cc. of a saturated alcoholic solution of magnesium nitrate (95 per cent alcohol saturated with $Mg(NO_3)_2 \cdot 6H_2O$ crystals) was added, and the sample placed on the steam bath. A tough surface layer was formed by heating overnight. The dish was then placed on an electric hot plate and carefully heated until the sample charred; too rapid heating was avoided. The charred sample was then placed in a cool muffle furnace and heated to 600° C. for three hours or until completely oxidized. The evaporating dish was then cooled and the fluffy ash carefully moistened with water and then dissolved with concentrated hydrochloric acid. The solution was transferred to a 250 cc. beaker, evaporated to a small volume, filtered, and the filter washed free from chlorides with hot water. Filtrate and washings were collected in another 250 cc. beaker and the volume adjusted by evaporation. The phosphorus was determined in the total sample according to the volumetric method described in the Official and Tentative Methods of Analysis of the A.O.A.C.[1]

For convenience in expressing the results it was assumed that all of the phosphorus present in the samples was combined in a stearyloleyl lecithin having the formula $C_{44}H_{88}O_9NP$ and containing 3.85 per cent phosphorus. The data thus obtained are shown in Table 1.

TABLE 1

Car Number	Days in Storage	Per cent Lecithin in Top Oil	Per cent Lecithin in Middle Oil	Per cent Lecithin in Bottom Oil	Description of Bottom Oil* Amount of Precipitate	Description of Bottom Oil* Color
1**	112	0.72	0.65	5.65	Medium	Normal
2	104	0.77	0.83	20.17	Large	Very light
3	100	0.85	0.85	6.48	Medium	Dark
4	97	1.15	1.11	10.63	Small	Dark
5	93	1.07	1.05	13.80	Medium	Dark
6	89	1.18	1.14	22.35	Large	Light
7	73	1.08	1.04	6.55	Large	Normal
8	69	1.28	1.29	9.33	Medium	Light
9	66	1.45	1.45	5.36	Large	Light
10	64	1.41	1.41	7.28	Trace	Dark
11	46	1.25	1.24	3.92	Trace	Normal
12	29	1.14	1.16	4.37	Trace	Normal
13	16	1.40	1.34	1.60	None	Normal
14	6	1.37	1.32	1.45	None	Normal
15	3	1.27	1.34	3.08	Trace	Normal

*All of the Top Oil and Middle Oil was free of precipitate and of normal color.
**This car was first loaded 315 days before sampling and the oil has been refiltered four times, the last time 112 days before sampling.

Discussion. In Table 1 the cars are arranged according to the time of loading, the cars filled first being at the top of the table and those filled most recently being at the bottom. When considered according to the age of the oil these cars can be divided into three groups. The cars which have been standing more than seventy-five days consist of an upper layer of oil containing less phosphatides than the fresh oil normally contains, and a layer on the bottom of the car which contains much more phosphatide than a normal oil contains. The cars which have been standing for a period of twenty-five to seventy-five days show this same accumulation of the phosphatides in the lowest layer, but to a lesser extent; in most cases the solid material is just beginning to accumulate on the bottom of the car. The oils which have been standing less than twenty-five days are still fairly uniform in composition, resembling the fresh oil throughout. Comparison of the surface and middle oil shows that the upper half of the car is of uniform composition in every case.

The large differences in phosphatide content of the bottom layers of oils of about the same age cannot be accounted for satisfactorily due to lack of data. However, it has been shown that when moisture is mixed into the oil there is a separation of yellowish hydrated phosphatides.[7] It is probable that the cars containing a thick sludge having a high phosphatide content are those which contained some moisture when the oil was loaded and that this moisture has hydrated the phosphatides of the oil and produced a more rapid separation than would occur in ordinary settling.

SUMMARY

Fifteen carloads of crude soybean oil which had been in storage from three to 112 days were sampled at three levels and the total phosphorus content of each sample determined. The oil from the surface to the middle of the car had a uniform phosphatide content. The phosphatide content of the material on the bottom of the car increased with the increasing length of the storage period and this increase was accompanied by a decrease in the phosphatide content of the oil in the upper layers of the car.

ACKNOWLEDGMENT

The author desires to thank Mr. Young and Mr. Adkins of Ralston Purina Company for their help in making the samples available.

BIBLIOGRAPHY

1. Association of Official Agricultural Chemists. "Official and Tentative Methods of Analysis." 3rd ed., p. 16, 1930.
2. Eastman, W. H., "The Industrial Utilization of Soybean Oil and Soybean Oil Meal," an address given before the Annual Convention of the American Soybean Association, Washington, D. C., September 2, 1932.
3. Gardner, H. A., Paint Manufacturers' Association of the U. S., Circular No. 67, August, 1919.
4. Jamieson, G. S., and McKinney, R. S., Oil and Soap **12**, 70-2, 1935.
5. Junker, M., Allge. Oel- und Fett Zeitung **30**, 492-5, 1933.
6. Pickard, G. H., "Soy Bean Oil," American Paint Journal Co., St. Louis.
7. Smith, R. L., Master of Science Thesis, Purdue University, June, 1931, and H. R. Kraybill, U. S. Patent 2,069,187, Jan. 26, 1937.

PROPOSED METHOD FOR COLD TEST ON REFINED OILS

Four ounce sample bottles which are perfectly dry are used in this test. If there is any doubt as to the dryness of the bottle, place it in an oven at 105° C. for one hour and cool in a desiccator. These precautions are necessary because a small amount of moisture will vitiate the test.

If the oil sample is received during cold weather and seems to have been chilled already, heat to 35°, and hold it 4 to 5 hours at about 25° C. (or preferably over night) to put the oil in proper condition for the test.

The sample must always be tested as received (except as modified in the preceding paragraph where the sample has been received in cold weather or has been chilled already).

However, if it is desired to determine whether a failure to pass the test is due to moisture in the oil, heat the oil to 105° C. and hold at this temperature until the last traces of moisture have been removed, then cool to 25° C. and hold overnight at this temperature before repeating the test.

Fill the bottle half full of the oil, insert a cork stopper tightly and cut off the end of the latter flush with the top of the bottle. Seal well with paraffin. Immerse in a container filled with cracked ice and just sufficient cold water to fill the container to the top of the

ice. Draw off water and replenish the ice as necessary to keep the container solidly filled with cracked ice. At the end of five and one-half hours from the time of immersion, remove the bottle and examine the oil. Winter oil must be clear, brilliant and limpid at the conclusion of the test.

The bottle must not be disturbed during the five and one-half hour period, nor must it be agitated when it is removed from the bath at the end of the test, as agitation will occlude air bubbles in the cold, viscous liquid. If it is desired to examine the oil during the duration of the test, additional samples should be introduced into the bath and examined when desired. The results, however, must be reported on a sample which is not removed until the five and one-half hour period has elapsed.

Editor's Note: This method is being considered by the Uniform Methods and Planning Committee, as a substitute for the present Cold Test Method.

ABSTRACTS

Oils and Fats

Edited by
W. F. BOLLENS and M. M. PISKUR

Determining the number of double bonds in oils and waxes. E. Rossmann. *Angew. Chem.* **50,** 187-90 (1937). A review of methods. The Br vapor method is emphasized.

The benzine point, a new characteristic for castor oil. W. Leithe and H. J. Heinz. *Fette u. Seifen* **44,** 33-4 (1937). Procedure: Weigh 6 g. castor oil in a 25 c.c. Erlenmeyer flask, add exactly 10 c.c. benzine (d^{20} 0.706, $n_D(20°)$—1.3956). This is stoppered with a stopper contg. a thermometer graduated in 0.2°. The flask is heated while being shaken in a warm water bath until the contents become clear. It is then removed from the bath and shaking is continued until turbidity appears. The temp. at which turbidity appears is called the benzine point. This figure was 32.2 to 34.0° for 20 commercial samples. Adulteration of castor oil with 5% linseed, sunflower, peanut, soybean, or rape oil reduces this figure 2.7-3.2°; 10% adulteration causes a reduction of from 4.2 to 6.5°.

Characteristics of fat from mold. (Citromyces spec.). K. Taufel, H. Thaler and H. Schreyegg. *Fette u. Seifen* **44,** 34-8 (1937). The composition of the fat was glycerin 4.9, unsapon. 9.9, palmitic acid 5.8, stearic acid 10.0, oleic acid 34.4 and linoleic acid 34.4%.

Fat from yeast (Saccharomyces spec.). K. Taufel, H. Thaler and H. Schreyegg. *Z. Untersuch. Lebensm.* **72,** 394-404 (1936). The fat of yeast, *Saccharomyces spec.,* is composed of: glycerin 5.3, steam volatile acids 5.2, palmitic acid 9.5, stearic acid 5.9, oleic acid 47.6, linoleic acid 2.9, and unsapon. 19.6% (stearin 3.3 and squalen 16.3).

The detection of animal fats and oils especially hardened train (marine animal) oil in fat mixtures. S. H. Bertram. *Öle, Fette, Wachse, Seife, Kosmetik* **1937,** No. 2, 13-14. In an investigation on the *Tortelli-Jaffe reaction* the author noted that (1) the green color reaction with hardened train oil occurred when the prescribed AcOH was omitted; and (2) when the prescribed $CHCl_3$ was replaced with CCl_4, CH_3CHCl_2, or MeI, the reaction was negative; when it was replaced with MeBr or C_6H_5COCl the color reaction was weak; while replacing the $CHCl_3$ with perchloroethylene (C_2Cl_4) or $C_2H_2Cl_2$ the reaction was significantly stronger. With a purified $C_2H_2Cl_2$ the reaction was weak. Because of the weaker reaction in this purified solvent a check was made to ascertain whether $CHCl_3$ acted similarly. When pure $CHCl_3$, prepd. from alc., was used in the test on train oil, the reaction was negative. Use of impure $CHCl_3$ solvents, *i.e.,* contg. aldehydes yielded excellent positive reactions. A new procedure proposed for the detection of animal oils (except hog fat) was:—1 cc. of oil or fat is mixed with about 3 g. crystd. trichloroacetic acid in a test tube and heated 5 mins. at 60° in an oil bath. The tube is removed from the oil bath and 10 cc. $CHCl_3$ are added. Development of a violet color indicates the presence of animal oil or fat except for hog fat, for which the reaction is negative. An intense violet color is obtained with whale, seal, herring, pilchard, shark and egg oils; the reaction being equally good for the partially hardened and unhardened oils. Beef fat, butter fat, sperm oil and horse fat give weak reactions. A green color with strong fluorescence occurs with ergosterol. Pure cholesterol and pure phytosterol yield no color with the procedure. (*Chem. Abs.*)

The detection of arachis oil in olive and almond oils. Norman Evers. *Analyst* **62,** 96-100 (1937). Olive oil—A modified Bellier test was carried out: One cc. of oil is saponified with 5 c.c. of 1.5 *N* alcoholic KOH soln. by heating on a water-bath for 5 min.; 50 cc. of 70% alc. are added, followed by 0.8 cc. HCl (sp. gr. 1.16). Soln. is warmed and then cooled in water, while stirring with thermometer, so that the temp. falls 1° C. per min. If oil remains clear at 9° C., arachis oil may be regarded as absent. Almond oil—The test is identical except that the soln. must remain clear to 4° C. In both cases, olive and almond oils, 5% arachis oil will be detected. If the tests are positive, a usual confirmatory test for arachis oil should be carried out.

Proof for small amounts of butter fat in presence of cacao fat. J. Grossfeld. *Z. Untersuch. Lebensm.* **72,** 434-5 (1936). The butyric acid value was used as a criterion for detg. the percentage of butter fat in prepared samples of cacao fat contg. 0.8 to 11.3% butter fat. Calculations to ± 0.3 were possible using the formula: butter fat = 5.12B — 0.12R. B represents the butyric acid value and R the "restzahl" (residual value). The "residual value" is defined as the No. of c.c. of 0.01 *N* NaOH corresponding to the total amount of fatty acids in 0.5 g. sample which was not pptd. by $MgSO_4$ minus the butyric acid value.

Refractometric determination of fat in chocolate. J. Stanley. *Ind. & Eng. Chem., Anal. Ed.* **9,** 132-135 (1937). A rapid refractive method for detg. total fat in chocolate is described. The data necessary for use