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Application News

GC-MS GCMS-QP2020 NX

Using GCMS to Determine the 2,3-Butanedione, 2,3-Pentanedione, and Acetoin Content in E-liquid

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User Benefits

- Users can analyze impurities in e-liquid, which will be regulated in China.
- Users can analyze the content of 2,3-butanedione, 2,3-pentanedione, and acetoin in e-liquid with high sensitivity, good repeatability, and with simple sample preparation.

■ Abstract

A method of using a Shimadzu GCMS-QP2020 NX gas chromatograph mass spectrometer to determine the 2,3-butanedione, 2,3-pentanedione, and acetoin content in e-liquid was devised. Samples were dissolved in and adjusted to volume with ethanol, vortexed, filtered through a membrane, and then analyzed. Results of that analysis revealed that calibration curves for 2,3-butanedione, 2,3-pentanedione, and acetoin had good linearity in the range of 0.2 to 2.0 mg/L, with linear correlation coefficients greater than 0.998. The detection limit for each component was within 0.05 to 1.27 mg/kg. In the recovery test using actual samples, the recovery of each component was 75.8 to 112.5 % for a spike concentration of 6 mg/kg. This method involves simple sample preparation, offers high sensitivity and good repeatability, and can be used to determine the 2,3-butanedione, 2,3-pentanedione, and acetoin content in e-liquid.

■ Introduction

An e-cigarette is an electronic product that simulates a cigarette, by using an atomizer to heat e-liquid (vape juice) in a chamber/tank to produce vapor that resembles smoke from a cigarette. E-liquid mostly consists of a base (glycerin, propylene glycol, water, etc.) and additives (flavorings, plant extracts, etc.). The type of flavoring determines the flavor of the e-liquid. Manufacturers of e-liquid usually choose to add food flavorings commonly used in the food industry, such as 2,3-butanedione, 2,3-pentanedione, and acetoin. However, excessive intake of these flavorings can adversely affect human health.

With the rapid increase in e-cigarettes, a growing number of countries are becoming aware of their health risks. The China Electronics Chamber of Commerce is developing an organizational standard, "Technical specification for safety of e-liquid" (draft for comment), that strictly limits many impurities in e-liquid. That standard stipulates that 2,3-butanedione, 2,3-pentanedione, or acetoin content may not exceed 20 mg/kg.

Based on the "Technical specification for safety of e-liquid" (draft for comment), a method of determining the 2,3-butanedione, 2,3-pentanedione, and acetoin content in e-liquid using a Shimadzu GCMS-QP2020 NX gas chromatograph mass spectrometer was devised. The method involves simple sample preparation, offers high sensitivity and good repeatability, and can be used to effectively monitor the 2,3-butanedione, 2,3-pentanedione, and acetoin content in e-liquid.

■ System

Analytical conditions and compound information are shown in Table 1 and Table 2, respectively.

Table 1 Analytical Conditions

GCMS System	: GCMS-QP2020 NX gas chromatograph mass spectrometer
Analytical Column	: SH-Rxi-624 Sil MS, 60 m × 0.32 mm × 1.8 µm
Column Temp. Program	: 60 °C (2 min) - 10 °C/min - 120 °C (2 min) - 25 °C/min - 240 °C (12 min)
Injection Temp.	: 240 °C
Carrier Gas Control Mode	: Constant linear velocity (25.8 cm/sec)
Injection Mode	: Split
Split Ratio	: 20:1
Ionization Mode	: EI
Interface Temp.	: 250 °C
Ion Source Temp.	: 200 °C
Data Acquisition Mode	: SIM

■ Sample Preparation

Accurately weigh 1 g of e-liquid in a 10-mL volumetric flask, fill to volume with ethanol, insert a stopper, and then place the flask in a vortex mixer for agitation and extraction at 2,000 rpm for 5 min. Collect the liquid extract, filter it through a membrane, and then analyze the filtrate.

Table 2 Compound Parameters and Information

No.	Compound	CAS No.	Retention Time (min)	Quantitative Ion (m/z)	Qualitative Ion (m/z)
1	2,3-butanedione	431-03-8	7.897	86	43
2	2,3-pentanedione	600-14-6	10.271	100	57
3	Acetoin	513-86-0	11.388	88	46

■ Calibration Curves

Fig. 1 and Fig. 2 show TIC chromatogram and mass chromatogram for the standard sample, respectively.

A propylene glycol/glycerin (80/20) mixture was used to prepare a standard stock solution of 2,3-butanedione, 2,3-pentanedione, and acetoin at a concentration of 1,000 mg/L.

Standard solutions for calibration curve concentrations of 0.2, 0.4, 0.5, 1.0 and 2.0 mg/L were prepared using ethanol as solvent. A calibration curve based on external standards was plotted for each compound with the mass concentration on the horizontal axis and the peak area on the vertical axis. The calibration curves for respective compounds are shown in Fig. 3. Based on data for the 0.2 mg/L standard solution, detection limits and quantitation limits for each target were calculated with a 3-fold and 10-fold signal-to-noise ratio (S/N). Results are shown in Table 3.

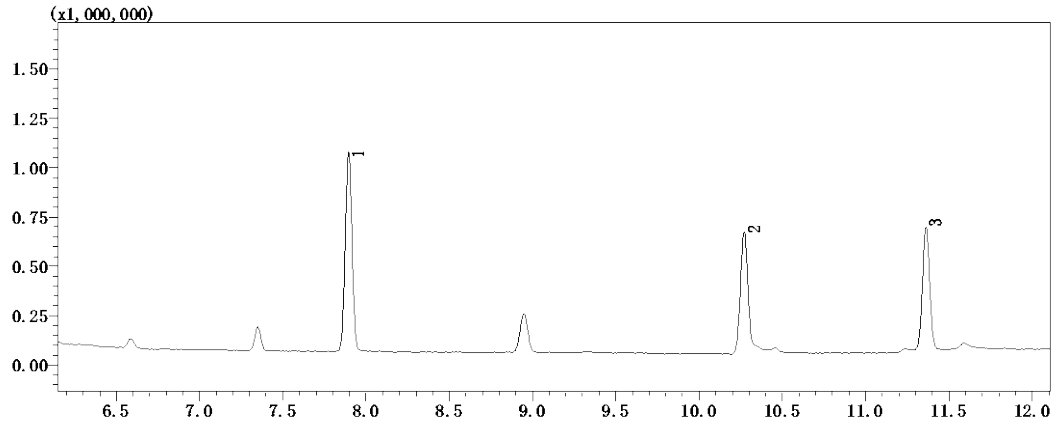

Fig. 1 TIC Chromatogram for Standard Sample

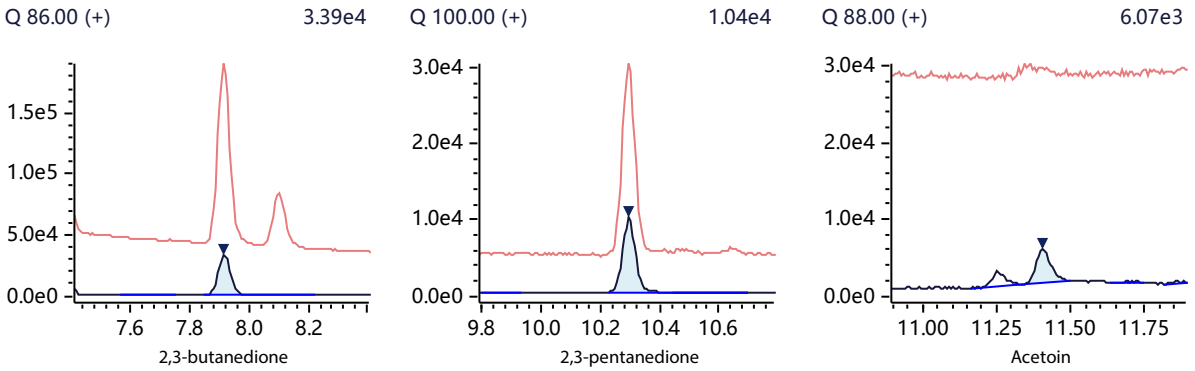

Fig. 2 Mass Chromatograms for the Compounds (0.4 mg/L)

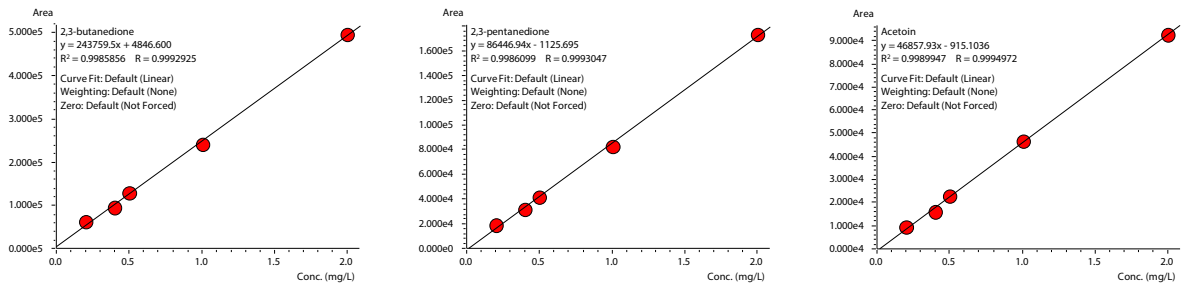

Fig. 3 Calibration Curves (0.2 - 2.0 mg/L)

Table 3 Calibration Curve Information, Detection Limits, and Quantitation Limits

No.	Compound	Correlation Coefficient	Detection Limit (mg/kg)	Quantitation Limit (mg/kg)
1	2,3-butanedione	0.9992	0.05	0.17
2	2,3-pentanedione	0.9993	0.13	0.44
3	Acetoin	0.9994	1.27	4.25

■ Testing of Repeatability

A standard solution at a concentration of 0.2 mg/L was repeatedly analyzed six times to determine instrument repeatability. The peak area and RSD% values for each compound are shown in Table 4.

■ Sample Testing and Recovery Test

Four samples of icy orange-flavored e-liquid were prepared, with two subjected to sample testing and two subjected to recovery testing at a spike concentration of 6 mg/kg. Chromatograms of the e-liquid samples are shown in Fig. 4. Results of sample testing and recovery rates are shown in Table 5.

■ Conclusion

A method of using a Shimadzu GCMS-QP2020 NX gas chromatograph mass spectrometer to determine the 2,3-butanedione, 2,3-pentanedione, and acetoin content in e-liquid was devised. Calibration curves for each component had good linearity in the range of 0.2 to 2.0 mg/L, with linear correlation coefficients greater than 0.9992. The detection limit for each component was within 0.05 to 1.27 mg/kg and can be accurately assessed for compliance with regulations. In the recovery test using actual samples, the recovery of each component was 75.8 to 112.5 % at a spike concentration of 6 mg/kg. The method involves simple sample preparation, offers high sensitivity and good repeatability, and can be used to determine the 2,3-butanedione, 2,3-pentanedione, and acetoin content in e-liquid.

Phthalate Esters in e-liquid will be also regulated in China. The analysis method is introduced in Application News "Using GCMS to Determine the Phthalate Content in E-liquid" (03-GCMS-407-EN).

Table 4 Repeatability Results

No.	Compound	Peak Area						RSD (%)
		1	2	3	4	5	6	
1	2,3-butanedione	44032	42671	44494	43469	41203	40174	4.0
2	2,3-pentanedione	14231	14620	14838	14102	13377	14157	3.5
3	Acetoin	6695	6314	6386	6456	6479	6118	3.0

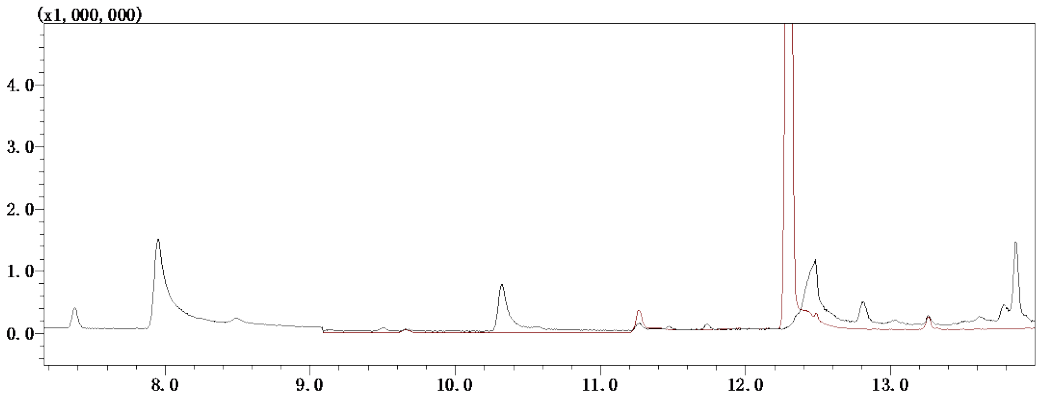

Fig. 4 Chromatograms for Icy Orange-Flavored E-liquid Samples

Table 5 Results of Sample Testing and Spiked Recoveries

No.	Compound	Results of Sample Testing (mg/kg)	Results of Spiked Recovery Testing (mg/kg)	Recovery Rate (%)
1	2,3-butanedione	3.7	10.2	108.3
2	2,3-pentanedione	4.1	8.6	75.8
3	Acetoin	N.D.	6.8	112.5

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