Vapor Pressure of Hexane-Soybean Oil Solutions at High Solvent Concentrations

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THE VAPOR pressure-temperature relations of hexane solutions of soybean oil at solvent concentrations from zero to five weight percentage were reported in a previous paper (1). This range of concentration is encountered in steam stripping residual solvent from solutions which have been concentrated by evaporation. During evaporation the solvent content of the original miscella from soybean extraction is reduced from about 80% to an amount determined by the final temperature and pressure. As knowledge of the properties of solutions in this concentration range, above 5%, is of practical importance in design and operation of preliminary solvent recovery equipment, the original investigation was extended. This paper presents vapor pressure data for the higher concentrations of interest at temperatures of 167°, 212°, and 257°F.

Apparatus

The selection of an experimental method was discussed previously (1), and it was pointed out that a dynamic method was desirable at concentrations above 5% solvent. Limitations of or difficulties experienced with several dynamic methods led to the design of a new apparatus which proved to be simple in operation and yielded reproducible results of adequate accuracy. Although the apparatus is applicable to vapor pressure measurements with any volatile solvent-oil solution, it is of specific value for use with solvents, such as commercial hexane, having a boiling range. For these solvents it is necessary to measure the conditions at equilibrium between the whole solvent and the oil.

The apparatus, shown in Figure 1, includes a 125-ml. flask *A* into which a sample of oil is weighed. The flask has, as an integral part, a device for circulating the oil by means of a vapor-lift *B*. Circulation of the oil is accomplished by solvent entering the apparatus from the reservoir *C*. The flask and connections to the vapor-lift are immersed in a mineral oil thermostat held at a constant temperature by means of a mercury regulator. The solvent, dripping from the reservoir, is vaporized in the tube leading to the flask and enters the vapor-lift through a nozzle. The head of oil in the flask, being greater than that in the vapor-lift which is filled with vapor and oil, causes circulation of oil as in a conventional air-lift pump. The whole solvent dissolves in the oil during the ap-

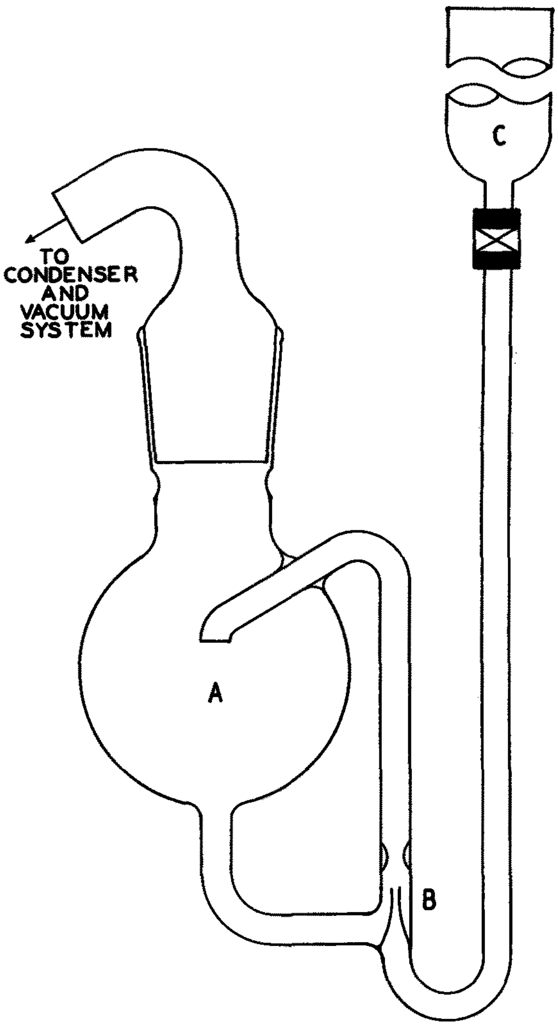

FIG. 1. Vapor pressure apparatus.

TABLE I

Vapor Pressures of Hexane-Soybean Oil Solutions

Temperature								
167°F.			212°F.			257°F.		
Hexane Concentration		P mm. Hg	Hexane Concentration		P mm. Hg	Hexane Concentration		P mm. Hg
Wt. %	Mol. Frac.		Wt. %	Mol. Frac.		Wt. %	Mol. Frac.	
5.97	0.39	231	5.05	0.35	366	3.02	0.24	381
6.92	0.43	271	6.18	0.40	419	3.43	0.27	425
8.87	0.50	318	6.32	0.41	429	4.03	0.30	499
10.95	0.56	364	6.74	0.42	453	4.48	0.32	535
13.85	0.62	415	7.61	0.46	482	4.54	0.33	545
18.70	0.70	505	8.28	0.48	521	5.08	0.35	576
23.55	0.76	562	9.35	0.51	571	5.19	0.36	603
30.07	0.81	630	10.26	0.54	606	5.41	0.37	618
39.50	0.87	716	10.72	0.55	625	5.61	0.38	648
......	......	......	11.30	0.56	646	5.77	0.38	655
......	......	......	11.67	0.57	678	6.02	0.39	688
......	......	......	12.90	0.60	718	6.36	0.41	706

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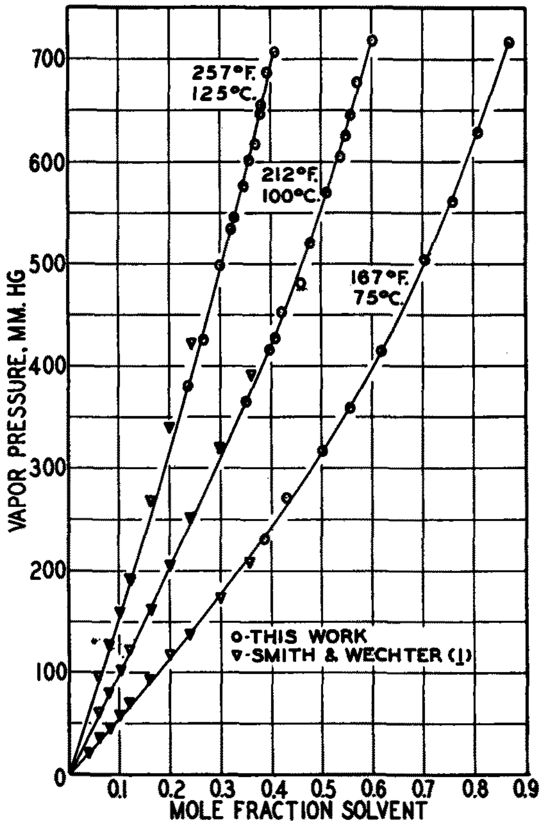

FIG. 2. Experimental vapor pressure data for soybean oil-hexane solutions.

proach to equilibrium and is in contact with the oil after that condition exists. Excess solvent distills from the flask into the condenser and is collected in a receiver cooled by ice.

After an arbitrary circulation period of two hours the solvent content of the oil was obtained by reweighing the flask. A check on the equilibrium time was made in initial runs by replacing the flask after weighing and continuing the run for various periods of time. Pressure on the apparatus was maintained by a vacuum pump operating through a regulator sensitive to ± 1 mm. A single sample of oil could be used for the measurement of several equilibrium concentrations at a constant temperature by starting at a low pressure and increasing the pressure in 50- to 100-mm. increments. The capacity of the flask limited the number of pressure-concentration values which could be obtained with one oil sample.

The crude extracted soybean oil was the same product as that used in the previous investigation. The hexane was Eastman practical grade, having a vapor pressure of 742 mm. at 153.1°F. The hexane used previously had a vapor pressure of 740 mm. at 155.3°F.

Experimental Results

The vapor pressures given in Table I are the experimental values multiplied by the ratio of the vapor pressure of the hexane used in this work to that of the hexane used in the previous work at low concentrations. The tabulated data are comparable therefore with the vapor pressures given previously for low concentrations. Justification of this method of comparing results obtained with different samples of hexane is given in a following paper. Good agreement of the two sets of data is shown by the smooth curves in Figure 2. The agreement between the data obtained by the two methods is apparently as good as that between individual results of each method. The maximum departure of experimental points from smoothed curves is about ± 2% for the concentration range covered in Table I. Interpolation of data between the experimental temperatures at high concentrations is best accomplished by plotting temperature against concentration at constant pressure.

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Solidification Point Curves of Binary Acid Mixtures. VII. Undecanoic to Pentacosanoic Acids[1]

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ALTHOUGH the present state of our knowledge on the occurrence of monocarboxylic *n*-acids of uneven number of carbon atoms in fatty oils probably lessens the practical interest in the solidification of point-curves of mixtures of their adjacent pairs as analytical tools, still there exists an academic interest in them if for no other reason than comparison with those of their "even" homologs.

The mixed melting point curve of binary mixtures of the C_{23} and C_{25} fatty acids which had been prepared by Chibnall and associates (1) for the purposes of reference in the identification of the acids of phrenosin and kerasin of brain tissue suggests studies which are comparable—they are not similar—to those herein reported. Even so however it could not be predicated, *a priori*, that the shape of this diagram sets the pattern for that of the solidification-point curves of binary mixtures of adjacent pairs of the lower homologs of these acids. Those of the C_8 to C_{16} group of this series are characterized by well defined inflections. These inflections are not evident however in the upper areas (4). One school of thought has described them as eutectic points at which is indi-

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