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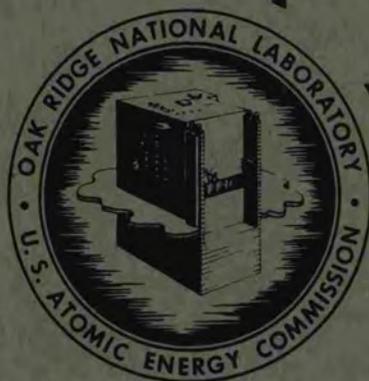
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OPERATIONS DIVISION

THE PRODUCTION OF C¹⁴ BY THE Be₃N₂ PROCESS

R. P. Shields

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CONTENTS

SUMMARY	1
INTRODUCTION	1
HISTORY OF C ¹⁴ PRODUCTION BY THE AMMONIUM NITRATE AND CALCIUM NITRATE PROGRAMS	1
DEVELOPMENT OF BERYLLIUM NITRIDE PROCESS	1
Initial Work by the Chemistry Division	1
Experimental Work by the Radioisotope Development Group	2
PRESENT PROCEDURE AND EQUIPMENT	4
Decanning	4
Preparing the Equipment	4
Dissolving the Nitride	8
Precipitation of Product	9
Summary of Operating Data	9

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THE PRODUCTION OF C¹⁴ BY THE Be₃N₂ PROCESS

R. P. Shields

SUMMARY

The first C¹⁴ produced under the Radioisotope Program of the Atomic Energy Commission was made by the ORNL Physics Division with equipment that circulated a saturated solution of ammonium nitrate through the graphite reactor. The process was discontinued when a program for irradiating calcium nitrate was started in 1946. The calcium nitrate program was terminated by December 1950, at which time the beryllium nitride process was under way.

In the beryllium nitride process, sulfuric acid is used to dissolve the beryllium nitride, thereby releasing the organic compounds and carbon dioxide which contain the C¹⁴. These gases are carried by a stream of nitrogen through a gas train, which includes a CuO furnace to oxidize the organic compounds, and through sodium hydroxide scrubbers to absorb the carbon dioxide. The product is obtained by precipitating the carbonate from the scrubber solution with barium hydroxide.

INTRODUCTION

Production of the C¹⁴ isotope from irradiated beryllium nitride by the present method has proved satisfactory during the past four years of operation (January 1951 through December 1954), and, for the present, no further developmental work is anticipated. This report is presented primarily for the purpose of recording the following: a brief history of C¹⁴ production before the Be₃N₂ process was developed, the essential steps in the development of the beryllium nitride process, and a detailed description of the procedure now used.

HISTORY OF C¹⁴ PRODUCTION BY THE AMMONIUM NITRATE AND CALCIUM NITRATE PROGRAMS

The first C¹⁴ to be processed and sold under the Radioisotope Program was produced by the Physics Department of ORNL.¹ The equipment was designed to produce C¹⁴ continuously by circulating a saturated solution of ammonium nitrate through the Graphite Reactor.² During the operation

period of several months, approximately 300 mc of the product was made and information regarding the half life of C¹⁴ was obtained.³ For several reasons, explained elsewhere,⁴ it was decided to discontinue this operation. The imminence of the calcium nitrate programs was one incentive for ending the experiment.

The ORNL Graphite Reactor was loaded in the spring of 1946 with about 1 ton of dehydrated calcium nitrate which had been compressed and sealed in aluminum cans. Total C¹⁴ production by this program was 8200 mc. Details of the canning of the calcium nitrate are described by Beall,⁵ and the development of the chemical procedure is outlined by Collier and Pressly.⁶ The last of the C¹⁴ produced by this method was processed and sold by December 31, 1950.

DEVELOPMENT OF BERYLLIUM NITRIDE PROCESS

Initial Work by the Chemistry Division

For some time prior to the completion of the calcium nitrate program, many other target materials were being investigated.⁷ The more important materials were several organic compounds, aluminum nitride, calcium nitride, lead nitrate, and beryllium nitride. Of these, beryllium nitride

¹ Physics progress reports from Oct. 15, 1944 through Nov. 30, 1946.

² L. D. Norris and A. H. Snell, *Science* 105, 265 (1947).

³ A. H. Snell and L. D. Norris, ORNL-388 (Oct. 19, 1949) (Confidential).

⁴ L. W. Nordheim, *Physics Division, Report for Month Ending October 31, 1946*, MonP-196 (Secret) and L. W. Nordheim, *Physics Division Monthly Report for January, 1947*, MonP-250 (Feb. 19, 1947) (Secret).

⁵ S. E. Beall, MonT-163 (Sept. 4, 1946) (Secret).

⁶ J. E. Collier and R. S. Pressly, *Development of the Production Scale Process for Separating Carbon-14 from Irradiated Calcium Nitrate*, MonT-431 (Dec. 4, 1947).

⁷ Physics Division progress reports M-CP-1436 (April 30, 1944) (Secret), M-CP-1858 (July 31, 1944), M-CP-1883 (August 15, 1944), and M-CP-2019 (August 15, 1944) (Secret).

appeared to be the most promising. Its advantages are stability, high nitrogen content (50%), and the very low cross section of beryllium for neutrons.

In 1946 the Chemistry Division of ORNL began the development of a method for processing beryllium nitride.⁸ Since this material is very hard and refractory, the initial problem consisted in finding a suitable method for dissolving the material to release the C¹⁴. During the investigation, three methods were found to be suitable.

These three processes were studied and compared in detail by the Chemistry Division at ORNL.⁹ The essential difference was in the method of dissolving the nitride and oxidizing the organic compounds which were released or formed during the dissolving steps. In each method, the gas used to sweep the activity from the dissolver was passed through a chain which consisted of traps for ammonia and water, a copper oxide furnace, and sodium hydroxide or barium hydroxide scrubbers. During this development of the method, scrubbers were changed following each major step to help identify the compounds released or formed during the dissolving. In the following paragraphs a brief description of these processes is given.

The NaOH-KMnO₄ process¹⁰ is begun by boiling a slurry of beryllium nitride and water. While the slurry is being agitated, an excess of 6 N NaOH is slowly added. The active compounds released are swept from the dissolver with oxygen, and then the hot alkaline solution is treated with KMnO₄; continued sweeping removes the activity released. The solution is acidified with 18 N H₂SO₄ to complete the removal of the C¹⁴.

The H₂SO₄-CrO₃ process¹⁰ also starts with a boiling slurry of beryllium nitride and water. Approximately 100 ml of 18 N H₂SO₄ per gram of nitride is slowly added to dissolve the nitride. Nitrogen is used as the sweep gas during the dissolving because considerable hydrogen is formed from the free metal and an explosive mixture might be formed in the copper oxide furnace. Following the dissolving, enough CrO₃ (dissolved in water) is added to make the dissolver solution about 0.2 M

in CrO₃. The chromate oxidizes the nonvolatile organic compounds. At this point oxygen replaces the nitrogen as the sweep gas during the oxidation.

In the NaOH-H₂SO₄-CrO₃ process¹¹ the slurry of beryllium nitride is dissolved by slowly adding 15 N NaOH. Heat generated by the dissolving must be removed by cooling the dissolver. The activity is removed by sweeping the system with carbon dioxide-free air. Next, the temperature is raised and held at 80 to 90°C while the dissolver solution is carefully acidified with 36 N H₂SO₄. When the activity released by this step has been removed, a great excess of CrO₃ is added to oxidize the nonvolatile organic compounds.

After the details of the three processes were considered, it was concluded that the H₂SO₄-CrO₃ process appeared to be the most practical means of recovering the C¹⁴ from the irradiated beryllium nitride. At this stage of development the Radioisotope Development Group of the Operations Division took over the work, in the latter part of 1948.

Experimental Work by the Radioisotope Development Group

Initial work by the Radioisotope Development Group was the building of equipment in which to scale up the amount of material that could be processed by the method suggested. The dissolver (Fig. 1) is a 2-liter flask equipped with a motor-driven agitator, the shaft of which extends through a mercury seal. Reservoirs for 20 N H₂SO₄-chromium acid solution and for distilled water feed into the dissolver through a dip-tube, which extends to the bottom of the flask. The gas train, beginning at the dissolver, consisted also of a reflux condenser, a copper oxide furnace heated to 750°C, drying traps, an ion chamber to operate an electrometer and a Brown recorder, a sodium hydroxide scrubber, and a barium hydroxide trap.

The first step in making a run consisted in melting the aluminum jackets from the beryllium nitride. An electric furnace with a sealed tube, through which nitrogen could be circulated, was used to remove the metal from the slugs. The nitrogen gas that was swept through the furnace was carried through the gas train of the equipment shown in Fig. 1. An assay of the C¹⁴ collected

⁸ Chemistry Division progress reports from MonN-229 (Nov. 15 to Dec. 15, 1946) (Secret) through ORNL-176 (June, July, and August 1948) (Secret).

⁹ J. R. Coe and E. H. Taylor, *Report of the Chemistry Division for the Months June, July, and August 1947*, MonN-370, p 15 (Secret).

¹⁰ B. A. Fries, BC-43 (March 7, 1947) (Secret).

¹¹ J. R. Coe and E. H. Taylor, *Report of the Chemistry Division for the Months March, April, and May 1947*, MonN-311 (June 24, 1947) (Secret).

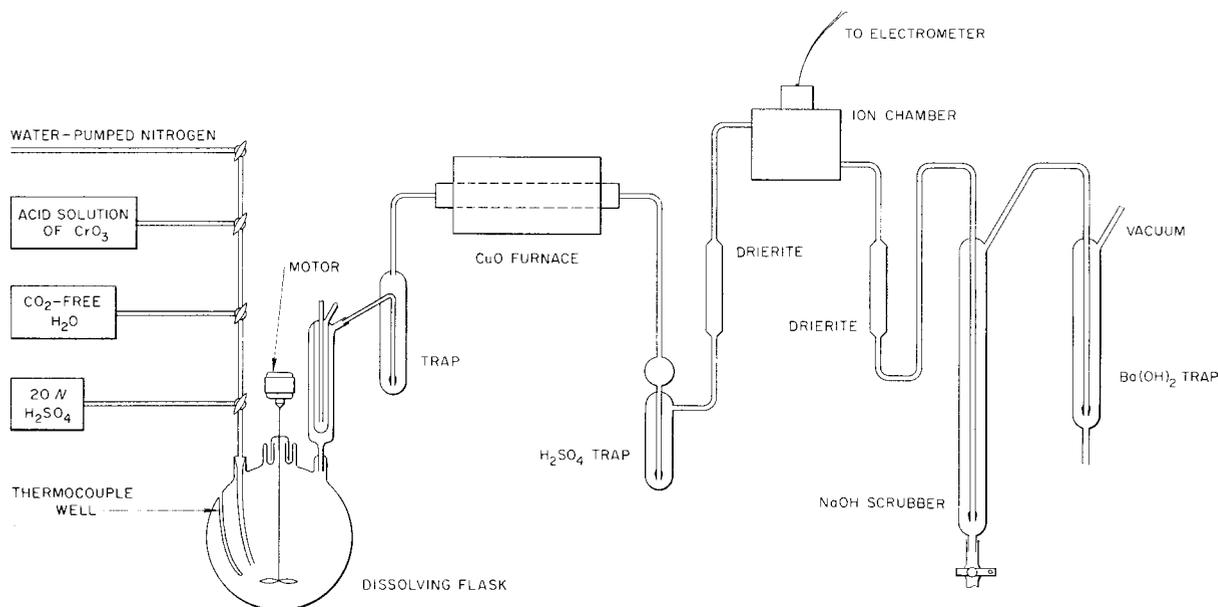


Fig. 1. Flow Diagram of First Production Equipment of the Be₃N₂ Process.

indicated that the losses were insignificant and that there was no need for including the furnace in the gas train of subsequent equipment.

After the metal was removed, the pellets of beryllium nitride were crushed in a stainless steel pipe, about 1½ in. in diameter and 10 in. in height, which was maintained in a vertical position by three legs. The crushed material fed through ¼-in. holes in a removable anvil, which was located in the bottom end of the tube, into a container under the pipe. A circular Stellite hammer, with two cutting wedges across the diameters at right angles, was driven by a rod extending from the top of the pipe. At first, the hammer was operated manually, but the extreme hardness of the pellets made this a slow process. After experimenting with the use of a carpenter's hammer, it was decided to use an electric hammer, which worked very well as long as an edge could be maintained on the crusher hammer. The resulting beryllium nitride dust created a health hazard; therefore the equipment was operated in a closed hood, and the manipulations were done with long-armed rubber gloves fastened into the front of the hood.

The difficulties of the above operation led to experiments on dissolving the slugs without crush-

ing them. It was found that the reaction proceeded smoothly. Since the dissolving rate had to be limited by the rate at which the reflux condenser could remove the heat of reaction, the decreased surface exposed to the reaction was of no consequence and all future runs were made without crushing the slugs.

The dry nitride was transferred to the dissolver, where it was dissolved and treated as explained in the H₂SO₄-CrO₃ process. By increasing the amounts of nitride in each run, the size of the run was scaled up to 200 g, which represented the practical limits of the equipment.

During this development work several methods of collecting the CO₂ were tried. It was hoped that the scrubbers could be filled with Ba(OH)₂ so that the BaCO₃ could be collected on a detachable filter at the bottom of scrubbers. This, however, proved unsatisfactory because of the barium carbonate which stuck to the walls of the scrubber. A solution of sodium hydroxide was used in place of the barium hydroxide and proved satisfactory. The NaOH solution was drained from the scrubber at the end of the run. Precipitation of the activity as barium carbonate during this phase of the work was accomplished in the

same manner as it is now with the production equipment, and details of this process will be given later in this report.

In the initial stages of this development, some effort was made to recover the beryllium as basic beryllium carbonate from the pellets. Freeing the beryllium from the chromium was very troublesome and led to experiments to replace the sulfuric-chromic acid solution as a reagent in the process. It was found that addition of 30% H_2O_2 removed as much activity as did the chromate. Later experiments showed the removal by H_2O_2 to be due largely to the flushing action of the many fine oxygen bubbles although there may have been some removal by oxidation.

One further major change was the elimination of mechanical agitation. Convection currents and the gas formed during the reaction were found to provide ample agitation.

PRESENT PROCEDURE AND EQUIPMENT

The following part of this report discusses in detail the method now used to remove the C^{14} from the irradiated beryllium nitride processed in amounts of 500 to 700 g per run.

Decanning

The first step in the process of C^{14} recovery is the decanning operation. The pellets are contained in aluminum cans with pieces of lead in each end that made the specific gravity of the cans correct for reactor conditions during irradiation. The Be_3N_2 is freed of the lead in an electric furnace which is mounted at an angle to permit the molten metal to pour out. Decanned pellets are pushed out by new slugs being charged into the furnace; the pellets then travel over the molten-metal container on a wire track to the container used for collecting the pellets.

The electric furnace, molten-metal containers, pellet containers, and other decanning equipment are mounted inside a steel hood which contains a lining of lead bricks to shield the operator from radiation from the impurities in the pellets and lead slugs (Fig. 2). The prime offenders in this case are Fe^{59} and Co^{60} .

Preparing the Equipment

The equipment for the preparation of the carbonate is shown by a flow diagram (Fig. 3) and two photographs (Figs. 4a & 4b). The chemical process

starts in the dissolver, which is a three-neck, twelve-liter, round-bottom flask seated in a heating mantle. The flask is equipped with a dip tube for admitting nitrogen and other chemicals, a thermocouple well, and a reflux condenser. To start a run, 2500 to 3000 ml of distilled water is drawn into the dissolver through line A (Fig. 3) and ten 50-g pellets of Be_3N_2 are carefully lowered into the water through the large opening in the top of the flask.

The atmosphere CO_2 is now removed from the system by flushing for about 2 hr with water-pumped nitrogen. The pressure difference for gas flow is obtained by a positive pressure of 1 in. of water on the dissolver and a reduced pressure at the outlet end.

Constant pressure with variable gas flow is maintained by an automatic vacuum control (Figs. 4b and 5). A decrease in the pressure on the gas train will cause the mercury to cover part of the sintered glass disc to reduce gas flow. Increasing pressure lowers the mercury to allow a greater flow of gas.

After the nitrogen flushing, the scrubbers are filled with carbonate-free sodium hydroxide. This is accomplished by attaching a reservoir of sodium hydroxide to the sodium hydroxide scrubbers and increasing the vacuum on the gas train until the sodium hydroxide rises in the scrubber to the 80-ml mark. Increased vacuum is obtained by isolating the dissolver by means of a pinch clamp on the Tygon tubing at point B (Fig. 3) and lowering the mercury reservoir on the automatic control until the desired pressure is obtained. When the first scrubber is filled, the Tygon tubing at the lower end is clamped, the ball joints are uncoupled, and the reservoir is attached to the next scrubber down stream, and so on until all the scrubbers are filled. The vacuum obtained while filling the first scrubber is sufficient to cause the remaining scrubbers to fill.

When the scrubbers are filled, the system is brought to proper operating pressures as follows: The vacuum-control reservoir is raised to a cutoff position, and nitrogen is fed slowly into the dissolver until the water manometer on the dissolver indicates about 2 in. of water pressure. Then the pinch clamp at point B is slowly opened just enough to maintain the 2 in. of pressure while allowing the scrubbers to receive nitrogen until the pressure above the first scrubber just balances the dissolver pressure. At this point, the nitrogen

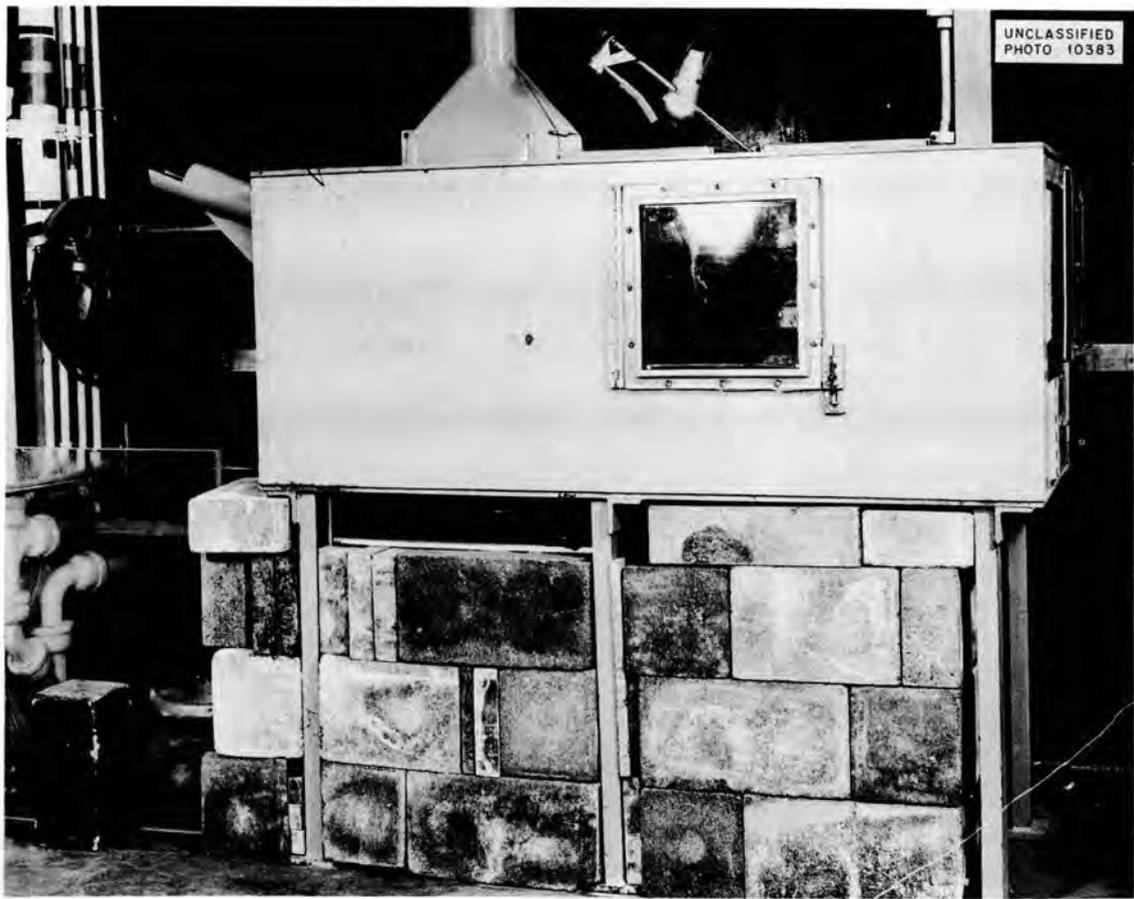


Fig. 2. Hood Containing Decanning Equipment.

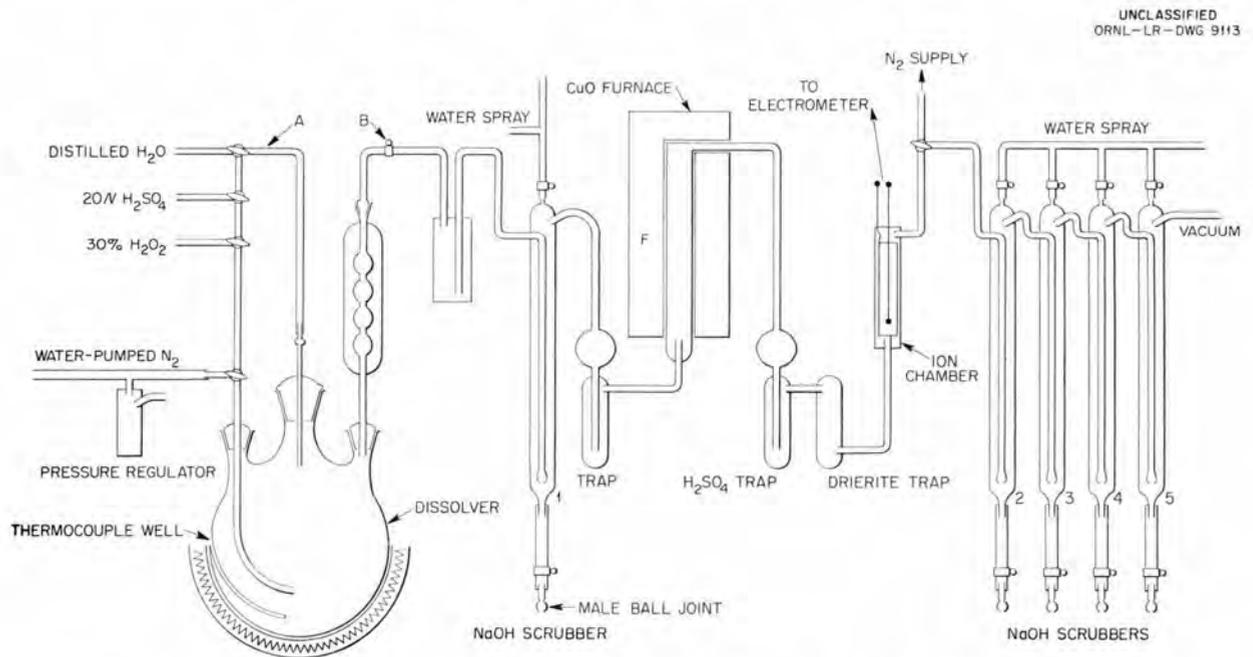


Fig. 3. Flow Diagram of Present Production Equipment of the Be_3N_2 Process.

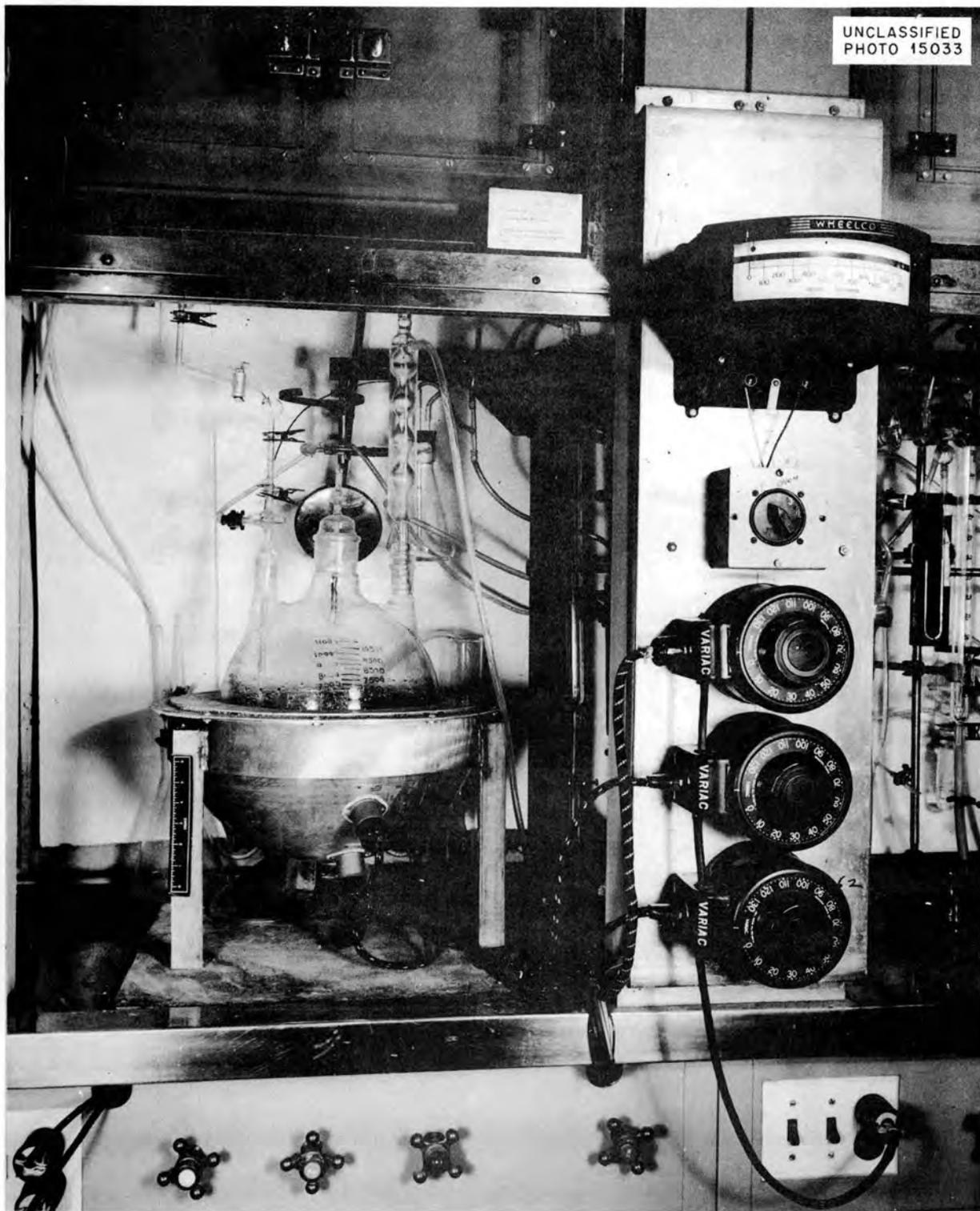


Fig. 4a. Photograph of Present Production Equipment – Dissolver Section.

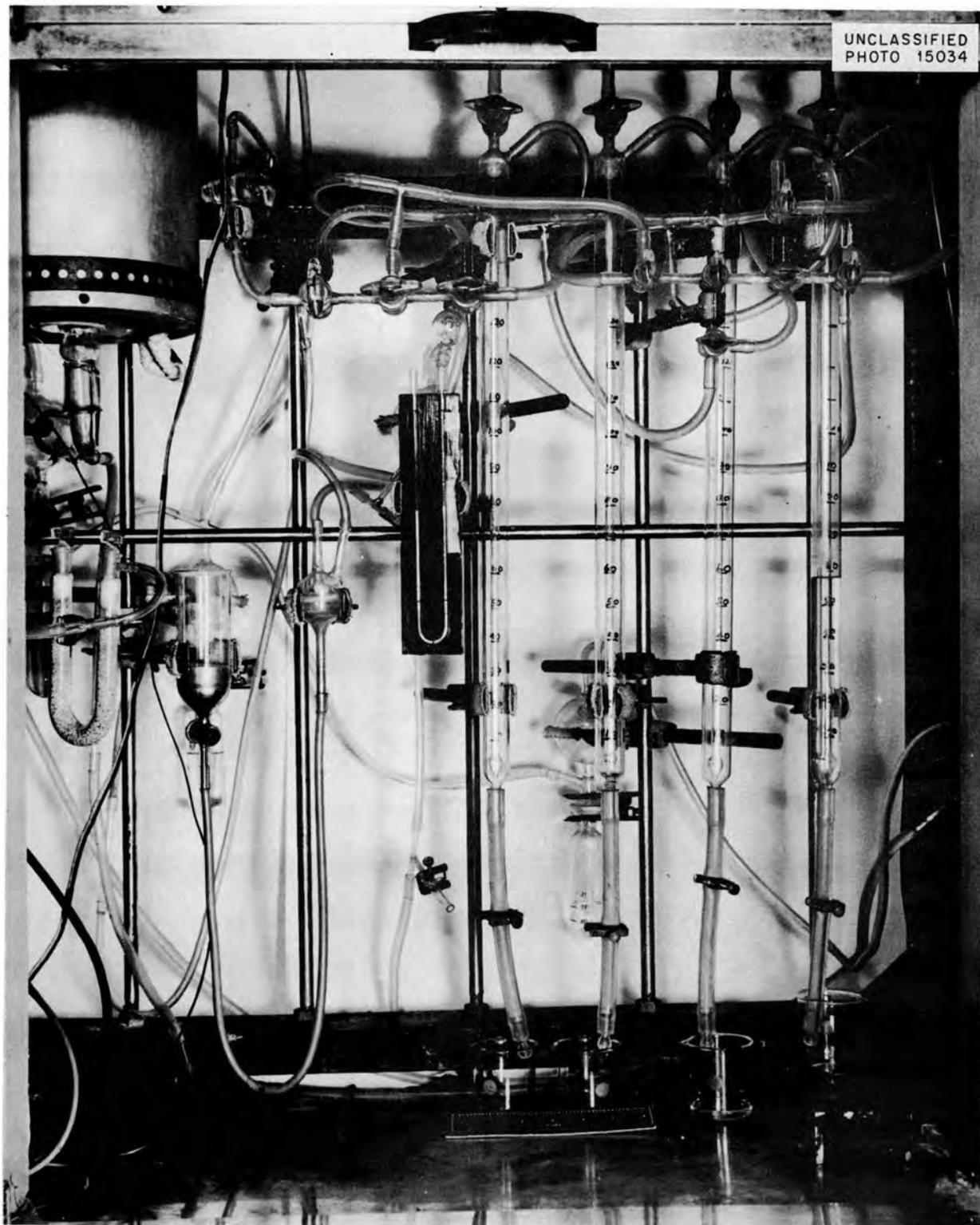


Fig. 4b. Photograph of Present Production Equipment - Gas Train.

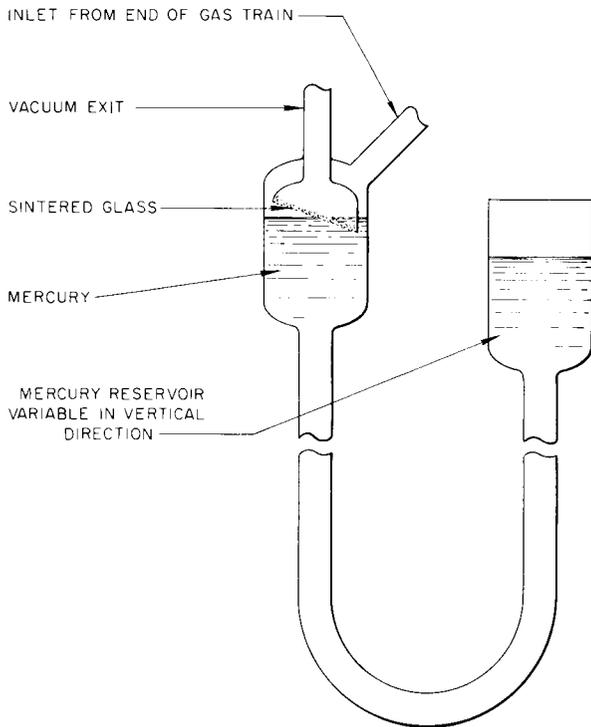


Fig. 5. Automatic Vacuum Controls for C¹⁴ Production Equipment.

supply is cut off, and the clamp is completely released.

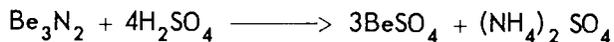
Dissolving the Nitride

With the system in this preparatory condition, the vacuum-control reservoir is slowly lowered until the pressure in the dissolver is brought to about 1 in. of water pressure. The voltage on the mantle is set at about 70 v and the charge is allowed to heat. The rate at which the water can be brought to the boiling point is dictated by the rate at which the gas over the water can expand through the scrubbers; the heating rate is controlled by Variacs on the mantle. When the water reaches the boiling point and the system comes to equilibrium, no bubbles pass through the scrubbers. The heat is adjusted until a very slow reflux rate is indicated by the drops returning from the condenser.

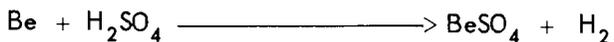
It is necessary to allow the equipment to come to equilibrium as described above before adding

acid because the reaction gives off heat and is accelerated by heat. The acid flow must be adjusted to the rate at which the reflux condenser can remove the heat. Four to five liters of 20 N sulfuric acid is used for each run (8 ml per g of beryllium nitride). When the run is ready for acid addition, the voltage on the mantle is lowered about 10 to 15 v, and the acid is allowed to start dropping in at about 20 to 30 drops per min. A glass bulb containing a dripping tip is in the acid addition line to enable the flow of acid to be observed. After the acid has been added for about 5 min at the above rate, the flow is stepped up about 25% and the voltage on the mantle is lowered another 10 to 15 v. This procedure is continued until the acid is entering in a small stream, and the voltage is completely turned off. If the charge becomes too hot, condensate will appear in the lines past the reflux condenser, and the acid flow must be reduced.

The reaction of the acid on the nitride is:



This reaction releases the C¹⁴ compounds which react with the H₂SO₄ as follows:



The gases from the above reactions are carried with water-pumped nitrogen from the dissolver through the reflux condenser into the gas train. A slow stream of these gases pass through the first scrubber, where the carbon dioxide and hydrogen cyanide are removed. The methane and other organic gases are passed through a copper oxide furnace (F, Fig. 3), which is held at a temperature of 750°C, to burn the organic compounds to carbon dioxide and water. The gases are dried by a sulfuric acid trap and a Drierite trap before being monitored by an ion chamber, which actuates an electrometer and a Brown recorder. The recorder serves to indicate the rate at which the activity is being released and aids in determining the time at which the sweeping can be stopped.

After being monitored the C^{14} dioxide is absorbed by sodium hydroxide solution in scrubbing columns.

As the acid addition nears completion, much of the nitride is dissolved and the reaction slows down, with a consequent decrease in the amount of heat liberated. Therefore, the voltage on the mantle must be gradually increased to maintain the charge at a boiling point. Also, near the end of acid addition, nitrogen flow is gradually increased to maintain a flow of gas through the system to sweep out the active gases. This also ensures the removal of hydrogen from the copper oxide furnace before oxygen is allowed in the system.

As soon as the system is safe for oxygen, 30% hydrogen peroxide is added dropwise and enters the solution near the bottom of the dissolving flask where it decomposes and forms many fine bubbles of oxygen. Some oxidation of organic compounds may take place, but the principal value of the H_2O_2 addition is the flushing action of the many small bubbles. The fact that the ion chamber indicates a large peak of activity during this operation indicates that most of the organic compounds flushed out are not oxidized because they pass through a sodium hydroxide scrubber before reaching the ion chamber. From 250 to 300 ml of the hydrogen peroxide are added, and, following this treatment, nitrogen is swept through the solution until activity is near background.

When the run is completed, the dissolver is emptied into the drain and is recharged with ten pellets. The capacity of the scrubbers is sufficient to absorb the gas from 50 pellets and they are usually emptied after this number of pellets have been processed.

Precipitation of Product

Recovery of the carbonate from the scrubbers is accomplished as follows. A flask fitted with a Tygon inlet tube and an Ascarite-filled vent tube is filled with nitrogen and attached successively to all the scrubbers to collect the active solution. Usually, more than one flask is used so that the solution from some scrubbers may be collected separately. The scrubbers can be vented at the top with nitrogen and have a water spray which washes out the last of the sodium hydroxide. When it is desirable to completely saturate the solution with carbonate, the scrubber following

the copper oxide furnace is charged with only a small amount of NaOH and a whole run is passed through it. This solution is then drained into a nitrogen-filled plastic bottle and retained as active sodium carbonate.

The flask containing the scrubber solution is placed in a large plastic box (Fig. 6) which has a nitrogen atmosphere. Openings in the box have long surgical gloves which permit working inside the box. While in this carbon dioxide-free atmosphere, barium hydroxide is added to precipitate the activity as barium carbonate. The barium carbonate is collected on a sintered glass disc and washed free of excess barium ion before it is taken out of the box to be dried and assayed.

Analyses show the isotopic abundance of the product made during the four years covered by this report to range from 4.8 to 29.0%. More recently, products containing ~40% C^{14} have been obtained. The following data show the proportion of the various ranges of isotopic abundance in the $BaCO_3$ produced during this period.

Isotopic Abundance (%)	Proportion of Total Carbonate Product (%)
4.8- 9.9	37.53
10.0-14.9	36.04
15.0-19.9	12.32
20.0-24.9	9.01
25.0-29.0	5.10

Variations in such factors as time of exposure, position in reactor, and carbon content of the beryllium nitride before irradiation account for the spread of values shown above.

SUMMARY OF OPERATING DATA

The present beryllium nitride- C^{14} processing equipment has been in operation since January 1951. The operating data for the period from January 1951 through December 1954 are summarized in Table 1.

The term "slug" as used in this report means the amount of beryllium nitride which was irradiated in one can. Each can contains two pellets of approximately 50 g each.

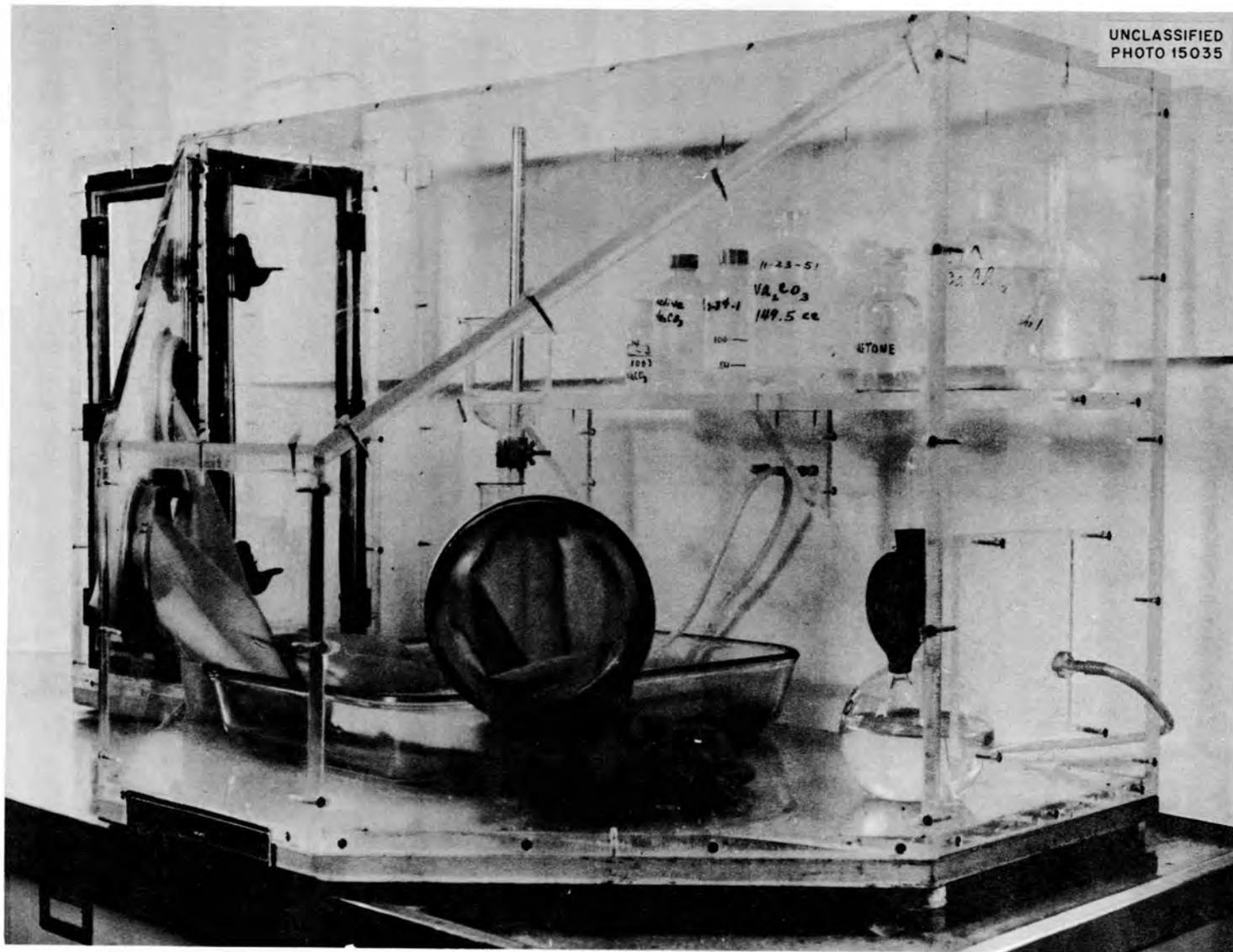


Fig. 6. Dry Box with Nitrogen Atmosphere for Precipitation and Collection of the C¹⁴ Product.

TABLE 1. OPERATING DATA FOR THE PERIOD JANUARY 1951 THROUGH DECEMBER 1954

Run No.	Number of Slugs	Be ₃ N ₂ (g)	20 N H ₂ SO ₄ (ml)	30% H ₂ O ₂ (ml)	BaC ¹⁴ O ₃ (mg)	C ¹⁴ (mc)	Isotopic Abundance (%)
1	2	199	1,600	130	2,902	106.0	10.3
2	5	547	4,375	250	6,262	155.0	6.9
3	5	495	3,960	250	4,780	123.0	7.3
4	5	492	3,940	250	4,607	157.0	9.6
5	15	1,441	11,543	750	9,426	397.0	11.9
6	15	1,496	12,036	750	7,231	415.0	16.2
7	11	1,070	8,560	500	9,507	160.7	4.8
8	25	2,542	20,427	1,200	11,593	578.6	12.6, 15.45
9 (Rework)							
10	20	1,957	15,660	1,000	28,225	1,813.9	22.9, 24.3, 17.3
11	21	2,245	17,939	750	21,002	1,049.0	16.4, 14.1, 12.7
12	21	2,283	18,255	750	22,533	937.0	14.2, 9.7
13	19	2,070	16,540	725	18,005	626.0	8.6, 10.9
14	18	1,874	14,992	950	19,371	638.6	8.5, 10.0
15	15	1,616	12,928	800	15,753	570.0	11.87, 8.91
16	20	2,225	17,196	1,000	20,464	736.0	8.8, 12.3
17	13	1,486	11,893	510	23,866	782.0	7.4, 10.1
18	10	976	7,840	375	8,018	541.0	13.2, 25.2
19 (Rework)							
20	27	2,874	22,978	1,450	9,603	1,284.0	
21 (Rework)							
22	15	1,560	12,500	750	15,873	301.0	10.3, 7.87
23	25	2,655	21,221	1,500	33,771	1,116.0	8.2, 10.0
24	25	2,636	21,078	1,500	30,228	1,201.0	8.0, 13.6
25	25	2,641	21,140	1,500	34,848	1,046.1	5.8, 10.6
26	25	2,648	21,182	1,500	26,415	951.0	9.1, 10.1
27	20	1,980	15,810	1,200	27,593	2,226.0	28.2, 18.6
28	10	1,051	8,550	600	13,457	702.0	15.2, 14.3
29	15	1,459	12,000	900	15,307	1,240.9	21.1, 25.4
30	15	1,411	11,400	900	17,140	1,472.1	20.2, 29.0
31	10	1,009	8,070	600	10,845	523.9	13.3, 14.1
32	10	996	7,968	600	16,740	773.0	10.1, 18.6
33	20	2,343	18,780	1,200	24,722	1,764.0	18.1, 20.0
34	25	2,621	21,120	1,500	23,025	1,637.0	18.8, 22.7
35	15	1,547	12,450	900	19,557	703.0	7.34, 13.3
Total	522	54,445	436,522	27,540	552,670	26,726.8	

In Table 1, the column referring to isotopic percentage has more than one value in most runs because the solutions from the individual scrubbers may be collected in more than one precipitating flask. The solution from the scrubber preceding the copper oxide furnace is usually kept separate and occasionally the solutions from the remaining four scrubbers may be collected in two or more flasks before the carbonate is precipitated.

The following information summarizes from Table 1 the pertinent data regarding the beryllium nitride processed during this period.

Totals	
Slugs used, number	522
Beryllium nitride, g	54,445
20 N H ₂ SO ₄ , liters	436.5
30% H ₂ O ₂ , liters	27.5
Copper oxide, lb	12.0
BaC ¹⁴ O ₃ , g	552.67
C ¹⁴ in above BaC ¹⁴ O ₃ , mc	26,726.8
Averages	
Beryllium nitride per slug, g	104.3
C ¹⁴ per slug of beryllium nitride, mc	51.2
C ¹⁴ per gram of beryllium nitride, mc	0.49