

Rapid Quantitative Analysis of Trans-fatty acid of extremely low concentration by Using the Cell for Liquid

Introduction

As a method of rapid quantitative analysis of trans-fatty acid in the food, which is becoming a big concern due to the influence to human health, the measurement by FTIR together with thermostatted ATR accessory has been suggested^{1,2)}. This is specified as an official method by AOAC (American Organization of Analytical Chemists) and AOCS (American Oil Chemists' Society) due to its extremely short analysis time within 1–2 minutes, which is much shorter than the time required by a method using GC (Gas Chromatography) that requires lousy sample preparation as well. However in official method of AOCS, the quantitation limit by ATR method is described as around 1.0%²⁾, which was also demonstrated in our FT/IR Application data¹⁾ under the same experiment conditions specified by AOCS.

Meanwhile, according to “The Guideline for Disclosure of Information on Trans-fatty acid Content” issued by the Consumer Affairs Agency of Japan, it is allowed to show “Zero” in the labeling if the content of trans-fatty acid / 100 g of food (100 ml in case of beverages) is less than 0.3%. In other words, 0.3% is required as the quantitation limit. Furthermore, the similar requirement in other countries is also reported (USA: < 0.5 g / meal, Taiwan: < 0.3 g / 100 g, Korea or South American: < 0.2 g / meal) and accordingly, a rapid and precise method is required for quantitative analysis of trans-fatty acid content in food in less than 1.0%. The method for quantitation of trans-fatty acid described in the Guideline is GC (AOCS Ce1h-05 or AOAC996.06), or other methods which need to have the equivalent performance of this method. The analysis by GC method not only requires the sample preparation before measurement, including the separation after having methylated the fat (being extracted) with BF₃, but also takes more than 1 hour for measurement. This report, describing the quantitative analysis of trans-fatty acid of lower than 1.0% by FTIR transmission method, shows the possibility that the equivalent result by GC method can be obtained in an extremely short time.

Experimental

The peak indicating the absorption at 966 cm⁻¹ by the trans-fatty acid is used for the quantitative analysis (Fig. 1). This is also adopted in official method such as ATR method²⁾.

Calibration curve is created by 5 samples with different content of trielaidin, which are prepared by adding the trielaidin (green color spectrum in Fig. 1), being known as an isomer of trans-type of triolein, into the cis-triolein (orange color spectrum in Fig. 1) which contains only cis-type.

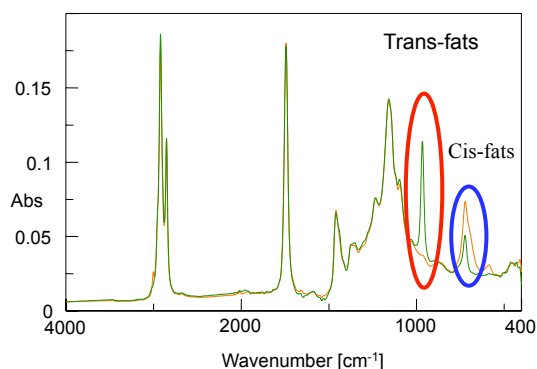


Fig. 1. IR spectrum of cis-fats and trans-fats(ATR)

1) JASCO FT/IR Application data 050-AT-0215 (2010)

2) AOAC Official Method 2000.10, AOCS Official Method Cd 14d-99

Measurement Conditions

Instrument: FT/IR-4100
 Detector: DLATGS Resolution: 4 cm⁻¹
 Accumulation: 64 times Apodization: Triangle
 Temperature: 25°C (ambient temperature)
 Mode: Transmission (method of solution)
 Cell: Sealed liquid cell *NaCl (thickness: 0.1 mm)
 Standard sample: Triolein, Trielaidin (0.05, 0.1, 0.2, 0.5, 1.0%)
 Peak calculation: Area within 945 - 990 cm⁻¹

* The sealed liquid cell with KBr can be also used. Please contact local JASCO distributor if the cell needs to be thermostatted.

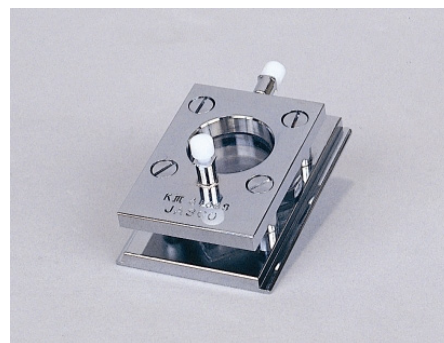
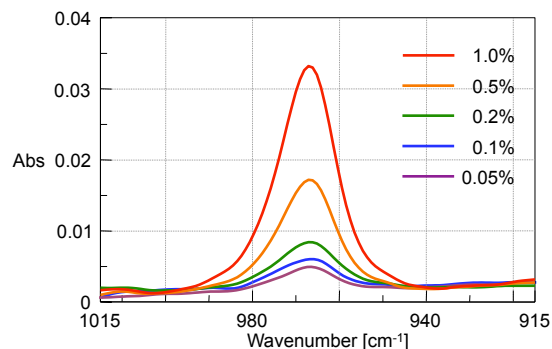


Fig. 2. Sealed liquid cell

Result and Discussion

Transmission spectra of 5 standard samples (with different concentration of trielaidin: 0.05, 0.1, 0.2, 0.5, 1.0%) are displayed in Fig.3. The correlation coefficient of 0.9998 of the calibration curve obtained shows the good linearity of trans-fatty acid concentration to the peak height or area around 966 cm⁻¹ (Fig. 4). Since the S/N (Peak-to-Peak) of the peak used for 0.05% quantitative calculation is about 15:1, which is much better than the value of 10:1 being normally accepted as the quantitation limit (Fig. 5), this method is considered as the accurate approach to the quantitative analysis of the sample whose concentration is extremely low, such as 0.05%.


 Fig. 3. Trans-fats peak at 966 cm⁻¹

The above result indicates that the quantitative analysis of 0.05 - 1.0% trans-fatty acid can be performed by the method of FTIR transmission measurement. Although this method has the advantages of no need of sample preparation and fast measurement comparing with the GC method, the cell has to be washed after the measurement each time. From such viewpoint, it is not so convenient as compared with the ATR method. Accordingly, it is considered to be the fast, convenient and accurate method to perform the screening with the ATR method first, and then apply FTIR transmission method only for the sample with concentration of lower than 1.0%.

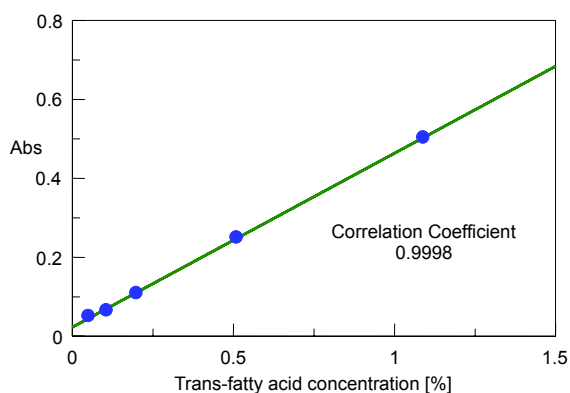


Fig. 4. Calibration curve

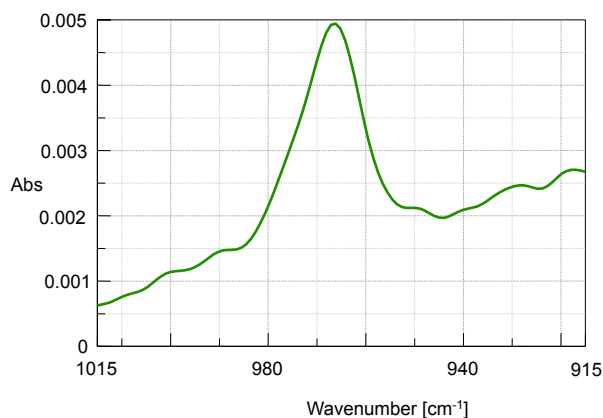


Fig. 5. Spectrum of trans-fats of 0.05%