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Application Data Sheet

No.136

GC-MS
Gas Chromatograph Mass Spectrometer

Off-Flavor Analysis in Chemical Material Using a Thermal Desorption Method

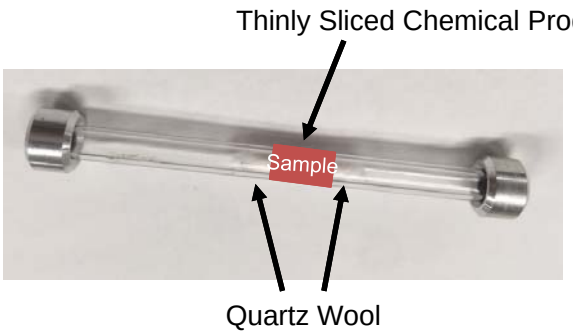
In recent years, there has been an increase in claims related to food and chemical products. Analysis via GC-MS(/MS) has been used as a method of specifying off-flavor causing substances. However, knowledge of the off-flavor causing substance (quality of the odor, offensive odor threshold value, and other information) is required; as a result, inexperienced analysts cannot perform such an analysis. In addition, off-flavor claims must be addressed quickly, so samples in a variety of forms must be preprocessed quickly and conveniently.
The thermal desorption (TD) method is a form of pretreatment in which an adsorbent or the sample itself is heated to a high temperature, and the gases produced are loaded into a GC-MS(/MS), enabling samples to be pretreated quickly and conveniently. In addition, in a GC/MS off-flavor analysis system, information on the parameters needed for off-flavor analysis and the main off-flavor causing substances is contained in a database, allowing analysts with no expertise or experience with off-flavor analysis to perform the analysis.
In this investigation, off-flavor samples were pretreated with the TD method, and chemical products were analyzed using a GC/MS off-flavor analysis system.

Experiment

A chemical product involved in an off-flavor claim (hereinafter the defective product) was sliced thinly, and a TD glass tube (SHIMADZU, P/N: S223-57119) was filled with approx. 40 mg of this sample. Both ends were fastened with 5 mg of quartz wool, it was heated at 250 °C for 30 minutes, and the gases produced were loaded into a GC-MS(/MS). In addition, a chemical product not involved in an off-flavor claim (hereinafter the normal product) was pretreated using the same procedures. The sample loaded was analyzed in GC-MS/MS Scan/MRM mode. The analysis conditions are shown in Table 1, and the analysis samples are shown in Fig. 1.

Table 1: Analysis Conditions

[Instrument Configuration]			
GC-MS/MS:	GCMS-TQ™ 8040		
Sample Loader:	TD-30		
Workstation (GCMS-TQ™8040):	GCMSsolution™ Ver.4.45		
Workstation (TD-30):	TD-30 Control Software		
Database Software:	GC/MS Off-Flavor Analysis System		
Column:	InertCap™ Pure-WAX (30 m x 0.25 mm I.D., df = 0.25 μm) (GL Sciences Inc.)		
[TD-30]		[MS]	
Tube Desorption Temperature:	250 °C	Ion Source Temperature:	200 °C
Tube Desorption Flowrate:	120 mL/min (5 min)	Interface Temperature:	250 °C
Trap Cooling Temperature:	-20 °C	Measurement Mode:	Scan/MRM Simultaneous Measurement
Trap Desorption Temperature:	250 °C	Scan Mass Range:	m/z 45 to 500
Joint Temperature:	250 °C	Scan Event Time:	0.1 sec.
Valve Temperature:	250 °C	Scan Speed:	5000 u/sec.
Transfer Line Temperature:	250 °C	MRM Event Time:	0.3 sec.
		MRM Transition:	Using GC/MS Off-Flavor Analysis System Transitions
[GC]			
Control Mode:	Pressure		
Pressure:	83.5 kPa		
Injection Mode:	Split 1:5 (Split Flowrate 7.2 mL/min)		
Column Oven Temperature:	50 °C (5 min) – (10 °C/min) – 250 °C (10 min)		



If the TD method is used, pretreatment can be performed simply by adding the sample to a glass tube. In addition, the gases produced by the sample can be loaded directly, so the loaded amount can be adjusted.

Fig. 1: Analysis Sample

Analysis Results

When the defective product was analyzed, 48 components were detected.
Calibration curve information on the components registered is contained in the GC/MS off-flavor analysis system, so approximate quantitative values for the detected components can be calculated automatically, without measuring a standard. The concentrations of the detected components can be calculated by dividing the quantitative values obtained by the weight of the measurement sample. In addition, offensive odor threshold values for the components registered are contained in the GC/MS off-flavor analysis system. By calculating the ratio of concentrations and offensive odor threshold values, and then comparing them across the components detected, it is possible to estimate which components are responsible for the odor. Of the 48 components detected, 15 components were identified with particularly large ratios of concentration to offensive odor threshold values. The results are noted in Table 2.

Table 2: List of 15 Components with Particularly Large Ratios of Concentration to Offensive Odor Threshold Value

I.D.	Component Name	Odor Quality	Offensive Odor Threshold Value (pg/mg)	Quantitative Value (pg/mg)		Ratio (-)	
				Normal Product	Defective Product	Defective Product Quantitative Value ÷ Offensive Odor Threshold Value	Defective Product Quantitative Value ÷ Normal Product Quantitative Value
1	2-Undecanone	Cheese, Fruity	10.000	183.060	144.354	14.44	0.79
2	Vanillin	Vanilla	1.000	12.902	11.382	11.38	0.88
3	Acetic Acid	Vinegar	1000.000	704.685	2086.625	2.09	2.96
4	Diacetyl	Butter, Butterscotch Candies	10.000	70.411	17.361	1.74	0.25
5	Lauric Acid; Dodecanoic Acid	Oils, Butter	100.000	298.020	158.269	1.58	0.53
6	Enanthic Acid	Cheese, Dry Fruits	10.000	14.094	13.283	1.33	0.94
7	p-Ethyl Guaiacol	Smoke, Scorched Materials	0.100	0.136	0.107	1.07	0.79
8	Salicyl Aldehyde	Scorched Resin, Hot Spices	1.000	6.978	0.935	0.94	0.13
9	2-Methyl Butyric Acid	Acids, Soles of the Feet, Blue Cheese	10.000	7.405	8.129	0.81	1.10
10	Guaiacol	Smoke, Beechwood Extract	1.000	1.314	0.754	0.75	0.57
11	3-Ethyl-4-Methyl Pyridine	Tobacco, Pyridine	1.000	1.095	0.707	0.71	0.65
12	Capric Acid	Wax	10.000	12.340	6.988	0.70	0.57
13	Propionic Acid	Vinegar, Acetic Acid, Butyric Acid	1000.000	134.265	539.701	0.54	4.02
14	Butyric Acid	Cheese, Yogurt	1000.000	59.323	385.369	0.39	6.50
15	Eugenol	Trees	1.000	0.516	0.339	0.34	0.66

In order to narrow down the candidate off-flavor causing substances, the quantitative values for the defective product were compared to the quantitative values for the normal product. From the results, the difference in the quantitative values for the defective product and the quantitative values for the normal product was significant for acetic acid, butyric acid, and propionic acid, indicating that they were most likely the off-flavor causing substances.

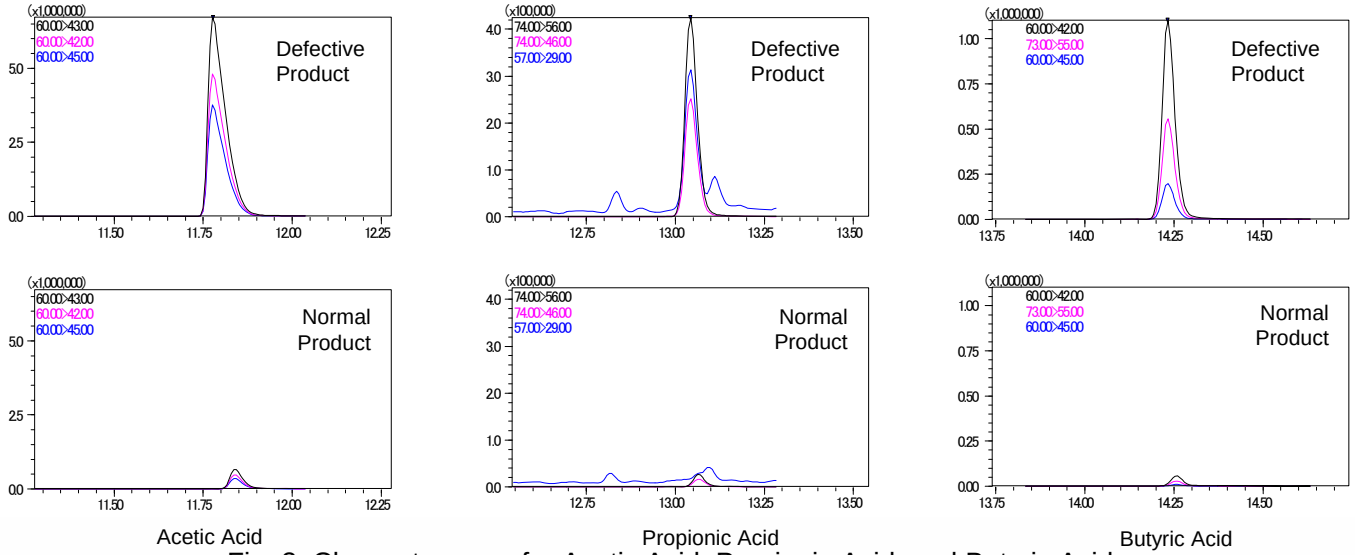


Fig. 2: Chromatograms for Acetic Acid, Propionic Acid, and Butyric Acid

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