prior to flaking, and cooking by the modified hydraulic method developed for use with the filtration-extraction process. Commercial hexane was used as the extraction solvent. The experiments were carried out by procedures which eliminated the effects of any variables other than the method of preparing the meats for extraction.

The results of the studies showed that the method used in preparing cottonseed meats for extraction had a significant effect on the yields of crude oil obtained but that the yields of neutral oil, the valuable constituent of crude oils, were virtually unaffected. Analyses of the crude oils showed that the differences in crude oil yields were caused by the relative amounts of non-neutral oil materials in the crudes from the differently prepared meats. The greatest yields of crude oil were obtained from raw flakes, intermediate yields from tempered flakes, and the smallest yields from cooked flakes. The impurities content in the respective crude oils followed the same order, *i. e.*, crudes from raw flakes were highest in impurities and lowest in neutral oil, crudes from tempered flakes were lower in impurities and higher in neutral oil, and the crudes from the cooked meats were outstandingly low in impurities and high in neutral oil. Virtually equal amounts of neutral oil were obtained from equivalent quantities of comparable meats regardless of the method used in preparing the meats for extraction.

Acknowledgment

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Procedure and Apparatus for Plasticizing Fats in the Laboratory[1]

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THE POUND-CAKE VOLUME BAKING TEST is an important initial guide for evaluation of a new shortening. Variations of this test also are valuable production control tools in the manufacture of shortenings.

The eight ounces of shortening used in this bake test must be plasticized and tempered unless some other consistent means of obtaining a satisfactory physical state is known. A fast, dependable procedure for plasticizing such a small amount of shortening has been a problem in the research laboratory.

In the plant the shortening is chilled rapidly with sufficient agitation to accumulate 10–15 cc. of air per 100 g. of fat. The air content is controlled, principally to improve the appearance of the shortening. The plasticized shortening is then tempered at least 48 hrs. at 80–90°F. (room temperature).

We have found a procedure whereby shortenings can be so plasticized in the laboratory as to give pound-cake volume values closely approximating those given by the same fats plasticized in plant equipment. The products of our process contain more air than those produced in plant equipment, and consequently they are not comparable in texture or appearance. Our process must be considered strictly as a method of preparing the shortening for the pound-cake volume test.

Apparatus

The process is carried out in a Hobart Kitchen Aid Mixer (Model K-4-B), which is of the planetary type, and the bowl is cooled in a water bath in which the entire assembly is set as shown in Figure 1.

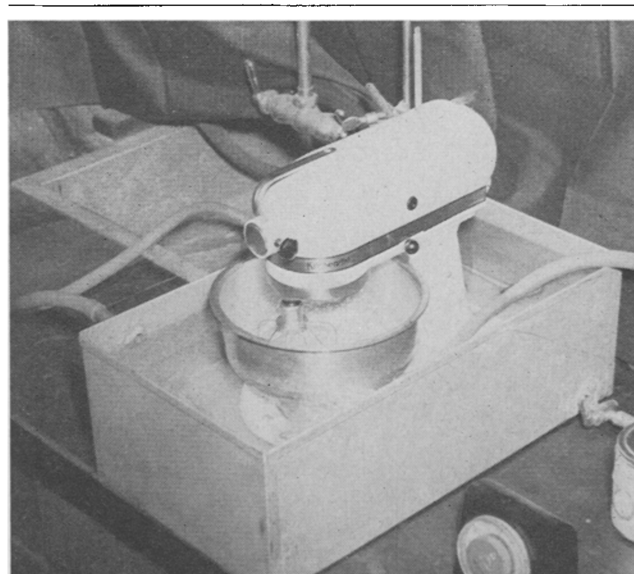

FIG. 1. Apparatus used for plasticizing fats.

[1] Presented at the fall meeting, American Oil Chemists' Society, Philadelphia, Pa., Oct. 10–12, 1955.

The stainless steel water bath is equipped with a) an overflow to maintain a predetermined constant water level, approximately 2½ in. below the top of the mixing bowl; b) a perforated air hose to aid in water circulation (a stirrer could be used); c) a thermometer; and d) a water outlet.

Ice or dry ice can be easily added to the water bath for temperature control. A combination of the two works well. The bubbling effect of a small amount of dry ice aids the water circulation.

As shown in the photograph, the mixer is placed in the water bath. (Caution: None of the electrical equipment should be in direct contact with water at any time. Both mixer and bath must be grounded for safety.)

The mixer is equipped with the following features: a) a metal bowl that is stationary during mixing but can be easily unscrewed from the base; b) a wire beater of the planetary type;[2] c) ten controlled speeds; d) a lock to hold the beater in position; and e) a base made of non-rusting material.

Experimental

Samples of a plant-plasticized flaked lard and of a hydrogenated vegetable oil shortening were melted and plasticized in the Kitchen Aid Mixer. By varying the water bath temperature, the speed of the beater, and the time of mixing, enough data were accumulated to establish the following detailed method.

One pound (or less) of sample is heated to 60°C. in a 600-ml. beaker. The sample is then cooled with stirring to 40°C. and transferred to the Kitchen Aid Mixer, where it is plasticized under the following conditions: temperature of water bath, 10°C.; speed setting, 4; and time of mixing, 10 min.

After this procedure the mixing bowl is removed from the water bath, and the plastic portion of the sample is spooned into a glass jar. Some of the shortening will have congealed on the upper surface of the bowl because of splashing in the first stages of the mixing. The plastic portion of the fat and the congealed portion do not differ in iodine value or Wiley melting point, indicating that the portions are identical in composition.

The temperature of the shortening as it comes out of the mixing bowl is 66–70°F., and the air content varies from 30 to 50 cc. per 100 g. of shortening.

The plasticized sample is tempered 24 hrs. at 85–90°F. before being submitted to the pound-cake volume baking test.

The standard pound cake test procedure used in this laboratory involves the use of the shortening under test in baking a cake of the following formula:

1 lb. fine granulated sugar	8 oz. whole eggs
8 oz. shortening	8 oz. milk
½ oz. salt	1 lb. fine cake flour

The conditions of mixing and baking are in each instance identical. The volume of the cake in milliliters divided by its weight in grams, multiplied by 100, gives a figure which is representative of the cake-volume-producing characteristics of the shortening being tested. Maximum experimental error in this baking test should not exceed an 8–10 point difference between duplicate samples or a ±5 point deviation from the average. The data upon which the process described was based follow.

TABLE I
Comparison of Plant Laboratory Samples of Flaked Lard and of Hydrogenated Vegetable Oil Shortening

	Bath temp. (°C.)	Speed setting	Time (min.)	Pound-cake volume value (ml./100 g.)	
				Lard	Hyd. veg. oil short'ng
Series I					
Plant plasticized	....	....	....	146	239
Laboratory plasticized	10	4	5	148	245
Laboratory plasticized	10	4	10	151	243
Laboratory plasticized	10	4	15	153	240
Series 2					
Plant plasticized	....	....	....	151	234
Laboratory plasticized	15	4	5	181	250
Laboratory plasticized	15	4	10	170	247
Laboratory plasticized	15	4	15	170	251
Series 3					
Plant plasticized	....	....	....	153	235
Laboratory plasticized	20	4	5	170	247
Laboratory plasticized	20	4	10	177	249
Laboratory plasticized	20	4	15	170	254
Plant plasticized sample (series 1, above)	....	....	....	146	239
Plant plasticized sample (series 2, above)	....	....	....	151	234
Plant plasticized sample (series 3, above)	....	....	....	153	235
Plant plasticized sample	....	....	....	....	239
Plant plasticized sample	....	....	....	....	240
Plant plasticized sample	....	....	....	....	238
Plant plasticized sample	....	....	....	....	239
Plant plasticized sample	....	....	....	....	240
Plant plasticized sample	....	....	....	....	244
Plant plasticized sample	Average..............................			150	239

Table I shows the results of three series of experiments, using a plant-processed flaked lard and an all-purpose hydrogenated vegetable oil shortening of intermediate quality. They show the effects of variation in bath temperature and time of mixing at speed setting 4. The speed of mixing was not varied because in the preliminary tests it was established that rapid mixing is desirable, and speed setting 4 gives the most rapid mixing that can be attained without excessive splashing of fat in the early stages.

Samples of the plant-processed flaked lard and shortening were melted and replasticized in the mixer at 10, 15, and 20°C. in 5, 10, and 15 min. at each temperature, and the remainder was used as received, as a standard.

Each group of samples was prepared in a single day, and the pound-cake volume values of each group, including that of the plant-plasticized sample, were determined in a single day.

The average pound-cake volume value for the plant-plasticized sample of lard was 150, and the spread between the individual determinations was well within the experimental error of the baking test.

The plant-plasticized sample of hydrogenated vegetable oil shortening, which was submitted to the baking test on nine different days, produced an average value of 239. All nine results were again within the experimental error of the baking test.

It is obvious from the results for series 1 that a bath temperature of 10°C. produces results most similar to those produced by the plant procedures and that mixing time may vary between 5 and 15 min.

From the data in Table I 10°C. was established as the desired water bath temperature, and a mixing time of 10 min. was selected. In 10 min. the softer shortenings have enough time to become plastic, and

[2] At speed setting 4, at which the process is operated, the revolutions per minute of the beaters of two machines about their own axes were 467 and 477, and their revolutions per minute about the central axes were 140 and 143, respectively. There was no significant difference between the results produced by the two mixers.

the harder shortenings do not become too hard.

Table II shows the results of experiments on various types of commercially manufactured shortenings, including four samples of hydrogenated vegetable oil shortening covering a range of quality.

A portion of each sample was melted and replasticized by the laboratory method, and the remainder was used as received, as a standard.

TABLE II
Comparison of Plant and Laboratory Samples of Various Types of Shortenings

Shortening	Pound-cake volume values	
	Plant-plasticized	Laboratory-plasticized
	ml./100 g.	*ml./100 g.*
1. Hydrogenated vegetable oil shortening, No. 1	234	244
2. Hydrogenated vegetable oil shortening, No. 2	257	266
3. Hydrogenated vegetable oil shortening, No. 3	271	268
4. Hydrogenated vegetable oil shortening, No. 4	239	243
5. Modified lard-hydrogenated vegetable oil shortening	258	259
6. Emulsifier type, modified lard-hydrogenated vegetable oil shortening	259	261
7. Emulsifier type, hydrogenated vegetable oil shortening	230	234
8. Experimental modified lard blend	269	261
9. Hydrogenated vegetable oil for deep fat frying	277	243
10. Flaked lard	146	151

The results obtained on the plant-plasticized and laboratory-plasticized samples are in good agreement. A statistical analysis of the data shows, with one exception, no significant difference in the pound-cake volume values.

Only in item 9 is a significant discrepancy evident. This was a sample of a deep frying fat, which is not customarily used for baking because of its short plastic range.

If the minimum pound-cake volume value specified for a high quality hydrogenated vegetable oil shortening is 250, it is obvious from the results that samples 1 and 4 plasticized by either method are readily classified as substandard whereas samples 2 and 3 are shown to be above standard in quality.

Table III shows the variation between pound-cake volume values of duplicate laboratory-plasticized samples of hydrogenated vegetable oil shortenings.

Portions of each commercially prepared hydrogenated vegetable oil shortening were melted and replasticized by the laboratory method, and the remainder was used as received, as a standard.

TABLE III
Variation Between Pound-Cake Volume Values of Duplicate Samples of Hydrogenated Vegetable Oil Shortening

	Pound-cake volume values
Sample 1	*ml./100 g.*
Plant-plasticized	240
Laboratory-plasticized	243, 246
Sample 2	
Plant-plasticized	257
Laboratory-plasticized	265, 267
Sample 3	
Plant-plasticized	270
Laboratory-plasticized	264, 272
Sample 4	
Plant-plasticized	238
Laboratory-plasticized	234, 233

Results obtained on the duplicate laboratory-plasticized samples show no significant difference in pound-cake volume values. These results, compared with those for the plant-plasticized sample, are in good agreement.

In addition to the data in Table III many other analyses confirm the conclusions that the laboratory method gives reproducible results comparable to those obtained with plant-plasticized samples.

Summary

A procedure for plasticizing fats in the laboratory, using a Hobart Kitchen Aid Mixer (Model K-4-B), has been described. The data indicate that this procedure is applicable to the common types of baking shortening for determining their pound-cake volume value and gives results in agreement with those obtained on plant-plasticized samples. The apparatus is relatively inexpensive and easy to assemble and operate. The procedure is fast and simple and has proven to be a helpful research tool in evaluating experimental shortenings.

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Recent Advances in in-Situ Epoxidation Reactions with Resin Catalysts[1]

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IN THE LAST SEVERAL YEARS the preparation of epoxy or oxirane compounds by the epoxidation of unsaturated fatty acids or esters with hydrogen peroxide or peracetic acid has assumed the role of a unit operation. Although perbenzoic acid was employed in the original synthesis of epoxides in 1909 (10), this reaction has been carried out with other peracids. The most prominent among these has been peracetic acid (3, 4, 6, 12).

Recently *in-situ* epoxidation procedures have been developed in which hydrogen peroxide is used as the oxidant. This eliminates the necessity of using a preformed peracid. Peracid formation and reaction with the double bond occur simultaneously with approximately stoichiometric amounts of hydrogen peroxide.

The first published *in-situ* process was described in the patent literature (9) and employed formic acid and hydrogen peroxide.

A procedure, developed in our laboratories, for the *in-situ* epoxidation of unsaturated fatty acid esters, employing acetic acid and hydrogen peroxide, has been described (4). In this process hydrogen peroxide is added to an acetic acid solution of the unsaturated ester in the presence of an acid catalyst

[1] Presented at the 29th fall meeting of the American Oil Chemists' Society, October 10–12, 1955, Philadelphia, Pa.