SHIMADZU

Application News

Gas Chromatography Mass Spectrometry No.M265

Analysis of 2-Alkylcyclobutanones by GC/MS - Detection of Irradiated Foods -

■ Introduction

Food irradiation is a technology in which foods that are to be stored for long periods of time are exposed to X-rays or gamma rays to kill any microorganisms adhering to the surface of the food. This technology has been used worldwide for such foods as spices and meats, but in Japan, except for the purpose of preventing the germination of potatoes, its use has not been approved.

2-alkylcyclobutanones are formed in the fats contained in food as a result of irradiation. The alkyl groups formed in the 2-alkylcyclobutanones differ depending on the fatty acid composition in the fat; 2-dodecylcyclobutanone (2-DCB) derived from palmitic acid and 2-tetradecylcyclobutanone (2-TCB) derived from stearic acid are known to be principle products generated due to irradiation. Since these are not detected in non-irradiated foods, they are used as radiation detection indicators in the European Standards (EN1785) and in one of the detection methods for irradiated food established by the Japan's Ministry of Health, Labour and Welfare (Alkylcyclobutanone Method; Notice No. 0330 Article 3 of the Pharmaceutical and Food Safety Bureau, Ministry of Health, Labour and Welfare, March 30, 2010).

In this Application News, we introduce an example of analysis of 2-alkylcyclobutanone in foods from which fat can be extracted (beef, pork, chicken, salmon, and Camembert cheese, etc.) according to the Alkylcyclobutanone Method.

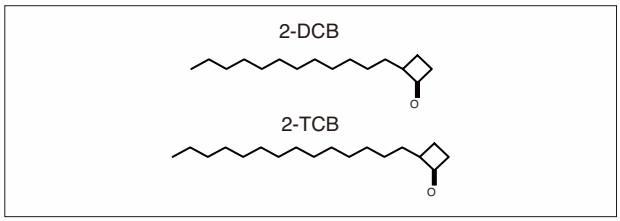

Fig. 1 Structures of 2-DCB and 2-TCB

■ Materials and Method

Using commercially-available ground pork as samples, one sample was irradiated with 1 kGy of cobalt-60 as the γ-ray source under room temperature, and the other was not irradiated. The sample preparation procedure is shown in Fig. 2.

SIM measurement is required for quantitation in this GC/MS test method, and SCAN measurement is required for qualitative identification. Here, the measurement was conducted in a single analysis using the FASST SCAN/SIM mode, and quantitation of 2-DCB and 2-TCB in the sample was conducted from the SIM measurement results using the internal standard method. The GC/MS analytical conditions are shown in Table 1.

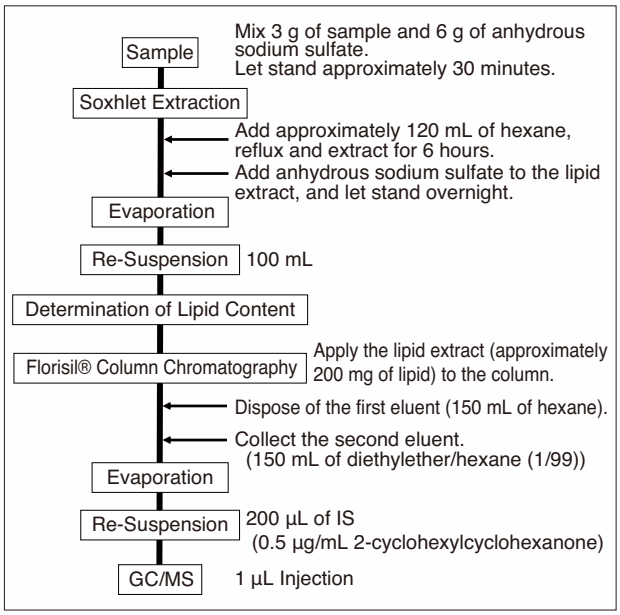

Fig. 2 Sample Preparation

Table 1 Analytical Conditions

Model	: GCMS-QP2010 Ultra		
–GC–		–MS–	
Column	: Rxi-5MS (30 m × 0.25 mm I.D.df=0.25 µm)	I.F. Temp.	: 280 °C
Col.Temp.	: 60 °C (1 min) -10 °C/min-300 °C (10 min)	I.S. Temp.	: 230 °C
Carrier Gas	: He (46.3 cm/sec, 100 kPa)	Ionization	: EI
Carrier Gas Mode	: Constant Linear Velocity Mode	Mode	: SCAN/SIM
Injection Temp.	: 250 °C	Scan Range	: *m/z* 95-115
Injection Method	: Splitless Injection (1 min)	Scan Interval	: 0.3 sec
Injection Volume	: 1 µL	Monitor Ion	: *m/z* 98, 112
		SIM Interval	: 0.2 sec

■ Sample Analysis

The SIM chromatograms of the irradiated and non-irradiated samples are shown in Fig. 3 and 4, respectively, and the mass spectra of the target analytes in the irradiated samples are shown in Fig. 5. In the irradiated samples, both 2-DCB and 2-TCB peaks were detected, and their concentrations in fat were 0.077 µg/g and 0.151 µg/g, respectively. The peaks were not detected in the non-irradiated samples.

According to the Alkylcyclobutanone Method, if the following 4 conditions are satisfied, the test result is considered to be positive for irradiation of food products.

1) Peaks with S/N ratio greater than 3 are recognized at *m/z* 98 and *m/z* 112 at the same retention times as in the standard solution.
2) The relative ion intensity ratio of *m/z* 112 with respect to *m/z* 98 is within ±20 % of the relative intensity ratio of a standard solution with a concentration level near that concentration.
3) When SCAN measurement is conducted in the vicinity of the retention time within the range of *m/z* 95 to *m/z* 115, *m/z* 98 and *m/z* 112 are the main ions (total intensity of the 2 ions is at least 50 %).
4) Even if the above items are satisfied, the quantitative values must be greater than the concentration calculated using S/N ratio=3 of the standard solution.

The irradiated food measurement results obtained here satisfy all of the above criteria, and therefore the results were “positive” for irradiation.

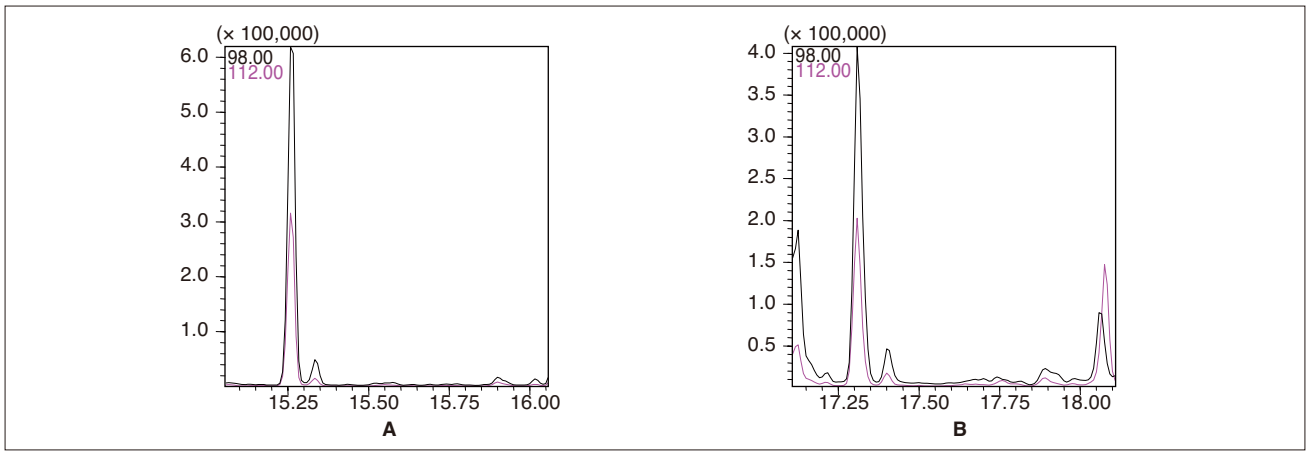

Fig. 3 SIM Chromatogram of Non-Irradiated Sample

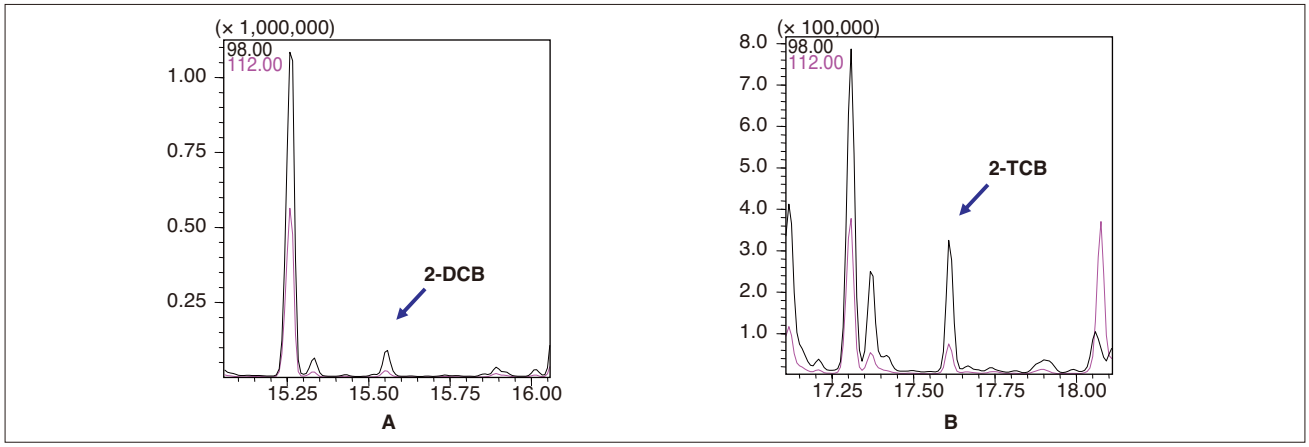

Fig. 4 SIM Chromatogram of Irradiated Sample

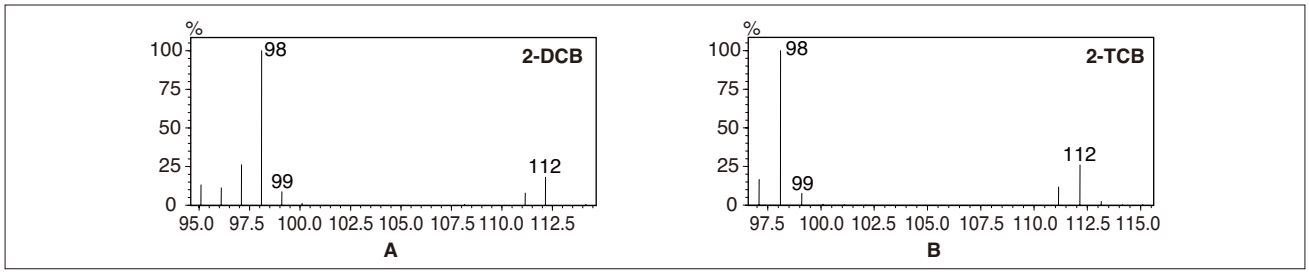

Fig. 5 Mass Spectra of 2-DCB and 2-TCB (Irradiated Sample)

[References]
Notice No. 0330 Article 3 of the Pharmaceutical and Food Safety Bureau, Ministry of Health, Labour and Welfare of Japan, March 30, 2010 “Detection Method for Irradiated Foods”

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