

LIQUID HOLDUP AND FLOW PATTERNS
IN PACKED TOWERS

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Thesis for the Degree of M. S.
MICHIGAN STATE UNIVERSITY
Rodney David Wood
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LIQUID HOLDUP AND FLOW PATTERNS
IN PACKED TOWERS

By

Rodney David Wood

AN ABSTRACT

Submitted to the College of Engineering
Michigan State University of Agriculture and
Applied Science in partial fulfillment of
the requirements for the degree of

MASTER OF SCIENCE

Department of Chemical Engineering

1959

Approved Randall W. Luedt

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ABSTRACT

This thesis reports the results of an experimental investigation of the effects of liquid-solid interfacial tension on liquid holdup and flow patterns in packed towers. Two towers were used: one, 3 inches in diameter, and packed with 1/4 inch glass spheres to a depth of 18 inches; the other, 6 inches in diameter, and packed with 1/2 inch glass spheres to a depth of 36 inches.

Variation in interfacial tension was obtained by using, in the one case, clean glass spheres, and in the other, spheres which had been treated with Beckman Desicote. Liquid holdup was measured at a variety of liquid flow rates by continuous weighing of the tower and its contents. Flow patterns were studied by introducing a small stream of potassium permanganate into various locations at the top of the packing. The path of the permanganate down through the packing was then observed.

Neither liquid holdup nor the pattern of flow was found to be affected by the change in interfacial tension, for either the 6 inch or the 3 inch column.

There was a definite difference between the behavior of the small tower and that of the large one, however. At equal liquid flow rates (in lb./hr. sq. ft.) the smaller tower displayed much higher values of holdup (per cubic foot of packing) than did the 6 inch column.

A graph of holdup versus flow rate was prepared. Over a considerable range, the curve for the 3 inch column is very nearly a straight line, whereas that for the larger tower shows no linear portion.

The most striking difference between the two columns was in the flow patterns observed using the permanganate tracer. The following points were noted:

- 1) In both columns, there appeared to be a tendency for the water to proceed from the center of the packing toward the tower wall.

- 2) Both towers showed definite channeling of the flow at low liquor rates, and no discernible channeling at high flow rates.

- 3) The region of transition from channeling to well-mixed flow occurred at roughly the same value of holdup for both towers. These points were, of course, at greatly different flow rates.

- 4) The same channels were observed at all flow

Rodney David Wood

rates below the transition range for each tower. This suggests that channeling is influenced more by the packing than by the liquid.

5) One quantity measured (though admittedly only approximately) was the vertical distance required for the permanganate to reach the wall of the tower from the center. A similar measurement was taken with tracer introduced near the wall. In all cases, at all flow rates, distribution was about twice as rapid in the 3 inch tower as in the 6 inch column.

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He is also indebted to Dr. James L. Dye, who provided advice on certain aspects of the experiments; and to W. B. Clippinger, who assisted him greatly in the design and fabrication of equipment.

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INTRODUCTION

The chief problem in the design of packed towers is to determine the size required for the mass transfer operation in question. There has been for several decades a continuing search for reliable means of predicting performance. The quest has not been an unqualified success.

Considerable assistance was given by the two-film theory of mass transfer between phases, which was advanced by Whitman³⁴ in 1923. Since then, an enormous amount of work has gone into the measurement and correlation of gas-film, liquid-film, and overall coefficients. Even so, Rixon²¹ appraised the situation in 1948 as follows: "The published data on the absorption and desorption of carbon dioxide from gases in water in packed towers is (sic) characterized by inconsistency, contradiction, and absence of general correlation." Since the system of carbon dioxide and water had been studied quite frequently in the 1930's and 1940's,^{8, 12, 17, 18, 24, 28} one can imagine the state of affairs with other systems.

In the course of seeking methods of tower design, a great number of factors have been given attention. Some of these, such as packing surface area, void

fraction, and packing size, are characteristics of the device itself. Others include properties of the fluid streams. Finally there are aspects of the tower operation, such as loading and flooding velocities, liquid distribution, and the amount of liquid contained within the tower. This last quantity is termed "liquid holdup" and will be more fully discussed in the next chapter.

For the present work, it was decided to examine holdup and the patterns of liquid flow, and to determine, if possible, the manner in which these are affected by the liquid-solid interfacial tension, the packing size, and the liquid flow rate. The reasons for this choice will be set forth in the chapter on The Choice of Variables.

LIQUID HOLDUP

General Considerations

Liquid holdup is defined as the quantity of liquid contained within the packed volume of the column. Three types of holdup are commonly recognized.

Static holdup is the quantity of liquid which remains within the tower after the packing has been thoroughly wetted and then permitted to drain. The time of drainage is a factor, because large columns continue to drain (at a very slow rate) for several hours or even days.^{10, 26, 27} It has been found, however, that the rate of drainage quickly drops to a small, constant value; and the practice has usually been to take the drainage time as ten minutes.

Static holdup is frequently called the portion of the liquid which is independent of the flow rate. This description is not entirely accurate. It has been shown^{10, 26, 27} that all the liquid in the column participates in the flow. Work done in connection with this thesis indicates that this is particularly so at high flow rates.

Total holdup is the entire amount of liquid within the packed volume when the column is operating under any given conditions.

Operating holdup is defined as total holdup minus static holdup.

Values of holdup are customarily expressed in cu. ft. of liquid per cu. ft. of packed tower volume.

Some writers have made use of a "free volume," though this has not been nearly so common. Mention is made of a "drained free volume," which is equal to the void fraction of the dry bed minus static holdup, and an "operating free volume," which is the void fraction less total holdup.

Methods of Measurement

Early investigators generally used the following technique to determine values of holdup.

The tower was packed dry. The packing was then drenched with a measured quantity of liquid, and the excess liquid was collected and measured. The original quantity of liquid minus the excess yielded static holdup.

To determine operating holdup, provision was

made^{10, 17} for shutting off the liquor feed and diverting the effluent simultaneously. The liquid was drained into a receptacle and weighed. Since the static holdup remained on the packing, operating holdup was measured directly. Total holdup could then be computed, if desired.

More recently, the practice has been to mount the column so that it is connected to a weighing device.^{26, 27} The liquid distributor and drain are mounted separately. The column and its contents can then be weighed continuously. In addition to convenience, this method has the advantage of direct measurement of holdup. It also facilitates design of the packing support and drain. (See the chapter on Equipment.)

Holdup and Mass Transfer Rates

Early in the 1930's, investigators began to report that absorption rates appeared to be associated with liquid holdup. A considerable part of the interest in holdup has been due to this fact.

Payne and Dodge¹⁷ were among the first to perceive this. They examined the absorption of carbon dioxide in aqueous media, and concluded that the increase

in absorption rate with increase in liquor rates is proportional to total holdup. About the same time, Bennetch and Simmons² derived and tested a correlating equation for mass transfer coefficients. In this expression, the coefficient is inversely related to free volume, which would correspond to a direct relationship with holdup. Free volume was assumed to be constant for a particular tower, irrespective of operating conditions. Simmons and Osborn²⁸ later reported that this equation produced a more satisfactory correlation if the operating free volume -- which manifestly was not constant -- were employed. These same men did some work on the factors affecting operating free volume. They concluded that it is independent of the type of packing, tower size, or the gas flow rate.

Furnas and Bellinger⁸ performed experiments in a 12 inch tower, 10 feet high, over a considerable range of liquor rates. They expressed the overall mass transfer coefficient as a function of liquid rate and holdup. Also given was the dependence of the coefficient on holdup when taken as a function of holdup alone. The overall coefficient varied as the 0.6 power of holdup for 3/8 inch Raschig rings, the 1.18 power for 1 inch rings, and the 0.97 power for 1 inch Berl saddles.

White and Othmer³³ reported overall coefficients and holdup for Stedman packing as functions of liquid rate, finding that both increase with an increase in liquor rate. Between 2000 and 6000 lb./hr. sq. ft. holdup varied linearly with liquid rate, while below 2000 lb./hr. sq. ft. it fell off sharply. No attempt was made to correlate mass transfer coefficients with holdup, however.

By 1950, a mass of evidence attested the connection between holdup and absorption rates. It was also suspected that both were related to the degree and manner in which the liquid wetted the packing. According to Pratt,¹⁹ who attempted to correlate a great deal of data, coefficients increase rapidly with liquid rate until a minimum effective liquor rate is attained. This, he concluded, appears to be the lowest liquid rate at which the packing is completely wetted, or as nearly completely wetted as it will get.

Some research was also done on the effect of holdup on the performance of packed fractionating towers. Fenske, Tongberg, and Quiggle⁷ discussed the relation of holdup to separation in distillation columns. They maintained that, for sharp separations, holdup should

be small. These gentlemen, incidentally, presented values of holdup for many types of packing which have not yet seen wide usage in commercial towers. Notable among these were carding teeth, jack chain, pieces cut from wire mesh, and several kinds of rivets. Their findings contradicted the claim of Simmons and Osborn that holdup is independent of the type of packing.

A somewhat different conclusion was reached by Rose, Williams, and Prevost,²² who studied batch distillation. They found that, at a certain "critical" reflux ratio, holdup has no effect on the sharpness of the separation. At ratios greater than the critical, increasing holdup is detrimental. Below the critical ratio, the reverse is true.

Holdup, Pressure Drop, and Flooding

An early investigation of flooding velocities and pressure drop was made by White.³² He mentions the difficulty of obtaining consistent results, citing differences in pressure drop up to twenty per cent when a column is unpacked and repacked. Part of his trouble may have been caused by using packing which was not

sufficiently small in comparison with the tower diameter. (More will be said of this later.) One interesting point is that a tower wetted with static holdup liquid may have a resistance to gas flow fifty per cent greater than the same tower when dry.

The first thorough correlation of flooding conditions was done by Sherwood, Shipley, and Holloway.²⁵ The equation they derived is still used widely today. Lobo and others¹³ later contributed some useful data and suggested some modifications.

The relationship of holdup to flooding was first closely tested by Elgin and Weiss.⁶ They found that an abrupt increase in holdup coincides with a similarly sharp increase in pressure drop. This was confirmed a year later by Piret, Mann, and Wall,¹⁹ and has been verified several times since.^{26, 27} Elgin and Weiss decided, therefore, that holdup affords a measure of flooding velocity. They also reported that, without gas flow, holdup varied linearly with liquid rate above 3000 lb./hr. sq. ft., but not below. (Cf. White and Othmer.)

About the most recent development in flooding is due to Newton, Metcalfe, and Mason,¹⁶ who stated that flooding velocities are affected by the depth of the

packing. They plotted results using the original coordinates of Sherwood et al. with packing depth as a parameter. Few data are given. The differences found are very small, but are claimed to be significant.

Factors Affecting Holdup

Once it was established that holdup is an important variable, work was done to determine how it is influenced by the various properties of the fluid streams, the packing characteristics, and the conditions of operation. Some information on this matter was obtained by workers to whom reference was made earlier in the text.^{6, 17, 19, 28, 33} These, however, were incidental to other investigations, and not at all comprehensive. Moreover, the findings were inconsistent in many respects. The bulk of the research on holdup itself is presented in three articles not yet cited.

The first of these is by Jesser and Elgin,¹⁰ who investigated the effects of packed height, liquor rate, surface tension, viscosity, and density on operating holdup for seven different packings. Neither static nor total holdup was measured. Because a number of earlier

writers^{6, 19, 28, 33} had reported that gas velocity has only a very slight effect below the loading point, their work was done without gas flow. In addition to reporting the effects of liquid properties, they showed rather conclusively that holdup is not a linear function of liquid rate. Of particular interest to the author was their findings concerning the surface tension of the liquid: the effect on holdup varies from the 0.1 power to the 0.4 power, depending upon liquor velocity.

Jesser and Elgin also examined visually the mechanism of liquid flow and holdup, and concluded that there exist three regions of flow. At low rates -- below 4000 lb./hr. sq. ft. -- the liquid is held chiefly at the points of contact between pieces of packing. Next, the liquid begins to flow in a film over the packing. The area and thickness of the film increases until a flow rate of about 18,000 lb./hr. sq. ft. is reached. At this point liquid begins to leave the packing and fall freely through the space between pieces.

The other two articles were published simultaneously by Shulman and coworkers.^{26, 27} Six different packings were used in a 10 inch tower. Gas flow was extensively investigated. Total and static holdup both

were reported, and the results concerning total and operating holdup are in very good agreement with those obtained by Jesser and Elgin. Static holdup, which Jesser and Elgin did not measure, was found to be affected by all factors except gas flow and liquid flow rate.

These same two articles contain some discussion of the nature and significance of holdup. It was found that, when the tower was run on a dye solution for a short period and then returned to clean water, the dye was displaced from regions around contact points very slowly. It is suggested that this semistagnant liquid may be comparatively ineffective in absorption, and yet be important in rectification.

A few other articles have treated holdup. Struck and Kinney²⁹ found that the holdup in packed fractionating columns varies nearly linearly with the distillation rate. Bloodgood, Teletzke, and Pohland³ measured holdup in order to determine contact time in trickling filters. Leva¹¹ has discussed the significance of surface tension, and points out that the general trend of data indicate that holdup is proportional to the 0.2 power of surface tension at flow rates below about 8000 lb./hr. sq. ft.

In summary, previous work has shown that:

1. Holdup is closely associated with absorption coefficients.

2. Total and operating holdup are affected by: the packing characteristics; the liquor rate; the density, viscosity, and surface tension of the liquid; and, near flooding, the gas rate.

3. Static holdup is independent of gas or liquid rate, but does depend on the other factors mentioned above.

LIQUID FLOW AND DISTRIBUTION

It is now generally recognized that the nature of liquid flow in packed towers must have a profound effect on absorption. Nevertheless, very little work has been done. The behavior of the liquid is scarcely better known or understood today than it was thirty years ago.

Early work on liquid flow was done by Baker, Chilton, and Vernon.¹ They examined the effects of liquid distribution at the top of the packing, measuring the resulting distribution at the bottom by collecting liquid in a receiver which had been divided into annular segments. A number of different packings were used, in towers of various diameters and heights. All work was performed using a flow rate of 500 lb./hr. sq. ft., which is quite a small value. They concluded that if the ratio of tower diameter to packing size exceeds eight, uniform distribution is readily achieved and, once achieved, persistent. If this condition is not met, there is a tendency for the liquid to flow toward the wall of the column. They maintained that, in large towers, the only requirement for uniform liquid flow is even distribution at the top.

It is fair to point out that Baker et al. only measured the distribution of liquid as it emerged from the column, and thus learned nothing of the flow within the packing. It is by no means certain that the receiver did not interfere with the flow. A flat packing support cannot avoid influencing the configuration of the pieces immediately above it.

An ingenious experiment designed to study the pattern of flow within the tower itself was carried out by Mayo, Hunter, and Nash.¹⁵ They constructed 1/2 inch and 1 inch Raschig rings of paper, and placed them in a tower which had been lined with paper. They then operated the tower for a short period, using a liquid containing a red dye. The paper rings were removed and dried, and the area marked by the dye was measured.

It was concluded that the wetted area increases with increasing liquor rate. Just below flooding the packing is still not completely wetted, but becomes so abruptly as the tower floods.

They found, moreover, a definite tendency for the liquid to move toward the wall. For several reasons, however, the results are open to question. In the first place, the use of paper neglects capillary movement of

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They found, moreover, a definite tendency for the liquid to move toward the wall. For several reasons, however, the results are open to question. In the first place, the use of paper neglects capillary movement of

the liquid. Second, the column diameter (3 inches) was quite small for the size of packing. Finally, they reported flooding at 11,000 lb./hr. sq. ft., even with no gas flow. No other investigator corroborates so low a value. Even so, it was an interesting test.

Tour and Lerman^{30, 31} performed experiments with a column 20 inches in diameter and three or four feet high. The unusual shape was used so that the liquid, which was introduced by a single central pipe, would not reach the walls. The effluent was collected in a grid at the bottom. The results partially verified a distribution expression derived from the laws of probability.

A similar series of tests was conducted later by Cairns,⁵ using an even shorter and wider tower. A number of packings were studied at two flow rates, both very low. No attempt was made to correlate the results; Cairns merely observed that the distribution was low in degree, and not very uniform. He also mentioned that, with larger packings, distribution is wider but less uniform.

The material on flow patterns, then, is scanty. No mention whatever was found of work on individual streams

within the packing. Since it seems likely that the bulk of the liquid does flow in several distinct streams -- which may, of course, split and recombine -- this is a conspicuous omission.

There have been a few attempts to study the behavior of liquid streams in absorption processes. These have been conducted with wetted-wall columns, however, and the relevance of the results to packed towers has not been established. They are nevertheless interesting. Two are described here.

Grimley⁹ examined the phenomena of ripple formation and film breakup, using a number of different liquids. He concluded that ripples assist absorption, and are related to the surface tension of the fluid. He also points out that surface active agents used to alter surface tension may interfere with mass transfer across the interface.²³ He does not, however, describe the basis of his contentions. His correlation for ripple formation involves the eighth power of the Reynolds number.

Bond and Donald⁴ observed that the absorption of ammonia is accompanied by the breaking up of the liquid film. This they associate with ripple formation. The explanation advanced is that because the film is

thinner in the trough than in the crest, the ammonia concentration is higher in the trough. The effect of surface tension is to draw trough liquid into the crest, thus causing the film to break.

THE CHOICE OF VARIABLES

By now it should be fairly clear that the behavior of the liquid within the packing is highly important. Three aspects of this behavior are evident:

- 1) The amount of liquid present upon the packing, i.e., holdup;
- 2) The manner in which the liquid is held upon the packing;
- 3) The manner in which the liquid moves over the packing.

The two preceding chapters were intended to give the reader an appreciation of the type and extent of research on these problems up to the present. In this chapter are set forth the reasons for choosing liquid-solid interfacial tension as a variable for investigation.

Such factors as the wetted area of the packing, and the quantity of liquid associated with the wetting, are certainly important. It was reasonable to expect that these would be affected by the surface tension of the liquid, and indeed this has been shown to be the case.^{10, 27} The phenomenon of wetting, however, is not a function of the liquid alone, but of the liquid-solid

pair forming the interface in question. Pure water is commonly said to "wet" clean glass, whereas mercury does not, but these are not properties solely of the liquids. Water on waxed paper has been observed by the author, and it behaves very much as does mercury on glass.

At this point a digression is in order. The surface tension of liquids is seldom measured as such. Usually it is the interfacial tension between the liquid and some gas, such as air or its own vapor. More explicitly, it is the work required to increase the liquid-gas interfacial area by one unit. The gas ordinarily accounts for a very small portion of this work, however, and hence the quantity is termed simply liquid surface tension.

When a liquid, a solid, and a gas are in mutual contact, the following equation¹⁴ relates the three interfacial tensions (see Fig. 1, page 21):

$$\gamma_{lg} \cos \theta = \gamma_{sg} - \gamma_{ls}$$

As may be seen from this equation, it is not possible to evaluate the liquid-solid interfacial tension γ_{ls} , but only $\gamma_{sg} - \gamma_{ls}$. If the nature of the solid surface is altered, as in this investigation, the correct

thing would be to speak of the change in the difference between the gas-solid and the solid-liquid interfacial tensions.

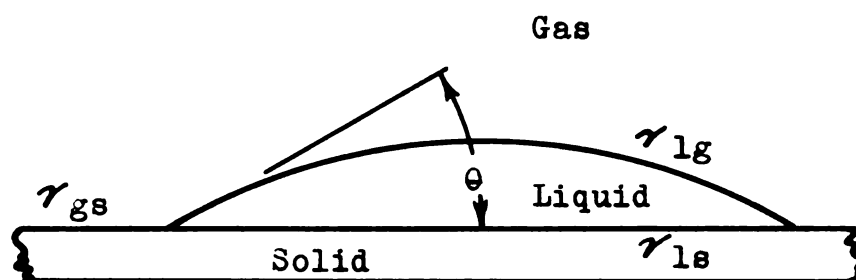


Fig. 1. Illustration of Contact Angle

To return to the main discussion, the reader is asked to consider the following argument. Let a small amount of water, with a surface tension of about 75 dyne/sq. cm., be in contact with a solid such as porcelain or glass. The contact angle will be small and, to a good approximation,

$$\gamma_{lg} = \gamma_{sg} - \gamma_{ls} = 75 \text{ dyne/sq. cm.}$$

Now let a wetting agent be added to the water in sufficient amount to reduce its surface tension to, say, 35 dyne/sq. cm. Again,

$$\gamma_{lg} = \gamma_{sg} - \gamma_{ls} = 35 \text{ dyne/sq. cm.}$$

But γ_{sg} has not been changed. Therefore, γ_{ls} must have increased by approximately 40 dyne/sq. cm., even though the solid surface is unchanged. Thus it appears that the action of wetting agents, commonly interpreted as a decrease in the liquid surface tension, is equally well expressed as an increase in the liquid-solid interfacial tension.

Now the liquid-solid interfacial tension may also be changed by altering the solid surface. It seems entirely reasonable to expect, therefore, that varying the solid surface should produce effects comparable to those produced by varying liquid surface tension. The main purpose of this investigation was to determine whether this were true.

Only one previous example of related research could be found, and it generated hope for affirmative results. Sherwood and Holloway²³ performed absorption experiments using packing coated with paraffin, and ascertained that the coefficients are considerably lower

than those for uncoated packing.

Inasmuch as this was to be rather an exploratory experiment, it was decided to include equipment size and liquid rate among the variables. Holdup was chosen as a criterion of effect because it was known to depend upon liquid surface tension, and because its measurement was relatively easy and reliable. Moreover, gas flow was not required, thereby simplifying problems of apparatus. Flow patterns were selected chiefly because so little had been done previously.

The range of flow rates was set as zero to 15,000 lb./hr. sq. ft. Higher values have been examined, but not frequently. Above this rate, loading occurs at even small rates of gas flow.^{6, 12, 24, 25, 26} Commercial towers frequently are designed to operate near the loading point.^{12, 18, 24}

EQUIPMENT

A diagram of the apparatus used is given in Fig. 2, page 25. The scheme was identical for both towers.

The columns and packings used were chosen so as to be, as nearly as possible, geometrically similar. Both were Pyrex glass pipe. The larger was 6 1/8 inches in diameter, and was packed with 1/2 inch glass spheres to a depth of three feet. The other, 3 1/16 inches in diameter, contained 1/4 inch glass spheres, and the packed height was 18 inches.

The packing support was designed to permit the best possible drainage from the packing. It consisted of a ring, integral with the mounting, with two parallel crossbars. Upon this rested a circle of wire mesh, which served as the support proper. The size of the mesh was half the size of the spheres. It is felt that this is about the best compromise between larger openings, which might be plugged by the packing pieces, and small ones, which tend to impede flow. Several holes were drilled in the ring to permit ready drainage of any liquid which might run down between the ring and the tower wall.

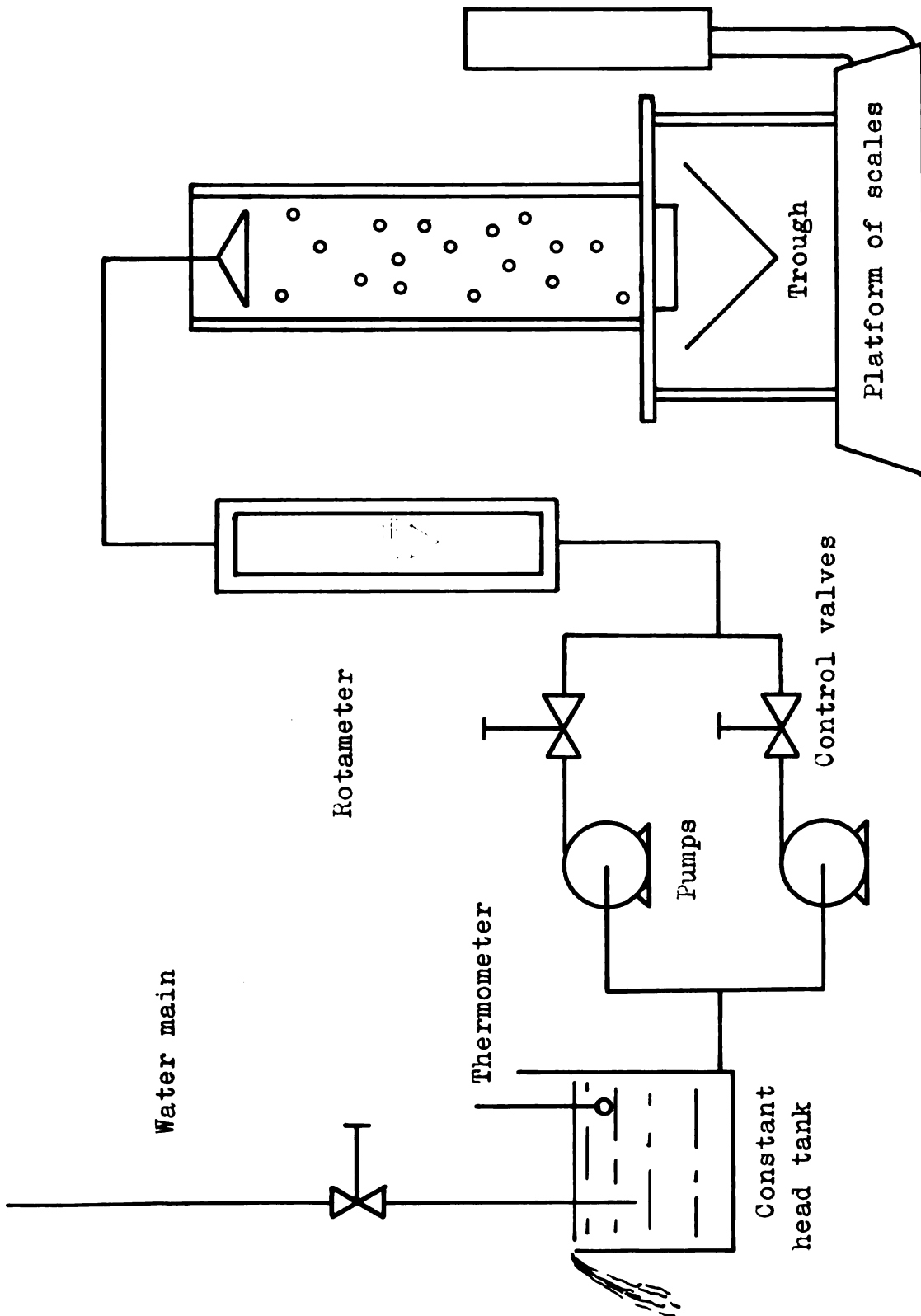


Fig. 2. Schematic Diagram of Equipment

The liquid distributor used for the 6 inch tower was a perforated plate type of shower head with 113 holes. The distributor for the smaller tower had six adjustable slotted stream guides, which were intended to provide six streams each. It was found, however, that the device was not reliable at the flow rates required when 36 streams were used. It was therefore fully opened, so that each stream guide gave one large stream instead of six small ones. This proved quite satisfactory.

The tower and its mountings rested directly upon the platform of scales. The scales used for the 6 inch column were dial type, graduated in two ounce divisions, and could therefore be read to about one-half ounce. For the small tower, a slide-weight platform balance was used. It had the disadvantage of requiring balancing whenever a weight was taken, but this was not serious. It was graduated in divisions of one gram, and with patience could be made to weigh that accurately.

The response and sensitivity of these devices were checked during the operation of the tower. When a 1/4 ounce weight was added to or removed from the larger one, the scales responded perceptibly, and within half a second had settled into the new position. The smaller

one immediately responded to a change of one gram.

Below the tower mounting a trough was placed to carry away the emerging liquid. This had its own supports, and thus did not affect readings in any way.

For each tower, a rotameter bob was designed so that the range of flow rates to be covered would occupy approximately the full rotameter scale. These were calibrated by measuring the time required to collect a certain weight of water.

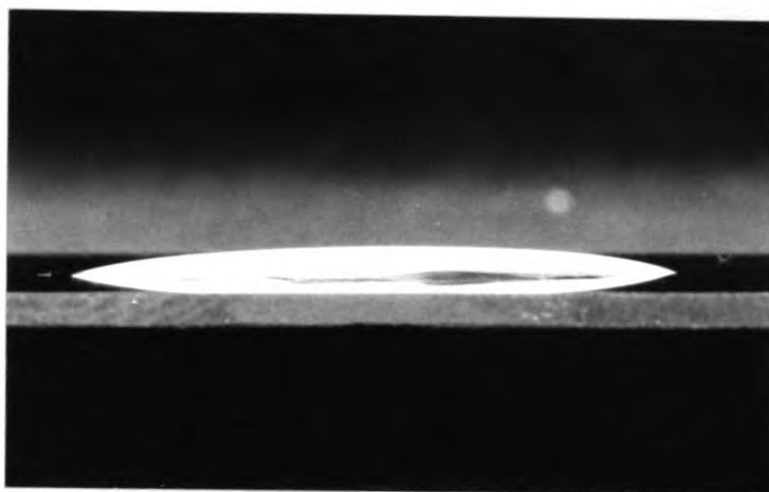
The two small pumps used were driven by synchronous motors, and gave quite constant rates of flow.

At the top of the column was placed a means of introducing small streams of a coloring agent to various locations at the top of the packing. This consisted of two small glass tubes, fitted to funnels. One of the tubes was led to the center of the tower. The other, following a suggestion by Dr. R. A. Zeleny, was given a slight bend so that its tip could be placed at the wall or $1/2$ inch in from the wall ($1/4$ inch in the small tower.) The flow rate of liquid through these leads was checked, and found to correspond to about 250 lb./hr. sq. ft. This is small in comparison with all but the lowest tower rates.

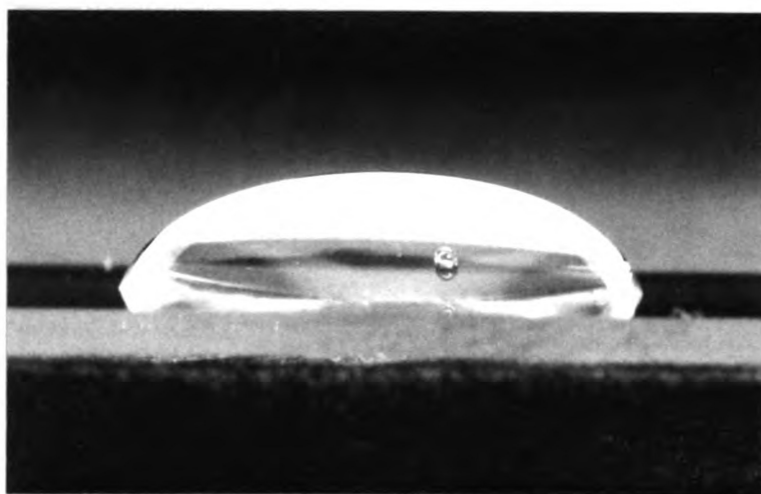
Potassium permanganate was selected as a tracer. A solution of one gram per 100 ml. may be diluted a thousandfold and yet exhibit clearly the characteristic color. It seemed entirely satisfactory in this case.

Variation in the nature of the packing surface was obtained by using, first, clean glass spheres, and second, glass spheres which had been treated with Beckman Desicote. This material is an organo-silicone which forms a bond with the glass, leaving a monolayer of organic chains extending from the surface. The effect of Desicote on the contact angle is depicted in Fig. 3, page 29. Measurement reveals that the contact angle is 14° for clean glass, and 67° for glass treated with Desicote.

11



A. Water on Clean Glass



B. Water on Desicote Treated Glass

Fig. 3. Illustration of the Effect of Desicote

PROCEDURES

In all essential respects, the experimental procedure was the same for each of the four series of tests conducted.

The first step was to level the scales and column support, so that the tower would be vertical. The tower was then bolted in place. Final adjustments were made in the location of the liquid distributor to insure that it did not touch the column. The empty tower, together with its mountings and packing support, was then weighed. It was then packed to the desired depth, and weighed again.

The weight of packing was needed in order to calculate the void fraction. Also required was the average density of the spheres. The volume of a weighed sample of spheres was determined by measuring the volume change when the sample was added to a graduated cylinder partially filled with water. In addition, a sample of each size of spheres was measured with a micrometer. Three mutually perpendicular diameters were measured in order to obtain a good indication of the average size.

Prior to packing, the spheres were washed in detergent, thoroughly rinsed, and carefully dried. Gloves were worn to avoid contact with the hands. When the spheres were to be treated with Desicote, they were, in accordance with the manufacturer's instructions, washed, rinsed, and dried. After treatment with Desicote, they were rinsed and dried again. The towers were put through identical procedures. For details on the use of Desicote, see the Appendix.

In all cases the column was packed dry in order to determine static holdup. For the 3 inch tower, the spheres were simply poured in in batches of about half a pound. To avoid chipping the spheres in the larger column, the pieces were first put into a pouch, a few pounds at a time, and lowered into the tower with a string. A second string upset the pouch, spilling the spheres.

Difficulty was encountered at first with the liquid distributor in the large tower. When first installed, it was out of the horizontal by about five degrees, and the liquid showed a marked tendency to flow down one side of the column. Correction was imperative. With the shower head horizontal, a vast improvement was evident.

In recording values of holdup, the first thing done was to check the presumption that static holdup is independent of the liquid rate. This was quickly confirmed. It was thus possible to record the weight of the tower at several flow rates without shutting down and draining the tower for each one. The importance of beginning at high flow rates to insure thorough wetting of the packing has been pointed out before,²⁶ and the precept was followed here. Following the recording of data at the last flow rate, the water was shut off, and the tower was allowed to drain. The weight was recorded after five minutes, and again after ten minutes had passed. In no case did these two values differ measurably.

To examine the patterns of flow within the tower, the tracing device (which was not in place during holdup runs) was positioned, care being taken to locate the tubes properly. Permanganate solution was allowed to run in until the pattern was established. This took about ten seconds. Then, while tracer continued to run in for another several seconds, the tower was observed, and an attempt was made to measure certain quantities.

Standing off a few feet and watching the tower while tracer was running into the center of the column,

it was possible to pick out an average level at which the permanganate appeared first to reach the wall of the column. The distance to this level from the top of the packing was recorded.

With the tracer introduced at or near the wall, a similar measure of distribution was obtained by noting the extent to which the permanganate spread out around the wall by the time it reached the bottom of the column. At the risk of seeming repetitious, let it be again stated that these observations were made after the pattern had become stabilized.

After the flow patterns had been examined at a variety of liquor rates, the water was shut off, and permanganate was allowed to run into the drained (but not dry) tower, again at the center, at the wall, and near the wall. Notes were taken on the appearance of the column.

Finally, the tower was operated briefly on diluted permanganate solution at a flow rate of about 8,000 lb./hr. sq. ft., and then permitted to drain. The tower was examined during and after the run.

During the experiments concerning flow patterns a lack of uniformity in tracer concentration across the

column was noted repeatedly. This is thought to be related to channeling, and is discussed more fully in the following chapters. No quantitative criterion of channeling has been suggested by other writers, and none has occurred to the author. He therefore is limited to verbal description.

RESULTS

Before presenting the principal results of the investigation, it is desired to record here some of the equipment characteristics which are frequently of interest.

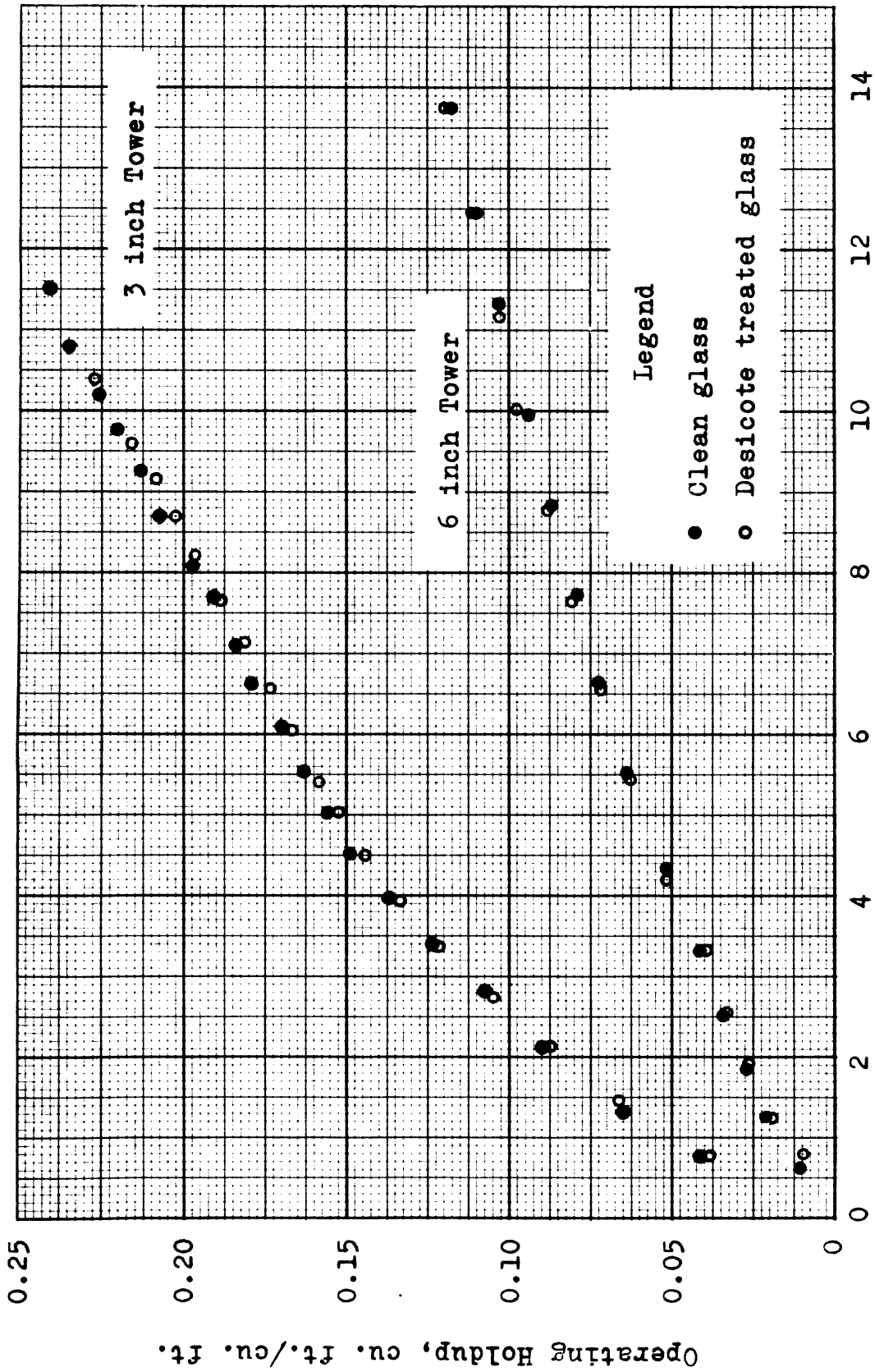
Table 1. Characteristics of Columns.

	Large Tower	Small Tower
Actual inside diameter, inches	6.125	3.0625
Actual packed height, inches	35.5	18
Average sphere diameter, inches	0.5136	0.2419
Void fraction, cu. ft./cu. ft.		
Clean glass	0.404	0.379
Desicote treated glass	0.401	0.379

No explanation is available for the small but consistent difference in void fraction for the two towers.

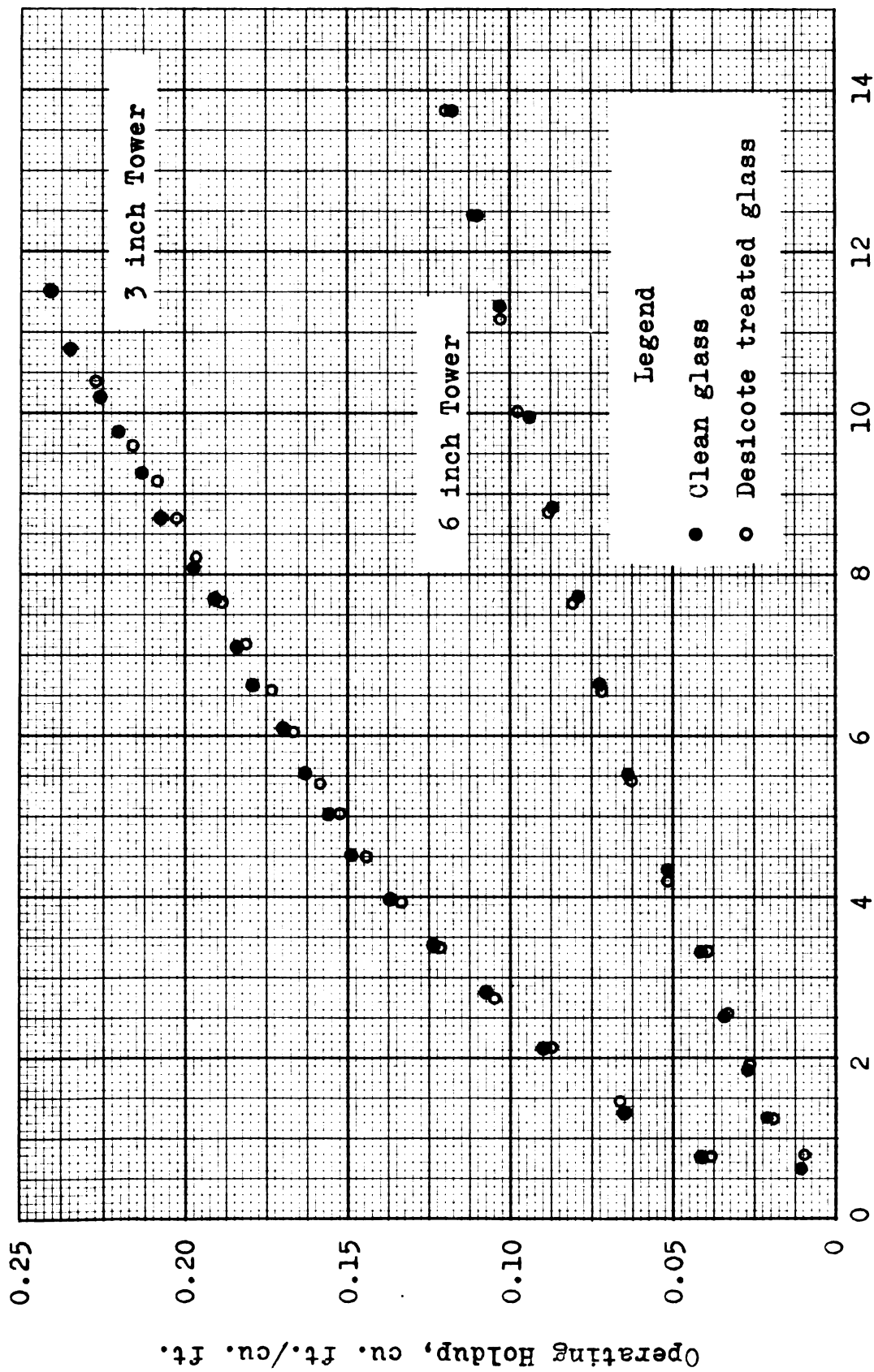
Holdup

The results of the experiments on holdup are presented in Fig. 4, page 36, and Table 2, page 37.



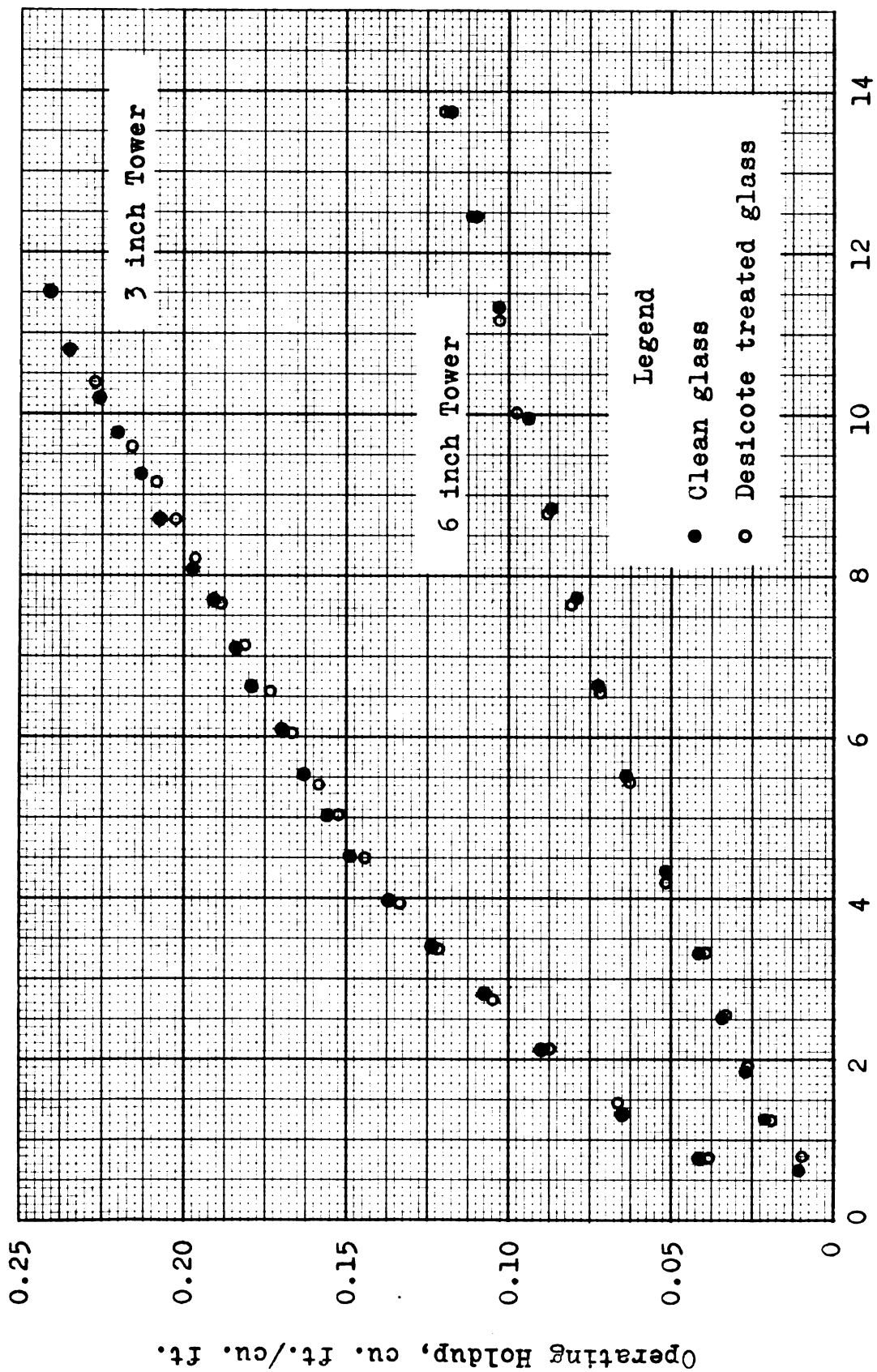
Liquid Flow Rate, Thousands of lb./hr. sq. ft.

Fig. 4. Graph of Operating Holdup Versus Liquid Flow Rate



Liquid Flow Rate, Thousands of lb./hr. sq. ft.

Fig. 4. Graph of Operating Holdup Versus Liquid Flow Rate



Liquid Flow Rate, Thousands of lb./hr. sq. ft.

Fig. 4. Graph of Operating Holdup Versus Liquid Flow Rate

1

The values used to plot the curves in Fig. 4 are tabulated in the Appendix, along with the original data.

Table 2. Static Holdup, cu. ft./cu. ft.

	Large Tower	Small Tower
Clean glass	0.0448	0.0401
Desicote treated glass	0.0400	0.0371

It will be seen that the change in liquid-solid interfacial tension results in a small change in static holdup. Operating holdup, however, cannot be said to be affected. By definition, total holdup must be altered by an amount corresponding to the change in static holdup.

Before turning to the results of other inquiries, the author wishes to report that the small tower would not accept flow rates much in excess of 10,000 lb./hr. sq. ft. If greater rates were attempted, a layer of liquid formed at the top of the packing. A corresponding point for the large tower was not encountered, although it was operated at rates well above 30,000 lb./hr. sq. ft.

Liquid Distribution and Flow Patterns

When the permanganate was introduced at the center of the top of the packing, it reached the wall of the tower approximately three inches from the top in the small column, and eleven inches from the top in the 6 inch tower. This value did not seem to be affected by liquid rate or the character of the packing surface.

When tracer was allowed to run into the drained tower, the resulting pattern was very irregular. The small tower was worse than the large one; in one case, one side of the wall was barely touched by the permanganate. In the 6 inch column, a semblance of uniformity could be seen near the bottom of the packing. The wall near the bottom was quite well covered by tracer, only a few spots being skipped. No such beginnings of order were to be noted in the small tower. The only consistent behavior observed was that liquid streams which reached the wall tended to stay there. Clean packing seemed to give slightly more uniform distribution, but since there was no quantitative means of measuring this, one cannot be certain.

Tracer introduced at the wall, with liquid

flowing, tended to move downward and around the wall, but very little evidence indicated movement across the packing. In the 6 inch column, the path of the permanganate spread out to cover about half of the tower girth by the time it reached the bottom. In the smaller tower, the tracer showed considerably more movement. It usually spread out around the entire tower perimeter before reaching the bottom, often completing the circle four or five inches above the bottom. In both columns, tracer running into the drained column for the most part simply ran down the wall, spreading out to about a third of the circumference at the bottom. There was usually no indication that tracer had traversed the interior of the packing beyond the region delineated by the wall spread; but twice, small, lone rivulets reached the wall directly across the tower only a short distance from the top.

Similar patterns resulted when the tracer stream was moved one packing diameter away from the wall. The significant change was that the tracer showed a greater tendency to move across the tower through the interior packing. Partly because of this, the total spread around the wall was slightly greater than in the case with tracer at the wall. Again, with water flowing, the small tower

showed about twice the dispersion of the larger. Typically, the permanganate would reach two-thirds of the wall circle in the large tower, and completely traverse the small one by the time it had flowed three-fourths of the way down. Running tracer into the drained tower produced effects quite similar to those obtained with tracer at the wall. A very small amount of liquid flowed into the packing, but the bulk remained near the wall.

Whether the tracer was introduced at the wall or one sphere in, no effect on the quantities measured could be ascribed to either interfacial tension or liquid velocity.

Operating the tower on a dilute permanganate solution yielded a few facts worth noting. While the 6 inch column was on stream, it could be seen clearly that the liquid was not uniformly distributed in the lower part of the tower, in spite of the fact that the distribution at the top should have been, and indeed appeared to be, excellent. After the tower had drained, two things were perceived. First, the clean packing supported a film of liquid, whereas the treated spheres were covered with very small beads of liquid. Second, the small tower had trapped in some crevices rather large

bodies of liquid. This gave it a spotty appearance not found in the larger column.

Channeling

Although the phenomena treated in this section were observed during the experiments already described, they are presented separately because the author considers them the most interesting part of the investigation. These effects were observed chiefly with tracer running in the center of the column, using both clean and treated packing.

The 6 inch tower was studied first, with a liquid flow rate near the maximum of 15,000 lb./hr. sq. ft. It was immediately noticed that the permanganate never became well mixed with the main liquid, even though it had attained distribution out to the wall in a third of the packing depth. As the liquor rate was decreased, this lack of uniformity slowly became more acute, without a perceptible change in the overall spread. Moreover, the general pattern of channeling did not change; it was rather like a hazy scene which slowly becomes more distinct.

As the flow rate approached zero, the channeling abruptly increased in severity, and the pattern began to break down. With no flow, there was no system whatever to the path of the tracer.

Attention was then turned to the small tower, and again studies were begun at the high liquid rates. Now no sign of channeling could be detected. The permanganate quickly mixed with the main stream, and the lower two-thirds of the column appeared quite uniform. As the flow rate was decreased, no channeling appeared until the flow was lowered to about 3200 lb./hr. sq. ft. At 2800 lb./hr. sq. ft. channeling was clearly present, and the behavior from there on down paralleled that of the 6 inch column.

This development suggested that channeling in the larger tower might disappear at sufficiently high flow rates. The tower was therefore reassembled, and the rotameter bob altered to provide the necessary liquor rates. Only a brief search was needed to establish that a transition did occur in the region of 21,000 to 23,000 lb./hr. sq. ft. Operating holdup in this range was about 0.153 cu. ft./cu. ft.

One last point should be brought out. The term

channeling is not used here merely to convey that liquid flowed through some of the possible openings between spheres and not others. Perhaps a better name would be "lack of mixing." A single region of conspicuously heavy or light flow was frequently several times the size of a packing piece. By some means, large streams (two to four times the diameter of a packing piece) evidently managed to form and retain their identity over appreciable distances. This the author found most interesting.

DISCUSSION OF RESULTS

Liquid Holdup

The investigation has yielded little that provokes comment. The results, for both clean and treated packing, are, with one exception, quite in line with those of earlier works.

Liquid-solid interfacial tension apparently has no significant effect on operating holdup. A modest change in static holdup is evident, and there is, of course, a similar effect on total holdup. It was expected, however, that the influence of interfacial tension would vary with liquor rate, and this anticipation has not been confirmed.

The data for 1/2 inch spheres agree well with the values obtained by Jesser and Elgin for the same packing. These gentlemen also measured holdup for 3/4 inch and 1 inch spheres, and the present data for 1/4 inch spheres fit quite well the trend for the various sizes.

One difference between the two towers is to be noted in the holdup-flow rate curves. That for the small column is very nearly linear over a considerable

range of liquor velocity, whereas that for the 6 inch column shows no linear portion. This is in opposition to the results of Jesser and Elgin, who stated that no portion of the curve is linear.

It is interesting that, although values of operating holdup for the small column are more than twice those for the larger, static holdup is slightly less. This, however, merely corroborates similar findings by Shulman et al. on various sizes of rings and saddles.

One important inference may be drawn from these data. The greater operating holdup of the 3 inch column cannot be due simply to the greater surface area of the packing. Were this true, static holdup should also be greater. It seems much more reasonable that the increase in holdup is due to the increase in the resistance to flow of the smaller spheres. This is supported by the fact that the maximum attainable liquid rate for the small column was less than 12,000 lb./hr. sq. ft., whereas the larger column could pass liquid at a rate well in excess of 30,000 lb./hr. sq. ft.

Liquid Distribution and Flow Patterns

All the evidence indicated that the smaller

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packing provides about twice the degree of distribution obtained with the 1/2 inch spheres, as measured by the rapidity of spread of the permanganate. For example, tracer introduced at the center reached the wall of the small column in about one diameter, but required two diameters in the larger tower. Similarly, permanganate introduced at or near the wall of the 6 inch column had spread out to cover a little more than half of the tower circumference upon reaching the bottom. In the 3 inch tower, however, it had spread all the way around before reaching the bottom. This, and the fact that the distribution was not affected by flow rate or the liquid-solid interfacial tension strongly suggests that spreading is a function of packing geometry and size.

Running permanganate into the center of the drained tower was similar to the experiments performed by Cairns, but his conclusions are not supported. Indeed, the reverse appeared true: it was found that the smaller packing was characterized by wider, but less uniform, distribution.

The second major inference drawn from the observations is that flow tended to proceed toward and concentrate near the wall. Consider, for example, the

large column. If tracer moves from the center to the wall in two diameters, it should cross the tower from wall to wall in four diameters, or two feet. However, such was not the case. Extrapolating the tower pattern mentally, it would take about ten feet for the tracer to accomplish the crossing. Similar arguments may be advanced for the case of the 3 inch tower. Finally, as has been said, when tracer was led into the drained tower, it was observed that streams which reached the wall nearly always stayed there. Very seldom did a stream running down the wall turn into the packing interior.

This last is not too surprising. At the point of contact between a piece of packing and the wall, the geometry does not usually favor a stream's leaving the wall. It is surprising, however, that the stream, once away from the wall, did not proceed inward nearly so rapidly as it proceeded outward. That it did not was demonstrated by the patterns obtained when the tracer was introduced one particle diameter away from the wall.

Channeling

It was hoped that the region of transition from

channeling to well-mixed flow might be related to other variables. Liquor velocity alone is not a criterion, because the values for the two towers differed immensely. Values of operating holdup corresponded more closely -- 0.153 for the 6 inch column, 0.115 for the 3 inch. This is close enough to suggest a connection, but is still a difference of 30%, and is not in itself considered satisfactory. It appears, then, that the transition region cannot be characterized on the basis of information presently available.

That the same channel patterns were observed at all flow rates between the transition point and the very low rates at which the patterns were disrupted suggests that, at least in this region, the phenomenon of channeling depends upon the packing geometry more than the properties and flow rate of the liquid. The disintegration of the patterns at very low liquor velocities is probably related to the fact that, at these rates, the stream of permanganate was no longer small in comparison with the overall stream.

The most important consequence of the presence of channeling is that it provides grounds for challenging the validity of earlier work on liquid distribution.

This chapter will be concluded with the exposition of such an argument.

Ideal liquid flow in a packed bed would be characterized by the presence of an individual stream through each space, or over each possible path, between pieces, all streams being of equal size. (At high rates these streams would, of course, tend to run together.) This is the situation which would provide the greatest liquid surface area. Departure from the ideal might consist of one or more of the following effects.

- 1) There might be flow through all possible paths, but with variation in stream size from path to path.

- 2) Substantial segments of the tower cross-section might experience conspicuously heavy or light flow.

- 3) Several streams might coalesce into a single large stream, completely surrounding several pieces of packing. This was observed by the author.

- 4) Particularly at low flow rates, many of the possible paths might not bear flow at all.

Now if liquid distribution is measured by collecting the effluent liquid in a segmented receiver, these

effects might easily go undetected. Each segment will automatically average the streams within the area which it subtends; and if a large stream is split by a divider, the lack of uniformity will not be nearly so apparent. The work of Baker, Chilton, and Vernon has evidently been accepted by most writers as demonstrating the uniformity of flow. However, they used a receiver divided into only four annular segments, and it scarcely seems likely that moderate channeling effects would be evident in the results.

Finally, it might be noted that although Baker et al. were only concerned with the general rate of spreading from the liquid distributors above the packing, their article has for twenty years been adduced as evidence of uniform flow under any conditions so long as the tower diameter is at least eight times that of the packing. This is unwarranted.

CONCLUSION

By way of summary, the principal conclusions based on this investigation will be collected here.

1) The nature of the solid surface has not been shown to affect any aspect of tower operation except static holdup.

2) The size of the equipment affects static and operating holdup, and the degree and uniformity of liquid distribution.

3) There is evidence that the liquid flowing down through a packed column tends to move toward the wall.

4) There exists, for a packed tower, a small region of liquid flow rate, above which channeling is absent, and below which it is pronounced.

5) Over a wide range of flow rates the channeling patterns do not change appreciably.

Although the experiments were designed primarily to examine the effects of liquid-solid interfacial tension, the author now feels that their chief value has been the quite unexpected results on flow patterns and channeling. He would therefore like to close this thesis

by suggesting a few topics for study for those who may be interested.

One thing which is badly needed is a criterion of channeling. This should take into consideration the number of individual streams per unit area of tower section, and the variation in the size of the streams, as well as their location. If such a quantitative measure could be defined and practically applied, it should certainly lead to some interesting results.

With or without such a criterion, there is much that could be done in the matter of pure information. A careful search of the literature has failed to reveal a single example of the study of individual streams within the packing. What might be done is to repeat the experiments of Mayo, Hunter, and Nash, with certain refinements. First, the packing should be hand stacked in a definite pattern, so that the effects of several variables might be studied. Second, the packing should be removed piece by piece after the run, and the individual streams traced through successive layers of packing. Variations of this are possible, too. One might trace the dispersion from a point source (essentially what was done crudely in the present studies) with and without overall

liquid flow. The location of the point source could be varied. The type, size, and pattern of packing might be changed. Wall effects should be examined. The list is long.

The author is persuaded that little is to be learned by studying the distribution of the liquid as it emerges from the bottom of the tower, and he has similar feelings about further investigations on holdup. These quantities are gross aspects of the column as a whole, and give no indication of internal processes. Visual study using glass towers only gives information concerning behavior near the wall, and it is not at all certain that phenomena in the center of the column are similar. It is hoped that this material will induce the curious to direct their attention to the interior of the packing. That is the only place where the nature of the liquid flow can be determined.



APPENDIX

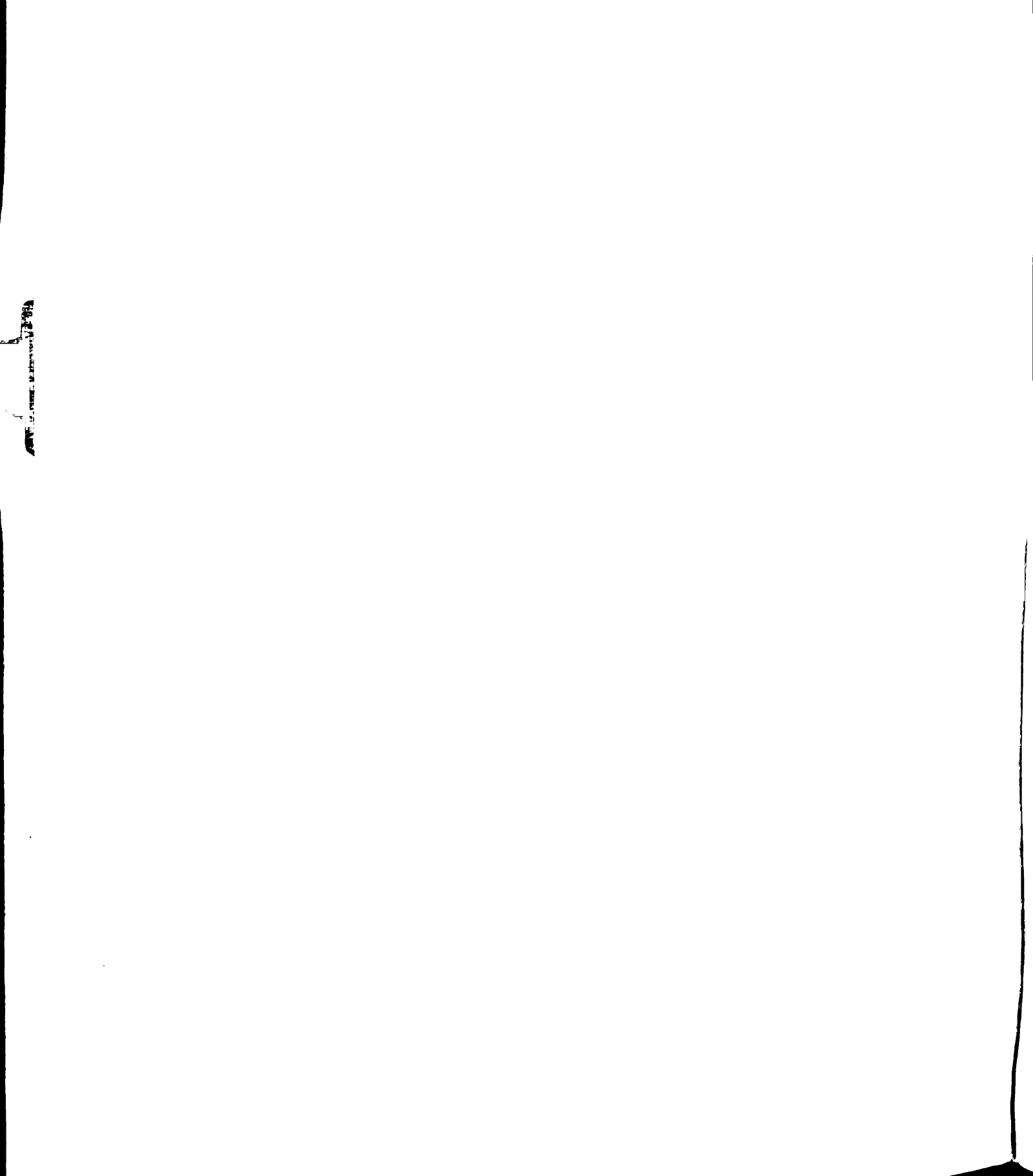


Table 3. Data Taken on 6 Inch Tower, Clean Glass Packing

Flow Rate lb./hr. sq. ft.	Total Weight of Tower and Contents		Operating Holdup cu. ft./cu. ft.
	lb.	oz.	
Empty Tower	34	2	--
Dry Tower and Packing	89	11 $\frac{1}{2}$	--
13,750	95	15	0.1176
12,460	95	10 $\frac{1}{2}$	0.1102
11,310	95	6	0.1029
9,960	95	0 $\frac{1}{2}$	0.0939
8,840	94	12	0.0866
7,730	94	7 $\frac{1}{2}$	0.0791
6,640	94	3 $\frac{1}{2}$	0.0726
5,510	93	14	0.0637
4,340	93	6 $\frac{1}{2}$	0.0515
3,320	93	0 $\frac{1}{2}$	0.0417
2,510	92	12	0.0343
1,860	92	7 $\frac{1}{2}$	0.0270
1,260	92	4	0.0212
620	91	13 $\frac{1}{2}$	0.0106
Drained 5 min.	91	7	--
Drained 10 min.	91	7	--

Table 4. Data Taken on 6 Inch Tower, Desicote Treated Packing

Flow Rate lb./hr. sq. ft.	Total Weight of Tower & Contents		Operating Holdup cu. ft./cu. ft.
	lb.	oz.	
Empty Tower	34	2	--
Dry Tower & Packing	90	2	--
13,750	96	3	0.1183
12,460	95	14 $\frac{1}{2}$	0.1110
11,160	95	9 $\frac{1}{2}$	0.1029
10,010	95	6	0.0972
8,780	95	0 $\frac{1}{2}$	0.0881
7,650	94	12	0.0808
6,550	94	6 $\frac{1}{2}$	0.0718
5,450	94	1	0.0628
4,190	93	10	0.0514
3,330	93	4	0.0416
2,540	92	15	0.0334
1,920	92	10 $\frac{1}{2}$	0.0261
1,240	92	6 $\frac{1}{2}$	0.0196
800	92	0 $\frac{1}{2}$	0.0098
Drained 5 min.	91	10 $\frac{1}{2}$	--
Drained 10 min.	91	10 $\frac{1}{2}$	--

Notes on Tables 5 and 6

In order to obtain greater accuracy from the balance used with the small column, a separate determination of the packing weight was made first. Then the balance was set, using weights and an auxiliary slider, so that it was just short of balancing. All balancing during the runs was then done with the marked slide-weights.

The weights of the packing for the two runs were as follows:

Clean glass spheres	3414 grams
Desicote treated spheres	3415 grams

Table 5. Data Taken on 3 Inch Tower, Clean Glass Packing

Flow Rate lb./hr. sq. ft.	Balance Reading, Grams	Operating Holdup cu. ft./cu. ft.
Dry Tower & Packing	11	--
11,520	623	0.2414
10,830	607	0.2345
10,200	588	0.2257
9,760	578	0.2209
9,260	560	0.2127
8,700	549	0.2076
8,080	526	0.1970
7,700	519	0.1913
7,100	497	0.1837
6,640	487	0.1791
6,100	466	0.1697
5,540	452	0.1630
5,030	437	0.1563
4,510	422	0.1493
3,990	396	0.1373
3,400	368	0.1242
2,820	333	0.1073
2,130	294	0.0903
1,310	239	0.0649
770	193	0.0439
Drained 5 min.	98	--
Drained 10 min.	98	--

Table 6. Data Taken on 3 Inch Tower, Desicote Treated Packing

Flow Rate lb./hr. sq. ft.	Balance Reading, Grams	Operating Holdup cu. ft./cu. ft.
Dry Tower & Packing	8	--
10,400	583	0.2273
9,600	557	0.2157
9,160	542	0.2085
8,700	529	0.2022
8,220	516	0.1967
7,660	499	0.1884
7,130	483	0.1812
6,570	466	0.1737
6,040	452	0.1670
5,400	433	0.1583
5,030	420	0.1522
4,500	403	0.1445
3,940	379	0.1333
3,360	353	0.1216
2,740	318	0.1053
2,130	280	0.0878
1,470	233	0.0663
780	173	0.0388
Drained 5 min.	89	--
Drained 10 min.	89	--

Table 7. Typical Measurements on Flow Patterns Formed by Permanganate Tracer

	6 Inch Column	3 Inch Column
Vertical distance to reach wall from center	11 in.	3 in.
Spread from wall	Half of tower circumference at bottom	Complete encirclement 2 in. above bottom
Spread from one particle dia. away from wall	Two-thirds of circumference at bottom	Encirclement 5 in. above bottom
Channeling transition range, lb./hr. sq. ft.	2800-3200	21,000-23,000
Operating holdup at transition, cu. ft./cu. ft.	0.153	0.115

Table 8. Data to Determine the Average Density of the Spheres

	1/2 Inch Spheres	1/4 Inch Spheres
Weight, gm.	2513	129.54
Volume, ml.	1016	51.4

Notes on the Use of Desicote

This material is not intended to supplant the manufacturer's instructions, but rather to clarify a few points.

The directions state that Desicote may be diluted with acetone. What is not stated is that, when this is done, a white precipitate forms. This precipitate is thought to be finely divided silica. The mixture may be used, precipitate and all, but the precipitate tends to coat the surface of glassware, and is rather troublesome to remove.

Better results are obtained by allowing the precipitate to settle and decanting the clear liquid. Diluted solutions of five, ten, and twenty parts of acetone to one of Desicote were tried, and all seemed satisfactory. A ten to one dilution was used in most of the author's work.

The instructions state also that one normal solution of sodium hydroxide, hot or cold, remove Desicote in several minutes. The author recommends several hours in hot solution.

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