

TECHNICAL NOTES

Method for Extracting Fat from Dry Whole Milk

Abstract

A method for extracting fat from dry whole milk is described in which the only solvent is methylene chloride. Fat is freed in the powder by preliminarily mixing with powdered ice cooled to -30°C . A specially designed extraction apparatus is described. Average yields of 97% of the total fat are obtained with 4-hr extractions. Steam deodorizations of fat samples give distillates suitable for gas chromatographic and organoleptic examinations free from interferences encountered with alcohol-extracted fat.

Introduction

In connection with off-flavor studies, we needed a method for extracting representative samples of fat from dry whole milk. Published procedures by Lampitt and Bushill (5), Whitney and Tracy (7), and Emery and Schwartz (1) appeared unsuitable because they required either a long hydration period or the use of an alcohol. A procedure by Greenbank and Pallansch (2) was otherwise satisfactory but gave us lower yields of fat than desired. In this report we describe an extraction with only methylene chloride and water which gives high yields of milk fat.

Experimental Procedures

Dry milk was prepared (3) and methylene chloride purified (6) as previously described. Powdered ice at -30°C was prepared by holding fine ice in a clean cloth against dry ice for about 15 min and then rapidly crushing to a powder. Fat in milk powders was determined by the Mojonnier method and in extracts by removing the solvent from a weighed aliquot by the Mojonnier procedure. Moisture was determined by toluene distillation. All analyses were in duplicate.

Dry milk (100 g) and powdered ice (10 g) were mixed vigorously with a spoon for 10 to 15 min. The uniform mixture was then transferred to the column of an extractor (Fig. 1), 400 ml methylene chloride added to the column, and the extraction begun. During extraction the system was open to the atmosphere by way of a solvent trap cooled with dry ice in alcohol. The solvent was distilled by heating in a water bath held at 55 to 60°C . The filtration rate at 350 ml/hr was regulated by a Teflon plug stopcock to maintain the

solvent in the extraction tube at a level above that of the powder. At completion of extraction the Teflon plug stopcock was closed and the solvent trap connected to the house vacuum by a line containing a nearly closed needle valve. The solvent was removed from the fat until it no longer dripped from the bottom of the condenser—the fat being maintained at 40°C or less by adjusting the needle valve.

Results and Discussion

The effect of extraction time on recovery of fat is in Table 1. We considered the 4-hr extraction satisfactory and adopted this period in further work. Table 2 shows reproducibility of the method.

By maintaining a head of solvent above the powder, channeling is avoided and the powder is bathed at all times in a maximum volume of continuously supplied fresh solvent. This should be more efficient than the intermittent

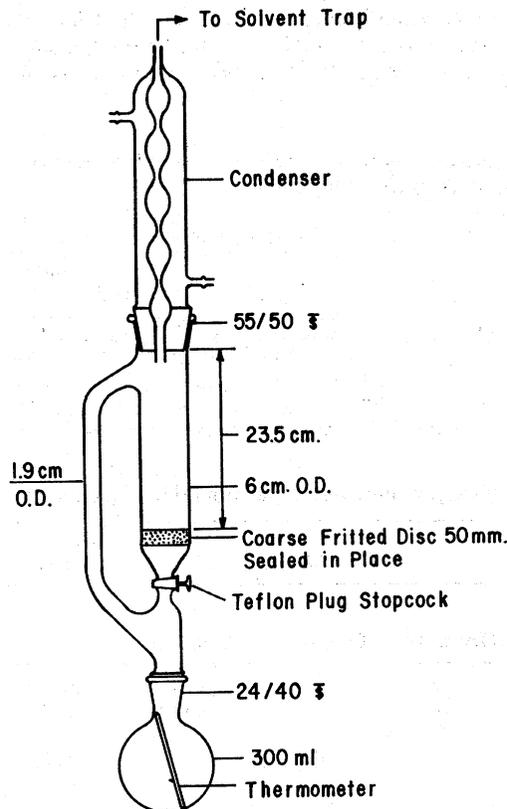


FIG. 1. Extraction apparatus.

operation of a Soxhlet extractor and, of course, requires less solvent than in a repeated mixture-filtration sequence.

Alcohol was avoided as a solvent since it interferes with both organoleptic evaluations and gas chromatographic analyses. We believe that the method described by Whitney and Tracy (7) for removing residual alcohol would entail large losses of flavor compounds. This difficulty and the hazards of purifying large quantities of ether are avoided with methylene chloride.

Methylene chloride, as do other relatively nonpolar solvents, requires a preliminary crystallization of lactose to extract effectively the fat from dried milk. If water is added to a slurry of dry milk in methylene chloride the recovery of fat is unsatisfactory — presumably because of the poor miscibility of the two liquids. Nor can water and dry milk be uniformly and rapidly mixed by adding water directly to the powder. By using ice cooled to -30°C , a satisfactorily uniform mixture with the powder can be obtained before melting.

The extracted milk fat is suitable for flavor evaluations after steam deodorization but not before. Steam distillates can be used for gas chromatographic investigations either before or after separation into flavor compound classes (4, 6). As these procedures employ methylene

chloride for extracting the aqueous distillate, no complication has been introduced by using the same solvent for extracting the fat sample. We found that 20 μ liters of methylene chloride from concentrating 400 ml of the purified solvent (6), is not detectable organoleptically in 1 liter of milk, so extracts of the steam distillates can also be used for flavor evaluations.

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TABLE 1. Effect of extraction time on recovery of fat from dry whole milk.

	Extraction period (hours)				
	0-1	1-2	2-3	3-4	Combined 0-4
Dry milk	Percent of total fat extracted				
A	81.9	11.5	3.0	1.5	97.9
B	83.9	11.6	2.4	0.5	98.4
C	80.9	11.6	2.8	0.9	96.2

TABLE 2. Recovery of fat from dry whole milk.*

Dry milk	Moisture (%)		Fat content	Percent of total fat extracted				Average
	Original	Adjusted	%	Replicate				
				1	2	3	4	
A	2.0	11.2-11.8	26.7	96.7	98.5	99.3	97.4	98.0
B	1.9	11.4-11.9	26.7	95.9	95.5	96.8	97.8	96.5
C	1.6	11.3-11.6	24.8	95.8	98.9	97.3	95.8	97.0
D	2.8	11.0-11.4	24.5	97.8	96.9	97.1	..	97.3
E	2.8	11.2-11.6	26.3	96.5	98.2	97.1	96.8	97.2

* 4-Hour extraction.