

Analysis of Pesticides in Tap Water with GC/MS

In 1993, the Japanese Ministry of Health and Welfare designated of guide line values and testing methods for the quality of tap water.

Subsequently in June 1999, the notification "Establishment of standards for the quality of tap water" was partially revised to increase the number of monitored items from 26 to 32 (nitrite nitrogen, uranium, bentazone, carbofuran, 2,4-dichlorophenoxy acetic acid (2,4-D) and triclopyr were added). Guide line values were revised for six items (boron, dichloroacetic acid, chlorothalonil (TPN), propyzamide, dichlorvos (DDVP) and fenobucarb

(BPMC)). Table 1 lists the components, guide line values and measurement methods.

Among the newly added pesticides bentazone, carbofuran, 2,4-dichlorophenoxy acetic acid (2,4-D) and triclopyr, carbofuran is analyzed by HPLC.

This article introduces measurement of bentazone, 2,4-D, triclopyr and 2,4,5-trichlorophenoxy acetic acid (2,4,5-T), which is an analogous compound for 2,4-D. All these compounds require methylation to analyze using GC/MS and cannot be analyzed simultaneously with other pesticides. Table 2 shows the pretreatment processes.

Table 1 List of Monitored Items in Tap Water

Compounds			Compounds		
		Guide line value			Guide line value
1	trans-1,2-Dichloroethylene	0.04mg/L	17	Chloral Hydrate	0.03mg/L
2	Toluene	0.6mg/L	18	Isoxathion	0.008mg/L
3	Xylene	0.4mg/L	19	Diazinon	0.005mg/L
4	p-Dichlorobenzene	0.3mg/L	20	Fenitrothion(MEP)	0.003mg/L
5	1,2-Dichloropropane	0.06mg/L	21	Isoprothiolane	0.04mg/L
6	Di(2-ethylhexyl)Phthalate	0.06mg/L	22	Chlorothalonil(TPN)	0.05mg/L
7	Nickel	0.01mg/L	23	Propyzamide	0.05mg/L
8	Antimony	0.002mg/L	24	Dichlorvos(DDVP)	0.008mg/L
9	Boron	0.2mg/L	25	Fenobucarb(BPMC)	0.03mg/L
10	Molybdenum	0.07mg/L	26	Chlornitrofen(CNP)	0.005mg/L
11	Uranium	0.002mg/L	27	Iprobenfos(IBP)	0.008mg/L
12	Nitrite Nitrogen	0.05mg/L	28	EPN	0.006mg/L
13	Formaldehyde	0.08mg/L	29	Bentazone	0.2mg/L
14	Dichloroacetic Acid	0.02mg/L	30	Carbofuran	0.005mg/L
15	Trichloroacetic Acid	0.3mg/L	31	(2,4-Dichlorophenoxy)acetic Acid	0.03mg/L
16	Dichloroacetonitrile	0.08mg/L	32	Triclopyr	0.006mg/L

Table 3 shows the analysis conditions. Fig. 1 shows the TIC (10ppm) of the three newly added components and 2,4,5-T. Fig. 2 shows the mass spectrum of each component. The internal standard substances used in these analyses are phenanthrene-d10 and pyrene-d10.

Fig. 3 shows the calibration curves for 10ppb to 500 ppb and Fig. 4 shows the SIM chromatograms at 50ppb. The samples were pesticide standard samples diluted in solvents. Since the actual samples are analyzed after concentration at 1000 times, guide line values are easily cleared.

Table 2 Pretreatment Processes

Solid phase column conditioning Sequentially deliver 5mL dichloromethane, 5mL methyl alcohol and 5mL purified water.
Adjust test water to pH 3.5 with hydrochloric acid (1+10).
Deliver 500mL of test water to the solid phase column (10 or 20mL/min.).
Aspirate the solid phase column for at least 30 minutes.
Deliver 5mL of dichloromethane to the solid phase column (pesticide desorption)
Spray dichloromethane solution with N2 and concentrate to 0.1mL.
Add 0.1mL of diazomethane for methylation.
Leave stand for 30min.
Add 0.25mL of internal standard solution (9-bromoanthracene).
Add dichloromethane to make it 0.5mL. Use this as the test liquid.

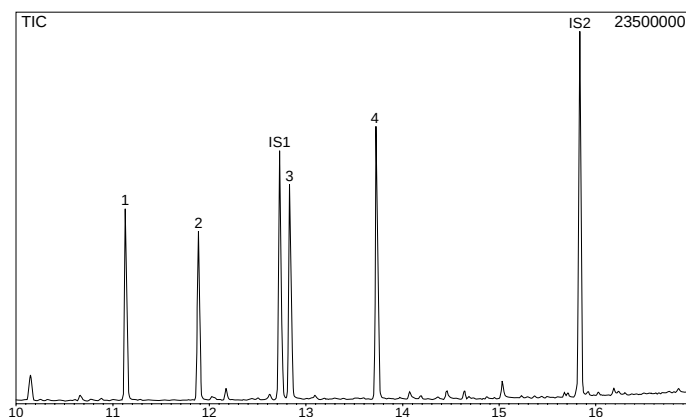
Table 3 Analytical conditions

Instrument : Shimadzu GCMS-QP5050A
 -GC-
 Column : DB-1 (30m × 0.32mm I.D. df=0.25μm)
 Column Temp. : 50°C(2min)-20°C/min-100°C-10°C/min
 -200°C-20°C/min-300°C(3min)
 Injector Temp. : 300°C
 Carrier Gas : He 40kPa
 Injection Method : Splitless(Sampling Time=2min)
 -MS-
 Interface Temp. : 300°C
 Scanning Range : m/z 35~400

Selected ion

	Pesticides	SIM(m/z)
1	Methyl 2,4-D	199.00,234.00,236.00
2	Methyl Triclopyr	109.95,111.95,269.00
3	Methyl 2,4,5-T	233.00,235.00,268.00
4	Methyl Bentazone	212.05,105.00,254.00
IS-1	Phenanthrene-d10	188.00,160.10
IS-2	Pyrene-d10	212.10,106.10

Sample name : Four newly designated pesticide components in tap water (10ppm)
Method file name : TapWaterNEW4.MO2



- 1. methyl 2,4-D
- 2. methyl Triclopyr
- IS1 Phenanthrene-d10
- 3. methyl 2,4,5-T
- 4. methyl Bentazone
- IS2 Pyrene-d10

Fig.1 TIC chromatogram of pesticides (10ppm)

Sample name : Four newly designated pesticide components in tap water (10ppm)
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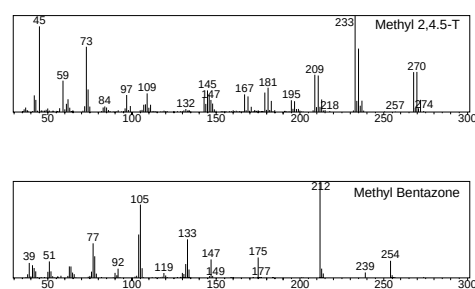
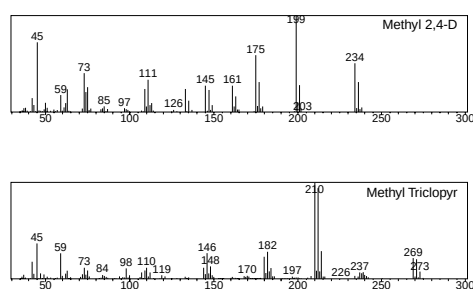
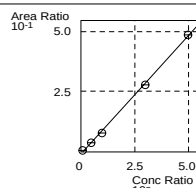


Fig.2 Mass Spectra of Pesticides

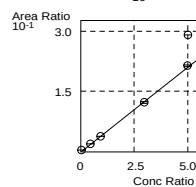
m/z 234 Methyl 2,4-D

1	10.000	0.005
2	50.000	0.037
3	100.000	0.077
4	300.000	0.277
5	500.000	0.489



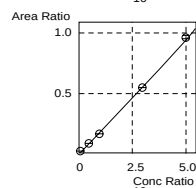
m/z 269 Methyl Triclopyr

1	10.000	0.004
2	50.000	0.019
3	100.000	0.038
4	300.000	0.122
5	500.000	0.214



m/z 233 Methyl 2,4,5-T

1	10.000	0.015
2	50.000	0.079
3	100.000	0.161
4	300.000	0.549
5	500.000	0.972



m/z 212 Methyl Bentazone

1	10.000	0.030
2	50.000	0.159
3	100.000	0.320
4	300.000	1.091
5	500.000	1.930

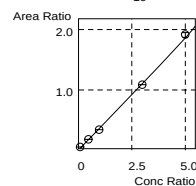


Fig.3 Calibration Curves(10ppb~500ppb)

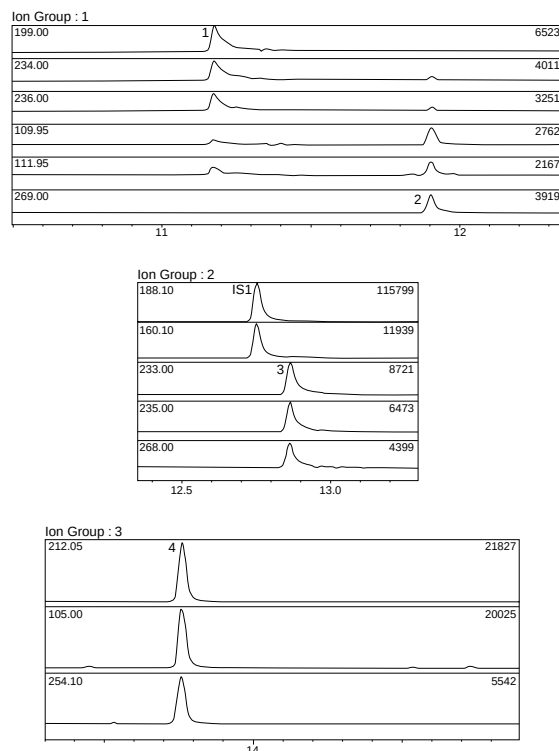


Fig.4 SIM chromatogram of pesticides (50ppb)



SHIMADZU CORPORATION, International Marketing Division

3. Kanda-Nishikicho 1-chome, Chiyoda-ku, Tokyo 101-8448, Japan Phone: 81(3)3219-5641 Fax: 81(3)3219-5710
Cable Add.: SHIMADZU TOKYO

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