SHIMADZU
Excellence in Science

GC-MS AOC™-6000 Plus / GCMS-TQ™8040 NX

Application News

Quantitative Analysis of Furan and Alkylfuran in Processed Food Using a Triple Quadrupole GC-MS System and an SPME Arrow

Yuto Nakasuji and Yu Nagao

User Benefits

- Using an SPME Arrow allows for highly sensitive analysis of furan and alkylfuran in processed food.
- The MRM mode enables highly reliable analysis even in complex matrices.
- An AOC-6000 Plus autosampler helps increase productivity and repeatability through automated pretreatment.

■ Introduction

Furan and alkylfuran compounds are commonly found in many foods, including coffee. They are known as volatile organic compounds that contribute to aroma. However, recent studies in various countries have reported health concerns regarding these compounds and monitoring of furan and alkylfuran has been recommended in the EU[1)].

These compounds are frequently measured using the HS-GC/MS method, which is convenient and effective when present at relatively high concentrations. However, for samples containing low concentrations, pretreatment for concentration is necessary. This Application News article describes a method using an SPME Arrow and GC-MS/MS system that enables more consistent analysis even at trace concentrations in complex matrix samples.

Fig. 1 AOC™-6000 Plus + GCMS-TQ™8040 NX

■ Instrument Configuration and Analytical Conditions

The analysis was performed using an AOC-6000 Plus multifunctional autosampler and a GCMS-TQ8040 NX triple quadrupole gas chromatograph mass spectrometer system (Fig. 1). The AOC-6000 Plus is a multifunctional autosampler that can automatically switch between various sample injection methods, such as liquid injection, headspace injection, and solid-phase microextraction (SPME), depending on the sample and purpose (Fig. 2). In this analysis, an SPME Arrow, which offers improved durability compared to a traditional SPME fiber, was used for sample analysis.

When using sampling methods involving concentration, such as the SPME method, matrix-derived impurities may also be concentrated, making it difficult to improve sensitivity due to interference from impurities. In this analysis, high sensitivity for analyzing furan in processed foods was achieved by using the SPME method in combination with the MRM mode of the triple quadrupole GC-MS system.

Detailed analytical conditions are indicated in Table 1.

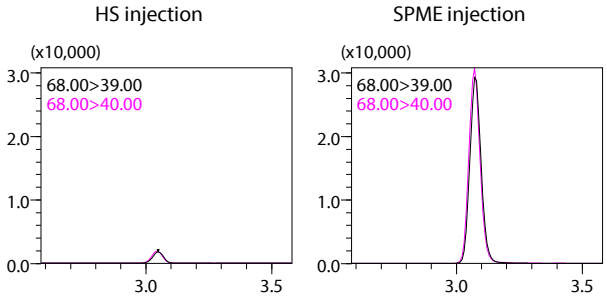

Fig. 2 Chromatograms of Furan (1 ng/mL) by AOC-6000 Plus HS and SPME Modes

Table 1 Analytical Conditions

AOC-6000 Plus		GC	
SPME Arrow:	Carbon WR/PDMS, 120 µm	Injection Mode:	Split
	(P/N : 227-35331-01)	Split Ratio:	20
Conditioning Temperature:	280 °C	Carrier Gas:	He
Pre-Conditioning Time:	5 min	Carrier Gas Control:	Constant linear velocity (30.0 cm/s)
Incubation Temperature:	50 °C	Septum Purging:	3.0 mL/min
Incubation Time:	10 min	Column:	SH-I-5 MS (P/N 227-36022-02)
Agitator Speed:	250 rpm		(30 m × 0.25 mm I.D., 1.00 µm)
Stirrer Speed:	250 rpm	Column Temp.:	40 °C (2 min) - 3 °C/min - 60 °C (0 min)
Sample Extraction Time:	20 min		- 20 °C/min - 220 °C (5 min)
Sample Desorption Time:	1 min		
Post-Conditioning Time:	5 min	MS	
		Ion Source Temp.:	200 °C
		Interface Temp.:	250 °C
		Acquisition Mode:	MRM
		Loop Time:	0.5 sec
		Tuning Mode:	Normal

■ Sample Preparation

In this analysis, the six components listed in Table 2 were analyzed. Initially, a standard solution was prepared by diluting each component to 0.05 - 50 µg/mL in methanol. Next, 4.5 g of sodium chloride and 10 mL of ultrapure water were weighed into 20 mL screw vials and then 2 µL of each concentration of standard solution was added to prepare aqueous solutions of concentrations 0.01 to 10 ng/mL, which were used as standards for analysis. To prevent volatilization of furan compounds during preparation, an Ice-Free VOC Preparation Kit from GL Sciences was used when preparing standard solutions.

Commercial baby food was used as the unknown sample. 1 g of a homogenized sample was weighed into a 20 mL screw vial containing 4.5 g of sodium chloride, 9 mL of ultrapure water was added immediately, the vial was sealed, and the mixture was well mixed with a vortex mixer to prepare the sample for analysis.

Table 2 List of Target Compounds and Monitor Ions

Compounds	Ion 1 *m/z*	Ion 2 *m/z*
Furan	68.00 > 39.00	68.00 > 40.00
2-Methylfuran	82.00 > 54.00	81.00 > 53.00
3-Methylfuran	82.00 > 54.10	81.00 > 53.00
2-Ethylfuran	96.00 > 81.00	96.00 > 53.00
2,5-Dimethylfuran	96.00 > 81.00	96.00 > 53.10
2,3-Dimethyl furan	96.00 > 81.10	96.00 > 67.00

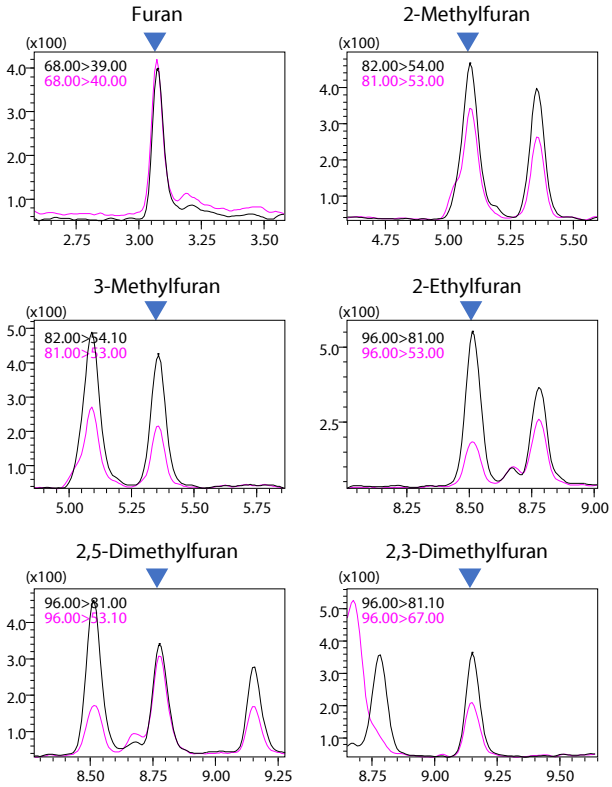

Fig. 3 MRM Chromatograms at minimum concentrations of Calibration Curve (0.01 ng/mL)

■ Analysis Results of Standards

Fig. 3 shows the MRM chromatogram of each component at the lowest concentration of 0.01 ng/mL. Sufficient sensitivity was obtained and the results of six repeated analyses at a concentration of 0.01 ng/mL showed that the area %RSD for all components was below 10 %, indicating adequate repeatability even at very low concentrations.

Fig. 4 shows the calibration curves for each component. As the range of quantification was wide in this analysis, the calibration curves were weighted by 1/C for quantification calculations. All components showed sufficient linearity (R > 0.996).

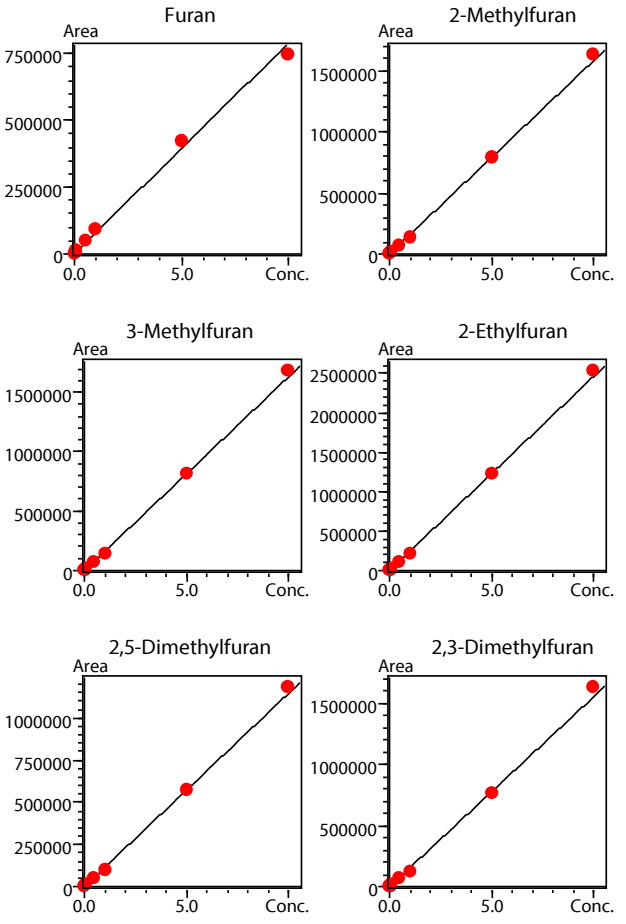

Fig. 4 Calibration Curves for Respective Targets (0.01 – 10 ng/mL)

■ Analysis Results of Unknown Samples

Unknown samples were analyzed using both the external standard method and the standard addition method to evaluate the effects of the matrix. The quantitative results of both methods are shown in Table 3 as weight-based concentrations in the samples.

Comparing the quantitative results of both methods indicates that the results were generally consistent, but differences were observed for some compounds due to the influence of matrix differences between the standard and unknown samples. This suggests that further dilution or correction using deuterium-labeled internal standards is necessary to suppress matrix effects.

Table 3 Quantitative Results of Furans in Baby food

	External Standard (µg/kg)	Standard Addition (µg/kg)
Furan	7.9	9.1
2-Methylfuran	0.37	0.37
3-Methylfuran	0.14	0.11
2-Ethylfuran	0.31	0.43
2,5-Dimethylfuran	< Lim.	< Lim.
2,3-Dimethylfuran	N.D.	N.D.

Note: "N.D" means not detected and "< Lim." means out of calibration range

■ Comparison of SIM and MRM

To confirm the improvement in selectivity with MRM, analysis using the SIM mode was also conducted. Generally, the MRM mode of a triple quadrupole GC-MS system offers better selectivity compared to the SIM mode, leading to higher sensitivity and more consistent detection.

Fig. 5 shows the SIM and MRM chromatograms of four components detected in baby food. While each component was detectable with both modes, the MRM mode provided higher selectivity, whereas the SIM mode was more susceptible to interference from impurities. Therefore, manual peak integration or changes to the quantitative ions may be required in the SIM mode depending on the sample.

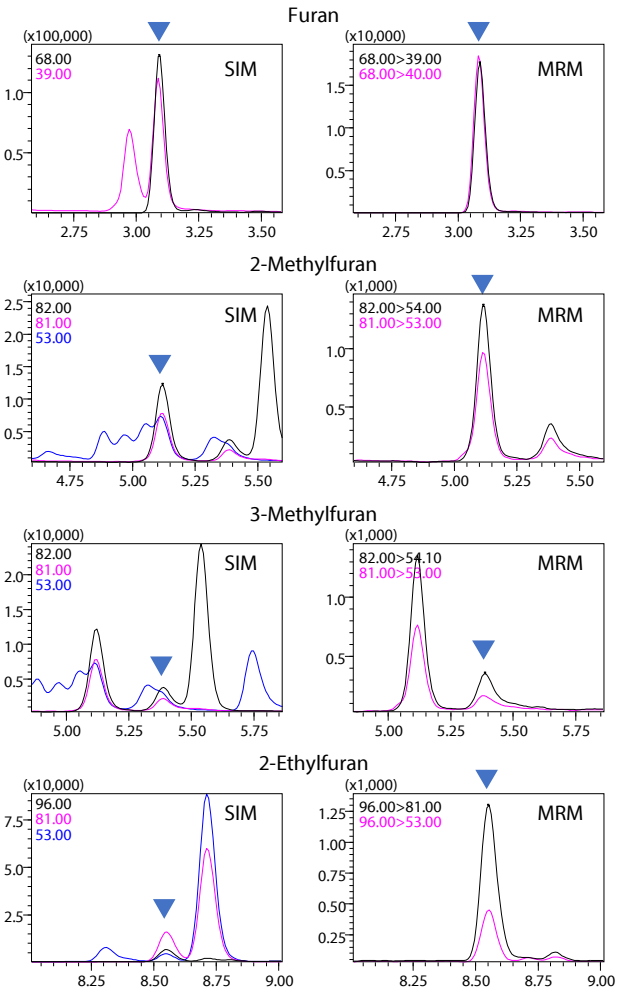

Fig. 5 Comparison of SIM and MRM Chromatograms of Furans in Babyfood

■ Conclusion

Quantitative analysis of furan and alkylfuran in processed food was conducted using an SPME Arrow and a triple quadrupole GC-MS system. The AOC-6000 Plus multifunctional autosampler allows for highly sensitive and reproducible quantification of furan compounds using the SPME Arrow method. Additionally, by enhancing selectivity with the MRM mode of the triple quadrupole GC-MS system, more reliable analytical results were obtained.

<References>

1) Commission Recommendation (EU) 2022/495 of 25 March 2022 on monitoring the presence of furan and alkylfurans in food
https://eur-lex.europa.eu/eli/reco/2022/495/oj/eng
(Accessed Feb. 4, 2025)

01-00876-EN First Edition: Mar. 2025

SHIMADZU
Shimadzu Corporation
www.shimadzu.com/an/

› Please fill out the survey

Related Products Some products may be updated to newer models.

› GCMS-TQ™8040 NX

Triple Quadrupole Gas Chromatograph Mass Spectrome···

› AOC-6000 Plus

AOC-6000 Plus Multifunctional Autosampler

Related Solutions

› Food and Beverages

› Food Dietary Restrictions

› Price Inquiry

› Product Inquiry

› Technical Service / Support Inquiry

› Other Inquiry