ice bath while mixing. Mixing time was 10 min. The results were not good, and duplicates did not agree. It is suspected that the jar used was too large and that too much air space existed above the meal sample and that hexane vapors were lost in transfer and also leaked out around the seal for it is rather difficult to form a vapor-tight seal around a moving shaft.

A second method of mixing was by natural diffusion. The miscella was added in small amounts throughout the entire meal sample at the time of weighing. The finished sample was placed in a jar with the sample completely filling the jar. The sample was allowed to stand at room temperature for one hour, then refrigerated for two hours. The results were better than the first method of mixing but were not considered reliable as duplicates did not agree more closely than 24°F. at the .1% hexane level.

A third method of mixing was tried, using a combination of the other two methods described. A sample of meal was weighed out. Miscella was added continuously during weighing so that miscella was rather thoroughly distributed throughout the sample. The sample was then placed in a jar just large enough to contain the entire sample. Mixing was accomplished by stirring, using the apparatus described in Method 1. Much better results were obtained by using this method. In one case duplicates agreed exactly, *i.e.*, 120°F. each at the 0.1% level of hexane.

In general, it was found that by using hexane in the form of miscella instead of "raw" that the flash points were from 20° to 30°F. higher for any given percentage hexane in the ranges selected. Also the same thing held true for miscella as for hexane in the matter of attenuation because of time. Samples kept any length of time, such as over-night, in a refrigerator flashed at higher temperatures than when run immediately after mixing and cooling. In the lower concentrations of hexane it was found that no flashes resulted after 24 hrs. where flashes had been found previously or when the samples were fresh.

In conclusion, it is thought that the flash-point tester has some merit but likely is not the ultimate answer to this problem. A great deal more work should be forthcoming on this and other methods in order to assure an accurate and reliable test for this pressing problem.

JAMES K. SIKES

[Received October 21, 1959]

The Determination of the Neutral Oil Content of Crude Vegetable Oils

R. BASU ROY CHOUDHURY and LIONEL K. ARNOLD, Iowa Engineering Experiment Station, Iowa State University of Science and Technology, Ames, Iowa

THE VARIOUS ATTEMPTS to devise methods for determining the refining loss of crude vegetable oils have resulted in three generally recognized methods: acetone-insoluble, Wesson, and chromatographic. The acetone-insoluble and the Wesson methods are not only time-consuming and elaborate but require considerable skill. Neither the Wesson method nor the A.O.C.S. cup method (3) give the actual percentage of crude oil. The chromatographic method, which is receiving increasing attention, uses a very flammable solvent, ether, which makes it undesirable, particularly for routine use.

The current method was based on the silicic acid column chromatographic method (1). Instead of using the usual column, the determinations were carried out in Erlenmeyer flasks. The apparatus used was 125-ml. Erlenmeyer flasks, sintered glass funnels, and a vacuum oven. The reagents were reagent-grade chloroform and reagent-grade powdered silicic acid. The soybean oil was a crude expeller-produced oil with a free fatty acid content of 0.6%.

Five grams of soybean oil were shaken for 10 min. in a flask with 50 ml. of chloroform and varying amounts of silicic acid and were filtered under vacuum. The silicic acid on the filter was washed with varying amounts of chloroform. The neutral fat content was also determined by the chromatographic method of Linteris and Handschumaker (2). The results are shown in Table I. Very good agreement with the results from the chromatographic method was obtained when the silicic acid was washed with a minimum of 300 ml. of chloroform.

TABLE I

Amounts of Neutral Oil Recovered from 2-g. Samples of Soybean Oil by Different Volumes of Wash Chloroform

Silicic acid in grams	25	50	100
Wash chloroform in ml.	Neutral oil in percentages		
100	88.0	87.8	87.5
150	88.8	89.0	88.7
200	89.6	89.5	89.0
250	92.6	92.5	92.4
300	93.1	93.0	92.9
500	93.1	93.1	93.0

Recovered by chromatographic method, 93.1%.

To determine if satisfactory results could be obtained with less chloroform, 2-g. samples were run with 25 g. of silicic acid and 50 ml. of chloroform. Results, using 250 ml. of wash chloroform (Table II), checked with those using 300 ml. of chloroform with the 5-g. oil samples.

Neutral oil was determined by this method on samples of corn oil and cottonseed oil. The corn oil was crude, produced from corn germs processed by prepressing, followed by solvent extraction, and had a free fatty acid content of 4.3%. The cottonseed oil was a crude expeller-produced oil with a free fatty acid content of 1.7%. Results are shown in Table III.

As the result of this work the following method for

TABLE II

Amounts of Neutral Oil Recovered from 2-g. Samples of Soybean Oil by Different Volumes of Wash Chloroform. Silicic Acid: 25 g.

Wash chloroform in ml.	Neutral oil in percentages
100	91.0
150	92.9
200	93.0
250	93.1
300	93.1
500	93.1

Recovered by chromatographic method, 93.1%.

the determination of neutral oil is proposed. Shake 2 to 3 g. of crude oil, 25 g. of silicic acid, and 50 ml. of chloroform in a 125-ml. Erlenmeyer flask for 10 min. Filter through a sintered glass funnel under vacuum, washing the silicic acid with five 50-ml. lots of chloroform. Combine the filtrate and washings, and evaporate the chloroform on a water bath. Heat to constant weight at 105°C. in a vacuum oven or in an atmosphere of nitrogen. The percentage of neutral oil equals the weight of the extracted oil over the sample weight times 100.

TABLE III

Neutral Oil Content of Cottonseed and Corn Oils

Oil	Wt. of oil in grams	Percentages of neutral oil
Corn	2.0043	89.9
Corn	2.5125	89.8
Corn	3.1238	89.8
Recovered by chromatographic method, 89.8%.		
Cottonseed	2.6279	95.0
Cottonseed	2.6279	94.9
Cottonseed	3.2587	95.0
Recovered by chromatographic method, 95.0%.		

The phospholipid contents of the crude oils were determined by washing the silicic acid residues with methyl alcohol, followed by evaporation of the alcohol and weighing. The results were as follows: soybean oil, 6.25%; corn oil, 5.83%; and cottonseed oil, 3.25%.

The method outlined for neutral oil does not require learning special or complicated techniques or the use of special apparatus. The chloroform, used as a solvent, is nonflammable, and its vapors are only moderately toxic. The results check well with the conventional chromatographic method.

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[Received September 21, 1959]

Factors Affecting Oil Particle Size in the Freezing and Thawing of Fat Emulsions[1,2]

W. S. SINGLETON, RUTH R. BENERITO, and J. L. WHITE, Southern Regional Research Laboratory,[3] New Orleans, Louisiana

EMULSIONS OF VEGETABLE OILS, for example, cottonseed, have considerable potential use as high caloric materials for intravenous alimentation. Such emulsions usually contain 10–15% of oil finely dispersed in an isotonic solution of dextrose, with emulsifying agents present to promote and maintain dispersion. Lipomul-I.V. is such a product, as reported by Meyer *et al.* (3).

One of the critical requirements of these emulsions is that the size of the dispersed oil particles be sufficiently small and remain so on prolonged storage. Meyer and co-workers report the size of the oil particles of Lipomul-I.V. to be predominantly 0.5 to 1 micron in diameter, with not more than 0.02% of the particles larger than 1.5 microns. The size distribution of the oil particles is maintained during storage of the emulsion at 5°C. However if Lipomul-I.V. and emulsions similar to Lipomul-I.V. either inadvertently or deliberately are allowed to freeze, many of the dispersed oil particles after thawing of the emulsion can be observed to have increased in diameter to 7 microns or more. Emulsions containing particles of this size are no longer suitable for use in intravenous alimentation.

In an investigation of the mechanism whereby freezing broke an emulsion of a benzene-carbon tetrachloride mixture in water, Rochow and Mason (4) determined that, as ice crystals form, they are separated from droplets of the internal phase by a membrane. This membrane thins and breaks, and the destruction of the membrane, rather than mere freezing of the continuous phase, is reported to be essential to coalescence of the oil droplets on thawing. Berkman and Egloff (2) explain the breaking of an emulsion by freezing as a withdrawal of free water from the films between droplets which are in contact, and coalescence of the oil droplets with thawing of the ice.

The present investigation was for the purpose of determining whether the increase in the size of some of the oil particles, such as generally occurs when emulsions of the type mentioned are frozen and thawed, could be prevented or minimized. The effects of control of the rates of freezing and thawing of emulsions, type and melting point of oils constituting the oil phase, concentration of the oil phase, and two systems of emulsifying agents were investigated.

[1] Presented at the 33rd Fall Meeting, American Oil Chemists' Society, Los Angeles, Calif., September 28–30, 1959.

[2] This investigation was supported by funds from the Office of the Surgeon General, U. S. Army.

[3] One of the laboratories of the Southern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture.