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CHEMISTRY DIVISION

Section C-VII

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AN AUTOMATIC DISTILLATION COLUMN WITH CONTROLLED TAKE-OFF

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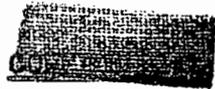
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Abstract

A laboratory column with an efficiency of 100 theoretical plates has been built using $3/64$ inch helices as the packing material.

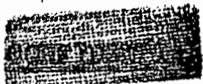


1. Introduction

This column was constructed to provide a source of pure organic liquids for radiation chemical studies. For convenience of operation, the column is designed to function automatically and requires attention only at the beginning of distillation. Its immediate use was to purify samples of solvents which are contemplated for the solvent-extraction process planned for the new pile reactor. Part of the program of the radiation chemistry section is to test the effects of high intensity radiation on such solvents. Experiments with hexone had earlier shown the importance of using pure compounds in such studies. It was found, for example, that the decomposition rate of commercial hexone exposed to radiation was much greater than that shown by the purified compound. It is generally preferable to study a pure compound since such studies provide unambiguous information of future application, and studies on one of lower purity furnish information valid under very limited conditions only.

2. Experimental

The packed section of the column (shown in Figure 1) consists of a 5 foot length of 14 mm Pyrex tubing. It is tightly packed throughout this length with 3/64 inch, single turn, stainless steel helices (obtained from Fenske at Penn State). The column packing is supported on a circular piece of 20 mesh, stainless steel gauze ~ 20 mm in diameter. A short length of 23 mm tubing was sealed onto the base of the 14 mm column and the gauze inserted. Its position is fixed since the diameter of the column



narrows again to 14 mm below this point. The helices were added by shaking them through a container with a screen wire bottom into a large funnel leading into the top of the column. The screen, about 10 mesh, forced the helices to separate into groups containing not more than 1-2 members and the column was thus uniformly filled. Tamping the helices during the filling operation aided in their interlocking to yield the maximum density of packing.

A length of 5 mm tubing which leads to a mercury manometer is sealed a little below the mesh support. This manometer serves two functions: (1) It allows observation of the flooding process when distillation is begun (repeated until the maximum pressure is obtained to insure complete wetting of the packing); (2) When the column is set for automatic operation an increase in the manometer (indicating an over-supply of heat to the pot) actuates a relay (Figure 2) which reduces the amount of current supplied to the pot heater. In other words, after the flooding operation has been completed, switch S_2 is closed; this operation connects the grid of the 12J5 to the adjustable contact of the mercury manometer. Any increase in pressure sufficient to raise the mercury to this contact will ground the grid and bias the tube sufficiently negatively that it will cease to conduct, thus releasing the plate circuit relay and throwing an additional resistance into the pot heat circuit. When the mercury level falls again, the process is reversed. In actual operation, sufficient current is supplied to the pot to keep the column on the point of flooding and the adjustable contact is set to counteract this at ~ 1.5 cm Hg pressure.



The glass is wrapped with 3/4 inch asbestos tape to bring its diameter up to ~ 1 inch and was inserted in a 5 foot length of 1 inch (I.D.) steel pipe. To provide for adiabatic conditions during distillation, the outside of the pipe was first insulated electrically with asbestos tape, then three independent heating sections (~ 100Ω each) were wound on it using 28 gage Chromel wire. The current supplied to these heating sections is adjusted by means of a Variac and variable resistances. Six thermocouples (Chromel P-Alumel, Class M matched wires) are placed along the column in such a way that one junction is in the center of each heater winding; directly opposite each, against the glass wall of the column, is another junction. During distillation, sufficient current is supplied to the heater windings so the temperature of the jacket at the three points is maintained equal to that shown by the corresponding thermocouples against the column wall. A two gang, multiple position switch connects a Rubicon potentiometer to any desired thermocouple.

The pipe containing the column was insulated with several more layers of asbestos tape and mounted rigidly to the laboratory wall by two extension pipe clamps. The entire length of the column was then fitted with standard magnesia steam pipe insulation, bringing the overall diameter up to ~ 3 inches. This type of insulation functions so well that the outside is barely warm to the touch when distilling a compound boiling at 100°C. Two lengths of 1/2 inch pipe, each 7 feet long, were mounted vertically on either side of the column and fixed to rods extending 6 inches from the wall. These protect the column from inadvertent physical shock and also

support various rings and clamps necessary to hold the pot, receiver, etc.

Figure 1-B shows details of the still head which was sealed onto the top of the column after addition of the helices. The take-off is arranged so the column is operated on total reflux except when a small sample of the distillate is taken at periodic intervals. This is achieved by sealing an iron nail (\sim 3 cm long) in a 5 mm glass tube which has a small glass loop at the top. This pendulum swings freely on a glass hook forming the drip-tip of the condensate return and is positioned so that all the condensate passes over it. In its normal position, the pendulum passes the condensate back down the column. By means of a Flexopulse timer (Eagle Signal Corp.) which periodically energizes an electro-magnet located close to the glass, the pendulum is forced to swing over to the exit tube and deliver a drop of distillate. The exit tube protrudes through the still head wall sufficiently that any condensate caught by the wall cannot enter it, but must flow around the tube. The reflux ratio can be controlled by setting the timer from 0 to 120 seconds in either the ON or OFF position.

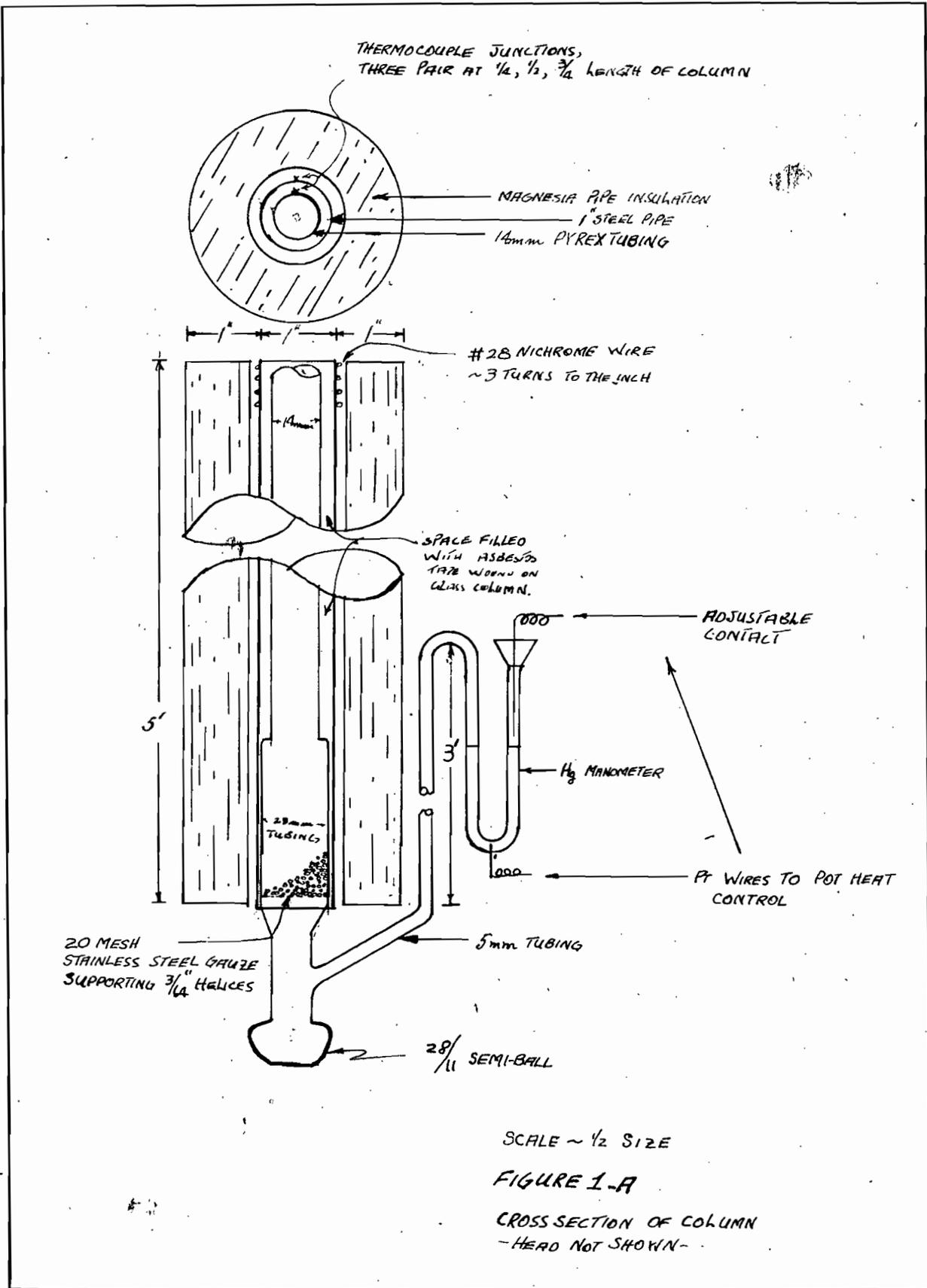
The still pot is connected to the main column with a 23/11 semi-ball joint. This joint is lubricated with a stop-cock grease made by mixing bentonite with glycerine to the proper consistency. Two removable blocks of magnesia insulate the joint and flask neck and prevent condensation of the vapor at this point. The temperature of the distillate is estimated to 0.05° C by means of an Anschutz thermometer suspended centrally in the still head to a depth sufficient for coverage by the vapors. Barometric readings are taken with a standard wall barometer and appropriate

corrections are applied. When the column is operating under a head equivalent to 1.5 cm Hg. the through-put and hold-up are 3-4 cc/min and 18 cc, respectively.

The number of theoretical plates and H.E.T.P. were determined by the method described in Bur. Standards J. Res. 22, 523 (1939). References are also given in the paper to the original work of several authors. The n-heptane and methyl-cyclohexane used in this determination were each fractionated singly and samples were checked carefully against the known boiling points and refractive indices. A mixture of the two reagents (containing \sim 0.25 mole fraction of n-heptane) was placed in the still pot and the column flooded three times. After operating on total reflux overnight, small samples were withdrawn simultaneously from the pot and still head. Composition of the pot and still head liquids was determined by index of refraction and the data of Bromily and Quiggle, Ind. Eng. Chem. 25, 1136 (1933). Two determinations, made with one day total reflux intervening, yielded 99 and 104 Theoretical Plates and an average H.E.T.P. of 1.5 cm.

Acknowledgments

The column was designed principally from a similar one constructed in Chicago by Dr. J. V. Flanagan and differs only in some circuit details and the manner of supporting the helices. The control circuit and panel mount were built by K. W. Forrestal of the 717-B instrument section. Photographs of the final column assembly were made with the aid of Q. V. Larson.



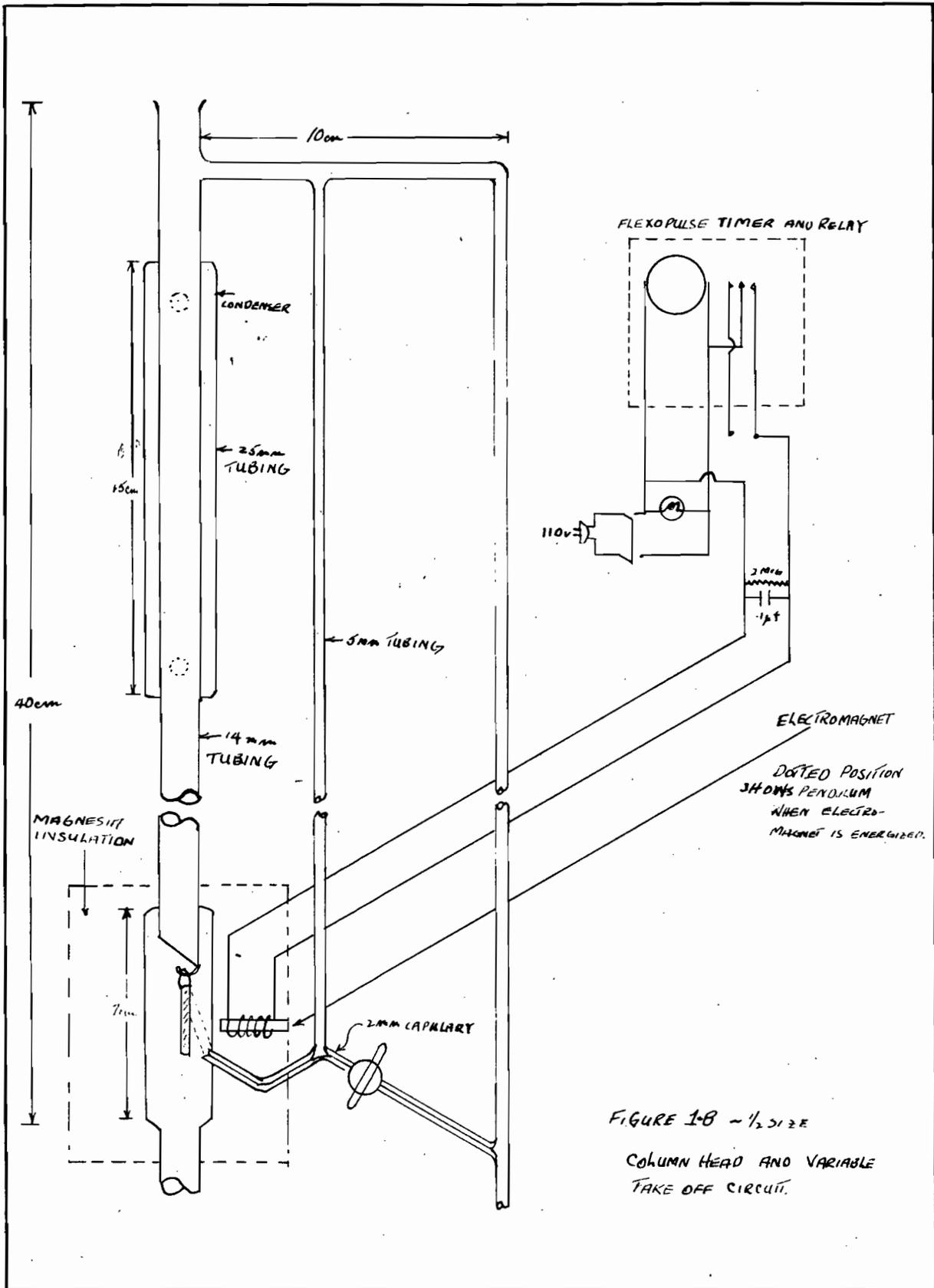
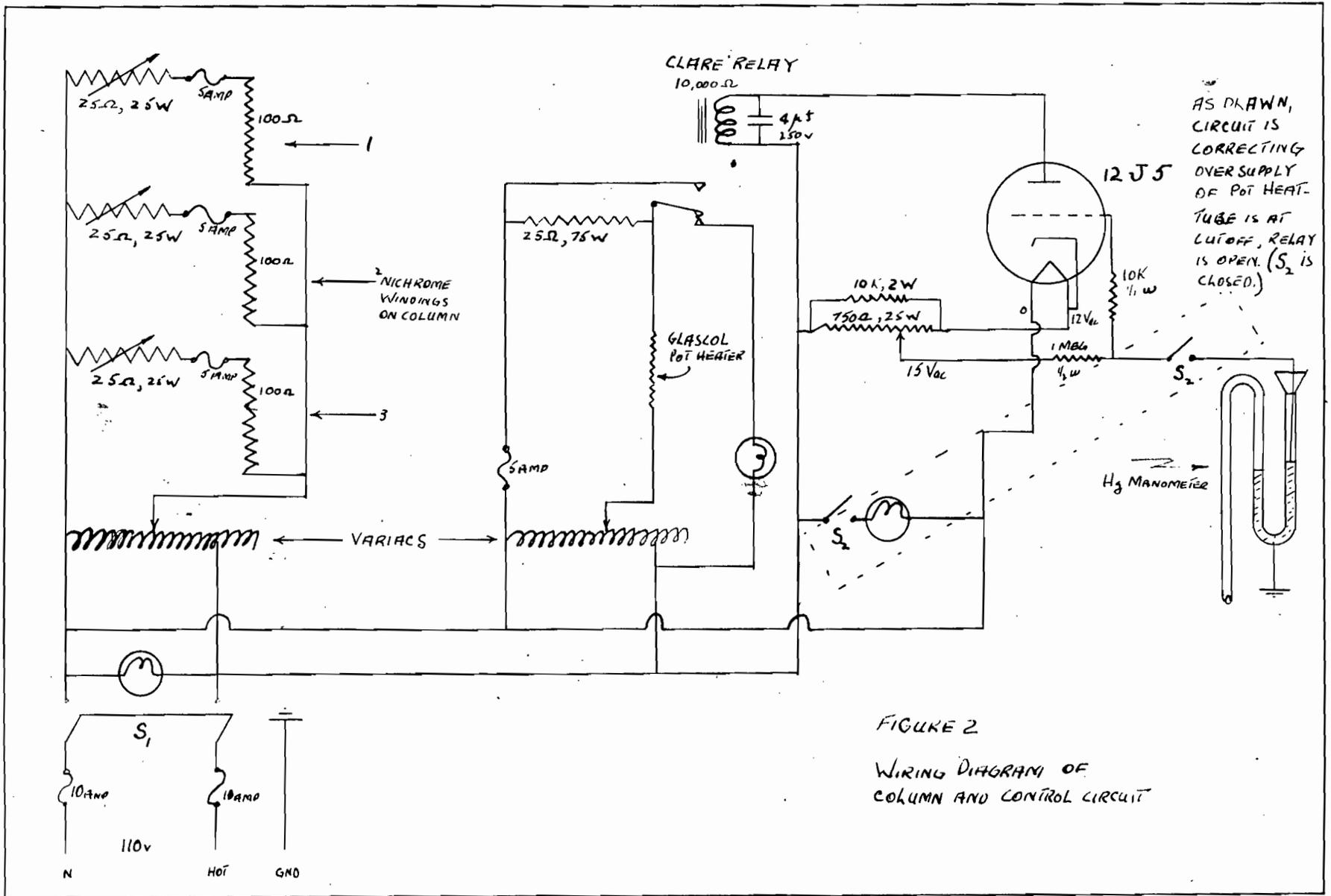


FIGURE 1-B ~ 1/2 SIZE
COLUMN HEAD AND VARIABLE TAKE OFF CIRCUIT.



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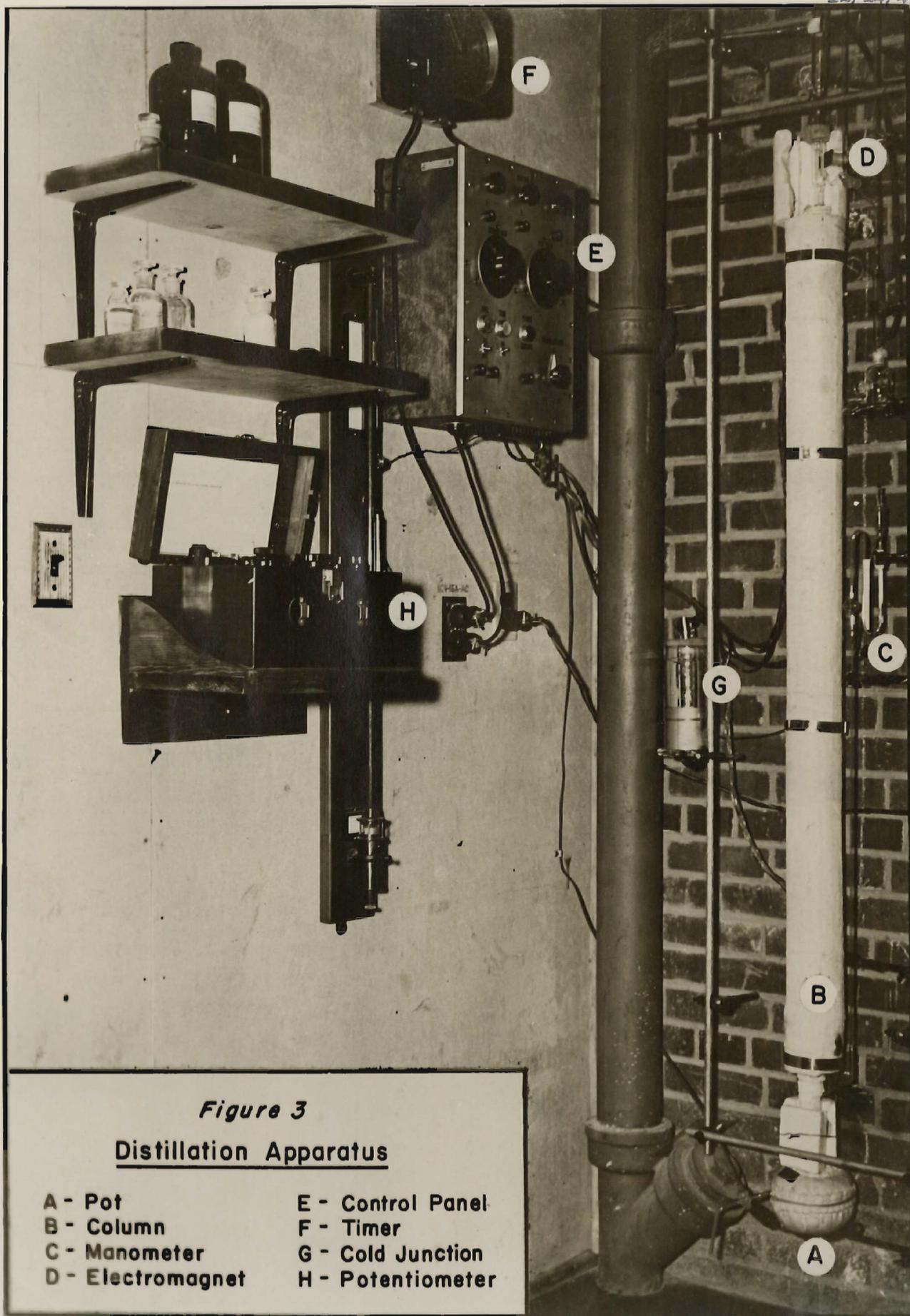


Figure 3

Distillation Apparatus

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|-------------------|-------------------|
| A - Pot | E - Control Panel |
| B - Column | F - Timer |
| C - Manometer | G - Cold Junction |
| D - Electromagnet | H - Potentiometer |



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