

A photoelectric colorimeter may be used, provided it is equipped with a proper interference filter.

**Seed and Meal Analysis Committee,  
T. H. Hopper, chairman**

**Urease Activity in Soybean Meal Subcommittee,  
T. J. Potts, chairman**

A new method for "Urease Activity" in soybean meals, soybean flour, and mill feeds is recommended for adoption as "Tentative." If approved, it will be placed in Section Ba under "Oil Seed By-Products." This method, which is that of Caskey and Knapp, under various modifications has been in use for several years. It has been standardized and collaboratively tested by members of the soybean industry and the Association of Cereal Chemists, by whom it will be used. The Uniform Methods Committee approves its adoption with two minor additions as follows. At end of D-1 add this sentence: "Do not invert the tube during the process of mixing." At end of B-1 add this sentence: "The useful life of the buffer solution, prepared as described, is less than 90 days."

**Fat Analysis Committee  
V. C. Mehlenbacher, chairman**

**Subcommittee on Continuous Flow Method of Sampling,  
L. R. Brown, chairman**

At the 1955 Spring Meeting a proposed method of continuous flow sampling was rejected by the U.M.C. as being "too cumbersome." The chief objection was that, for each tank car loading or unloading, from 25 to 40 gallons of surplus oil are accumulated and the method contained no provision for its disposal, preferably by return to storage.

Since that time the method has been used by several large shippers and users of soybean oil, and a convenient means of disposal of the excess oil has been devised. The method has been found to be satisfactory in all respects so far as the drawing of an accurate sample is concerned. Accordingly a revised

method, with the incorporation of certain improvements has been submitted for approval.

The Uniform Methods Committee has studied the proposed revised procedure and has approved of its adoption as a Tentative Method to replace present "Petcock Method," C 1-47, D (a), but with the following minor changes and additions:

- a) In order to promote more rapid and complete drainage and to eliminate, in most cases, the need for the cleaning prescribed in D-5, the bottom of the drum should be replaced by an inverted cone bottom welded securely in place. Samples should be drawn off through the "draw-off line," presently shown in the drawing of the proposed Continuous Flow Sampler, but surplus oil to be returned to storage should be drained off through a connection to the apex of the conical bottom.
- b) The cone bottom should have an apex angle of approximately 120°, the two other angles with the horizontal being about 30° each. These angles should be shown in the revised drawing.
- c) Whenever a different type or kind of oil is to be sampled, the drum should be flushed thoroughly with the oil to be sampled. To accomplish this conveniently a one-inch line with a 45° swing joint should be installed to deliver oil from the loading line to the top center of the drum in such a way that by rotating the swing joint the oil may be used to flush down the sides of the drum into the cone bottom.

These changes should be worked out with the subcommittee on Continuous Flow Method of Sampling and must be reapproved by the subcommittee of the Fat Analysis Committee and by the Uniform Methods Committee.

With the above changes the Uniform Methods Committee approves adoption of this proposed replacement for C 1-47, D (a).

All of the above recommendations by the Uniform Methods Committee were approved by vote of the members present at the business session on October 2, 1957.

J. J. GANUCHEAU	R. J. HOULE
D. L. HENRY	R. R. KING
T. H. HOPPER	T. C. SMITH
J. T. R. ANDREWS, chairman	

## An Improved Liquid-Liquid Extractor<sup>1</sup>

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FOR STUDIES of thermal polymerization of vegetable oils and investigations of the viscosity of the resultant polymer, a method for fractionating heat-bodied oils became essential. The solvent fractionation method of Bernstein (1) was used, but removal of last traces of the high-boiling alcohols from the heavier fractions proved to be troublesome. Because of the marked influence of temperature on the solubility of thermally polymerized oils in acetone, extraction at progressively increased temperatures was tried as an alternative. This type of fractionation is possible in the apparatus to be described.

A number of liquid-liquid extractors having the advantages of thermostatic control (8), internal stirring (2, 3, 6), or all-glass construction (2, 4, 6) have been described. None however embodies all these features. An all-glass extractor was therefore constructed in which these and other advantages are incorporated.

In the present apparatus (Figure 1) stirring is effected by a Teflon-enclosed bar magnet, and channelling of the lighter liquid is further restricted by four baffles (2 cm. long and 1 cm. deep) set at approximately 30° from the vertical. To obviate the problem of alignment the central tube is sealed into the extractor wall. A 2.0-mm. hole at the bottom of the solvent delivery tube permits satisfactory solvent dispersal. The extraction area is surrounded by a water jacket, through which water from a constant-temperature bath is circulated.

The liquid return-line has been kept small and the vapor path large to direct the vapor to the condenser and thus avoid heating the condensate. To permit equalization of pressure in the event of a blocked, liquid return-line, a 2-mm. hole was made in the upper part of the solvent delivery tube near the seal.

The temperatures within the extractor, when acetone was the solvent, were measured one hour after the start of the experiment with the water jacket empty or containing water at various temperatures (Table

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TABLE II  
 Fractionation of Polymerized Linseed Oil with Acetone

Properties	Extraction temperature, °C.						
	10	20	30	40	50	55	55 <sup>a</sup>
$d_4^{25}$ .....	0.9416	0.9464	0.9558	0.9689	0.9739	0.9657	0.9608
$n_D^{25}$ .....	1.48212	1.48412	1.48738	1.49245	1.49377	1.48720	1.49549
$\eta_{30}$ (stokes).....	3.542	5.592	15.06	80.70	177.3	too viscous	too viscous
M.W. <sup>b</sup> = 300.....	14.83%	10.38%	4.84%	2.10%	0.70%	—	0.97%
M.W. = 600.....	6.36	6.83	7.38	10.88	1.33	—	0.24
M.W. = 900.....	42.56	38.04	27.80	18.26	7.13	—	1.90
M.W. > 900.....	36.25	47.75	59.98	68.74	90.83	—	96.89

<sup>a</sup> Insoluble in acetone at 55°C. <sup>b</sup> From molecular distillation (6).

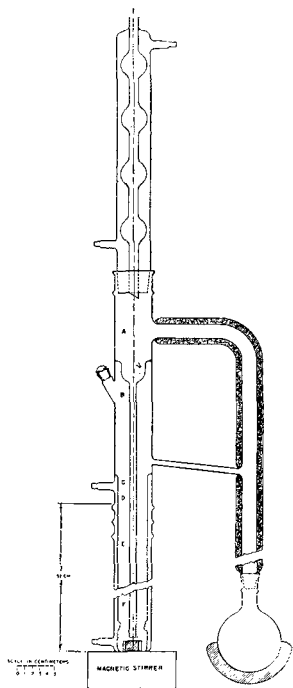


FIG. 1. All-glass, thermostated, liquid-liquid extractor.

1). In the absence of thermostatic control the temperature of the liquid being extracted rose to within 5°C. of the boiling point of the solvent because the solvent vapor heated the area above the surface of the liquid. The limited reduction in temperature when the liquid return-line was cooled (Table I) suggests that hot vapor enters the extractor through the pressure-equalizing hole as well as the liquid return-line. However the extractor has been operated successfully within 1°C. of the boiling point of the solvent, acetone or pentane.

The extractor has been used to separate heated lin-

 TABLE I  
 Variation in Liquid Temperature with Jacket Temperature <sup>a</sup>

Jacket temperature °C.	Temperature, °C., at various thermocouple positions					
	A	B <sup>b</sup>	C	D	E	F
Air at 25°C.	56	55	55	52	30 <sup>c</sup>	30 <sup>c</sup>
Water at 10°C.	56	54	35	24	10	10
Water at 20°C.	56	54	35	22	20	20
Water at 30°C.	56	54	37	33	31	30
Water at 40°C.	56	54	45	42	41	40
Water at 50°C.	56	54	51	50	50	50

<sup>a</sup> Temperature measured one hour after start of experiment.

<sup>b</sup> 2°C. cooler when liquid return-line cooled at 24°C.

<sup>c</sup> Rose to 50°C. over-night.

seed oil into fractions on a temperature basis (Table II). The data show that this equipment permits extraction over a wide range of operating conditions and demonstrate the advantages of extracting at different temperatures in the event of mutual solubility of the solvent and raffinate.

The apparatus has also been used to remove heat-labile material, *e.g.*, ether-soluble substances from liver homogenates, and to extract mono- and dicarboxylic acids from oxidation mixtures (5). In both of these operations low boiling-point solvents are required, and the ability of this apparatus to operate to within 1°C. of the boiling point of the solvent made these extractions possible.

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# ABSTRACTS . . . R. A. REINERS, Editor

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## • Oils and Fats

Absorption of fatty acids from aqueous solutions by active charcoal. Kentaro Ito and Kinnosuke Fukao (Sci. Res. Inst., Tokyo). *Kagaku Kenkyusho Hokoku* **33**, 41-4 (1957). Adsorp-

tion of valeric acid, caproic acid, and heptylic acid by sugar charcoal was studied at various steps of activation. The longer the chain of the fatty acid the lower seems the degree of adsorption by the same charcoal when compared at the concentrations of half saturation. The calculated monolayer adsorption value, and also the heat of adsorption tend to in-