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A STUDY OF

THE PILOT PLANT RECOVERY OF FORMEX SOLVENTS

FROM WIRE BAKING OVENS

A dissertation presented to the Department of Chemistry of Union College in partial fulfillment of the requirements for the degree of Master of Science in Chemistry by

Name Gerald W. Young

Approved by ____________________

May 17, 1948.

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I INTRODUCTION

Magnet wire is used in the manufacture of electric motors, generators, transformers, magnetic coils and many other types of electrical equipment. This insulated copper wire is made by passing bare copper wire from a supply spool through a bath of varnish into a baking oven and to a pickup spool. The bare copper wire is continuously coated with varnish. In the baking oven the solvent is evaporated leaving a tough insulating film of Formex resin on the copper wire.

For many years over a million dollars worth of Formex solvent per year has been allowed to pass up the stacks of several hundred tall vertical baking ovens. The commercial recovery of vapors is of relatively recent origin. The increased cost of the solvents themselves as well as their increasing scarcity in many cases has only in the last few decades forced industry to attempt their recovery.

II PURPOSE OF INVESTIGATION

The purpose of the present investigation was to recommend the most practical solvent recovery equipment for recovering Formex varnish solvent.

The investigation was carried out in three steps:

1. Theoretical study of recovery methods.
2. Operation of a glass laboratory model.
3. Operation of a pilot plant for one oven.

III THEORETICAL CONSIDERATIONS

In general there are four methods of solvent recovery; namely, cooling, liquid adsorption, solid adsorption, and electrostatic precipitation.

A - Cooling Methods

The cooling methods (1) consist of cooling the mixture of solvent vapor laden air to a temperature below its solvent dew point causing the solvent to condense and separate as a liquid from the air. Only water coolers are mentioned here because they are the most common. The cooling method may be subdivided into two general classes:

1. Indirect cooling takes place when the solvent vapor and cooling water do not touch each other. For example, the vapor laden air may pass between banks of pipes containing cooling water. Refrigeration (2) and compression in conjunction with indirect cooling increase the solvent recovery yields. For concentrations of solvent vapor in air below the explosive limit, the increased yield will usually not pay for the power required to produce the refrigeration or drive the compressor. The explosive limit for Formex solvent, 40% cresylic acid, 60% high flash naphtha, is 1 to 8% by volume vapor in air.

2. Direct cooling occurs when the solvent vapor is brought into intimate contact with the cooling water. The indirect tube cooler never requires a distillation to separate liquid solvent from cooling water. The direct contact cooler usually requires distillation for separation of a soluble solvent from cooling water. An insoluble solvent is simply decanted from water. There are four types of direct cooling scrubbers:

(a) - The packed column water cooler consists essentially of a long vertical tube filled with packing. The packing (3) may be crushed stone, pebbles, hollow cylinders called Raschig rings, or other shapes. Intimate contact is provided between cooling water flowing down over packing and vapor laden air rising up the column.

(b) - A bubble cap column(4) borrowed from distillation practice can be used to insure good condensation of solvent vapor in cooling liquid. Air to be cooled enters at the bottom, passing up through the tower and bubbling through the liquid on each plate. The water is fed at the top and overflows from one bubble cap plate to the one below through vertical overflow pipes connecting each plate with the next immediately beneath it. The air enters the caps through risers from below, and depressing the liquid level inside the caps escapes into the liquid on the plate through notches cut in the periphery of each cap.

(c) - A bubbler column is similar to a single large bubble cap plate. The air with solvent vapor is led to the bottom of a vertical water tank. The air bubbles rise from the bottom to the top of tank through the water.

(d) - Spray chambers (5) are constructed in such a manner that the air holding vaporized solvent passes through the various water sprays.

B - Liquid Absorption

The liquid absorption method(6) consists of dissolving the solvent vapor from air into a less volatile liquid absorbent and subsequently separating the liquid solvent from the liquid absorbent. A packed column is often employed to cause intimate contact between the vapor laden air rising up through the wet packing and the absorbing liquid flowing down over the packing. A bubble cap column may be effectively used to approach a state of absorption equilibrium by allowing the air with solvent to be in contact with liquid absorbent for a longer time. Separation of the liquid solvent from the less volatile liquid absorbent is usually effected by fractional distillation. The addition of a third liquid to the one phase liquid consisting of solvent in absorbent liquid may cause the formation of two immiscible layers making decantation of the solvent possible. An inverse temperature solubility relationship allowing decantation again or other means would then be necessary to separate the added liquid from the absorbent liquid in order to re-use the absorbent again. Purification of the absorbent liquid eventually is necessary.

C - Solid Adsorption

The third method makes use of the property of a solid adsorbent(7) such as activated charcoal, to adsorb on its surface the solvent vapor. The solvent is later removed by high temperature vaporization(8) and condensed. Solvent recovery systems based on the principle of adsorption on activated charcoal have been successfully used in many industries(9) for the recovery of low boiling solvents.

D - Electrostatic Precipitation

The fourth method of recovery gives a charge to the low vapor pressure solvent entrained in the air by passing the vapor near a negatively charged wire or sheet and the charged solvent particles then deposit and run down on an oppositely charged sheet into a collection tank. Electrostatic precipitators or Cottrell precipitators are not generally associated with successful industrial recovery of low or medium boiling solvents. However, many such installations readily precipitate acid mists, oil droplets and dust particles.

E - Advantages of a Packed Column

A packed column water cooler was selected for recovering Formex solvents since a good recovery yield can be realized for these low vapor pressure Formex solvents at river water temperatures. By careful selection of new solvents having less vapor pressure than present solvents, greater recovery yields can be obtained.

A packed column water scrubber is simple and inexpensive to construct, operate and maintain. Decantation of insoluble solvents from water saves heat energy required for distillation from solid or liquid adsorbents.
A packed column provides greater cooling per pound of water and also per unit surface than indirect tube coolers.

Resinous deposits cause less trouble in a packed column as indicated in table below:

Equipment	Effect of Tar Formation
Packed Column	- cuts down cross sectional area of column slightly. Does not decrease heat transfer.
Tube Cooler	- Cuts down heat transfer coefficient greatly decreasing cooling capacity.
Activated Carbon	- slowly decreases capacity of adsorbent to hold solvent.
Liquid Oil Adsorbent	- deposits sludge in oil requiring distillation purification of oil in nitrogen under reduced pressure.
Electrostatic Precipitation	- causes current leakage on insulators finally resulting in arc-over which may in turn explode solvent vapor in air.

Furthermore, less resin decomposition products or tars will deposit on a packing which is constantly washed with water. Solvent pumped in the place of water will dissolve resin on Raschig ring packing. The solvent can be re-used for several clean-up cycles. Then the solvent can be evaporated from resins and the condensed solvent re-used for several more clean-up cycles.

IV EXPERIMENTAL LABORATORY PACKED COLUMN

The first laboratory packed column water scrubber to recover Formex enamel solvent effectively is shown in figure 1. Wherever practical all the parts of the system were made adjustable in order to study each variable concerned in solvent recovery.

A - Procedure

The glass column is packed with Raschig rings and assembled in place. The Formex enamel solvent (40% cresylic acid, 60% high flash naptha) is pumped from a burette in a thin stream into a flask on a hot plate. The solvent rate is adjusted by varying the speed of the pump motor. Air is also passed into flask and the rate of air flow is adjusted by a valve in the air line near the air meter. By varying the solvent in relation to the air flow, the concentration in any wire enamel oven can be duplicated for investigation. The water pump is adjusted to recirculate cooling water at a desired rate. The water travels down the packing into the decanter, through the coiled water cooler back to the top of the column and down the packing again. After running for several hours the water, air and solvent are shut off. All the water is drained out of the packing, pipes and pump. The solvent is decanted from the top of the water.

PACKED COLUMN WATER SCRUBBER FOR SOLVENT RECOVERY

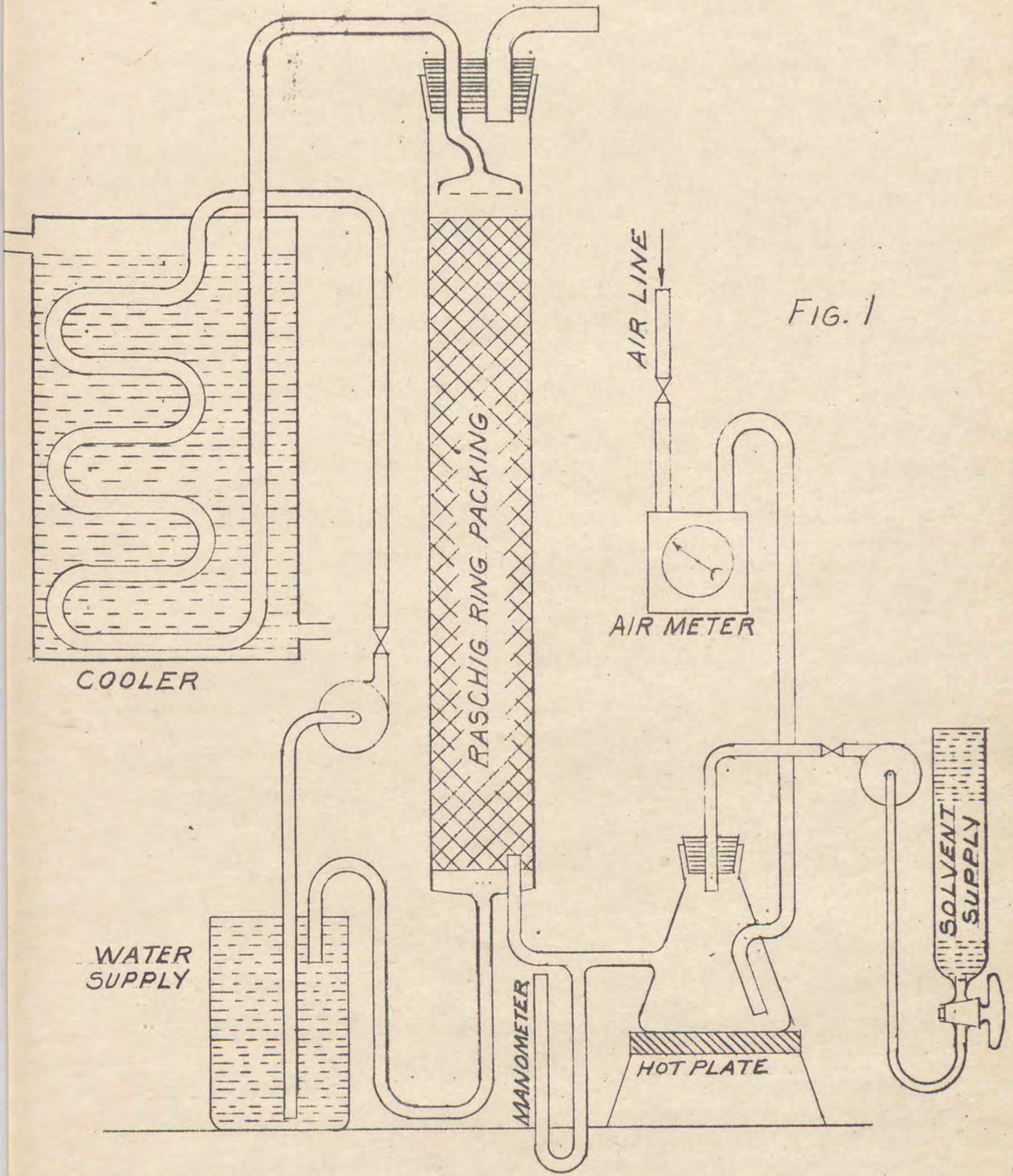


FIG. 1

B - Results

1. Solvent Recovery Yields

The curves in graph I indicate that if the air flow through an oven is properly controlled to produce a concentration of 0.8% by volume of solvent vapor in air, the following recovery efficiencies can be maintained:

(a) - By water scrubbing 62% of all the Formex solvent evaporated during the wire baking operation can be recovered. This represents a saving of 72% of the cost of solvent because the more expensive component, cresylic acid, is more readily condensible than the high flash naptha.

(b) - At the same concentration, 0.8% by volume, practically all (96.5%) of the solvent evaporated can be recovered by connecting an activated charcoal system in series to back up the water scrubber system.

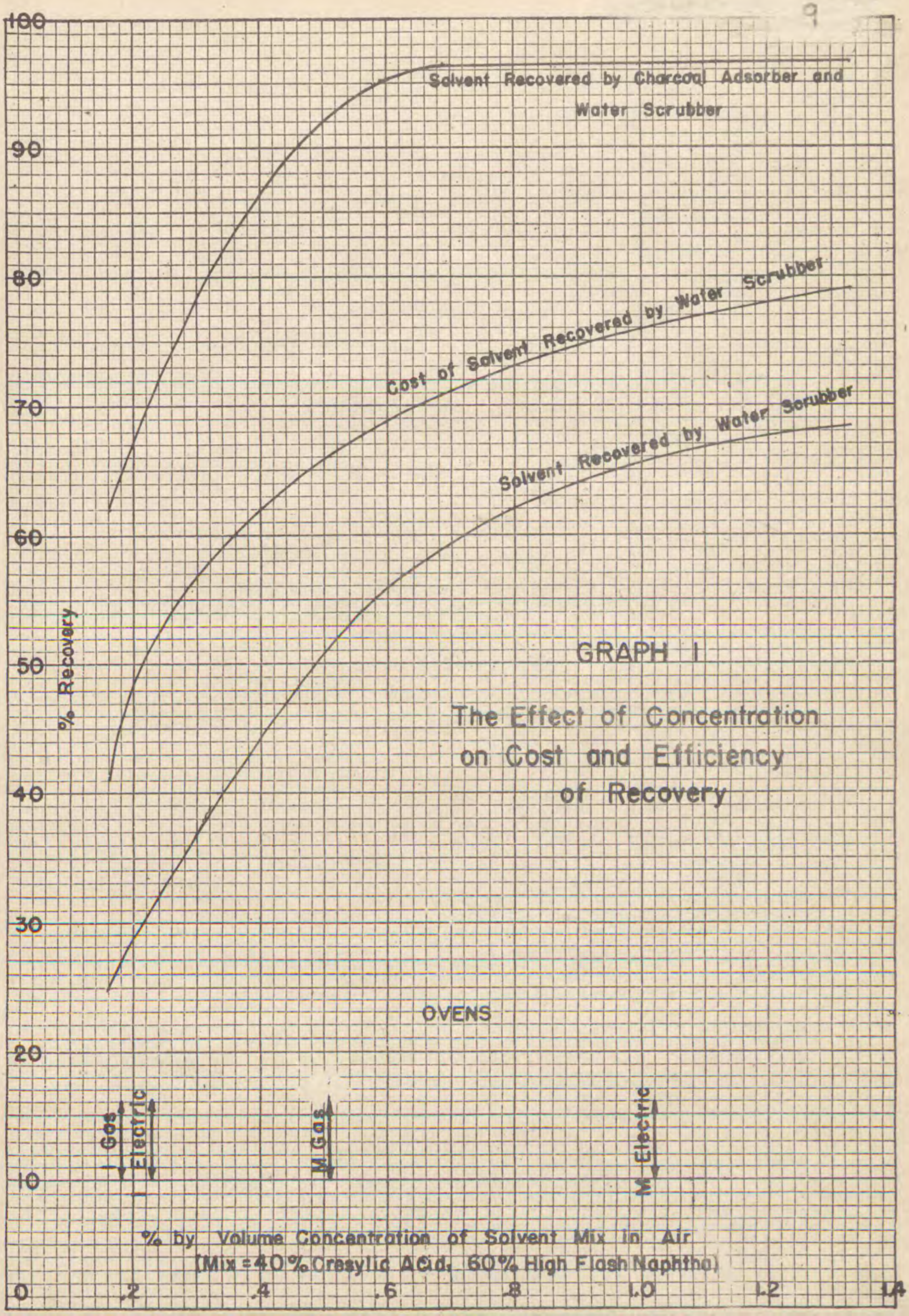
The runs plotted in graph 1 indicate that for a given wire enamel oven as the flow of air through the oven is decreased to increase the concentration of solvent vapor in the air, more solvent can be recovered by water scrubbing.

Graph 2 shows solvent recovery efficiencies broken down into the two main solvent components of Formex solvent.

2. Effect of Variables on the Design of a Packed Column

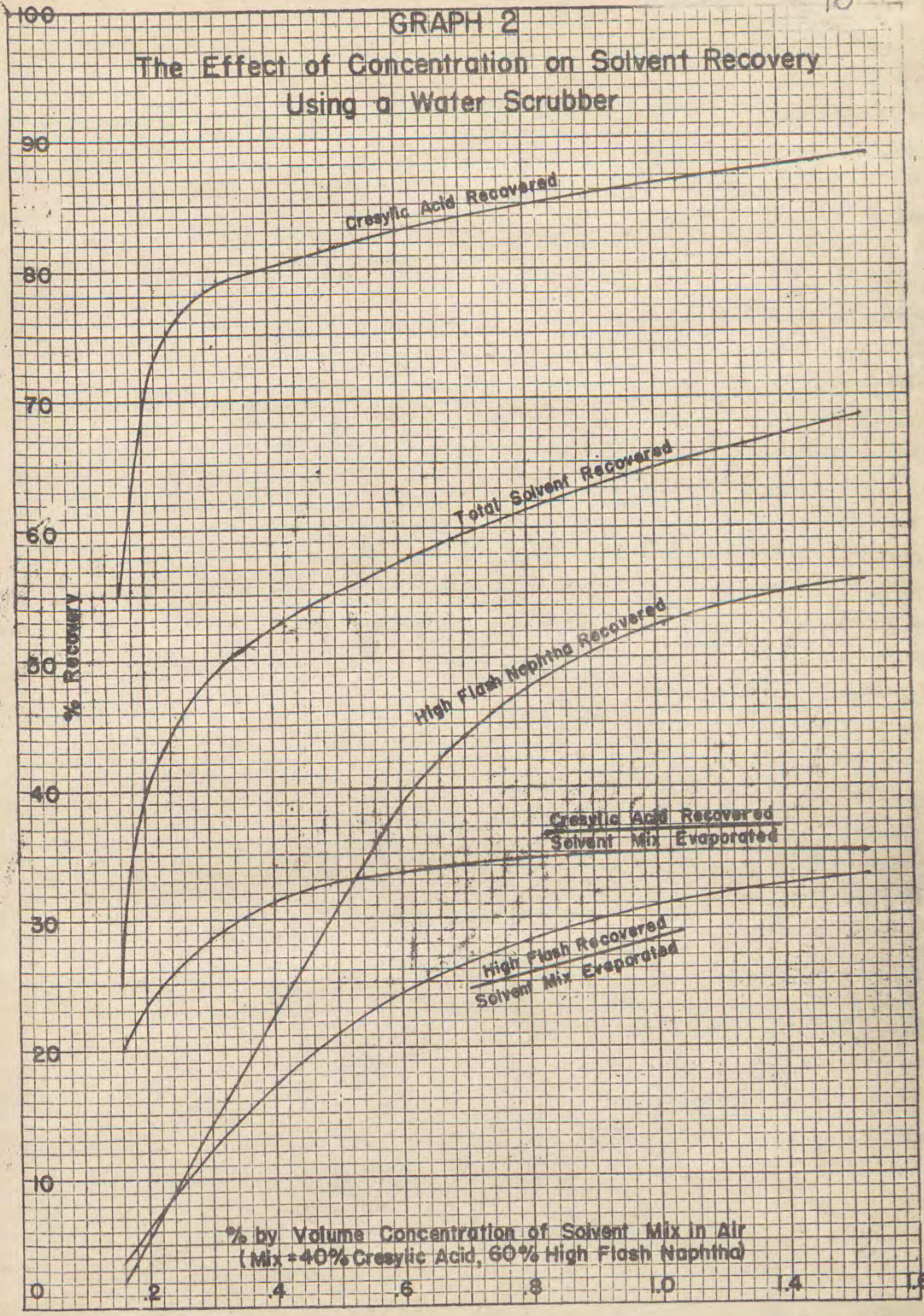
(a) - Height of Column: As the height of the packed column increases, the efficiency increases until a height is reached where equilibrium exists between the solvent vapor in the air and the cold water temperature.

(b) - Diameter of Column: The diameter of the column affects only the capacity. As the diameter is increased, larger volumes of air containing solvent can be handled.



GRAPH I

The Effect of Concentration on Cost and Efficiency of Recovery


GRAPH 2
The Effect of Concentration on Solvent Recovery
Using a Water Scrubber
Cresylic Acid Recovered
Total Solvent Recovered
High Flash Naphtha Recovered
Cresylic Acid Recovered / Solvent Mix Evaporated
High Flash Recovered / Solvent Mix Evaporated
% Recovery
% by Volume Concentration of Solvent Mix in Air
(Mix = 40% Cresylic Acid, 60% High Flash Naphtha)
0
10
20
30
40
50
60
70
80
90
100
.2
.4
.6
.8
1.0
1.4
1.6

(c) - Velocity of Air Containing Solvent Vapor: The effect of air velocity on recovery efficiency is not critical for moderate velocities. However, as the velocity is increased above a limiting velocity (dependent upon the height of the column), the recovery efficiency will fall off as illustrated in graph 3. The air velocity should be kept low enough to insure against exceeding the load point.

At a given water rate there is an air rate which will cause flooding. Likewise at a given air velocity, there is a rate of water flow which will cause flooding. See graph 4.

The loading point(10) represents the air velocity at which drops of water are carried up out of the tower by the air. The flooding point designates the air velocity at which the water cannot penetrate the tower but is held as a layer on top of the packing.

As either air or water velocity is gradually increased, the water holdup in the packing is increased. Bubbles of air begin to appear in the water. Further increase in air or water velocity increases the liquid held up with an increase in bubble formation until the whole mass begins to bubble violently accompanied by a lifting of Raschig rings. A condition is finally attained where the pressure drop rises sharply with even a slight increase in air velocity. Entrainment of water in air leaving column also increases sharply. This is the flood point.

Below the load point there is little change in the channel area. Above the loading point due to an increase in liquid retention the channels decrease in area as the air velocity increases causing a greater pressure. The loading point is believed to be the point at which the water holdup becomes sufficiently marked to cause reduction in the area available for air flow. The flooding point occurs when the channels become sufficiently clogged by extreme water retention so that the tower acts as a bubbler over its entire length.

(d) - Water Recirculation Rate: The water rate for a given velocity of air flow should be below the load point to avoid unnecessary loss of solvent and water by entrainment. See graph 5. The lower the water rate, the less power is required for pumping water. Since the air flow pressure drop decreases with decreasing water flow, a low water rate requires less power for air blowing.

The water recirculation rate should be great enough to wet a large portion of the packing in the column. As the flow of water is increased, the area wetted increases since the water film thickness remains practically constant except at the very low water rates. If the water rate is less than 5 lbs. per minute per $ft.^2$ packing area, the packing will not be wetted enough and channeling will occur resulting in a decrease in recovery efficiency.

(e) - Type and Size of Packing: Raschig rings are the most commonly used form of tower packing. The ring is a hollow cylinder of the same length as diameter of the cylinders. The walls are as thin as the material will permit without crushing in order to provide a larger free cross sectional area and a larger free volume. Ceramic Raschig rings in practice vary from about 3/8" to 6" in diameter and have a wall thickness varying from about 1/16" to 5/8". Rings containing one or two interior webs increase the surface area without greatly decreasing the free cross section.

The product of free space and surface area divided by 100, parallels the efficiency and the overall utility of a given packing. The efficiency, in general, varies inversely with the packing diameter. When the packing elements are of the same size and shape, the probability of the wetting liquid forming channels becomes a minimum. For hollow cylinders a ratio of the length to the diameter of 1:1 causes a minimum of channeling.

The maximum allowable water rate for normal non-flooded operation decreases with increasing gas velocity and is apparently independent of the height of the column to the diameter of the column ratios and also independent of the diameter of the column to diameter of packing ratios.

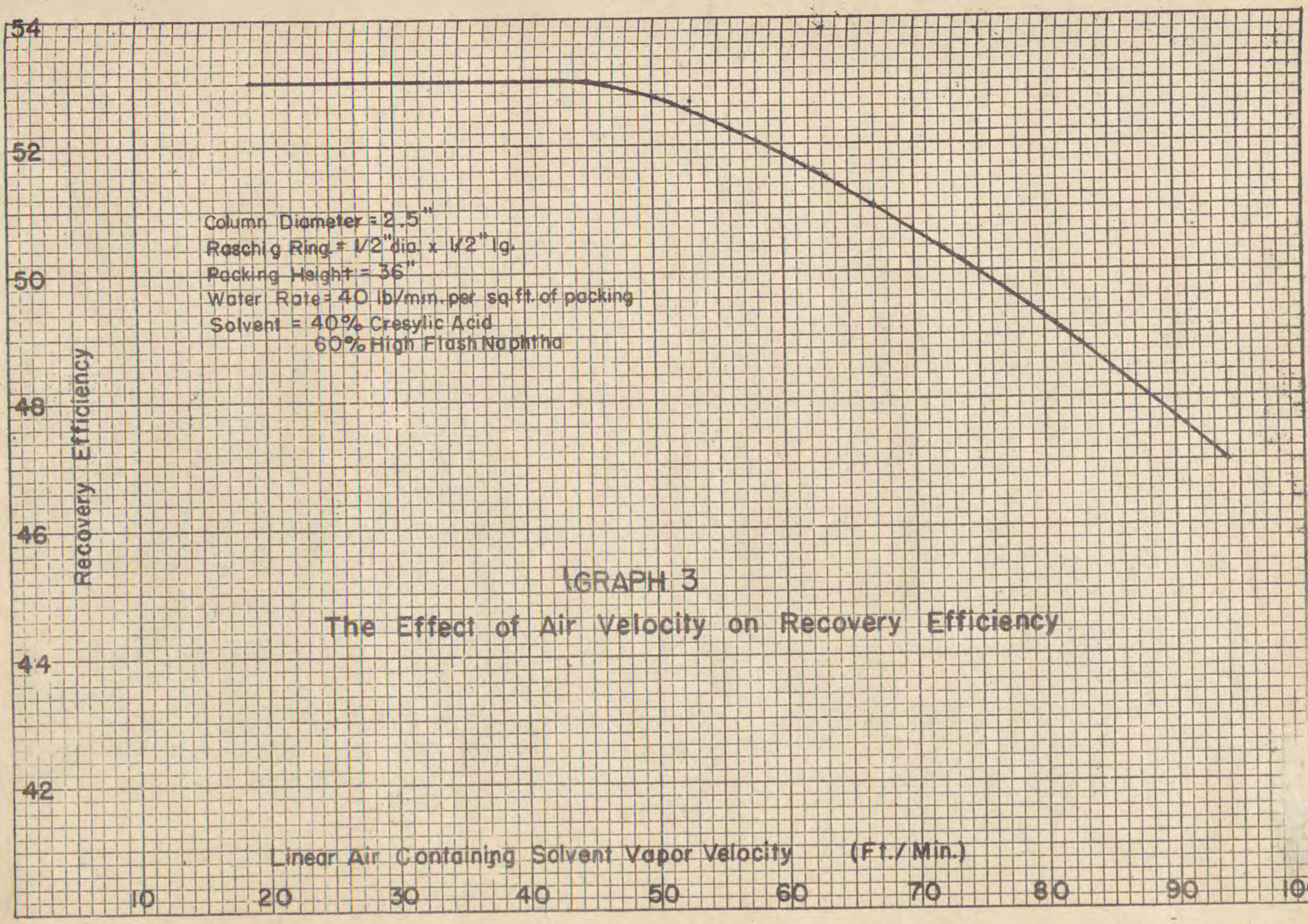
The ratio of column diameter to ring packing diameter must be 8:1 to prevent concentration of flow in outermost zone by the column walls. When the packing elements are large compared to the tower diameter, there is a marked tendency for the water to concentrate near the walls and for the center of the column to run somewhat dry.

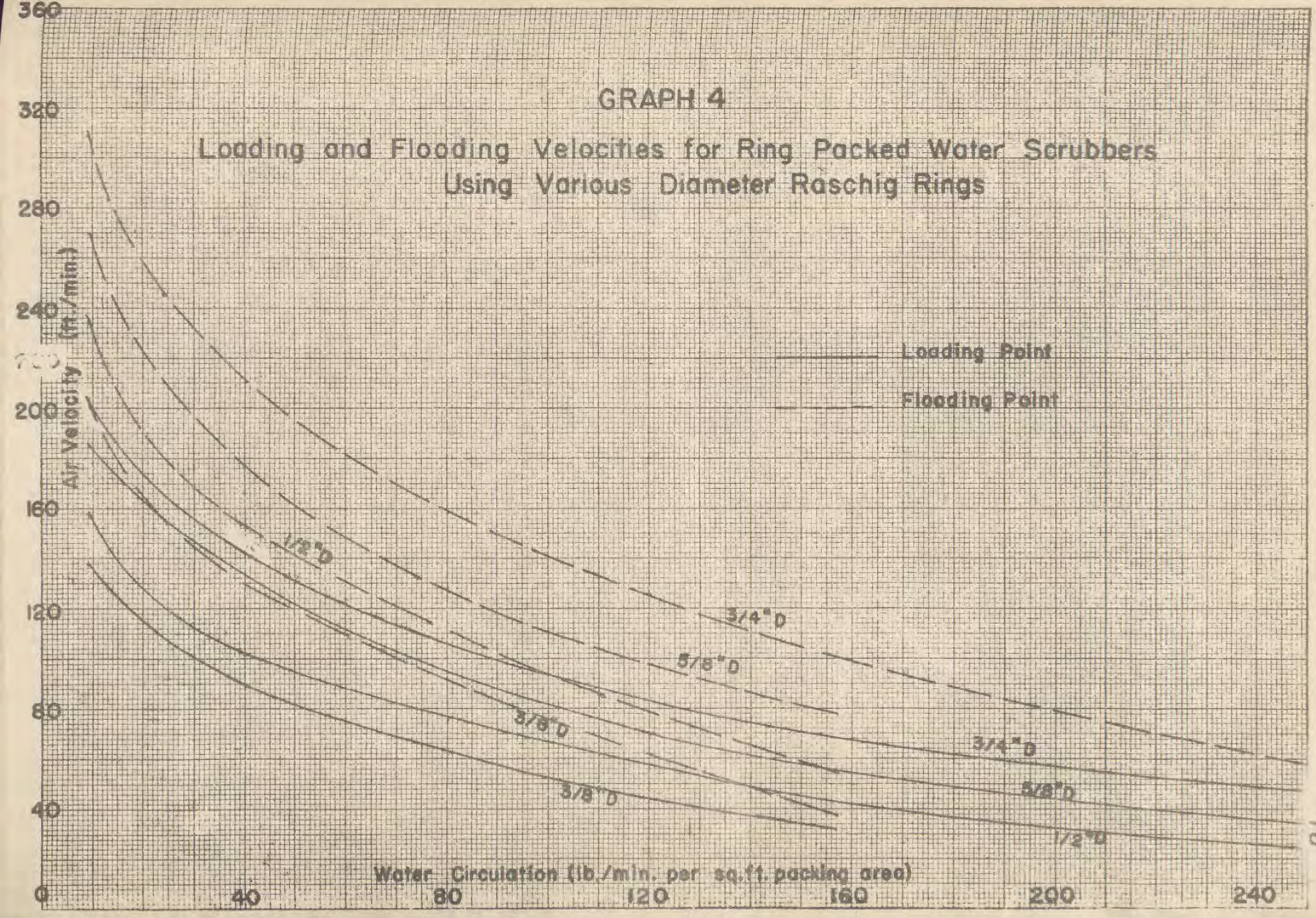
At all rates of liquid flow below the flooding point, the wetting liquid runs down the packing in thin streams, the major portion being diverted to the column walls owing to the increased free space there due to the displacement of the packing by the column wall. The packing next to the tower wall is not so closely packed as the center portions. There is a lowering of resistance to air flow in the central portions of the column with an increased rate of water flow near the column wall. When the air velocity just below that causing flooding is reached, the air stream up the wall will support the main water stream down the wall against the force of gravity and hence tend to push the liquid stream toward the center of the packing. At the center of the packing the liquid will again be held up by a limiting air velocity and complete flooding will occur. Flooding, therefore, most probably takes place first at the tower wall and then spreads inward toward the center of the packing.

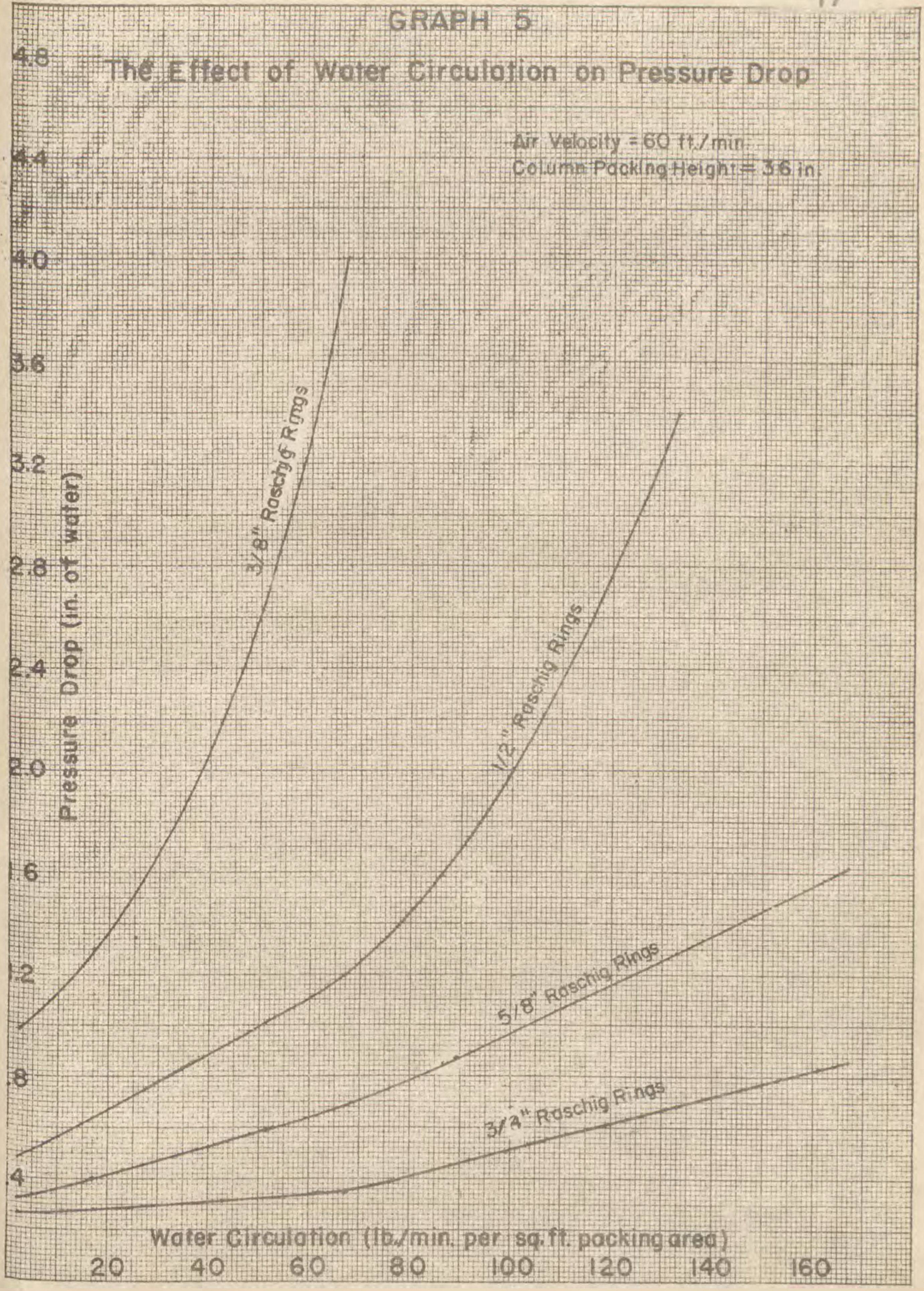
(f) - Distribution of Feed Circulating Water: In a packed column some device is necessary to distribute the water fairly evenly over the top layers of the packing. Flow from a single stream is not uniformly distributed until four or five tower diameters have been traversed. Four streams aimed parallel to column and evenly spaced on a mean circumference can be effectively used for fairly uniform distribution. Weir boxes or perforated plates can also be used. Spray nozzles give good results if pointed toward large central portions of column packing. However, sprays which are pointed outward so as to allow a large portion of water to concentrate on the column walls are even less effective than a single central stream because the water has a natural tendency to seek the walls in preference to the packing.

Distribution here refers to the distribution of the thin water streams over the packing and walls. The packing can be only partially wetted even though the water streams are well distributed over the cross sectional area of the packing.

As the column length is increased, a greater portion of the wall surface compared to Raschig ring surface is wetted.


54
52
50
48
46
44
42
Recovery Efficiency
Column Diameter = 2.5"
Raschig Ring = 1/2" dia. x 1/2" lg.
Packing Height = 36"
Water Rate = 40 lb/min. per sq.ft. of packing
Solvent = 40% Cresylic Acid
60% High Flash Naphtha
GRAPH 3
The Effect of Air Velocity on Recovery Efficiency
Linear Air Containing Solvent Vapor Velocity (Ft./Min.)
10
20
30
40
50
60
70
80
90
100


GRAPH 4
Loading and Flooding Velocities for Ring Packed Water Scrubbers
Using Various Diameter Raschig Rings
Loading Point
Flooding Point
Air Velocity (ft./min.)
Water Circulation (lb./min. per sq.ft. packing area)
3/4"D
5/8"D
1/2"D
3/8"D
3/4"D
5/8"D
3/8"D
1/2"D
0
40
80
120
160
200
240
280
320
360
0
40
80
120
160
200
240
16


GRAPH 5
The Effect of Water Circulation on Pressure Drop
Air Velocity = 60 ft./min.
Column Packing Height = 36 in.
3/8" Raschig Rings
1/2" Raschig Rings
5/8" Raschig Rings
3/4" Raschig Rings
Pressure Drop (in. of water)
Water Circulation (lb./min. per sq.ft. packing area)
4.8
4.4
4.0
3.6
3.2
2.8
2.4
2.0
1.6
1.2
.8
.4
20
40
60
80
100
120
140
160

V PACKED COLUMN PILOT PLANT

The packed column water cooler for a six strand wire coating oven consists of a four inch inside diameter tube eight feet long packed with 1/2 inch Raschig rings. A coiled pipe water cooler in series with the column maintains a low temperature in water flowing over packing. A settling tank also in series separates the insoluble floating solvent from the water. See figure II.

A - Procedure

To recover solvent, the pilot plant is operated as follows:

1. The water recirculating rate, measured by a rotameter, is set at a predetermined flow.
2. The air blower pulls air from the wire oven through the packed column. It is adjusted to a speed great enough to prevent oven air leaking out of the top of the hood where the wires pass through a narrow slot before passing over the sheaves. For our equipment, this speed corresponds to a negative pressure drop of eight to sixteen inches of water measured by the manometer between the blower and the column.
3. The speed of copper wire through the Formex enamel in the applicator and the oven above the applicator is adjusted to produce a good wire coating.
4. The volume of air through the oven and the column is regulated by a valve near the air rotameter to give the desired concentration of solvent vapor in air.

The total amount of solvent evaporated from the wire during the eight hour run is calculated from the Formex enamel consumed in the applicator. The solvent evaporated per minute and the air flow per minute are the basis for determining the solvent concentration in air. The solvent recovered is continuously decanted and the water is recirculated to save the solvent required to saturate water.

Previously, it was demonstrated that the portion of the solvent not condensed out by the packed column water cooler could be recovered by an activated carbon adsorber following cooler. (See graph 1). An electrostatic precipitator connected in series following the packed column water cooler was operated for several runs to recover the portion of solvent not condensed out by the cooler. Since the solvent vapors following the packed column water cooler are present as a gas rather than as entrained or suspended particles, no solvent was recovered in this manner.

The ability of a solvent to be condensed is inversely proportional to its vapor pressure. Vapor pressure data on our solvents were not available so we obtained the values plotted in graph 8 from apparatus shown in figure III.

The needle valve is adjusted to produce a given vapor pressure indicated by the manometer. The temperature at the set pressure is taken from thermometer. (See graph 8). The thermometer reads the liquid-vapor equilibrium temperature since the thermometer bulb is in the solvent vapor with solvent liquid percolating onto the bulb.

B - Results

1. The recovery efficiency is increased by:

   (a) Increasing the height of column. (See graph 6)

   (b) Increasing the rate of water flow. (See graph 7)

   (c) Increasing the concentration of solvent vapor in air. (See graph 6).

   (d) Selection of solvents with lower vapor pressure to use in place of present solvents. The less the vapor pressure of the solvent, the greater is the recovery by condensation methods. (See graph 6). Vapor pressures for a few solvents are plotted. (See graph 8).

2. Since the more expensive component, cresylic acid, is recovered in greater relative amount, the dollar value recovered is greater than the volume recovery (See graph 9).

3. The electrostatic precipitator following the packed column did not precipitate any solvent.

4. The Formex enameled wire produced by the newly designed oven is good and passed all Formex specifications.

5. Data from the pilot plant approximately checks data from the laboratory packed column. (See graph 6).

VI GENERAL SUMMARY AND CONCLUSIONS

At least two-thirds the cost of the present Formex enamel solvent can be recovered by the installation of a properly designed and operated packed column water cooler. S uch a column should contain ten to twelve feet of packing and the diameter should be large enough to allow a maximum air velocity of sixty feet per minute through the column. The water recirculation rate should be two hundred pounds per minute per square feet of packing area or more. The air intake to the oven should be cut down until concentration of solvent in air is approximately 0.80 per cent by volume. The blower should supply sufficient negative pressure to prevent leakage of stack air out of the hood slot at top of oven, but should not unnecessarily dilute the concentration of the solvent in air below about 0.80 per cent by volume.

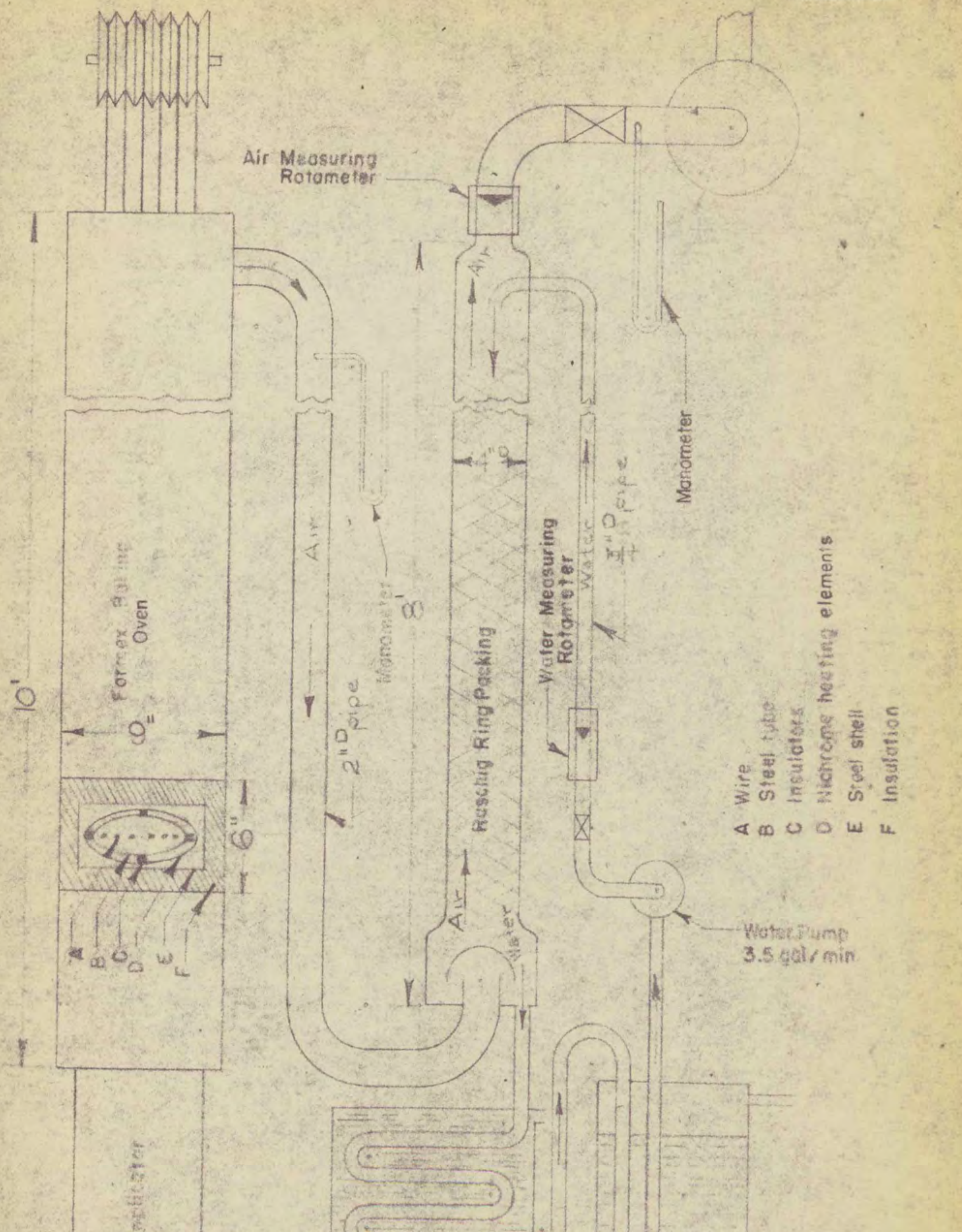


Figure II
Solvent Recovery Pilot Plant

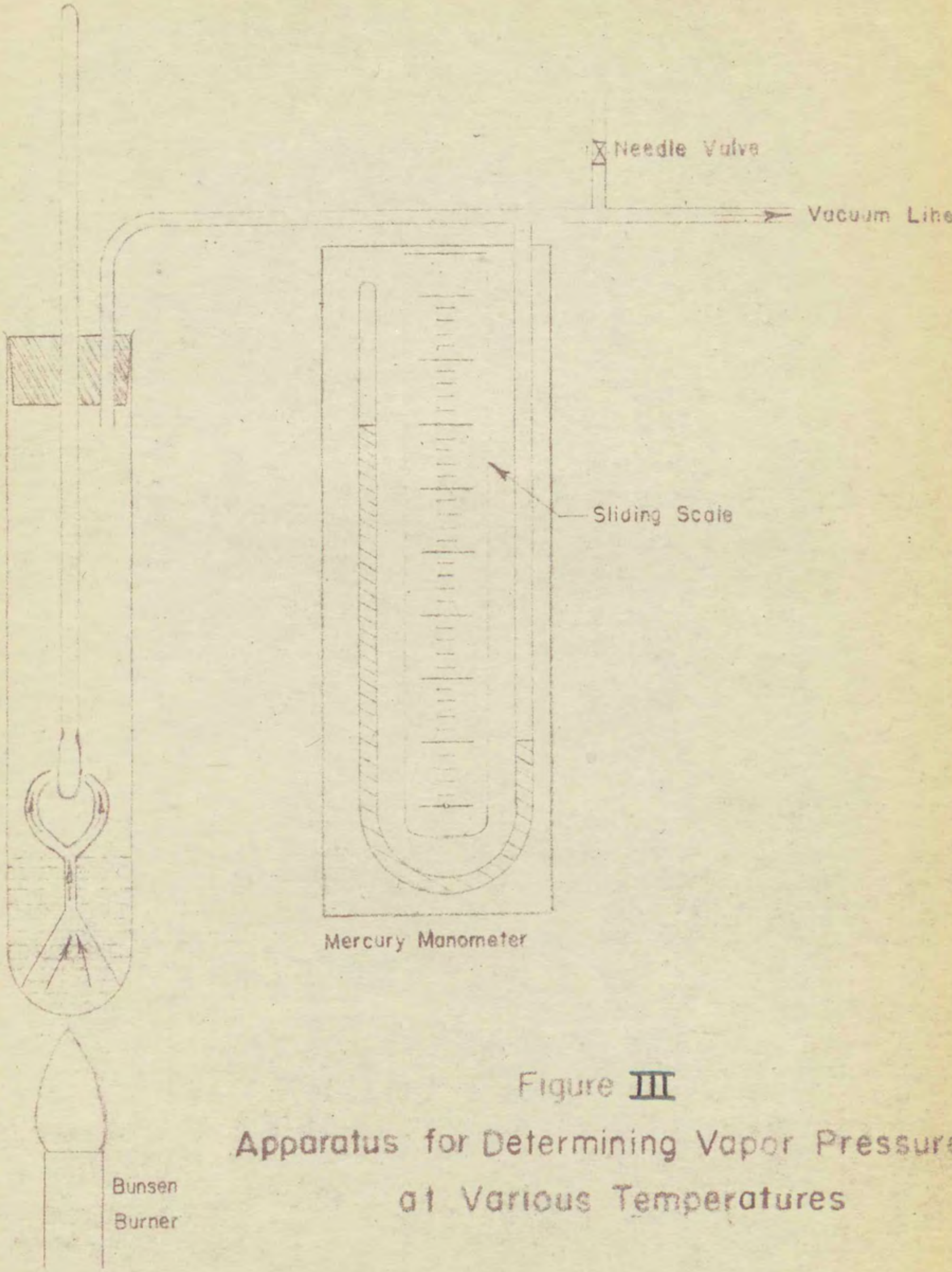
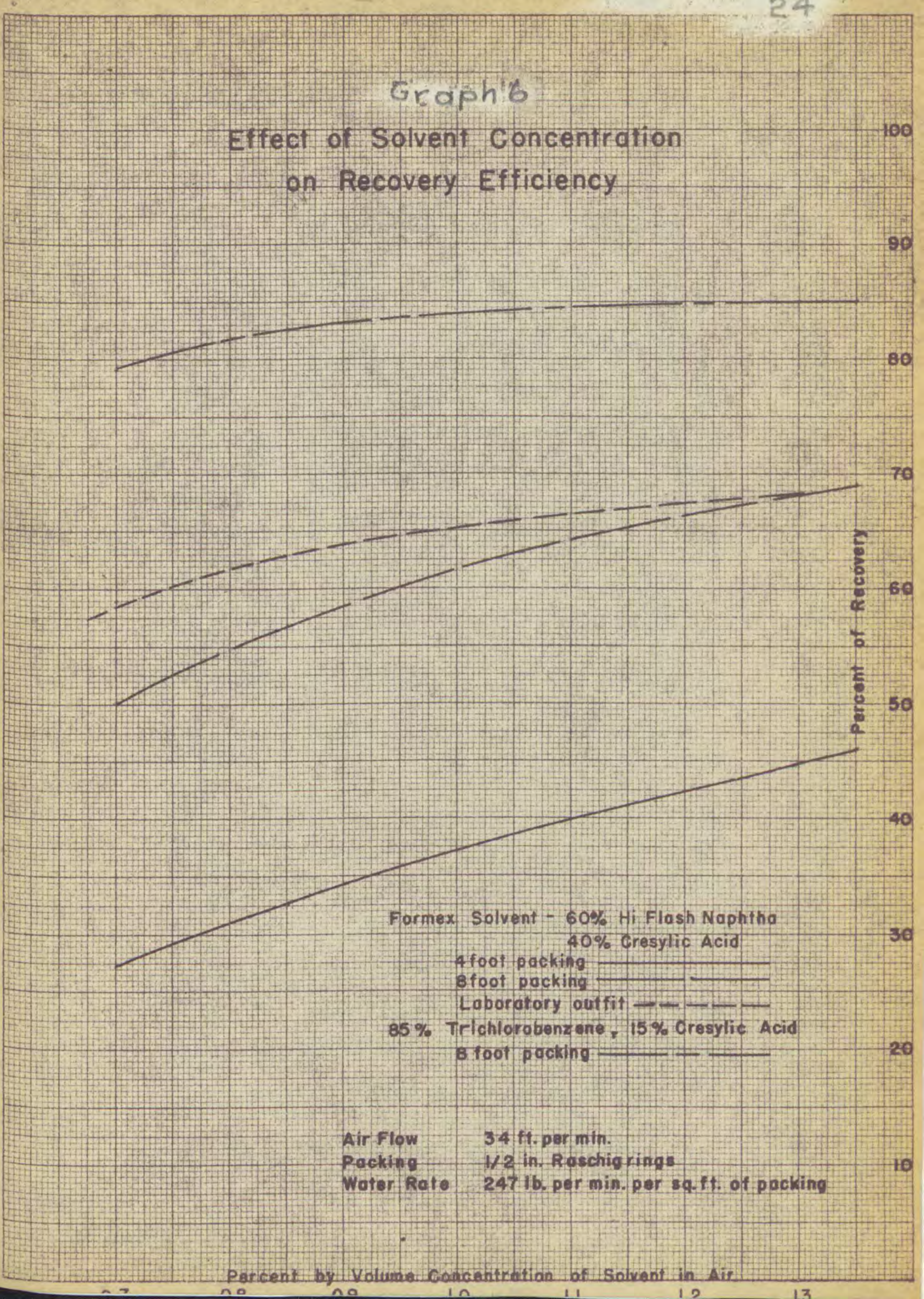
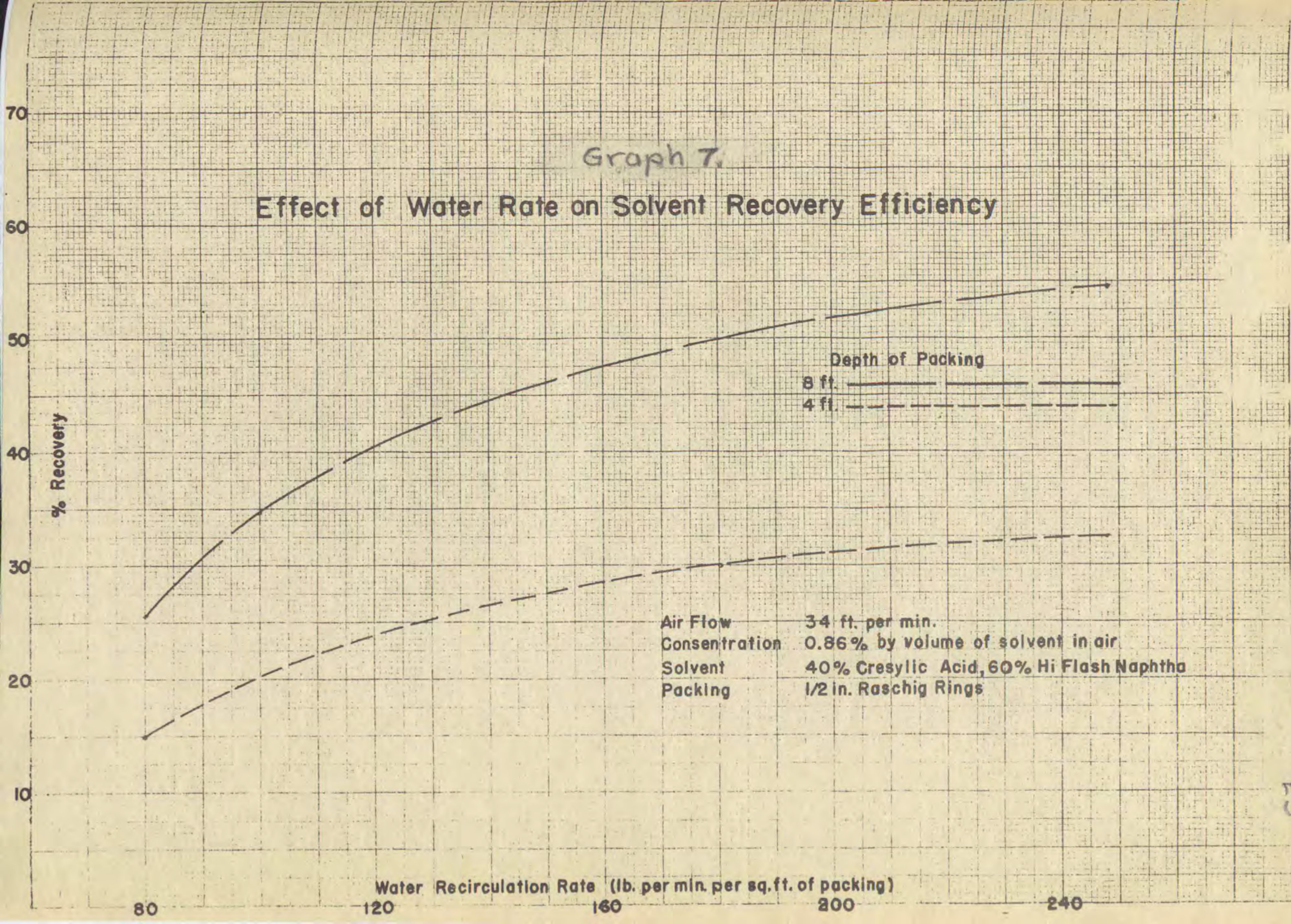
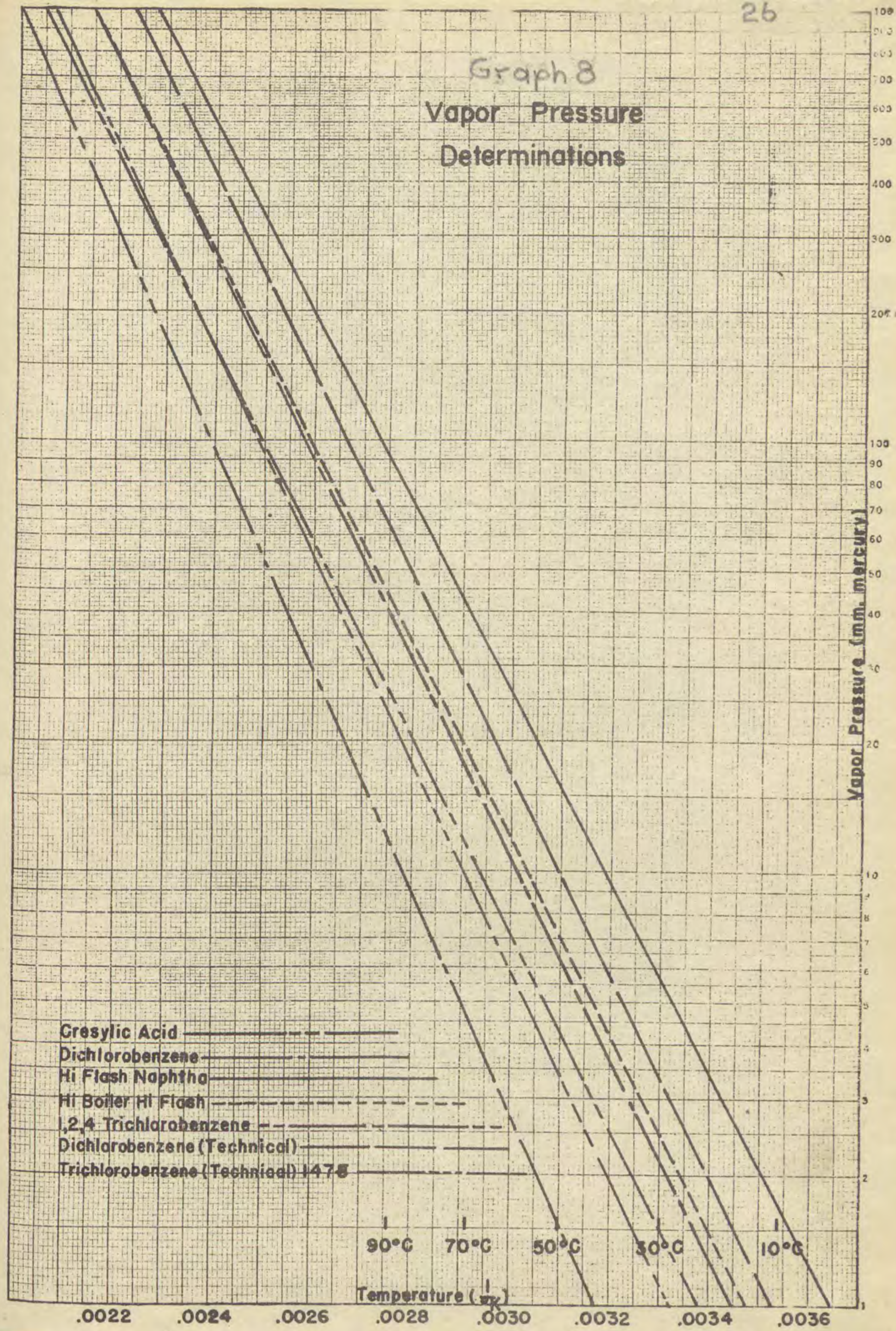


Figure III
Apparatus for Determining Vapor Pressures at Various Temperatures


Graph 6
Effect of Solvent Concentration
on Recovery Efficiency
100
90
80
70
60
50
40
30
20
10
Percent of Recovery
Formex Solvent - 60% Hi Flash Naphtha
40% Cresylic Acid
4 foot packing
8 foot packing
Laboratory outfit
85% Trichlorobenzene, 15% Cresylic Acid
8 foot packing
Air Flow 34 ft. per min.
Packing 1/2 in. Raschig rings
Water Rate 247 lb. per min. per sq. ft. of packing
Percent by Volume Concentration of Solvent in Air


Graph 7.
Effect of Water Rate on Solvent Recovery Efficiency
Depth of Packing
8 ft.
4 ft.
Air Flow 34 ft. per min.
Consentration 0.86% by volume of solvent in air
Solvent 40% Cresylic Acid, 60% Hi Flash Naphtha
Packing 1/2 in. Raschig Rings
% Recovery
10
20
30
40
50
60
70
Water Recirculation Rate (lb. per min. per sq. ft. of packing)
80
120
160
200
240


Graph 8
Vapor Pressure
Determinations
Cresylic Acid
Dichlorobenzene
Hi Flash Naphtha
Hi Boiler Hi Flash
1,2,4 Trichlorobenzene
Dichlorobenzene (Technical)
Trichlorobenzene (Technical) 1476
90°C
70°C
50°C
30°C
10°C
Temperature
.0022
.0024
.0026
.0028
.0030
.0032
.0034
.0036
Vapor Pressure (mm. mercury)
1000
900
800
700
600
500
400
300
200
100
90
80
70
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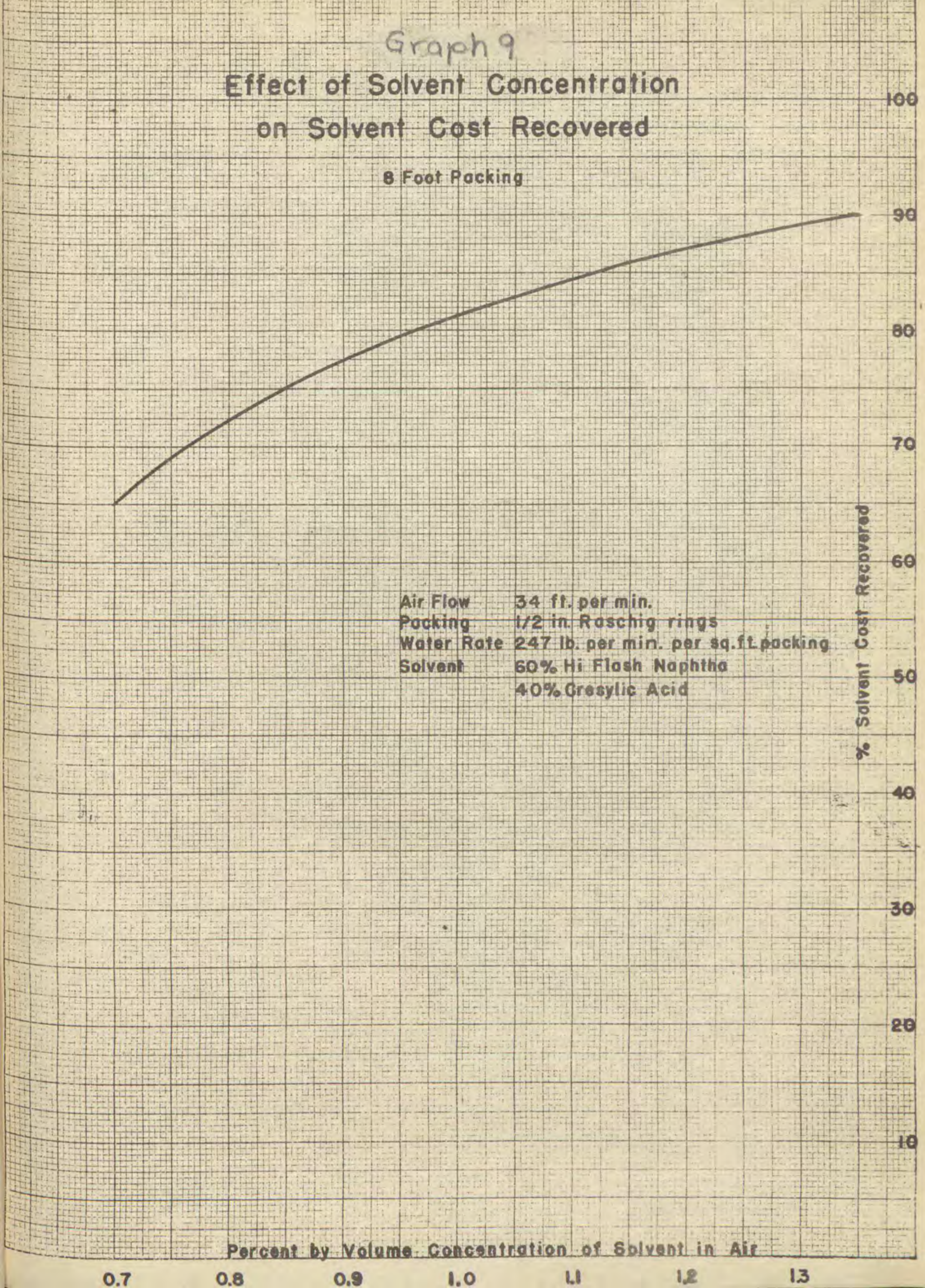
Graph 9
Effect of Solvent Concentration
on Solvent Cost Recovered
8 Foot Packing
Air Flow 34 ft. per min.
Packing 1/2 in. Raschig rings
Water Rate 247 lb. per min. per sq. ft. packing
Solvent 60% Hi Flash Naphtha
40% Cresylic Acid
% Solvent Cost Recovered
100
90
80
70
60
50
40
30
20
10
Percent by Volume Concentration of Solvent in Air
0.7
0.8
0.9
1.0
1.1
1.2
1.3