Density-Composition-Temperature Data for Rice Bran Oil-Commercial Hexane Miscellas

FRANK C. MAGNE, RENE L. DURR,[1] and EVALD L. SKAU, Southern Regional Research Laboratory,[2] New Orleans, Louisiana

THE increasing interest in rice bran oil and the application of hexane-extraction in its recovery has emphasized the need for systematic density-composition-temperature data for mixtures of rice bran oil and commercial hexane. This information is essential, for example, as a basis for a rapid method of determining the concentration of hexane-oil miscellas for control purposes in industrial processing.

In order to obtain such data accurate density determinations were made at 10°, 25°, and 40°C. for 11 different concentrations from 0 to 100% of a refined rice bran oil in a commercial hexane. The experimental procedure was the same as previously described (1). The rice bran oil had an iodine value of 102.3. The absolute densities at 25°C. of the rice bran oil and the commercial hexane used were 0.9154 and 0.6801 grams per milliliter, respectively. The experimental results expressed in absolute density units are given in Table I.

TABLE I

Density Data for Mixtures of a Rice Bran Oil and Commercial Hexane

Weight % of oil	10°C.	25°C.	40°C.
	g./ml.	*g./ml.*	*g./ml.*
0.00	0.6931	0.6801	0.6664
9.96	0.7110	0.7003	0.6864
19.41	0.7293	0.7178	0.7054
29.99	0.7514	0.7400	0.7287
39.31	0.7715	0.7601	0.7488
49.23	0.7941	0.7830	0.7722
58.47	0.8147	0.8045	0.7932
68.91	0.8411	0.8301	0.8200
78.73	0.8658	0.8557	0.8454
89.74	cloudy	0.8854	0.8754
100.00	cloudy	0.9154	0.9046

These basic data can be used to calculate the density corresponding to any given concentration at any selected temperature between 10° and 40°C. (1). It was thus possible to construct Table II which gives the complete smoothed specific gravity data at 5% intervals of concentration and at 10 Fahrenheit-degree intervals of temperature. The specific gravity values are with respect to water at 60°F. so as to correspond to the units usually read from commercially available hydrometers. These data can be converted to other density units, if desired, by multiplying by a factor. For conversion to pounds per gallon the factor is 8.337, for pounds per cubic foot it is 62.37, and for absolute density in grams per milliliter it is 0.99905.

By interpolation in Table II it is possible to estimate the specific gravity of any rice bran oil mixture in commercial hexane at any chosen temperature if its concentration is known. Similarly the table can be used to determine the concentration of any rice bran oil mixture if its specific gravity at any temperature is known.

[1] Present address: U. S. Navy.

[2] One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

In general, it is more convenient to interpolate graphically in a large-scale plot of the data. For this purpose the density values of Table II, in the desired units, are best plotted against the temperature, and lines are drawn through all the points representing a given oil concentration. These lines are practically straight and are 21 in number, representing 5% steps in concentration from 0 to 100%.

Though no major error is usually involved from the practical point of view, attention should be called to the fact that the data of Table II are absolutely accurate only for a rice bran oil and a commercial hexane whose specific gravities at 70°F. are 0.9191 and 0.6838, respectively. Specific gravities of rice bran oils or of commercial hexane vary slightly depending upon their sources. If great accuracy is desired, a correction can be made for the error thus incurred so that the data of Table II apply exactly.

To make this correction it is necessary only to know the specific gravity at any temperature of the rice bran oil and of the hexane being used. If the rice bran oil had a specific gravity at 70°F. of 0.9201 instead of 0.9191 as in Table II, it can be assumed that the values for the 100% oil concentration at all temperatures will be 0.0010 higher than the table values. The resulting error for other oil concentrations will be proportional to the percentage of oil in the given mixture (2). For example, the value for a 40% mixture would be 0.0004 higher than the value read from the table at the desired temperature. Similarly if the hexane used had a specific gravity at 70°F. of 0.6828, i.e., 0.0010 lower than the value given for 100% solvent in Table II, the value for the specific gravity of a mixture containing 40% of oil (60% of hexane) would be 0.0006 lower than the table value. The resultant total error is the algebraic sum of the two errors. Thus a 40% mixture of the hypothetical oil in the hypothetical hexane at 90°F., for example, would be the value found from the table, 0.7569, plus 0.0004 minus 0.0006 or 0.7567.

In general, the specific gravity, S, of a mixture containing P% of a given rice bran oil in a given commer-

TABLE II

Specific Gravity °F./60°F. for Rice Bran Oil-Hexane Mixtures

Weight % oil	50°F.	60°F.	70°F.	80°F.	90°F.	100°F.
0	0.6937	0.6887	0.6838	0.6788	0.6738	0.6689
5	0.7028	0.6980	0.6932	0.6884	0.6837	0.6789
10	0.7119	0.7073	0.7027	0.6981	0.6935	0.6889
15	0.7213	0.7168	0.7123	0.7078	0.7033	0.6989
20	0.7313	0.7269	0.7224	0.7180	0.7136	0.7092
25	0.7416	0.7372	0.7329	0.7286	0.7242	0.7199
30	0.7520	0.7477	0.7435	0.7392	0.7349	0.7307
35	0.7626	0.7584	0.7542	0.7500	0.7459	0.7417
40	0.7735	0.7693	0.7652	0.7611	0.7569	0.7528
45	0.7847	0.7806	0.7764	0.7723	0.7682	0.7641
50	0.7961	0.7920	0.7879	0.7838	0.7798	0.7757
55	0.8078	0.8036	0.7997	0.7957	0.7916	0.7876
60	0.8197	0.8157	0.8117	0.8077	0.8038	0.7998
65	0.8318	0.8279	0.8240	0.8201	0.8162	0.8123
70	0.8442	0.8403	0.8365	0.8327	0.8288	0.8250
75	0.8569	0.8531	0.8493	0.8455	0.8418	0.8380
80	0.8698	0.8661	0.8623	0.8586	0.8549	0.8512
85	0.8829	0.8792	0.8756	0.8719	0.8682	0.8646
90	0.8967	0.8930	0.8893	0.8856	0.8820	0.8783
95	cloudy	.....	0.9038	0.9000	0.8962	0.8924
100	cloudy	.....	0.9191	0.9151	0.9111	0.9071

cial hexane can be calculated by means of the following equation:

$$S = S_{II} + \Delta S_{oil}\left(\frac{P}{100}\right) + \Delta S_{hex}\left(\frac{100\text{-}P}{100}\right)$$

where S_{II} is the value read from Table II by interpolation; ΔS_{oil} is the specific gravity of the given oil at any temperature minus that read from the table; and ΔS_{hex} is the specific gravity of the given hexane at any temperature minus that obtained from Table II.

It is apparent that if Table II is used for determining the concentration of a random rice bran oil-hexane miscella an accuracy of one percentage unit will be attained even if the value of $S - S_{II}$ is as large as $\pm$ 0.0018 at low oil concentrations and $\pm$ 0.0030 at high oil concentrations. If a commercial hexane having a specific gravity appreciably different from that in Table II is to be used extensively, it may be desirable to calculate corrections for all the values in the table and thus construct a new table which will be directly applicable with a correction only for an appreciable difference in the specific gravity of the random rice bran oil.

It was found that satisfactory agreement was obtained when Table II was applied to a hexane miscella of crude rice bran oil. This miscella, obtained from a pilot-plant rice bran extraction, had a specific gravity at 77°F. of 0.7042. Analysis by stripping off the solvent showed that it contained 11.84% by weight of oil having a specific gravity at 77°F. of 0.9165. The specific gravity of the original solvent at 77°F. was 0.6812.

The uncorrected specific gravity read from Table II for an 11.84% oil mixture is 0.7031, which is in satisfactory agreement with the experimental value, 0.7042, considering the accuracy attainable by means of commercial hydrometers. Conversely, if the experimental specific gravity, 0.7042, is applied to determine the concentration by use of the table, 12.42% is obtained instead of 11.84%.

Better agreement is obtained if correction is made for the fact that the specific gravities at 77°F. of the oil and the hexane used were 0.9165 and 0.6812, respectively, instead of 0.9163 and 0.6803, the corresponding values obtained from the table. Thus S_{II} in the above equation is 0.7031, ΔS_{oil} equals + 0.0002, and ΔS_{hex} equals + 0.0009, and the calculated specific gravity, S, equals 0.7039, which agrees within experimental accuracy with the value found. The experimental values were determined before the dissolved waxes had time to come out of solution.

Summary

Complete density-composition-temperature data have been obtained for binary systems of a refined rice bran oil with a commercial hexane. They have been presented in the form of a table which gives the specific gravities at 5% intervals of concentration and at 10 Fahrenheit-degree intervals of temperature and can readily be converted to other density units. These data can be used to determine the specific gravity, knowing the composition and the temperature, or, conversely, the composition, knowing the specific gravity and temperature, and should be useful in commercial processing and control. For high accuracy a small correction must be applied for random refined and crude rice bran oils and for different commercial hexanes.

REFERENCES

1. Magne, F. C., and Skau, E. L., Ind. Eng. Chem., *37*, 1097-1101 (1945).
2. Magne, F. C., Hughes, E. J., and Skau, E. L., J. Am. Oil Chem. Soc., *27*, 552-555 (1950).

[Received May 1, 1952]

Rice Bran Oil. VIII. Tank Settlings From Crude Rice Bran Oil as a Source of Wax[1,2]

EDWIN R. COUSINS, S. P. FORE, H. J. JANSSEN, and R. O. FEUGE, Southern Regional Research Laboratory,[3] New Orleans, Louisiana

IN the extraction of oil from rice bran a certain amount of wax is removed with the oil. The yield of wax varies with the temperature at which the extraction is made, the solvent used, source and history of the bran, and a number of other factors. Yields of 3-9%, 5.7%, and 6.4%, on a total lipid basis, have been reported (8, 9, 13). It is probable however that most of the crude rice bran oil presently produced in the United States contains only 1 or 2% of wax because no attempt is made to extract it with the oil.

In spite of the estimated yields of 1 or 2% several facts indicate the wax may become a regular article of commerce. Rice bran oil is being produced in the cars of crude oil per year, and at least two new extraction plants are scheduled to begin operation in the near future. Not only is a sizeable amount of crude wax already available with more becoming available, but it is at present regarded somewhat as a nuisance. The high-melting fraction of the wax is quite insoluble in the oil and settles out as a voluminous, oil-rich sludge which at present has little value.

Tank settlings domestically available, which are generally from crude oil extracted with commercial hexane, vary in composition and quality, as might be expected. Those from crude oil extracted from bran of good quality are usually tan to brown in color and possess the rather pleasant odor characteristic of crude rice bran oil. Their consistency varies from that of soft butter to that of a heavy, viscous liquid.

The solid and liquid portions of tank settlings can not readily be separated by filtration. Any filter fine enough to retain the solids clogs almost immediately on use. The addition of hexane to settlings permits

[1] Report of a study made under the Research and Marketing Act of 1946.
[2] Presented at the 43rd Annual Meeting of the American Oil Chemists' Society, Houston, Tex., April 28-30, 1952.
[3] One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.