THE PREPARATION OF ULTRA FINE BERYLLIUM POWDER BY THE AMALGAM PROCESS

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TECHNICAL REPORT

November 1965

NASA Contract NASW 1099

GENERAL ASTROMETALS CORPORATION

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This investigation was financed by and performed under the auspices of the National Aeronautics and Space Administration, Contract No. NASW 1099, with General Astrometals Corporation as prime contractor.

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ABSTRACT

A pilot plant designed to produce 0.5 lb of micron and submicron beryllium metal powder from commercial $BeCl_2$ per 8-hr shift is described. The process involves the electrolysis of beryllium out of a molten eutectic mixture of $BeCl_2$-NaCl at 300° C, using a mercury cathode and a carbon anode. Separation of the quasi-amalgam formed, recovery of beryllium by pressure removal of the majority of the mercury and vacuum distillation of the residual mercury complete the process.

Author

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INTRODUCTION

Electrolysis of an aqueous solution using a mercury cathode has been employed in preparing amalgams, from which pure metals are recovered by distilling off the mercury[1, 2]. Metals which will yield ultrafine powders by electrolysis into a mercury cathode are those that have very low solubility in mercury. These metals are suspended in the mercury in the form of fine particles; the suspension is called quasi-amalgam. The powder particles in a quasi-amalgam are highly reactive and if the quasi-amalgam is exposed to air, oxidation of the powder particles will occur rapidly. Metal powders with particle sizes in the micron to sub-micron range can be produced by the amalgam process.

Iron and nickel powders of extremely fine particle size and consequently high reactivity were prepared by the amalgam process[3, 4]. Because nickel forms compounds with mercury, while iron does not, the nickel powder obtained is much finer than the iron powder. The breakdown of the nickel compound during distillation of the mercury results in the reduced particle size[5].

In the mercury phase, the suspended particles undergo a process of aging, i.e., agglomerate spontaneously, even at room temperature. If a high enough temperature is used to distill off the mercury, sintering may take place. The sintered particles may be bonded together, even as low as 400° C as in the case of iron[6] and cannot easily be crushed to fine powders. Therefore sintering should be minimized by distilling under high vacuum at as low a temperature as possible. Further, since the sintering temperature of a metal powder is lower the lower the melting point of the metal, it follows that the amalgam process is really suitable only for relatively high melting

point metals.

Beryllium has a solubility of 0.001 wt % of mercury[7]. It is not yet known whether or not it forms compounds with mercury. It is known however, that when a beryllium salt is electrolyzed using a mercury cathode, a quasi-amalgam is formed. Because of the high oxidation potential of beryllium, it cannot be electrolyzed out of an aqueous solution.

The preparation of ultrafine beryllium powder by electrolysis of a molten salt using a mercury cathode was investigated by Kells, Holden, and Whitman [8, 9] as the result of a patented process utilizing this principle by Kopelman and Holden[10]. The process makes use of a molten $BeCl_2$-NaCl electrolyte, a graphite anode and a mercury cathode. It is of course necessary that the electrolyte be molten below the boiling point of the mercury. At first a 5-in. diameter cell was operated, and later, a continuous process was developed using a 10-in. diameter cell. During electrolysis the mercury cathode was continuously stirred and the amalgam continuously extracted from the cell. The amalgam was filtered and the mercury recycled to the cell. Stirring of the mercury was necessary to constantly expose fresh cathode surface. The dilute amalgam was concentrated to 2 wt % Be by filtration; further concentration was achieved by pressing at relatively high pressure. The powder could also be recovered by distilling the concentrated amalgam under vacuum.

The beryllium powder produced by the amalgam process of Kopelman et al had a much smaller particle size than beryllium powders produced by other methods. Such ultrafine powder may be especially suitable for certain powder metallurgical applications as well as for incorporation into a potential rocket fuel. If the proper precautions are taken to exclude

oxygen and water from all operations, the powder should be of purity at least equivalent to commercial beryllium and with a particle size of about 0.1-10μ. This earlier work showed that it was also possible to produce metal compacts directly by hot pressing the concentrated amalgam[8, 9]. The heavy mercury vapor envelopes and protects the compact from oxidation during pressing.

One important advantage of utilizing a mercury cathode is the belief that the product will not be contaminated with electrolyte as is the case when a solid cathode is used. Therefore no washing step is necessary to remove the electrolyte.

PROCESS DESCRIPTION

The present investigation was instituted using a larger cell than that originally used by Kells et al. It is hoped that experience applicable to large-scale production of beryllium powder by the amalgam process will be obtained. At the same time sufficient powder will be produced for evaluation of its metallurgical potential and its applicability as a rocket fuel additive.

Beryllium cannot be produced by aqueous electrolysis, because the beryllium ion is strongly hydrated in solution and the only product obtained is $Be(OH)_2$. The reduction potential of the beryllium ion in aqueous solution is not favorable. Electrolysis in organic solvents has been tried but has been unsuccessful due to low power efficiency, low yield, and product contamination[11]. Electrolysis using fused salts is, therefore, the apparent route for producing beryllium. Pure molten beryllium salts are not appreciably ionized and usually have a high vapor pressure. They are mixed with an

alkali or alkaline earth salt to form a low-melting eutectic, with increased conductivity and lowered vapor pressure.

An electrolyte to be used for beryllium electrolyte must contain no component with a decomposition potential lower than that of beryllium. A $BeCl_2$-NaCl eutectic mixture meets these requirements. Fig. I shows the $BeCl_2$-NaCl phase diagram according to Schmidt[12]. It will be seen that the eutectic occurs at 50 mol % $BeCl_2$ and melts at 215°C.

A flowsheet of the overall process is shown in Fig. 2 which indicates that, first, $BeCl_2$ is purified by sublimation at 485°C. The purified $BeCl_2$ is fed to the electrolysis cell where an approximately eutectic mixture of $BeCl_2$-NaCl is electrolyzed at about 275-300° C. The beryllium collects in the mercury cathode in the form of a quasi-amalgam. The quasi-amalgam is drawn off to a filter where a paste of quasi-amalgam is collected; the mercury passing through the filter is pumped to a holding tank ready for recirculation to the electrolysis cell. The paste is pressed to squeeze out more mercury and the mercury which remains after pressing may be removed by distillation leaving beryllium powder.

RAW MATERIALS

In order to produce a pure powder, all raw materials must be of the highest possible purity. Because most impurities in the fused salt electrolyte are electrolyzed at a decomposition potential below that of beryllium, they will contaminate the product.

Beryllium Chloride

Commercially available $BeCl_2$ may be of sufficient purity to produce pure beryllium metal by the amalgam process. Due to its high reactivity

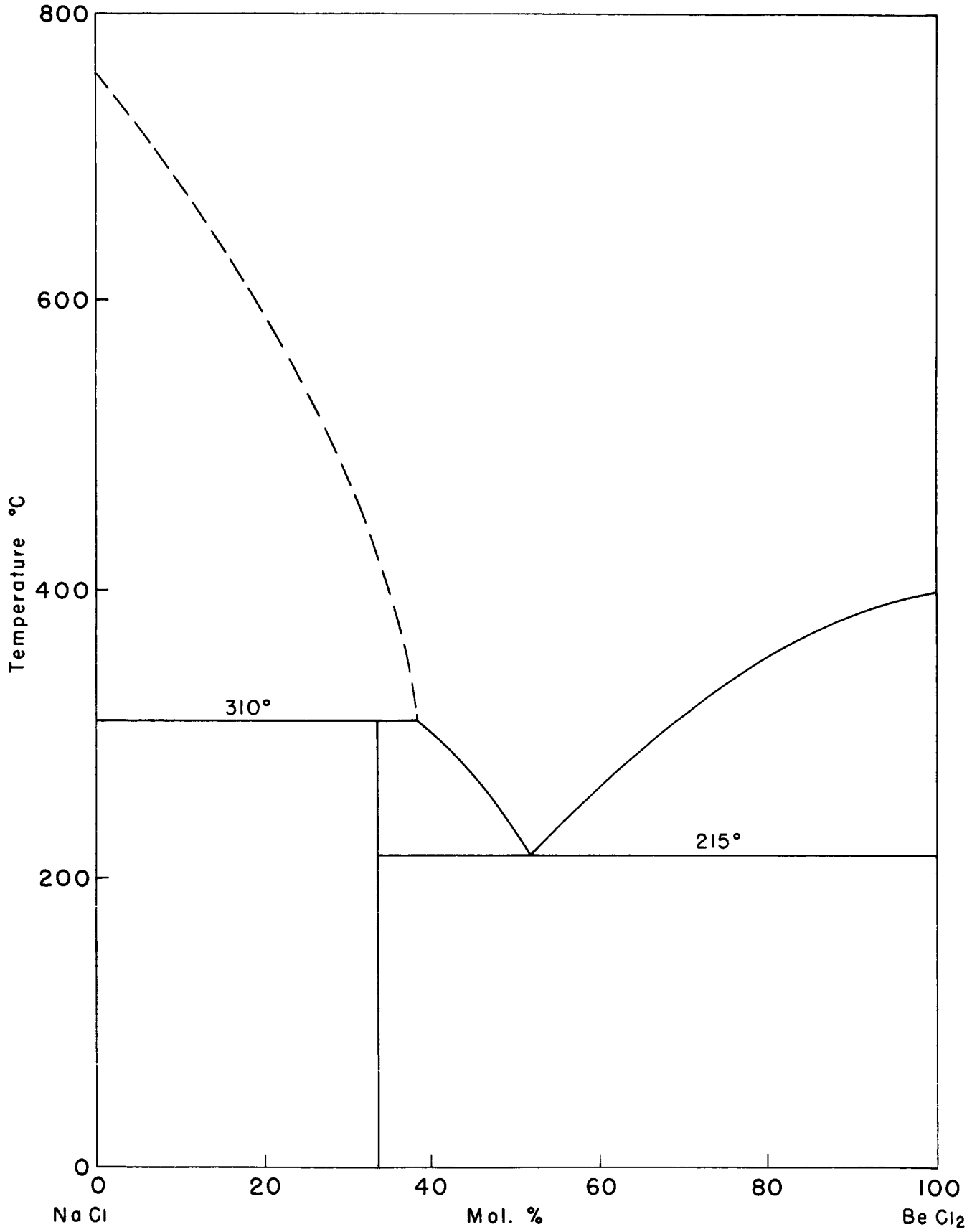


Fig. 1 — Phase diagram $BeCl_2$–NaCl

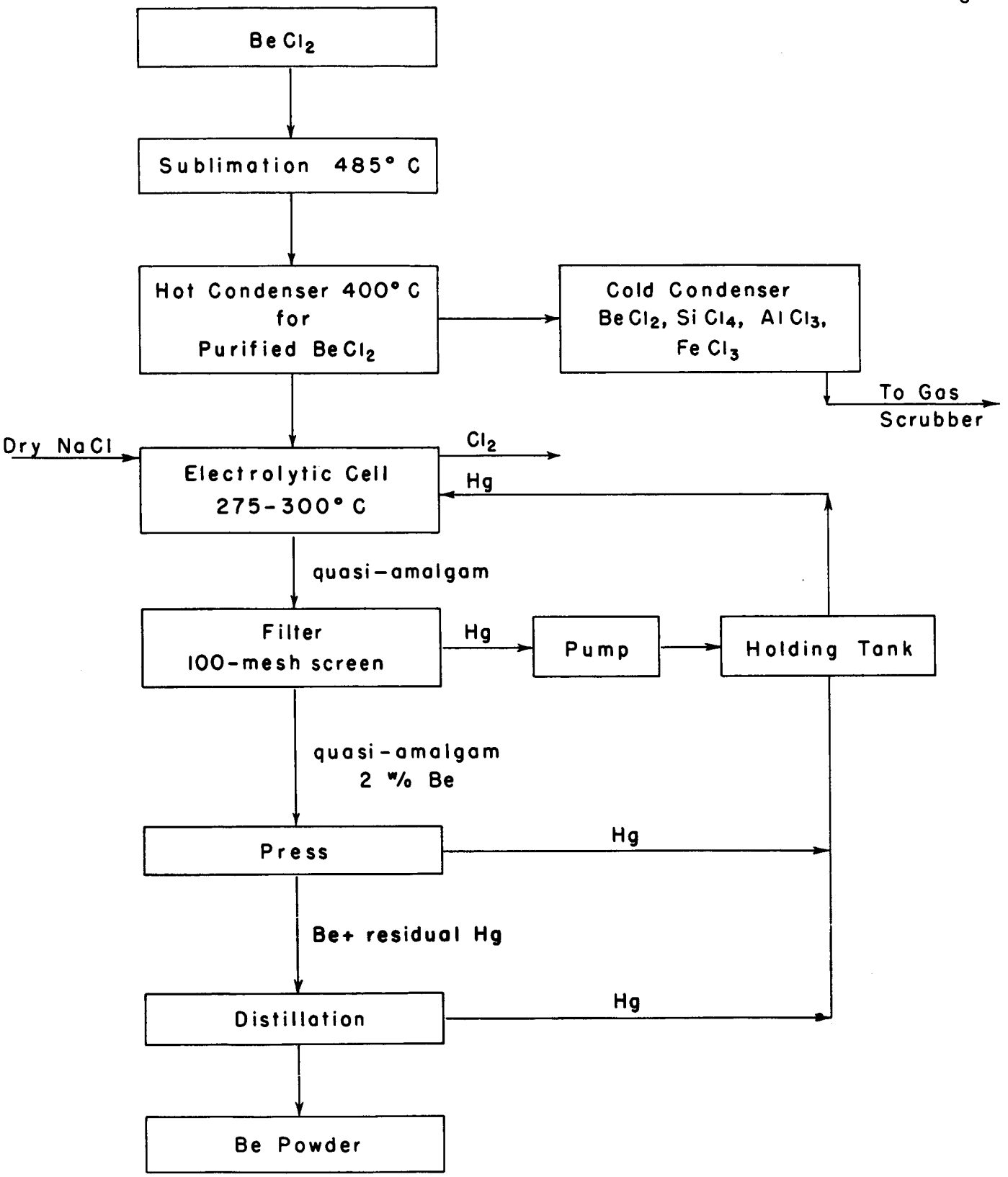


Fig. 2 — Flow Sheet of Amalgam Process

with air and moisture, non-volatile oxychlorides may be present. Sublimation and fractional condensation is a convenient method to purify $BeCl_2$[14] because of its relatively low sublimation temperature compared to the boiling temperatures of other impurity chlorides (Table I). Here, $BeCl_2$ was sublimed primarily to eliminate water and oxides.

TABLE I

Melting Points and Boiling Points of Some Chlorides [15]

Chloride	M.P. °C	B.P. °C
$SiCl_4$	-70	57.57
$AlCl_3$	190	177.8 (subl)
$FeCl_3$	282	315
$BeCl_2$	440	520 (subl)
$NiCl_2$	1001	973

Sodium Chloride

Reagent-grade sodium chloride was acquired from J. T. Baker Chemical Company. The supplier's analysis is shown in Table II.

TABLE II

Impurities in NaCl.

Impurity	ppm
Arsenic	5
Bromine	30
Iron	0.5
Heavy Metals	40
Iodine	10
Insol. residue	30
Potassium	4
Nitrous oxides	10
Phosphate	1
Sulfate	5

Mercury

The mercury was spectographically comparable to reagent-grade triple-distilled mercury.

Inert Gas

An inert dry atmosphere is needed to prevent oxidation and hydrolysis of the $BeCl_2$, of the quasi-amalgam and of the beryllium metal powder. Since beryllium is not reported to form nitrides at low temperatures, nitrogen can be used as the inert gas. It was obtained in low-pressure liquid form and the supplier's analysis of the gas was 99.997% purity and with a dew-point -84°F. Argon may replace nitrogen in the future if the ultra fine beryllium produced should be reactive to nitrogen.

EQUIPMENT

The pilot plant consists of two sections: a sublimation unit and an electrolytic cell system. All equipment and accessories are located in the General Laboratory Building, of The Anaconda Company, Anaconda, Montana. Fig. 3 shows the floor plan.

The $BeCl_2$ Sublimation Unit

The sublimation unit is designed to purify a 5-lb batch of commercially pure $BeCl_2$ by sublimation and fractional condensation as shown in Fig. 4. The furnace (1) is raised and lowered around the charge crucible by a jack. The charge crucible (2) is made of nickel and is clamped to the furnace lid (3). Carrier gas enters the crucible to transport the hot vapors to condensers. The furnace is housed in a hood (Fig. 5) to prevent toxic vapors from entering the room.

The condensers are 12-in. diameter nickel cylinders with tapered bottoms to match 8-in. diameter crucibles. The hot condenser (5) is heated by strip heaters, and is insulated to effect the fractional condensation of $BeCl_2$ while

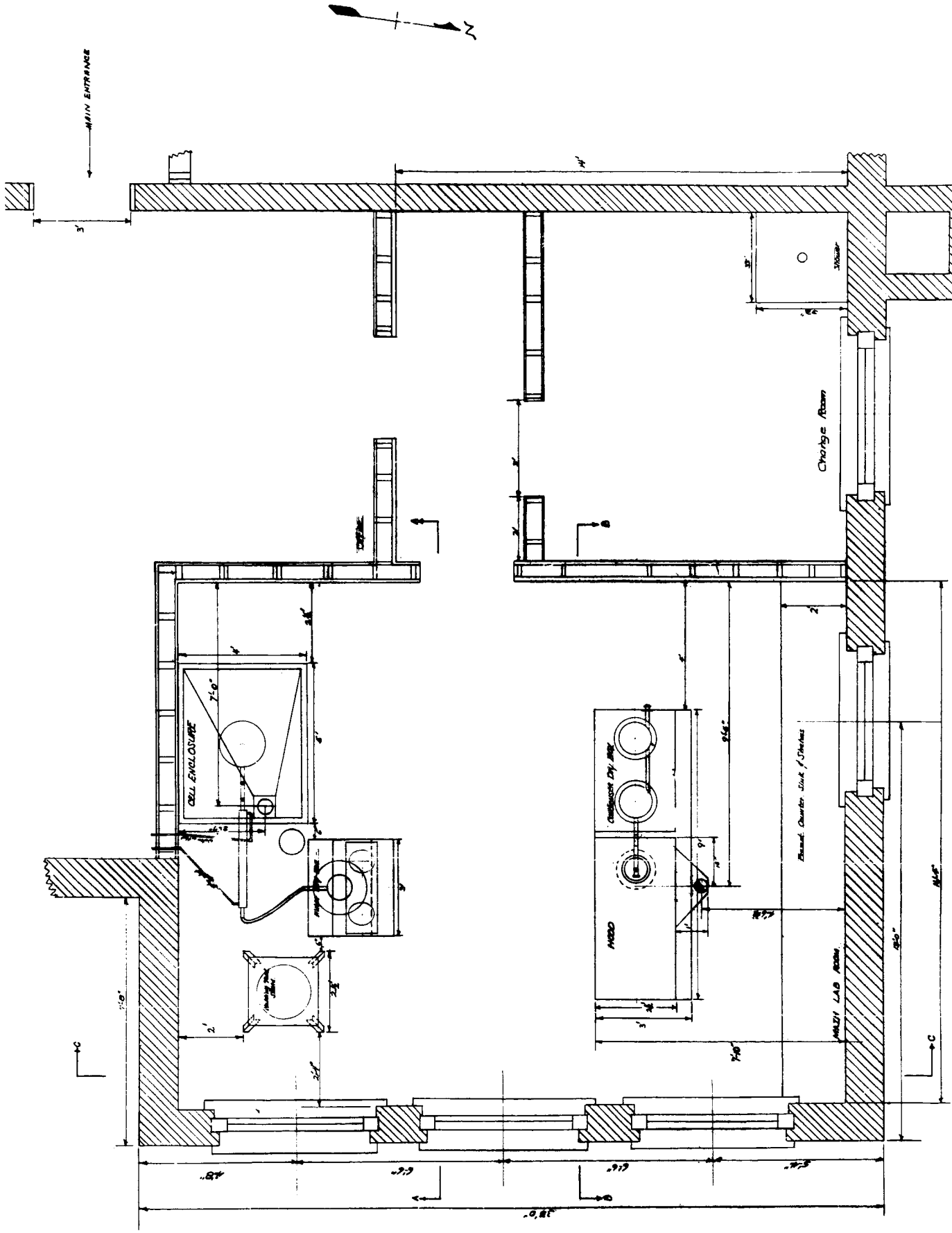


Fig. 3 Floor Plan

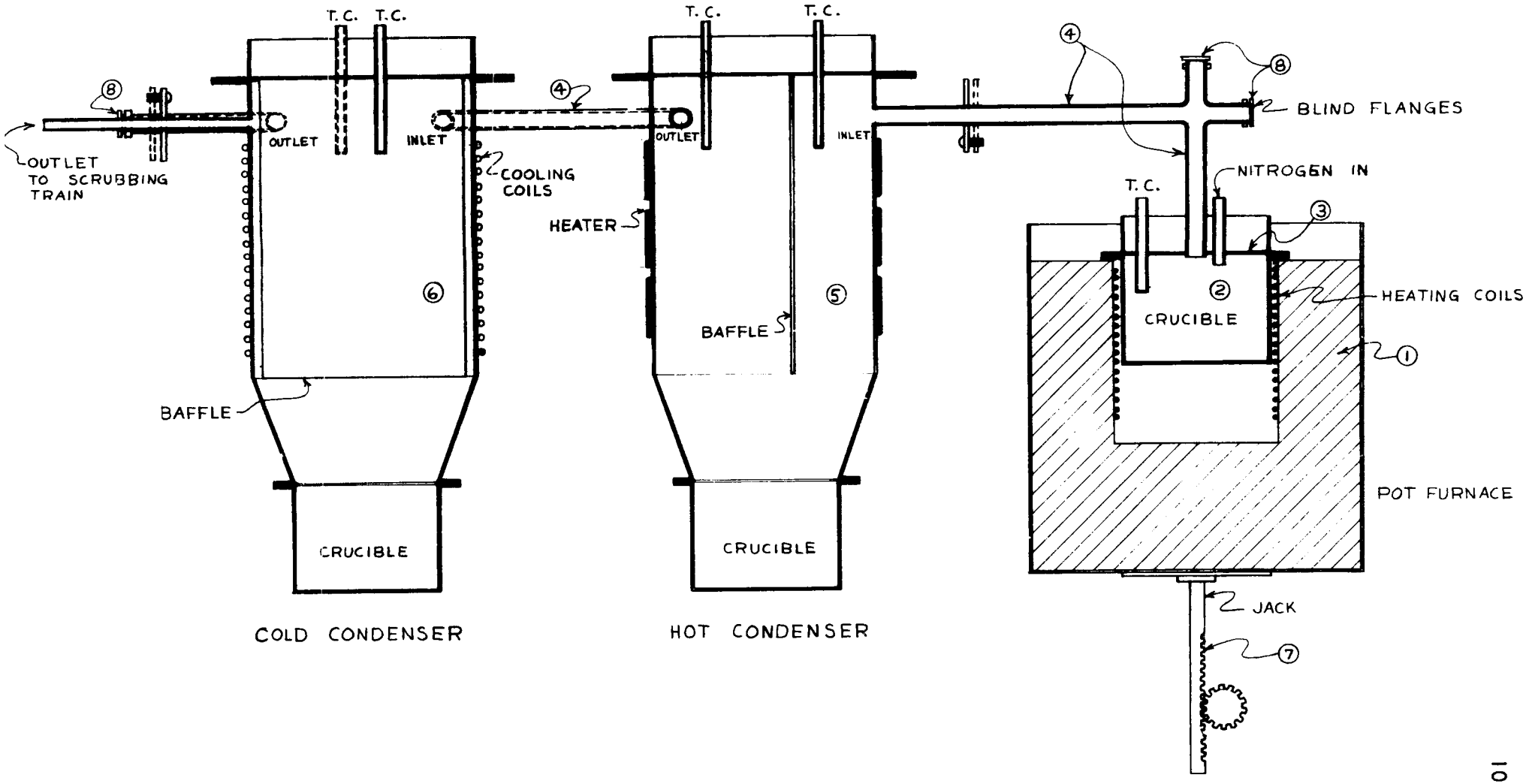


FIG. 4—SUBLIMATION UNIT ASSEMBLY

the cold condenser (6) collects any fine $BeCl_2$ particles and impurities which escape from the hot condenser. The condensers are contained in a stainless-steel, nitrogen atmosphere box (Fig. 5) to prevent oxidation and hydrolysis of the purified $BeCl_2$. To prevent premature condensation of $BeCl_2$, which can cause clogging, the connecting pipes (4) are kept near 300°C by heating tapes.

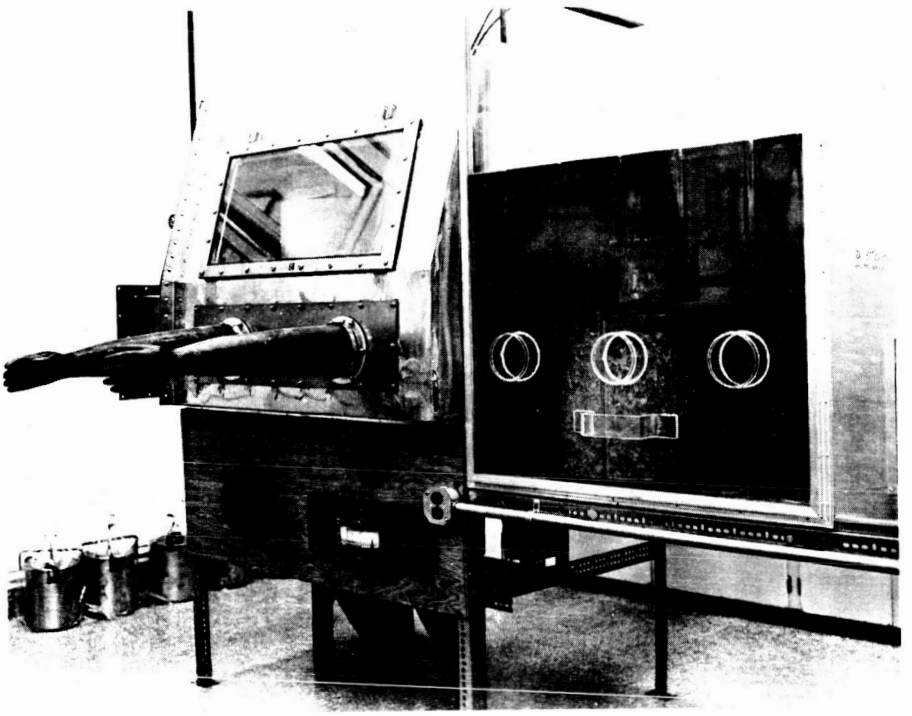


Fig. 5. Dry Box and Hood

The outlet of the cold condenser is connected to the gas train. The gas scrubbing train consists of a large carboy to collect most of the fine dust leaving the cold condenser, an anhydrous sulfuric acid trap, and a sodium hydroxide scrubber. The gases leaving the gas train are vented to the stack.

Electric power to the pot furnace is controlled manually with a variable transformer. The sublimation unit contains five thermocouples located as follows: pot furnace, hot condenser inlet, hot condenser outlet, cold condenser inlet, and cold condenser outlet. All thermocouples are connected to a recorder, while the hot condenser outlet thermocouple is connected to a controller, which regulates the power input to the strip heaters.

Electrolytic Cell System

A schematic outline of the electrolytic cell system is shown in Fig. 6.

The cell (1) was designed with a capacity of 500-550 lb of mercury and 100 lb of electrolyte to be capable of producing at least 0.5 lb of beryllium powder per 8-hr shift. To prevent any toxic vapors from entering the room, the cell is contained inside a large wooden hood, which is connected to an exhaust system for safe disposal of the vapors.

The cell (Fig. 7) consists of a 15-3/4 in. inside diameter stainless-steel cylinder 22-1/2 in. deep. It is covered by a 5-in. deep lid which is attached to the cell by a bolted flange. A false lid which fits in a groove inside the flange separates the cell from the lid, allowing for a gas purge. The flange is gasketed with a neoprene O-ring, and cooled by a copper water-coil.

The cell is sleeved inside with a Pyrex cylinder 15-3/8 in. outside diameter and 18-1/8 in. depth. This sleeve restes on brackets 3-1/2 in. up from the inside bottom of the cell. There are two Teflon blocks on the cell bottom to seat the stirrer shafts. Two stirrer shafts extend through the lid, sealed by rotary mercury seals, into the cell, where they are protected from the salt by glass sleeves, and into the mercury. The electrolyte is prevented from contacting the cell wall by the glass cylinder.

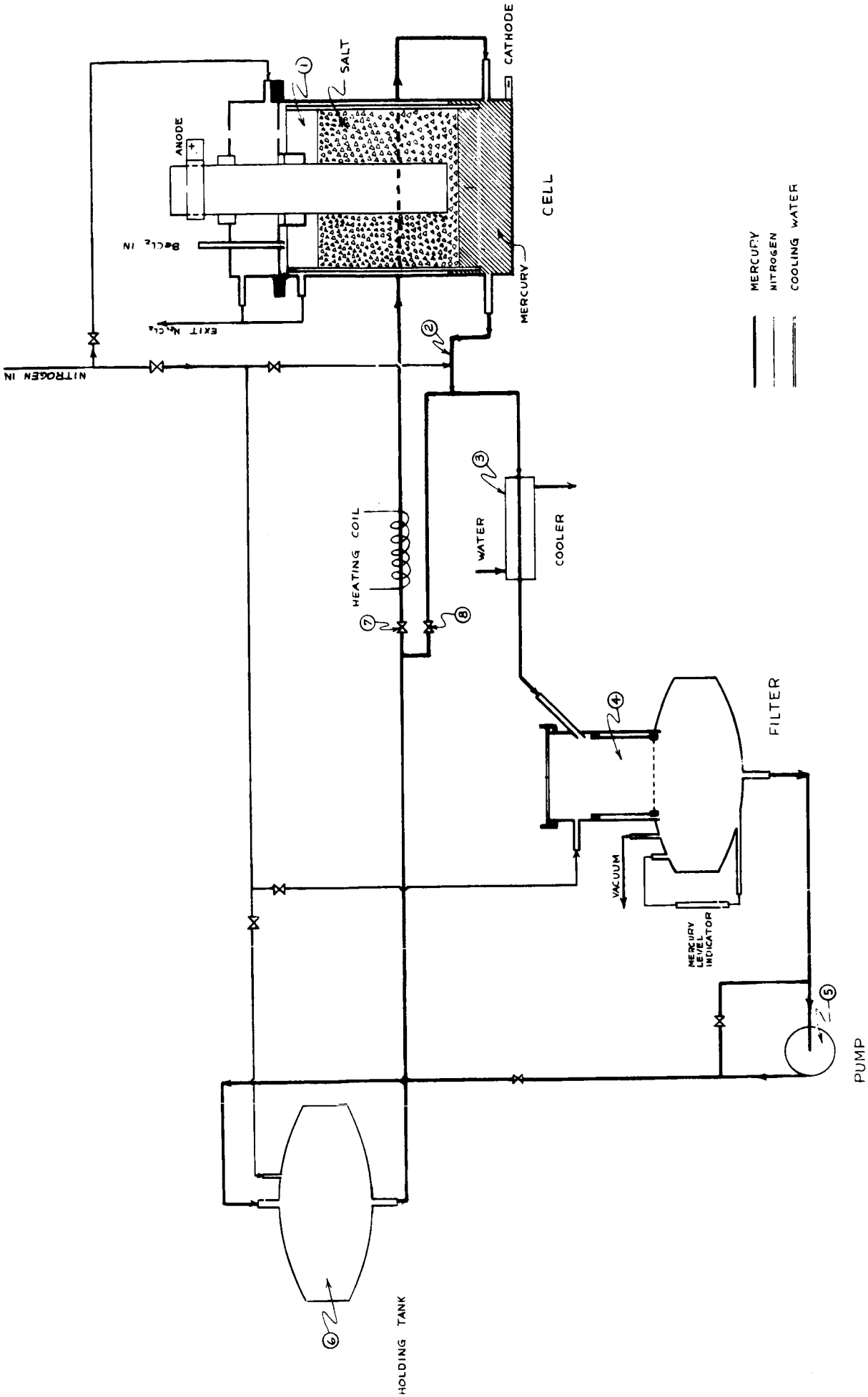


FIG. 6 — ELECTROLYTIC CELL SYSTEM SCHEMATIC

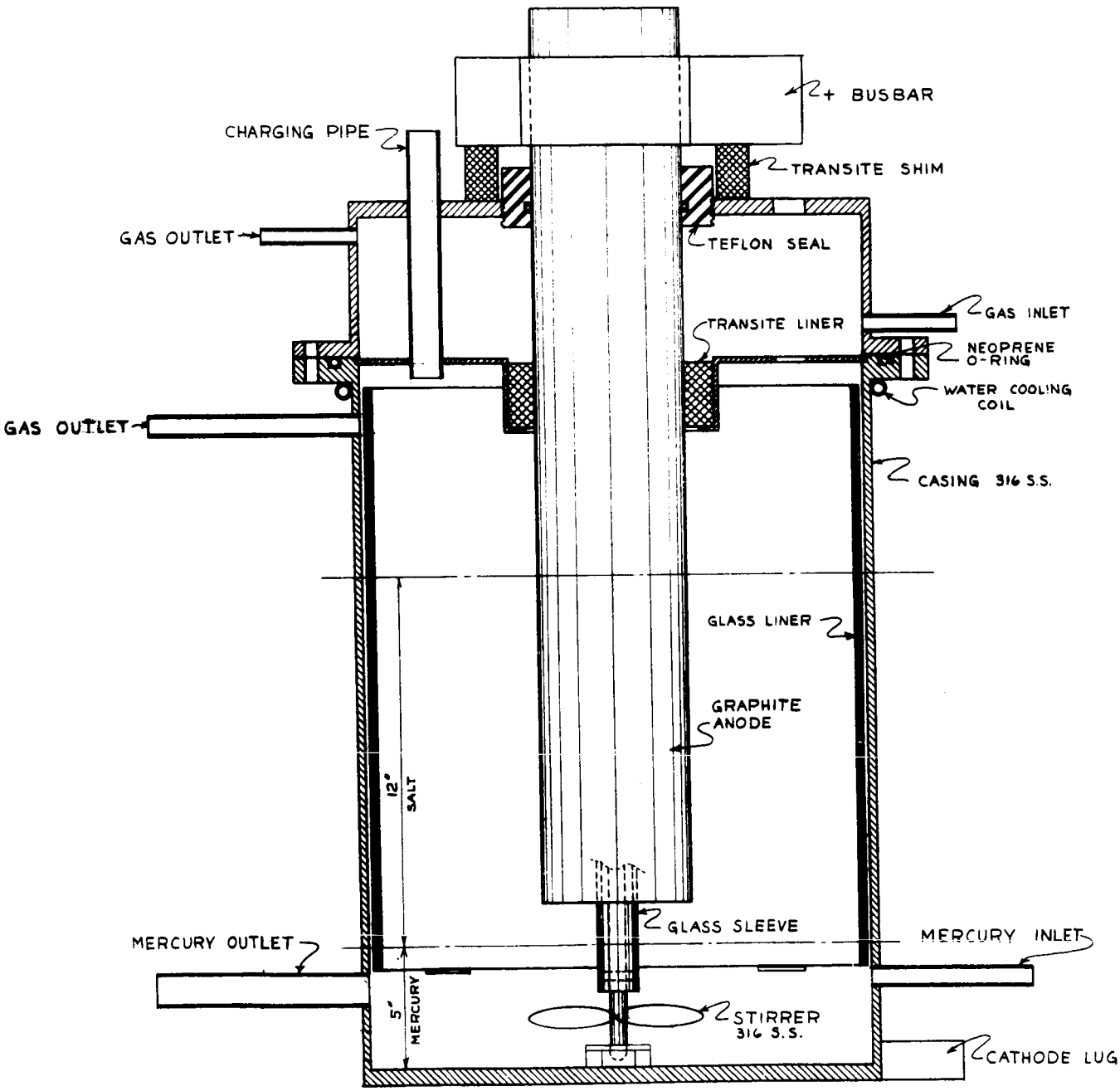


FIG. 7— ELECTROLYSIS CELL

The anode, a 4-5/8 in. diameter doubly-impregnated graphite rod, extends through the cell lid, false lid, and thence into the salt. The distance between the anode and cathode which determines the amperage at any given voltage, is approximately two inches. The anode is sealed and insulated through the lid by a Teflon sleeve containing a Teflon O-ring, and is insulated from the false lid by a Transite liner.

The level of the mercury in the cell is controlled by an overflow U-trap (2). In (Fig. 6) a valve on the inlet side permits mercury to enter the cell, thus forcing the amalgam out of the cell, through a cooler (3) and finally into the vacuum filter (4). The cooler can be washed with clean mercury by opening another valve to bypass the cell.

A mercury level indicator (Fig. 8) is used inside the cell so that fluctuations in mercury level may be observed. It consists of three platinum-tipped, glass-shielded probes: in the mercury, the salt, and at the interface. The probes in the salt and mercury are connected in parallel while the probe at the interface is common to both circuits. The two independent circuits indicate mercury level changes by a variation in the intensity of the lamp in each circuit. Normally the lamp in circuit with the probe in the mercury is bright, while the other lamp is noticeably dim due to lowered conductivity of the salt. If the mercury level goes down, the lamp in circuit with the mercury probe grows dimmer but if the mercury level goes up, the lamp in circuit with the probe in the salt becomes brighter.

The cell also contains three thermocouples: One is located in the mercury and is connected to a heater controller while the other two are located at different depths in the electrolyte. All three are connected to a recorder.

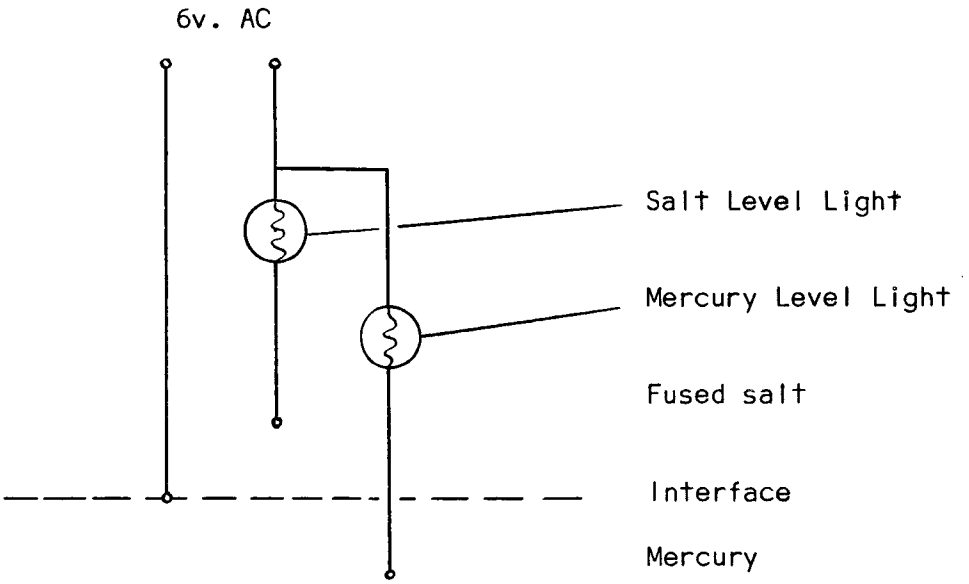


Fig. 8. Mercury Level Indicator Schematic

The filter assemby (Fig. 9) consists of an outer filter tank; a removable cup inside the filter tank which contains the 100-mesh filtering screen supported by heavy gauge screens and a lid on top of the filter tank which is fastened down by a single bolt clamp. The mercury exits from the filter tank, through a pump (5), and into a holding tank (6).

The filter tank has a capacity of 640 lb of mercury and the cup a capacity of 75 lb of amalgam cake. The screens are kept in place by a retaining ring on the cup bottom, and the cup may be removed through the top of the tank. Mercury and nitrogen enter above the screen cup, while the vacuum exit and mercury outlet are below the screens in the filter tank.

The filter is contained inside a nitrogen-filled controlled atmosphere box to prevent oxidation of amalgam during handling. The filter cup can be transferred to another dry box for storage or further handling. The

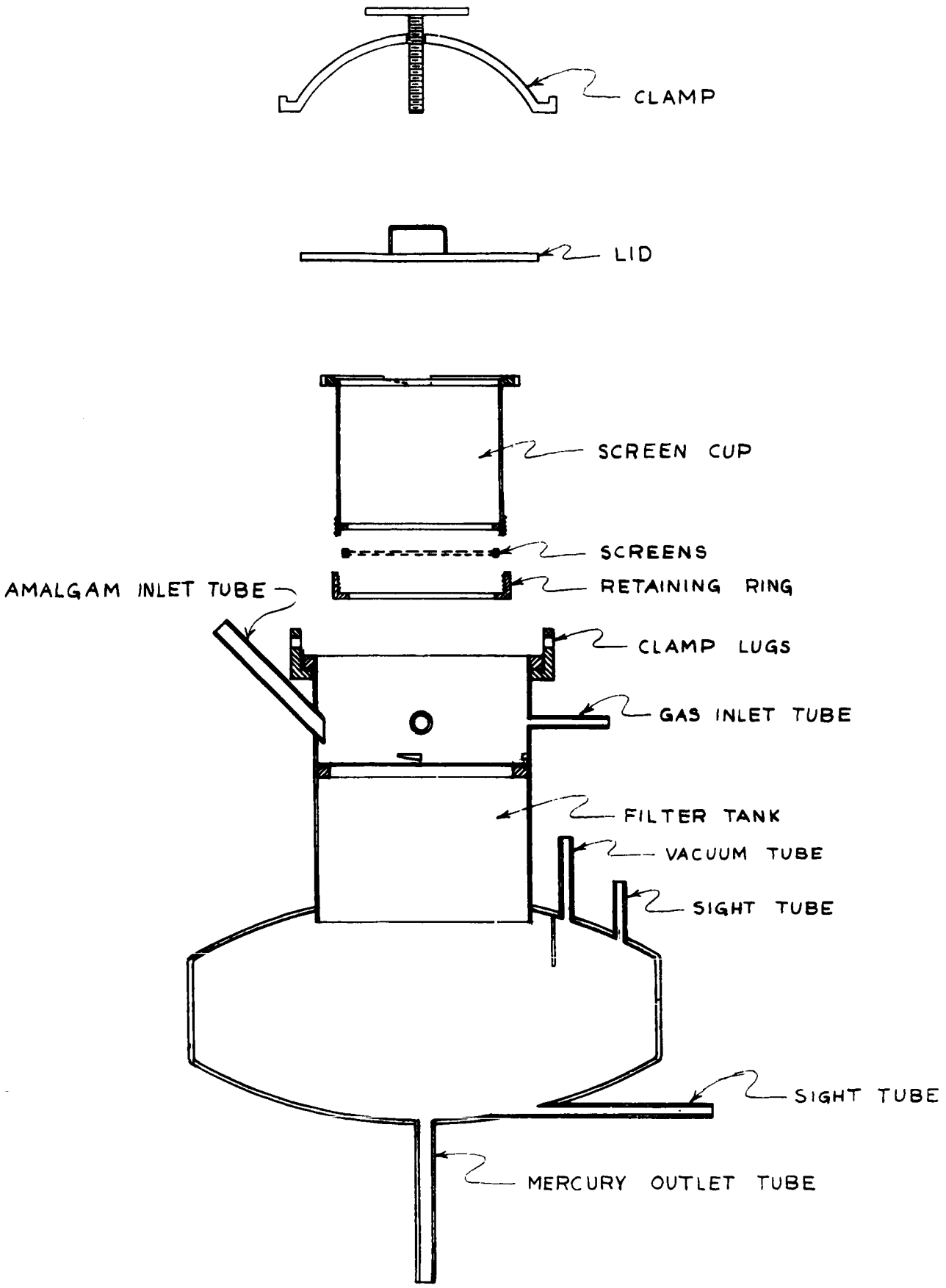
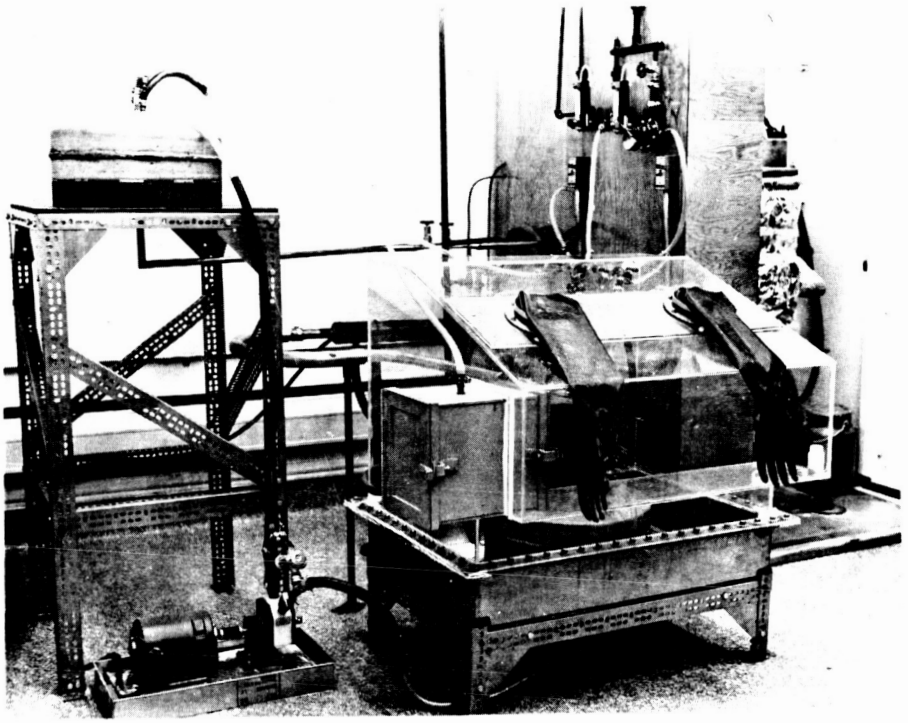
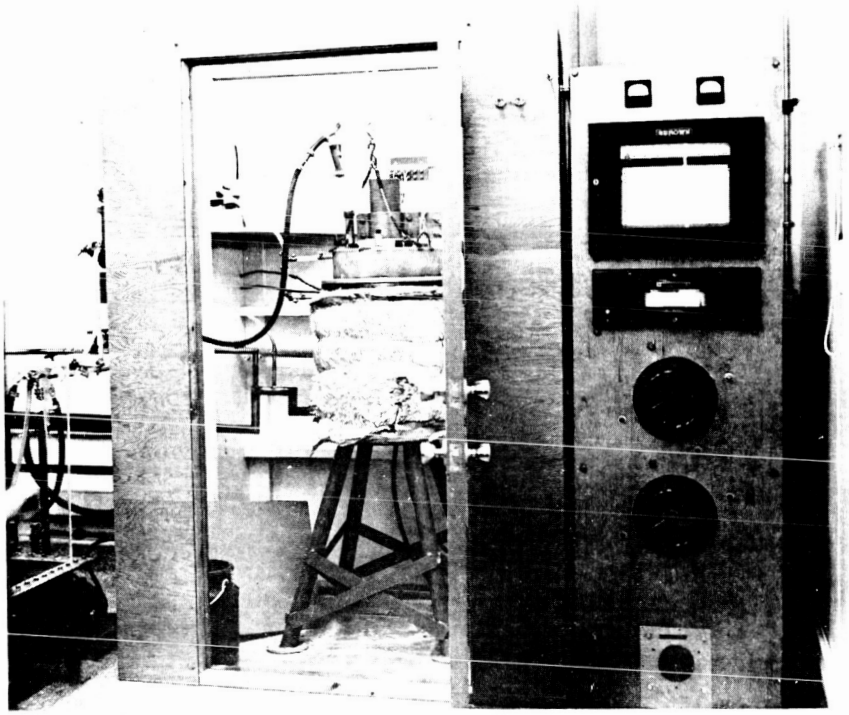


FIG. 9 — FILTER ASSEMBLY



Filter Dry Box, Pump, Holding Tank, and Nitrogen Controls



Cell, Enclosure, and Control Panel

FIG. 10 — ELECTROLYTIC CELL SYSTEM

amalgam may be also removed from the screens and transferred to containers inside the plastic dry box.

The mercury pump is a peristaltic type, and is specified to sustain a mercury flow of 0.1 gpm against a head of 60 in. This allows storage of mercury above the cell level so that tapping can be made to occur by gravity flow. The mercury leaving the holding tank is controlled by the inlet valve (7) or the bypass valve (8).

The electrolytic cell system is shown in Fig. 10.

Powder Recovery Unit

The oxygen-sensitivity of both the amalgam and powder necessitates that both be handled in a vacuum dry box. By pressing the amalgam in a press outfitted with the proper accessories, it is possible to remove approximately 90% of the mercury from the amalgam. The pressed beryllium amalgam must then be transferred to a vacuum distillation unit to remove the residual mercury. The mercury still (Fig. 11) was selected to permit removing the lid from the retort for charging the amalgam paste and collecting the powder after distillation.

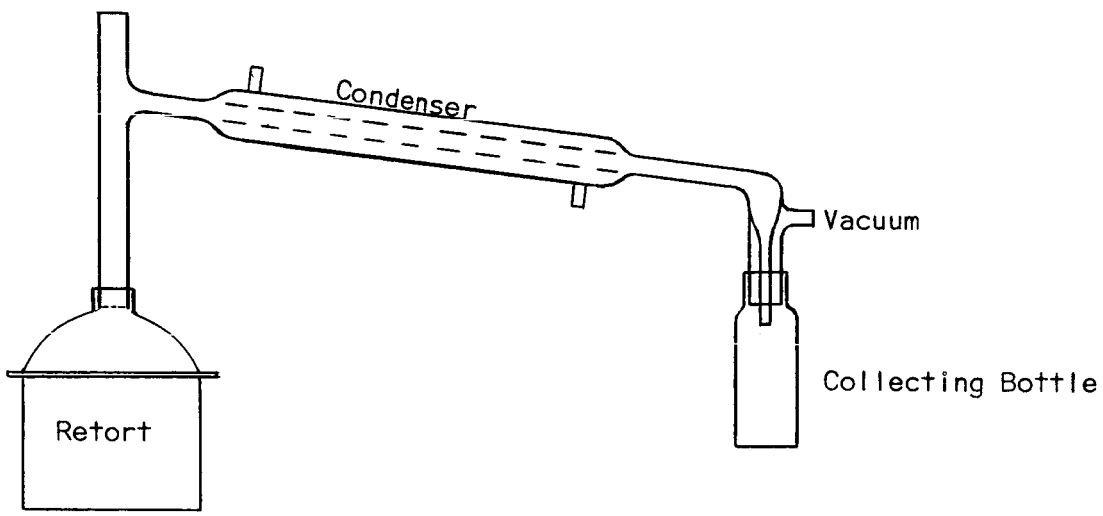


Fig. 11. Vacuum Distillation Unit

RESULTS

$BeCl_2$ Purification

The sublimation unit was operated successfully for over a month on its initial trials. The main difficulty was plugging of the connecting pipes. This usually occurred when the furnace temperature was 225-300°C. It was found that with slow heating rates, high nitrogen flow rate and controlling the hot condenser temperature at 350-400°C, the appearance of the purified product was improved, i.e., a spongy, loose-packed, acicular deposit was produced.

For analysis the purified $BeCl_2$ was dissolved in water, NH_4OH added to precipitate $Be(OH)_2$, which was taken to dryness and ignited to BeO. Spectrographic analysis of this material is given in Table III.

TABLE III
Analysis of BeO from Purified $BeCl_2$

(Values in Ppm)

	Sample 1	Sample 2	Sample 3	Sample 4
Al	104	64	40	70
Ag	5	3	2	9
B	Trace	Trace	Trace	Trace
Ca	180	200	95	260
Cd	53	110	18	24
Co	420	150	70	250
Cr	360	1300	150	41
Cu	490	52	300	550
Fe	300	255	100	150
Mg	110	160	60	170
Mn	23	50	9	11
Mo	47	52	17	17
Na	290	150	120	200
Ni	310	650	105	140
Pb	115	82	68	45
Si	850	480	370	730
Ti	190	105	70	160
Zn	320	170	110	220

Operation of the Cell

Two electrolytic runs have been made to date. The first lasted 3 hours, 24 minutes and averaged 190 amperes, 6 volts, for an ampere-hour total of 646. The second operated for 4 hours 15 minutes and averaged 250 amperes, 7 volts, for an ampere-hour total of 1062. The ampere-hour total of 1708 is equivalent to .443 pound Be or a depletion of 3.94 pound of $BeCl_2$. Analysis of the salt showed a decrease of 2.35 pound of $BeCl_2$, yielding a current efficiency of 59.7%. Because of the vapor losses, this current efficiency is only approximate. A more accurate means of determining current efficiency is planned in the future by determining the chlorine con-content in the exit gases.

Analysis of Product

A sample of the quasi-amalgam obtained was exposed to the air and the black powder which separated was collected. Part of the powder was calcined, and both calcined and uncalcined samples were analyzed spectrographically with the results shown in Table IV.

TABLE IV
Analysis of Oxidized Powder from Quasi-Amalgam

(Values in Ppm)

	Oxidized (Black Powder)	Calcined Product
Al	2500	1300
Ag	200	250
B	4	13
Ca	140	240
Cd	4	6
Co	60	120
Cr	700	1500
Cu	180	450
Fe	6000	15000
Mg	20	60
Mn	280	660
Mo	300	700
Na	800	300
Ni	1200	3000
Si	50	150
Zn	90	170

Since most metals have lower decomposition potentials than beryllium, the bulk of the impurities in the electrolyte were electrolyzed in the initial electrolysis. The quasi-amalgam sample contained mainly beryllium from the first electrolytic run and was expected to be high in impurities. Subsequent X-ray diffraction and chemical analysis revealed that the black powder was actually about 90% metallic beryllium, and the remainder BeO. Comparison of the black powder with a nickel powder of known particle size at approximately 500 X has indicated, tentatively, that the black powder has a particle size of about 1 micron.

FUTURE WORK

It is believed that all the equipment is operable and that future runs will optimize efficiency of electrolysis and purity of product.

It is planned to produce three products; black powder, concentrated quasi-amalgam from which a considerable amount of the mercury has been removed by filtration and pressure compaction, and beryllium metal powder produced by distillation away of residual mercury from the concentrated amalgam.

Hot pressing and metallurgical evaluation will be conducted on the first two products. By supplying small quantitites of the black powder and of the beryllium metal powder to selected laboratories, which are investigating solid propellant fuels, we hope to determine the potential of these products as rocket fuel additives.

ACKNOWLEDGEMENTS

The help of Mr. W. G. Lidman, Technical Director, General Astrometals Corp., Dr. B. Kopelman, Consultant, and Dr. F. L. Holderreed, Director of Metallurgy, Anaconda Company, is gratefully acknowledged.

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APPENDIX

OPERATION OF THE UNITS

Due to the toxicity of $BeCl_2$ and of mercury vapor, great care must always be taken to confine any vapors to the hooded and ventilated dry-box areas. Any doubt about the condition of the air demands wearing respiratory equipment. Any contamination of the working area is cleaned immediately.

Hands should be thoroughly covered with talcum powder before putting on dny box gloves to allow easy removal if required. Sharp objects, heat, and rough edges are avoided when working in the dry boxes since the gloves rupture easily. Ruptured gloves should be changed immediately to prevent contamination of the laboratory.

Special clothing is used in the plant and showers are taken before leaving the work area to prevent outside contamination. Gloves and protective aprons are worn. In the case of suspected air contamination, or if working inside the pot furnace hood or cell enclosure, respirators or gas masks are worn. Respirators remove only fine dust particles from the air, while the gas mask will remove chlorine and mercury vapors.

The plant area and adjacent rooms are constantly tested for mercury vapor by a mercury vapor detector and for beryllium content by a suction type air sampler. The maximum level of permissible beryllium concentration in the air, 2 μg/cu. m., was rarely exceeded. However, ventilation and handling practices are constantly studied for further improvement.

A change room adjacent to the plant room contains lockers, sink, toilet, and shower facilities.

PURIFICATION OF $BeCl_2$ BY SUBLIMATION

Cleaning and Charging. Before beginning sublimation, the connecting pipes in the unit, the hot condenser, and the charge crucible are all cleaned. The connecting pipes are cleaned by removing the end caps and inserting a pipe or cleaning tool. Care must be taken to be sure that all the sublimate is removed from the walls of the pipes. The hot condenser is cleaned by removing the crucible from the bottom, and inserting a spatula inside the condenser. The sulfuric acid and caustic soda solutions in the gas train should be checked. When the solutions appear turbid, they should be discarded and fresh solutions added.

Before filling the charge crucible, any scale and lumps should be removed. Occasionally the charge crucible should be washed with dilute hydrochloric acid to remove any adherent scale. About 5 lb of impure $BeCl_2$ is charged in the crucible, the crucible clamped to the furnace lid, and the pot furnace is raised around the crucible.

Sublimation. The flow of water to the cold condenser and the operation of the heating tapes should first be checked. The temperature controller is set at a predetermined temperature, and the hot condenser heaters turned on. While the hot condenser is warming up, the pot furnace control is set to 25%, and the nitrogen flowing into the furnace adjusted to 1.68 lb/hr on the flowmeter scale.

When the temperature of the hot condenser reaches 325-375°C, the pot furnace power and the nitrogen flow into the furnace are increased. When the pot furnace temperature is between 225°C and 300°C plugging of the pipes with sublimate may occur. This is observed by an increased pressure drop across the sublimation unit. If the plugging cannot be relieved by momen-

tarily reducing the nitrogen gas flow, the pipes must be opened and cleaned without interrupting the operation.

Sublimation of the bulk of $BeCl_2$ occurs when the pot furnace temperature reaches 475-480°C. When sublimation starts, the pot furnace power should be reduced so that the $BeCl_2$ will not sublime too rapidly; otherwise the product will be lumpy. During sublimation, the pot furnace temperature will remain constant. When it begins to fall, this is an indication that the sublimation is practically complete. The pot furnace power and condenser heaters may then be turned off, and the nitrogen flow reduced.

By using asbestos gloves, the crucible containing purified $BeCl_2$ can be unclamped from the hot condenser and any dense lumps of $BeCl_2$ removed. The purified sublimate, which should be fibrous, is transferred to a storage beaker, which is closed tightly and removed from the dry box. If the sublimate is to be charged directly to the cell, the charging cone (see Electrolysis Section) is attached to the crucible, clamped tightly and removed from the dry box. The dense lumps which were collected cannot easily be charged to the cell, so they should be resublimed.

Starting with a cold unit, a complete purification takes about 2 hours; but if the unit is hot, the operation can be completed within 1 1/2 hours. Two complete purification cycles including cleaning the equipment and transferring the product can be completed in one eight-hour shift.

ELECTROLYSIS

Charging the Cell. The cell is charged with an arrangement illustrated in Fig. 12. The charging cone is placed on a crucible full of $BeCl_2$ which has been crushed fine and is in a protective atmosphere; the cone and crucible arrangement is attached to the cell charging pipe. The charging pipe is then

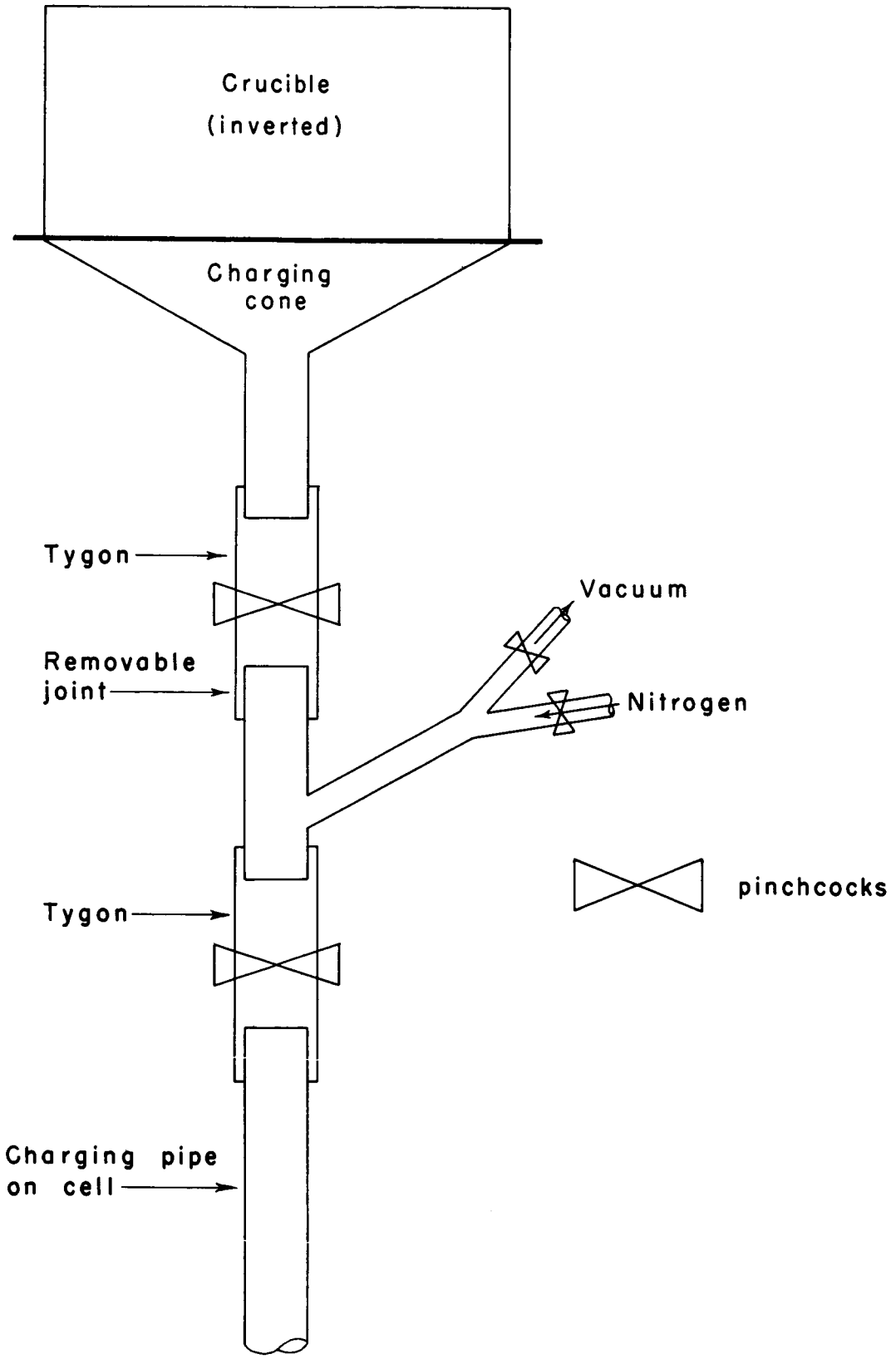

Fig. 12— Charging Arrangement

evacuated and backfilled with nitrogen to remove any air. The pinchcocks are opened and the $BeCl_2$ is charged to the cell. When all the $BeCl_2$ is in the cell, the pinchcocks are closed and the crucible removed.

The cell is initially charged with a 5-in. layer of mercury (approximately 550 lb) and a 12-in. layer of electrolyte of eutectic composition (55.5 lb $BeCl_2$, 40 lb NaCl). The initial heating takes about 6 hrs and must be attended closely to prevent any rapid temperature rise. The salt mixture will begin fusing when the salt temperature reaches 225°C, and fusion proceeds rapidly at 250°C. To avoid excessive vaporization, the mercury temperature should not exceed 250° C during the initial heating.

Before starting electrolysis, the $BeCl_2$ content in the cell should be increased to 60 lb, to prevent electrolyte freezing due to $BeCl_2$ depletion during electrolysis. Since the eutectic salt composition is 50 mol % (58 wt %) $BeCl_2$, the melting point of an electrolyte containing 60 wt% $BeCl_2$ will decrease during electrolysis until the eutectic composition is reached.

Operating the Cell. Before electrolysis, the salt concentration should be in the range of 58-60 wt % $BeCl_2$. Samples should be taken before and after electrolysis to control this concentration. The rectifier can supply a maximum of 250 amps, 9 volts; higher amperage will overload the rectifier. The voltage is adjusted with the variable transformer on the control panel, but the amperage should initially be adjusted by changing the anode to cathode distance. This can be done by attaching the overhead pulley to the anode bus clamp and placing Transite shims under the bus clamp to secure the anode. The stirrers should be turned on while the amperage is adjusted.

During electrolysis, the salt temperature should be above 275°C, but no higher than 330°C to prevent mercury vapors and fogging. The top heaters

on the cell are adjusted as required to avoid a temperature rise during electrolysis. Since the anode to cathode distance is not changed during electrolysis, the amperage can be held constant by varying the voltage. Cathode stirring is essential to successful electrolysis.

When electrolysis has proceeded for the desired length of time, the rectifier is shut off, the heaters adjusted, and the temperature idled at about 250°C between electrolytic runs. This avoids undue mercury evaporation. If electrolysis is allowed to proceed for a long period of time, greater than 24 hours, the amalgam accumulated in the cathode will become pasty and tapping will be difficult. Once heated, the cell should never be allowed to cool unless circumstances require a complete shutdown.

Collecting the Amalgam. The amalgam should be removed from the cell after electrolysis. The heater tape on the mercury inlet line should be turned on, the water flow through the exit cooler checked, and the mercury holding tank should be full before tapping the cell.

The inlet mercury valve can be opened slowly, and the flow adjusted by visual observation of the exiting amalgam flow through the Tygon line. If the cell is tapped slowly, the amalgam will likely drain on the filter screens. Tapping should continue until the exiting mercury appears to be beryllium free. If more mercury is needed in the holding tank to finish tapping, it can be pumped up from the filter tank.

When tapping is completed, the cooler can be flushed by admitting mercury through the bypass vaive. To further concentrate the collected amalgam, or to relieve a buildup of mercury on the screens, the vacuum pump is switched on and vacuum is applied at the filter tank. The mercury inlet line, the vent line, and the mercury outlet line at the filter should

all be clamped to isolate the vacuum from the rest of the circuit. When vacuum is applied, nitrogen should be flowing slowly into the filter above the screens.

Vacuum in the filter tank below the collecting screens will be evident due to collapsing of the Tygon level-tube. When any of the Tygon lines which enter the filter tank above the screens begin to collapse, it is an indication that the "amalgam cake" on the screens has cracked and filtration is practically complete. The vacuum line should be closed and nitrogen allowed to flow into the filter until the pressure equalizes with the remainder of the system. To empty the filter, unscrew the single bolt clamp, remove the lid, lift out the screen cup, place it on the dry box shelf, and transfer the amalgam to an airtight container.

To prevent screen damage, only a plastic spoon should be used for transferring the amalgam. After the screen cup is replaced and the filter closed, all the mercury can be pumped to the holding tank. Any time mercury is flowing in the system, all vent lines should be open.

Control Analysis. Samples of electrolyte taken before and after each electrolytic run were analyzed as follows:

1. Weigh about 1 gm in a tared, covered weighing bottle.

2. Transfer the sample to a beaker and add 50 ml of a previously prepared alcohol solution (80% secondary butyl alcohol and 20% ethyl alcohol).

3. Boil slowly for 3-4 hr, intermittently crushing the sample with a stirring rod. When the sample appears white and granular, the $BeCl_2$ is completely dissolved.

4. Using a vacuum flask, filter the sample through a tared sintered-glass filter. Wash with the alcohol solution.

5. Dry the sintered-glass filter containing the residue at 110°C to constant weight and record the weight. The residue is NaCl.