

6.2 Analysis of Volatile Organic Compounds (VOC) in Soil (1) – GC/MS

•Explanation

Following on from the amendments to the water quality standard in tap water in December 1992 and the environment standard for environment water in March 1993, a further amendment was also implemented for the wastewater standard in February 1994. Next, based on these water standards, the environment standard related to pollution of soil was amended in February 1994. The head space method and purge trap method are used for analysis of the compounds concerned. The head space method features ease of use, provides good reproducibility, can accommodate an auto sampler, and carry over (conforming pollutant level of device caused by concentration level of elements) is minimal. The purge trap method allows analysis of low-concentrate samples at high sensitivity as samples can be concentrated using a collecting agent, which provides excellent sensitivity of 10 to 100 times more than that of the head space method.

References

- (1) Drinking Water Test Method & Explanation, Japan Water Works Association volume
- (2) Environmental Water Analysis Manual, Environmental Science Research Group volume
- (3) New Wastewater Standards and Other Analysis Methods, Environmental Science Research Group volume
- (4) Concerning additional items for environment standard related to soil pollution (Verdict) Central Environment Think Tank January 14th, 1994

•Pretreatment

Creation of Sample

↓ Gravel and pieces of wood exceeding a granular diameter of 5 mm are removed from collected soil.

Sample Solution Preparation

↓ 50g of sample and 500mL of solvent (purified water: water without VOC) are placed in a 500mL conical flask with threaded inlet and agitator placed inside, and the flask sealed immediately.

Solving Out

↓ The prepared sample is kept at room temperature and atmosphere (20°C and 1 atmosphere) while being continually stirred for 4 hours using a magnetic stirrer.

Creation of Test Solution

When the above operations are completed, let the sample solution stand for 10 to 30 minutes, then pass it through a membrane filter with perforation diameter of 0.45 μm to make the measuring sample solution.

•Analytical Conditions

Head Space Method

Sample volume	: 10mL+NaCl 3g
Sample temperature	: 60°C
Heat retaining time	: 30min
Needle temperature	: 120°C
Line temperature	: 150°C
Pressurization time	: 2min
Injection time	: 0.20min

Shimadzu GCMS-QP5050A

Carrier gas	: He 120kPa
Column	: DB-624 60m × 0.32mm I.D., 1.8μm(df) : 40°C(2min)-10°C/min -200°C(2min)

Purge Trap Method

Tekmar-Dohmann LSC3000

Sample volume	: 5mL
Adsorption tube	: G-2 (Tenax-Silica gel)
Purge time	: 5min (room temp)
Dry purge time	: 2min
Purge flow rate	: 30mL/min
Desorption time	: 3min (200°C)
Injection	: 2min (200°C)
Baking	: 220°C (30min)
Transfer	: 150°C

Shimadzu GCMS-QP5050A

Carrier gas	: He 120kPa
Column	: DB-624 60m × 0.32mm I.D., 1.8μm(df)
Column temperature	: 40°C(5min)-4°C/min-80°C -6°C/min-140°C-8°C/min-180°C -40°C/min-200°C (2min)

6.2 Analysis of Volatile Organic Compounds (VOC) in Soil (2) – GC/MS

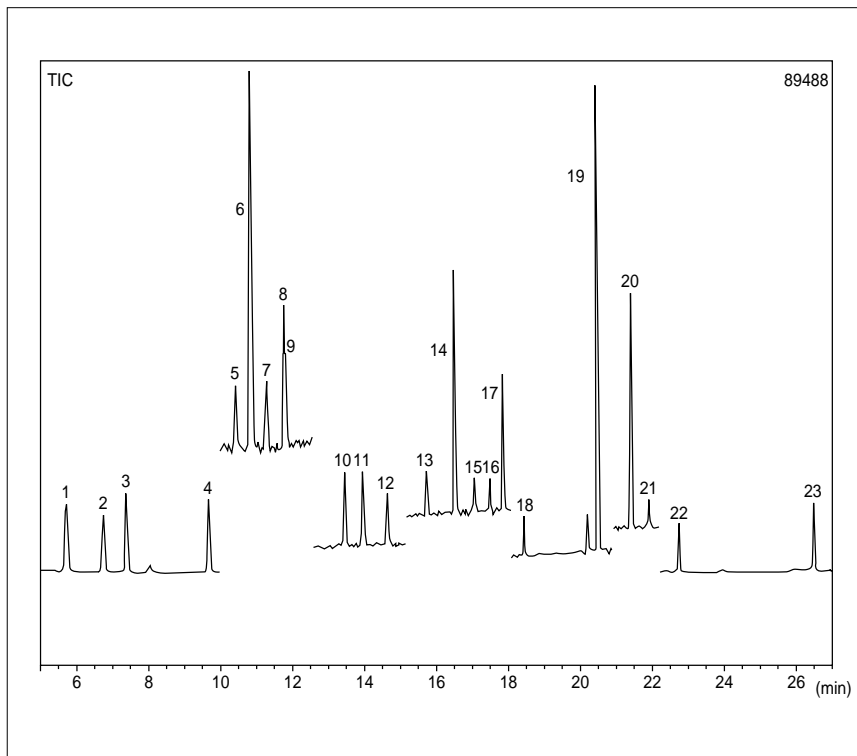


Fig. 116 SIM chromatogram (head space method) for sample with 23 standard elements (1µg/L) added to soil

ID	Name	SIM Selected Ions
1	1,1-dichloroethene	96,61
2	Dichloromethane	84,86
3	trans-1,2-dichloroethene	96,61
4	cis-1,2-dichloroethene	96,61
5	Chloroform	83,85
6	1,1,1-trichloroethane	97,99
7	Tetrachloromethane	117,119
8	Benzene	78,77
9	1,2-dichloroethane	62,64
10	Trichloroethene	130,132
11	1,2-dichloropropane	63,62
12	Bromodichloromethane	83,85
13	cis-1,3-dichloropropene	75,110
14	Toluene	92,91
15	trans-1,3-dichloropropene	75,110
16	1,1,2-trichloroethene	97,99
17	Tetrachloroethene	166,164
18	Dibromochloromethane	129,127
19	m,p-xylene	106,91
20	o-xylene	106,91
21	Bromoform	173,175
22	p-bromofluorobenzene(IS)	174,176
23	p-dichlorobenzene	146,148

Chart 15 SIM selected ions

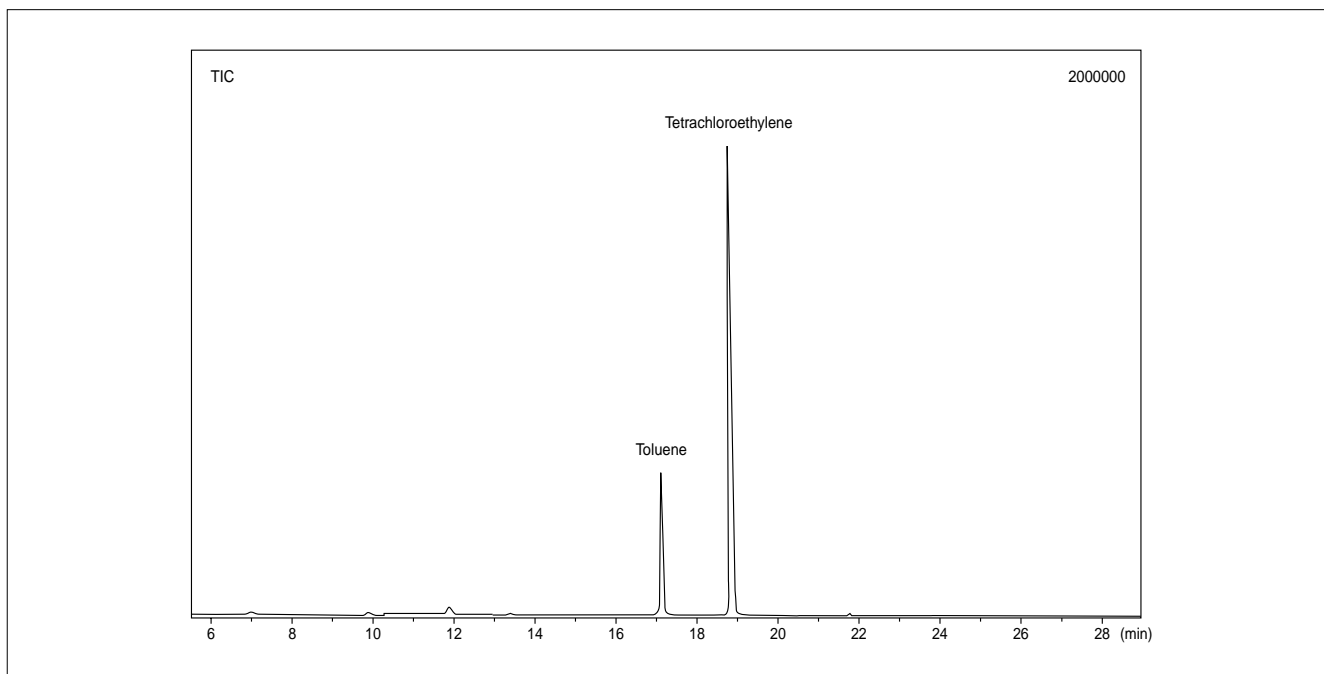


Fig. 117 SIM chromatogram of soil (purge trap method)