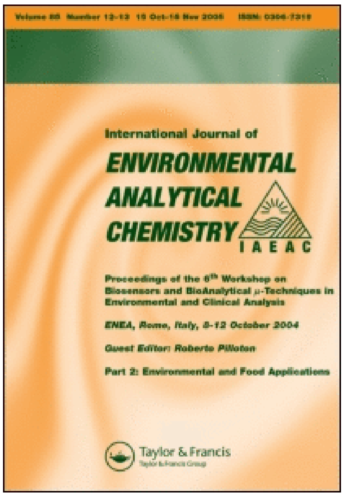

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Determination of bismuth in natural water samples by ICP-AES after preconcentration on dithiocarbamates-coated amberlite XAD-7

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A column solid-phase extraction procedure for separation and preconcentration of bismuth in natural water samples using sodium diethyldithiocarbamate (NaDTTC) or piperidene dithiocarbamate (pipDTC)-coated Amberlite XAD-7 resin prior to their determination by inductively coupled plasma atomic emission spectrometry (ICP-AES) is reported. The results showed that solution pH and flow rate would affect the sorption of bismuth. The sorbed bismuth was eluted with 4 M/L of HNO_3. The extractor system has good sorption capacity values of 9.82 mg/g of Bi on NaDTTC-coated resin and 9.56 mg/g of Bi on pipDTC-coated resin. The preconcentration factor was 150 and 170 for pipDTC-coated resin and NaDTTC-coated resin, respectively. The detection limits are 0.9 and 1.2 μg/L for resin-coated with NaDTTC and pipDTC, respectively. The results showed that resin coated with NaDTTC performs slightly better in the recovery of bismuth than resin coated with pipDTC. The accuracy of the proposed method was investigated by determining the bismuth in spiked water samples. The relative standard deviations of the determination of bismuth were below 5%. The proposed procedure was applied for the determination of bismuth in natural water and seawater samples.

Keywords: Bismuth; Piperidine dithiocarbamate; Sodium diethyldithiocarbamate; Inductively coupled plasma atomic emission spectrometry; Preconcentration

1. Introduction

Bismuth-containing compounds have been used for different medicinal purposes, especially for the treatment of syphilis, gastritis, and ulcer. As the use of bismuth in medicines is increasing, it has spread in the environment, and the exposure of organisms to

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bismuth has increased [1]. However, a number of toxic effects in humans have been attributed to bismuth compounds such as nephropathy, osteoarthropathy, hepatitis, and neuropathology [2]. The concentration of bismuth in natural waters is very low; hence the determination of bismuth by spectroscopic methods requires a suitable preconcentration procedure. The preconcentration step not only provides lower detection limits but also helps in separating analyte from the matrix interferences. Several preconcentration methods like co-precipitation [3], liquid–liquid extraction [4, 5], ion exchange [6], and solid-phase extraction [7, 8] are used for the determination of bismuth in various matrices. Among the above methods, solid-phase extraction has received more attention because of a number advantages including preconcentration from a larger sample volume, establishing higher concentration factors, simple storage, and transportation of pretreated sample [9].

Chelating sorbents are important metal ion collectors and suitable for metal-ion enrichment because of their higher selectivity and sensitivity. Hence, there is a growing need to develop selective chelating sorbents in current research. Therefore, several workers attempted various sorbents like, silica gel [10], C-18 [11] activated carbon [12–14], polyurethane foam [15–17], and Amberlite XAD resins [18–22] immobilized with suitable reagents used as solid supports to preconcentration and separation of bismuth from various matrices. Based on good physical properties like porosity, uniform pore size distribution, high surface area, and the chemically homogeneous non-ionic structure of Amberlite XAD resins, several authors used these resins functionalized with a variety of chelating ligands for the preconcentration of trace metals [23–25]. It was, therefore, thought worthwhile to modify Amberlite XAD-7 resin by attaching the dithiocarbamates and investigate the bismuth uptake behaviour of the new matrix. The strong metal-binding property of dithiocarbamates combined with the insolubility of metal complexes and the capacity of molecules to form chelate complexes are the advantages for the wide application of dithiocarbamate compounds as ligands in heavy-metal determination.

In the present work, a simple and sensitive separation and preconcentration method for the determination of bismuth from aqueous samples using Amberlite XAD-7 resin coated with sodium diethyl dithiocarbamate (NaDTTC) or piperidene dithiocarbamate (pipDTC) by inductively coupled plasma atomic emission spectrometry (ICP-AES) has been developed. Ramesh *et al.* [26] have studied the preconcentration of trace elements on a support, polyurethane foam impregnated with pipDTC and reported a good preconcentration factor for different trace metals. The developed method has been applied to the determination of trace amounts of bismuth in natural water and seawater samples. The accuracy of the proposed method has been tested with standard reference materials.

2. Experimental

2.1 *Instrumentation*

Atomic emission spectrometer (ICP-AES, Varian Liberty Series (II), Australia) with a Wipro Acer computer was used. The operating conditions and wavelengths of emission lines are given in table 1. A pH meter with combined glass electrode (LI-129) was employed for measuring pH values.

Table 1. ICP-AES operating parameters.

Photomultiplier tube voltage (V)	700
Plasma glass flow (L/min)	15.0
Auxiliary gas flow (L/min)	1.5
Incident power (kW)	1.1
Observation height (mm)	14.0
Sample uptake time (s)	30
Pump rate (rpm)	15
RF generator (MHz)	27.12
Wavelength of bismuth	223.061

2.2 *Chemicals and reagents*

All the reagents used were of analytical grade. Deionized, doubly distilled water was used throughout the experimental study. The Amberlite XAD-7 resin (Rohm & Hass, Aldrich) with a particle size of 20–50 mesh and surface area of 450 m^2/g was used. Before use, the resin was washed with methanol-4 M/L HCl to remove the metal impurities. Sodium diethyldithiocarbamate was obtained from Fluka. HNO_3 (Glaxo Exeelah, India) was used without additional purification.

Sodium salt of piperidine dithiocarbamate was prepared by slowly adding 80 g of carbon disulphide to a solution of piperidine (85 g) in 25 mL of water at 5°C with constant stirring, followed by 40 g of sodium hydroxide dissolved in 20 mL of water. The product was warmed to room temperature and washed repeatedly two or three times with purified acetone. Finally, the reaction product was purified by recrystallization in acetone. A standard solution of bismuth (100 µg/mL) was prepared from the dilution of ICP standard (Merck, Germany), and the working standards were prepared by diluting the standard solution.

The glass column (with a stopcock), 15 cm long and 0.90 cm in diameter, was used. In the lower part of the column, a small amount of glass wool was used to prevent the loss of resin during the sample loading. The column was conditioned by acetate buffer solution before passing the sample solution.

The acetate buffer solution was prepared by mixing of 8.2 g of sodium acetate in 800 mL of distilled water, and the pH was adjusted to 5.0 with glacial acetic acid.

2.3 *Preparation of chelating resin*

The washed Amberlite XAD-7 resin (25 g) was added to 100 mL of aqueous solution containing 0.03 M NaDTTC or pipDTC, respectively. This solution was shaken at room temperature for 2 h, filtered, and washed with deionized water. Then, the washed resin was dried at room temperature for 24 h in a vacuum dry oven.

2.4 *Preconcentration procedure*

One gram of pipDTC or NaDTTC-coated Amberlite XAD-7 resin was loaded in the column. One hundred millilitres of water samples containing bismuth in the concentrations of 0.1–200 µg/L filtered through a 0.45 µm membrane filter was taken, and the pH was adjusted to the desired value by 1 : 1 HCl or 1 : 1 ammonia followed by 2 mL of acetate buffer. Then, the sample solution was passed through the column at a flow rate of 2 mL/min. After passing this solution, the column was washed with 10 mL of distilled water. Thereafter, the metal ions sorbed on the resin were eluted with 10 mL of 4 M/L HNO_3 at a flow rate of 2 mL/min and determined by ICP-AES. The operating parameters of ICP-AES are given in table 1.

2.5 *Determination of trace metals in natural water and sea water samples*

Natural water and seawater samples were collected in screw-cap polyethylene bottles from two different places in and around Tirupati and Bay of Bengal near Pulicot Lake in south India, respectively. The samples were acidified with 5 mL of concentrated nitric acid per litre of sample and filtered through a 0.45 µm cellulose membrane filter. One hundred millilitres of the sample was taken, and the pH was adjusted to 5.0 ± 0.2 with 1 : 1 HCl or 1 : 1 ammonia followed by 2 mL of acetate buffer and passed through the pipDTC-coated Amberlite XAD-7 resin at a flow rate of 2 mL/min. After passing this solution, the column was washed with 10 mL of deionized water. Thereafter, the metal ions sorbed on the resin were eluted with 10 mL of 4 M/L HNO_3 at a flow rate of 2 mL/min and determined by ICP-AES.

3. Results and discussion

3.1 *Effect of pH*

The effect of pH on the retention of bismuth was studied by passing 50 µg of metal through the dithiocarbamates-coated Amberlite XAD resin (1.0 g) loaded columns at different pH values and determined as described in the general procedure. The data are presented in figure 1. The results demonstrated that over 95% recovery of bismuth

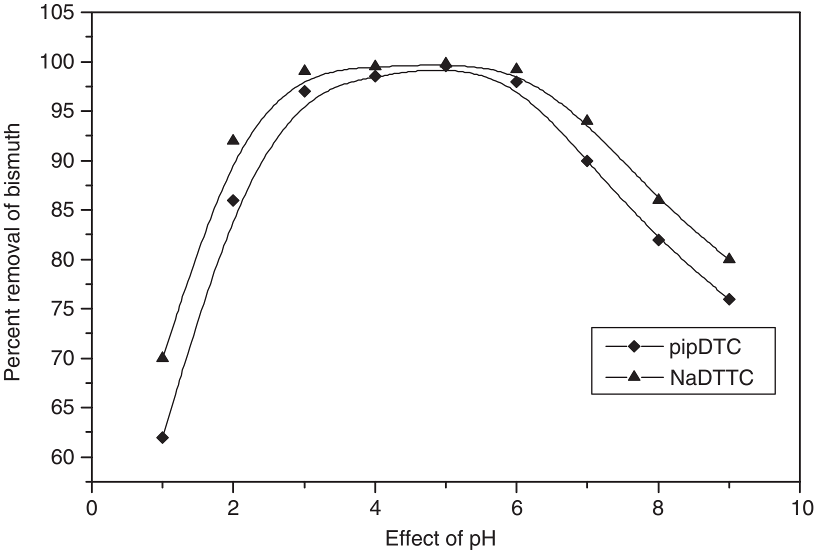

Figure 1. Effect of pH on complexation of bismuth with NaDTTC or pipDTC-coated Amberlite XAD-7 resin.

was obtained in the pH range of 3.0–6.0 and maximum at pH 5.0. Hence, pH 5.0 was used for further studies.

3.2 *Effect of flow rate on retention and recovery of bismuth*

The effect of flow rate on the retention and recovery of bismuth on dithiocarbamates-coated Amberlite XAD-7 resin was examined by varying the flow rate from 2.0 to 5.0 mL/min under optimal conditions. The results showed that, in the range of 2.0–5.0 mL, the retention of bismuth (>95%) is not affected considerably by the sample flow rate. On the other hand, the quantitative recovery of bismuth was needed for larger volumes of 4 M/L HNO_3 at high flow rates. Hence, a 2.0 mL/min flow rate was used for elution of bismuth from resin. Flow rates slower than 2 mL/min were not studied so as to avoid long analysis times.

3.3 *Kinetics of sorption*

The rate of uptake of bismuth on NaDTTC or pipDTC-coated Amberlite XAD-7 was studied by the batch method. Ten milligrams of bismuth was added to 1.0 g of resin and stirred for 2, 5, 10, 20, 30, 40, 50, 60, 90, and 120 min at room temperature. The concentration of metal ions in the supernatant was analysed by ICP-AES as described in the standard procedure. The saturation of resin with time is graphically presented in figure 2. The results in the figure indicate that the time needed to reach 50% of loading capacity ($t_{1/2}$) is almost the same both for the NaDTTC-coated resin and for the pipDTC-coated resin.

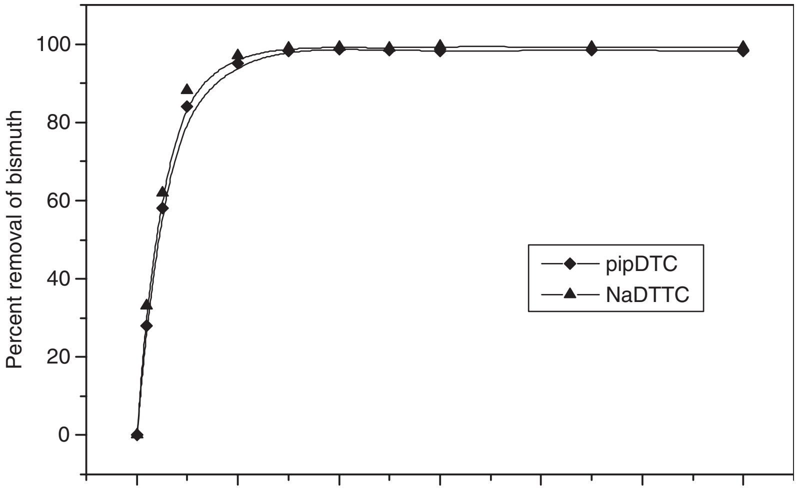

Figure 2. Effect of contact time on percentage removal of bismuth.

Table 2. Experimental results of modified Amberlite XAD-7 resins.

	Experimental results of bismuth	
Experimental parameter	Resin coated with NaDTTC	Resin coated with pipDTC
Sorption capacity (mg/g)	9.82	9.56
Preconcentration factor	170	150
Detection limit (ng/mL)	0.9	1.2
Loading half time ($t_{1/2}$) (min)	3.8	4.1
Flow rate (mL/min)	2.0	2.0

3.4 *Sorption capacity*

The sorption capacity of the resin was calculated by passing increasing concentrations of bismuth solution in 1.0 g of NaDTTC or pipDTC-coated Amberlite XAD-7 resin containing columns. The sorbed bismuth was eluted with 4 M/L HNO_3 at a flow rate of 2 mL/min and determined as described in the general procedure. The results are presented in table 2. These results demonstrated that NaDTTC-coated Amberlite XAD-7 resin has a marginally higher adsorption capacity than pipDTC-coated Amberlite XAD-7 resin.

3.5 *Preconcentration factor*

The preconcentration factor was examined by passing the increasing volumes of bismuth solution into 1.0 g of NaDTTC or pipDTC-coated resin, keeping the total bismuth amount at 10 µg, and analysed by general procedure. The results showed that the recovery values were above 95% up to 1700 and 1500 mL of sample volumes for NaDTTC and pipDTC-coated Amberlite XAD-7 resins, respectively, and the results showed that the NaDTTC-coated resin has a slightly higher preconcentration factor than the pipDTC-coated resin.

3.6 *Effect of HNO_3 concentration on elution*

The bismuth–dithiocarbamate complex elution from NaDTTC or pipDTC-coated Amberlite resin was studied using different strengths of HNO_3 at a flow rate of 2 mL/min, and the results are presented in figure 3. These data indicate that the highest recovery of bismuth was obtained at an HNO_3 concentration of 4 M/L. Hence, 4 M/L HNO_3 is used for further experimental studies.

3.7 *Effect of matrix ions*

The effect of matrix ions on the sorption of bismuth was studied. Different amounts of various cations and electrolytes were added to the 50 µg of bismuth solution in 100 mL and the solution passed through the column containing 1.0 g of pipDTC-coated Amberlite XAD-7 resin. The concentration which caused ±2% error is given table 3. The results in table 3 demonstrate that the anions and cations do not affect the sorption of bismuth under the reported conditions. The alkaline and alkaline earth metals were almost not retained at all through the column because of the low solubility constant of their chelates, and the small amounts of retained matrix ions were removed by washing

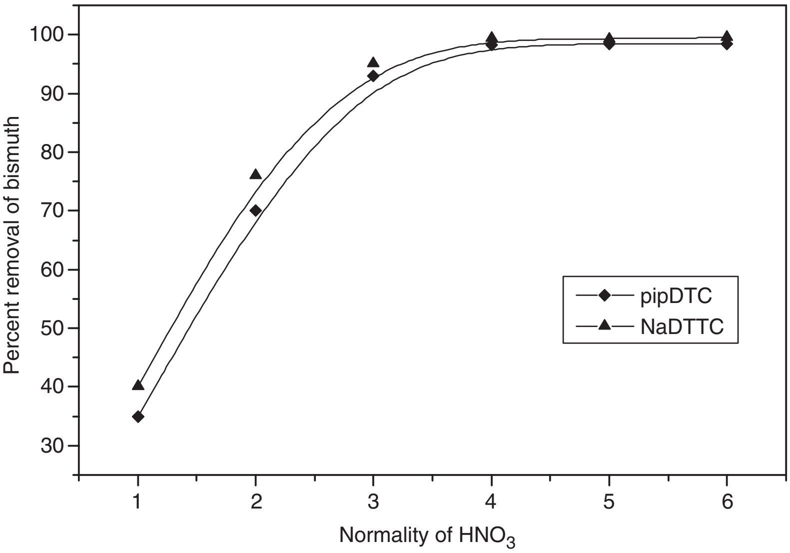

Figure 3. Effect of HNO_3 concentration on elution of bismuth.

Table 3. Tolerance limits of matrix ions for the recovery of bismuth.

Matrix ion	Tolerance limit (μg/mL)
Ca^{2+}	40 000
Mg^{2+}	35 000
Na^+	50 000
K^+	60 000
SO_4^{2-}	40 000
Cl^-	60 000
NO_3^-	40 000

Table 4. Recovery of bismuth from spiked water samples after preconcentration on NaDTTC- and pipDTC coated Amberlite XAD-7 resins.

Spiked bismuth concentration	Resin coated with NaDTTC			Resin coated with pipDTC		
(μg/L)	Found (μg/L)	Recovery (%)[a]	RSD (%)[a]	Found (μg/L)	Recovery (%)[a]	RSD (%)[a]
10	9.936	99.36	3.38	9.796	97.96	3.36
25	24.82	99.28	3.14	24.30	97.22	3.74
50	49.74	99.48	3.56	49.07	98.15	3.62
100	99.13	99.13	3.26	98.04	98.04	4.55

[a] Percentage recovery and percent RSD for five determinations.

with 10–15 mL of buffer solution, as reported by Uzun *et al.* [21]. The accuracy of the method was investigated by determining the bismuth in spiked water samples, and the results are presented in table 4. The detection limit of bismuth was expressed as the amount of analyte in μg/L giving a signal-to-noise ratio of 3. The detection limit of bismuth was reported in table 2.

3.8 *Efficiency of multi-element preconcentration*

The efficiency of multi-element preconcentration together with bismuth was studied by taking 50 µg of individual metal ions like As, Zr, Sn, Hg, Zn, Ni, Cd, and Bi in 100 mL and passed through the resin coated with 1 g each of NaDTTC or pipDTC. The adsorbed metal ions on resin coated with NaDTTC or pipDTTC were eluted with 4 M/L of HNO_3 and analysed using the general procedure described in the experimental section. The results are presented in table 5. The results indicated that Bi, Zr, Zn, Ni, and Cd have a higher recovery than Sn, As, and Hg. The data in table 5 also indicate that the recovery of bismuth slightly decreased because of the presence of other metal ions in solution.

3.9 *Determination of bismuth in standard reference materials*

The accuracy of the proposed method was ascertained by the determination of bismuth in standard reference materials (NIST-SRM), 1643e and 3106, respectively, and the data are given in table 6. Both SRMs are aqueous solutions, and the concentration of bismuth was analysed by following the general procedure described in the experimental section. The experimental results are in good agreement with standard reference values.

3.10 *Application of the method*

The proposed method was applied to the preconcentration and determination of bismuth along with other trace-metal ions in natural water samples. The treatment

Table 5. Recovery of metal ions from spiked water samples after preconcentration on NaDTTC- and pipDTC-coated Amberlite XAD-7.

Metal	Concentration of element added (µg/100 mL)	Resin coated with NaDTC Found (µg/100 mL)	Recovery (%)[a]	RSD (%)[a]	Resin coated with pipDTC Found (µg/100 mL)	Recovery (%)[a]	RSD (%)[a]
As	50	40.16	80.32	5.23	40.95	81.90	5.15
Bi	50	49.22	98.44	3.76	48.74	97.48	3.94
Cd	50	49.24	98.48	4.02	49.07	98.14	4.18
Hg	50	35.18	70.36	7.29	34.28	68.56	7.85
Ni	50	49.12	98.24	4.14	48.94	97.88	4.31
Sn	50	48.03	96.06	5.48	47.75	95.50	5.62
Zn	50	49.60	99.20	3.91	49.51	99.02	4.04
Zr	50	48.61	97.22	4.08	48.31	96.62	4.16

[a]Percentage recovery and percentage RSD for five determinations.

Table 6. Recovery of bismuth from standard reference sample after preconcentration on NaDTTC- and pipDTC-coated Amberlite XAD-7 resins[a].

Name of the SRM	Certified value	Analysed by ICP-AES	After preconcentration on NaDTTC-coated resin	After preconcentration on pipDTC-coated resin
1643e	14.09	14.02 ± 0.13	13.92 ± 0.12	13.73 ± 0.14
3106	10.00	9.96 ± 0.14	9.91 ± 0.13	9.72 ± 0.15

[a]All values are in ppb. The values reported are the means of five readings ± SD.

Table 7. Determination of trace metals in natural water and seawater samples after preconcentartion on pipDTC-coated resin[a].

	Present method (μg/L)		Standard method (μg/L)		Present method (μg/L)		Standard method (μg/L)	
Metal	Natural water sample 1	Natural water sample 2	Natural water sample 1	Natural water sample 2	Seawater sample 1	Seawater sample 2	Seawater sample 1	Seawater sample 2
Bi^{3+}	2.0 ± 0.04	1.7 ± 0.08	2.0 ± 0.03	1.5 ± 0.06	1.4 ± 0.08	n.d.	1.4 ± 0.06	n.d.
Cd^{2+}	6.0 ± 0.5	8.0 ± 0.4	7.0 ± 0.2	8.0 ± 0.4	14 ± 0.5	13.8 ± 0.5	14.6 ± 0.3	14 ± 0.4
Cu^{2+}	31.0 ± 1.6	28.0 ± 1.3	32.0 ± 1.1	27.0 ± 1.1	20.5 ± 2.8	21.3 ± 2.5	20.4 ± 2.0	20.8 ± 2.2
Mn^{2+}	25.0 ± 1.4	27.0 ± 1.5	24.0 ± 1.8	25.0 ± 1.2	4.8 ± 1.9	5.4 ± 2.2	4.7 ± 1.6	5.2 ± 1.8
Ni^{2+}	23.0 ± 1.3	20.0 ± 1.2	20.0 ± 1.1	18.0 ± 1.2	11.4 ± 1.0	10.5 ± 1.4	11.4 ± 1.0	11.3 ± 1.1
Pb^{2+}	37.0 ± 1.9	34.0 ± 1.3	31.0 ± 1.6	33.0 ± 1.5	10.0 ± 1.3	9.7 ± 1.5	9.8 ± 1.0	10.0 ± 1.2
Zn^{2+}	820.0 ± 4.5	842.0 ± 3.4	825.0 ± 4.1	846.0 ± 3.2	189.2 ± 4.8	194.1 ± 3.6	190.1 ± 4.2	192.7 ± 3.4

[a] The values reported are the means of five readings ± SD. n.d.: not detectable.

and sampling procedure of natural water samples are given in the experimental section. The results are presented in table 7. The accuracy of this method was ascertained by comparing these results with the GFAAS method.

4. Conclusions

The studies reported here demonstrate that the dithiocarbamates-coated Amberlite XAD-7 resin has a good enrichment factor for preconcentration of bismuth from natural water samples. The results of adsorption capacity, detection limits, and RSD values showed that resin coated with pipDTC has a similar enrichment factor to the resin coated with NaDTTC. Hence, pipDTC is a good alternative for complexing bismuth in water. The method has high tolerance limits from the matrix ions. The results of the standard addition method revealed that the proposed method has a good accuracy. The proposed method was applied for the determination of trace-metal ions in natural water samples, and the results obtained agreed well with the standard method.

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