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DECLASSIFIED

Operational Manual

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FILE

Route List

1. ~~F. Prudicts~~
2.
3. ~~H. M. Walker~~
4. ~~Buck~~ 15124
5.

DATE Undated

SUBJECT OPERATIONAL MANUAL

OPERATION 5

To File

FROM Author not given

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Operational Manual

Operation 5

Index

Operation 5

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The function of Operation 5 is to prepare plutonium tetrafluoride from purified plutonium oxalate. The main steps in the hydrofluorination operation are:

I. Receiving of material from Operation 4 and preparation for slurry transfer.

II. Transfer of the plutonium oxalate slurry from the purification container into Pt-Rh boats.

III. Drying and ignition of the oxalate slurry to the oxide.

IV. Cooling and weighing of the oxide.

V. Hydrofluorination of the oxide to the tetra-fluoride.

VI. Weighing of the fluoride.

VII. Transfer of the fluoride to the reduction bottle and sending it to storage or Operation 6.

VIII. General clean-up of the apparatus.

I. Preparation for Slurry Transfer:

Purpose:

The plutonium is received as an oxalate slurry in a 1000 ml. calibrated florence flask. The total volume of material varies from 300-800 ml., depending on the partical size and the amount of excess water. A very bulky oxalate will be dark green in color while the more dense oxalate will be black.

The slurry flask sets inside of a lucite can which is delivered in a boron lined can. The boron lined can is placed on a space unit square which is painted on the floor in front of the dry box unit.

[redacted]

Procedure:

1. Obtain a data sheet (see appendix, Fig. VI) from the file at the control desk. Record lot number, time in, boat weights and numbers, and ~~schedule number~~.

2. Turn on vacuum pumps and hood lights. The switches are on the outside of the hood.

3. Remove cover from boron lined can.

4. Open air lock door M, Fig. I. Take lucite can from the boron lined can and place on the shelf attached to door N, Fig. I.

5. Close door M, Fig. I.

6. ~~Using the glove ports in the wet transfer box, remove the protecting tube from the stirring rod and delivery tube and place it in the pan in the bottom of the wet transfer~~ box. Open door N, Fig. I, take the lucite can and place it in the holder on the transfer device.

7. Unscrew the four wing-nuts and remove the cover from the lucite can. Place the cover and nuts in the tray in the bottom of the wet transfer box.

8. Remove the ground glass jointed cover from the slurry flask and place in the tray.

9. Referring to Fig. I, raise the slurry bottle by use of hand-wheel E until the ground glass joint of the slurry flask forms an air-tight seal with the ground joint of the transfer apparatus at H.

[redacted]

II. Wet Transfer Operation:

Purpose:

The oxalate slurry containing about 100 ml. of excess water is transferred by means of a compressed air-vacuum system from the slurry bottle through the delivery tube into two Pt-Rh boats (7"x 2"x 1½"). The boats are held in a nickel scow which sets in a cradle (A, Fig. I). This cradle can be moved horizontally by turning wheel B, Fig. I. The valves and levers operating the transfer apparatus are located on the outside of the wet transfer box as shown at C and D, Fig. I.

All but 100-200 mg. of the oxalate can be transferred.

Procedure:

1. Adjust the delivery tube so that its tip extends to the bottom of the slurry flask.
2. Adjust the height of the out-lot tip of the delivery tube, by use of handwheel I, Fig. I, so that it is about 1/4" below the top of the boats.
3. Start stirrer. Switch is on the outside of the hood (J, Fig. I).
4. Open air pressure valve at O, Fig. I and start forcing the slurry through the delivery tube into the boats. Regulate the pressure by use of the bleed (K, Fig. I).
5. After the excess water has been forced over into the boats, close the air pressure valve (D, Fig. I), and open the vacuum line at C, Fig. I. By adjusting the height of the tip of the delivery tube with the handwheel (I, Fig. I), suck the excess water back into the slurry flask.

[redacted]

II. Procedure (cont.)

6. Repeat steps 4 and 5 until as much of the slurry as possible has been transferred to the boats, being careful to move the position of the boats between passes, by means of wheel B, Fig. I, so that there is approximately the same amount of material in each boat. About 10-15 passes will probably be required.

7. Suck as much of the excess water as possible from the slurry in the boats, being sure to leave no liquid in the delivery tube when finished.

8. Open sliding door L, Fig. I and run the boats out on to the cradle in the air lock. Place boat covers on the boats.

9. Start the drying and ignition operation. Complete this operation after the drying and ignition has been begun.

10. Lower slurry flask on transfer device with handwheel E, Fig. I.

11. Wash stirring rod and delivery tube with distilled water. Add water to bring the volume in the slurry flask up to 150 ml.

12. Place glass cap on slurry flask and fasten the lucite cover in place.

13. Remove the lucite can from the wet transfer box through door N, Fig. I and then out of the air lock through door M, Fig. I. Place in the boron lined can and send to Operation 4.

7

III. Drying and Ignition

Purpose:

The scow and the boats are moved out of the air lock into the hood unit into another cradle. Here the scow is slid out of the cradle into a ~~quartz~~ Ni tube which sets inside of an electric furnace. The temperature of the furnace is controlled by a Wheelco program controller. This program, (D-2), gives the following temperature-time cycle:

130° C	for	3 hrs.
130 - 250	in	1/2
250 - 310	in	1-3/4
600	for	3/4
		6 hrs. total

During the drying cycle air is drawn over the boats at a constant rate of one liter per minute by means of a vacuum pump. The hot gases from the furnace tube pass through a water cooled condenser followed by a glass wool filter before reaching the pump.

Procedure:

1. Move cradle (containing nickel scow with boats of oxalate slurry) through door R, Fig. II into the hood unit and in front of the drying and ignition furnace, Fig. II.
2. Close air lock door R, Fig. II.
3. Slide the nickel scow out of the cradle and into the ~~quartz~~ Ni tube (A, Fig. II).
4. Take tube cover from pan B, Fig. II and place over the end of the quartz tube.
5. Adjust the air flow in the furnace by means of the valve I, Fig. II so that the flowmeter J, Fig. II indicates a flow of one liter per minute.

[redacted]

III. Procedure (cont.)

6. Open circulating water valve near air valve I, Fig. II until the rotasight (X, Fig. II) indicates a flow of one liter per minute.

7. Rotate program disk E, Fig. V to the start of the drying and ignition program.

8. Standardize program controller (L, Fig. V). See Wheelco Instructions in appendix.

9. Start the drying and ignition program by throwing switch I, Fig. V down.

10. Record the necessary data on the lot data sheet at the control desk A, Fig. V.

11. Continue with step 10 under the wet transfer procedure.

12. Take readings every half hour; recording the data on the log sheet at the control desk.

13. At the completion of the temperature-time cycle:

    a. Shut off switch I, Fig. V at control panel.

    b. ~~Remove quartz tube cover and place in pan B, Fig.~~ V.

    c. Pull nickel scow containing boats with the oxide out of the furnace and onto the cradle.

14. Replace ~~quartz tube cover~~.

15. Shut off vacuum pump (switch is on outside of hood).

16. Shut off condenser water.

17. Condensate collected during the run is to be sent to Operation 2[illegible].

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[redacted]

Operation 5

IV. Cooling and Weighing of the Oxide

Purpose:

After the cradle containing the scow, boats and oxide has cooled to room temperature, it is moved onto the balance (Fig. II) and weighed to the nearest 0.1 gram.

Knowing the weight of the empty boats, scow and cradle, the weight of oxide is determined by difference. From this weight the amount of metal in the lot is calculated and also the theoretical amount of fluoride. (See sample data sheet and calculations in appendix.)

Procedure:

1. After the material has cooled to room temperature, move the cradle and its contents onto the long pan of the torsion balance (Fig. II).
2. Unlock the balance and weigh to the nearest 0.1 gram.
3. Lock balance.
4. Record weight on the lot data sheet at control desk.
5. Slide cradle in front of hydrofluorination furnace (Fig. II).

V. Hydrofluorination

Procedure:

1. Open reaction tube cover (C, Fig.

2. Slide scow containing boats with the oxide on to the cradle and into the nickel reaction tube (D, Fig. II).

3. Bolt on reaction tube cover (C, Fig. II).

4. Adjust the water flow to the absorption tower (F, Fig. II) to two liters per minute as indicated by the rotasight (G, Fig. II).

5. Adjust O_2 flow to 300 cc per minute by means of valve J, Fig. V, and hot wire flowmeter H, Fig. V at control panel.

6. Check program disk E, Fig. V to see that it is set for the hydrofluorination cycle.

7. Turn on controller by use of switch I, Fig. V at control panel.

8. When recorder B, Fig. V shows furnace temperature to be about 200° C, open valve K, Fig. V, thereby turning in H.F. Check the HF flow with the hot wire meter at control box H, Fig. V.

9. Note H.F. line pressure at C, Fig. V. If it falls below 3 p.s.i. cut in a fresh tank.

10. Take readings of HF pressure, water flow to absorption tower, O_2 flow, HF flow, and furnace temperature every half hour and record on data sheet at control desk.

11. At the completion of the ~~10~~ 6 hour temperature-time cycle turn off switch I, Fig. V.

12. Open the top half of the furnace as shown in Fig. II and turn on cooling fan H, Fig. II.

V. Procedure (cont.)

13. When the reaction tube has shown by recorder B, Fig. V, shut off [illegible] ([illegible], Fig. V).

14. Wait at least one minute after the [illegible] has been turned off, then shut off O_2 flow (valve J, Fig. V).

15. Shut off fan and water flow to the absorption tower.

16. Open reaction tube cover (C, Fig. II).

17. Pull out nickel scow, containing boats and the fluoride, onto the cradle in front of the furnace and allow to cool to room temperature.

18. Replace reaction tube cover (C, Fig. II).

19. Close top half of furnace on reaction tube.

VI. Weighing of the Fluoride

Purpose:

The fluoride is moved onto the torsion balance, Fig. II, and weighed. From this weight and the previous weight of the oxide, the per cent conversion can be calculated. (See appendix A).

Procedure:

1. Move the cradle and its contents onto the long pan of the balance, Fig. II.
2. Unlock the balance and weigh to the nearest 0.1 gram.
3. Lock balance.
4. Record weight on data sheet at the control desk and calculate the per cent conversion.
5. Remove weights and slide cradle in front of air lock door R, Fig. II.

VII. Removal of the Fluoride

Purpose:

The fluoride is moved through the air lock into the dry transfer box, Fig. I. Here the material is transferred from the boats into the reduction bottle with the aid of a lucite dumping apparatus. The fluoride is then removed from the apparatus and sent to Operation 6.

Procedure:

1. Start vacuum pump.
2. Obtain a boron lined can which goes between Operation 5 and Operation 6.

[redacted]

VII. Procedure (cont.)

3. Remove the reduction bottle from the can and place it in the air lock through door M, Fig. I.

4. Move the bottle through door O, Fig. I into the dry transfer unit and place it in the dumping device at P, Fig. I.

5. Move the scow through door R, Fig. II into the air lock, then onto platform S, Fig. I in the dry transfer box. Remove boat covers in air lock.

6. Take one boat from the scow and place it in the dumping apparatus at T, Fig. I.

7. Dump the fluoride in the boat by rotating transfer cover 180°.

8. Remove the boat from the dumping apparatus and loosen any material clinging to the boat.

9. Repeat steps 6 and 7.

10. Remove the boat and place in scow.

11. Dump contents of the second boat (steps 6, 7, 8, 9 and 10).

12. Remove reduction bottle from the dumping apparatus and break up any lumps.

13. Move cradle containing the scow and boats into the air lock.

14. Screw on cap and remove reduction bottle from the dry box and place in the boron lined can. 7

15. Move scow and the boats into the wet transfer box.

16. Send the fluoride with the proper date to Operation 6.

Data required:
a. lot number
~~b. schedule number~~
c. weight of Pu
d. weight of Fluoride
e. per cent conversion
f. container number

[redacted]

VIII. General Clean-Up of Equipment

The outside of the hood and dry box unit is cleaned with special solutions furnished by the H.I. group.

After the cleaning is completed the H.I. group will check the unit for hot spots.

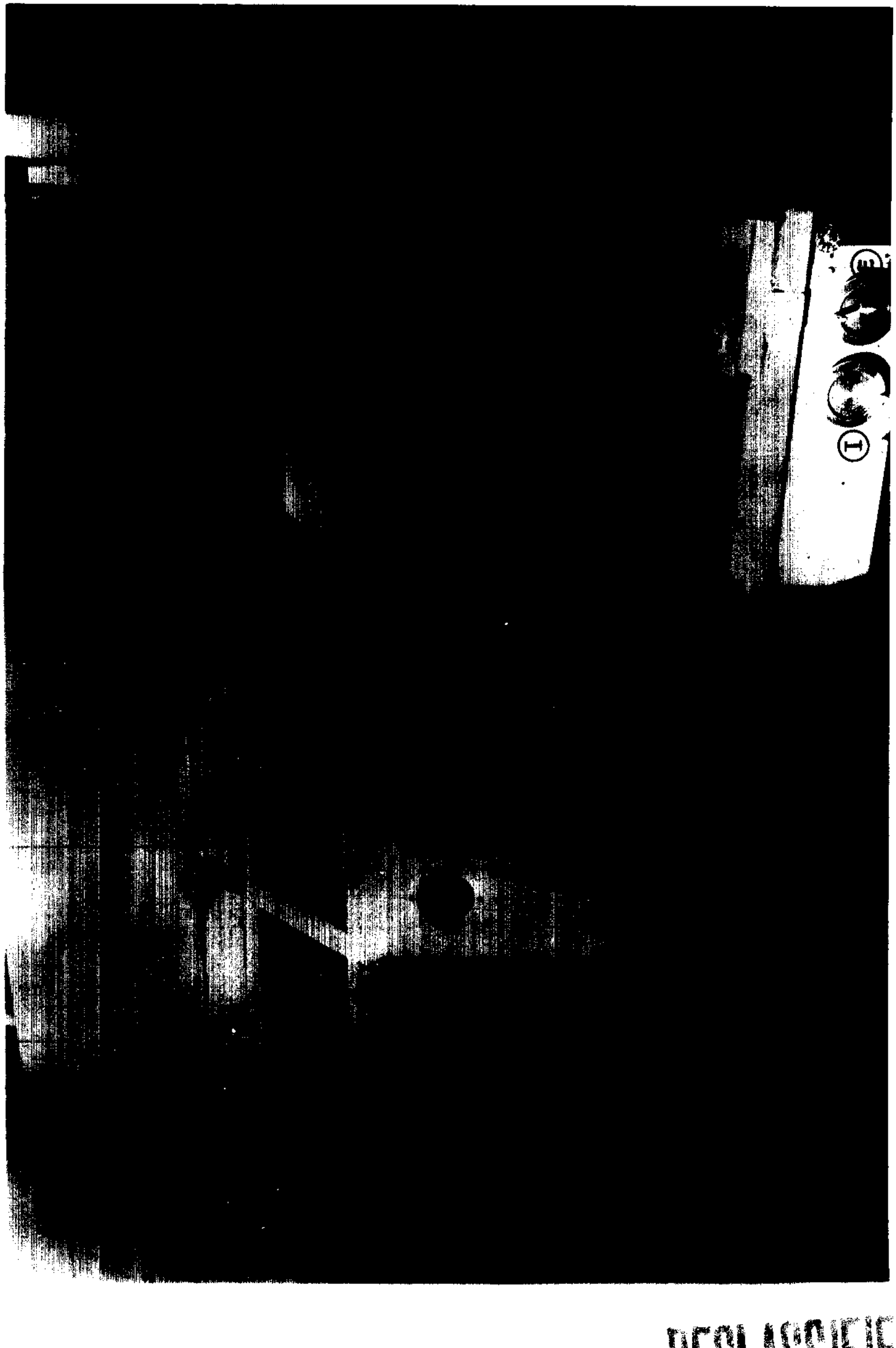


FIGURE I

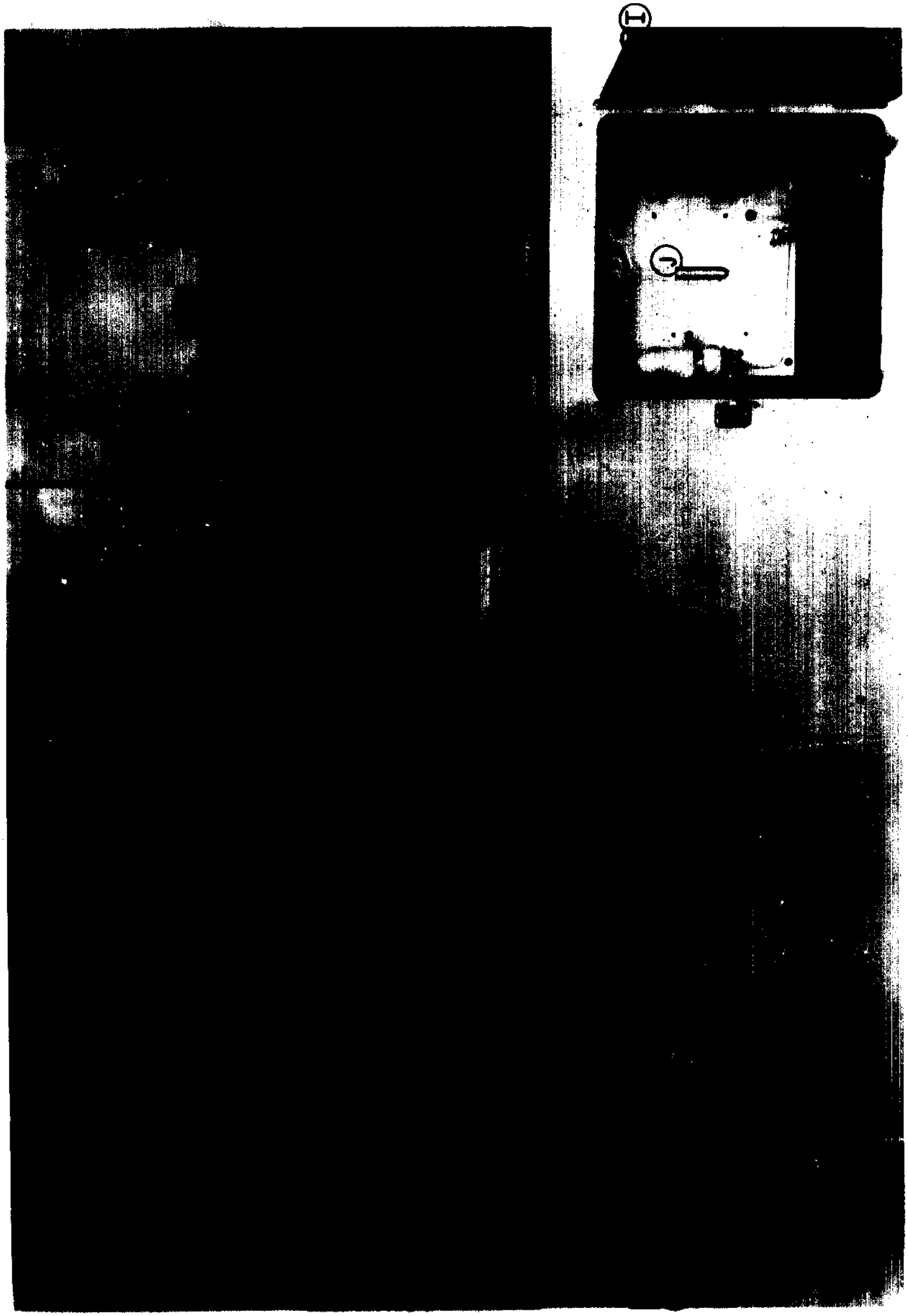
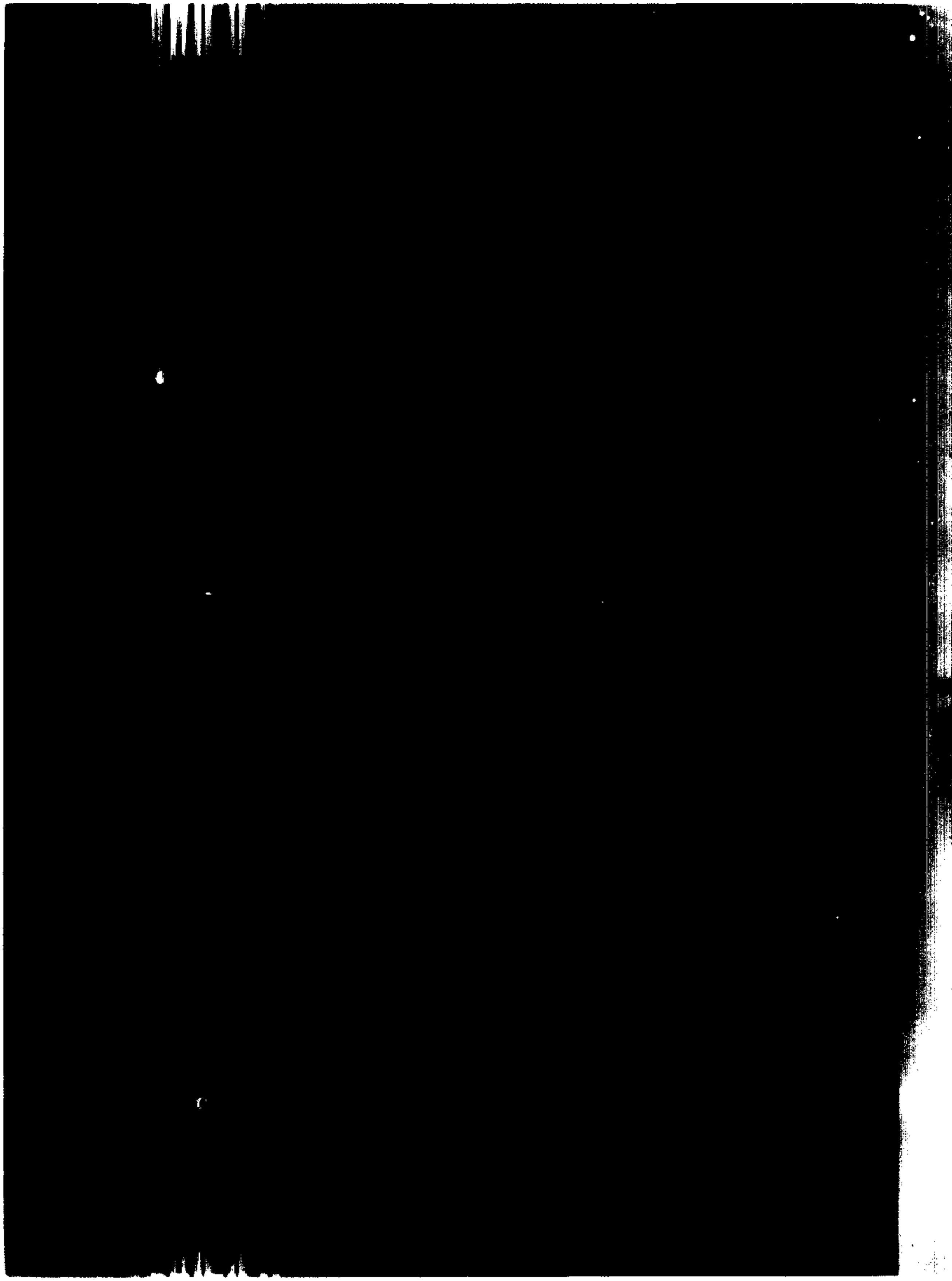


FIGURE 2

 [redacted]



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Appendix

A. Lot Data Sheet, Graph and Calcul[illegible]

B. Hot Wire Flowmeter; principal of operation and construction

C. HF and O_2 Supply

D. Wheelco Instruction Sheet

E. List of Equipment

A. Calculations, Operation 5

Upon arrival of the oxalate slurry, the Lot, [redacted], and Source Numbers are noted at the top of the data sheet. The time of arrival, the "L" box containing the batch, and the unit in which the batch is to be processed are also noted.

The weight of the empty boats and cradle is determined. After the oxalate has been dried and converted to oxide in the desired number of hours, the boats, cradle and oxide are weighed. The difference in weights is the weight of oxide, assumed to be 100% converted.

Because of poorer hydrofluorination yields on oxides which have been ignited at 900° C, it has been found expedient to heat to only 600° C, and then apply a correction factor to the weight of oxide at 600° C to convert to equivalent weight at 900° C. It was found by testing several 160-gram batches that an average of 0.65% of 600° C oxide weight was lost in igniting to 900° C. To correct the weight of oxide to 900° C then, the weight of oxide determined by weighing is multiplied by 99.35% or 1/1.0065. This new oxide weight is then called the "corrected weight of the oxide."

Since the molecular weight of the oxide is 271 and the molecular weight of "element" is 239, the equivalent weight of "element" in the oxide is found by multiplying the corrected oxide weight by 239/271 or 1/1.1339. Similarly the molecular weight of tetrafluoride is 315 and the theoretical weight of tetrafluoride (100% conversion) is determined by multiplying the corrected weight of oxide by 315/271 or 1/0.86032.

After the oxide has been hydrofluorinated for the desired time,

[redacted]

A. (cont.)

the product is removed and the sum of "boats, cradle and crude tetrafluoride" determined by weighing. The original weight of empty "boats plus cradle" is subtracted and the difference is the weight of "Tetrafluoride."

The ratio of metal in the fluoride is found by dividing the weight of element by the weight of crude tetrafluoride and the result, multiplied by 100, is the "% metal." From a graph previously constructed to cover the range of conversions from 58% to 100%, the "% conversion" is found. Such a graph, plotting "% metal" versus "% conversion" accompanies this outline.

A summation of the critical facts concerning the batch is then made by noting "weight of element," "% conversion" and "weight of tetrafluoride" at the top of the data sheet. The 5 "[redacted]" box is which the batch is removed, the time of removal, and the total time in "dry chemistry" of that batch are also noted.

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[redacted] [redacted]

[redacted] 647-P WT. ELEMENT [redacted]

SO. MD. # 19-o % CONVERSION 98.4%

SOURCE # 131-H WT. FLUORIDE 195.8

DRYING PROC # D-2 6 hrs. HF PROC # HF-3 10 hr's.

L BOX IN 4-1 TIME IN 0800, 30/10/45

L BOX OUT 5-1 TIME OUT 0315, 31/10/45

UNIT # 1 TOTAL TIME 19½ hr.

Boat # 11 and 12	TOTAL
Boats + Cradle + Oxide	847.5
Boats + Cradle	677.4
Wt. Oxide	170.1
Cor. Wt. Oxide ($^1/_{1.0065}$)	168.8
(1) Element ($^1/_{1.1339}$)	148.9
Th. Wt. F_4 ($^1/_{.86032}$)	196.2
Boats + Cradle + F_4	873.2
Boats + Cradle	677.4
(2) Wt. F_4	195.8
% Metal (1)/(2)	76.0%
% Conversion	98.4%

Fig. VI

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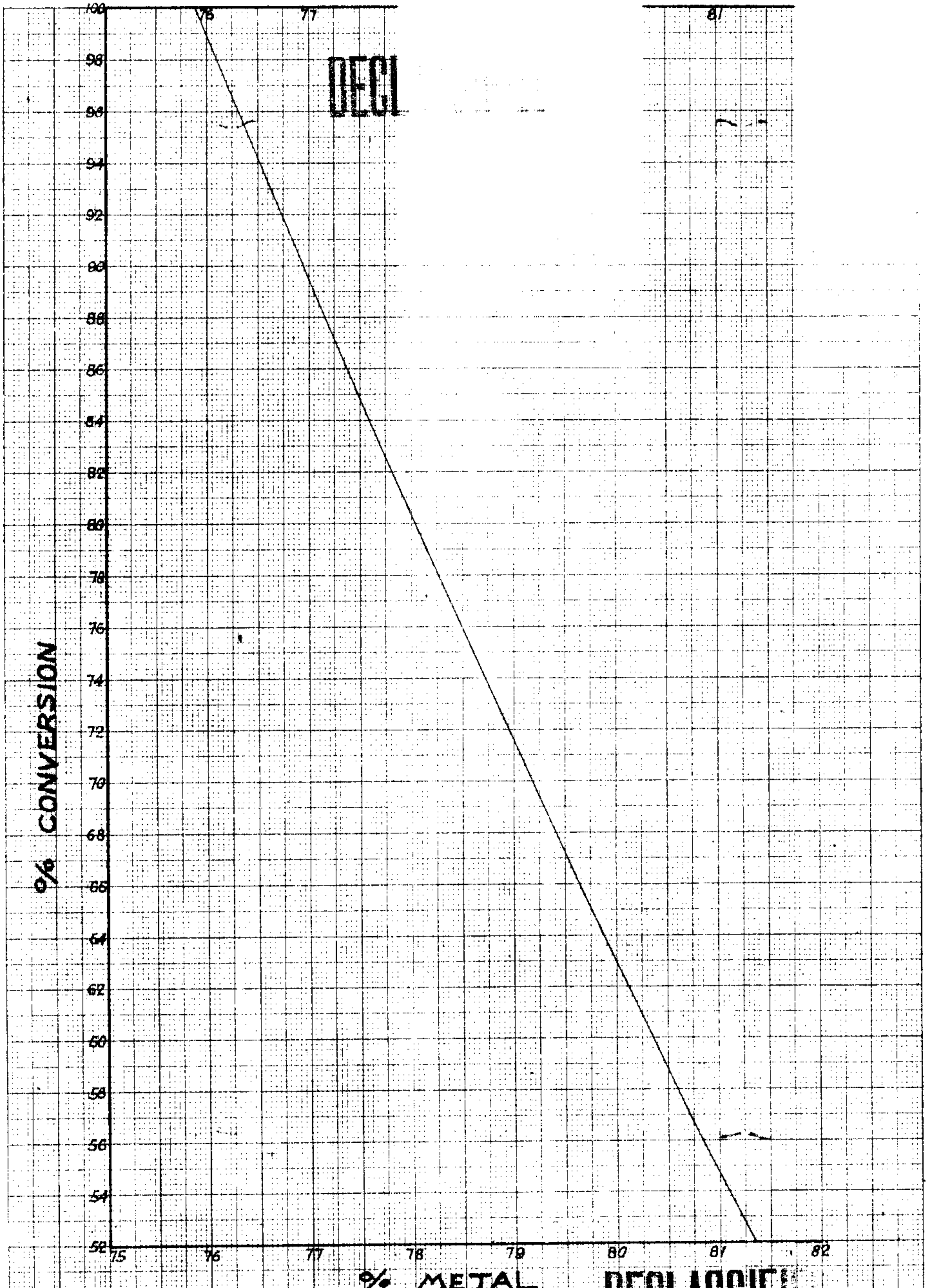
[redacted]


% CONVERSION
% METAL
100
98
96
94
92
90
88
86
84
82
80
78
76
74
72
70
68
66
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81
82

[redacted]

B. <u>The Thermocouple Type Hot-wire Flowmeter</u>

The thermocouple type hot-wire flowmeter is primarily suited for measuring rates of gas flow. In Operation 5 it is used to meter the oxygen ~~and the HF~~ to the hydrofluorination furnaces.

A constant current is passed through a 12 mil platinum heater wire which is perpendicular to the gas flow. A special gold-palladium, platinum-rhodium thermocouple is spot-welded to the heater wire. An orifice plate is placed in the line ahead of the wires and is lined up so that the gas passing through the orifice will flow across the thermocouple-heater wire junction.

Drawings number 38, 39, 40 and 41 on the following pages give the detailed construction of the meter. Drawing number 42 shows the wiring diagram of the six point control box used in Operation 5. To operate, the desired meter is selected by the six point selector switch. While the push button is held in the variac is adjusted until the ammeter indicates a flow of ~~6~~ 8 amperes. The millivolt meter is read and the e.m.f. reading referred to the calibration curve. After the reading has been taken the variac should be turned back to its zero position.

Using a heater wire 0.5 inches long, a current of ~~6~~ 8 amperes will heat the junction to ~~3~~7 00-400° C with no gas flowing. This temperature will produce an e.m.f. of 30-40 millivolts in the thermocouple circuit. As gas flows over the junction, the heater wire is cooled, in proportion to the amount of gas, velocity (depends on orifice size) and the thermoconductivity of the gas. Accordingly, the change of e.m.f. can be calibrated in terms of flow.

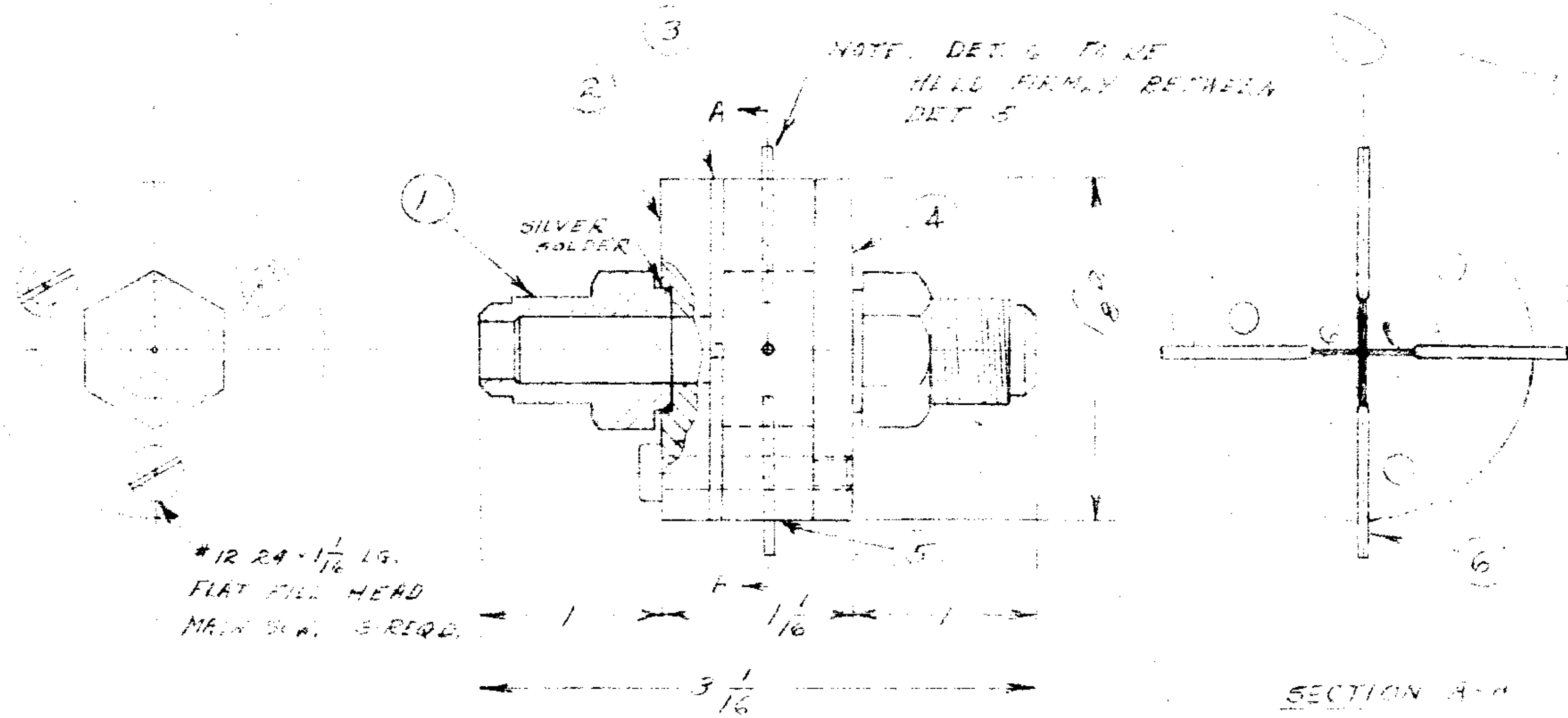
[redacted]

B. (cont.)

In Operation 5 several meters are wired to one electric control box so that one can select and read the desired meter by means of selector switches.

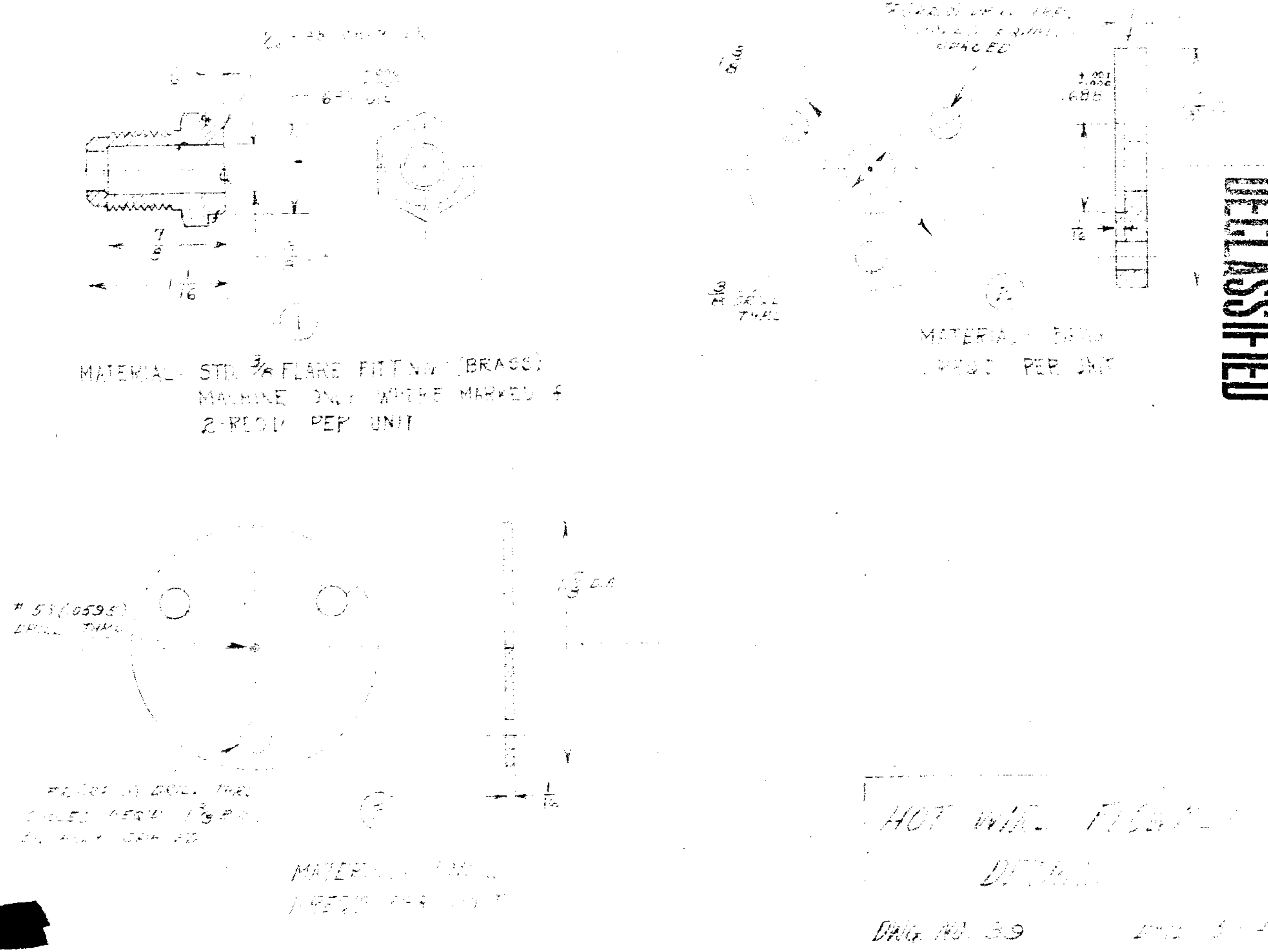
The accuracy of the meter is comparable to other types of flow-meters. Tests have shown these meters give readings within 5% of the true flow over the entire range of the meter.

[redacted]



HOT WIRE FLOW METER

ASSEMBLY



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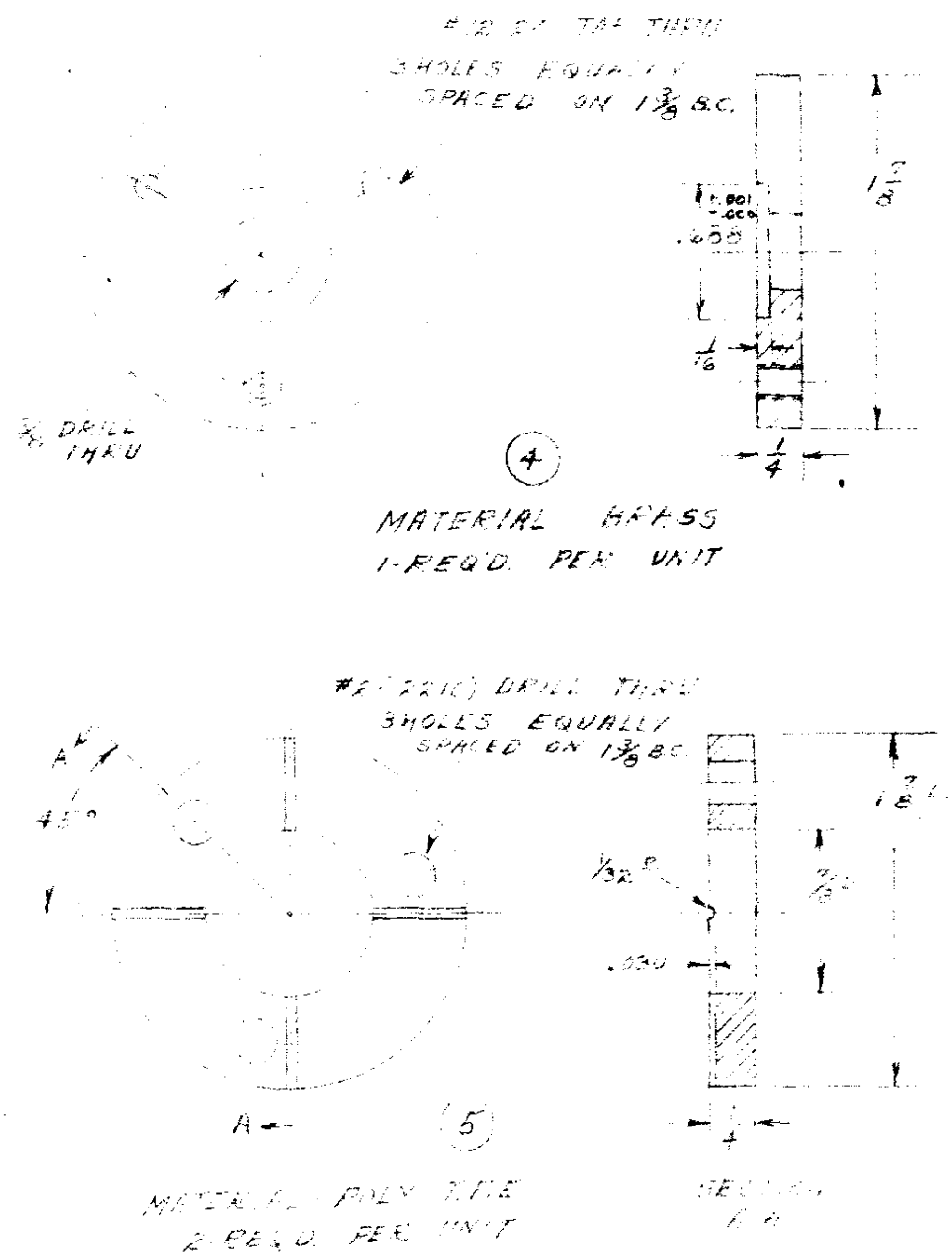

HOT WIRE ...

DETAILS

DWG NO 40

GEH-21,268

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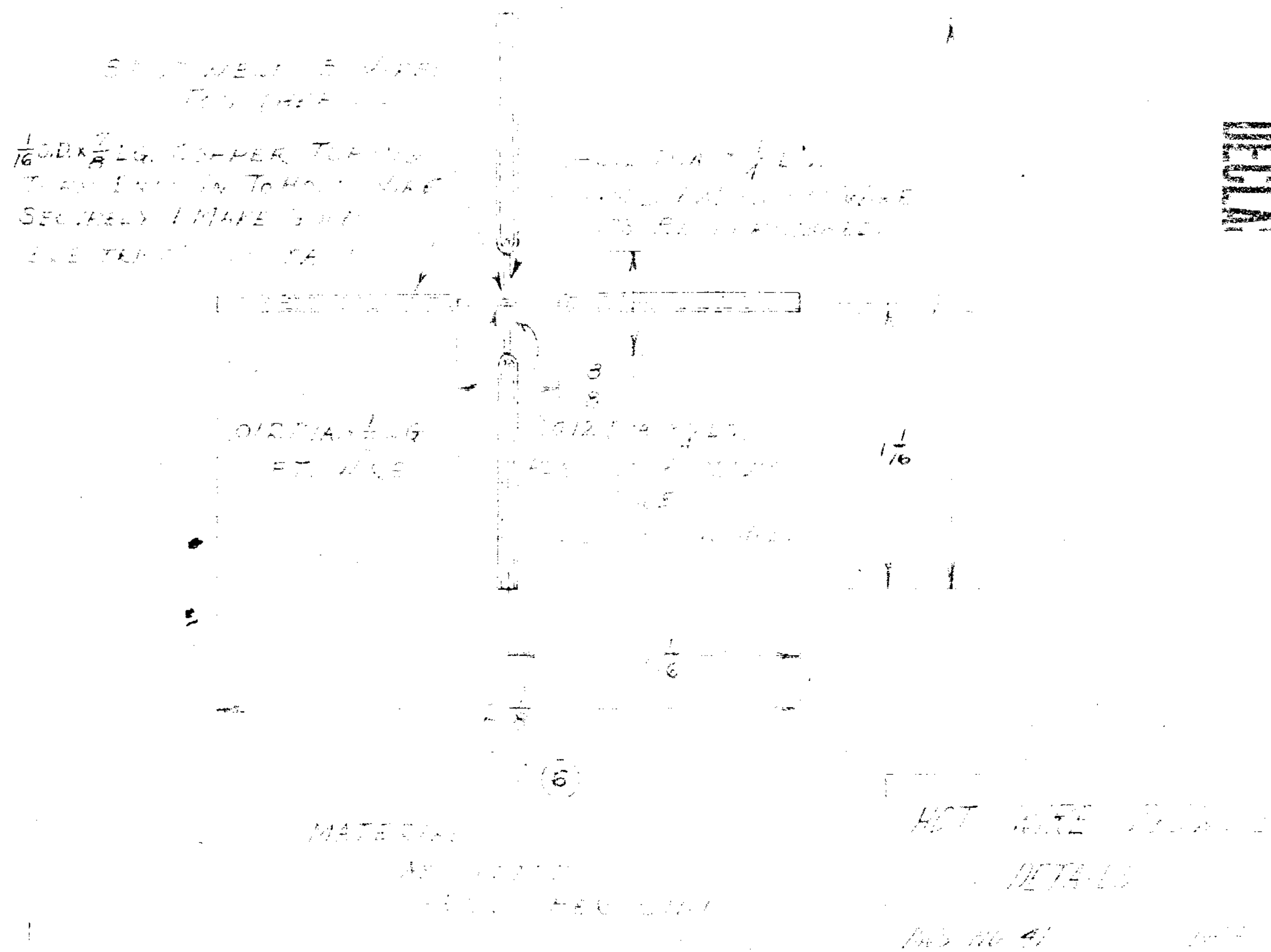

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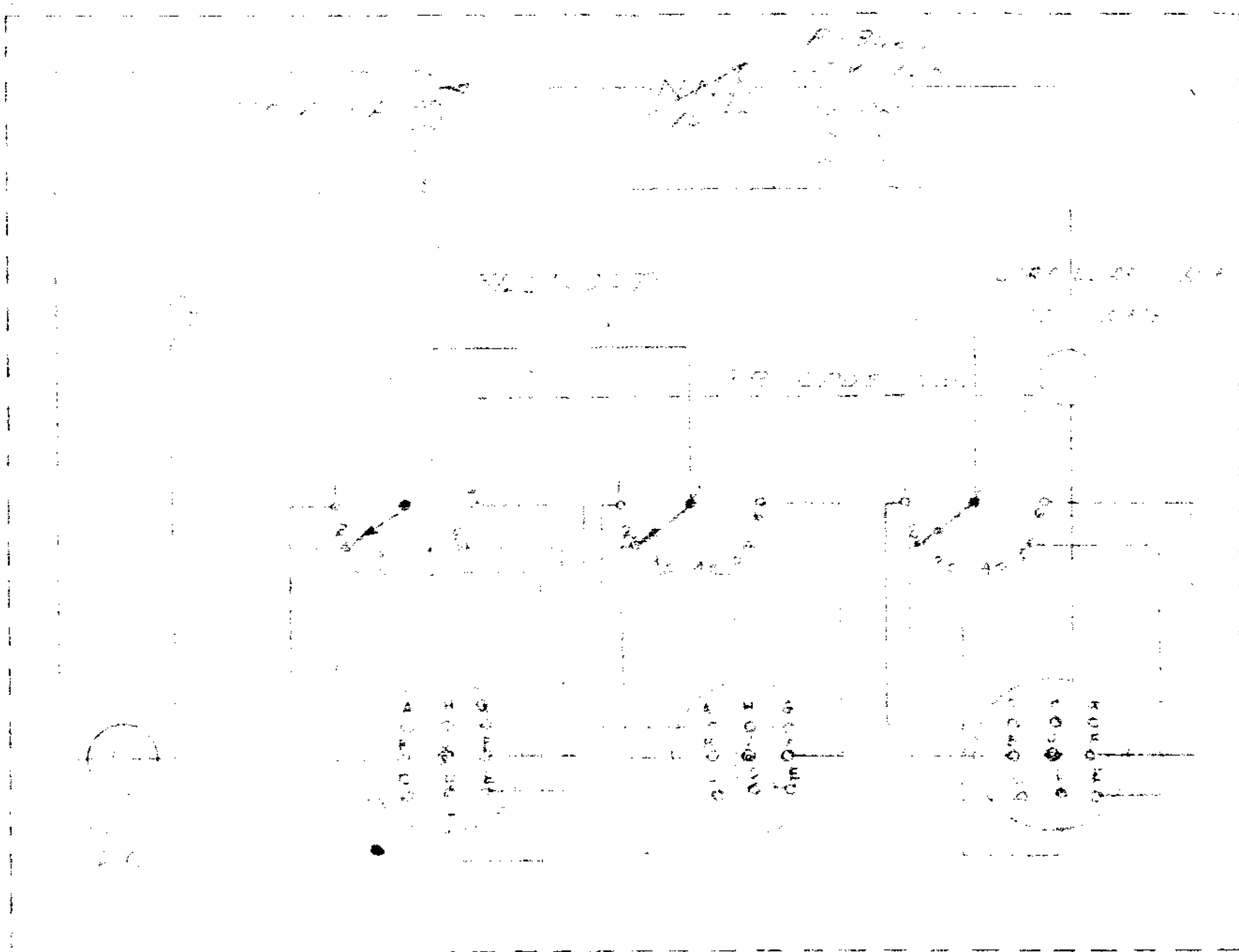

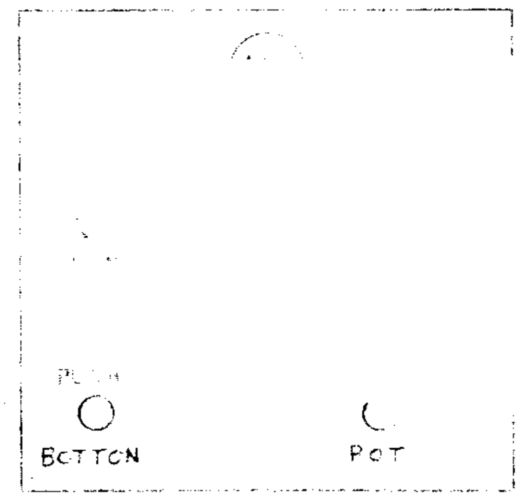

FIT BOX FOR FLUSH MOUNTING

NO DIAL NEEDED ON RE... AND IS OPTIONAL ON THE VARIAC.

MODEL 263 WESTON
0-50 MILLIVOLTS

... TO BE MADE

WIRING DIAGRAM FOR
FLOW METER

DWG NO. 43

C. Oxygen and HF Supply

The oxygen and HF necessary for Operation 5 is supplied from cylinders located in the equipment room.

The oxygen is supplied from one of the two tanks shown in Fig. IV. (The second tank is a spare.) The pressure is reduced to about ~~25~~ 5 p.s.i. by use of the reducing valves connected to the tanks. ~~This pressure is further reduced to 4 p.s.i. by means of a reducing valve located in the main line behind the control panel in the process room.~~

A special distilled anhydrous HF is supplied in cylinders containing 80-85 pounds of HF. The cylinders are connected to a six tank manifold as shown in Fig. III. The manifold is arranged so that a new cylinder can be bled, to remove excess hydrogen, by means of ~~the water aspirator~~ shown at the extreme left in Fig. II.
absorption tower

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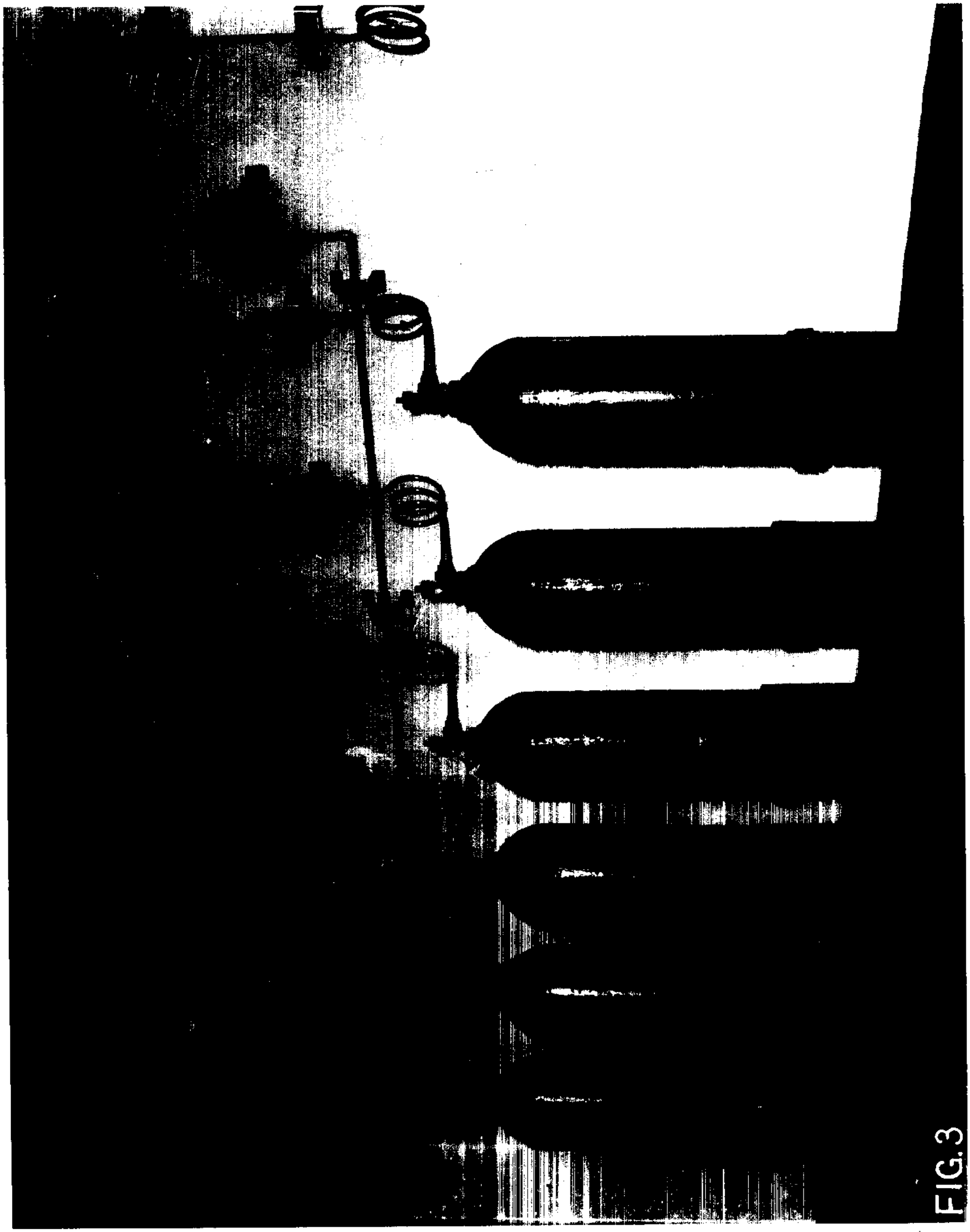

FIG.3

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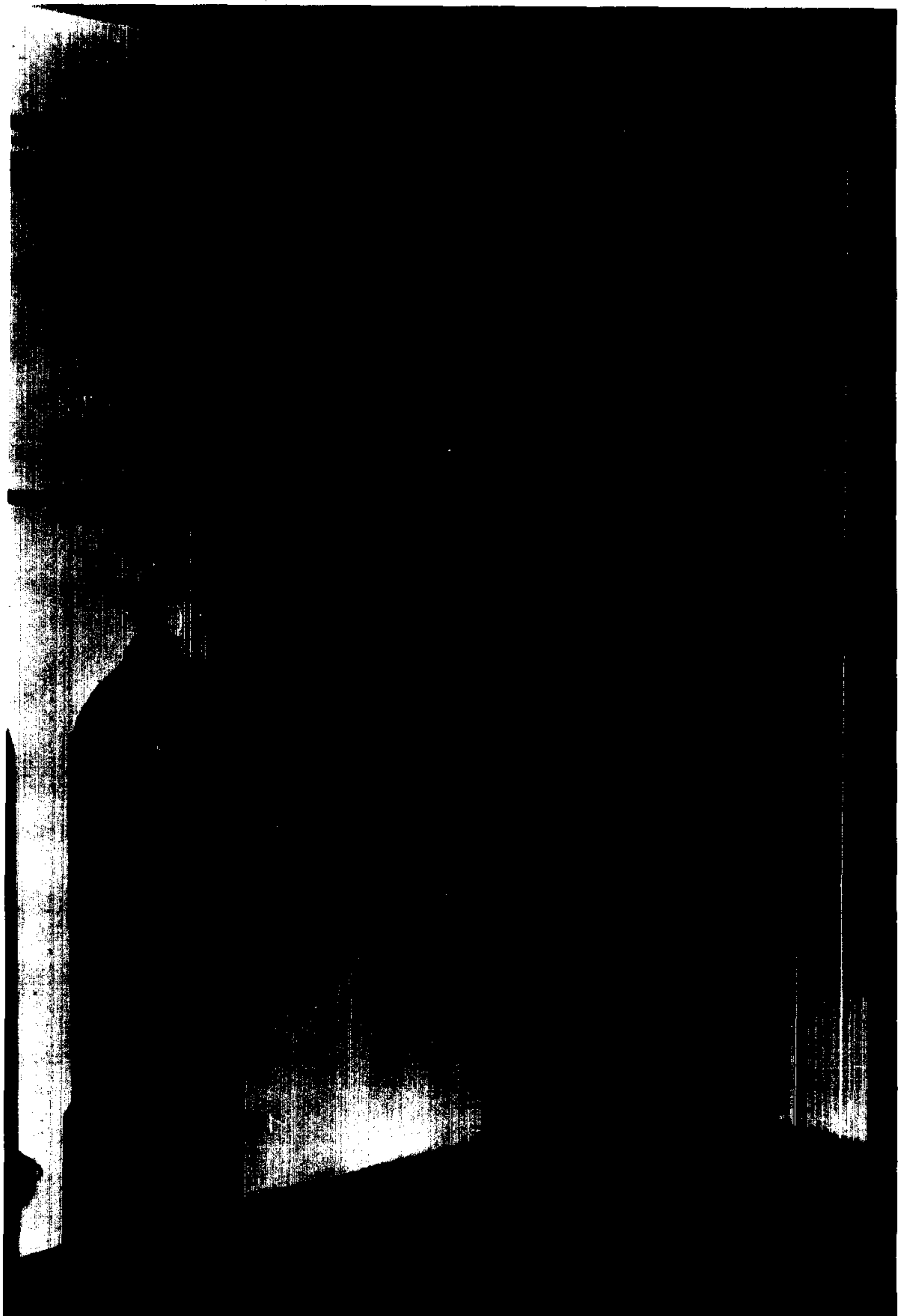

FIGURE 4

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D. Checking and Adjust

On the inside o[illegible] is a standardizing switch lever at the back of the meter [illegible]. This lever has three positions, forward, backward and neutral or reading position.

1. Zero Reading. With standardizing switch lever backward, the galvanometer pointer should come to rest on the zero mark on the right hand side of the scale. To adjust pointer for zero reading, slowly turn the slotted screwhead on bottom of the instrument.

2. Standardizing. Pull and hold standardizing switch forward. The galvanometer pointer should come to the zero reading and come to rest. This should be carefully adjusted by the standardizing rheostat (above the standardizing switch).

3. To check THERMOCOUPLE BREAK PROTECTION (On Models With This Feature) Move setting index all the way up scale. Disconnect thermocouple from connector block, simulating a broken thermocouple. Pointer is supposed to go to the right past the zero mark, operating the control. (If pointer goes to left, T.C.B. leads are either open or reversed).

4. Replacement of DRY CELL. When standardizing rheostat reaches limit of adjustment - replace the dry cell.

Instruction Sheet No. 5044A

Supersedes Sheet No. 5044

WHEELCO INSTRUMENTS CO.