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GC-MS GCMS-QP™2050

Application News

Analysis of Formic Acid in Acetone Using GC/MS System

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User Benefits

- The GCMS-QP2050 enables highly accurate measurement of formic acid in solution.
- The SIM mode enables the detection of formic acid at sub-ppm levels.
- The Scan mode enables the qualitative analysis of unknown components other than formic acid.

■ Introduction

In research on artificial photosynthesis, the analysis of impurities in chemical products and raw materials has increased the demand for high-sensitivity analysis of formic acid. Previous examples of formic acid analysis using GC-BID and GC-FID (Jetanizer™) have been presented, but in this article high-sensitivity analysis of formic acid using the GCMS-QP2050 (see Fig. 1) is introduced.

Fig. 1 GCMS™ -QP2050

■ Insert, Column Phosphoric Acid Treatment

Formic acid is known to adsorb onto the injection port and column, making peak detection difficult. However, by performing phosphoric acid treatment on the glass insert and column before measurement, good peaks can be obtained. The procedure for phosphoric acid treatment of the glass insert is shown in Fig. 2, and the procedure for phosphoric acid treatment of the column is shown in Fig. 3.

1. Prepare the split insert (P/N 227-35007-01).
   ↓
2. Prepare a 0.3 % phosphoric acid acetone solution.
   ↓
3. Immerse the split insert of step 1 in the prepared solution of step 2 for more than 1 minute (see diagram on right).
   ↓
4. Dry at 50 °C for 1 hour.

Fig. 2 Phosphoric Acid Treatment Procedure for Glass Insert

1. Prepare a 100 ppm phosphoric acid methanol solution.
   ↓
2. Measure four times under the analytical conditions shown in Table 1.
   ↓
3. Measure methanol ten times at 150 °C in the column (with all other conditions the same as in Table 1) to stabilize the column interior.

Fig. 3 Phosphoric Acid Treatment Procedure for Column

■ Analytical Conditions

Table 1 shows the analytical conditions.

Table1 Analytical Conditions

GC-MS Model:	GCMS-QP2050 (GC-2030)
Autoinjector:	AOC-30i
[GC]	
Column:	SH-PolarWax (0.32 mm I.D. × 30 m, d.f. = 1.0 µm) P/N 227-36252-01
Column Temp.:	80 °C – 5 °C/min – 180 °C (3 min) (Total 23 min)
Injection Temp.:	180 °C
Purge:	10 mL/min
Injection Mode:	Split
Split Ratio:	2
Carrier Gas Controller:	Constant Linear Velocity (He)
Linear Velocity:	60 cm/sec
Injection Volume:	1 µL
[MS]	
Pumping Speed:	255 L/s
Acquisition Mode:	Scan, SIM
Ion Source Temp.:	200 °C
Interface Temp.:	200 °C
SIM monitoring *m/z*:	Target: 46, For confirmation: 29
m/z range:	20 - 300
Event time:	0.3 sec

■ Analysis of Formic Acid in Acetone Using SIM Mode

The SIM chromatogram for formic acid in acetone at 0.2 ppm is shown in Fig. 4, the calibration curve for 0.2 ppm to 20 ppm is shown in Fig. 5, and the repeatability of the analysis at 0.2 ppm (n = 5) is presented in Table 2.

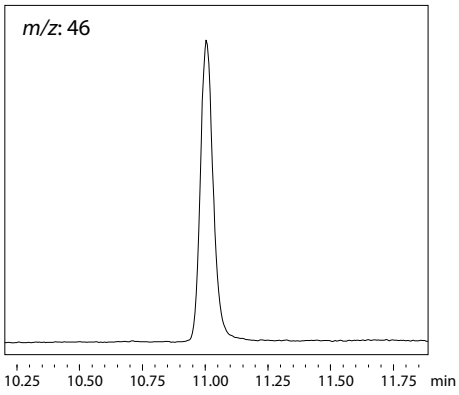

Fig. 4 SIM Chromatogram of 0.2 ppm Formic Acid

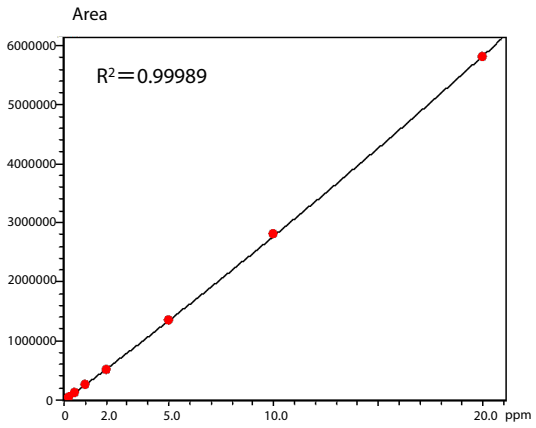

Fig. 5 Calibration Curve from 0.2 ppm to 20 ppm

Table 2 Repeatability of Analysis at 0.2 ppm (n = 5)

Data	Area
Data 1	40,394
Data 2	40,833
Data 3	40,733
Data 4	41,084
Data 5	42,828
Area Average	41,174
%RSD	2.3

■ Analysis of Formic Acid Using Scan Mode

The Total Ion Chromatogram (TIC) chromatogram of a 20 ppm formic acid standard solution is shown in Fig. 6.

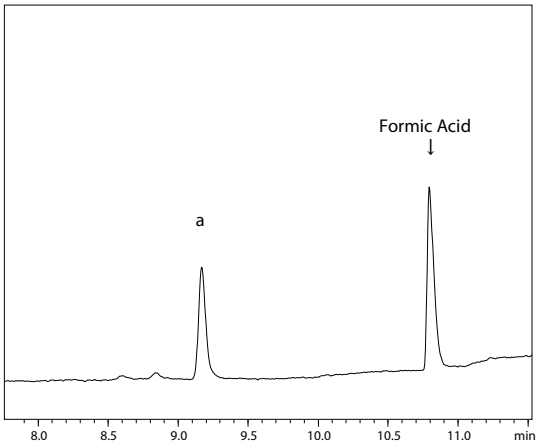

Fig. 6 TIC Chromatogram of a 20 ppm Formic Acid Standard Solution

A peak labeled “a” other than formic acid was observed. By conducting a library search using the mass spectrum of peak a, acetic acid was identified. Thus, by performing analysis in Scan mode, it is possible to identify unknown components.

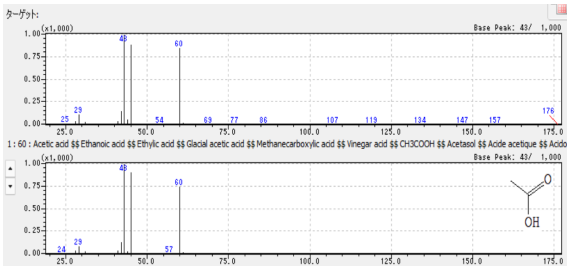

Fig. 7 Library Search Results for Peak a

■ Comparison of Formic Acid Analyses

A comparison of formic acid analyses using GC-BID, GC-FID (Jetanizer), and GC-MS is shown in Table 3.

Table 3 Comparison of Formic Acid Analysis Across Different Analytical Methods

	GC-BID	GC-Jetanizer	GC-MS*2
Phosphoric acid treatment	Needed	Needed	Needed
Conc. Range*1	Approximately 1 ppm to several 1000 ppm	Approximately 1 ppm to several percent	Approximately 0.1 ppm to several ppm
Carrier Gas	He only	He, N_2, H_2	He recommended

*1 The concentration range varies depending on analytical conditions, samples, and the environment.
*2 The values for GC-MS are for the SIM mode.

■ Conclusion

By performing phosphoric acid treatment on the insert and column, it became possible to analyze formic acid in SIM mode down to 0.2 ppm. Furthermore, good results could be obtained for both linearity and repeatability. Additionally, by analyzing in Scan mode, identification of unknown peaks other than formic acid was also achieved.

<Related Applications>

1. Improvement of Sensitivity and Repeatability in Analysis of Formic Acid Application News No. G279
2. High-Sensitivity Analysis of Formic Acid in Methanol Solution Using Jetanizer™ Application News 01-00833-en

01-00957-EN First Edition: Sep. 2025

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