

Analysis of Phenols in Tap Water Using GC/MS

The method specified by the Minister of Health, Labour and Welfare (Notice No. 261, Ministry of Health, Labour and Welfare, 22 July 2003) in accordance with the provisions of ministerial ordinances on water-quality standards was partially revised on 30 March 2005 (Notice No. 125, Ministry of Health, Labour and Welfare). In the analysis of phenols, the elution solvent used after solid-phase extraction was changed from dichloromethane to ethyl acetate, and a concentration procedure was added after elution.

The following six phenols are the targets of analysis: phenol, 2-chlorophenol, 4-chlorophenol, 2,4-dichlorophenol, 2,6-dichlorophenol, and 2,4,6-

trichlorophenol. After measuring the concentration of each constituent, these concentrations are converted into phenol concentrations, and then added together to obtain the total phenol concentration. The limit is 5 µg/L. Although the toxicity of phenol itself is a problem, even a low concentration of chlorophenols can give rise to an unpleasant odor and taste, and so measurement is considered indispensable.

An example of phenol measurement performed using solid-phase extraction, derivatization, and GC/MS is presented here. This method complies with Notice No. 261 issued by the Ministry of Health, Labour and Welfare (partially revised in 2005 by Notice No. 125).

■ Outline of Analysis

Hydrochloric acid is added to 500 mL of test water to reduce the pH value to 2 or less and then this solution is passed along a styrene-divinylbenzene-vinylpyrrolidone copolymer solid-phase column. After the solid-phase column is rinsed with pure water, it is dehydrated by aeration, and elution is performed with 5 mL of ethyl acetate. After the eluate is dehydrated with anhydrous sodium sulfate, a 4 mL sample is taken and concentrated to a volume of approximately 0.8 mL

by spraying with pure nitrogen gas.

A derivative reagent (N,O-bis (trimethylsilyl) trifluoroacetamide : BSTFA) is then added and the sample is left to stand for at least 1 hour. After that, an internal standard is added, the volume is increased to 1 mL using ethyl acetate, and the sample is subjected to GC/MS-SIM measurement. Fig.1 shows the overall flow of analysis.

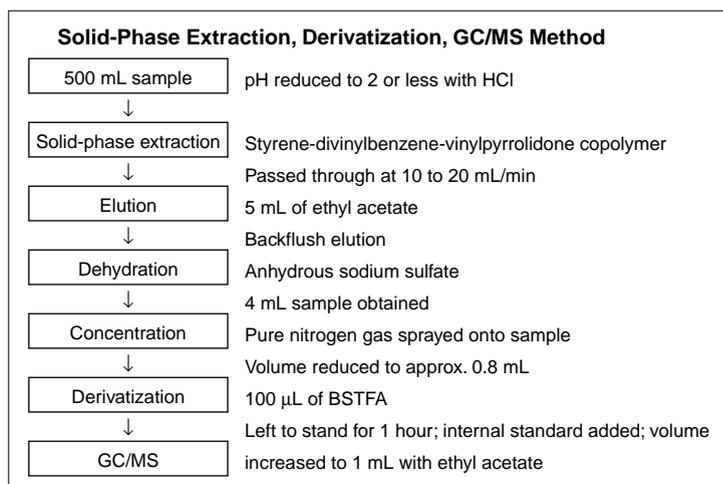


Fig.1 Analysis Flowchart

Table 1 Analytical Conditions

Model : GC/MS-QP2010			
-GC-		-GCMS-	
Column	: Rtx-1 (30 m × 0.25 mm I.D. df = 1.0 µm) (100 % demethylpolysiloxane)	Interface Temp.	: 250 °C
Column Temp.	: 50 °C (2 min) - 10 °C/min - 250 °C (5 min)	Ion box Temp.	: 200 °C
Carrier Gas	: He (Constant linear velocity mode)	Ionization Method	: EI
Linear Velocity	: 45 cm/s	-- Scan Mode --	
Injector Temp.	: 250 °C	Scan Range	: m/z 35 - 350
Injection Method	: Splitless (Head Pressure = 250 kPa at Injection)	Scan Interval	: 0.5 s
Injection Volume	: 1 µL	-- SIM Mode --	
		SIM Monitoring Ion	: refer to Table 2
		SIM Sampling Interval	: 0.2 s

■ Analysis with EI Scan Mode

Fig.2 shows the TIC (total ion chromatogram) obtained by derivatizing a standard sample containing the 6 constituents to be measured, each at concentrations of 2.5 mg/L (concentration in sample water: 5 µg/L),

■ Analysis with EI SIM Mode

SIM (selected ion monitoring) enables high-sensitivity measurement.

Table 2 is the monitor ion table. Fig.4 shows the SIM chromatogram obtained for the derivative of a standard sample containing each constituent at a concentration of 0.25 mg/L (concentration in sample water: 0.5 µg/L). Fig.5 shows the calibration curve for phenol derivative (concentration in sample water: 0.1 to 5.0 µg/L).

Table 2 Monitor Ion Table

Constituent	Selected ion
Phenol	151,166
2-chlorophenol	185,200
4-chlorophenol	185,200
2,6-dichlorophenol	219,234
2,4-dichlorophenol	219,234
2,4,6-trichlorophenol	253,268
Acenaphthene-d10	164,162

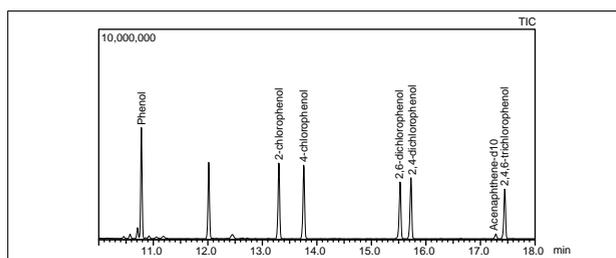


Fig.2 TIC of 2.5 mg/L Standard Sample
(Concentration in Sample Water : 5 µg/L)

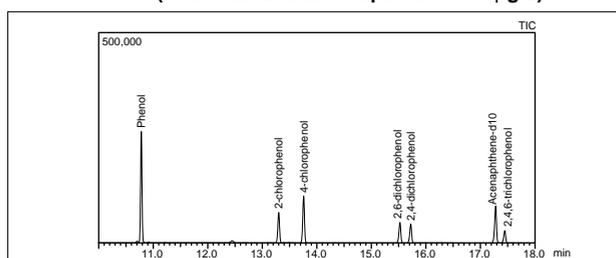


Fig.4 SIM Chromatogram of 0.25 mg/L Standard Sample
(Concentration in Sample Water : 0.5 µg/L)

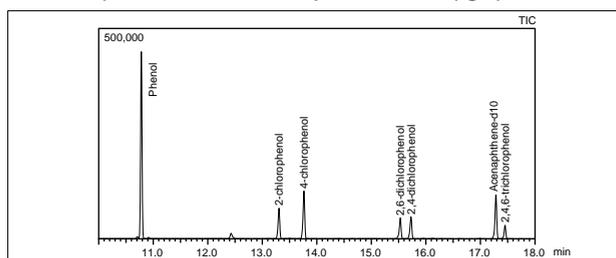


Fig.6 SIM Chromatogram of Tap Water Spiked with Standard
(Equivalent Concentration in Sample Water : 0.5 µg/L)

adding acenaphthene-d10 as an internal standard, and performing measurement in scan mode. Fig.3 shows the mass spectra for each constituent and Table 1 gives the analytical conditions.

A sample was created by adding a standard sample containing each constituent at a concentration equivalent to 0.5 µg/L to tap water. This was then subjected to solid-phase extraction, derivatization, and analysis. Fig.6 shows the resulting SIM chromatogram. It can be seen that even concentrations lower than the limit (5 µg/L) can be reliably measured.

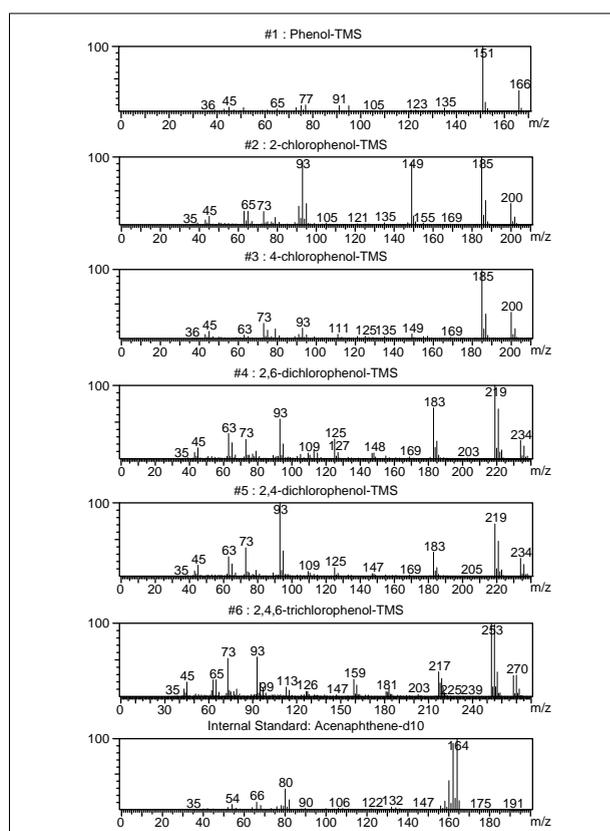


Fig.3 Mass Spectra of Phenols and Internal Standard

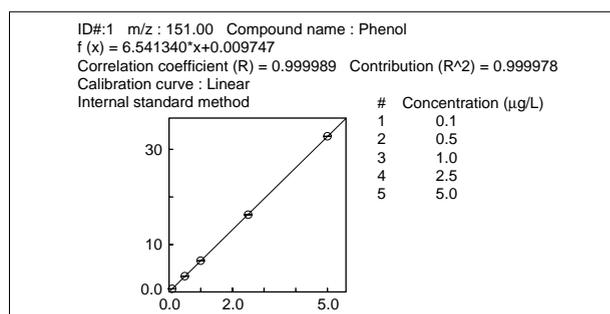


Fig.5 Calibration Curve for Phenol

NOTES:

*This Application News has been produced and edited using information that was available when the data was acquired for each article. This Application News is subject to revision without prior notice.



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