NASA-CR 86330

Interim Scientific Report

on a simple, versatile

THERMOSTAT

for small objects and the temperature

range of 100°K to 400°K

designed and developed as an

outgrowth of research performed under

the sponsorship of the

NASA Electronics Research Center

under grant

NGR 05-002-100

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August 29, 1969

I. SUMMARY

The thermostat described in this report has been designed to meet the following specifications: (i) temperatures ranging from about 100°K to 400°K, (ii) a stability and an absolute error of ≤ 1°K, (iii) the ability to easily set and rapidly acquire (≤ 10 min.) any temperature within that range, (iv) a modest chamber volume of the order of 100 cm^3, (v) an easy access to the chamber (by light, magnetic fields, high frequency signals, etc.) for flexibility in application, (vi) an inexpensive and simple construction.

This system has been extensively and successfully used in studies of electrical properties of solids. To facilitate the construction of replicas in laboratories with similar needs, pictures, graphs, circuit diagrams, lists of parts and suppliers, as well as calibration procedures are provided. Liquid nitrogen is the primary temperature bath. Gaseous nitrogen serves as transfer medium. An electronic control system -- also fully described -- provides automatic regulation.

II. INTRODUCTION

A number of cryostats have been described in the recent past[1 - 11] or are commercially available, but none satisfies the conditions we wanted to meet[12]: (i) a temperature range extending approximately from 100°K to 400°K, (ii) a stability and accuracy of about 1°K, (iii) a modest active volume of the order of 10 cm^3, (iv) a fast response ($\leq$10 min.) to changes in the due temperature and simple means to set this due temperature, (v) a flexible design which could easily be adapted to varying experimental conditions, such as access by light, magnetic fields, fast electronic signals, etc., (vi) an inexpensive and simple construction. We feel that these conditions are frequently encountered in other laboratories and thus report here in detail the features of our thermostat.

To meet the requirement of flexibility and easy access to the chamber, a streaming nitrogen gas is used as temperature bath. This method has the advantage that the primary cooling agent (liquid nitrogen) can be separated from the object under study, which then can be placed easily in a magnet, a fast electronic gear, etc. In addition to this high flexibility in lay-out, a streaming gas assures an intimate thermal contact with the object independently of its mechanical support. These advantages are significant particularly for small objects, such as are frequently encountered in the study of physical properties of crystals and solid-state devices. The streaming gas also maintains a dynamic, rather than a static thermal equilibrium. This warrants a speedy response to changes in the due temperature.

Simple means of construction are incompatible with vacuum insulation. The design is thus based on closed-cell polyethylene foam as the main element of thermal insulation. With vacuum insulation the system will almost reach 77°K, but at a loss of flexibility and mechanical simplicity. In addition, the cooling capacity of the gas falls off significantly. For these reasons, evaporative cooling of liquid nitrogen should be given the preference at temperatures between 100°K and 77°K. On the other hand, only minor improvements in the thermal insulation of the chamber are required to extend the temperature range beyond 400°K.

The electronic control system maintains the temperature fluctuations at the object within 0.5°K, provided a Chromel-Alumel thermocouple with the necessary intimate thermal contact to the object is available. The digital dial selecting the temperature simulates the emf expected from that thermocouple at that temperature. The reading is calibrated absolutely in volts, so that standard tables of thermocouple voltages can be used directly to select the desired temperature. The regulator is carefully optimised for its performance. Significant improvements will necessitate major additional sophistication.

Our experience with the system shows that, if built and operated properly, it will perform reliably over extended periods of use. The pages below have been written in the hope that other laboratories will be able to share this experience. An abbreviated description of the thermostat and a presentation of some of its performance and characteristics is also given in Vol. 41 (1970) of the Review of Scientific Instruments.

The report is subdivided as follows:

III. PRINCIPLE OF OPERATION

Figures 1 and 2 show pictures of the thermostat. A 4 liter dewar with wide neck (Fig. 3) contains liquid N_2. Purified air or nitrogen gas from an external supply flows through spirals of copper tubing (Fig. 4) immersed in the liquid. The cold gas is transferred to the thermostat chamber through a brass tube (Fig. 3) which is thermally insulated against the room temperature by three layers of 1/2" thick polyethylene foam and one outer layer of 1/4" thick sheet of the same material (Fig. 2). The transfer tube is rigidly attached to a phenolic plate (Fig. 4). This plate itself is clamped onto the top of the dewar and serves as a tight lid to it. A hole in the lid serves both as a vent for the evaporating nitrogen and for refilling liquid N_2 during long runs. In Figs. 1-3 the transfer tube has the shape of an elbow to be able to reach between the poles of a magnet or over the top of a bench. The whole unit can be raised or lowered with a little jack and moved on four cart wheels (Fig. 1).

The thermostat chamber (Fig. 5) housing the heater (Fig. 6) and the active volume is attached to the end of the transfer tube with a phenolic flange. Polyethylene also surrounds this joint (located under the thickened segment of polyethylene in Fig. 2) and the chamber assembly (open ended head section of polyethylene in Fig. 2). With this layout, different head assemblies are easily interchanged to suit different needs. The object under study is attached to a front cover of phenolic which seals off the chamber with an O-ring. The front cover is thermally insulated with polyethylene as well. (This cover is removed in Figs. 1 and 2 to show the chamber opening.) A sensing thermocouple in good thermal contact with the object is also inserted into the chamber through the cover, as well as other leads and feed throughs necessary in the experiment. The sensing thermostat controls an electronic regulator which provides electrical power to the heater.

For fast response to changes in the due temperature, the wall chambers are made of thin brass tubing and the heater consists of printed circuit-type meanders of a thin metal strip on a flexible, heat-resistant substrate about 1" wide and 4-1/2" long (Fig. 5 and 6). This substrate is wound in a spiral at the inner end of the chamber (Fig. 6 and 7). The cold gas is forced to circulate within the spirals before entering the chamber proper. The electrical connections to the heater and a supplementary thermocouple are visible in Figs. 1 and 2 also.

The gas leaves the chamber through holes located towards the front in the thin brass wall (Fig. 5). The gas then flows back along the outer side of the wall before escaping to the outside through the tube pointing downwards in Fig. 7. This reverse-flow arrangement improves the uniformity of the temperature within the chamber and cuts down the requirements on gas consumption. A straight pattern of flow can also be adopted, at some loss of performance. This eliminates the double wall of the chamber and simplifies the mechanical design still further. For operation above room temperatures the dewar is left empty.

The electronic control system (Fig. 8 and 9) contains a commercial component oven for the reference junction of the thermocouple. A temperature compensated Zener diode provides a standard from which a voltage is derived whose value equals that expected from the thermocouple at the thermostat temperature desired. This voltage is subtracted from the actual emf of the thermocouple, the difference is amplified up to 10^6 times and fed back into the heater. For convenience, the potentiometer selecting the voltage is referenced to a virtual electrical 0°C point and is equipped with a digital dial calibrated in millivolts. Standard thermocouple tables can then be used directly to set the temperature.

IV. GENERAL DESCRIPTION AND LIST OF SUPPLIERS

The complete description of the control system is given in the circuit diagrams of Figs. 10 - 12. Figure 10 shows the overall schematic. Figure 11 gives the diagram of the control chassis, and Figure 12 that of the reference chassis. Figure 8 is a picture of the front panel.

The overall mechanical construction of the thermostat is shown in the pictures of Figs. 1 and 2. Figures 3 to 6 are pictures of separate parts, and Fig. 7 gives a drawing of the thermostat chamber and heater assembly.

A descriptive list of parts is added to each picture. A list of the suppliers of special items follows:

Thermos vacuum jar; Laboratory Big Jack

Van Waters and Rogers
1363 South Bonnie Beach Place
Los Angeles, California 90023 Phone: (213) 269-9311

Polyethylene foam

Arlon Products Incorporated
2394 South Vermont Ave.
Harbor City, California 90170 Phone: (213) 325-9700

Flow meter

Brooks Instrument Division
407 West Vine Street
Hatfield, Pennsylvania 19440 Phone: 855-5174

Brooks Instrument Division
3106 West Beverly Blvd.
Montebello, California 90640 Phone: (213) 685-5980

Armstrong epoxy A-12

Lunar Products, Inc.
10782 Weaver Street
South El Monte, California Phone: (213) 442-4630

Thermofoil heater

Minco Products Inc.
7300 Commerce Lane
Minneapolis, Minnesota 54432 Phone: (612) 786-3121

Representative
The Pentagon Company
1010 Fair Oaks
South Pasadena, Calif. 91030 Phone: (213) 799-7141

Amphenol Microdial

Amphenol Controls Division
Bunker-Ramo Corp.
120 South Main Street
Janesville, Wisconsin 53545 Phone: (608) 754-2211

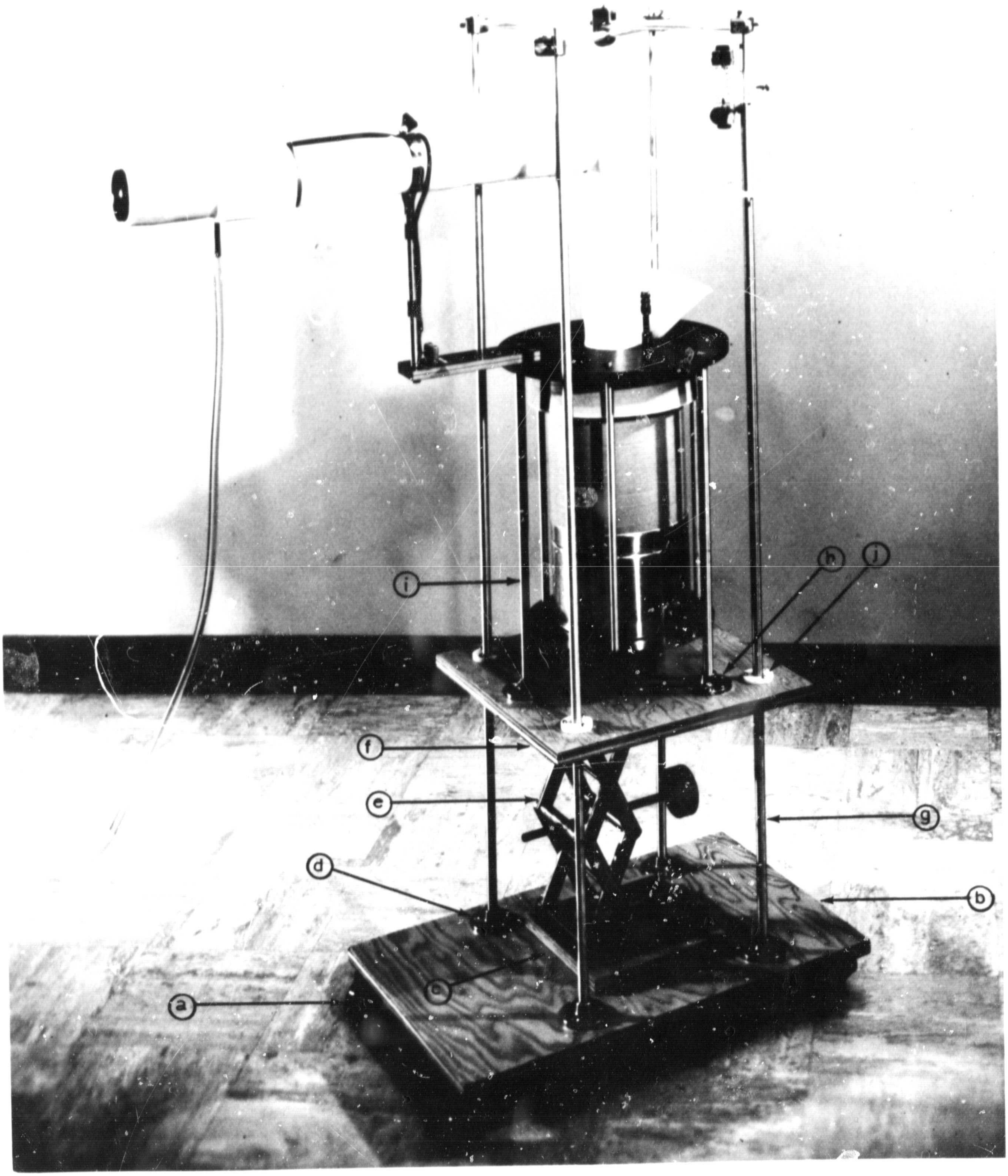
Thermocouple Connector

Barber-Colman Co.
Rockford, Illinois 61101 Phone: (815) 877-0241

This instrument has been designed and constructed under the sponsorship of NASA Electronics Research Center, Cambridge, Mass. It has been developed from a prototype built in connection with research performed under NGR 05-002-100, and with Dr. John Hopkins as technical officer.

V. FIGURE CAPTIONS AND FIGURES

Figure 1	Thermostat
Figure 2	Upper Portion of Thermostat
Figure 3	Liquid Nitrogen Dewar
Figure 4	Gas Cooling Spirals
Figure 5	Thermostat Chamber and Heater Assembly
Figure 6	Heater and Heater Element
Figure 7	Schematic of Chamber and Heater Assembly
Figure 8	Control Panel
Figure 9	Control System Block Diagram
Figure 10	Control System Overall Schematic
Figure 11	Control Chassis
Figure 12	Reference Chassis


i
h
j
f
e
g
d
b
c
a

Thermostat

a. Plate caster (4x).

b. 3/4" Plywood 14" - 22".

c. 3/4" Plywood 7 1/2" - 7 1/2" (2x) bottom and top of jack.

d. Flexaframe foot (4x).

e. Platform, laboratory Big Jack.

f. 3/4" Plywood 14" - 14".

g. 48" aluminum rod 1/2" OD. (4x).

h. Flexaframe foot (3x).

i. 17" aluminum rod 1/2" OD. (3x).

j. Nylon bushing (4x).

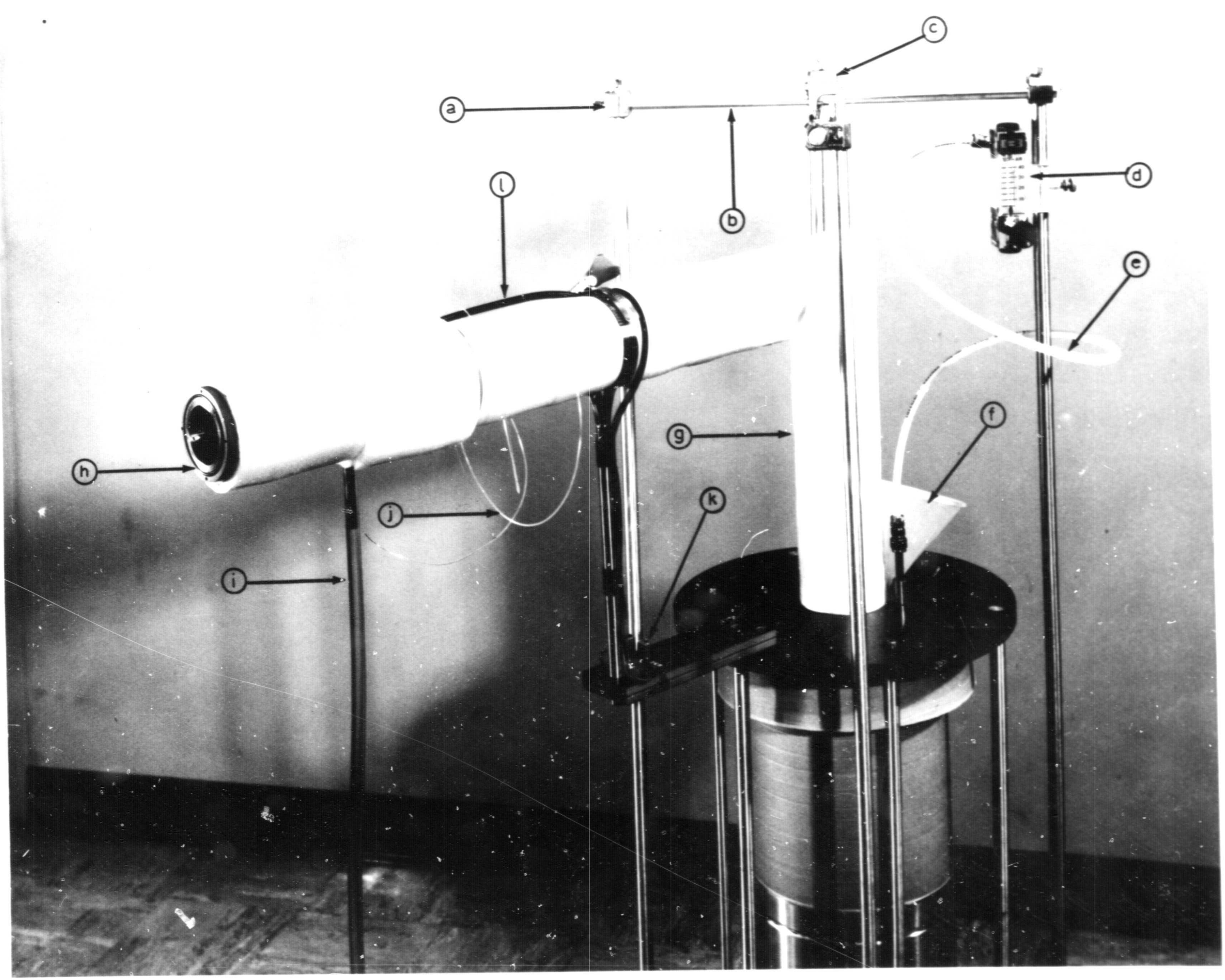


Figure 2

Upper Portion of Thermostat

a. Flexaframe connector (4x).

b. 15" aluminum rod 1/2" OD. (2x).

c. Flexaframe hook connector.

d. Brooks-mite flowmeter model 2001-V capacity 40 S.C.F.H.

e. Poly-Flo tubing 1/61" OD.

f. Fumel.

g. 1-1/4" thick polyethylene foam, closed-cell, Type No. 9A Arlon

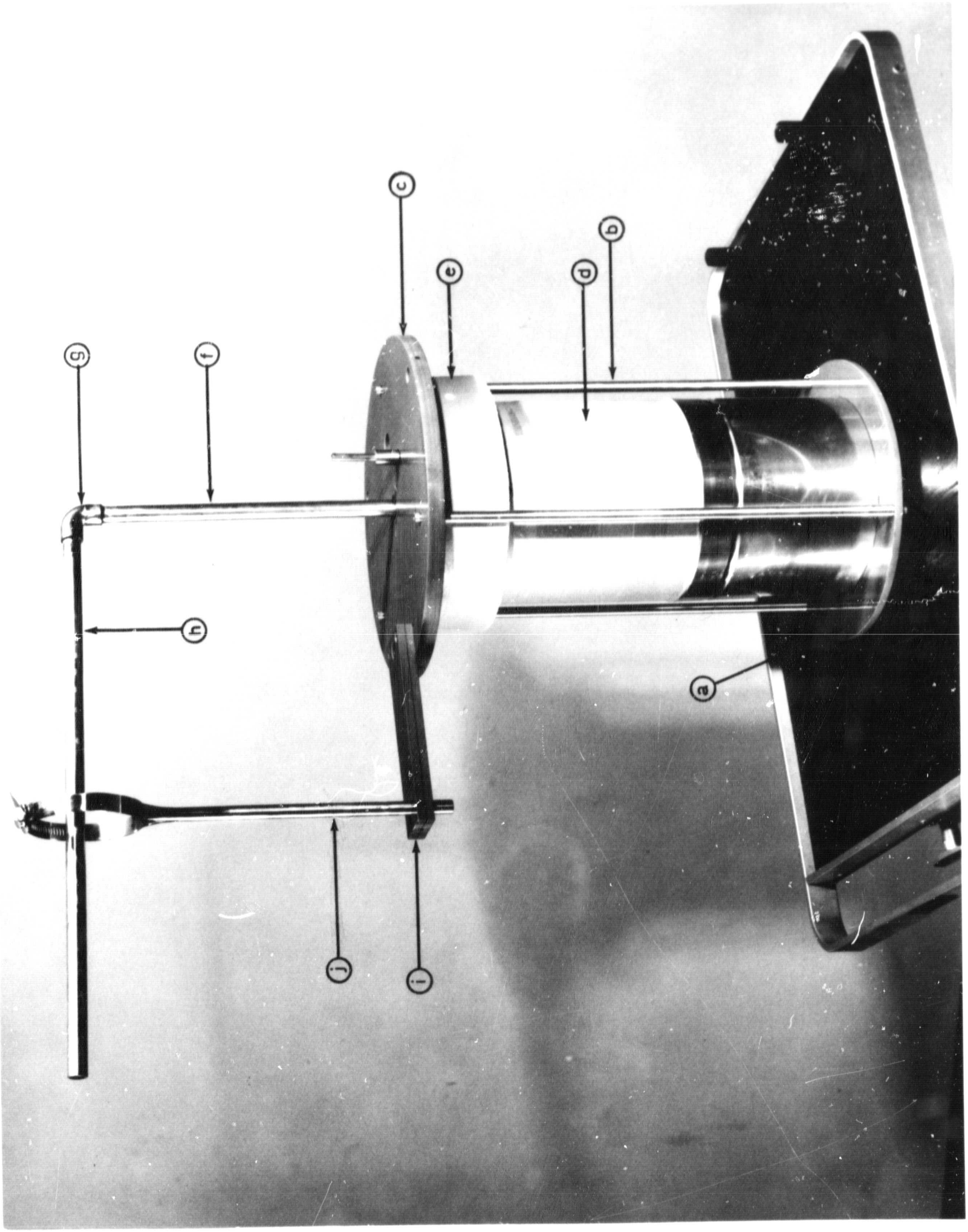
h. Chamber.

i. Flexible PVC tubing 1/61" ID.

j. Thermocouple wires.

k. Binding post (2x).

Figure 3



Liquid Nitrogen Dewar

a. 1/4" Phenolic 9 1/2" OD.

b. 13 7/8" aluminum rod 3/8" OD. (3x).

c. 1/2" Phenolic 11 1/2" OD.

d. Thermos vacuum jar, pyrex glass 4300 ml.

e. Polyethylene foam.

f. 22" Brass tube 9/16" OD. 18 GA.

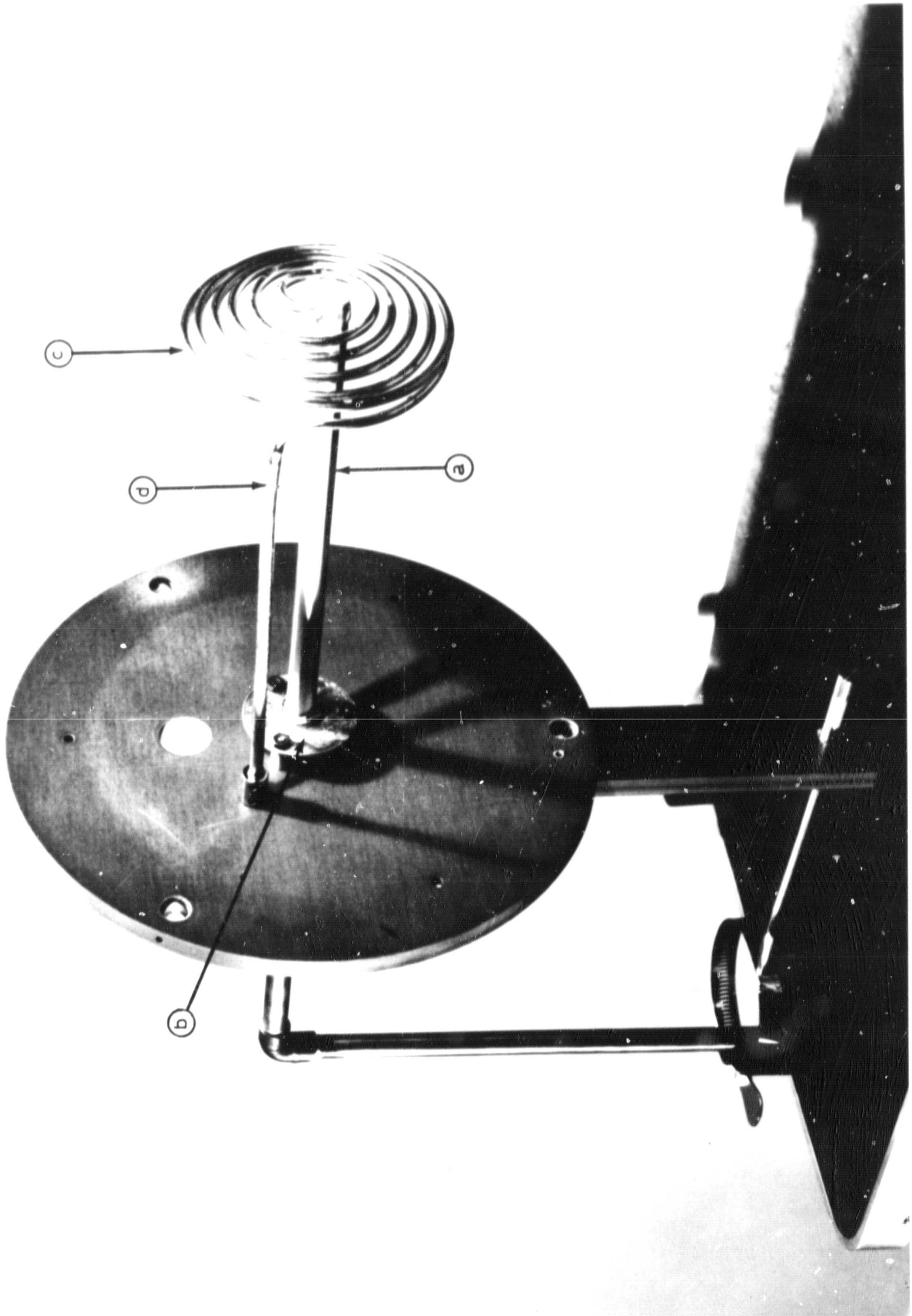
g. Brass, elbow.

h. 18" Brass tube 9/16" OD. 18 GA.

i. 1/2" Phenolic 2"-6 1/2".

j. Varigrip SS. clamp.

Figure 4



Gas Cooling Spirals

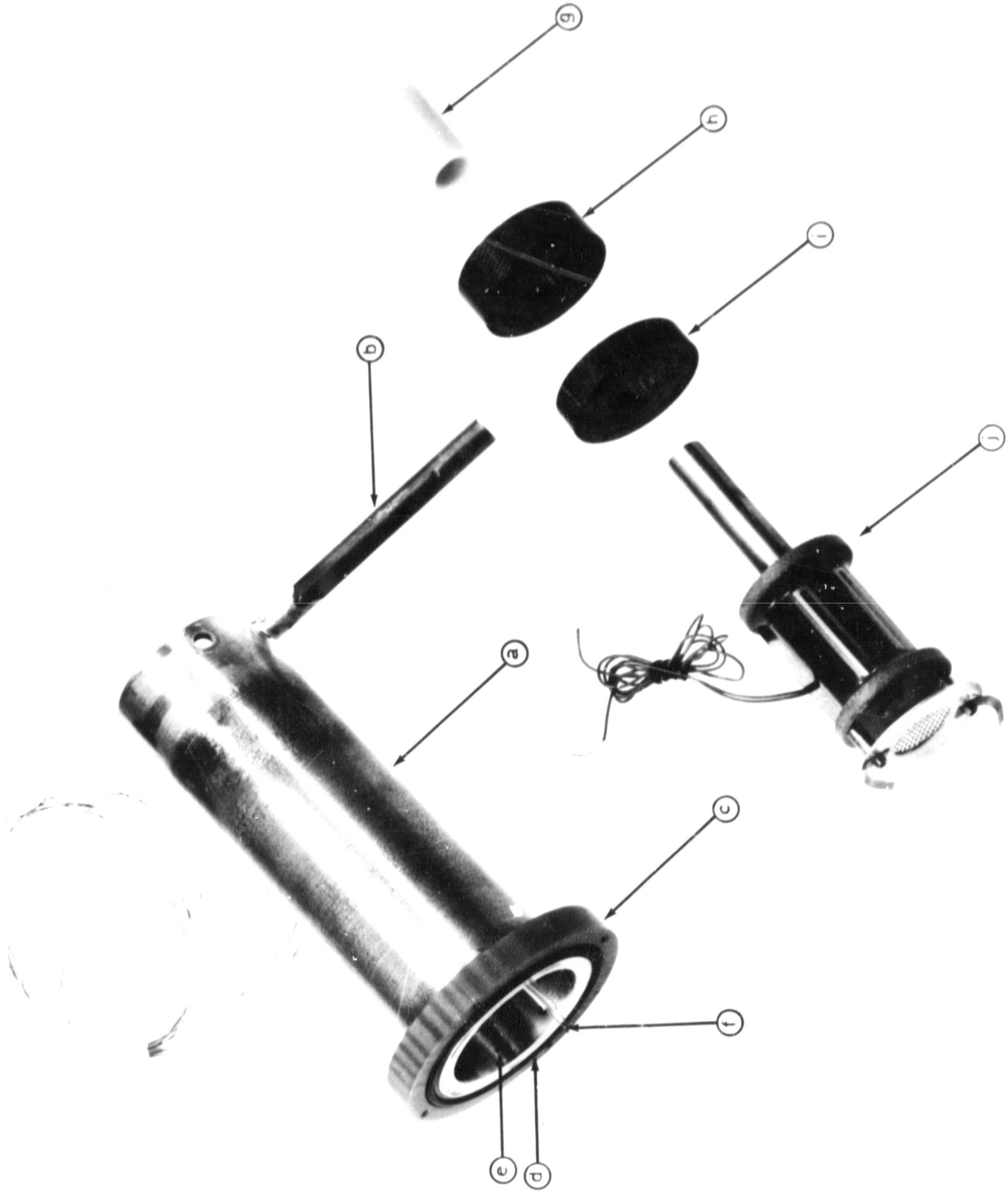
a. 22" Brass tube 9/16" OD. 18 GA.

b. Brass mounting flange.

c. 60" Copper tubing 3/16" OD.

d. Copper tubing 1/4" OD.

Figure 5



Thermostat Chamber and Heater Assembly

a. 4" Brass tube 1.290 OD. .020 thick.

b. 2 1/2" Brass tube 1/4" OD. 22 GA.

c. 1/4" Phenolic.

d. Viton O ring No. 2-29.

e. 4" Brass tube (inner) 1 1/8" ID. .020 thick.

f. Thermocouple wires No. 28 chromel-alumel.

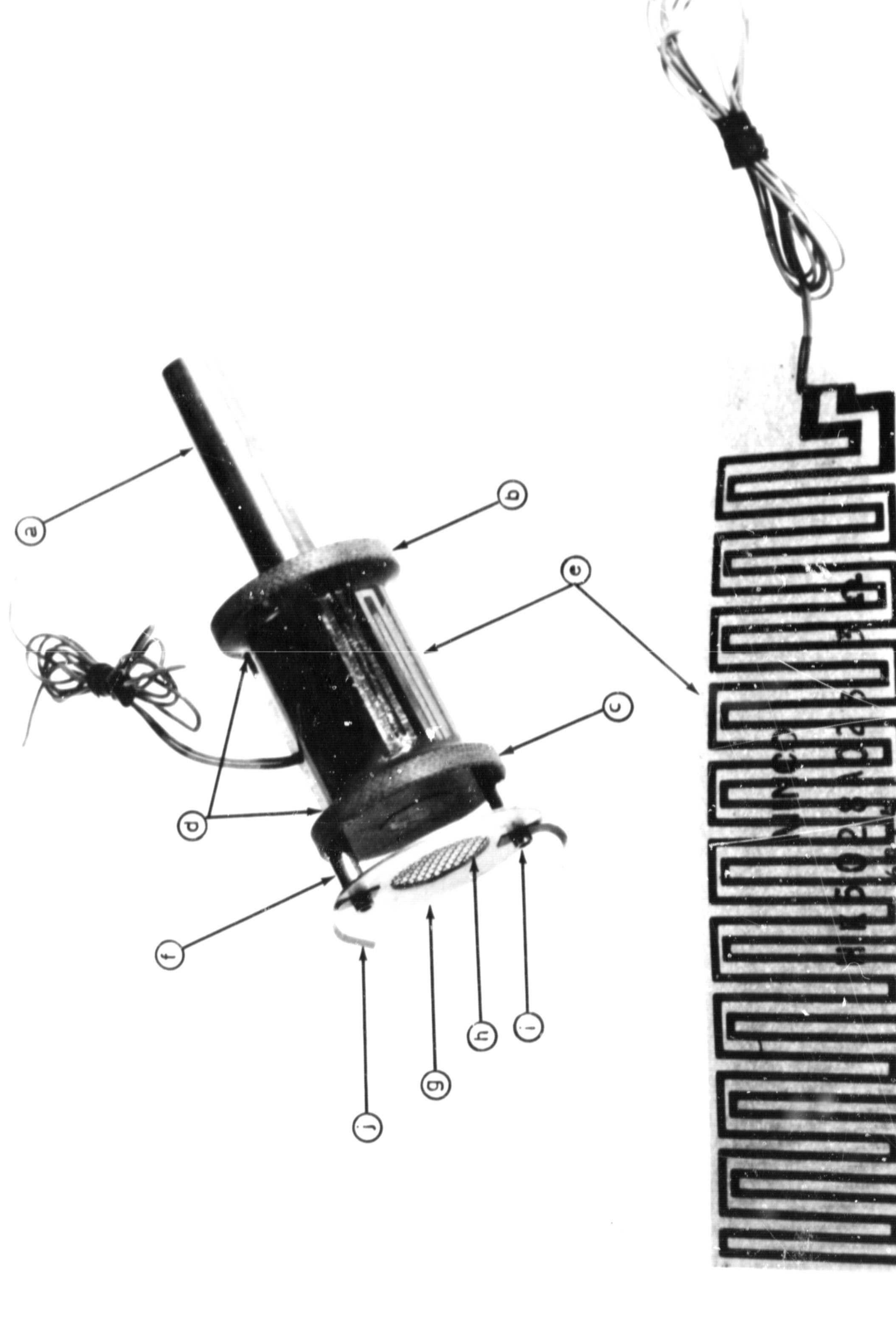
g. 881 RTV. rubber.

h. 3/8" Phenolic 1 1/8" OD.

i. 1/4" Phenolic 1 1/8" OD.

j. Heater assembly.

Figure 6



Heater and Heater Element

a. 2 3/4" Brass tube 5/16" OD. 24 GA.

b-c. 1/8" Phenolic .960 OD.

d. Armstrong epoxy adhesive A-12.

e. Minco thermofoil heater No. HK 5028 23.3 ohms max. 30.6 watts.

f. 1/4" Brass tube 1/8" OD. 25 GA.

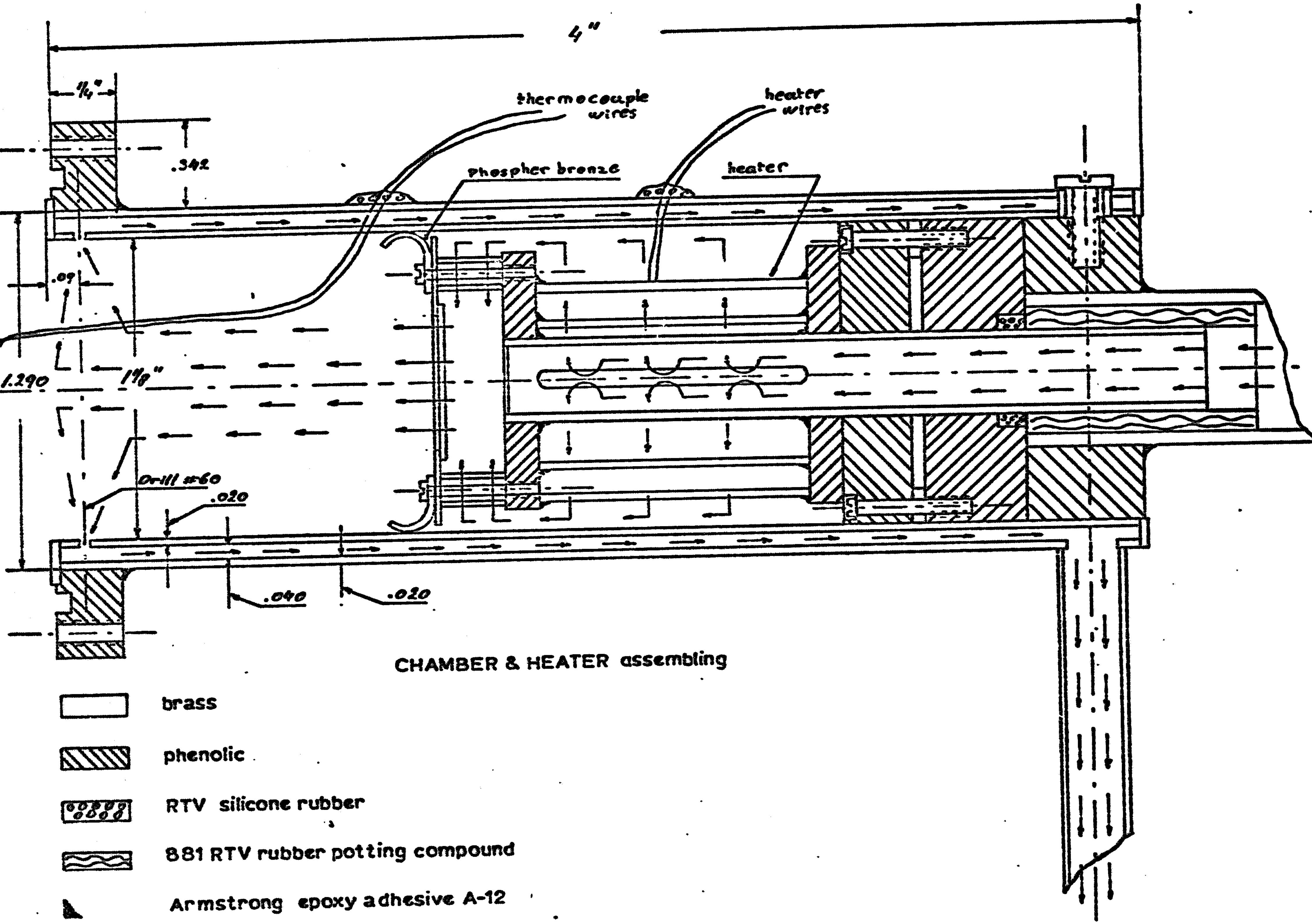
g. .020 Brass 1.075 OD.

h. Screen SS.

i. 0-80 screws SS.

j. Phospher bronze spring contacts.

Figure 7



CHAMBER & HEATER assembling

brass

phenolic

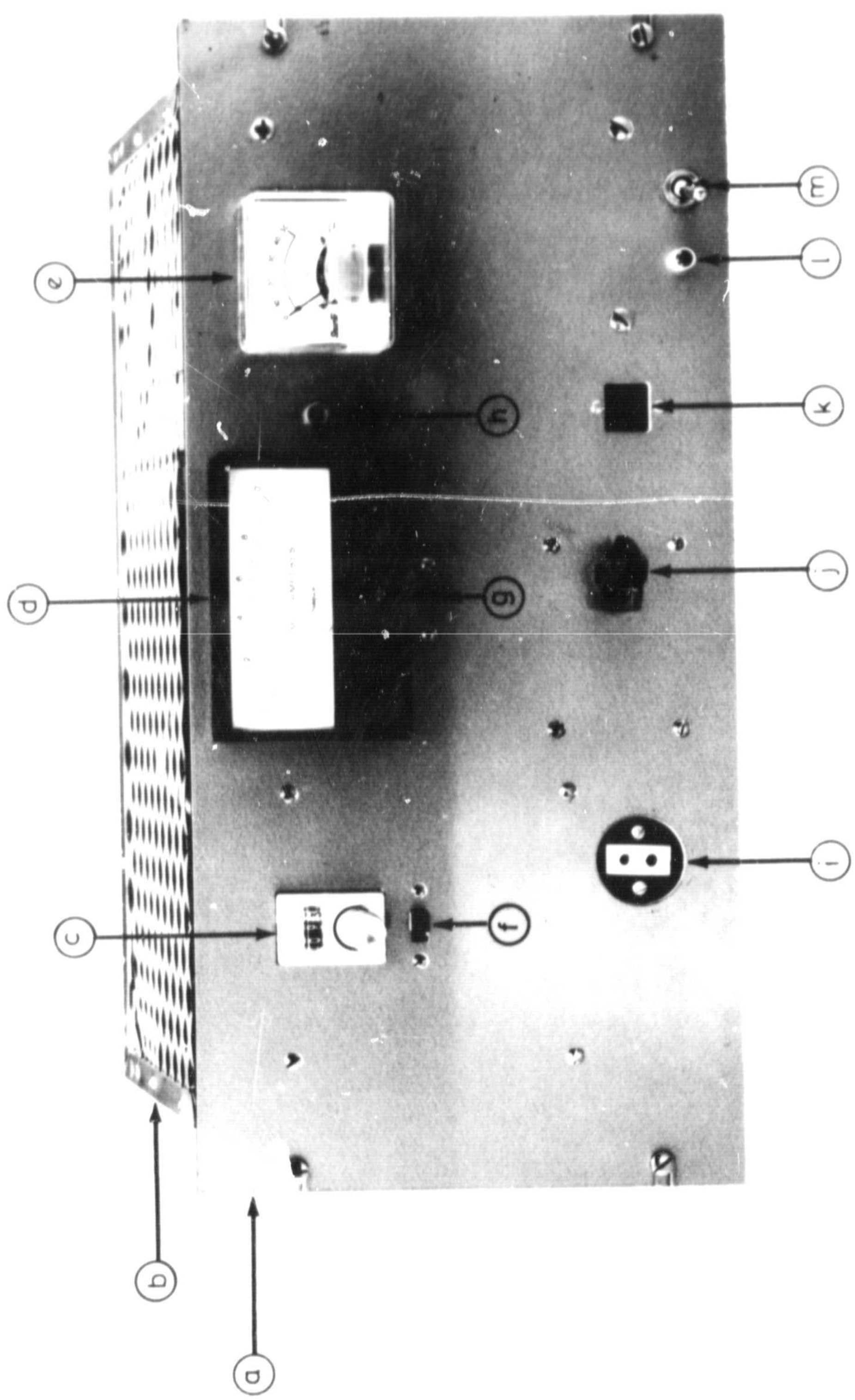
RTV silicone rubber

881 RTV rubber potting compound

Armstrong epoxy adhesive A-12

air flow

Figure 8



Control Panel

a. Aluminum gray hammertone rack panel, 8-3/4" - 19".

b. Aluminum panel-mounting chassis, 8-3/4" - 17".

c. Temperature selector, calibrated in units of 10^{-4}Vdc of a chromel-alumel thermocouple referenced to 0^{o}C; Amphenol Microdial 3141.

d. Temperature indication (in the final versions the face of the meter is redrawn in ^{o}C); Simpson Meter Model 524, 0-1mAdc.

e. Heater voltage indication; Shurite Meter Model 850, 0-50Vdc.

f. Selector switch of the temperature dial for operation below or above 0^{o}C.

g. Selector switch of the temperature indication for operation below or above 0^{o}C.

h. Recorder output.

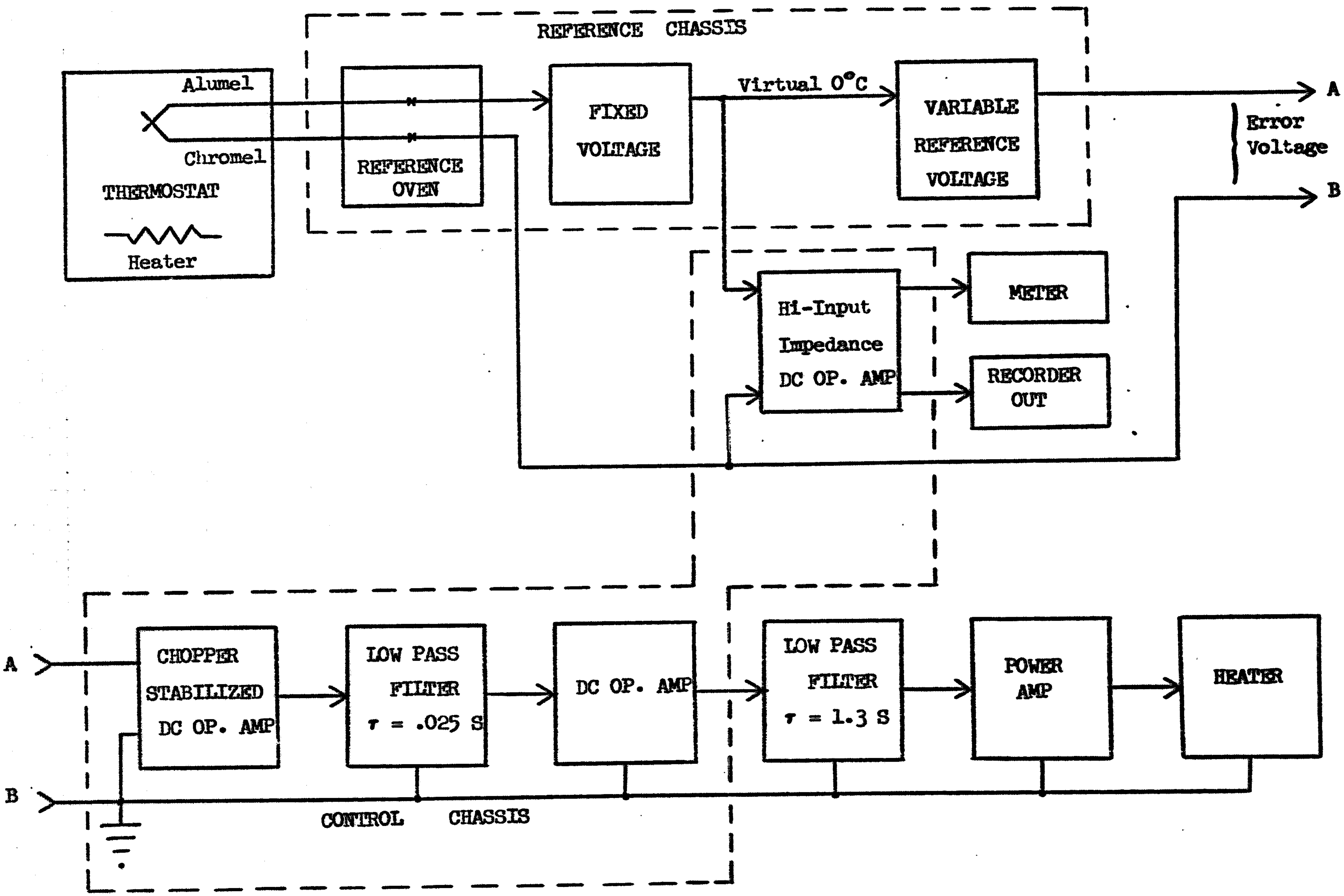
i. Chromel-Alumel thermocouple connector; Barber-Colman Co.

j. Selector switch for the gain of the control amplifier, in steps of (x1, x3, x10, x30, x100, x300) $x10^{3}$.

k. Heater output.

l. Pilot light.

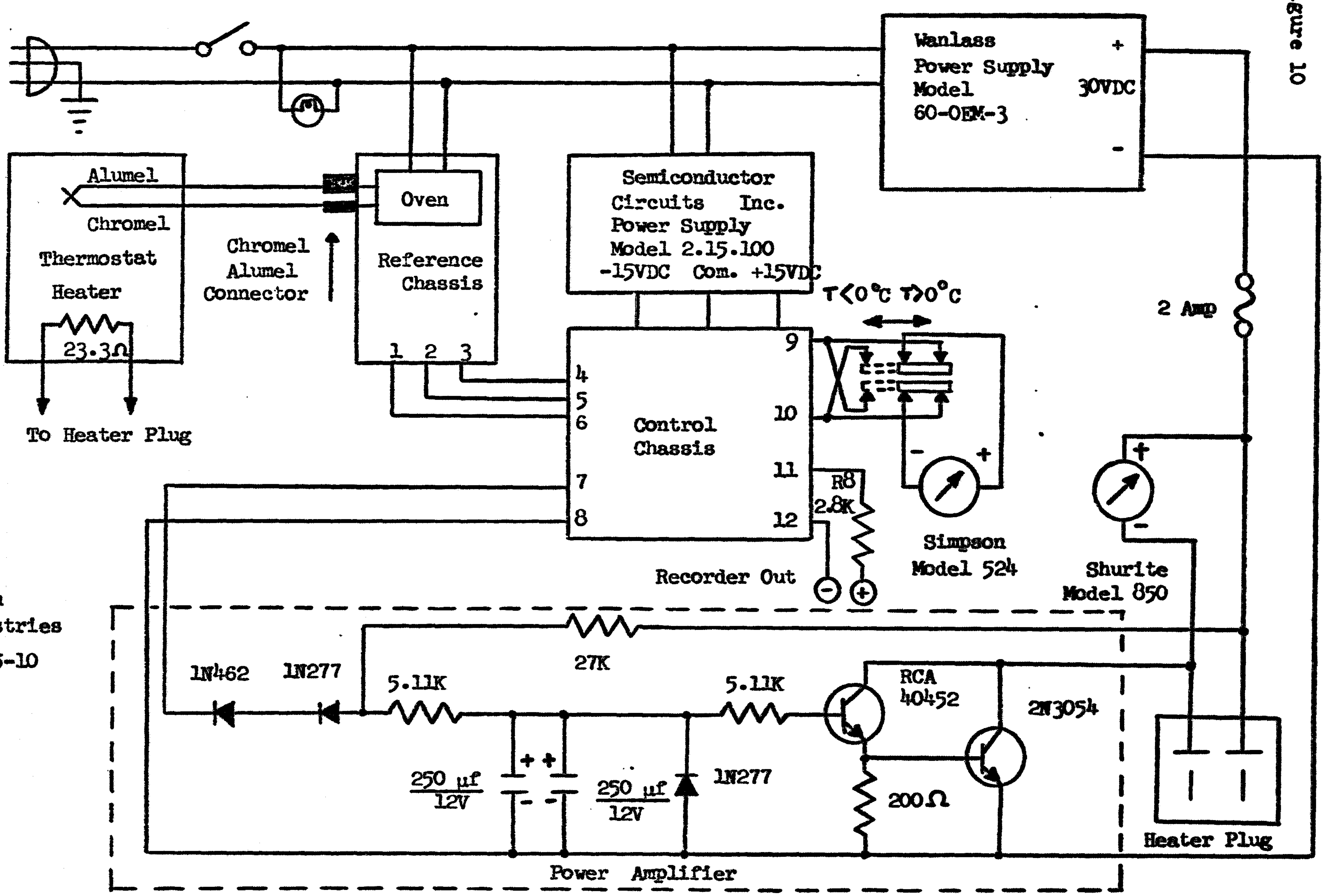
m. Power switch.



CONTROL SYSTEM BLOCK DIAGRAM

Figure 9

Figure 10



Oven is an
Oven Industries
Model 1765-10

CONTROL SYSTEM OVERALL SCHEMATIC

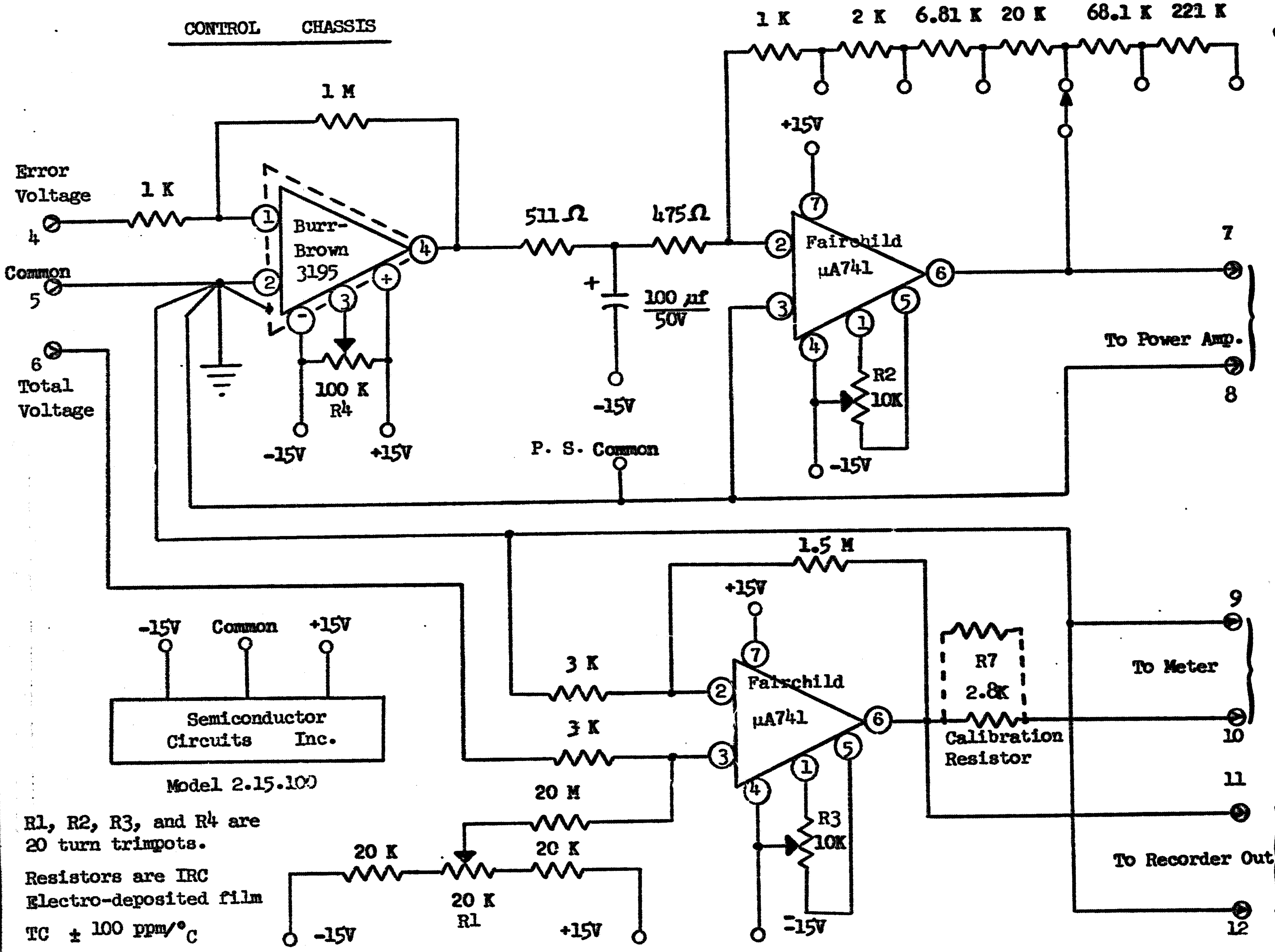
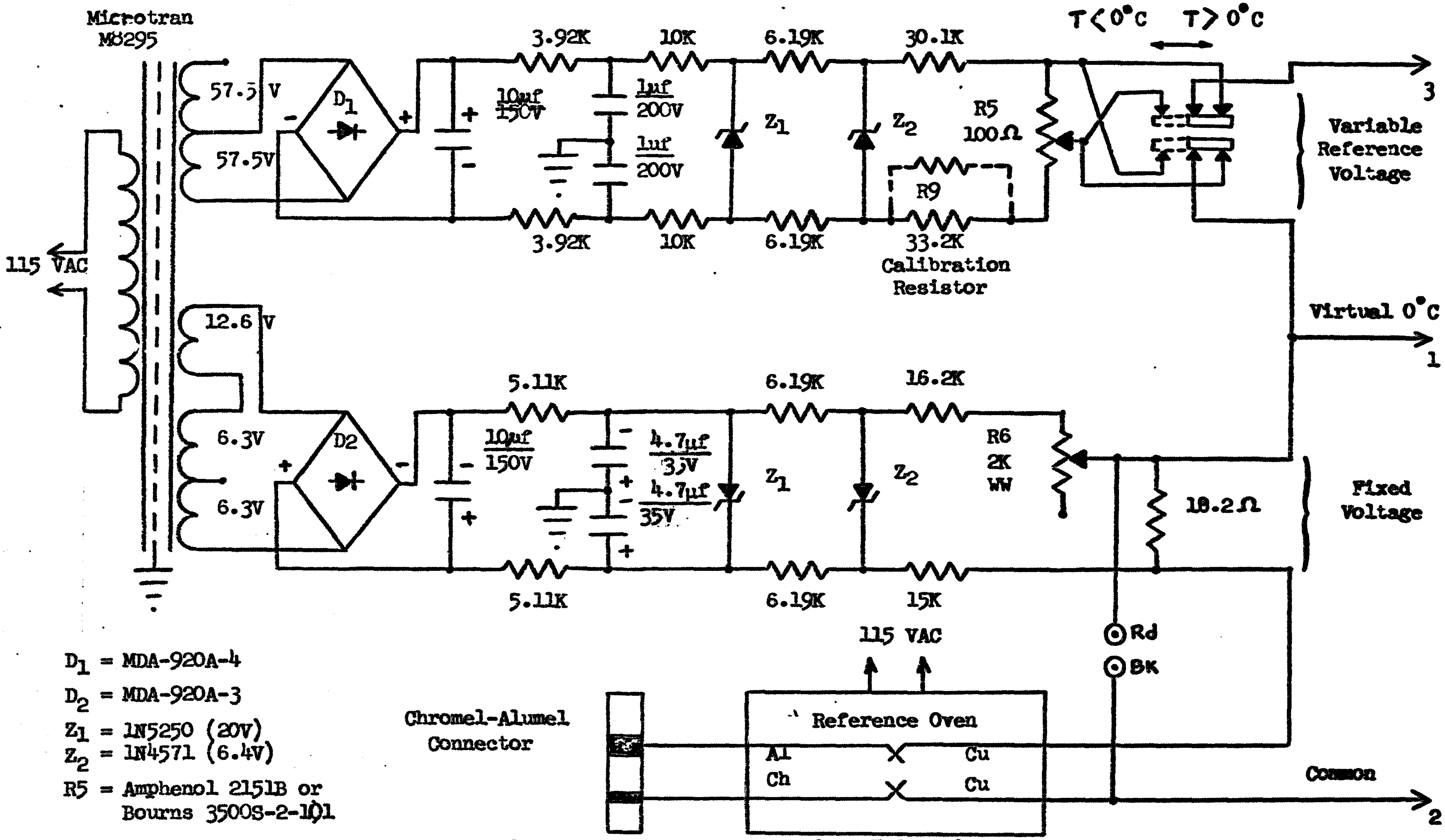

CONTROL CHASSIS
1 K
2 K
6.81 K
20 K
68.1 K
221 K
1 M
Error Voltage
4
1 K
Burr-Brown 3195
Common
5
6
Total Voltage
100 K
R4
-15V
+15V
511 Ω
475 Ω
+
100 µf
50V
-15V
P. S. Common
+15V
Fairchild µA741
R2
10K
-15V
7
To Power Amp.
8
1.5 M
+15V
-15V
Common
+15V
Semiconductor Circuits Inc.
Model 2.15.100
3 K
3 K
Fairchild µA741
R7
2.8K
Calibration Resistor
R3
10K
-15V
9
To Meter
10
11
To Recorder Out
12
20 M
20 K
20 K
20 K
R1
-15V
+15V
R1, R2, R3, and R4 are 20 turn trimpots.
Resistors are IRC Electro-deposited film
TC ± 100 ppm/°C

Figure 11



D_1 = MDA-920A-4

D_2 = MDA-920A-3

Z_1 = 1N5250 (20V)

Z_2 = 1N4571 (6.4V)

R5 = Amphenol 2151B or
Bourns 3500S-2-101

Resistors are IRC Electro deposited film
TC ± 100 ppm/°C

REFERENCE CHASSIS

Figure 12

VI. CALIBRATION AND ADJUSTMENT PROCEDURE

Reference Chassis (See Figure 12)

Virtual 0°C:

The reference oven temperature cannot be adjusted by the user. Therefore a separate voltage supply with a screw-driver adjustable variable resistor R6 is supplied in the reference chassis ("Fixed voltage" on the Control System Block Diagram, Fig. 9), which is adjusted to produce zero μV between output 1 ("virtual 0°C") and 2 (common) of the reference chassis, when the sensor thermocouple is plunged into an ice-water mixture. (Note that the reference oven temperature is cycling with a peak-to-peak temperature equivalent to about 10 μV.) This adjustment which requires a microvoltmeter, may be repeated every two months, to compensate for a possible drift of the oven. Points 1 and 2 are easily accessible through the two banana jacks on top of the reference chassis.

Variable reference voltage:

A separate power supply is designed to produce 10.00 mV across a ten-turn potentiometer with a specified nonlinearity of ± 0.25%. To adjust this voltage it is necessary to solder (a) resistor(s) parallel to R9 (calibration resistor). A digital voltmeter capable of < 0.1% accuracy on a 10 mV-scale is desirable for this procedure. The voltage between output 3 and 1 on the reference chassis is adjusted to 10.00 mV when the dial is set to 10.00 mV (do not measure across the ends of the potentiometer). During this procedure, the output 3 is to be disconnected from the control chassis.

Control Chassis (See Figure 11)

This chassis houses the error signal amplifier and the thermocouple voltage amplifier. Operational amplifiers are used throughout. Compensation for their offset voltages and currents are provided.

Error signal amplifier, first stage:

A Burr-Brown chopped operational amplifier is used for this stage. Its specified bias current of < 1nA is of no concern. The offset voltage is compensated with R4. To achieve this a millivoltmeter is connected to the output of the Burr-Brown (with second stage disconnected), the error voltage input 4 and 5 is shorted and R4 is adjusted until the DC-voltage at the output is minimized. A slow millivoltmeter is desirable in order to reduce the fluctuations due to noise.

Error signal amplifier, second stage:

The gain of the Fairchild μA741 used in this stage is variable between x1 and x300. Again, there is no compensation for the bias current. The voltage offset is compensated with R2 by short-circuiting the input (in front of the 511Ω resistor) and adjusting for minimum DC-output. The gain values are only coarsely adjusted. They are not critical.

Thermocouple voltage amplifier:

Since this stage is connected to the input, provisions are made to prevent offset currents from flowing out of this stage back into the input. Thus, a bias current adjustment (R1) is provided as well as an offset voltage adjustment. First, input 3 of this op amp is connected to common and the output 6 is zeroed through R3. Next, the input in front of the 3KΩ-resistor is connected to common, and the output 6 is again zeroed through R1.

Thermocouple voltmeter and recorder:

Calibration resistor R7 is adjusted to provide a calibration of the meter.

To do this the sensor thermocouple is plunged into liquid nitrogen (the heater output may be disconnected), the dial of the reference voltage is set to 5.70mV and R7 is paralleled with other resistors, until the meter reads 99.0% of full scale. The recorder output is connected to the output of the opamp through a 2.8KΩ resistor. The output current into the recorder should not exceed 3mA if this resistor were to be changed.

VII. OPERATING INSTRUCTIONS

Close the thermostat chamber, which must contain a sensing Chromel-Alumel thermocouple in good thermal contact with the object under investigation.

BEFORE turning on the power at the panel:

(a) - connect the sensing Chromel-Alumel thermocouple to the front panel input.

(b) - position the switch of the temperature dial to $T < 0$.

Turn on the power. The heater dial must come to rest on zero. This is the stand-by position of the control system (maintaining the reference oven at steady state).

Failure to comply with (a) will cause latch-up of the Burr-Brown op amp. Turn off the power and start again after 5 minutes. Failure to comply with (b) may turn on the heater power.

BEFORE operating the thermostat:

(c) - blow out the heat exchanger (copper spirals) with room temperature gas for a few minutes (to eliminate moisture possibly accumulated in the coils). Then set the gas flow rate to approximately 20 scfh.

(d) - set the temperature dial to the desired Chromel-Alumel thermocouple reading.

(e) - fill the dewar with liquid N_2 (omit for temperatures above that of the room).

(f) - connect the heater to the front panel output.

NEVER connect the heater to the front panel output without a finite flow rate of gas through the thermostat. The heater output handles enough power to damage the heater element if heat dissipation is insufficient.

To stop the thermostat, either:

(a) - disconnect the heater, and

(b) - turn off the gas flow;

or (for T < room temperature)

(c) - let the supply of liquid N_2 run out. The system will turn off the heater automatically and slowly reach room temperature, then

(d) - disconnect the heater, and

(e) - turn off the gas flow.

ALWAYS disconnect the heater before turning off the gas flow.

Open the chamber.

VIII. SPECIFICATIONS

Power Requirements:	115 Vac; 60 Hz: 70 W maximum at full heater power.
Warm-up Time:	30 minutes (required to establish steady state conditions in the reference oven).
Maximum Heater Power:	30 W (as specified by manufacturer).
Maximum Operating Temperature:	90°C (determined by the softening point of the polyethylene foam insulating the thermostat chamber).
Minimum Operating Temperature:	approximately -173°K (100°K, and determined by the maximum acceptable flow rate of gas).
Short-Term Fluctuations:	≤ 0.5°C
Long-Term Fluctuations:	≤ 0.5°C
Absolute Error:	approx. 1°K

Typical Consumption of N_2 gas at 10 psi pressure

thermostat temperature	scfh (standard cubic feet/hour)	ℓ/h
355°K (+82°C)	20	660
200°K (-73°C)	20	660
115°K (-158°C)	> 40	> 1320

Typical Gas Pressure:	10 psi (0.68 atm)
Typical Consumption of liquid N_2:	1 ℓ/h (at 20 scfh flow rate of gas).
Recorder Output:	± 2.8 Vdc at full scale deflection of temperature meter, and directly proportional to meter deflection; 2.8KΩ source resistance (determined by R8, which is connected to the red output terminal.
Temperature Sensing:	Chromel-Alumel thermocouple.

IX. REFERENCES

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12. D. H. Lee and M-A. Nicolet, Phys. Rev. 184, 806 (1969).

X. APPENDIX: WARNING AND SIMPLE IMPROVEMENT

Warning:

In a few cases, the reference oven in the reference chassis of the electronic regulation failed to regulate, and its temperature ran away to well above 100°C (from the specified setting tolerance of 85°C ± 3°C). In this case, the temperature indicated on the panel (with sensor thermocouple at room temperature) will be well below room temperature, and the reference oven case will be quite warm (well over 40°C). It is suggested that the supplier (see manual) is then contacted, and the oven is replaced by a better model.

A Simple Improvement:

To achieve quick increases in temperature (e.g. -150°C to +80°C in a few minutes) the modification in layout sketched below is useful.