TABLE III

Synthesis of Compounds Related to Arylstearic Acids

Compound	Yield %	Boiling Point, °C.	n_D^{25}	d_4^{25}	Molecular Refractivity		% C.		% H.	
					Found	Theoretical	Found	Theoretical	Found	Theoretical
Xylyloctadecanol [a] (1.03 moles aluminum chloride added to 0.78 mole of oleyl alcohol and 5.7 moles of xylene; max. temp., 53°)	78	201-20 at 0.3 mm.	1.4925	0.8985	121.1	120.2	83.25	83.35	12.28	12.38
Phenyloctadecyl Phenylstearate [b] (0.1 mole each of the alcohol and acid; refluxed in xylene solution in presence of dry hydrogen chloride for 14 hours)	51	274-80 at 0.3 mm.	1.4935	0.9146	219.0	218.3	83.66	83.65	11.84	11.70
Methyl Phenylenedistearate [c] (1.65 moles aluminum chloride added to 0.5 mole of phenylstearic acid and 1.0 mole of oleic acid in petroleum ether. Product subsequently esterified with methanol in presence of dry hydrogen chloride)	29	238-73 at .04 mm.	1.4874	0.9283	207.2	204.9	78.67	78.75	11.82	11.72

[a] % OH, found, 4.65; theoretical, 4.54; color, Lovibond, 1" cell = 0.5Y.
[b] Sapon. equiv.; found, 685.0; theoretical, 688.7.
[c] Sapon. equiv.; found, 339.8; theoretical, 335.5.

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Components of "Soybean Lecithin"[1]

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DESPITE years of production and use, information as to the composition of "soybean lecithin" is surprisingly meager. Recent texts and papers repeat the statement that soybean phosphatides consist of approximately 35% lecithin[3] and 65% cephalin (1,2,3,). While lecithin is readily prepared in high purity from the alcohol-soluble fraction through the cadmium salt precipitation (4,5), the cephalin fraction or alcohol-insoluble portion has been poorly characterized. Phosphatides other than lecithin and cephalin are known to be present in soybean oil. McKinney, Jamieson, and Holton (6) have reported the presence of diamino-monophosphoric acid- and monamino-diphosphoric acid-phosphatides and of a glycosidal lecithin complex. Levene and Rolf (7) have reported a fraction which they describe as resembling cuorin. They regarded this fraction as being formed by partial hydrolysis of lecithin and cephalin, but in the light of present knowledge it seems probable that it contained a large amount of inositol-containing phosphatides. As early as 1939 Klenk and Sakai (8) isolated inositol and inositol-monophosphoric acid from soybean cephalin hydrolyzates, but it was not until 1943 that Woolley (9) isolated an inositol-containing phosphatide which he called lipositol. More recently, in 1947, Folch (10) reported the presence of a phosphatide containing both inositol and glycerol. However, insufficient information is available as to the number and composition of inositol-containing phosphatides to permit an estimate of the composition of soybean "lecithin."

Lack of adequate methods for fractionation of the complex mixture of soybean phosphatides has hindered study of their composition. Adsorption and partition chromatography have been found inapplicable (11,12). The technique of "countercurrent distribution," developed by Craig (13,14) and applied with outstanding success in the separation of the penicillins, offered a new method of fractionation. This technique has been highly effective, and some preliminary analyses of soybean phosphatides obtained by this procedure are presented.

Analytical Methods

The procedures used for the determination of nitrogen, phosphorus, choline, amino nitrogen, sugar, and inositol were dictated in part by the size of samples available from the distribution apparatus and in part by complexity of the materials being analyzed.

Total nitrogen was determined by the micro-Kjeldahl procedure. Phosphorus determinations reported in Table I were obtained gravimetrically as ammonium phosphomolybdate. Owing to the small size of

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[3] Except when enclosed by quotation marks to denote the common usage in the trade, the word lecithin refers specifically to phosphatidyl choline.

TABLE I

Analysis of Phosphatide Preparations

Preparation	Per Cent of total weight	Nitrogen	Phosphorus	Molar ratio	Choline nitrogen	Amino nitrogen	Sugar	Inositol
		%	%	P/N	%	%	%	%
Crude phosphatides	100	1.24	3.37	1.27	0.42	0.62	3.96	4.18
Fraction I	50.7	1.65	3.54	0.97	0.81	0.62	2.38	0.26
Fraction II	4.4	1.09	2.87	1.19	0.04	0.81	7.08	2.80
Fraction III	6.6	1.24	3.10	1.12	0.17	1.14	5.52	1.66
Fraction IV	38.3	0.79	3.30	1.89	0.00	0.62	5.15	11.3

the samples obtained from countercurrent distribution, phosphorus in the samples from this apparatus was measured spectrophotometrically by Burmaster's method for total phosphorus (15). In the oil itself phosphorus was determined by a modification of the method of Truog and Meyer (16). Both of the last mentioned methods involve the measurement of optical density of the molybdenum blue complex. Choline was measured by the reineckate method of Glick (17).

Amino nitrogen in the preparations in Table I was determined by the micro method of Van Slyke (18) after refluxing the sample for 24 hours with $2N$ H_2SO_4, and filtering. It was found that the Burmaster periodate oxidation method (19) gave results agreeing with those obtained by the Van Slyke method on the alcohol-soluble phosphatides. Consequently, the Burmaster method was used for the samples from the countercurrent distribution of the alcohol-soluble fraction because of the much smaller sample required. For convenience in handling the large number of samples from this distribution these samples were hydrolyzed with $2N$ H_2SO_4 at 120°C. in an autoclave overnight. In dealing with the alcohol-insoluble phosphatides, the Burmaster method gave results lower than the Van Slyke method, and the Burmaster method was therefore not applicable to this fraction. This is in agreement with the observation of Burmaster (20) that low amino nitrogen values were obtained in fractions of calf brain phosphatides containing inositol, when compared with values obtained by the Van Slyke method.

Sugar was determined by first hydrolyzing the phosphatides for approximately seven hours with $.6N$ H_2SO_4 on a steam bath. After the hydrolyzate was filtered and neutralized, sugar was measured by the method of Stiles, Peterson, and Fred (21). Since Woolley has reported that galactose is present in soybean lipositol (9), all sugar was uniformly calculated as galactose although other sugars were probably present.

Inositol was estimated by a modification of the Atkin, Schultz, Williams, and Frey (22) microbiological method for the assay of pyridoxin. Samples were hydrolyzed with 20% HCl at 120°C. for 16 hours prior to assay.

Preparation of Fractions

The phosphatides used in this work were separated from the oil by passing steam into 10 liters of commercial crude hexane-extracted soybean oil and allowing the hydrated phosphatides to precipitate as a sludge. After removal of this sludge, the steaming, settling, and removal steps were repeated twice. The combined sludges were centrifuged to separate the phosphatides from oil and water. The crude phosphatides were dissolved in diethyl ether, and the resulting emulsion was broken by the addition of sodium sulfate. After evaporation of the ether solution to small volume, the phosphatides were precipitated from the oil with acetone. The precipitate obtained was extracted twice by stirring with acetone, redissolved in ether, and reprecipitated with acetone. The precipitate was washed with successive portions of acetone until the preparation became waxy. Four more extractions were made with the aid of a Waring Blendor. The total weight of the phosphatides was 124 grams. Analytical data on this crude phosphatide preparation are shown in Table I.

Since the crude oil contained 0.072% phosphorus and, after steaming, 0.0035% phosphorus, 94% of the phosphorus in the crude oil was removed and 63% was recovered in the phosphatide preparation.

The crude phosphatides were first separated into alcohol-soluble and alcohol-insoluble fractions. Of the crude material 114 grams were extracted with 350-ml. portions of absolute alcohol six times in a Waring Blendor. The alcohol-soluble portion was further fractionated by removing the solvent under vacuum at 50°C. and again adding absolute alcohol. The alcohol was evaporated from the clear supernatent solution, leaving 55 grams, which are designated in Table I as Fraction I. A small portion, 4.8 grams, which did not redissolve in the alcohol, is designated as Fraction II. The alcohol-insoluble portion was extracted twice in the Blendor with absolute alcohol warmed to 50°C. This alcohol solution was evaporated under reduced pressure at 50°C., leaving 7.1 grams residue designated as Fraction III. The alcohol-insoluble material remaining weighed 41.5 grams and is designated as Fraction IV. (Analytical data on these fractions is also given in Table I.)

The phosphatide fractions were protected from oxidation by keeping them under carbon dioxide as much as possible during their preparation and by storing them at —16°C. in a vacuum desiccator.

Countercurrent Distribution of Fractions I and IV

In principle, the countercurrent distribution apparatus consists of a series of separatory funnels, each funnel containing a pair of immiscible solvents. In the actual apparatus the separatory funnels consist of a series of tubes or holes bored in a cylindrical metal block, and the upper phase of each tube may be readily transferred to the adjacent tube containing a lower phase, thus achieving countercurrent movement of solvents.

To resolve a mixture of solutes, the mixture is introduced into one of the tubes. By shaking and allowing the solvents to separate, each solute of the mixture is distributed between the two solvents in the tube according to its partition coefficient. The alternation of countercurrent movement and mixing of solvents tends to separate individual solutes of the mixture into particular tubes since the rate of movement of each solute through the tubes depends upon its specific partition coefficient. When the desired

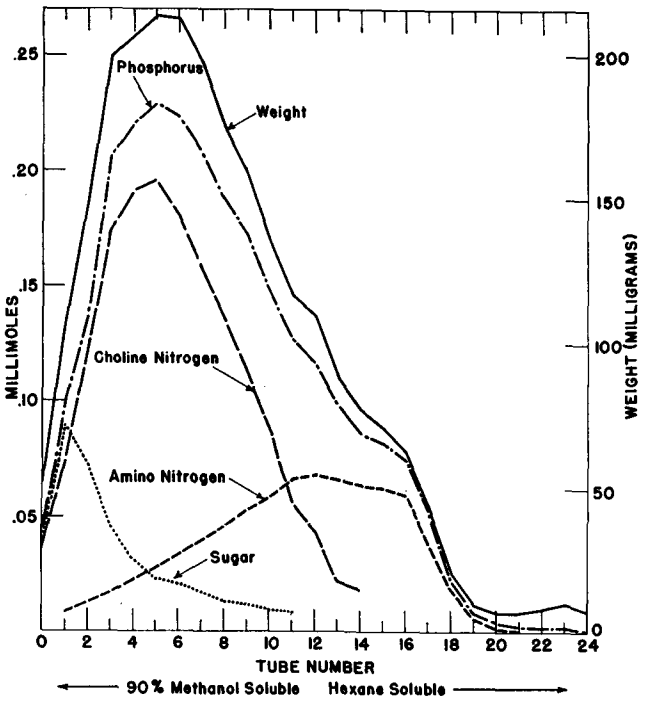

FIG. 1. Results of the countercurrent distribution of the alcohol-soluble fraction.

number of transfers have been performed, the contents of the individual tubes are removed and, after evaporation, the residues are weighed and analyzed for constituents. Choice of the pair of immiscible solvents to be used is made with regard to the partition coefficients of the compounds to be fractionated and with regard to the rapidity with which the emulsion breaks. Because of limitations imposed by the design of the distribution apparatus, it is also critical that the volume of the lower phase does not change on introduction of the sample.

For work with the alcohol-soluble fraction, hexane and 90% methanol were found to be a suitable pair of solvents. Fraction I was dissolved in hexane, and an aliquot containing 497 mg. was distributed between hexane and 90% methanol in the apparatus. After the sample had been carried through the 24 distributions which can be performed in our 25-tube apparatus, it was found that very little material had reached tube 24. In order to obtain better fractionation six additional distributions were applied using the withdrawal technique described by Craig (13). In this technique the contents of tube 24 are withdrawn and replaced by fresh solvent after each distribution.

The distribution operation just described was repeated four times, and the contents of the respective tubes from each experiment were combined. After the solvents were evaporated, the residues were left in a vacuum desiccator until constant weights were obtained. The weights of the fractions are shown plotted against tube number in Fig. 1. Weights of fractions 25-30 are small and are not plotted on the graph. Phosphorus, choline nitrogen, amino nitrogen, and sugar were determined on the fractions wherever a sufficient amount of material was obtained. These results were calculated as millimoles and are also shown in Fig. 1.

For the countercurrent distribution of the alcohol-insoluble fraction, hexane and 95% methanol were found to comprise a suitable pair of solvents. A solution of Fraction IV was made with hexane, and an aliquot containing 500 mg. was distributed between the two solvents in one of the tubes. After the distribution the solvents were badly emulsified, and the contents of this tube were removed and centrifuged to give a clear upper and lower layer with some insoluble material at the interface. The entire sample was then returned to the apparatus with care to avoid re-emulsification. In the succeeding distributions this procedure for breaking emulsions was unnecessary. After the 24 distributions had been performed, the fractions were removed from each tube. This complete distribution operation was repeated as just described. The contents from the respective tubes were then combined and the solvent was evaporated. The residues were left in a vacuum desiccator until they reached constant weight. Phosphorus, total nitrogen, sugar, and inositol were determined wherever a sufficient amount of material was obtained. The analytical values expressed as millimoles are plotted against tube number in Fig. 2 along with the total weight of the fractions in milligrams.

Discussion

Choline- and inositol-containing phosphatides are separated from each other quite satisfactorily on the basis of solubility in absolute alcohol as shown in Table I. However, amino nitrogen constituents are present in both the alcohol-soluble and alcohol-insoluble fractions. Since only a trace of inositol is present in Fraction I and since this fraction has a phosphorus to nitrogen ratio of .97:1, it is concluded the amino nitrogen constituent of the alcohol-soluble fraction is cephalin. Fraction IV is estimated to contain 11% inositol. Also this fraction has a phosphorus-to-nitrogen ratio of 1.89:1, and a phosphorus-to-inositol ratio of 1.70:1. Since cephalin has a phosphorus-to-nitrogen ratio of 1:1, it appears that only a small amount of cephalin (approximately 5%) remains in the alcohol-insoluble fraction. This is contrary to the common idea that lecithin and cephalin are separated on the basis of their solubility in alcohol.

The results of the countercurrent distribution of the alcohol-soluble fraction (Fraction I) are given in Fig. 1. It is apparent from this data that lecithin, cephalin, and a sugar-containing compound are present. When a pure compound is distributed among immiscible solvents in the countercurrent distribution apparatus and the amount of material in each tube is plotted against the tube number (as in Figs. 1 and 2), a Gaussian distribution curve should be obtained (13). In the analysis of a mixture the curve will be the sum of the curves for each component. Thus the weight curves in Figs. 1 and 2 represent the sum of the weights of the components in the mixture. The phosphorus curves represent the sum of the phosphorus contents of all the compounds.

The weight and phosphorus curves of Fig. 1 each show a peak coincident with the peak in the choline nitrogen curve and a hump corresponding to the peak in the amino nitrogen curve. The shape of the choline and amino nitrogen curves are that which might be expected for groups of closely related compounds, such as the lecithins and cephalins containing fatty acids differing in degree of unsaturation and chain

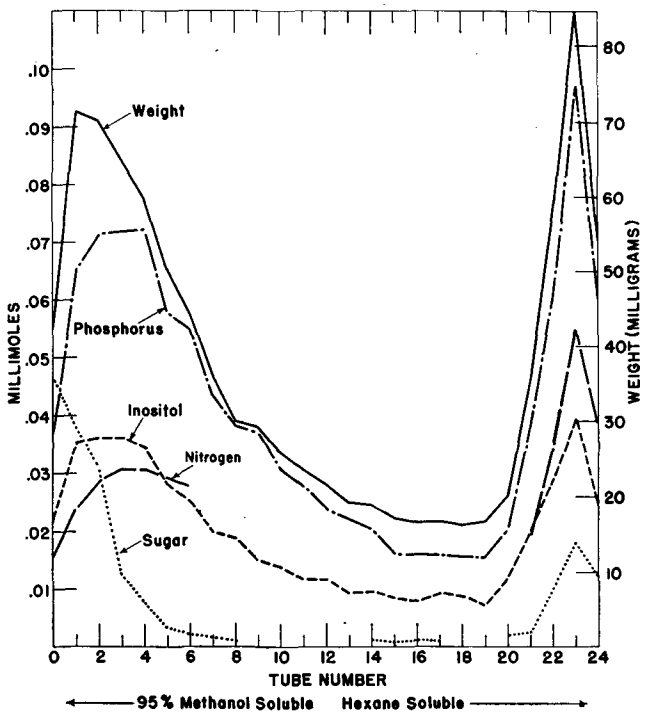

FIG. 2. Results of the countercurrent distribution of the alcohol-insoluble fraction.

length. However, the concentrations of solutes present in these distributions is sufficiently high that partition coefficients may be changing with concentration (23) and in part account for the deviation from the theoretical type curve. It is also probable that complexes exist between lecithin and cephalin and between the phosphatides and the sugar components. Such association effects or mutual solubility effects would also distort the curve.

The countercurrent distribution data for Fraction IV, the "cephalin" or alcohol-insoluble fraction, are given in Fig. 2. It is immediately apparent that the weight curve is composed of two peaks, one in the hexane-soluble portion of the curve and the other in the methanol-soluble portion. The shape of the curve for the hexane-soluble material approaches the theoretical for a single substance while the shape of the curve for the methanol-soluble components is sharp on the left but trails to the right, the latter being indicative of a mixture of related compounds. The most surprising part of the data is that within the limits of experimental error the phosphorus-to nitrogen-to-inositol ratio of 2:1:1 is present in all the tubes, both in the hexane-soluble phosphatides and in the 95% methanol-soluble phosphatides.

The sugar content at the left of the curve is high, 19% of the dry weight in tube 0, and decreases to 0.6% in tube 8. Sugar is also found in the peak at the right of Fig. 2 but in smaller amount. In tube 23 there is 3.9% or one mole of sugar to 5.4 moles of phosphorus. It appears doubtful, therefore, that sugar is a constituent of these phosphatides. Rather, it may be complexed or held by secondary valence forces.

No chemical evidence has yet been obtained which would account for the varying solubility of the inositol phosphatides. The presence of glycerol (10) or differences in number and unsaturation of associated fatty acids may possibly account for the difference in properties. Whether the sugar compounds are present in a sugar phosphatide complex or whether sugar is a component of the inositol phosphatides, the high sugar fractions are more alcohol-soluble, as would be anticipated. The phosphorus-to-nitrogen and phosphorus-to-inositol ratios and the low sugar content of certain fractions are not in agreement with data of Woolley (9) and Folch (10) on the inositol-containing phosphatides from soybean lecithin. Further study is necessary before conclusions can be drawn as to the number and structure of inositol-containing phosphatides.

A rough estimate of the composition of the crude phosphatides used in this work can be calculated from the analytical data in Table I and the information obtained by countercurrent distribution. It should be pointed out that the crude phosphatides examined contained only 63% of the phosphorus of the crude oil. This estimate is distinctly tentative and will be altered as more information becomes available. From the choline nitrogen content of the crude phosphotides, lecithin is calculated as 24% if the molecular weight of 278.3 is assumed for the fatty acids as found by Thornton, Johnson, and Ewan (5).

If the inositol phosphatides are considered as containing one nitrogen for each inositol, and if the remainder of the non-choline nitrogen is assigned to cephalin, an estimate of cephalin content can be made. Assuming the same molecular weight for the fatty acids as was used with lecithin, the crude phosphatides are estimated to contain 25% cephalin.

In a calculation of the amount of inositol-phosphatides some estimate of the percentage of inositol in the pure inositol-phosphatides is necessary. At least two inositol-containing phosphatides appear to be present. However, the inositol and weight curves of Fig. 2 indicate that each tube contains approximately the same percentage of inositol. If the inositol content of Fraction IV is corrected for sugar and cephalin impurities, the inositol containing phosphatides are estimated to contain 12.5% inositol. [Woolley's lipositol was reported to contain 16% inositol (9).] Based on this figure of 12.5%, the crude phosphatides contain 33% inositol phosphatides.

The values calculated above are percentage by weight in the crude phosphatides. The remainder of the crude phosphatide preparation, approximately 17%, can only partially be accounted for as sugars, sterol glycosides, etc. Excluding these materials, 29% of the phosphatides present in this crude phosphatide preparation is lecithin, 31% is cephalin, and 40% is inositol-containing phosphatides. This agrees with the accepted value for lecithin, but it appears that much of the phosphatides formerly considered to be cephalin are actually inositol-containing phosphatides.

Summary

The acetone-insoluble material from soybean "lecithin" has been fractionated by submitting alcohol-soluble and alcohol-insoluble portions to countercurrent distribution between hexane and methanol. The alcohol-soluble portion was found to contain lecithin, cephalin, and sugars or glycosides; the alcohol-insoluble portion was separated into two major inositol-containing phosphatides and sugars or glycosides. While the commonly accepted value of 30-35% for lecithin in the phosphatides was confirmed, it appears that the accepted value of 65% of cephalin

needs revision. The approximate composition for one sample of soybean phosphatides is estimated to be 29% lecithin, 31% cephalin, and 40% inositol-phosphatides.

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Laboratory Extraction of Cottonseed With Various Petroleum Hydrocarbons*

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Summary

Solvent extraction of cottonseed with various hydrocarbons in the C_5 to C_7 range was studied to determine comparative yields and quality of oil produced with pure and commercial hydrocarbon solvents. The solvents used were Pure Grade (minimum 99 mol per cent purity) isopentane, normal pentane, cyclohexane, normal heptane, and benzene; Technical Grade (minimum 95 mol per cent purity) neohexane, diisopropyl, 2-methylpentane, 3-methylpentane, normal hexane, methylcyclopentane, and a 90 mol per cent cyclopentane; and Commercial Grade normal pentane, isohexanes, normal hexane, isoheptanes, and normal heptane.

The effects of precooking the seeds and of the inclusion of hulls on yield and color of extracted oil were determined. Yields and colors of crude oils from cottonseed with both high and intermediate free fatty acid content were studied, and characteristics of the oils determined and compared. Refining losses, refined colors, and bleach colors of oils extracted with various hydrocarbons were compared. Color comparisons were made with a Gardner Color Comparator and a Fisher Electrophotometer. Other test methods used were modifications of official A.O.C.S. methods adapted to small samples. In general normal paraffins and isoparaffins were found to have some advantages over cyclic hydrocarbons.

Introduction

IN the course of the development of extraction processes for vegetable oil production many improvements have been made in the type and quality of hydrocarbon solvents available to the solvent extraction industry. Major improvements in solvent quality have been achieved by narrowing the boiling range and eliminating the undesirable sulfur and nitrogen compounds and unsaturated hydrocarbon components. However, only limited attention has been given to the relative merits of the individual hydrocarbons contained in current commercial petroleum solvents.

The present commercial solvents used by the vegetable oil extraction industry have usually been so selected to include a major proportion of normal hexane. This selection has certain favorable technical and economic aspects. Commercial plant operations have demonstrated that the use of n-hexane cuts is economical from the standpoint of extraction efficiency, particularly when the boiling range is adjusted so as to maintain solvent losses to a minimum. Furthermore, a n-hexane fraction is obtainable in relatively good yields from natural gasoline which has long been the primary source of many types of solvents and pure hydrocarbons. The users of n-hexane solvent have, therefore, benefited by the improved separations such as the Superfractionation process, which has been applied to natural gasoline to yield products of ever-increasing quality.

Developments within the petroleum industry during the past several years have altered to some extent the former concepts regarding the availability of various hydrocarbons. Many less familiar isomers of the five, six, seven, and eight carbon compounds have been synthesized and/or separated on a commercial basis. This is particularly true of the isoparaffins which have become commercial products as the result of the development of the newer alkylation and isomerization processes for the production of blending components for aviation gasoline.

As a result of these developments, it was considered advisable to study, on a laboratory scale, a group of the more readily available cycloparaffins, isoparaffins, normal paraffins, and aromatic hydrocarbons as solvents for cottonseed oil extraction. The experimental solvents were confined to those boiling near n-hexane since most existing commercial cottonseed oil solvent extraction facilities have been designed for operation with n-hexane fractions.

* Presented at the 39th annual meeting of the American Oil Chemists' Society in New Orleans, May 4-6, 1948.