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Abstract. In certain research studies involving the determination of the refining loss of vegetable oils, such as soybean, the 500-gram samples required by the official method may not be available. The chromatographic method, while giving consistent results on a given sample, does not give results which check the official method. A modification of the official cup method was developed using a cup of special design with a 50-gram sample. Good checks with the official method were obtained. It was found that variations in evaporation losses were more important than mixing speeds in affecting results.

By definition the official refining loss method (Mehlenbacher *et al.*, 1959), for vegetable oils "determine the loss of free fatty acids, oil and impurities when the sample is treated with alkali solutions under the specific conditions of the test." This method requires 500 grams of oil for each sample. In certain areas of research, such as small scale extraction studies, the amount of available oil may be too small for its use. Studies were therefore made to develop a method which would require a smaller sample.

The chromatographic method (Linteris and Handschumaker, 1950), was investigated using 2- to 3-gram samples of oil with 25 grams of ethyl ether as a solvent and activated alumina as the adsorbent. Reproducible results were obtained by the use of considerable care. These results were lower than those obtained by the standard cup method. This agrees with Sipos (1958), who has developed an empirical relationship between the two methods. This relationship holds only for soybeans of a given crop year and is not general. A modified chromatographic method in which a chloroform solution of the oil is shaken with silicic acid in a flask and then filtered was also developed in this laboratory (Choudhury and Arnold, 1960). This method checked well with that of Linteris and Handschumaker. However, neither supplied the refined oil needed for other tests. An attempt was next made to modify the official method as it applied to extracted soybean oil to allow the use of a 50-gram sample.

Since the speeds at which the oil and the sodium hydroxide solution are agitated at two different temperatures are specified at 250 ($\pm$ 10) rpm and 70 ($\pm$ 5) rpm, it was assumed that the speeds used are very important to producing a suitable soap which could be readily separated from the oils. Thus, it would be expected that in

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a smaller refining cup it would be necessary to increase the angular speed to secure the proper linear velocity. In our modified method 125-ml stainless steel beakers approximately 2 inches in diameter by 2¼ inches high were used. The weight of the soybean oil sample was 50 grams. The speeds used in agitating are shown in Table 1. Except for the changes shown in the table the official method was followed. The weight of soap produced did not vary significantly with the variation in speed. However, the evaporation loss was definitely higher, resulting in a high refining loss. Attempts to reduce the evaporation loss by reducing the time in the hot bath did not give satisfactory results.

Table 1

Refining Loss By the Official Method and a Modified Method

	Official Method	Modified Method			
Speed in cold bath, rpm	250	200	250	450	1000
Speed in hot bath, rpm	75	50	75	110	250
Weight of oil, gm.	500	50	50	50	50
Weight of 14°Be, lye, gm.	28.5	2.82	2.83	2.90	3.00
Evaporation loss, gm.	1.2	0.60	0.31	0.67	1.81*
Weight of soap, gm.	43.8	4.37	4.59	4.34	4.42
Loss in weight, gm.	16.5	2.15	2.07	2.11	3.23*
Refining loss, percent	3.3	4.30	4.14	4.22	6.46*

*Some splashing from high speed

Table 2

Refining Loss of Soybean Oil By the Two Methods

	Official Method	Modified Method
Weight of oil in grams	500	49.9970
Weight of 14° lye, gm.	28.5	2.7525
Evaporation loss gm.	1.2	0.1480
Weight of soap, gm.	43.8	4.2185
Loss in weight. gm.	16.5	1.6140
Refining loss, percent	3.3	3.25

It was next assumed that the high evaporation loss resulted from a relatively high ratio of exposed area of oil surface to volume in the beaker as compared with that in the standard refining loss cup. A special cup was made from a stainless steel tube 1¼ inches in diameter and 4½ inches high. A stirrer was made by soldering a thin plate of stainless steel ¾ inches by 9/16 inches to a small shaft attached to a laboratory stirrer motor. As originally constructed, the cup weighed about 365 grams making it impossible to weigh it on a conventional analytical balance. By thinning down the cup walls on a lathe, the weight was reduced to about 140 grams. This allowed the cup with a 50-gram sample to be weighed on an analytical balance. The refining loss secured using the modified cup and stirrer with a 50-gram sample of soybean oil under conditions specified in the official method averaged 3.25 percent. The refining loss by the

official method on the same oil was 3.30 percent (Table 2). This is considered a satisfactory check.

Apparently the conditions of agitation for this test are not as critical as assumed. Mixing needs to be good but excessive speeds cause spattering, resulting in losses of oil or soap. The purely arbitrary evaporation losses needed to check results by the official method can be obtained by the use of the new cup. By carrying out the weighings on an analytical balance, it is possible to secure results on 50-gram samples comparable to those secured by the official method on 500-gram samples.

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