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GCMS – Food – Food Safety

Determination Of 4 Banned Substances In Weight-Loss Food By GC-MS

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Abstract

Sibutramine and other banned substances in weight-loss foods pose significant health risks, making reliable detection methods essential to safeguard consumer health. In this Technology Brief, Shimadzu's single quadrupole GCMS-QP2020 NX was utilized to detect four banned substances, including sibutramine, in weight-loss foods. The extraction process involved methanol ultrasonic extraction and membrane filtration, followed by GC-MS analysis.

The method demonstrated excellent linearity, reproducibility, and recovery rates for all target components, with correlation coefficients exceeding 0.997 across a concentration range of 0.2 to 5.0 mg/L. Injecting a standard solution at 0.2 mg/L six times resulted in a relative standard deviation of peak areas below 4%. Recovery rates at a spiking level of 20 mg/kg ranged from 102% to 115%. Overall, this straightforward method provides a reliable reference for determining banned substances in weight-loss foods.

Ensuring food quality for consumer health

Keywords: Gas Chromatography-Mass Spectrometry, Weight-Loss Food, Banned Substances

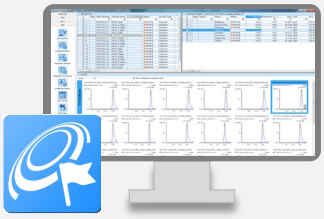
Highlights

- *Simultaneous analysis of 4 banned substances in weight-loss food with ease.*
- *Exceptional sensitivity with GCMS-QP2020 NX ensures reliable results even at low limit of detection.*
- *Simple and straightforward method enables robust recovery rates of 102% to 115%.*

Technologies Featured

GCMS-QP2020 NX

LabSolutions™ Insight



1. INTRODUCTION

Food adulteration, particularly in weight-loss products, presents serious health risks due to the inclusion of undeclared pharmaceutical ingredients. Marketed with claims of burning fat, promoting effective weight loss, and suppressing appetite, these products often contain banned substances like sibutramine, which significantly increase the risk of heart attack and stroke.

Generally, weight-loss components in foods fall into three categories: appetite suppressants, absorption inhibitors, and metabolism boosters. Among these, appetite suppressants are the most concerning. These include drugs like phenylpropanolamine, methamphetamine, and other catecholamine-like neurotransmitters, as well as serotonin-like drugs such as sibutramine and fenfluramine. Consuming weight-loss products containing these banned substances can lead to severe side effects, including insomnia, irritability, fatigue, gastrointestinal disorders, abnormal blood pressure, and, in extreme cases, liver and kidney diseases or even cancer.

To detect banned substances in weight-loss products, various testing methods are employed, including spectroscopy, capillary electrophoresis, and quantitative techniques like gas chromatography-mass spectrometry (GC-MS). Among these, GC-MS stands out for its accurate qualitative and quantitative analysis, offering enhanced selectivity against interference in complex matrices.

This Technology Brief introduces an analytical method using Shimadzu's single quadrupole GCMS-QP2020 NX to detect four banned substances in weight-loss products. The method is straightforward, highly reproducible, and effective in determining the content of sibutramine and three other compounds in these products.

2. EXPERIMENT

2.1 Experimental Condition

System	: GCMS-QP2020 NX
Injection Mode	: Split injection, 5:1
Injection Volume	: 1 µL
Injection Port Temp	: 320°C
Carrier Gas Control Mode	: Constant linear velocity, 41.1 cm/sec
Column	: SH-I-5Sil MS, 30 m × 0.25 mm × 0.25 µm
Column Temp Program	: 100°C (1 min) → 15°C /min to 250°C → 25°C /min to 300°C (4 min)
Ionization Mode	: Electron Ionization (EI)
Ion Source Temperature	: 230°C
Interface Temperature	: 320°C
Acquisition Mode	: SIM (Selected Ion Monitoring)

2.2 Sample Pre-treatment

Take an appropriate amount of the sample and mix well (for solid samples, grind into a fine powder).

↓

Weigh 0.5 g of sample (accurate to 0.1 mg) into a 15 mL centrifuge tube.

↓

Add 10 mL of methanol and ultrasonicate for 10 minutes.

↓

Stand for 5 minutes
Filter the solution via syringe filter.

↓

Perform GC-MS analysis.

Fig. 1 Sample preparation workflow

3. RESULTS AND DISCUSSION

3.1 Chromatogram of Standard Solution

The chromatogram of the mixed standard solution containing four banned substances is shown in Figure 2, and the mass chromatogram is shown in Figure 3 (with concentrations of sibutramine, fenfluramine, and prasterone at 0.2 mg/L, and fluoxetine at 1.0 mg/L). The corresponding compound information is provided in Table 1.

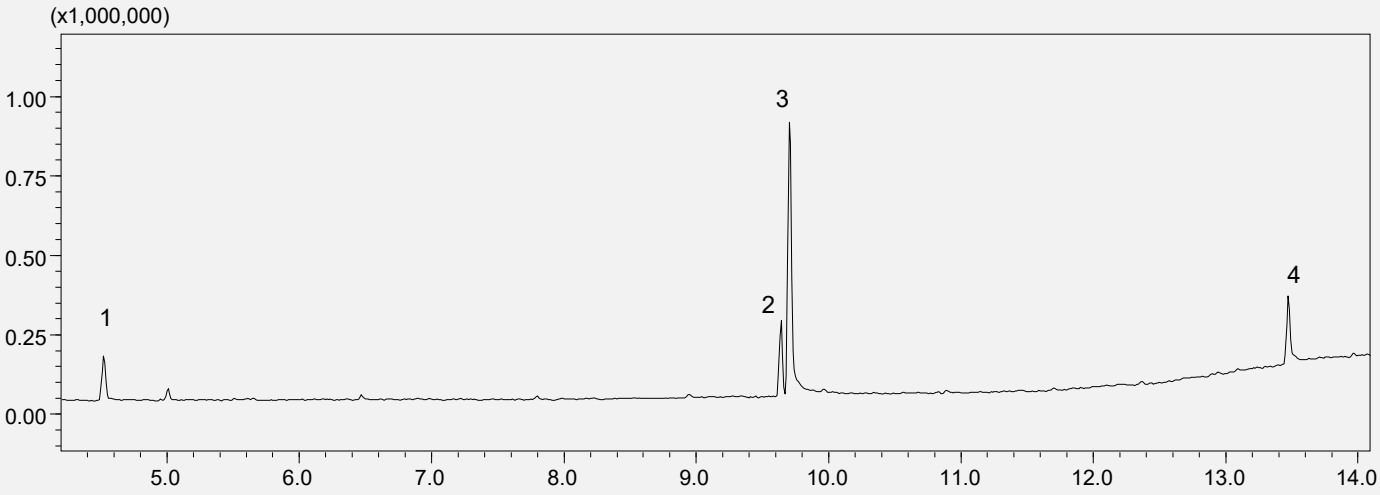


Fig. 2 Chromatogram of standard solutions of four banned substances

Table 1 Compound information on four banned substances

No.	Compound	CAS No.	Retention Time (min)	Quantitative Ion (*m/z*)	Qualitative Ion (*m/z*)
1	Fenfluramine	458-24-2	4.154	72	159, 216
2	Sibutramine	106650-56-0	9.631	114	72, 115
3	Fluoxetine	54910-89-3	9.706	104	148, 309
4	Prasterone	53-43-0	13.467	288	255, 270

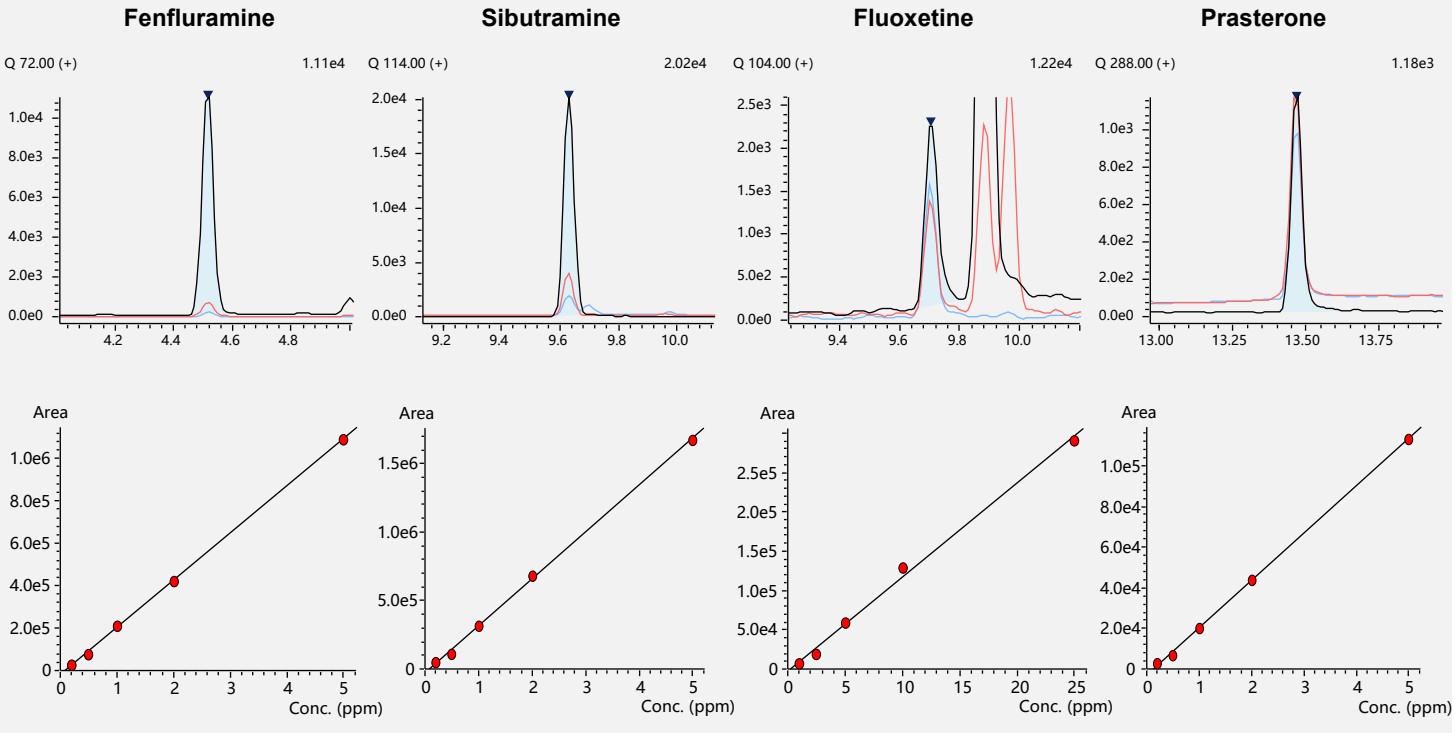


Fig. 3 Mass chromatogram and standard calibration curves for four banned substances

3.2 Standard Calibration Curves and Limit of Detection

Using methanol as the solvent, standard solutions were prepared at concentrations of 0.2, 0.5, 1.0, 2.0, and 5.0 mg/L (with fluoxetine at 1, 2.5, 5.0, 10, and 25 mg/L) to examine the correlation coefficients of the calibration curve regression equations for each compound. The limit of detections were calculated based on the spectrum of the 0.2 mg/L standard solution (1 mg/L for fluoxetine) using a signal-to-noise ratio of 3. The standard curves for each compound are shown in Figure 3, and the linear correlation coefficients and limit of detection are presented in Table 2.

Table 2 Correlation coefficient and limit of detection for four banned substances

No.	Compound	Correlation Coefficient (R)	Limit of Detection (mg/L)
1	Fenfluramine	0.9998	0.003
2	Sibutramine	0.9995	0.001
3	Fluoxetine	0.9970	0.094
4	Prasterone	0.9997	0.003

3.3 Reproducibility

A standard solution with a concentration of 0.2 mg/L (fluoxetine at 1.0 mg/L) was injected six times to assess the reproducibility of the instrument. The reproducibility results are shown in Table 3.

Table 3 Reproducibility results for four banned substances

No.	Compound	Peak Area						Peak Area RSD (%)
		1	2	3	4	5	6	
1	Fenfluramine	31096	30264	30253	29437	29479	30208	2.0
2	Sibutramine	49753	48391	48576	48310	48270	49622	1.4
3	Fluoxetine	6750	7103	7114	6961	7432	7318	3.4
4	Prasterone	2796	2818	2839	2734	2767	2907	2.1

3.4 Spiked Recovery Rate

Blank samples were taken for a spiking recovery experiment, following the pretreatment procedure outlined in Figure 1, with three parallel samples prepared. The recovery rate results are shown in Table 4.

Table 4 Recovery results for four banned substances

No.	Compound	Spiking Amount (mg/kg)	Measured Value of Spiked Sample (μg/kg)			Average Recovery Rate (%)
			1	2	3	
1	Fenfluramine	20	22.77	23.27	22.42	114.1
2	Sibutramine	20	23.14	22.68	22.05	113.1
3	Fluoxetine	100	102.47	104.91	100.62	102.7
4	Prasterone	20	22.85	22.95	22.02	113.0

3.5 Commercial Sample Testing

A commercial food sample was obtained and analyzed for the presence of the four banned substances. None of the banned substances were detected and the chromatogram is shown in Figure 4.

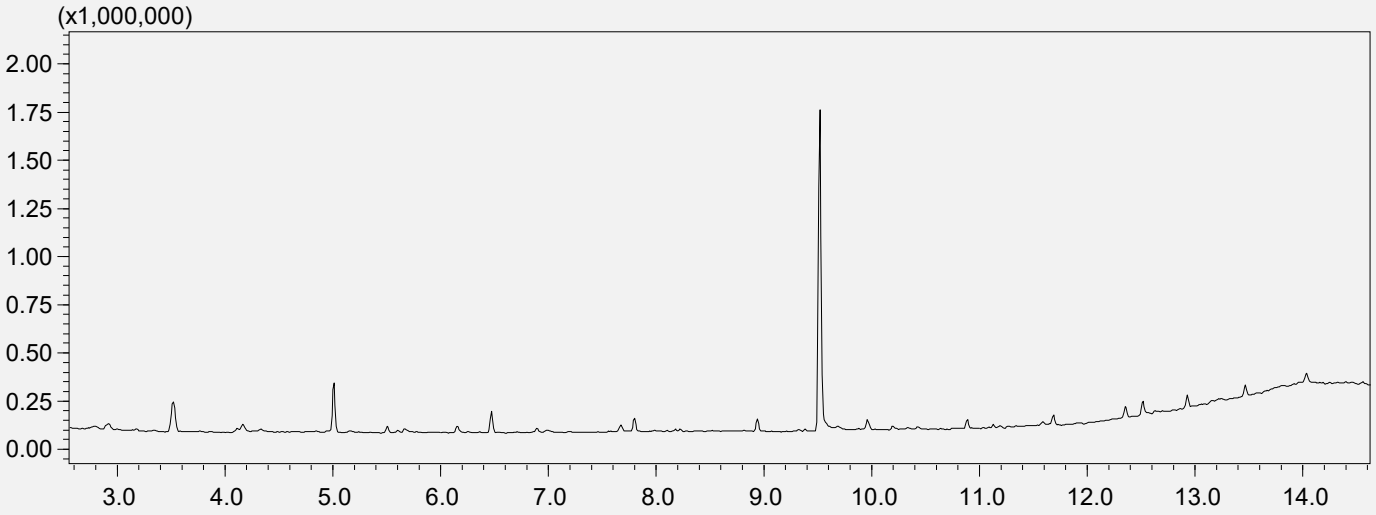


Fig. 4 Chromatogram of commercial sample

4. CONCLUSION

A detection method for four banned substances, including sibutramine, in weight-loss foods was established using Shimadzu GCMS-QP2020 NX. The method demonstrated excellent linearity, reproducibility, and recovery rates for all target components. Within the concentration range of 0.2 to 5.0 mg/L, the correlation coefficients were above 0.997. A standard solution at a concentration of 0.2 mg/L was injected six consecutive times, with relative standard deviation of the peak areas below 4%. At a spiking level of 20 mg/kg, the recovery rates ranged from 102% to 115%. This method is straightforward and can provide a reference for identifying banned substances in weight-loss foods.

READ MORE

GCMS-QP2020 NX

Shimadzu single quadrupole GCMS-QP2020 NX gas chromatograph-mass spectrometer (GC-MS) delivers exceptional performance, efficiency, and reliability, setting a new standard in its class. Excelling in both ease of use and robustness, the user-friendly instrument ensures accurate and ultra-fast analysis, making it an essential tool for diverse applications such as environmental pollution monitoring, forensics, material sciences, and more. Discover how GCMS-QP2020 NX can enable reliable, high-quality results to meet your analysis needs today.

Learn More

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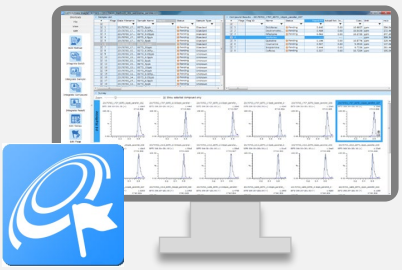

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