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DETERMINATION OF FAT CONTENT IN GHEE, HYDROGENATED OILS, BUTTER AND POWDERED MILK BY IR SPECTRA

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Abstract. A quick method, using IR spectrophotometry has been investigated for the analysis of (%) fat content in ghee, hydrogenated oils (substituted-ghee), butter and powdered milk. To avoid long procedure of chemical analysis for the determination of fat, this method uses simple preparation of solutions in carbon tetrachloride, under controlled physical conditions, which can be directly used for IR studies. The quantitative estimation has been made by using the 1745 cm^{-1} absorption frequency due to the carbonyl group of glycerides. Correlation of the results obtained by this method as compared with the chemical analysis shows an average variation of $\pm 1\%$ for total fat-content.

In conventional methods[1] for determining the fat-contents of animal and plant materials samples are extracted with suitable solvents and the extracted lipid weighed after removing the solvents. These methods are time-consuming and may not completely extract polar lipids and, therefore, are unsuitable for routine use in quality control laboratories. Refractometric methods[2,3] for determining the fat-content of foodstuffs are also unsuitable because of the interference from nonlipid components and extremely sensitive to fluctuations in temperature.

IR method of analysis offers a means of overcoming these disadvantages. D'yachenko and Samsonov[4] have carried out the analysis of fat by IR using KBr pellet technique. Keisuky and Saito[5] have suggested a thin-film technique using AgCl plate for the analysis of fat in milk which involves a long procedure of sample preparation. The present method uses simple and quick preparation of milk solution in carbon tetrachloride under controlled physical conditions. IR spectra of solution overcome the drawbacks of KBr pellet. Also difficulties may arise for the sample particles to be uniformly distributed in the matrix, which is very important for precise quantitative analysis.

Experimental

Preparation of Standard Fat and Powdered Milk Solutions. Pure butter fat (0.1 g) dried at 100°C was dissolved in 25 ml CCl_4 to provide a standard stock solution A. Two-, four- and eight-fold dilutions of solution A were used as working standards.

Powdered milk (2.8 g, 26% fat) was dissolved in 25 ml CCl_4 containing 2 drops of dil HCl, by warming to 50°C and stirring for 25 min. To remove the moisture, the mixture was filtered into a 25–ml volumetric flask through layer of sodium sulphate and the flask was filled up to the mark with CCl_4. Ten, twenty- and forty-fold dilutions of the above solution were used as working standards, corresponding to 26, 13 and 6.5% fat in powdered milk.

Preparation of Sample Solutions. Sample of ghee (0.1 g), substituted-ghee or butter was dissolved in 25 ml CCl_4 and filtered through a layer of sodium sulphate into a 25-ml volumetric flask after which it was filled up to the mark by CCl_4. The absence of —OH absorption in the region 3635-3610 cm^{-1} confirmed the absence of moisture in the solution.

For the analysis of powdered milk, 2.8 g sample was treated as described for standard powdered milk solution.

Spectra. The spectra were recorded on a Perkin–Elmer 237 spectrophotometer which had a wavelength range from 4000 to 635 cm^{-1} divided into two ranges by different gratings. The ordinate was 0–100% in linear transmittance with an accuracy of $\pm 1\%$ and the reproducibility for identical sampling conditions was within $\pm 1\%$ in the first range used in this work.

Matched cells of 1 mm thickness with NaCl windows were used. Purity of CCl_4 used was checked by IR spectra after redistillation.

Quantitative estimation was made by using baseline measuring technique for absorbance values of the standards and the samples.

Results and Discussion

The standard fat solutions described in the experimental section have been used for drawing a calibration graph against their corresponding ab-

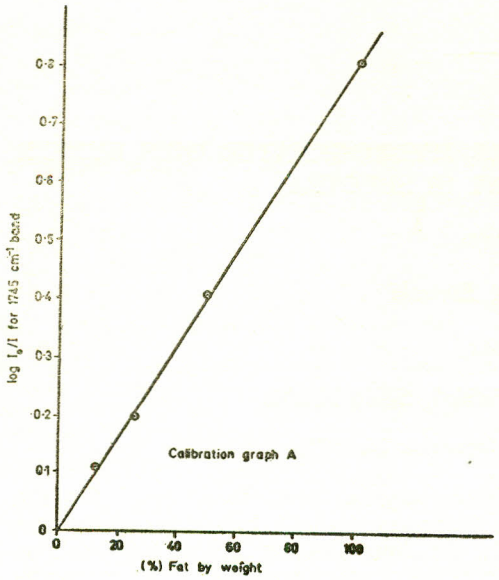

Fig. 1. Absorbance against known percentage of butter fat in CCl_4.

TABLE 1. ANALYSIS OF THE SAMPLES OF GHEE AND BUTTER.

Origin of the sample	log Io/I for 1745 cm^{-1}	Fat (%) Determined by present method	Fat (%) Determined by chemical method
Ghee (Holland)	0·81	100	100
Hydrogenated oil (Pakistan)	0·66	84	85
Butter (Denmark)	0·63	79	80
Butter (Australia)	0·58	72	72
Butter (Pakistan)	0·63	79	78

sorbance (log Io/I) for the C—o stretching frequency at 1745 cm^{-1} due to the carbonyl group of glycerides. In order to use this method for routine testing only 0.1 g fat sample is dissolved in CCl_4 and recording the absorbance at 1745 cm^{-1} in 1-mm cell, the fat percentage is noted directly from the calibration graph (Fig. 1).

Table 1 shows the analysis of different type of fat samples by the present method alongwith their analysis by chemical method. The average variation between the two methods is not more than $\pm 1\%$ for total fat content.

Estimation of (%) Fat in Powdered Milk. The determination of fat in powdered milk required suitable conditions for preparation of solution in CCl_4. Several attempts were made and finally the method described in the Experimental section was adopted. The calibration graph B (Fig. 3) was plotted from the concentration against absorbance (log Io/I) values of 1745 cm^{-1} (Fig. 2 shows the spectra from which absorbance values have been calculated).

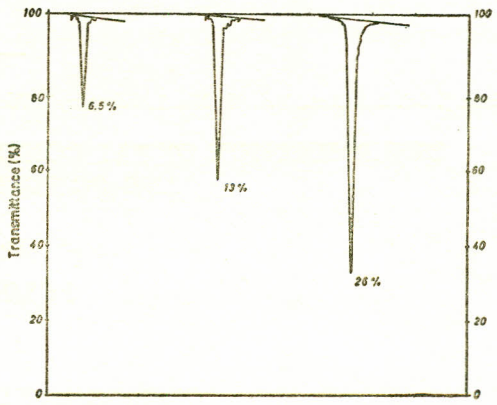

Fig. 2. Transmittance for 1745 cm^{-1} frequency of known percentage of fat in dried milk.

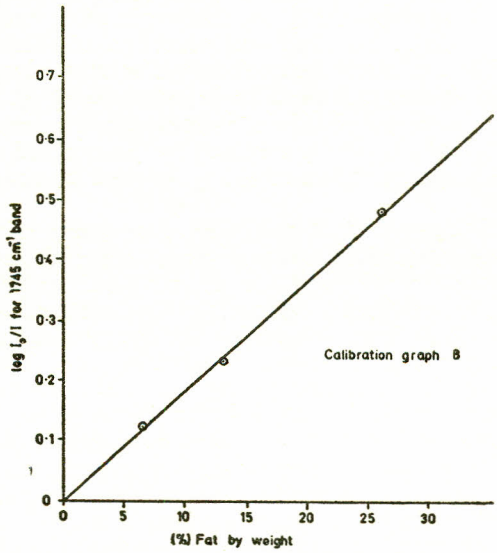

Fig. 3. Absorbance against known percentage of fat of dried milk in CCl_4.

TABLE 2. ANALYSIS OF THE SAMPLES OF POWDERED MILK.

Origin of the samples	log Io/I for 1745 cm^{-1}	Fat (%) Determined by present method	Fat (%) Quoted on the label
Cow & Gate (Red Cow (England)	0·55	30	28
Cow & Gate (Pakistan)	0·45	25	26
Comila (Belgium)	0·51	27	26
Dano (Denmark)	0·34	18	18
Bebelac (Holland)	0·29	16	18
Arinco, (Denmark)	0·47	26	26
German Export (Germany)	0·51	27	26 (min)
Similac (Holland)	0·45	25	26

Eight samples of different brands of milk have been tested by this method and results are shown in Table 2 alongwith the percentage of fat given on the label of the containers. Three samples have also been tested by chemical method and show a variation of $\pm 1\%$ for total fat, as compared by this method.

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