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GC-MS HS-20 NX/GCMS-QP2050

Application News

Quantitative Analysis of Furan and Alkylfurans in Processed Food Using Headspace GC-MS

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User Benefits

- The compact instrument design reduces the installation footprint.
- Maintenance requirements are reduced due to the DuraEase ion source, which is designed for durability and ease of maintenance.
- The HS-20 NX headspace sampler offers automated preparation of samples for improved productivity and repeatability.

■ Introduction

Furan and alkylfurans are aromatic compounds with a five-membered ring structure. Furan and some alkylfurans, such as 2-methylfuran, 3-methylfuran, and 2,5-methylfuran (furan with a hydrocarbon chain attached), are found in processed food. They are generated through a variety of pathways during heat treatment of the food. Some countries have studied these compounds and concluded they are a health concern.[1)]

At present, no country has set permissible levels for furan or alkylfurans in food. But since they are harmful substances that are generated unintentionally during the processing of food, analyzing them is becoming increasingly important. In this Application News, the levels of furan and alkylfurans in commercially available processed food were measured.

■ Equipment

The GCMS-QP2050 gas chromatograph mass spectrometer has a compact design for a smaller installation footprint. When combined with the HS-20 NX headspace sampler, the GCMS-QP2050 can simply and accurately measure furan and alkylfuran levels in processed food. The DuraEase ion source, which is specifically designed for durability and ease of maintenance, also provides reduced maintenance requirements.

Fig. 1 HS-20 NX + GCMS-QP2050

■ Preparation of Calibration Standards

The following six compounds were selected as measurement targets: furan, 2-methylfuran, 3-methylfuran, 2,5-dimethylfuran, 2,3-dimethylfuran (reagent grade, Tokyo Chemical Industry), and 2-ethylfuran (special grade, FUJIFILM Wako Pure Chemical). A methanol mixed standard solution was prepared by diluting the six compounds in methanol. Calibration standards were prepared by adding 4 g of NaCl (special grade, FUJIFILM Wako Pure Chemical), 10 mL of mineral water, and the methanol mixed standard solution to a 20-mL headspace vial and sealing the vial. Each compound was added to an equivalent concentration of 20, 50, 100, 500, 1000, 2000, 5000, 10000, 20000, and 50000 µg/kg.

■ Preparation of Unknown Samples

Test samples were prepared by adding 4 g of NaCl, 9 mL of mineral water, and 1 g of homogenized sample to a 20-mL headspace vial, sealing the vial, and thoroughly mixing the contents. Measurements were performed using both an external standard method and a standard addition method to verify the effect of the sample matrix on analysis. The spiked sample for the standard addition method was prepared as described above, but the methanol mixed standard solution was added before sealing the vial and mixing the contents. The amounts of each compound added for the standard addition method were 14000 and 35000 in coffee, 2000 and 5000 in baby food, and 20000 and 50000 in canned mackerel (units: µg/kg).

■ Headspace GC-MS Analysis

The amounts of each analyte in the calibration standards and unknown samples were measured by headspace GC-MS. The analysis conditions are shown in Tables 1 and 2. Given the concern that heat treatment increases levels of furan and alkylfurans in food, the food samples used in this study were held at 60 °C for 15 minutes, a heating protocol reported to have no effect on furan and alkylfuran levels.[2)]

Table 1 Analysis Conditions

Model	: GCMS-QP2050
Autosampler:	: HS-20 NX
HS-20 NX	
Oven Temp.	: 60 °C (15 min)
Sample Line Temp.	: 150 °C
Transfer Line Temp.	: 150 °C
Vial Stirring	: OFF
Vial Pressurization Time	: 1.0 min
Pressure Equilib. Time	: 0.1 min
Loading Time	: 0.5 min
Load Equilib. Time	: 0.1 min
Injection Time	: 1.0 min
Needle Flush Time	: 5.0 min
Vial Pressure	: 100 kPa (N_2)
Injection Volume	: 1.0 mL
GCMS-QP2050	
Injection Mode	: Split
Split Ratio	: 5
Carrier Gas	: He
Carrier Gas Control	: Constant Linear Velocity (30 cm/sec)
Column	: SH-I-5MS (P/N 227-36025-03) (60 m × 0.32 mm I.D., 1.0 µm)
Column Temp.	: 50 °C (3 min) – 10 °C/min –110 °C –200 °C– 20 °C/min –200 °C (5 min)
Ion Source Temp.	: 200 °C
Interface Temp.	: 250 °C
Mode	: SIM (See Table 2)
Event Time	: 0.3 s
TMP Evacuation Rate	: 255 L/sec

Table 2 SIM Settings

	m/z
Furan	68, 39
2-Methylfuran	82, 81, 53
3-Methylfuran	82, 81, 53
2-Ethylfuran	96, 81, 53
2,5-Dimethylfuran	96, 95, 53
2,3-Dimethylfuran	96, 95, 81

■ Results from Measuring Calibration Standards

Fig. 2 shows the results obtained from the calibration standards (prepared by adding the ethanol mixed standard solution to the vial) after being held at 60 °C for 15 minutes, headspace sampling, and GC-MS analysis. The results show good separation between the six organic solvents. The calibration curves created by analyzing standard samples under fixed conditions also show good linearity. Fig. 3 shows the calibration curve for furan as a representative example.

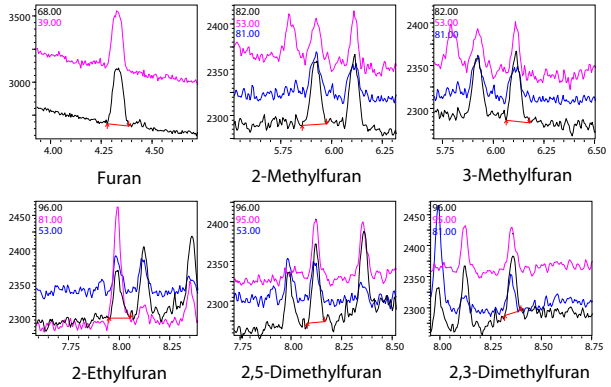
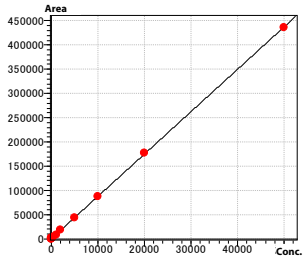


Fig. 2 SIM Chromatograms of Mixed Standard Solution (equivalent to 20 µg/kg)



Y = 8.73167X + 614.941
R^2 = 0.9998286
R = 0.9999143

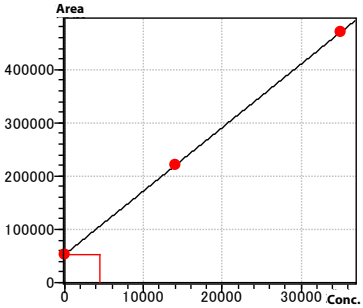
External standard
Calibration curve: Linear
Passes through origin: No
Weighting: 1/C

Fig. 3 Furan Calibration Curve
(20, 50, 100, 200, 500, 1000, 2000, 5000, 10000, 20000, 50000 µg/kg)

Verifying peak area repeatability using the lowest concentration on the calibration curve (20 µg/kg) gave %RSD results (n = 5) of 10 % or below for every compound (Table 3).

Table 3 Peak Area Repeatability (%RSD)

	m/z	1	2	3	4	5	Mean	RSD(%)
Furan	68	1,508	1,520	1,501	1,468	1,480	1,495	1.4
2-Methylfuran	82	233	204	213	254	229	227	8.5
3-Methylfuran	82	249	259	275	244	224	250	7.5
2-Ethylfuran	96	181	164	139	165	157	161	9.4
2,5-Dimethylfuran	96	234	214	217	244	238	229	5.8
2,3-Dimethylfuran	96	223	198	217	210	231	216	5.8



Y = 11.9006X + 53.025
R^2 = 0.9999880
R = 0.9999940

Measured level: 4500

Fig. 4 Analysis of Coffee A by Standard Addition Method:
3-Methylfuran Calibration Curve

■ Results from Food Sample Analysis

The results from analyzing six samples of three different foods (coffee, baby food, and canned mackerel) are shown in Tables 4–6. Quantitative analysis of unknown samples by the standard addition method was performed using two standard spiked samples with different spike concentrations. Fig. 4 shows the calibration curve of Coffee A for 3-methylfuran obtained by the standard addition method. The results obtained by the external standard method and the standard addition method were almost the same for coffee, but there was a large discrepancy with the baby food and canned mackerel. If a deuterium-labeled substance is not used as an internal standard (it was not used in this analysis), the standard addition method must be used to obtain an accurate quantitative result.

Table 4 Quantitative Analysis of Coffee (µg/kg)

Compound Name	Coffee A		Coffee B	
Quantitative Method	Standard addition	External standard	Standard addition	External standard
Furan	56000	54000	60000	64000
2-Methylfuran	140000	130000	100000	120000
3-Methylfuran	4500	4500	4400	4500
2-Ethylfuran	460	620	340	500
2,5-Dimethylfuran	3400	3500	2700	2800
2,3-Dimethylfuran	----	----	----	----

Table 5 Quantitative Analysis of Baby Food (µg/kg)

Compound Name	Baby food A		Baby food B	
Quantitative Method	Standard addition	External standard	Standard addition	External standard
Furan	10000	7800	71000	32000
2-Methylfuran	330	260	1500	1200
3-Methylfuran	160	140	910	750
2-Ethylfuran	1000	760	450	360
2,5-Dimethylfuran	----	----	350	270
2,3-Dimethylfuran	----	----	----	----

Table 6 Quantitative Analysis of Canned Mackerel (µg/kg)

Compound Name	Canned mackerel A		Canned mackerel B	
Quantitative Method	Standard addition	External standard	Standard addition	External standard
Furan	130000	99000	200000	120000
2-Methylfuran	19000	8400	41000	15000
3-Methylfuran	2600	920	4000	1400
2-Ethylfuran	88000	31000	650000	110000
2,5-Dimethylfuran	610	76	330	75
2,3-Dimethylfuran	570	38	180	44

■ Conclusion

In this Application News, headspace GC-MS analysis was used to measure levels of furan and alkylfurans in processed food. Analysis of standard samples produced good results in terms of separation, calibration curve linearity, and repeatability. Analyzing food samples and comparing the results obtained by the external standard method and the standard addition method showed that the matrix effects differed depending on the sample.

<References>
1) Food Safety Commission, Alkylfuran Fact Sheet:
https://www.fsc.go.jp/sonota/kikansi/25gou/25gou_3.pdf

2) Japan Food Research Laboratories, Motoki Ogiso et al.:
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01-00906-EN First Edition: Aug. 2025

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