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Tracy T. Word

(M. S. Thesis)

September 1, 1961

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ABSTRACT

A 2-ft-i.d. packed gas-absorption column was constructed for the purpose of obtaining longitudinal mixing data on both the liquid and gas phases under nonabsorbing operating conditions. The equipment is described in detail and the operating procedure is given. The calculation of the liquid-phase Peclet number of an early run is reported.

I. INTRODUCTION

General

Individual particles of a fluid stream passing through a packed bed do not all have the same residence time in the bed. In other words, if a given group of fluid packets enters a bed at the same time, they will not leave the bed simultaneously. Some particles will appear to have a velocity greater than the average stream velocity, and others will appear to travel more slowly than the average. This phenomenon is commonly referred to as longitudinal mixing or longitudinal dispersion.

In industrial equipment, when a nonhomogeneous fluid passes through a packed bed, longitudinal mixing reduces any axial concentration gradient that would exist in the bed if plug flow were realized. This becomes important when, for instance, in fixed-bed catalytic reactors the point concentrations of the reactants within the bed are reduced by longitudinal dispersion, with a corresponding decrease in the reaction rate.

Longitudinal dispersion also occurs in two-phase countercurrent systems such as absorption and extraction columns. In this case the concentration gradients in both phases are lowered by longitudinal dispersion, and the driving force for mass transfer may be decreased considerably. Therefore, a knowledge of the extent of longitudinal mixing is necessary for the precise design of any fixed-bed apparatus.

One way of presenting longitudinal-mixing data is to give values of the dimensionless Peclet number, Ul/E, determined for the system under consideration. Here U is a characteristic velocity of the fluid, l is a characteristic length of the system, such as the diameter of a packing particle or the length of the packed bed, and E is the eddy dispersion coefficient. Miyauchi[1] and other workers have shown how HTU and NTU vary with the Peclet number in extraction columns.

Although radial mixing had been studied for some time, the first axial-mixing data reported in the literature were published by Danckwerts in 1953.[2] Other workers were soon to follow suit.

Kramers and Alberda,[3] Carberry and Bretton,[4] Jacques and Vermeulen,[5] and Ebach and White[6] have determined values of the Peclet number for water flowing through beds of various packings over a wide range of Reynolds numbers. McHenry and Wilhelm,[7] and Robinson,[8] were among those who determined Peclet numbers for gas streams flowing through packed beds. These data indicate that the Peclet number in single-phase flow (a) is a function of Reynolds number, (b) has different values in laminar and turbulent flow, and (c) passes through a transition region with respect to Reynolds number, corresponding to the change from laminary to turbulent flow in the bed.

Until recently there has been comparatively little work done on longitudinal dispersion in two-phase systems. In the paper referred to above, Jacques and Vermeulen used a water-kerosene system to determine Peclet numbers in both phases in a packed extraction column. They found the Peclet numbers in two-phase flow to be equal to or less than the corresponding single-phase values in laminar flow. The two-phase Peclet numbers obtained can be correlated in terms of the flow rates in the two phases. In a later report Jacques et al.[9] present additional data that agree with their previous results for two-phase systems.

De Maria and White[10] studied mixing in an air stream flowing through beds of various sizes of raschig rings irrigated by water. They found that the axial Peclet number of the gas flowing through wet packing was always less than that for dry packing.

Recently Stemerding[11] reported data on axial diffusion coefficients of the continuous water phase determined in an air-water system passing through a column packed with 13-mm raschig rings. He found the axial diffusion coefficient rather insensitive to liquid flow rate, and that it passed through a maximum value when plotted against increasing gas flow rate. The coefficients determined were in the range of 8 to 15 cm^2/sec.

Theoretical

A method of determining the Peclet number for flow systems, independent of mass-transfer behavior, is to analyze the breakthrough curve of a tracer element injected into the stream under consideration. This requires that the tracer be injected in the form of a known mathematical function (i.e., step, pulse, sine, etc.), and that the concentration of the tracer be determined as a function of time at a known distance downstream. The Peclet number can be evaluated by substituting the properties of the experimental breakthrough curve of tracer concentration into a theoretical equation representing the concentration of tracer as a function of time at the sample point.

Three models for deriving the theoretical shape of the breakthrough curve are the random-walk model, the diffusion model, and the cell-mixing model. The concentration profiles for these models converge for large values (greater than 10) of the ratio of the length of the bed to the longitudinal mixing length. This ratio, which is also the number of mixing lengths in the bed, will be referred to as N. The longitudinal mixing length is of the order of one packing particle diameter and, therefore, the values of N at the sampling points in the column described in this report should be about 30 or 60, depending on the sampling point under consideration. This means that one should obtain the same Peclet numbers when evaluating the data from this column by using equations derived from any one of the three models listed above. Equations based on the random-walk model were used in evaluating the data in this work, and these equations are the only ones that will be discussed now.

Random-Walk Model

A fluid packet moving through a packed bed must continually change its speed and direction as it encounters successive packing particles, and the packet may be thought of as "randomly walking" through the bed. As was mentioned before, fluid packets entering a packed bed simultaneously do not pass an arbitrary point downstream

simultaneously. The probability that a given packet will be a given number of mixing lengths from the entrance to the bed after a certain period of time can be expressed mathematically. Following Einstein, equations developed by Jacques et al.[9] give this probability in terms of dimensionless quantities. If $N = h/H$ is the number of mixing lengths, where h is the bed length and H is the longitudinal mixing length, t is the time the packet has been in the bed, U is the characteristic velocity, $T = Ut/H$ is the dimensionless time, and n is the number of jumps in the random walk, the probability is

$$P_T(N) = \sum_{n=0}^{n=\infty} [\exp(-N-T)] \cdot \frac{N^n}{n!} \cdot \frac{T^n}{n!} \quad . \tag{1}$$

By using this equation, the above authors obtain an expression for the relative concentration at a point within the bed when a step-function input of constant concentration, C_0, has taken place at $N = 0$ and $T = 0$. If C is the point concentration and C_0 is the concentration of the step function at $N = 0$, then we have

$$x = \frac{C}{C_0} = \int_0^{T'} \exp(-N-T)\, I_0(2\sqrt{NT})\, dT \quad . \tag{2}$$

The slope of a breakthrough curve based on relative concentrations may be obtained by differentiating Eq. (2) with respect to T/N:

$$s = \frac{\partial x}{\partial (T/N)} = N \left(\frac{\partial x}{\partial T} \right) \quad . \tag{3}$$

This can be written

$$s = \frac{N I_0 (2\sqrt{NT})}{\exp (\sqrt{N} - \sqrt{T})^2 \exp (2\sqrt{NT})} \quad . \tag{4}$$

It is convenient to determine a breakthrough curve slope, s', relative to a time scale of $t/t_{(x = 0.5)}$. This is related to s as follows:

$$s' = \frac{dx}{d(t/t_{0.5})} = \frac{T_{(x=0.5)}}{N} \cdot s . \qquad (5)$$

Expanding terms in s we have

$$s' = \frac{\sqrt{N}}{2\sqrt{\pi}} \left(1 + \frac{3}{8N} + \frac{1}{16N^2} + \cdots\right) . \qquad (6)$$

The solution of this equation for N has been found to be

$$N = 4\pi(s')^2 - 0.80, \qquad (7)$$

to a close approximation.[9]

As was stated earlier, $N = h/H$ is the number of mixing lengths between the tracer injection point and the sampling point. The eddy dispersion coefficient may be defined as $E = UH$, and therefore

$$N = h/H = hU/E, \qquad (8)$$

which is the column Peclet number. For comparison with results calculated from diffusion theory equations, we can determine the packing Peclet number

$$N_{Pe} = Nd_p/h = d_pU/E, \qquad (9)$$

where d_p is the packing particle diameter.

Statement of the Problem

Data on the longitudinal mixing in two-phase systems flowing through packed beds are not as plentiful as those for single-phase flow. Therefore it has seemed desirable to obtain longitudinal-mixing data on both phases of a packed liquid-gas absorption column. The purpose of the work described in this report was to construct an absorption column from which such data could be obtained.

In order to make the data obtained more accurate and useful, the column was to have the following characteristics:

a. The packing material could be changed with a minimum of difficulty, so that mixing data for several types and sizes of packing could be taken.

b. Injection and sampling of the tracer element would be done within

the packed bed, so that the data would not have to be corrected for end effects.

c. The column would be of near-industrial size.

II. DESCRIPTION OF APPARATUS

The experimental system consisted of a cylindrical steel column, a positive-displacement blower, a liquid feed pump, liquid- and gas-tracer injection systems, and the necessary indicating, controlling, and recording devices. The interrelationship of these components is indicated in the schematic diagram of the system shown in Fig. 1. A detailed description of the components is given below.

The blower and pump in this system were those used in previous experiments by Brown.[12]

Packed Column

The column was constructed in three flanged cylindrical sections, each with a 2-ft i. d. The walls of these sections were formed from 1/8-in. mild steel sheet, and the flanges were cut from 1-1/2-in. steel plate. The interior surfaces of all the sections were coated with epoxy resin to prevent rusting, and the exterior surfaces were painted a dull gray. Figure 2 is a photograph of the assembled column with its accessory equipment, and Fig. 3 is the column assembly drawing.

Lower section

The lower section, which served as a liquid feed reservoir, was 4 ft tall. A sight glass indicated the liquid level in this section. Attached to the floor of the section was the return line to the liquid feed pump. A screen was fitted over the liquid outlet in the lower section to prevent bits of solid material from entering the return line. Two 2-in. welding-neck flanges were attached to the wall of this section. One flange was fitted with a valve and was used as a liquid supply inlet, and the other was used as a passageway for a tube which carried the gas tracer element into the column.

Packed section

The next section was 6 ft high and contained the packing material. Between the packed section and the lower section was a packing support, that consisted of a piece of Irving Industrial Grating, type CC, welded inside a 2-ft i. d. flange. This grating was chosen because it had great

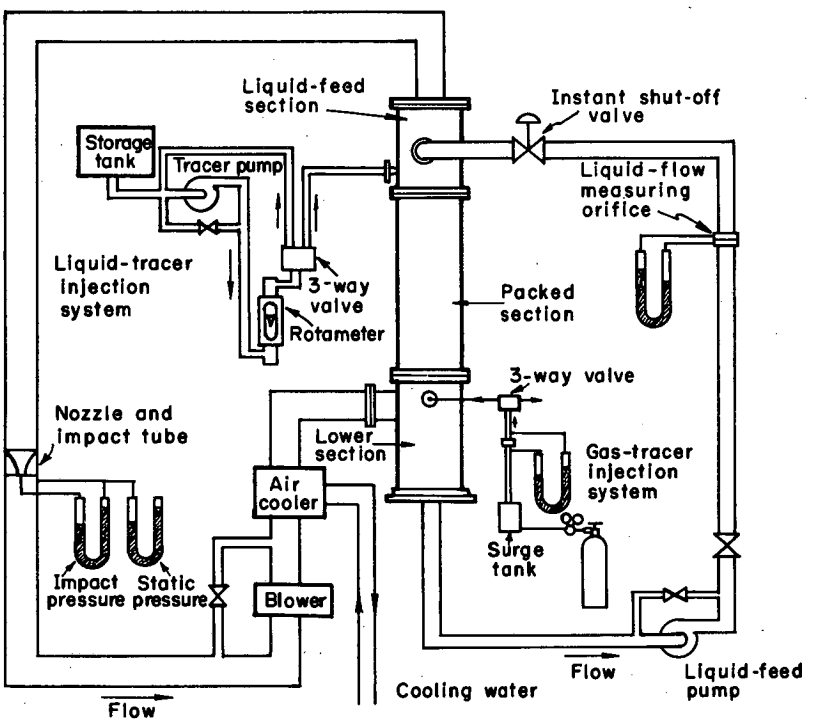

Fig. 1. Schematic diagram of the experimental apparatus.

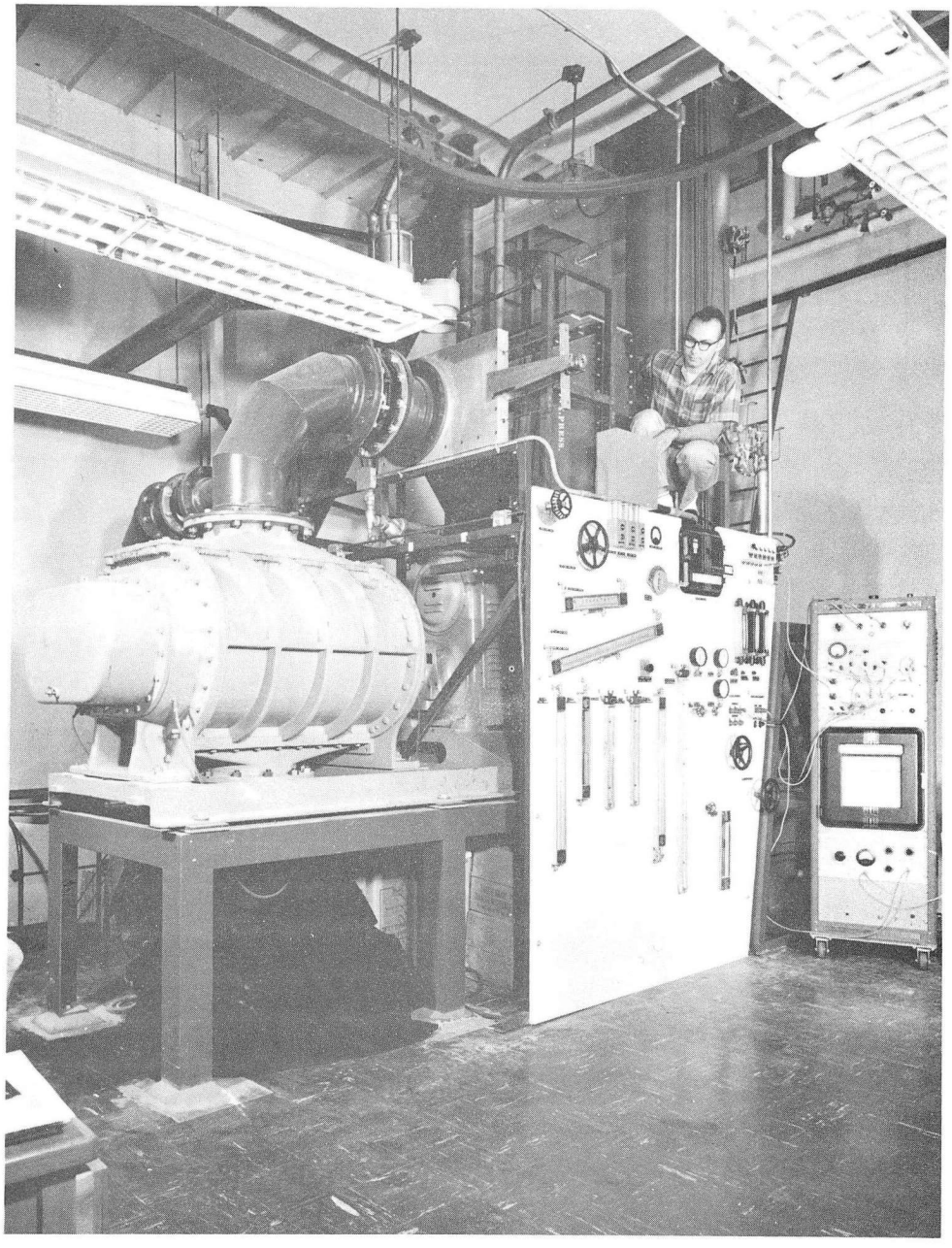

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Fig. 2. Over-all view of the experimental apparatus.

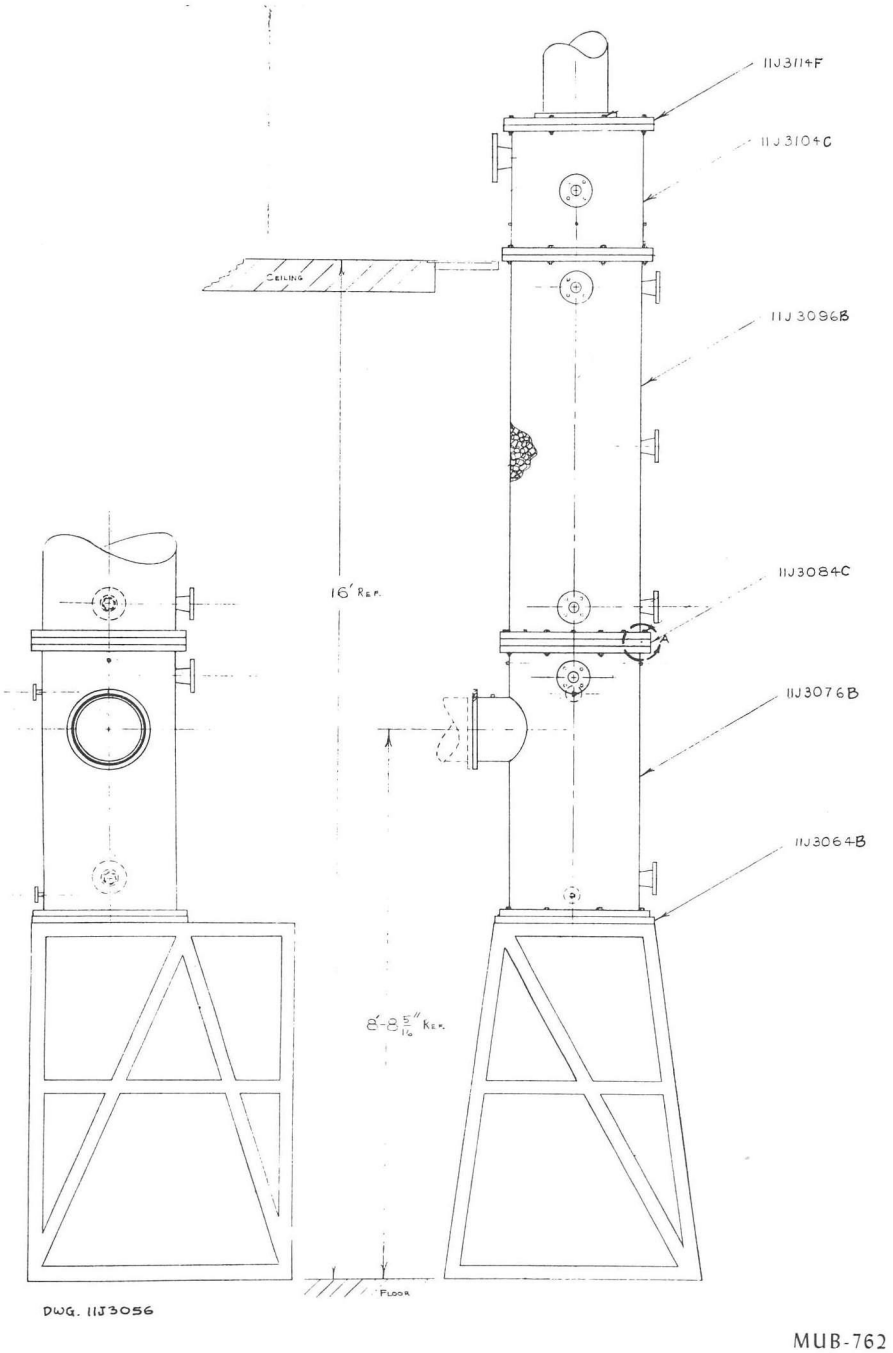


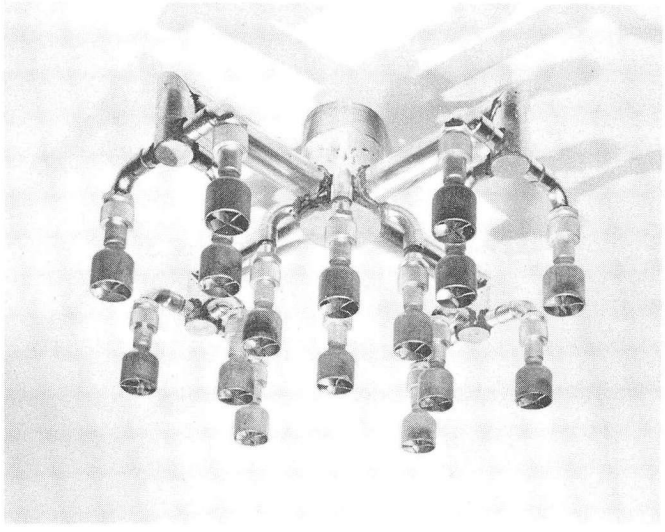
Fig. 3. Assembly drawing of packed column.

strength (415 lb/ft^2 allowable safe load) and a high percentage of free area (83%). The packing support flange had eight bolt holes and eight tapped holes, whereas the bottom flange of the packed section had sixteen bolt holes. When the column was assembled, eight bolts were passed through the bottom flange of the packed section, the packing support flange, and the top flange of the lower section, for binding these three flanges together. Eight bolts were also passed through the bolt holes in the packed-section flange and into the tapped holes of the packing support flange, for fastening the packing support to the packed section. When it was desired to change packing, the eight bolts holding all three flanges together were removed, and the packed section, packing material, and the packing support flange were removed as a unit by a crane. The packed section was then placed in a bin, and the eight bolts fastening the packing support flange to the packed section were removed. When the packed section was raised again, the packing material dropped out on top of the packing support in the bin.

Pairs of 2-in. welding-neck flanges were placed at points 6 in., 36 in., and 66 in. from the bottom flange of the packed section to permit liquid- and gas-sampling probes to be inserted into the column. These probes are discussed later, in the description of the tracer injecting and sampling systems.

Water-feed section

The top, or water-feed, section was about 3 ft high. This section contained the liquid distributor (Fig. 4). This distributor consisted of a number of brass pipes that supplied water to 16 copper bell reducers. The four feed lines to the bell reducers were 1-1/2-in. brass pipes that were soldered at right angles to a 3-in. brass pipe. Each feeder extended 8-1/2 in. from the center of the 3-in. pipe. A splash plate that resembled a four-bladed propeller was soldered in the mouth of each bell reduced to break up the water stream in the bell reducer. Leva[13] states that the number of distribution points necessary



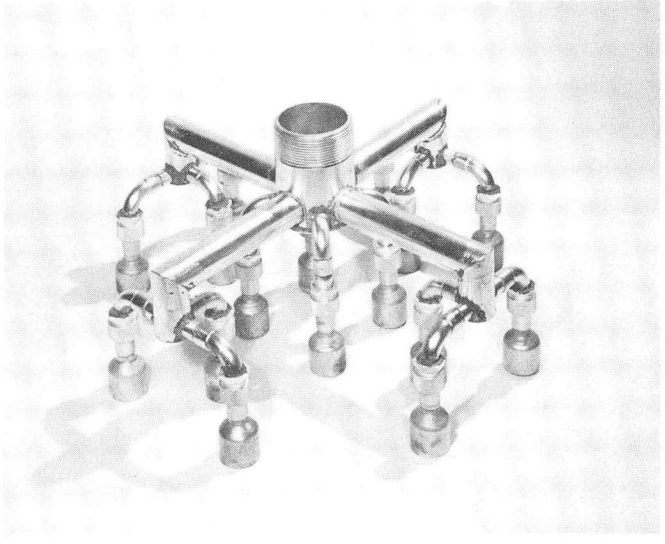


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Fig. 4. Liquid-feed distributor.

for adequate distribution in a packed column is $(D/6)^2$, where D is the inside diameter of the column in inches. On this basis the distributor was designed to have $(24/6)^2$, or sixteen, distribution points. This particular design was visually observed to give even liquid distribution at both high and low liquid flow rates, and at the same time resulted in a large free area to reduce liquid entrainment at large gas flowrates. Another important feature of this distributor was that it resulted in a low pressure drop at high liquid flowrates. This was necessary because the liquid feed pump was only capable of low fluid heads.

The total height of the column was about 13 ft. The column was mounted on a stand about 6 ft tall; this made it necessary to extend the upper part of the column through the ceiling and into the room above, as may be seen in Fig. 5.

The water feed section was fitted with one 4-in. and one 2-in. welding-neck flange. A 3-1/2-in. o.d. copper pipe that brought feed water to the liquid distributor was inserted through the 4-in. flange. The liquid-tracer feed tube was inserted through the 2-in. flange. This feed tube was connected to the liquid injection system which is discussed later in this section. The air-outlet plate, which covered the water-feed section, had four lucite windows. A waterproof light was mounted inside the column, and by looking through the windows the operation of the liquid distributor could be observed.

Gas Circulation System

Blower

The blower was a Sutorbilt model-1436, driven by a U. S. Electrical Motors 20-hp motor. The air leaving the blower first passed through an air cooler that removed the heat of compression of the blower and the heat supplied by the steam heating coils. It then passed into the lower section of the column, up through the packed section and the water-feed section, and entered the return-air duct which carried it back to the blower. Since the air circulated in a closed

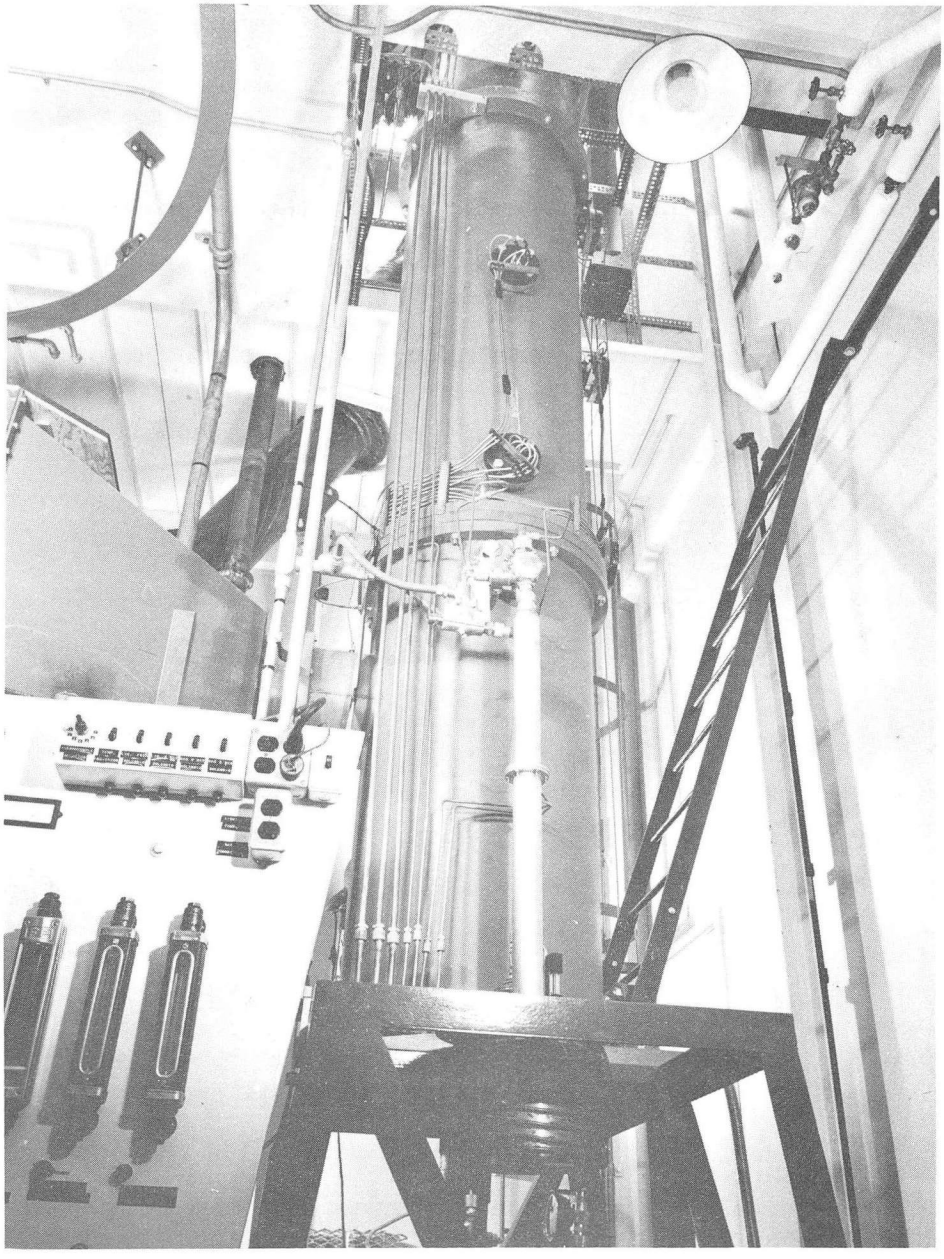

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Fig. 5. Packed column (looking upward).

system, it soon became saturated with liquid. The steam heating coils referred to above were located in the section of the air duct immediately above the column. These coils heated the gas to approximately 100°F and prevented condensation in the return duct, which might lead to rust formation in the blower that would cause contact between the rotating lobes and the case.

Air-flow metering nozzle

The return-air duct was fitted with an impact tube mounted in the center of a nozzle throat. Bean et al.[14] have shown that the velocity is essentially uniform over the entire cross section. Thus the velocity determined from the impact-tube reading times the cross-sectional area of the nozzle gave the volumetric flow of gas through the test section. The impact tube was used because its alignment in the direction of gas flow is not as critical as the alignment of a pitot tube. The nozzle used in this equipment had the dimensions of Bean's C-2 nozzle;[14] its cylindrical throat was 5.003 in. in diameter. The air flow through the column was controlled by adjusting the blower speed and the butterfly valve in the blower bypass duct.

Liquid Circulation System

The liquid feed pump was an Ingersoll-Rand Motor-Pump, model 4RVL, equipped with an 8-in. -diam impeller. The liquid passed upward from this pump through a 4-in. copper pipe to the liquid distributor in the water-feed section of the column.

Just before the liquid entered the column, it passed through a Johnson Service model V-95 reverse-acting pneumatic 4-in. valve. This two-position valve was controlled by a Johnson Service V-20 three-way solenoid valve. When the solenoid was de-energized, 25 psi air was applied to the valve diaphragm and held the valve in the open position. The energizing of the solenoid cut off the air pressure, and allowed the diaphragm cavity to exhaust to the atmosphere; then the valve rapidly closed under the pressure of its spring. By rapidly closing the 4-in. valve, the liquid flow was stopped, and the operating

liquid holdup was determined by noticing the increase of the liquid level in the lower section of the column. Located in the 3.89-in. i.d. copper pipe was a standard 2-15/16-in. ASME flow-measuring orifice that was fitted with flange taps (specifications are given in Ref. 15).

Liquid-Tracer Injection System

The liquid-tracer injection system consisted of a stainless steel storage tank, an Eastern Industries model-F centrifugal pump, two Fisher-Porter Flowrators, a Skinner model-L3DB5150XP three-way solenoid valve, a spider injection manifold, and the necessary copper lines. A schematic representation of this system is shown in Fig. 6.

Flow meters

After leaving the storage tank the 1N sodium nitrate tracer solution passed through the pump, and then to one of the two rotameters. The larger rotameter was equipped with a Flowrator tube No. B5, with a capacity of 1.96 gpm of water at 70°F. The smaller one had a No. 2F tube, with a capacity of .21 gpm. The combination of the two meters permitted almost a 100-fold range in flow measurement.

Solenoid valve

After leaving the rotameters the tracer encountered the three-way solenoid valve. If the solenoid was de-energized the tracer returned to the suction side of the pump, but if the solenoid was energized the tracer was diverted into the column via the spider injection manifold.

Injection manifold

The spider injection manifold, shown in Fig. 7, consisted of 16 1/8-in. -i.d. stainless steel tubes connected to a 3-in. -diam by 1-1/2-in. -deep copper box, much as the legs of a spider are attached to its body. The longest arms extended 9-1/2 in. from the side of the box, and all the arms extended 6 in. deep into the packing. The ends of the stainless tubes were fitted with 1/4-in. copper tubing tees, so that when the tracer was injected it would not have a velocity component

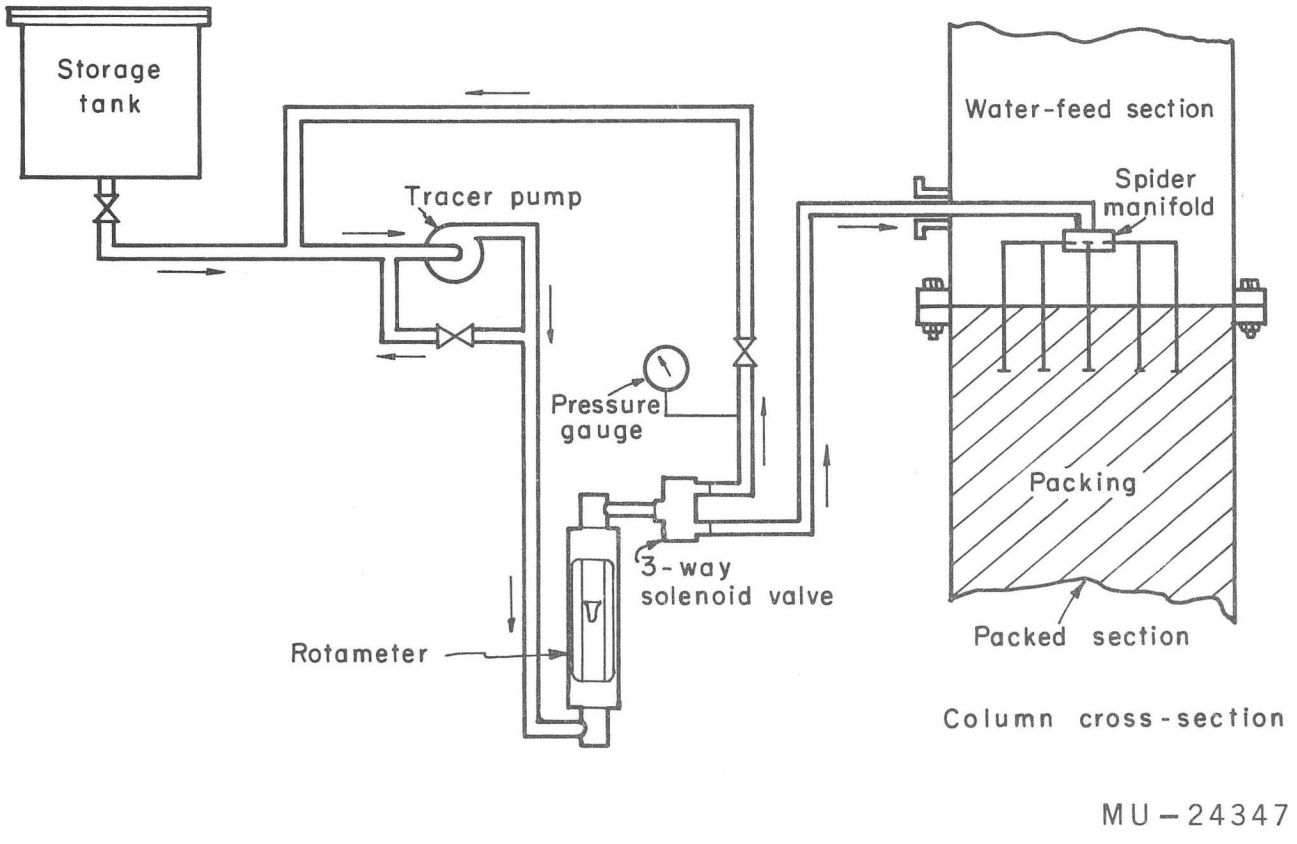

Fig. 6. Schematic diagram of the liquid-tracer injection system.

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Fig. 7. Spider injection manifold.

along the length of the column and thus affect longitudinal dispersion in the liquid. Stainless tubes with small inside diameters were chosen so that the surface tension of the tracer would keep the manifold full even when it was not in use. Tracer flow control was maintained by means of valves installed close to the rotameters and by a valve located in the bypass line around the pump.

Gas-Tracer Injection System

The gas-tracer injection system consisted of four 215-scf helium cylinders connected to a surge tank which in turn fed a log-type injection manifold. A schematic diagram of the system is shown in Fig. 8 and a photograph of the log manifold is shown in Fig. 9.

Helium supply system

The helium cylinders were connected to a National Welding Equipment Company manifold which in turn was connected to a National model-765 pressure regulator. This regulator fed helium at 60 psig to a Matheson model-M2 "pancake" regulator which was rated at 15 scfm at discharge pressures of 0 to 250 psig. The discharge side of the Matheson regulator was connected to the cylindrical stainless steel surge tank. This tank was 10 in. diam by 10 in. deep, and had a volume of about 800 $in.^3$—more than 8 times the volume of the log injection manifold. The purpose of this tank was to provide a reservoir of helium at the expected operating pressure of the log manifold.

Helium metering orifice

Leading away from the top of the surge tank was a 2-in. -i. d. brass pipe which contained a 1/4-in. sharp-edged orifice. This orifice met the design criterion specified by Stearns et al.[16]

Solenoid valves

Above the orifice the 2-in. pipe mated with the normally closed port of a Skinner model-L3ADB5150 three-way solenoid valve with 3/4-in. orifices, whose operation is described below. A Skinner model-L2DB5150 3/4-in. two-way solenoid valve was connected by a copper pipe to the normally open port of the three-way valve. The

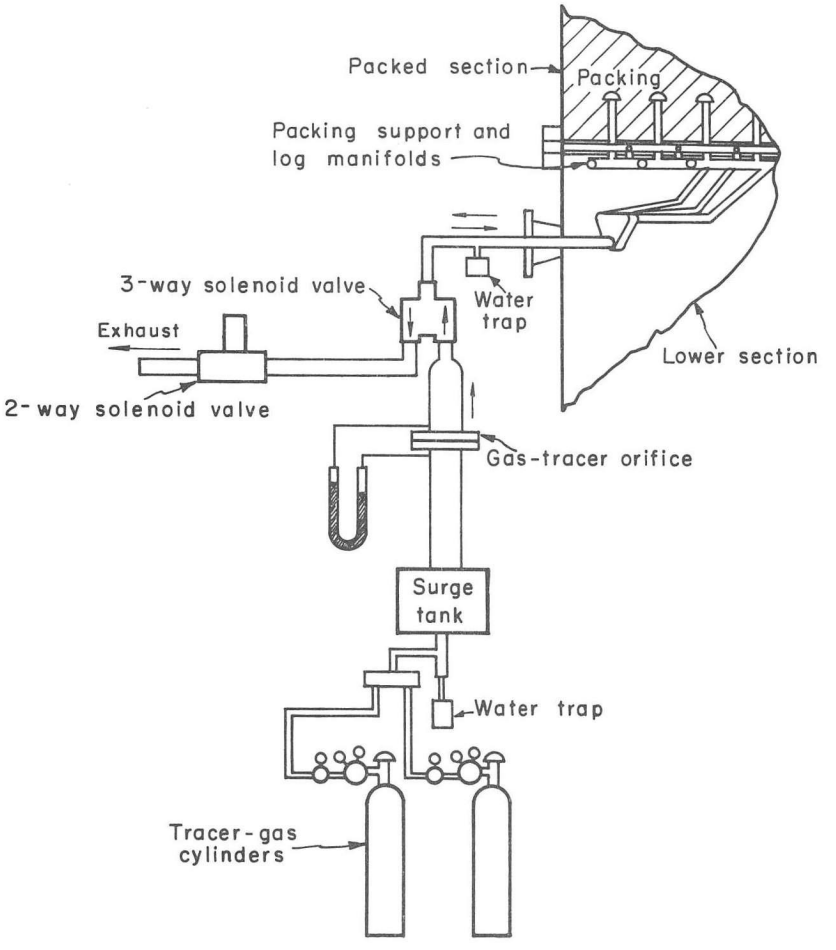

Fig. 8. Schematic diagram of the gas tracer injection system.

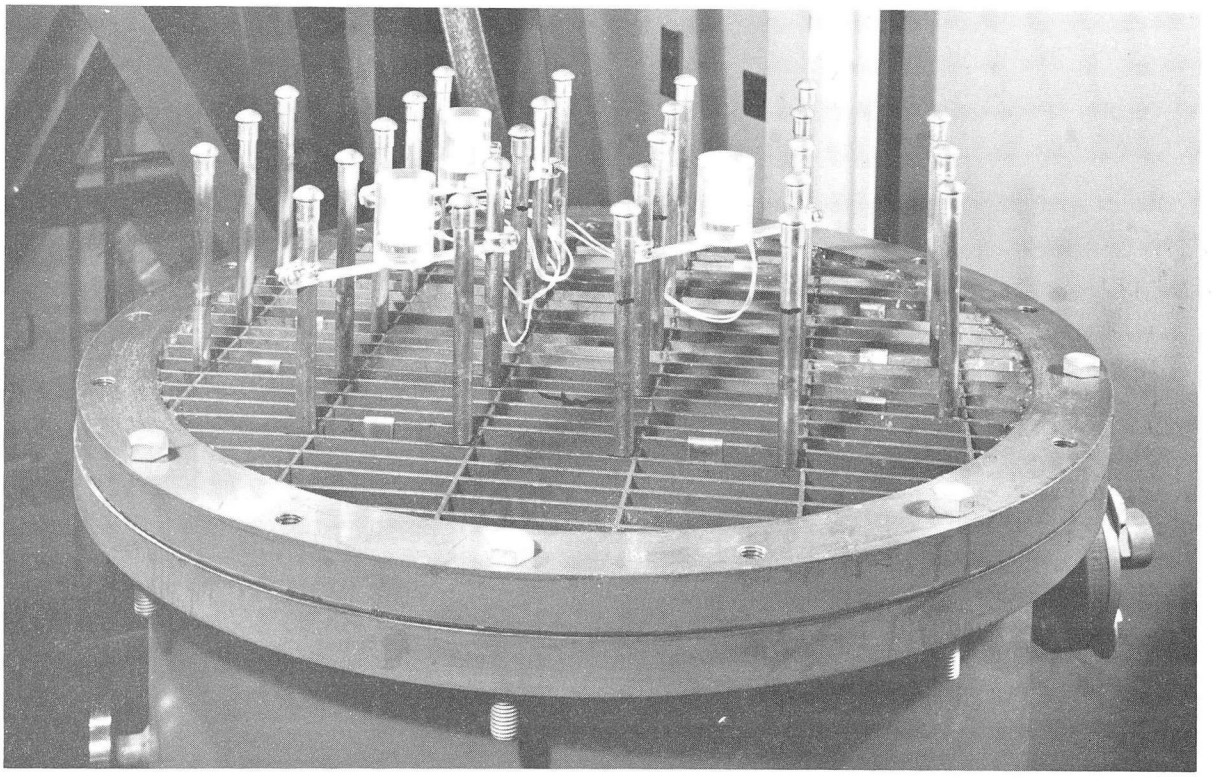

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Fig. 9. Gas-tracer injection manifold.

common port of the three-way valve was connected through a 2-in. welding-neck flange to the log-type injection manifold.

Helium purge system

A 1/4-in. -o. d. copper refrigerator tube led from the surge tank to a Conoflow purge regulator, and then connected with the log manifold feed tube just outside the welding-neck flange. At times when data on the gas phase were being taken, the log manifold was kept full of helium by a purge of about 50 cm^3/min.

Tracer injection manifold

A photograph of the injection manifold attached to the packing support is shown in Fig. 9. It consisted of 26 1/2-in. -o. d. copper injection tubes soldered on 4-in. centers to five 3/4-in. -o. d. copper tubes. These 3/4-in. "logs" were suspended from the packing support grating bars by metal hangers. The distance between the logs was also 4 in. A feed line ran from the center of each of the 3/4-in. tubes to a triangular box manifold, 3 in. on a side and 1-1/2 in. thick. The feed line coming from the three-way solenoid valve entered the apex of one side of this inverted triangular box, and the five feed tubes left the base of the other triangular side. Each of the 5/8-in. injection tubes was capped by a 1/2- by 1/4-in. bell reducer, to which an umbrella-like copper lid had been soldered. Four 0.018-in. -diam holes were drilled in each bell reducer at such an angle that a jet of tracer gas leaving the hole would strike just inside the edge of the umbrella. The tubes extended into the packing so that the injection holes were 6 in. above the packing support. Small holes were drilled so that the manifold could be kept full of helium by a small purge stream, and so that when the tracer was being injected the manifold would have a finite back pressure. The copper umbrellas served to deflect some of the liquid away from the holes and also to deflect the helium tracer and diminish its velocity component along the axis of the column.

Liquid drainage

The 3/4-in. tubes of the log manifolds were slightly lower in the center than at the ends, so that any liquid which entered the manifold would flow to the center of the log, then into the feed tubes, and then into the inverted triangular box. The water flowed from the apex of the box to a tube fitted with a transparent Lucite water trap outside the column. A second Lucite water trap was located at the lowest point in the system, below the surge tank.

Method of operation

It was desirable to have an injection system which would deliver the correct flow of tracer almost instantaneously, and which would also permit rapid interruption of the tracer flow. When an injection was begun the three-way solenoid valve was energized, opening its 3/4-in. orifice and starting tracer flow from the surge tank. The surge tank had been pressurized to the estimated back pressure of the injection manifold at the desired flow rate. The helium leaving the surge tank was replaced as the helium pressure regulators admitted more gas to the surge tank to maintain the selected pressure. Since the injection manifold was kept full of helium by the purge regulator, the tracer entered the column immediately. The two-way valve was then opened to the atmosphere. When the tracer had been injected for the required time, the three-way valve was de-energized, closing the line to the surge tank and opening the injection manifold to the atmosphere via the two-way valve. Since the column operated at a slight gauge pressure, the tracer flow was stopped immediately. The two-way valve was then closed, and the manifold was filled with helium through the purge system in preparation for another run.

Column Controls

The controls necessary for the operation of the apparatus were located on a single panel board by means of sprockets, chains, gears, extension shafts, and remote-operated electrical devices. A photograph of the instrument panel is shown in Fig. 10. Manometers were

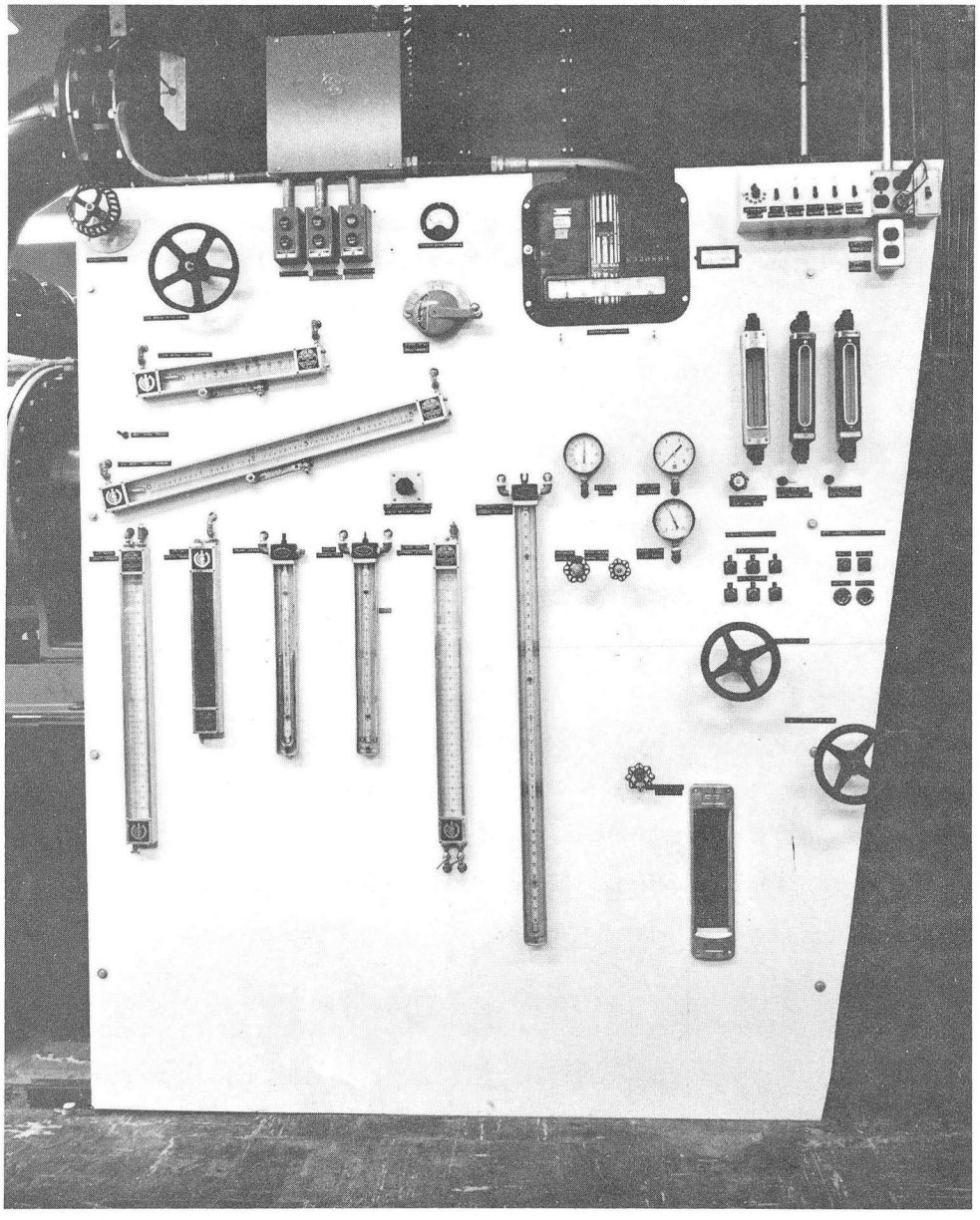

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Fig. 10. Instrument panel.

used for flow metering as well as for pressure indication. These gauges were grouped according to function and are discussed as they appear on the board.

Manometers and draft gauges

Two draft gauges and a manometer were used to indicate the impact pressure in the air duct flow metering nozzle. These gauges were manufactured by Uehling Instrument Company and indicated pressure ranges of 0 to 1, 0 to 5, and 0 to 20 in. of water, respectively. The draft gauges could be isolated from the impact tube pressure by means of a quick-closing Hoke valve labeled Draft Gauge Shutoff. It was necessary to use Uehling indicating oil (sp gr 0.795) in the draft gauges to obtain accurate readings. The black manometer second from the left on the board gave the air duct static pressure in inches of water. Two 15-in. Meriam manometers gave the pressure below the packing in inches of mercury, and the pressure drop across the packing in inches of water. Located fifth from the left was a 20-in. Uehling manometer that indicated the pressure difference across the liquid flow metering orifice. This manometer was also connected to the building compressed-air system through a pressure regulator. The liquid orifice was located about 6 ft above the manometer, with the result that the manometer would ordinarily fill up with liquid. By adjusting the air pressure above the two liquid legs in the manometer, their interfaces could be controlled to any desired position. The last manometer, a 36-in. Meriam, gave the pressure difference across the helium tracer orifice.

Valves

A number of valve wheels can be seen on the board. In the upper left-hand corner of the panel is one marked Blower Bypass Valve. This controlled the butterfly valve in the bypass air duct, and was used to give better control of the air flow. To the right of that is the Air-Cooler Water Valve that controlled the flow of water to the air cooler. In the center of the board are the Tracer Return-Line Valve and the

Rotameter Bypass Valve. A 3/8-in. needle valve, which can not be seen in Fig. 10, was installed behind the panel in the line which extended from the liquid-tracer three-way solenoid valve to the spider injection manifold. By adjusting these three valves, a steady flow of sodium nitrate tracer solution could be maintained when the tracer injection was started.

In the lower right-hand section of the panel were two valves which controlled the flow of liquid. The 4-in. Powell gate valve located on the discharge side of the liquid feed pump was labeled Liquid Feed Valve. The 2-in. Lunkenheimer gate valve located in the bypass line around the pump was labeled Liquid-Feed Bypass Valve.

Switches

At the upper edge of the board to the left of center were three push buttons that started the Tracer Feed Pump, Liquid Feed Pump, and Blower Motor. In the upper right-hand corner of the board was a battery of switches. These were a thermocouple-selector switch, the temperature-indicator motor switch, and four switches which controlled the solenoid valves.

The thermocouple-selector switch connected a Leeds-Northrup Micromax temperature indicator-controller to five iron-constantan thermocouples. These thermocouples measured the temperature in the two gas thermal-conductivity cells, the air temperature at the column inlet and outlet, and the air temperature in the flow-metering nozzle.

Rotameters

Four Fisher-Porter Flowrators were mounted on the panel. The two smallest had F-P No. 02F tubes, with ranges of 2600 and 975 cm^3/min of air at 70°F and atmospheric pressure. These rotameters could be operated singly or in parallel, and were used to measure the flow of sample gas drawn through the thermal-conductivity cells. The other two, with capacities of 0.21 and 1.96 gpm of water at 72°F, have been discussed in the description of the liquid tracer system.

Pressure gauges

Three J. H. Marsh pressure gauges were located in the center of the panel. These indicated the liquid-tracer return-line pressure, the helium surge-tank pressure, and the thermal-conductivity sample pressure.

Cell terminals

Signal cables from the liquid and gas sampling cells were brought to a terminal panel at the right of the board. Other cables connected the terminal of the desired cell to the appropriate amplifier-recorder system in the instrument cabinet.

Sampling, Amplifying, and Recording Equipment

Liquid sampling cells

The change in concentration of sodium nitrate tracer in the liquid stream was determined by measuring the electrical conductivity of the liquid. One of the conductivity cells, used is shown in Fig. 11. A cross section of a typical cell is shown in Fig. 12. These cells consisted of 0.0935-in.-diam rhodium-plated nickel pins set in 1-1/2-in.-o.d. by 1-in.-i.d. lucite cylinders. The bottom of each cylinder was closed and the top was open. Inserted in the top of the cylinder was a Teflon plug which was cut in such a way that liquid entering the top of the cell had to flow down into the cell, across the conductivity pins, up the other side, and pass out of the cell through a 1/4-in. lucite tube. Therefore the conductivity pins were always submerged in fluid and gave continuous readings. The pins were located 0.0935 in. from the bottom of the lucite cylinder on 0.28-in. centers. Teflon-coated wires were soldered to the pins. These wires extended outside of the column through lucite plugs which were fitted in 2-in. welding-neck flanges. Microphone cables connected these wires with the terminal board. The plugs were designed so that when the column packing was dumped the plugs could be detached from the flanges; and packing, cells, and plugs were dumped together. Three cells were located, as shown in Figs. 13 and 14, on each of two levels in the column: 6 in. and 30 in. above the packing support. The cells were anchored to stainless steel screens to keep them upright.

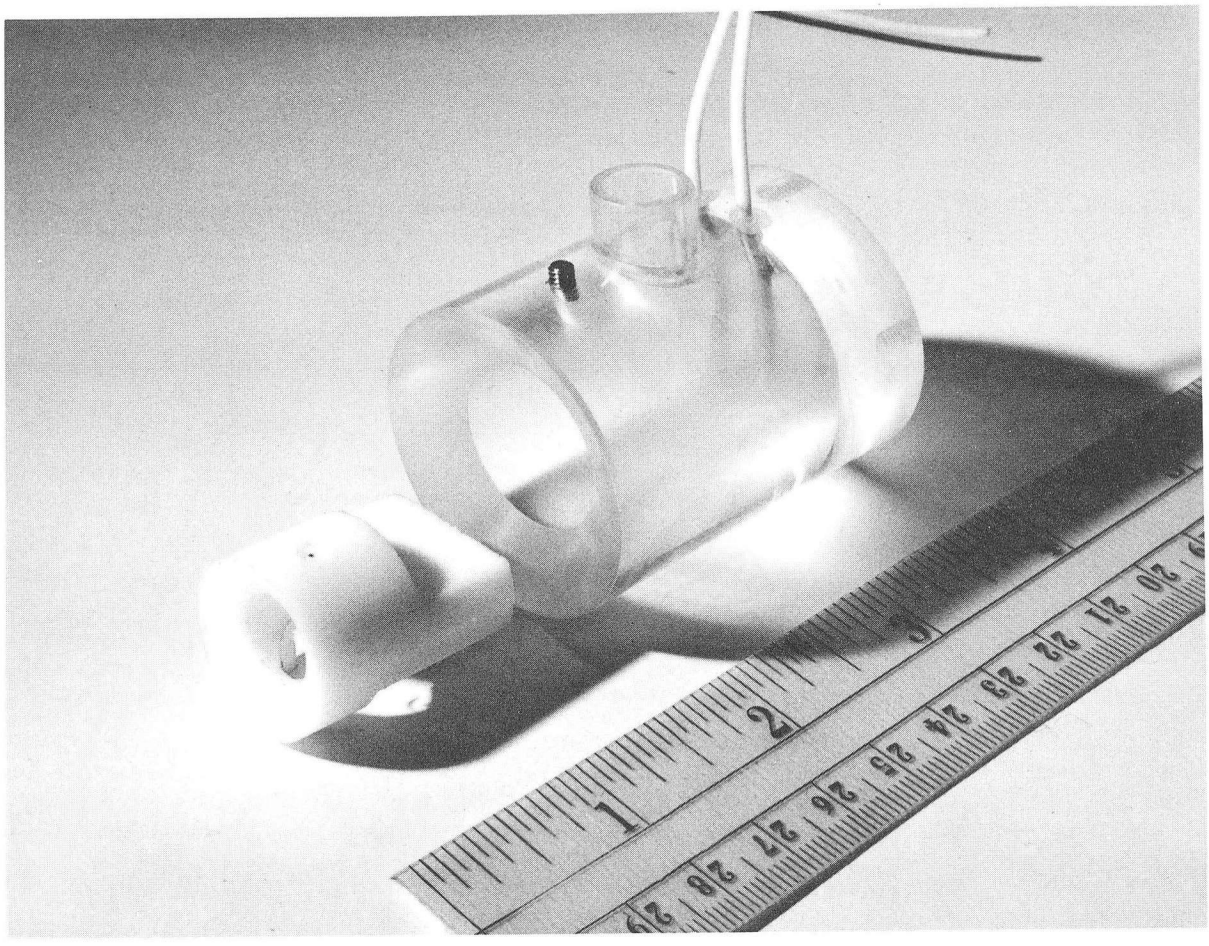

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Fig. 11. Liquid-conductivity cell.

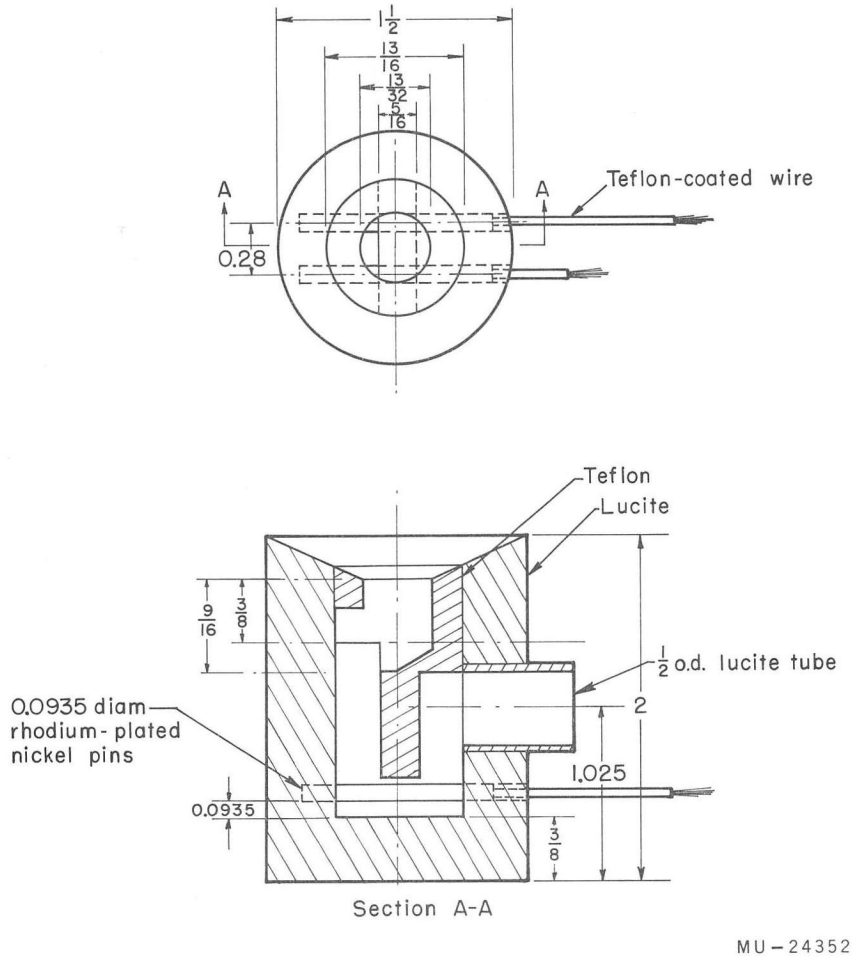

Fig. 12. Cross-sectional drawing of a liquid-conductivity cell.

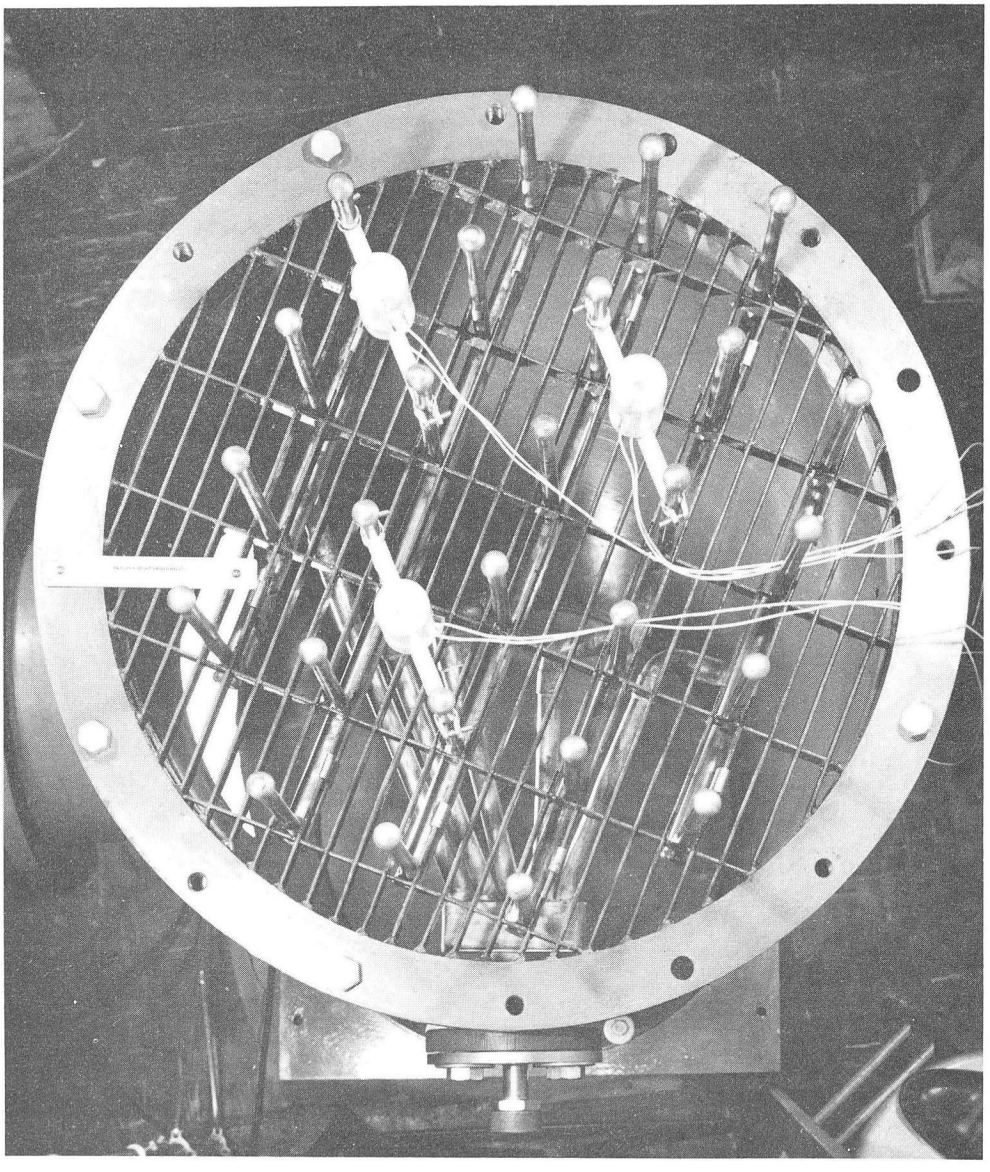

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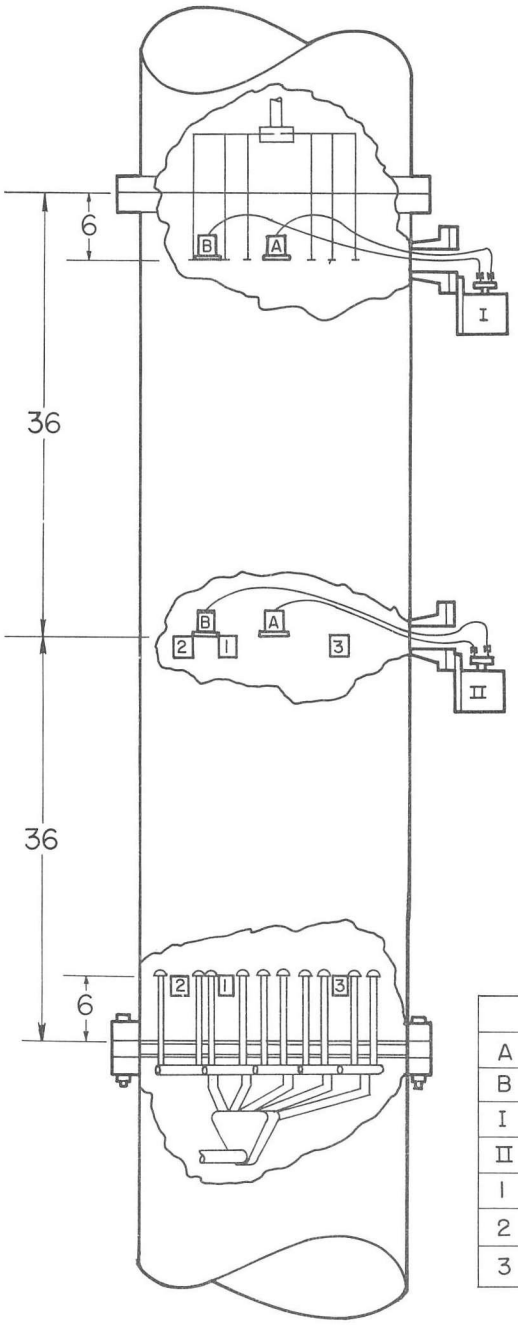
Fig. 13. Liquid-conductivity cells, gas-tracer injection manifold, and packing support plate (showing radial location of liquid cells).



	Unit	Radius
A	Gas sampler	0
B	Gas sampler	4 in.
I	Therm. cond. cell	
II	Therm. cond. cell	
1	Liq. cond. cell	4 in.
2	Liq. cond. cell	7 in.
3	Liq. cond. cell	5 in.

MUB-761

Fig. 14. Location of sampling probes (schematic diagram).

Liquid-cell amplifying and recording system

The electrical resistance between the conductivity pins in a cell was the variable used to determine the tracer concentration in the liquid stream. The electronic system used to receive, amplify, and record the signal from the conductivity cells is shown in the diagram in Fig. 15. It was modeled after the system described by Jacques and Vermeulen.[5]

Basically, the system consisted of a 1000-cycle power source that applied a voltage across any two of the six cells in the column. An amplifying system amplified and rectified the signals from the two cells and sent these signals to a Leeds and Northrup Speedomax type-G dual-channel recorder. The amplifying and recording equipment for the liquid and gas phases was housed in a metal cabinet as shown in Fig. 16. A relay was wired to the switch of the solenoid valve that controlled the tracer flow. When this switch was thrown, a small signal traveled to the recorder via the relay, and a spike was made on the recorder strip chart to indicate the beginning of a run. The change in cell resistance, and consequently the change in tracer concentration in the liquid stream, was recorded on the strip chart.

Five adjustments could be made on both channels of the recorder-amplifier system. These were the zero, sensitivity, range, attenuation, and gain. Before each set of runs, both channels were zeroed for no-signal conditions by disconnecting the signal cables and turning the zero knob until zero voltage was indicated on the meter in the upper left-hand corner of the LICONAMP (Liquid Conductivity Amplifier) panel. It must be emphasized that the zero adjustment was only made for no-signal conditions. Adjusting the zero knob to make the recorder read zero when there was a finite signal input to the recorder could cause the recorder response to be an unknown nonlinear function of tracer concentration.

The sensitivity and range adjustments were made only once, so that a calibration curve of recorder response versus concentration of

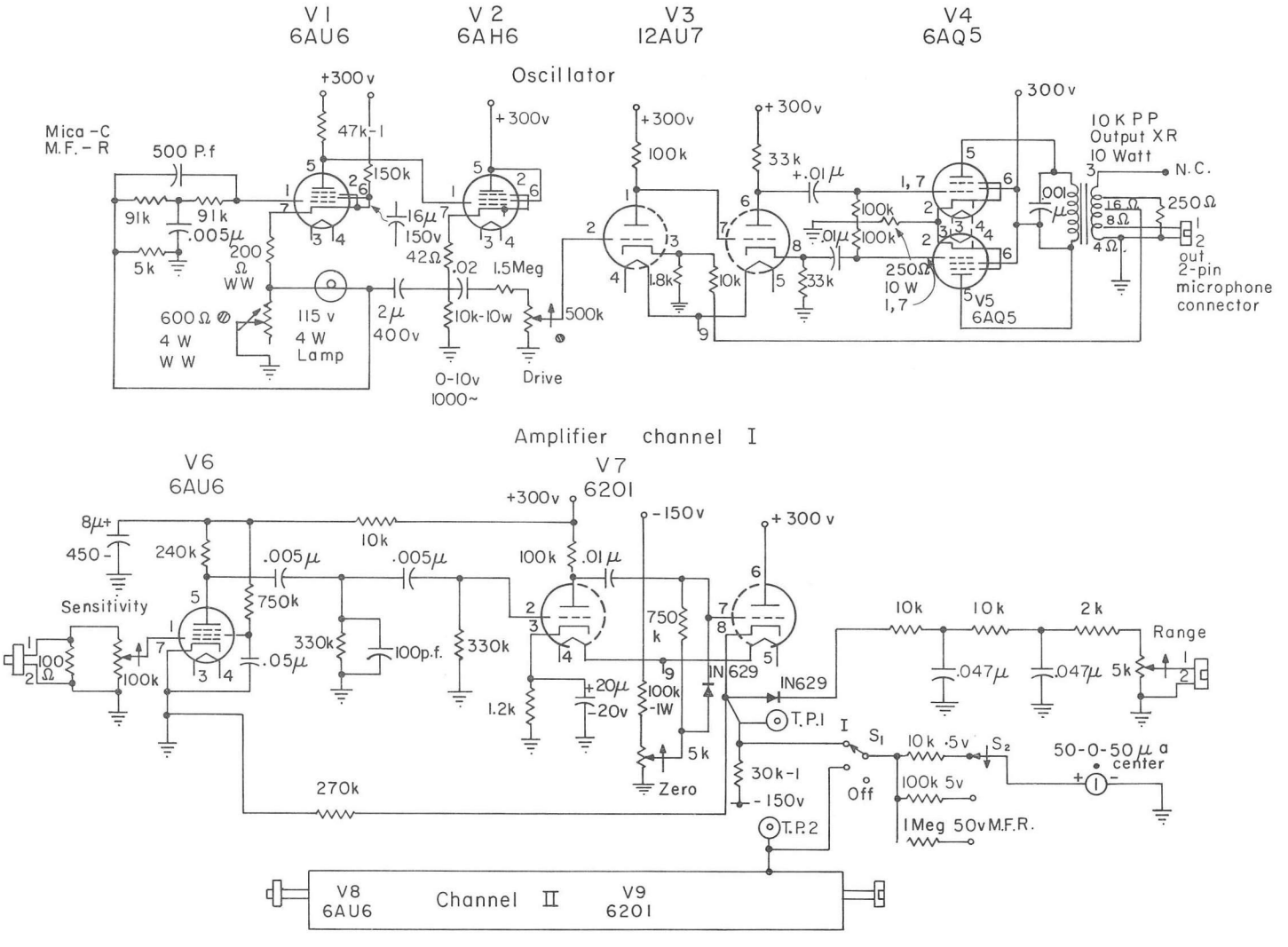


Fig. 15. Wiring diagram for liquid-conductivity oscillator and amplifier.

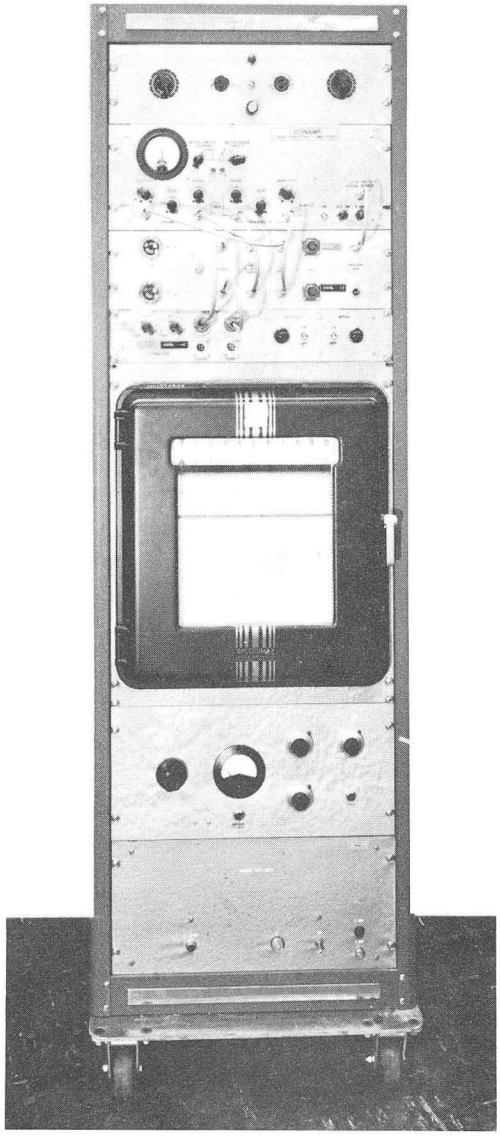


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Fig. 16. Cabinet housing electronic gear.

tracer in a given cell could be used. Changing these adjustments would change the slope of such a curve.

The attenuator adjustment had the same function as the range with the exception that changes in the attenuation of a recorder channel were made in integral steps. The range adjustment controlled the amount of recorder response for a given amount of conductivity, and this could be varied within certain limits. By adjusting the attenuator knob the operator could cause the recorder response to be divided by a factor of 2, 5, 10, 20, 50, etc. This was desirable when a signal exceeded the recorder scale of 10 mv. In this work most of the runs were made with the attenuator at 100 so that a full scale reading on the recorder represented 100×10 mv = 1 v. If a signal of 1.2 v was sent to the recorder, the attenuator was turned to 200 and a reading of 6 mv was indicated on the recorder scale. The validity of a calibration curve was not impaired by changing the attenuator setting since the recorder response was always an accurately known fraction of the input signal.

The use of very high attenuator settings with relatively concentrated solutions (greater than about 0.05% by weight of $NaNO_3$) is not recommended, however, for two reasons. First, conductivity is a linear function of $NaNO_3$ concentration only in very dilute solutions and, second, the high conductivity of a concentrated solution might demand a greater current than the 1000-cycle power source could produce at its set voltage. This problem was avoided by never taking data when the concentration of $NaNO_3$ in the liquid stream was above about 0.035%, although the maximum permissible concentration was not determined.

Violent fluctuations of the recorder pens could be controlled by reducing the recorder gain. In the case of the 30-in. -level pen this was done by turning the black knurled knob, marked X_2, located above and to the right of the recorder chart. The gain for the 60-in. -level pen was controlled by a slotted screw mounted on the recorder-amplifier chassis directly in front of the converter. Turning these adjustments counter-clockwise reduced the gain and the pen fluxuations.

This was done with caution, however, since reducing the gain of a recorder pen increased its response time and could result in inaccurate breakthrough curves.

Gas-sampling probes

The change in concentration of helium tracer in the air stream was determined by continuously monitoring the thermal conductivity of a small gas stream that was withdrawn from the column through brass sampling probes fitted with porous Teflon disks. These disks removed any entrained water in the sample. One of the sampling probes is shown in Fig. 17. The sampling probes were 1-in. -i.d. by 1-1/2-in. -o.d. brass cylinders which were fitted with threaded caps. A stainless steel screen was soldered to the outside of each cap. Porous Teflon wafers were cut from material obtained from Porous Plastic Filter Corporation and were held in place over the mouth of the brass cylinders by the threaded caps. A 1/8-in. -o.d. copper tube was soldered to the closed end of each cylinder, and this tube was connected to one inlet port of a thermal-conductivity cell. Two probes were attached to stainless steel screens and placed in the column at each of two levels, 30 in. and 60 in. above the packing support, as shown in Fig. 14. At each level, one cell was placed in the center of the column, and the other at a distance of 4 in. from the center toward the side of the column to which the sight glass was attached.

Thermal-conductivity cells

Figure 18 shows a photograph of one of the thermal-conductivity cells. These cells were made from 2-in. -diam aluminum bars, and were 3 in. long. A 1/8-in. hole was drilled along the axis of the bar. Inlet and outlet holes, also 1/8 in. in diam, intersected the axial hole at right angles 1/2 in. from each end. A cross section of the cell is shown in Fig. 19. A 0.00045-in. -diam tungsten wire was stretched along the axis of the cell and anchored to terminals in the Bakelite end pieces. Keulemans[17] indicates that a cell of this design should have a very rapid response.

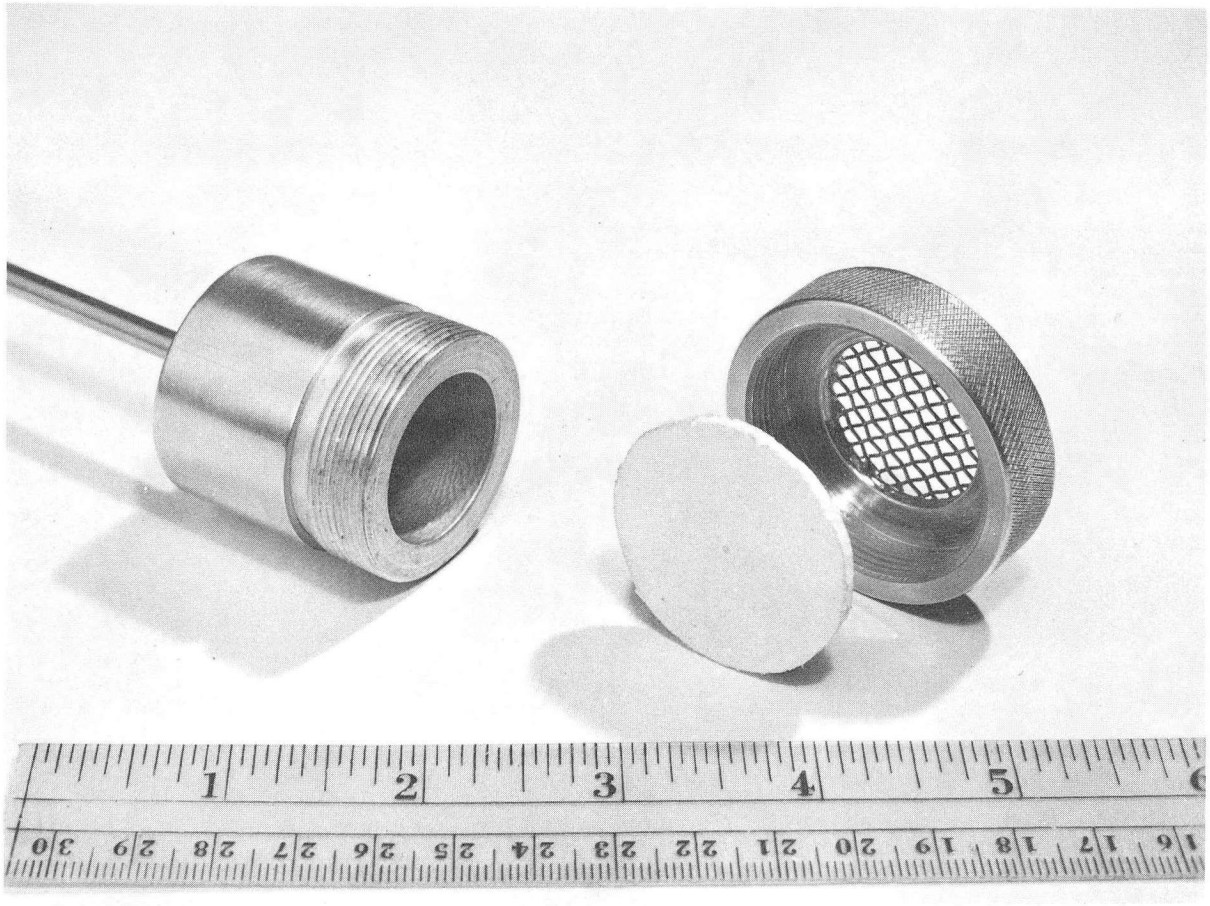

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Fig. 17. Gas-sampling probe.

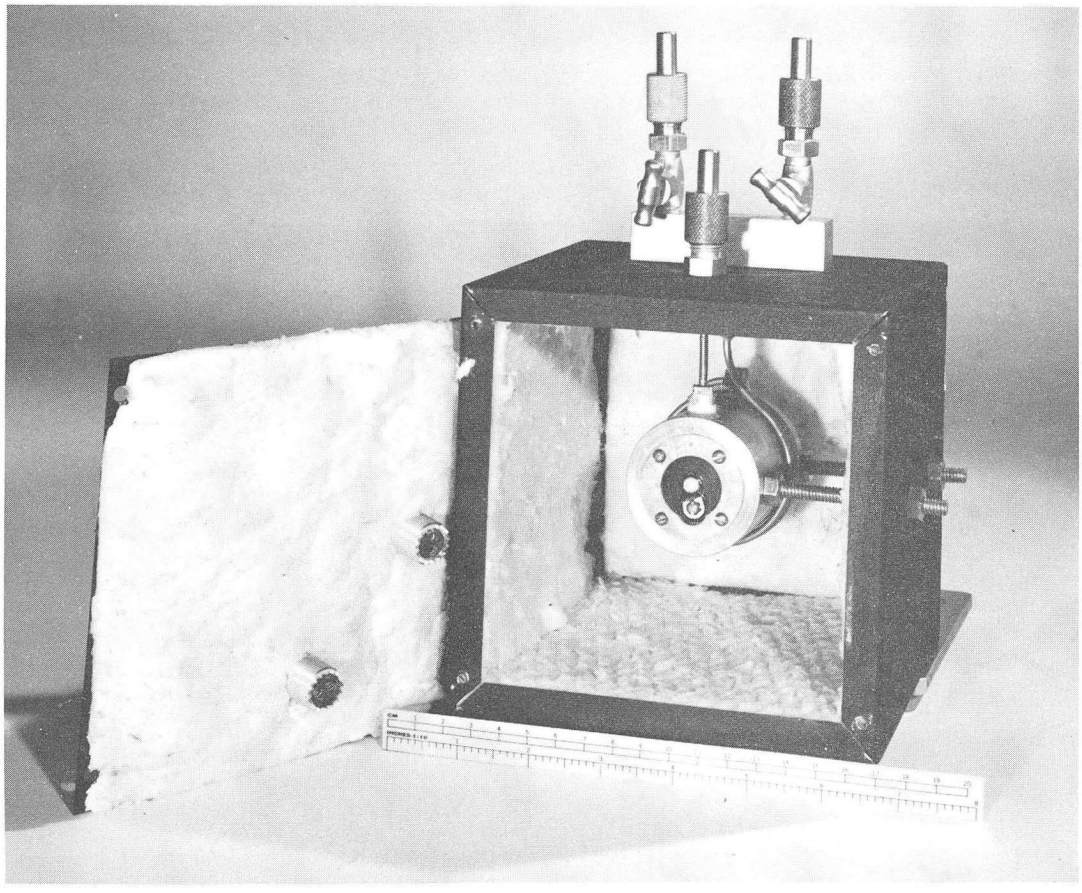

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Fig. 18. Thermal-conductivity cell.

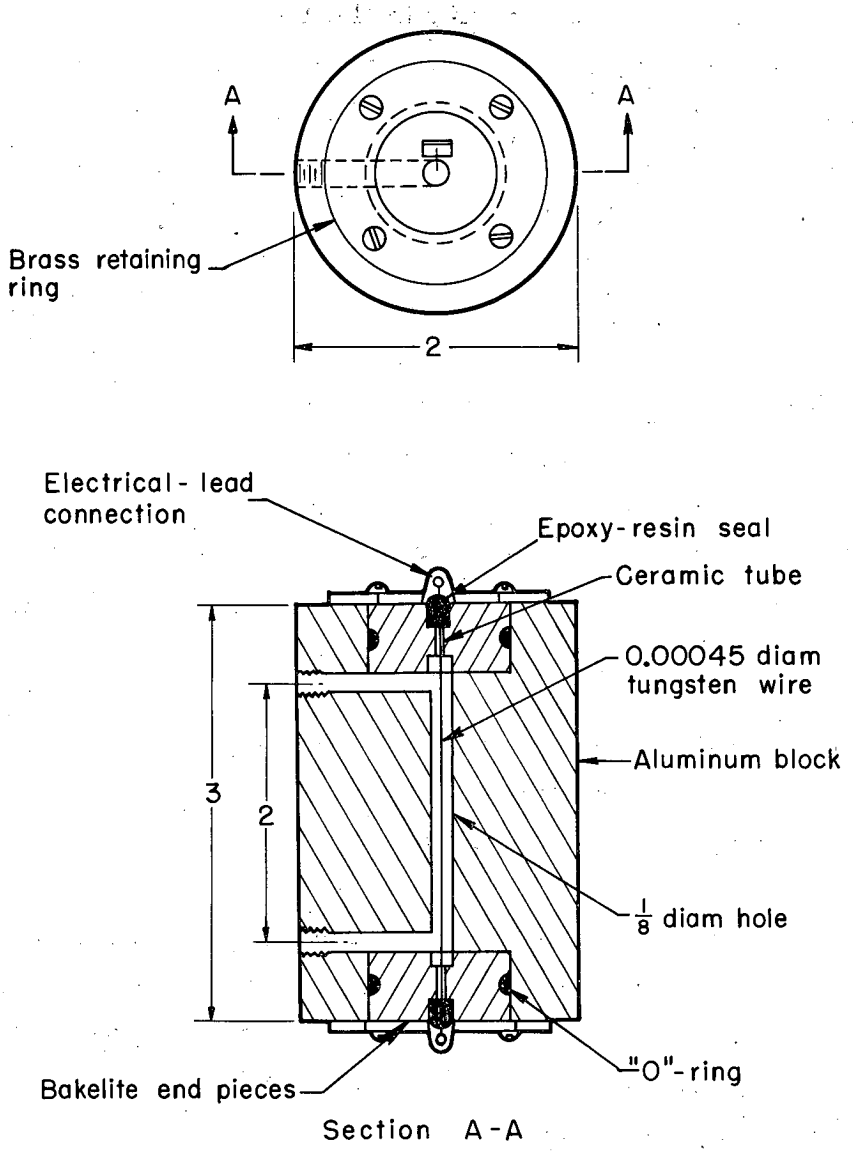

Fig. 19. Cross-sectional drawing of a thermal-conductivity cell.

The aluminum cell was mounted in a metal box which measured 6 in. on a side as shown in Fig. 18. A heating tape was wrapped around the cell and the empty cavity in the box was filled with glass wool insulation. The electrical connections for the cell and the heating tape were brought outside the box by threaded connections and were extended back to the main instrument panel by means of Belden 8422 microphone cables. Current to the heating tapes was controlled by two 230-w Powerstats mounted in the electronic-instruments cabinet. A thermocouple was inserted into a hole in each cell, and the cell temperature was read on the temperature indicator on the instrument panel.

Gas-cell amplifying and recording system

The amplifying system for the thermal-conductivity cells was simpler than that for the liquid-conductivity cells, as may be seen from the circuit diagram in Fig. 20. It consisted of a bridge made up of three variable resistors and a thermal-conductivity cell connected in series with a large variable resistor and a 300-v dc power source. Since the major part of the voltage drop occurred across the large resistor, the current to the bridge was very constant. The current passing through the tungsten wire in the thermal-conductivity cell heated the wire to a certain temperature. The heat generated in the wire was conducted through the gas in the cell to the cell walls, which were maintained at a constant temperature. As long as the composition, and therefore the thermal conductivity, of the gas remained constant, the temperature and resistance of the wire also remained constant. When the concentration of helium in the gas began to increase, the thermal conductivity of the gas increased, the wire cooled, and the cell resistance decreased. The resulting changed bridge resistance caused an out-of-balance voltage; this was sent to a Brush BL932 amplifier and then to a Brush BL202 oscillograph recorder. A relay (connected to the switch of the gas-tracer three-way solenoid valve) sent a small signal to the recorder at the beginning of each run, which put a spike on the recorder chart. The Brush

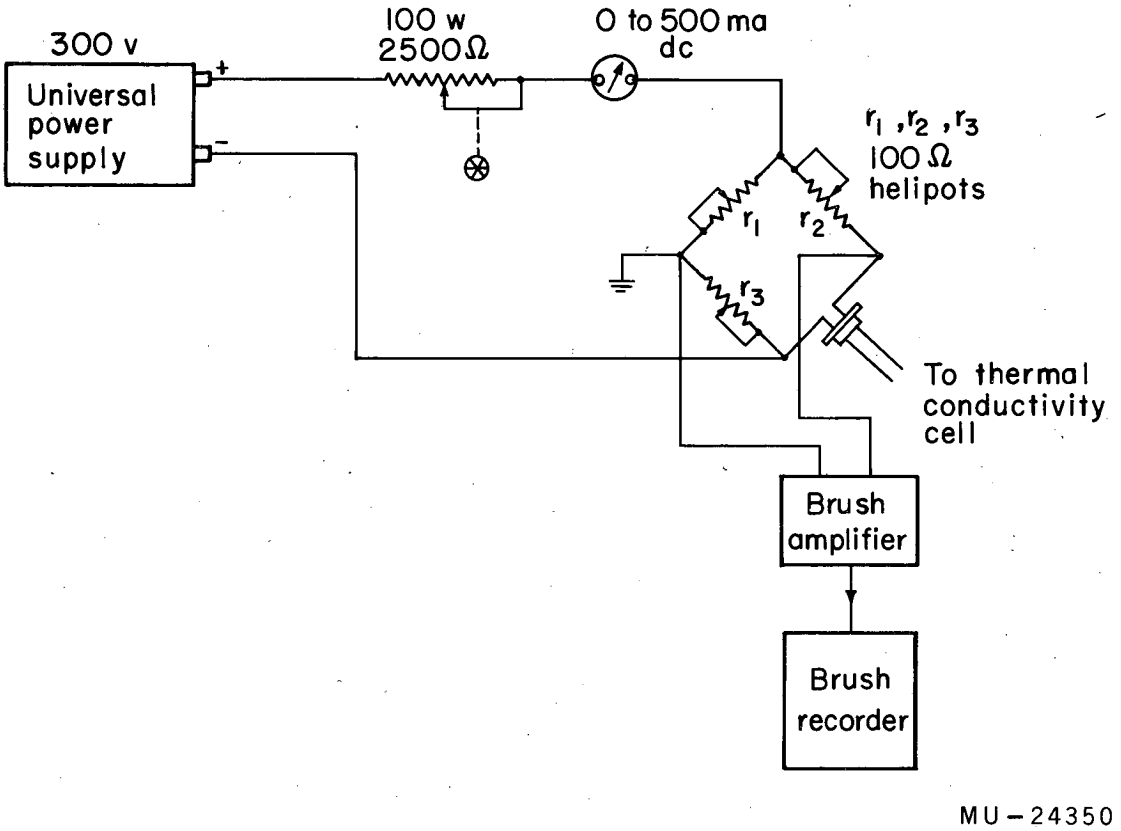

Fig. 20. Wiring diagram for the thermal-conductivity recording and amplifying system.

recorder had a rapid response and a fast chart speed (125 mm/sec), which were necessary to measure the change in concentration of the helium tracer in the fast-moving gas stream.

III. OPERATING PROCEDURE

The purpose of the equipment described in the previous section was to obtain recorder-chart traces (breakthrough curves) of the change in concentration of a tracer element at a point in the packed column downstream from the injection point. The operating procedure necessary to obtain these traces is discussed below. The procedure for obtaining data on the liquid and gas phases will be discussed separately, although the equipment permitted data on both phases to be taken simultaneously.

Attainment of Steady-State Conditions

The first step in taking data for either phase was to put the column into steady-state operation. First a plastic hose was connected to a hose coupling on the lowest 2-in. welding-neck flange on the lower section of the packed column. The lower section was filled to a depth of about 2 ft as measured on the sight glass with water from a wall tap.

While the lower section was filling with water, the steam to the coils in the return-air duct was turned on. It was important that these coils be operating before the blower was started, to prevent any water condensation in the return duct. The liquid feed pump was started, and the desired flow rate attained by adjusting the control valve on the pump. The bypass valve was ordinarily left fully open. Next, the blower was started, with the butterfly valve in the bypass duct set in the neutral position. The blower speed was adjusted to give a flow rate slightly less than the desired value, and the butterfly valve was then slowly closed until the proper flow was realized.

The temperature indicator was turned on and the air temperatures at the column inlet and outlet were compared. It was assumed that the gas at both points was saturated with water. The cooling water to the air cooler was adjusted until the inlet and outlet temperatures were identical.

If data were to be taken on the gas phase, the thermal-conductivity cell heaters were turned on, and their Powerstats were adjusted to the estimated current requirements. A scale reading of 30 on the Powerstat usually served to bring the cell to 30° C in about 5 min. When the cells reached the desired temperature the heaters were turned off. The helium cylinders were checked to see if they contained sufficient gas for the desired runs. The three-way solenoid valve was opened for about 10 sec at a surge-tank pressure of about 2 psi, to flush the air out of the tracer-gas injection manifold, and the purge regulator was adjusted to give the desired amount of helium flow to the manifold.

If data were to be taken on the liquid phase, it was necessary to prepare the sodium nitrate injection system. The tracer level in the surge tank was checked to see if there was sufficient tracer for the desired runs. The tracer pump was started, and the tracer was allowed to circulate through the pump.

Usually by this time the column had come to equilibrium. The final adjustments of flows and temperatures were made, and then the column data were recorded. The data taken were as follows:

a. Date, run number, packing material
b. Air-nozzle impact pressure
c. Air-duct static pressure
d. Air-nozzle temperature
e. Liquid orifice pressure difference
f. Air inlet and outlet temperature
g. Column pressure below packing
h. Pressure drop across the packing

The column was then ready to receive a tracer injection.

Liquid-Phase Procedure

The liquid tracer flow was adjusted by means of needle valves close to the rotameters. The quantity of 1N sodium nitrate tracer solution used was between 0.1% and 0.5% of the liquid flow in the column.

The resistance in the return line from the liquid three-way solenoid valve to the tracer pump was adjusted to be equal to the resistance in the tracer injection line and the spider injection manifold. This was done by adjusting the rotameter bypass valve, the tracer return line valve, and the tracer injection-line valve. Experience showed that the tracer flows, before and after the injection was started, could most easily be made equal by adjusting the tracer-injection-line valve.

The recording and amplifying equipment was turned on and allowed to warm up. When data were being taken several times a week the equipment was left on continuously to reduce drift of the recorder zero point; otherwise, at least a 45-min warm-up period was required. Microphone cables connecting the amplifier to the conductivity cells were attached to the terminals of the desired cells, and the number and position in the column of both cells were recorded on the data sheet.

When all the equipment was ready, the switch to the liquid-tracer three-way solenoid valve was closed. This started the tracer injection to the column and also put a spike on the recorder chart. The tracer was allowed to flow into the column until its concentration at the sample point reached a steady-state value, whereupon the solenoid valve was de-energized, another mark was made on the recorder chart, and the tracer was flushed out of the column. Both the salt-in and salt-out breakthrough curves could be used to determine the Peclet numbers.

Gas-Phase Procedure

Helium tracer flow was usually about 3% of the volumetric air flow rate. When the amount of helium tracer to be used had been determined, the regulators on the helium cylinders were adjusted to maintain a pressure in the gas-tracer surge tank that was approximately equal to the back pressure of the gas-tracer manifold when it was delivering the desired tracer flow.

The 300-v dc power source and the Brush amplifier were turned on and allowed to warm up for at least 45 min. The current to the cell bridge was adjusted to about 100 ma by selecting the proper value of the large variable resistor. The amount of current determined the sensitivity of the bridge. A 100-ma current would usually give a full-chart-width deflection when 3% by volume of helium was being drawn through the thermal-conductivity cell.

The temperatures of the thermal conductivity cells were checked to make sure that they were 30°C. This cell temperature was maintained by adjusting the current to the cell-heating tapes. It was necessary that the temperature of the cell block remain constant during a run.

While the current passed through the bridge, the three variable resistors were adjusted until the bridge was balanced. This was done by determining the recorder pen position when a shorted phone plug was inserted in the amplifier-input jack, and then adjusting the resistors until the signal from the bridge resulted in the same pen position. The thermal-conductivity cells had resistances of about 60 ohms each when drawing 100 ma current, and this value was used as a rough estimate in the first stages of balancing the bridge. The pen of the Brush recorder normally rested in the center of the chart for no-signal conditions; but the signal from the bridge could be biased, so that the recorder pen would rest at the left-hand side of the chart, by adjusting the balance knob on the Brush amplifier. This was done so that for a full-scale deflection the pen would travel the full width of the chart. Finally, just before the helium injection was begun, the desired chart speed of 5, 25, or 125 mm/sec was selected by pulling out a knob on the side of the Brush recorder to the proper position.

When all the preliminary steps had been taken, the switch to the gas-tracer three-way solenoid valve was closed. This started the helium flow into the column and put a spike on the recorder chart. The two-way solenoid valve was then opened. When the helium

concentration at the sampling point had become constant, the three-way valve was de-energized, allowing the injection manifold to exhaust to the atmosphere, and the tracer injection was stopped. The two-way valve was closed, and the manifold was refilled with helium in preparation for the next run.

Determination of Liquid Holdup

The liquid holdup in the packing was determined for each set of flow conditions. This was done in the following manner.

When the desired flow conditions had been obtained and the liquid level in the lower section had become constant, the height of liquid in the sight glass was marked with a piece of tape. The solenoid valve controlling the instant shutoff valve in the liquid feed line was energized, closing the instant shutoff valve and stopping the liquid flow. With the pump still running, the liquid-feed control valve was closed, and the liquid was allowed to circulate through the pump via the 2-in. bypass line. The pump and blower were then shut off and the liquid was allowed to drain from the packing.

When all the liquid had drained, the increase in the liquid level in the lower section was measured. From this height and from the cross-sectional area of the lower section, the liquid holdup was determined.

Shutdown Procedure

Shutting down the column was quite simple. After the last run had been made, sodium nitrate solution was poured into the column, through a fitting in one of the windows in the air-outlet plate, until the concentration of $NaNO_3$ in the liquid was 1% to 3%. The purpose of this was to prevent rusting. The solution was circulated through the column for a few minutes, then the solenoid controlling the instant-shutoff valve was energized, closing the valve. With the solenoid energized and the pump still running, the valve controlling the air supply to the diaphragm of the instant-shutoff valve was closed. Next the liquid-feed control valve was closed. The pump was shut off, and the solenoid was de-energized.

This procedure resulted in the system being filled with sodium nitrate rust-inhibitor solution from the lower section of the column up to the inlet side of the instant-shutoff valve. When the rest of the equipment had been turned off, the equipment was secured until the next set of data had to be taken.

ACKNOWLEDGMENTS

I would like to thank Dr. C. R. Wilke, Dr. T. Vermeulen, and L. R. Lucas for their guidance and constructive criticism in the design of the column and its accessories. I am also indebted to G. G. Young and M. K. Flamm for their ingenuity and industry in the construction of the apparatus.

This work was done under the auspices of the U. S. Atomic Energy Commission.

APPENDICES

A. Nomenclature

C	Point concentration of tracer inside the column
C_0	Concentration of tracer after an infinite injection period
d_p	Particle diameter
E	Dispersion coefficient
h	Height of packed bed
H	Longitudinal mixing length
HTU	Height of a transfer unit
I_0	Bessel function of zero order, with imaginary argument
l	Characteristic length
n	Number of jumps in a random walk
N	Number of longitudinal mixing lengths; column Peclet number
NTU	Number of transfer units
N_{Pe}	Particle Peclet number
s	Slope of breakthrough curve
s'	$s(T_{0.5}/N)$
t	Time
T	Dimensionless time
U	Characteristic velocity
x	C/C_0

B. Calculation Procedure

The calculation procedure used was that devised by Jacques and Vermeulen,[10] and was based on Eq. (7). This procedure consisted of determining the slope, s', of a plot of C/C_0 versus $t/t_{0.5}$, and substituting s' into Eq. (7) to calculate N_{Pe}. This is most easily understood by considering a specific example.

Figure 21 is a recorder chart tracing for Run 108. For each run, values of the concentrations shown on the recorder chart were recorded in convenient units together with the time elapsed since the beginning of tracer injection (which could be measured on the chart). The time required to attain $C/C_0 = 0.5$ (designated $t_{0.5}$) was also measured. These values were recorded in tabular form as shown in Table I. Next, values of the reduced concentrations and reduced elapsed times were obtained by dividing the concentrations and times by C_0 and $t_{0.5}$, respectively. A breakthrough curve of C/C_0 versus $t/t_{0.5}$ was then plotted, and its slope, s', at the midpoint ($C/C_0 = 0.5$) was measured graphically (Figs. 22 and 23). The value of s' was substituted into Eq. (7) and a value of N was obtained. N_{Pe} was calculated by multiplying N by d_p/h for each level.

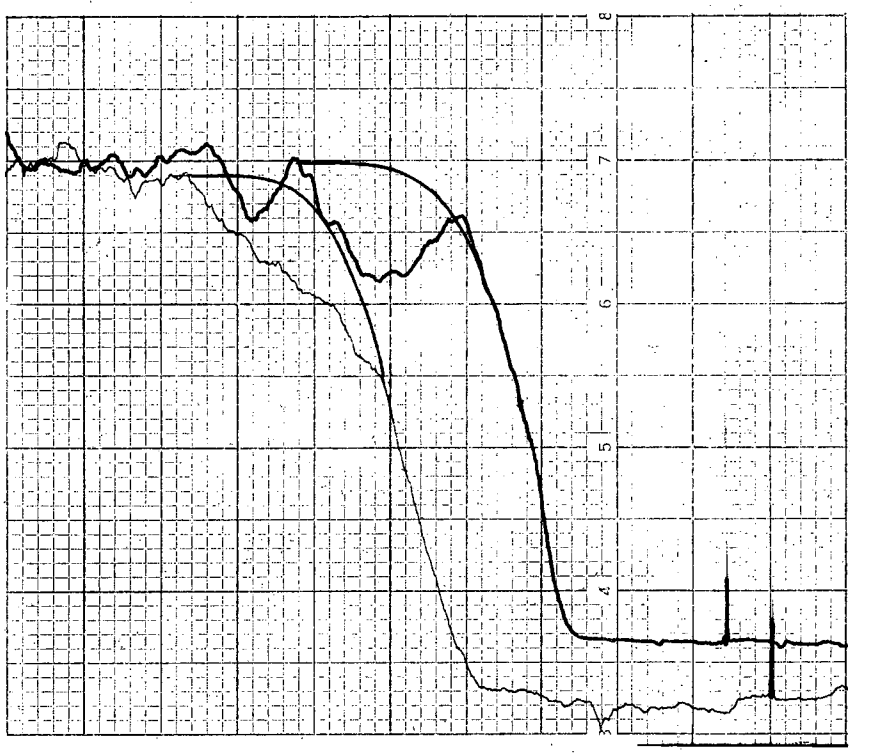

Fig. 21. Recorder chart tracing for Run No. 108.

Table I. Calculation of Peclet numbers for Run No. 108.*

Sampling-cell level = 60 in.				Sampling-cell level = 30 in.			
$t_{0.5}$ = 12.4 sec				$t_{0.5}$ = 6.7 sec			
t (sec)	$t/t_{0.5}$	C (mv)	C/C_0	t (sec)	$t/t_{0.5}$	C (mv)	C/C_0
10.1	0.815	0.3	0.079	5.65	0.844	0.37	0.1098
11.0	0.888	0.8	0.21	6.0	0.896	0.87	0.258
11.65	0.94	1.3	0.342	6.35	0.948	1.37	0.407
12.3	0.992	1.8	0.474	6.9	1.03	1.87	0.555
12.8	1.032	2.3	0.606	7.6	1.132	2.37	0.704
13.4	1.08	2.8	0.737	8.7	1.299	2.87	0.853
14.5	1.17	3.3	0.869	∞	∞	3.37	1.0
∞	∞	3.8	1.0				
$s' = 2.96$				$s' = 1.98$			
$s'^2 = 8.75$				$s'^2 = 3.94$			
$N = 4\pi s'^2 - 0.80$				$N = 4\pi s'^2 - 0.80$			
$= 111 - 0.80 = 110.2$				$= 49.3 - 0.80 = 48.5$			
$N_{Pe} = \frac{110.2}{60} = 1.84$				$N_{Pe} = \frac{48.5}{30} = 1.62$			

*Gas flowrate: 290 lb/hr-ft^2
Liquid flowrate: 13,500 lb/hr-ft^2
Packing: 1-in. raschig rings

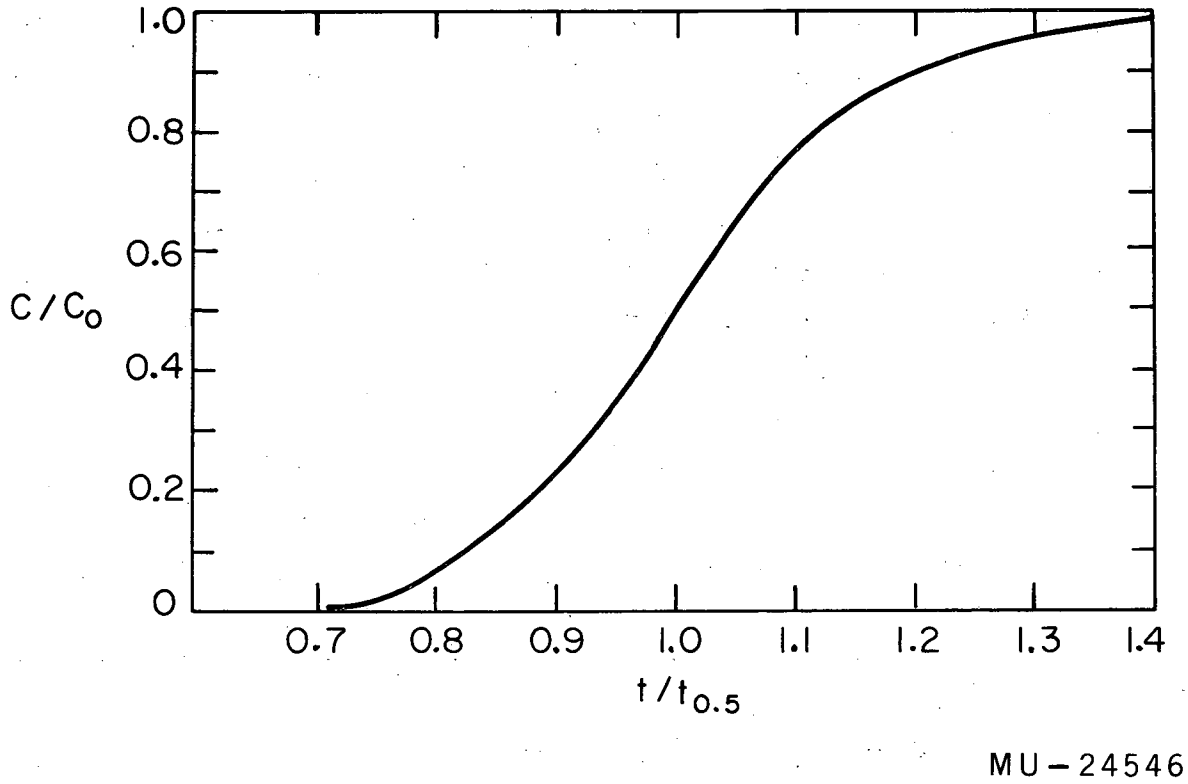

Fig. 22. Breakthrough curve for Run No. 108, 60-in. level.

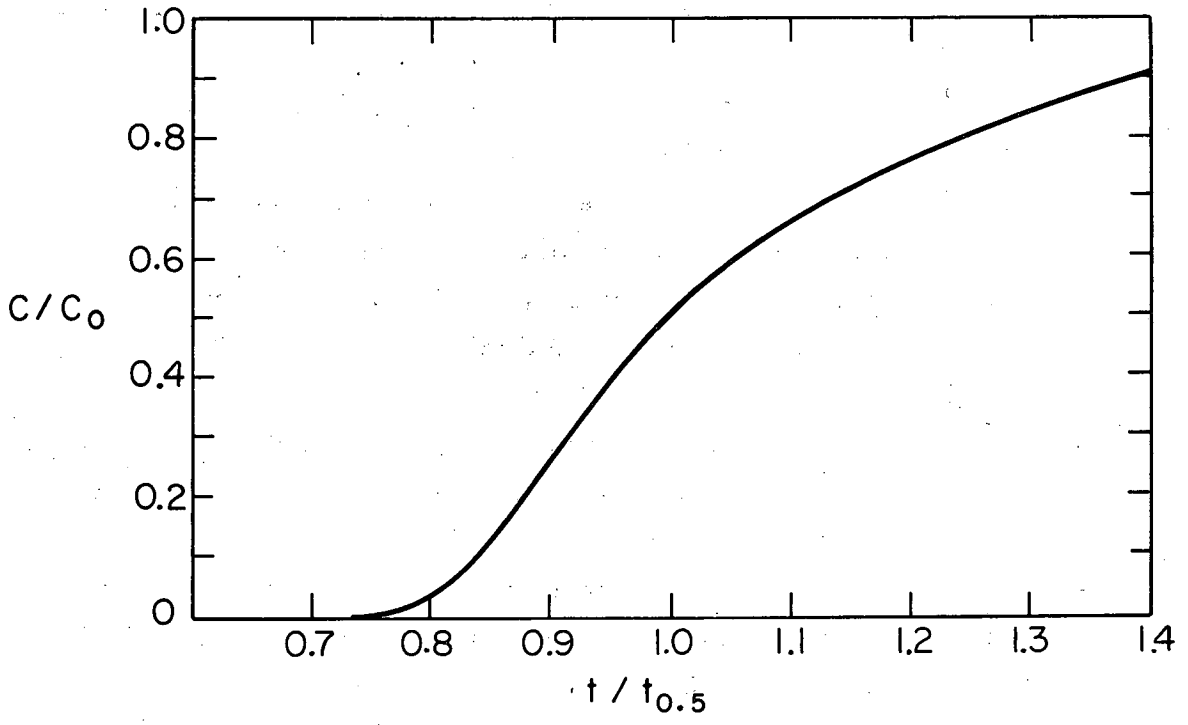

Fig. 23. Breakthrough curve for Run No. 108, 30-in. level.

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