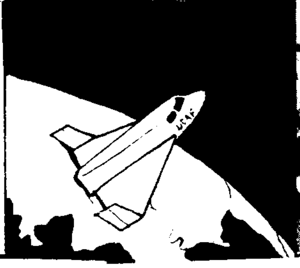

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GPO PRICE $ ______

CFSTI PRICE(S) $ ______

Hard copy (HC) 2.00

Microfiche (MF) .50

ff 653 July 65

N66-14296

(THRU)

34 (PAGES)

1 (CODE)

CR 68975 (NASA CR OR TMX OR AD NUMBER)

06 (CATEGORY)

BROMINE TRIFLUORIDE METHOD FOR OXYGEN IN LIQUID ALKALI METALS

CONTRACT NO.: NASw-882

PERIOD COVERED: (Second Quarter) April 1, 1964 to June 30, 1964

AUTHORS: H Kirtchik/T Lajcik/SL Culp

Materials Development Laboratory Operation

Advanced Engine Technology Department

GENERAL ELECTRIC

CINCINNATI 15, OHIO

July 17, 1964

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Second Quarterly Report
April 1, 1964 to June 30, 1964

H Kirtchik
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SL Culp

Contract No. NASw-882
Control No. 10-2379

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Materials Development Laboratory Operation

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PREPARED FOR: Chief of Materials Research Program, Code RRM, Office of Advanced Research and Technology

NASA, Washington, D. C.

TITLE: BROMINE TRIFLUORIDE METHOD FOR OXYGEN IN LIQUID ALKALI METALS

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W. H. Chang, Manager
Materials Applied Research

DATE: July 17, 1964

ABSTRACT

14296

This report describes the completed apparatus for determining oxygen in potassium by the BrF_3 method. The potassium extruder, the all monel BrF_3 handling apparatus, and the oxygen measuring device are described and pictured.

Calibration of the extruder was done under a vacuum of 4×10^{-8} torr and the weight of potassium extruded can be determined with less than a 1% error in the range between 0.25 and 1.0 grams.

The calibration of the Brady apparatus has shown that less than one part per million of oxygen in a helium stream can be detected and measured.

Author

BROMINE TRIFLUORIDE METHOD FOR OXYGEN IN ALKALI METALS

INTRODUCTION

The whole system now has been completely constructed and assembled. The Brady oxygen measuring system has been calibrated. The extruder turn versus sample weight calibration has been completed. The vacuum requirements of the extruder have been met.

EXPERIMENTAL

I Calibration of Oxygen Measuring System

The Brady apparatus (see Figure 10, First Quarterly Report, April 20, 1964) was calibrated by the coulometric generation of known amounts of oxygen in a specially designed electrolytic cell. The cell was attached to the carrier gas inlet on the apparatus. A known constant current passing through the cell (containing 10% sulfuric acid) for a given period of time yields a definite amount of oxygen. This amount, in accordance with Faraday's law, may be expressed by the equation:

$$\text{Micrograms Oxygen} = .08287\ I \cdot t$$

where I is expressed in milliamperes and t in seconds.

In order to achieve precise results, special techniques (previously not reported) had to be developed in order to eliminate errors arising from:

1. Atmospheric oxygen dissolved in the electrolyte.
2. Incomplete recovery of generated oxygen from the electrolyte.
3. Inability to recover oxygen which clung to the anode in the form of bubbles.
4. Errors due to faulty current regulation resulting from polarization.

Initially, erratic results were obtained due to dissolved atmospheric oxygen introduced into the electrolyte when the liquid level was lowered to free bubbles of oxygen tending to stick to the platinum wire anode. Also, the recovery of oxygen from the cell was very slow since it could only be removed by helium passing over the top of the solution. These difficulties were overcome, as shown in Figure 1, by modifying the previous electrolysis cell (Figure 8, First Quarterly Report). A fine fritted glass tube was sealed into the cell in such a way that purified helium (from the NaK and Ti pre-train) could be bubbled directly through the electrolyte. The tube was positioned so that bubbles of helium rising from it would sweep over the anode, thus removing oxygen bubbles which may cling to the anode. Hoke #482 bellows valves were positioned so that the ratio of helium passing through and over the electrolyte could be controlled. By means of this arrangement, it became unnecessary to change the electrolyte level or expose it to the atmosphere. The removal of dissolved oxygen was also accelerated and amounts of electrolytically generated oxygen in excess of 180 micrograms could be recovered completely in about 45 minutes using a flow rate of about 100 cc/minute.

The problem of current regulation was solved by a special circuit used in conjunction with a standard power supply. A schematic of this circuit is shown in Figure 2. By means of the 3000 ohm potentiometer, the milliammeter may be preset to a given current level. Thereafter, whenever the circuit is closed, a precise current flow can be maintained regardless of the degree of polarization in the electrolysis cell. Current measurements were made with a Simpson Model 37 milliammeter reading from 0-10 milliamperes. The meter was calibrated by measuring the potential drop across a standard one ohm resistance placed in series with it. A precision L & N potentiometer was used to make the measurements. A 1.5 volt dry cell in series with a 10 K ten turn potentiometer was used to supply current for calibration purposes. The resulting calibration curve is shown in Figure 3. While the manufacturer claims an accuracy of 2% of full scale for this meter, it can be seen from the calibration curve that in this case an error of 2.5% was observed at the ten milliampere reading.

FIGURE 1 ELECTROLYSIS CELL
FOR BRADY APPARATUS

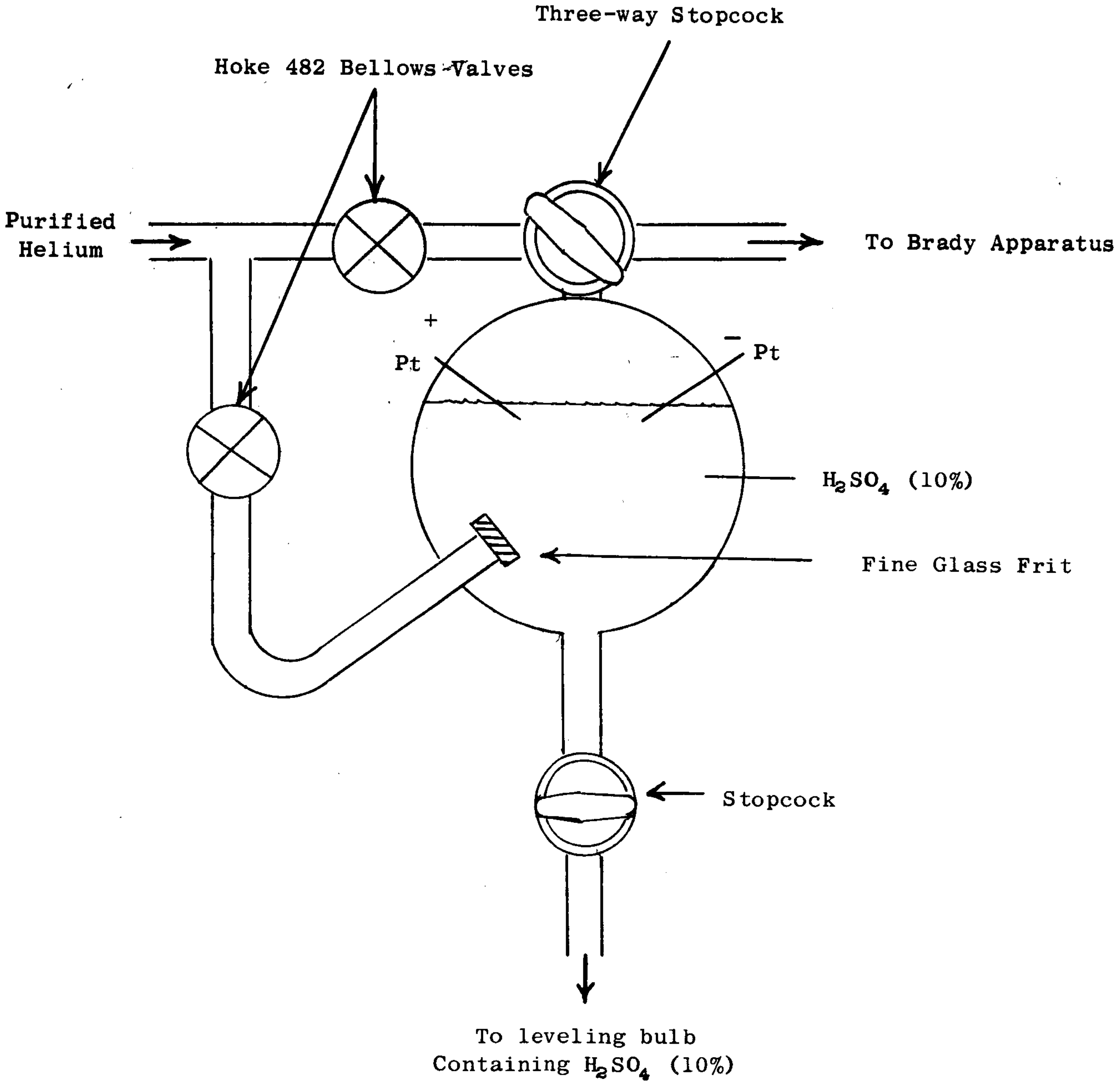
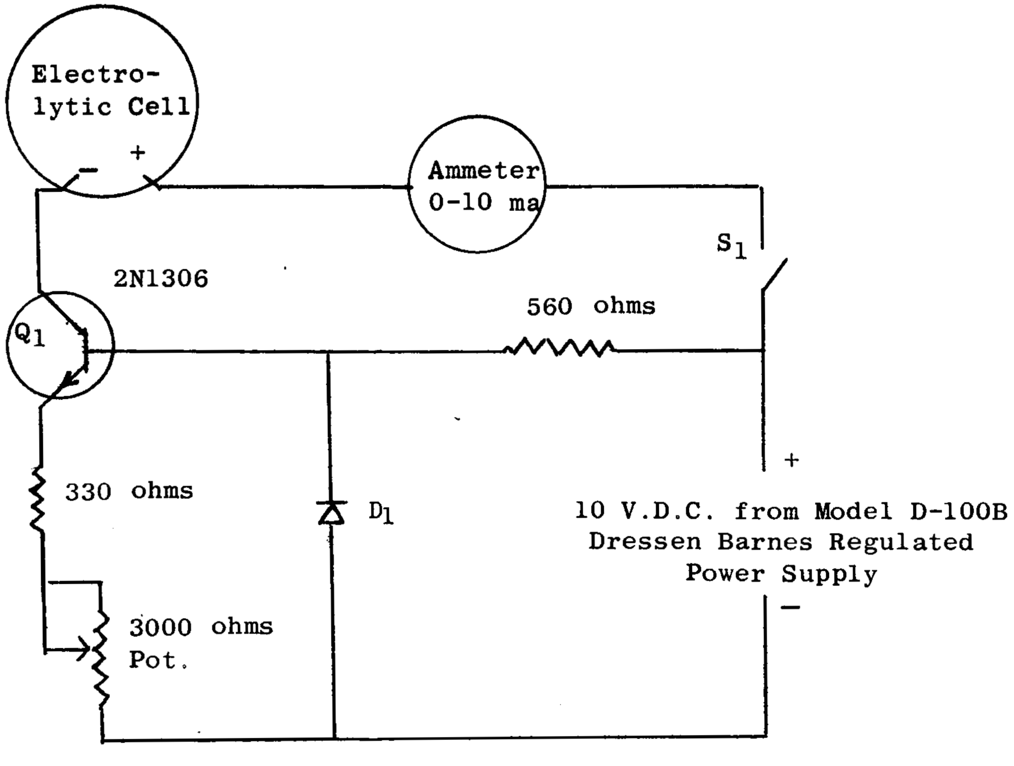


FIGURE 2 CURRENT REGULATING CIRCUIT FOR ELECTROLYSIS CELL
ON BRADY APPARATUS



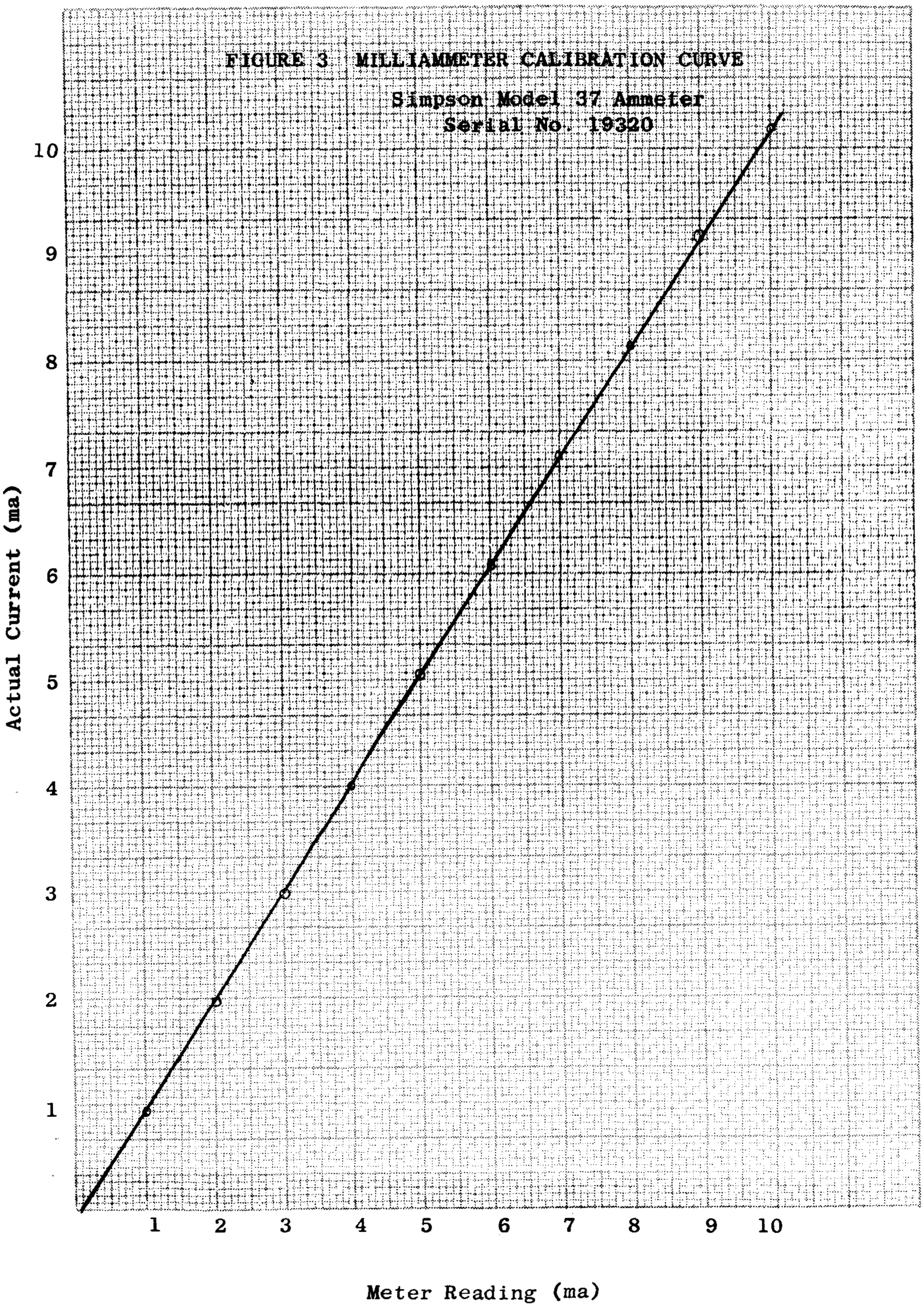


FIGURE 3 MILLIAMMETER CALIBRATION CURVE

Simpson Model 37 Ammeter
Serial No. 19320

The actual oxygen calibration values obtained are listed in Table I and displayed graphically in Figure 4. For purposes of clarity, values obtained in the range 0-90 micrograms of oxygen are also plotted on a larger scale in Figure 5.

As can be seen from Table I, most of the calibration points were obtained with a generating current of 6.10 ma. At this current level, oxygen is being generated at a rate of about 0.5 micrograms per second. In order to be certain that none of the oxygen was passing through the sodium anthraquinone-β-sulfonate solution without reacting, a number of tests were conducted using a 2.00 ma generating current. Since both calibrations resulted in the same curve, this was regarded as proof that all the oxygen was recovered. All of the values listed were obtained using a flow rate of about 108 cc/minute. Several values were also obtained using a flow rate of about 203 cc/minute at the 6.10 ma current level. At 25 micrograms of oxygen, the same absorbance value was obtained; however, at the 100 microgram level, the results obtained were about 6 micrograms low, while at 160 micrograms, the result was about 10 micrograms too low. In the course of an actual analysis, however, it might be possible to use higher flow rates since the oxygen recovered from the sample would be contained in a much larger volume, thus, undergoing considerable dilution with helium. Should it prove necessary to run at higher flow rates, the completeness of recovery could be checked with synthetic samples. Since flow rates below about 100 cc/minute were not considered practical, no determinations were made at lower flow rates.

The stepwise procedure employed in the calibration of the Brady apparatus was as follows:

The level of the 10% H_2SO_4 in the electrolytic cell was raised so that the two electrodes were just covered. By means of the Hoke #482 bellows valves, the helium flow was regulated so that a steady stream of helium bubbles flowed through the electrolyte while, at the same time, some helium flowed across the

TABLE I

CALIBRATION DATA - BRADY APPARATUS

Micrograms Oxygen	Absorbance	Generating Current (ma)
5.06	.984	6.10
10.1	.962	6.10
14.9	.940	2.00
20.2	.915	6.10
25.0	.892	2.00
35.4	.851	6.10
40.1	.830	2.00
50.6	.790	6.10
60.6	.742	6.10
70.4	.708	2.00
80.9	.650	6.10
90.0	.621	2.00
101.1	.574	6.10
121.2	.500	6.10
130.0	.465	2.00
141.6	.420	6.10
150.0	.388	2.00
161.9	.347	6.10
180.0	.283	2.00
182.0	.274	6.10
187.0	.258	6.10

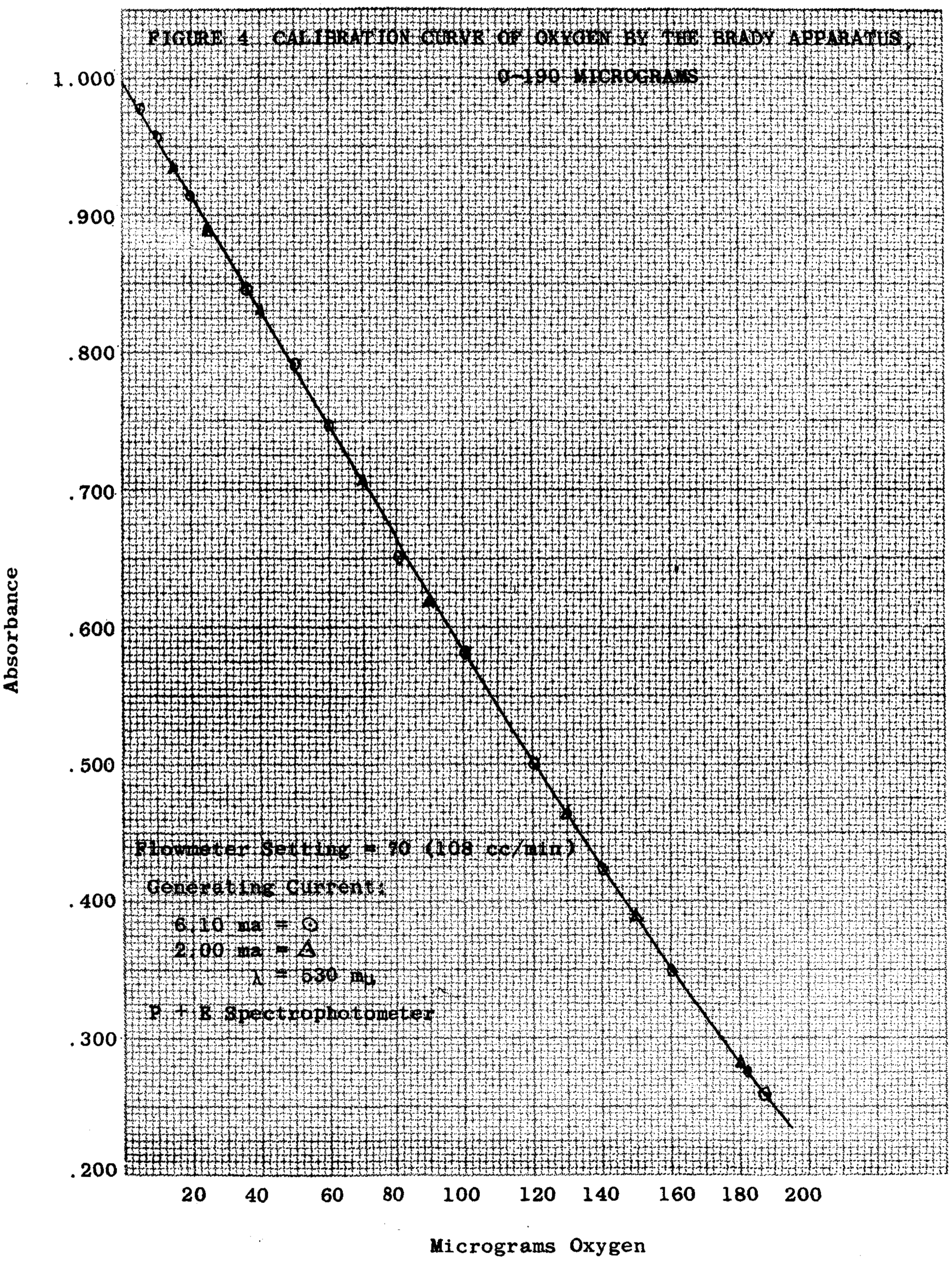


FIGURE 4. CALIBRATION CURVE OF OXYGEN BY THE BRADY APPARATUS, 0-190 MICROGRAMS

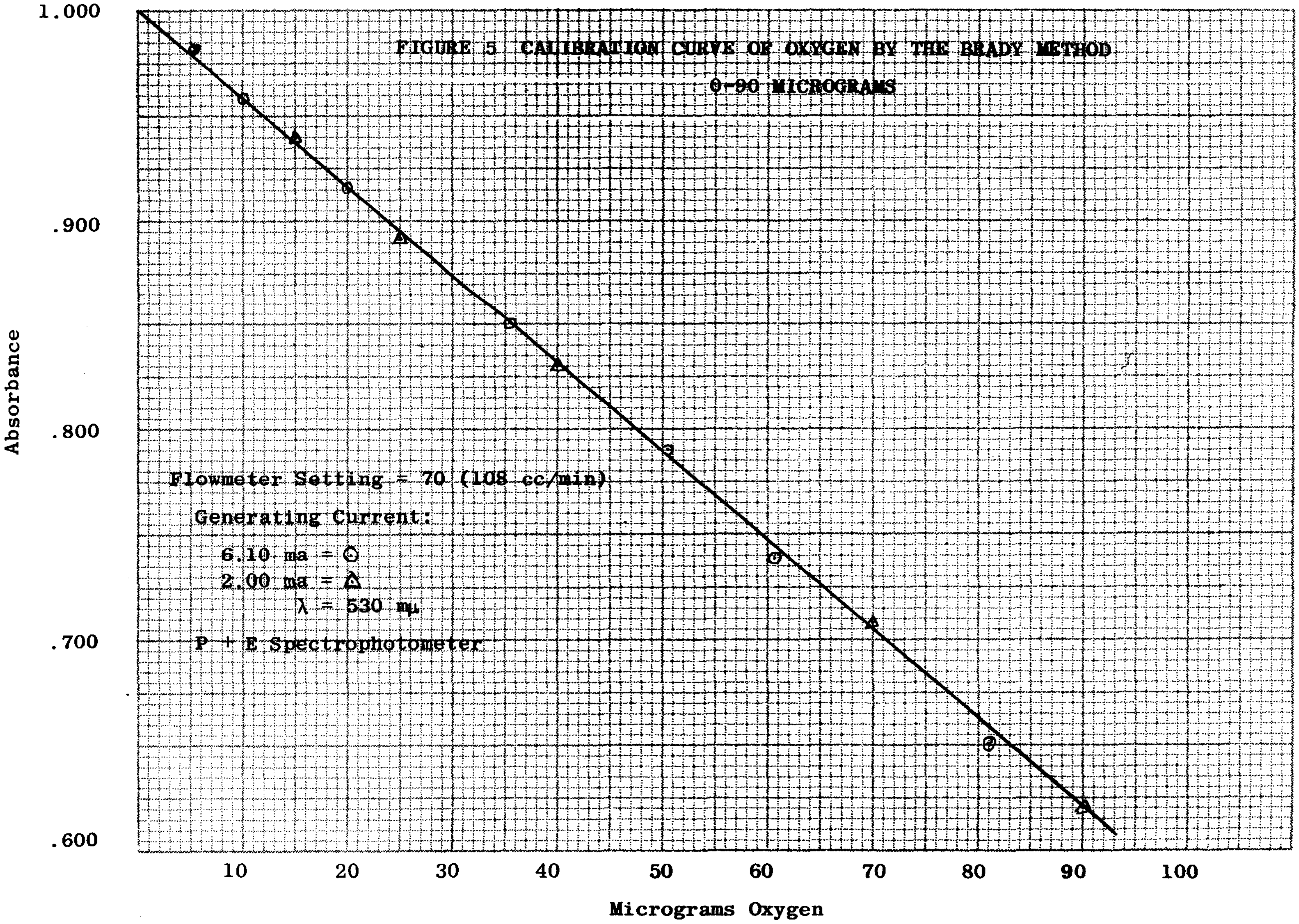


FIGURE 5 CALIBRATION CURVE OF OXYGEN BY THE BRADY METHOD

0-90 MICROGRAMS

top of the cell (see Figure 1). The three-way stopcock was set in a position so that all parts were interconnected. The total flow through the Brady apparatus may be finely adjusted by means of the needle valve in the Matheson No. 602 flowmeter inserted between the exit end of the Brady apparatus and the wet test meter. By suitable manipulation of the valves a steady flow can be obtained. The flow reading on the R-2-15-D flow tube was held at 70 mm (108 cc/minute).

While the flow was being regulated, the solution in the Brady apparatus was circulated through the zinc amalgam reducing column. The Perkin-Elmer Spectrophotometer and the Varicord Model 43 chart recorder were turned on and the absorbance monitored. With the slit set at about 0.12 mm, the absorbance reached a maximum absorbance value of about 1.000 or slightly less.

After the absorbance has reached its maximum value and the gas flow has attained a steady state, the Teflon stopcocks of the Brady apparatus are adjusted so as to by-pass the reducing column. By suitable manipulation of these stopcocks, the level of solution in the gas separator can be adjusted to the reference mark. It is important that this be done with the flow regulated exactly at 70 mm on the flowmeter so as to obtain the same quantity of reagent solution each time. Once the level has been set and the stopcocks closed, changes in the flow rate do not affect the quantity of solution in the analytical section, even though the level of the solution in the gas separator will change due to differences in hydrostatic pressure.

The chart recorder trace was observed for a period of about thirty minutes. During this time, if there are no leaks in the system, the absorbance should not change noticably. The decrease in absorbance during a 45 minute blank run seldom exceeded 0.001 absorbance unit. At this level, the blank is negligible since one microgram of oxygen produces an absorbance change of about 0.005 units.

After the blank level (if any) has been determined, the absorbance reading on the chart recorder was noted. Then the power supply to the electrolysis cell was actuated, noting the time interval with a stopwatch. After about 30 to 45 minutes (depending on the amount of oxygen generated) the chart recorder trace will have leveled off at some lower absorbance value. The difference in absorbance, corrected for any blank, is substracted from 1.000 and plotted against the micrograms of oxygen generated.

The Hitachi Perkin-Elmer Model 139 Spectrophotometer yields its best accuracy when the concentration of the sample is such that the absorbance is in the range of 0.3 to 0.7. For samples of high absorbance, a selector switch is provided with settings of x $\sqrt{10}$ and x10. When set on the x $\sqrt{10}$ scale, the true absorbance is obtained by adding 1.0. During all calibration runs made at oxygen levels of less than 70 micrograms, the selector switch was placed on x $\sqrt{10}$ thus giving an initial absorbance reading of about 0.5. When using the Varicord Model 43 chart recorder which has a linear absorbance scale from 0 to 1.000, identical values were obtained with the selector switch at x1 (normal full scale setting) and x $\sqrt{10}$.

Following a calibration run, the solution in the Brady apparatus may be regenerated by circulating through the reducing column prior to the next run. At the lower oxygen levels, several runs may be made before the solution is regenerated. For best accuracy, however, the absorbance should not be allowed to drop below about 0.3 during the course of a run.

Because of the excellent stability of the Hitachi Perkin-Elmer spectrophotometer and Varicord chart recorder combination, the absorbance in the range of 0-1.000 can be estimated to within 0.002 absorbance units with considerable confidence. This accuracy, coupled with the fact that the sample cuvette is fixed and that only differences in absorbance, rather than absolute absorbance values need be measured, indicate that the relative error should be very small by spectrophotometric standards--probably less than one percent.

II Calibration of Weight of Potassium Sample by Extruder Turns

In order to correlate the number of extruder turns to the weight of potassium extruded, the following procedure was used:

Samples of potassium (from NASA, Cleveland) were obtained in nominal 3/8" OD x 6-3/4" stainless steel tubes having one flared end. After removal of the end caps, a tube was placed in the extruder in the normal fashion*. With the extruder under high vacuum (4×10^{-6} torr), a small number of turns was first made to expose the oxidized end. This exposed length then was cut off with one of the two cutting wires and discarded into the waste pan. Several potassium samples were then extruded, cut off, and held by the other cutting wire. The sizes of the potassium samples corresponded to randomly chosen extruder turns in the range of 5 to 20 turns. The ball valve, connecting the extruder and the reaction cell (which had been evacuated previously by means of the rough vacuum system) was opened and the cutting wire heated (using the 1.5 V battery), causing the sample to drop into a 150 ml glass beaker contained within the reaction cell. The ball valve was then closed and the reaction cell brought to atmospheric pressure.

After removal from the cell, the potassium was immediately transferred to a tared 100 ml platinum dish and covered with hexane. The potassium was then carefully reacted by dropwise additions of ethanol to convert it to the alkoxide. Any potassium adhering to the glass beaker was treated similarly and then transferred quantitatively to the platinum dish.

After completion of the reaction with ethanol, the sample was treated with an excess of concentrated hydrochloric acid to convert all of the potassium to the chloride. The organic solvents then were removed by evaporation, using infrared lamps. The platinum dishes containing the chloride were placed in a drying oven at 120°C for at least one hour then the dried samples were cooled to room temperature in a desiccator and weighed as potassium chloride. Subsequently, the equivalent amount of potassium was calculated. The results are shown in Table II and Figure 6. Including the point at 17 turns, one can expect a weight determination with less than 2% error. Excluding this point, the error is less than 1%.

---

*The NASA extruder as modified here will be described more completely in the next quarterly report. Several additional minor changes are being made prior to completion of drawings.

TABLE II

DETERMINATION OF SAMPLE WEIGHT OF POTASSIUM BY EXTRUSION*

Extruder Turns	Wt. of Potassium (g)
5	0.2543
7	0.3692
10	0.5124
12	0.6231
15	0.7800
17	0.9007
18	0.9365
19	0.9814
20	1.0376

*Obtained by converting K to KCl and weighing.

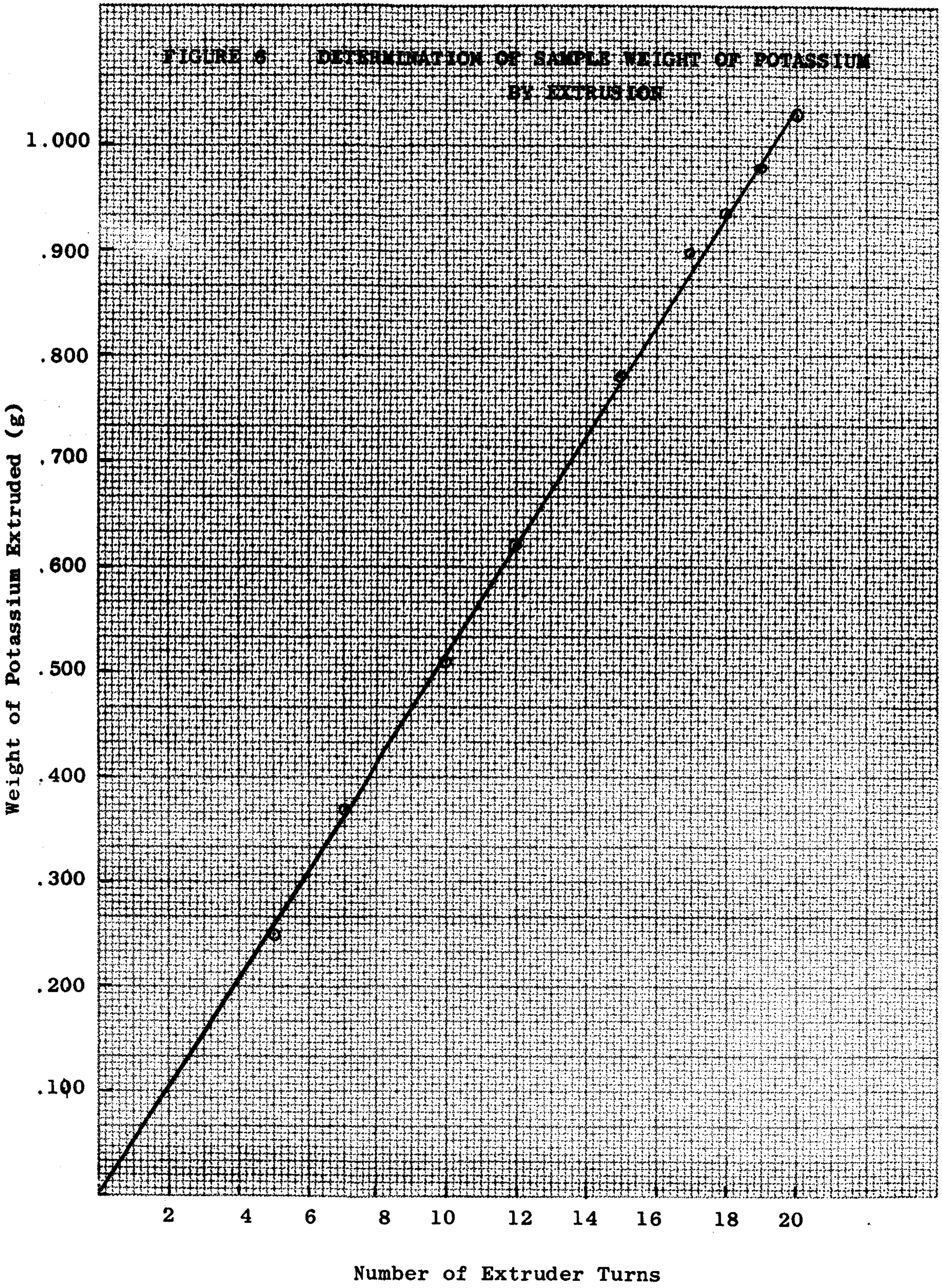


FIGURE 6 DETERMINATION OF SAMPLE WEIGHT OF POTASSIUM BY EXTRUSION

III Apparatus Construction

The complete assembly, exclusive of the helium purification train and the Brady oxygen measuring section, is shown in Figure 7. The helium inlet can be noted at the top left of the photograph. The helium plus oxygen outlet to the Brady apparatus is shown at the top right. All connections that see BrF_3, other than threaded joints and welds, are made by monel Cajon connectors with Teflon gaskets. The joints in copper lines where only helium passes are all silver or soft soldered.

Figure 8 shows the bromine trifluoride purification and storage cell whose construction is shown in Drawing #4012286-181. The modified flange (G) is from Drawing #4012093-784. All tubings are joined through Cajon fittings to all monel Durco Sleeveline vacuum plug valves having Teflon gasketing. These 1/4" screwed-end valves are used through the apparatus. Figure 9 shows an expanded view of this valve. All threaded connections to these are made with Teflon tape.

In Figure 8 an inverted Matheson #4 cylinder of bromine trifluoride is connected to the storage cell first through a Matheson #55A-670 monel needle valve (A) with a 1/4" SAE flare fitting and then through a Durco valve (B). A manifold connects valve (C) to the rough trap (J). An exit valve on (J) is connected through a jacketed liquid nitrogen glass trap to a rough vacuum pump by rubber covered Teflon tubing. The rough trap design is detailed in Drawing #4012286-171. Lines (H) and (I) to the rough trap manifold connect to the Kel-F trap and the adapter which are described later. Valve (C) allows the removal of volatile impurities from the bromine trifluoride by vacuum pumping. Valve (D) serves the dual purpose of allowing purified helium to be bubbled through the BrF_3 and out through valve (C) for purification purposes, and also for transferring, by pressure, liquid BrF_3 through valve (E) to the Kel-F trap.

A TEG-12 Conax Connector (F) with a Teflon insert and 1/8" nickel rod is mounted on the center of flange (G). The height of the liquid BrF_3 within the cell can be ascertained by raising or lowering the nickel rod until electrical contact is made. Conductivity will be ascertained with a simple Simpson meter. In practice, the nickel rod will be preset to indicate a low volume of BrF_3, which will signal the necessity for refilling.

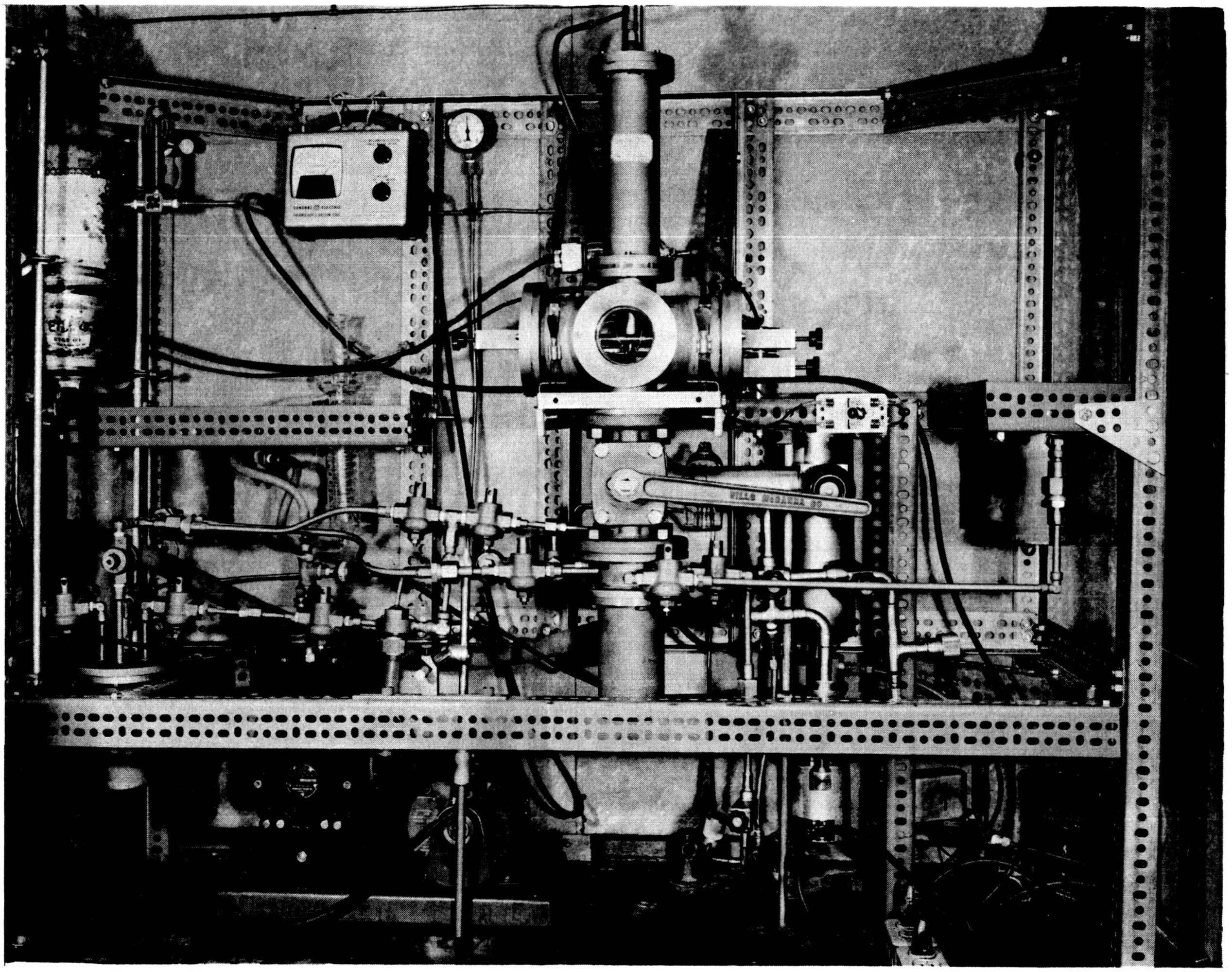


FIGURE 7 System for the Analysis of Oxygen in Potassium (C 64062554)

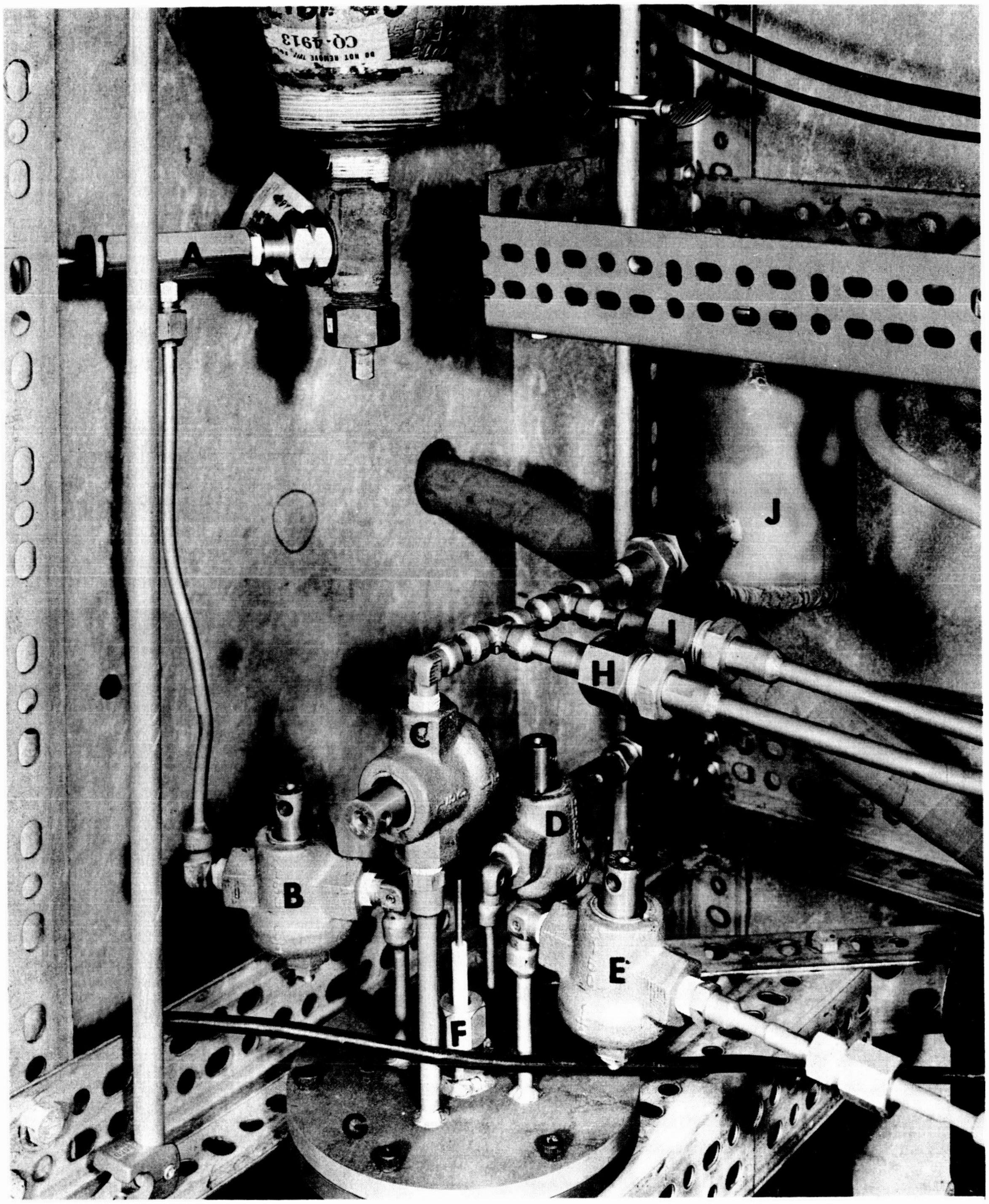


FIGURE 8 Bromine Trifluoride Purification and Storage Cell (C 64062553)

REVISIONS				
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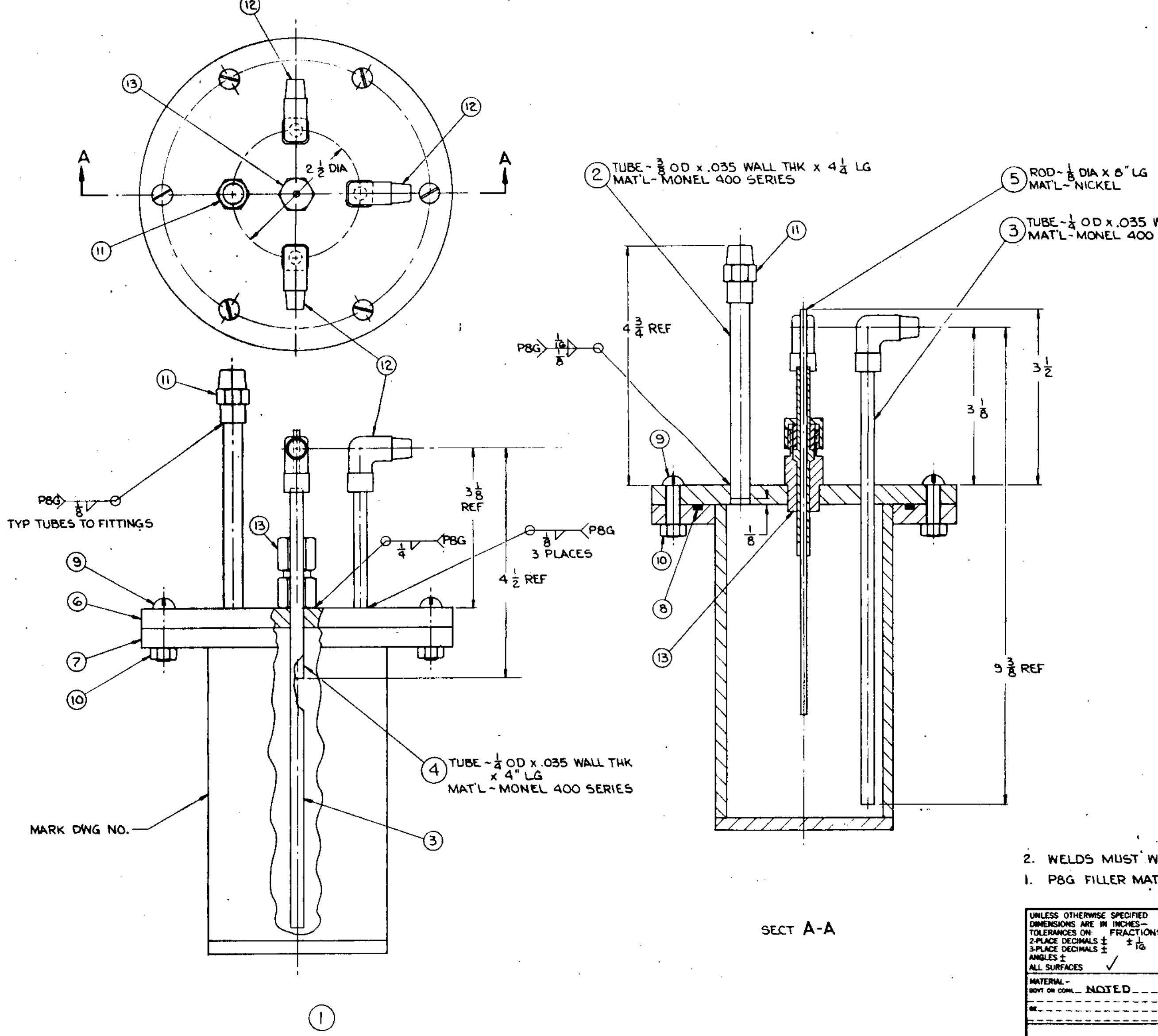


SECT A-A

ITEM	IDENTIFICATION NO.	DESCRIPTION OR NAME	ZONE	GROUP NO. & QTY 1	2	3	4
1		ASSY	A9	X			
2	4012286-181 P2	TUBE	F7	1			
3	P3		F4	2			
4	P4		B8	1			
5	P5	ROD	F4	1			
6	4012093-784P1	FLANGE	D10	1			
7	4012093-787G1	TRAP	C10	1			
8	MS29513-242	O-RING	D6	1			
9	COM'L STN STL	RD HD CAP SCR 1/4 UNC x 1" LG	D10	6			
10	COM'L STN STL	HEX NUT 1/4 UNC	C10	6			
(A) 11	MGSW-1-6	MALE CONNECTOR	F6	1			
(A) 12	M4SW-2-4	MALE ELBOW	E8	3			
(B) 13	TEG-12	ELECTRODE GLAND (NO ELECTRODE)	C6	1			

(A) CAJON COMPANY CLEVELAND, OHIO
(B) CONAX CORPORATION BUFFALO, NEW YORK

2. WELDS MUST WITHSTAND 10^{-5} - 10^{-6} TORR WITH NO LEAKS
1. P8G FILLER MAT'L - INCO 60

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES—
TOLERANCES ON: FRACTIONS ± 1/16
2-PLACE DECIMALS ±
3-PLACE DECIMALS ±
ANGLES ±
ALL SURFACES ✓
MATERIAL -
GOVT OR COML NOTED

SIGNATURES	DATE
Morris D. Penn	6/20/64
Morris D. Penn	6/30/64
F. R. Uester	June 30, 1964
[illegible]	6/30/64
[illegible]	6/23/64
[illegible]	6/20/64

GENERAL ELECTRIC
AETD DEPT LOC EVENDALE PLANT

TITLE
STORAGE CELL
FOR BROMINE TRIFLUORIDE

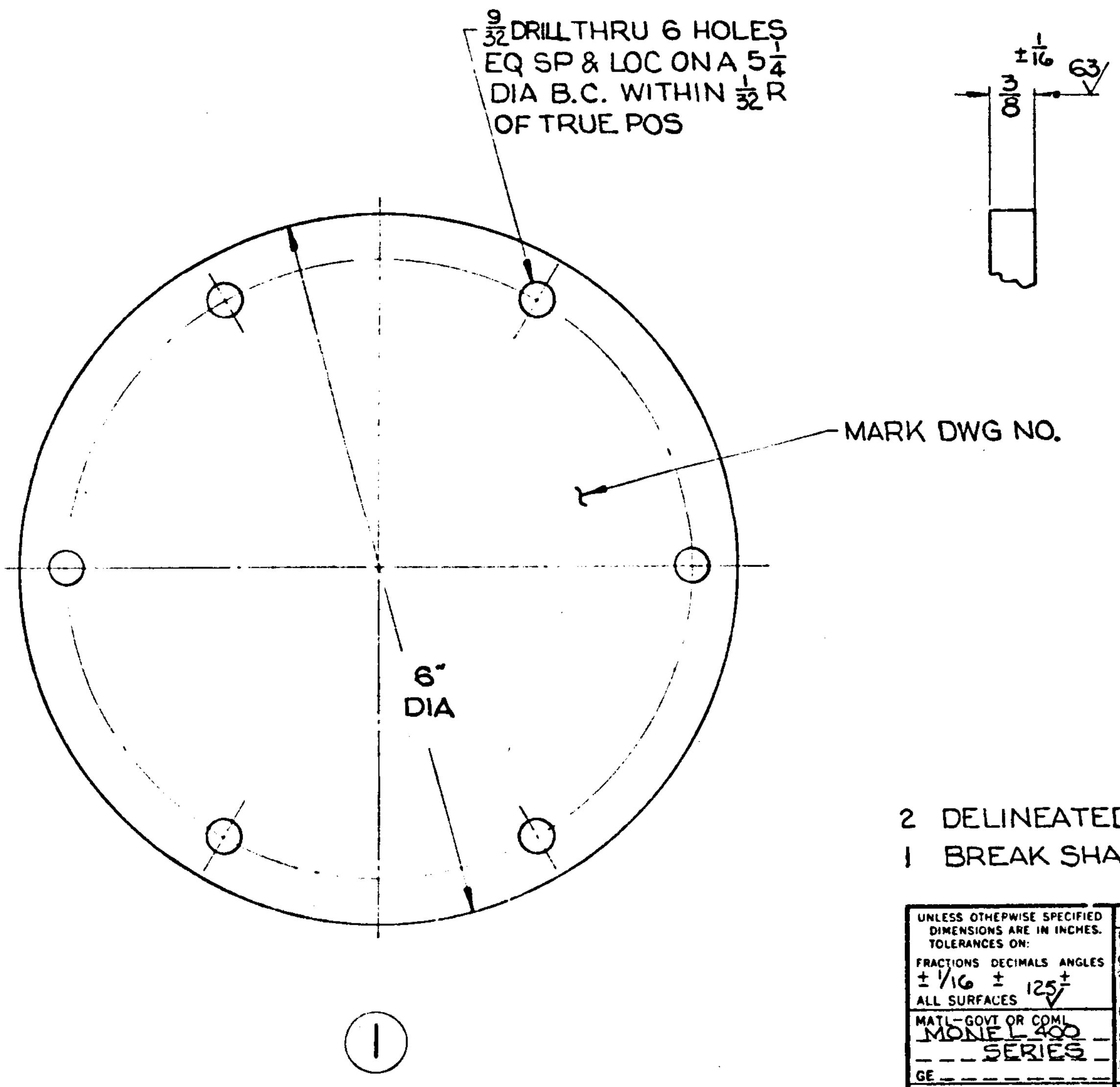
CONTRACT NO.
CODE IDENT NO. SIZE D DWG NO. 4012286-181
SCALE 1/1 WT CALC ACTUAL SHEET 1 OF 1
57

PL-189

SIZE		SHEET	REV
B	4012093-784		

REVISIONS

SYM	DESCRIPTION	DATE	APPROVED



2 DELINEATED RIGHT ANGLES 90°± 30' UNLESS OTHERWISE SPECIFIED

1 BREAK SHARP CORNERS .015 MAX RAD OR CHAM

UNLESS OTHEPWISE SPECIFIED DIMENSIONS ARE IN INCHES. TOLERANCES ON: FRACTIONS ± 1/16 DECIMALS ± ANGLES ± ALL SURFACES 125	SIGNATURES	DATE	GENERAL ELECTRIC AET DEPT LOC EVENDALE
MATL—GOVT OR COML MONEL 400 SERIES	DRAWN C. H. Kennedy	2/19/64	TITLE FLANGE
GE	CHECKED C. H. Kennedy	2/20/64	CONTRACT NO.
	ISSUED F. R. Vester	2.21.64	CODE IDENT NO. 06083
	ENGRG	2.20.64	SIZE B
	MFG R. A. Eckels	2/20/64	DWG NO. 4012093-784
	MATLS H. Kirtchuer	2.20.64	SCALE 1/1
			WT CALC ACTUAL
			SHEET

AR-394

57

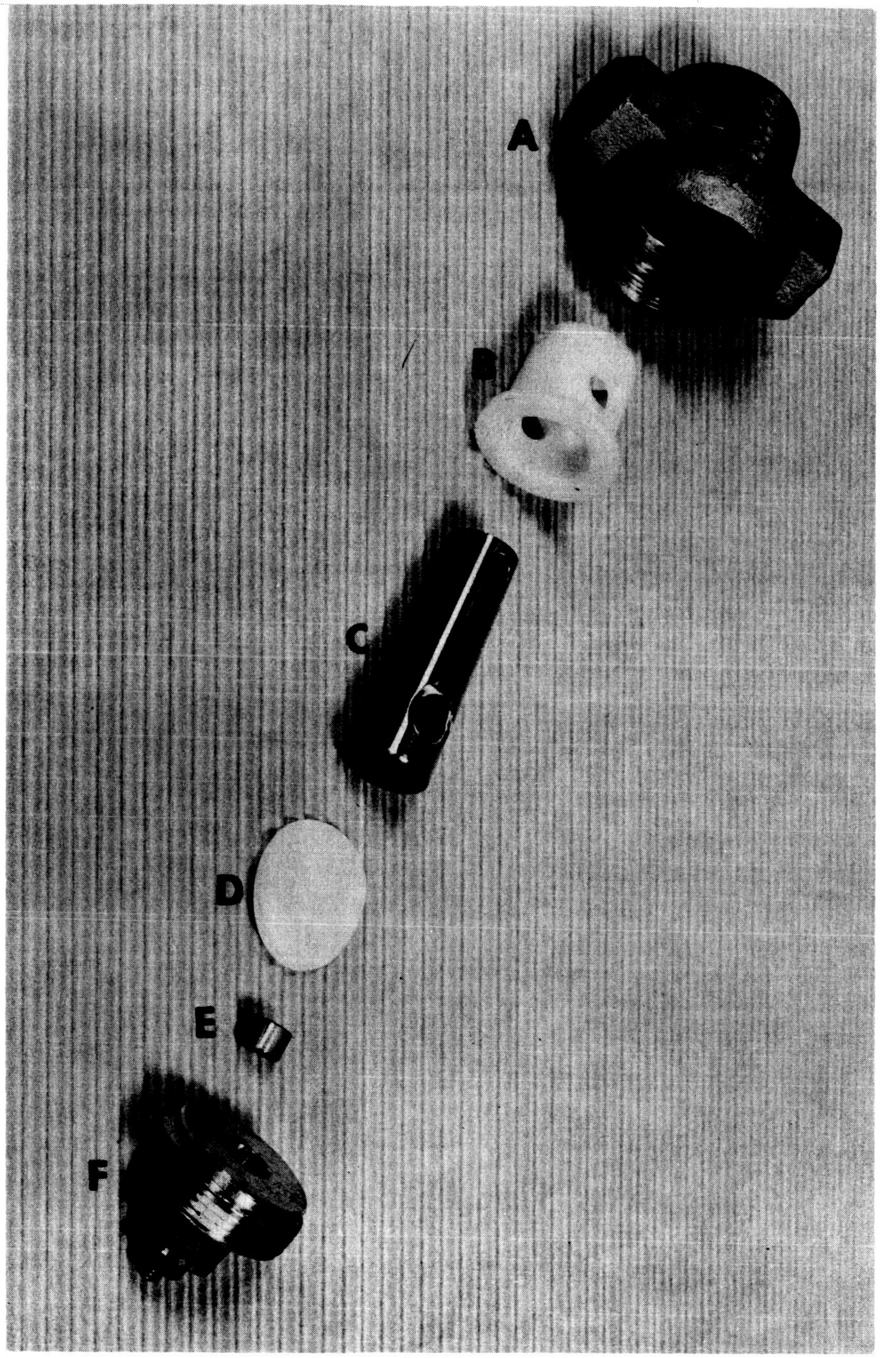
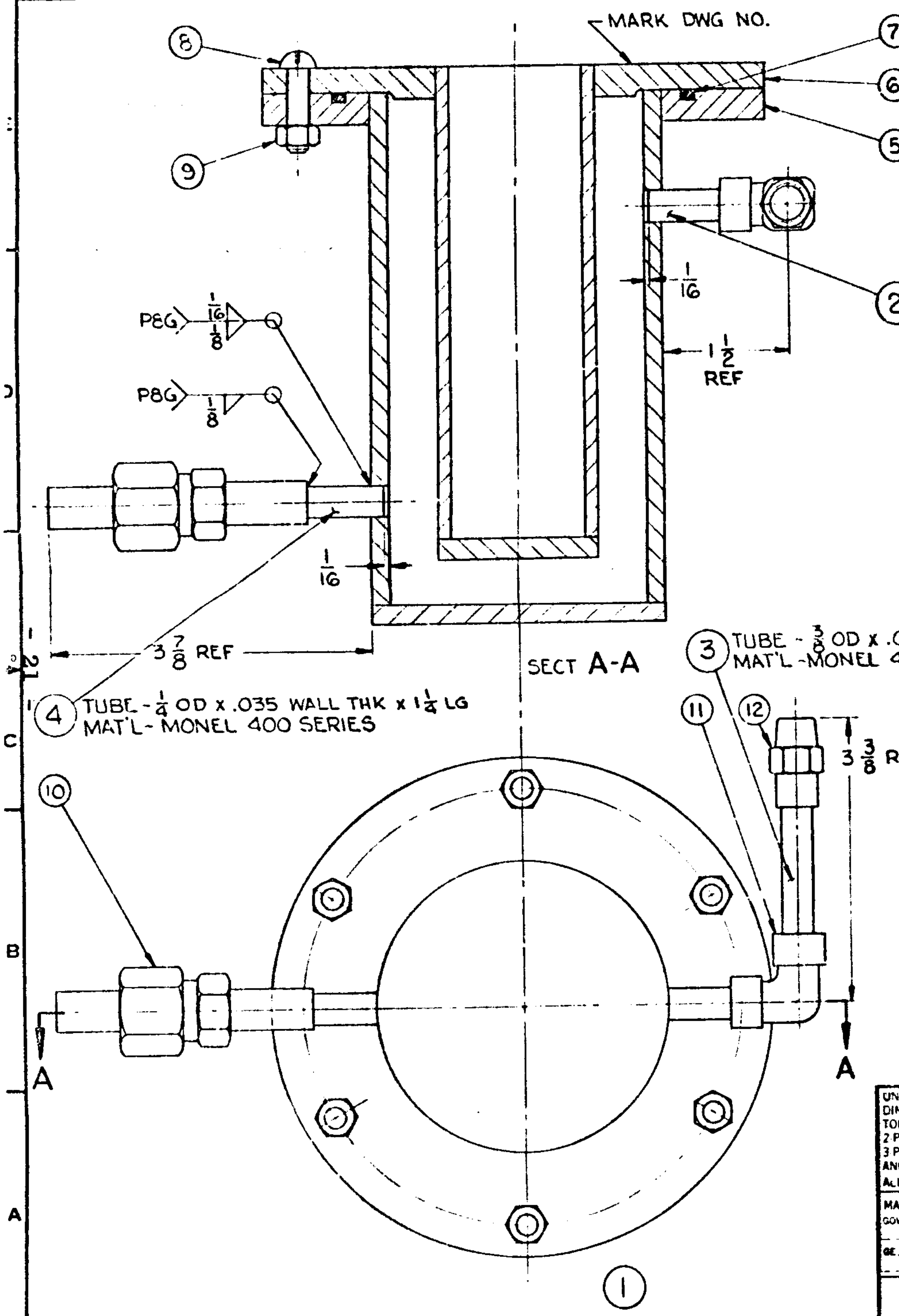


FIGURE 9 Expanded View of All Monel-Teflon Durco Vacuum Plug Valve (C 64070101)

C 4012286-171

REVISIONS				
SYM	ZONE	DESCRIPTION	DATE	APPROVED



ITEM	IDENTIFICATION NO.	DESCRIPTION OR NAME	ZONE	GROUP NO. & QTY 1	2	3	4
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3	P3	TUBE	C5	1			
4	P4	TUBE	C7	1			
5	4012093-787G1	TRAP	E4	1			
6	4012093-786G1	INNER TRAP MEMBER	E4	1			
7	MS29513-242	O-RING	E4	1			
8	COM'L STN STL	RD HD CAP SCR 1/4 UNC x 1" LG	E7	6			
9	COM'L STN STL	HEX NUT 1/4 UNC	E7	6			
10	M6VC	VACUUM COUPLING (A)	C7	1			
11	M4SW-9	ELBOW (A)	C5	1			
12	M6SW-1-6	MALE CONNECTOR (A)	C5	1			

(A) CAJON COMPANY CLEVELAND, OHIO

2. WELDS MUST WITHSTAND 10^{-5} - 10^{-6} TORR WITH NO LEAKS

1. P8G FILLER MAT'L - INCO 60

UNLESS OTHERWISE SPECIFIED
DIMENSIONS ARE IN INCHES
TOLERANCES ON FRACTIONS
2 PLACE DECIMALS ± ± 1/16
3 PLACE DECIMALS ±
ANGLES ±
ALL SURFACES ✓

MATERIAL - GOVT OR COML NOTED

SIGNATURES	DATE
Morris G Penn	6/20/64
Morris G Penn	6/30/64
F R Vester	June 30 1964
W. Fitch	6/30/64
KA Ecobetts	6/25/64
W. Fitch	6/30/64

GENERAL ELECTRIC
AETD DEPT LOC EVENDALE PLANT

TITLE ROUGH TRAP

CONTRACT NO
CODE IDENT NO | SIZE C | DWG NO 4012286-171
SCALE 1/1 | WT | SHEET 1 OF 1

57

PL-189

C 4012286-171

The Kel-F trap shown in Figure 10 is used for measuring known volumes of liquid BrF_3 as received from the storage cell (Figure 8) and for transferring it to the reaction cell. Drawings #4012286-191, -192, and -193 detail the construction. The Kel-F trap (B) may be evacuated through valve (C) connected to the rough trap manifold noted previously. BrF_3 is admitted through valve (A) and may be forced into the reaction cell through valve (E) by admitting purified helium through valve (D).

Figure 10 shows valve (F), used for the rough evacuation of the reaction cell. Connection (G) joins the exit end of the liquid nitrogen glass trap (after the rough trap) to the vacuum pump. A thermocouple vacuum gauge tube at this connection (G) serves the purpose of determining whether all volatile matter has been removed from the lines.

Figure 11 shows the adapter (C) assembled to the ball valve (G) and reaction cell (D). Also shown is the lower portion of the extruder (A) and the Veeco vacuum system which connects to it. The vacuum system also connects to the adapter through valve (F). The double-pole double-throw switching arrangement (H) is for heating either of the two wire cutters within the extruder. The exit gases, helium, oxygen, and some bromine trifluoride pass through valve (E) toward the trapping system and the Brady apparatus. Helium may be bubbled through or over the BrF_3 in the cell. The adapter assembly construction is detailed in Drawing #4012286-182.

Figure 12 shows the double trap section. The construction is shown in Drawing #4012286-180. The two traps (A) are connected together by a Durco valve (B). The exit end of the trap is connected through valve (C) and monel tubing to copper tubing with silver solder. This copper tubing goes directly to the Brady apparatus. The helium inlet joins the adapter through a Cajon connector. In traps (A), pentane slush will be used to condense any bromine trifluoride vapors from the helium and oxygen before this stream reaches the Brady apparatus.

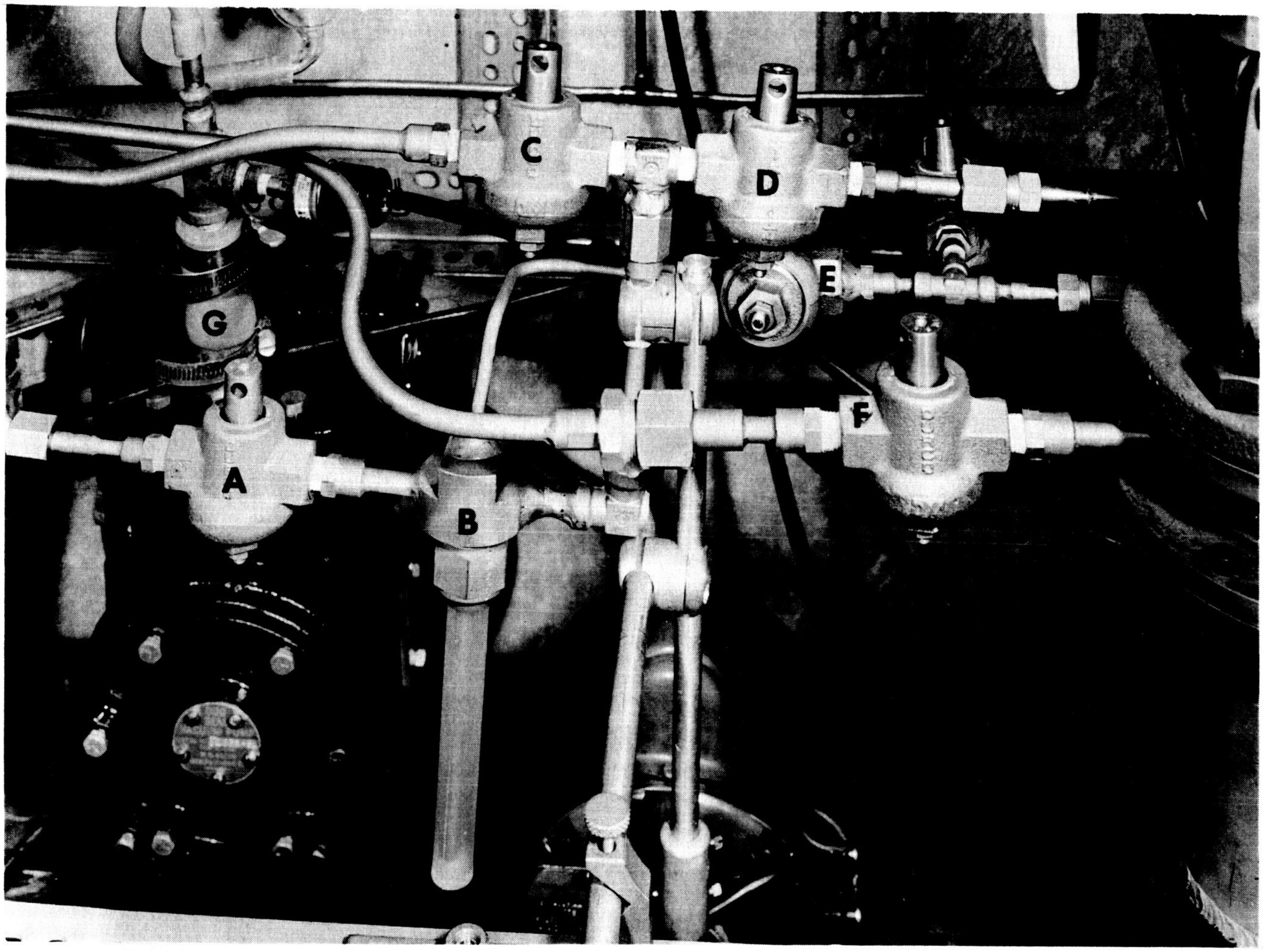
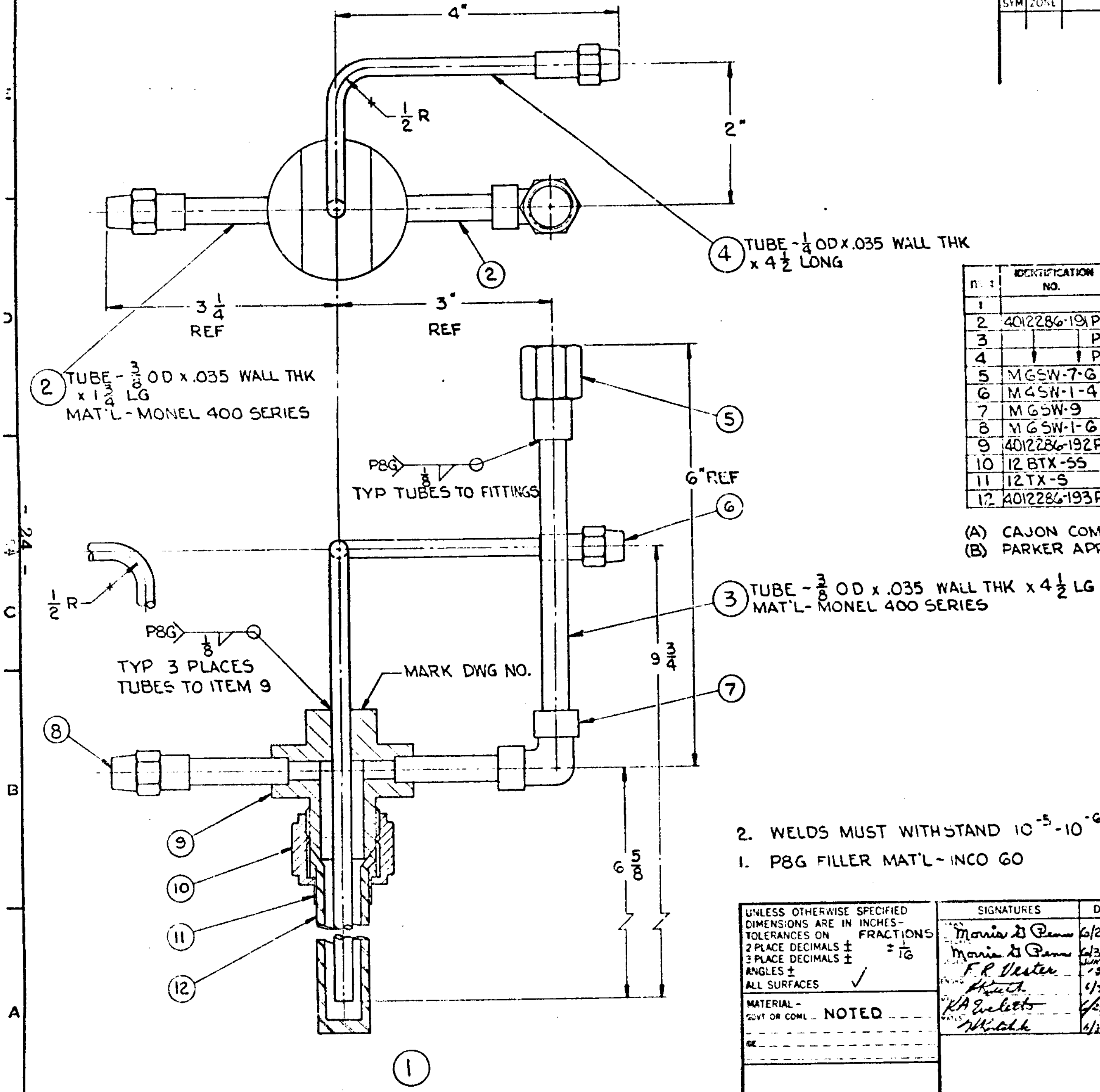


FIGURE 10 Kel-F Trap for Transfer of Purified Bromine Trifluoride (C 64062556)

SIZE		REV
C	4012286-191	

REVISIONS

SYM	ZONE	DESCRIPTION	DATE	APPROVED



ITEM	IDENTIFICATION NO.	DESCRIPTION OR NAME		ZONE	GROUP NO. & QTY 1	2	3	4
1		ASSY		A6	X			
2	4012286-191P2	TUBE		D7	2			
3	P3	TUBE		C4	1			
4	P4	TUBE		D4	1			
5	MGSW-7-6	FEMALE CONNECTOR	(A)	D4	1			
6	M4SW-1-4	MALE CONNECTOR	(A)	C4	1			
7	MGSW-9	ELBOW	(A)	B4	1			
8	MGSW-1-6	MALE CONNECTOR	(A)	B7	1			
9	4012286-192P1	FITTING FOR KEL-F TRAP		B7	1			
10	12 BTX-SS	NUT	(B)	B7	1			
11	12 TX-S	SLEEVE	(B)	A7	1			
12	4012286-193P1	KEL-F TUBE		A7	1			

(A) CAJON COMPANY - CLEVELAND, OHIO
(B) PARKER APPLIANCE COMPANY - CLEVELAND, OHIO

2. WELDS MUST WITHSTAND 10^{-5}-10^{-6} TORR WITH NO LEAKS

1. P8G FILLER MAT'L - INCO 60

UNLESS OTHERWISE SPECIFIED
DIMENSIONS ARE IN INCHES-
TOLERANCES ON FRACTIONS ± 1/16
2 PLACE DECIMALS ±
3 PLACE DECIMALS ±
ANGLES ±
ALL SURFACES ✓

MATERIAL -
GOVT OR COML NOTED

SIGNATURES	DATE
Morris G. Penn	6/20/64
Morris G. Penn	6/30/64
F.R. Vester	June 30 1964
	6/30/64
K.A. Evelets	6/29/64
	6/30/64

GENERAL ELECTRIC
AETD DEPT LOC EVENDALE PLANT

TITLE
KEL-F TRAP
FOR BROMINE TRIFLUORIDE

CONTRACT NO
CODE IDENT NO
SIZE C
DWG NO 4012286-191
SCALE 1/1
SHEET 1 OF 1

57

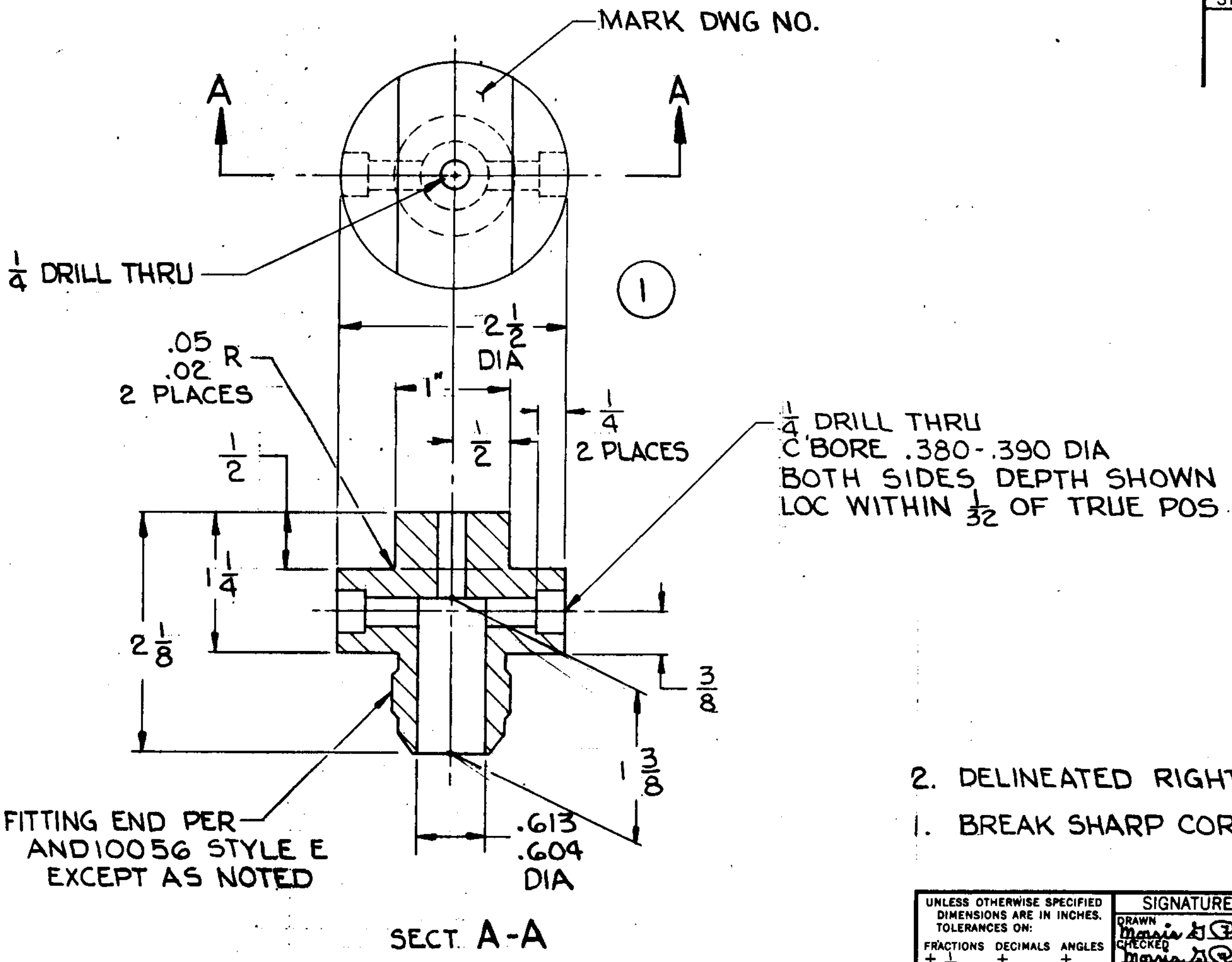
PL-189

C 4012286-191

SIZE		SHEET	REV
B	4012286-192	1	

REVISIONS

SYM	DESCRIPTION	DATE	APPROVED



2. DELINEATED RIGHT ANGLES ARE 90° ±15'

1. BREAK SHARP CORNERS .015 MAX RAD OR CHAM

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES.
TOLERANCES ON:
FRACTIONS ± 1/32 DECIMALS ± ANGLES ±
ALL SURFACES 63 √

MATL—GOVT OR COML
MONEL
SERIES 900
GE

SIGNATURES		DATE
DRAWN	Morris G Penn	6-27-64
CHECKED	Morris G Penn	6-30-64
ISSUED	F. R. Vester	JUNE 30 1964
ENGRG		6/30/64
MFG	L A Evelette	6/29/64
MATLS		6/30/64

GENERAL ELECTRIC
AETD DEPT LOC EVENDALE

TITLE
FITTING/ FOR KEL-F TRAP

CONTRACT NO.

CODE IDENT NO.	SIZE	DWG NO.
	B	4012286-192

SCALE 1/1 | WT CALC ACTUAL | SHEET 1

PRINTS TO 57

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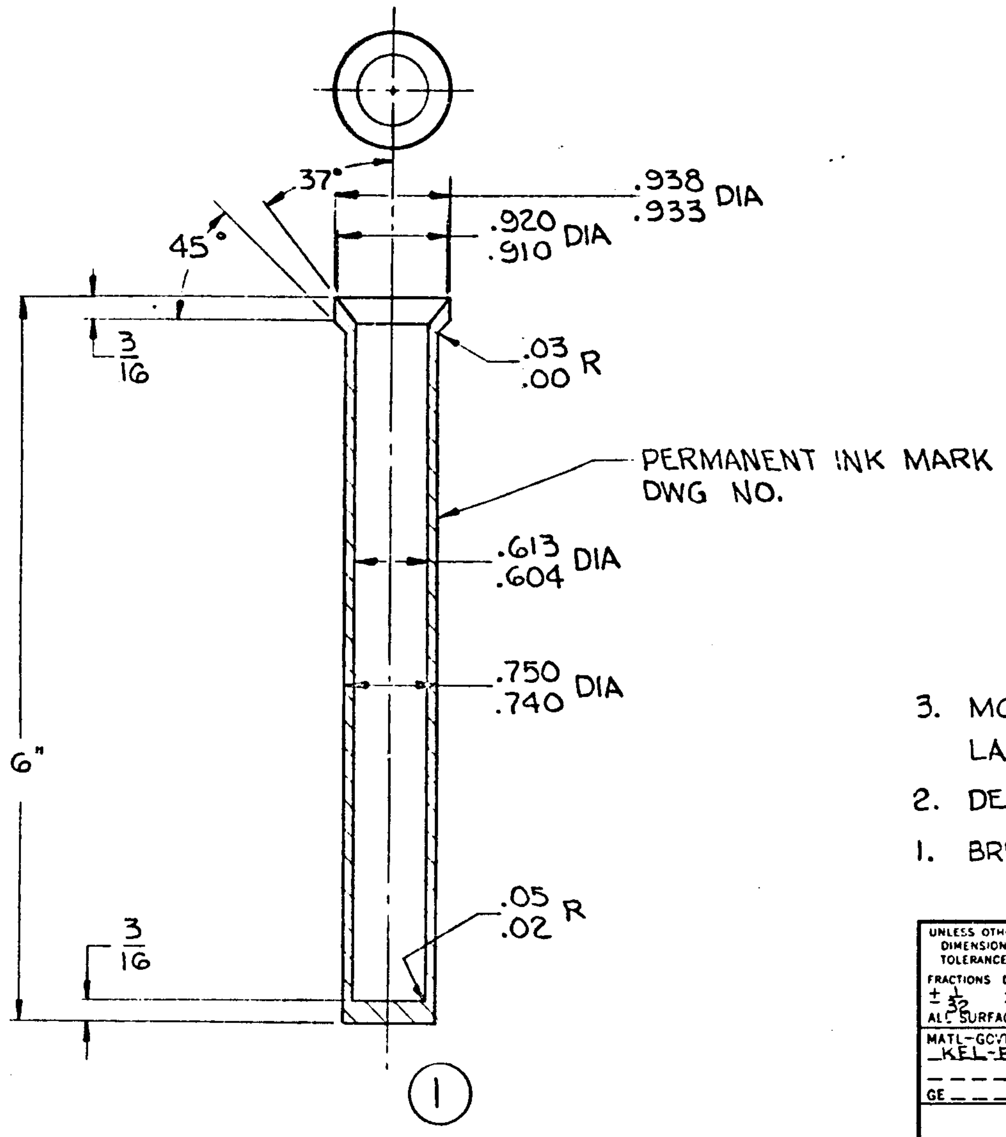
SIZE		SHEET	REV
B	4012286-193	1	

REVISIONS

SYM	DESCRIPTION	DATE	APPROVED



3. MOLDED PART AVAILABLE FROM ARGONNE NATIONAL LABS. CHICAGO, ILL.
2. DELINEATED RIGHT ANGLES ARE 90° ± 15'
1. BREAK SHARP CORNERS .015 MAX RAD OR CHAM

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES. TOLERANCES ON:
FRACTIONS ± 1/32 DECIMALS ± ANGLES ± 1°
ALL SURFACES 125 √
MATL–GOVT OR COML KEL-F
GE

SIGNATURES		DATE
DRAWN	Morris G. Penn	6-27-64
CHECKED	Morris G. Penn	6-30-64
ISSUED	F. R. Vester	JUNE 30 1964.
ENGRG	[illegible]	6/30/64
MFG	K A Everett	6/29/64
MATLS	[illegible]	6/30/64

GENERAL ELECTRIC
AETD DEPT LOC EVENDALE
TITLE KEL-F TRAP TUBE
CONTRACT NO.

CODE IDENT NO.	SIZE	DWG NO.
	B	4012286-193

SCALE 1/1	WT CALC ACTUAL	SHEET 1

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PRINTS TO	57

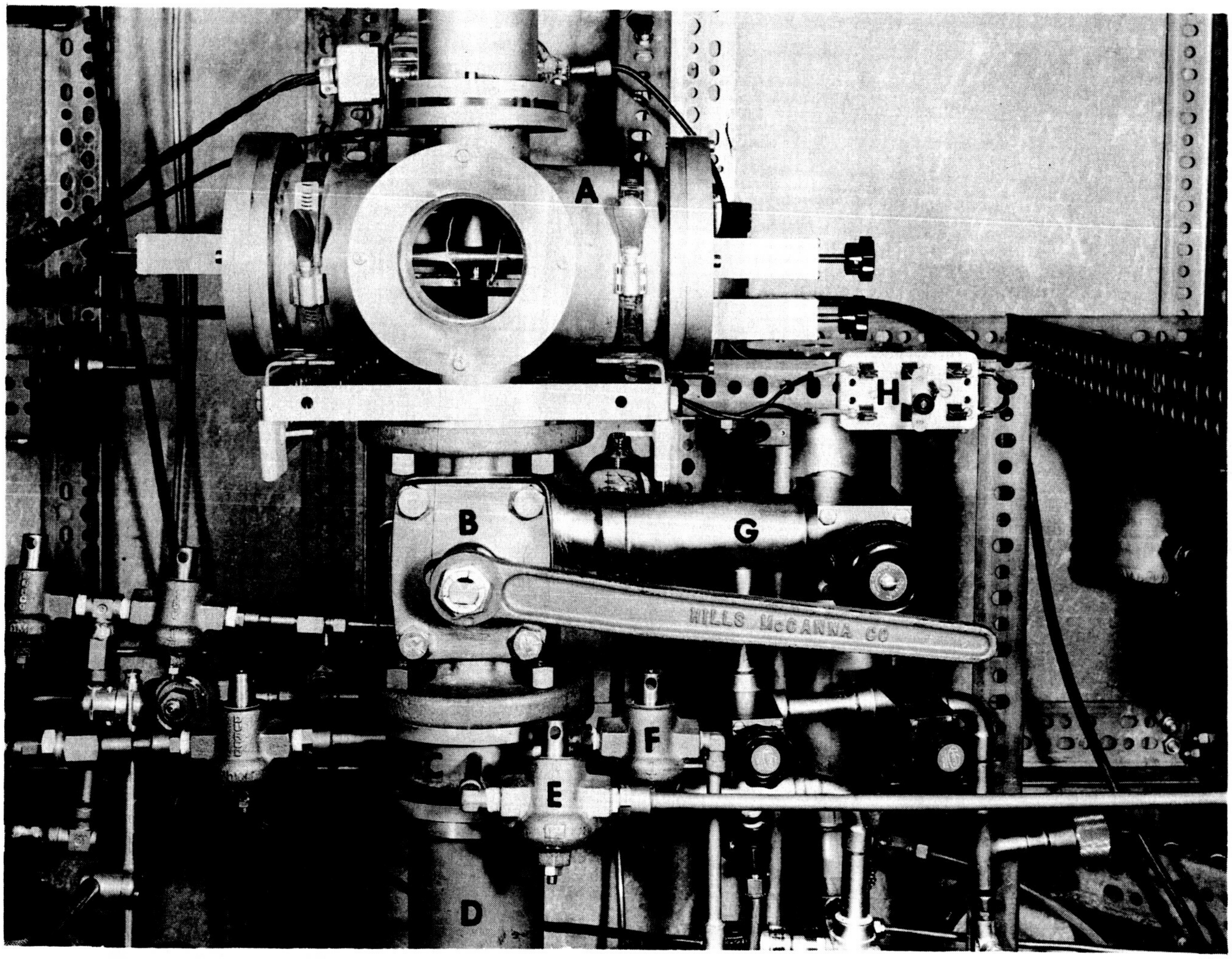
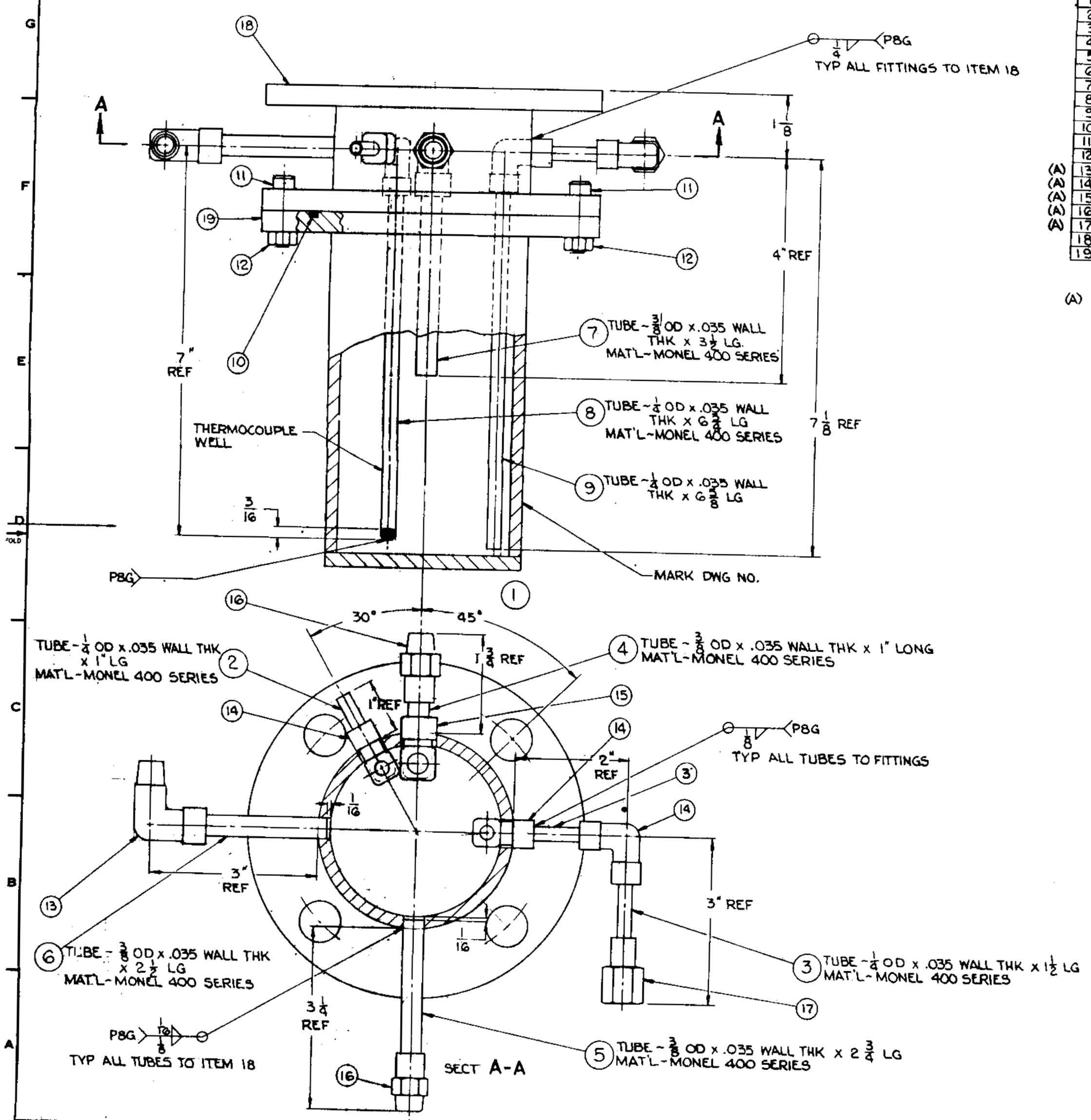


FIGURE 11 Extruder, Ball Valve, Adapter and Reaction Cell Assembly (C 64062552)

D 4012286-182



	ITEM	IDENTIFICATION NO.	DESCRIPTION OR NAME	ZONE	GROUP NO. & QTY 1	2	3	4
	1		ASSY	D9	*			
	2	4012286-182P2	TUBE	C10	1			
	3	P3		B7	2			
	4	P4		C8	1			
	5	P5		A8	1			
	6	P6		B11	1			
	7	P7		E8	1			
	8	P8		D8	1			
	9	P9		D8	1			
	10	MS 29513-242	O-RING	E10	1			
	11	COM'L STN STL	SOC HD CAP SCR 1/4 UNC x 1" LG	F10	6			
	12	COM'L STN STL	HEX NUT 1/4 UNC	F10	6			
(A)	13	M6SW-2-6	MALE ELBOW	B11	1			
(A)	14	M4SW-9	ELBOW	C8	3			
(A)	15	M6SW-9	ELBOW	C8	1			
(A)	16	M6SW-1-6	MALE CONNECTOR	A10	1			
(A)	17	M4SW-7-4	FEMALE CONNECTOR	A7	1			
	18	4012093-785G1	BALL VALVE ADAPTER	G10	1			
	19	4012093-787G1	REACTION CELL OR TRAP	F10	1			

(A) CAJON COMPANY - CLEVELAND, OHIO

REVISIONS

SYM	ZONE	DESCRIPTION	DATE	APPROVED

2. WELDS MUST WITHSTAND 10^{-5}-10^{-6} TORR WITH NO LEAKS
1. P8G FILLER MAT'L - INCO 60

UNLESS OTHERWISE SPECIFIED
DIMENSIONS ARE IN INCHES—
TOLERANCES ON: FRACTIONS ± 1/16
2-PLACE DECIMALS ±
3-PLACE DECIMALS ±
ANGLES ± 1°
ALL SURFACES ✓
MATERIAL-
GOVT OR COML NOTED

SIGNATURES	DATE
Dennis R. [illegible]	6/28/64
Dennis R. [illegible]	6/30/64
F. R. Uester	[illegible]
[illegible]	6/30/64
K. [illegible]	7/2/64
[illegible]	6/30/64

GENERAL ELECTRIC
AETD DEPT LOC EVENDALE PLANT
TITLE
ADAPTER ASS'Y
CONTRACT NO.
CODE IDENT NO. SIZE D DWG NO. 4012286-182
SCALE 1/1 WT CALC ACTUAL SHEET 1 OF 1
PL-189

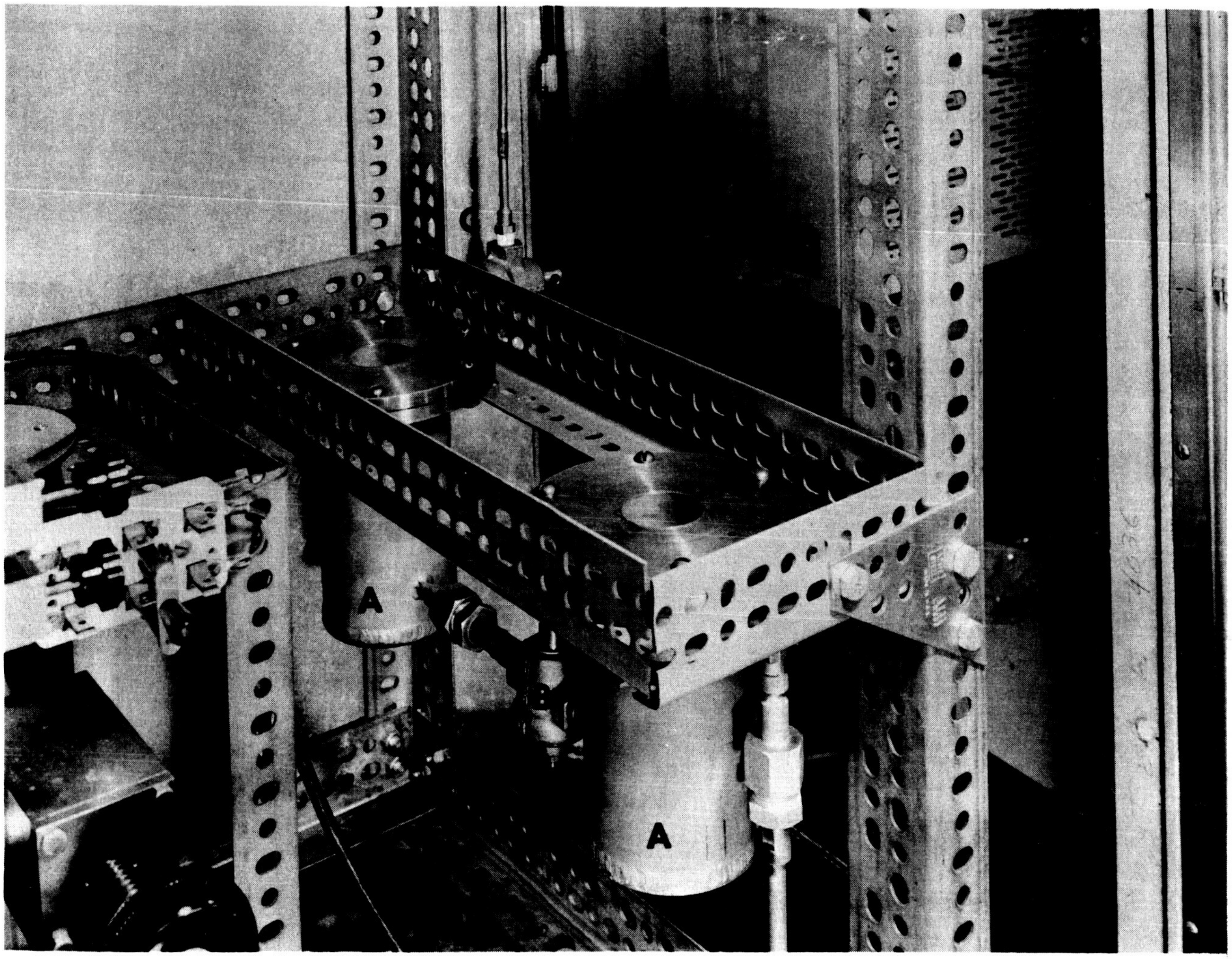
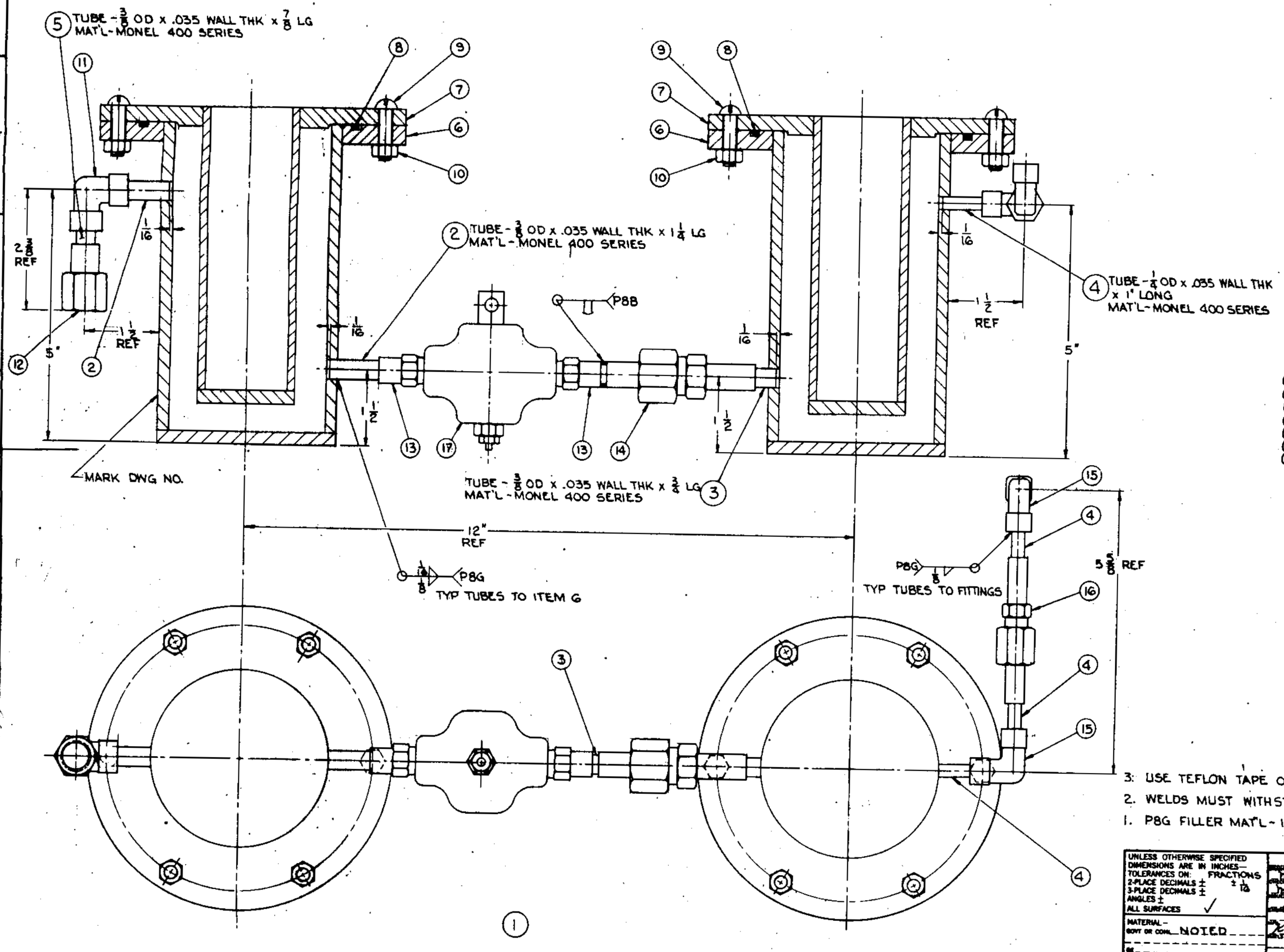


FIGURE 12 Double Trap Section for Bromine Trifluoride Removal (C 64062555)



ITEM	IDENTIFICATION NO.	DESCRIPTION OR NAME	ZONE	GROUP NO. & QTY 1	2	3	4
1		ASSY	AB	X			
2	4012286-180P2	TUBE	E9	2			
3	P3	TUBE	D7	2			
4	P4	TUBE	E4	3			
5	P5	TUBE	G11	1			
6	4012093-787G1	REACTION CELL OR TRAP	F9	2			
7	4012093-786G1	INNER TRAP MEMBER	F9	2			
8	MS29513-242	O-RING	G9	2			
9	COM'L STN STL	RD HD CAP SCR 1/4 UNC x 1" LG	G9	12			
10	COM'L STN STL	HEX NUT 1/4 UNC	F9	12			
(A) 11	M6SW-9	ELBOW	F11	1			
(A) 12	M6SW-7-6	FEMALE CONNECTOR	E11	1			
(A) 13	M6SW-1-6	MALE CONNECTOR	D9	2			
(A) 14	M6VC	VACUUM COUPLING	D8	1			
(A) 15	M4SW-9	ELBOW	D4	2			
(A) 16	M4VC	VACUUM COUPLING	C4	1			
(B) 17	COM'L	1/4 SLEEVELINE PLUG VALVE MONEL TEFLON	D9	1			

(A) CAJON COMPANY - CLEVELAND, OHIO

(B) DURCO COMPANY - CLEVELAND, OHIO

3. USE TEFLON TAPE ON THREADED JOINTS ON PT 17
2. WELDS MUST WITHSTAND 10^{-5} - 10^{-6} TORR WITH NO LEAKS
1. P8G FILLER MAT'L - INCO 60

REVISIONS				
SYM	ZONE	DESCRIPTION	DATE	APPROVED

UNLESS OTHERWISE SPECIFIED
DIMENSIONS ARE IN INCHES—
TOLERANCES ON: FRACTIONS ± 1/16
2-PLACE DECIMALS ±
3-PLACE DECIMALS ±
ANGLES ±
ALL SURFACES ✓
MATERIAL—
GOVT OR COML NOTED

SIGNATURES | DATE
F.R. Vester
6/20/64
6/30/64
June 30 1964

GENERAL ELECTRIC
A.E.T.D. DEPT LOC EVENDALE PLANT

TITLE
TWO TRAP ASS'Y
FOR BROMINE TRIFLUORIDE

CONTRACT NO.
CODE IDENT NO. | SIZE D | DWG NO. 4012286-180
SCALE 1/1 | WT | SHEET 1 OF 1

D 4012286-180

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WORK PLANNED FOR THIRD QUARTER

1. Determination of the blank or leak rate with bromine trifluoride in the system is now underway. With the objective being a leak rate of less than 10 micrograms of oxygen per hour, the welding of several minute leaks was necessary.

2. At least two different potassium samples (two levels of presumed oxygen content) will be obtained from NASA, Cleveland and used for the bulk of the work. Work will commence on determining: a) the precision and sensitivity of the method; b) the effect of sample size on the accuracy and bias; and c) the number of samples that may be run with a specific quantity of BrF_3.

3. The range of the method, with or without potassium carbonate addition, may require special samples not now available.

4. A method will be devised for the quantitative introduction of K_2CO_3 samples into the reaction cell.