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MANHATTAN DISTRICT HISTORY

BOOK VIII, LOS ALAMOS PROJECT (Y)

VOLUME 3, AUXILIARY ACTIVITIES

CHAPTER 3, ACTIVITIES OF OHIO STATE UNIVERSITY CRYOGENIC LABORATORY

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MANHATTAN DISTRICT HISTORY

BOOK VIII, LOS ALAMOS PROJECT (Y)

VOLUME 3, AUXILIARY ACTIVITIES

Chapter 3, Activities of Ohio State Cryogenic Laboratory

PART I

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FOREWORD

This portion of the history is written to record the activities of The Ohio State University Cryogenic Laboratory on the "Uranium Project".

It relates to the initial OSRD contract for a program of work correlated with the Columbia University program under the direction of Professor H. C. Urey. Although it is written as of 15 July 1946, the activities described were terminated some time before this date. This portion of the history is closely related to the subsequent Manhattan District contract with the Cryogenic Laboratory, which was a part of the Los Alamos program, and is written up as Part II of this Chapter.

15 July 1946

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

Chapter 3, Activities of Ohio State University Cryogenic Laboratory
Part I

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SUMMARY

1. The du Pont Company planned to build a plant for distilling liquid hydrogen in conjunction with their ammonia plant at Belle, W. Va. Basic data were lacking for proper design for efficient and safe operation of this plant and the Ohio State University low temperature laboratory was to be the research laboratory in which the problems that presented themselves in connection with the large scale production and distillation of liquid hydrogen were to be worked out. The heavy water so produced was to be used as moderator material in plutonium piles, and for other purposes in connection with the "Uranium Project".

2. The Ohio State University had the equipment for liquefying hydrogen, but no building in which to set it up; although, such a building was under construction. A contract was signed by the Ohio State University Research Foundation and the War Department on November 15, 1942. Work progressed rapidly, and on February 2nd liquid hydrogen was made. Research was gotten under way immediately.

3. Tests were made of various thermal insulating materials suitable for use at liquid air and liquid hydrogen temperatures. The impact strength of several types of wood and plastic materials at these temperatures was determined. Also, apparatus was designed and built to obtain data necessary to the removal of nitrogen from high pressure hydrogen, and planned a new design of liquefier intended to reduce difficulties resulting from nitrogen impurities.

4. In late March the laboratory was informed that the plan for

fractional distillation of liquid hydrogen had been abandoned because of discovery that the du Pont source of hydrogen contained only half the normal concentration of heavy hydrogen. However, research at liquid hydrogen temperatures and on the liquefaction of hydrogen was continued for another very important division of the "Uranium Project".

5. The working staff on this first project consisted of nine persons: a director, three research associates, one research assistant, one chief mechanic, one machinist, one machinist's helper, and a secretary.

MANHATTAN DISTRICT HISTORY

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VOLUME 3, AUXILIARY ACTIVITIES

Chapter 3, Activities of Ohio State University Cryogenic Laboratory
Part I

SECTION 1 - INTRODUCTION

1-1. The du Pont Company planned to build and operate a plant for distilling liquid hydrogen at Belle, W. Va., adjacent to the large ammonia plant located there and owned and operated by the du Pont Company.

1-2. In connection with the production of synthetic ammonia a mixture of steam and air in suitable proportions is passed over heated coke. The coke removes the oxygen from the air to form carbon dioxide (with some residual traces of carbon monoxide) and hydrogen. This produces a gas mixture which consists principally of hydrogen, nitrogen and carbon dioxide. The carbon dioxide is removed by absorption in water or other suitable absorbent. This then leaves a gaseous mixture whose composition is approximately 70% hydrogen, 30% nitrogen, and small amounts of carbon monoxide and of oxygen. The plan was to take this mixture of hydrogen and nitrogen, which normally goes into the catalytic reactors for producing ammonia, and send it to the special plant for obtaining pure heavy hydrogen. In this plant the nitrogen would be condensed out by refrigeration to yield a gas which would be 98% hydrogen, with perhaps 2% of residual nitrogen. This gas was then to be liquefied and fractionally distilled to remove its content of heavy

hydrogen. The proportion of heavy hydrogen in normal hydrogen (as in normal water) amounts to about 1 part of heavy hydrogen in 6,000 parts of ordinary hydrogen. This du Pont operated plant was intended to remove this small fraction of heavy hydrogen by the process of fractional distillation. The ordinary light hydrogen would then be remixed with the nitrogen originally condensed out of the ammonia gas mixture and was to be returned to the ammonia plant for the production of ammonia. By this overall process the plant was thus to extract the small concentration of deuterium from the ammonia hydrogen and not sensibly affect the production of ammonia. The production of hydrogen at the Belle Ammonia Plant was such that it was planned to use a battery of more than 20 large compressors, each driven by a 2,300 hp. motor, to supply the hydrogen liquefiers. This, therefore, represented a hydrogen liquefying plant of approximately 50,000 hp. size. When it is considered that hydrogen has heretofore been liquefied only on a laboratory scale - with very pure hydrogen (less than 0.02 of 1% impurity in the hydrogen)- and that the largest laboratory liquefier was no larger than about 60 hp., the enormity of the proposed industrial scale of liquefaction is seen.

SECTION 2 - PROBLEMS

2-1. Many difficult problems were anticipated in the successful and safe operation of this plant. At the temperature at which a hydrogen liquefier runs, air, nitrogen, carbon monoxide or other impurity freezes to a solid and plugs the coils carrying the high pressure hydrogen from the compressors. That involves the problem of removing such impurities from the hydrogen before its liquefaction, or of designing a liquefier in which interference from these impurities can be eliminated. Since this liquid boils at only 20°C above the absolute zero (-423°F), and has a very low latent heat of vaporisation and very low specific gravity (density only 1/12 that of water) there are problems in the nature of heat insulation and in the mechanical performance of materials. (Some materials become extremely brittle at this low temperature and almost all known lubricants freeze before this temperature is reached.) The high pressure at which the liquefier would have to operate (somewhere between 1500 lbs. and 2500 lbs. per square inch) and the highly explosive nature of hydrogen and air posed a serious problem in the safety of the process.

2-2. The final objective of the overall program was the preparation of large quantities of pure heavy water to be used as moderator material in plutonium piles, and for other purposes in connection with the "Uranium Project".

SECTION 3 - CONTRACT

3-1. At Ohio State University there was equipment for liquefying hydrogen on a large laboratory scale (60 hp. compressor capacity) and for carrying out research at the temperatures of liquid hydrogen. At the time that the University was approached by Professor Urey of the Columbia University Division of War Research, and member of the OSRD Executive Committee, the hydrogen liquefier equipment had not yet been set up. In fact, the University was awaiting the completion of a laboratory building in which the low temperature laboratory was to be included. Only the framework of the building was completed, no walls had been constructed either externally or internally, floors had not been poured and no piping or other service installed. The University was urged to proceed with the laboratory and its research program with all possible haste. The contract, No. OEMsr-786, was set up through the Ohio State University Research Foundation under date of November 15, 1942, for an initial period of six months, with a budget of $25,000 (subsequently, increased to $30,000).

SECTION 4 - PERFORMANCE

4-1. The laboratory staff immediately went to work to get the hydrogen liquefier in operation and to get the research program underway. During December and January the staff worked in overcoats while the hydrogen liquefying plant was being installed in the War Research Building. There were no windows and no doors in the building during December and during the early weeks in January. Although water lines were installed in early January, it was impossible to turn them on until after January 20th when heat was first supplied to this portion of the building. An effort was made by university authorities and the contractors to complete the space in the building that the hydrogen liquefying plant would occupy in advance of the rest of the building.

4-2. Liquid hydrogen was made for the first time on February 2nd. On that first trial it was possible to produce between 2 and 3 liters per hour only, but the next day, by changing some adjustments in the liquefier it was possible to step up the production to nearly 15 liters per hour. A few weeks later the production had been built up to 25 liters per hour. This production represents at least a 20% higher yield than any other hydrogen liquefier constructed up to that time, and represents close to the theoretical maximum with a 60 hp. compressor.

4-3. Following the successful liquefaction of hydrogen on February 2nd and 3rd, investigations at liquid hydrogen temperatures began. Among the first of these were measurements of the mechanical strengths of metals and other constructional materials at liquid air

and liquid hydrogen temperatures. Apparatus was planned and constructed for measuring the efficiency with which nitrogen could be condensed out of a mixture of high pressure gaseous nitrogen and hydrogen at temperatures down to 60° above the Absolute Zero (40°C above the boiling point of hydrogen) and for measuring the solubility of solid nitrogen in liquid hydrogen. These investigations were necessary to the problem of removing nitrogen impurity from the high pressure hydrogen.

4-4. Also, tests were begun of various thermal insulating materials suitable for use at liquid air and liquid hydrogen temperatures. A rough plan was outlined for removing nitrogen and carbon monoxide impurity from the hydrogen by means of a double liquefying cycle. This was to consist of one compressor and liquefier operating at high pressure on very pure hydrogen, whose evaporation would be used to condense the impure hydrogen in a second low pressure system, from which impurity could be more easily removed than from the high pressure system.

4-5. A new type of thermal conductivity cell was developed, which was patented, in connection with the program of measuring heat conduction of thermal insulators at liquid hydrogen and liquid air temperatures.

4-6. The impact strength of several types of wood and plastic materials at liquid hydrogen and liquid air temperatures was determined.

SECTION 5 - TERMINATION

5-1. In late March the laboratory was informed that the plan for producing deuterium by fractional distillation of hydrogen had been abandoned due to the discovery that the production of deuterium in the mixture of nitrogen and hydrogen produced at the du Pont Ammonia Plant was only 1 part in 12,000 in place of the normal 1 part in 6,000. Research turned to other methods which are mentioned in the Smyth Report (chemical exchange method and fractional condensation of steam). The original six months OSRD contract was continued until its expiration on May 15, but emphasis was placed on research at liquid hydrogen temperature, and on the liquefaction of hydrogen in connection with another very important division of the 'Uranium Project'.

SECTION 6 - ORGANIZATION

6-1. Research Staff. - The project was directed by Professor H. L. Johnston, who took leave of absence from University teaching during the period January 1, 1943, to April 1, 1943, in order to expedite the program more fully. Half time assistance was given by Dr. George E. MacWood, who was responsible for the charpy impact test and for the early measurements of thermal conductivity. The rest of the research staff consisted of Mr. Wesley M. Jones, Mr. P. G. Wilkinson and Mr. C. B. Hood. Mr. Jones was responsible for the calibration of thermocouples and collaborated with Professor Johnston and Mr. Hood in the design of a precision apparatus for measuring thermal conductivities. Mr. Wilkinson developed improved methods of analyses for impurities in hydrogen - sensitive to 1 part in 10,000,000. Mr. Hood assisted Dr. MacWood in the early thermal conductivity measurements and assisted Mr. Jones in the calibration of thermocouples.

6-2. Shop Staff. - The shop program was under the direction of Mr. Gustave Nuessle, as chief mechanic. Mr. Jacob Myers (machinist), Mr. Hugh E. Hockway (part time machinists' helper) and Mr. Ralph M. Robbins (general mechanic) assisted with the shopwork, which included the construction and installation of units in the hydrogen liquefying plant as well as the construction of research apparatus.

PART II

PART II

FOREWORD

This portion of the history is written to record the activities of the Ohio State University Cryogenic Laboratory on the Los Alamos program of the Manhattan District project.

As a record of these activities, it is a sequel to that written to record the activities of the laboratory on its original OSRD contract correlated with the Columbia Project under Professor H. C. Urey, described in Part I of this Chapter. The record extends through the completion of the Manhattan District contract in April 1946.

15 July 1946

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Chapter 3, Activities of Ohio State University Cryogenic Laboratory
Part II

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SUMMARY

1. Introduction. - In 1943, when the Los Alamos plant was being developed, the Ohio State University was contacted in connection with problems affecting the design and operation of a liquid deuterium plant at Los Alamos and in connection with the storage and handling of liquid deuterium. A contract for this work was signed by the Manhattan Engineer District and the Ohio State University Research Foundation in May, 1943. This contract was continued until April, 1946. The Manhattan Engineer District helped procure material and equipment for the laboratory by assigning prioritiee, etc. and by direct expediting from their office in Washington.

2. Research Problems. - Several of the most significant problems which presented themselves in connection with this program are listed below.

(a) Development of a Liquefier.
(b) Diffusion of Air Through Gas Holder Sealing Fluids.
(c) Exchange of Deuterium Atoms with Sources of Ordinary Hydrogen.
(d) Rate of Ortho-Para Deuterium Conversion.
(e) Thermal Insulating Materials for Preserving Liquid Deuterium.
(f) Joule-Thomson Cooling in the Expansion of Compressed Gaseous Deuterium.
(g) Latent Heat of Vaporization of Liquid Deuterium.
(h) Gas Density of Deuterium at Various Pressures and Temperatures.
(i) Production of Deuterium Gas from Heavy Water as a Source.
(j) Factors Met in the Storage and Handling of Deuterium Gas.
(k) Thermal Properties of Gaseous Deuterium Under Pressure.
(l) Two Cycle Liquefying System.

3. Security. - The natural irritation caused by the necessity of withholding the nature of the work from responsible university officials contributed to the difficulties presented by security. Some examples of annoying secrecy incidents are related. However, it was possible to keep

the secret so well that even after the official announcement that Ohio State University had engaged in research on the Atomic Bomb few persons on the campus identified the work with the low temperature laboratory.

4. Physical Security. - The hazardous nature of the research required that all personnel connected with experiments be covered by extra hazard insurance. However, there were no accidents that resulted in either personal injury or significant property damage.

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VOLUME 3, AUXILIARY ACTIVITIES

Chapter 3, Activities of Ohio State University Cryogenic Laboratory
Part II

SECTION 1 - INTRODUCTION

1-1. Laboratory. - The Ohio State University Cryogenic Laboratory was started in 1942, to foster a research program long planned at the University. Installation of the hydrogen liquefying equipment for the Laboratory was completed early in 1943. Liquid hydrogen was made on a practical scale on February 2, 1943. The Laboratory was devoted exclusively to the 'Uranium Project' from November, 1942, until July, 1945. Between July, 1945, and the expiration of the Manhattan Project contract in April, 1946, the Laboratory worked both on the Manhattan Project and on a new jet propulsion project for the Army Air Corps. During the first six months of its operation (November, 1942, to May, 1943) the work of the Laboratory was directed to problems associated with the large scale liquefaction of hydrogen. (See Part I of this Chapter.)

1-2. Background of Contract. - In connection with the development of the 'Los Alamos' project, directed by Dr. J. R. Oppenheimer, Dr. McMillan and Dr. Kennedy visited Ohio State University in February, 1943, to discuss the feasibility of preparing and handling large quantities of liquid deuterium (heavy hydrogen). The liquid deuterium was needed in connection with the development of the 'Super Bomb', which was to be a long range, super secret, development within the Los Alamos program. Two alternatives were discussed: (1) the production of liquid deuterium in quantity in our Laboratory in Columbus and shipment to Los Alamos on a

specially chartered fast train, and (2) the inclusion of a liquid deuterium plant in the program at Los Alamos. The latter proposal seemed to be the preferable one and was adopted. Subsequently, a low temperature group was organized at Los Alamos and a plant set up there for liquefying deuterium. However, this plant was set up on a pilot scale and the cooperation of the Ohio State University low temperature laboratory was asked in connection with problems affecting its design and operation, and in connection with storing and handling liquid deuterium.

1-3. Contract. - A seven and one-half months contract (W-7405 eng-93) starting May 15, 1943, was signed by the Manhattan Engineer District and the Ohio State University Research Foundation. This contract - which followed Contract No. OEMsr 786 - was extended for two additional twelve month periods. A further four months extension, to April 30, 1946, was granted to permit completion of computations and the preparation of a final report. (This report, "Final Report on Contract W-7405 eng-93 - 'Thermal Studies'", No. AM-2398, dated 1 July 1946, is in the AEC files.) These contracts were awarded because of the special facilities provided at the Ohio State University for carrying on research at the temperatures of liquid hydrogen, and because of the experience of personnel associated with the Ohio State University Cryogenic Laboratory. In fact, the Ohio State University Cryogenic Laboratory was one of the best equipped in the country for work with liquid hydrogen. The gross amount of these contracts (May 14, 1943 to April 30, 1946) totalled $198,000.

1-4. Procurement of Equipment and Supplies. - Much of the equipment required in the development and operation of the low temperature laboratory was of a type which was particularly difficult to obtain during

the war years. This included high pressure compressors, vacuum pumps, electric motors, etc. A great deal of effort was often required to obtain delivery within the time required for efficient performance under the contract. Major A. C. Johnson and the other officers associated with him in the Washington office of the Manhattan District, U. S. Corps of Engineers, were extremely cooperative and helpful in assisting the laboratory in different purchases both by way of making triple A priority assignments when necessary, and by direct expediting from their office. The contracting officer, Colonel S. L. Stewart, was also extremely cooperative and helpful in matters of procurement as well as in other matters in which it was necessary to obtain his help. In many instances - as for example, of the purchase of the air compressors for making liquid air - necessary laboratory equipment of major character was purchased with funds of the Ohio State University but the cooperation of the Manhattan District was given in assisting priority and otherwise expediting these purchases by the University.

1-5. Purpose. - The purpose of the research program under the Manhattan Contract W-7405 eng-93 was to serve as a research laboratory in connection with the development of the super bomb.

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It was necessary to obtain data that would aid in handling deuterium efficiently and with a minimum of contamination because of its relative cost and rarity.

SECTION 2 - RESEARCH PROGRAM

2-1. General. - A wide variety of problems presented themselves in connection with the production, handling, storage and transportation of liquid deuterium. These arose due to the relative scarcity of deuterium as compared with ordinary hydrogen and the fact that it had never before been liquefied except as a curiosity, and that it had never been worked with in quantities of the magnitude that would be required in connection with the super bomb. Several of the most significant of these problems are presented below.

2-2. Development of a Liquefier. - Up to the time of the contract probably no liquefier in the world had liquefied more than 50 to 75 liters of liquid hydrogen in a single operation. The plan was to develop a liquefier that could liquefy in excess of 1000 liters of liquid deuterium in a continuous operation and within a reasonable period of time. The principal drawback to liquefaction in large quantity lies in the presence of impurity in the hydrogen - notably air and products formed in the compressor. These impurities freeze out as solids at temperatures above the liquefaction point of hydrogen (or of deuterium) and cause erratic operation and eventually plugging of the liquefier. The laboratory, therefore, set as its goal either the removal of these impurities or modifications of liquefier design such that these could be tolerated without seriously interrupting performance of the liquefier.

a. One of the earliest investigations had to do with analytical means of detecting small quantities of impurities in hydrogen (or in deuterium) and with determining the character of impurities found

in the normal operation of a hydrogen liquefier and their sources. It was found that the purest source of hydrogen (or of deuterium) was electrolysis of an alkaline solution of water (or of heavy water). By passing the hydrogen generated in a good commercial electrolyzer through a reduced nickel catalyst at a suitable temperature and rate of flow a representative hydrogen was obtained which analyzed two thousandths or three thousandths percent oxygen and about one thousandth percent nitrogen. However, it was found that the nitrogen contamination usually rose to a value between .01 percent and .02 percent before storage was accomplished in high pressure cylinders. The nitrogen contamination was even greater as the hydrogen was cooled slowly so that it remained for a longer period of time in the gas holder from which it was pumped into storage cylinders. There was little or no increase in the oxygen contamination. The source of this added nitrogen contamination was traced to diffusion of atmospheric nitrogen through the oil usually used as gas holder sealing fluid. Contamination by air which might enter at the packing glands of the compressor was readily eliminated by keeping the packing glands under high pressure from an auxiliary oil pump.

b. During the liquefaction of hydrogen, nitrogen contamination continued to enter by diffusion through the gas holder sealing fluids. Added contamination of another sort occurred at the compressor. This was the result of cracking of the oil which lubricated the compressor cycles and by interaction of the oil and its cracking products with both the nitrogen and the hydrogen. These reactions occurred under the conditions of relatively high temperatures and high pressures in the compressor cycles. Methane and other hydrocarbon products were identified. Among

the contaminants formed in the compressor ammonia and pyrridine-like substances were also identified.

c. On the basis of these observations two methods of dealing with nitrogen contamination presented themselves: (1) search for a gas holder sealing fluid more impervious than oil to the diffusion of atmospheric nitrogen, and (2) improvement in liquefier design that would either freeze out nitrogen in a suitable trap or that would permit its passage through the liquefier without interruption of operation. It was found that most of the contamination produced in the compressor could be removed by a trap immersed in a bath of liquid air. It was also planned to make a more careful study of the reaction in the compressor cycle - particularly as a function of the kinetics of lubricating oil used in the compressor and of operating pressures and temperatures. However, the war ended before it was possible to carry out this phase of the program.

d. In order to carry out analyses for the principal improvement listed above - namely nitrogen and oxygen - new analytical procedures were developed for minute traces of these substances as contamination in hydrogen gas. The analytical sensitivity for these two contaminents approximated one part of these contaminents in ten million parts of hydrogen. This sensitivity is 100 to 1000 fold greater than that obtained by methods previously recorded.

e. It was found that the liquefier with which the laboratory started - which was the equal of any then in existence - would not accommodate much more than about .02 of 1% nitrogen contamination without plugging after three or four hours of operation. By making certain improvements in the design it was possible to attain the goal of liquefying

more than 1000 liters of liquid hydrogen in a continuous operation even with hydrogen that contained much more than .02 percent of nitrogen as impurity. In the final test run on this liquefier development success was attained in liquefying 1000 liters of liquid hydrogen in 41 hours elapsed time. This corresponds to almost full capacity of the liquefier (25 liters per hour) at unhindered operation through the full period of the run. It was even possible to continue liquefaction successfully while nitrogen impurity was added at a relatively high rate to the intake of the compressor.

2-3. Diffusion of Air Through Gas Holder Sealing Fluids. - Efforts were made to find gas holder sealing fluids that would be superior to oil. In this investigation several liquids were examined with respect to diffusion rates of both oxygen and nitrogen gas through the liquefiers and with respect to solubilities of these gases. Ninety percent glycerine proved to be much superior to oil since the diffusion rate of nitrogen through glycerine was only about 1% of that through oil. However, glycerine has the disadvantage of being hydroscopic in character and so absorbs moisture to reduce its concentration. In the more dilute glycerine the diffusion rate of nitrogen increased rapidly. There are also the disadvantages that water vapor is thus introduced into the dry hydrogen and that undesired cracking products are formed in the compressor cycles. Tricresyl phosphate was found to be almost as good as glycerine in reducing the rate of nitrogen diffusion without possessing the disadvantages mentioned for glycerine. Although tricresyl phosphate is a relatively expensive liquid and so would be ordinarily inadvisable for gas holder uses, its use represents an economy in handling the relatively

costly deuterium, since the deuterium in a single super bomb might represent a market value of more than One Million Dollars.

a. It was found to be a universal rule that nitrogen diffuses more readily than oxygen through the several liquids that were tested as sealing fluids. The only contamination that should be present in troublesome quantities in a hydrogen liquefier is, therefore, nitrogen - provided the piping and compressor packing are tight.

2-4. Exchange of Deuterium atoms with Sources of Ordinary Hydrogen. It is important, for success of the super bomb, that the deuterium (heavy hydrogen) be free of appreciable amounts of ordinary hydrogen. If ordinary hydrogen enters the deuterium as a contaminant it is extremely difficult to remove since this represents an isotopic separation. The sources of possible hydrogen contamination are represented by the gas holder sealing fluids and by contaminants which may enter through the compressor cycles. The rate was measured at which the ordinary hydrogen atoms in tricresyl phosphate may exchange with the heavy hydrogen atoms with the deuterium gas. Results indicated that this exchange rate is slow and that deuterium may be kept in contact with tricresyl phosphate for several days with no more than a few tenths of 1% contamination. It was found that this exchange rate was much slower than that between the ordinary hydrogen of water and the atoms of deuterium gas. The amount of ordinary hydrogen contamination that might enter in the compressor cycles is relatively small. This contamination may enter either by exchange with the oil on the cylinder walls or with its decomposition products or by association with water which may reach the cylinder through leaky gaskets. The intention was to make a study of the amount of hydrogen contamination that might enter through either one of these sources in the course of

sustained compressor operation. However, termination of the war interrupted this program.

2-5. Rate of Ortho-Para Deuterium Conversion. - Deuterium, like hydrogen, exists in two modifications known as ortho and para forms. In both liquid hydrogen and liquid deuterium one of these forms converts into the other upon standing. This conversion is accompanied by the evolution of a considerable amount of heat which, therefore, causes evaporation of the liquid. This results in evaporation loss comparable to that lost by heat leak through ineffective insulating materials. It was found that with pure liquid deuterium the normal rate of this change is so slow as to be insignificant in the absence of solid catalysts. The influence of various metals and metal oxides which might be present in containers used for storage of liquid deuterium or in components of the super bomb was tested. In this manner information was obtained regarding the preservation of liquid deuterium.

a. The changes between ortho and para forms of hydrogen or of deuterium may also occur in the gas phase when the gas is passed over metals which are catalytic. Measurement was made of the rate at which this change occurs in ordinary hydrogen that is passed at high pressure through charcoal cooled to the temperature of liquid air. This measurement was made because the process of passing the high pressure gas through charcoal at liquid air temperatures is one of the methods of removing nitrogen contamination from the gas. It was planned to make a similar investigation with deuterium gas but these plans were interrupted by the termination of the war.

2-6. Thermal Insulating Materials for Preserving Liquid Deuterium.

Liquid deuterium used in the super bomb will generally have to be stored for a period of several hours before usage, may have to be shipped over seas and may even be stored in the bomb itself for some hours while the bomb is being carried to its destination. Reduction of evaporation loss of highly volatile liquid deuterium was, therefore, important in connection with the super bomb program. For that reason considerable time was given in the research program to the subject of heat leak through thermal insulating materials. A new and superior type of cell was developed for comparing the insulating value of different materials at temperatures of liquid air and liquid hydrogen. This represented a continuation of a program started under the original OSRD contract (OEMsr - 786). With this cell a considerable amount of data was obtained on the performance of several insulating materials both in vacuum and with various pressures of air added to the insulator. Excellent results were obtained which make it possible to predict not only the relative merits of these insulating materials at these extreme temperatures but also to predict the relative heat loss with various amounts of air in the insulators.

2-7. Joule-Thomson Cooling in the Expansion of Compressed Gaseous Deuterium. - The principle ordinarily used in the liquefaction of hydrogen depends upon the effect (first observed by Joule and Thomson) that gases change temperature when allowed to expand freely from a region of high to a region of low pressure. The sign of this temperature change and its magnitude depend upon both the pressure and the temperature from which expansion takes place. To design properly a liquefier for either hydrogen or deuterium it is necessary to know the exact values of these temperature changes, as a function of both the temperature and pressure.

a. Elaborate apparatus is required to obtain reliable results in this type of investigation. A relatively simple apparatus was designed which proved to give results of high reliability. In order to save the precious deuterium while the apparatus was being perfected Joule-Thomson effects for ordinary hydrogen were first measured. These data were themselves valuable since no similar data exist for hydrogen. After the preliminary work with hydrogen, Joule-Thomson effects for deuterium were measured at the desired range of temperature and pressure. In comparison with the determination of the latent heat of vaporization of liquid deuterium (cf. seq.) these data permit computation of the expected performance of deuterium liquefaction as a function of the pressure and temperature at which it is operated.

2-8. Latent Heat of Vaporization of Liquid Deuterium. - A calorimeter was constructed similar to that improved several years ago by Giauque and Johnston, which made accurate determination of the heat of vaporization of oxygen and of nitric oxide. With this apparatus a highly accurate value was determined for the heat of vaporization of deuterium in conjunction with the Joule-Thomson data (see infra). This made it possible to calculate the expected performance of a deuterium liquefier.

2-9. Gas Density of Deuterium at Various Pressures and Temperatures. The measurements of gas density as a function of temperature and pressure provided a substitute to Joule-Thomson measurements for computing the expected performance of liquefiers. These measurements also provided data needed for the design of heat interchangers in the liquefier.

a. An apparatus for these measurements was designed and constructed. It was first used with ordinary hydrogen in order to prove

its operation and then the wanted data on deuterium were obtained. Results with both ordinary hydrogen and with deuterium were highly satisfactory.

2-10. Production of Deuterium Gas from Heavy Water as a Source. - The number of means of liberating the deuterium from heavy water were investigated. Among these were included the passage of hot vapor over heated metal turnings. It was found that this method is a good one when only small quantities of deuterium gas are wanted, but that the reaction between the hot vapor and the metal becomes extremely slow as soon as a thin coating of oxide covers the metal surface. Experiments were continued with various types of electrolytic cells. Best results were obtained with a cell constructed on the pattern of the commercial Shriver filter type cell. With this type of cell rapid production of deuterium with relative high currents through the cells was obtained. There was no source of contamination and almost no loss of deuterium in this operation.

2-11. Factors Met in the Storage and Handling of Deuterium Gas. - Experience was gained in methods of handling relatively large quantities of deuterium under pressure. With ordinary hydrogen it is economical to tolerate some loss of the gas through valve packings, compressor packings, etc. Because of the relatively greater cost of deuterium it is important to avoid these losses, which are tolerated in the handling of ordinary gases. The experience with both valves and threaded connections indicated that only packless valves of the diaphragm type should be used, and that silver soldered joints should be substituted for tapered threaded connections on the high pressure side of the system. Streamlined copper pipe with sweated joints should be used on the low pressure side of the

system. It was found that equilibrium in the ortho-para conversion is attained within a few hours when ordinary hydrogen is stored in ordinary steel cylinders at a pressure of several hundred pounds per square inch. The war was terminated before it was possible to carry out a similar test with deuterium.

2-12. Thermal Properties of Gaseous Deuterium under Pressure. - Heat capacities, with contents and entropies of high pressure deuterium were computed from the Joule-Thomson measurements and the gas densities. These data contributed to the design of an efficient liquefier.

2-13. Two Cycle Liquefying System. - Some plans were formulated for a liquefying system that would utilize deuterium at relatively low pressure (500 p.s.i.). The advantage of this cycle is two fold: (a) It would reduce loss of deuterium by leaks which might develop at valves on compressor packing or at fittings and (b) it would reduce the likelihood of plugging in operation of the liquefier. The plan was to use a fixed supply of very pure hydrogen in a high pressure liquefier to obtain the cooling effect necessary to liquefy the deuterium. The cold deuterium, compressed to 500 pounds by a second compressor, would form liquid without necessity of carrying it through a Joule-Thomson expansion. The hydrogen which liquefied in the high pressure cycle is re-evaporated in the process of cooling the deuterium and returns to the compressor for re-cycling.

a. Plans for developing this two cycle deuterium liquefier were discontinued upon termination of the war.

SECTION 3 - SECURITY

3-1. General. - The laboratory was especially handicapped in the early weeks of the 'Uranium Project' work by the fact that the personnel were setting up apparatus and continuing their research in a building which was only partially constructed and which had not been turned over to the University by the contractor. They were further handicapped by the fact that they were not permitted to divulge either the character or the objectives of the research program to their superiors in the univereity, nor to the administration officials of the Ohio State University Research Foundation with which the contract was drawn up. This frequently led to delays - particularly when it was necessary to call on the university Service Department for service calls to the building - and sometimes led to irritating and embarrassing situations. Another impediment in the enforcement of security regulations arose from the fact that the guards who were put on duty at the War Research Building to safeguard the work were employed by the University Service Department and were responsible, for their instructions, to that Service Department, although the director of the project, Professor H. L. Johnston, was personally responsible for the enforcement of security regulations.

3-2. Specific Security Problems. - A few illustrations of awkward situations which arose out of the above handicaps are the following:

a. During the early weeks of the original work on an OSRD contract (OEMsr - 786) the Laboratory was not equipped with doors and it was necessary to admit workmen to complete work on the sub-contracts of the building. Instructions of the Security Officer for this area were followed, to admit workmen only at times when their presence would jeopardize neither security nor the progress of the research. On one

occasion a curious workman, who had no duty in the Laboratory space, sauntered into look over the research activities and refused to heed warning signs or admonitions to 'Stay Out'. Although the workman was finally reprimanded for his conduct, question was raised by the University Architect's Office regarding the authority to deny access of any employee of the contractor's.

b. When one of the building guards was reported for careless talk in a public restaurant and his dismissal was recommended by the Area Security Officer, following an investigation he was retained by the University Service Department and was a source of irritation by his presence throughout the duration of the contract.

c. A small fire broke out in the Laboratory in December, 1944, The fire was quickly extinguished by the laboratory staff. However, a mistake was made by one of the men who was newly employed in putting in a call to the city fire department. When the firemen arrived they insisted on coming into the Laboratory although the small fire (no more than $5,00 damage) was already out. The guard admitted a large and curious crowd into the building and up to the door of the Laboratory and also admitted the firemen to all of the Laboratory space in violation of the instructions of the director. On leaving the Laboratory the firemen refused to sign the visitors' register. A university official who had no direct authority over the laboratory supported the firemen and the guard in this. In distorted form and in humorous vein the account of this fire was published in a Columbus paper and eventually attained a paragraph in the Saturday Evening Post in an article dealing with Manhattan District Security.

3-3. Secrecy. - In spite of these handicaps the character of the program on which the laboratory was engaged was successfully kept secret and not even the University officials to whom the personnel were responsible in the administration of the contract and in the use of university space and equipment were aware of the character or of the objectives of the research, or of the existence of an Atomic Bomb program, until the official announcements were made after Hiroshima.

3-4. Physical Security. - The work was hazardous both because of

the high pressures (up to 3500 p.s.i.) and of the explosive character of hydrogen, when contaminated with air. It was especially so because of the fact that in some of the investigations the laboratory was under the necessity of 'pushing' the apparatus beyond the limitations of performance which were customary in laboratory experimentation. All of the investigators were covered by extra hazard insurance. The personnel were constantly on the alert to guard against personal hazards that were not necessary to the program. In a period of almost three years they liquefied and worked with more than 12,000 liters of liquid hydrogen and with the equivalent of more than 2,000 cylinders of high pressure hydrogen. 80,000 liters of liquid air were also prepared in the laboratory at operating pressures of 3500 p.s.i. In the entire period of the investigation no accident was experienced with liquid hydrogen or liquid air or with high pressure gases. No injury more severe than an injured finger was experienced by anyone engaged on the project. Two or three small fires occurred. However, these were of very minor character - such as may occur in any laboratory - and property damage by fire never exceeded the $5.00 damage referred to in the section above. There was no substantial material damage to any apparatus or equipment used in the program. Probably the most expensive single items which were lost through accident were large glass thermos bottles valued at about $50.00 each, which were lost through breakage.

SECTION 4 - ORGANIZATION

4-1. Research Staff. - Professor H. L. Johnston directed the project throughout its duration. The organization of the research staff, with the approximate periods of service, are shown in App. A1. Grilly assisted in setting up apparatus for work at high pressures. Hood carried out thermal conductivity measurements with the new cell. Wirth, Douglas and Plumlee developed techniques for measuring the catalytic conversion of hydrogen and of liquid deuterium. Bezman was responsible for the high pressure PVT measurements on hydrogen. Rubin, Corak and Rifkin later carried these out for deuterium. Wallace and Satterthwaite measured the heat of vaporization of liquid deuterium, although the computation of their data was subsequently carried out by Rubin and Rifkin. Swanson was responsible for the preparation of pure deuterium, pure heavy water, and for carrying out the experimental work on the ortho-para conversion of liquid deuterium. Haag was responsible for the measurements of diffusion rates of O_2 and N_2 through gasholder sealing fluids, and for the solubility determinations. Brooks and Greifer were responsible for the charpy impact tests at liquid air and liquid hydrogen temperatures.

a. Hood, Douglas, Bezman, Wirth and Johnston operated on shifts in developing the hydrogen liquefier to the point that continuous runs leading to 1000 liter production were accomplished.

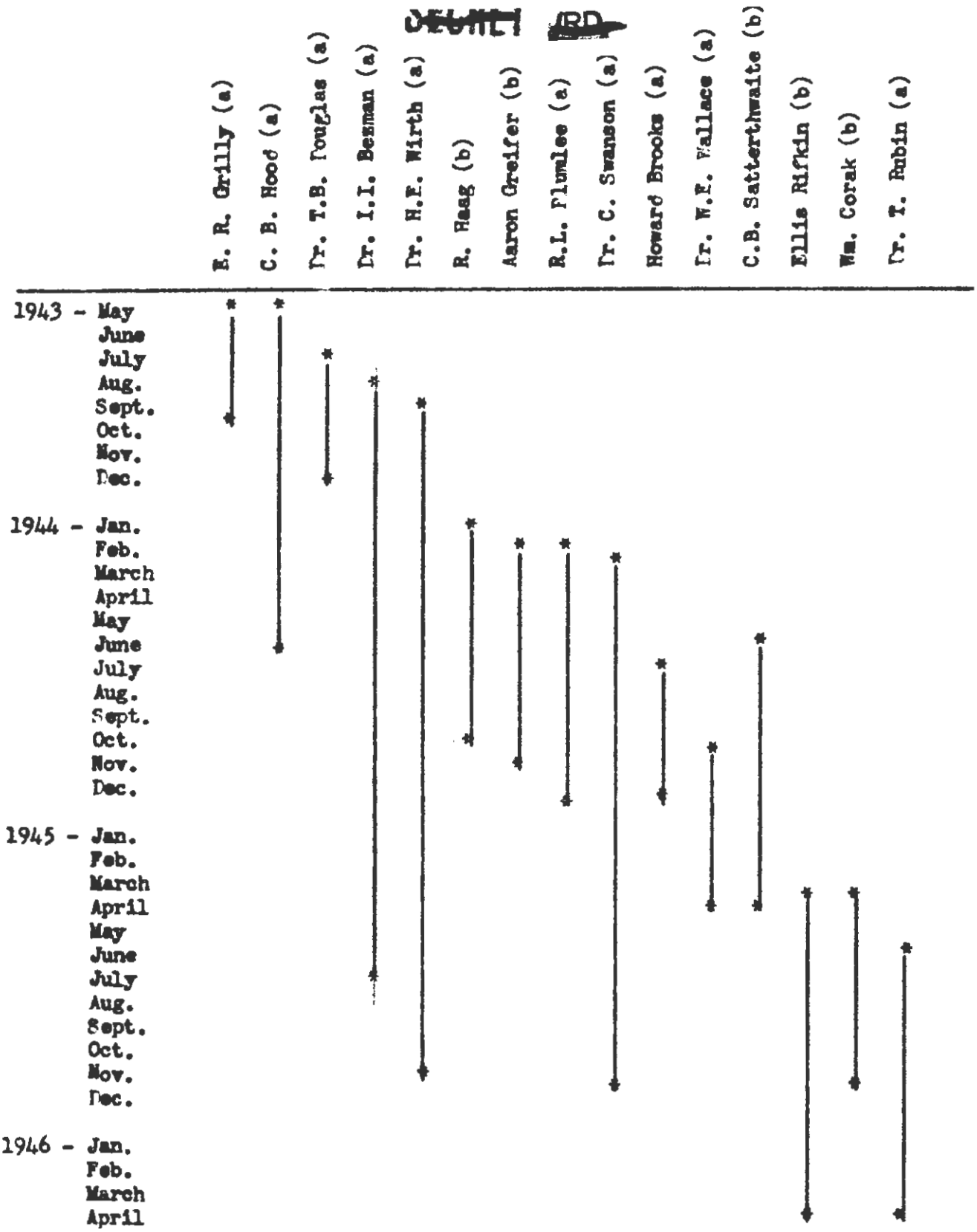
b. Professor Johnston designed the Joule-Thomson apparatus; Hood, Bezman, Wirth, Wallace, Satterthwaite, Haag and Greifer carried out the Joule-Thomson determination on hydrogen and on deuterium.

c. Bezman, Wirth, Greifer, Haag and Swanson were

responsible for the investigations on removal of impurities by charcoal at liquid air temperature, and the ortho-para conversion rate.

4-2. Shop Staff. - The shop staff - responsible for the construction and repair of apparatus - consisted of Mr. L.F. Cox, shop foreman, Mr. Jacob Myers and Gustave Nuessle (machinists). Mr. Ralph N. Robbins, Mr. Gwynne Wright and Mr. Wm. V. Johnston (part time) maintained the heavy equipment and made liquid air and liquid nitrogen for the project. One draftsman was included in the organization throughout the period of the project. This position was held by Miss Joy Phalor, Miss Rosella Horine and Mr. Maurice Lautenslager, in turn. Miss Eileen Davis served as a computer from September 1945, to April 1946.

a. One full time secretary assisted Professor Johnston in the administration of the project, preparation of requisitions, submission of reports, etc. This position was held successively by Miss Phyllis Greenwood, Miss Mona Lou Franken and Miss Margarette Coffee. Miss Olga Sobkiewicz (part time) served as accountant in keeping records of expenditures.



(a) Research Associate
(b) Research Assistant