The Determination of Conjugated *cis-trans* and *trans-trans* Methyl Octadecadienoates by Infrared Spectrometry[1]

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CONJUGATED DIENES usually are determined by measurements of light absorption in the ultraviolet region near 233 mμ. The conjugated diene system however may exist in a *cis-cis, cis-trans,* or *trans-trans* configuration, and each of the three geometrical isomers differs from the others in the exact location and intensity of its absorption maximum. In unsymmetrical compounds a fourth isomer with a *trans-cis* configuration is possible. However, when the conjugated system is removed from other functional groups, such as in conjugated linoleic acids, the spectral characteristics of this compound should be identical with those of the *cis-trans* material.

Most studies with conjugated octadecadienoates have been confined to the 9,11- and 10,12-compounds obtained from linoleic or ricinoleic acid. Conjugated *trans-trans* octadecadienoic acids have an absorption maximum at 231 mμ (1), and absorptivities from 113 to 120 have been reported for these compounds (2, 3, 4, 5, 6, 7). The *cis-trans* isomers absorb at 233 mμ, and two sets of values can be found in the literature for their absorptivities, depending on the location of the double bonds (1, 2, 8). Recently methyl 10-*cis*,12-*cis* octadecadienoate was synthesized by Allen (9) and was found to have an absorption maximum of 235 mμ, with an absorptivity of 95.0.

The direct determination of diene conjugation by ultraviolet spectroscopy is only approximate therefore, both because of the difference in position of the absorption peaks of the possible isomers and of the uncertainties of the true absorptivities for the pure compounds. However, while this investigation was in progress, Tolberg and Wheeler (10) reported that total diene conjugation could be measured by ultraviolet or infrared spectrometry, using equilibrium mixtures obtained by isomerizing the conjugated compounds with iodine.

In the infrared region, conjugated *cis-trans* octadecadienoates absorb sharply at 10.18 and 10.54 μ while the *trans-trans* isomers show a single stronger peak at 10.12 μ (8). Tolberg and Wheeler (10) have used these absorption characteristics to measure these compounds but gave no detail of their procedure. The present report describes in detail such a method used to determine, separately, conjugated *cis-trans* and *trans-trans* methyl octadecadienoates in mixtures with other fatty esters. The conjugated *cis-cis* octadecadienoate of Allen (9) has no specific absorption between 2 and 15 μ intense enough for its independent determination, and this isomer must be estimated by difference.

The esters of saturated or of *cis* unsaturated fatty acids have only a minor general absorption between 10 and 11 μ so that, in the absence of other *trans* unsaturation, the peaks characteristic of *cis-trans* and *trans-trans* diene conjugation occur in a region reasonably free of extraneous absorption. The fatty acids however have a strong and broad absorption band near 10.7 μ, and in these compounds the conjugated *cis-trans* and *trans-trans* peaks are superimposed on the steep slope of the acid absorption. For this reason the quantitative determination of *cis-trans* and *trans-trans* conjugation by infrared spectrometry is preferably done with esters rather than with acids.

Since the conjugated *trans-trans* compounds have the highest absorptivity in the ultraviolet region, their isolation and purification can be followed conveniently by measuring the light absorption of the fractions at 233 mμ. With the conjugated *cis-trans* isomers however high absorptivities near 233 mμ would indicate the presence of *trans-trans* compounds while low values would result from contamination with unconjugated or with conjugated *cis-cis* materials. Ultraviolet spectroscopy therefore is not suitable for measuring the purity of *cis-trans* conjugated dienoates, but infrared absorption between 10 and 11 μ can be used for this purpose. Contamination with unconjugated or with conjugated *cis-cis* material would result in a proportionately equal decrease in the intensities of both peaks at 10.18 and 10.54 μ without changing the ratio of the absorptions at these two wavelengths. On the other hand, the presence of *trans-trans* isomers would result in decreased absorption at 10.54 μ and in an increase in absorption near 10.1 μ because of the additive effect of the *cis-trans* and the more intensely absorbing *trans-trans* compounds. In this case the ratio of the intensity at 10.1 μ to that at 10.54 μ would be high and would increase very rapidly with increasing *trans-trans* content. Consequently high purity of *cis-trans* fractions should be indicated by a high absorptivity at 10.54 μ and a low value for the ratio $A_{10.1}/A_{10.54}$.

Experimental Procedures

Preparation of Methyl 10-trans,12-trans Octadecadienoate. Castor oil was dehydrated in the laboratory at 220°C., with 2% sodium bisulphate as a catalyst, and the methyl esters were prepared from the dehydrated product by methanolysis. The unconjugated fraction was removed by fractional distillation in a Podbielniak hypercal column and recrystallized several times from acetone at −70°C., essentially as described by Jackson *et al.* (8), to concentrate the 9-*cis*,12-*trans* material. The separation was followed by infrared spectrometry, and the fractions with high isolated *trans* absorption near 10.35 μ were combined and alkali-isomerized as follows. A solution of 15% sodium hydroxide in ethylene glycol (165 g.) was heated to 195°C. under nitrogen for 10 min., and 50 g. of the ester were added slowly to the hot solution, with stirring. Heating and stirring were continued for 30 min., after which the soaps were cooled and dissolved in water. The conjugated fatty acids were recovered by acidification and extraction with petroleum ether and were recrystallized repeatedly from Skellysolve B at −20°C. The filtrates from each crys-

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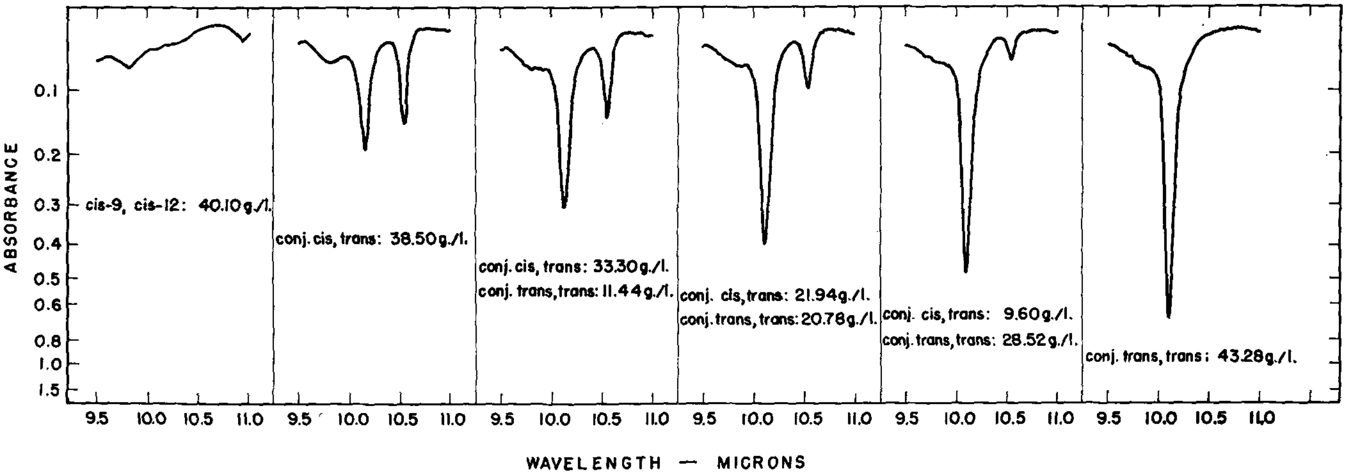

FIG. 1. Infrared spectra, between 9.5 and 11.0 μ, of methyl linoleate, of pure conjugated *cis-trans* and *trans-trans* octadecadienoates, and of mixtures of conjugated *cis-trans* and *trans-trans* octadecadienoates.

tallization were concentrated and recrystallized from Skellysolve B, and progress of the separation was followed by measuring the spectral absorption of the fractions at 231 mμ. Several nearly pure precipitates ($a_{231\ m\mu} = 115$ to 117) were combined and esterified with diazomethane. The methyl esters were recrystallized several times from Skellysolve B at −20° C., finally yielding a precipitate of essentially pure 10-*trans*,12-*trans* octadecadienoate: $a_{231\ m\mu} = 112.7$ (calculated for the acid $a_{231\ m\mu} = 118.3$), melting point 23–24.9°C.

Preparation of cis-trans Octadecadienoates. The crude fatty acids (1,000 g.) from safflower seed oil (iodine value 147.3), containing approximately 75% linoleic acid, were added, with stirring, to a hot solution of 1,600 g. of urea in 4,000 ml. of methanol. Stirring of the precipitate which formed immediately was continued for about 10 min., and the mixture was allowed to stand under nitrogen over-night, at room temperature, and filtered. The filtrate was diluted with 2 volumes of cold 0.5 N aqueous HCl and the liberated acids were extracted with petroleum ether. After washing, drying, and removing the solvent, the fatty acids were distilled rapidly through a short Vigreux column. The main fraction had an iodine value of 178.7 (theory for linoleic acid, 181.0) and contained 97.3% linoleic acid.

The linoleic acid concentrate was isomerized with alkali, as described for the dehydrated castor oil acids, and the conjugated acids ($a_{233\ m\mu} = 85.1$) were esterified with methanol. The methyl esters were recrystallized from methanol according to the scheme of Nichols *et al.* (1). A fraction melting at −9.4 to −10.5°C., with an absorptivity at 233 mμ of 88.2, was obtained in 8% yield. The fraction was saponified, and the fatty acids were recrystallized once from petroleum ether and twice from acetone at −50°C., yielding a final acid fraction corresponding to Nichols' fraction 18a: melting point 23.2–23.9°C., and $a_{233\ m\mu} = 94.6$. These constants agree with values reported for pure 10-*trans*,12-*cis* octadecadienoic acid (1, 2), but the absorptivity of the methyl ester at 10.54 μ was lower, and the ratio of the absorbance at 10.18 to that at 10.54 μ was higher than corresponding values obtained previously in this laboratory on a sample of nearly pure methyl 9-*cis*,11-*trans* octadecadienoate (11). Although these observations may indicate that the 10,12- and 9,11-*cis-trans* compounds have different absorptivities in both the ultraviolet and infrared regions, the differences are also what would be expected if the 10,12-*cis-trans* material contained small amounts of *trans-trans* isomers. It is felt that the latter alternative is the more likely possibility and that the relatively high-melting 10-*trans*,12-*cis* octadecadienoate is difficult to obtain completely free of *trans-trans* isomers by the crystallization methods employed here and by earlier workers.

Very little material corresponding to fraction 20 of Nichols *et al.* was obtained. However the infrared spectrum of a relatively large fraction corresponding to their fraction 19 indicated that it contained a high proportion of conjugated *cis-trans* octadecadienoate and no or very little *trans-trans* isomer. After three additional crystallizations from methanol (20 ml. per g.) at −45°C., the spectral characteristics of this fraction remained unchanged, indicating that it was essentially pure conjugated *cis-trans* material, presumably the 9-*cis*,11-*trans* octadecadienoate: melting point −36 to −30°C.; $a_{233\ m\mu} = 90.37$; $a_{10.16\ \mu} = .345$; $a_{10.55\ \mu} = .288$; $a_{10.16}/a_{10.55\ \mu} = 1.198$. Although the melting range of this fraction was appreciable, the absorptivities at 10.16 and 10.55 μ indicated that its content of conjugated *cis-trans* material was higher than that of any other fraction obtained during this study or reported in the literature, and it was used without further purification.

The absorption coefficients of Tolberg and Wheeler (10) are higher than the values reported here, but they were calculated by using a different base line from absorbances obtained with uncalibrated cells. Using their base line (equal to absorbance at 11 μ), the absorptivity of our *trans-trans* compound (1.411) agrees well with that of Tolberg and Wheeler (1.46), but, as indicated by these authors (10), our *cis-trans* material ($a_{10.56} = 0.316$) appears purer than theirs ($a = 0.295$).

Spectrophotometric Measurements. A Perkin-Elmer Model 21 infrared spectrophotometer was used, and no special effort was made to obtain maximum resolution. The controls were set as recommended by the manufacturer for routine quantitative measurements, and the mechanical slit width, as indicated by the counter, varied from 118 μ at 10.10 μ to 125 μ at 10.55 μ. The same pair of 0.1-mm. cells was used all through the study, and their exact thicknesses, measured by the interference fringe method (12), were 0.0985 mm. for the reference cell and 0.1066 mm. for the sample cell. Carbon disulfide solutions were used, and the concentrations of the materials were adjusted so that the absorbance of the samples did not exceed 0.7. Pure carbon disulfide was used in the reference cell. The absorbance of carbon disulfide is negligible in this region, and no correction was applied for the difference in thickness of the two cells.

Fats containing conjugated dienes usually contain appreciable quantities of unconjugated materials also, and in routine measurements an approximately constant amount of total fat is used for each determination. Therefore it was thought advisable to determine the effect of unconjugated constituents on the absorptivities and on the determination of the conjugated dienoates.

Solutions containing various concentrations of the pure *cis-trans* or *trans-trans* isomers in pure carbon disulfide, or with natural 9-*cis*,12-*cis* linoleate added to keep the total fat concentration approximately constant, were used to obtain the absorptivities of the pure compounds. Mixtures containing different proportions of the two isomers at several concentrations with and without added linoleate were prepared also. All spectra were recorded between 9.5 and 11.0 μ at a chart speed requiring 2 min. per micron.

Results and Discussion

Figure 1 shows the spectra of natural 9-*cis*, 12-*cis* linoleate, of the pure *cis-trans* and *trans-trans* conjugated compounds, and of representative mixtures of the conjugated isomers all at about the same total concentration in carbon disulfide (approximately 40 g. per liter).

With our instrument the *cis-trans* isomer absorbed at 10.55 and 10.16 μ and the single peak of the conjugated *trans-trans* compound was recorded at 10.11 μ. In mixtures of these two compounds the 10.11 and 10.16 μ bands are not resolved but occur as a single peak at an intermediate wavelength.

In all cases the 10.55 μ *cis-trans* peak is well separated from the other bands and receives no contribution from them. Consequently the content of conjugated *cis-trans* material can be determined easily from the absorption at 10.55 μ.

To determine the *trans-trans* isomers however the total absorption at 10.11 μ must be corrected for the contribution of the *cis-trans* material. Since, in some mixtures, the absorption maximum of the *trans-trans* compound is on the steep slope of the merged peaks, it is very important that intensity measurements in this region be made at exactly 10.11 μ. In these determinations it was assumed that the long wavelength *cis-trans* peak always occurred at 10.55 μ, and it was used as a calibration point to determine, within 0.005 μ, the exact location of the other peak. In this manner, errors caused by small variations in the reproducibility of the instrument and failure to place the pen exactly on the chart were minimized.

Since unconjugated linoleate shows a small but significant increasing absorption between 9.9 and 10.7 μ, a sloping base line drawn across the base of the absorption peaks of the conjugated compounds should best approximate the background values to be used for calculating the absorbance of the conjugated isomers. In some mixtures a single straight line drawn across both peaks is not tangent to the low absorption point between the two peaks. In these cases a second value may be obtained by drawing a base line across each individual peak. Base line values obtained by both methods were used to calculate the absorptivities of the pure *cis-trans* and *trans-trans* materials and for estimating the composition of mixtures. Most consistent values were obtained when the base line was drawn across each individual peak, and the results reported here were obtained by this method.

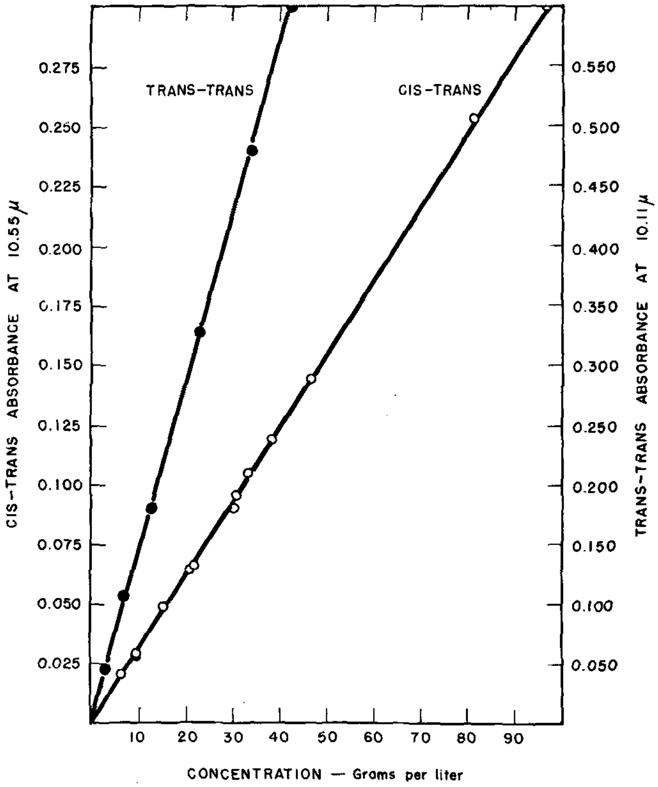

FIG. 2. Absorbance of conjugated *cis-trans* and *trans-trans* octadecadienoates.

Determination of Conjugated cis-trans Octadecadienoate. A plot of absorbance at 10.55 μ against *cis-trans* concentration for 13 samples containing the *cis-trans* compound, either alone or in mixtures, yielded a straight line, shown in Figure 2, and the absorptivity of the *cis-trans* material at 10.55 μ was 0.288.

The amount of *cis-trans* material present in solutions of unknown samples can be calculated readily by the following equation:

$$X = A_{10.55}/0.288\ b \quad (1)$$

where X is the content, in grams per liter, of *cis-trans* octadecadienoate, in the solution examined, $A_{10.55}$ is the observed absorbance (peak absorbance − base line absorbance) at 10.55 μ, and b is the thickness of the sample cell in cm. (in this case 0.01066).

Determination of Conjugated trans-trans Octadecadienoate. A plot of absorbance at 10.11 μ *vs. trans-trans* concentration for six samples containing conjugated *trans-trans* material but no conjugated *cis-trans* is shown in Figure 2 also. Again a straight line relationship going through the origin was ob-

TABLE II

Analysis of Mixtures of Conjugated *cis-trans* and *trans-trans* Octadecadienoates

Sample	Known composition			Spectral measurements					
	Linoleate	Conjugated *cis-trans*	Conjugated *trans-trans*	10.55 μ peak absorptivity	10.1 μ peak		Calculated composition		
					Location	Absorptivity	Conjugated *cis-trans*	Conjugated *trans-trans*	
								From absorbance at 10.11 μ	From absorbance at peak
	%	%	%	*l/g cm.*	μ	*l/g cm.*	%	%	%
9	0	74.49	25.51	0.218	10.125	0.515	75.58	27.99	25.27
10	0	51.36	48.64	0.143	10.120	0.753	49.56	49.02	48.27
11	0	25.18	74.82	0.074	10.115	1.053	25.63	75.47	75.13
12	20.65	60.80	18.55	0.178	10.125	0.390	61.85	18.97	18.24
13	22.40	38.87	38.73	0.109	10.120	0.598	37.80	39.50	38.63
14	19.97	19.74	60.29	0.052	10.110	0.838	18.19	60.31	60.33
				Standard deviation[a]			1.32	0.87	0.25

[a] Calculated from the difference between known composition and calculated composition.

tained, and the absorptivity of the *trans-trans* compound at its maximum was 1.330.

When *cis-trans* material is absent, as indicated by the absence of a peak at 10.55 μ, the *trans-trans* content can be determined from the absorbance at 10.11 μ by an equation identical to equation (1) but using 1.330 for the absorptivity of the pure *trans-trans* compound instead of the 0.288 value for the *cis-trans* isomer.

In mixtures of the two isomers however it is necessary to correct for the contribution of the *cis-trans* material to the *trans-trans* absorption. With samples containing no *trans-trans* compounds, a plot of absorbance at 10.11 μ against concentration of *cis-trans* conjugation gave a straight line also, from which the absorptivity of conjugated *cis-trans* diene at 10.11 μ was found to be 0.187.

The conjugated *trans-trans* content of unknown samples containing both *cis-trans* and *trans-trans* compounds is calculated as follows:

$$Y = \frac{(A_{10.11}/b) - 0.187\,X}{1.330} \qquad (2)$$

where Y is the content, in grams per liter, of *trans-trans* octadecadienoate and $A_{10.11}$ is observed absorbance (peak absorbance − base line absorbance) at 10.11 μ.

For accurate analysis of many mixtures where the 10.11 μ *trans-trans* maximum is located on the steep side of the combined bands, extreme care must be used in reading both wavelength and absorbance on the chart. For routine determinations the use of absorbance at the maximum of the combination peak is more desirable because it is less subject to error although the calculations are slightly more complicated. Theoretically, when peak absorbance is used, the absorptivities of the *cis-trans* and *trans-trans* components to be employed in the calculations should vary with the location of the peak. In practice however it was found that the use of *trans-trans* absorptivity values varying with peak location gave grossly erroneous results. On the other hand, values in excellent agreement with known composition were obtained when *trans-trans* absorptivity at 10.11 μ was used in the calculations, regardless of the position of the peak. There is no theoretical justification for using absorptivity at 10.11 μ to calculate *trans-trans* content from absorbance at some other wavelength, and consequently this method must be considered empirical. In this case the *trans-trans* content is calculated as follows:

$$Y = \frac{(A_{peak}/b) - a_x X}{1.330} \qquad (3)$$

where A_{peak} is the absorbance of the combination peak (peak absorbance − base line absorbance) and a_x the absorptivity of the *cis-trans* material at the exact wavelength of the combination peak. This value is taken from Table I.

TABLE I

Absorptivity of Conjugated Methyl *cis-trans* Octadecadienoate Between 10.11 and 10.16 μ

Wavelength (μ)	Absorptivity
10.11	0.188
10.12	0.219
10.13	0.251
10.14	0.282
10.15	0.314
10.16	0.345

Table II shows the results obtained when these equations were applied to model mixtures of conjugated *cis-trans* and *trans-trans* octadecadienoates with and without natural *cis-cis* linoleate. The average error for the determination of the *cis-trans* compounds is greater than for the *trans-trans* measurement although the latter requires a correction depending on the calculated *cis-trans* content. No explanation for this unexpected result is offered.

It was thought that the absorptivity-wavelength curves of the *cis-trans* and *trans-trans* compounds near 10.1 μ might cross at a wavelength at which the total concentration of these two isomers could be measured. However the absorptivity of the *trans-trans* material is so much greater than that of the *cis-trans* compounds and the two peaks are so close together that the curve for the *cis-trans* compound lies completely above that of the *trans-trans* isomer, and no isosbestic point exists.

In complex fat mixtures other types of *trans* unsaturation may be present. Compounds containing isolated *trans* double bonds absorb strongly near 10.33 μ, and a correction for the contribution of this structure to the *cis-trans* and *trans-trans* absorption peaks probably would be required. Conjugated trienes with *trans* double bonds absorb very strongly near 10.08 μ and, in addition, may have weaker bands near 10.4 or 10.5 μ (13). The presence of these substances would seriously interfere with the determination of conjugated *cis-trans* and *trans-trans* diene described here.

Obviously this method cannot replace the determination of total conjugated diene by infrared or ultraviolet spectroscopy, but it can supplement it, and it is the only method presently available to measure separately conjugated *cis-trans* and *trans-trans* compounds. When used in conjunction with a total diene determination, an estimate of conjugated *cis-cis* unsaturation may be obtained by difference.

The method has been used to follow the conversion of conjugated *cis-trans* octadecadienoate, obtained by alkali isomerization of linoleic acid, to the *trans-trans* isomer under the influence of several catalysts and under different conditions.

Summary

Pure conjugated methyl *cis-trans* and *trans-trans* octadecadienoates have been prepared and used to develop a method for the determination of these compounds by infrared spectrometry. *Cis-trans* compounds alone or in the presence of *trans-trans* conjugation may be determined directly by the absorption at 10.55 μ. In the absence of *cis-trans* isomers conjugated *trans-trans* octadienoates may be determined directly from the absorption at 10.11 μ. When both types of compounds are present however, a correction must be applied for the contribution of the *cis-trans* isomers to the 10.11 μ absorption of the *trans-trans* material.

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The Design, Construction, and Operation of a Pilot-Plant-Size Plasticizer for Shortening and Margarine

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IN 1940 THE SENIOR AUTHOR of this paper designed and constructed a small plasticizer for plasticizing 12-ounce samples of shortening. This unit was in use in our pilot plant for a number of years in our experimental work. In 1957 it was decided that a larger unit would have greater utility, serving both for our experimental work and the preparation of actual samples for sales distribution. It was agreed that a three-pound size would have the greatest utility. Further, some refinements in design and construction seemed in order. Accordingly a unit was designed and built. Figure 1 shows a picture of the unit in its entirety except for the bath of cooling water. Figure 2 shows the cross-section of the chilling unit itself. Essentially it is a jacketed cylinder cooled with circulated ice water, in which a piston actuated by a steam pump moves at a rate of 12 strokes per minute. The product being plasticized is forced through a small adjustable slot in the piston as it moves from one end of the cylinder to the other. At the completion of the desired period of time, the slot is closed, a port is opened at the top end of the cylinder, and the product is forced into a receiving can.

To operate, the melted sample is placed in container A with the piston E at the top of the stroke. Valve C is closed. Valve B is opened, and shaft D is tightened down. The lever is engaged to the speed reducer, and the piston E is pulled down. The lever is disengaged temporarily as the sample is drawn into the chilling chamber F. Valve B is then closed, and shaft D is rotated one full turn. The lever actuating the steam pump is engaged, and the piston goes up and down 12 times per minute, forcing the product through orifice G. At the completion of the cycle, shaft D is tightened when the plunger is at the bottom and the lever actuating the pump is disengaged on the upstroke. Valve C is opened, and the product is forced into a convenient receiver. Air may be admitted near the end of the cycle by opening valve B momentarily as the piston E is on the downward stroke. Some of the dimensions of the various parts are given in Figure 2.

The cooling is effected by allowing brine to pass through a copper coil submerged in a water bath. The usual temperature of the water is 34–36°F. The water is circulated with a one-half-in. Viking pump. The motor used to run the pump is one-fourth horse power. The motor also actuates the cam (through a speed reducer), which engages the lever for actuating the steam pump. The steam pump used is a Burnham 6⅛ x 12 in. Obviously other types would serve just as effectively. We have 125–140 p.s.i. steam pressure available in our pilot plant. The stroke is designed to bring the piston flush with the top and lower surfaces of the chamber. Retention of the product in the unit is less than 1%, which is easily removed by allowing the piston to move freely with the aperture closed by depressing shaft D and alternately opening and closing valves B and C. Where flavor is of prime importance, the unit may be flushed with salad oil or with some of the product to be plasticized.