ELSEVIER

Journal of Chromatography A, 911 (2001) 75–83

JOURNAL OF CHROMATOGRAPHY A

www.elsevier.com/locate/chroma

Gas chromatographic–mass spectrometric determination of hydrophilic compounds in environmental water by solid-phase extraction with activated carbon fiber felt

Kuniaki Kawata[a,*], Tsuyoshi Ibaraki[a], Akiko Tanabe[a], Hiroaki Yagoh[a], Akiko Shinoda[b], Hiroshi Suzuki[b], Akio Yasuhara[c]

[a]*Niigata Prefectural Research Laboratory for Health and Environment, 314-1 Sowa, Niigata 950-2144, Japan*
[b]*Showa Denko Co. Ltd., 5-1 Ogimachi, Kawasaki-ku, Kawasaki, Kanagawa 210-0867, Japan*
[c]*The National Institute for Environmental Studies, 16-2 Onogawa, Tsukuba, Ibaraki 305-0053, Japan*

Received 19 September 2000; received in revised form 12 December 2000; accepted 13 December 2000

Abstract

Simple gas chromatographic–mass spectrometric determination of hydrophilic organic compounds in environmental water was developed. A cartridge containing activated carbon fiber felt was made by way of trial and was evaluated for solid-phase extraction of the compounds in water. The hydrophilic compounds investigated were acrylamide, *N*,*N*-dimethylacetamide, *N*,*N*-dimethylformamide, 1,4-dioxane, furfural, furfuryl alcohol, *N*-nitrosodiethylamine and *N*-nitrosodimethylamine. Overall recoveries were good (80–100%) from groundwater and river water. The relative standard deviations ranged from 4.5 to 16% for the target compounds. The minimum detectable concentrations were 0.02 to 0.03 μg/l. This method was successfully applied to several river water samples.

Keywords: Environmental analysis; Water analysis; Solid-phase extraction; Carbon fiber felt; Acrylamide; Dimethylacetamide; Dimethylformamide; Dioxane; Furfural; Furfuryl alcohol; Nitrosodiethylamine; Nitrosodimethylamine; Amides; Amines

1. Introduction

Hydrophilic organic compounds such as *N*,*N*-dimethylformamide and 1,4-dioxane are polar solvents, miscible with water as well as several organic solvents. Determination of these toxic compounds in water is important for estimating their effect on human health and better understanding the complicated chemistry involved in the chlorination of water [1]. Among these compounds, 1,4-dioxane (an anticipated carcinogen [2]), was frequently detected in leachate from wastes landfills [3], sea water, river water, groundwater [4,5] and rain water [5]. *N*,*N*-Dimethylacetamide, *N*,*N*-dimethylformamide and furfural were also detected in environmental water samples [5–7] as well as leachates from wastes landfill [5]; *N*-nitrosodimethylamine was detected in groundwater [8], sea water and river water [5].

Although the purge-and-trap technique [9] has been used for the preconcentration of 1,4-dioxane, it suffers from interferences by some substances [10]. Headspace analysis of *N*,*N*-dimethylacetamide, *N*,*N*-

*Corresponding author. Tel.: +81-25-263-9417; fax: +81-25-263-9410.
E-mail address: kkawata@mail.goo.ne.jp (K. Kawata).

0021-9673/01/$ – see front matter
PII: S0021-9673(00)01252-8

dimethylformamide and 1,4-dioxane [11] gave insufficient lower detection limits. These hydrophilic compounds in water can be extracted by solid-phase extraction (SPE) for determination by gas chromatography–mass spectrometry (GC–MS) in many cases [1,6–8,12–14]. Activated carbon has been frequently used as a solid phase rather than alkylated porous silica [1,6–8,12–15]. Kadokami et al. [1] reported a granular activated carbon in a PTFE tube as a device for SPE of 15 hydrophilic organic compounds such as *N*,*N*-dimethylacetamide, *N*,*N*-dimethylformamide, 1,4-dioxane, furfural, *N*-nitrosodiethylamine and *N*-nitrosodimethylamine. Cartridges containing granular activated carbon, which are commercially available, have been also used for SPE of *N*,*N*-dimethylformamide [7] and 1,4-dioxane [12] in water, because of their easy preparation as well as reliable collection efficiency. Prior to SPE using these cartridges, water samples were filtered through glass-fiber filters with 1-μm pore size to remove suspended particulates [6,7]. However, these cartridges cause high resistance to large flow in extraction, because they are easily clogged with suspended particulates smaller than 1 μm in diameter and coextractives from water samples, resulting in impossible extraction. In contrast, activated carbon fiber felt, which has been used to collect pesticides in air [16], hardly clogs during the extraction procedure. However, a part of the felt easily breaks into fibers during extraction.

Therefore, we produced new cartridges containing activated carbon fiber felt and evaluated them for SPE of hydrophilic organic compounds in environmental water. This report presents the successful determination of eight hydrophilic organic compounds in environmental water using the activated carbon fiber felt cartridge for SPE and GC–MS. The hydrophilic compounds investigated were acrylamide, *N*,*N*-dimethylacetamide, *N*,*N*-dimethylformamide, 1,4-dioxane, furfural, furfuryl alcohol, *N*-nitrosodiethylamine and *N*-nitrosodimethylamine. Among the target compounds, *N*-nitrosodiethylamine and *N*-nitrosodimethylamine have been reported to form naturally in the environment by the reaction of nitrous acid with amines or amides [5,17] as well as by the cooking of fish or processed meat [17,18]; *N*-nitrosodimethylamine is a contaminant and an oxidative degradation product of unsymmetrical dimethylhydrazine, a component of rocket fuel [8]. The IARC has evaluated *N*-nitrosodiethylamine and *N*-nitrosodimethylamine to be regarded for practical purposes as if they were carcinogenic to humans [19,20]. The other target compounds are mainly used as solvents as well as raw materials of paint (acrylamide) or plastics (furfural and furfuryl alcohol).

2. Experimental

2.1. Apparatus and materials

A Waters Sep-Pak Concentrator and a J&W SPE Manifold were used for SPE and elution, respectively. A gas chromatograph–mass spectrometer model, Shimadzu GCMS-QP5050A was used for quantitative analysis. GC–MS conditions were as follows: column, a fused-silica column J&W DB-WAX (0.5 μm film thickness, 30 m×0.25 mm I.D.); column temperature programmed from 35°C (held for 3 min) to 190°C (held for 3 min) at a rate of 5°C/min; injector temperature, 200°C; injection mode, splitless; injection temperature, 170°C; carrier gas pressure programmed from 6 to 70 kPa (held for 2 min) at a rate of 200 kPa/min, then back to 6 kPa at the same rate and increased to 39 kPa (held for 3 min) at a rate of 1.1 kPa/min; ionization current, 300 μA; electron energy, 70 eV.

Reagents were purchased from Wako (Osaka, Japan) and Kanto (Tokyo, Japan). Standard solutions of a mixture of target compounds (100 and 20 μg/ml) were prepared in acetone. An acetone solution containing 40 μg/ml of 1-bromo-4-fluorobenzene was prepared as an internal standard solution. A surrogate solution (20 μg/ml) of [$^2H_7$]*N*,*N*-dimethylformamide (*N*,*N*-dimethylformamide-d_7) and [$^2H_8$]1,4-dioxane (1,4-dioxane-d_8) in acetone (20 μg/ml) was prepared as a surrogate solution.

Purified water for washing the extraction cartridges as well as blank tests was prepared by passing water from a Milli-Q system (Millipore, Bedford, MA, USA) through a glass column (35 cm×10 mm I.D.) packed with 10 g of Activated Carbon Beads-L (20–30 mesh; specific surface area, 800–1200 m^2/g; the surface is considered hydrophobic), which was purchased from GL Sciences (Tokyo, Japan). A 1-μm pore size glass-fiber filter,

Toyo GA-100, with 47 mm diameter was heated at 450°C for 4 h before use. A Waters Sep-Pak Plus C_{18} cartridge containing 360 mg of octadecylsilane-bonded silica was washed with 5 ml of acetone, followed by 5 ml of the purified water prior to use.

Ground water (pH 7.0; containing sulfates at 4.3 mg/l, chlorines at 3.6 mg/l, sodium at 3.8 mg/l, potassium at 0.4 mg/l, magnesium at 1.3 mg/l and calcium at 6.9 mg/l) was collected for recovery tests at Yuzawa Town, Niigata Prefecture, Japan. River water (pH 7.2; containing biochemical oxygen demand at 1.6 mg/l and suspended substances at 1.4 mg/l) was also collected for the recovery tests from Kamo River, Niigata Prefecture. They were filtered through the glass fiber filter before use. No target compounds were determined from both the water samples.

Activated carbon fiber felt KF Type 1500, purchased from Toyobo (Osaka, Japan), was employed for the solid phase, because the fiber felt has the widest specific surface area of 1500 m^2/g among activated carbon fiber felts available. Average length, diameter, pore size and apparent density of the fiber felt are 300 μm, 14–23 μm, <14 nm and 58 kg/m^3, respectively; the surface of the fiber felt is considered hydrophobic. The extraction cartridge used was made of a polypropylene barrel (8 ml volume, 12 mm I.D.) and packed with 0.5 g of the fiber felt at a density of 440 kg/m^3 supported by polyethylene frits at both sides of the felt as well as a paper frit at a side (Fig. 1).

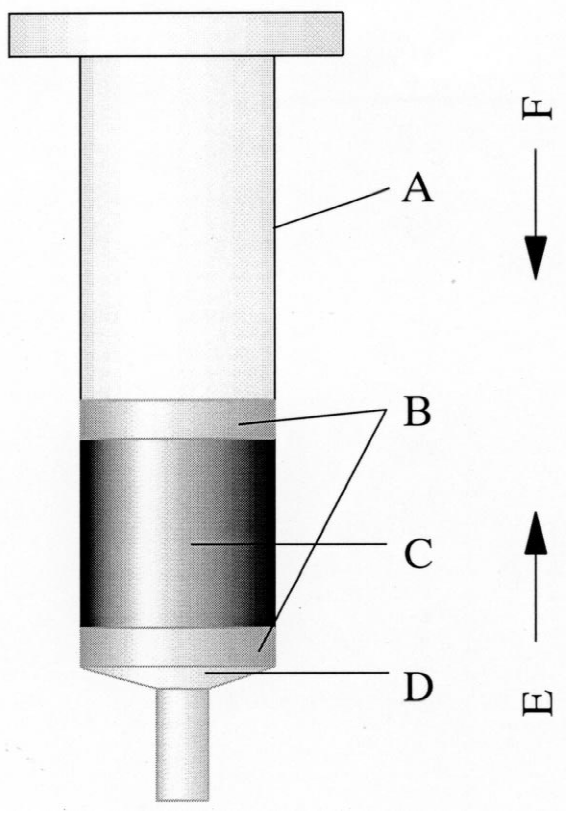

Fig. 1. Activated carbon fiber cartridge. A = Polypropylene syringe of 8 ml; B = polyethylene frit; C = activated carbon fiber felt; D = paper frit; E = direction for extraction; F = direction for elution.

2.2. Determination procedure

Water samples were filtered through the glass-fiber filter. A 500-ml volume of the filtered water spiked with 25-μl of the surrogate solution was passed through a C_{18} cartridge and three extraction cartridges in series at 10 ml/min in series. After the extraction cartridges were washed with 10 ml of purified water, they were dried by passing air for 2 min under 2.7 kPa using an aspirator followed by centrifugation at 3000 rpm (1700 *g*) for 10 min. The target compounds collected on the cartridges were eluted with 5 ml of acetone at 0.5 ml/min in the opposite direction of the extraction (Fig. 1). The eluates were concentrated to 3 ml by blowing nitrogen gas. A 10-μl volume of the internal standard solution was added to the solution and a 4-μl aliquot of the resulting mixture was analyzed by GC–MS. The monitored ions for quantification of the compounds are listed in Table 1, together with their retention times. The ratios of peak areas of the monitor ions to those of the internal standards were used for quantification of the compounds.

3. Results and discussion

3.1. GC–MS analytical conditions

Fused-silica columns bonded with 100% dimethylpolysiloxane [10], 5% diphenyl–dimethylpolysiloxane [9] or polyethylene glycol [1,6,11] have been used for comparison of gas

Table 1
Retention times, selection ions for GC–MS determination and minimum detection limits of target compounds

Compound	Functional group or use	t_R[a] (min)	m/z		MDL[d] (μg/l)
			Q[b]	I[c]	
Acrylamide	Amide	34.75	71	55	0.02
N,*N*-Dimethylacetamide	Amide	22.34	87	44	0.02
N,*N*-Dimethylformamide	Amide	19.98	73	44	0.03
1,4-Dioxane	Ether	11.26	88	58	0.03
Furfural	Aldehyde	23.56	96	95	0.02
Furfuryl alcohol	Alcohol	28.47	98	81	0.02
N-Nitrosodiethylamine	Amine	21.64	102	56	0.02
N-Nitrosodimethylamine	Amine	19.34	74	42	0.02
N,*N*-Dimethylformamide-d_7	Surrogate	20.09	80	–	–
1,4-Dioxane-d_8	Surrogate	11.21	96	–	–
1-Bromo-4-fluorobenzene	Internal standard	20.50	174	–	–

[a] Retention time.
[b] Quantitation ion.
[c] Confirmation ion.
[d] Minimum detection limit.

chromatographic separation of hydrophilic organic compounds. Among these columns, DB-WAX, a fused-silica column bonded with polyethylene glycol, gave a predominant separation. Therefore, DB-WAX was chosen for this investigation. Column temperature program was modified to raise column temperature slower than that in our previous work [7] for better separation of the target compounds. Carrier gas pressure was raised from 6 to 70 kPa and maintained at 70 kPa for 2 min after injection to introduce the sample injected to the column under the high pressure; after reduction the pressure to 6 kPa, the pressure was increased from 6 to 39 kPa at a rate of 1.1 kPa/min for a constant flow of helium at 1.2 ml/min. Thereby, the target compounds, the surrogates and the internal standards were separated completely within 35 min under the GC–MS conditions described in Section 2.1.

3.2. Desorption solvent

Desorption efficiencies for the target compounds and surrogates from the extraction cartridges were determined by passing 50 ml of purified water spiked with 1 μg of the compounds through the cartridges. After the cartridges were washed with 10 ml of purified water and dried, the compounds were eluted from the cartridges by using acetone and dichloromethane. The results are given in Table 2. At 4–6 ml acetone, the recovery of all eight analytes exceeded 91%. By contrast, when dichloromethane was used, the recovery of furfural and furfuryl alcohol did not exceed 60%, while those for *N*,*N*-dimethylacetamide and *N*,*N*-dimethylformamide did not exceed 83%. Dichloromethane would be considered an acceptable substitute for acetone only for 1,4-dioxane, *N*-nitrosodiethylamine, and *N*-nitrosodimethylamine. Therefore, a 5-ml volume of acetone was recommended for the elution of the compounds from the extraction cartridge. No target compounds were observed in the solvent or in the procedure blanks.

3.3. Interference by water in eluate for GC–MS determination

When water from the solid phase is present in the eluates, some compounds provide wide and tailing peaks; insufficient drying of the extraction cartridge before elution of target compounds resulted in poor quantitation [21]. Therefore, the effect of water on the peak of the target compounds, surrogates and the internal standard was elucidated. Among these compounds, the peak shapes of 1,4-dioxane and 1,4-dioxane-d_8 became much less sharp with water. Fig. 2 illustrates the chromatograms of *N*,*N*-dimethylformamide and 1,4-dioxane. While the peak shape of

Table 2
Desorption efficiencies from extraction cartridge

Compound[a]	Desorption efficiency (%)											
	Volume of acetone (ml)						Volume of dichloromethane (ml)					
	1	2	3	4	5	6	1	2	3	4	5	6
Acrylamide	26	69	89	92	93	93	<5	<5	<5	<5	<5	<5
N,*N*-Dimethylacetamide	34	91	100	100	101	101	56	76	83	83	83	83
N,*N*-Dimethylformamide	<5	68	88	91	91	91	68	80	80	80	80	80
1,4-Dioxane	32	64	90	95	96	96	93	100	100	100	100	100
Furfural	10	48	85	92	93	93	10	24	34	42	51	59
Furfuryl alcohol	20	66	91	92	92	92	7	18	25	29	35	38
N-Nitrosodiethylamine	23	71	93	94	94	94	100	115	115	115	115	115
N-Nitrosodimethylamine	<5	61	89	91	92	93	90	97	97	97	97	97
N,*N*-Dimethylformamide-d_7	<5	73	91	93	93	93	63	76	77	77	77	77
1,4-Dioxane-d_8	12	62	93	97	97	98	95	102	102	102	102	102

[a] Added 1 μg.

N,*N*-dimethylformamide did not change by adding water, that of 1,4-dioxane dulled as water content increased from 3.8 to 14%. Moreover, retention time of *N*,*N*-dimethylformamide changed in the presence of more than 1.6% of water (Fig. 2). Margins of retention times of the compounds with water to those without water are shown in Fig. 3. Although the retention time of 1-bromo-4-fluorobenzene decreased

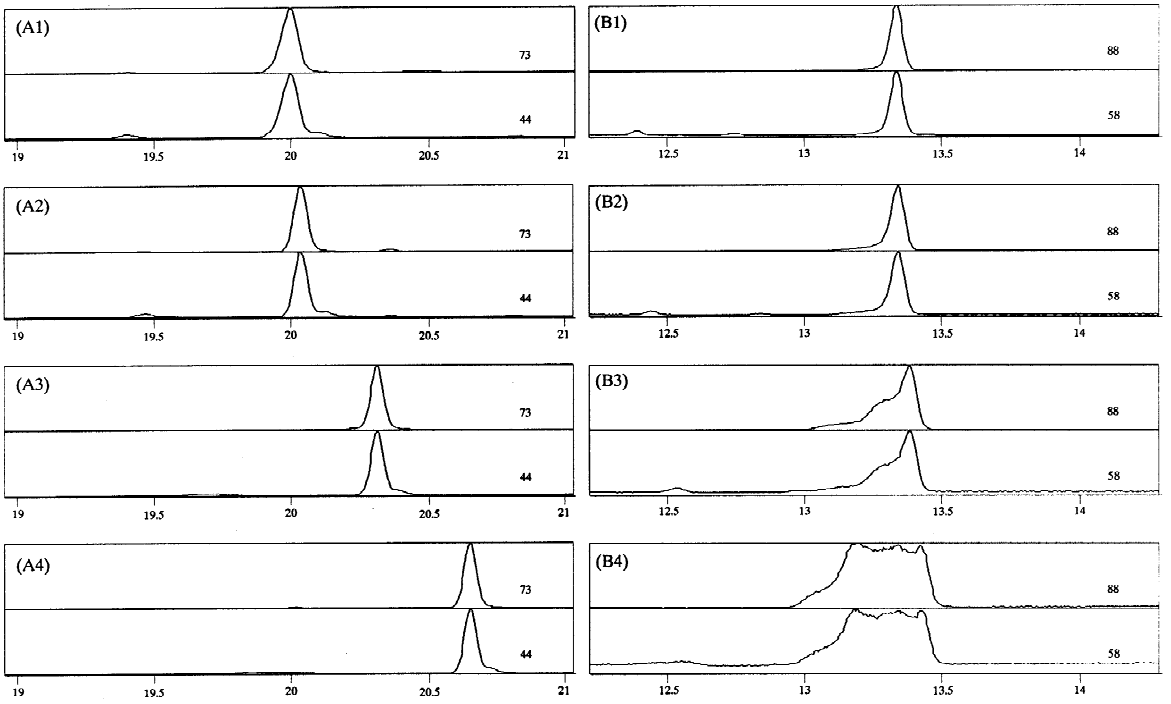

Fig. 2. Effect of water in acetone solution on peak shapes of *N*,*N*-dimethylformamide (A) and 1,4-dioxane (B). Mass chromatograms of the confirmation ion and the quantitation ion are given in the upper and lower rows, respectively. Water contents are 0% (A1 and B1), 1.6% (A2 and B2), 7.6% (A3 and B3) and 14% (A4 and B4).

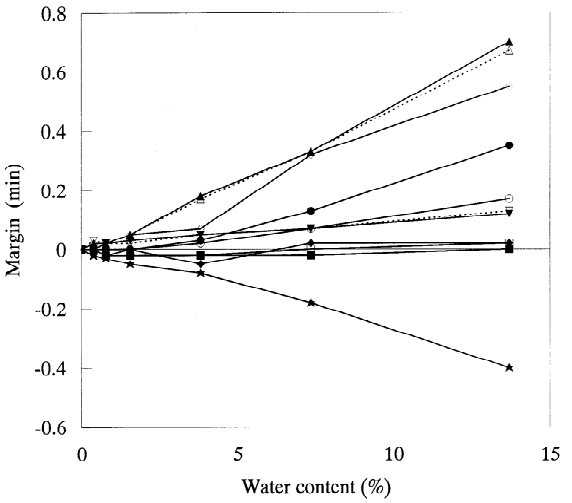

Fig. 3. Effect of water in acetone solution on retention time. Margin is the difference of retention time in the presence of water and that in the absence of water for each compound. ■ = Acrylamide; ● = *N*,*N*-dimethylacetamide; ▲ = *N*,*N*-dimethylformamide; ▼ = 1,4-dioxane; ♦ = furfural; □ = furfuryl alcohol; ○ = *N*-nitrosodiethylamine; △ = *N*-nitrosodimethylamine; ▽ = *N*,*N*-dimethylformamide-d_7; ◇ = 1,4-dioxane-d_8; ★ = 1-bromo-4-fluorobenzene.

with the increase of water content, those of the other compounds except for acrylamide increased accordingly with the increase of water content.

The water content in the eluate decreased to <1.4% when the extraction cartridge was dried by passing air for 2 min using an aspirator followed by centrifugation at 3000 rpm (1700 *g*) for 10 min. Hence, this procedure prior to the elution from the extraction cartridge is necessary.

3.4. Breakthrough

Breakthroughs of the target compounds and the surrogates from the extraction cartridge were evaluated as follows. Purified waters of 250 ml, 500 ml and 1000 ml, to which 1 μg of each compound was added, were passed through a series of three extraction cartridges. The compounds were eluted from the cartridges under the conditions described in Section 2.2. Recoveries of the target compounds as well as the surrogates are shown in Fig. 4. Although the recoveries were good (86–106%) in case of 500 ml extraction, they were rather low (58–82%) in

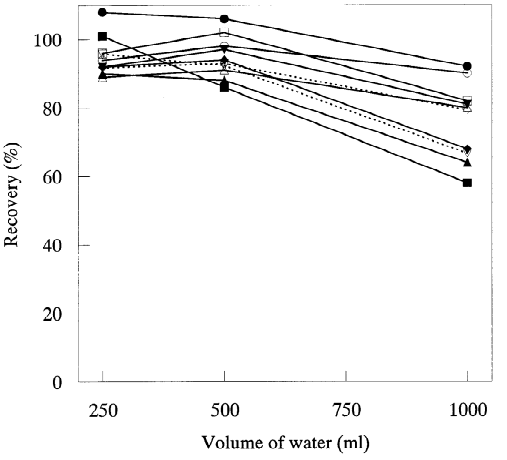

Fig. 4. Recoveries vs. volume of sample water. ■ = Acrylamide; ● = *N*,*N*-dimethylacetamide; ▲ = *N*,*N*-dimethylformamide; ▼ = 1,4-dioxane; ♦ = furfural; □ = furfuryl alcohol; ○ = *N*-nitrosodiethylamine; △ = *N*-nitrosodimethylamine; ▽ = *N*,*N*-dimethylformamide-d_7; ◇ = 1,4-dioxane-d_8.

case of 1000 ml extraction except for *N*,*N*-dimethylacetamide (92%) and *N*-nitrosodiethylamine (90%). The relation between the recoveries from 500 ml of water and the mass of the activated carbon fiber felt is presented in Fig. 5. The felt of 1.5 g was necessary for good recoveries of the compounds. In cases of

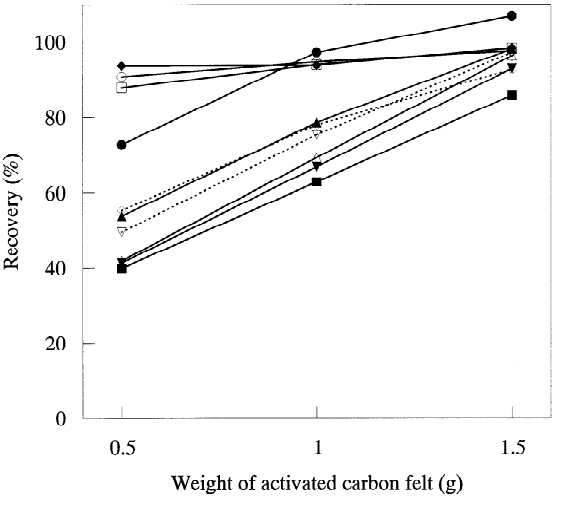

Fig. 5. Recoveries vs. mass of solid phase. ■ = Acrylamide; ● = *N*,*N*-dimethylacetamide; ▲ = *N*,*N*-dimethylformamide; ▼ = 1,4-dioxane; ♦ = furfural; □ = furfuryl alcohol; ○ = *N*-nitrosodiethylamine; △ = *N*-nitrosodimethylamine; ▽ = *N*,*N*-dimethylformamide-d_7; ◇ = 1,4-dioxane-d_8.

granular activated carbon, 1 g of the granule was necessary for extraction of *N*,*N*-dimethylformamide [7] and 1,4-dioxane [11]. Therefore, the felt has rather weaker adsorptive power than the granule. Consequently, sufficient extraction of the compounds in 500 ml water required three extraction cartridges in series. While the use of the three 0.5 g cartridges in series was appropriate to developing new methods, a cartridge containing 1.5 g of the felt will be more suitable for routine extraction procedure because of a lower susceptiblity to leaks and because of a simpler procedure. Hence, we would like to produce cartridges containing more than 0.5 g of felt for further evaluation.

3.5. Recovery test

Overall recoveries of the target compounds from 500 ml of the filtered groundwater and river water were investigated by adding 0.2 μg of each standard compound to the waters, stirring the waters for 10 min, and extracting the compounds from the waters using a C_{18} cartridge and three extraction cartridges in series under the conditions described in Section 2.2. A C_{18} cartridge was attached onto the extraction cartridges to eliminate hydrophobic compounds [1]. Namely, the C_{18} cartridge pre-extracted impurities such as pesticides and hydrocarbons from sample water. Thereby, the eluates from the extraction cartridges were measured clearly without any clean-up procedures [5]. No target compounds and the surrogates were detected from the C_{18} cartridge. The results of recovery test are shown in Table 3. Recoveries from the waters were good (80–100%). The relative standard deviations (RSDs) ranged from 4.5 to 16% for the target compounds. Consequently, the target compounds were determined satisfactorily by this method.

For calculation of minimum detection limits (MDLs) of the target compounds, recoveries from 500 ml purified water spiked with 0.1 μg of each target compound were investigated. The MDLs of the target compounds were estimated by using the results of the recovery study for the lower spiked level ($n=5$) according to the following equation:

$$\mathrm{MDL} = S \times T(n-1, 1-a=0.99)$$

where S is the standard deviation of the replicate analysis in μg/l, a is the level of significance, $T(n-1, 0.99)$ is the T value at the 99% confidence level with $n-1$ degrees of freedom, and n is the number of replicate analyses. The calculated MDLs of the target compounds were 0.02 to 0.03 μg/l as given in Table 1. Calibration curves were linear ($r>0.998$) between 0.03 and 1 ng for *N*,*N*-dimethylformamide and 1,4-dioxane, and 0.02–1 ng for the other target compounds.

3.6. Application to environmental samples

This method was applied to determination of the

Table 3
Overall recoveries of target compounds (0.2 μg)

Compound	Recovery (%)			
	Groundwater		River water	
	Mean (%)	RSD (%)	Mean (%)	RSD (%)
Acrylamide	82	16	80	13
N,*N*-Dimethylacetamide	84	6.9	91	4.5
N,*N*-Dimethylformamide	93	7.6	100	6.1
1,4-Dioxane	95	6.9	98	9.1
Furfural	82	5.5	89	12
Furfuryl alcohol	89	9.3	96	11
N-Nitrosodiethylamine	86	11	84	14
N-Nitrosodimethylamine	83	8.9	82	5.0
N,*N*-Dimethylformamide-d_7	98	8.1	90	9.6
1,4-Dioxane-d_8	97	7.5	92	12

hydrophilic organic compounds in waters from four rivers in the Niigata Prefecture, Japan. Water samples were collected from the Shinano River, the Nishi River, the Shin River and the Agano River in June 2000. Among the target compounds, *N*,*N*-dimethylformamide and 1,4-dioxane were detected; concentrations of *N*,*N*-dimethylformamide and 1,4-dioxane were 0.78 μg/l and 0.23 μg/l at the Shinano River, 0.29 μg/l and 0.13 μg/l at the Nishi River, 0.73 μg/l and 0.18 μg/l at the Shin River, and 0.38 μg/l and 0.17 μg/l at the Agano River, respectively. Concentrations of *N*,*N*-dimethylformamide and 1,4-dioxane in river water or sea water in Japan ranged from <0.1 to 6.6 μg/l and from <0.1 to 16 μg/l, respectively [4,5,22]. The values of *N*,*N*-dimethylformamide and 1,4-dioxane in Niigata were 8–20-times and 70–120-times less than the highest concentrations reported. Although there is no regulation for the target compounds in river water in Japan, Reitz et al. reported an upper bound estimate of $1 \cdot 10^{-5}$ lifetime risk for 1,4-dioxane exposures up to 20 000 μg/l in water [2]. Therefore, the concentrations of 1,4-dioxane in Niigata were estimated to be extremely low risk.

Typical selected ion monitoring (SIM) chromatograms of the compounds are shown in Fig. 6. Every compound was determined without interferences. Method blanks of the target compounds, evaluated by using the purified water (in Section 2.1) were below their MDLs.

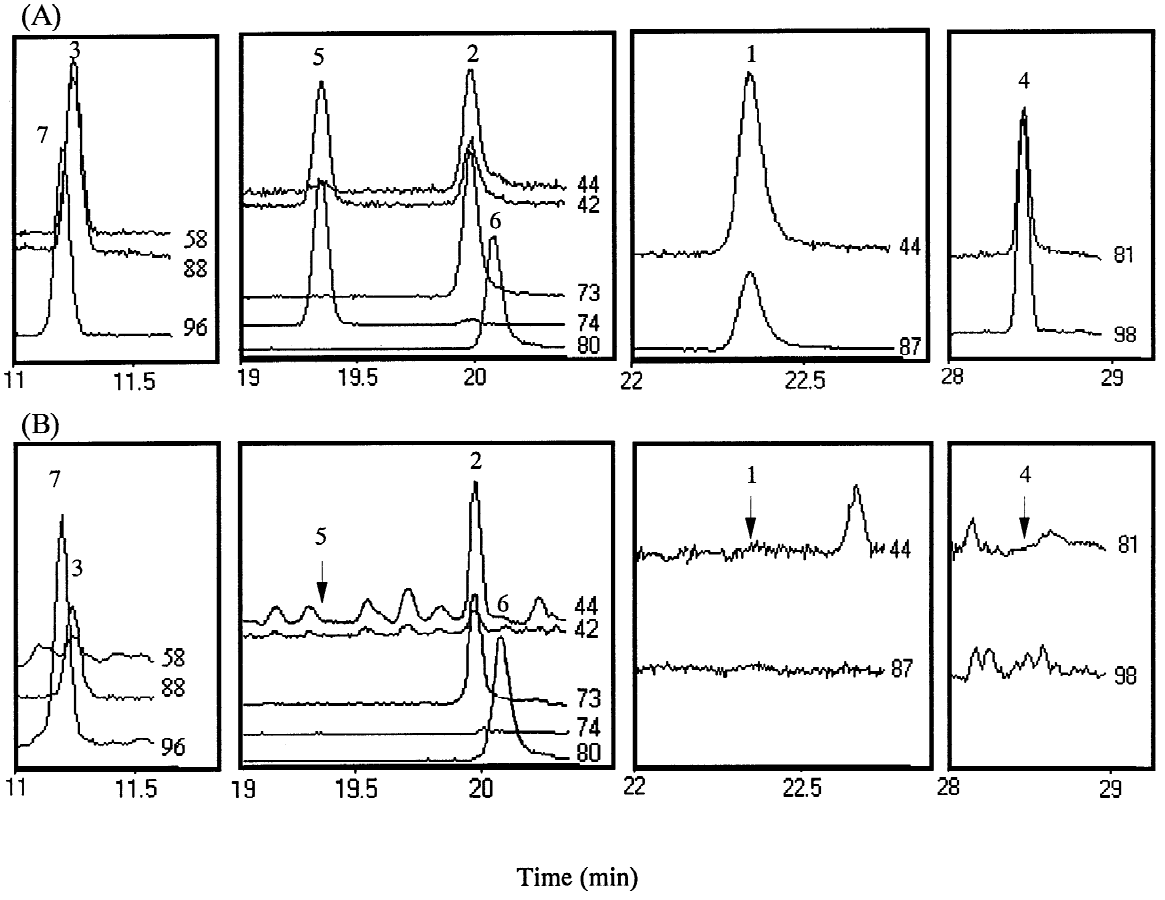

Fig. 6. Selected ion monitoring chromatograms of a standard (A) and a river water sample from the Shinano River (B). 1=*N*,*N*-Dimethylacetamide; 2=*N*,*N*-dimethylformamide; 3=1,4-dioxane; 4=furfuryl alcohol; 5=*N*-nitrosodimethylamine; 6=*N*,*N*-dimethylformamide-d_7; 7=1,4-dioxane-d_8.

References

[1] K. Kadokami, M. Koga, A. Otsuki, Anal. Sci. 6 (1990) 843.
[2] R.H. Reitz, P.S. McCroskey, C.N. Park, M.E. Andersen, M.L. Gargas, Toxicol. Appl. Pharmacol. 105 (1990) 37.
[3] A. Yasuhara, Toxicol. Environ. Chem. 51 (1995) 113.
[4] A. Abe, Sci. Total Environ. 227 (1999) 41.
[5] K. Kadokami, K. Sato, M. Koga, J. Environ. Chem. 3 (1993) 15.
[6] Y. Okamoto, N. Ohashi, H. Sasano, J. Environ. Chem. 2 (1992) 45.
[7] T. Ibaraki, H. Mitobe, K. Kawata, M. Sakai, J. Environ. Chem. 9 (1999) 407.
[8] B.A. Tomkins, W.H. Griest, Anal. Chem. 68 (1996) 2533.
[9] S. Yoshikawa, Y. Shibata, S. Miyajima, Y. Kurosawa, J. Jpn. Soc. Water Environ. 21 (1998) 879.
[10] M. Song, S. Zhang, J. Chromatogr. A 787 (1997) 283.
[11] P.J. Streete, M. Ruprah, J.D. Ramsey, R.J. Flanagan, Analyst (London) 117 (1992) 1111.
[12] A. Abe, J. Environ. Chem. 7 (1997) 95.
[13] V.Y. Taguchi, S.W.D. Jenkins, D.T. Wang, I.-P.F.P. Palmentier, E.J. Reiner, Can. J. Appl. Spectrosc. 39 (1994) 87.
[14] S.W.D. Jenkins, C.J. Koester, V.Y. Taguchi, D.T. Wang, I.-P.F.P. Palmentier, K.P. Hong, Environ. Sci. Pollut. Res. Int. 2 (1995) 207.
[15] C. Crescenzi, A. Di Corcia, G. Passariello, R. Samperi, M. Isabel, T. Carou, J. Chromatogr. A 733 (1996) 41.
[16] S. Suzuki, Bunseki Kagaku 41 (1992) 115.
[17] M. Matsui, K. Kaya, J. Environ. Chem. 2 (1992) 1.
[18] M. Matsui, H. Ohshima, T. Kawabata, Bull. Jpn. Soc. Sci. Fish. 46 (1980) 587.
[19] IARC, in: Monographs on the Evaluation of Carcinogenic Risks to Humans, Supplement 17, IARC, Lyon, 1978, p. 83.
[20] IARC, in: Monographs on the Evaluation of Carcinogenic Risks to Humans, Supplement 17, IARC, Lyon, 1978, p. 125.
[21] A. Tanabe, H. Mitobe, K. Kawata, M. Sakai, A. Yasuhara, J. AOAC Int. 83 (2000) 61.
[22] Chemicals in the Environment, Environmental Agency Japan, 1996.