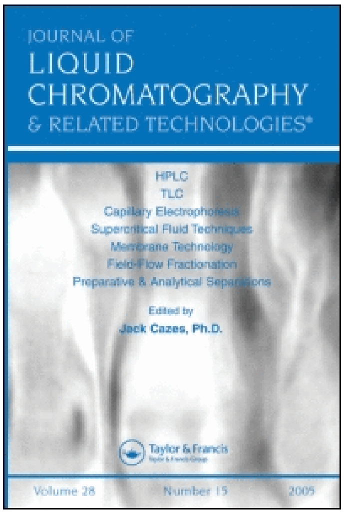

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Susumu Ichinoki[a]; Masashi Kodani[a]; Youichi Fujii[a]
[a] Faculty of Pharmaceutical Sciences, Hokuriku University, Kanazawa, Japan

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Selective and Sensitive Determination of Mercury(II) Ions in River Water by an Automatic HPLC System Combined with On-Line Column Enrichment as 2-Mercaptopyrimidine Chelate

Susumu Ichinoki, Masashi Kodani, and Youichi Fujii

Faculty of Pharmaceutical Sciences, Hokuriku University, Kanazawa, Japan

Abstract: A selective and sensitive determination method for mercury (Hg) ions in river water by reversed phase high performance liquid chromatography (HPLC) has been developed. Two mL of water sample containing Hg(II) ions was put into a 4 mL sample tube. To the sample tube, 500 μL of 2 mol/L (M) acetate buffer solution (pH 4.5), and 50 μL of 0.01 M 2-mercaptopyrimidine (MP) were added to form a neutral Hg chelate. The Hg chelate subsequently became preconcentrated in a reversed phase mini column. By switching the valve, the Hg chelate was separated and detected at 255 nm. These processes occur automatically, except for the addition of water samples, buffer, and MP solutions. The correlation coefficients of the calibration curves obtained with 2 mL of mercury standard were more than 0.999 over the range of 20 ng/mL (ppb) to 10 μg/mL (ppm). The detection limit of Hg ions in 2 mL solution was 1.6 ppb, which corresponded to 3 times the standard deviation ($N = 10$) of the blank peak area. Effects of foreign ions on the determination of 0.2 ppm Hg were investigated with 57 metal ions. Almost none of the ions interfered except for Au(III), Pd(II), Ag(I), and Co(II) ions. The recoveries with spiked river water samples for 5, 0.5, and 0.05 Hg ion ($N = 7$) were 99.7 ± 0.4%, 100.3 ± 0.3%, and 100.7 ± 0.7%, respectively.

Keywords: 2-Mercaptopyrimidine (MP), Column enrichment, High performance liquid chromatography (HPLC), Mercury (Hg) ion, Photometric detection, River water

Correspondence: Susumu Ichinoki, Faculty of Pharmaceutical Sciences, Hokuriku University, Ho 3, Kanagawa-machi, Kanazawa 920-1181, Japan. E-mail: s-ichinoki@hokuriku-u.ac.jp

INTRODUCTION

Mercury (Hg) is well known as one of the most toxic metals. For mercury analysis, inductively coupled plasma atomic emission spectrometry (ICP-AES) and atomic absorption spectrometry (AAS) combined with a reducing vaporization technique are routinely used. Inductively coupled plasma mass spectrometry (ICP-MS) is sometimes used for more sensitive analysis. However, ICP-AES and ICP-MS require expensive apparatuses, and the detection sensitivity of AAS and ICP-AES differ considerably according to the metal. On the other hand, the application of high performance liquid chromatography (HPLC) in the separation and determination of metal ions has increased in recent years.[1–5] The HPLC apparatus is not too expensive and the running cost is very low. In addition, operation of the HPLC is easy and a specific skill is not required. Many metal ions have been determined as metal chelates with various chelating reagents.

The authors have reported metal determination methods by reversed phase HPLC with photometric detection using dithiocarbamate,[6,7] β-diketones,[8,9] salicylideneamino- 2-thiophenol,[10] and α-furil dioxime[11] as precolumn chelating reagents.

The 2-mercaptopyrimidine (MP) was used as a chelating reagent for some metal ions such as lead(II),[12] osmium(VIII),[13] and ruthenium-(IV)[14] ions. On the other hand, the MP was used as an optical chemical sensor for the Hg ion.[15] However, the analytical application of MP for Hg determination by HPLC is not found. We found that MP reacted with the Hg(II) ion, and the Hg-MP chelate was stable in a reversed phase column when a certain amount of MP was added into an eluent.

In this paper, pretreatment and HPLC conditions were studied for sensitive determination of Hg(II) ions in river water. The linear working range of calibration curves and detection limits were also investigated. The effect of 57 foreign ions on the determination of Hg ions was also examined. The concentration of Hg ions in a spiked river water sample were determined by the HPLC method.

EXPERIMENTAL

Instrumentation

The HPLC system consisted of two Shimadzu (Kyoto, Japan) LC-10A*i* pumps (inert pump, as pump A and B), a Shimadzu SCL-10A system controller, a Shimadzu SIL-10A auto injector, a Shimadzu SPD-10AV *VP* photometric detector, a Shimadzu PCV-12AH six port valve, a Develosil Ph-UG-5 enrichment column (10 × 4.0 mm ID, stainless steel, Nomura Chemical Co. Ltd., Seto, Japan), a Cosmosil 5 PE-MS (phenyl ethyl, monomeric type) analytical column (150 × 4.6 mm ID, stainless steel, Nacalai Tesque, Kyoto, Japan), and a Shimadzu C-R4A integrator. Micropipettes were used for 2.5 mL or less volume of solutions.

A Develosil ODS-UG-5 enrichment column (10 × 4.0 mm ID), a Cosmosil 5 C_{18} AR-II analytical column (250 × 4.6 mm ID), and a Thermo Minder SX-10 R thermostat water bath (Taitec Co., Koshigaya, Japan) were used for a screening test.

Reagents

All reagents used were of analytical reagent grade unless otherwise stated. Milli-Q water was used for aqueous solution preparation. Nitric acid was of super special grade for toxic metal analysis (Wako Pure Chemicals Industries, Osaka, Japan). Methanol was distilled and filtered through a membrane filter (pore size, 0.45 µm). The chelating reagent 2-mercaptopyrimidine (MP) was obtained from Tokyo Kasei Kogyou Co. Ltd. (Tokyo, Japan). Further details are shown in Figure 1. A 0.01 mol/L (M) MP solution was prepared as follows: the MP of 0.112 g was dissolved in 20 mL of 2 M ammonia- 2 M ammonium chloride buffer solution (pH 9.0), and diluted to 100 mL with water. The 0.01 M MP was preserved in a refrigerator (about 4°C). A 2.0×10^{-4} M MP was prepared by dilution of the 0.01 M MP with water. All metal standard solutions of 1000 µg/mL (ppm) for atomic absorption spectrometry were obtained from Kanto Chemical Co., Inc. (Tokyo, Japan). The 1000 ppm Hg standard solution consists of $HgCl_2$ and 0.1 M HNO_3. The other Hg standards were prepared by dilution of the above solution (1000 ppm) with 0.1 M HNO_3. Acetate buffer solutions (pH 3.0 to 6.0) were prepared with 2 M acetic acid and 2 M sodium acetate. Ammonia-ammonium chloride buffer solutions (pH 8.0 to 11.0) were prepared with 2 M ammonia and 2 M ammonium chloride solutions. River water was collected from the Tomuro River (branch of the Asano River, Kanazawa, Japan). The river water was filtered through a membrane filter (0.45 µm pore size), and used as the river water sample for HPLC analysis.

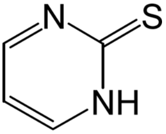

Figure 1. 2-mercaptopyrimidine (MP). $C_4H_4N_2S = 112.15$, CAS No. 1450-85-7.

Recommended Procedure and HPLC Conditions

Put a sample solution or an Hg standard solution of 2.0 mL into a 4 mL sample tube with a stopper. Add 500 μL of 2 M acetate buffer solution (pH 4.5) and 50 μL of 0.01 M MP solution, and stopper the tube. Shake the contents for 10 seconds by hand, and leave standing for at least for 1 hour. Then, determine the Hg-MP chelate under the following HPLC conditions and a time program shown in Table 1.

Enrichment and analytical columns were described above. Eluent B (for enrichment): methanol/water/0.01 M MP (10:90:0.5, v/v), eluent A (for analysis): methanol/water/0.01 M MP (54:46:0.5, v/v), flow rate of eluent A and B: 1.0 mL/min. Injection volume: 1500 μL, detection wavelength: 255 nm, analysis time of HPLC (stop time): 10.3 min, column temperature: ambient, conditioning time: 3 min, rinse time: 6 min.

Chelate Composition of Hg-MP Chelate

To a sample tube, 2000 μL of water, x μL of 2.0×10^{-4} M Hg standard solution, $(200 - x)$ μL of 2.0×10^{-4} M MP, 500 μL of 2 M acetate buffer solution of pH 4.5 were added. After shaking for 10 seconds by hand, the peak area of Hg-MP chelate was measured by the recommended procedure. Where, (x, y) were (0, 200), (20, 180), (40, 160), (60, 140), (66.7, 133.3), (80, 120), (100, 100), (120, 80), (140, 60), (160, 40), (180, 20), and (200, 0). The mole fractions of [Hg]/([Hg] + [MP]) were 0, 0.1, 0.2, 0.3, 0.333, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, and 1.0, respectively.

Effects of Foreign Ions

The effects of foreign ions on the determination of 0.2 ppm Hg ion were tested with 57 metal ions. Each foreign metal ion and 80 μL of 5 ppm Hg

Table 1. Time program for automatic preconcentration and HPLC analysis

Time (min)	Command	Value	Action
5.90	ZERO A		Adjust absorbance to zero
6.00	RV. A	1	Change valve for elution position
6.00	START		Start integrator
15.00	RV. A	0	Change valve for concentration position
15.00	S. PRET		Start sample injection procedure
18.00	STOP		Stop time program (Analysis is repeated.)

The time program is prepared for a Shimadzu SCL-10A system controller.

standard were put into a sample tube, and diluted to 2000 µL with water. To the sample tube, 500 µL of 2 M acetate buffer solution of pH 4.5 and 50 µL of 0.01 M MP were added. After standing at least for 60 minutes, the concentration of Hg ion in the solution was determined by the recommended procedure and HPLC conditions. The recovery percentage was calculated from the peak area and that of the Hg standard containing no foreign metal ions.

Recovery Tests with a River Water Sample

A river water sample of 1800 µL and 200 µL of Hg standard were put into a sample tube. The concentrations of the Hg standards were 50, 5, and 0.5 ppm. Thus, the concentrations of added Hg ion in the 2 mL solutions were 5, 0.5, and 0.05 ppm. To other sample tubes, water and Hg standards of varying concentration were added, and the calibration curves were prepared by the recommended procedure. The Hg concentrations in spiked river water samples were determined from the peak areas and the equations of calibration curves, and recovery percentages were calculated from the results.

RESULTS AND DISCUSSION

Sample Pretreatment and Enrichment Conditions

The screening test indicated that Hg, cobalt (Co), and palladium (Pd) ions were extracted as metal-MP chelates from a weak alkaline solution, and gave chromatographic peaks as shown in Figure 2 (a). The chromatogram a) was obtained with a Cosmosil 5 C_{18} AR-II analytical column (250 × 4.6 mm ID) and an eluent of methanol/water/0.01 M MP (55:44:1, v/v). Thus, the effect of reaction pH on peak area of the metal chelates was examined, and the results are shown in Figure 3. Constant peak areas were obtained for Hg ion in the range of pH 3 to 9, and the 2 M acetate buffer solution of pH 4.5 was employed for determination of Hg ion considering the buffer capacity. Because the Co peak did not appear under the conditions, a shorter column was employed. The chromatograms of Figure 2b, c, and d were obtained with Cosmosil 5 PE-MS column (150 × 4.6 mm ID) and recommended procedure and HPLC conditions.

The effect of the MP amount on the peak area of Hg chelate was also investigated over the range of 5 to 300 µL. As constant peak areas were obtained in the range of 10 to 150 µL, 50 µL was employed in the recommended procedure.

Effect of the reaction time was also investigated for 20 to 120 min. The results indicated that the peak areas obtained from 20 and 40 min

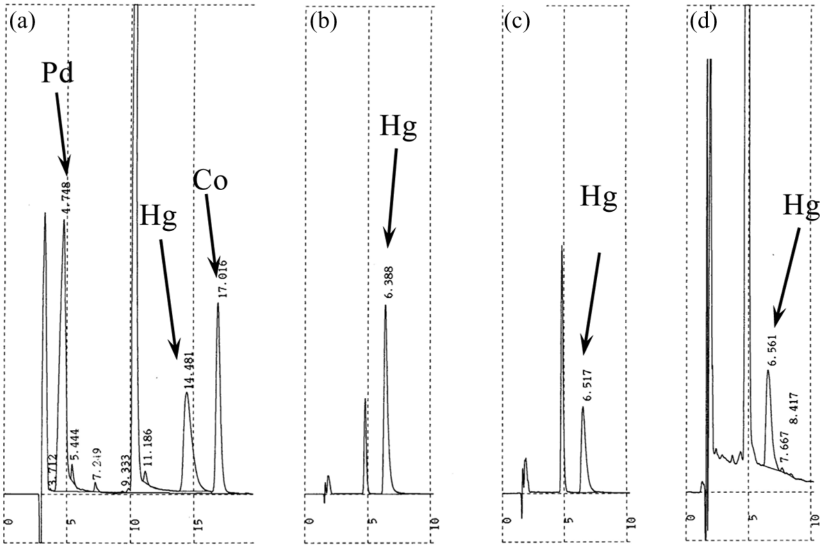

Figure 2. Chromatograms of metal-MP chelates. (a): Pd-, Hg-, and Co-MP chelates extracted at pH 9, (b): 5 ppm Hg, (c): 0.5 ppm Hg, (d): 0.05 ppm Hg. The chromatograms of b, c, and d were obtained with Hg spiked river water samples. The spiked Hg ion was extracted as Hg-MP chelate at pH 4.5. More details are in the text.

were slightly small compared with 60 to 120 min. Thus, 60 min or more time is required for quantitative chelation of Hg-MP.

Before injection of a pretreated sample solution, the six port valve should be switched to concentration position, and the eluent B should

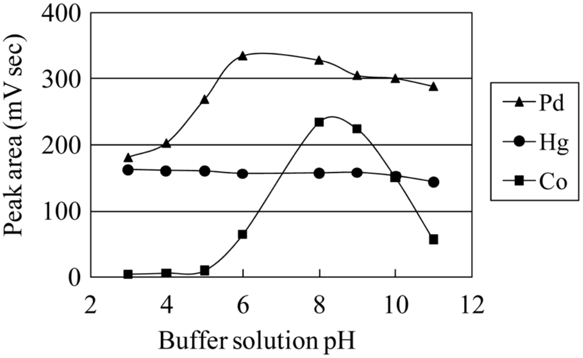

Figure 3. Effect of reaction pH on peak area of the metal-MP chelates. Each peak area was plotted against the pH of the buffer solution. Column: Cosmosil 5 C_{18} AR-II analytical column (250 × 4.6 mm ID), eluent A: methanol/water/0.01 M MP (55:44:1 v/v), eluent B: water/0.01 M MP (99:1 v/v), detection wavelength; 255 nm.

be made to flow to the enrichment column for several minutes, otherwise, a part of the Hg chelate was eluted from the enrichment column due to the high methanol content in the eluent A. This conditioning time should be optimized. After injection of the pretreated sample solution into a sample loop, the eluent B was made to flow for several minutes in order to inject the sample solution into the enrichment column, and to elute large amounts of buffer solution and MP. This rinse time was also investigated with 1 ppm Hg standard according to the recommended procedure.

The results of the conditioning and rinse times are shown in Figure 4. By the results of Figure 4, conditioning time and rinse time were set at 3 and 6 min, respectively.

HPLC Separation Conditions

The composition of eluent A (methanol/water/0.01 M MP) was investigated concerning the retention time of Hg chelate, stability of Hg, and Pd chelates. Addition of the MP into eluent is required for stabilizing

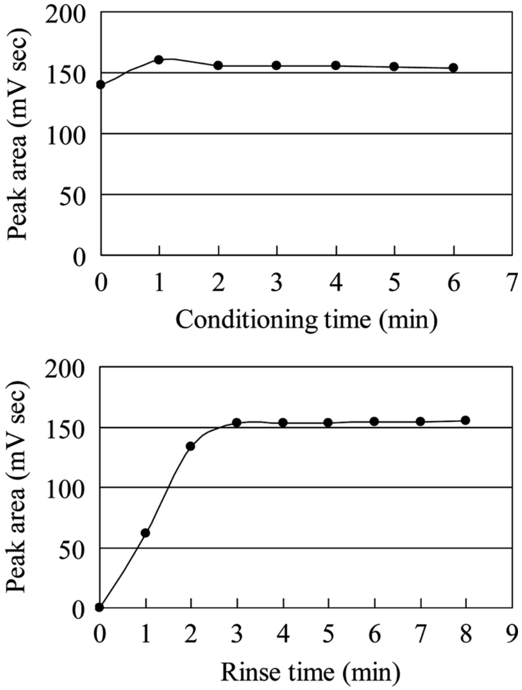

Figure 4. Effects of conditioning time and rinse time on peak areas of Hg-MP chelate. Concentration of Hg(II) ion in a 2 mL solution was 1 ppm. The other conditions are in the text.

the Hg chelate. However, higher concentration of MP gave large base line noise and Pd chelate peak. Thus, the eluent of methanol/water/0.01 M MP (54:46:0.5, v/v) was employed as eluent A. The composition of the eluent B (for enrichment) was also investigated with the methanol/water/0.01 M MP mixtures of x: (100−x): 0.5, where x was 0, 10, 20, and 30. The mixture of methanol/water (10:90:0.5, v/v) was selected for eluent B, because the best peak shape of the Hg chelate was obtained.

The detection wavelength was set at 255 nm, which is the maximum absorption wavelength of the Hg-MP chelate. The Cosmosil 5 PE-MS (150 × 4.6 mm ID) column was used at ambient temperature, because column pressure was not so high.

Hg-MP Chelate Composition

The composition of the Hg-MP chelate was investigated by Job's method. The peak areas were plotted against the mole fractions of [Hg]/([Hg] + [MP]) as shown in Figure 5. The maximum peak area was obtained at a mole fraction of 0.333 (that is [Hg]:[MP] = 1:2). The results indicated that the MP ionized to H^+ and MP^-, then reacted with the Hg^{2+} ion to form $Hg(MP)_2$ chelate.

Calibration Curve and Detection Limit

Calibration curves for Hg(II) ions were prepared with an Hg standard of varying concentrations according to the recommended procedure and HPLC conditions. The correlation coefficients of the calibration curves were more than 0.999 over the concentration range of 20 ng/mL (ppb)

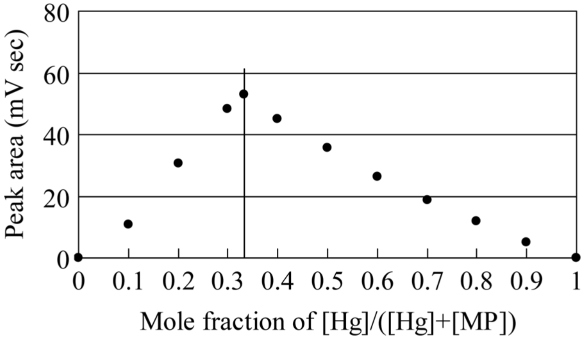

Figure 5. Determination of Hg-MP chelate composition by Job's method. Experimental conditions are in the text.

Table 2. Calibration curves for Hg(II) ion

Concentration range	Equation of line	Correlation coefficient	Measuring point (ppm)
1–10 ppm	$y = 28.614x - 0.3212$	0.9999	0, 1, 2, 4, 6, 8, 10
0.1–1 ppm	$y = 88.172x - 2.4189$	0.9999	0.1, 0.2, 0.4, 0.6, 0.8, 1.0
0.02–0.1 ppm	$y = 763.15x - 7.0028$	0.9991	0.02, 0.04, 0.06, 0.08, 0.10
Calibration curves for Hg ion obtained on another days			
1–10 ppm	$y = 28.586x - 1.5802$	0.9999	0, 1, 2, 4, 6, 8, 10
0.1–1 ppm	$y = 88.796x - 2.0153$	0.9999	0.1, 0.2, 0.4, 0.6, 0.8, 1.0
0.02–0.1 ppm	$y = 773.7x - 7.8413$	0.9992	0.02, 0.04, 0.06, 0.08, 0.10

y: peak area (mV sec), x: concentration of Hg ion (ppm).

to 10 ppm, as shown in Table 2. The correlation coefficients of the calibration curves obtained on other days were more than 0.999. The wide linear range was compared with that of ICP-AES.

The detection limit of the Hg ion in a 2 mL solution was 1.6 ppb, which corresponded to 3 times the standard deviation ($N = 10$) of the blank peak areas.

Effects of Foreign Ions

The effects of 57 foreign metal ions on the determination of the 0.2 ppm Hg(II) ion (2 mL) were investigated. Table 3 shows that 48 metal ions did

Table 3. Effects of foreign metal ions on determination of 0.2 ppm Hg(II) ion

Tolerance limit	Metal ion
200 ppm	Al(III), As(III), Ba(II), Be(II), Ca(II), Cd(II), Cs(I), Ce(III), Cr(VI), Cs(I), Cu(II), Dy(III), Er(III), Eu(III), Fe(III), Ga(III), Gd(III), Ge(IV), Ho(III), In(III), K(I), La(III), Lu(III), Mg(II), Mn(II), Mo(VI), Na(I), Nb(V), Nd(III), Ni(II), Pb(II), Pr(III), Pt(IV), Rh(III), Se(IV), Si(IV), Sm(III), Sr(II), Ta(V), Tb(III), Te(IV), Tl(I), Tm(III), V(V), W(VI), Y(III), Yb(III), Zr(IV)
100 ppm	Zn(II)
40 ppm	Sb(III), Sc(III)
20 ppm	Bi(III), Ti(IV), Sn(II)
2 ppm	Co(II)
1 ppm	Ag(I)
0.1 ppm	Au(III), Pd(II)

Note: The tolerance limit value of the foreign ion concentration was taken as the value that caused an error of less than 10% in the recovery of Hg(II) ion (0.2 ppm).

Table 4. Recovery tests for Hg ion with a river water sample

No. of run	Sample (ppm)	Added (ppm)	Found (ppm)	Recovery (%)	Equation of calibration curve	Correlation coefficient
5 ppm						
1	0.00	5.00	4.98	99.6	$y = 26.779x - 0.3852$	0.9997
2	0.00	5.00	4.96	99.1		
3	0.00	5.00	4.97	99.4		
4	0.00	5.00	5.01	100.3		
5	0.00	5.00	5.00	100.0		
6	0.00	5.00	4.99	99.8		
7	0.00	5.00	4.99	99.9		
Av.[a]	0.00	5.00	4.99	99.7		
SD[b]			0.02	0.4		
RSD[c]			0.4	0.4		
0.5 ppm						
1	0.000	0.500	0.505	100.9	$y = 80.436x - 4.557$	0.9995
2	0.000	0.500	0.500	99.9		
3	0.000	0.500	0.501	100.3		
4	0.000	0.500	0.501	100.2		
5	0.000	0.500	0.499	99.8		
6	0.000	0.500	0.505	100.9		
7	0.000	0.500	0.501	100.1		
Av.[a]	0.000	0.500	0.502	100.3		
SD[b]			0.002	0.4		
RSD[c]			0.4	0.4		
0.05 ppm						
1	0.0000	0.0500	0.0509	101.9	$y = 1054.1x - 7.0617$	0.9991
2	0.0000	0.0500	0.0505	101.0		
3	0.0000	0.0500	0.0498	99.6		
4	0.0000	0.0500	0.0505	100.9		
5	0.0000	0.0500	0.0505	100.9		
6	0.0000	0.0500	0.0501	100.2		
7	0.0000	0.0500	0.0503	100.6		
Av.[a]	0.0000	0.0500	0.0504	100.7		
SD[b]			0.0004	0.7		
RSD[c]			0.7	0.7		

Detector response was changed with the Hg concentration.

[a] Average.

[b] Standard deviation.

[c] Relative standard deviation (%).

not interfere at 1000 times (200 ppm), or more, the concentration of the Hg ion. The tolerance limits of Au(III) and Pd(II) were 0.1 ppm. The Ag ion reacted with the chloride ion of Hg standard, and formed a white precipitant of AgCl, because the Hg standard consisted of $HgCl_2$ and 0.1 M HNO_3. More details are shown in Table 2.

Recovery Tests with River Water Sample

Hg concentrations in the collected river water were lower than detection limits (1.6 ppb) of the presented HPLC method. Therefore, Hg standards were added to the river water sample, and the Hg ions in the solution were analyzed. Table 4 shows the results of the recovery test.

The recoveries obtained for 5, 0.5, and 0.05 ppm Hg ions were 99.7 ± 0.4, 100.3 ± 0.4, and 100.7 ± 0.7%, respectively. As shown in Table 4, correlation coefficients of the calibration curves were more than 0.999. The recoveries obtained on other days were 98.5 ± 0.7% for 5 ppm, 100.3 ± 0.2% for 0.5 ppm, and 99.6 ± 1.3% for 0.05 ppm Hg, respectively. Thus, the two results obtained on other days showed good agreement with each other. The high recovery indicated that the ions in typical river water did not interfere with the Hg determination.

CONCLUSION

In this paper, an automatic HPLC method for the ppm level of Hg(II) ion determination has been shown. In this method, the Hg ion is preconcentrated as the Hg-MP chelate. The enrichment and HPLC analysis carried out automatically. The HPLC method was applied to the determination of the ppm levels of Hg ions in spiked river water samples with precise results.

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