Operation of Viscometer

The filling operation is effected by means of the filling tube illustrated in Figure 3. The asphaltic liquid is warmed to a temperature (60° to 82.22° C., 140° to 180° F.) sufficient to permit its being poured into the 100-mesh copper funnel sieve. The liquid passes through the sieve into the receiver arm of the viscometer and catches at point P where the narrow connecting tube joins the receiver tube reservoir. The flow down through the connecting tubing is controlled by means of pressure changes caused by a roller device (devised by V. Lantz, of these laboratories, and shown in Figure 2) which squeezes a rubber tubing connected to the left arm of the viscometer. To control the rate of flow from the filling tube into the viscometer and to adjust the flow so that the upper surface of the liquid approaches mark M about the same time that the lower surface approaches mark M' on the lower capillary, the ground-glass cap with control rubber tubing is placed on the filling tube. In this way complete control is obtained for the filling operation.

The lower level of the liquid is not immediately brought up to M' but left somewhat below, while the upper level is brought to 1 or 2 mm. above M into the expanded part of the receiving arm. When temperature equilibrium is attained, the lower level is brought exactly to the mark by means of the control roller device and tubing connected to the left arm of the viscometer. Finally, the upper level is brought down to M by use of the suction leveling tube shown in Figure 3.

TABLE II. COMPARATIVE VISCOSITY MEASUREMENTS

Samples	Time of Flow, Modified Ostwald Viscometer	Viscosity: Modified Ostwald viscometer	Viscosity: Koppers viscometer	Difference
	Seconds	*Centistokes*		%
1	56.5, 56.6	21.7 (3)	21.8 (1)	0.37
2	371.3, 370.7	142.6	142.3	0.21
3	121.4, 121.5	395.4	397.9	0.63
4	233.2, 233.8	760.3	762.4	0.28
5	55.5, 55.7	2,119	2,125	0.28
6	104.8, 104.6	3,990	3,972	0.45
7	589.5, 584.7	22,371	22,252	0.53

Observation indicates that the liquid is drawn off sharply at the end of the leveling tube tip and that the hydrostatic head can be reproduced to better than 0.02 cm. Since the least hydrostatic pressure is for the upper bulb and amounts to 7.0 grams per sq. cm., the error in viscosity determination due to the possible error of 0.02 cm. in hydrostatic head is not more than 0.3 per cent; and the error is less than this for the lower bulbs where the hydrostatic head is greater.

After a viscosity measurement has been made, the liquid may be removed readily by applying a vacuum to the leveling tube. Benzene or carbon tetrachloride may be introduced into the left arm, and as it is sucked through the instrument it sweeps the viscometer clean of oil. As the vacuum is continued, the cleaning solvent is soon evaporated. The cleaning operation is thus effected simply without removal of the viscometer from the thermostat bath.

The viscometer is readily calibrated by the use of oils of known viscosity (such as the alpha and beta oils of the American Petroleum Institute) at low enough temperatures to get good flow times. Since the effect of temperature on the calibration constants is less than the experimental error for any usual range in temperature, the bulbs may be calibrated with a single oil by choosing several different temperatures to give the appropriate viscosity range.

Precision and Accuracy

To illustrate the reproducibility of measurement and accuracy of determination of viscosity, data are given in Table II for measurements made with both this modified Ostwald instrument and the Koppers instrument on cutback asphalts. The reproducibility of measurement can be estimated by examination of the column in Table II giving repeated measurements for the times of flow. It is seen that the reproducibility is generally within 0.1 to 0.2 per cent. The modified Ostwald and Koppers instruments yield viscosity determinations that agree within 0.2 to 0.6 per cent over a range of viscosities from 16 to 22,000 centistokes which will include all the liquid road materials from the 0 grade to the 6 grade (example: SC-0 to SC-6).

With a little experience the viscometer can be used as easily and as rapidly as the Saybolt Furol viscometer.

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A Large Spinning-Band Fractionating Column

For Use with Small Quantities of Liquids

ROBERT H. BAKER, CHAS. BARKENBUS, AND C. A. ROSWELL
University of Kentucky, Lexington, Ky.

A 70-plate spinning-band column, which has a holdup of 0.1 cc. per plate has been constructed. The column is practical and inexpensive, and may be used for analytical separations on small quantities of liquid.

MANY types of fractionating columns have been described in recent years to effect the separation of liquid mixtures by distillation. The packed column of the Fenske type (*6*), the all-glass bubble-cap type described by Bruun (*3*), and the recently described spinning-cone type first used by Urey (*9*) and further investigated by Mair (*8*) seem to represent the most efficient ones proposed, if judged by their plate rating or height of equivalent theoretical plate.

The one inherent weakness in all the columns mentioned is their relatively large holdup, which necessitates a fairly large volume of liquid to be fractionated. The value of a fractionating column for analytical separations, especially where small quantities of liquids are to be distilled, is primarily a function of its holdup per theoretical plate. Since very little attention has been given this point, the authors determined the characteristics of several columns which, experience indicated, had a small holdup and a fair separating power.

The results, listed in Table I, clearly show that the spinning-band type of column described by Lesesne (*7*) has the smallest holdup, height of equivalent theoretical plate, and holdup per theoretical plate. It is peculiar that all the columns except the spinning-band type give approximately the same height of equivalent theoretical plate regardless of the mixture used in making the determination, while the spinning-band type

TABLE I. HOLDUP AND PLATE VALUE OF COLUMNS

Column	Length	Plate Value by CCl_4-Benzene	H.E.T.P.	Plate Value by *n*-Heptane–Methylcyclohexane	H.E.T.P.	Holdup per Theoretical Plate
	Cm.		*Cm.*			*Cc.*
Podbielniak	137	10.0	13.7	9.6	14.2	0.30
Vigreux	30	5.1	6	4.2	7.3	0.33
Widmer	36	4.4	8.2	4.4	8.2	0.49
Spinning band	44	19.7	2.2	6.2	7.1	0.12

shows a widely differing value when *n*-heptane–methylcyclohexane is used instead of carbon tetrachloride–benzene. No similar observation could be found in the literature, though Fenske states that the process of enrichment is different for carbon tetrachloride–benzene than for *n*-heptane–methylcyclohexane. In the packed columns used by Fenske, carbon tetrachloride–benzene gives a slightly lower height of equivalent theoretical plate than *n*-heptane–methylcyclohexane. The difference is small, however, compared to the large difference observed in this case.

These preliminary data indicated the desirability of constructing a large column of the spinning-band type. Though the mechanical features do not seem to warrant the easy construction of a long column of this type, nevertheless a 545-cm. (18-foot) column has been built which has proved to be practical and inexpensive.

Construction of Still

The supporting frame of the column was made from two 5-cm. (2-inch) pipes held together at intervals of 73 cm. by crosspieces of boiler plate, welded in place and designed to facilitate the placing of the outer jackets. This frame was bolted to the wall and was sufficiently stable to be used as a ladder as well as a support.

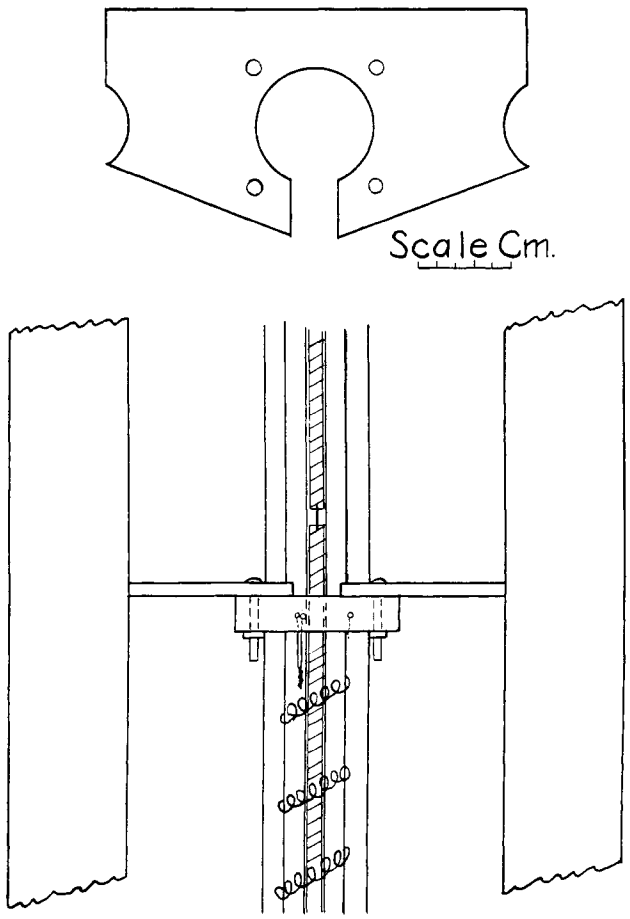

FIGURE 1. CROSS SECTION OF TRANSITE BLOCK
Heater and insulating tubes in place. Top view of iron crosspiece

The column consisted of a glass inner tube, about 6.7-mm. in inside diameter and 545 cm. long, not including the still head. It was made from 150-cm. (5-foot) lengths of Pyrex tubing which were easily fused together after lining them up in the frame of the still. The spinning band which revolved inside this long glass tube was made from strips of Monel metal 30 cm. long, 6 mm. wide, and 1 mm. thick, held together by links made from soft Monel metal wire having a diameter of 0.20 cm. This long spinner with no bearing but the glass tube was fastened to the pulley shaft by a piece of the same kind of wire. The spinner had a clearance of approximately 0.6 mm. and at speeds of 950 r. p. m. had practically no vibration. The still has been run continuously for more than a week at a time with no apparent wear.

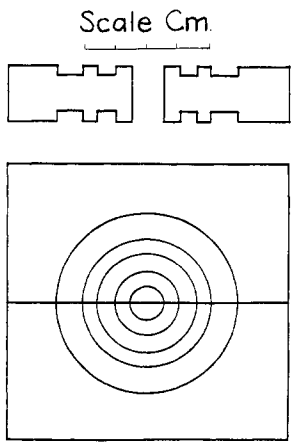

FIGURE 2. END AND TOP VIEW OF TRANSITE BLOCK

Though the column is rather long, it was decided to have the heater coil jacket and insulating jacket made of glass in order to observe conditions inside the tube. This was made possible by constructing the two outer jackets in sections which could be slipped over the inner tube. The heater coil for each section was wound around a Pyrex glass tube, outside diameter 3 cm., which was 72.5 cm. long. The coil, which was made from 24 B. S. gage Nichrome wire 7.2 meters (24 feet) long (40 ohms), was first wound into a tight coil 3 mm. in diameter and then stretched and wound diagonally around the glass tube. Pieces of asbestos paper were placed under the coil and bent up partly to cover the coil. The outer insulating jackets were made from glass tubes of the same length and 5.2 cm. in diameter.

The boiler plate crosspieces placed at 73-cm. intervals between the 5-cm. (2-inch) pipes of the frame were 6 mm. thick and designed as shown in Figure 1. The hole in the center was large enough to allow the large outer glass jacket to go through, thus enabling one to slip the heater and insulating jackets over the lower end of the inner continuous tube and build the sections from the top down.

A side and top view of the Transite blocks which held the outer and inner tubes at each section end is shown in Figure 2. A hole was bored in the center of the block to fit the inner tube fairly tightly. Two circular grooves were cut on both the top and bottom of the blocks in which the outer jackets fitted. The blocks were then cut in half and, after placing them around the inner tube, were bolted to the iron crosspiece. Before the inner tube was built, the holes in the blocks were lined up and the holes to bolt the blocks to the frame were bored to make sure that the inner tube was perfectly in line.

A cross section of the Transite block with the heater and insulating tubes in place is shown in Figure 1, which is drawn to scale. Heater coil wires and thermocouple wires came out through holes in the Transite blocks. With the arrangement all-glass columns of any length can be readily constructed and easily taken apart for repairs.

The bottom end of the inner tube had an interchangeable ground-glass joint, while the still head was designed as shown in Figure 3. It was constructed with a trap at the bottom of the reflux condenser to enable all of the returning liquid to flow through the side tube. This allows one to take off a representative fraction at all times and to measure the return to the still. With a simple screw adjustment the rate of take-off can be readily regulated.

By fitting the bearing of the spinner with a packing, the still can be used for vacuum distillation. It has been run at a pressure of 1 mm. for several hours without any difficulty in maintaining a vacuum. A 150-liter (40-gallon) drum was placed in the system to keep the pressure constant when fractions were taken off through the fraction cutter. The pressure above the boiler liquid, as measured by a manometer filled with butyl phthalate and calculated to mercury, was only 1.7 mm. This small pressure drop enables the still to be used for vacuum work.

TABLE II. DETERMINATION OF TIME TO REACH EQUILIBRIUM

Hours after Operating Conditions Reached	Fraction No.	Throughput per Min.	Refractive Index 20° Head	Refractive Index 20° Pot	Mole Fraction *n*-Heptane Head	Mole Fraction *n*-Heptane Pot	Theoretical Plates	H. E. T. P.
		Cc.						*Cm.*
0.0[a]	1	2.7	1.4010	1.4178	0.60	0.125	35.0	15.3
2.0	2	2.7	1.3933	1.4180	0.820	0.120	51.8	10.5
3.0	3	2.7	1.3906	1.4182	0.910	0.115	64.5	8.5
10.5	4	2.7	1.3904	1.4190	0.920	0.095	69.4	7.8
17.0	5	2.7	1.3898	1.4192	0.930	0.090	72.3	7.5
21.0	6	2.7	1.3908	1.4197	0.900	0.085	68.0	8.0

[a] One hour required before refluxing started and 0.5 hour later rate of throughput was adjusted to 2.7 cc. per minute. This was called zero time. Speed 980 r. p. m.

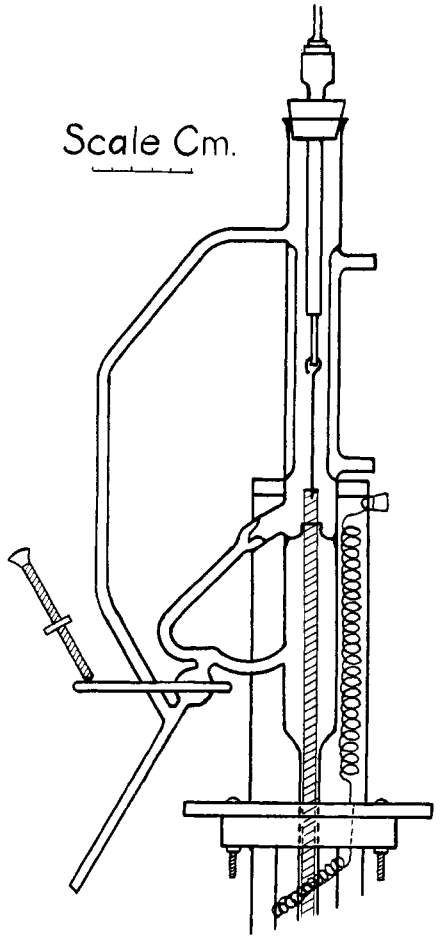

FIGURE 3. STILL HEAD

Characteristics of Column

The theoretical plate value of the still was determined by using a mixture of *n*-heptane and methylcyclohexane. Both compounds were distilled through the still and only those fractions were used which agreed with the index of refraction given in the literature. The plate value was calculated using the formula derived by Fenske (*5*) and a relative volatility value of 1.07 (*1*). The mole per cent values of the two constituents in the head and pot fractions were determined from the data of Bromiley and Quiggle (*2*).

Table II gives the data collected to determine the time necessary for equilibrium to be established. The still in 3 hours reached a plate value of 64.5 plates and then slowly climbed to an average of about 70 plates. These data were taken at a throughput of 2.7 cc. per minute. At a throughput of 5 cc. per minute the value at equilibrium was 48 plates. This approaches the maximum throughput, as slight flooding was observed at the joints of the spinner. Lesesne (*7*) also observed that the plate value fell off with increased throughput. Increasing the speed to 1900 r. p. m. did not appreciably affect the plate value, which is also characteristic of the spinning-cone type of column.

The total holdup of the column, including the still head as measured by the method of Fenske (*10*), was only 7.9 cc. Subtracting the nondrainable holdup of the head—namely, 0.8 cc.—leaves only 7.1 cc. as the holdup of the column. The nondrainable holdup of the still and head is 2.8 cc. A holdup as small as 7.1 cc. or 0.1 cc. per plate would allow one to fractionate as little as 25 cc. of liquid and at the same time have the separating power of a still rating approximately 70 theoretical plates.

All descriptions of columns in the literature stress the low height of equivalent theoretical plate and fail to mention the relationship between holdup and number of plates. This relationship controls the sharpness of separation and, when dealing with small quantities of liquids, is just as important as the height of equivalent theoretical plate. Where head room is available it is much better to have a column rating one theoretical plate for 7.0 cm. of height but holding 0.1 cc. of liquid than to have one theoretical plate for 1.0 cm. of height but holding 0.7 cc. of liquid. The data for this column indicate that it is outstanding in low holdup per theoretical plate and at the same time has a high separating power.

Characteristics of Column under Operating Conditions

In using a fractionating column one is particularly interested in the rate of take-off and how it affects the separating power of the column. In order to find this effect, the still was brought to equilibrium at a constant throughput and then successive fractions were taken off at a constant rate from both the top of the column and the pot. Using a large volume of liquid to start with, the small fractions removed did not noticeably affect the pot composition. From these data the plate equivalences can be calculated for different reflux ratios. The term "plate equivalence" as used here is defined as equal to the number of theoretical plates which would be required at total reflux to produce the observed separation. In Table III these data are collected and are expressed as per cent of the number of theoretical plates at total reflux—that is, percentage plate equivalences. The data clearly indicate

TABLE III. PERCENTAGE PLATE EQUIVALENCES[a] AT VARIOUS THROUGHPUTS

(A value of 70 for the number of theoretical plates at total reflux is assumed)

Distillation rate, cc. per hour:	1.25	2.4	6.0	3	6	10.9	24
	Throughput 160 cc. per hour			Throughput 300 cc. per hour			
Distilled *Cc.*							
1	95	81	..	68	70	69	65
2	83	90	98	62	62	62	57
3	82	80	66	61	53	55	51
4	76	70	45	57	50	50	45
5	75	61	36	57	47	43	40
6	78	61	32	..	45	40	36
7	71	64	32	..	44	36	32
8	73	64	31	..	42	34	29
9	69	..	..	..	40	32	28
10	69	..	..	..	37	31	24
11	..	..	..	..	36	30	24

[a] Arbitrarily defined as $\frac{\text{plate equivalence at operating conditions}}{\text{theoretical plates at total reflux}} \times 100$.

TABLE IV. THEORETICAL PLATES AND COLUMN EFFICIENCIES COMPARED TO TOTAL REFLUX

Reflux Ratio, L/D	Composition of Mole Fractions of Methylcyclohexane Pot	Head	Theoretical Plates at Operating Conditions	Minimum Theoretical Plates at Total Reflux	Efficiency, %
		Throughput 160 cc. per hour			
122.7	0.830	0.165	56	47	84.0
67.5	0.820	0.175	91	45	50.0
27	0.475	0.170	..	..	..
		Throughput 300 cc. per hour			
100	0.740	0.160	51	40	80.0
50	0.470	0.135	41	26	63.4
27.5	0.375	0.125	..	..	..
12.5	0.275	0.110	..	..	..

that the separating power of the column rapidly decreases to a minimum and that this value is dependent on the reflux ratio. Being a batch still, this minimum value will not remain constant but the rate of change will be much slower than the change preceding this steady state.

To find the true theoretical plate value of the column under operating conditions, the method of Dodge and Huffman (4) was used. Only data at the point where the rate of change was very small were used, since it was evident that the number of theoretical plates under operating conditions decreased to this minimum value. These data are listed in Table IV in the fourth and fifth columns. The ratio of these two values expressed as per cent represents the efficiency of the column for the indicated operating conditions.

The data gave conditions that were not operative when the reflux ratio was less than 50. The enrichment is too large, indicating that a steady state had not been reached. If a larger number of fractions had been taken, the enrichment would probably have decreased to an operative condition. The number of theoretical plates at operating conditions of reflux ratio 67.5 and a throughput of 160 cc. per hour is rather high. The record of this run shows that some trouble was experienced in keeping the throughput constant, because of changes in room temperature. This would indicate that a steady state had not been reached and probably this number would decrease and approach the 84 per cent efficiency noted at the reflux ratio of 122.7. The data indicate that at the highest reflux ratios used the over-all efficiency is approximately 80 per cent regardless of throughput. At reflux ratios between 50 and 67.5 the over-all efficiency is probably near 60 per cent.

Acknowledgment

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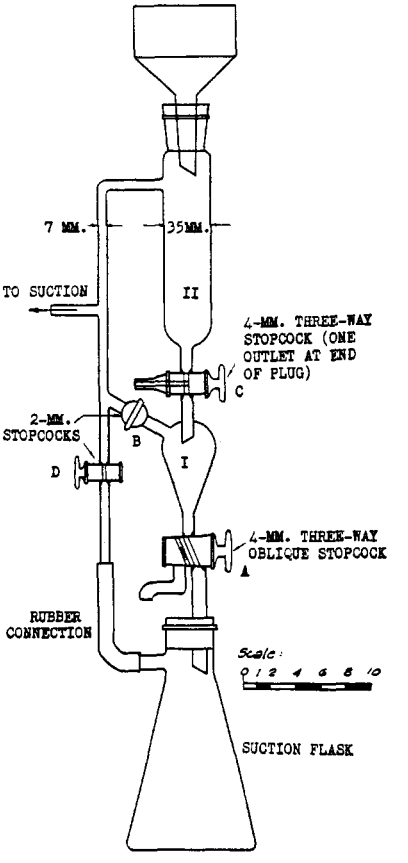
Suction Filtration Apparatus for Sampling Filtrates under Constant Pressure

EDWARD T. FUKUNAGA AND L. A. DEAN
Hawaii Agricultural Experiment Station, University of Hawaii, Honolulu

IN MANY laboratory operations requiring suction filtration, it is necessary to remove small portions of the filtrate in rapid succession for purposes of testing.

In order to obtain clear filtrates continuously while handling many materials on Büchner funnels, constant pressure on the paper must be maintained; a sudden release of the pressure results in a cloudy filtrate when the filtration is resumed. The apparatus illustrated has been satisfactorily used in this laboratory for several years. It eliminates slow and clumsy manipulations with a suction flask and allows rapid removal of aliquots of the filtrate without any disconnections or changes in pressure.



For normal operation, all stopcocks are opened, allowing the filtrate to flow into the suction flask. To sample the filtrate, close stopcock A, allow chamber I to fill with the desired quantity, close stopcock B, turn stopcock C allowing chamber I to come to atmospheric pressure, and draw off filtrate by turning stopcock A to the second position. Then close A, turn C to the off position, and slowly open B. The liquid, which has collected in chamber II, may be run into chamber I and drawn off, or it may be run into the suction flask. The suction flask may be removed at any time after A and D are closed.

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