bands at 3040 w (ring C-H, Str, -CH - CH-), 1720 ($\underline{C}OOCH_3$), 1190, 1160, 1090, 1025 cm^{-1} (C-O). The band at 1025 and 3040 cm^{-1} in the IR spectrum established the presence of cyclopropane moiety and a prominent band at 1160 cm^{-1} indicated the C-O stretching of an ether function. The assigned structure of methyl 4-methoxy-*trans*-2,3-methylenehexadecanoate [2] was further confirmed by the NMR spectrum. It gave signals at δ 3.66 s (3H, -COO$\underline{C}H_3$), 3.6 s (3H, -O-$\underline{C}H_3$), 3.35 (1H, -$\underline{C}H$-OCH_3), 1.69 m (1H, -CH - $\underline{C}H$-$COOCH_3$), 1.3 br,s (chain-$\underline{C}H_2$) and 0.85 m (6 protons, -CH - CH-$COOCH_3$; and terminal -$\underline{C}H_3$).

Compound [3] was also obtained as an oil analyzed for $C_{18}H_{34}O_3$. This composition can give rise to 2 possible structures [3 and 4], which can be derived from [1*b*] under the reaction conditions. The IR and NMR spectra of the product support structure [3]. The IR spectrum gave bands at 3430 (OH), 3030 w (ring C-H, Str, -CH - CH-), 1725 ($\underline{C}OOCH_3$), 1175, 1025 (C-O). The NMR spectrum gave signals at δ 4.55 br (1H, O$\underline{H}$, disappeared on D_2O addition), 3.65 s (3H, -COO$\underline{C}H_3$), 3.5 m (1H, C$\underline{H}$-OH), 1.71 m (1H, -CH - $\underline{C}H$-$COOCH_3$), 1.3 br,s (chain-$\underline{C}H_2$) and 0.9 m (6 protons, -CH - CH-$COOCH_3$; and terminal -$\underline{C}H_3$).

In both cases one of the cyclopropane protons appeared downfield (δ 1.69 and δ 1.71), which is understandable since one cyclopropane proton is α to the ester carbonyl group. After addition of D_2O the spectrum of [3] was slightly modified.

In conclusion, it may be added that it has been reported that the ester carbonyl suppresses cyclopropanation of the conjugated double bond (3) and the yields are ca. 20-25%. However, the presence of an allylic hydroxyl group increases the double bond reactivity of the α,β-unsaturated ester and this might be one of the reasons for the formation of cyclopropane derivative in high yield (∼90%) in relatively less reaction time.

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Detection of Goat Body Fat in Ghee by Differential Thermal Analysis[1]

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ABSTRACT

The differences in the melting diagrams and crystallization patterns of goat body fat and ghee, as determined by differential thermal analysis, provide a basis for the determination of adulteration in cow or buffalo ghee. The new endothermic peak on the melting diagram in samples having more than 10% goat body fat can be used for qualitative detection and the crystallization diagram can be used for quantitative estimation.

INTRODUCTION

Ghee (clarified butterfat) is a popular indigenous product in India prepared from cream or butter by a heat clarification process (1-3). Like butter, ghee is sometimes adulterated with animal or vegetable fat; a common adulterant is animal body fat (4, 5). In the past, several methods have been tried to detect such adulteration based on color test (6) or chromatography (7, 8). This paper offers a method for detecting goat body fat in ghee using differential thermal analysis (DTA).

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MATERIALS AND METHODS

Milk samples were obtained from a cow or buffalo maintained as National Dairy Research Institutes herds. Ghee samples were prepared either from cow milk or buffalo

TABLE I

Mixtures of Ghee and Goat Body Fat Analyzed by Differential Thermal Analysis

Sample no.	Sample composition
1	Goat body fat (pure)
2	Buffalo ghee (pure)
3	Cow ghee (pure)
4	Buffalo ghee + 5% goat body fat
5	Buffalo ghee + 10% goat body fat
6	Buffalo ghee + 20% goat body fat
7	Cow ghee + 5% goat body fat
8	Cow ghee + 10% goat body fat
9	Cow ghee + 20% goat body fat

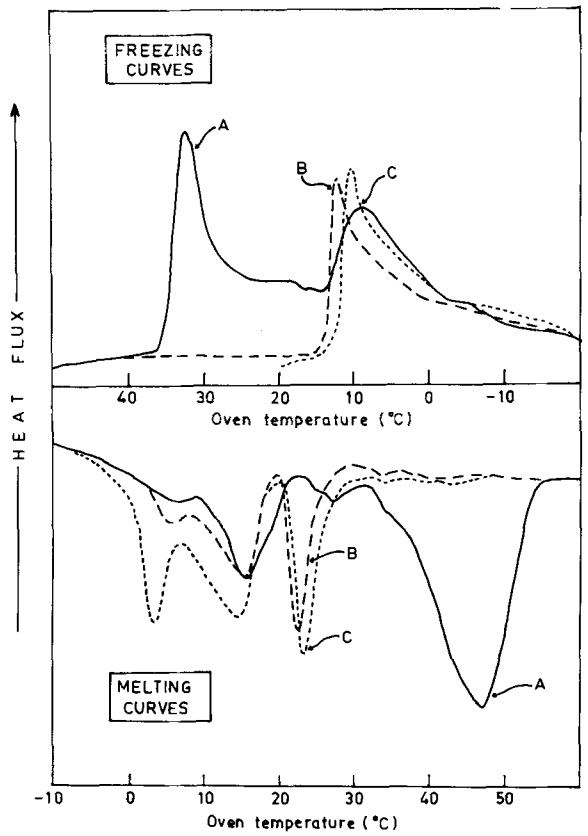

FIG. 1. The freezing and melting diagrams of goat body fat (A), buffalo ghee (B) and cow ghee (C).

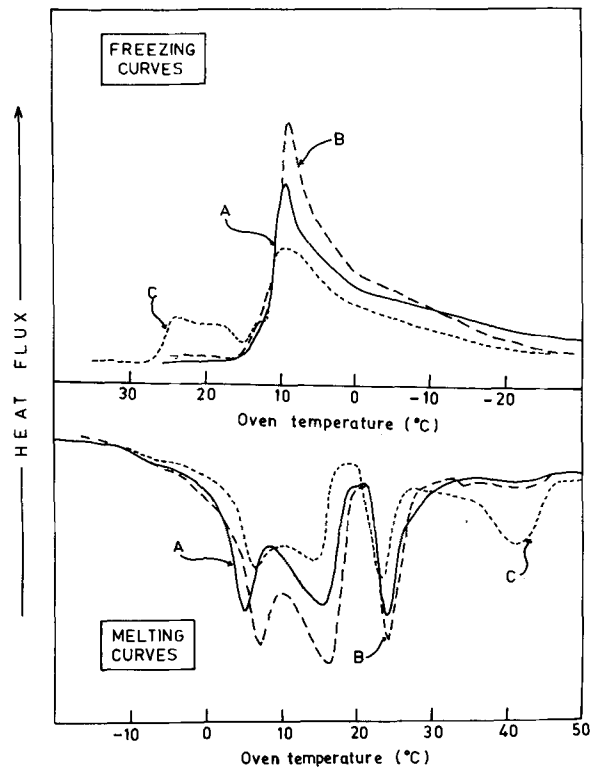

FIG. 2. The freezing and melting diagrams of cow ghee samples adulterated with goat body fat at 5% (A), 10% (B) and 20% (C) levels.

milk by the creamery method (1). Fresh cream was heated over an electric heater in a stainless steel vessel to 119-120 C and kept at this temperature for ca. 5 min, strained through a double-fold muslin cloth, filled in bottles, cooled and stored in a refrigerator at a temperature of 4-5 C for analysis. The moisture content of the ghee samples was 0.25%. Goat body fat was prepared by melting adipose tissue (120-125 C) obtained from a local slaughter house, strained through a double-fold muslin cloth and used without any further purification. The moisture content of the sample was 0.2%. The mixtures prepared from ghee and body fat for analysis are shown in Table I. These samples were then analyzed by DTA according to the following procedure. About 5 mg of fat was heated from -30 to 70 C at a heating rate of 2 C/min and then cooled from 70 to -30 C at a cooling rate of 2 C/min. The DTA was carried out using the Mettler & Co. Model TA-2000B having a temperature range between -170 and 550 C. The curve areas were measured by planimeter.

RESULTS AND DISCUSSION

It is clear (Fig. 1) that the DTA diagrams of the pure goat body fat is different from the 2 pure milk fats. Melting diagram of the goat body fat shows a big endothermic peak between 30 and 60 C, whereas the 2 other fats are completely molten at temperatures higher than 30 C. There is also some difference in the melting diagram in ghee from cow and buffalo milk which could result from the difference in its triglyceride composition (9,10). Crystallization of goat body fat begins at ca. 35 C, whereas the 2 other fats begin to crystallize at 15 C. These differences provide a basis for the determination of adulteration in cow or buffalo ghee using DTA.

Adding 5% of goat body fat does not provide any significant difference in the DTA diagrams of cow (Fig. 2) or buffalo ghee (Fig. 3). But adding 10% or more goat body

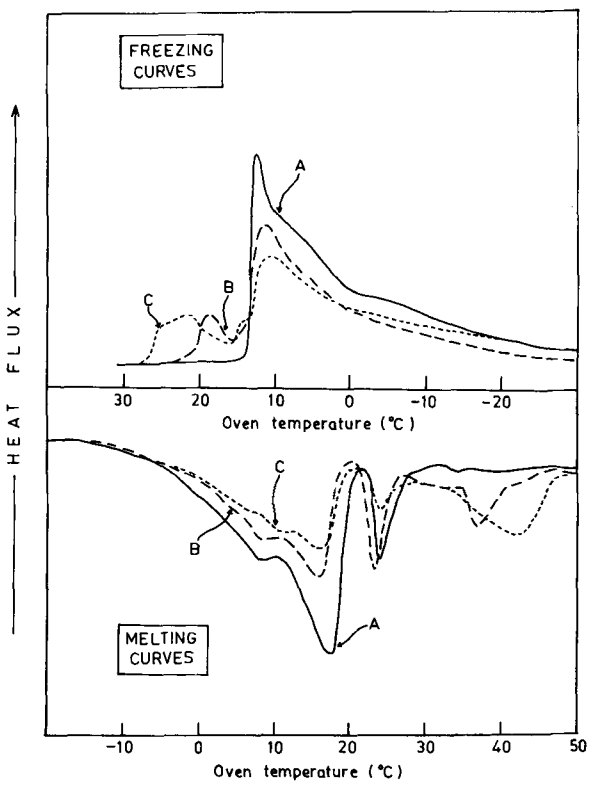

FIG. 3. Conditions same as in Fig. 2 for buffalo ghee samples.

TABLE II

Quantitative Determination of Goat Body Fat in Buffalo and Cow Ghee by Differential Thermal Analysis Freezing and Melting Curves

Goat body fat added (%)	Goat body fat (%) by DTA			
	Buffalo ghee		Cow ghee	
	Freezing	Melting	Freezing	Melting
10	9.9	13.7	–	3.9
20	21.0	27.0	19.8	20.3

fat to buffalo or cow ghee involves significant modification of the corresponding diagrams with greater effect for buffalo ghee. If goat fat is added, a new endothermic peak appears on the melting diagrams at temperatures higher than 30 C and a new exothermic peak on the crystallization diagrams at temperatures higher than 15 C. This means that above 10%, goat body fat can be qualitatively determined.

Further, based on the area percentage of this new peak appearing in the crystallization diagram, it is possible to determine goat body fat quantitatively above a level of 10% in buffalo ghee (Fig. 3) and above a level of 15-20% in cow ghee (Fig. 2). These data are given in Table II. Quantitative determination of goat body fat by measurement of the corresponding peak areas in the melting diagrams is theoretically possible, but requires previous calibration.

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