

Tempering Method for Chocolate Containing Milk-Fat Fractions

S. Yella Reddy¹, N. Full², P.S. Dimick*, and G.R. Ziegler

Department of Food Science, The Pennsylvania State University, University Park, Pennsylvania 16802

ABSTRACT: Anhydrous milk fat (AMF) was fractionated by a two-stage dry fractionation process to produce three fractions—high- (HMF), middle-(MMF), and low-melting (LMF). The effect of replacing 12.2–40% by weight of cocoa butter with these fractions on the tempering profile of milk chocolate was studied. Degree of temper was evaluated by differential scanning calorimetry, and expressed as the ratio of enthalpies of melting for higher-stability polymorphs to those of lesser stability. The degree of temper was dependent on the crystallization time and temperature, and the type and quantity of milk-fat fraction in the formulation. Chocolates containing AMF or its fractions in concentrations of up to 20 wt% (total fat basis) were tempered after a conventional thermocycling tempering process (50°C/30 min, 27.7°C/4 min, 31°C/2 min) to obtain products with good contraction and mold release properties. For those milk chocolate formulations that did not temper by the conventional method and resulted in poor contraction and mold release, a new tempering protocol was developed. Lower crystallization temperatures and/or longer holding times were required at concentrations of AMF, MMF, or LMF above 20%. Chocolate containing HMF required slightly higher crystallization temperatures because of high viscosity. Chocolates containing up to 35% HMF and up to 40% of the total weight of fat in the chocolate of AMF, MMF, and LMF were successfully tempered by adjusting crystallization time and temperature.

JAACS 73, 723–727 (1996).

KEY WORDS: Chocolate, differential scanning calorimetry, dry fractionation, milk fat, tempering.

In chocolate manufacture, the tempering process is prerequisite for quality, significantly influencing both sensory and physical properties, such as gloss, snap, texture, heat resistance, and bloom stability (1). Tempering is a process by which the fat phase in chocolate is conditioned by thermal and mechanical means to drive the fat into the desirable stable polymorphic form in proper number, size, and at the proper time. Chocolate that is in good temper consists of the maximum number of small crystals in the right crystalline form, well distributed throughout the mass. Failure to produce suffi-

cient crystals in a stable form results in handling problems for the manufacturer, gives the product poor setting characteristics, and results in quality defects (2). Prior to enrobing or molding, chocolate must be tempered to contain the stable β_2 -3 form (Form V, m.p. = 34–35°C) with a high degree of maturity (2). Currently, commercial-scale tempering is a two-stage crystallization process: precrystallization and bulk crystallization (1). Precrystallization is carried out by a cooling/reheating process, in which the molten chocolate is first cooled from about 50 to 26–27°C, then reheated to 30–31°C. Thereafter, bulk crystallization is accomplished at about 8–15°C to produce the end products (3). Precrystallization ensures proper polymorphic form of the nucleated seed crystals. Initial cooling at 26–27°C crystallizes both the metastable and stable polymorphs of cocoa butter (CB) (1). The metastable forms, which cause undesirable solidification behavior, are transformed to the more stable forms during subsequent reheating at 30–31°C. CB is crystallized in Form V by this cooling/reheating process (3). The amount of CB crystallized during tempering has been reported to range from 0.1 to 0.5% (4,5). The depositing or enrobing temperature depends on the quantity of crystalline fat and its polymorphic form. The precise degree of temper needs rapid scientific confirmation if large machines are to work consistently. Differential scanning calorimetry (DSC) has been successful in measuring the degree of temper in precrystallized chocolate, which is based on determination of the stable seed crystals present in the fat phase (6). Also, DSC is useful for predicting tempering behavior of different CB samples by plotting melting enthalpy vs. percentage contribution of the polymorphic forms (6).

In the United States, milk chocolate is produced in greater quantity than sweet chocolate. Milk fat contributes to the flavor and smooth texture of milk chocolate, is less costly than cocoa butter, and aids in preventing fat bloom (7–12). Milk fat delays the crystallization and lowers the melting points of the polymorphic forms of CB, thus chocolates containing higher concentrations of milk fat require lower temperatures and longer times during tempering (7,9,13). It has been reported that the incorporation of the high-melting fraction of milk fat into chocolate reduces fat bloom (11,12). Barna *et al.* (13) observed distinct differences in tempering profiles for chocolates that contain different levels of solvent-fractionated milk fat. They also reported that chocolates containing high-melting fraction (HMF) at 20% and middle-melting

¹Current address: Lipid Technology Department, Central Food Technological Department, Mysore-570 013, India.

²Current address: M & M Mars, Inc., Elizabethtown, PA.

*To whom correspondence should be addressed at Department of Food Science, 116 Borland Lab, The Pennsylvania State University, University Park, PA 16802

fraction (MMF) and low-melting fraction (LMF) at 30% replacement levels of CB were untemperable. In the present study, the tempering procedures were determined for milk chocolates formulated with anhydrous milk fat (AMF) and its fractions as a replacement of CB at different concentrations.

MATERIALS AND METHODS

Trace moisture present in commercial AMF procured from Tedford/Tellico (Concord, TN) was removed by heating the fat to 60°C, adding anhydrous sodium sulfate, and filtering. Low-heat nonfat dry milk, sucrose, chocolate liquor (West Africa and Ecuador), and CB (Nigeria) were all obtained from commercial sources. Lecithin (Clearate B-70) was obtained from the W.A. Cleary Corp. (Somerset, NJ).

Fractionation of AMF. AMF was fractionated by a two-stage dry fractionation process to produce three fractions (14). The fat was fractionated first at 29°C for 6 h to obtain the HMF (m.p. 42°C, yield 20%). The resultant olein was re-fractionated at 18°C for 6 h to yield the MMF (m.p. 33°C, yield 20%), and LMF (m.p. 16°C, yield 60%) fractions.

Milk chocolate formulation. Milk chocolate with 30% total fat was prepared by using the following formulation: 15% chocolate liquor (54.7% fat), 15.5% nonfat dry milk, 47.3% sucrose, 21.8% fat blend containing CB and AMF or its fractions, and 0.4% lecithin. Total batch size was 500 g. AMF or its fractions were incorporated at 12.2, 20, 25, 30, 35, and 40% by weight on a total fat basis, resulting in a range from 3.66 to 12% in the milk chocolate formulation. The ingredients minus lecithin were mixed with 60% of the total fat in a Hobart mixer (Hobart Corporation, Troy, OH) and refined in a horizontal 3-roll refiner (Lehmann Maschinenfabrik, Aalen/Wurt, Germany) to a median, volume-based diameter of $11.6 \pm 1.4 \mu\text{m}$. The remaining fat and lecithin were added, and the mass was conched at 60°C for 6 h in a jacketed Hobart mixer with a circulating water bath. The chocolate was tempered in a thermostatically controlled tempering kettle (Little Dipper, Hilliard's Chocolate System; JIMSAN Enterprises, Inc., West Bridgewater, MA). A conventional batch tempering process was employed. Molten (50°C) milk chocolate was cooled in 20–25 min to the crystallization temperature of 27.7°C, and held for the crystallization time of 4 min, after which a sample (10–15 mg) was removed from the kettle for DSC analysis to determine temper. The inclusive time required to draw, weigh, and place the sample on the DSC head was one minute. If proper temper was not observed after four minutes at the crystallization temperature, samples were taken from the kettle at longer time periods (e.g., 8, 12, 20 min) until proper temper was attained. If the crystallization time exceeded 60 min, the milk chocolate mass was reheated to 50°C, held for 30 min, and retempered at a lower crystallization temperature. The final molding temperature was approximately 31°C. Chocolate was molded in preheated (23–25°C) stainless-steel molds and placed in a cooling cabinet at 12–15°C (air temperature) for one hour to complete the solidification process. All experiments were conducted in duplicate.

DSC. The DSC (model 7; Perkin-Elmer Corp., Norwalk, CT) was calibrated with Indium (m.p. 156.6°C, ΔH_f 28.45 J/g). Chocolate samples (10–15 mg) were accurately weighed (Autobalance; Perkin Elmer Corp.) into aluminum DSC pans and immediately introduced onto the DSC head at -5°C. Thermograms were obtained from -5 to 50°C at 20°C/min.

RESULTS AND DISCUSSION

The degree of temper, expressed as the ratio of ΔH_f (J/g) of Peak 2 (higher-melting polymorphs) to Peak 1 (lower-melting polymorphs), was calculated from DSC endotherms (Fig. 1). Mold release, snap, and surface gloss were evaluated visually. Good mold release, snap, and gloss were obtained when the Peak 2/Peak 1 ratio was ≥ 0.4 . Poor mold release and snap were observed below this value; therefore, a degree of temper ≥ 0.4 was considered adequate. The degree of temper was dependent on the crystallization time and temperature and on the type and quantity of milk-fat fraction in the formulation. Chocolates containing AMF or its fractions in concentrations of up to 20 wt% (fat basis) were adequately tempered after 4 min at 27.7°C. However, chocolates containing concentrations of AMF at >20 wt% did not develop a sufficient quantity of high-melting stable crystals for adequate temper at the above conditions (Table 1). At four minutes at 27.7°C temper temperature, crystallization of CB was noticeably delayed by the presence of LMF, MMF, and HMF. To adequately temper and mold products that were untemperable or unmoldable after four minutes at 27.7°C, the crystallization time and temperature were altered. The crystallization time had to be lengthened as the concentration of AMF increased (Fig. 2) to maintain the degree of temper above 0.4. Milk chocolate containing 40% AMF required lowering of the crystallization temperature in addition to lengthening of time (Fig. 2). Chocolate containing 50% of AMF could not be tempered because it did not develop a sufficient quantity

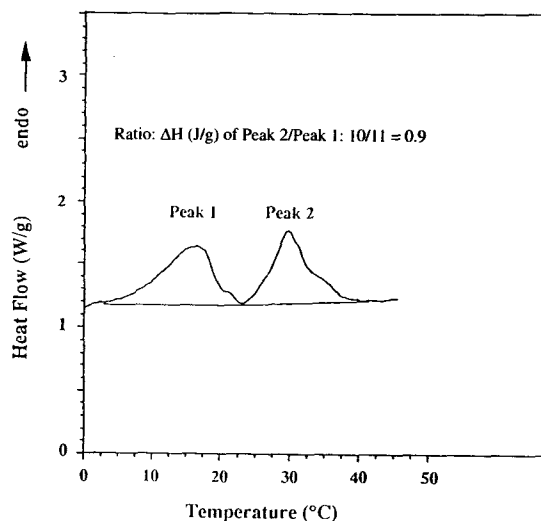


FIG. 1. Typical differential scanning calorimetry thermogram of unstable and stable crystals generated during tempering of chocolate.

TABLE 1
Enthalpy of Crystal Forms and Their Ratios Generated During Tempering of Chocolates
Containing Various Proportions of AMF^a

AMF (%)	Enthalpy (ΔH , J/g) at tempering temperature 27.7°C after:								
	4 min			8 min			25–30 min		
	Peak 1	Peak 2	Ratio Peak 2/Peak 1	Peak 1	Peak 2	Ratio Peak 2/Peak 1	Peak 1	Peak 2	Ratio Peak 2/Peak 1
12.2	17.0	14.5	0.85						
20.0	16.2	6.4	0.40	15.0	10.2	0.70			
25.0	13.0	3.6	0.27				13.8	9.7	0.70
30.0	12.0	0.3	0.03	14.0	3.0	0.20	14.4	8.6	0.60
40.0	11.5	0.5	0.04	8.0	2.5	0.30	12.2 ^b	7.7	0.60
50.0	9.0	0.3	0.03				11.2 ^c	2.4	0.20

^aAnhydrous milk fat.

^bTempered at 26.6°C for 60 min.

^cTempered at 26.6°C for 70 min.

of high-melting crystal polymorphs, even after 70 min at 26.6°C (Table 1).

Products containing MMF, except at 40%, were tempered under similar conditions to those containing AMF. The degree of temper increased from 0.1 to 0.7 in chocolates containing 30% MMF as the time at 27.7°C increased from 8 to 30 min (Fig. 3). Chocolates containing concentrations of MMF above 25% showed broad melting peaks that extended to a higher temperature (34°C). However, tempering at 28.8°C did not produce a sufficient quantity of high-melting crystal polymorphs in these products to yield good gloss and mold release properties. The degree of temper decreased at normal tempering conditions as the concentration of LMF increased. Chocolates with 30 and 40% LMF developed few high-melting crystals after 8–10 min at 27.7°C, which resulted in poor mold release properties. However, by increasing crystallization time and lowering the temperature, choco-

lates containing up to 40% LMF were well tempered (Fig. 4). For example, when crystallization temperature was lowered to 26.6°C and the time increased to 30–60 min, chocolate containing 30–40% LMF exhibited good mold release properties.

Products containing HMF behaved differently than those with AMF, MMF, or LMF. Although the degree of temper was adequate, chocolate mass with 20–35% of HMF was too viscous under normal tempering conditions to deposit easily into the molds. To lower viscosity of the mass, the crystallization temperature was increased to 30–31.1°C, but insufficient high-melting crystals developed at Peak 2 (Fig. 5), even though higher-melting endotherms (38–39°C) were observed. The degree of temper was sufficient to yield well-tempered chocolate when the crystallization temperature was lowered to 28.8°C (Fig. 6). Additional shoulders or endotherms at higher temperatures (>32°C) appeared in thermograms of

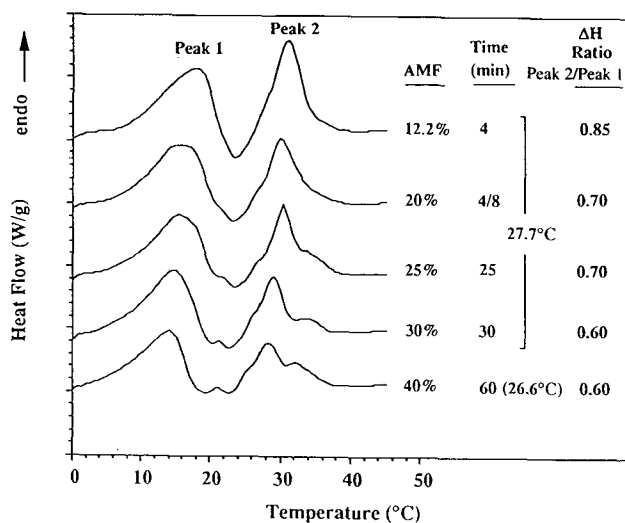


FIG. 2. Differential scanning calorimetry thermograms (heating rate 20°C/min) of chocolates containing various concentrations of anhydrous milk fat (AMF) recorded at different tempering crystallization temperatures and times.

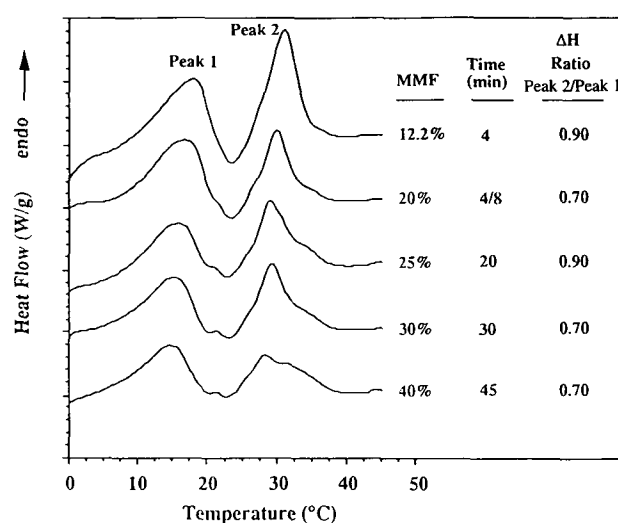


FIG. 3. Differential scanning calorimetry thermograms (heating rate 20°C/min) of chocolates containing various concentrations of middle-melting fraction (MMF) of anhydrous milk recorded at different tempering times.

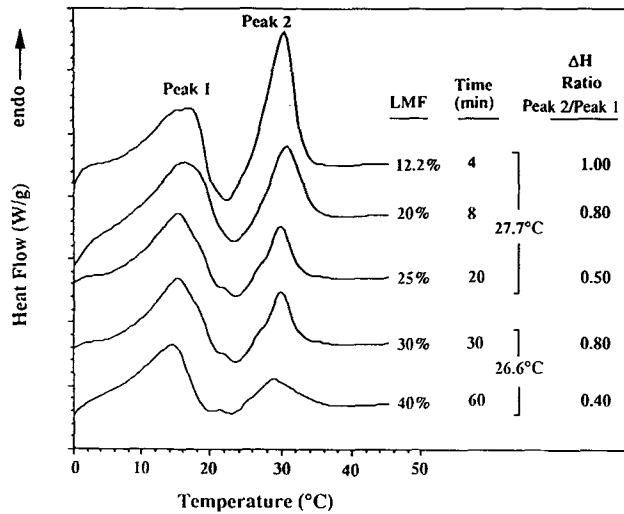


FIG. 4. Differential scanning calorimetry thermograms (heating rate 20°C/min) of chocolates containing various concentrations of low-melting fraction (LMF) of anhydrous milk fat recorded at different tempering crystallization temperatures and times.

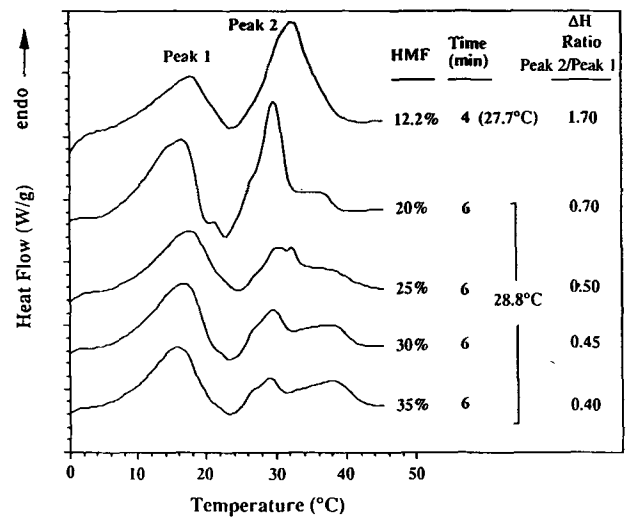


FIG. 6. Differential scanning calorimetry thermograms (heating rate 20°C/min) of chocolates containing various concentrations of high-melting fraction (HMF) of anhydrous milk fat recorded at different crystallization tempering temperatures and times.

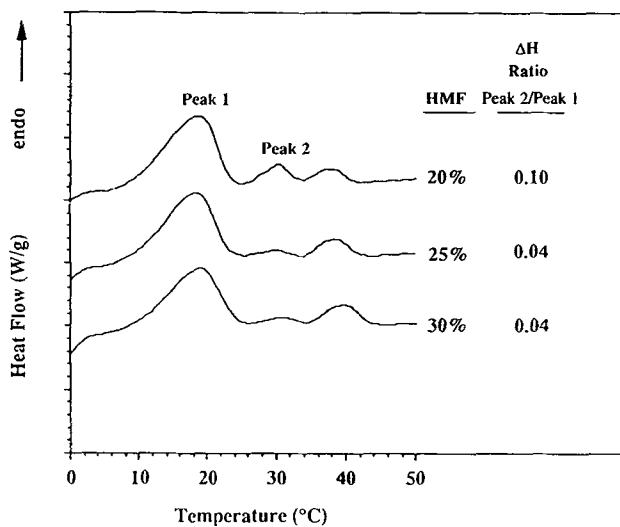


FIG. 5. Differential scanning calorimetry thermograms (heating rate 20°C/min) of chocolates containing high-melting fraction (HMF) of anhydrous milk fat recorded during tempering at 31.1°C after 10 min.

chocolates that contained 20–35% HMF, likely due to the high-melting triacylglycerols of milk fat (Dimick, P.S., S.Yella Reddy, and G.R. Ziegler, unpublished data), and may be responsible for the high viscosity of the mass. Chocolates with higher proportions (>35%) of HMF were too viscous, even at 28.8°C, to be easily molded. However, the high viscosity may be advantageous in such product applications as drop depositing (14).

Because AMF or its fractions lowered the peak melting temperature of CB, the degree of temper decreased when the samples were heated to the normal final molding temperature (31°C). Therefore, the molding temperature was lowered to

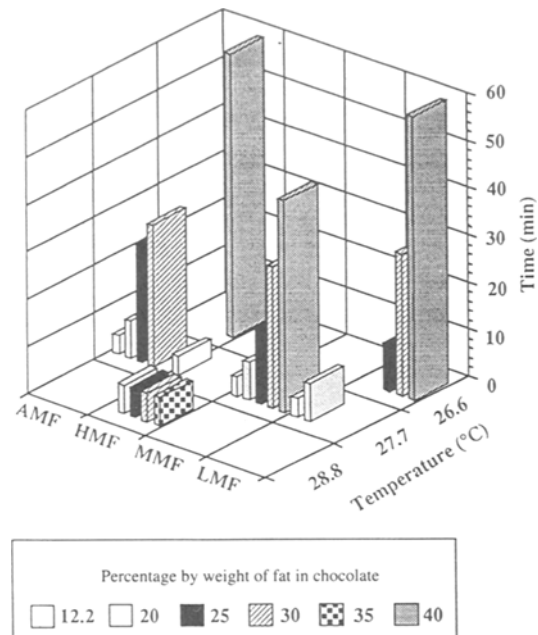


FIG. 7. Tempering profiles for milk chocolates containing various proportions of milk-fat fractions. See Figures 2–6 for abbreviations.

just below that of the melting temperature observed for the high-melting crystals (Peak 2). These results confirm earlier reports that milk fat delays the crystallization of CB, lowers the melting temperature of CB, and that chocolates containing milk fat require lower temperatures and longer times for tempering (7,10–13).

The final tempering profiles for chocolates containing various proportions of milk fat or its fractions are shown in Figure 7. Chocolates with HMF could be tempered at slightly

higher temperatures. Chocolates with AMF and MMF could be tempered under similar conditions. Products containing LMF required tempering at lower temperatures and for prolonged periods to overcome the dissolution effect of LMF on CB. We have observed that, as the concentration of LMF was increased in a mixture with CB, the melting endotherm of CB gradually disappeared and the amount of crystallized material was lower (Dimick, P.S., S.Yella Reddy, and G.R. Ziegler, unpublished data). By adjusting both time and temperature of the process, chocolate with up to 40% of AMF, MMF, and LMF, and up to 35% of HMF on a total fat basis were successfully tempered to obtain milk chocolate with good mold release properties and gloss. The products were free from fat bloom upon storage (15). This is in contrast with reports that chocolates with 20% of HMF and 30% of MMF and LMF of milk fat were untemperable (13). DSC was found to be a good and rapid analytical tool for evaluating the degree of temper in chocolate during the tempering process. Molding or enrobing temperatures could be determined based on the melting temperature.

ACKNOWLEDGMENT

The work was supported in part by the National Dairy Promotion and Research Board, Arlington, VA.

REFERENCES

1. Martin, R.A., Jr., Chocolate, in *Advances in Food Research*, Vol. 31, edited by C.O. Chichester, E.M. Mark, and B.S. Schweigert, Academic Press, New York, 1987, pp. 211–342.
2. Talbot, G., Chocolate Temper, in *Industrial Chocolate Manufacture and Use*, 2nd edn., edited by S.T. Beckett, Chapman and Hall Ltd., London, 1994, pp. 156–166.
3. Hachiya, I., T. Koyano, and K. Sato, Seeding Effects on Solidification Behavior of Cocoa Butter and Dark Chocolate. I. Kinetics of Solidification, *J. Am. Oil Chem. Soc.* 66:1757–1762 (1989).
4. Campbell, L.B., and P.G. Keeney, Temper Level Effect on Fat Bloom Formation on Dark Chocolate Coating, *Food Technol.* 22:1150 (1968).
5. Koyano, T., I. Hachiya, and K. Sato, Fat Polymorphism and Crystal Seeding Effects on Fat Bloom Stability of Dark Chocolate, *Food Structure* 9:231–240 (1990).
6. Merken, G.V., S.V. Vaeck, and D. Dewulf, Determination of the Technological Properties of Cocoa Butter by Means of Differential Scanning Calorimetry, *Lebensm. Wiss. u. Technol.* 15:195–198 (1982).
7. Chapman, G.M., E.E. Akehurst, and W.B. Wright, Cocoa Butter and Confectionery Fats. Studies Using Programmed Temperature X-Ray Diffraction and Differential Scanning Calorimetry, *J. Am. Oil Chem. Soc.* 48:824–830 (1971).
8. Hendrickx, H., H. DeMoor, A. Huyghebaert, and G. Janssen, Manufacture of Chocolate Containing Hydrogenated Butterfat, *Rev. Int. Choc.* 26:190–193 (1971).
9. Lohman, M.H., and R.W. Hartel, Effect of Milk Fat Fractions on Fat Bloom in Dark Chocolate, *J. Am. Oil Chem. Soc.* 71:267–275 (1994).
10. Hogenbirk, G., Influence of Milk Fat on the Crystallization and Melting Behavior of Cocoa Butter and Cocoa Butter Alternatives, *Proc. of Ann. Production Conf. of PMCA* 44:53–60 (1990).
11. Jebson, R.S., The Use of Fractions of Milk Fat in Chocolate, *XIX Int. Dairy Congress*, International Dairy Federation, Brussels, Belgium, 1974, p. 761.
12. Dimick, P.S., L.N. Thomas, and C. Versteeg, Potential Use of Fractionated Anhydrous Milk Fat as a Bloom Inhibitor in Dark Chocolate, *INFORM* 4:504 (1993).
13. Barna, C.M., R.W. Hartel, and S. Martin, Incorporation of Milk Fat Fractions in Milk Chocolate, *The Manufacturing Confectioner* 72:107–116 (1992).
14. Urbanski, J., Chocolate Manufacturing: Drops, Chips, Chunks, Etc., *Ibid.* 74:47–54 (1994).
15. Full, N., Physical and Sensory Properties of Milk Chocolate Made with Dry-Fractionated Milk Fat, M.S. Thesis, The Pennsylvania State University, University Park, 1995.

[Received October 5, 1995; accepted February 20, 1996]