will be converted entirely into 1-2 diglycerides. This involves the loss of about 40% of the total palmitic acid and about 6% of the total oleic acid.

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♣Factors Affecting Cloud Point Analysis of Palm Oleins

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ABSTRACT

Some of the factors affecting cloud point determination of palm oleins are described. These are the type of container used, method of stirring, rate of stirring and bath temperature. The repeatability and reproducibility standard deviations of the method are determined from collaborative trials. Recommendations for the test are made to reduce the large variations among laboratories.

INTRODUCTION

Cloud point determination of palm olein is an important test in the palm oil industry. It provides an indication of the unsaturation of the sample and of the stability of the sample to crystallization at room temperature. The test forms part of the required specifications of the customer.

When a sample of melted fat is steadily cooled and agitated to low enough temperature, turbidity is induced due to the formation of fat crystals. Cooling is continued until there are sufficient crystals to form an opaque cloud (defined as the point when the thermometer in the sample is no longer visible). The temperature at which this opaque cloud is observed is designated as the cloud point.

When testing samples of unknown cloud point, the cooling bath should be started at a sufficiently high temperature to avoid supercooling and the temperature lowered slowly, keeping a gradient of 2-5 C between the bath and the sample. In the AOCS Methods Cc 6-25, a second determination is made with the bath maintained at 2-5 C below the cloud point obtained in the preliminary determination.

This paper describes some of the factors affecting such determinations. It provides an indication of the errors that could arise from conducting the test as described above. The repeatability and reproducibility standard deviations obtained through several collaborative trials also are reported.

METHOD

The reference method used in this study was the AOCS Method Cc 6-25.

MATERIALS

Samples studied include crude palm oleins, physically refined palm oleins, degummed bleached oleins and alkali refined oleins. The oleins were from the single fractionation process.

RESULTS AND DISCUSSION

Effects of Sample Container

The recommended container in the AOCS Method Cc 6-25 is vaguely defined as oil sample bottles, 115 ml, 4 oz. It is observed that different sample containers gave varying cloud point depending on the thickness of the walls or bottle diameter. Table I shows the difference obtained with different types of containers. Lower cloud point values were observed with thin walled vessels such as Nessler tubes, and higher values obtained with thicker walled vessels such as sample bottles. The repeatability of analysis varied depending on the thickness of the walls of the vessels. The greater fluctuations obtained with the use of beakers probably are due to some supercooling occurring in the thin walled vessel. Sample containers should therefore be strictly standardized.

Effect of Stirring Angle and Rate of Stirring

The effect of stirring at an angle as against upright stirring is

TABLE I

Effect of Container

	Cloud point °C	Standard deviation °C	No. of analyses
Sample bottle (2.5 mm thickness)	9.4	0.07	6
Beaker (2.0 mm thickness)	7.7	0.29	6
Nessler tube (1.8 mm thickness)	5.9	–	1

TABLE II

Effect of Stirring

Stirring	Cloud point °C	Standard deviation °C	No. of analyses
Upright	9.4	0.09	6
Slanted angle	8.1	0.17	6
60 rpm	9.0	0.23	6
240 rpm	8.9	0.12	6

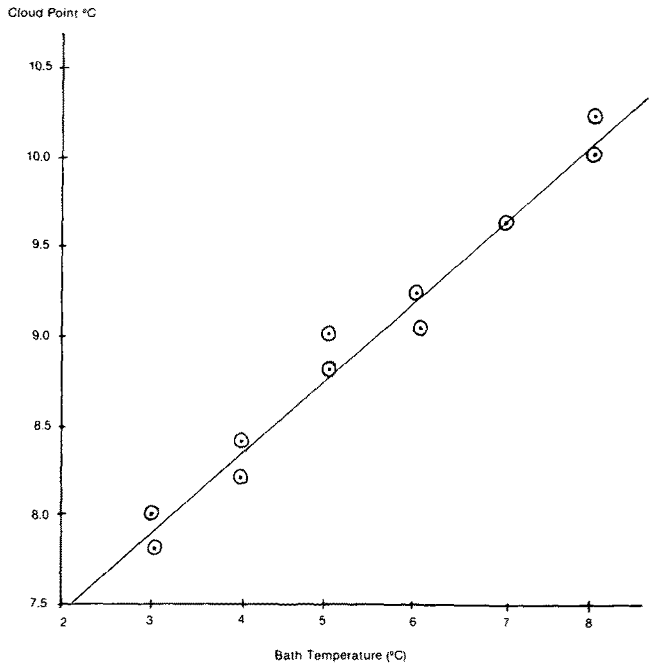

FIG. 1. Effect of bath temperature on cloud point using a beaker as container for measurement.

shown in Table II. The slanted angle of stirring gave a slightly higher repeatability standard deviation between analyses. Different hand stirring speed of approximately 60 rpm and 240 rpm also affects the cloud point measurements. A fairly rapid and steady stirring rate is recommended in view of the smaller standard deviation obtained between analyses. When a slower stirring rate is used, the initial cloud of crystals formed is not as efficiently distributed in the olein as when a faster rate of stirring is used. However, care must be taken to prevent any incorporation of air bubbles from the rapid rate of stirring.

Effects of Bath Temperatures

The AOCS Method Cc 6-25 procedure recommends a bath temperature ranging from 2-5 C below the expected cloud point. A preliminary measurement usually is made to determine the correct bath temperature required.

Figures 1 and 2 show the effect of bath temperature on cloud point values using beakers and sample bottles, respectively. The bath temperature has a greater effect on cloud point analysis when beakers are used. A lower cloud point is obtained with low bath temperatures, and higher values are observed with higher bath temperatures (Fig. 1). Figure 2 shows that when sample bottles are used, there is a small range of temperatures over which the cloud point remains fairly constant. Outside this range, a temperature increase resulted in higher cloud point values. The constant cloud point bath temperature range differs for different samples, as explained further below (Fig. 2). For most palm oil or olein samples this range lies between 0-5 C.

The cloud point of sample X is 14 C. The required bath temperature lies in the range 9-12 C. Cloud point changes within this range are minimal. Similar trends are observed with Sample Y. For sample Z with a cloud point of 8.5 C, the cloud point measurement was taken at a temperature between 3-6 C. Measurement at 3 C gives a cloud point of 8.5 C while at 6.5 C, the cloud point is 9.2 C. The effect of bath temperature is evident, and there will be some discrepancies in the results, particularly if the bath temperatures are not well controlled.

Hence, three points may be noted from the above observations:

(i) The effect of bath temperatures on cloud point is less significant when sample bottles are used.

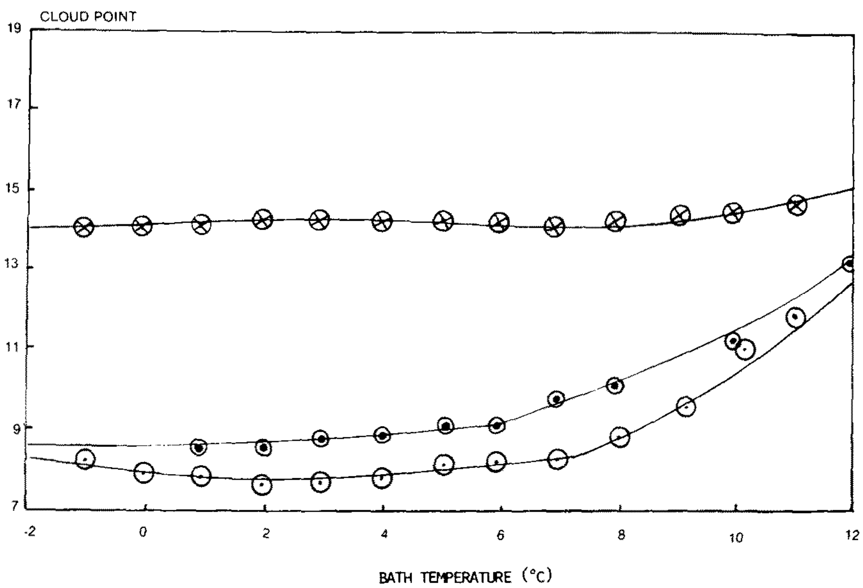

FIG. 2. Cloud point vs bath temperature. ⊗, sample X; ⊙, sample Y, and ◉, sample Z.

(ii) The recommendation that bath temperatures be 2-5 C below the expected cloud point, while suitable for other oils, is not very appropriate with palm olein.

(iii) A single temperature, preferably at 5 C, may be more appropriate for palm olein.

The Effect of Bath Temperatures on Clouding and Nucleation Time

The clouding time is defined as the time in seconds required for the sample to attain the cloud point; the nucleation time is the corresponding time in seconds when the first nucleus or crystal is observed.

Figures 3 and 4 show the nucleation time and clouding time versus bath temperature (respectively). Fairly similar behaviors are observed with nucleation time and clouding time against bath temperatures. It is observed that the bath temperatures in the 9-12 C range (Sample X) and 3-6.5 C (Sample Y) lie at the region of the graph where the greatest changes occur in clouding time and nucleation time (Region G). In fact, the 9 C and 3 C of sample X and Y were at the edge of the region of least time changes (Region L). These graphs support the recommendation of measuring the cloud point at bath temperature 5 C below the expected value.

Effect of Heating Time on Volatile Matter and Cloud Point of Filtered and Unfiltered Samples

Table III shows the effect of heating times of 3, 5, 10 and 30 min on the volatile matter and cloud points of filtered and unfiltered oil samples.

Filtering the oil samples using Whatman No. 1 filter paper reduces the amount of volatile matter. The cloud point of unfiltered sample varies with the heating time, but remains fairly constant with filtered sample. It is important to filter samples before measurement in order to avoid variations due to moisture and dirt levels.

Effect of Light

A difference of about 1.5 C is observed when samples are

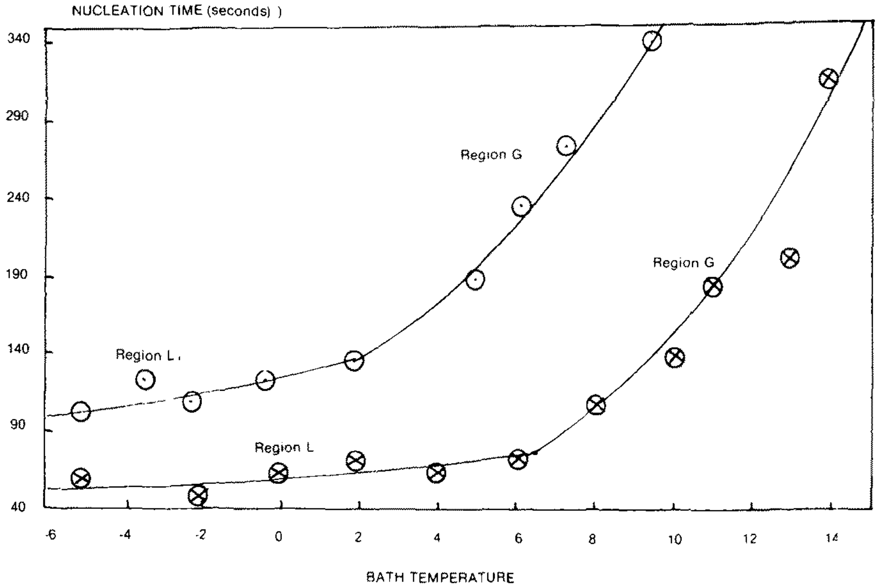

FIG. 3. Nucleation time vs bath temperature. ⊗, sample X, and ⊙, sample Y.

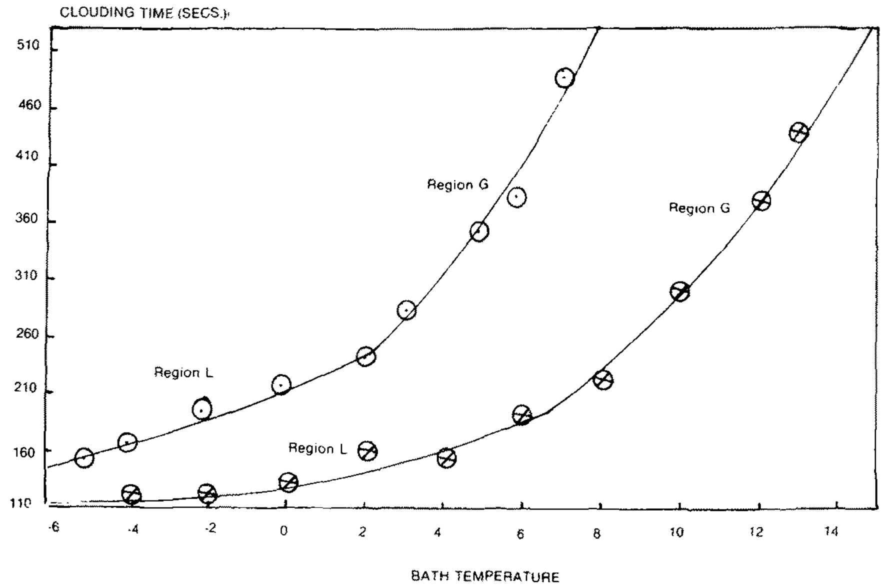

FIG. 4. Clouding time vs bath temperature. ⊗ sample X, and ⊙, sample Y.

TABLE III

Effect of Heating Time on Volatile Matter and Cloud Point

Heating time (min)	3	5	10	30
Vol. matter (%) (unfiltered oil)	0.034	0.016	0.032	0.029
Vol. matter (%) (filtered oil)	0.012	0.011	0.011	0.006
Cloud point (° C) (filtered oil)	7.0	7.0	7.2	7.2
Cloud point (° C) (unfiltered oil)	6.3	5.9	5.7	5.5

TABLE IV

Repeatability and Reproducibility Standard Deviations

	Collaborative trials		
	No. 1	No. 2	No. 3
Bath temperature	2-5C below expected cloud point	5 C	0 C
Sample container	Variable	Beaker	Beaker
Cloud point (C.P., ° C)	8.6	8.6	8.4
No. of participants	29	31	31
Sr (repeatability s.d., ° C)	0.19	0.22	0.25
Sb (laboratory bias, ° C)	1.01	0.69	0.88
Sd (reproducibility s.d., ° C)	1.02	1.00	1.26

viewed with and without a suitable light source. This is especially important in rooms which are dimly lit.

The light source should be placed at a distance of about 60 cm away from the sample bottle.

Repeatability and Reproducibility Standard Deviations

The standard deviations within and between laboratories are determined from several collaborative trials (Table IV). The systematic errors as shown by Sb are greatest when participants carried out measurement at bath temperatures ranging from 2-5 C below the cloud point determined initially. This was reduced when a fixed temperature was used. When 0 C was used, the slightly higher repeatability standard deviation (Sr) observed probably was due to effects of supercooling. Hence, fixing a bath temperature between 2 and 5 C (Fig. 2) would offer certain advantages such as reduced analysis time and offer better reproducibility between laboratories.

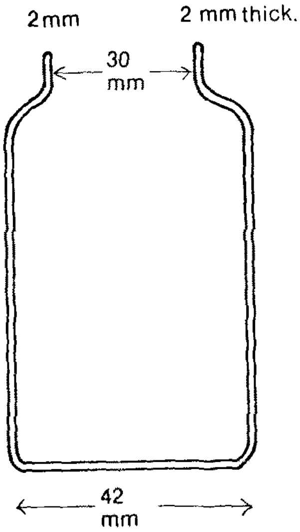

FIG. 5. Illustration of the 155-ml, 4-oz bottle used for cloud point measurements.

Recommendations

For standardization of the procedure for single fractionation palm oleins the following points are recommended and considered useful for inclusion as notes in the method description.

(i) A sample bottle, 115 ml, 4 oz (Beatson bottle) with diameter of 42 mm and wall thickness 2 mm is recommended (Fig. 5).

(ii) The bath temperature is recommended to be at least 5 C below the cloud point determined initially. For palm oleins, a suitable temperature fixed at 5 C could be used in order to reduce interlaboratory variations.

(iii) The samples should be filtered using a Whatman No. 1 filter paper prior to measurements.

(iv) A suitable hand stirring speed of approximately 240 *rpm is suggested.*

(v) A light source (15 watt) placed at a distance of 60 cm from the bottle is recommended when viewing the thermometer in the sample.

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