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M-356(Book II)

OPERATING INSTRUCTIONS -- PROCESS FOR PRODUCTION OF CRYSTALLINE BORON PRODUCT 891A

By

R. D. Anderson
W. M. Bowes
E. L. Carpenter
G. I. Cathers
J. K. Dixon
K. M. Milton
B. C. Rowley
R. C. Ryder
M. Sonnino
R. T. Swain

July 1946

Work performed under Contract No. W-7401-Eng-91

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ACTUAL MS PG. GALLEY NO.

TYPIST

SAME SIZE TIS Form 138 (Rev. Sep. 1953)

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2 INVESTIGATION NO. R. U. 232.
PROBLEM NO. 64.

M-356
(Book II)
~~PAGE~~ cy. 1

Step 4

Notes: (1) References to Drawings 396-19-0 and 386-19-0 interchangeable. (Dwg. 386-19-0 usually intended.)

(2) Tube manometers and valves referred to Dwg. 392-19-0 are shown in Photograph 6.

[July 1946]

Step 4

Chemistry of Process

General Description

Pure Chlorthane received from the distillation after Step 3 is mixed with an excess of pure hydrogen in the ratio of one mole to seventeen, and the resulting mixture is allowed to react at the surface of an electrically heated tantalum-tungsten (TAW) wire heated to 1260°C. The reaction process taking place liberates hydrochloric acid gas and deposits Product 891 as a coating on the surface of the heated wire.

$$2 \text{ Chlorthane} + 3 H_2 \rightleftharpoons 2 \text{ 891} + 6 HCl$$

The metallic Product 891 so produced is then submitted for the Separation and Grinding process to be described in the next section.

The wire is maintained carefully at a temperature of 1260°C. since at lower temperatures the Product 891 tends to deposit upon the surface of the wire as a brown, amorphous product. It will be recalled that the specification for 891 calls for a crystalline product and if the temperature is maintained close to 1260°C., the brown, amorphous form is avoided entirely. If the wire temperature falls below 800°C., the reaction does not proceed at all. Although the effect of wire temperatures has not been studied thoroughly above 1300°C., there was indication that the higher temperatures tended to cause the Product 891 to adhere comparatively strongly to the wire, thus making a clear-cut separation of the two somewhat difficult. If the separation is not clean-cut, the purity of the Product 891 is adversely affected.

A control of hydrogen to Chlorthane ratio is important because it has an effect on the percentage conversion to 891 of the Chlorthane per pass over the wire and it affects the ease of separation of the Product 891 from the wire and, finally, the percentage recovery of the unconverted Chlorthane (see section on Variations for Step 4).

Hydrogen employed in the reaction must be carefully purified to remove oxygen because otherwise this gas would form water on the hot wire which would, in turn, react with the Chlorthane to form boric oxide. The copper catalyst effectively removes traces of oxygen and the water thus formed is removed in the Lectro Drier. Not only would boric oxide formation lower yields, but this oxide would also be deposited on the walls of the glass tube and the optical pyrometer readings of wire temperature would be in error.

The catalyst used to remove traces of oxygen in the hydrogen was one consisting of a reduced copper oxide deposited on an activated alumina carrier. The catalyst is operated at 300°C. during this period. The original catalyst prepared, operated satisfactorily over two years, producing about 230-240 kg. of finished Product 891.

a) Main Reaction

$$2 \text{ Chlorthane} + 3 H_2 \rightleftharpoons 2\ 891 + 6\ HCl$$

This reaction occurs when Chlorthane in the presence of an excess of hydrogen (say 17/1) is led over a hot wire (Ta). The product, 891, is deposited on the wire.

The wire temperature should be about 1260°C. Lower temperatures tend to cause the formation of a brown, amorphous product, and at temperatures below about 800°C. the reaction does not proceed at all. At temperatures much higher than 1260° (1300+), the wire and product tend to adhere more and more closely together.

The ratio of hydrogen to Chlorthane is very important in relation to the percentage yields, the ease of product and wire separation, and the percentage recovery. (See section on Variations in Step 4.)

b) Side Reactions

Reaction with Oxygen

The removal of all oxygen from the incoming gases is important, since in the presence of the heated wire, boric oxide is formed if oxygen is present. This side reaction lowers yield and causes the glass tube to become cloudy, which results in an incorrect temperature reading.

The oxygen is removed from the hydrogen by passing it over a catalyst at 300°C. The catalyst is a reduced copper oxide deposited on an alumina carrier. The cuprous oxide (or copper) takes on the oxygen to form cupric oxide. The cupric oxide is then reduced back again to cuprous oxide and metallic copper by the hydrogen with the formation of water.

$$2\,Cu + O_2 \longrightarrow 2\,CuO$$

$$2\,Cu_2O + O_2 \longrightarrow 4\,CuO$$

$$2\,CuO + H_2 \longrightarrow Cu_2O + H_2O$$

$$Cu_2O + H_2 \longrightarrow 2\,Cu + H_2O$$

The water is thoroughly removed by passing the gas through a Lectrodryer (activated alumina system).

The oxygen is removed from the Chlorthane tank by an extensive flushing period previous to the start of the run.

Reaction with Water

Any water that comes in contact with Chlorthane results in the formation of boric oxide.

Reactions forming Hydrides

Besides the main reaction of forming 891, Chlorthane and hydrogen also react to give various hydrides. A few of the more important are:

B_2H_6 - Colorless gas; results by heating of B_4H_{10}. F. P. = -165.5°C.; B. P. = -92.5°C. Very sensitive against water but otherwise is stable.

B_4H_{10} - Colorless liquid; F. P. = -120°C.; B. P. = +18°C.; decomposes quickly into H_2 and a great number of other boron hydrides, among them B_2H_6 and B_5H_9.

B_5H_9 - Colorless liquid; results by heating B_4H_{10}; F. P. = -47°C.; quite stable; reacts slowly with water.

Reactions of TAW Wire

The TAW wire in the course of the "hydriding" period and the run loses its elasticity and its tensile strength. In the process, some soluble form of the T and W are formed; probably either a hydride or a boride. The only basis for this assumption is that a test of T is obtained in the analysis of the 891. As pure T will not dissolve in the reagent used in the analysis, the T must be present as a compound. No knowledge of the exact nature of this compound is available.

Film Deposit Next to Wire

The first deposit which is laid down on the wire may be of various compositions. It is usually a brown, white, or gray (ordinary 891) layer. The reason for the formation of these various deposits is not known.

The brown deposit is an indication that handpicking will be difficult - many attachments of 891 and T.

The white layer has so far always been an indication of very good handpicking - wire does not chip and crumble and form attachments.

The absence of either a brown or white layer results in poor handpicking also - wire brittle and forms attachments.

A high hydrogen ratio (approximately 15/1) and the use of TAW wire are the conditions which will most likely result in easy handpicking.

Table XV

Properties of Materials

	Source	B.p., °C.	Density	Viscosity (micropoises)
Hydrogen	Electrolytic			
Chlorthane	Step 3			93 at 20°C.
Liquid nitrogen	Air Reduction Co.	-195.8	1.43 0°/4	
HCl	-			
Boron hydrides	-			

Table XVI

Time Analysis for Operations in Step 4

A.	Setting up 7 tubes		
	1.	Cleaning tubes, trays, and rods	1.5 hrs.
	2.	Bending and straightening wire	0.2
	3.	Actual setting up	2.0
		Total	3.7 hrs.
B.	Actual time of reaction		8.0 hrs.
C.	Other duties carried out with (A) and (B)		
	1.	Cooling condensing system	0.3 hr.
	2.	Filling precondenser with dry ice and trichlorethylene	1.0
	3.	Cooling kettle	0.5
	4.	Venting system with N_2 and H_2	0.2
	5.	Testing for leaks and quality of mixture	0.1
	6.	Venting with N_2 after a run	0.1
	7.	Weighing and filling CT tanks	0.5
	8.	Regeneration of Lectrodryer	5.0
	9.	Draining recovered CT	0.7

Equipment

The equipment used for the conversion of Chlorthane to 891 is located at the east end of the building. The unit is shown diagrammatically in Flow Sheet, Dwg 386-19-0. There are three fundamental parts to the system: (1) the gas purification and feed lines; (2) reaction tubes; and (3) the condenser unit. Nearly all the controls, except those for the electric current used to heat the wires in the reaction tubes, are located on a panel board in the center of the room. Drawing 394-19-0 shows the arrangement of the instruments on this board.

The hydrogen purification system (Dwg 386-19-0) consists of a heated column containing a reduced copper oxide catalyst, a heat exchanger and a Lectrodryer. After passing through these the gas is metered into the reaction tubes through a Rotameter. A Mercoid Pressuretral, set for 20 lbs. gage, is connected by high pressure tubing to the manifold in the hydrogen truck. If the pressure in the manifold falls below 20 lbs., the alarm light on the panel board flashes on. A Bristol controller maintains the temperature of the catalyst in the purifier at 300°C.

The Chlorthane bombs are heated to maintain a constant pressure, and this gas also is metered into the reaction tubes through a rotameter. A Mercoid Pressuretral controls the immersion heater in the Chlorthane tank. Since the line to the Mercoid is beyond the valves which shut off the tanks, care must be taken to open all lines from the Chlorthane storage tank to the Mercoid before heat is applied to the former.

One of the two condensing units is shown in detail in Dwg. 386-14-0. The other, differing from the one shown only in that the freezer is in a vertical rather than horizontal position, normally is kept as a spare. There are two parallel vents through Hg lutes to relieve pressure in the system if either the condensing or freezing unit plugs and the pressure in the system builds up to 45 cm. of Hg. Also, there are two warning horns connected to the system, one of which sounds if the pressure reaches 3 cm. and the other if it reaches 12 cm. of Hg. A Micromax controller and recorder connected to a thermocouple in the liquid nitrogen pipe just before the condenser controls the flow of liquid nitrogen to the freezer and the condenser. Details of this system are shown in Dwg. 399-19-0, p.666.

Dwg. 392-19-0 and Photograph 8, pp. 651 and 625 show the details of the reaction tubes. Temperatures of the wire are read with a Leeds and Northrup optical pyrometer. No corrections are made to the scale reading on this instrument for the emissivity of the wire, or for other such factors.

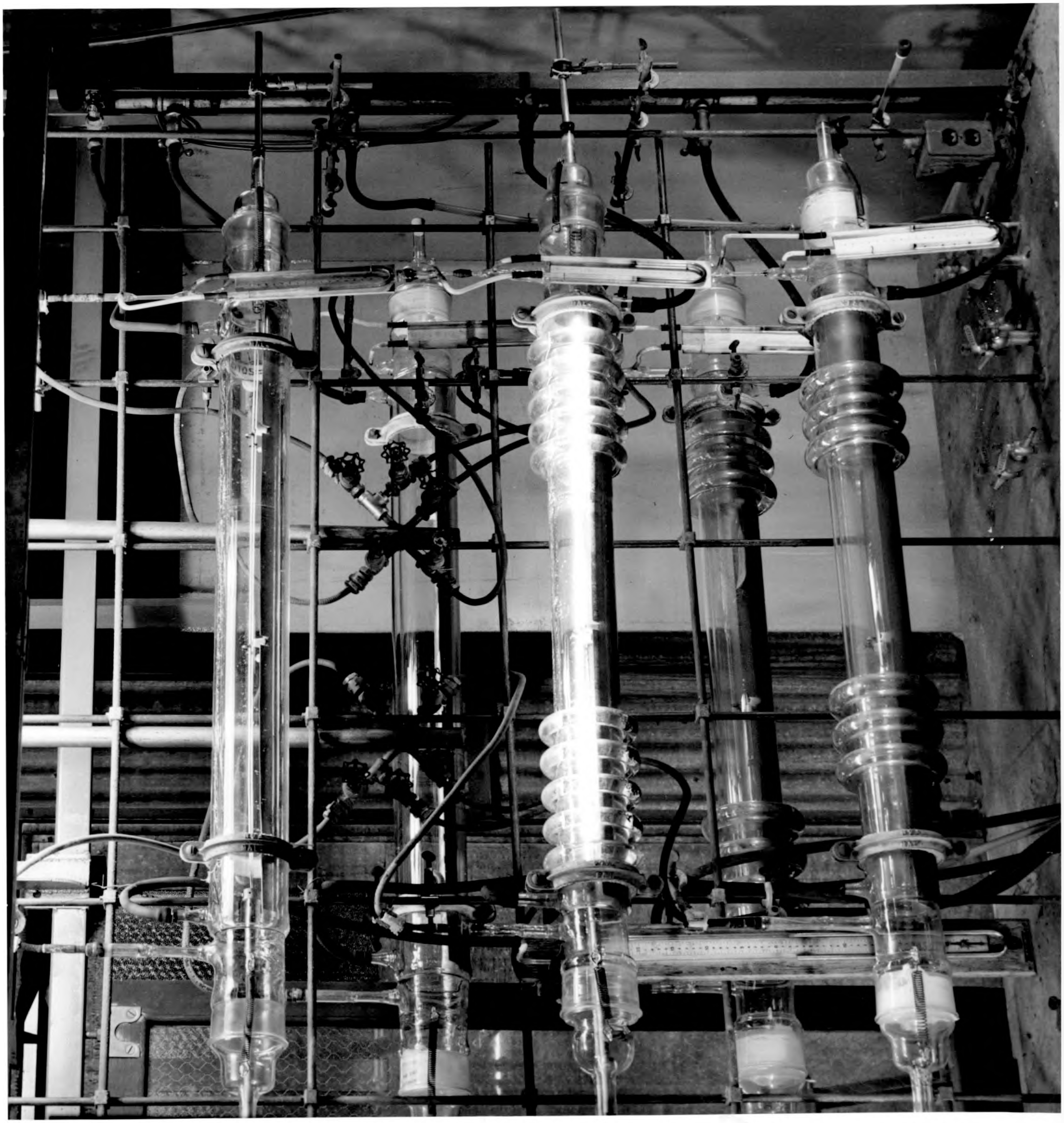


Photo #6

Operational Procedure

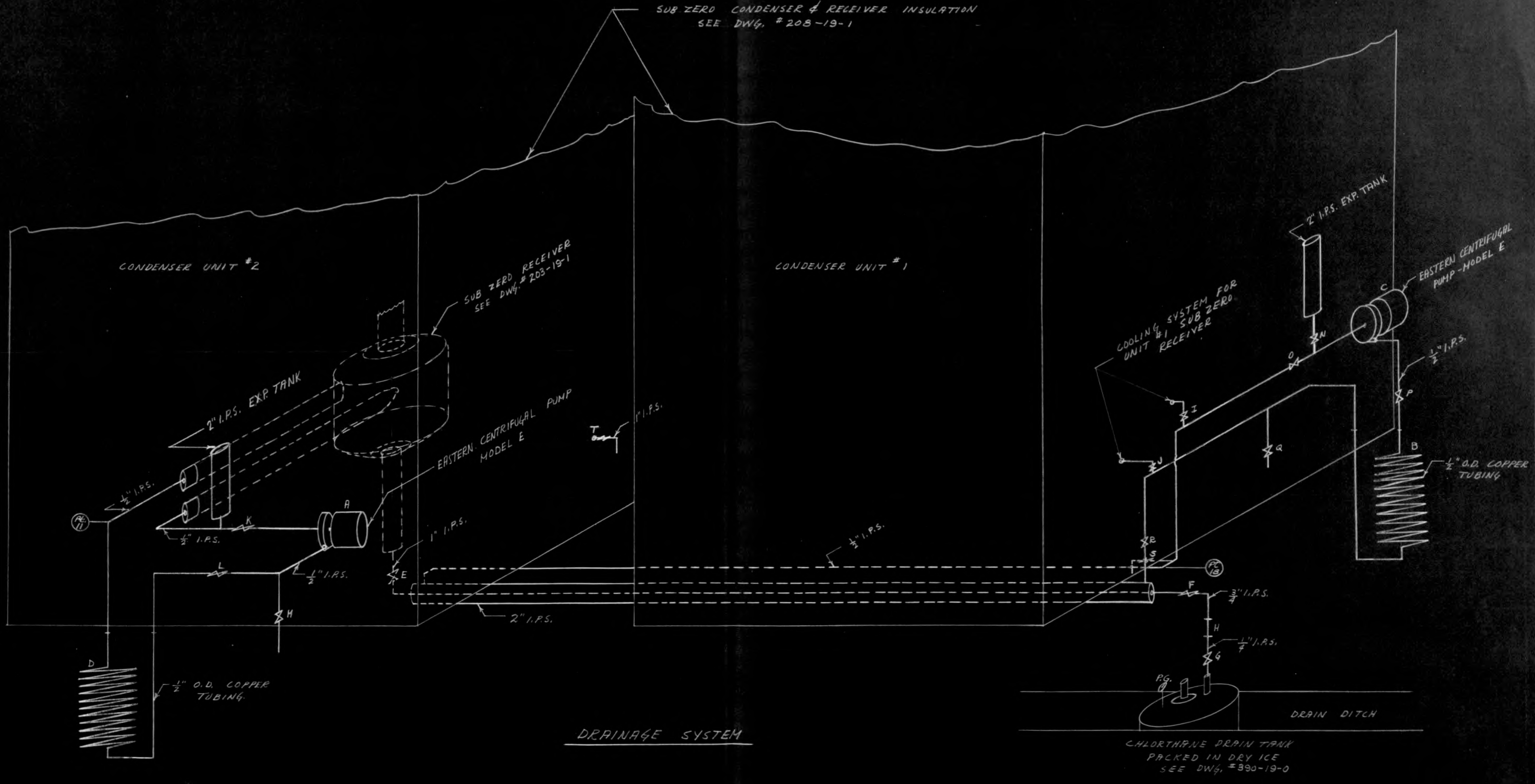
1. Draining Kettle (Dwg. 398-19-0; photograph 7).

Assuming a run has been made the previous day, which is the usual case, the first step in the day's operation is to drain the kettle at the bottom of the sub-zero condenser. The HCl has been distilling off all night through the low Hg lute (4.5 cm.). The pump on the circulating system for the kettle (Point A in Dwg. 398-19-0, p.628) has been running all night with trichlorethylene and dry ice around the coil. About 6:30 A. M. the watchman has instructions to remove the cooling flask and allow the pump to run with just air around the coil. About 7:45 A. M. the temperature of the kettle (Point 10 on the 18 point potentiometer) will usually be around -70°C. (Valves K, L open and valve M shut, Dwg. 398-19-0).

The coil for the drain-line cooling system (Point B, Dwg. 398-19-0) is placed in a Dewar flask containing a mixture of trichlorethylene and dry ice and the circulating pump (C, Dwg. 398-19-0) started. (Valves N, O, P and R should be open and valves Q, S, I and J should be closed - Dwg. 398-19-0.) After weighing, the drain bomb is connected to the end of the drain line (this operation is usually performed the preceding day at the close of the run to save time). A bucket of dry ice is ground up and the ice placed around the drain bomb. Temperature Point 10 should have warmed up to about -55°C. by this time; if not, steam should be used to heat the coil on the kettle's circulating system (D, Dwg. 398-19-0).

When Point 10 reaches -55°C., the temperature of the drain line (Point 18 [selector switch on 6]) is about -65°C. or at least 3 degrees colder than the kettle, and the drain bomb is extremely cold (usually a vacuum of about 26 inches of Hg is created on cooling). The kettle is ready to drain into the bomb.

Photo #7



DRAINAGE SYSTEM

REVISION	REFER. DWG.
	203-19-1
	208-19-1
	386-19-0
	390-19-0
	396-19-0

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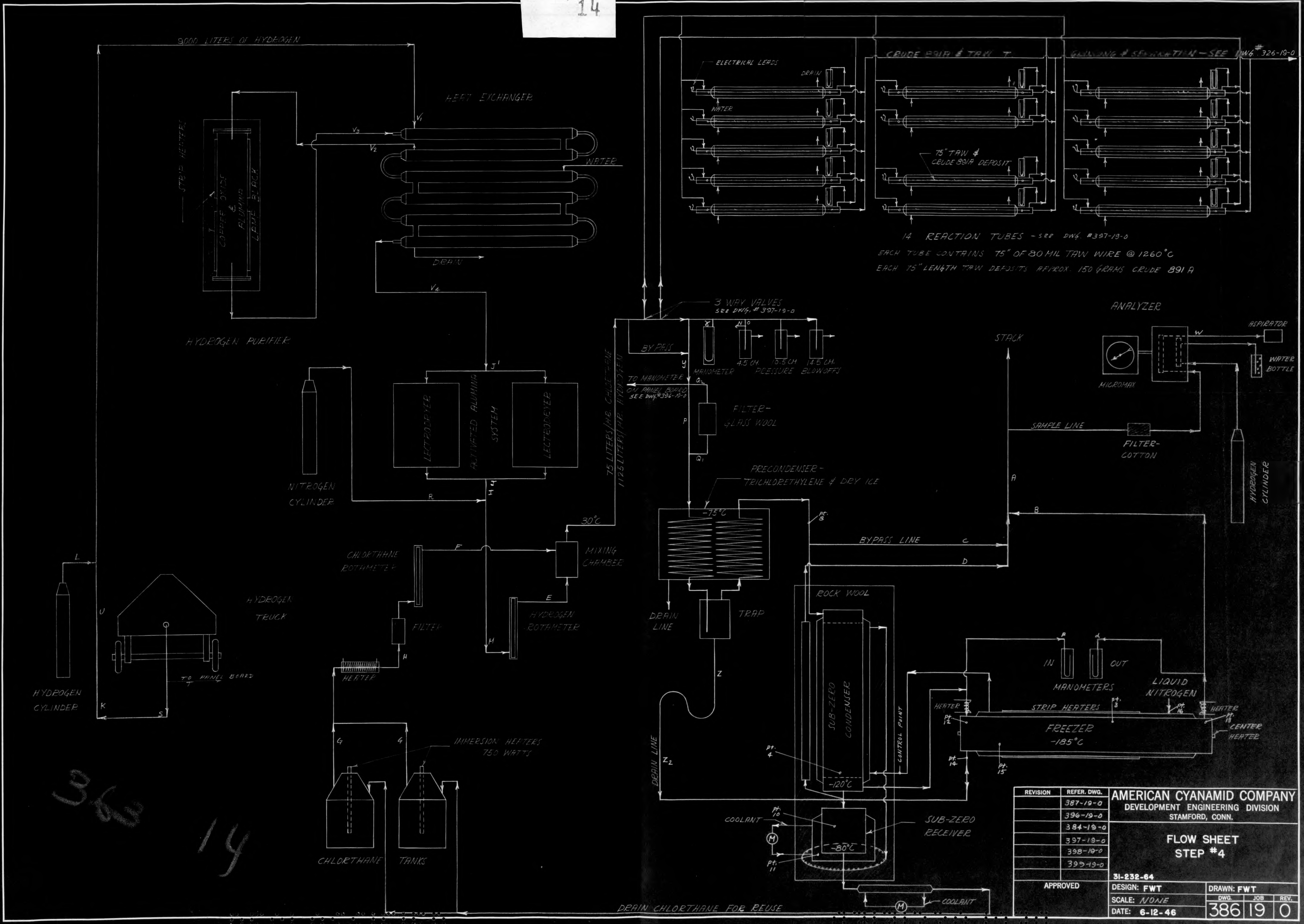
STAMFORD, CONN.

PIPING - SHEET # 3
STEP # 4

31-232-64

DESIGN: FWT	DRAWN: FWT
SCALE: NONE	
DATE: 6-24-46	

DWG.	JOB	REV.
398	19	0



REVISION	REFER. DWG.
	387-19-0
	396-19-0
	384-19-0
	397-19-0
	398-19-0
	399-19-0

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DEVELOPMENT ENGINEERING DIVISION
STAMFORD, CONN.

FLOW SHEET
STEP #4

31-232-64

DESIGN: FWT
DRAWN: FWT
SCALE: NONE
DATE: 6-12-46

DWG.	JOB	REV.
386	19	0

363

14

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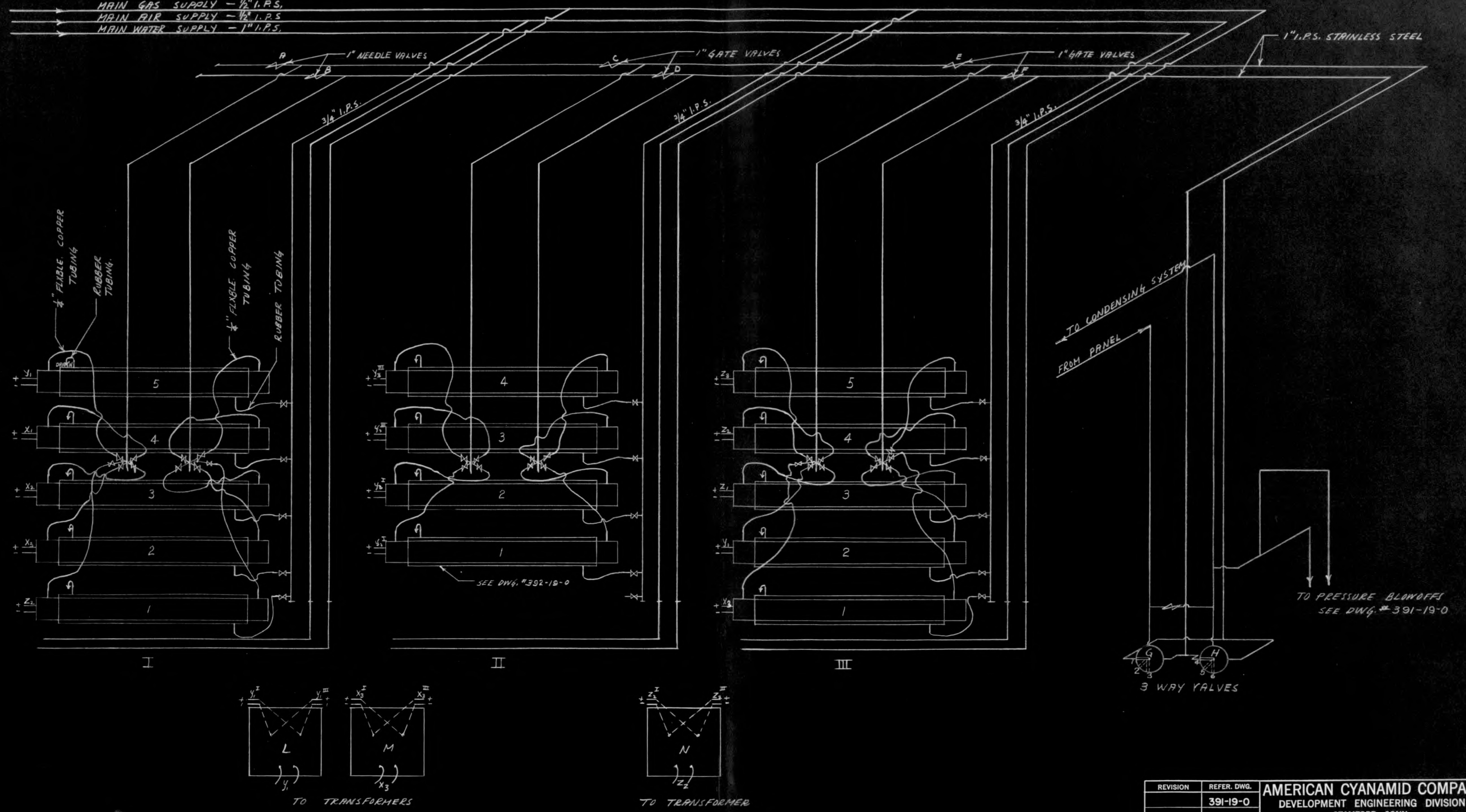
After shutting off Pump A the valve just below the kettle is opened first (E), then the valve on the end of the drain line (F), and lastly the valve on the drain bomb (G). Any leaks in the system may be detected more easily by opening the valves in this order. The kettle is allowed to drain until no more liquid Chlorthane is in the drain pipe. This may taken any time from 5 to 30 minutes depending on the amount and the ease of draining. A simple test to be used to determine whether any liquid remains in the drain pipe is as follows: Wipe off the frost from the 1/4 inch horizontal pipe at the end of the drain line (H, Dwg. 398-19-0). If the pipe frosts up again, liquid is still present in the pipe. If no more frost forms, and/or the pipe begins to thaw, the liquid is all in the bomb and the drainage is complete. Valve E (Dwg. 398-19-0) is closed, the Dewar flask removed from the drain coil (B, Dwg. 398-19-0), and a bucket of hot water placed around the coil instead. When Point 18 reaches 40°C., the pump (C, Dwg. 398-19-0) is stopped and Valve F closed. A protective covering of rags is laid over the drain bomb and then the line from Valve G to Valve F (Dwg. 398-19-0) is steamed. When the pipe reaches a temperature of about 50°-60°C. (pipe hot to touch), Valve G may be closed. The drain bomb is then disconnected from the line, freed of all ice, and then weighed. The difference between the initial and final weights is the amount of Chlorthane recovered from the preceding day's run.

2. <u>Flushing and Cooling Systems</u> (Photograph 8 , p.632).

While the kettle is draining it is possible to flush the large glass reaction tubes holding the hot wires for reduction. This permits the cooling of the condensing system to be started as soon as drainage is complete. Valve B, Dwg. 397-19-0, should be opened. Valve A is always kept closed. If over five tubes are being run, Valves E and F will always be open. If the tubes on racks I and II were run the preceding day, Valves C and D will be open. If the tubes on racks II and III were



36 16

REACTION PIPING & ELECTRICAL SYSTEMS

REVISION	REFER. DWG.
	391-19-0
	392-19-0
	396-19-0
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STAMFORD, CONN.

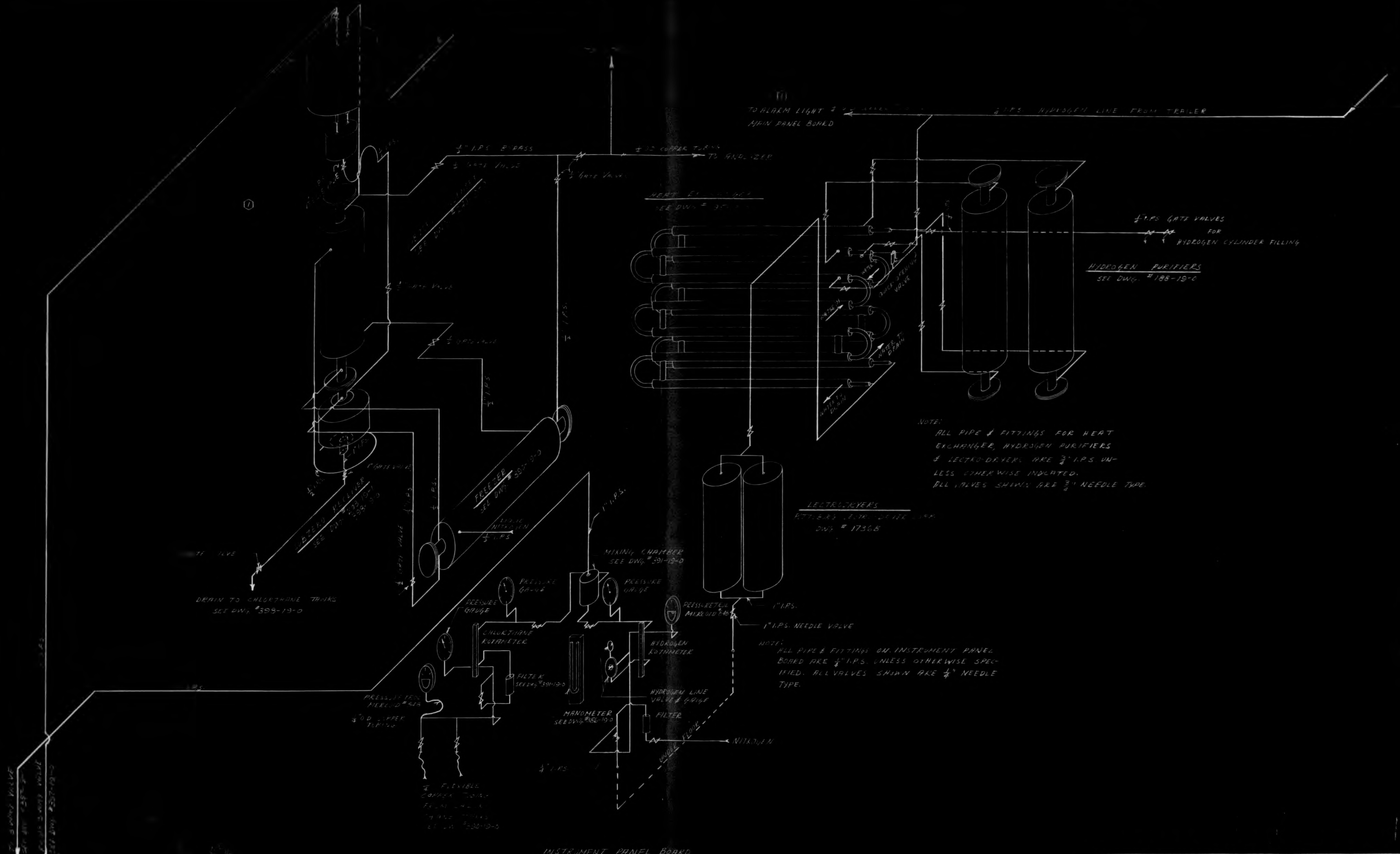
PIPING-SHEET #2
STEP #4

31-232-64

DESIGN: FWT
DRAWN: FWT
SCALE: NONE
DATE: 6-24-46

DWG.	JOB	REV.
397	19	0

Foto #8363-17



NOTE:
ALL PIPE & FITTINGS FOR HEAT EXCHANGER, HYDROGEN PURIFIERS & LECTRO-DRYERS ARE ¾" I.P.S. UNLESS OTHERWISE INDICATED.
ALL VALVES SHOWN ARE ¾" NEEDLE TYPE.

NOTE:
ALL PIPE & FITTINGS ON INSTRUMENT PANEL BOARD ARE ¼" I.P.S. UNLESS OTHERWISE SPECIFIED. ALL VALVES SHOWN ARE ¼" NEEDLE TYPE.

NOTE:
ALL CHLORTHANE & CHLORTHANE-HYDROGEN MIXTURE LINES ARE 18-8 S.S. TYPE 3[illegible]

REVISION	REFER. DWG.
① 12-18-46	185-19-0 188-19-0
	203-19-1 240-19-0
	386-19-0 387-19-0
	390-19-0 391-19-0
	394-19-0 397-19-0
	398-19-0 399-19-0

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DEVELOPMENT ENGINEERING DIVISION
STAMFORD, CONN.

PIPING - STEP #4

31-232-64
DESIGN: FWT
DRAWN: FWT
SCALE: NONE
DATE: 6-27-46
DWG. 396 JOB 19 REV. 1

8

used the preceding day, then the 1/4 inch valves (inlet and outlet) leading to Tubes 1, 2, 3, 4 and 5 on rack I and Tubes 1 and 2 on rack II must be opened for a 7 tube run. If racks I and II were used the preceding day, the valves leading to tubes 1, 2, 3, 4 and 5 on rack III and tubes 3 and 4 on rack II should be opened. The 3-way valve G should be put in the number 1 position. The nitrogen tank behind the panel and valve R (Dwg. 396-19-0) are opened, putting a nitrogen pressure on the system up to 1/4 inch control valve E (Dwg. 396-19-0). (The line valve M is set at the correct pressure - 15 lbs. and left open from day to day.) Valve E (Dwg. 396-19-0) is opened to give a flow of about 50 on the hydrogen rotameter. The nitrogen then flows from the panel, through 3-way valve G (Dwg. 397-19-0), through line ECA (Dwg. 397-19-0), through the tubes, and through line FDB, and out to the atmosphere. The tubes should be flushed with nitrogen about 5 minutes.

A hydrogen truck (Dwg. 395-19-0, p. 650) has 22 cylinders arranged in four banks - 7, 6, 5 and 4 cylinders per bank from bottom to top, respectively. The valves for the individual cylinders are kept open as well as the line valve for the bank which is in use. In the morning the main valve is opened and the pressure regulator S (Dwg. 396-19-0) set for 25 lbs. Valves V_1, V_2, V_3 and V_4 (Dwg. 396-19-0) are always left open; and valves T, K and U (Dwg. 396-19-0) are always open except when hydrogen truck is being changed. Then T, K and U are closed and valve L (Dwg. 396-19-0) leading to the auxiliary hydrogen cylinders is opened; otherwise valve L is kept closed. The 4-way valves JJ' (Dwg. 396-19-0) are opened to the correct side of the Lectrodryer. (Each side of the dryer is used for three full runs and then the other side is run while the first side is being reactivated. Reactivation instructions are contained in booklet on Lectrodryer. See Photograph 9.) The hydrogen pressure of 25 lbs. is now up to valve I (Dwg. 396-19-0).

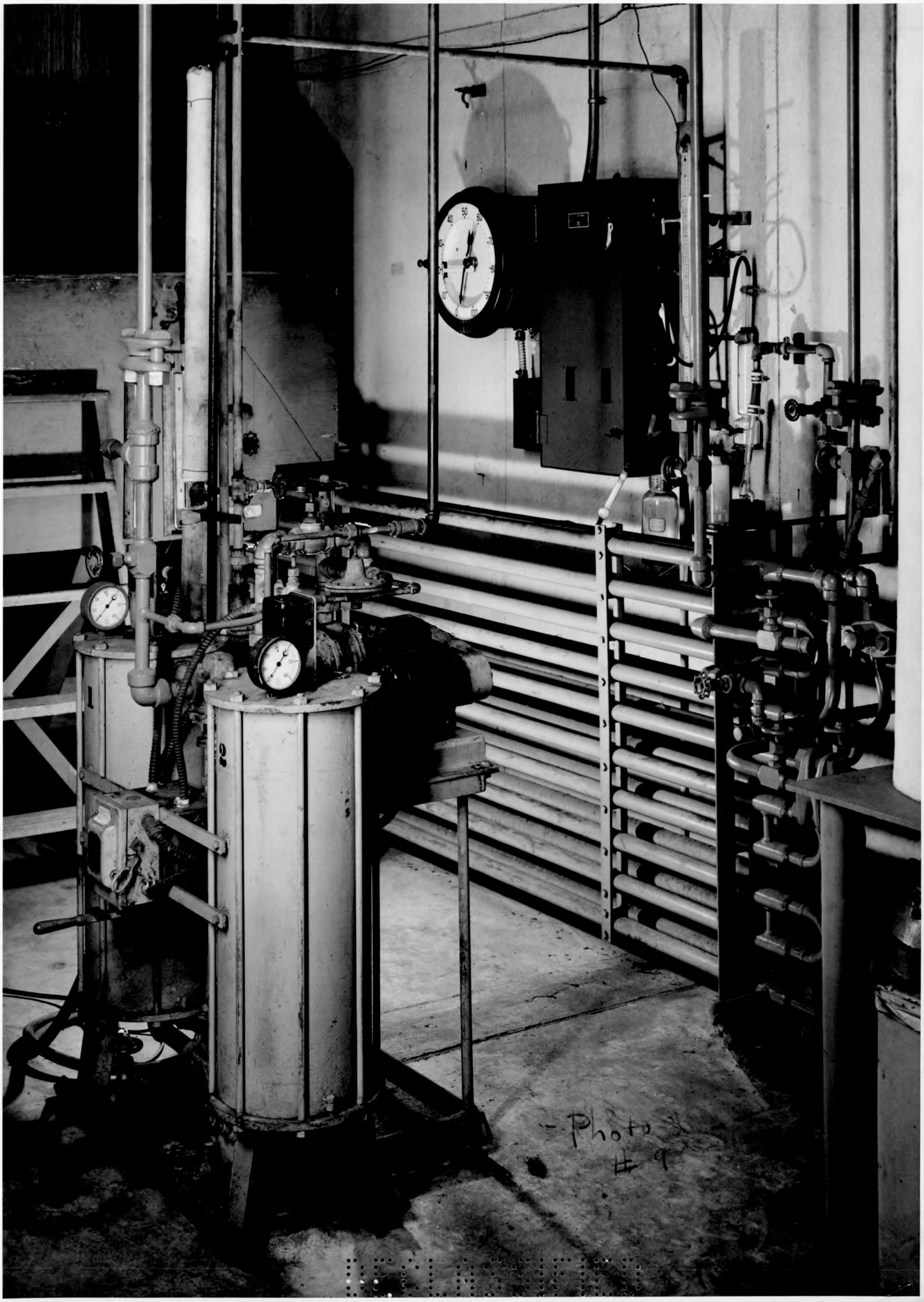

Photo #9

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363-20

The nitrogen flushing is stopped by shutting valve R (Dwg. 396-19-0) and the nitrogen tank. Hydrogen is now allowed to flush tubes by opening valve I (Dwg. 396-19-0) for a maximum of 10 minutes and a minimum of 5 minutes at a setting of 50 on the hydrogen rotameter. A flow of 100 bubbles per minute is put through the standard hydrogen side of the analyzer (Photograph 9; Dwg. 396-19-0) with a pressure of 5 lbs. on low side of Multistage. When the flushing period is over, the control valve E (Dwg. 396-19-0) is closed, and valve B (Dwg. 397-19-0) is closed. If racks I and II are to be used, then the system is ready. If racks II and III are to be used, then valves C and D (Dwg. 397-19-0) must be closed. The Bristol controlling #1 hydrogen purifier should be set at 300°C. and its powerstat (Dwg. 394-19-0) set at 60.

As soon as the drain is completed and valve E (Dwg. 398-19-0) is closed, the cooling of the system may be started. The liquid nitrogen can is placed on its stand next to freezer; the delivery tube D (Dwg. 399-19-0) placed in can; connection made to the freezer E (Dwg. 399-19-0), and to the gaseous nitrogen supply F (Dwg. 399-19-0). The nitrogen cylinder is turned on and a pressure of about 15 lbs. put on the low pressure side of the Multi-stage Gage. Valve C (Dwg. 399-19-0) is open all the way and valve B (Dwg. 399-19-0) is partially open. A pressure of 15 lbs. is now on the nitrogen system from the gage up to the ASCO 3-way valve (Dwg. 399-19-0). The stopper A (Dwg. 399-19-0) is removed from the pipe on the top of the condenser jacket. After making sure the stopper G (Dwg. 399-19-0) is in place, the Micromax Recorder and Controller (Dwg. 394-19-0) is turned on. This is accomplished by throwing the solenoid switch (Dwg. 394-19-0) and the two switches on top of the Micromax. A nitrogen flow of about 45 is set on the rotameter (Dwg. 399-19-0) by using valve B (Dwg. 399-19-0). The rate of approach on anticipatory control (Micromax Electronic Control) (Dwg. 399-19-0) is set at 100. (Inside the instrument, the settings for the throttling range (3), the rate of droop correction (6) and the impulse rate (4) are approximately correct for the prevailing conditions and will not have to be moved.) If at this point the line manometer on the panel (Dwg. 394-19-0) shows a difference (either pressure or vacuum) of over 3 cm. of Hg, then the three-way valve G (Dwg. 397-19-0) should be placed in the number 2 position and the bypass valve just above (Dwg. 396-19-0) should be opened. If there is a large pressure on the system, the liquid N_2 feeding into the freezer will overcome this quickly. If there is a large vacuum on the system, then H_2 should be fed into the system by opening valve E (Dwg. 396-19-0). When the difference reaches a value under 3 cm., valves G and H (Dwg. 397-19-0) may be put in positions 1 and 4, respectively and the by-pass valve closed. If after turning on the liquid nitrogen, the manometer on the panel reads under 3 cm. difference, then valve G (Dwg. 397-19-0) may be left in the number 1 position, valve H (Dwg. 397-19-0) put in the number 4 position (from number 5) and the by-pass valve left closed. H_2 is metered into the condensing system (through the tubes) as the system cools using valve E (Dwg. 396-19-0) to control the flow, and keeping a small vacuum (1-2 cm. Hg) on the system as read on the open-end Hg manometer.

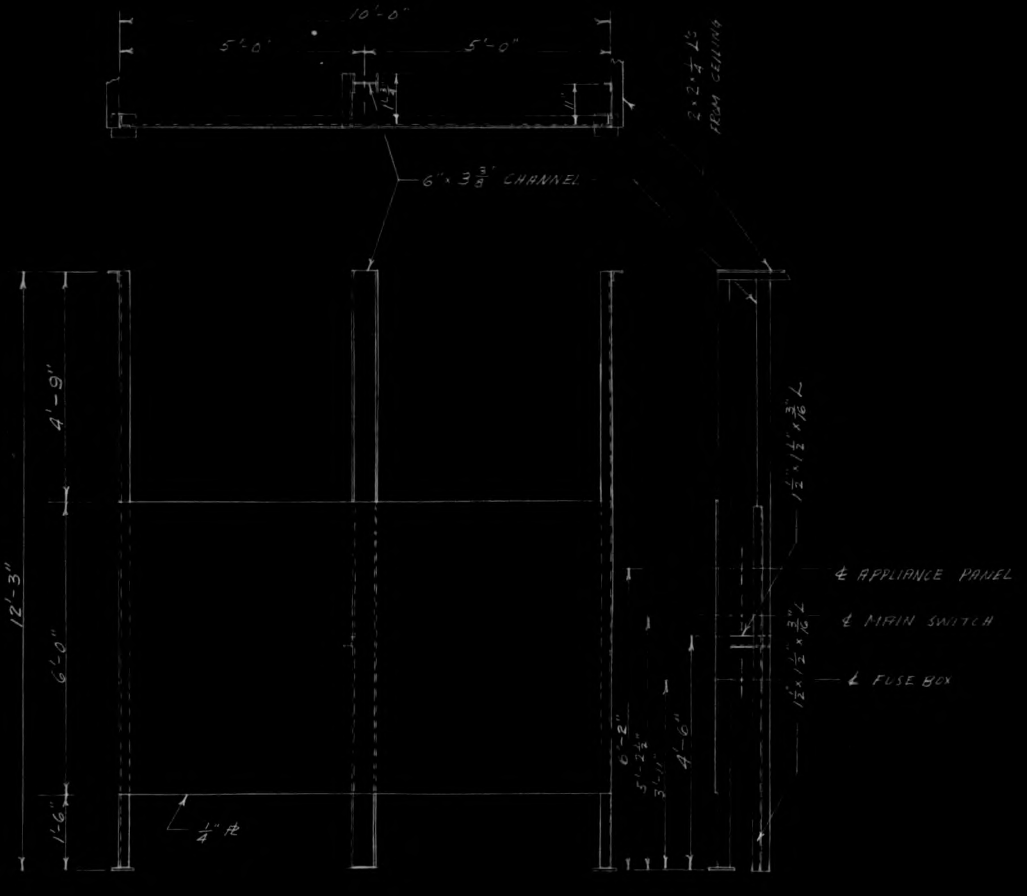
When point 3 reaches -180°C., a small positive pressure is put on the system; and with a small H_2 flow on the rotameter, the main vent valve A (Dwg. 396-19-0) and the freezer outlet valve B (Dwg. 396-19-0) are opened. (Make sure valves C and D, Dwg. 396-19-0, are closed, and drain line valves Z_1 and Z_2 open). The H_2 rotameter is set at the correct reading for the number of tubes in run, and the pressure gage above rotameter set at 15 lbs. by adjusting line valve M (Dwg. 396-19-0). When point 4 reaches -90°C., the condensing system is ready to run. The analyzer, Photograph 9, is turned on right after the system is opened to the atmosphere. This is done by opening stopcock W (Dwg. 396- 19-0), turning on the aspirator, and flipping on the switches on the Micromax.

While the condensing system is being cooled a number of things may be accomplished and, at any rate, the following things must be done before the run may be started: The switch for the gas alarm (Dwg. 394-19-0) should be turned on. The switch for the H_2 Pressuretrol (Dwg. 394-19-0) should be turned on. A weighed CT tank should be attached to the flexible Cu line behind the panel (Photograph 10). After making sure valve H (Dwg. 396-19-0) is closed, the valve on the tank and valve G (Dwg. 396-19-0) on the same line are opened. The tank heater is plugged to the appropriate Variac and the tank heater switch (Dwg. 394-19-0) turned on. The hot plate beneath the Chlorthane tank should be plugged in and its Variac set at about 60. The Chlorthane lines switch should be turned on and its Variac set at about 30. The Bristol controlling the freezer exit (Point 13) should be turned on and set for -80°C. (Photograph 8). The switch for the freezer exit heater should be turned on and its Variac set at 60. If racks I and II had been run the previous day, the electrical leads Y_1^I must be exchanged for leads Y_1^{III} in box L (Dwg. 397-19-0); the leads X_3^I exchanged for leads X_3^{III} in box M (Dwg. 397-19-0); and leads Z_2^I exchanged for leads Z_2^{III} in box N (Dwg. 397-19-0). On rack II, leads Y_3 changed from tube 2 to tube 4 and Y_2 from tube 1 to tube 3. If racks II and III had been run the previous day, the above process would be reversed. The cooling water for the tubes to be run that day should be turned on. The rate of flow should be set so that while the tube is in operation, the water temperature on the outlet side is less than 50°C. After point 3 has reached -180°C., the rate of approach dial on the anticipatory control (Dwg. 394-19-0) should be turned to 50, the flow in the N_2 rotameter (Dwg. 399-19-0) controlling the liquid nitrogen set at 30, stopper A (Dwg. 399-19-0) put in pipe, the trap (Dwg. 386-19-0) opened by opening valves Q_1 and Q_2

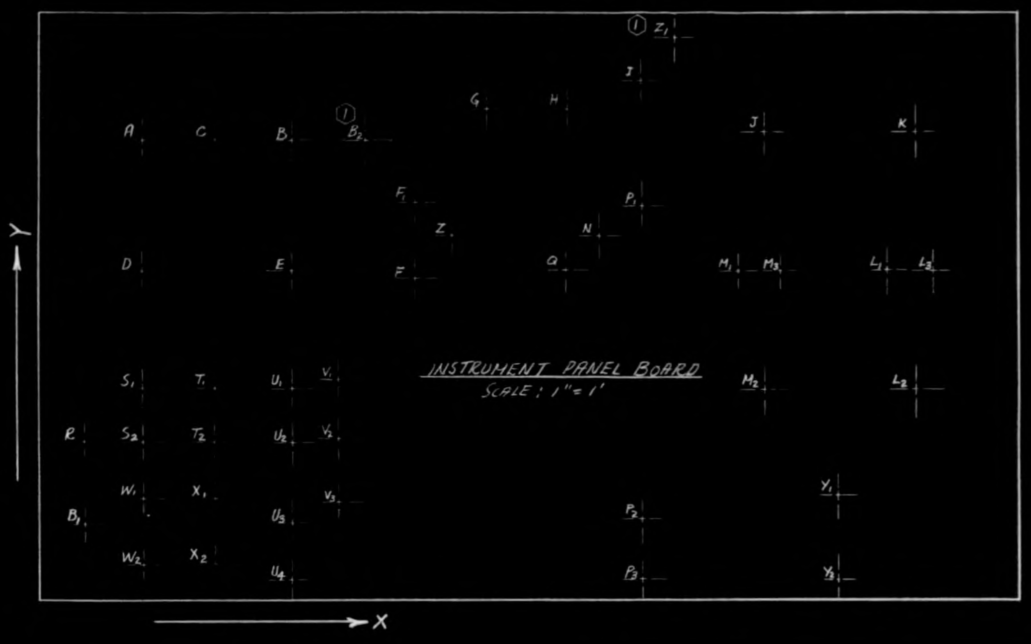
and closing valve P (Dwg. 396-19-0), the Dewar flask containing trichlorethylene and dry ice put around coil D (Dwg. 398-19-0) and the kettle circulating pump A (Dwg. 398-19-0) started. A sample of the gas passing through the tubes must be taken and tested to determine whether or not an explosive mixture is present. After making sure valve O (Dwg. 396-19-0) is open, valve N (Dwg. 396-19-0) is opened and valve Y (Dwg. 396-19-0) closed down until a fair amount of gas is flowing through the rubber tube fastened on beyond valve N. The escaping gas is allowed to displace the air from a large inverted test tube. When the gas has displaced all the air, the test tube is stoppered, valve Y opened, and valve N shut. The gas sample is then ignited. If the hydrogen burns normally, the system is free of air and the run may proceed; but if the sample explodes, the tubes must be flushed longer until a sample of gas gives a negative test.

3. Making a Run

After checking the ends of the tubes for leaks with the MSA Explosimeter, and inspecting the tube manometers (Dwg. 392-19-0) to make sure each tube has a flow of gas through it, the run may be begun. If racks I and II are to be run, go to the rear of the electrical panel board (Dwg. 393-19-0) and throw the switches on both the 100 amp. and the 32 amp. circuit breakers (in that order) for X_1, X_2, X_3, Y_1, Y_2, Y_3 and Z_2. If racks II and III are to be run, the circuit breakers for X_3, Y_1, Y_2, Y_3, Z_1, Z_2 and Z_3 will be thrown. (Assuming a 7 tube run in each case.) Go around to the front of the panel board and turn the seven Powerstats quickly to give a reading of 100 amps. (See Photograph 11.) Now start the Chlorthane flow by opening valves F and H (Dwg. 396-19-0). Two operators are needed during these operations. Valve H is the main control valve for the Chlorthane flow. Valve F is used to maintain a pressure of 1 lb. on the gage above the Chlorthane rotameter (Dwg. 394-19-0). The starting time of the run is



INSTRUMENT PANEL BOARD [illegible]
SCALE: ½"=1'



INSTRUMENT PANEL BOARD
SCALE: 1"=1'

DESIGNATION	DESCRIPTION	CO-ORDINATES X *	Y *	REFERENCE
A	FREEZER INLET CONTROLLER	1'-½"	4'-8½"	DWG. #386-19-0, Pt. #12, BRISTOL #E-486
B	FREEZER OUTLET CONTROLLER	2'-7"	4'-8½"	DWG. #386-19-0, Pt. #13, BRISTOL #E-486
B₁	FREEZER OUTLET HEATER RELAY	5½"	9½"	
C	SIXTEEN POINT SELECTOR SWITCH	1'-9½"	4'-8½"	BROWN INST. CO.
D	EIGHTEEN POINT INDICATOR	1'-½"	3'-4½"	LEEDS & NORTHRUP CO.
E	MICROMAX CONTROLLER & RECORDER	2'-7"	3'-4½"	LEEDS & NORTHRUP CO.
F	TANK HEATER PRESSURETROL	3'-10"	3'-3½"	MERCOID #DA-231
F₁	TANK HEATER PRESSURE GAUGE	3'-10"	4'-¾"	
G	PRESSURE GAUGE - CHLORTHANE ROTAMETER	4'-7"	5'-¼"	DWG. # 386-19-0
H	PRESSURE GAUGE - HYDROGEN ROTAMETER	5'-5"	5'-¼"	DWG. # 386-19-0
I	PRESSURE GAUGE - HYDROGEN TRUCK	6'-2"	5'-3¾"	DWG. # 395-19-0
J	① FREEZER-HEATER CONTROLLER	7'-5"	4'-8½"	DWG. #386-19-0
K	#1 HYDROGEN PURIFIER CONTROLLER	8'-11½"	4'-8½"	DWG. #386-19-0, BRISTOL #E-486
L₁	#1 HYDROGEN PURIFIER RELAY	8'-8"	3'-4½"	
L₂	#1 HYDROGEN PURIFIER POWERSTAT	8'-11½"	2'-2"	SUPERIOR ELECTRIC CO.
L₃	#1 HYDROGEN PURIFIER SWITCH	9'-1½"	3'-4½"	
M₁	① FREEZER-HEATER RELAY	7'-1¾"	3'-4½"	
M₂	① FREEZER-HEATER POWERSTAT	7'-5"	2'-2"	SUPERIOR ELECTRIC CO.
M₃	① FREEZER-HEATER SWITCH	7'-7"	3'-4½"	
N	HYDROGEN ROTAMETER	5'-8¾"	3'-9"	FISCHER-PORTER #D3-1133
P₁	HYDROGEN PRESSURETROL	6'-2"	4'-¼"	MERCOID #DA-221
P₂	LOW PRESSURE HYDROGEN RELAY	6'-2"	10"	
P₃	LOW PRESSURE HYDROGEN SWITCH	6'-2"	2½"	
Q	HYDROGEN LINE VALVE	5'-5"	3'-4½"	
R	FREEZER INLET HEATER RELAY	5½"	1'-7½"	
S₁	FREEZER INLET HEATER POWERSTAT	1'-½"	2'-2"	VARIAC - TYPE 200-CM
S₂	FREEZER INLET HEATER SWITCH	1'-½"	1'-7½"	
T₁	FREEZER OUTLET HEATER POWERSTAT	1'-9½"	2'-2"	VARIAC - TYPE 200-CM
T₂	FREEZER OUTLET HEATER SWITCH	1'-9½"	1'-7½"	
U₁	CHLORTHANE TANK HEATER POWERSTAT	2'-7"	2'-2"	VARIAC - TYPE 200-CM
U₂	CHLORTHANE TANK HEATER SWITCH	2'-7"	1'-7½"	
U₃	CHLORTHANE TANK HEATER RELAY	2'-7"	9½"	
U₄	CHLORTHANE TANK HEATER PILOT LIGHT	2'-7"	2½"	
V₁	MICROMAX SOLENOID SWITCH	3'-¾"	2'-3"	
V₂	MICROMAX SOLENOID RELAY	3'-¾"	1'-8"	H-B TYPE MP-1-M
V₃	MICROMAX SOLENOID PILOT LIGHT	3'-¾"	1'-0"	
W₁	CENTER FREEZER HEATER POWERSTAT	1'-½"	1'-½"	SUPERIOR ELECTRIC CO. TYPE 116
W₂	CENTER FREEZER HEATER SWITCH	1'-½"	4½"	
X₁	CHLORTHANE LINE HEATER POWERSTAT	1'-9½"	1'-½"	VARIAC - TYPE 200-CM
X₂	CHLORTHANE LINE HEATER SWITCH	1'-9½"	4½"	
Y₁	GAS ALARM	8'-2"	1'-1"	MINE SAFETY APP CO. - TYPE I2-376
Y₂	GAS ALARM SWITCH	8'-2"	2½"	
Z	CHLORTHANE ROTAMETER	4'-2¾"	3'-9"	FISCHER-PORTER #D5-1147
① B₂	ANTICIPATORY CONTROL	3'-4"	4'-8½"	
① Z₁	ALARM LIGHT	6'-6"	5'-9"	

LEGEND - INSTRUMENT PANEL BOARD

① NOTES:

1. CO-ORDINATES X & Y REFER TO CENTER LINES OF INSTRUMENTS & LOCATE THEM.
2. DWG. 394-19-0 ERRONEOUSLY REFERS TO PTS. J, M_1, M_2, M_3 AS HYDROGEN PURIFIER #2 ACCESSORIES.

REVISION	REFER. DWG.
① 12-19-46	386-19-0
	395-19-0
	387-19-0
	396-19-0

APPROVED

AMERICAN CYANAMID COMPANY
DEVELOPMENT ENGINEERING DIVISION
STAMFORD, CONN.

INSTRUMENT PANEL BOARD
STEP #4

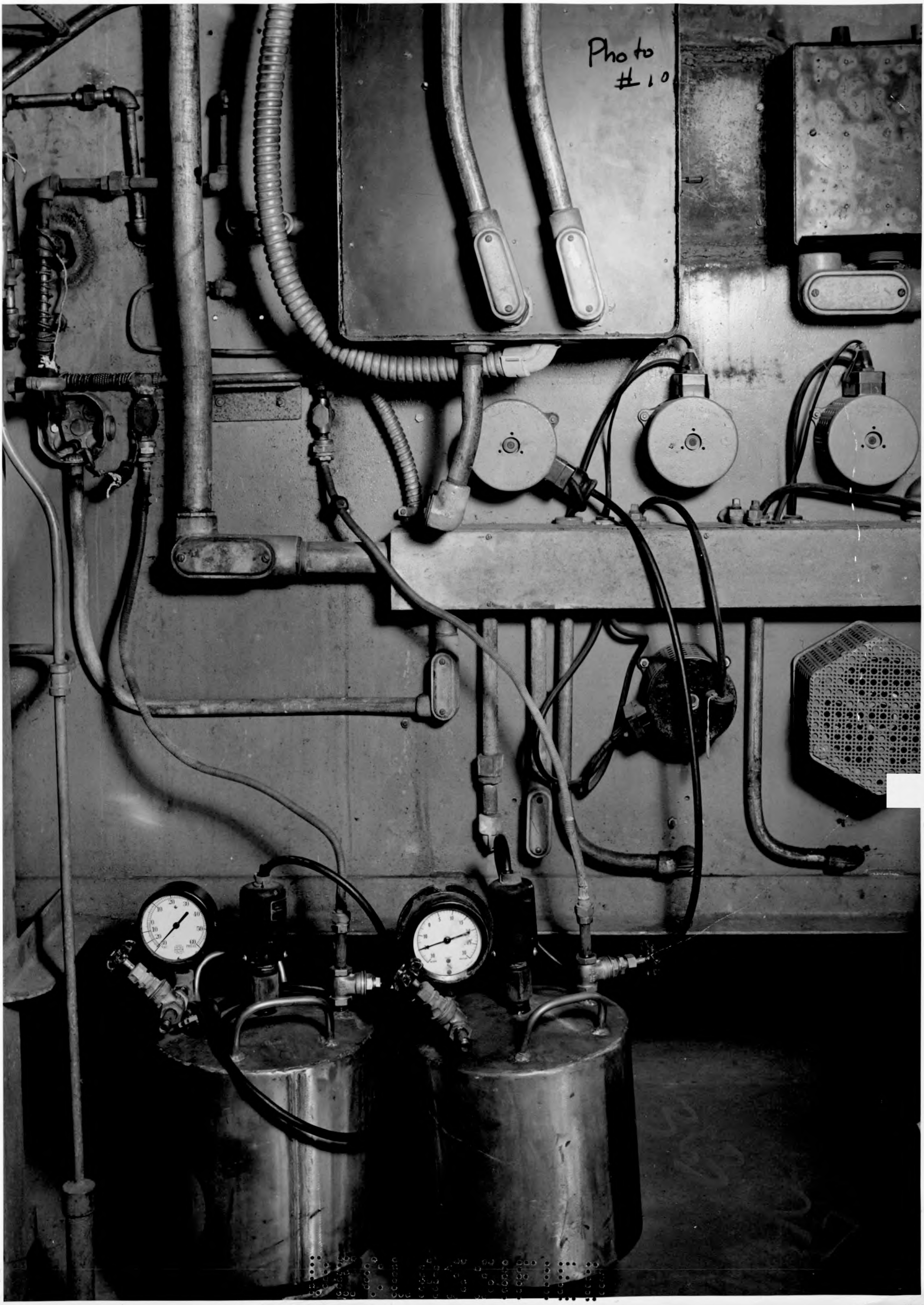
31-232-64

DESIGN: FWT
DRAWN: FWT
SCALE: AS SHOWN
DATE: 6/19/46

DWG.	JOB	REV.
394	19	1

noted on the clock when the float in the Chlorthane rotameter reaches the correct value, i.e., 8.9 for seven tubes. As soon as the Chlorthane reaches the wire, a noticeable cooling effect on the wire will be noticed. When this "cold spot" has covered the entire wire, the Powerstats are turned up again until the wires reach a temperature of about 1260°C. (a reading of about 125 amperes). A Leeds and Northrup optical pyrometer #8622C is used to determine the temperature of the wires. Complete directions for operating this instrument are to be found in the booklet attached to the pyrometer. An inspection of the manometers on the tubes (Dwg. 392-19-0) is made to determine whether or not the rate of flow through each tube is the same. If not, the flows are made equal by partially closing one of the 1/4 inch gate valves (Photograph 6) leading to the individual tubes. (This applies to each tube in which the rate is higher than the others.)

The mean temperatures of the wires are kept at about 1260°C. by increasing the voltage on the tubes by means of the Powerstats (Dwg. 393-19-0). The section of the wire on the inlet (gas) side of the tube will tend to grow colder than the other end. When the difference in temperature between the ends of the wires becomes 60-70°C., the direction of flow is reversed by quickly changing the positions of the three-way valves G and H (Dwg. 397-19-0) from 1 to 3 and from 4 to 6 (or vice versa), respectively, without opening the by-pass valve (Dwg. 396-19-0). At all times the wire temperature at any point should be above 1200°C. and not over 1300°C. After each reversal of flow, the tube manometers should be checked and any deviations corrected. This flow reversal will have to be performed quite often at the beginning of the run, but toward the end, once every 1 or 2 hours will be found to be sufficient. Readings of the wire temperatures (both left and right sides) should be recorded each



363-27

hour as well as the voltage and amperage for each tube on a mimeographed data sheet. Also the tubes should be constantly watched for signs of overheating and deformities of the wire which might cause an electrical short or cause the wire to touch the side of the glass tube; and if there appears to be any real danger of the tube breaking, it should be shut down. If the hairpin end of the wire curves upward during the first two or three hours of the run to a point where it might become dangerous, it may be caused to return to a horizontal position (usually) by lowering the temperature of the wire to about 900°C. for about 5 minutes and then raising the temperature back to normal. Toward the end of the run, the above treatment will not work, and the only alternative is to shut down the tube for that particular wire. If for some reason (short circuit, broken tube, etc.) a tube has to be shut down, the power for that tube is turned off first by throwing the switches on the circuit breakers behind the electrical panel board (Dwg. 393-19-0). The H_2 and the Chlorthane are left flowing through the rest of the tubes, but both the 1/4 inch valves (Dwg. 392-19-0) leading to that tube are closed. The rates of flow for the hydrogen and the Chlorthane are adjusted to meet the new conditions by turning down on valves E and F (Dwg. 396-19-0). The water for that tube may be turned off.

As soon as the drain bomb has been weighed, it may be placed on a hot plate and connected to the Chlorthane line behind the main panel board (Photograph 19. (It also may be placed in dry ice chest until ready for use.) One must make sure the nipple and half union are perfectly dry before connecting the bomb to the copper tubing. At this time, the bomb may have a vacuum or even a slight pressure. At any rate, when the bomb starts to warm to room temperature, a pressure will develop and be indicated on the gage attached to the bomb. When the pressure is in excess of six lbs., the valve on the bomb may be completely opened and then the valve G (Dwg. 396-19-0) on the same line cracked. (At this time the run has been going at least an hour.) The bomb is allowed to vent slowly until it reaches approximately room temperature, at which time the other Chlorthane bomb may be turned off and the valve to the drain bomb opened wide. The tank heater cords are switched and the hot plate under the other bomb unplugged. It may be calculated quite closely from the weight of Chlorthane in a bomb and from the rotameter calibrations just how long the Chlorthane in a bomb will last. All bombs (except the recovery bomb in which 2-3 lbs. are left) were used to a weight down to within a half pound of the tare.

The following table was made from the calibration curves of hydrogen (Fig. 1) and of Chlorthane (Fig. 2).

Table XVII

Calibration Chart

(Chlorthane rate 75 l./hr. - H_2 ratio 15/1
at 1 lb. gage pressure)

No of Tubes Operating	Setting of H_2 Rotameter	Setting of Chlorthane Rotameter	lbs. CT/hr.
7	130.5	8.9	6.02
6	112.4	7.5	5.16
5	93.6	6.3	4.30
4	75.0	4.77	3.44
3	56.0	3.45	2.58
2	37.1	2.15	1.72
1	18.5	0.91	0.86

If during the run the pressure on a bank in the H_2 truck falls below 20 lbs., the alarm light will come on. (Drawing 394-19-1 , p. 639). One must then shut the valve on the empty bank, and then quickly open the valve on another bank (Dwg. 395-19-0). If the truck goes empty or a new truck arrives, it will be necessary to shift to the auxiliary H_2

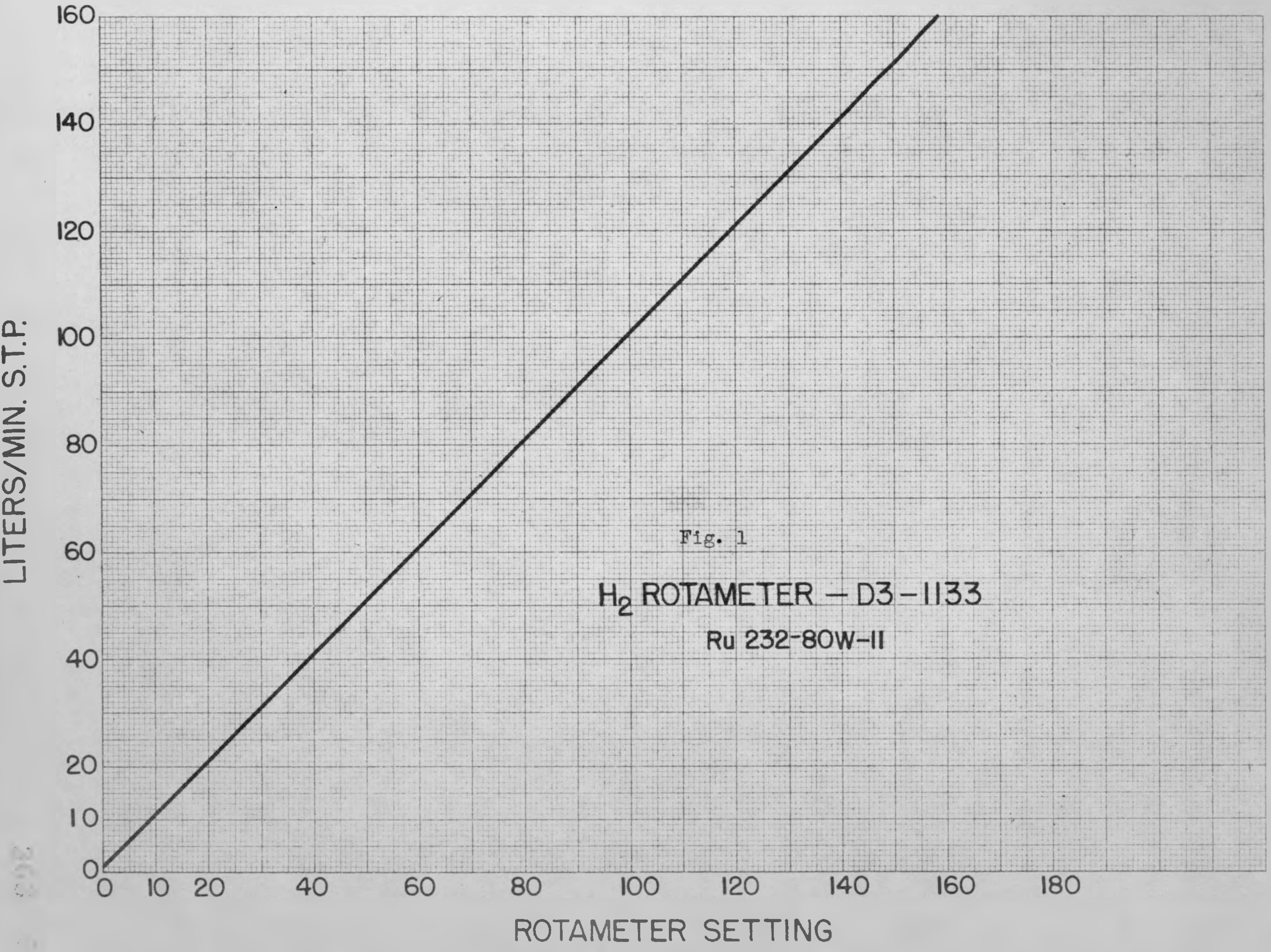


Fig. 1

H$_2$ ROTAMETER — D3-1133

Ru 232-80W-11

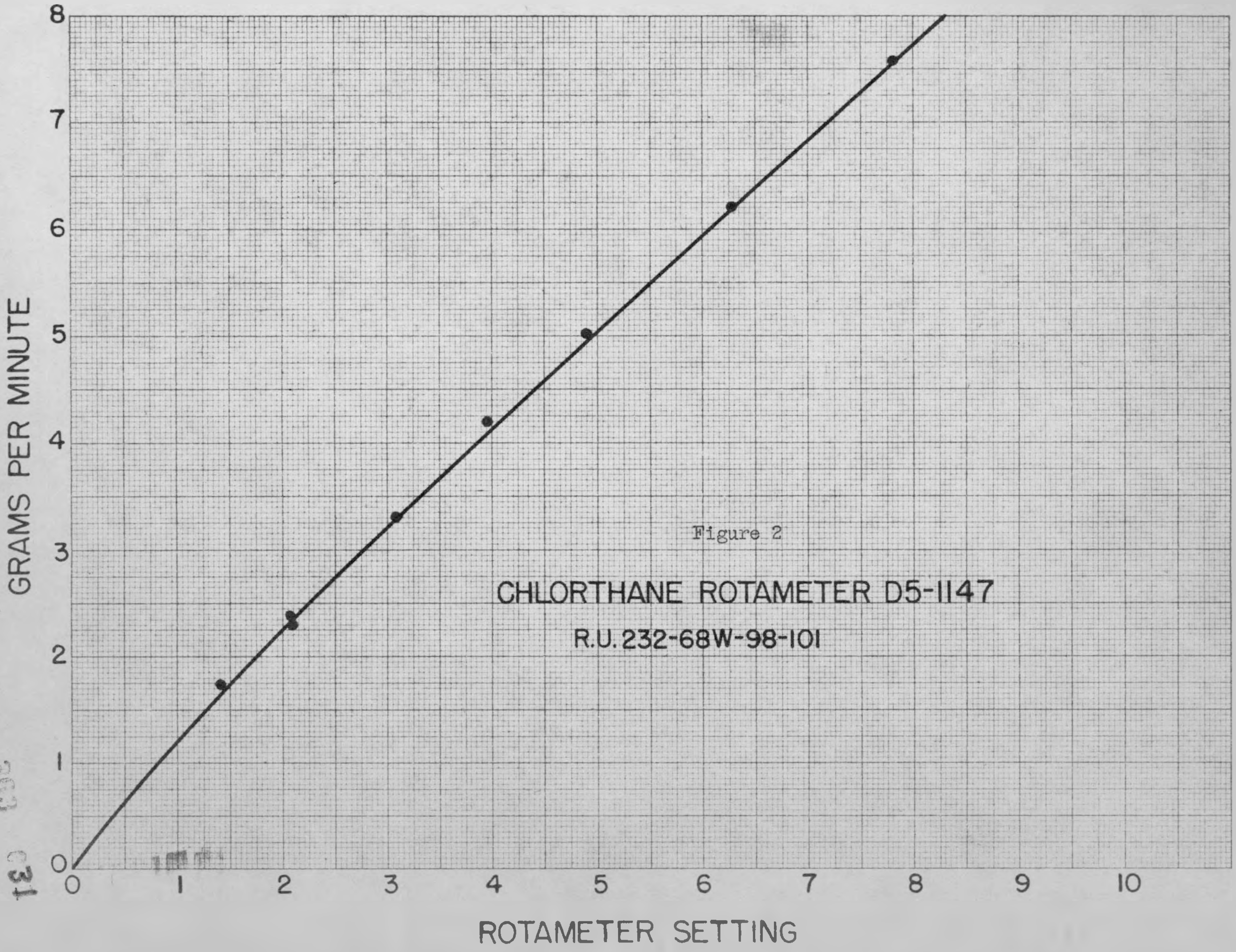


Figure 2

CHLORTHANE ROTAMETER D5-1147

R.U. 232-68W-98-101

cylinders (Dwg. 386-19-0, p.629). When the truck is about half empty, the amount of H_2 left and how long it will last are calculated; and a new truck ordered from American Oxygen, Newark, N. J. At least three days' notice is required. The cylinders are opened, the Multi-stage Gage turned up to 25 lbs., and the needle valve just beyond the gage (Dwg. 396-19-0) opened. The truck is now shut off and the auxiliary cylinders are supplying all the H_2. Valves K and T (Dwg. 396-19-0) are shut, the Multi-stage Gage S (Dwg. 396-19-0), A (Dwg. 395-19-0) released, all the bank valves on the truck closed, the high pressure gage line B (Dwg. 395-19-0) disconnected, and the main flange connection C (Dwg. 395-19-0) unbolted. After bolting on a protective cap in place of the flange and disconnecting the electrical ground calbes, the empty truck is ready to be taken away. The flange union on the new truck is wiped clean with a rag and some very fine emery paper and the line connection bolted on. Great care should be taken to get the flange on square in order to prevent leakage. The high pressure gage line is connected, and then the system,should be inspected for leaks. One bank valve is opened, the main line valve opened which puts a pressure of about 2000 lbs. on the flange, and the high pressure gage line connection. The MSA Explosivemeter is used for the detection of leaks. The flange should be straightened, tightened, and even lapped if necessary in order to get a leak-proof system. After the electrical ground leads have been attached, the new truck is ready to be put into operation. The Multi-stage Gage is turned down to give a pressure of 25 lbs. on the low side. Valves T and K (Dwg. 396-19-0) are opened. Valve L (Dwg. 396-19-0) is closed and valve U (Dwg. 396-19-0) quickly opened; the shift back to the truck is now complete. The auxiliary H_2 cylinders may be closed off.

4. Setting up the Tubes (Photograph 6)

After the run is under way, the tubes used in the previous day's run may be taken down, cleaned, and set up again. The iron wire is taken off the stopper, the clamp removed, and the tension on the spring released, on the right side of the tube (Dwg. 392-19-0) first. The electrical leads are removed. The iron wires are removed from the stoppers on the left side. The clamps are taken down and the rubber stoppers removed. The spring is taken off the side of the tube and the two-holed end removed gently along with the copper rods. After wiping out the grease, one goes to the right end of the tube, removes the single hole end, wipes out the grease, and pushes the tray about six inches toward the left side of the tube. One then removes the tray from the left side of the tube and places it, including the copper tension rod D (Dwg. 392-19-0), in a tared aluminum pan (four of these may be found in the grinding room - 6" x 45" x 1-1/2"). The wire and the product are removed from the tray and rods, and then weighed on the scale in the grinding room. The above procedure is followed for each of the tubes. All the wires and products are placed in the same tray but the latter is weighed after each time. The final weight minus the weight of the wires and minus the tare of the tray is the weight of 891 produced in the day's run.

The orifices of the tubes are cleaned by the use of a small pipette brush wet with acetone. The tubes are cleaned first with water and then dried with acetone by pushing a cloth-covered swab through them. The caked grease should be removed from the ends (usually just the bottom third) and fresh grease applied (Dow-Corning stopcock grease is used). The trays (A, Dwg. 392-19-0) should be washed off (Bon Ami),

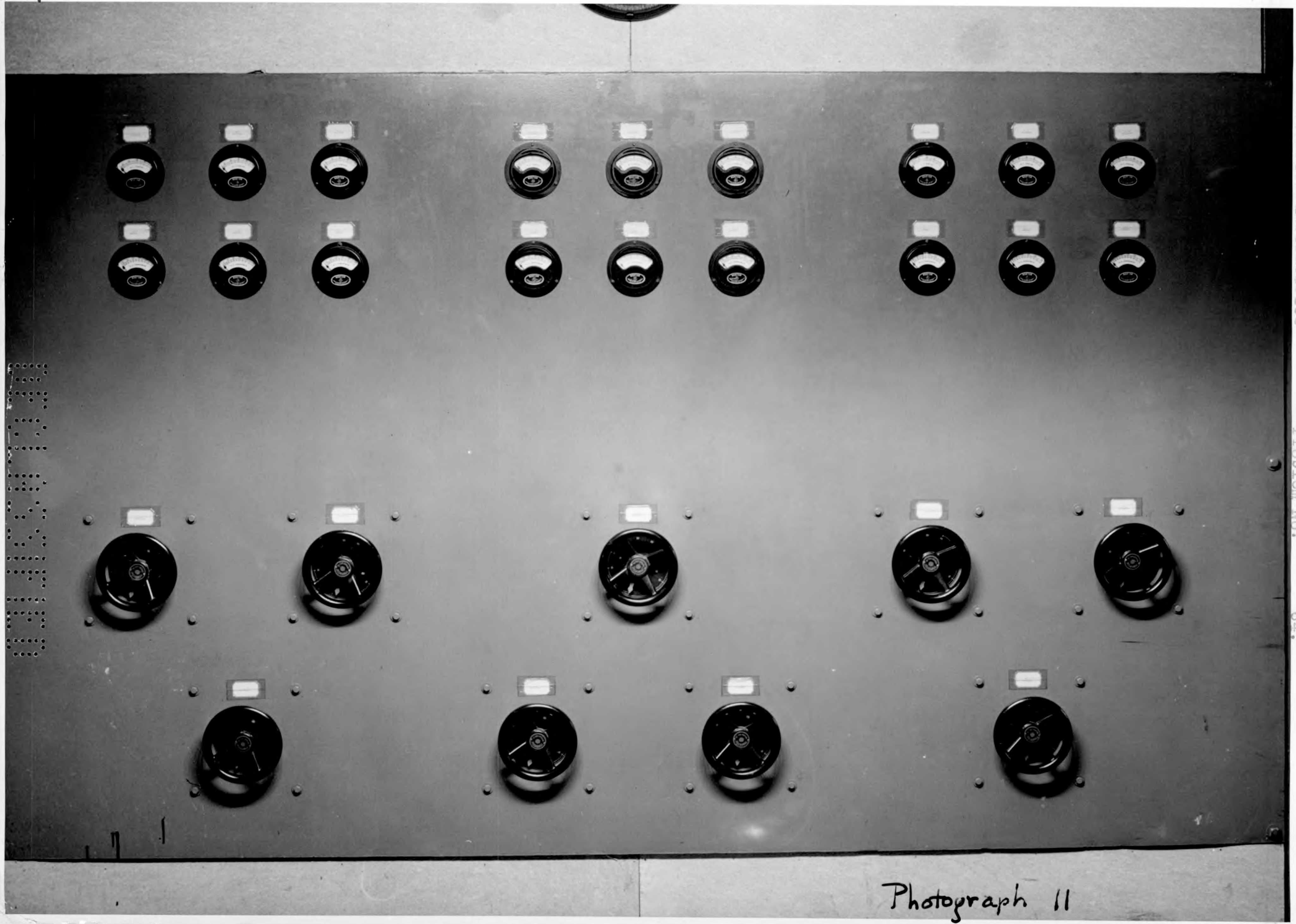
363-34

American Cyanamid Company
Stamford Laboratories

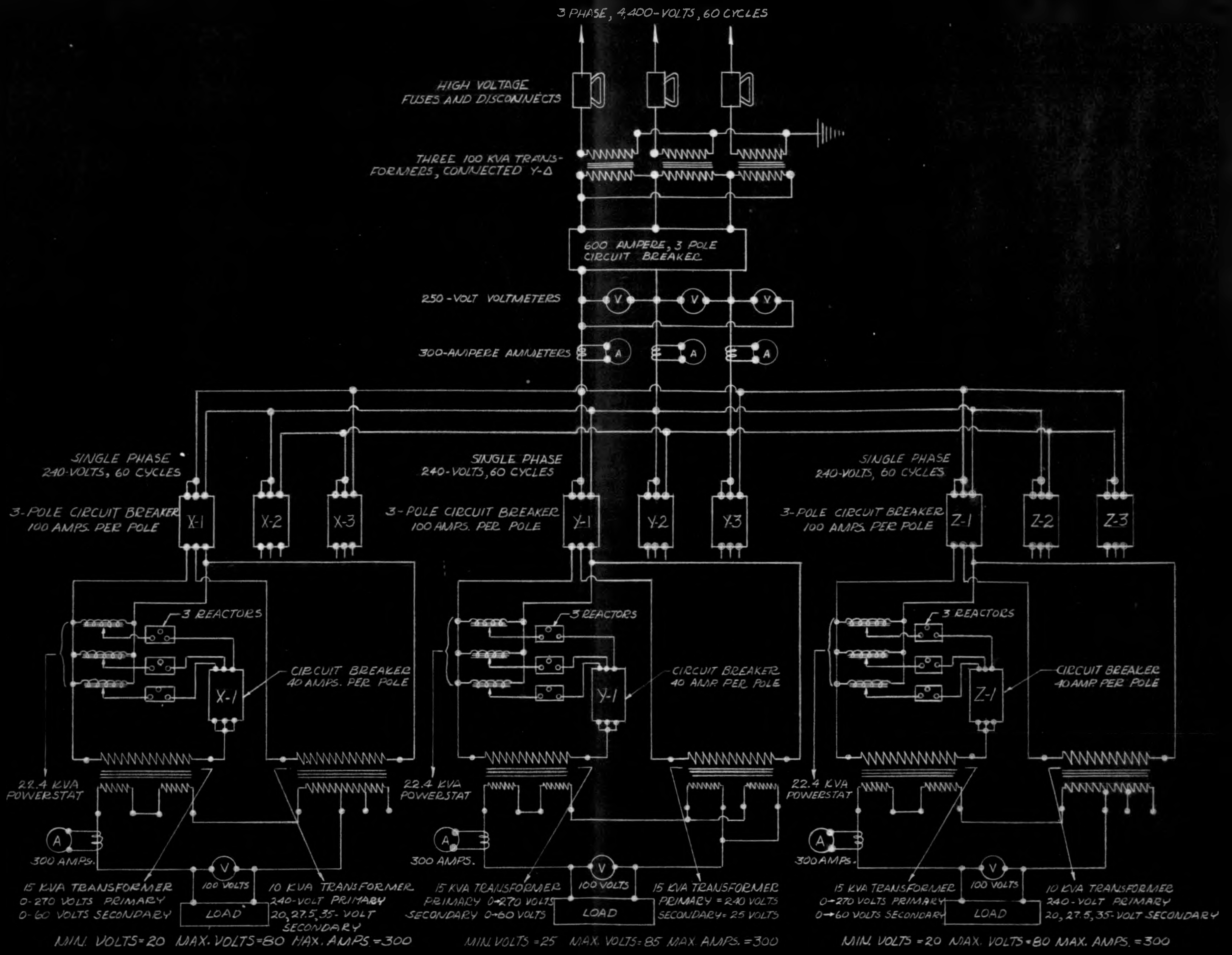
34

Investigation No. R. U. 232.
Problem No. 64.

Page 648enc-91



Photograph 11



AMERICAN CYANAMID CO.
STAMFORD, CONN.

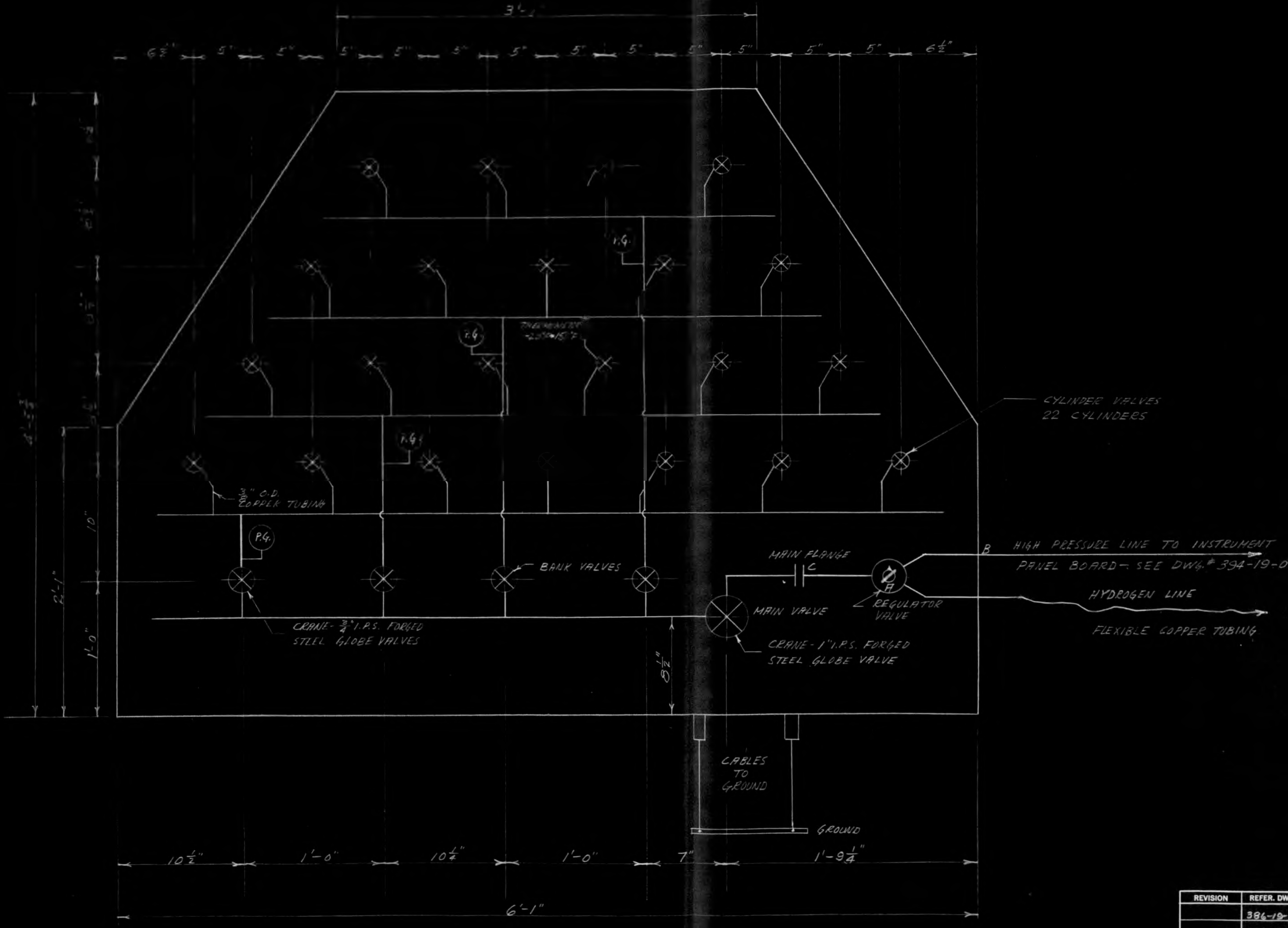
ISOLATED OPERATIONS NO.2 WIRING

REF. DWG REV.
397-19-0,

31-232-64
STEP #4

DESIGN: D. R.
DRAWN: D.R. – E.K.M.
SCALE: NONE
DATE: JAN. 2, 1945
DWG. 266
JOB 19
REV. 0

FILE 10-2



REVISION	REFER. DWG.
	386-19-0
	394-19-0

APPROVED

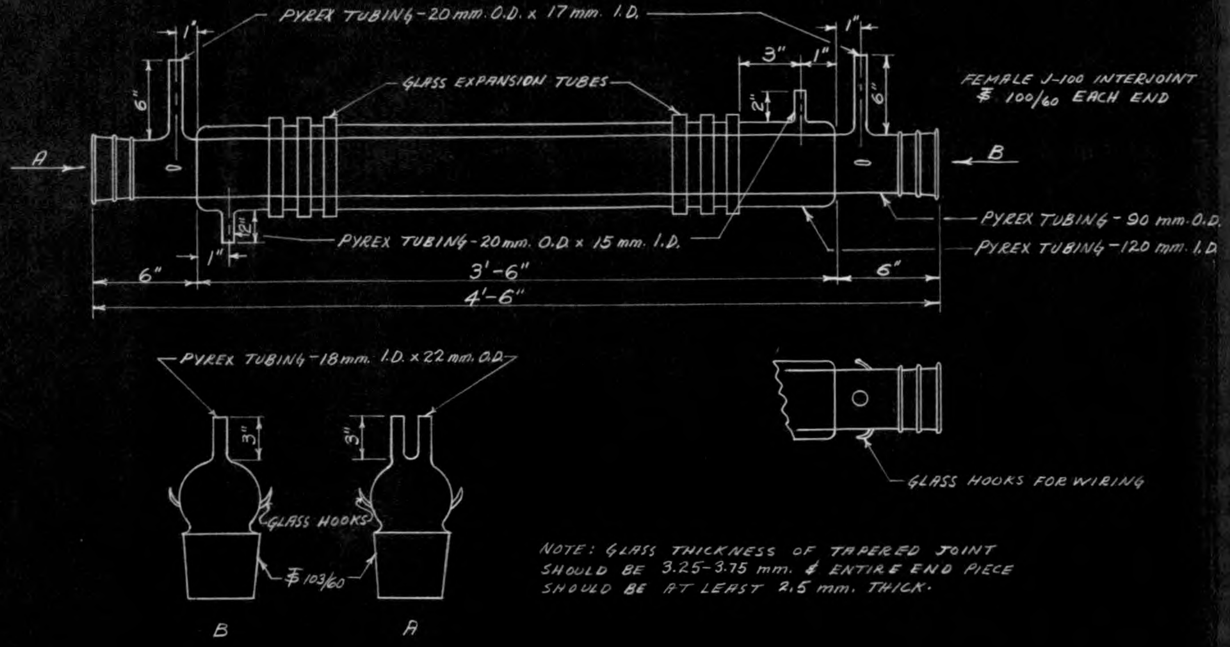
AMERICAN CYANAMID COMPANY
DEVELOPMENT ENGINEERING DIVISION
STAMFORD, CONN.

HYDROGEN TRAILER
STEP #4

31-232-64

DESIGN: FWT
DRAWN: FWT
SCALE: NONE
DATE: 6-18-46

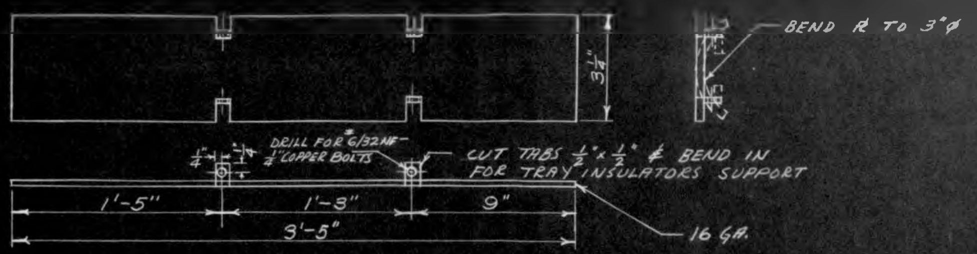
DWG.	JOB	REV.
395	19	0



NOTE: GLASS THICKNESS OF TAPERED JOINT SHOULD BE 3.25-3.75 mm. & ENTIRE END PIECE SHOULD BE AT LEAST 2.5 mm. THICK.

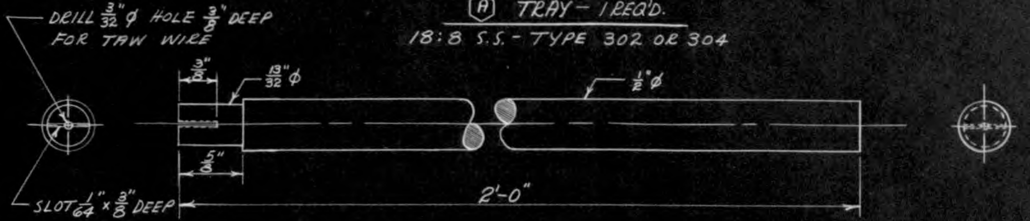
PYREX GLASS JACKET

ACE GLASS CO.



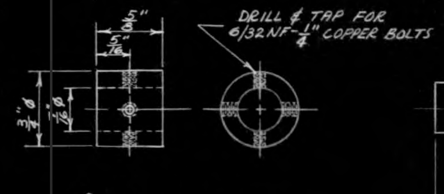
Ⓐ TRAY - 1 REQ'D.

18:8 S.S. - TYPE 302 OR 304



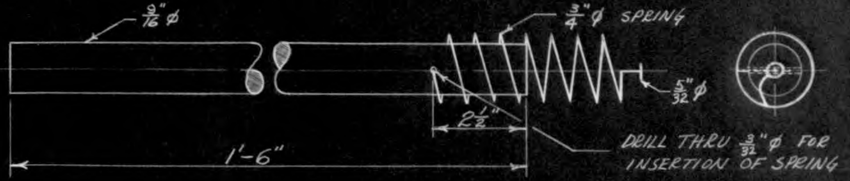
Ⓑ COPPER ROD LEADS

2 REQ'D.



Ⓒ COPPER COLLAR

2 REQ'D.



Ⓓ COPPER TENSION ROD & SPRING

1 REQ'D.

Ⓔ INSULATOR FOR TENSION ROD

MEISSNER 1" - 1 REQ'D.

Ⓕ HOOK FOR TENSION SPRING

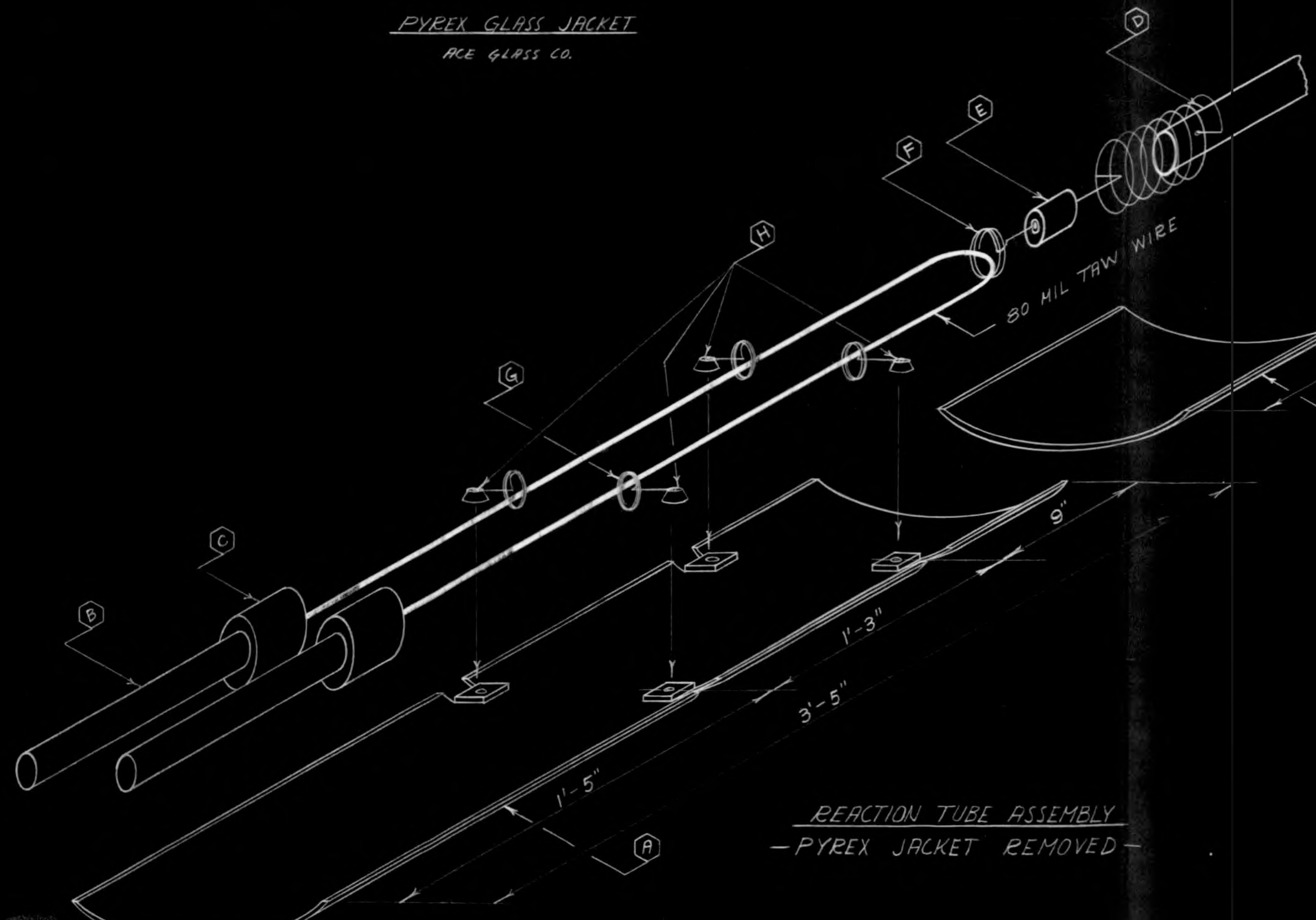
80 MIL T WIRE - 1 REQ'D.

Ⓖ WIRE SUPPORTS FOR TRAY INSULATORS

60 MIL T WIRE - 4 REQ'D.

Ⓗ TRAY INSULATORS

JOHNSON STEATITE CONE INSULATORS - CAT. #500 - 4 REQ'D.



REACTION TUBE ASSEMBLY

-PYREX JACKET REMOVED-

Ⓘ MOLYBDENUM SHIELD - 1 REQ'D.

REVISION	REFER. DWG.
	266-19-0
	386-19-0
	387-19-0
	396-19-0
	397-19-0
APPROVED	

AMERICAN CYANAMID COMPANY
DEVELOPMENT ENGINEERING DIVISION
STAMFORD, CONN.

REACTION TUBE
STEP #4

31-232-64

DESIGN: FWT
SCALE: NONE
DATE: 6-21-46
DRAWN: FWT

DWG.	JOB	REV.
392	19	0

dried and any broken tray insulators (H, Dwg. 392-19-0) or wire supports (G, Dwg. 392-19-0) replaced. The copper leads (B, Dwg. 392-19-0) should be cleaned with steel wool. The copper tension rods and springs (D, Dwg. 392-19-0) cleaned and any broken insulators (E, Dwg. 392-19-0) or hooks (F, Dwg. 392-19-0) replaced. A 75 inch TAW wire which is received in straight lengths and so ordered is bent in a hairpin shape and the other sections of the wire straightened. The wire is weighed on the balance in the grinding room, passed through the hook (F, Wg. 392-19-0) on the copper tension rod (D, Dwg. 392-19-0), and inserted in the supports on the tray (A, Dwg. 392-19-0) with the hairpin being on the side of the tray in which the supports are only nine inches from the end. The tray, wire and copper tension rod are placed in the tube from the left side (Dwg. 392-19-0) - hairpin end first, and pushed in until the tray is just flush with the left end of the tube. The copper collars (C, Dwg. 392-19-0) are placed on the wire, the ends of the wire stuck into the slots in the copper rod leads (B, Dwg. 392-19-0), and the collars screwed down tight on the rods, thus holding wire in place. The glass insulators (Dwg. 392-19-0) are put on the rods and the rods and the tray pushed into the tube until the tray is flush with the water jacket. The extreme ends of the rods are supported on a clamp attached to the rack. The shield (I, Dwg. 392-19-0) and the single-holed ground glass end are placed into the right end of the tube (Dwg. 392-19-0) with the copper tension rod extending through the hole. The external spring is attached (Dwg. 392-19-0). Now working from the left side of the tube, the tray is pushed into the tube until it establishes contact with the shield, and then the two-holed ground glass end is put into the tube with the copper rods extending through the holes and the external spring attached. No. 3 rubber stoppers with holes just fitting the rods (holes made with #8 cork borer) are used to hold the rod and seal the tube. Still working on the left end of the tube, the rubber stoppers are fitted over the rods and into the glass tubes to make a gas-tight seal. After making sure the rods extend over the end of the tray about an inch and the hairpin end of the wire is horizontal and in the center of the tube, the rubber stoppers are wired on with #18Ga. galvanized iron wire. Going to the right side of the tube, a rubber stopper is fitted over the copper tension rod and inserted in the glass tube; a fairly good tension put on the spring by pulling back the rod, and the stopper wired tight. The rod is clamped into a position in the center of the tube and slightly off the horizontal plane - the outside end higher than inside end (Photograph 6). Now going to the left side of the tube, the copper rods are clamped into position by the use of four burette clamps and short pieces of asbestos tape for insulation (photograph 6), taking care that they are approximately horizontal and that they are parallel (Dwg. 392-19-0) (a space of half an inch or more between them). With the attachment of the proper electrical leads (Dwg. 397-19-0), the tube is completely set up. An identical procedure is used for the rest of the tubes. A number of similar tasks may be performed at the same time and thus speed up the procedure; i.e., all the tubes may be cleaned at the same time, all the tube ends degreased together, all the trays cleaned together, etc.

5. Operation of the Condenser System

There are three main parts to the condenser system: the precondenser coil (Dwg. 391-19-0), which is held at a temperature of -75°C. by dry ice-trichlorethylene mixture, the secondary condenser (Dwg. 240-19-2) operating at a temperature of -110 to -125°C., and the third condenser or freezer (Dwg. 390-19-0) operating at a temperature of -180°C. The temperatures of the second and third condensers are controlled by a thermocouple point which is located on the outside of the liquid air pipe just before it reaches the condenser (Dwg. 399-19-0). At the start of the run, the red control pointer on the Micromax Recorder (Dwg. 394-19-0) should be set at about -175°C. As soon as point #4 reaches -120°C., the control point may be set down to -160° (6 or 7 tubes). (The greater the number of tubes, the colder the control point will have to be kept.) An indication of how the condenser system is operating is shown by the analyzer (Dwg. 386-19-0). The system should be kept cold enough to keep the analyzer Micromax reading close to zero (especially in the first half of the run). If point #4 is kept between -120 and -127°C., the analyzer will give low readings. Toward the end of the run the reading may rise to 4 or 5 without any harm being done. As the run progresses, the control pointer on the Micromax recorder will have to be moved toward the cold end in order to maintain point 4 temperature and a low analyzer reading. The control point should be kept at the warmest point in which the desired temperatures and readings may be maintained.

When a liquid nitrogen can runs empty during a run, the recording point temperature and point 16 (Dwg. 399-19-0) will fall off. The nitrogen rotameter (Dwg. 399-19-0) is set up to 100. When point 16 reaches -140°C., the solenoid switch (one on left) on the top of the Micromax recorder is turned off. This cuts off the gaseous nitrogen flow, and the can may be exchanged for a full one. The unions E and F (Dwg. 399-19-0) are broken and the delivery tube (Dwg. 399-19-0) removed and a #4 rubber stopper put in the other half of the union at E (Dwg. 399-19-0). The empty can is taken down and a full one put in its place. The delivery tube is slowly inserted in the can to prevent wasting the liquid nitrogen. When the delivery tube is in place, the stopper may be removed and the unions E and F (Dwg 399-19-0) put together and tightened (not too tight- just snug). The solenoid switch on the recorder may be turned on and the float on the rotameter (Dwg. 399-19-0) set at 30.

If the line manometer on the panel shows an increase of pressure during the run, a plug of some sort has appeared in the system. The plugs, their detection and removal, will now be discussed for the various ones which have arisen so far during this project.

If both manometers α and β (Dwg. 396-19-0) show a pressure, the plug is somewhere in the exit pipe beyond the freezer. The plug so far has been at the point where the gas enters the atmosphere. One may climb up on the roof and poke the line free with a rod.

If manometer β (Dwg. 396-19-0) indicates a pressure but not α, then the plug must be in the freezer. The center heater switch (Dwg. 394-19-0) is flicked on and the powerstat turned up to 60 (maximum). The liquid nitrogen is turned off by the solenoid switch on the Micromax recorder (Dwg. 394-19-0). This heater should quickly melt any minor plug in the freezer. If the freezer plugs solidly and the gas pressure goes up to 4.5 cm. of Hg and starts blowing out the lute (Dwg. 386-19-0), one must crack the "still" valve (D, Dwg. 396-19-0). The freezer strip heaters should be turned on by throwing the switch (Dwg. 394-19-0), turning on the Bristol controlling the freezer strip heaters on the panel, and turning up the freezer heater powerstat to about 50. If point 3 warms up to above -170°C., and the system is still plugged, the freezer valve (B, Dwg. 396-19-0) should be closed. The still valve D (Dwg. 396-19-0) should be turned down until a pressure of 3.5 to 4 cm. of Hg is registered on the panel manometer. The freezer is allowed to warm up until the plug is melted. Valve B (Dwg. 396-19-0) is cracked from time to time to check on the plug. When opening valve B causes the pressure to drop right off, then the plug is gone. The center heater and the strip heaters may be turned off by their respective switches. The Bristol for the freezer heater is turned off. Valve B (Dwg. 396-19-0) is left closed and the liquid nitrogen turned on by the switch on the recorder. When point 3 reaches a temperature of -150°C., Valve B (Dwg. 396-19-0) may be opened and valve D (Dwg. 396-19-0) closed.

If the panel manometer shows a pressure but neither α or β show any, the plug is before the freezer. If point 4 suddenly gets very cold (-160°C.), the condenser is frozen. The liquid nitrogen should be shut off and the rubber stopper G (Dwg. 399-19-0) should be removed and any surplus liquid nitrogen allowed to drain out. If point 4 is still getting colder, stopper A (Dwg. 399-19-0) is removed, an air hose placed in pipe G (Dwg. 399-19-0) and a stream of air sent through the jacket of the condenser. When the condenser freezes, the pressure blows the liquid Chlorthane out of the trap in the drain line and the gas bypasses the condenser and flows through the drain line. One of the valves on the drain line (either Z_1 or Z_2, Dwg. 396-19-0) should be closed down until a pressure of 4 cm. appears on the panel manometer. The air is allowed to blow until the plug melts. When it melts, the back pressure on the panel manometer will drop off to normal and point 4 will warm up quickly. The air is then turned off, stoppers G and A (Dwg. 399-19-0) put in place, the the switch for the liquid air turned on.

If a pressure develops and is not detected by manometers α or β (Dwg. 396-19-0), and point 4 remains constant, the chances are that there is a solid plug of oxide forming somewhere. Valve P (Dwg. 396-19-0) may be opened. If this lowers the pressure the filter should be shut off and cleaned and new glass wool put into it. If opening valve P does nothing, bypass valve C (Dwg. 396-19-0) may be opened. If this lowers the pressure a great deal, the plug is some place beyond the point where the bypass line leaves the system and the condenser (Dwg. 386-19-0). This plug cannot be fixed while the system is running. If the pressure gets too great, the run must be stopped. The next day after a good drain is obtained and all the system warmed up, the rock wool will have to be taken out of the top section of the box, the pipe taken out and cleaned before it can be put back, and another run begun. If the bypass valve is opened and no pressure drop is apparent, the coils of the precondenser are plugged (Coil #1, Dwg. 396-19-0, for sure and maybe coil #2). If opening the by-pass line has no effect and if when a drain line valve Z (Dwg. 386-19-0) is closed down the pressure increases, coil #2 is plugged. In any case when a solid plug occurs in the coils, they cannot be fixed until the unit is shut down.

On the morning in which a plug is to be cleaned out, the kettle is drained as usual, with this difference. Before stopping the drain by closing valve E (Dwg. 398-19-0), the kettle (Pt. 10) should be heated up to room temperature by putting steam on the coil D (Dwg. 398-19-0) while running the kettle circulation system pump A (Dwg. 398-19-0). The freezer should be heated above room temperature by the use of the center heater and the freezer strip heaters. The trichlorethylene should be drained from the precondenser system by opening valve T (Dwg. 398-19-0) in the drain line (Dwg. 386-19-0) and allowing the liquid to run into five-gallon containers (in small side room). The dry ice may be scopped out by hand methods and placed in a large cover so that the trichlorethylene clinging to the ice may be recovered. When the precondenser system is empty, the outside of the coil should be steamed. Air should be blowing through the secondary condenser jacket and the freezer - in at E and out at A (Dwg. 399-19-0). When the system is thoroughly warmed up (usually takes all morning), valve E (Dwg. 398-19-0) may be closed and the drain bomb removed.

The top section of the box (south side) should be taken off and all the rock wool removed (the wet wool is kept separate from the dry wool) from around the precondenser tank and a working space cleared around the unions underneath. All four unions on the top and bottom of the two precondenser coils are opened, and rubber stoppers quickly placed in the pipes leading to the rest of the system. Steam is then blown up through each of the coils in turn for a period of half hour or so. After a coil has been steamed clean, air is blown through it while it is still hot until the pipe is dry. When everything is dry, the rubber stoppers are removed and the unions refastened. A pressure of about 16 cm. of Hg is put on the system to test for leaks by: making sure valves A, B, C, and D (Dwg. 396-19-0) are closed; closing off the tubes by putting valves G and H (Dwg. 397-19-0) in positions 2 and 5 respectively; opening the bypass valve above the three-way values (Dwg. 396-19-0); opening value R (Dwg. 396-19-0) and the nitrogen cylinder behind the instrument panel; and building up the pressure by opening valve E (Dwg. 396-19-0) until the desired pressure is reached. The fittings should then be inspected for leaks (any leak will be evident as Chlorthane will escape and leave a white smoke of oxide). If any leaks are found, the pressure should be released by opening valves A and B (Dwg. 396-19-0) slowly; the fittings broken, wiped off, realigned, etc. and then put back together again. When the system is tight, the side is put on, the box is filled with dry rock wool, and the trichlorethylene and dry ice are replaced.

The flexible copper lines attached to the tubes may become partially plugged. This fact will show up when the tube's manometer (Dwg. 392-19-0) reads low and the valves on the rest of the tubes will have to be turned down in order to force a flow through the conjested tube. When the tube is shut down, the oxide plug may be removed by poking a rod up the copper tube.

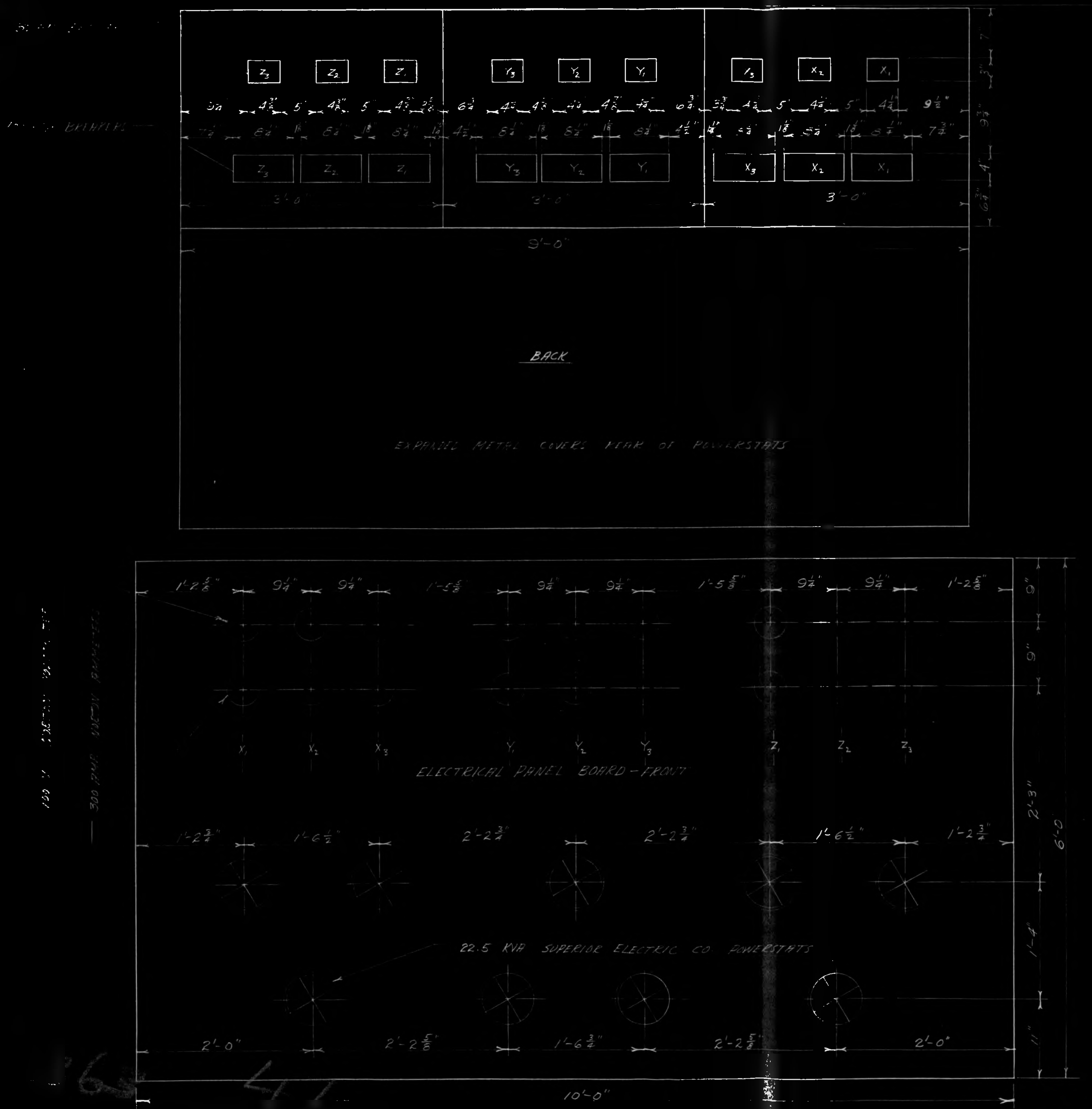
6. Shutting Down Operations

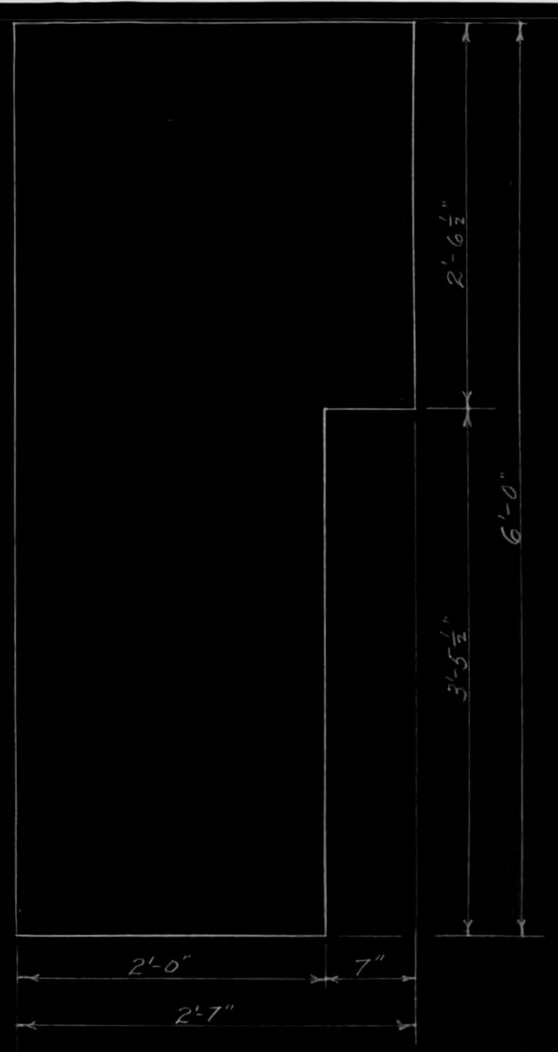
Ten minutes before the end of the run the flask containing dry ice and trichlorethylene can be placed around the cooling coil to the Chlorthane receiver cooling system and the circulating pump started at a slow speed (Dwg. 398-19-0). Five minutes before the end of the run the liquid nitrogen is shut by throwing both switches on top of the Micromax recorder and shutting off valves on the nitrogen cylinder (Dwg. 399-19-0).

When the tubes are to be shut down (usually at the end of eight hours), the Chlorthane line valve (Valve E or F, Dwg. 396-19-0) is shut. Then as soon as flow through the Chlorthane rotameter has stopped, valve H (Dwg. 396-19-0) is shut. Then allow 45-60 seconds for the Chlorthane in the line to be swept into the tubes. While waiting for the line to clear, variacs and switches to Chlorthane line and tank heater can be switched off, and also switches Nos. 12 and 13 (Dwg. 394-19-0). Also begin to cool Chlorthane tank by setting it in a box of dry ice.

Now the variacs controlling power to the filaments (Dwg. 393-19-0) are gradually turned back to zero setting (this operation should require about one minute), and the appropriate circuit breakers are thrown to cut the power completely (Dwg. 393-19-0). The normal hydrogen flow is continued until the filaments are cold. The hydrogen flow is then changed to nitrogen flow at rotameter setting of 90 for 10 minutes. This change is made by closing valve J and valve I (Dwg. 396-19-0) on Lectrodryer and opening valve R (Dwg. 396-19-0) and the valve on the nitrogen cylinder behind control board.

During the 10-minute flush period the liquid nitrogen can is disconnected and covered. The gas analyzer is turned off by switching off the two toggle switches inside the Micromax, shutting the stopcock in the suction line, turning off the aspirator and closing valves on the hydrogen cylinder connected to analyzer. The valves to the glass wool filter are shut, Q_1 and Q_2, after valve P (Dwg. 396-19-0) is opened. The electrical leads to the reacting tubes should be changed for the next day's run. If racks I and II have just been turned off, then leads Y_1^{I} must be exchanged for leads Y_1^{III}, in box L (Dwg. 397-19-0); the leads X_3^{I} exchanged for X_3^{III} in box M (Dwg. 397-19-0); and leads Z_2^{I} exchanged for leads Z_2^{III} in box N (Dwg. 397-19-0). On rack II, leads Y_3 changed from tube 2 to tube 4 and Y_2 changed from tube 1 to tube 3. If racks II and III have just been turned off, the above process is reversed. The cooling water to the reacting tubes should be shut off. The HCl exit stack (beyond pressure lutes) can be cleared at point opening to the atmosphere by poking several times with the attached ram rod.





REVISION | REFER. DWG.
266-19-0
387-19-0

APPROVED

AMERICAN CYANAMID COMPANY
DEVELOPMENT ENGINEERING DIVISION
STAMFORD, CONN.

ELECTRICAL PANEL BOARD
STEP #4

31-232-64

DESIGN: FWT
DRAWN: FWT
SCALE: 1-1/2" = 1'
DATE: 6-18-46
DWG. 393
JOB 19
REV. 0

After flushing for 10 minutes, the nitrogen is turned off by closing valves E and R (Dwg. 396-19-0), and valves A and B (Dwg. 396-19-0), the stack and freezer exit valves. Next the two three-way valves G and H (Dwg. 397-19-0) are closed to positions 2 and 5, respectively, followed by all the 1/4" gate valves leading to the individual tubes (Dwg. 392-19-0).

Thawing of the freezer is now started by turning on the freezer center heater variac at a setting of 45 and the external freezer strip heaters by switching on Bristol freezer heater, freezer strip heater switch, and setting freezer heater powerstat at a setting of 55. Thawing is further hastened by blowing air into freezer jacket at G (Dwg. 399-19-0) and exiting at E and A (Dwg. 399-19-0), after removing rubber stoppers. This warms both the freezer and the condenser. This heating process is continued until the solid material in the freezer melts and drains through the drain line into the Chlorthane receiver. About 30-40 minutes' heating is required to thaw the freezer. This draining can be detected by watching points Nos. 10 and 14 on the 18-point indicator (Dwg. 394-19-0). At the time of this draining, point 14 will drop from -80° to -90°C. to -105°-115°C. and point 10 (internal temperature of Chlorthane receiver) will drop from about -75° to -78° to -83° to -87°C. At this time, all the heaters used for thawing are switched off. Turn off air to freezer and replace stoppers at A, E and G (Dwg. 399-19-0).

As soon as the system is entirely closed, before starting the thawing procedure, pressure will immediately start to build up and in a few minutes will reach a pressure of 4.5 cm. kg., and will begin blowing HCl through the low pressure Hg lute (Dwg. 386-19-0). At the time the freezer drains into the Chlorthane receiver the pressure in the system will likely fall below 4.5 cm. and bubbling of HCl will stop. Bubbling of HCl through the Hg lute should commence again within a few minutes.

Plugging of the mercury lute or the HCl exit stack occurs occasionally. If the lower pressure lute set at 4.5 cm. mercury becomes plugged, pressure will build up to about 10.5 cm. mercury and the HCl will then pass through the 10.5 cm. lute. If this lute becomes plugged, pressure will build up to 14.5 cm. mercury and then pass through the 14.5 cm. lute and out the No. 2 exit pipe (Dwg. 386-19-0). The high pressure alarm horn will blow if this happends. If the No. 1 exit pipe becomes plugged, pressure will build up to 14.5 cm. mercury and the HCl will exit through the 14.5 cm. lute and out the No. 2 exit pipe. Again the high pressure alarm horn will blow. If both exit pipes or all of the lutes should become plugged, which is not very likely to happen, then the pressure will continue to build up beyond 14.5 cm. mercury. The alarm horn will continue to blow. This condition must be remedied at once by attaching a rubber hose to the 1/4" pipe coming from the 1/4" gate valve (N, Dwg. 396-19-0) leading the hose to outside the building, and opening valve N (Dwg. 396-19-0). The HCl can then be discharged through this hose until the plugs can be removed.

The flask of dry ice cooling the Chlorthane receiver is now filled and a wooden bucket of chopped dry ice left for the watchman with the instructions: "Keep filled with dry ice until midnight. Then fill well and add no more ice. At 6:30 a.m. remove flask from coil and also any ice remaining on coil." By following this procedure the HCl will distil off at the proper rate for draining of the Chlorthane the following morning. The variac operating the circulating pump is set at 80 (Dwg. 398-19-0).

While thawing the freezer, the Chlorthane tank can be

DECLASSIFIED

removed (after pulling a vacuum of at least 10 in. Hg. by cooling with dry ice) and weighed to determine the amount fed to the tubes during the run. The Chlorthane recovery tank can be attached to the drain-line for draining the following morning.

All heater circuits on the instrument panel board are turned off except the heater on the catalyst tower (purifier), the powerstat of which is lowered to setting of 50, and the Bristol controller set at 250°C. (Dwg. 394-19-0).

The hydrogen trailer is turned off by closing the main valve and the regulator valve (Dwg. 395-19-0). The valves on the individual cylinders are all left open, and also the bank valve on the bank being used.

7. Repairs

The following comments indicate conditions or apparatus which might need repairs or changes.

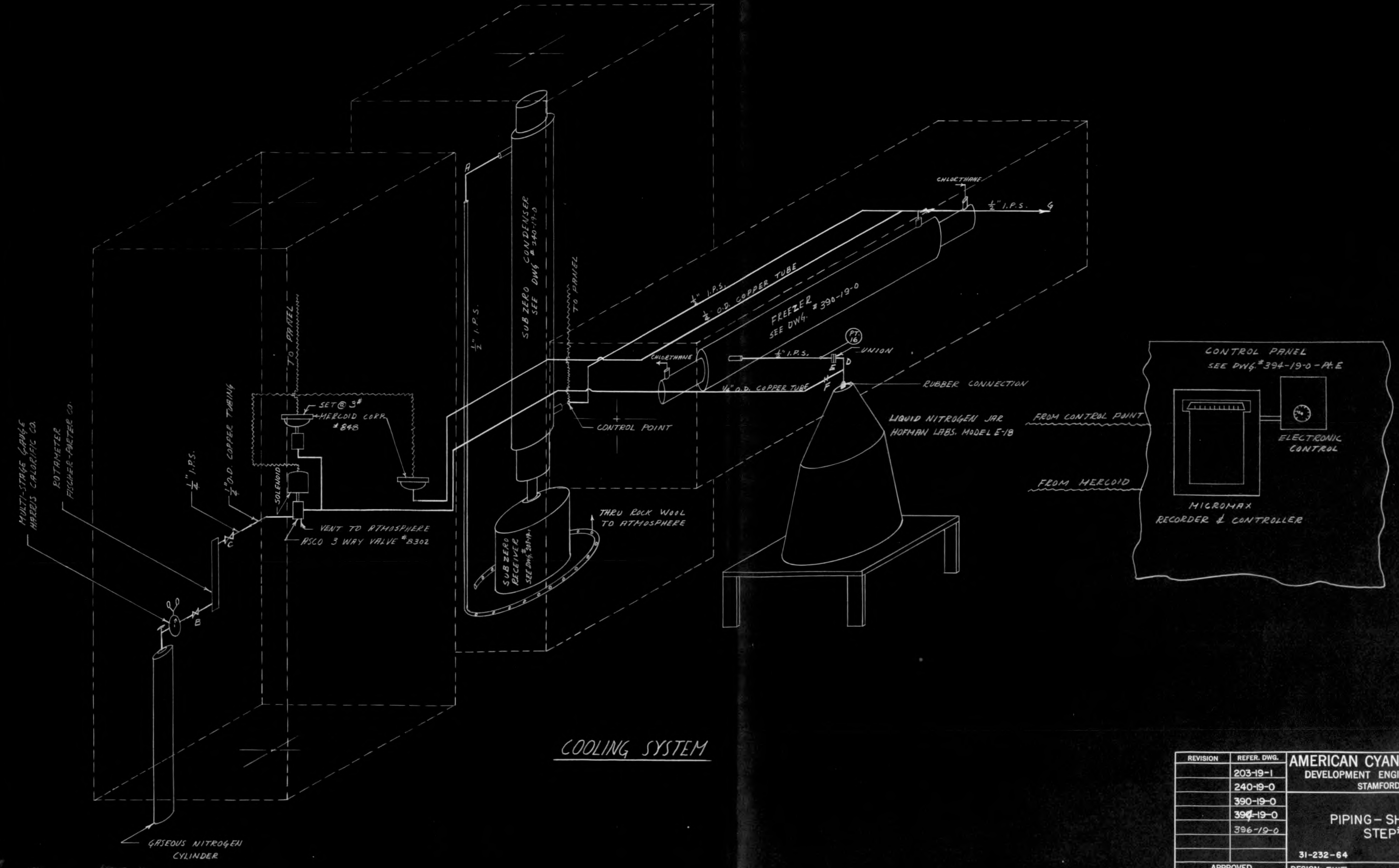
During the last 17 runs of the operation the daily liquid nitrogen consumption was considerably above normal. Normal liquid nitrogen consumption averaged 200 l. per day. On Run 395 consumption suddenly jumped to an average of 230 l. per day and continued at that level. Suspecting a leak in the liquid nitrogen lines, examination of lines from the delivery tube to the freezer and from the freezer outlet to the sub-zero condenser indicated all pipes and joints were intact. The jacket of the freezer could not be checked without interrupting the operation. The outside of the freezer box on the bottom had a heavy frost coating about six inches wide from end to end. There was also more frost than usual on the outside bottom of the subzero condenser. This suggests either a leak in the liquid air system or ineffectiveness of the rock wool insulation because of accumulation of moisture in the rock wool. Before operating further, the liquid nitrogen system should be completely checked and the rock wool examined, and replaced if necessary with dry rock wool.

The Chlorthane control valve on the panel board (H, Dwg. 396-19-0) leaks around the packing and must be repacked or replaced.

On the hydrogen Lectrodryer the reactivating pump leaks hydrogen around the packing. In order to reduce the amount of gas escaping, we have tightened down the packing nuts as far as possible, but the leaking continued, although slower. New packing is necessary to put the pump in operating condition.

Three new reacting tubes are needed on the racks. This involves the copper-glass seals, which must be seated onto the glass inlet tubes of the reacting tubes. The tubes needed are: Tube 2, Rack I; tube 1, rack II; and tube 3, rack II (Dwg. 397-19-0).

Periodically the precondenser requires cleaning by blowing each of the two coils a half hour with steam, and then blowing with air until dry. Forty runs have been made since the precondenser was last cleaned, which is about the average number of runs that can be made between cleanings. This cleaning procedure should be done before operating further. It appears that cleaning the condenser, Chlorthane receiver and drain line with steam and drying with air would be well worth while. This part of the condensing system has not been cleaned since installing, and is probably coated with solid material which reduces the efficiency of the condenser. The freezer, too, has never been cleaned and very likely contains considerable solid material. It should be steamed thoroughly, then dried by using the center heater in the freezer and blowing with air.



COOLING SYSTEM

REVISION	REFER. DWG.
	203-19-1
	240-19-0
	390-19-0
	39~~0~~4-19-0
	396-19-0

APPROVED

AMERICAN CYANAMID COMPANY
DEVELOPMENT ENGINEERING DIVISION
STAMFORD, CONN.

PIPING – SHEET #4
STEP #4

31-232-64

DESIGN: FWT
DRAWN: FWT
SCALE: NONE
DATE: 6-25-46

DWG.	JOB	REV.
399	19	0

The reaction tubes (Photograph 6, p.625) are held on the rack by pipe hangers which are held by Fisher castelloy clamps to the rack. Many of these clamps are bent downward and need to be replaced.

Both of the pressure alarm horns are in poor working order, due to corrosion of the moving parts, and need to be cleaned and adjusted. The low pressure alarm should blow at 3.0 cm. mercury and the high pressure alarm at 14.0 cm. mercury (Dwg. 396-19-0).

Variables and Variations

a) Chlorthane Feed Rates

A rate of 75 liters per hour per tube was chosen to be the optimum rate. Although slower rates would tend to increase the conversion, the overall yield in grams per hour would drop off and the amount of 891 in grams per wire would not meet production requirements. With the 75 l./hr. rate, conversions of over 60 percent have been obtained when a high percentage of recovered Chlorthane has been circulated.

Higher rates of 100, 125 and 180 liters per hour have been tried with the only advantage being that the yield in grams per hour is increased. These higher rates tend to decrease the conversion and make the condensing operation more difficult, with the result that greater losses occur through the vent.

b) Hydrogen Feed Rate

A ratio of 15/1 (H_2/CT) was chosen as the optimum. Lower ratios (12/1 or so) tend to increase the conversion slightly and also help the recovery, but it has a detrimental effect on the ease of handpicking. Ratios higher than 15/1 up to a certain point (not determined exactly, but approximately 20-25/1) lead to slightly easier handpicking, but also reduce the conversion and the recovery is made more difficult. With a Chlorthane rate of 75 l./hr., a hydrogen rate of 15/1 is about the maximum that can be handled by the present condenser system if seven tubes are in operation. A number of operations such as higher Chlorthane rates with lower hydrogen ratios and high hydrogen ratios during the first part of the run have been tried but so far 75 l./hr. and 15/1 seem to be the most desirable conditions.

c) Length of Run

As long a run as possible is desired since the rate of deposition goes up very rapidly in the latter hours of the run. An eight hour run has been decided upon as an optimum. After 8-1/2 or even 8-1/4 hours, the TAW wires show a tendency to become brittle and even break before the end of the run. Also, by staggering shifts, the 8-hr. run fits nicely into a working day with three operators.

d) Electrical Leads for Runs Under 7 Tubes

1. A Six-Tube Run

When six tube runs are being made day after day, one tube only on Rack II (Dwg. 397-19-0, p.631) is used per day and thus the leads Y_2 and Y_3 will not have to be shifted from one tube to another. It still will be necessary to switch the leads in the boxes L, M and N (Dwg. 397-19-0). Thus if Racks I and II were to be used, tubes 1, 2, 3, 4 and 5 on rack I and tube 3 on rack II, and electrical power to X_1, X_2, X_3, Y1, Y_2 and Z_2 would be in operation. (Valves C and D, Dwg. 397-19-0, open.) If racks II and III were to be used, tubes 1, 2, 3, 4 and 5 on rack III and tube 4 on rack II and electrical power to X_3, Y_1, Y_3, Z_1, Z_2 and Z_3 would see operation. (Valves C and D, Dwg. 397-19-0, closed.)

2. A Five-Tube Run

If five tubes were to be run day after day, rack II would not have to be used. Lead Z_2 would be left in box N (Dwg. 397-19-0) attached to the third rack. Lead X_3 would be left in box M (Dwg. 397-19-0) attached to the first rack. Thus only the leads in box L would have to be changed from day to day. Lead Y_2 would be moved to rack I and lead Y_3 moved to rack III. Thus if rack I were in use, tubes 1, 2, 3, 4 and 5 in rack I and leads X_1, X_2, X_3, Y_1 and Y_2 would be employed. (Valves C, D, E and F, Dwg. 397-19-o, open.) If rack III were in use, tubes 1, 2, 3, 4 and 5 on rack III and leads Y_1, Y_3, Z_1, Z_2 and Z_3 would be employed. (Valves E and F, Dwg. 397-19-0, closed.) Four tubes and under may also be run by using only one rack and leaving the leads fixed.

e) The Analyzer (Dwg. 386-19-0).

This instrument has a standard cell and an unknown cell. The Micromax is "zeroed" by running pure hydrogen through both cells and setting the dial marked "zero" at a point (now 53) where the instrument is just in balance, i.e., at 53 the pointer is stationary, at 53.5 the pointer moves up the scale, and at 52 would give a low result. With a sample of the exit gas (Dwg. 386-19-0) running through the unknown cell and pure hydrogen in the standard cell, the Micromax will indicate how close the conductivity of the sample is to pure hydrogen. The Micromax has not been calibrated too exactly. A reading of 2 or 3 at the beginning of the run will result in about a 4 or 5 percent loss of Chlorthane; while near the end of the run, the same reading will result in an exceedingly small (under 1%) loss. This difference is mainly due to the varying percentage of HCl in the escaping gas. The instrument provides a sensitive method of observing the efficiency of the condensing system, which is so important for high yields.

Operation: Combustible Gas Alarm (Dwg. 394-19-0).

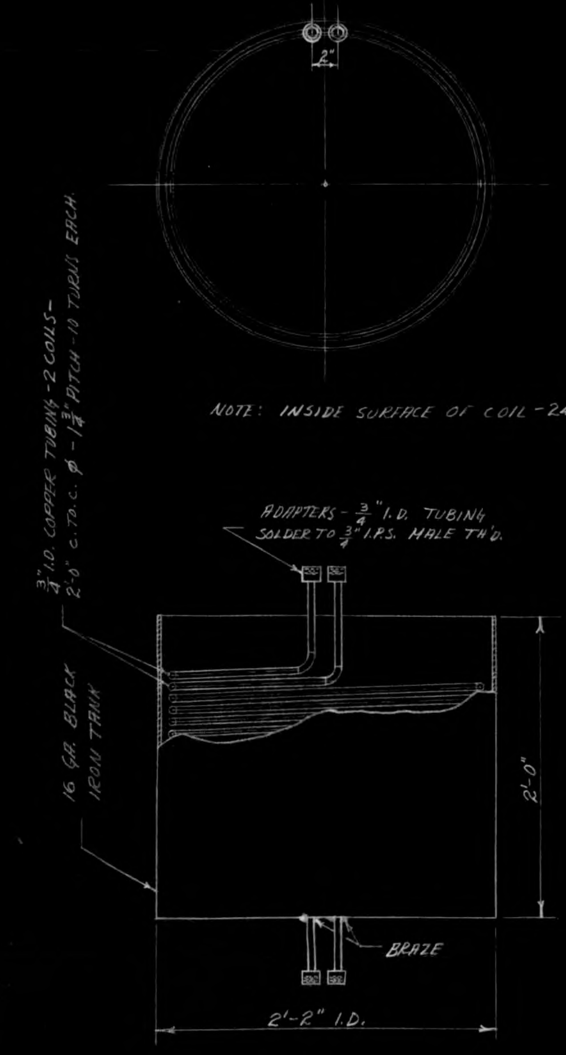
1. Turn "Reset" knob to its extreme clockwise position.
2. Switch on power to the line leading to the instrument. The motor will start.
3. Turn sampling line valve knob to admit fresh air. This knob is marked to indicate the passages through the valve and the following illustrations give the positions used:


Air Only

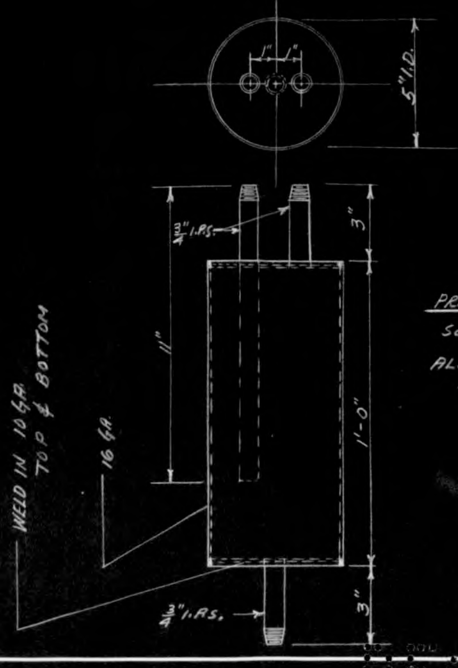

Gas Only


Gas and Air

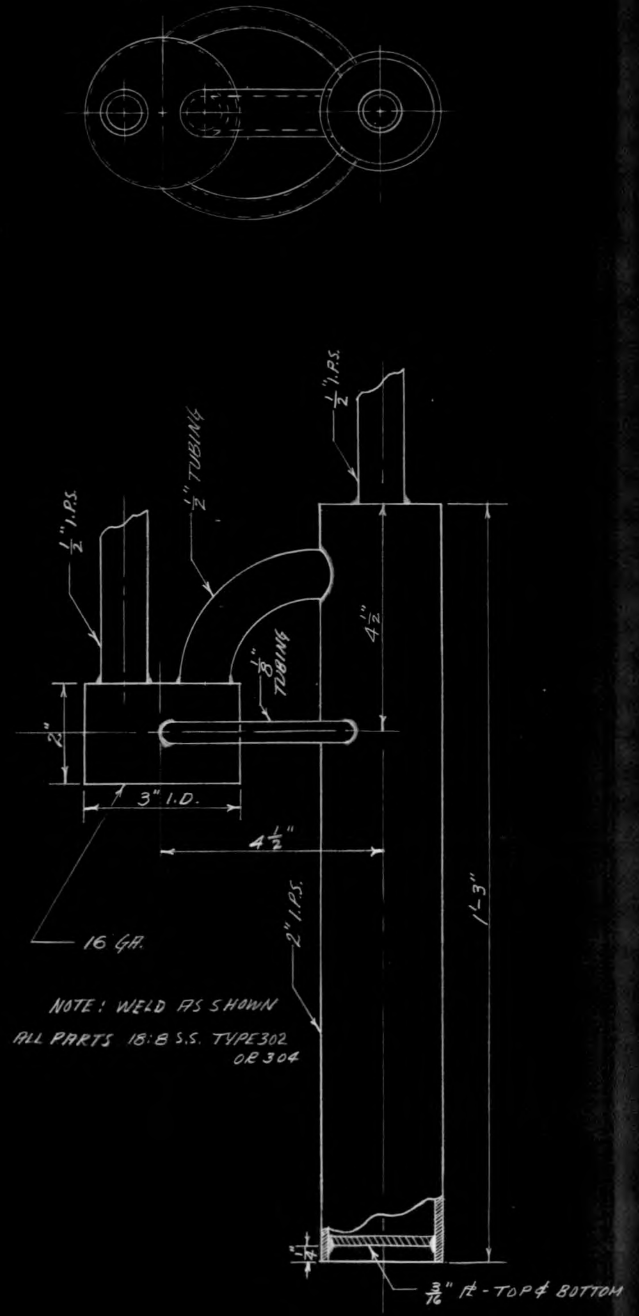
4. Slowly turn "Reset" knob in a counter-clockwise direction until the two white pilot lights glow.
5. Adjust zero: Turn "Zero" knob until sensitrol pointer is at either the a, b or c mark on the scale, depending upon the percentage of L.E.L. (Lower Explosive Limit) at which it is desired to set the alarm signal. The percentage of L.E.L. for each mark is given on the nameplate.
6. Turn "Reset" knob to its extreme counter-clockwise position.
7. Turn sampling line valve knob to admit gas only.
8. Danger Signal: When the white pilot lights go out, the red light goes on and the alarm sounds, it indicates that the concentration of gas has reached the limit for which the instrument has been set. The space from where the sample is being taken must be ventilated at once or the gas concentration reduced by other means.
   NOTE: The alarm also sounds if the detector unit burns out. This alarm signal can be differentiated from an alarm caused by a high gas concentration by the fact that the white pilot light will only glow very faintly when the instrument is reset and, of course, the danger signals will come into operation, even though the sampling line valve is turned to admit air only.
9. After thorough ventilation, turn sampling line valve to admit air only. Turn "Reset" knob in a clockwise direction until the sensitrol pointer is slightly to the right of the red line on the scale, and the white lights glow.
   NOTE: Reset knob should not be turned to its extreme clockwise position. After running on air for one or two minutes, the pointer should detach itself from the pin which was pushing it away from the contact and go back to where it was originally set. If after two minutes on air it does not go back, adjust by means of the "Zero" knob.
10. Reset instrument as per 6 and 7. Unless the concentration of gas has been reduced below the value for which the instrument is set, the danger signals will again come into operation and more ventilation is necessary.
11. The instrument can be completely shut down by turning the "Reset" knob to the extreme clockwise position.



PRECONDENSER SCALE: 1 1/2" = 1'

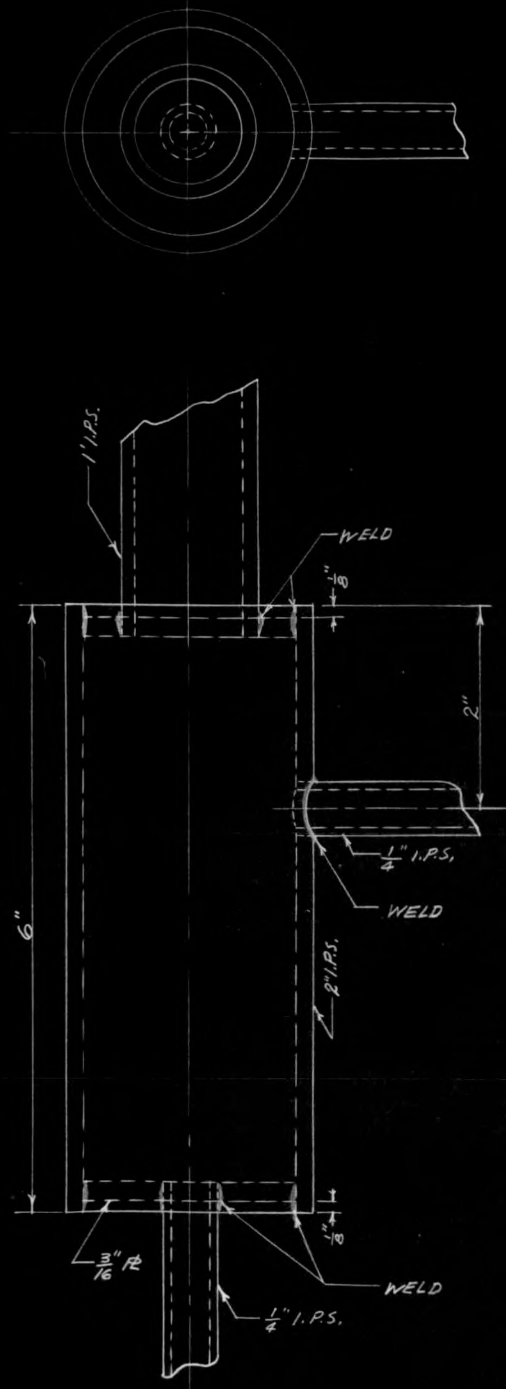


PRECONDENSER TRAP
SCALE: 3" = 1'
ALL PARTS 18:8 S.S. TYPE 302 OR 304

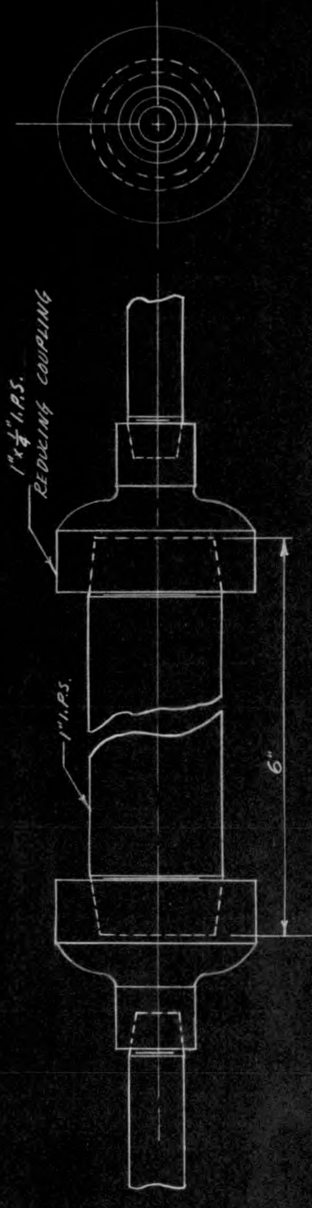


NOTE: WELD AS SHOWN
ALL PARTS 18:8 S.S. TYPE 302 OR 304

PRESSURE BLOWOFF
SCALE: 6" = 1'

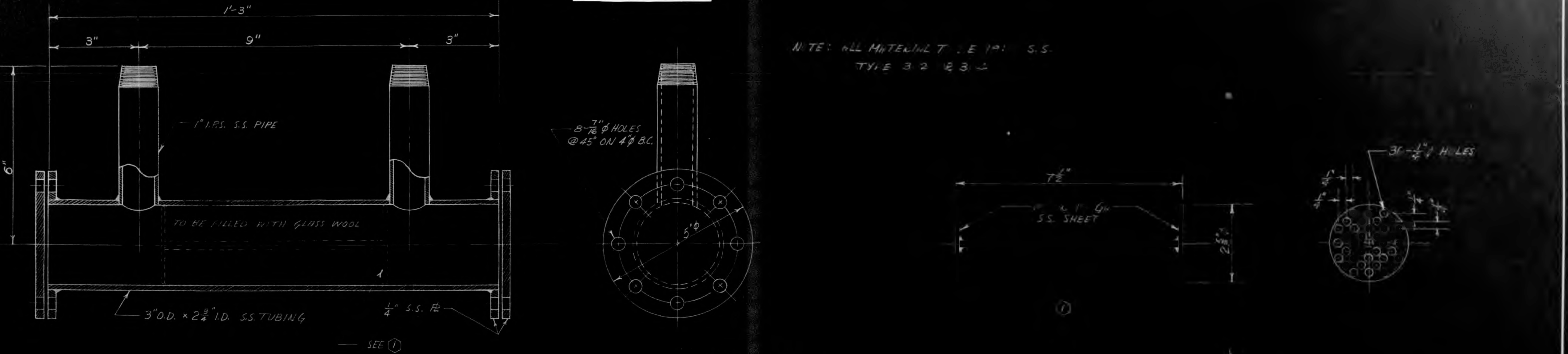


MIXING CHAMBER
FULL SCALE
ALL PARTS 18:8 S.S. TYPE 302 OR 304



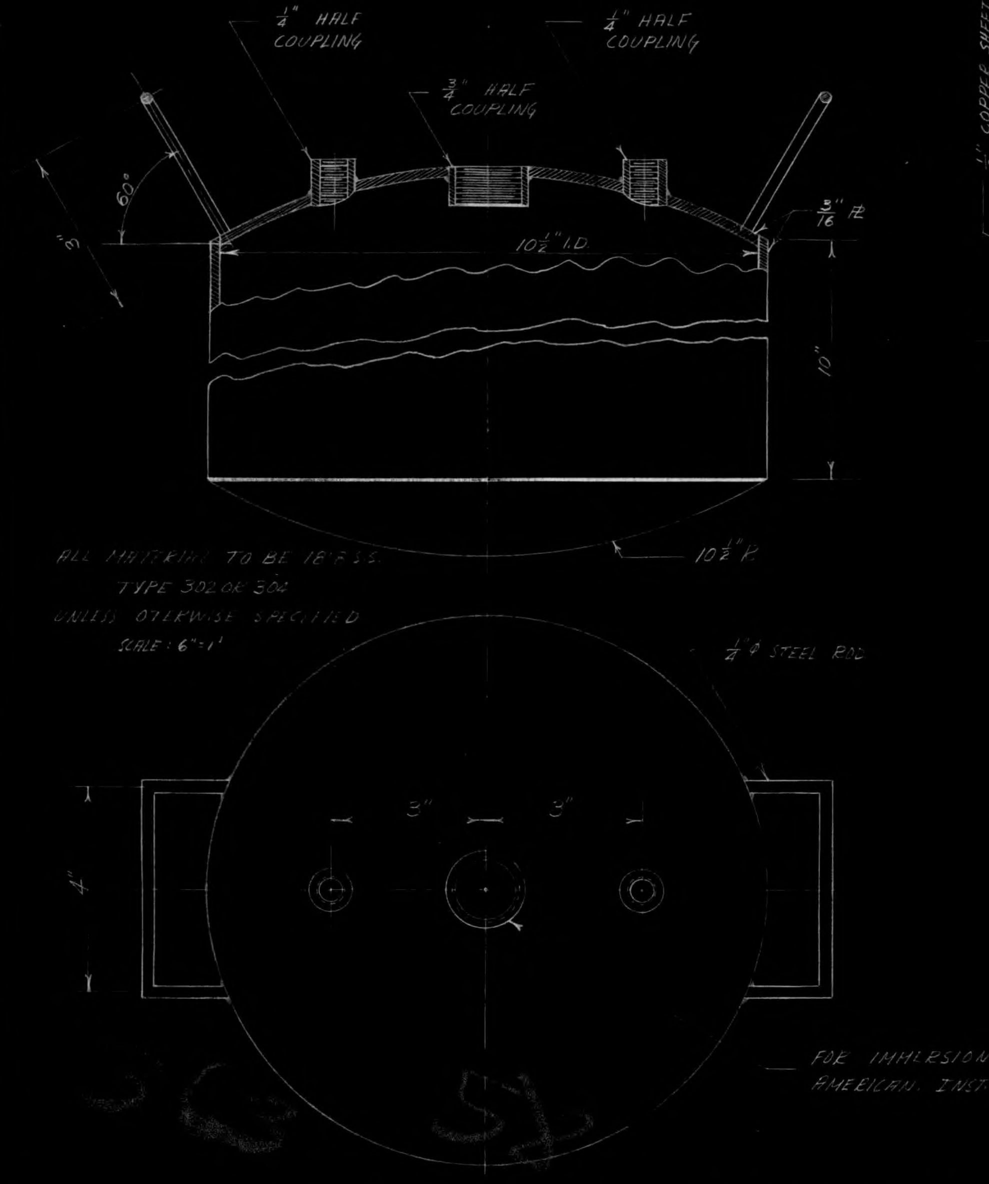
CHLORTHANE FILTER
FULL SCALE
ALL PARTS 18:8 S.S. TYPE 302 OR 304

REVISION	REFER. DWG.	AMERICAN CYANAMID COMPANY DEVELOPMENT ENGINEERING DIVISION STAMFORD, CONN.
	386-19-0	
	387-19-0	AUXILIARY EQUIPMENT STEP #4 - SHEET #2
		31-232-64
APPROVED		DESIGN: FWT SCALE: AS SHOWN DATE: 6-17-46
		DRAWN: FWT DWG. 391 JOB 19 REV. 0



FILTER-SS

SCALE: 6" = 1'



CHLORTHANE TANK

FREEZER

SCALE 1" = 1'

REVISION	REFER. DWG.
	386-19-0
	387-19-0
	396-19-0
APPROVED	

AMERICAN CYANAMID COMPANY
DEVELOPMENT ENGINEERING DIVISION
STAMFORD, CONN.

AUXILIARY EQUIPMENT
STEP #4 - SHEET #1

31-232-04

DESIGN: FWT
DRAWN: FWT
SCALE: 6" = 1'
DATE: 6-14-46

DWG.	JOB	REV.
390	19	

NOTES:

STRUCTURE TO HAVE $\frac{3}{4}$" PLYWOOD PANELS ON ALL SIDES & BOTTOM - TOP TO BE LEFT OPEN

AMERICAN CYANAMID CO.
STAMFORD, CONN.

SUB-ZERO CONDENSER
INSULATION

31-232-64

REF. DWG	REV.
240-19-0, 203-19-1	
387-19-0, 396-19-0	
	◇ 6-20-46

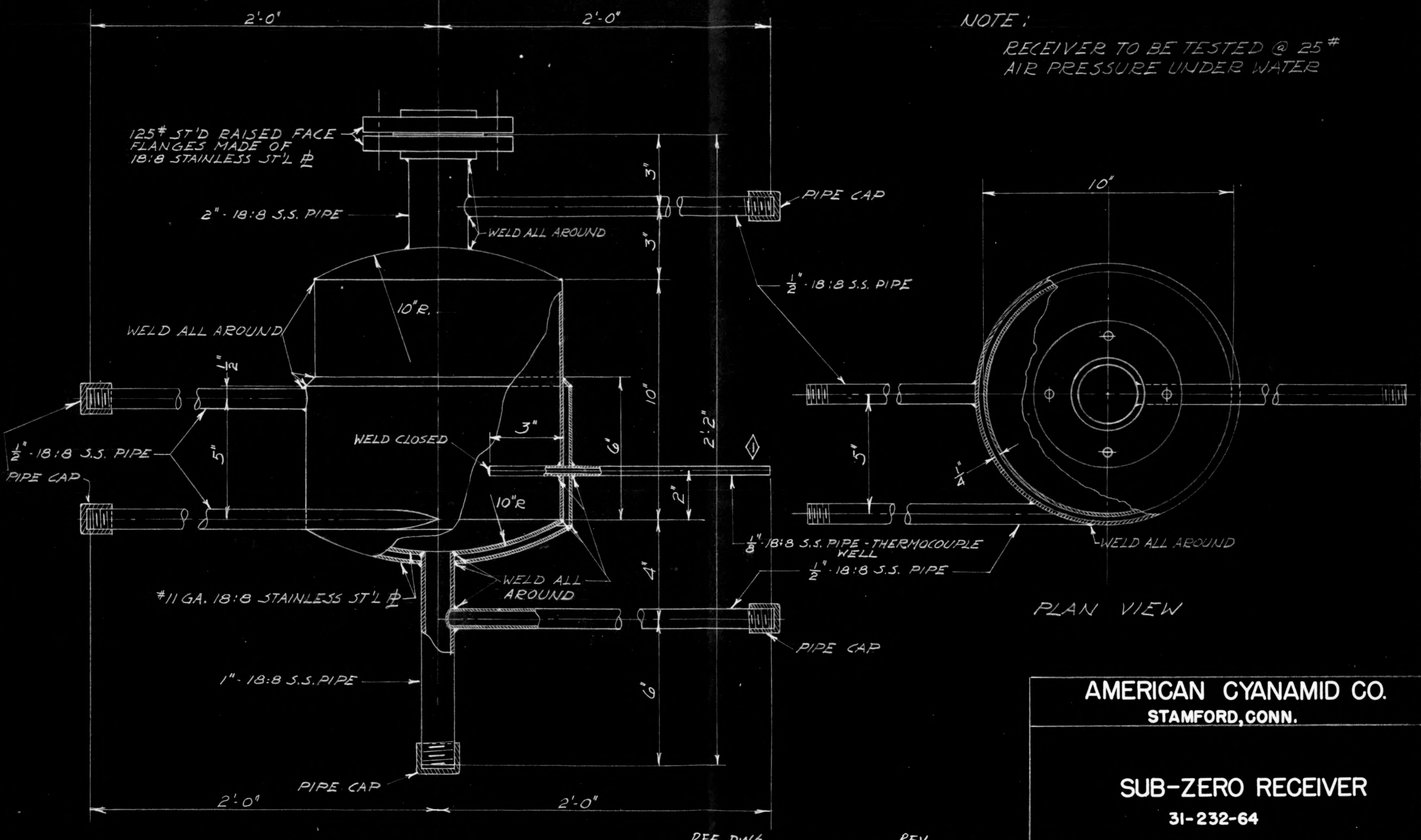
DESIGN: W.F.C.
DRAWN: M.S.
SCALE: 1" = 1 FT.
DATE: MAY 9, 1944

DWG.	JOB	REV.
208	19	1

FILE 9-2

362 59

DECLASSIFIED



NOTE:
RECEIVER TO BE TESTED @ 25# AIR PRESSURE UNDER WATER

NOTE: IRON PIPE CAPS TO BE PROVIDED AT EACH THREADED PIPE END

AMERICAN CYANAMID CO.
STAMFORD, CONN.

SUB-ZERO RECEIVER

31-232-64

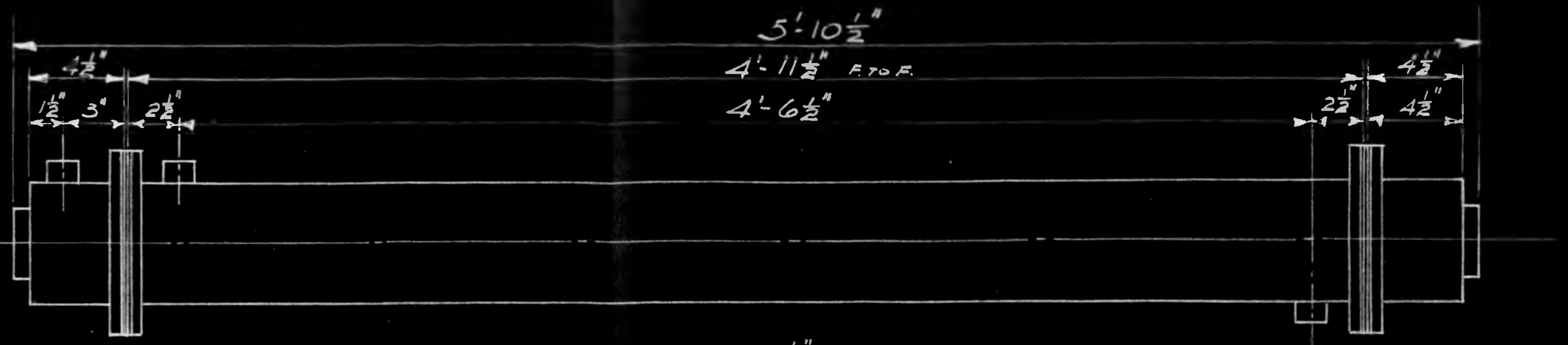
REF. DWG.	REV.
387-19-0	
396-19-0	
	REV. ◇1 MAY 8, '44

DESIGN: W.F.C.	DRAWN: M.S.		
SCALE: 3" = 1 FT.	DWG.	JOB	REV.
DATE: MAY 1, 1944	203	19	1

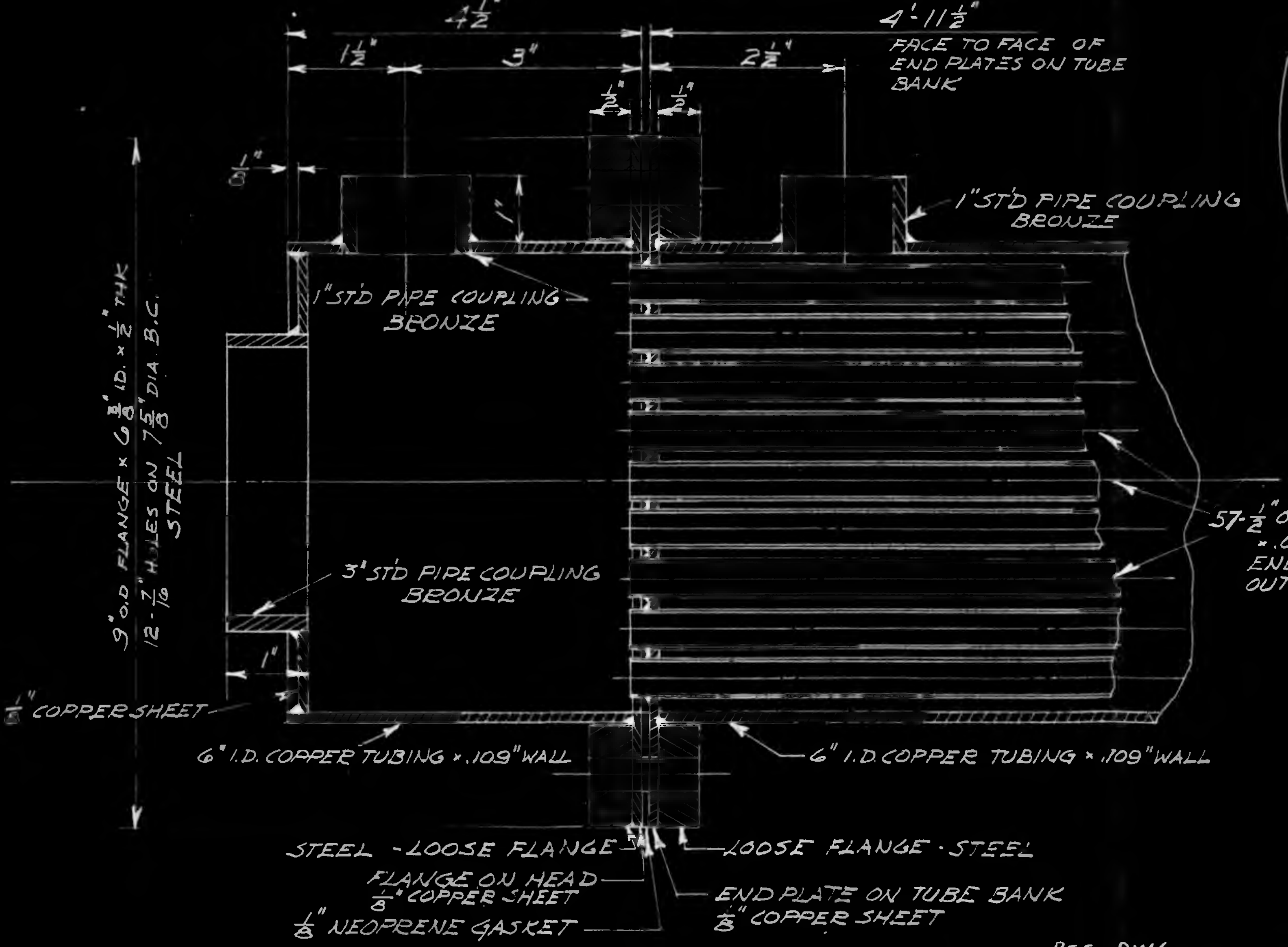
DECLASSIFIED

NOTE

OUTSIDE SURFACE OF ½" TUBES = 36.8 SQ. FT.

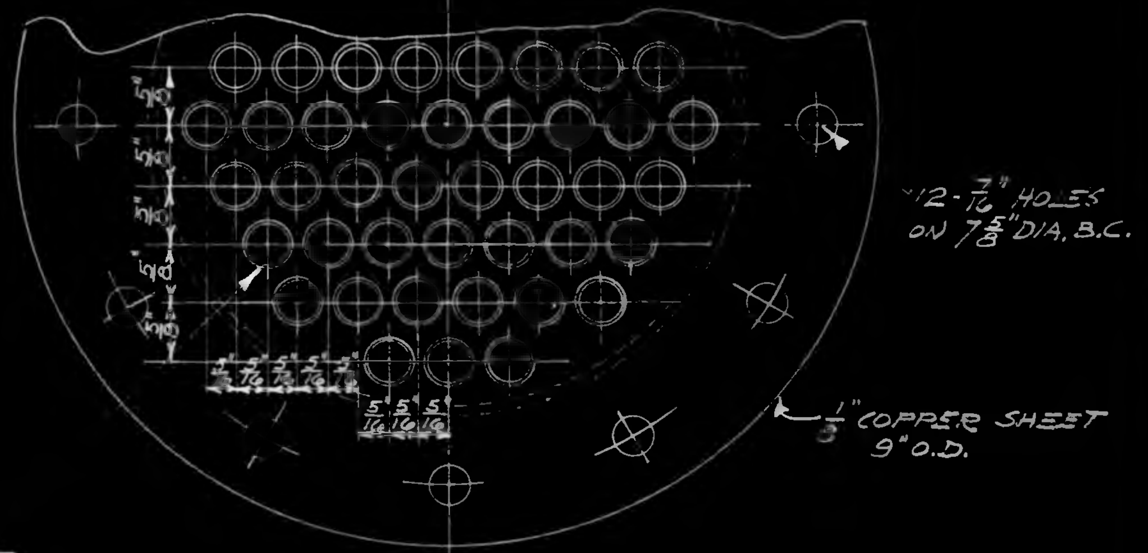


SCALE 1½" = 1 FT.



DETAIL OF ENDS

SCALE : HALF SIZE



DETAIL OF END PLATE ON TUBE BANK

SCALE : HALF SIZE

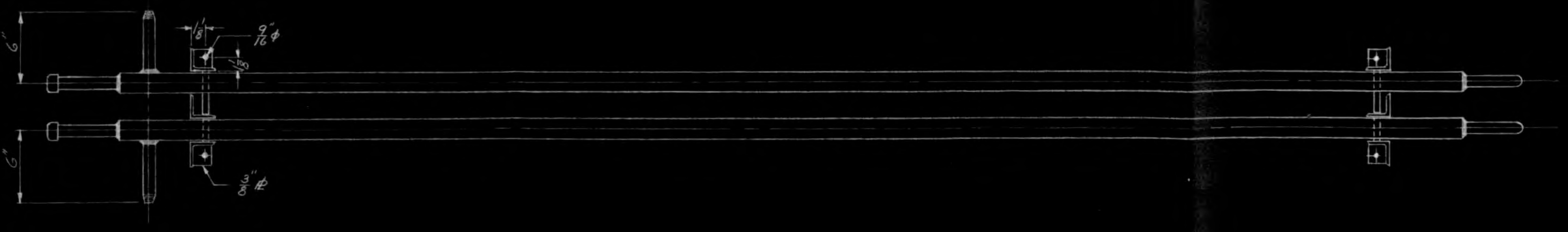
REF. DWG.	REV.
387-19-0, 396-19-0	

AMERICAN CYANAMID CO.
STAMFORD, CONN.

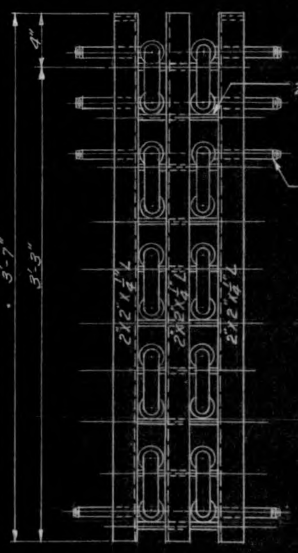
SUB-ZERO
CONDENSER
31-232-64

DESIGN:	DRAWN: M.S.		
SCALE: AS NOTED	DWG.	JOB	REV.
DATE: AUG. 3, 1944	240	19	0

FILE: 7



NOTE: TUBE-TURN 180° RETURN
BENDS MAY BE USED IF
DESIRED. SHORTER RADIUS
BENDS ACCEPTABLE.



1/2" BOLTS ON 4" CTRS.
TOP & BOTTOM BOLTS TO
HAVE PIPE SPACERS

CAP ENDS OF ALL NIPPLES

REF. DWG.	REV.
386-19-0, 387-19-0,	
396-19-0	

AMERICAN CYANAMID CO.
STAMFORD, CONN.

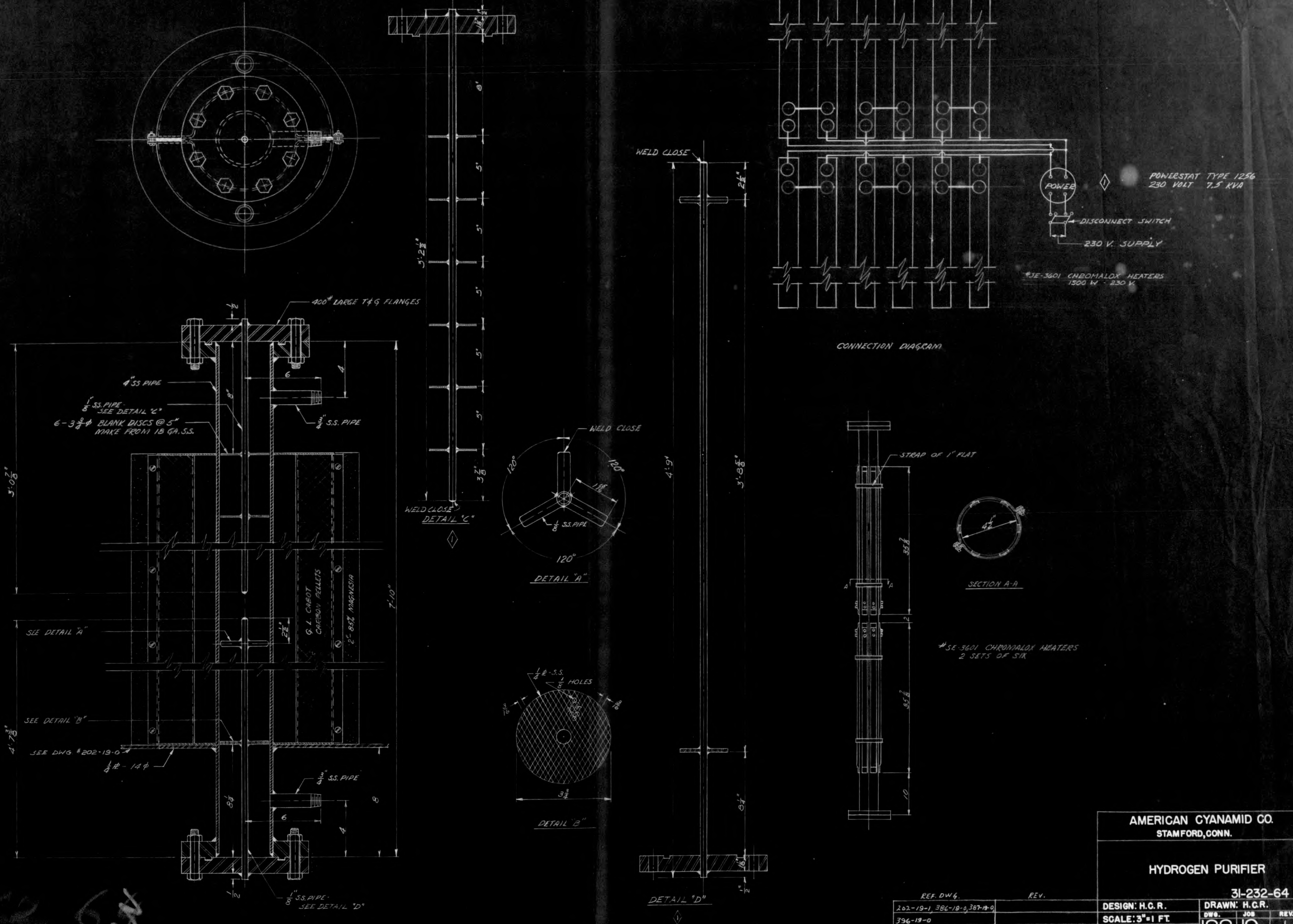
HEAT EXCHANGER

31-232-64

DESIGN: H.C.R.
DRAWN: H.C.R.
SCALE: 1-1/2" = 1 FT.
DATE: MAR. 3, 1944

DWG	JOB	REV.
185	19	0

FILE 9-2



AMERICAN CYANAMID CO.
STAMFORD, CONN.

HYDROGEN PURIFIER

31-232-64

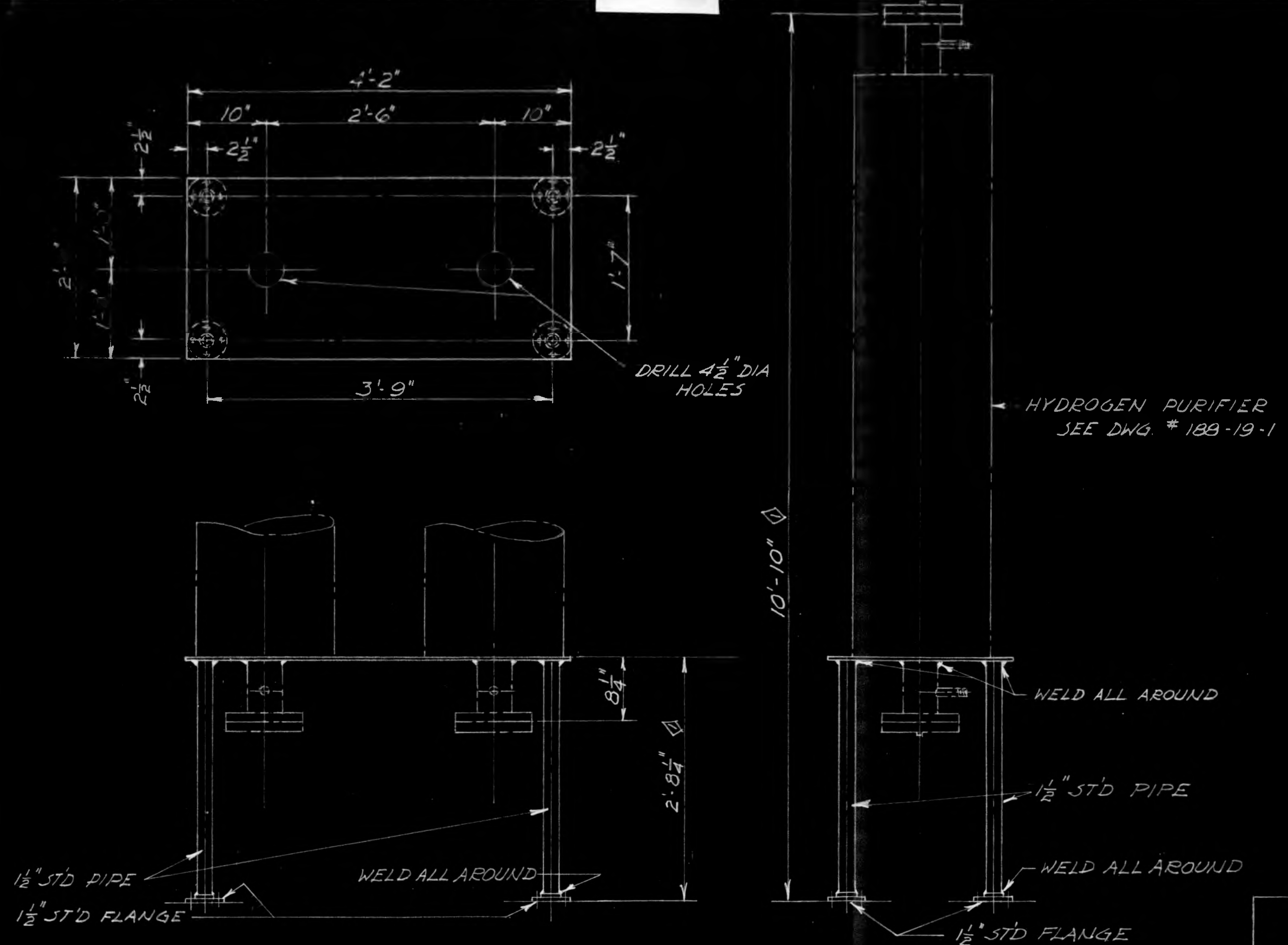
REF. DWG.	REV.
202-19-1, 386-19-0, 387-19-0,	
396-19-0	
	REV. 1 APRIL 28, 1944

DESIGN: H.C.R.
DRAWN: H.C.R.
SCALE: 3" = 1 FT.
DATE: MAR. 16, 1944

DWG.	JOB	REV.
188	19	1

202.6

64



REF. DWG.	REV.
188-19-1, 386-19-0, 387-19-0, 396-19-0	◇ 5-1-44

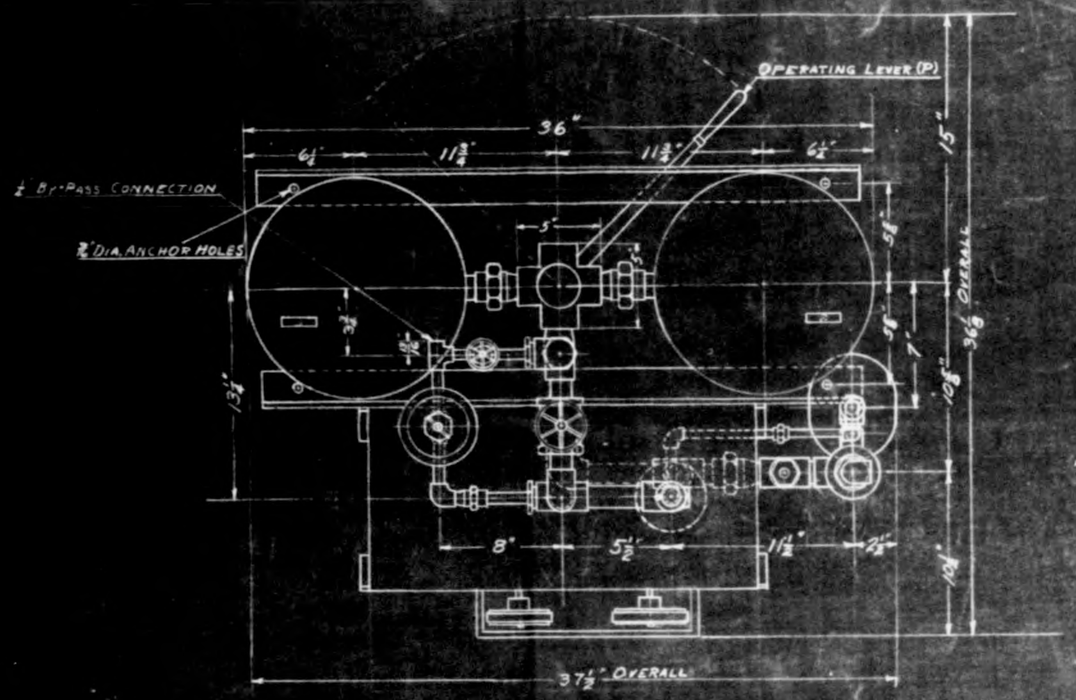
AMERICAN CYANAMID CO.
STAMFORD, CONN.

SUPPORT FOR HYDROGEN PURIFIER

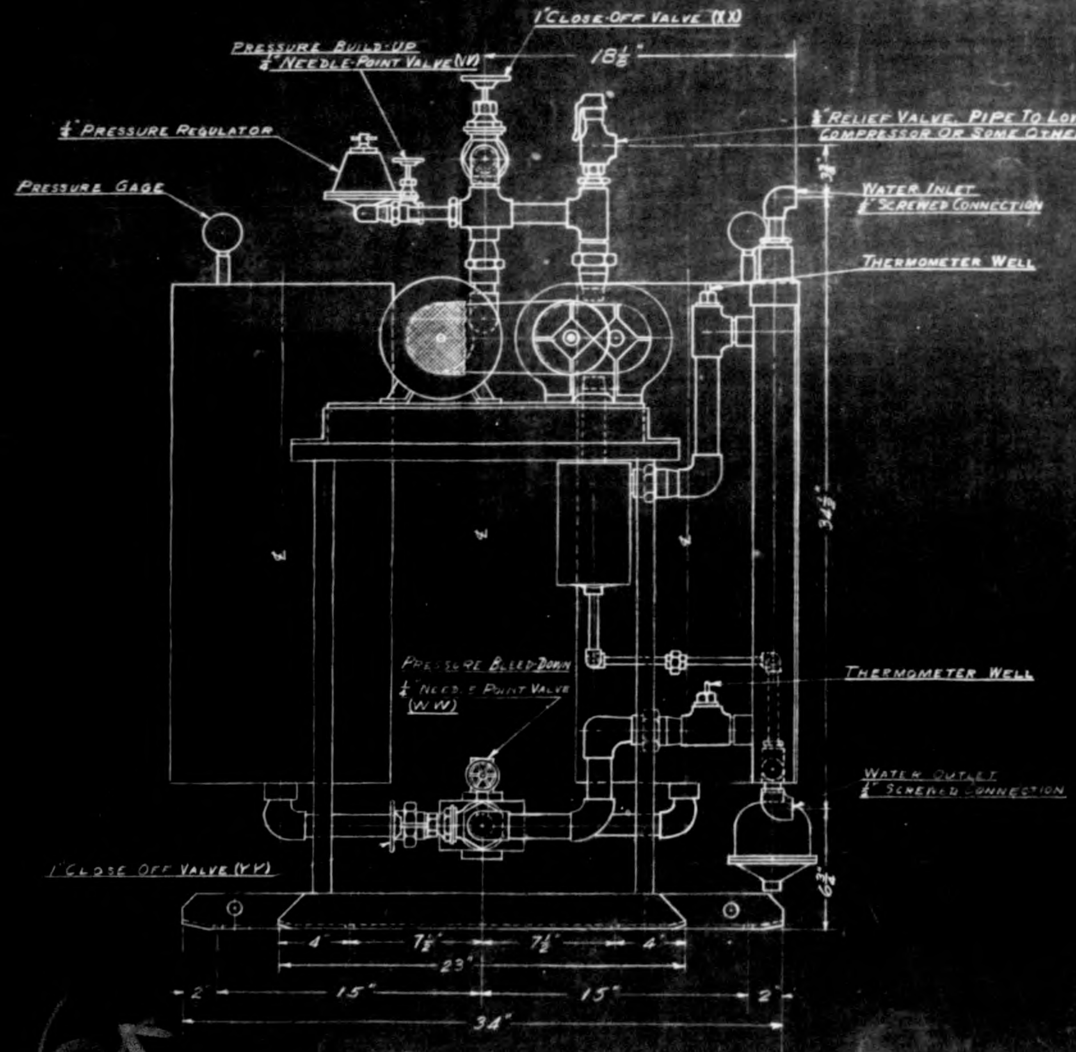
31-232-64

DESIGN: H.C.R.	DRAWN: M.S.		
SCALE: $\frac{3}{4}$ = 1 FT.	DWG.	JOB	REV.
DATE: APRIL 29, 1944	202	19	1

64

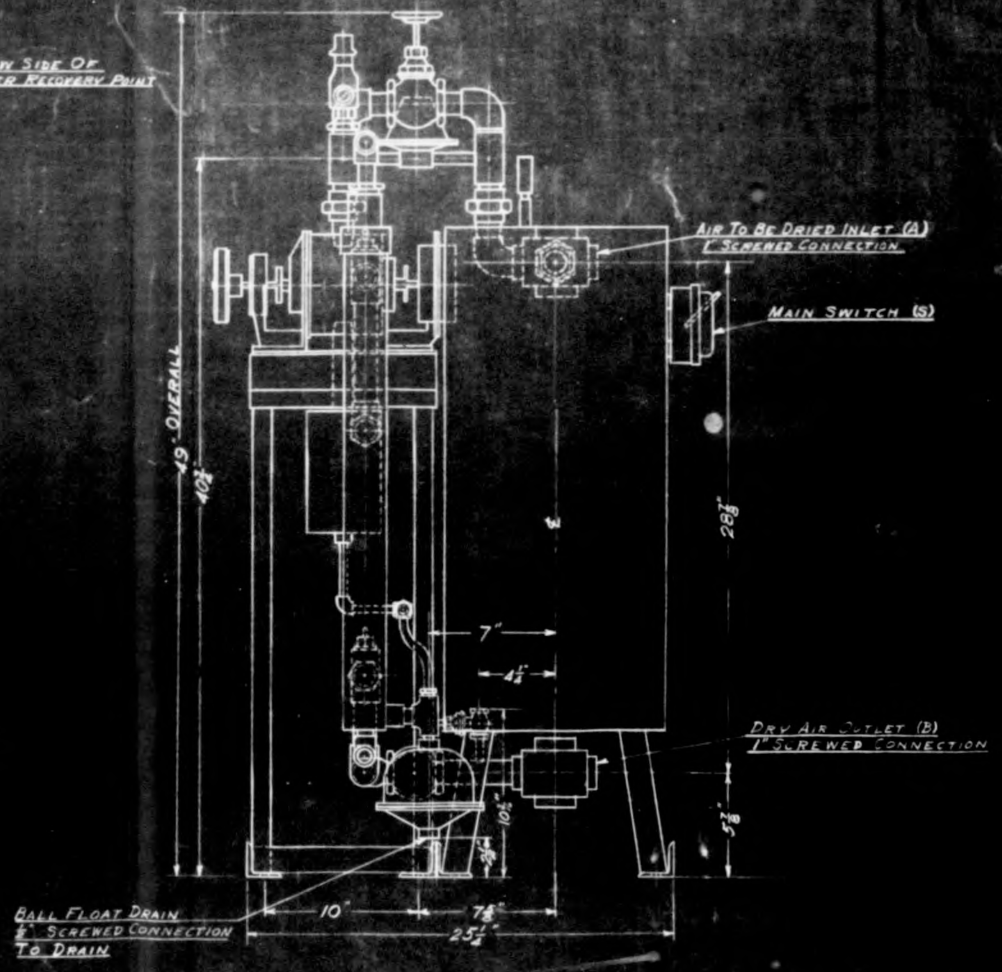


TOP VIEW



65

REAR VIEW



SIDE VIEW

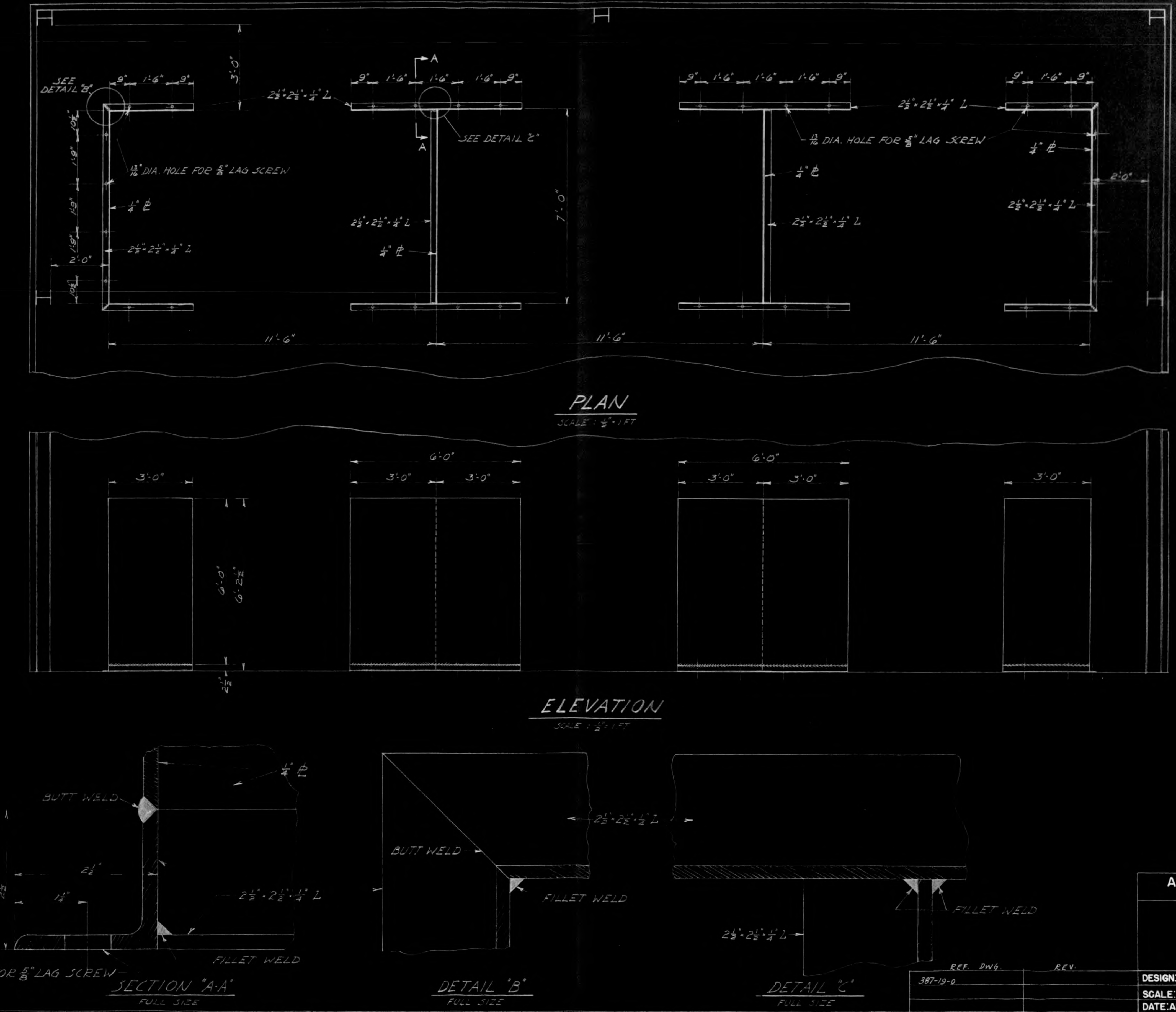
PITTSBURGH LECTRO-
DRYER CORPORATION
PITTSBURGH U.S.A.

DIMENSION DRAWING FOR
BAC-50 LECTRODRYER WITH
INTERCOOLER
25 LBS WORKING PRESSURE

DRAWN 4-6-44 R.W.B. CHECKED
TRACED APPROVED
BILLS OF MATERIAL
SCALE 3"=1'-0"

1736 B

23393 D 4-22-44



3266

REF. DWG.	REV.
387-19-0	

AMERICAN CYANAMID CO.
STAMFORD, CONN.

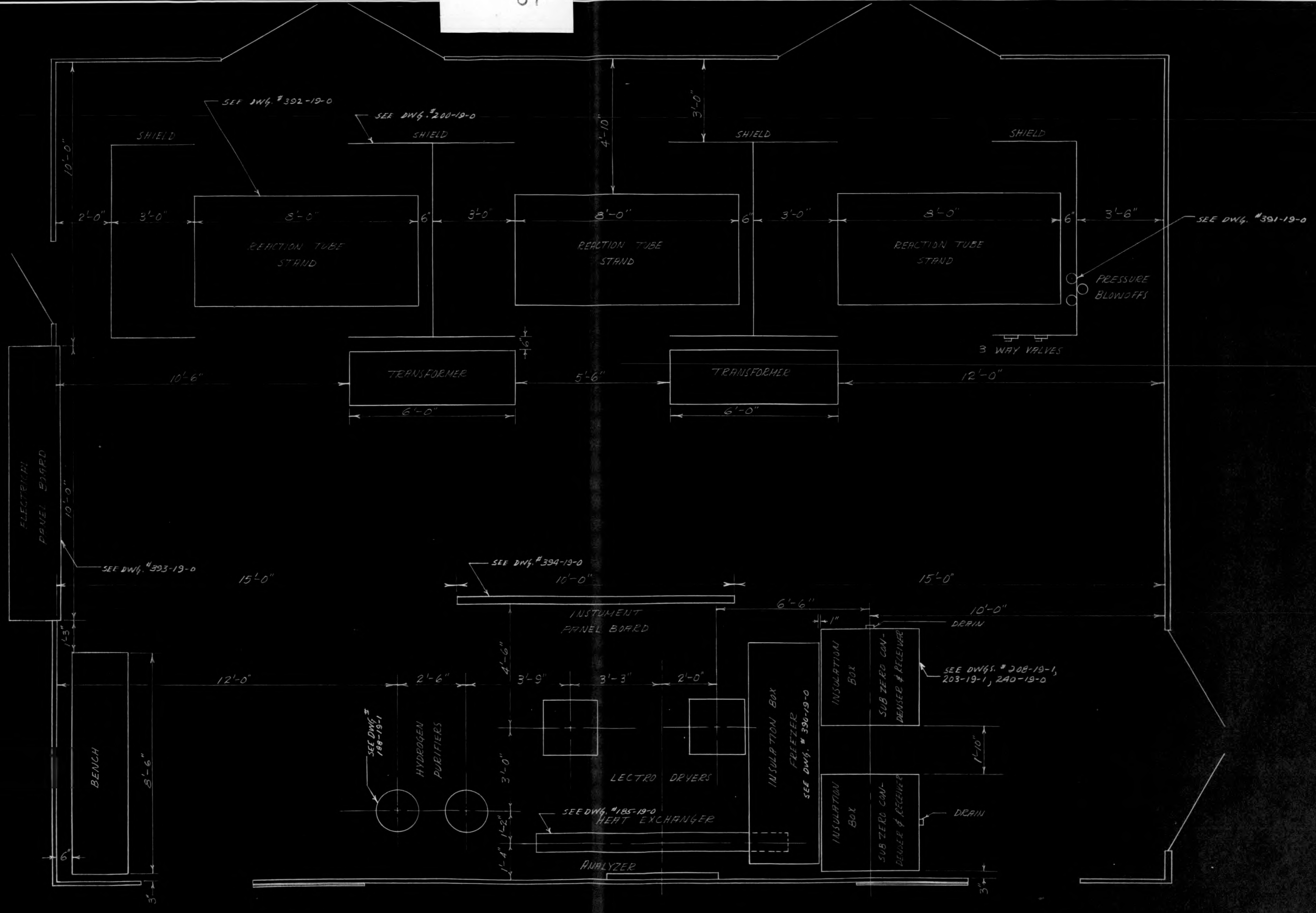
SAFETY SHIELDS

31-232-64

DESIGN: M.S.
DRAWN: M.S.
SCALE: 1/2" = 1FT.
DATE: APRIL 25, 1944

DWG.	JOB	REV.
200	19	0

FILE 92



REVISION	REFER. DWG.
	266-19-0
	384-19-0
	386-19-0
	396-19-0

APPROVED

AMERICAN CYANAMID COMPANY
DEVELOPMENT ENGINEERING DIVISION
STAMFORD, CONN.

FLOOR PLAN
STEP #4

31-232-64

DESIGN: FWT
DRAWN: FWT
SCALE: 1/2" = 1'
DATE: 6-13-46

DWG.	JOB	REV.
387	19	0

67

Grinding and Separation Operations

Grinding and Separation Operations

1. Introduction

The 891 produced on the TAW wire in Step 4 is cracked off the wire manually. The TAW core[5,6], containing a small percentage of 891 (approximately 5%), is saved for recovery (see section on Recovery of 891 from Wire). The pieces of 891 are separated by hand into two groups - those with some TAW attached and the clean pieces. The two groups are separately passed through a Sturtevant Roll Crusher and screened through Tyler standard screens until 100% passes 100 mesh and 90% passes 200 mesh. The grinding operation adds 2-2-1/2% iron from the rolls of the crusher. The 200 mesh material obtained from grinding the clean pieces of 891 is then passed through a Stearns Magnetic Separator to remove the iron. The non-magnetic fraction should meet specifications for shipment, namely 98% 891A and less than 0.3% Fe. The magnetic fraction is treated with hot 6 N hydrochloric acid to remove the iron. The 200-mesh dust obtained from grinding the 891A (collected from the vacuum cleaner) is treated with hot 6 N hydrochloric acid. If these two dust fractions pass specifications, they are added to the non-magnetic fraction above.

The attachments containing TAW are ground in a similar manner as the clean 891A pieces except that the 200 mesh material is acid-treated with hot 6 N hydrochloric acid and then subjected to two heavy media separations. The "float" from the second separation should pass specifications for shipment. The "Middlings" and "Sink", containing most of the TAW, and the TAW wire core are chlorinated with gaseous chlorine to recover the 891A as Chlorthane, which in turn is redistilled and passed through Step 4.

The equipment used in the Grinding step is located in the middle room of the southern section of the building. The Flow Sheet for the process is shown in Figure 326-19-0. When TAW wire was substituted for the T wire used originally, there were less attachments of 891A to the wire core, thus resulting in less attachments to work up in some rather involved separation procedures. All steps for the T wire are shown in the flow sheet, but the dashed lines indicate those that were eliminated by use of TAW alloy wire.

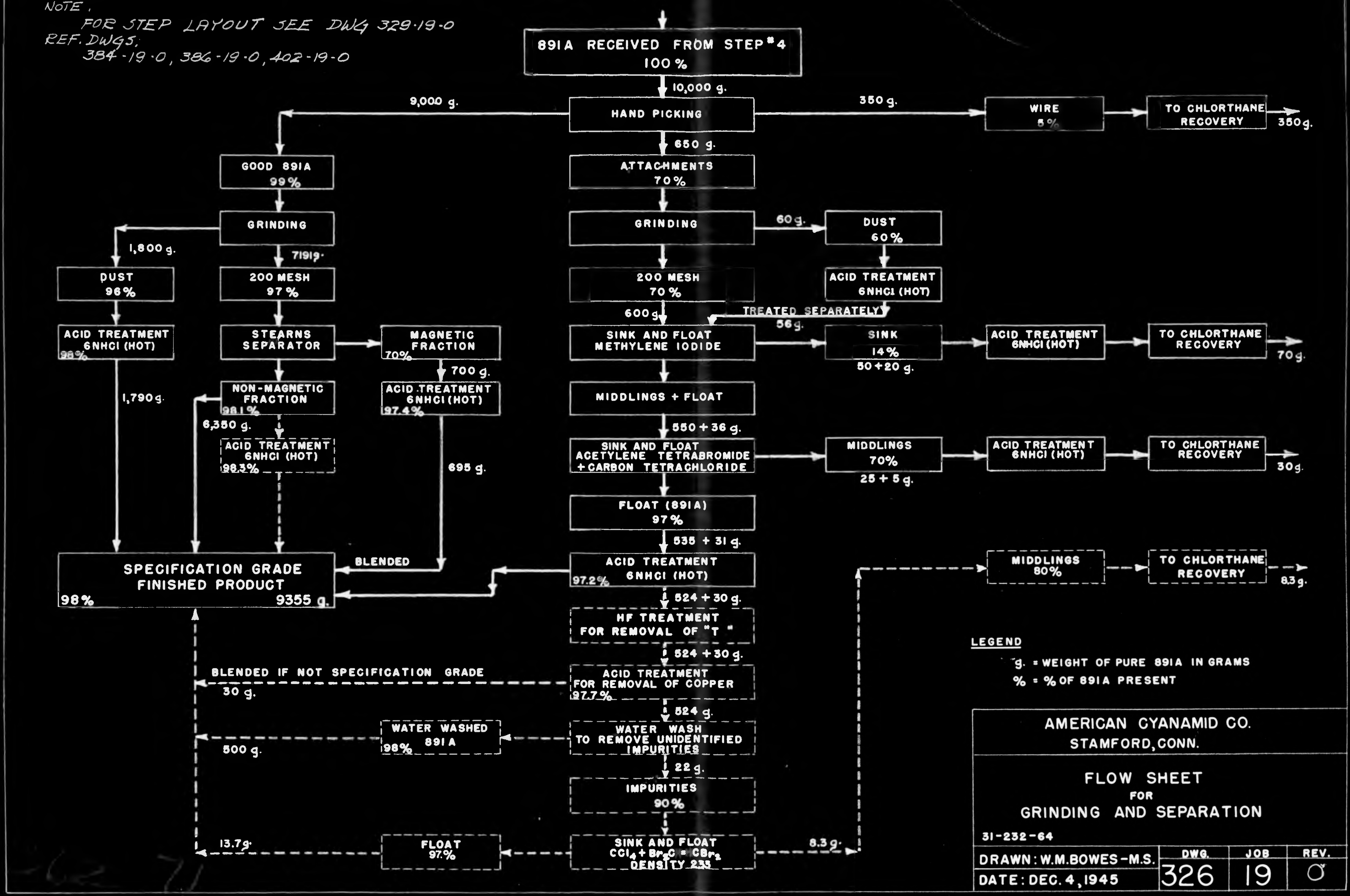
2. Hand Picking the 891A from Step 4

The 891A as received from Step 4 is deposited on the tantalum-tungsten alloy wires and must be removed by hand before the grinding operation. The wire and 891A is placed in a metal tray (Fig. 411-19-0, p. 710). To remove the core the material is broken off the wire by bending. Canvas gloves are worn to protect the hands during this operation. In most cases merely bending the wire was sufficient to crack off the 891A without damaging the wire or causing formation of attachments to an abnormal degree. Where the 891A was not easily broken off the wire, pliers had to be used to strip off the 891A. When the wire was not broken and no attachments were present, the 891A was collected and weighed.

If considerable attachments were present, each piece of 891A had to be carefully "inspected" to separate the good pieces of 891A from the attachments. Scoops and tweezers are needed for collecting the 891A and attachments.

The "attachments" obtained from the above procedure are treated according to producers outlined under grinding, acid treatment and sink and float. If there were only a few attachments from

NOTE,
FOR STEP LAYOUT SEE DWG 329-19-0
REF. DWGS.
384-19-0, 386-19-0, 402-19-0



LEGEND
g. = WEIGHT OF PURE 891A IN GRAMS
% = % OF 891A PRESENT

AMERICAN CYANAMID CO.
STAMFORD, CONN.

FLOW SHEET
FOR
GRINDING AND SEPARATION

31-232-64

DRAWN: W.M.BOWES-M.S.	DWG.	JOB	REV.
DATE: DEC. 4, 1945	326	19	0

each run and time permitted, the TAW in the attachments was broken away from the 891A with the pliers.

The wire that was separated was stored in bottles for the wire recovery operation (see p.730) for recovery of the dissolved or attached 891A on the wires. The 891A so recovered as Chlorthane was distilled and then reprocessed in Step 4.

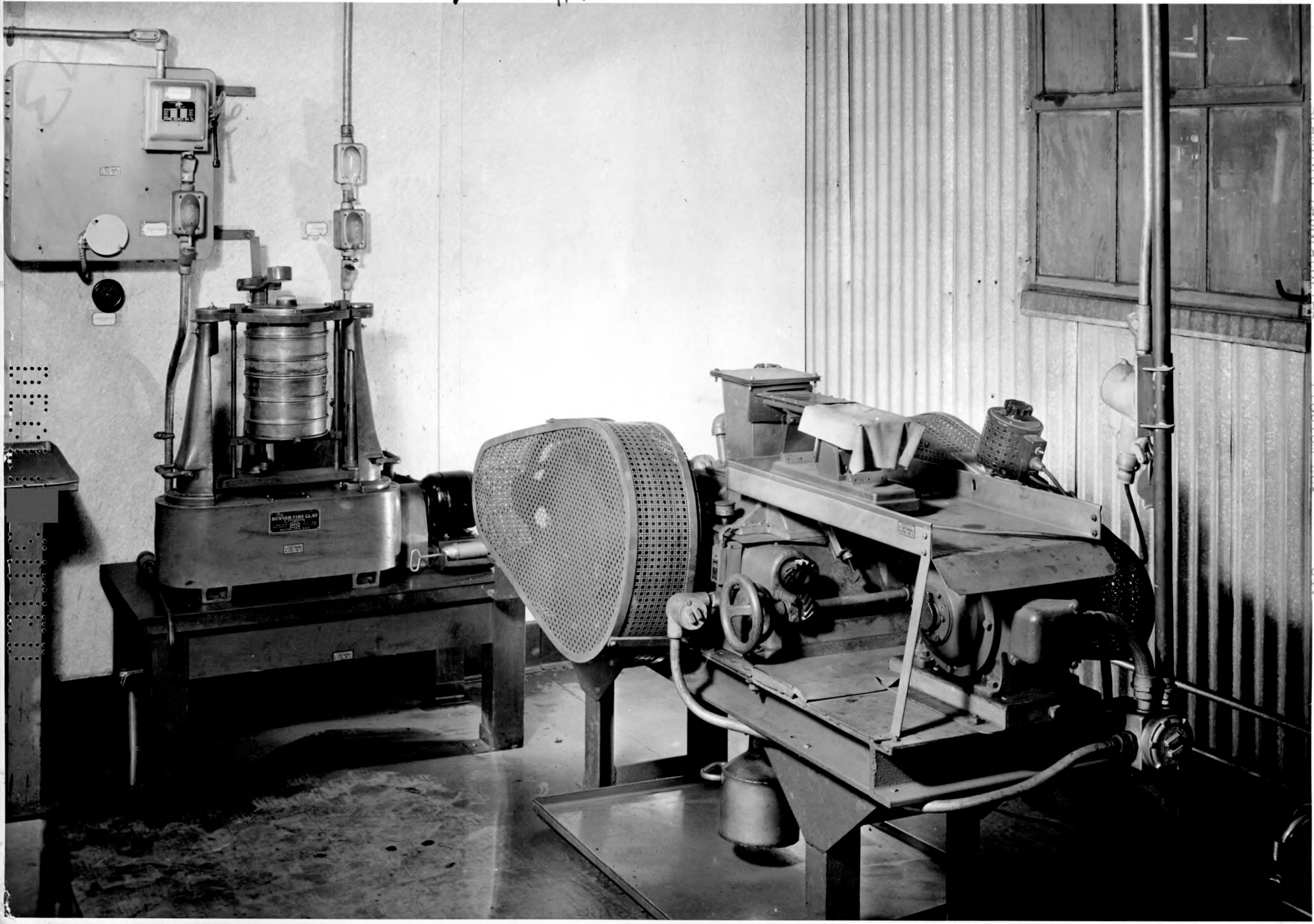
Care should be taken in removing the 891A from the core since sharp splinters often fly. Safety goggles should always be worn to prevent getting splinters into the eyes and gloves also should be worn at all times.

3. Grinding

The grinding is done in a Sturtevant Mill Crusher, shown in Dwg. 218-16, p.704. The charge of hand-picked 891A to be used was weighed and then placed in the vibrating feeder. The mill was started by throwing the starter switch on the panel board. If the rolls did not rotate immediately, the switch was shut off and the rolls adjusted so that they did not touch each other. The mill was restarted. The rolls were drawn together until they just touched, then backed off 3/4 of a turn. This was the proper setting for the first grind of the 891A. If the rolls were any closer it was found that it wore the rolls excessively, thus adulterating with more iron. Before the feeder was started, the rolls were clamped to prevent any change in the setting. The clamp is located on the far end of the shaft connected to the adjusting wheel. Next the Variac for the vibrating feeder was adjusted so that the 891A was fed at a moderate rate. Too rapid a feed increased the wear on the rolls. The vacuum cleaner was started and the valve adjusted to control the amount of air-intake.

363-73

Photo #12



If dust appeared around the feed hopper, the valve was opened a little wider. Occasionally a large piece of material (891) will catch on the rolls and start to scratch them. When this occurs, it is important to try to release it by opening the rolls wider or charging the material a little faster until it is crushed. If it cannot be freed, then the machine is stopped and the cover lifted up, and the piece removed. A fast feed rate will not increase the amount of grinding and may cause choking of the rolls which will stop the machine, so care should be exercised in choosing a feed rate that is most suitable.

If dust seeped out of the feed hopper at any time, the air control valve on the vacuum cleaner was opened further. When the valve was opened wide and dust still appeared, the feeder, rolls and vacuum cleaner were stopped. The cloth bag in the vacuum cleaner was replaced with a clean one. The dust collected from the bag was screened to remove the coarse materials.

The 891A having passed through the rolls once was collected from the receiver under the grinder and a new receiver was put in its place. The material from the receiver was placed in the feed trap. The rolls were reset to a point about 1/4 turn on the adjusting wheel from the point where they were touching each other. Then the adjusting wheel was clamped.

The above procedure was repeated several times until all the large pieces were crushed. As the material became finer the wheel which controls the gap between the rolls was tightened until the rolls were touching. After every third or fourth pass through the mill, the material was screened as described in the section below on "Screening-'Rotap'". Each screening removed some 200 mesh material. When all the material had been ground to 100 mesh or better, the rolls were placed under pressure by tightening the wheels until the blocks at the side of the machine had a gap of 1/16 inch in order to get maximum crushing power. When all but a small amount of material, 500 g. or less, had been ground to 200 mesh, it was saved and added to the next batch. If the machine was used to grind attachments, then it was cleaned out with the vacuum cleaner, both inside and out. All trays and vacuum bags were also cleaned. If the machine was used for grinding clean 891A, it was cleaned out after every third batch.

For care of the machine both during grinding and after each run, see the section on Maintenance, p.703.

4. Screening - "Rotap".

A 100 and a 200 mesh screen were placed in series on top of the Rotap receiver, and approximately 200 g. of crushed 891Awas charged to the 100 mesh screen. After the cover and top plate had been put on, the set was securely fastened in position on a Tyler-Rotap Screen Shaker (Dwg. R-211, p.707). The tapping arm was placed on top of the top plate and the machine turned on.

After fifteen minutes the machine was stopped and the screens removed. All the material which did not pass the 200 mesh screen was removed and recharged to the Sturtevant mill. The material passed the 200 mesh screen and was collected for the next step of magnetic separation of iron. The purpose of the 100 mesh screen is to prevent clogging of the 200 mesh screen. Also, it was allowable to have in the final product 10% of material which passes the 100 mesh screen but not the 200 mesh screen. Since some of the 891A was removed in later operations, a maximum of 8% was added to the 200 mesh material. The 200 mesh material plus the 8% of

100-200 mesh material was ready for the next step described below.

The dust collected from the vacuum cleaner during the grinding operation was similarly screened to remove the coarse material. By experience it has been found that this dust "Rotapped" very slowly. Also, it contained less than 5% of 100-200 mesh material, so after Ro-tapping for 20 minutes it was found that the 100 mesh and 200 mesh material could actually be combined as 200 mesh material. Moisture collected on the very fine dust particles and prevented them from passing through the 200 mesh screen. In fact moisture also prevented efficient magnetic separation of the iron from the 891A dust, so another procedure had to be used with the clean 891A dust, as described in the section entitled "Acid Treatment".

After each screening operation it was necessary to clean the screens by brushing both sides of the screen with a stiff brush. The screen was then turned over and the sides of the screen were tapped to clean off the 891A caught in the holes of the screen. If this was not done some of the 200 mesh material was not screened out. This meant that the 200 mesh material was re-ground and the amount of dust obtained in the vacuum cleaner greatly increased.

5. Stearns Magnetic Separator

A Stearns Magnetic Separator is used to remove the iron added during grinding of the clean pieces of 891A. This machine and the various adjustments on it are illustrated in Dwg. C-731, p.709. The 891A is placed on an inclined vibrating tray on the machine which feeds the 891A and causes it to pass near the pole of an electro-magnet. The magnetic particles (Fe) are attracted to the pole and fall into a differchute than to the non-magnetic particles (891A) which are not deflected from the normal vertical downward growth.


Photo

The method for feeding the machine was to first turn on the current on the vibrating feed tray and then set the leveling vane at the end of the feed tray until it almost touches the vibrating tray. Next enough 891A was added to the tray to fill the section behind the vane. The flow was adjusted so that a small thin layer of material flowed under the vane. If the rate of flow was too slow, it was increased by two methods. The first was to increase the gap between the vibrating tray and the leveling vane. The second was to increase the amplitude of the vibrations of the feed tray by advancing the Variac controller. It is recommended that the amplitude of the vibrator be kept at a minimum.

During operation the pole of the magnet became coated with iron particles sufficiently to interfere with the separation of the magnetic particles. When this condition occurred, it was necessary to release the iron by stopping the machine. Automatically the dividing vane which split the material into magnetic and non-magnetic fractions was released and moved to a position such that all the material passed into the magnetic fraction. The iron which had been collecting on the nose of the magnet pole piece fell off and dropped into the magnetic chute fraction.

After all the material had been separated once, the non-magnetic fraction was recycled two times without shutting off the magnet. The final non-magnetic fraction should pass specifications. The magnetic fraction was also passed three times through the Stearns Separator and the 891A recovered added to the next batch to be separated. The final magnetic fraction which contained between 10-25% iron was then subjected to acid treatment as will be described in the next section.

6. Acid Treatment

The purpose of acid treatment was to remove all iron present in the 891A. As was often the case iron could not be removed completely by mechanical means and had to be treated with 6 N hydrochloric acid. The dust from "good 891A" was always acid treated, since the Stearns Magnetic Separator would not give a sharp separation because of electrostatic charge causing agglomeration of iron and 891A. The Middlings, which were to be recovered by chlorination, had to be free of iron, since iron chloride plugged the reactor tube. "Sink" fractions likewise were acid treated to remove iron before the material was chlorinated. The attachments would not pass the 98% specification for 891A; therefore the material was acid treated first to take out the iron. This material was then subjected to Sink and Float heavy media separation. When, on occasion, the non-magnetic fraction did not pass specifications, this acid treatment removed all iron and increased the % 891A up to specification. This did not have to be done when the fraction passed specifications.

The procedure for acid treatment of any of the above materials was substantially the same in all cases. The dust (not over 1000 g.) or other material was put in a three-liter Erlenmeyer flask, which was surrounded by a metal receiver. Approximately 500 cc. of 6 N acid was added. Without stirring the material was allowed to react slowly. When bubbling had subsided the mixture was stirred and more acid was added. Enough acid was added to leave 100% excess after the iron had reacted.

When foaming in the flask finally ceased, the flask and metal receiver were placed on a hot plate and the mixture was heated to

90-100°C. A stirrer was placed in the flask to keep the 891A in suspension during the acid treatment. It was usually found necessary to heat the material for 6-8 hours to assure complete removal of the iron. The flask was cooled and the 891A filtered off to remove the acid. The 891A was washed with several volumes of water to remove the acid, then washed with 2B alcohol. The washed material was dried in the oven at 110°C. for 12 hours. The clean 891A dust should pass specifications for shipment. The acid-treated magnetic fraction also should pass specifications. The attachments after acid treatment must be subjected to the procedure described in the section entitled "Sink and Float" in order to increase the 891A content above 98%.

7. Sink and Float Separation of 891A.[5]

The Sink and Float purification was usually performed on a composite of materials obtained from three sources: (1) 200 mesh material from the grinding of 891A with attachments; (2) the 200 mesh dust obtained from the vacuum cleaner which was collected during grinding of TAW-891A attachments; (3) the so-called "Middling" fraction from previous Sink and Float operations. These three were subjected to acid treatment with hot 6 N hydrochloric acid before purification by "Sink and Float". The purification was carried out in two stages. In the first a medium of methylene iodide (density = 3.31 g./cc.) was used to separate part of the tantalum and tungsten with a little 891A included as the "Sink" fraction. In the second step a mixture of acetylene tetrabromide and carbon tetrachloride (d = 2.35 g./cc.) was the medium. The rest of tantalum and tungsten along with some 891 sinks in this separation and is called the "Middlings". The "Float", after drying, should consist of 891A meeting specifications. The "Middlings" were added to the next sink and float batch, while the first "Sink" was kept for recovery. These separations were carried out in a centrifuge as shown in Photo. 14). Details of the operations are given below.

The two 250 ml. centrifuge cups loaded with equal portions of the 891A (50 to 100 g. in each), and the cups were filled to 1/2 - 3/4 inch from the top with the first medium (methylene iodide). The cups were placed in the centrifuge and the covers placed on the cups. The centrifuge was started by releasing the brake and pushing the starting switch with the rheostat set at zero. The rheostat setting was slowly increased up to 21, and after two minutes the centrifuge speed was checked to make sure that it did not exceed 2000 R.P.M.

After the samples had been spun for 15 minutes, the motor was shut off and the centrifuge allowed to stop <u>without use of the brake</u>. When the centrifuge had stopped, the upper layers in the cups were reslurried and the process repeated.

This time at the end of the operation the upper layer was decanted through a Büchner funnel. The medium and the "Sink" material left in the tubes were poured through another Büchner. The medium was removed from the flasks and the solids washed thoroughly with carbon tetrachloride. The two fractions were then dried for 3 to 8 hours, depending on the quantity of material.

The dried material from the upper layer was placed in the two cups and the above procedure repeated, this time with a mixture of acetylene tetrabromide and carbon tetrachloride as the medium. The mixture was made from 42 parts by volume of carbon tetrachloride and 58 parts of acetylene tetrabromide, and had a density of 2.35 at 25°C. A 250 cc. graduate was used to check the density to the nearest 0.1 unit.

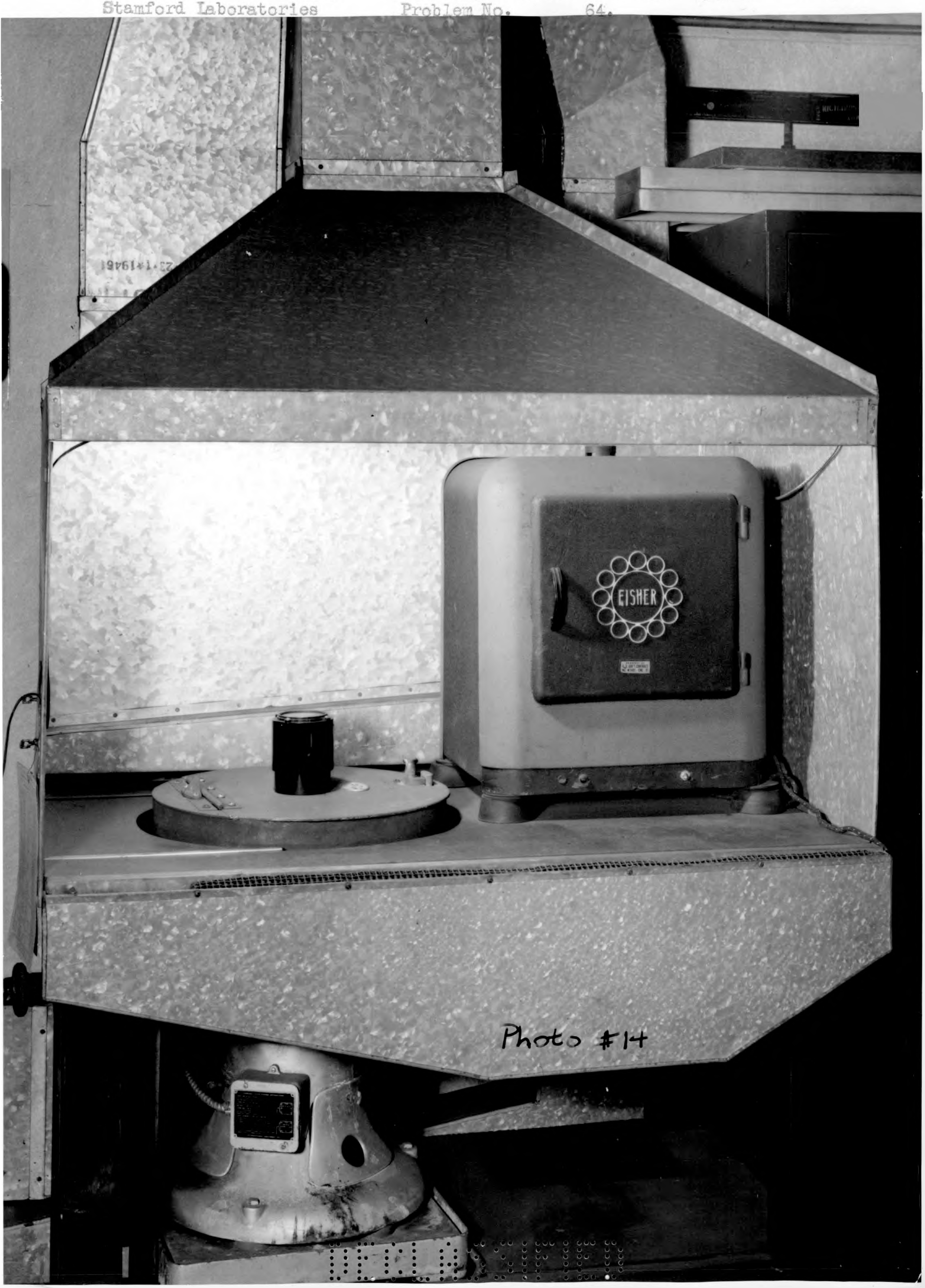


Photo #14

308-082

363-82

The upper layer obtained this time was the "Float" and the lower layer was the "Middlings". After washing as described above, the materials were dried overnight at 105-110°C. to remove the last traces of medium and washing solvent. The "Float", if it met specifications, was not further treated. If it did not, it was subjected to HF treatment or water wash. The "Middlings" were recycled with the next run.

8. Hydrogen Fluoride Treatment.[4]

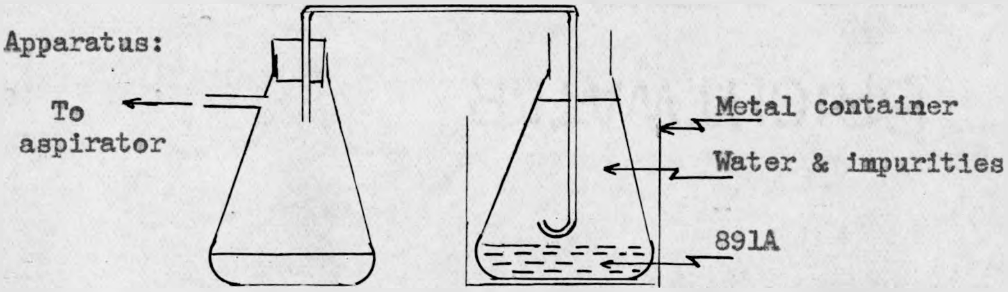
The "Float" from the so-called attachments after acid treatment and "Sink and Float", may not pass specifications for 891A (98%) and often requires further treatment. Hot hydrofluoric acid (48%) will dissolve all free tantalum, tungsten and silica, but will not attack 891A.

The 891A was placed in a copper beaker and the hydrofluoric acid (HF) added. Sufficient HF was added so that the 891A was completely covered and could be stirred easily. Since all iron had been removed previously, there was no appreciable evolution of gas. The mixture was heated carefully to boiling. Additional HF was added from time to time to replace that lost by evaporation. The slurry was stirred every 15-30 minutes for six hours by hand with a copper stirring rod. In the handling of HF extreme care should be taken. At all times wear rubber apron, rubber gloves and a shield. If any HF gets on the skin or clothing, wash area thoroughly with water and report to the Medical Department immediately. HF will cause severe burns and is extremely poisonous. Care should be taken not to inhale any of the fumes, since they are very injurious to the lungs. At all times keep HF liquid in the hood. The HF and 891A mixture after reaction was diluted with about three volumes of water (i.e., three times as much water as HF present) and allowed to cool and settle. The clear liquor was decanted off and thrown away. The procedure was to pour it down the sink using large volumes of water to dilute it. When it was decanted as far as possible without losing 891A, several volumes of water were added. The 891A was allowed to settle between each washing. The wash water was decanted off at least three times. Next the material left was filtered and washed three more times with water on the filter. At no time should any of the liquid be allowed to contact the skin or clothing, since it may still contain a small amount of HF. The cake, when dry, was washed with 2B alcohol, then placed in the oven to dry. This treatment removed all free tantalum, tungsten and silica but did not remove combined 891A. This treatment leaves objectionable amounts of copper in the 891A, so that it was acid-treated (6 N hydrochloric) after it had dried. To remove combined 891A another procedure must be used. This will be described in the section entitled "Water Washing of 891A".

9. <u>Water Washing of 891A[4,5]</u>.

The purpose of water-washing of the 891A is to remove the combined 891A present in the 891A after "Sink and Float" operations. The water-wash can be done either after the "Sink and Float" or after the HF treatment operation. Water-washed 891A will show an improved purity of the product of the order of 0.5 to 1.0%.

To water wash, the 891A was placed in a three-liter Erlenmeyer flask and the flask filled with water. The Erlenmeyer was put in a metal container as a safety measure in case of breakage. The material was mixed thoroughly and then allowed to settle for 2 or 3 minutes. As soon as the coarse 891A had settled, the upper layer was drawn off on a Büchner filter funnel with glass tube and rubber hose attached to the Büchner. The water was decanted off and then filtered.



The material left in the Erlenmeyer flask was slurried with fresh water. This procedure was repeated 8 times. The 891A left after 8 washings was filtered. The cake was washed with 2B alcohol and dried in the oven. This material should pass specifications for shipment, but if the washed 891A is slightly below specifications it will have to be blended with good 891A to bring it up to specifications.

10. Variables in the Process

Since the entire process is dependent on the ease with which the 891A can be removed from the wire and the amount of attachments formed on removal, it is important that the Chlorthane passed over the hot wires be as pure as possible and that the wires be free from grease, scratches and nicks. TAW wires gave the best results. If the recovered Chlorthane is not purified often enough, residues that accumulate in the recovery bombs seem to increase the attachments and make removal from the wire more difficult. As the attachments increase, the purity of the finished 891A is lowered. When more attachments are formed, the 891A must be subjected to many more operations such as "Sink and Float", acid treatment, HF treatment and water washing. When more attachments are formed more 891A is recycled as "Sink", "Middlings" and impurities from water wash. This is the most important phase of the process for which to watch out. Another variable is grinding of the 891A. With new rolls in the crusher approximately 2% iron is added, but should the rolls be badly worn the iron content may rise to 5 to 6%. The adding of more iron than is necessary will increase the magnetic fraction and ultimately lower the yield. The rolls should be changed when the iron concentration after grinding is higher than 5%. In order to determine the per cent iron added during grinding, an overall check on the material charged and the material recovered (after the machine has been cleaned) will give the increase in weight due to the iron added, less losses. Grinding losses normally run about 1%. When the iron content is 5% or over the new set of rolls should be installed. For directions on installing new rolls, see section entitled "Equipment".

Another variable in the process is the method of removing the iron added during the grinding. The best method is magnetic separation. In some cases when the humidity is high, the separation will be very poor. It is always advisable to keep the 891A in desiccators until it is to be used in the Stearns Magnetic Separator. Should the separation be so poor that the magnetic fractions are abnormally high, it will be necessary to recombine the fractions and wait until the humidity drops before continuing. Should the material be needed immediately an alternate procedure would be to acid treat the 891A with 6 N HCl. This would require at least two days to obtain the finished product, since it takes approximately two hours to add all of the acid and four hours to react at 90°C. After that it must be cooled, filtered and dried overnight in the oven. Since acid treatment does not give as high a purity as the magnetically separated 891A, it is usually advisable to wait until the humidity drops.

The "Sink and Float" of 891A is another variable which is dependent on the mesh size, the amount of combined 891A present, and the operator. For instance, dust from attachments will not separate very well, since it is so very fine. Even spinning in the centrifuge cups for a long time will not help very much. Approximately one-half of the dust from attachments must be recycled, which represents about 5% of the 891A in the attachments. If the attachments are very high, it is obvious that the amount of recycled 891A will be very high.

The "Sink and Float" of the attachments (not dust) will at best leave approximately 14% 891A with the "Sink" fraction and 60% 891A with the "Middlings". The "Sink" fraction represents approximately 15% of the total 891A present in the attachments. The amount left is dependent on the sharpness of the separation of the layers and the skill of the operator in removing the upper layers. (The above figures are for the normal separations.)

11. Time Schedule

For a normal run with the present process the following time schedule will illustrate the approximate time spent on each step.

	Weight Handled g.	Time Required hrs.	No. Days of Week Equipment is in Operation	% Time Requiring Attention
Hand Picking	1500	2	5	100
Grinding	2000	16	3-4	20
Rotapping	2000	16	3-4	10
Magnetic Separation	2000	8	2-3	15
Sink and Float	2000	20	2	75
HCl Treatment	1000	10	2	25
HF treatment	1000	10	0-1	50
Water Wash	1000	15	0-1	50
Sampling	2000	(20 min.)	1	100
Shipping of 891A	8000-10000	6	1/2	100
Inventory, Reports, etc.		5	1	100

Man hours required during the week - 80

This is assuming that there is only a small percentage of attachments present. Should the attachments increase considerably, as has been the case on occasion, the time requirements would increase rapidly.

12. Losses

Losses in the process are purely mechanical losses since no chemical reactions are involved. The losses are as follows:

Hand-Picking Losses. Material lost in breaking 891A off wires.

Grinding Losses. 891A collected on the bearings and on the grease used to lubricate bearings; 891A as dust and material not recovered from pans, trays and machine.

Rotap-losses. Material is lost as dust during the filling, screening and unloading of the screens.

Magnetic Separation Losses. Material as dust or otherwise spilled during the operation of the separator.

Sink and Float Losses. These losses are of two varieties: first, the material which must be recycled and converted to Chlorthane; secondly, the mechanical losses such as are incurred in centrifuging, on the filter paper, and during the handling of 891A.

Acid Treatment Losses. These consist of material in the filtrate as suspended 891A, material in the filter paper and any miscellaneous losses in handling.

Sampling Losses. Samples retained for analysis.

13. Data

All data should be recorded in a suitable notebook; all materials should be given a sample number to identify them for the succeeding steps. In that way, all materials can be traced back to its source. In addition to the data kept in a notebook, a chart should be kept to record all weights of the material handled in the process. This chart shows where all material goes and serves as a check on the physical inventory. It should show where losses are occurring and how much material is being recycled. The chart should show the amount received from Step 4 and the amount shipped. The analysis of the material shipped should also be recorded so that the amount of pure 891A shipped is known.

14. Sampling of 891A.

The procedure for sampling the finished 891A was as follows: The material was thoroughly mixed in a pan and then it was passed through the sample splitter slowly. On the side of the sample splitter there was a switch which operated the vibrator. This helped vibrate the material down the chutes and prevented clogging. The recommended method of running the material through the splitter was to pass the sample through the splitter, then to take one of the pans and dump it into another container. Material remaining was next passed through the splitter again and the pan that was on the opposite side of the splitter was dumped into the storage receiver. In this way any irregularities in sampling procedure were not cumulative. When the material in any pan was approximately 10 g., it was placed in a bottle and submitted for analysis. This bottle was labelled as follows: R. U. 232 - notebook number - page number; the second line was labelled with an identification of the material, such as non-magnetic fraction, float or acid-treated 891A; the third line gave the gross, tare and net weights; at the bottom, the date and the operator's initials were shown. The sample weight was recorded in the notebook beside the material sampled.

For very large samples, it was first necessary to divide the material in half in the pans before running it through the splitter. This was best done by mixing thoroughly the 891A from one pan to another, using a scoop and then finally dividing the 891A into two equal portions.

One of the two portions was taken and sampled in the same manner as previously outlined. Although this, of course, is not quite as accurate as splitting the whole amount, it was necessary to do this in order to reduce losses of 891A on sampling. Care should always be taken not to have any fans on when sampling, since these would blow away any considerable dust always present at such times. Shields were used to keep the dust from rising. Most of the dust could be collected after the sample had been taken by brushing the bench. In spite of all precautions, 2-3 g. of 891A usually was lost during sampling as dust.

15. <u>Shipments</u>

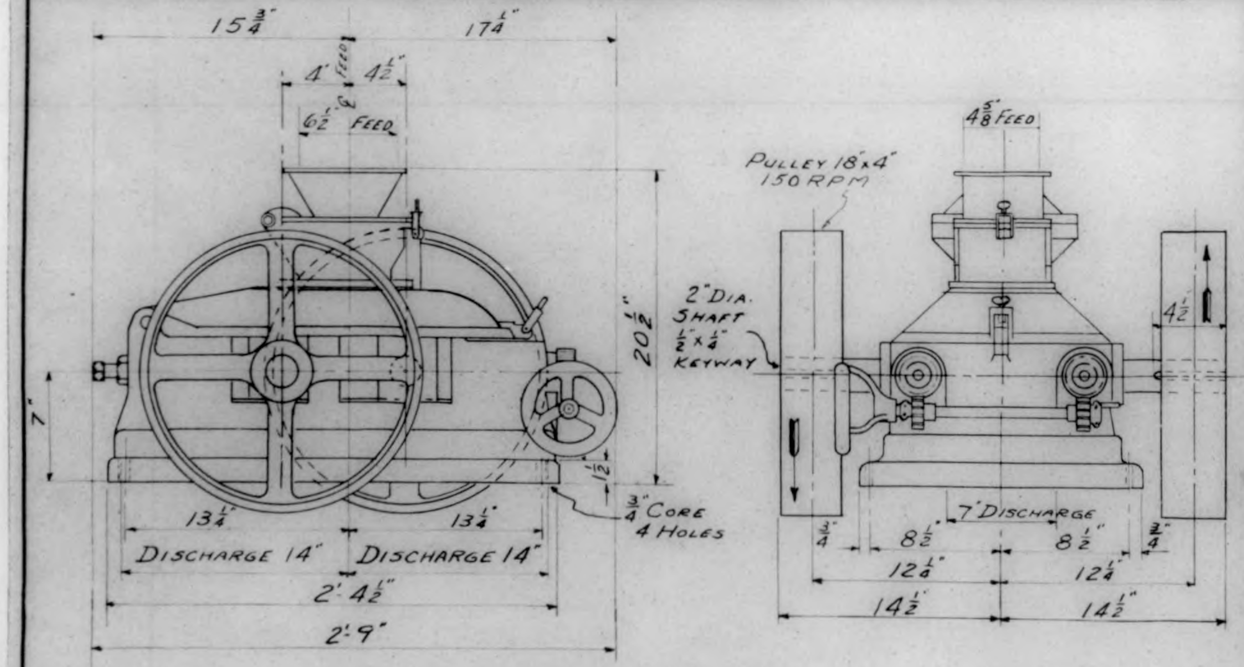
The material which passed specifications (891A 98%, Fe 0.3%) was placed in one quart tin cans (friction covers) and weighed to 1000.0 g. net. The cans were sealed with solder on two sides to prevent the covers from coming off during shipment. The only labels on the cans were as follows:

Lot No.........
R. U. 232 - Notebook No......-Page..............
This can contains.............kg.
Total weight...................kg.
Assay % 891...................% Fe...
As represented by the following batches........
of 890A..........% Date..................

16. <u>Equipment and Floor Plans</u>

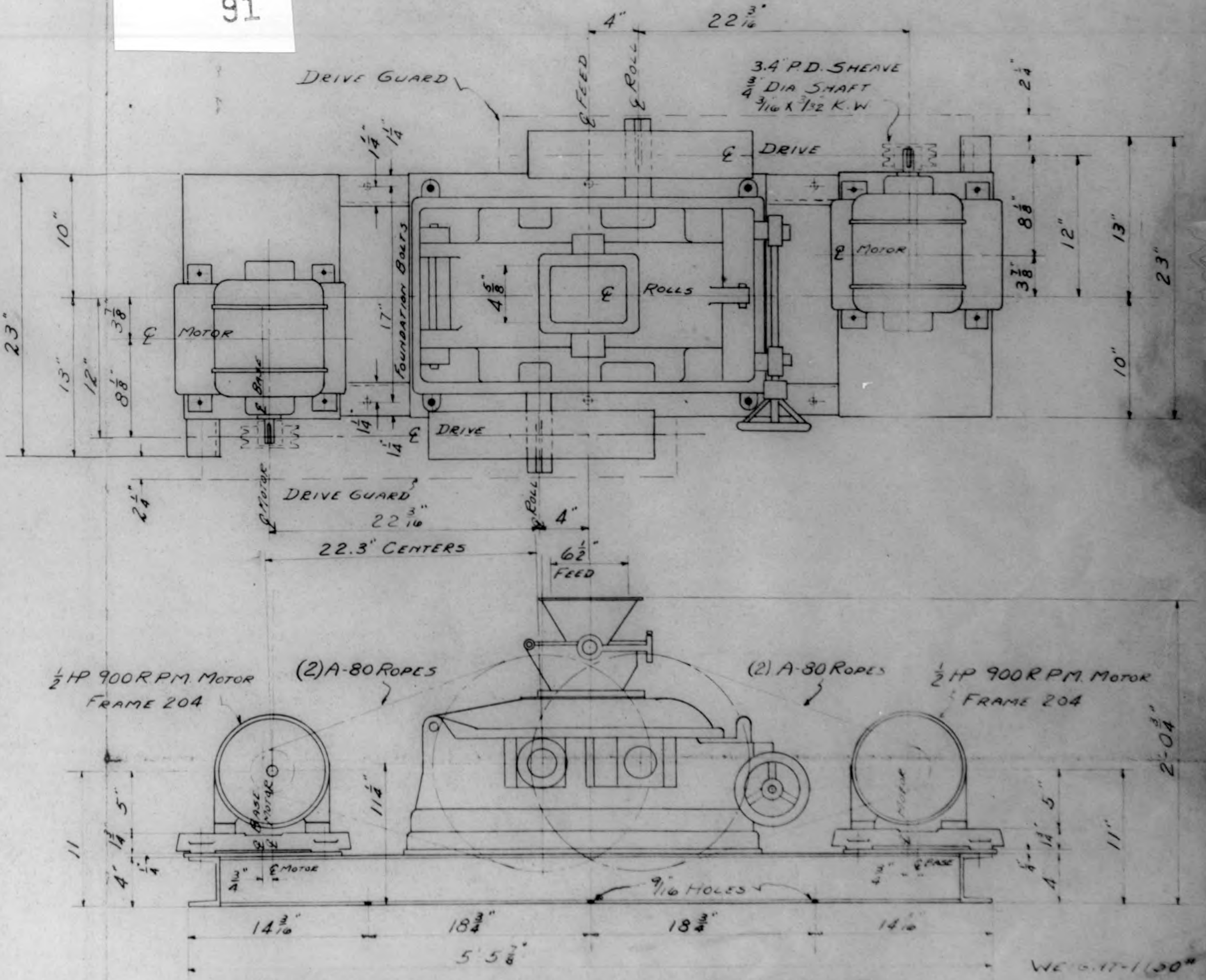
<u>Care and Maintenance of Crusher</u>

1. The Sturtevant Mill Crusher is a 8" x 5" laboratory roll crusher with special glass-hardened rolls R.C. 65. For details see Dwg. 21816 , p.704. It is driven by two induction, General Electric, horizontal, polyphase, explosion-proof motors, 1/2 HP, 900 RPM, using No. A, 80 V belts.



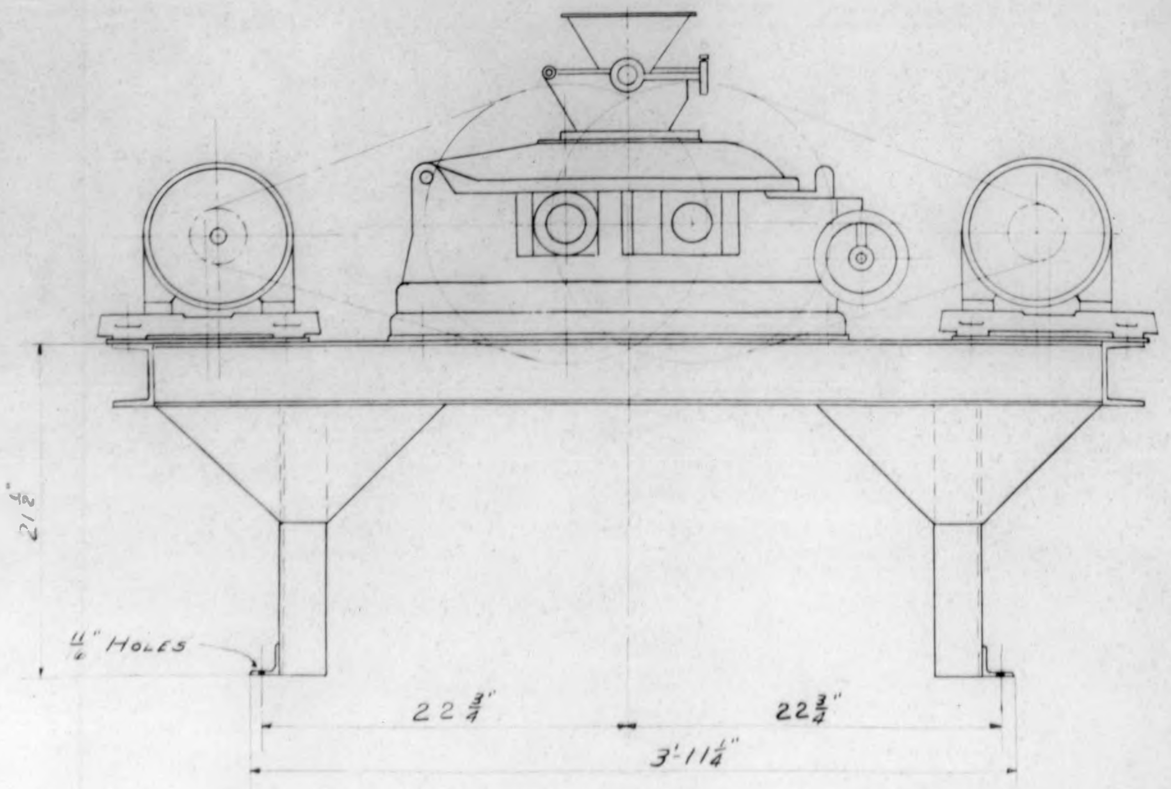
FIG"1 8"x5" LABORATORY ROLLS

1 HP
WEIGHT - 650 LBS



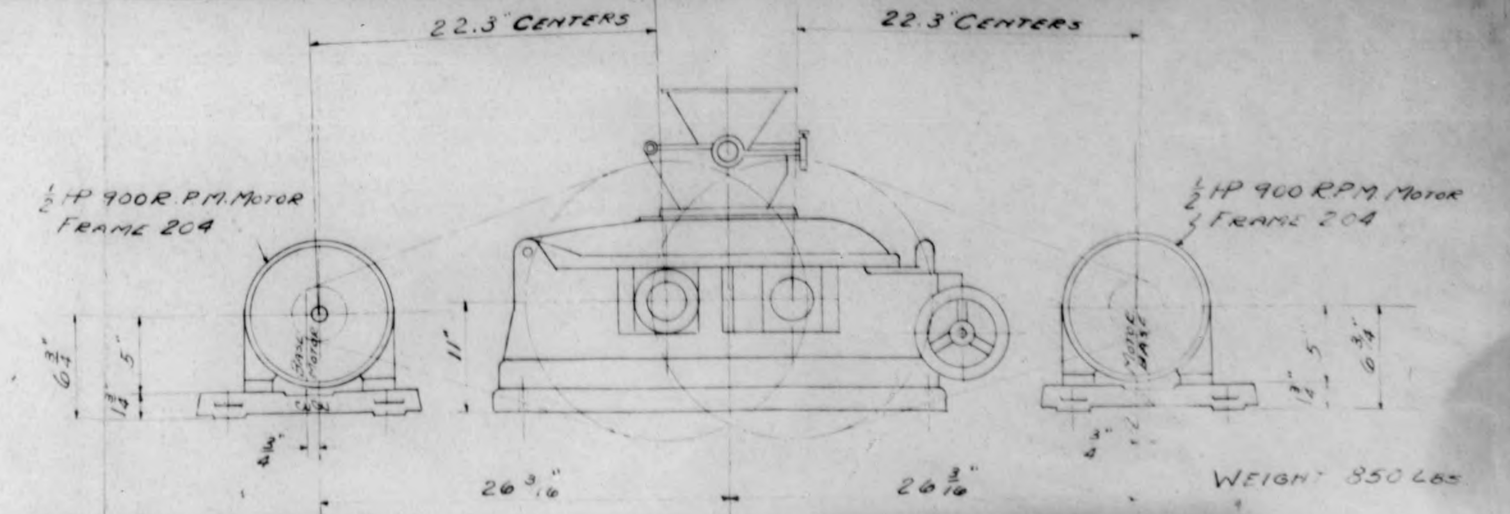
FIG"3 - 8"x5" LAB ROLLS, MOTOR, V-FLAT DRIVE AND BASE

WEIGHT - 1130"



FIG"2 - 8"x5" LAB. ROLL, MOTOR, V-FLAT DRIVE AND STAND

WEIGHT - 1200 LBS.



WEIGHT 850 LBS

FIG"4 8"x5" LABORATORY ROLLS, MOTOR AND V FLAT DRIVE

④	③	②	①	REVIS. NO.	DRAWN EFA 11-10-42	STURTEVANT MILL CO.	8"x5" LABORATORY ROLLS	SCALE 1 1/2"-1'-0"
				MADE BY	CHK'D	HARRISON SQUARE	OUTLINE ASSEMBLIES	21816
				DATE	APP.	BOSTON, MASS., U.S.A.		FILE NO. 5-16

363 091

DECLASSIFIED

The only maintenance required for the machine is the greasing of the shaft bearings each day by turning of the grease cups 1/2 to 1 turn and the oiling of the oil cup and motor twice a year. The rolls become worn and have to be replaced as indicated above. The procedure for installation of a new set of rolls is as follows: remove the guards on the fly wheels and remove the V belts. Using a wheel puller, remove the two fly wheels. Next open the hood on the machine and clean thoroughly with the vacuum cleaner to recover any 891A left in the machine. The springs at the rear of the machine are released by turning the two large bolts on the casing. The rolls next to the spring are then lifted out, including the journal boxes. Next turn the adjusting wheel or the other roll until the bolts are exposed, and lift the roll out, making sure not to turn the bolts, since otherwise they will roll out of alignment when the new set of rolls is installed. The journal boxes are removed and cleaned out, first scraping off excess grease and 891A. Store the latter in the bottle marked "scraps". Then wash bearings with carbon tetrachloride to remove all grease and 891A. New grease should be put in the grease cups. Wipe off the protective grease on the new rolls with carbon tetrachloride and then coat shafts only with grease to aid in replacing the journal boxes. The rolls are placed in position and the springs tightened on the rolls until there is a slight compression. The fly wheels are put back in position and the key put in.

This operation will take about a day, so that work should be scheduled to prevent loss of time while the rolls are being installed. The rolls that are worn are sent at once to the Machine Shop for redressing of the grinding surface. If the shafts are badly scored, they should also be resurfaced. A spare set of journal boxes should always be kept in readiness in the case bearings wear out.

Care and Maintenance of the Ro-tap

The W. S. Tyler Ro-tap is made by the W. S. Tyler Co., Cleveland, Ohio (No. 4977). For details, see Dwg. R211, p.707 . The Ro-tap must be greased every two weeks to prevent excessive wear of the parts. A grease gun should be kept in the bottom of the equipment cabinet. The excess grease that appears around the parts should be wiped off to prevent it from getting on the screens or the bench. Every six months the oil in the crankcase should be drained and fresh oil put back in. The level in the crankcase should be checked every three months. If it is below 2-3/4 inches, fill to 3 inches. Approximately two gallons of light oil will fill the machine to the proper level.

An occasional drop of oil on the motor bearings will be sufficient to prevent wear.

Stearns Magnetic Separator

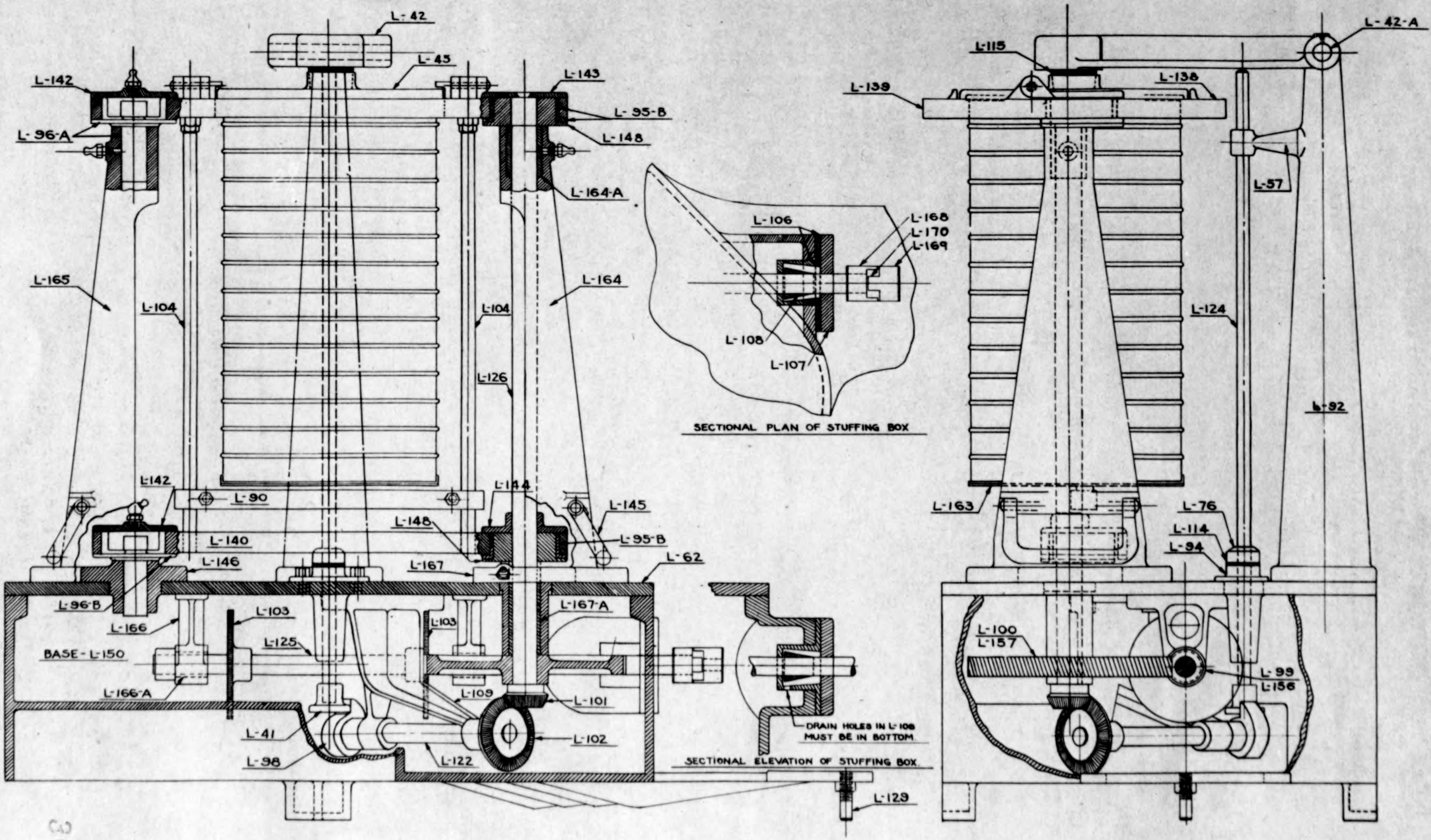
The stearns Magnetic Separator is a Type "KB" Separator with rectifier. The Stearns Magnetic Separator requires no maintenance. For detailed drawing, see Dwg. C-731, p.709.

International Centrifuge

The centrifuge is a size 1, type S, with reenforced steel guard. It requires no maintenance except an occasional filling with oil of the cup at the base of the machine. When installing a new head on the centrifuge, be sure the key is in the slot and that the set screw is tight. If it is not tight it will vibrate and might lift off at high speed.

Floor Plan and General Layout of Equipment

See Dwg. 329-19-0, p.711.



PARTS DRAWING FOR

RO-TAP TESTING SIEVE SHAKER

THE W. S. TYLER COMPANY
CLEVELAND, O.-ST. CATHARINES, ONT

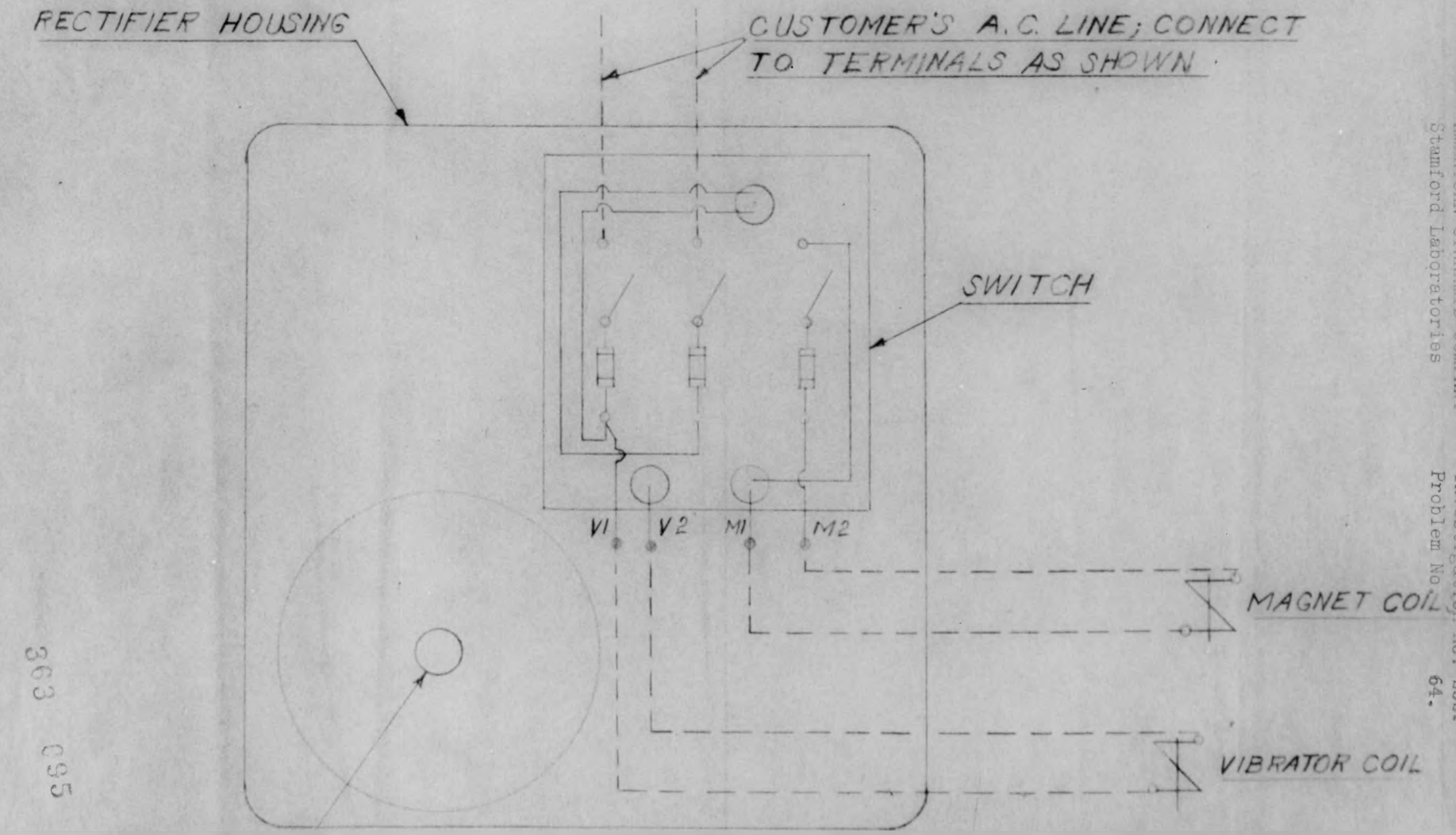
SCALE HALF SIZE
DRAWN BY
CHECKED BY
APPROVED BY
DATE 5-4-37

DRAWING
R-211

AMERICAN CYANAMID COMPANY
Stamford Laboratories

94
Investigation No. 232.
Problem No. 64.
Page 707 eng 91

363 094



NOTE :- CUSTOMER IS TO MAKE CONNECTIONS AS INDICATED BY DOTTED LINES

REVISIONS	NOTICE				DWG. NO.
A	THIS DRAWING IN DESIGN AND DETAIL IS OUR PROPERTY ALL RIGHTS OF DESIGN OR INVENTION ARE RESERVED	HIGH STEARNS DUTY STEARNS MAGNETIC MFG. CO. MILWAUKEE, WIS., U. S. A.	WIRING DIAGRAM FOR TYPE KB SEPARATOR WITH RECTIFIER	DATE 1-30-46	A-376-B
B				SCALE —	
C				BY G.R.S.	
D				ORD. 20664	

AMERICAN CYANAMID COMPANY Investigation No. 232.
Stamford Laboratories Problem No. 64.
Page 708 eng 91

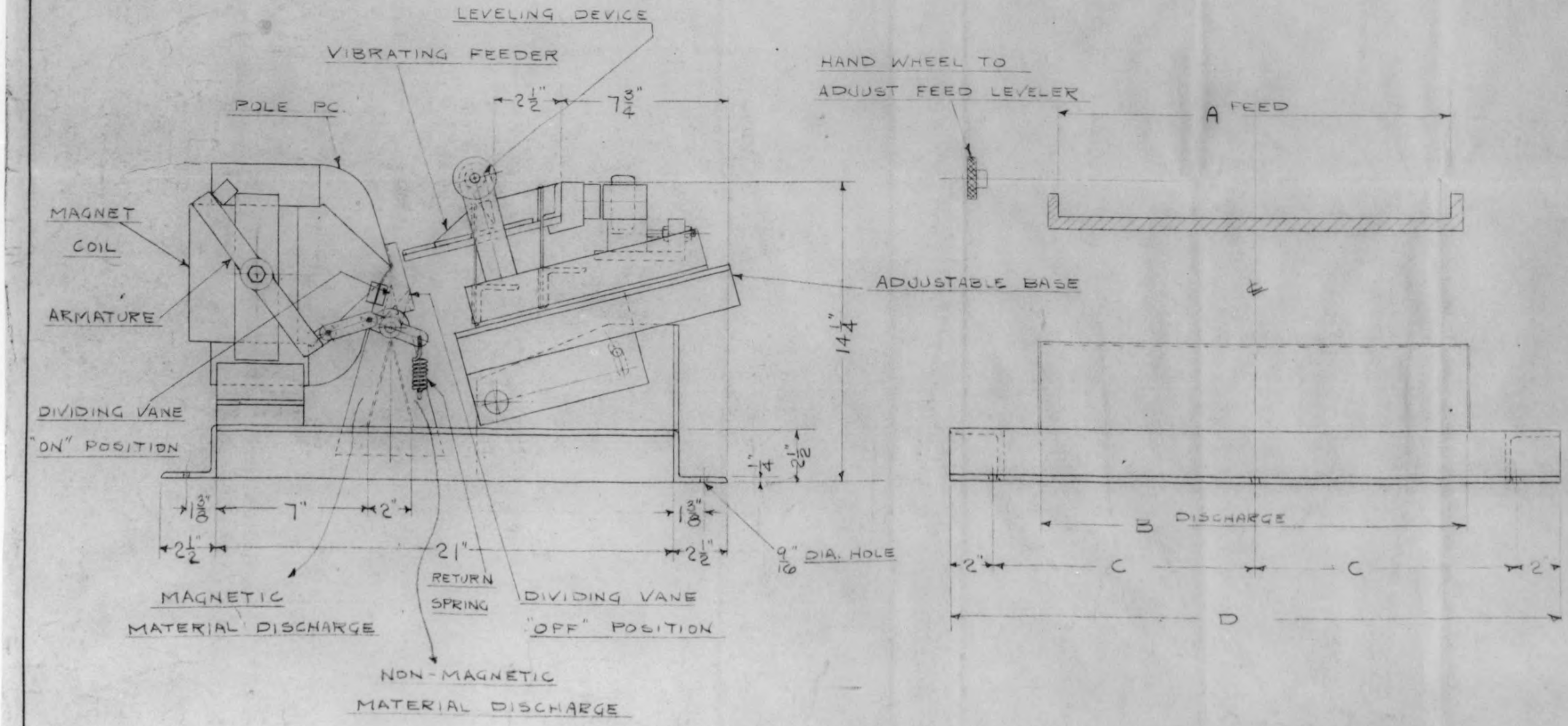
95

363

095

96

SIZE FEED A	DISCHARGE B	C	D
12"	13¼"	9"	22"
18"	19¼"	12"	28"
24"	25¼"	15"	34"



REVISIONS		DATE	BY	INCORP. ORDER NO.	REVISIONS		DATE	BY	INCORP. ORDER NO.
A					E				
B					F				
C					G				
D					H				

NOTICE
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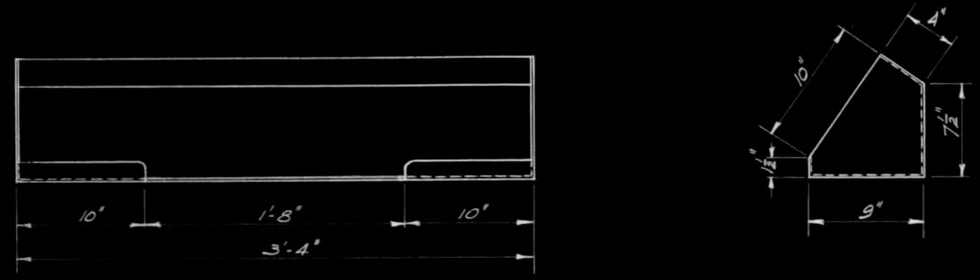
HIGH STEARNS DUTY
STEARNS MAGNETIC MFG. CO.
MILWAUKEE, WIS., U. S. A.

OUTLINE & DIMENSION
TYPE "KB" MAGNETIC
SEPARATOR

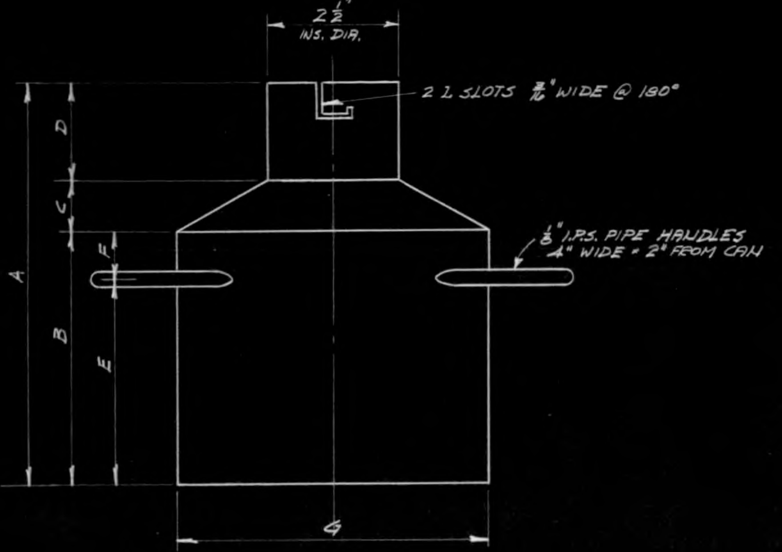
DATE 10-15-41
SCALE 3"
BY WGC
ORD.

DWG. NO.
C-731

303 090



PICKING TRAY
MAT: 18 GA. 18:8 STAINLESS ST'L



NO. REQ.	USE	A	B	C	D	E	F	G
2	STEARNS MAG. SEP.	7¾"	4½"	1"	2¼"	4"	½"	6"
2	STURTEVANT ROLLS	9½"	5½"	1½"	2½"	5"	½"	7"

RECEIVERS
MAT: 18 GA. 18:8 STAINLESS ST'L
NOT TO SCALE

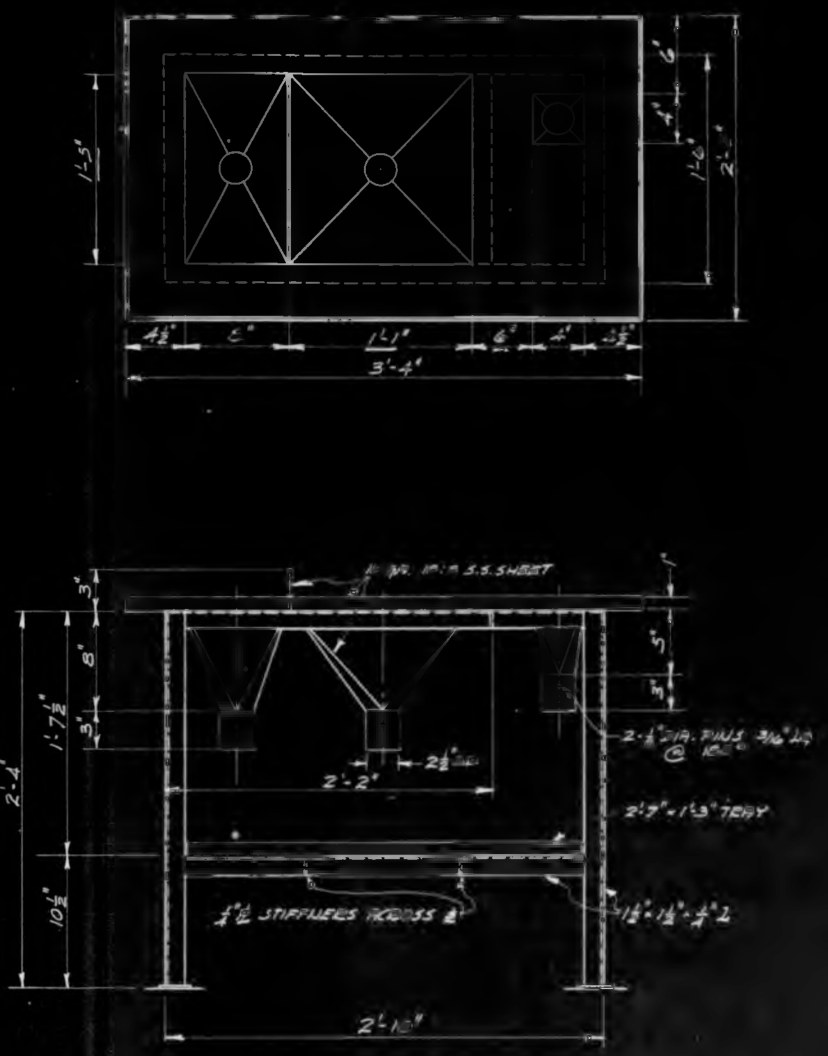


TABLE & HOPPER FOR STEARNS MAGNETIC SEPARATOR

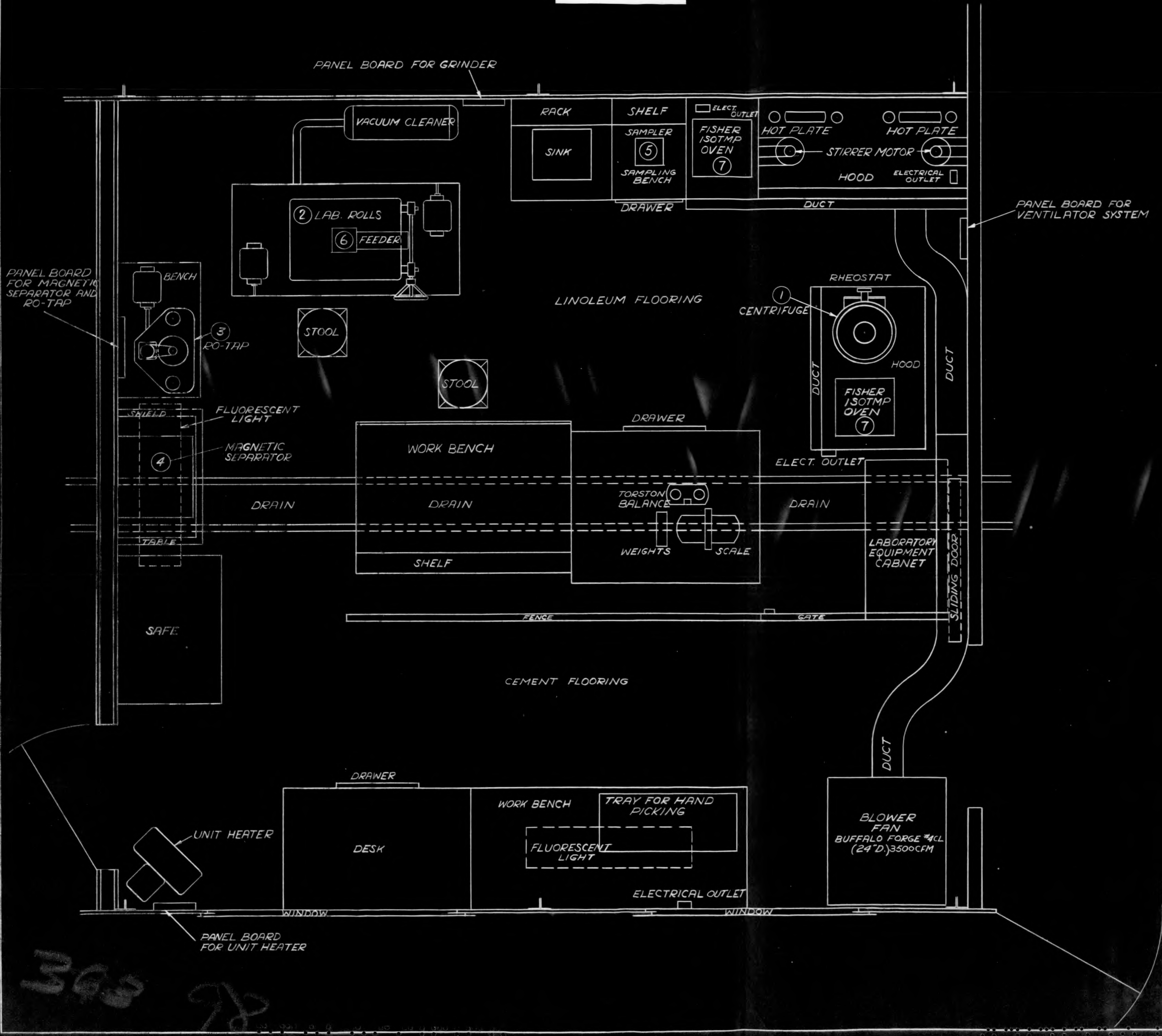
HOPPER FOR STURTEVANT LAB. ROLLS
MAT. 18 GA. 18:8 STAINLESS ST'L
ALL WELDS TO BE OUTSIDE

REVISION
REFER. DWG.
329-19-0
APPROVED

AMERICAN CYANAMID COMPANY
DEVELOPMENT ENGINEERING DIVISION
STAMFORD, CONN.

AUXILIARY EQUIPMENT FOR
GRINDING & SEPARATION

31-232-64
DESIGN: M.S.
DRAWN: M.S.
SCALE: 1-1/2" = 1 FT.
DATE: 6/26/46
DWG. 411
JOB 19
REV.



EQUIPMENT LIST
1 INTERNATIONAL EQUIPMENT CO. CENTERFUGE SIZE 1 TYPE SB
2 STURTEVANT MILL CO. LAB. ROLLS DWG. NO. 21816
3 W.S.TYLER CO. RO-TAP TESTING SIEVE SHAKER DWG. R-221
4 STERN'S MAGNETIC MFG. CO. TYPE KB MAGNETIC SEPARATOR DWG. NO. C-731
5 THE DENVER FIRECLAY CO. $7\frac{1}{2}$"X5" NO. 2DFC. S.S. SAMPLE SPREADER
6 JEFFREY MFG. CO. -JEFFREY-TRAYLOR NO. 1-B VIBRATING FEEDER WITH ENCLOSED S.S. PAN ($3\frac{3}{8}$"X$15\frac{7}{8}$")
7 FISHER ISOTMP OVEN NO. 13-245A 550 WATTS - 115V.

REV.	REF. DWG.
	326-19-0
	411-19-0

APPROVED

AMERICAN CYANAMID COMPANY
DEVELOPMENT ENGINEERING DIVISION
STAMFORD, CONN.

GRINDING & SEPARATION ROOM LAYOUT

31-232-64

DESIGN: M.S.
DRAWN: wel
SCALE: 3/4"=1'
DATE: 12-13-45

DWG.	JOB	REV.
329	19	0

363
78

98

DECLASSIFIED

17. References

1. Progress Report, August 1, 1945. The use of the vacuum cleaner.
2. Progress Report, June 1, 1945. Acid treatment.
3. Progress Report, August 1, 1945. Purification of Product 891A by extraction methods. p. 399.
4. Progress Report, November 1, 1945. pp. 435-437. HF treatment. Water washing.
5. December 1, 1945 Progress Report.
   Use of TAW wire. pp. 454-456.
   Removal of iron from 891A. pp. 456-457.
   Water wash. pp. 457-458.
   Float with heavy media. pp. 458-459.
6. Progress Report, February 1, 1946. p. 473. TAW wire.

890 Recovery

890 Recovery

Chemistry of Process

The liquid collected in the trap of Step 1 (see above, p.543; see also section on By-Products) is largely 890. The 891 content of this material is recovered as Tribnol in a four-step recovery process using water, hydrofluoric acid, potassium hydroxide and fuming sulfuric acid. The first three steps comprise the "890 Recovery".

(1) $890 + 3\ H_2O = 4\ (CH_3)_2O + 3\ HBF_4 + H_3BO_3$

(2) $H_3BO_3 + 4\ HF = HBF_4 + 3\ H_2O$

(3) $HBF_4 + KOH = KBF_4 + H_2O$

The decomposition of 890 by water, in the first step, proceeds so rapidly at room temperature that the two reactants must be mixed with care to prevent too violent an evolution of Monomer. This reaction is slightly exothermic. Variation of the molar ratio, H_2O:891 from 3:1 to 9:1 has little effect (Ref. 379-38) upon the reaction; however, even the smallest ratio tried represents a large excess of water. Smaller ratios were not experimented with because of the large amount of boric acid which sometimes crystallizes out at low dilutions.

The reaction with hydrofluoric acid is so highly exothermic that the reaction vessel has to be cooled in a water bath. The effect of temperature on the reaction, though, was never studied. The mol ratio of hydrofluoric acid to 891 was varied from 3:1 to 10:1 with no apparent effect, but again this relationship was not studied very closely (Ref. 379-381).

The final step, the neutralization of fluoroboric acid with potassium hydroxide, proceeds as would be expected. Of course a great deal of heat must be dissipated. It is preferable to make the final mixture quite basic to precipitate all the KHF_2 formed even though most of the metals are brought down as impurities in the strongly alkaline solution. These metals cause no trouble in the Salt Recovery whereas any KHF_2 remaining does introduce difficulties (Ref. 380). The salt is usually produced in 96-98% yields and is 94-96% KBF_4.

Table XIV

Properties of Materials in 890 Recovery

Material	Physical State	Molecular Wt.	Remarks
Recovered 890	Liquid	ca. 113.2[a]	Corrosive, irritating fumes in air.
Monomer	Gas	46.07	Inflammable and explosive
HF	60% solution	-	Extremely corrosive, irritating fumes.
KOH	45% solution	-	Corrosive
Salt-KBF_4	powder	125.2	May burn skin slightly when damp.

a. Rec. 890 averages 8.2-8.6% 891.

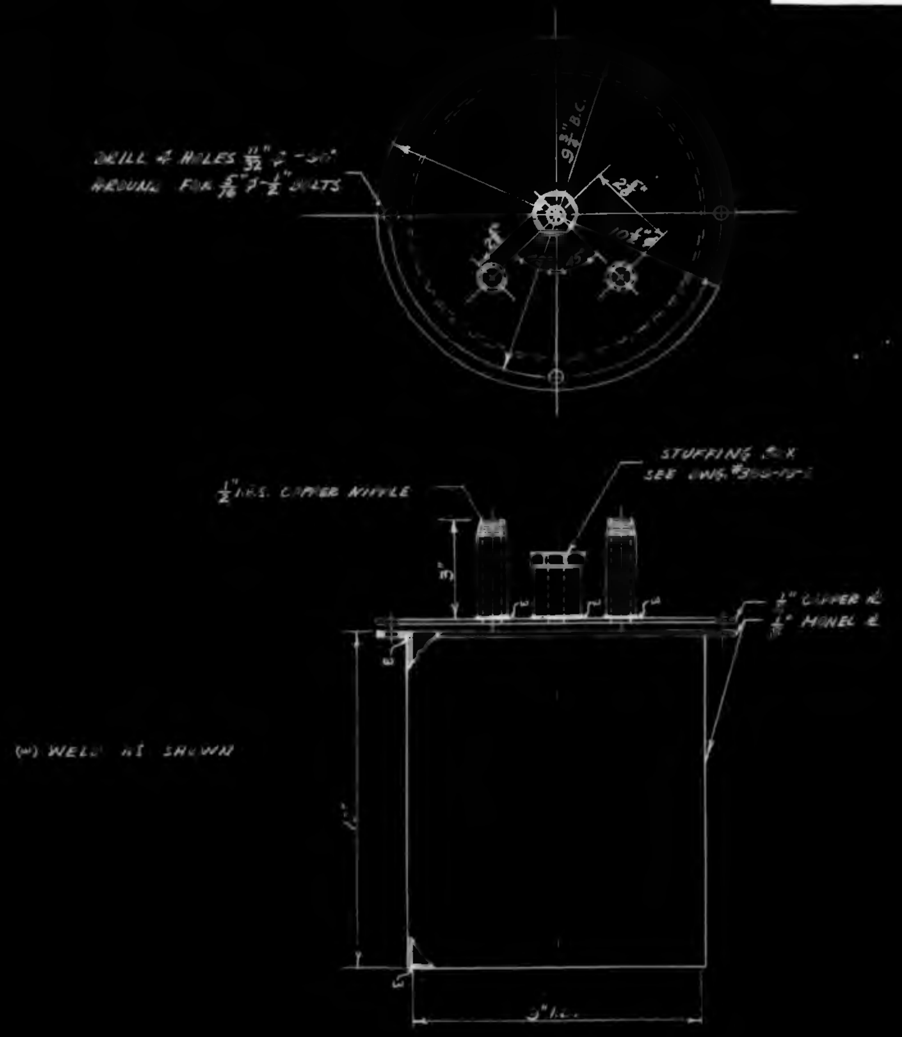
Table XV

890 Recovery Usage Figures (Run 69-186)

Wt. Rec. 890, kg.	2.1	
Vol. water, l.	1.0	(approx. 3 moles H_2O/mole 891)
Wt. 60% HF, kg.	1.6	(" 3 " HF/mole 891)
Vol. 45% KOH, l.	ca.3.8	
Wt. salt (94.3%), kg.	2.3	

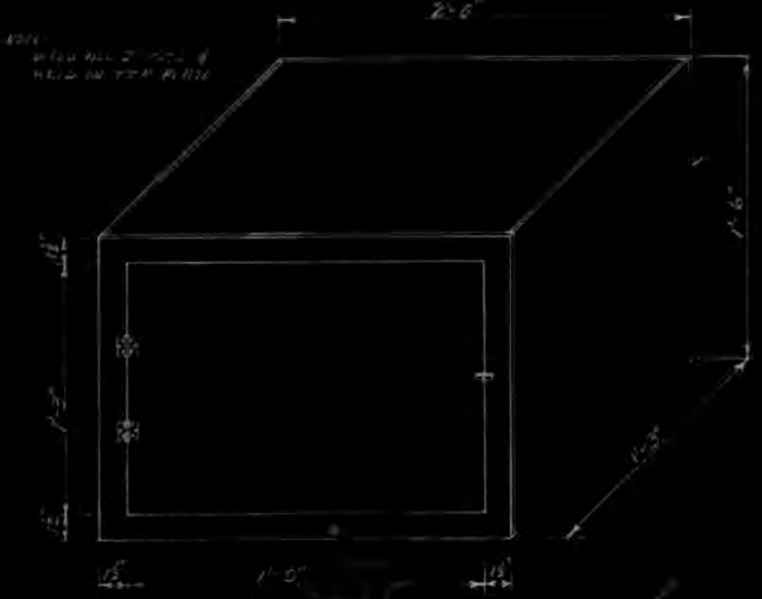
Equipment

The equipment for the 890 Recovery is located in or adjacent to the hood in the northwest corner of the center room (see p.545), and consists of a jar, a stirred monel pot, a cooling bath, and a centrifuge. (See Dwgs. 385-19-0 and 388-19-0).

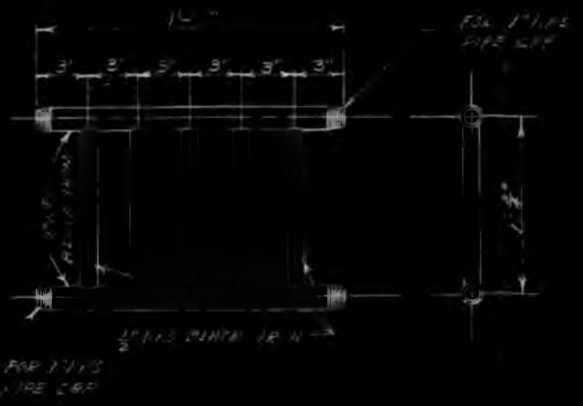


(w) WELD AS SHOWN

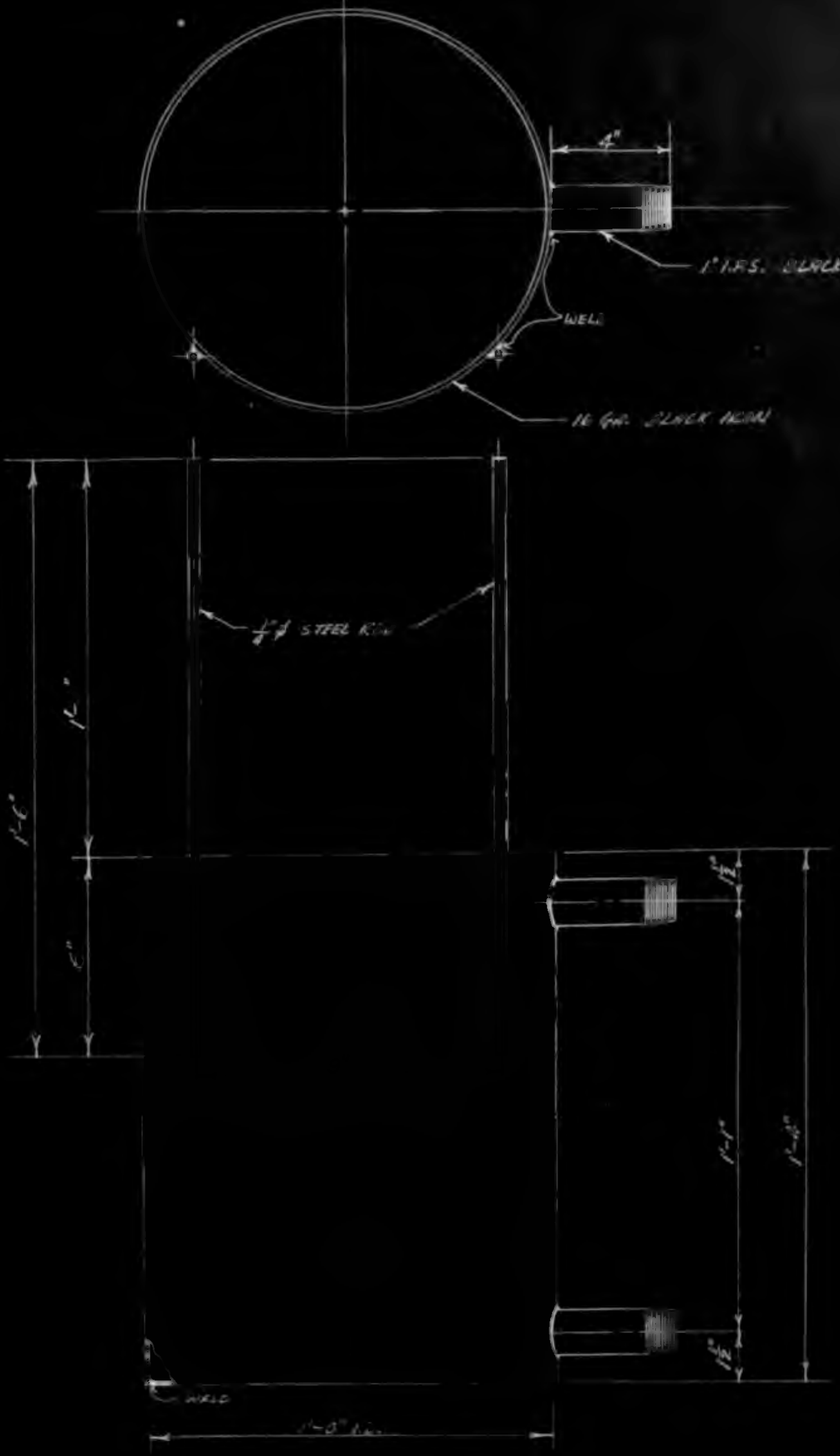
SALT RECOVERY POT
Scale: 2"=1'



OVEN
Scale 2"=1'
INSULATE THOROUGHLY WITH 3/4" ASBESTOS SHEET



OVEN HEATER
Scale: 2"=1'



JACKET FOR SALT RECOVERY POT
Scale: 4"=1'

REVISION	REFER. DWG.
	385-19-0
	389-19-0

APPROVED

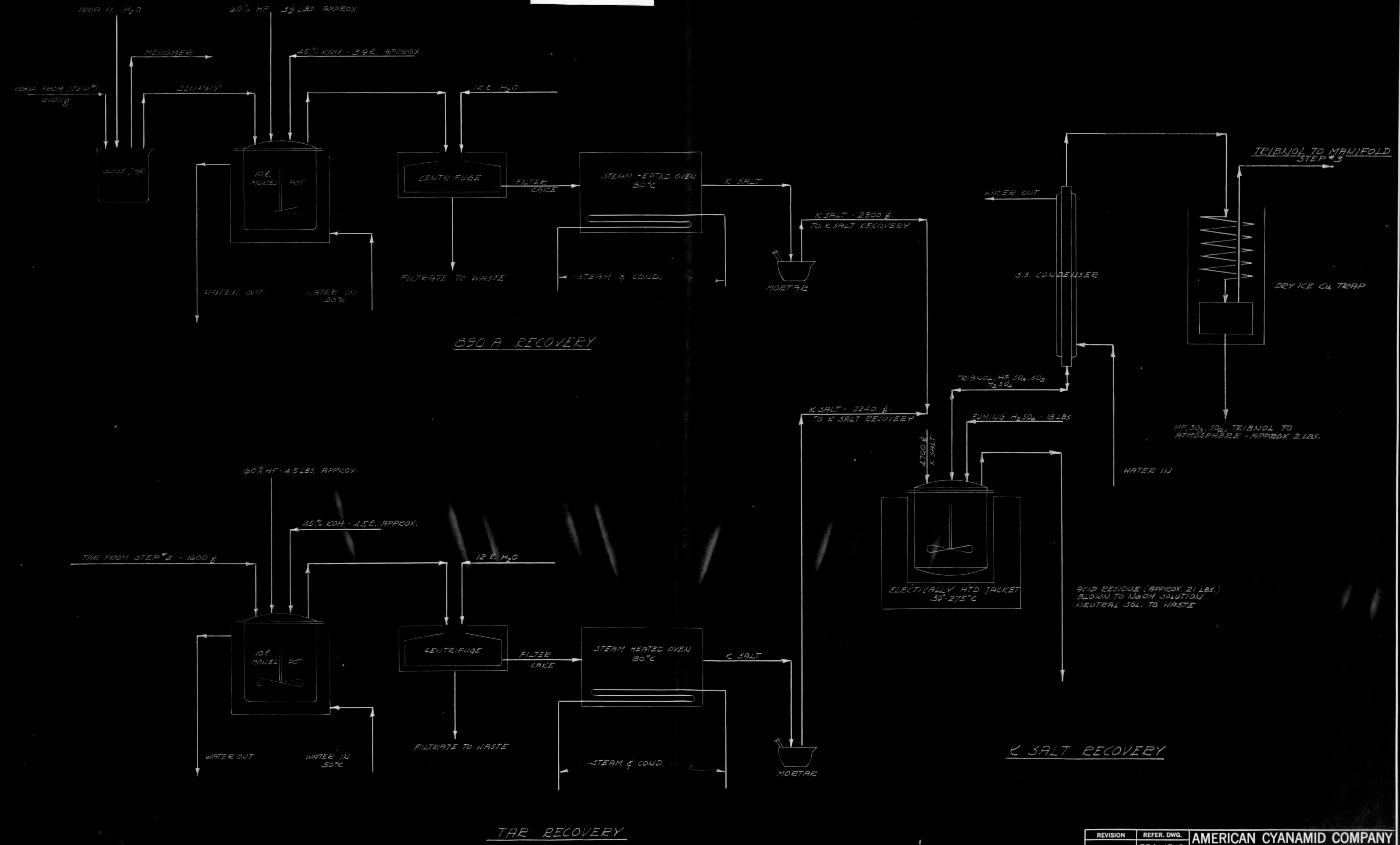
AMERICAN CYANAMID COMPANY
DEVELOPMENT ENGINEERING DIVISION
STAMFORD, CONN.

890A RECOVERY
EQUIPMENT

31-232-64

DESIGN: FWT
DRAWN: FWT
SCALE: AS SHOWN
DATE: 6-28-46

DWG.	JOB	REV.
388	19	0



REVISION	REFER. DWG.
	384-19-0
	334-19-0
	343-19-0
	344-19-0
	388-19-0
	389-19-0

APPROVED

AMERICAN CYANAMID COMPANY
DEVELOPMENT ENGINEERING DIVISION
STAMFORD, CONN.

SALT RECOVERY FLOW SHEET

31-232-64

DESIGN: M.S.
DRAWN: M.S.
SCALE: NONE
DATE: JUNE 11, 1946

DWG.	JOB	REV.
385	19	0

The Recovered 890 and water are mixed in a hood in an open container of suitable size (a one gallon glass jar is suitable). Monomer is given off which is explosive and inflammable! After the Monomer is given off, the slurry is added through a glass dropping funnel to the hydrofluoric acid in a 10 l. spun monel pot. A copper stirrer and cover were originally used but it was found that the copper corroded too rapidly. A monel stirrer as well as pot would be preferable. A powerful laboratory motor operates the stirrer. The heat of reaction is dissipated by placing the pot in a water bath through which tap water is circulated. The temperature is read on a thermometer placed in the thermometer well in the cover. The caustic is added through the same funnel to the mixture in the pot. Then the neutral mixture is centrifuged in a Size 1, Type S.B. Centrifuge manufactured by the International Equipment Co. (See Dwg. 329-19-0 in section on Grinding and Separation, p.711) , using an 11 inch stainless steel perforated basket with a filter cloth. The product is transferred to a porcelain tray, dried in the steam-heated oven, ground in an ordinary porcelain mortar and pastle, and stored until used in glass jars.

Operational Procedure

The first operation in this recovery is to collect the Recovered 890. It is normally drained from the doughmixer trap in Step 1 (see p.584ff) at the end of each run and stored in a monel receiver until a convenient quantity has been collected (approximately 8 doughmixer runs using 5 gal. 890 per run) to convert to K-salt. Since it is very hygroscopic and corrosive, its composition may change during storage. It should therefore be sampled for analysis just prior to its use.

The quantity of 890 desired is then weighed out, and from this value the amounts of water and 60% HF required are calculated on a basis of 3-4 moles of water and of HF per mole 891 in the 890.

The calculated quantity of 60% HF is then siphoned through a rubber hose from the storage drum to the monel pot on a scale. Great care should be exercised during this operation; i.e., acid boots, apron, gloves and hood should be worn. Safety equipment for this purpose is described in bulletins which can be obtained from HF suppliers like Harshaw Chemical Company. Immediately after use, the siphon should be placed in a bucket of water. The cover is then fastened on the pot, and the pot is placed in the water bath. Next the siphon, bucket and all the tools should be washed whether or not they came in contact with the acid. Throughout the rest of the operations, an acid apron, gloves and a face shield should be worn.

The 890 is then slowly added, with occasional shaking, to the calculated amount of water in an open vessel in the hood. The liberated Monomer boils vigorously out of the solution, and if the addition is too rapid, the solution will boil out of the vessel.

Crystals may or may not form during the above reaction. Contrary to Ref. 379, the solution is not filtered, but added, with stirring, directly to the hydrofluoric acid at such a rate as to keep the temperature below 50°C. The mixture is stirred for 10 minutes after the addition and then neutralized. A 45% KOH solution is then added, with stirring, slowly enough to keep the temperature below 50°C. At first the caustic must be added very slowly, but after about half has been used, the remainder can be run in fairly rapidly without the temperature increasing unduly. When almost all the expected amount of caustic has been added, the mixture is checked with pH paper to make sure the solution is alkaline. Caustic addition is then continued slowly until the solution is slightly basic. The mixture is allowed to stir 10-15 minutes and the pH again checked. If necessary, more caustic is used to produce alkalinity.

The pot is then taken from the cooling bath, the cover remove, and the mixture centrifuged. First the filter cloth is fitted to the basket, and the centrifuge started. A little water is poured through to settle the cloth. The centrifuge is run at about 2500 RPM. About 400-500 cc. of mixture is put through the centrifuge. Then the filtrate is collected and recentrifuged. It quite often happens that at the very start some salt gets through the cloth; hence the first portion of the filtrate is recentrifuged. The rest of the mixture is now centrifuged once, and the filtrate discarded. Often the filtrate will be very dark and turbid. This is not due to salt coming through the cloth but to metal precipitates. Recentrifuging the filtrate will not increase the yield but it will lower the purity by as much as 10%. When the filter cake is fairly dry, it is removed, placed on a porcelain tray and dried overnight in the steam-heated oven. The next morning the product is ground in a mortar and pestle, weighed, sampled for analysis and stored in a glass jar or other suitable container.

Variables and Variations

The amounts given above may be varied considerably. Even though there is a large excess of both water and hydrofluoric acid, such variation has little effect. One must be careful, however, not to exceed the capacity of the pot. Therefore in calculating the amounts of water and acid to use, one may round off the values obtained.

Tar Recovery

Chemistry of Process

The tarry liquid collected in the water-cooled trap in Step 2 (see above, p.558; see also By-Products section, p.535) is thought to be largely $BF_2(OH)H_2O$ (S.A.M. Report A-2121, p. 63). The 891 content of this material is recovered as Tribnol in a three-step recovery process using hydrofluoric acid, potassium hydroxide, and fuming sulfuric acid. The first two steps comprise the "Tar Recovery".

$$(1)\ BF_2(OH)H_2O + 2\ HF \longrightarrow HBF_4 + 2\ H_2O$$

$$\text{or}\ \ (BF_3.\text{tar}) + HF \longrightarrow HBF_4$$

$$(2)\ HBF_4 + KOH \longrightarrow KBF_4 + H_2O$$

The decomposition of tar by hydrofluoric acid proceeds very rapidly at room temperature and gives off so much heat that the reaction vessel must be cooled in a water bath. The effect of temperature on the reaction, however, has never been studied. The mole ratio of hydrofluoric acid to 891 has been varied from roughly 3:1 to 12:1 with apparently no effect (Ref. 243-249, 283-286; S. A. M. Report A-2121, p. 53-57, 62-65), but no conclusive studies have really been made. The neutralization proceeds exactly as in the 890 Recovery, and the yield is the same. The purity, however, usually is slightly higher than in the 890 Recovery.

Table XVIII

Properties of Materials in Tar Recovery

Material	State in Which Encountered	Mol. Wt.	Remarks
Tar	Viscous liquid	-	Corrosive, burns skin on contact
HF		See Table XIV	
KOH		" " "	
K-Salt		" " "	

Table XIX

Usage Figures in Tar Recovery

Wt. Tar, kg.	1.6	
Wt. 60% HF, kg.	2.0	(Approx. 3.5 moles HF/mole 891)
Vol. 45% KOH, l.	4.5	

Equipment

The same equipment is used in the Tar Recovery as is used in the 890 Recovery with the exception that no container is needed to mix the tar with water since this step is not required. (See Drawings 385-19-0 and 388-19-0, pp.715 and 714.)

Operational Procedure

Once the tar has been collected from Step 2, the recovery procedure is exactly the same as in the 890 Recovery described above, except that the Tar is not added to water but is added directly to the hydrofluoric acid. The proportions of reagents also differ slightly as described in Tables XVI and XVII.

Salt Recovery

Chemistry of Process

When the K-salt produced in the 890 and Tar Recoveries is mixed and heated with sulfuric acid, a mixture of Tribnol, hydrogen fluoride and potassium acid sulfate is formed.

$$KBF_4 + H_2SO_4 \xrightarrow{50-250^\circ C.} BF_3 + HF + KHSO_4$$

Apparently no reaction takes place at room temperature, but upon warming to 40-50°C., the reaction begins. In order to obtain complete reaction and a controllable, continuous flow of Tribnol, the temperature must be gradually raised to about 250°C. The flow abruptly ceases at this point. The evolution of Tribnol is a rather sensitive function of the temperature throughout the reaction.

Theoretically, concentrated sulfuric acid is sufficient for the reaction; however, studies on 97%, 100% and fuming (20% SO_3) sulfuric acid (Ref. 243-249, 283-286) indicated that fuming acid is required to obtain the maximum yield. The yields obtained were roughly 50-60%, 75-83%, 92-96%, respectively. Fuming acid is probably required because there is some water present in the salt and free SO_3 is needed to remove it by reaction.

The ratio of acid to Salt has not been extensively studied (Ref. 243-249, 283-286, 438, 449) and a ratio of 1.7 to 1 has been found most satisfactoy.

In normal operating procedure, the Tribnol generated is passed directly into the columns of Step 3 and converted to Chlorthane; hence it is impossible to calculate a Tribnol yield. The overall yield in converting Salt to Chlorthane should be 88-89% (Ref. 449-452, and 501).

Table XXIII

Properties of Materials in Salt Recovery

Material	State in Which Encountered	Mol. Wt.	Remarks
Salt	Powder	125.2	May burn skin slightly when damp.
Fuming H_2SO_4	Liquid	-	Very corrosive, irritating fumes.
HF, SO_3, SO_2, H_2SO_4 Mix	Liquid	-	Extremely corrosive, irritating fumes.
Tribnol	Gas	67.82	Irritating and toxic.

Table XXI

Usage Figures in Salt Recovery

Wt. Salt, kg.	4.7	(Approx. 95.5% KBF_4)
Wt. fuming H_2SO_4, kg.	8.0	(20% free SO_3)

Equipment

The equipment for the Salt Recovery is stored in the Step 1 doughmixer room, but is set up next to the hood in the northwest corner of the center room when in use.

The Salt and acid are mixed in a 10" diameter stainless steel kettle fitted with a stainless steel stirrer and thermocouple well, a gas inlet, pressure gauge, a loading hole, and an exit line. The kettle has a flanged top using "Garlock 900" for gasket material. DuPont's "Teflon", polytetrafluoroethylene, was found to be satisfactory for use as packing material around the stirrer shaft. At the end of 30 runs (all that were required), the kettle was badly corroded, the flanges eaten through, and the stirrer had practically disappeared. For future work, corrosion data should be obtained and new equipment of a different material - possibly one of the Hastelloys - should be made.

The kettle is heated in an electric furnace of 3 K.V.A. maximum capacity. The heat input may be varied by means of the nine heaters in the furnace or by varying the voltage with a Powerstat.

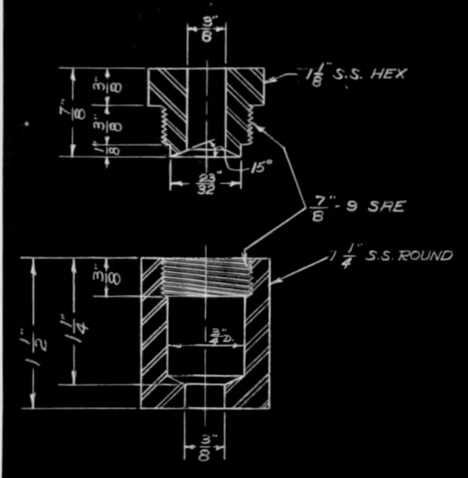
The Tribnol generated in the pot passes through a 30 inch stainless steel reflux condenser which is cooled by tap water. The reflux of boiling sulfuric acid is returned to the pot. The Tribnol then enters a copper trap which is cooled by a dry ice-trichloroethylene mixture, and supported by an iron frame so that the trap may be drained at the bottom. HF, SO_3, SO_2 and H_2SO_4 carried along with the Tribnol is condensed. A monel receiver is used to collect drainage from this trap at the end of each run. The Tribnol then passes through several feet of copper tubing, to a U-bend with a drain valve at the bottom. Occasionally some tar collects here and it must be drained out to keep the lines free. The gas is then led - via black iron pipe - from the hood to the lower manifold of the Step 3 column set-up. See Drawing 389-19-0 below and Ref. 349, 437-438 for additional information.

Operating Instructions

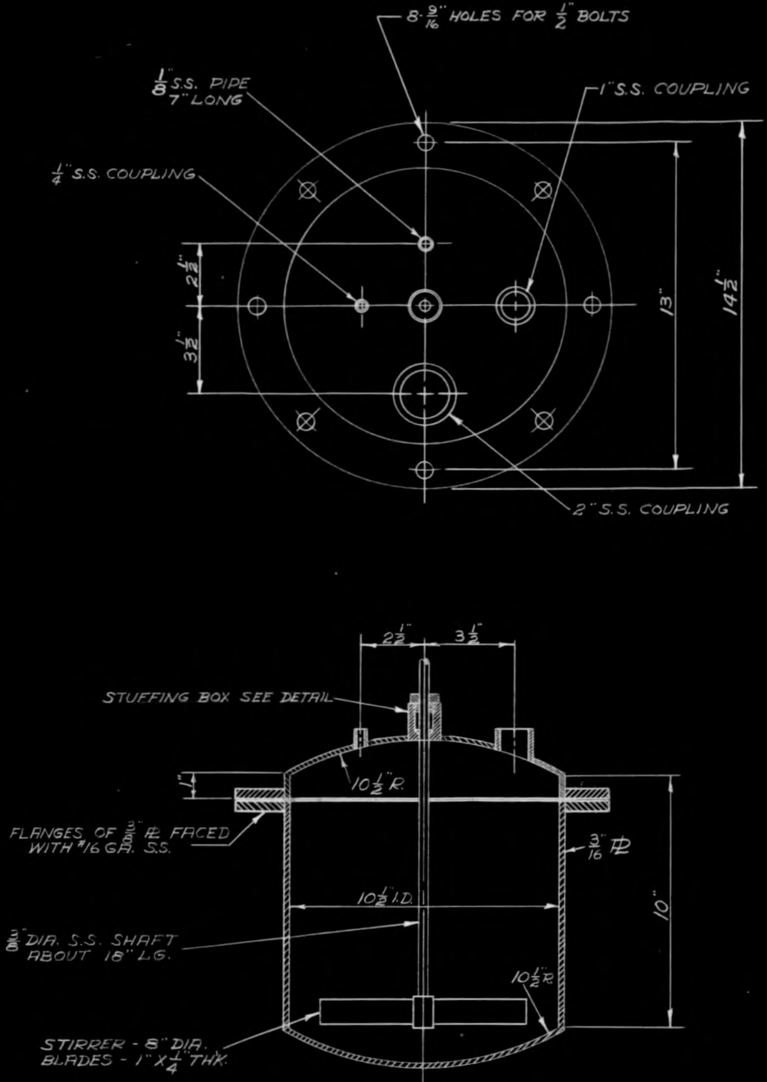
As mentioned above, the Salt Recovery consists of one process, the conversion of Salt to Tribnol, but it is inseparable from the Step 3 process which involves the conversion of Tribnol to Chlorthane. Since Tribnol cannot be condensed it must be used as formed in Step 3. This latter process has been thoroughly described above in the section entitled "Step 3" and will not be described in detail now. The first operation in the Salt Recovery, however, is the preparation of the columns, i.e., the heat is turned on, the dry-ice traps is filled, etc. (pp.583 and 585).

As soon as the columns of Step 3 are ready for use, the recovery dry-ice trap is cooled and the water turned on the reflux condenser. Then 4700 g. Salt is charged through the loading hole and 18 lbs. fuming sulfuric is poured in. The loading port is closed with a cap so that the latter is "hand tight" using some suitable pipe-joint compound. The top of the furnace and kettle is covered with rock wool, stirring started and the heat turned on.

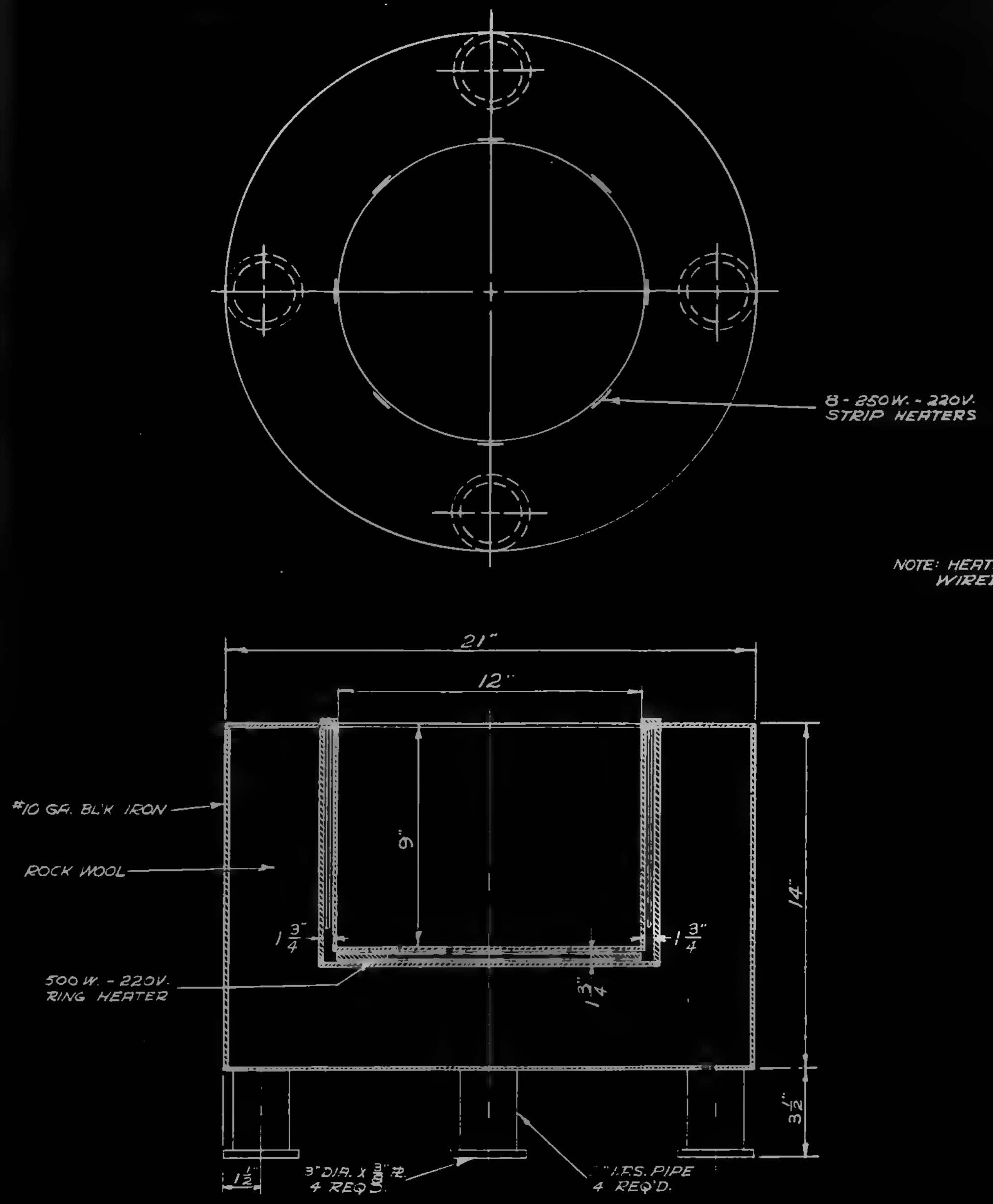
When the pressure in the pot equals the back pressure in the columns, all connecting valves are opened and a flow of Tribnol through the columns of Step 3 commenced. The temperature and pressure



STUFFING BOX
MAT. 18-8 S.S. - 1 REQ.
SCALE - FULL SIZE

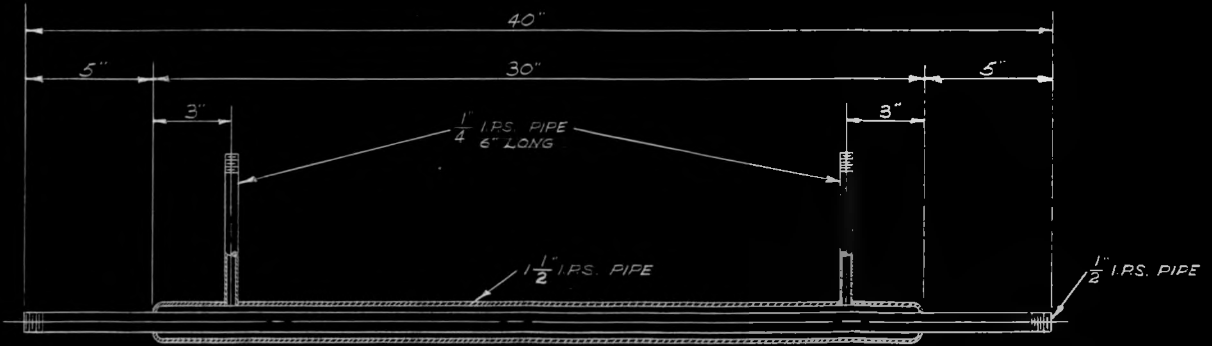


KETTLE
MAT. 18-8 S.S. - 1 REQ.
SCALE - 3" = 1 FT.



NOTE: HEATING JACKET TO BE
WIRED ON 3 HEAT SWITCH

HEATING JACKET
1 REQ'D.
SCALE - 3" = 1 FT.



NOTE: FOR DRY ICE TRAP [illegible]
SEE DWG. # 381-19-0

CONDENSER
MAT. 18-8 S.S. - 1 REQ.
SCALE 3" = 1 FT.

REVISION	REFER. DWG.
	385-19-0
	381-19-0
APPROVED	

AMERICAN CYANAMID COMPANY
DEVELOPMENT ENGINEERING DIVISION
STAMFORD, CONN.

K SALT RECOVERY
EQUIPMENT

31-232-64

DESIGN: M.S.
DRAWN: wel
SCALE: AS SHOWN
DATE: 6/28/46

DWG.	JOB	REV.
389	19	0

114

DECLASSIFIED

must be watched very closely at this point. Since the flow must be held down in the columns, as mentioned in Step 3, until Chlorthane begins to condense, the pressure tends to build up in the kettle. If such is the case, the heat must be reduced manually since the controls are not automatic.

The temperature of the pot is gradually raised to keep the desired flow in Step 3. As the temperature approaches 200°C., the temperature of the line from the condenser should be noted with the hand. If the line is hot, it is a sign that hot sulfuric acid is getting by the condenser. This acid mixture will solidify in the dry-ice trap and plug the system. If the run is almost completed, it should be stopped. Otherwise the pot should be cooled, opened, more fuming sulfuric acid added, and the run continued. Such an occurence will never happen if the salt is properly dried.

When the temperature reaches 235-250°C., the flow will suddenly drop. The heat is cut off at 265°C., the rock wool remved so the kettle can cool, and the system blown with dry air for twenty minutes at the same rate mentioned for Steps 2 and 3 (20 mm. on a "standard" flowmeter). This length of time is only sufficient to purge the kettle. If the columns are to be discharged, they must be blown separately according to regular procedure.

The final operation is to discharge the kettle and trap. To empty the kettle, the load hole is opened, and a discharge pipe which reaches to the bottom of the kettle is screwed on. This is connected, via black iron pipe, to a barrel, placed just outside the building, containing a caustic solution. Next the valve in the exit line is closed, and air is blown into the pot forcing the hot, liquid residue over into the caustic where it is neutralized. No one should be near the barrel, since the solution will spatter during the mixing operation if the acid residue is very hot, and the caustic solution very concentrated.

The liquid in the dry-ice trap is drained into a dry-ice cooled monel receiver. The receiver is removed, capped and weighed. It is then taken out-of-doors, and the acid mixture allowed to vaporize to the atmosphere. Great care must be exercised in handling this material. It is extremely corrosive and in contact with the skin results in an instantaneous and very painful burn.

The columns have to be discharged only every fifth run; hence they are not blown separately after each run. It is advisable to leave the heat on the columns while the kettle and trap are discharged. Then as soon as the kettle has cooled to 50°C., it may be recharged and another run commenced at once.

The time requirements are given on the data sheet below.

<u>Variables and Variations</u>

1. The acidic pot residue may be stored and disposed of at a later date, instead of neutralized and discarded. In such a case it must still be blown from the kettle while hot, because upon cooling it turns to a solid mass.

2. Oxide obtained from the Distillation step (see section on "Distillation", p.600) may be added to the salt and sulfuric acid. In this case more acid is required and less HF is formed. See Ref. 499 for a complete discussion of this case.

Table XXII

Log Sheet for Salt Recovery

RUN 69-199-1

CHARGE { 4700 g. Salt (191, 192)
18 lbs. fuming H_2SO_4
DISCHARGE
ANALYSIS
TRAP LIQUID

KETTLE: Salt Recovery

Date	Time	Kettle Temp.	Kettle Press.	Variac	Remarks	
4/16/46	2100	51			Start stirring.	
	2120			80	#3 heat on.	
	2230	80	15	80	To #2 heat.	
	2300	155	14	80	To #3 heat; full flow in Step 3.	
	2340		9	80		
	2430	224	9-1/2	80		
	0110	266	2	-	Heat off.	2537
	0130	265	1-1/2	-	Air on.	1704
	0200	-	-	-	Air off.	833 g. in HF trap.

RUN 69-199-2

CHARGE 4700 g. Salt (189, 190)
18 lbs. fuming H_2SO_4

Date	Time	Kettle Temp.	Kettle Press.	Variac	Remarks	
	0500	50	-	80	Start; #3 heat.	
	0530	65	5	80	To #2 heat.	
	0630	120	14-1/2	80		
	0700	150	8	80	To #3 heat; full flow in Step 3.	
	0730	173	6	80		
	0830	220	12	80		
	0850	250	-	-	Heat off.	4103
	0925	266	-	-	Air on.	3336
	0940	-	-	-	Air off.	767 g. in HF trap.