## A Binary Mixture for Evaluating Low Pressure Distillation Columns

#### Frederick E. Williams HERCULES POWDER COMPANY, WILMINGTON, DEL.

Vapor-liquid equilibria are presented for the di-n-butyl phthalate-di-n-butyl azelate system at 1 mm. pressure. This binary system is shown to be satisfactory for determining the number of theoretical plates in low pressure distillation columns having as many as thirty plates. Two types of low pressure columns were compared by means of the new test mixture.

NVESTIGATIONS on the design and efficiency of fractionating columns for operation in the neighborhood of 1 mm. pressure have been seriously hampered by lack of a suitable test mixture for measuring the rectification efficiency of such columns. Hickman (4) suggested di(2-ethylhexyl) phthalatedibenzyl phthalate for this purpose, but the high relative volatility of this combination restricts its use to a column of two or three theoretical plates. Bishop (1), after examining several binary pairs, recommended either tri-*m*-cresyl phosphate-tri-*p*-cresyl phosphate or di(2-ethylhexyl) phthalate-dibutyl Cellosolve phthalate. The analytical methods for determining the composition of these binary mixtures require relatively large samples and are subject to inaccuracies; in addition, there exists the possibility of ester interchange between the components of both mixtures.

A test mixture suitable for measuring rectification efficiency of low pressure columns should meet the following requirements: (a) The boiling points of the components must be close together and should be in the range 100-200° C. at 1 mm. pressure; (b) mixtures should not deviate too markedly from ideality; (c) any mixture of the two components should be accurately analyzable, preferably by a rapid and simple physical method which requires only a small sample; (d) to avoid operational difficulties in the use of the mixture, the components should remain liquid at temperatures well below their boiling points; (e) the components must be thermally and chemically stable at the distillation temperature; and (f) the components should be fairly readily available and easy to purify.

All of these requirements are met by mixtures of di-*n*-butyl phthalate and di-*n*-butyl azelate,  $C_4H_9OOC(CH_2)_7COOC_4H_9$ . The two esters boil approximately 5° C. apart at 1 mm. pressure; the phthalate ester boils at 150° C. and the azelate ester at 155° C. Mixtures of the two esters do not form ideal solutions, as can be seen from the calculated values of  $\alpha$  in Table I. The departure from ideality is not so extreme as to preclude the use of the mixture for efficiency tests. There is no evidence of the formation of an azeotrope of the two components to limit the usefulness of the mixture.

The azelate ester has a refractive index,  $n_D^{20}$ , of 1.4404 and the phthalate ester 1.4928, a difference of 0.0524 unit; this large difference (48% greater than the difference in refractive index of methylcyclohexane and *n*-heptane, the standard mixture used for evaluating columns at atmospheric pressure) permits accurate analysis of mixtures of the two esters. The esters are liquids at all ordinary temperatures so that no difficulties due to solidification are encountered either in the rectification efficiency test or in the subsequent refractive index analysis. Chemically, the esters have no tendency to react with each other since they are both *n*-butyl esters; the materials are also nonreactive with the metals commonly used in column construction. The thermal stability of the esters is entirely satisfactory. Azelaic acid and di-*n*-butyl phthalate are commercially available and inexpensive. The azelate ester is easily prepared from the acid, and both esters are readily purified by simple procedures.

#### MATERIALS USED

The di-*n*-butyl phthalate used in this work was ordinary commercial material purified by fractionation through a ten-plate column at 1 mm. pressure. The distillate had the following constants: acid number, 0.35; saponification number, 404 (theory, 403.1); and  $n_D^{20}$ , 1.4928.

The di-n-butyl azelate was prepared from azelaic acid obtained from the Emery Industries, Inc. The acid was first purified by crystallization from water using the following procedure: 40 grams of acid were dissolved in 1 liter of water at 80° C., and the water solution was decanted from a small amount of heavy oil. Five grams of decolorizing carbon were added to the solution, and the mixture was allowed to digest for an hour with occasional stirring. After filtering, the clear solution was cooled, allowed to crystallize for 24 hours and centrifuged; the acid was then airdried and recrystallized from water under the same conditions but without the use of decolorizing carbon. A yield of about 75% was obtained from the first crystallization and nearly 100% on the second.

To prepare the ester, the dry acid was mixed with three times the theoretical amount of commercial *n*-butanol, and 0.1% by

TABLE I. VAPOR-LIQUID EQUILIBRIUM DATA FOR THE DI-*n*-butyl Phthalate-Di-*n*-butyl Azelate System at 1.00 mm. of Mercury

Mole % Di-n-butyl Phthalate in:		Refra	Relative		
Liquid (still charge)	Vapor	Liquid (still charge)	Liquid (still residue)	Vapor	Volatility (Caled.)
$\begin{array}{c} 0.00\\ 4.90\\ 15.22\\ 25.20a\\ 25.22a\\ 35.03\\ 44.99\\ 55.02\\ 74.96\\ 89.93\\ 95.12\\ 100.0\\ \end{array}$	0.0 7.1 19.5 31.0 30.8 30.8 40.9 50.8 60.6 69.7 78.5 87.0 91.3 91.3 91.3 90.0	$\begin{array}{c} 1.4404\\ 1.4424\\ 1.4471\\ 1.4518\\ 1.4518\\ 1.4518\\ 1.4518\\ 1.4516\\ 1.4616\\ 1.4669\\ 1.4778\\ 1.4836\\ 1.4836\\ 1.4898\\ 1.4928\\ \end{array}$	$\begin{array}{c} 1.4424\\ 1.4471\\ 1.4518\\ 1.4518\\ 1.4518\\ 1.4518\\ 1.4566\\ 1.4616\\ 1.4669\\ 1.4778\\ 1.4836\\ 1.4836\\ 1.4898\\ 1.4898\\ 1.4898\end{array}$	$\begin{array}{c} 1.4434\\ 1.4491\\ 1.4546\\ 1.4545\\ 1.4545\\ 1.4595\\ 1.4698\\ 1.4698\\ 1.4748\\ 1.4798\\ 1.4874\\ 1.4874\\ 1.4874\\ 1.4874\\ 1.4801\\ \ldots\end{array}$	$\begin{array}{c} 1.483\\ 1.350\\ 1.333\\ 1.320\\ 1.284\\ 1.261\\ 1.258\\ 1.258\\ 1.216\\ 1.189\\ 1.177\\ 1.146\\ \dots\end{array}$
<sup>a</sup> Triplicate	runs on	three different	samples on th	ree differen	nt days.



Figure 1. Vapor-Liquid Equilibrium Diagram for the Di-nbutyl Phthalate-Di-n-butyl Azelate System at 1.00 Mm. of Mercury

weight (based on the azelaic acid) of p-toluenesulfonic acid was added as a catalyst. The mixture was slowly distilled through a ten-plate column with a special head which permitted removal of the lower layer of the heterogeneous azeotrope of *n*-butanol and water. Distillation was continued until the boiling point of nbutanol was reached. A concentrated solution of sodium hydroxide in dry n-butanol was then added to neutralize the acid catalyst as indicated by phenolphthalein. Excess n-butanol was distilled off to a liquid temperature of 150° C., and the last traces of alcohol were removed at 25 mm, using a slow sparge of carbon dioxide. A small amount of decolorizing carbon was then added to the ester and the mixture stirred for an hour at 150° C, with the carbon dioxide. The ester was then filtered free of decolorizing earbon and sodium salt. The yield of the crude ester  $(n_{p}^{20} =$ 1.4406) was nearly quantitative. Final purification was accomplished by distillation at 1 mm. pressure through a ten-plate column. The distillate had the following constants: acid number, 0.40; saponification number, 377 (theory, 373.5);  $n_{\nu}^{20}$ 1.4404.

#### EQUILIBRIUM DETERMINATIONS

The vapor-liquid equilibrium diagram was determined for mixtures of the two esters at 1 mm, pressure. The diagram is shown in Figure 1, and the experimental data for the eleven mixtures on which it is based are given in Table I.

APPARATUS. The equilibrium data were derived from measurements made with the apparatus shown in Figure 2. This is a modification of Othmer's  $(\theta)$  design. Condensation and fractionation were eliminated by holding the walls of the apparatus at a temperature 15° C. above the boiling point of the higher boiling component. Bumping was entirely eliminated by a mass of single-turn <sup>3</sup>/<sub>82</sub>-inch stainless steel helices in the boiling chamber. The volume of helices used was adjusted so that the top surface was submerged to a depth of about 2 mm. when a 50-ml. sample of test mixture was put into the equipment. The volume of the holdup loop was about 1.7 ml., or about 3.4% of the charge volume. This arrangement permitted rapid attainment of equilibrium and prevented any measurable change in the composition of the liquid in the boiler. A wide vapor delivery tube was used to avoid any significant pressure drop between the condenser and still, a matter of utmost importance for distillations at low pressures (4).

Close control of the still pressure was maintained by a thermostated manostat (Figure 3) operating on the principle of the Smits (7) manometer. Dibutyl phthalate saturated with diethylene glycol was used as the upper liquid, and the lower layer was diethylene glycol saturated with dibutyl phthalate. A small amount (about 0.5 %) of sodium nitrite was dissolved in the lower layer to make it sufficiently conducting to operate an electronic relay, which in turn operated a solenoid valve connecting the still to a 5-gallon pressure reservoir. The latter was held below 'I mm, pressure by the continuous operation of an oil pump.

This pair of liquids was chosen for their low vapor pressures, particularly the phthalate  $(10^{-3} \text{ mm. mercury at } 25^{\circ} \text{ C}_{\odot})$ , to permit the production of a reference vacuum of about  $10^{-3} \text{ mm. of}$  mercury, and for the low mutual solubilities of the two liquids. Low solubility of diethylene glycol in the phthalate was necessary in order to keep the concentration of sodium nitrite in the phthalate layer at a minimum and hence reduce the conductivity to a very low value. On the other hand, low solubility of the phthalate in the glycol was desirable in order to maintain the conductivity of the glycol sodium nitrite at a satisfactory value for operation of the Cincephone electronic relay. In actual practice a resistance of 1.5 to 2.0 megohms in the circuit from the manostat to the grid circuit of the relay was necessary to ensure precise control of the pressure.

A mercury vapor pump, backed by the pressure in the reservoir and connected to the manostat through a dry ice trap, provided a reference vacuum for the manostat. To minimize changes in density of the manostatic liquids and prevent changes in their



Figure 2. Diagram of Equilibrium Still



Figure 3. Low Pressure Manostat

mutual solubilities, the manostat proper was submerged in a constant temperature bath held at  $25.0^\circ \pm 0.1^\circ$  C. This control system held the operating pressure constant to at least  $\pm 0.01$  mm, of mercury.

EXPERIMENTAL PROCEDURE. Fifty milliliters of a mixture of known composition of the two esters were charged to the equilibrium still, and the filler tube was sealed off. The still was slowly heated and evacuated with an auxiliary pumping system to degas the liquid. After degassing, the still was connected to the pres-sure-controlling apparatus. As soon as distillate appeared in the condenser, the boiler heater was adjusted to give a distillation rate of one drop per second. The distillation was continued for 6 hours, after which the boiler heater was removed, a stream of compressed air was directed against the bottom of the still, and the wall and oven heaters were turned off. When the still cooled the liquid in the holdup loop and the boiler was removed and analyzed to establish the vapor-liquid equilibrium. No significant change in the composition of the boiler liquid occurred in any of the experiments.

A refractive index-composition curve (Figure 4), plotted from the data of Table I, was used to analyze mixtures of the two esters. The samples of known composition were made up by careful weighing of each component. Refractive indices were measured on a Valentine refractometer reading direct to the fourth decimal place. The refractometer temperature was held at  $20.00^{\circ} \pm 0.02^{\circ}$  C. The precision of these analyses was about  $\pm 0.20$ mole  $C_{c}$ .

#### EVALUATION OF LOW PRESSURE COLUMNS

When a mixture of the two esters is used to evaluate a low pressure column, the number of theoretical plates must be calculated by a graphical method such as that of McCabe and Thiele (5). The deviations from ideality were too large to permit the use of Fenske's (2) algebraic method. However, the mixture is applicable only to columns having thirty plates or less, and, since most low pressure columns have considerably fewer plates, the use of the graphical method is not cumbersome and is probably faster than the algebraic method.

In order to use the X-Y diagram for accurate determinations of plate values, it is desirable to use an enlarged diagram constructed from the experimental values in Table I. Graph paper,  $10 \times 10$  inches, was used in the present work. Low concentrations (about 15%) of the more volatile phthalate ester in the mixture is also helpful in extending the workable range of the X-Y diagram.

Two columns were tested at total reflux with the new test mixture. The first of these consisted of a vacuum-jacketed open tube 13 mm. in diameter and 180 cm. (6 feet) long. The boil-up rate was controlled automatically by a back-pressure manometer. The head pressure was maintained at 1.00 mm. of mercury by a system similar to that used with the equilibrium apparatus. For a given back pressure the column was kept at total reflux for 6 to 8 hours before withdrawal of samples for analysis. Just before the tests were discontinued but while the column was still operating, a pot sample was taken by means of a small glass bucket; this was attached to a metal rod passing through a rubber stopper, which was used to close the still pot.

The efficiency of this column at several back pressures is given in Table II; for comparison, the efficiency of the same column determined with methylcyclohexane-heptane mixture at 720 mm. of mercury is also given. The efficiency of this column at 1.00 mm. of mercury is only 4.5 plates at a back pressure of 42 mm. of propylene glycol (1 mm. propylene glycol = 0.08 mm. mercury), and the efficiency decreases with decreasing throughput. At atmospheric pressure the column has more plates, and the efficiency increases with decreasing throughput. This can probably be explained on the basis of the much higher back pressures required to make the column function at low pressures. Vapor

<sup>1</sup> The authors are aware that, in their tests of column efficiencies, they used pot pressures varying from twice to nearly four times the head pressure of 1 mm., the pressure at which vapor-liquid equilibria were made, and that the vapor-liquid equilibria, if measured at the higher pressures prevailing in the pot, might show significant differences from measurements made at 1 mm. pressure. There is as yet no information on this supposition. On the other hand, when studying the effect of changes in column design for which this test mixture was primarily intended, tests can be made at essentially the same back pressure (assuming comparable boil-up rates), to give reliable comparative results.



Figure 4. Refractive Index-Composition Diagram for Di-n-butyl Phthalate-Di-n-butyl Azelate Mixtures

<b>TABLE</b> II. 13 M	Efficiency [] [m. in Diamete:	Cests on . r and 180 (	an Open <b>-T</b> Cm. (6 Feet	ube Column ) Long
Head Pressure, Mm. Hg	Back Pressure, Mm. Propylene Glycol <sup>a</sup>	Pot	D Head	Theoretical Plates
1.00b 1.00b 1.00b 720c	$\begin{array}{c}13\\32\\40\\2\end{array}$	${}^{1.4437}_{1.4437}_{1.4437}_{1.4437}_{1.4437}_{1.4148}$	$1.4510 \\ 1.4533 \\ 1.4554 \\ 1.4118$	$2.9 \\ 3.8 \\ 4.5 \\ 5.9$
720¢	6	1.4148	1.4122	3.0

1 mm. propylene glycol = 0.08 mm. mercury.
 b Di-n-butyl phthalate-di-n-butyl azelate test mixture.
 Methylcyclohexane-n-heptane test mixture.

TABLE III. EFFICIENCY TESTS AT 1.00 MM. OF MERCURY HEAD PRESSURE ON AN ALUMINUM SPIRAL-PACKED COLUMN 40 MM. IN DIAMETER AND 120 CM. (4 FEET) LONG

Back Pressure, Mm. Pronylene	n <sup>20</sup> <sub>D</sub>		Theoretical
Glycola	Pot	Head	Plates
$     \begin{array}{c}       11 \\       20 \\       40 \\       50     \end{array} $	$1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1.4473 \\ 1$	1,4830 1,4806 1,4787 1,4790	$12.6 \\ 11.7 \\ 10.7 \\ 10.8$
<sup>a</sup> 1 mm. propylene	glycol = 0.08 n	nm. mercury.	

temperatures in the experiments at 1.00 mm. of mercury were higher by at least 15° C. than was to be expected for the operating pressure and indicated that a core of hot vapor, superheated so far as the head pressure was concerned, was passing up through the tube without contacting the refluxing liquid sufficiently to cause it to lose its superheat.

The second column, specifically designed for low pressure operation, consisted of a 40-mm. diameter Trubore tube fitted with a 120-cm. (4 feet) long spiral packing made by turning a square thread in a solid aluminum rod. The spiral had a root diameter of 12 mm., a pitch of two turns per 25 mm. (1 inch), and a thread thickness of 2 mm. Heat losses were minimized by a heated jacket. Boil-up rate was maintained manually and indicated by a back-pressure manometer. In other respects the spiral-packed column was similar to the open-tube column.

Rectification-efficiency tests were made at several back pressures (Table III). This column has 11 to 12.5 plates in its useful operating range, and no difficulties due to false head-temperature readings were encountered. The results of these tests were confirmed by actual distillations, in which the column performed very satisfactorily in applications where its limited efficiency was sufficient to accomplish separation.

In the event that low-pressure columns are encountered which are too efficient to be evaluated with the *n*-butyl esters of phthalic and azelaic acid, a test mixture based on the corresponding ethyl esters can probably be developed which will be applicable. According to the literature (3), ethyl esters of azelaic and phthalic acids have a difference in boiling points of 5°C. at atmospheric pressure, and the phthalate ester is the higher boiling. If these values are correct, the difference in boiling points would probably be somewhat less than  $5^{\circ}$  at 1 mm. pressure. An X-Y diagram of these esters would therefore permit efficiencies in excess of thirty plates to be determined.

#### ACKNOWLEDGMENT

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# Liquid-Vapor Equilibria in High Vacuum

### TWO PHTHALATE-SEBACATE SYSTEMS AT 0.10 MM.

#### Edmond S. Perry and Robert E. Fuguitt

DISTILLATION PRODUCTS, INC., ROCHESTER, N. Y.

O APPARENT interest in the determination of liquid-vapor equilibria in high vacuum has been evinced in the literature. The sole study<sup>1</sup> which has come to the authors' attention is that of Bishop (2, 11) who investigated the possible use of several high boiling compounds as components in binary test mixtures for high vacuum stills. The data were reported for three systems which were considered satisfactory; but extended use of these systems does not seem practical, since methods of analysis of the components were laborious and time-consuming.

Several equilibrium stills for the determination of liquidvapor equilibria have been described by Sameshima (10), Othmer

1 F. E. Williams (page 779) reported a new equilibrium still which is applicable at 1 mm. and which might be suitable for high vacuum studies

(8, 9), and others (3, 7). These stills, as described, would require modification for such determinations at pressures below a few millimeters, since it is well known (4, 5, 6) that the high vacuum distillation of high molecular weight oils requires a minimum pressure drop between the boiler and the condenser. The authors constructed an equilibrium still which extends the design of the tensimeter previously evolved in this laboratory (5) and used it to measure the vapor-liquid equilibria of high boiling binary mixtures at pressures ranging from about 0.01 mm. to any higher pressure commensurate with the thermal stability of the systems.

This equilibrium still was used in particular to measure the vapor-liquid equilibria at 0.1 mm. of two high boiling phthalatesebacate systems, which appeared to have a potential use in