INTRODUCTION

This report contains a description of the distillation laboratory at Bruceton, Pa., which was established to aid in the separation and identification of the products produced in the synthetic liquid fuel processes under study there. Compounds present in synthetic liquid fuels may include aliphatics from C_1 to C_{60} or greater. Alcohols, ketones, esters, aliphatic carboxylic acids, saturated hydrocarbons, olefins, and diolefins are all found in Fischer-Tropsch products. Coal-hydrogenation products contain, in addition to aliphatic compounds, a large proportion of naphthenic material and aromatic compounds ranging from benzene to coronene; in addition, appreciable quantities of tar bases and tar acids are present. In designing the laboratory, therefore, provision had to be made for the greatest amount of flexibility and versatility. A great deal of standardization of basic equipment had also to be designed to permit interchange of parts and services.

GENERAL

The distillation laboratory is in a bay 18 by 22 feet by 13 feet, 7 inches high off the main organic-chemistry laboratory. Along the windowless sides, a four-tier steel framework 10 feet high is mounted 9 inches from the wall. The wall supports the various services, whereas the framework supports the columns and accessory receivers, condensers, timer lines, and thermocouple services. The distillation flasks are supported on cradles bolted to the floor. The columns are spaced 2 feet 3 inches apart, provision being made for 14 columns in addition to other equipment. The arrangement of services for two adjacent column stations is shown in figure 1. A vertical steel rod, not shown in the diagram, is mounted 5 inches to the left of the vertical steel straps. This rod holds the clamps that support the receiver assembly and mercury leveling bulb for the back-pressure manometer and other accessory equipment, not shown in the figure.

COLUMN SET-UP

A typical set-up for a distillation column, showing connections to the appropriate services and illustrating the kind of equipment most generally used, is shown in figure 2. Glas-Col mantles furnish heat to the pots. The heat input (and hence the distillation rate) is controlled automatically by maintaining a constant pressure differential (back pressure) through the column. In addition to atmospheric pressure operation under nitrogen, the distillation may be conducted at any reduced pressure down to 15 mm. Hg by connecting the apparatus to one of the five manostated vacuum lines. Liquids boiling as low as -20° C. can be distilled by using the circulating coolant system. Non-condensable gases can be handled through a gas-collection system. Solids melting below 100° C. can be distilled through the use of steam lines and auxiliary electrical heating devices. Samples as small as 2 ml. and as large

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as 16 liters can be handled at one time with the equipment available. Each column is surrounded by a heating mantle controlled by a variable voltage transformer (not shown in any of the figures) that serves to protect the column and reduce the hazard to personnel from accidental breakage. In addition it allows the columns to operate much more adiabatically, especially in the case of high-boiling substances, by compensating for heat losses through the vacuum jackets. These jackets have been found very useful in reducing the starting-up period, for by presetting them overnight to the column temperature the length of time consumed in heating the packing is reduced considerably. The columns in use are listed in table 1. A spinning-band column (Piros Glover) 6 mm. in diameter is used for handling small samples at low pressures. A special vacuum-control unit for this micro column is used for pressures below 15 mm. down to about 1/2 mm. and is illustrated in figure 3. This figure also shows the services for the column. The reflux rate and reflux ratio are manually controlled in this column but are maintained constant by a constant voltage supply and rate of flow of condenser fluid.

TABLE 1

Station No.	Column type	Diameter, mm.	Height of packed sections, ft.
1.........	Podbielniak Heligrid	8	2
2.........	do.	25	2
3.........	do.	25	3
4.........	Oldershaw 40 plate	25	3
5.........	Special test	25	4
6.........	Podbielniak Heligrid	25	3-1/2
7.........	do.	25	9
8.........	do.	25	4
9.........	do.	13	6
10.........	Fenske 3/32-inch glass helices	22	3
11.........	Fenske 1/4-inch glass helices	25	9
12.........	Fenske 3/32-inch glass helices	25	4
13.........	Pertruded packing	25	6
14.........	Piros-Glover	50	6
		6	2

REFLUX RATE CONTROLS

The automatic controls for regulating the heat input are at the column station (see fig. 2) and are shown in detail in figure 4. When contact is made at the differential manometer, the voltage supplied by a high-voltage transformer is automatically replaced by that supplied by a lower-voltage transformer. This control can be by-passed by a switch, which allows the high-voltage transformer to remain in the circuit. Such an arrangement is necessary when it is desired to flood the column before use. A safety feature included in the system automatically switches to the low-voltage arrangement if there is any interruption of current to the rectifier.

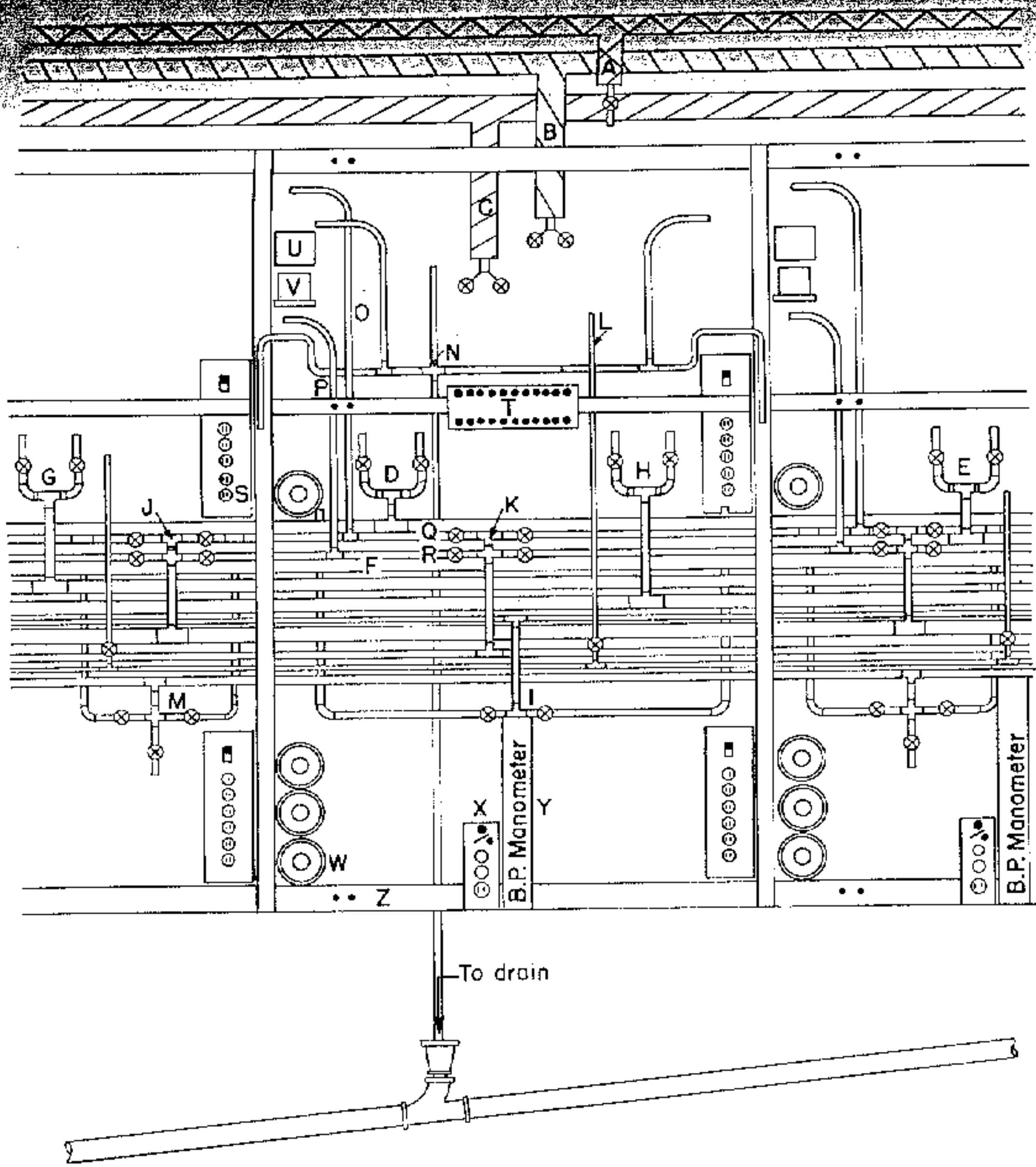


Figure 1.–Support frame and services for columns in distillation laboratory.

Legend for Figure 1

A. Steam line (magnesia insulated).
B. Circulating coolant feed line (rock cork insulated).
C. Circulating coolant return line (rock cork insulated).
D. No. 1 vacuum line.
E. No. 2 vacuum line.
F. No. 3 vacuum line.
G. No. 4 vacuum line.
H. No. 5 vacuum line.
I. Auxiliary vacuum line.
J. Cold water line (magnesia insulated).
K. Hot water line.
L. Nitrogen line.
M. Air line.
N. Cold water, hot water, air, and steam line vent to drain.
O. Cold water, hot water, and air to reflux condenser.
P. Cold water, hot water line to product cooler.
Q. Cold water, hot water, and air line to manifold line.
R. Cold water, hot water line to manifold line.
S. Six-gang power outlet boxes.
T. Timer outlet manifold.
U. Electromatic rectifier.
V. Mercury relay.
W. Variable voltage transformer.
X. Heat-input control panel.
Y. Back-pressure manometer.
Z. Thermocouple junction points.

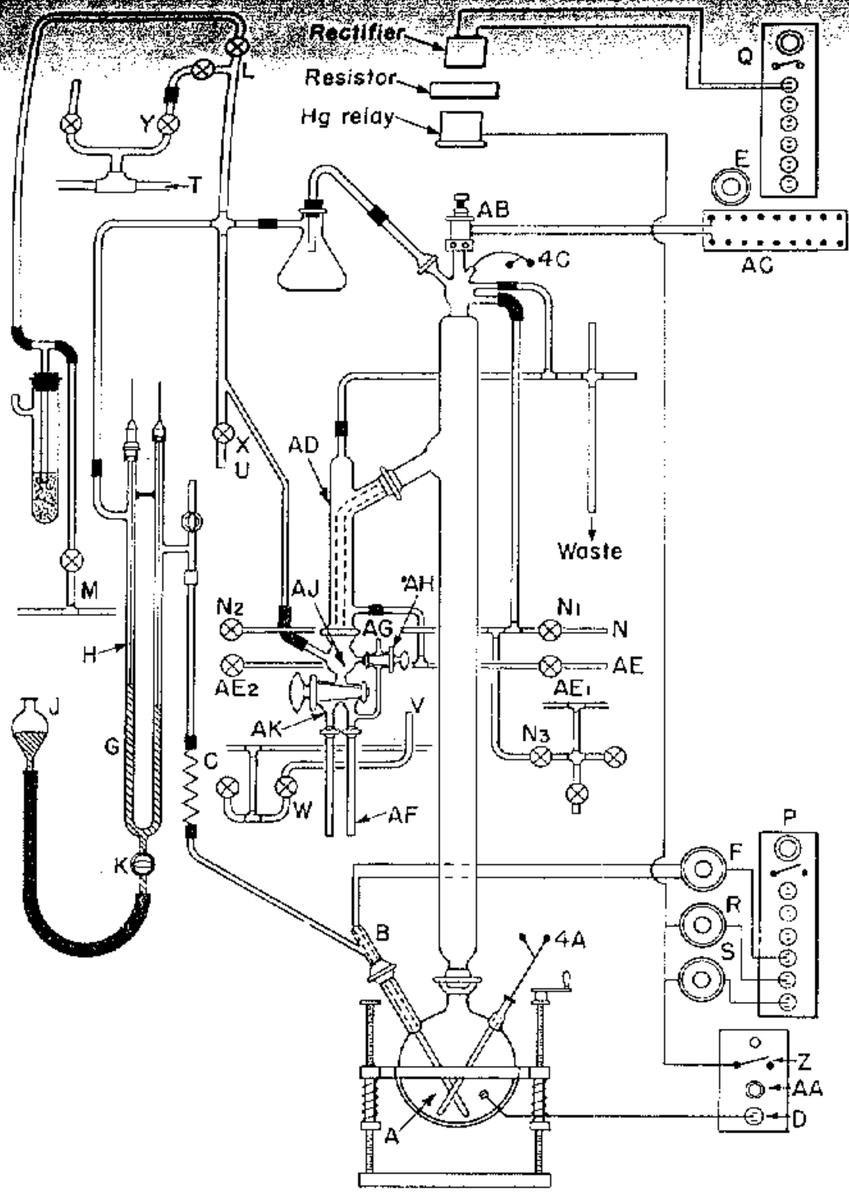


Figure 2.-Arrangement for services and control connections for laboratory still in distillation unit.

Legend for Figure 2

A. Bare wire heater coil.
B. Side-arm adapter.
C. Copper tubing, 1 mm.
D. Controlled power outlet.
E, F, R, S. Variable transformers.
4A. Thermocouple for pot temperature.
4C. Thermocouple for vapor temperature.
G. Back pressure manometer.
H. Contact wire.
J. Leveling bulb.
K. Stopcock.
M. Nitrogen line.
N. Service line for reflux condenser.
N_1. Cold water valve.
N_2. Hot water valve.
N_3. Compressed air valve.
P, Q. Six-gang electric outlets (110V AC).
T. Manostated vacuum line.
V. Auxiliary vacuum line.
W. Valve for auxiliary line.
X. Valve for connecting system to auxiliary vacuum line through connection U.
Y. Valve from vacuum line.
Z. Switch for releasing automatic back pressure controls.
AA. Indicator panel light.
AB. Solenoid for operating distillate valve.
AC. Junction box for timer circuits.
AD. Product cooler.
AE. Service line for product cooler.
AE_1. Cold water valve for product cooler.
AE_2. Hot water valve for product cooler.
AF. Receiver buret.
AG. Outlet for connection to auxiliary vacuum.
AH, AK. Three-way stopcock.
AJ. Receiver changer.

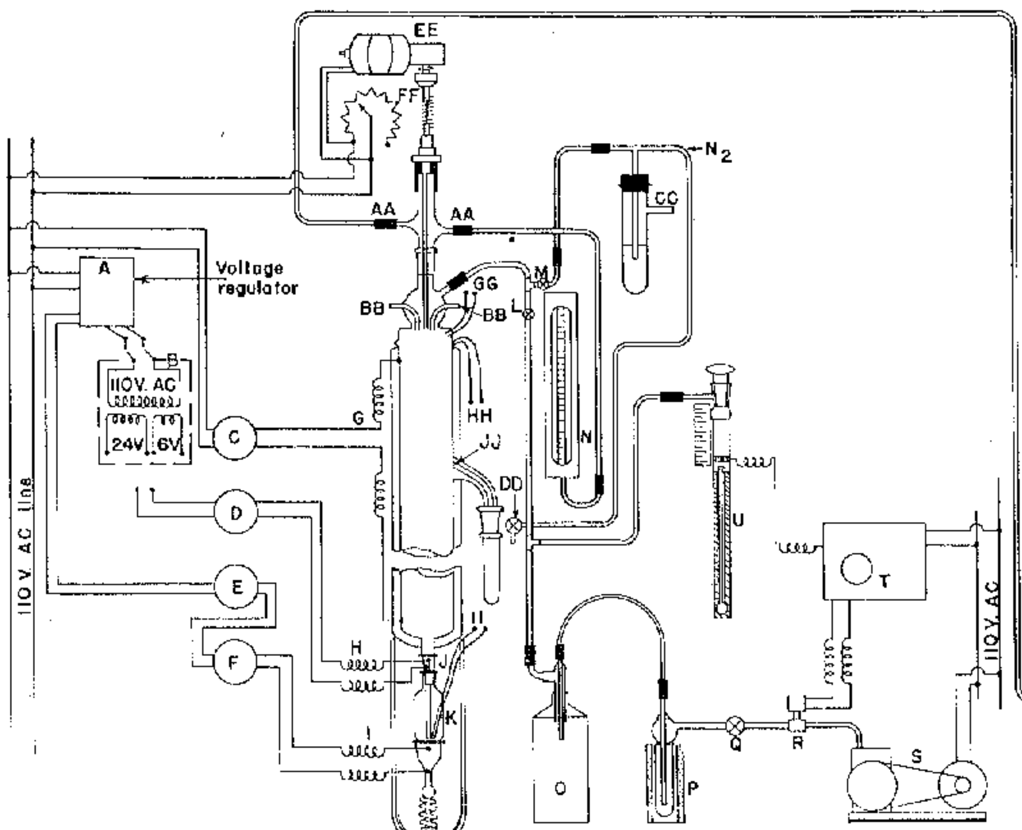


Figure 3.– Micro-still services diagram.

Legend for Figure 3

A. Constant voltage regulator (Sola Electric, primary 95-125 volts, secondary 115 volts).
B. Transformer.
C. Variable voltage transformer for jacket heater.
D. Variable voltage transformer for internal heater.
E,F. Variable voltage transformer for pot heat input.
G. Jacket heater load lines.
H. Internal heater lead lines.
I. Pot heater lead lines.
J. Internal heater adapter.
K. Flask thermowell.
L. Valve to vacuum line.
M. Valve to atmosphere or nitrogen line.
N. Rotameter.
O. Vacuum surge bottle (2 gallon).
P. Freezing trap.
Q. Needle valve.
R. Solenoid valve (Detroit Lubricator, Cat. No. 683-3).
S. Pressovac pump.
T. Thermocap relay (Niagara Electric).
U. Dubrovin mercury gage.
V. Air pressure regulator.
X. Hoke valve for regulator.
Y. Air vent valve.
Z. High pressure air line valve.
AA. Controlled air pressure through primary condenser.
BB. Water circulation through total condenser.
CC. Nitrogen bubbler.
DD. Nitrogen line.
EE. Stirring motor.
FF. Rheostat.
GG. Vapor temperature thermocouple junctions.
HH. Column temperature thermocouple junctions.
II. Pot temperature thermocouple junctions.
JJ. Commercially available Piros-Glover micro still supplied by H. S. Martin Co., Evanston, Ill., Cat. No. M-8441.

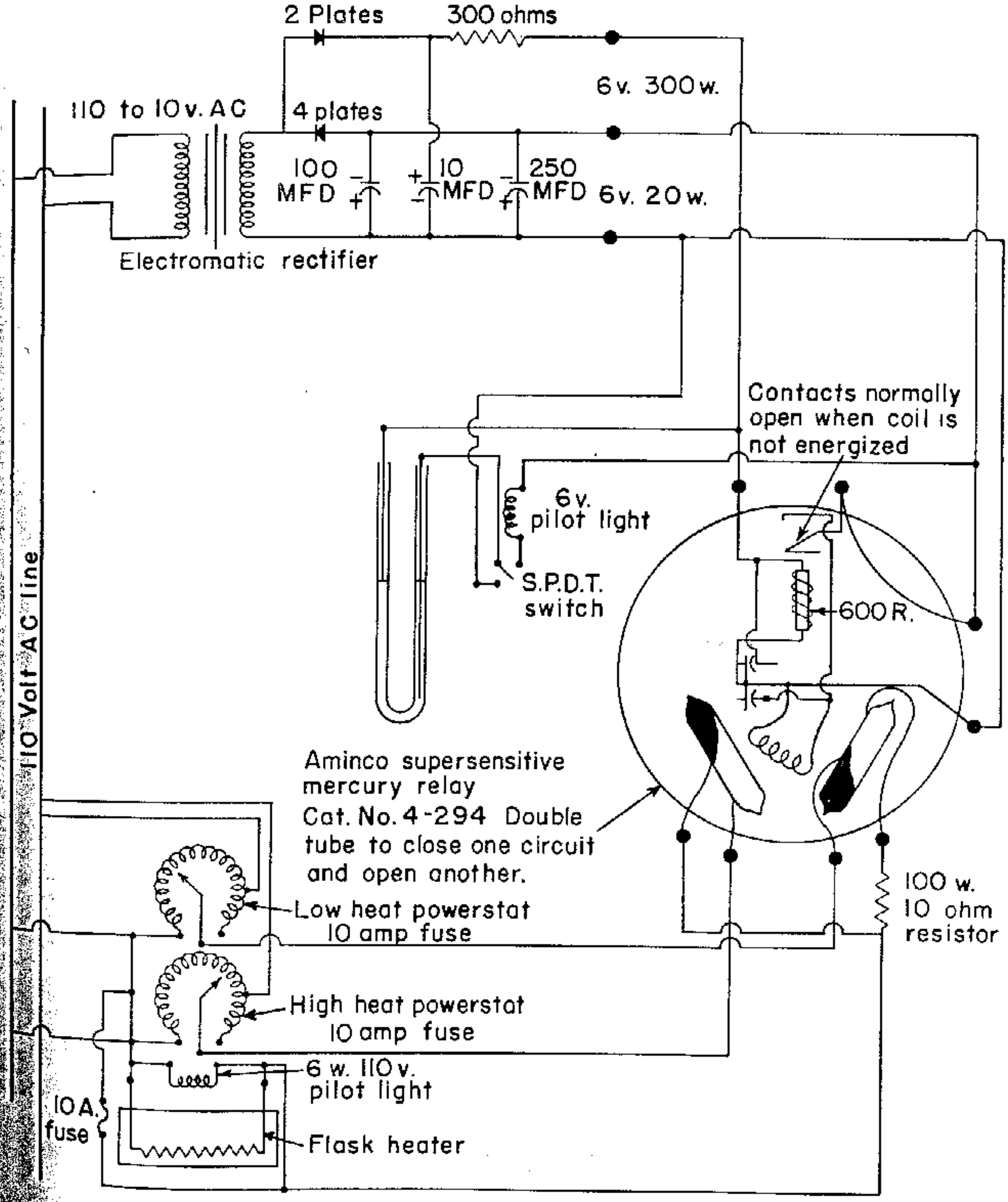


Figure 4.- Diagram for reflux rate control through differential pressure.

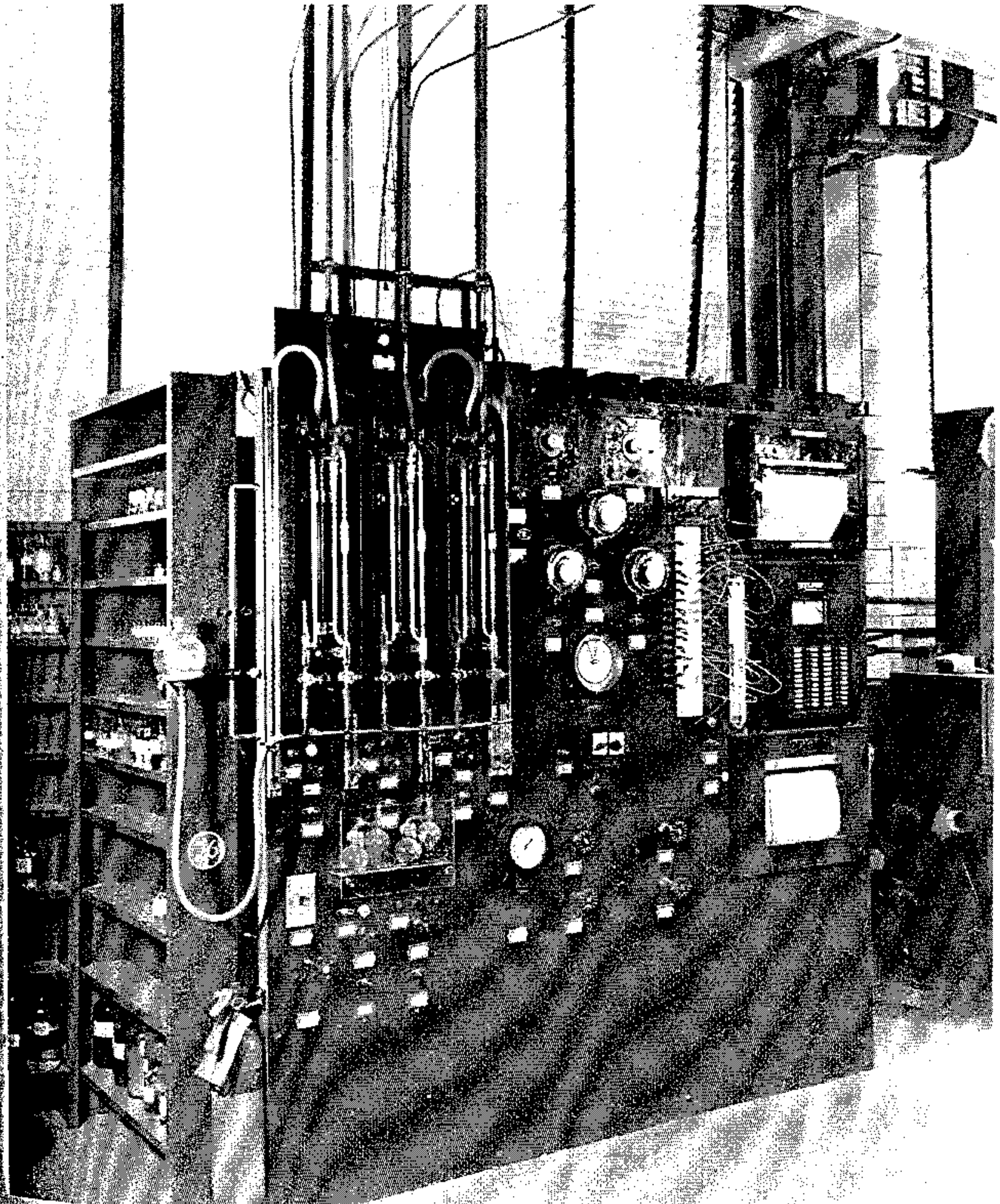


Figure 5. - Panel board.

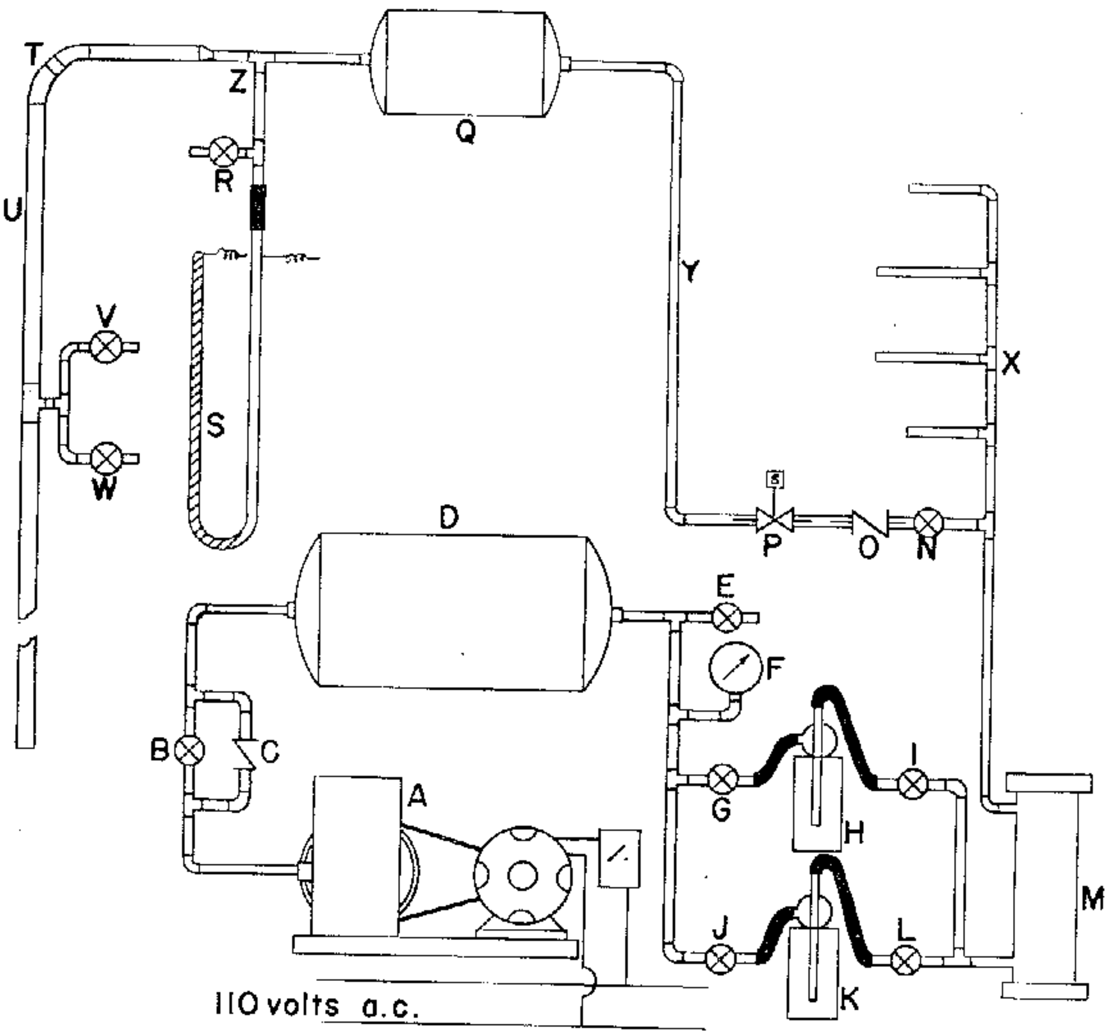


Figure 6.—Manostated reduced pressure system

A-Hypervac "20" pump
B-1/2" vacuum pump by-pass valve
C-1/2" vacuum pump check valve
D-20 gallon surge tank
E-Vacuum system vent
F-Pump side pressure gage
G-Pump side-1/2" cold trap valve No.1
H-Dry ice & acetone cold trap No.1
I-System side-1/2" cold trap valve No.1
J-Pump side-1/2" cold trap valve No.2
K-Dry ice & acetone cold trap No.2
L-System side-1/2" cold trap valve No.2
M-1/2 gallon silica-gel drying trap
N-1/4" throttle valve for vacuum line
O-1/4" check valve for vacuum line
P-3/8" solenoid 1/8" orifice
Q-5 & 10 gallon surge tanks for line
R-Air vent valve
S-Elec. control manostat & manometer
T-All bends in 1" line are 45°
U-1" copper streamline lines
V-1/2" streamline brass valves
W-3/8" brass tubulature
X-1/2" galvanized pipe
Y-3/8" copper tubing
Z-1/2" streamline copper lines

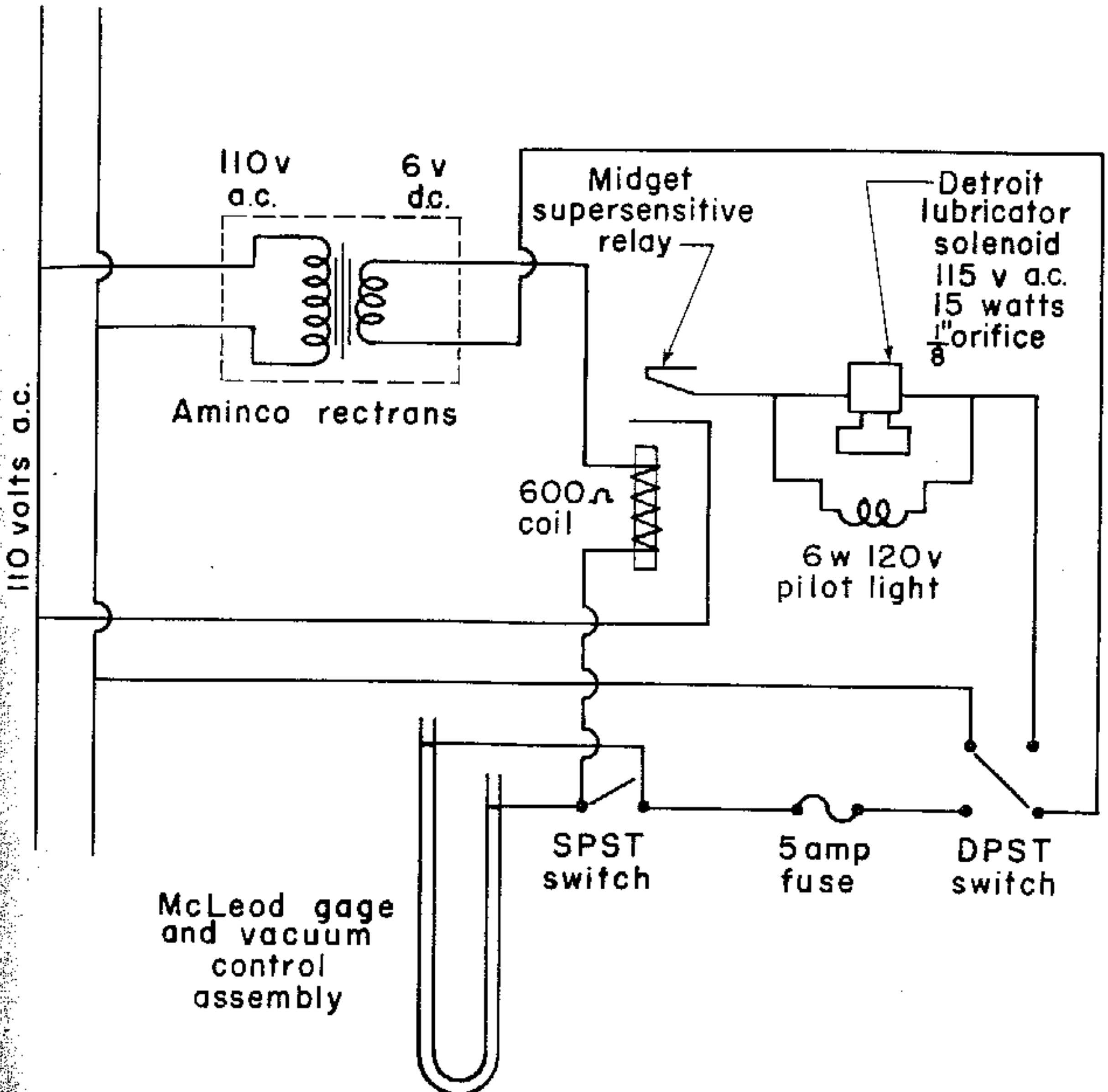


Figure 7.-Wiring diagram for mercury manostat control.

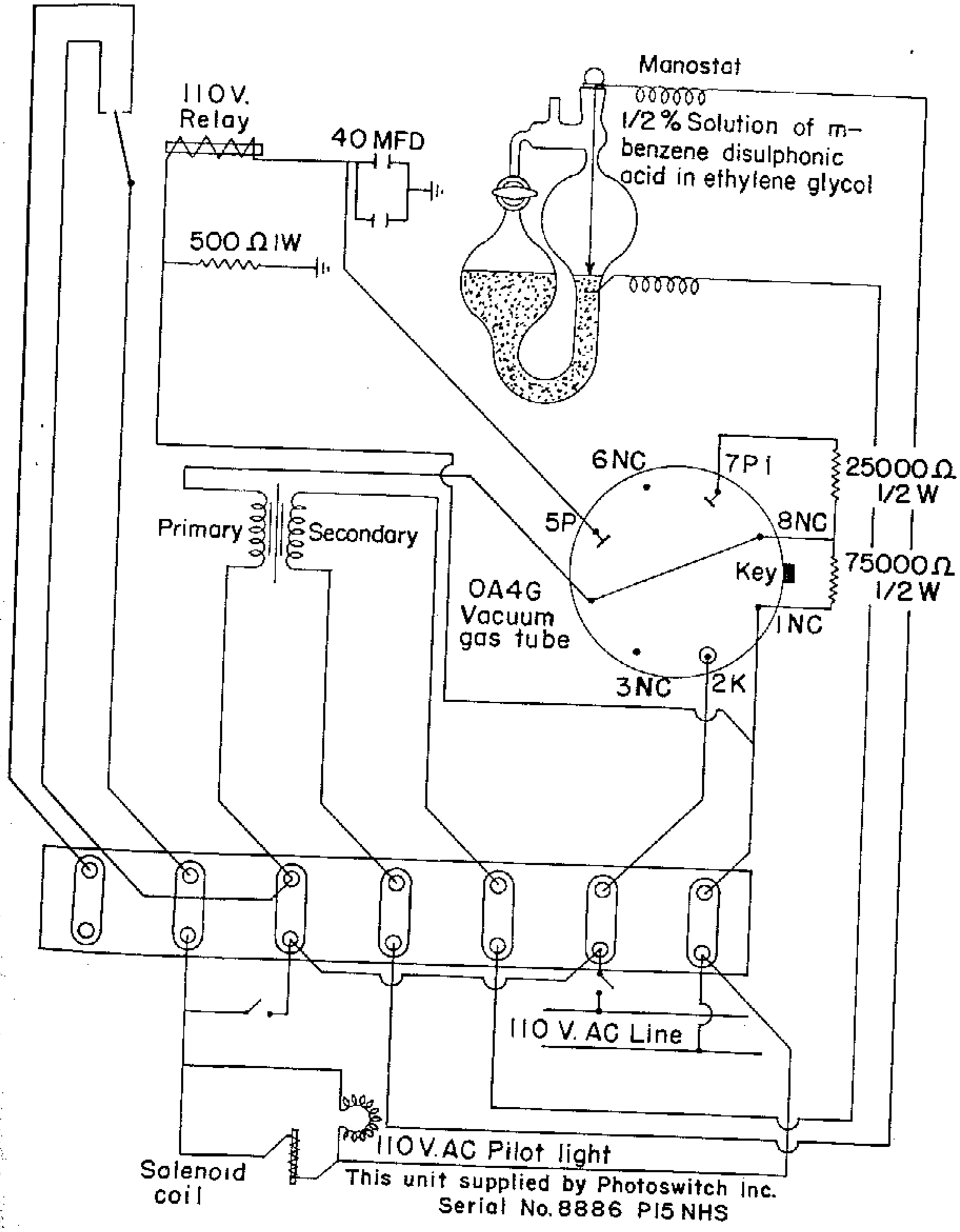
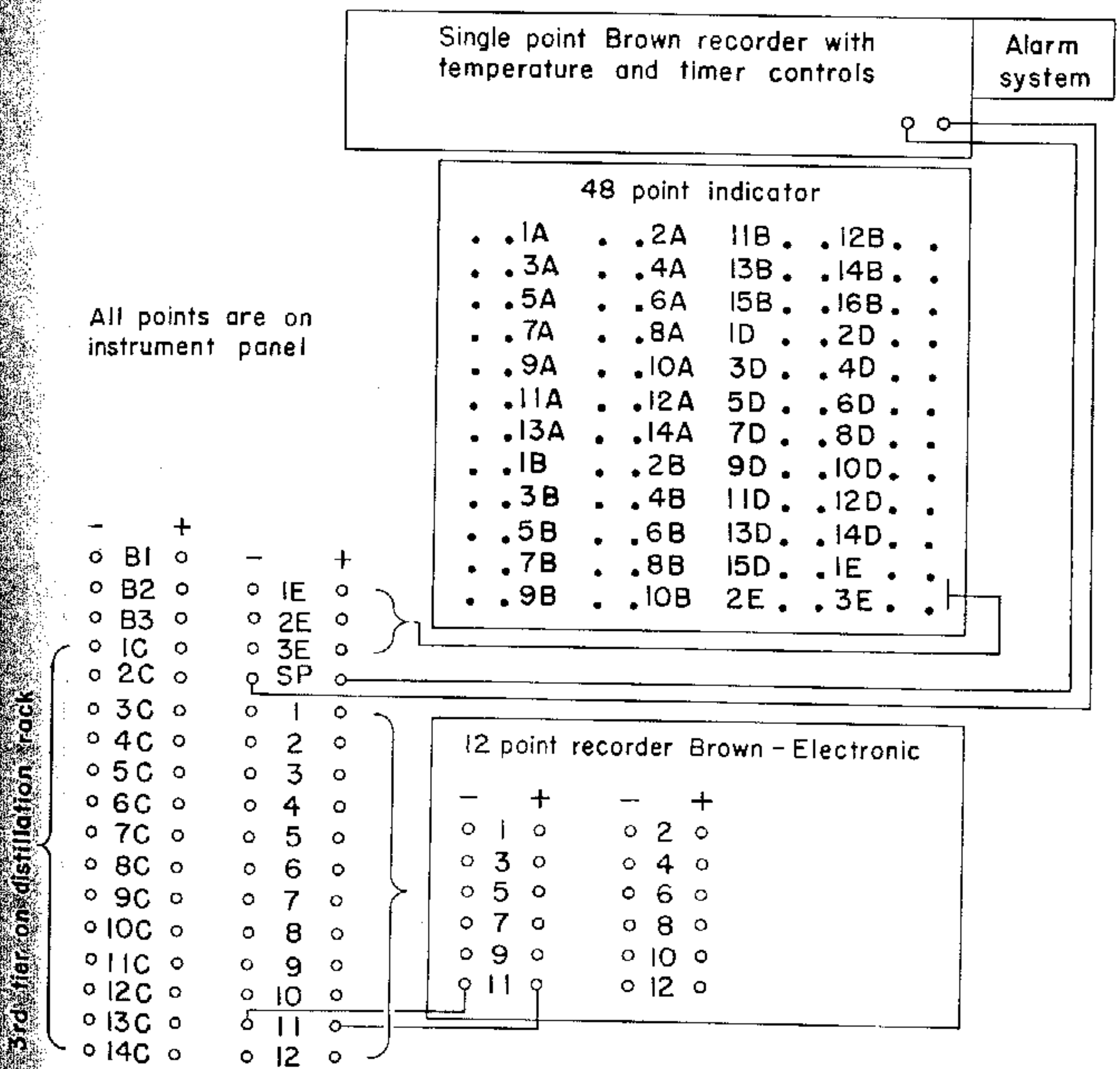


Figure 8.- Wiring diagram for ethylene glycol manostat control.



All thermo-couples are iron-constantan
Points 1A to 14A inclusive – Bottom tier of distillation rack
Points 1B to 16B inclusive – 2nd tier of distillation rack
Points 1D to 15D inclusive – 4th tier of distillation rack
Points 1E to 3E inclusive – Chemical bench through B1, 2, & 3
45 points 1A through 16B and 1D through 15D tied directing into 48 point Brown indicator
Points B1 through B3 and 1C through 14C can be jumpered to read in any one of the 3 instruments.

Figure 9.– Potentiometer thermocouple junctions for distillation laboratory

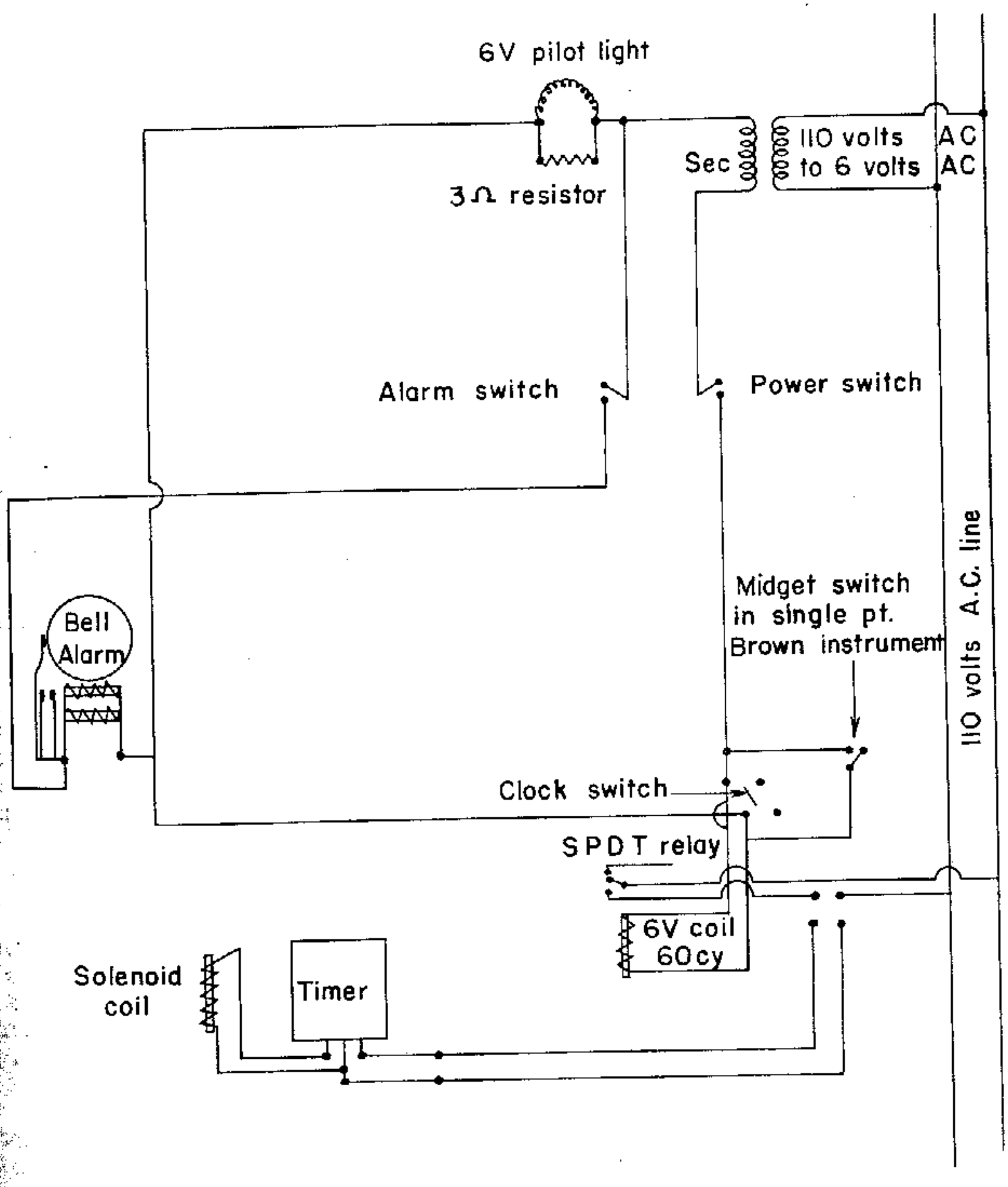


Figure 10.– Wiring diagram for alarm and control system for temperature and time.

PANEL BOARD

Vacuum controls, temperature recorders, and timers for operating the distillation valves are located on a panel board 6 feet 4 inches square. A photograph of this board is shown in figure 5.

VACUUM CONTROL UNIT

The source and distribution of the vacuum circuit is shown in figure 6. Two types of manostatic systems are in use. The first consists of a Podbielniak manostat with mercury as a contacting agent. This control circuit is shown in figure 7. The other consists of a vacuum tube circuit (fig. 8) in conjunction with a slightly conducting solution of benzene disulphonic acid in ethylene glycol. The glycol manostats are used generally for maintaining pressures constant above 90 mm.

Each of the five vacuum lines is connected through a throttled-down needle valve to the main vacuum line (X in fig. 6), which is always maintained at a pressure lower than any of the system lines. The solenoid valve is automatically opened whenever the pressure in the controlled system rises above its set point, and the valve is closed whenever the pressure drops to that value. The needle valve is throttled so that the change in pressure is gradual enough to avoid greatly overshooting the set point and yet is rapid enough to overcome the effects of leaks in the manostated system. This is accomplished by adjusting the valve so that the solenoid remains activated for a period of 3 to 30 seconds.

Two dry-ice traps connected in parallel are placed between the main vacuum pump and the distillation system to protect the pump from organic vapors that might otherwise condense in the pumping oil and interfere with the mechanical operation. This arrangement permits one trap to be shut down for cleaning without interfering with a vacuum distillation that may be in progress.

TEMPERATURE RECORDS

All temperatures are indicated or recorded by means of iron-constantan thermocouples. The thermocouples, which measure the vapor temperatures, are led along the next to the highest tier of the frame (4C in figs. 1 and 2) and can be connected by means of jacks to any of the points on a 0° - 300°C., 12-point, electronic recorder to three points on an electronic 48-point indicator (fig. 9) or to a single-point recorder and alarm system (fig. 10).

Temperatures of the pot are similarly transmitted through a thermocouple system on the bottom tier of the framework (4A, fig. 2) directly to the indicating instrument. Other thermocouple connections along the other two tiers tie in directly to the indicator and can be used for reading any other temperatures, such as jacket temperatures.

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TIMERS

All the columns, with the exception of the Piros-Glover micro column, are operated at total condensation with intermittent take-off. The valves that allow distillate to collect in the receivers are operated by any one of seven timers on the panel board. The wiring diagram for these is shown in figure 11. Solenoid lead wires can be plugged into the outlet boxes mounted near each column, each of which has nine outlets. Any or all of the columns can be operated by a single timer.

AUXILIARY VACUUM SYSTEM

A Pressovac pump is mounted behind the panel board. The switch and pilot light for its operation, together with a needle valve and manometer for regulating and reading the pressure, are mounted on the board. A 1/4-inch diameter copper seamless tubing connects to various column outlets. This auxiliary system (fig. 12) is used to evacuate receivers to column pressure before connecting them with the column and to evacuate columna to line pressure before connecting them with the manostated lines.

COLUMN OPERATION

All packed columns are first brought to reflux slowly and then flooded by increasing the reflux rate. The heat is then gradually reduced, so that reflux never ceases. Columns operating under vacuum are provided with small nichrome coils (A, B, fig. 2), which are immersed in the liquid near the bottom of the flask. These are heated by a variable transformer to a temperature just high enough to give continuous ebulliton without bumping. Effective operation cannot be obtained without constant ebullition.

Flasks sit on cradles in electric mantles and are held in place under spring loading. The long side arm prevents hot vapor from dissolving the grease at the joint.

Several views of the arrangement of columns in the laboratory are shown in figures 13 and 14.

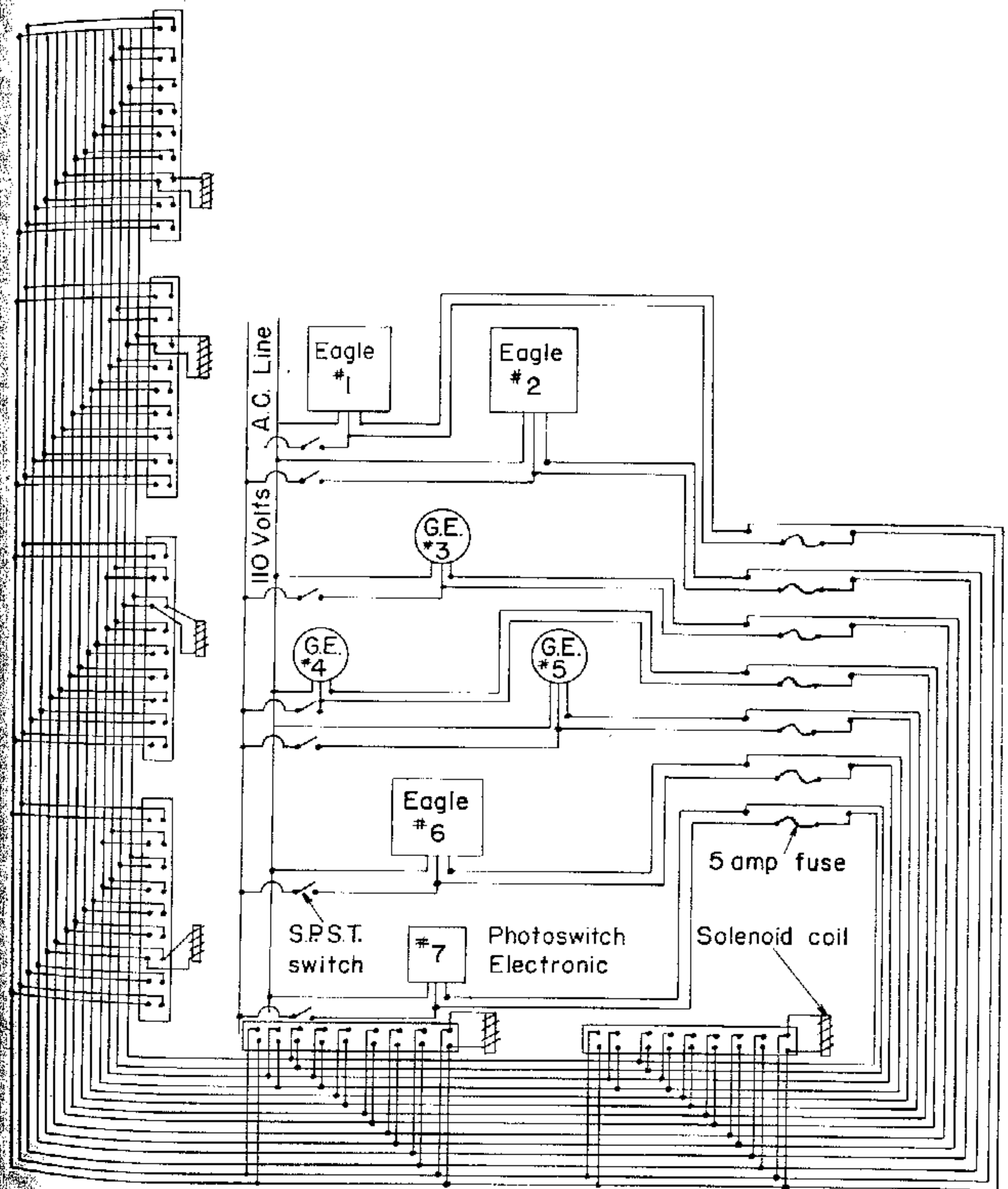

Figure 11. — Distillate valve control system

A– Cenco–Pressovac 4 pump
B– I gallon surge trap
C– Drying trap
D– Vent needle valve
E– U–type closed end manometer
F– Valve manifold
G– 3/8" copper lines

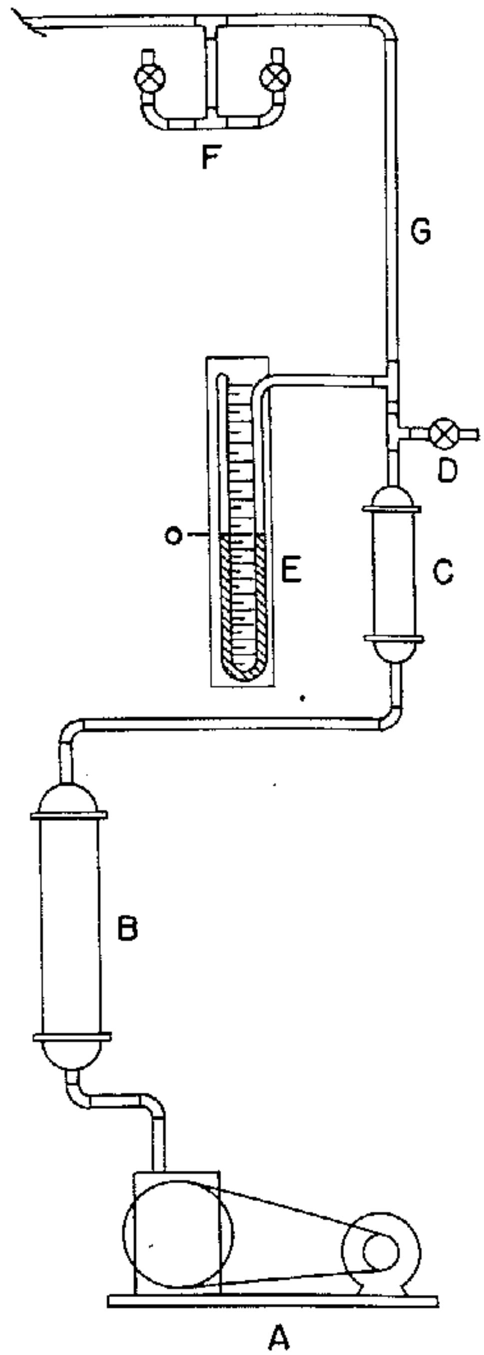

Figure 12.— Auxiliary vacuum system

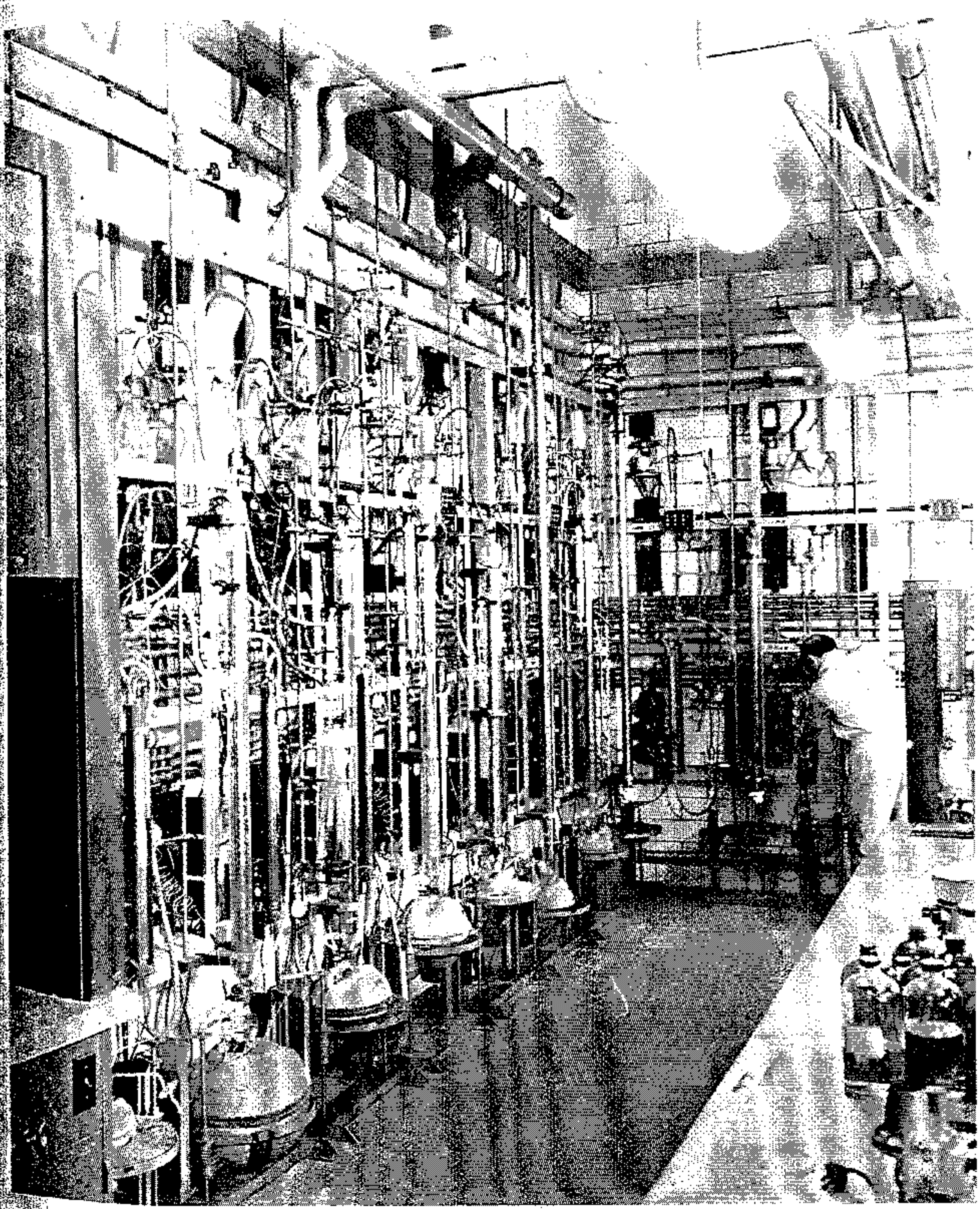

Figure 13. - Arrangement of columns in the laboratory.

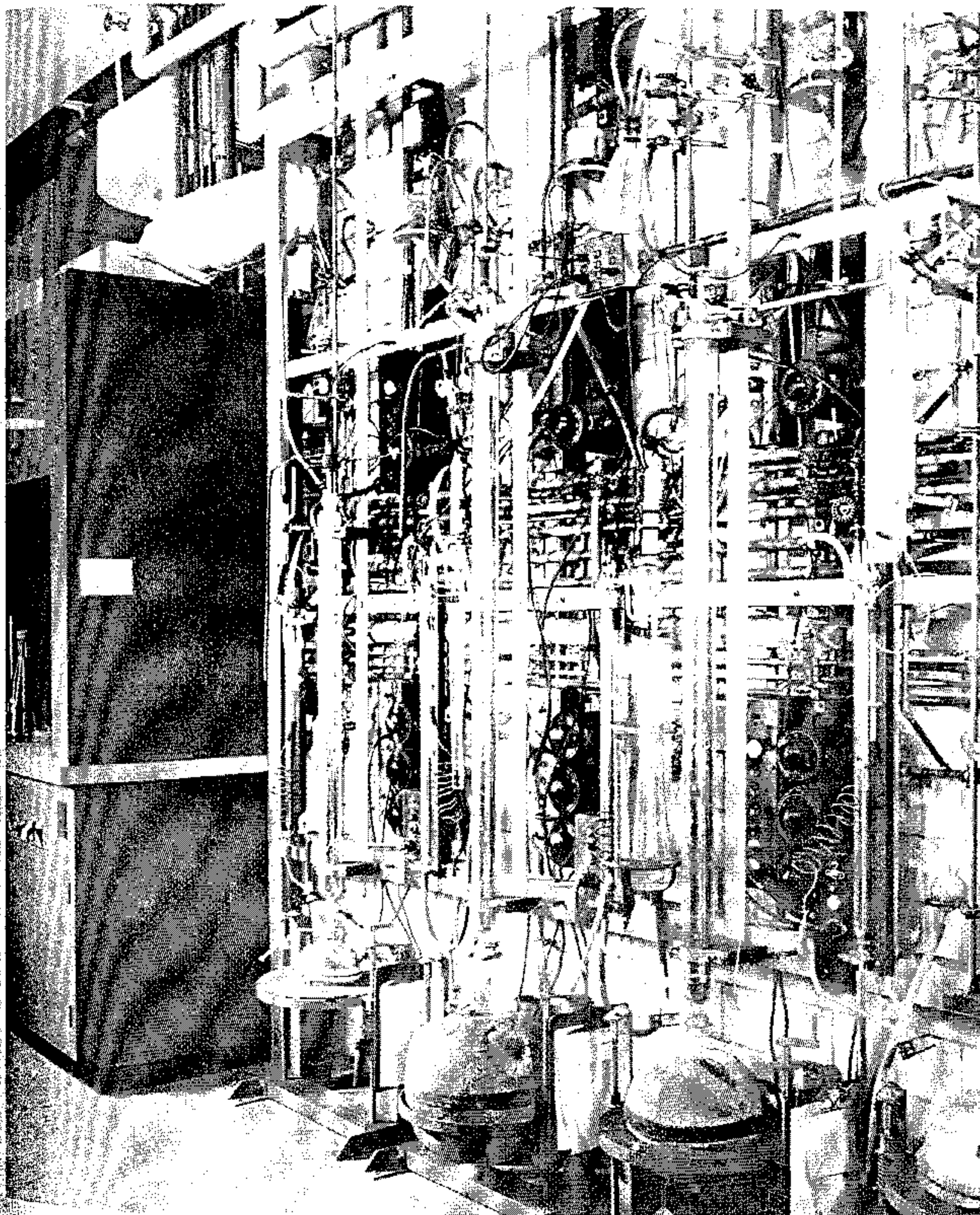

Figure 14. - Arrangement of columns in the laboratory.