SHIMADZU

Application Data Sheet

No.20

GCMS

Gas Chromatograph Mass Spectrometer

Analysis of Japanese Sake Utilizing the Solid Phase Micro Extraction GC-MS Method

Solid phase micro extraction (SPME) is a method for easily extracting and concentrating measurement components. It is applied to the extraction and concentration of gaseous phase fragrant components. This article introduces a sample analysis of the fragrant components of Japanese sake using the SPME method.

Experiment

Commercially-available Japanese sake was measured under the analysis conditions in Table 1.

Table 1: Analysis Conditions

SPME	: AOC-5000	GC-MS	: GCMS-QP2010 Ultra
[SPME]		[GC]	
Fiber	: PDMS/DVB 65μm	Vaporization chamber temperature	: 250℃
Sample amount:	: 10mL＋NaCl 2g (HS extraction)	Column	: Rtx®-5MS (30 mL. x 0.32 mmI.D., 1.0 μm)
Pre Inc Time	: 3 min	Column oven temperature:	50℃ (2 min)→(10℃/ min)→250℃ (5 min)
Incubat Temp	: 50℃	Carrier gas	: Helium
Extract Time	: 30 min	Carrier gas control	: Constant linear velocity (57.6 cm/sec)
Desorb Time	: 2 min	Injection mode	: Splitless (sampling time: 2 min)
		[MS]	
		Interface temperature	:250℃ Ion source temperature :200℃
		Measurement mode	:Scan Mass range: *m/z* 25-400
		Event time	:0.3 sec Emission current: 60 μA (normal)
		Detector voltage	:-0.1 kV (relative value)

Results

The total ion current chromatogram obtained and the identification results of the detected peaks are shown in Fig. 1.

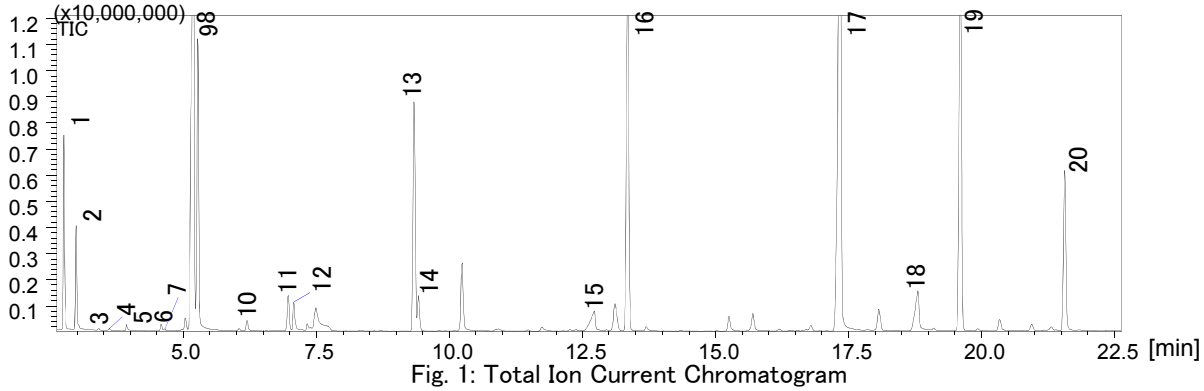

Fig. 1: Total Ion Current Chromatogram

1:Ethyl Acetate 2:Isobutyl alcohol 3:Butanal, 3-methyl- 4:1-Butanol 5:Acetic acid 6:Propanoic acid, ethyl ester
7:n-Propyl acetate 8:Isopentyl alcohol 9:sec-Butylcarbinol 10:Acetic acid, 2-methylpropyl ester
11:Butanoic acid, ethyl ester 12:2,3-Butanediol 13:1-Butanol, 3-methyl-, acetate 14:1-Butanol, 2-methyl-, acetate
15:Hexanoic acid 16:Hexanoic acid, ethyl ester 17:Phenylethyl Alcohol 18:Octanoic Acid
19:Octanoic acid, ethyl ester 20:Acetic acid, 2-phenylethyl ester

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