beryllium and beryllium compounds, but exact specifications, both physical and chemical, were classified as were the quantities of material being produced. In addition, as special production and fabrication

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improvements and techniques were developed specifically in order to meet new and highly specialized requirements of the Manhattan District, these techniques were classified Secret. Thus, for example, when the Brush Beryllium Company developed for the Manhattan District a process for simplified sintering of beryllium metal, a process which gave highly desirable physical properties to beryllium heretofore unobtainable, this process was classified Secret by the Manhattan District and special precautions to safeguard the process were set up.

Strict security measures continued to apply to all production development programs and metallurgical development programs pertaining to the supply and fabrication of beryllium since the large scale use of beryllium in the atomic program depended considerably on the success of these development programs. Furthermore, the work being performed, and the results being obtained from these development programs were such as to give this country an apparent substantial lead in the use of beryllium for nuclear purposes and in many cases reactors and other devices could be built of a higher efficiency and specialized purpose by the use of beryllium, provided, of course, that beryllium could be supplied in fabricated forms of such purity and physical characteristics as the specialized use of beryllium required. Hence, the development programs on beryllium, set up to develop techniques by which beryllium could be manufactured and fabricated in the specialized forms, were given full security treatment by the Manhattan District.

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CONTRACTUAL COMMITMENTS

In the beryllium program, through the end of 1946, approximately thirty-eight separate contractual actions were taken to provide for the production of beryllium metal, beryllium oxide, metal shapes, oxide shapes, and development work on production and fabrication of beryllium and beryllium oxide. During the same period, ten separate prime contracts were issued and maintained in order that this work could be carried out. A summary of all contractual actions concerning beryllium is given in Addendum No. 4 and includes contract numbers, contractor's names, contractual dates, values and brief descriptions of the scopes of work.

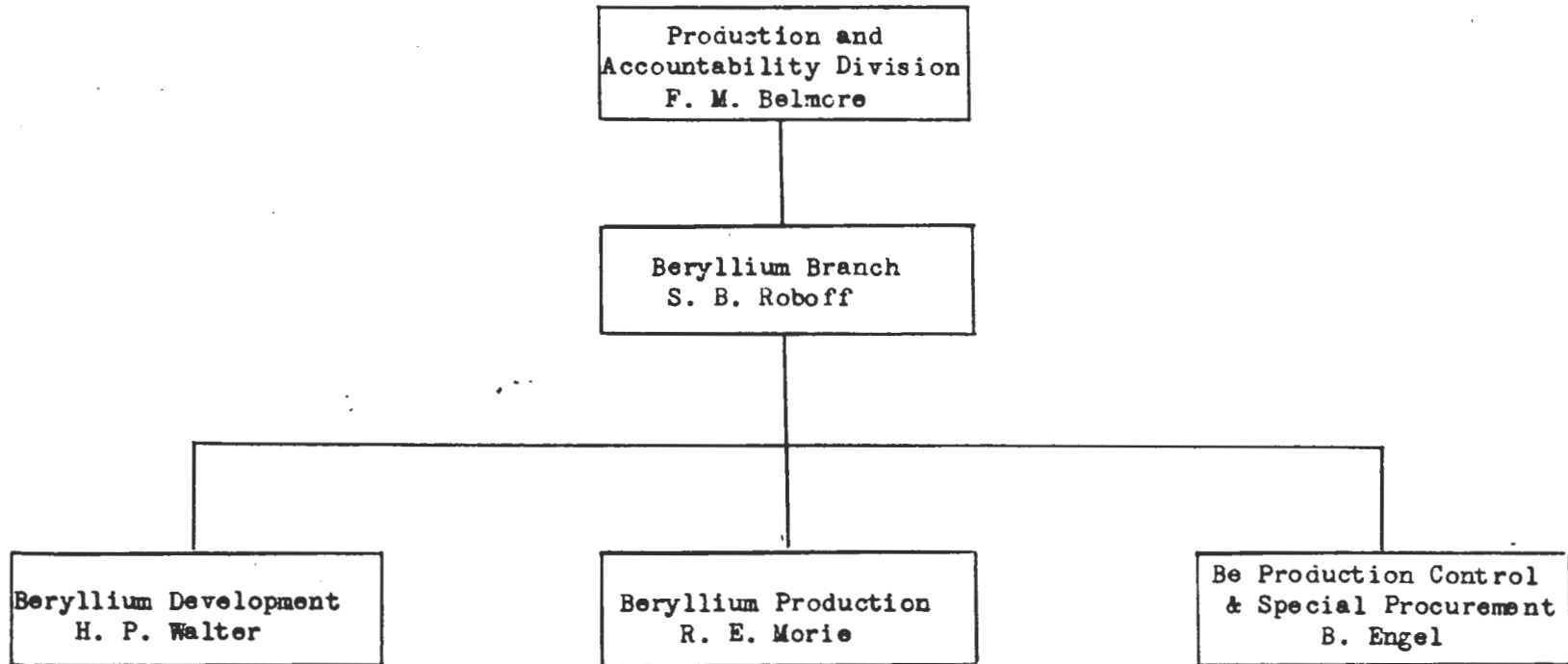
Wherever possible, it was the policy to have beryllium metal, beryllium oxide and beryllium shapes produced by the Manhattan District on unit price contracts. This was possible because there was sufficient production information on hand at the various producers to enable firm prices to be quoted to the Manhattan District. However, because of the very specialized nature of the work involved in the production, fabrication and development of beryllium, together with the security requirements, it was seldom if ever possible to issue invitation for bids. Therefore, whenever unit prices for beryllium products were quoted, cost breakdowns were made for each quoted price and were thoroughly reviewed and examined by Madison Square Area, in order that the unit prices quoted were substantiated in fact. Furthermore, as production under these unit price contracts was affected, checks were made on production costs in order that future price quotations would have up-to-date financial backing.

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In the case of development and experimental contracts, wherever possible it was the policy to issue such contracts of the lump sum type in order that much administrative handling could be eliminated. However, whenever such lump sum contracts were issued for development work, provisions were made in the contract to include downward revisions of costs upon conclusion of the work in order that savings in cost affected by the contractor during the course of the work could be realized by the Government. In the case of the Massachusetts Institute of Technology developmental contract W-7405 Eng-175, the work being performed was of such a varied nature and the work load was so unpredictable that it was virtually impossible to ascertain, with any great accuracy, lump sum amounts for which contracts should be written. Therefore, in this case the work was performed on a cost-plus-overhead (no fee) type of contract, wherein all costs incurred by the contractor were directly reimbursed by the Government. However, in this case expenditures by the contractor were carefully scrutinized (and subject to approval) by the Contracting Officer.

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ADDENDUM NO. 1



ORGANIZATION CHART  
Beryllium Branch  
December, 1946

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Description of the Clifton Process for Producing  
Beryllium Oxide and Beryllium Metal

Addendum No. 2

As of December, 1946

Beryllium ore is crushed so that it will pass through a 2 inch mesh screen in a jaw crusher and is ground dry in a ball mill operating in a closed circuit with an air classifier. The ground product is mixed with sodium carbonate where it is fed to an oil fired furnace, melted and then poured. The resulting frit is crushed and ground and then digested with sulfuric acid. The sulfated material is then dried in a rotary kiln. The dried product is passed through a hammer mill to break up any cake which may have formed, and it is then added to water. The resulting liquor is centrifuged and then clarified by filtration for removal of the silica. The filtrate is then concentrated by evaporation in steam-jacketed glass-lined tanks and ammonium sulfate is added. After further evaporation, the liquor is cooled and ammonium alum is crystallized out and removed by means of a centrifuge. The liquor is diluted with water and sodium carbonate is added to bring the solution to pH<sub>4</sub>. The iron is to be removed in this manner by precipitation as ferric hydroxide and subsequent filtration. The filtrate is brought up to pH<sub>8</sub> by further addition of sodium carbonate with the resultant precipitation of pure beryllium hydroxide, Be(OH)<sub>2</sub>. The hydroxide is removed from the liquor by means of the centrifuge, dried and ignited in an oil-fired furnace to yield beryllium oxide.

Clifton Products produces beryllium metal in the form of flake. This is accomplished by first chlorinating beryllium oxide in the presence of carbon. The beryllium chloride is placed in chrome-nickel-iron alloy

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pots, where it is fused at high temperature with sodium chloride. The alloy pots act as a cathode and a carbon electrode serves as an anode. Current is passed through the electrolyte until substantially nothing remains but pure sodium chloride. The beryllium metal is collected on the inside of the pot in the form of flakes.

At the present time, Clifton is capable of producing approximately 5,000 pounds of beryllium oxide per month, of which 2,000 to 3,000 pounds now goes directly for commercial use. The metal plant is not now in operation but could be set up within three to four weeks to produce 100 pounds of metal per month in flake form. Additional metal production capacity could be made available by increasing the electrolytic facilities.

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Description of the Brush Process for Producing  
Beryllium Oxide and Beryllium Metal

As of December, 1946

Beryllium ore in the form of beryl is crushed to 2 inch mesh size in a jaw crusher. The crushed ore is then fed to a carbon brick-lined 110 volt AC electric arc furnace. The ore is melted in this furnace and is tapped therefrom as a glassy substance, which is then quenched in water from a temperature of about 2900°F. The resulting frit is dried in a kiln from where it is passed to a ball mill operating in a closed circuit with an air classifier. The ball mill product is sulfated in a digester by the addition of hot sulfuric acid. The effluent from the digester passes to a Bird centrifuge, where the dehydrated silica,  $\text{SiO}_2$ , is removed. The liquor from the centrifuge is essentially aluminum sulfate,  $\text{Al}_2(\text{SO}_4)_3$ , and beryllium sulfate,  $\text{BeSO}_4$ , plus minor impurities, notably iron and alkaline earth metal sulfate. The liquor from the centrifuge is transferred to an evaporating kettle, and thence to a crystallizer where ammonium sulfate,  $(\text{NH}_4)_2\text{SO}_4$ , is added. The product in the crystallizer passes to a centrifuge where the precipitated ammonium alum,  $\text{NH}_4\text{Al}(\text{SO}_4)_2$ , is removed. The liquor from the centrifuge becomes essentially beryllium sulfate,  $\text{BeSO}_4$ . The beryllium sulfate is evaporated and is passed through a filter for the removal of finely divided ammonium alum and calcium sulfate. The filtrate is then crystallized in a tank and a pure beryllium sulfate is thus obtained. The beryllium sulfate is then calcined in a kiln to form beryllium oxide,  $\text{BeO}$ .

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If a pure oxide is desired, the beryllium sulfate obtained in the normal manner is dissolved in distilled water instead of being calcined in the kiln. This solution is then saturated with hydrogen sulfide, and impurities are precipitated or removed by filtration. The filtrate then passes to a glass-lined crystallizer and then to a centrifuge. The salt is then ignited in a batch furnace, resulting in the production of beryllium oxide of low iron content.

To convert beryllium oxide to beryllium metal by the Brush method, ammonium bifluoride,  $\text{NH}_4\text{HF}_2$ , is added to beryllium oxide in water. The liquor so obtained is treated with hydrogen sulfide, both on the acid and alkaline sides, to precipitate impurities. The filtered liquor is then passed to evaporators where ammonium beryllium fluoride,  $(\text{NH}_4)_2\text{BeF}_4$ , is crystallized out. The product from the crystallizer is centrifuged and the salts are then dried. The ammonium beryllium fluoride is heated in a gas-fired furnace where ammonium fluoride,  $\text{NH}_4\text{F}$ , is driven off and beryllium fluoride,  $\text{BeF}_2$ , remains as a liquid. This liquid is then allowed to solidify and is broken into lumps. The lump beryllium fluoride is then melted in a gas-fired furnace and reduced with magnesium, resulting in the production of beryllium metal and a slag consisting chiefly of magnesium fluoride. The beryllium metal floats to the top of the furnace and is allowed to cool. The cake is removed from the furnace and broken into lumps for further processing.

At the present time the Brush Beryllium Company is capable of producing approximately 15,000 pounds of regular grade beryllium oxide per month, or approximately 5,000 pounds of high grade beryllium oxide per

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month. By the end of January, 1947, it is anticipated that the production of beryllium metal at Brush will be about 1,500 pounds per month.

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Description of the Beryllium Corporation Process  
for Producing Beryllium Oxide and Beryllium Metal

As of December, 1946

The ore is first ground in a ball mill to about 70% -200 mesh. It is then mixed with sodium ferric fluoride and made into wet briquettes. These are heated for about one hour at 750°C. The reaction which occurs is the following:



The alumina, silica, and iron oxides are not <sup>acted</sup> attacked upon. Only enough fluoride is added to react with the beryllium oxide content of the ore. After the baking operation, the material is in a sintered, easily ground condition. Grinding is performed in a wet pebble mill. The sodium beryllium fluoride is the only soluble portion of the sinter and is leached out with water at room temperature at a concentration of approximately 3 grams per liter beryllium oxide. About 95% of the beryllium contained in the ore is put in the solution. The insolubles are filtered off and the solution is treated with caustic soda to precipitate beryllium hydroxide. Enough caustic soda is mixed with the solution to redissolve the precipitate and form a sodium beryllate solution. This is then heated to about 85°C and more sodium beryllium fluoride is added with good agitation, the temperature being maintained until all of the beryllium is precipitated as hydroxide. This is then filtered in water, the filtrate being a sodium fluoride solution. The hydroxide cake is ignited at a temperature of about 800°C, which converts it to the anhydrous oxide.

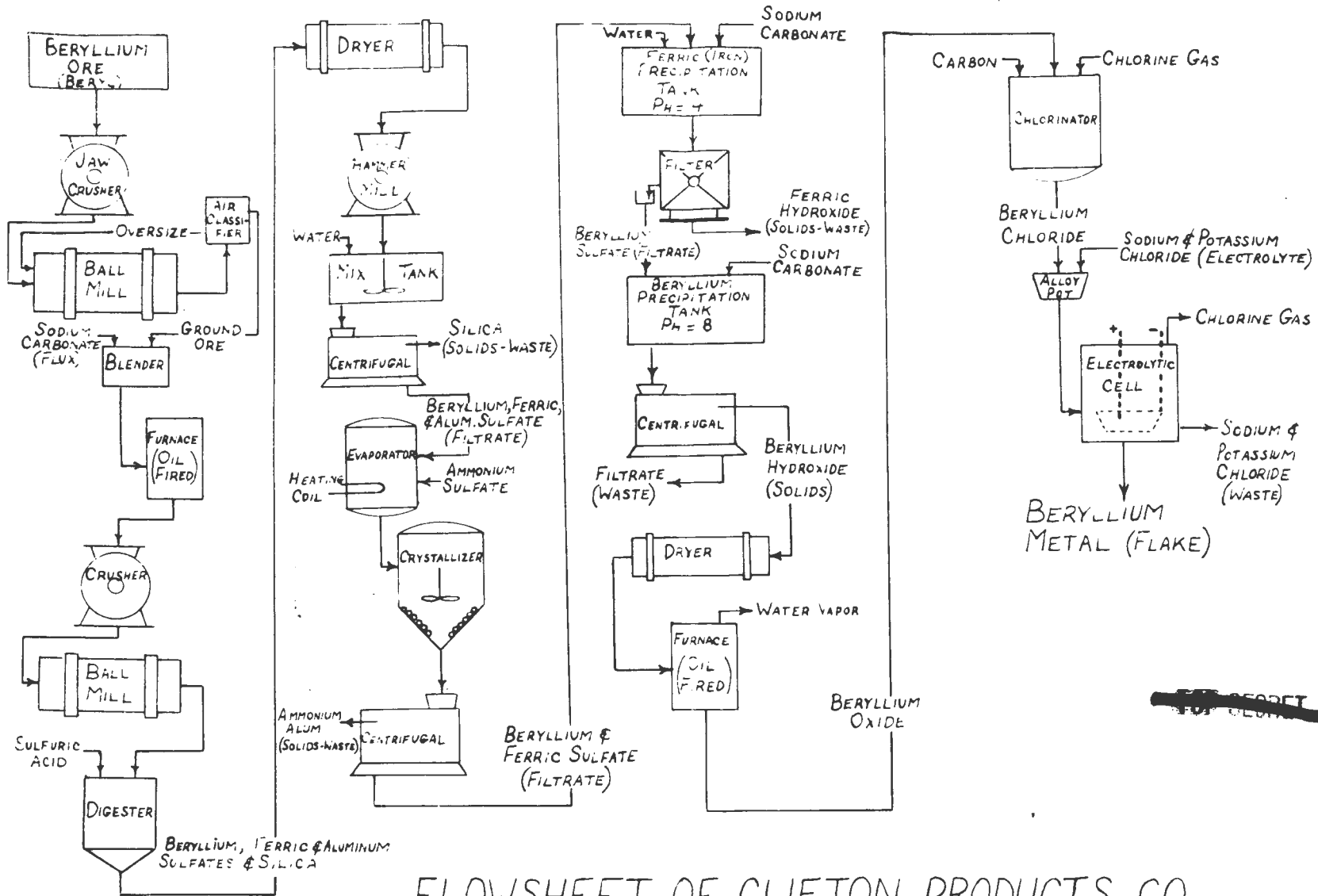
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At the present time, metal is produced in small quantities at The Beryllium Corporation in the following manner: Beryllium oxide is thoroughly mixed with ammonium bifluoride in nickel pans which are then placed within a magnesium-lined furnace and heated for 16-24 hours at a temperature of 450°C. The resultant beryllium fluoride is then placed in a gas heated furnace, heated beyond its melting point, and then reduced to beryllium metal by the addition of magnesium lumps. A change in the Beryllium Corporation process for producing metal may be put into operation within the next few weeks. This change would involve a conversion of beryllium hydroxide directly to beryllium basic acetate, and the subsequent fluorination of this acetate to beryllium fluoride. This fluoride is converted to metal in the same manner as described above. It is expected, however, that by using the beryllium acetate intermediate a beryllium metal of much greater purity will be obtained than that being produced at the present time. It is also anticipated that beryllium basic acetate could also be converted to a highly pure beryllium oxide.

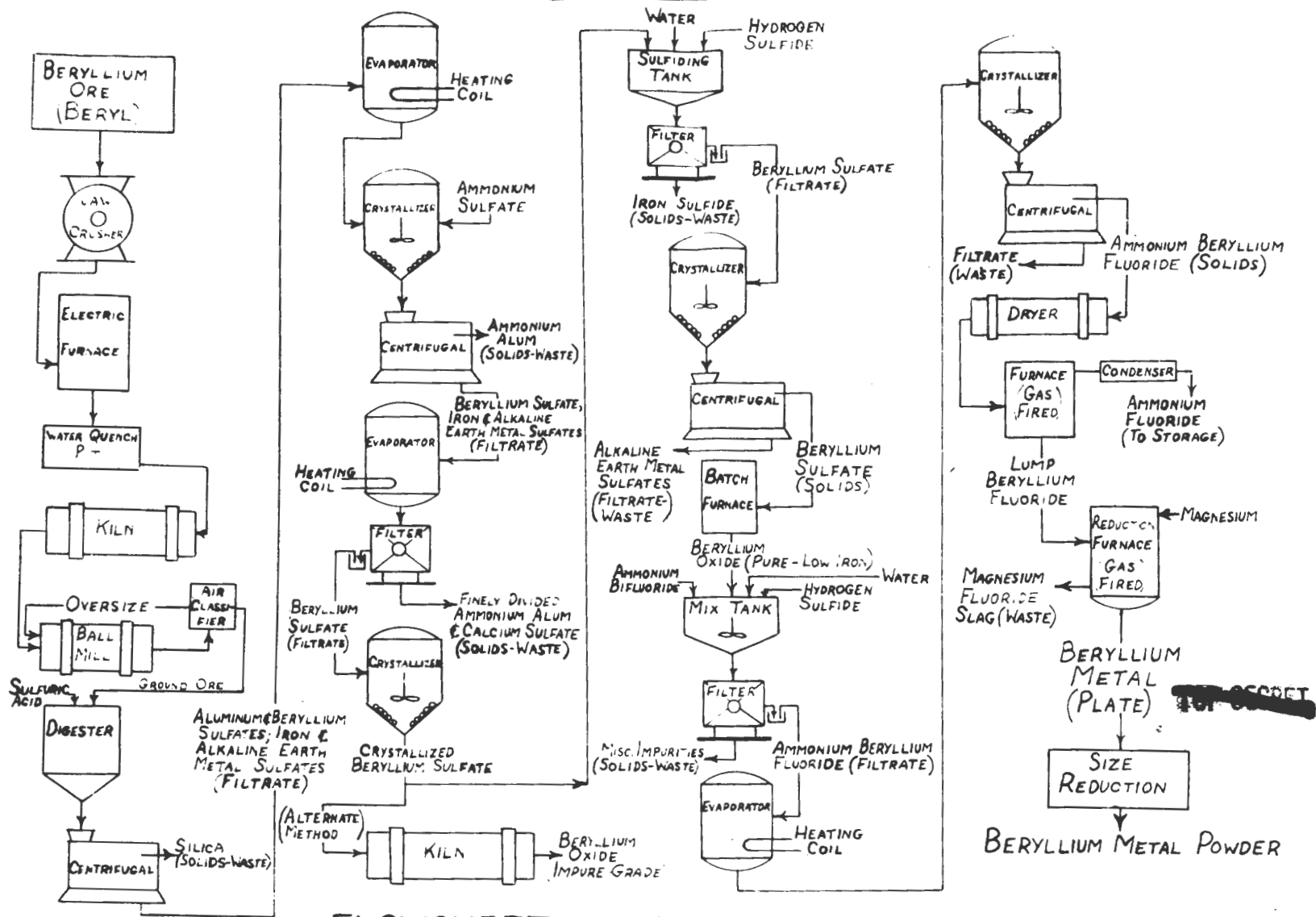
The Beryllium Corporation is not capable at the present time of producing beryllium oxide meeting project specifications. It is expected, however, that within three to four weeks The Beryllium Corporation will be capable of producing approximately 800 pounds per month of high purity beryllium metal. Facilities for producing high purity beryllium oxide and additional metal could be installed within four to eight weeks after notification has been given to The Beryllium Corporation.

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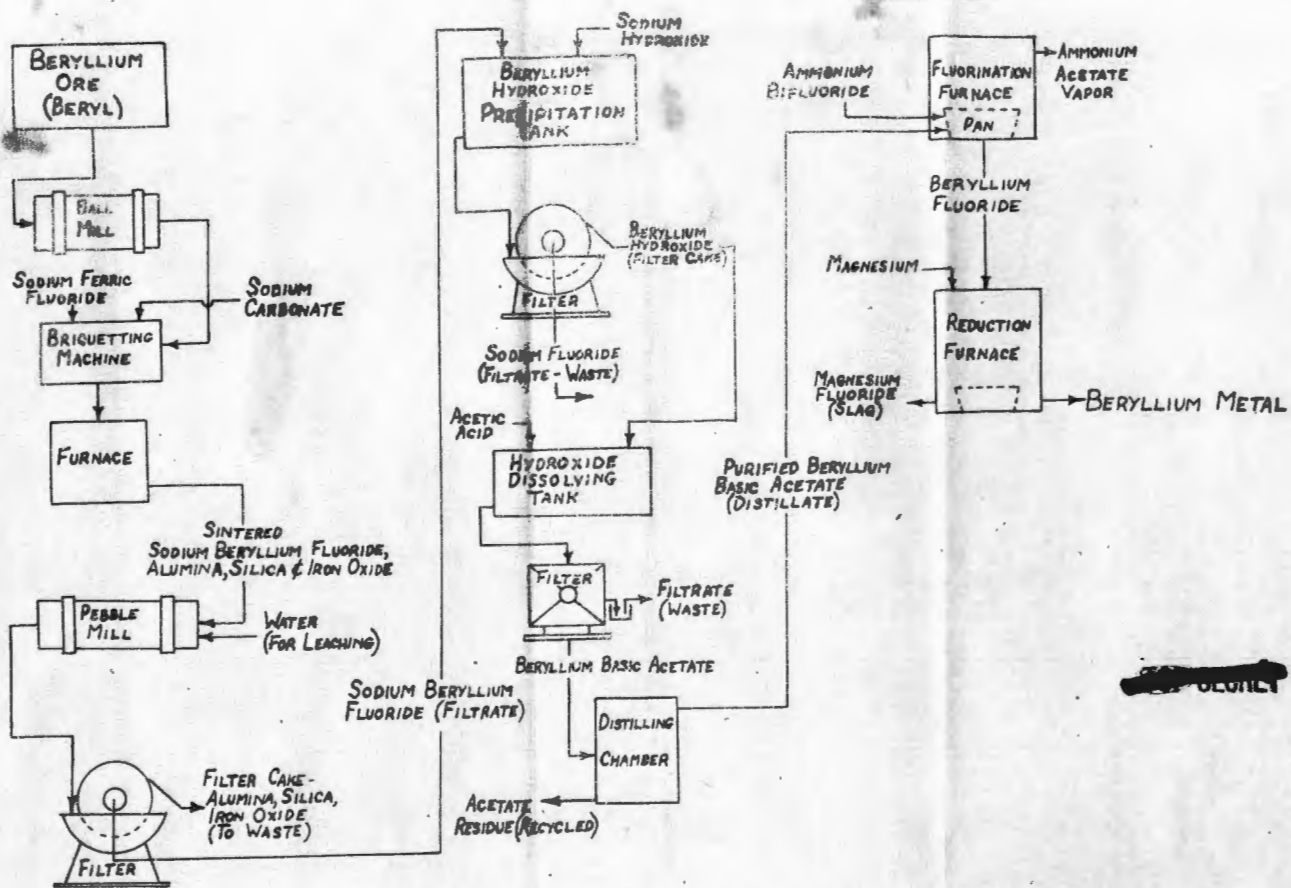


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FLWSHEET OF BERYLLIUM CORP.



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PROCUREMENT OF BERYLLIUM METAL AND OXIDE

Addendum 3

<u>ROCHESTER</u>	<u>Quantity</u>	<u>Source</u>	<u>Contract or Purchase Order</u>	<u>Final Shipment Date</u>
Beryllium Powder	1 lb.	Brush	44285	November 8, 1946
Beryllium Oxide	20 lbs.	Clifton	44286	
Beryllium Sulphate	20 lbs.	Brush	44291	
Beryllium Oxyfluoride	5 lbs.	Brush		
Beryllium Powder, technical grade	1 lb.	Brush	47022	November 22, 1946
<u>BERKELEY</u>				
Beryllium	7,000 grams	M.I.T.	W-7405 Eng-175	September 12, 1946
<u>AC SPARK PLUG</u>				
Beryllium Oxide, fluorescent grade	150 lbs.	Clifton	42942	December 11, 1945
Beryllium Oxide, SP grade	100 lbs.	Brush	43901	July 8, 1946
Beryllium Oxide	30 lbs.	Clifton	44099	August 22, 1946
Beryllium Oxide, fused, fluorescent grade, 30 mesh	10 lbs.	Clifton	44201	September 18, 1946
	20 lbs.			
	20 lbs.			
Beryllium Oxide, special grade	200 lbs.	Brush	44241	October 1, 1946
Beryllium Oxide, specially prepared	60 lbs.	Clifton	44333	November 7, 1946

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ADD 3

	<u>Quantity</u>	<u>Source</u>	<u>Contract or Purchase Order</u>	<u>Final Shipment Date</u>
<u>IOWA STATE COLLEGE</u>				
Beryllium Oxide	100 lbs.	Clifton	28192	August 28, 1944
Beryllium Fluoride	400 lbs.	Brush	29124	November, 1944
Beryllium Fluoride	50 lbs.	Brush	W-7401 Eng-60	October 9, 1944
Beryllium Oxide	900 lbs.	Brush	W-7401 Eng-78	1944
Beryllium Oxide	100 lbs.	Brush	W-7401 Eng-78, S5	March, 1945
Beryllium Oxide	110 lbs.	Clifton	-	July 7, 1945
Beryllium Fluoride, anhydrous	250 lbs.	Brush	43179	January 15, 1946
Beryllium Oxide, refractory grade, 200 mesh	225 lbs.	Clifton	43906	July 12, 1946
Beryllium Hydroxide, 99.9%	100 lbs.	Clifton	43906	July 12, 1946
Beryllium Oxide	560 lbs.	Brush	43735	September 26, 1946
Beryllium Oxide, refractory grade, 200 mesh	1,000 lbs.	Clifton	44101	December 4, 1946
Beryllium Oxide	20 lbs.	Clifton	28054	-
Beryllium Oxide	50 lbs.	Clifton	35645	-
<u>NEW MEXICO</u>				
Beryllium Oxide	2,000 lbs.	Brush	W-7401 Eng-78, S6	April 9, 1946
Beryllium Flake, (surplus V matl.)	100 lbs.	Middlesex	Middlesex	September 25, 1945
Product 58 (minimum assay 90%)	75 lbs.	Middlesex	Middlesex	November 8, 1945
Beryllium Oxide	4,000 lbs.	Brush	W-7401 Eng-78	1946
Beryllium Oxide, 61 mesh	50 lbs.	Middlesex	Middlesex	January 29, 1946
Beryllium Flake	100 lbs.	M.I.T.	-	April 5, 1946
Beryllium Metal, lump	200 lbs.	Middlesex	Middlesex	July 11, 1946
Beryllium Rods	-	M.I.T.	W-7405 Eng-175	July 29, 1947

	<u>Quantity</u>	<u>Source</u>	<u>Contract or Purchase Order</u>	<u>Final Shipment Date</u>
<u>NEW MEXICO (continued)</u>				
Beryllium Metal	6,000 lbs.	Brush	36812 (MIF) 36911 (Battelle) W-7401 Eng-60, SB W-22-075 Eng-10 W-22-075 Eng-10, SI	May 15, 1946

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	<u>Quantity</u>	<u>Source</u>	<u>Contract or Purchase Order</u>	<u>Final Shipment Date</u>
<u>NORTON COMPANY, CANADA</u>				
Beryllium Oxide, 60 mesh	225 lbs.	Middlesex	Middlesex	February 11, 1946
Beryllium Oxide, 200 mesh, Clifton fluorescent grade	60 lbs.	Clifton	43380	March 12, 1946
Beryllium Oxide, batch mixed, Brush S.P. grade	600 lbs.	Brush	43512	April 18, 1946
Beryllium Oxide, Clifton grade	200 lbs.	Clifton	43726	May 31, 1946
Beryllium Oxide, Brush, G.C.	2	Brush	43727	June 14, 1946
Beryllium Oxide, G.C.	200 lbs.	Brush	44356	October 18, 1946
Beryllium Oxide, Brush, G.C.	200 lbs.	Brush	47012	October 18, 1946
Beryllium Oxide, Densified, fluorescent	1-3/4 lbs.	Clifton	47107	November 30, 1946
Beryllium Oxide Blocks	125 lbs.	Norton	47235 (fabrication only)	December 3, 1946
Beryllium Oxide, fluorescent grade	30 lbs.	Clifton	47187	December 17, 1946
Beryllium Oxide Samples G.C. 1250°C	80 lbs.	Brush	47331	December 17, 1946

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CHICAGO

<u>Item</u>	<u>Source</u>	<u>Contract or Purchase Order</u>	<u>Final Shipment Date</u>	
Beryllium Fluoride	800 lbs.	Brush	W-7401 Eng-60	March 29, 1945
Beryllium Oxide	300 lbs.	Brush	W-7401 Eng-78, S5	March, 1945
Beryllium Oxide	1500 lbs.	Brush	W-7401 Eng-78, S7	September 5, 1945
Product 58	200 lbs.	Middlesex	Middlesex	October 17, 1945
Beryllium Oxide, GC grade	50 lbs.	Brush	42964	November 19, 1945
Beryllium Oxide, fluorescent grade	50 lbs.	Clifton	42965	November 19, 1946
Beryllium Oxide, S.P.	50 lbs.	Brush	43153	January 8, 1946
Beryllium Nuggets	50 lbs.	Middlesex	Middlesex	January 11, 1946
Beryllium Oxide, metal grade	50 lbs.	Clifton	43152	January 16, 1946
Beryllium Oxide, hi-fired	2,000 lbs.	Brush	42873 (Supl. 8 to Contract No. W-7401 Eng-78)	January 25, 1946
Beryllium Oxide, 60 mesh (sample)	2 lbs.	Middlesex	Middlesex	January 29, 1946
Product 88A, B, C, D	50 lbs.	Middlesex	Middlesex	April 29, 1946
Beryllium Metal, 97.5% (Product 88)	100 lbs.	Middlesex	Middlesex	May 3, 1946
Beryllium Oxide, fluorescent grade	10 lbs.	Clifton	43597	May 7, 1946
Beryllium Hydroxide	2 lbs.			
Beryllium Sulphate	5 lbs.			
Beryllium Nitrate	10 lbs.	Clifton	43475	May 19, 1946
		Brush	43474	

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	<u>Quantity</u>	<u>Source</u>	<u>Contract or Purchase Order</u>	<u>Final Shipment Date</u>
<u>CHICAGO (continued)</u>				
Product 89	100 lbs.	Middlesex	Middlesex	June 4, 1946
Beryllium Oxide, hi-fired, refractory	200 lbs.	Clifton	43728	June 5, 1946
Beryllium Nitrate, Mixture 36B, minimum nitrogen content 42%	10 lbs.	Brush	43912	August 13, 1946
Beryllium Oxide, metal-grade	30 lbs. 1 lb.	Clifton	44099 44100	August 22, 1946
Beryllium Oxide, Brush fused, special grade 300-325 mesh	200 lbs.	Brush	47013 (for grinding only)	November 22, 1946
Beryllium Metal	50 lbs./mo. thru 12/46	Middlesex	Middlesex	December, 1946
Beryllium Fluoride	200 lbs.	Brush	W-7401 Eng-60	-
Beryllium Oxide	200 lbs.	-	35283	-
Beryllium Nitrate, Mixture 36C	95 lbs.	Clifton	W-51-109 Eng-18	January 15, 1947
Beryllium Blocks	2,000 lbs. to 3,000 lbs.	Brush	W-22-075 Eng-12	July 10, 1947

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	<u>Quantity</u>	<u>Source</u>	<u>Contract or Purchase Order</u>	<u>Final Shipment Date</u>
<u>CLINTON LABORATORIES</u>				
Beryllium Sphere, solid w/radius about 4 1/2"	1 lb.	M.I.T.	W-7405 Eng-175	July 31, 1946
Beryllium Oxide				
GC	5 lbs.	Brush	44494	November 18, 1946
SP	5 lbs.	Brush	44494	
Fluorescent	5 lbs.	Clifton	44495	
Beryllium Metal, premium grade technical grade	100 lbs. 100 lbs.	Brush	W-22-675 Eng-10, S5	December 31, 1946
<u>BATTELLE</u>				
Beryllium Product 58P, high flux	75 lbs.	Brush	42586	September 20, 1945
Beryllium Metal, (min. assay 85%)	500 lbs.	Middlesex	Middlesex	October 22, 1945
Beryllium Oxide, pure, 60 mesh, high fired	50 lbs.	Middlesex	Middlesex	January 23, 1946

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	<u>Quantity</u>	<u>Source</u>	<u>Contract or Purchase Order</u>	<u>Final Shipment Date</u>
<u>BATTELLE (continued)</u>				
Beryllium Metal, VC	100 lbs.	Middlesex	Middlesex	October 18, 1946
Beryllium Metal	116 lbs.	-	36812	-
<u>MASSACHUSETTS INSTITUTE OF TECHNOLOGY</u>				
Beryllium Oxide				
325 mesh Clifton	100 lbs.	Clifton	43021	October 6, 1945
20 mesh	50 lbs.	Brush	43022	
Surplus Flake Beryllium (V material)	300 lbs. or more, 348 available)	Middlesex	Middlesex	October 10, 1945
Product 58, lump	100 lbs.	Middlesex	Middlesex	November 20, 1946
Beryllium Sulphate, anhydrous	10 lbs.	Clifton	43062	December 10, 1945
Virgin Light Metal Nuggets (88)	5,000 lbs.	Middlesex	Middlesex	January 11, 1946
Beryllium Oxide, Prod. 48				
Type 3	50 lbs.	Brush	43263 Brush	February 1, 1946
60 mesh	50 lbs.	Clifton	43264 Clifton	
8 mesh	25 lbs.	Clifton		
Brush Lump Metal	400 lbs.	Middlesex	Middlesex	February 4, 1946

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	<u>Quantity</u>	<u>Source</u>	<u>Contract or Purchase Order</u>	<u>Final Shipment Date</u>
<u>MASSACHUSETTS INSTITUTE OF TECHNOLOGY (continued)</u>				
<b>Beryllium</b>				
Lump Metal, highest quality	150 lbs.	Middlesex	Middlesex	March 7, 1946
Flake Metal, 98%	35 lbs.	Clifton	43293	
Flake Metal, 99%	27 lbs.	Clifton	43293	
<b>Beryllia Crucibles</b>				
Standard Brush BeO	5 lbs.	Brush	43356	April 22, 1946
Norton fused Brush BeO	5 lbs.			
<b>Beryllium Oxide</b>				
Product 48, Type 6	50 lbs.	Clifton	43594	May 4, 1946
	150 lbs.	Brush	43462	May 9, 1946
<b>3" diameter Rod, not less than 4" or greater than 8"</b>				
	1 lb.	Brush	43937	July 31, 1946
<b>Product 48, Type 6 High-fired from S.P. grade, 325 mesh</b>				
	150 lbs.	Brush	44102	September 26, 1946
<b>Beryllium Oxide, 8 mesh, refractory grade</b>				
	50 lbs.	Clifton	44402	November 1, 1946
<b>Beryllium Oxide, GC, (Norton Grade)</b>				
	10 lbs.	Brush	44496	November 21, 1946
<b>Product 48, Type 6</b>				
	150 lbs.	Brush	43874	September 26, 1946
<b>Process Q Scrap</b>				
	25 lbs.	Chicago	-	November 27, 1946

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	<u>Quantity</u>	<u>Source</u>	<u>Contract or Purchase Order</u>	<u>Final Shipment Date</u>
<u>MASSACHUSETTS INSTITUTE OF TECHNOLOGY (continued)</u>				
Beryllium Metal	100 lbs. immed. 100 lbs. monthly	Middlesex	Identification No. LSV-20	December, 1946
Process Q Material	20 lbs. (powder)	Brush	44242	February 26, 1947
2" x 4"	6 pieces			
1/2" x 4" x 4"	6 pieces			
3/8" x 4" x 6"	6 pieces			
Brush Beryllium Oxide	50 lbs.	Middlesex	Middlesex	-

GENERAL ELECTRIC, SCHENECTADY

Beryllium Oxide, high-fired	100 lbs.	Clifton	44480	November 11, 1946
Beryllium Oxide, low density	10 lbs.			
Beryl Ore	10 lbs.			

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Beryllium Contract Data - Manhattan Project, Beryllium Branch

<u>Contractor</u>	<u>Contract</u>	<u>Effective Contract Date</u>	<u>Value</u>	<u>Scope</u>
Brush Beryllium Company	W-22-075 eng-10	July 9, 1945	\$193,000	Purchase or manufacture of Schedule A equipment. Manufacture of 4390 lbs. of Be metal.
	" " S.1	Oct. 17, 1945	\$45,000 additional	Raises total Be to be manufactured to 5570 lbs.
	" " S.2	July 9, 1945	\$7,898	Additional Schedule A equipment (amendment of basic contract).
	" " S.3	April 5, 1946	None	Provides for rental payments on government-owned facilities.
	" " S.4	April 17, 1946	\$10,698.18	Purchase of 701.52 lbs. of remelted Be metal from Be metal supplied by Government.
	" " " "		\$630	For maintaining Schedule A equipment on standby basis.
	W-7401 eng-76	Sept. 15, 1943	\$16,200	Manufacture of 4000 lbs. of BeO.
	" " S.1	Dec. 27, 1943	\$257.30	Provides for overtime labor.
	" " S.2	May 5, 1944	\$9,406 additional	Purchase of 1256 additional pounds of BeO.
	" " S.3	May 12, 1944	(Government re-imbursed) (\$3,415.29)	Government sold to contractor 662 lbs. of BeO.
	" " S.4	Sept. 1, 1944	\$6,780.82	Purchased 1000 lbs. BeO
	" " S.5	Jan. 19, 1945	\$29,725	Purchased 2900 lbs. BeO
" " S.6	Apr. 23, 1945	\$10,250	" 1000 lbs. BeO	
" " S.7	July 11, 1945	\$19,987.50	" 1950 lbs. BeO	
" " (S.8)	Nov. 8, 1945	\$8,581.50	1430.25 lbs. fused BeO.	
" " (S.10)	Feb. 1, 1946			
" " S.11			Production of 5,000 lbs. GG BeO.	
" " S.12			Production of 5,000 lbs. SP BeO.	

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Beryllium Contract Data - Manhattan Project, Beryllium Branch (Continued)

<u>Contractor</u>	<u>Contract</u>	<u>Effective Contract Date</u>	<u>Value</u>	<u>Scope</u>
Brush Beryllium Company	W-7401 eng-60	Aug. 18, 1943	\$89,000	Manufacture of 2000 lbs. of Be metal and 100-200 lbs. of BeF <sub>2</sub> .
	" "	S.1 May 11, 1944	None	Provides for rental of Government facilities.
	" "	S.2 Sept. 1, 1944	None	Allows Contractor to assign rights to bank.
	" "	S.3 Sept. 11, 1944		Basic Contract amended as follows: Purchase of 1830 lbs. (max.) Be lump. 16-20 lbs. of Be billets 100-150 lbs. of Be billets 100-200 lbs. of BeF <sub>2</sub>
	" "	S.4 Jan. 17, 1945	\$5,500	1000 lbs. impure Be metal
	" "	S.5 Mar. 30, 1945	\$68,708	1500 lbs. of Be metal
	" "	S.6 Apr. 24, 1945	\$7,404	15 pieces each of Parts #1, #2A & #2B (Be-Al fabricated shapes) 16 pieces of Be-Al fabricated shapes
	" "	S.7 July 6, 1945	None	Restoration of Premise Clause added
	" "	S.8 July 27, 1945	\$66,975	1500 lbs. of Be Metal
	W-22-075 eng-11	Nov. 6, 1946	\$52,000	Research and development work on the production of hi-purity beryllium metal by reacting BeO with ammonium bifluoride and reducing the beryllium fluoride thus formed with magnesium to obtain the hi-purity metal.

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Beryllium Contract Data - Manhattan Project, Beryllium Branch (Continued)

<u>Contractor</u>	<u>Contract</u>	<u>Effective Contract Date</u>	<u>Value</u>	<u>Scope</u>	
Brush Beryllium Company	W-22-075 eng-12	May 2, 1946	\$400,000	Purchase of 4,000 lbs. of beryllium metal blocks fabricated by powder metallurgy.	
Clifton Products, Inc.	W-31-109 eng-18	July 10, 1946	\$8,750	100 lbs. Beryllium Nitride	
	S.1	Oct. 9, 1946	\$10,500	120 lbs. Beryllium Nitride	
	W-31-109 eng-19	Nov. 4, 1946	\$3,500	500 lbs. BeO, refractory grade	
	W-31-109 eng-16	Nov. 19, 1946	\$51,000	Research and Development of a low cost beryllium of extreme purity.	
Massachusetts Institute of Technology (Definitive contract not prepared in Madison Square Area)	W-7405 eng-175.S.1	May 1, 1943	Not to exceed \$60,000	Conduct studies and experimental investigations in metallurgy on a cost plus overhead basis.	
	" "	S.2	Jan. 1, 1944	Increased to \$312,200	Conduct studies and experimental investigations in metallurgy on a cost plus overhead basis.
	" "	S.3	July 1, 1944	Increased to \$1,032,500	Conduct studies and experimental investigations in metallurgy on a cost plus overhead basis.
	" "	S.4	July 1, 1945	Increased to \$1,800,000	Conduct studies and experimental investigations in metallurgy on a cost plus overhead basis.
	" "	S.5	March 15, 1946		Scope of work increased to cover the furnishing of personnel to render services in connection with the "Crossroads Project."

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Beryllium Contract Data - Manhattan Project, Beryllium Branch (Continued)

<u>Contractor</u>	<u>Contract</u>	<u>Effective Contract Date</u>	<u>Value</u>	<u>Scope</u>
Fansteel Metallurgical Company	W-7425 eng-27 " " S.1	Dec. 23, 1943) ) )	\$44,200	Production of 720 BeO bricks from Brush GC oxide by hot press process Bricks to be 3" x 3" x 6"

44-111

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Brush Beryllium Company Contract W-22-075 eng-10

Contract:

Definitive Contract W-22-075 eng-10 with Brush Beryllium Company was signed September 21, 1945 and called for a total quantity of 2195 units (2 lbs. = 1 unit) of Product 58 and/or Product 59. Price was stated as \$88.50 per unit for Product 58; \$83.20 for Product 59. (Products 58 and 59 are Be Metal.)

Correspondence:

A summary of important correspondence pertaining to the contract follows:

<u>Reference</u>	<u>Description</u>
No. 1	September 8, 1945, Memorandum to Files from J. Pinkston, Senior Engineer, subject "Future Supply of Be Metal", states that supply of metal to M.E.D. is dependent upon one company whose business foundation appears shaky and whose price is high due perhaps to inefficiency and large overhead. At the time Brush has an outstanding order for 5,000 lbs. at approximately \$45/lb., which should be filled by March 1, 1946.
No. 2	October 18, 1945, Major Greager to B. Kjellgren (Brush) sets up a method of paying for Be metal according to analysis.
No. 3	October 18, 1945, N. W. Bass (Brush) to S. B. Roboff states that contract called for 4390 lbs. Be, of which 1042 lbs. had been shipped, plus 1000 lbs. in process, leaving 2348 lbs. subject to change in price, specifications, etc.
No. 4	March 8, 1946, C. B. Sawyer (Brush) to Major Kelley states that Brush can produce 1200 lbs./month premium grade (99%) Be; approximately 2000 lbs./month for Product 88 (Be metal in lump form). Premium grade could be raised to 1500 in 4 to 6 weeks; quotes price of \$55/lb. for premium grade. States that contract Eng-10, Supplement 1, is nearing completion.
No. 5	March 15, 1946, Lt. Col. Kelley to F. M. Belmore, designates F. M. Belmore as duly authorized representative for Eng-10.
No. 6	August 21, 1946, R. Cobb (Brush) to Capt. Roboff, gives bid on wrecking A.E.C. installations at Chester Ave. - awarded to Broadway Housewrecking Company for \$675.

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Brush Beryllium Company Contract W-7401 eng-60

Contract:

Brush Contract W-7401 eng-60 provides for production and supply of Be metal and beryllium fluoride (BeF<sub>2</sub>). Supplements call for Be billets, beryllium-aluminum alloys, etc. This contract has been consolidated.

Correspondence:

A summary of important correspondence pertaining to the contract follows:

<u>Reference</u>	<u>Description</u>
No. 7	July 3, 1943, memo Lt. Col. Grenshaw, Corps of Engineers, to Col. K. D. Nichols. Gives requirements for Be for British and Canadian Government. States that at present Brush is only producer of metallic Be and has a capacity of 400-500 lbs./month. \$45/lb. for lump and \$55 for recast metal.
No. 8	August 9, 1943, memo to Lt. Col. Cornell from Major Russell, Corps of Engineers. Requests preparation of UP contract for supply of 2000 lbs. Be and 200 lbs. BeF <sub>2</sub> .
No. 9	September 6, 1943, memo to Mr. Levin (Legal Section) from Lt. Col. Ruhoff, Corps of Engineers. Desire to purchase 4075 lbs. BeO. Delivery of total by November 1943 at \$4.05/lb.
No. 10	August 7, 1943, Mr. Henderson, Chief, Be Section, Minerals Bureau, to Dr. Sawyer (Brush). Gives authority to increase production from present 350 lbs. per month to 600 lbs. per month.
No. 11	February 25, 1944, Major Russell to Brush Beryllium Company. Gives shipments of Be (type A) metal to February 10, 1944 as 403.25 lbs.
No. 12	March 30, 1944, memo to Major Greenstein from Frank Zeitlin. Provides for 40¢/lb. credit for all metal produced in government-owned facilities.
No. 13	December 8, 1944, Capt. Chapman to Area Engineer, Madison Square Area, Attention Mr. Frank Zeitlin. Gives Be estimates for first two quarters of 1945 as 2300 lbs. BeO, 5200 lbs. BeF <sub>2</sub> , and 1500 lbs. Be.

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Brush Beryllium Company Contract W-7401 eng-60 (Continued)

<u>Reference</u>	<u>Description</u>
No. 14	December 14, 1944, Frank Zeitlin, Principal Engineer, to Capt. Hecker. Gives summary history of contract. Explains that utilization requirements changed after signing of contract so that rapid deliveries are no longer required. In addition, Brush had trouble in meeting specifications, and certain changes in specifications covering part of the material were made. At the present time less than one-half of the contractual quantity has been shipped. The contract was written at the request of the Chicago Area.
No. 15	February 15, 1945, Frank Zeitlin to Dr. Sawyer (Brush). Requests quotation for 200 lbs. Be lump metal per month for five or six months <u>in addition</u> to that under Contract eng-60.
No. 16	March 7, 1945, memo F. Zeitlin, Principal Engineer, to Major Kelley. States that production at Brush is about 125 lbs./month metal, 700 lbs./month high fired oxide, plus large amounts of BeF <sub>2</sub> . Our requirement of 200 lbs. BeF <sub>2</sub> per month is being filled, but metal rate is too small.
No. 17	March 28, 1945, memo to Files from John T. Pinkston, Senior Chemist. Explains that remelting is necessary to produce 98% metal, but 96% metal can be produced without remelting.
No. 18	March 29, 1945, F. Zeitlin to Chicago Area Engineer. States that present production rate is 200 lbs./month.
No. 19	July 21, 1945, memo to Files from F. Zeitlin, subject "Be Requirements". Lists new Be requirements and states that expansion of Brush's facilities now in progress will increase production to 1500 lbs./month.
No. 20	October 29, 1945, Lt. Roboff to Capt. Hecker (memo). Requests consolidation of contract as contractor has completed obligations.

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Brush Beryllium Company Contract W-7401 eng-78

Contract:

Brush Contract W-7401 eng-78 provides for production of beryllium oxide (BeO) in various grades and amounts.

Correspondence:

A summary of important correspondence pertaining to the contract follows:

<u>Reference</u>	<u>Description</u>
No. 21	September 6, 1943, memo Major Hadlock to Capt. Spaulding. Allocation of BeO. Request for 4075 lbs. BeO, Grade GC.
No. 22	October 15, 1943, Major Russell to Dr. Sawyer (Brush). States that production of GC oxide in its normal form is at the rate of 300 to 400 lbs/week.
No. 23	October 22, 1943, memo to Files from Major Russell, Corps of Engineers. Concerns the present position regarding procurement of BeO. States that the only firm requirement thus far is for 4000 lbs. of GC oxide - delivery by Nov. 30, 1943. If high-firing or molding is required, contamination resulting may allow use of lower grade SP oxide. Absolute maximum production of GC grade oxide is 950 lbs./week; for SP oxide, 2000 lbs./week.
No. 24	November 27, 1943, Major Russell to Mr. Fletcher (Brush). States that all GC oxide produced in the future under Eng-78 is to be high fired and ground.
No. 25	December 3, 1943, Major Russell to Mr. Fletcher (Brush). States that Brush has practically completed production of oxide under Eng-78, and that high-firing of the oxide will start and continue at a rate of 700 lbs./week until the 4000 lbs. have been so fired. The fired oxide will also be ground and acid washed.
No. 26	January 6, 1944, Mr. Fletcher (Brush) to Major Russell. States that losses in firing are much greater than expected, and anticipates a revision of contract to meet increased costs.

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Brush Beryllium Company Contract W-7401 eng-78 (Continued)

<u>Reference</u>	<u>Description</u>
No. 27	March 18, 1944, Mr. Fletcher (Brush) to Dr. Zeitlin. Gives breakdown of costs incurred in production of 4362 lbs. high-fired oxide. Cost per lb. is stated to be \$3.62/lb. but this does not include any profit figure.
No. 28	April 29, 1944, memo to Legal Section from Frank Zeitlin. Requests preparation of a supplement to Eng-78 to authorize purchase of various quantities and grades of BeO.
No. 29	September 5, 1944, F. Zeitlin to Area Engineer, Chicago Area. Gives prevailing prices of high-fired BeO. Prices range from \$6.25/lb. for -20 mesh to \$10/lb. for -325 mesh.
No. 30	October 10, 1944, F. Zeitlin, memo to Files. Explains that majority of oxide was sent to Fansteel Metallurgical Corp. for fabrication into sintered shapes. Supplement No. 3 permits sale to Brush of unused as well as scrap BeO.
No. 31	January 8, 1945, Frank Humiston (Brush Comptroller) to Dr. Zeitlin. Suggests \$10.25/lb. as the price for 2800 lbs. BeO proposed in Supplement No. 5.
No. 32	July 4, 1945, memo to Capt. Hacker from F. Zeitlin. States that contract Eng-78 was completed approximately May 15, 1945.
No. 33	July 13, 1945, memo by F. Zeitlin to Legal Section. Requests preparation of Supplement No. 7.
No. 34	August 31, 1945, Bengt Kjellgren (Vice Pres. of Brush) to Dr. Zeitlin. Agrees to accept a contract calling for 1000 to 1500 lbs./month of high-fired BeO at \$8.50/lb. For orders less than 1000 lbs./month, the price will be \$10.25/lb.
No. 35	October 26, 1945, N. W. Bass (Brush's Sales Manager) to Lt. Roboff. Refers to proposed supplement to Eng-78 for 1500 lbs. BeO and proposes plan whereby Norton Company would fuse the oxide and reduce to -60 mesh.
No. 36	November 9, 1945, memo Lt. Roboff to Legal Section. States that Supplement No. 8 is being extended and provides for further production (15lb lbs.) of BeO.

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Brush Beryllium Company Contract W-7401 eng-75 (Continued)

<u>Reference</u>	<u>Description</u>
No. 37	January 30, 1946, H. W. Bass (Brush) to Mr. Belmore. States that except for 8 <sup>th</sup> lbs., Supplement No. 8 has been filled.
No. 38	April 4, 1946, H. W. Bass (Brush) to Lt. Fisher, U. S. Engineers Office, states that Supplement No. 10 has been signed.
No. 39	August 20, 1946, H. W. Bass (Brush) to Dr. Oliver Simpson, Metallurgical Laboratory, Chicago. Concerns a 5000 lb. order for rotary fired SF BeO for A G Spark Plug Company. States that basic price is \$3.40/lb.
No. 40	September 30, 1946, memo by Mr. R. Morie (Production Branch) to Legal Section. Requests Supplement No. 11 be prepared.
No. 41	December 2, 1946, J. S. Quidor to Dr. Sawyer (Brush). States Mr. Belmore is delegated representative authority for Eng-75.
No. 42	December 19, 1946, memo by Mr. R. Morie to Legal Section. Requests preparation of Supplement No. 12.

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Clifton Products, Inc. Contract W-31-109 eng-16

Contract:

Contract W-31-109 eng-16 with Clifton Products, Inc. is a research and development contract to develop a low cost beryllium of extreme purity.

Correspondence:

The following correspondence pertains to this contract:

<u>Reference</u>	<u>Description</u>
No. 43	July 3, 1946, F. M. Salmer to Mr. Wendecker (Clifton). Requests that a modification of a previous proposal for a research and development program be submitted. This program would endeavor to produce highly pure Be metal at low cost.

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Clifton Products, Inc. Contract W-31-109 eng-18

Contract:

Contract W-31-109 eng-18 with Clifton Products, Inc. provides for production and supply of beryllium nitride.

Correspondence:

A summary of important correspondence pertaining to the contract follows:

<u>Reference</u>	<u>Description</u>
No. 44	July 11, 1946, F. M. Belmore to E. T. McGown (Clifton). States that Contract Eng-18 is being issued to Clifton which provides for production of 100 lbs. of Mixture 360 (beryllium nitride) at \$87.50/lb.
No. 45	July 15, 1946, memo by Capt. Roboff to Legal Section. Explains that the Government will furnish Clifton with high purity Be metal (Product FLK) for the production of the high purity beryllium nitride (Mixture 360).
No. 46	August 8, 1946, F. M. Belmore to Mr. Windecker (Clifton). Gives suggested specifications for beryllium nitride, and points out that since Si, Mg, and Al tolerances are high, (1%), refractories of MgO, Al <sub>2</sub> O <sub>3</sub> , and SiO <sub>2</sub> are permissible.
No. 47	August 26, 1946, memo R. Morie to Miss A. Hodnett. Requests fiscal clearance for Supplement No. 1.
No. 48	December 12, 1946, Mr. R. A. Anderson, Deputy Advisor on Patent Matters, O.S.R.D. to F. M. Belmore. States that a type C patent clause will be incorporated into Eng-18.
No. 49	December 27, 1946, memo to Legal Section from R. E. Morie. Requests preparation of Supplement No. 2 to Eng-18.

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Clifton Products, Inc. Contract W-31-109 eng-19

Contract:

Clifton Products, Inc. Contract W-31-109 eng-19 provides for production and supply of refractory grade beryllium oxide (BeO).

Correspondence:

A summary of important correspondence pertaining to the contract follows:

<u>Reference</u>	<u>Description</u>
No. 50	November 29, 1946, memo by Col. Deeler to District Engineer, Manhattan District, requests approval to award Contract Eng-19 to Clifton Products, Inc. for 500 lbs. BeO at \$7.00/lb.
No. 51	December 6, 1946, Mr. Rentenbach to Area Engineer, Madison Square Area, approves award of Contract Eng-19 to Clifton Products, Inc. to furnish BeO.
No. 52	January 7, 1947, memo from R. Morie to A. C. Hodnett (Contract Section), requests preparation of Supplement No. 7 to Eng-19.

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Brush Beryllium Company Contract W-22-075 eng-11

Contract:

Brush Beryllium Contract W-22-075 eng-11 is a research and development contract pertaining to the production of high purity Be metal by means of reacting beryllium oxide ( $\text{BeO}$ ) with ammonium bifluoride ( $\text{NH}_4\text{HF}_2$ ) and reducing the resulting beryllium fluoride ( $\text{BeF}_2$ ) with magnesium ( $\text{Mg}$ ) to give Be metal.

Correspondence:

A summary of important correspondence pertaining to the contract follows:

<u>Reference</u>	<u>Description</u>
No. 53	<p>April 10, 1946, C. B. Sawyer (Brush) to Capt. James Cox, Manhattan District. Gives a suggested plan for production of high purity Be. Outlines Process Q, Process R, Process S, and Process T.</p> <p>Process Q: Aggregation of powdered Be into fine grained, high-density material.</p> <p>Process R: Low temperature vacuum sublimation of <math>\text{BeF}_2</math>.</p> <p>Process S: Production of Be powder from the Product of Process R by reaction with Na or Mg vapor.</p> <p>Process T: Production of crude <math>\text{BeF}_2</math> directly from the ore.</p> <p>Suggests that Manhattan District agrees to purchase certain amounts of materials produced by the above processes. The work would be carried out at Plancor Pilot Plant 669.</p>
No. 54	<p>May 1, 1946, Major Barnett to Capt. Roboff. Requests negotiations be started with Brush for a research and development contract leading to the production of high-purity Be metal.</p>
No. 55	<p>June 6, 1946, Memo Capt. Roboff to Legal Section, requests that letter contract Eng-11 be drawn up.</p>
No. 56	<p>June 6, 1946, Capt. Roboff to Legal Section, defines code words "Sawgran" and "Process RS" as follows:</p> <p>Sawgran - Be metal powder of certain specification.</p> <p>Process RS - Purification of <math>\text{BeF}_2</math> by sublimation, and direct production of Be powder (Sawgran) by reaction with Mg or Na vapor.</p>

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144



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Brush Beryllium Company Contract W-22-075 eng-11 (Continued)

<u>Reference</u>	<u>Description</u>
No. 57	July 1, 1946, Lt. Col. A. W. Oberbeck to District Engineer, Manhattan District, Attention: Dr. Chapman. Refers to final proposal for research and development program submitted by Brush, and explains that only Process R and S are to be investigated at this time.
No. 58	July 25, 1946, Mr. Bass (Sales Manager, Brush) to Col. Reeler, states that Supplement No. 1 has been signed.
No. 59	August 29, 1946, Mr. N. W. Bass (Brush) to Mr. Quidor states that Supplement No. 2 has been signed.
No. 60	September 22, 1946, Mr. Bass (Brush) to Mr. Quidor, states that Supplement No. 3 has been signed.

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Brush Beryllium Company Contract W-22-075 eng-12

Contract:

Contract W-22-075 eng-12 with Brush Beryllium Company calls for the production and supply of 4,000 lbs. of Be metal blocks fabricated by powder metallurgy.

Correspondence:

A summary of important correspondence pertaining to the contract follows:

<u>Reference</u>	<u>Description</u>
No. 61	May 17, 1946, Capt. Roboff to Legal Section. Requests that letter contract Eng-12 be drawn up.
No. 62	June 25, 1946, Mr. N. W. Bass (Brush) to Col. Seeler, U. S. Engineers Office, states that Supplement No. 1 to Eng-12 has been signed.
No. 63	July 25, 1946, N. W. Bass (Brush) to Col. Seeler, states that Supplement No. 2 has been signed.
No. 64	July 26, 1946, Mr. Humiston (Treasurer, Brush) to Capt. Roboff, quotes price of \$139.50/lb. of finished product for Be metal blocks. Price is subject to certain reductions.
No. 65	August 23, 1946, F. M. Belmore to N. W. Bass (Brush). Lists specifications for the Be metal blocks and states that maximum total impurity, exclusive of oxygen, be no greater than 0.4%.
No. 66	October 1, 1946, Mr. Bass (Brush) to Mr. Quidor, states that Supplement No. 4 has been signed.
No. 67	October 2, 1946, Mr. Humiston (Brush) to R. E. Norie. Gives price of Process Q billets from which protective coatings have been stripped as \$100.50/lb. Rough machined billets are \$114/lb.
No. 68	October 7, 1946, Mr. Roboff to Mr. Bass (Brush), suggests that work proceed immediately on fabricating bricks by a new sieving method.

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Brush Magnesium Company Contract W-22-075 eng-12 (Continued)

<u>Reference</u>	<u>Description</u>
No. 69	November 4, 1946, Mr. Bass (Brush) to Mr. Roboff. Gives quotation of \$94.50/lb. for "Process Q" billets, stripped but not machined.
No. 70	November 5, 1946, Mr. Musiston (Brush) to Mr. Roboff. Concerns proposed purchase of 25,000 lbs. of Mg from Dominion Magnesium, Ltd. Suggests that Government purchase Mg to avoid payment of duty.
No. 71	November 14, 1946, Mr. Bass (Brush) to Mr. R. E. Morie. Gives revised codes for identification of metal blocks.  Process Q - premium or remelt metal, and use of Wellman Press. QX - Same as Q except crushed and sieved metal is used instead of remelt. QT - Same as QX except that press is not used. QJ - Same as Q except that press is not used prior to sintering.
No. 72	November 20, 1946, memo Mr. M. Ancher to Files. Concerns a visit to the Brush Plant and gives a particularly clear description of their undertakings. Describes the two methods of making Be powder; methods for producing Be blocks from powder, etc.
No. 73	December 5, 1946, Mr. Roboff to Legal Section. Requests that Contract W-22-075 eng-12 be drawn up.
No. 74	December 13, 1946, E. W. Bass (Brush) to Mr. Quidor, states that Supplement No. 7 to Letter Contract eng-12 has been signed.

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Massachusetts Institute of Technology Contract W-7405 eng-175

Contract:

Contract W-7405 eng-175 with Massachusetts Institute of Technology is a research and development contract involving work with Be as well as other metals and compounds.

Correspondence:

A summary of important correspondence pertaining to the contract follows:

<u>Reference</u>	<u>Description</u>
No. 75	August 31, 1943, Major Harold Greenstein to N. McL. Sage (MIT). Returns a signed copy of Contract to M.I.T.
No. 76	February 17, 1944, Maj. Greenstein to McL. Sage (MIT). Requests return of signed Supplement No. 7 to contract.
No. 77	February 22, 1944, Maj. Greenstein to Area Engineer, Chicago (Capt. Karl). Transmits proposed Supplement No. 9 for approval.
No. 78	December 7, 1944, 1st Lt. Lord to Area Engineer, Madison Square Area, requests copy of Supplemental Agreement No. 2.
No. 79	August 21, 1945, M.I.T. to Revere Copper and Brass Co. Furnish 1250 ton press and labor for extrusion work.
No. 80	January 2, 1946, Col. Brown, Oak Ridge, to Area Engineer, Madison Square Area, discusses transfer of administrative responsibilities from Boston Area office to Manhattan Area when Boston closes. Transfer to take place Feb. 15, 1946 according to letter.
No. 81	March 8, 1946, memo from John Chipman to McL. Sage (MIT). Dr. Kaufmann has become Director of Project. The former separation of the project into divisions has been abandoned. Group leaders now report directly to Kaufmann.
No. 82	March 11, 1946, Maj. Grenger to Dr. Chipman, (MIT). Discusses plans for comprehensive Be analysis program.
No. 83	March 12, 1946, F. M. Belmore to Dr. Kaufmann. Acquaint representatives of Brush and Clifton with MIT method of making Be nitride.

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Massachusetts Institute of Technology Contract W-7405 eng-175 (Cont'd)

<u>Reference</u>	<u>Description</u>
No. 84	April 12, 1946, Dr. Kaufmann (MIT) to S. B. Roboff. Describes difficulty in producing extruded Be rods greater than 2" diameter. Require new melting equipment to produce larger billets.
No. 85	May 9, 1946, memo S.B. Roboff to J. Moran, Property Section, planned move of MIT to Hood Milk Co. Building.
No. 86	June 4, 1946, memo to files by Maj. Hearon. Mentions Eng-22, Eng-40, and Eng-175 to be terminated and replaced by contracts on cost plus percentage-for-overhead plus 7% fixed fee.
No. 87	June 29, 1946, Kaufmann (MIT) to S.B. Roboff. Estimates cost of producing finished extruded Be rod starting with Brush metal (\$14/lb.) as \$95/lb.
No. 88	Sept. 6, 1946, J. S. Lidor to District Engineer, Manhattan District, Oak Ridge. Encloses letter dated August 30, 1946, delegating representative authority to Mr. Belmore for approval.
No. 89	Sept. 12, 1946, Dr. Kaufmann to F. H. Belmore. Plans to test Brush Process 4 metal on behavior during fabrication.

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Beryllium Corporation - MCH

Correspondence:

A summary of important correspondence follows:

<u>Reference</u>	<u>Description</u>
No. 90	August 23, 1943, Lt. Col. Ruhoff to Mr. Gravely, Beryllium Corporation, requests analysis of BeO produced by Beryllium Corp.
No. 91	April 25, 1946, Mr. Gravely (Beryllium Corp.) to F. M. Belmore, states that Beryllium Corp. has developed a new process for production of high purity metal.
No. 92	September 30, 1946, memo to Files from R. E. Morie, states that Beryllium Corporation can produce 99.5% Be metal in pig, lump, or brick form at \$55/lb. Be powder is sold at \$85/lb. In addition, thin metal sheets can be produced. Production to date is on a laboratory scale, however.
No. 93	November 29, 1946, F. M. Belmore to Mr. Gravely, Beryllium Corp., gives specifications for 2,000 lbs. of Be metal blocks, and requests a price quotation.
No. 94	December 26, 1946, Memo to R. Turner, Safety Section from S. B. Roboff, states that the Government is about to enter into production contract and a research and development contract with the Beryllium Corporation.

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General Beryllium Files

Correspondence:

A summary of important correspondence from the General Beryllium Files follows:

<u>Reference</u>	<u>Description</u>
No. 95	July 20, 1945, memo to Dr. Chipman from Paul Gordon. States that Clifton flake Be can be successfully extruded by compacting in cans before extrusion. 1/2 inch rods were extruded at 900°C and 1000°C. Grain size is very fine and shows practically complete recrystallization.
No. 96	January 15, 1946, memo to Files from Capt. Roboff. Enumerates Be requirements for 1946; discusses various programs, anticipated as well as those in operation, for producing Be metal, Be shapes, Be compounds, etc. The total Be requirements for 1946 are estimated as 6,000 lbs.
No. 97	March 11, 1946, memo to Files from Capt. Roboff. Concerns a proposed standardization program for Be analysis. The National Bureau of Standards is to prepare standard samples which will then be distributed to various laboratories for analysis. It is thought that the assay of Be for free metal content is in very poor shape, and that a research job would be necessary.
No. 98	March 12, 1946, F. M. Belmore to Capt. Cox, Manhattan District. Gives types of Be metal available, price, and production rates: Be lump metal, 2000 lbs/month, \$45/lb. Be lump metal, premium grade, 1500 lbs/month, \$58/lb. Be flake metal, 600 lbs/month, \$100/lb.
No. 99	August 26, 1946, Lt. Col. Leber to Area Engineer, Madison Square Area. Concerns Be requirements for a proposed power pile which will require between 25,000 and 50,000 lbs. BeO in brick form. An order for 5,000 lbs. BeO from Brush has been placed and will be fabricated into bricks by the Horton Co. These bricks will then be used for experimental purposes.

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General Beryllium Files (Continued)

<u>Reference</u>	<u>Description</u>
No. 100	September 19, 1946, memo to Files from Lt. Comdr. Dunford. Concerns a trip to suppliers of BeO and fabric tons of BeO bricks for the purpose of coordinating procurement of the material for the power pile program. This is an extensive memo and covers all parts of the BeO brick program, including operations at Brush, Clifton, A C Spark Plug Co., Norton Co., etc.
No. 101	October 10, 1946, memo to Files from R. E. Morie. Concerns a Be conference held at Oak Ridge to clarify the Be procurement situation throughout the Manhattan District. This memo is very extensive and gives a breakdown of requirements according to each user. Specifications, types, uses, etc. are listed.
No. 102	October 11, 1946, memo to Col. Nichols from A. V. Peterson, Research Division Director. Recommends Dr. Spedding's (Iowa State) Be program be expanded to pilot plant stage.
No. 103	October 28, 1946, Col. Kirkpatrick to Area Engineer, New York, gives the approved procurement program for Be metal and BeO for 1947.
No. 104	October 28, 1946, memo to Mr. Kelley from S.B. Roboff. This is another extensive memorandum concerning the present status of the Be program.
No. 105	November 29, 1946, memo from Col. Beeler to District Engineer, Manhattan District. Concerns BeO requirements. Requirements for various installations are listed.
No. 106	December 3, 1946, memo to Mr. Turner from Mr. Morie. Lists all installations throughout Manhattan District using Be.
No. 107	December 5,-6, 1946, Paper entitled "Be Analysis Program" gives results of various methods of analysis of Be for Mg, Si, etc.

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General Beryllium Files (Continued)

<u>Reference</u>	<u>Description</u>
No. 108	December 16, 1946, memo to Area Engineer, New York from Mr. Turner. A comprehensive survey concerning Be health hazards.
No. 109	December 16, 1946, memo from G. W. Boyd to Mr. Simpson. Concerns certain functional tests performed upon Norton Company's BeO bricks. Tests were made by placing the bricks in a graphite pile and determining what setting of a control rod was required to maintain a given power level.
No. 110	December 26, 1946, memo from Col. Kirkpatrick to Area Engineer, New York. Confirms approval for a research and development program with Beryllium Corp. of America for a six month period.
No. 111	October 17, 1946, Mr. Merritt and Mr. Selfridge, memo to Files. Gives an exhaustive Be ore survey.
No. 112	November 15, 1946, memo from Mr. Roboff to Files. Concerns possible production of pure Be compounds directly from the ore by means of a newly developed procedure as conceived by Dr. Kerman of Phelps-Dodge Corp. In this process, the briquetted ore would be the anode of an electrical furnace operating with a Cl <sub>2</sub> atmosphere. Recommends initiation of a development program with Phelps-Dodge if possible.
No. 113	December 13, 1946, Col. Beeler to Mr. F. Rockwell, Office of Temporary Controls, Washington, D.C. Requests that the Metals Reserve Board consider raising their Civilian Deficiency of beryllium ore so that the Manhattan District may be sure to meet its 1947 requirements.
No. 114	January 15, 1947, Col. Beeler to F. G. Rockwell, Civilian Production Administration, Washington, D.C. States that Metal Reserve has increased the Civilian Deficiency of beryllium ore by the 2,000 tons as requested by the Manhattan District. Proposes that the 2,000 tons now be purchased directly by the A.M.C. from Metals Reserve.

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A-10

Special Listings

Reference

Description

No. 115

September 6, 1945, Report Lt. S. B. Bobeff to Major A. E. Kelley, subject "The Production and Fabrication of Beryllium." Gives comprehensive report on status of overall Beryllium Production and Development facilities throughout the U.S.A. together with recommendations for a coordinated Manhattan Project Program (on file in Madison Square Area).

No. 116

December 20, 1943, letter Major G. W. Russell to Dr. F. H. Driggs of Insteel Metallurgical Corp. concerning the production of 600 BeO bricks by the hot press method from Brush 90 BeO, (on file in correspondence folder W-7425 eng-27).

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Brush Beryllium Company - HCN

Correspondence:

A summary of important correspondence follows:

<u>Reference</u>	<u>Description</u>
No. 117	January 23, 1946, F. M. Belmore to Mr. Bass (Brush) states that maximum requirement for $\text{Be}_3\text{N}_2$ (Beryllium Nitride) would not be more than 600 lbs.
No. 118	January 29, 1946, Mr. Bass (Brush) to F. M. Belmore states that cost of beryllium nitride ( $\text{Be}_3\text{N}_2$ ) would not be less than \$50.00/lb.
No. 119	March 27, 1946, Mr. Bass (Brush) to Capt. Roboff. Quotes price of \$60/lb. plus \$500 additional charges for 5 lbs. of beryllium nitride.
No. 120	April 9, 1946, Mr. Humiston (Brush) to Capt. Roboff. Quotes a price of \$100 to \$150/lb. for various Process Q shapes rough finished.
No. 121	April 16, 1946, Mr. Humiston (Brush) to F. M. Belmore states that rental for building at 3771 Chester Avenue is \$200/month.
No. 122	May 1, 1946, Maj. Barnett, Corps of Engineers, to Capt. Roboff. Requests negotiations be started with Brush for a research and development contract to produce high-purity Be metal.
No. 123	June 14, 1946, Mr. Schwenzfeier, (Brush) to Capt. Rutman, Madison Square Area, gives a summary of analytical work for analysis of Be metal.
No. 124	August 6, 1946, Mr. Humiston (Brush) to Col. Beeler. Gives a cost breakdown for remelting Be metal. Cost is \$15.25/lb. for remelting.
No. 125	March 8, 1946, Mr. Sawyer (Brush) to Major Kelley. Gives an estimate as to maximum possible rate of production of premium grade metal and Product 88 as 1200/lbs/month and 2000 lbs/month respectively. Quotes a price of \$55/lb. for premium grade metal in large quantities.

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Brush Beryllium Company - NCN (Continued)

<u>Reference</u>	<u>Description</u>
No. 126	August 22, 1946, Mr. Bass (Brush) to R. E. Morie. Gives price list for all Brush chemicals. Of general interest are: BeO "GC" grade, 5-9 lbs. \$5.50/lb. Be metal lump tech. grade \$47.00/lb. Be metal lump premium grade \$62.00/lb. Be metal powder technical grade \$60.00/lb. Be metal powder premium grade \$80.00/lb. Beryllium Fluoride 25-49 lbs. \$8.00/lb.
No. 127	September 9, 1946, Mr. Bass (Brush) to F. M. Belmore. Gives production rates of beryllium oxide.
No. 128	September 23, 1946, Mr. Bass (Brush) to R. E. Morie. States that Brush is proceeding with production of 223 Process Q blocks for Dr. Zimm.
No. 129	August 27, 1946, Mr. Sawyer (Brush) to Col. Fields, War Dept., Washington, D. C. Concerns possibility of establishing Be industry in England, and raises the question of Brush's position along this line.
No. 130	October 18, 1946, Col. Beeler to Commanding General, Manhattan Project, Washington, D.C., Attention Col. Fields. Gives the status of patent agreements for all four Brush contracts.
No. 131	October 22, 1946, Lt. Col. Oberbeck to Lt. Col. Jannarone. Gives recommendations for a health program, including tolerances for alpha, beta, and gamma radiation, Radon gas, etc.
No. 132	October 31, 1946, Mr. Lavender, Patent Advisor, to Col. Fields, concerns feasibility of allowing Brush to disclose Process Q to British.
No. 133	December 17, 1946, Mr. Humiston (Brush) to Reconstruction Finance Corp., Office of Metals Reserve, Washington, D.C. suggests that a quantity of beryl ore now allotted to Civilian Deficiency be placed in storage at Brush's plant.

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Massachusetts Institute of Technology - NCN

Correspondence:

A summary of important correspondence follows:

<u>Reference</u>	<u>Description</u>
No. 134	May 31, 1944, Mr. McL. Sage (MIT) to Capt. Sturges. Concerns a special patent clause to be used for part-time workers.
No. 135	July 26, 1945, Lt. Rutman to Dr. Harrison, MIT. States that a new purchase order has been issued to enable the spectrographic laboratory to continue analyses of Metal Hydrides Company samples.
No. 136	August 18, 1945, John Chipman, Director MIT Metallurgical Project, to Major Greagor. Suggests that MIT attempt to extrude thorium in a manner similar to that used for flake Be.
No. 137	September 11, 1945, Mr. A. M. Gaudin, MIT to Lt. Duffey. Concerns reputed discovery of a high grade pitchblende ore at the N'Kana Mine in Northern Rhodesia.
No. 138	March 7, 1946, Major Barnett to Dr. A. R. Kaufmann states that an accounting system has been set up.
No. 139	April 15, 1946, Dr. Kaufmann (MIT) to Capt. Roboff. Mentions the planned move of MIT's activities to a new building.
No. 140	June 5, 1946, N. McL. Sage (MIT) to Col. Kelley. Gives a clause taken from the Signal Corps contract with MIT covering classified aspects and ability of the staff to publish.
No. 141	July 2, 1946, memo from N. McL. Sage (MIT) to Col. Beeler. Advances the argument that Government contracts should make a contribution in excess of costs to provide a margin of revenue for creation of facilities, support of research in unsponsored fields, etc.
No. 142	July 9, 1946, Col. Beeler to District Engineer, Oak Ridge, states that MIT requests a 7% fixed fee in future contracts, and since this deviates from all previous policy, requests information as to whether or not such negotiations can be authorized.

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Massachusetts Institute of Technology - NCR (Continued)

<u>Reference</u>	<u>Description</u>
No. 143	July 18, 1946, Col. Beeler to Mr. N. McL. Sage (MIT). Postpones decision on fixed fee question until return of the District Engineer from Bikini Tests.
No. 144	August 9, 1946, Col. Beeler to Mr. N. McL. Sage. States that activities of Manhattan District will shortly be handled by an Atomic Energy Commission to be appointed by the President, and therefore the Manhattan Engineering District would not like to make such a major change in contractual policy at this time.
No. 145	August 14, 1946, Mr. Burwell, Jr. to Col. Beeler. Requests permission for certain steel samples, used in a lubrication investigation at MIT, to be irradiated in the Clinton pile.
No. 146	October 8, 1946, T. S. Chapman to Dr. Kaufmann. States policy of Government concerning release of information to industrial concerns, etc.
No. 147	December 10, 1946, T. S. Chapman to Mr. Cammann. Transfers administrative authority to Madison Square Area Engineer.
No. 148	Dec. 17, 1946, Col. Beeler to N. McL. Sage, states that as of January 1, 1947 control of Madison Square Area's uranium inventory will be relinquished by the Manhattan District to the A.E.C.

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Clifton Products, Inc. - NCW

Correspondence:

A summary of important correspondence follows:

<u>Reference</u>	<u>Description</u>
No. 149	December 29, 1942, Lt. Quensau to Major Halle, Steel Section, Materials Branch, Resources Division. Describes a visit to Brush Beryllium Company and Clifton Products, and gives a good summary of plant processes.
No. 150	August 11, 1943, Mr. Windecker to Major Russell. Describes Be metal which Clifton plans to produce starting in September.
No. 151	August 7, 1943, Mr. Henderson, Chief, Be Section, Minerals Bureau to Mr. Drager, Defense Plant Corp., Washington, D. C. Concerns urgency for completion of Clifton plant for production of Be metal.
No. 152	September 25, 1943, Major Russell to Capt. Bassett. Describes inspection visit of Clifton.
No. 153	October 30, 1945, memo to Files from Lt. Roboff. Concerns procurement of high-fired BeO from Clifton.
No. 154	January 9, 1946, Capt. Davies, Jr. to The Judge Advocate General, U. S. Army. Includes copies of documents pertaining to a claim for relief filed by Clifton against Manhattan District, Army-Navy Munitions Board, etc. for #17,772-13.
No. 155	April 3, 1946, Col. Hulland, Contract Relief Advisory Committee to Maj. Gen. Groves. Gives a copy of the decision of the Appeal Board of the Office of Contract Settlement in which the Board approves Clifton's claim.
No. 156	May 2, 1946, Mr. Sturges' memo to Files. Preliminary Negotiation of Reimbursement for Clifton Products.
No. 157	June 6, 1946, Major Sturges to Mr. Windecker (Clifton). Requests Clifton to draw up a proposal for a research and development contract.
No. 158	July 22, 1946, Mr. Windecker (Clifton) to F. M. Belmore. Presents proposal for a research and development program. This program will involve a radically new electrolysis process for producing metallic Be.

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Clifton Products, Inc. - MCM (Continued)

<u>Reference</u>	<u>Description</u>
No. 159	March 7, 1945, Mr. Windecker (Clifton) to Mr. Zeitlin. Gives a summary of Clifton's experience in the production of flake metal.
No. 160	August 22, 1946, Mr. Windecker (Clifton) to R. W. Morie. Gives Clifton's position relative to production of Be metal. Clifton could start to produce flake metal by their old process in four months at 150 lbs/month but would require about \$15,000 to rehabilitate their plant.



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**INDEX**

(Abbreviation App. - Appendix)

- Accountability, 13.1  
African Metals Corp., 1.9, 2.1,  
App. F 2.4, 2.8, 2.9, 5.1, 7.2  
African sources, 1.8, 1.9, 2.1  
App. F1  
American sources, 1.9, 4.1  
App. F3  
Ames, Iowa, 10.1, 10.5  
Analytical program (See Quality  
control)  
Archer-Daniels-Midland Co.  
warehouse, 2.2  
Assaying, 2.7, 2.8, 2.9  
Atoms, 1.4  
Authorizations, 1.2
- Badger, E.B. & Sons, 8.4  
Beeler, Col. G.W., 1.16, 1.17,  
Belgian Congo, 1.3, 1.8, 2.1  
Beverly, Mass., 10.1, 10.7  
Billets, 1.13, 10.2., 10.7, 10.8  
Black Oxide, 1.2, 1.8, 2.4, 3.2,  
4.3, 4.4, 5.2, 7.1, 7.3, 7.4,  
8.5, 7.7, 7.8  
App. C2, D3, F  
Eldorado, 1.10, 3.2, 3.3, 7.1,  
7.2, 7.3, 7.4  
Linde, 1.10, 7.2, 7.5, 7.6,  
7.7, 7.8  
Vitre, 1.10, 7.1, 7.4, 7.5  
Bloomfield, W. J., 10.1, 10.9  
Brown Oxide, 1.11, 8.1, App.  
C3, D6, D7, D8  
du Pont, 1.11, 8.1, 8.2, 8.3,  
8.4, 8.5  
Linde, 1.11, 8.1, 8.5, 8.6, 8.7  
Mallinckrodt, 1.11, 8.1, 8.2  
8.3, 8.4  
Brush Beryllium Co., 10.9, 10.10  
Brush Laboratories, 1.11, 10.9,  
10.10  
Bush, V., 1.2  
By-products, 1.6, 1.13, 2.2, 4.1,  
6.1, 6.3, 8.7
- Canadian Government, 3.3  
Canadian Radium and Uranium Corp.,  
3.1, 5.1, 6.2  
Canadian sources, 1.9, 3.1,  
app. F2  
Cannonsburg, Pa., 7.4  
Carnotite, 1.3, 1.10, 4.1, 4.2, 4.3,  
4.4, 4.8  
Casting (See Recasting)  
Chicago (See University of)  
Cleveland, Ohio, 9.4  
Clinton Engineer Works, 2.6, 2.7  
Clinton Laboratories, 1.14  
Colorado Plateau region, 1.3, 4.1, 4.3  
Columbia University, 10.7  
Commercial uses of uranium, 1.4  
Conant, J.B., 1.2  
Concentrating of ores, 7.2  
U.S.V., 7.8, 7.9, 7.10, 7.11, 7.12  
V.C.A., 7.13  
Conservation Order M-285, 5.1  
Construction, 1.11  
Contract Data, App. F  
Contract negotiations  
African sources, 2.3  
American sources, 4.3  
Canadian sources, 3.2  
Market procurement, 5.1  
Radioactive materials, 6.1  
Contractors, graph, App. B4  
Contractual arrangements, 7.3, 7.5  
Control laboratories, 12.1  
Costs  
Black oxide, 1.9, 7.3, 7.7, 7.8  
App. D4, F  
Brown oxide, 8.3, 8.4, 8.5, 8.6, 8.8  
App. E.6, D.7, E.8  
Concentrating, 7.10, 7.11, App. D4  
Construction, 1.15  
Green salt, 9.2, 9.3, 9.4, 9.5, 9.6  
App. D.9, D.10  
Hexafluoride, 9.8, App. D.11  
Metal, 10.3, 10.4, 10.5, 10.6, 10.7,  
10.8, 10.9, 10.10, App. E.12 E.13  
Operation, 1.15

Costs, Continued  
Overall 1.15  
Oxyfluoride, 9.9  
Peroxide 8.8, 8.1  
Procurement, 1.15.3, 3.2  
3.3, 4.3, 4.7, 4.15.1, 5.2,  
6.1, App. F1 thru  
Quality Control, 1.1  
Radioactive Lead, 1, 6.3,  
App. F5  
Radium, .1, 6.2, p. F6  
Soda Sal, 7.5  
Tetrachloride, 9.1  
Crenshaw, Lt. Col. F., 1.16,  
1.17

Deepwater Point, W., 8.1, 8.4,  
8.8, 10.  
Development (See Research and  
development)  
Dioxide (see Brownide)  
du Pont & Nemours Co., E.I.  
1.11, 1.3, 8.1, 8.5, 8.7  
8.8, 8.9, 9.1, 9.2.3, 9.7  
9.8, 10.  
Durango, Colorado, 2, 4.5, 4.6  
7.2, 7.9, 7.10, 7.

Eldorado Gold Mines Ltd., 3.1  
Eldorado Mining and Refining, 1.3,  
1.10, 1.22, 2.3, 3.6.3, 7.1,  
7.2, 7.3, 7.4, App. F  
Electro Metallurgical Co., 1.12,  
1.21, 10.1, 10.0.4

File references, App. F  
French, Carl, 3.2  
Frick Chemical Laboratories, 2.9  
Flow diagrams, App. F  
Future considerations, 1.14

General Features of Program,  
Part A  
Glossary, App. A  
Grand Junction, Colorado, 4.5  
4.6, 7.2, 7.9, 7.1, 11  
Great Bear Lake, 1.1  
Green salt, 1.11, 9, App. C 5  
D 9, D 10

du Pont, 1.11, 9.1, 9.2, 9.3, 9.4  
Harshaw, 1.11, 9.1, 9.4, 9.5  
Linde, 1.11, 9.6  
Mallinckrodt, 9.1, 9.2  
Green sludge, 7.9  
Graphs, App. E

Hanford Engineer Works, 1.14, 12.4  
Harshaw Chemical Co., 1.11, 1.21, 5.1  
9.1, 9.4, 9.5, 9.6, 9.7, 9.8, 9.9  
Hexafluoride, 1.1, 9.1, 9.6, App. C6,  
D 11  
du Pont, 9.7  
Harshaw, 9.1, 9.6, 9.7, 9.8  
Hooker Electrochemical Co., 1.13

Introduction, 1.1  
Iowa State College, 1.11, 1.22, 10.1,  
10.2, 10.5, 10.6, 10.7, 11.1, 12.5  
Isotopes, 1.5

Jackson Laboratory, 1.11

K-25, 1.1, 1.15, 8.7, 9.5, 9.7, 9.8,  
App. G  
Kelley, Lt. Col., W.E. 1.16, 1.17,  
Kelly, Joseph A., 6.2, 6.3, App. F 6  
Klaproth, M.H., 1.3

LaBine, Gilbert A., 3.2, 3.3, App. F  
Lake Ontario Ordnance Works, 2.6  
Lead Oxide, 6.1  
Ledoux & Co., 2.8  
Linde Air Products Co., 1.10, 1.11  
1.21, 2.6, 7.2, 7.5, 7.6, 7.7,  
7.10, 8.1, 8.5, 8.6, 8.7, 9.6  
Lindsay Light & Chemical Co., 11.1

Mallinckrodt Chemical Works, 1.11  
1.21, 8.1, 8.2, 8.3, 8.4, 8.6, 9.1,  
9.2, 10.1, 10.2, 10.3, 10.4  
Market procurement, 1.10, 5.1,  
App. F 4  
Marshall, Col. J.C., 1.16  
Massachusetts Institute of Technology,  
1.22, 12.2, 12.3, 12.4  
Material balances, 13.1, 13.2  
Metal, 1.1, 1.11, 1.13, 1.14, 9.2,  
10.1, App. C 7, D 12, D 13

~~TOP SECRET~~

Metal Continued  
 Brush Labs, 1.11, 10.9, 10.10  
 du Pont, 1.11, 10.1, 10.5  
 Electromet, 1.11, 10.1, 10.3, 10.4  
 Iowa State College, 1.11, 10.1,  
 10.2, 10.5, 10.6, 10.7  
 Mallinckrodt, 1.11, 10.1, 10.2,  
 10.3, 10.4  
 Metal Hydrides, 1.11, 10.1, 10.2,  
 10.7, 10.8  
 Westinghouse, 1.11, 10.1, 10.8,  
 10.9  
 Metal Hydrides, 1.11, 1.21, 10.1,  
 10.7, 10.8, 12.3  
 Metals Reserve Corp., 4.1, 4.2,  
 4.3, 4.5, 4.6, 4.7, App. F 3  
 Middlesex warehouse, 2.6, 2.8, 2.9,  
 Monazite, 12.5  
 Monticello, Utah, 4.2, 4.7, 7.12  
  
 National Bureau of Standards, 1.22,  
 2.9, 10.7, 12.2, 12.3, 12.4, 12.5  
 Naturita, Colorado, 4.2, 4.6, 7.13  
 Niagara Falls, N.Y. 10.3  
 Nichols, Col. K.D., 1.16  
  
 Objective, 1.1  
 Occurrence of uranium, 1.3  
 Office of Scientific Research and  
 Development, 1.2, 1.11, 3.1, 1.5  
 8.1, 8.2, 9.1, 9.3, 9.4, 10.1, 9.7  
 10.5, 10.7, 10.9, 10.10, 12.1, 12.3  
 Oclan, Belgium, 2.2  
 Operations, 1.1, 1.7, 1.10, 1.13  
 1.14, App. C, D, F7, F 8  
 African sources, 2.1  
 American sources, 4.1  
 Black oxide, 7.1  
 Brown oxide, 8.1  
 Canadian sources, 3.1  
 Concentrating of ores, 7.8, 7.13  
 Green salt, 9.1  
 Hexafluoride, 9.1  
 Market procurement, 5.1  
 Metal, 10.1  
 Orange oxide, 8.1  
 Oxyfluoride, 9.9  
 Radioactive materials, 6.1  
 Refining of ores, 1.7, 1.10, 7.1  
 Soda Salt, 7.1  
 Tetrachloride, 9.5  
  
 Operations, Plan of, 1.7  
 Orange Oxide, 1.1, 8.1, 8.2,  
 App. C 1, C 3  
 Ores, 1.3, 1.7, 1.8, 1.10, 1.14,  
 1.15 (See Sources of raw material)  
 Occurrence, 1.3  
 Refining, 7.1  
 Organization and personnel, 1.15  
 1.16, 1.17, 1.18, 1.19, 1.20, 1.21,  
 1.22, Charts, App. B  
 Oxide (See Black oxide)  
 Oxyfluoride, 9.9  
  
 P-9, Miscellaneous Material for,  
 App. H  
 Perry Warehouse, 2.6  
 Personnel, 1.15, 1.17  
 Army, 1.17, 1.18, 1.19, 1.20, 1.21  
 Contractor, 1.22  
 Pitchblende, 1.2  
 Pitkin, Lucius, 2.8, 2.9, 12.3  
 Port Hope, Ontario, 3.1, 3.2,  
 7.1, 7.2  
 Pregel, Boris, 3.1, 3.3, 6.2  
 App. F  
 Prices (See Costs)  
 Princeton University, 1.22, 2.9  
 12.2, 12.3, 12.4  
 Processes (See Operations)  
 Process gas, 9.7  
 Procurement, 1.1, 1.8, 1.9  
 African sources, 2.1, App. F  
 American sources, 4.1, App. F 3  
 Canadian sources, 3.1, App. F 2  
 Market procurements, 5.1, App. F 4  
 Radioactive materials, 6.1  
 App. F 5, F 6  
 Production (See Operations)  
 Program, Development of, 1.1,  
 1.2, 1.7  
 Properties of uranium, 1.3, 1.4  
 Chemical, 1.5  
 Physical, 1.4  
 Radioactivity, 1.5  
 Relationship to radium, 1.5  
 Quality Control, 12.1  
 Radioactive lead, 1.9, 6.1, 6.3,  
 App. F 5  
 Radioactive materials, 1.5, 1.9,  
 6.1, App. F 5, F 6  
 Radioactivity, 1.5

~~TOP SECRET~~

Radium, 1.6, 1.9, 2.1	St. Louis Sash and Door Works, 9.2
4.1, 6.6	Site I, Procurement for, App. J
Costs, Demand	Standard Chemical Co., 7.4, 8-50., 1.1, 1.15, 9.5, 9.7, 9.8
Relation, 2.5	Stone and Webster Engineering Corp., 1.7, 1.8, 1.16, 3.2, 3.3, 7.5, 8.2, 9.4, App. F
Radium-2.6	Storage, 2.5
Radium-2.9, 5.2	Tailings, 1.9, 4.2, 4.4, 4.5
App. F	App. F 3
Radium O <sub>2</sub> , 6.3	Treatment of, 7.2, 7.8
Raw mate (See Costs)	Tetrachloride, 9.5
Recasting 0.6	Tetrafluoride (See Green salt)
10.7, 1.1	Thorium, 11.1, 12.5
Recovery	Tonawanda, N.Y., 7.2, 7.6, 8.5
Operat 3.8, 8.9,	Transportation of African ore, 2.5
6.10, 4.1	Treatment (See Refining operations)
Plant, Referenc	Trioxide (See Orange oxide)
Refining, 1.7, 1.10,	Turnings, 1.13, 10.2, 10.6, 10.7
1.11, 7.1, App. C1 C2,	
D 4, F	
Eldorado	
Linde,	
U.S.V.,	
V.C.A.,	
Vitro,	
Requires App. D 2	
Research 1.12, 1.14,	Union Carbide and Carbon Corp., 7.15, 8.7
1.15, 3.1, 9.3	Union Miniere du Haut Katanga, 1.3, 2.1, 2.2, 2.3
9.7, 10.4, 10.5, 10.6	U.S. Vanadium Corp., 1.22, 4.1, 4.7
10.7,	4.2, 4.3, 4.4, 7.2, 7.6, 7.8
Results	7.9, 7.10, 7.11, App. F
Ruhoff, 1.6	University of Chicago, 1.14, 7.5, 8.1, 12.2, 12.3
	Uranite, 1.3
Samplin	Uranium, 1.1, 1.2, 1.3, 1.4, 1.5, 1.6, 1.12, 1.13, 1.14
Scrap rry)	Dioxide (See Brown oxide)
Scope,	Hexafluoride (See Hexafluoride)
Seneca	Metal (See Metal)
Shinkol, 2.1, 2.2	Ores (See Sources of raw material)
2.3, 3.3	Oxide (See Black oxide)
Soda sa	Oxyfluoride (See Oxyfluoride)
Sodium alt)	Tetrachloride (See Tetrafluoride)
Sources 1.3, 1.8	Trioxide (See Orange oxide)
(See U	
Africa	
Americ	
Canadi	
Market	

~~TOP SECRET~~

Uranium Oxide, 1.1, 2.3,  
7.1

Uravan, Colorado, 4.2, 4.4,  
7.2, 7.9, 7.10, 7.11

Vanadium, 1.9, 1.15, 4.1, 4.3,  
4.4, 4.5, 4.6, 7.2, 7.8

Vanadium Corp. of America, 1.22  
4.1, 4.2, 4.3, 4.4, 4.5, 4.6,  
4.7, 7.13, App. F 3

Vanadium pentoxide, 1.15, 4.1,  
7.6, 7.9

Vitro Manufacturing Co., 1.10,  
1.22, 2.3, 4.3, 4.8, 5.1, 5.2  
7.1, 7.4, App. F

War Production Board, 5.1  
Weighing, 2.7

Westinghouse Electric and  
Manufacturing Co., 1.11,  
1.22, 10.1, 10.8, 10.9  
Westinghouse, Chicago, Ill.  
11.1

X-10, 1.1, 1.2, 1.12, 1.14,  
1.15, App. I

Y-12, 1.1, 1.14, 1.16  
Yale University, 1.22, 8.3, 8.9  
Yellow Sludge, 7.9, 7.10  
7.11

~~TOP SECRET~~

