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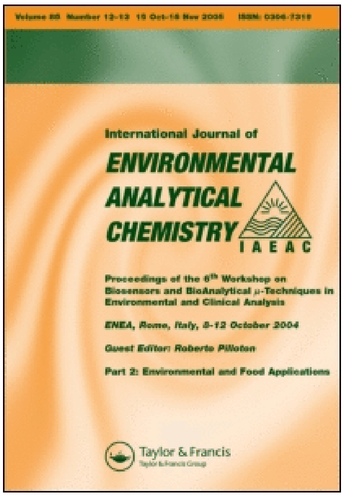

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Microdetermination of Mercury in Industrial Waste Waters by Stripping Voltammetry

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MICRODETERMINATION OF MERCURY IN INDUSTRIAL WASTE WATERS BY STRIPPING VOLTAMMETRY

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Low levels of mercury have been determined in industrial waste waters by differential pulse anodic stripping voltammetry with a precision of ±3.6 standard deviation. The limit of determination was observed to be 1.0 μg/L.

Keywords: Industrial waste waters; mercury; voltammetry

INTRODUCTION

Among the toxic trace metals, mercury is one of the most hazardous pollutants. [1] In dissolved state it enters the marine and aquatic food chains due to accumulation by the organisms. [2] The main sources of mercury in the environment and natural water systems are the industries of chemicals, mining, paper and applications of organometallic compounds as pharmaceuticals, fungicides and catalysts. [3] Thus it is appropriate to develop an analytical method of simple approach for the determination of mercury in industrial waste waters.

Advanced modes of voltammetry are among the few instrumental techniques for trace analysis which have good potential for detection, speciation and quantitation. [4] Stripping voltammetry with suitable solid electrodes provides superior sensitivity for the determination of a number of metal ions in different matrices. [5] Fukai et al have determined mercury by stripping voltammetry with

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*Corresponding author. Fax: +91-291-613434 a graphite electrode. [6] A simultaneous determination of copper and mercury in sea water has been described by Nurnberg et al.[7] Carbon paste and glassy carbon electrodes have also been employed for mercury determination. [8], [9] Andrew et al.[10] have shown the use of a gold electrode for determination of mercury at low concentrations. In the present studies authors have utilized a gold disc electrode in differential pulse anodic stripping voltammetry for micro determination of mercury in industrial waste water samples of the Basni and Marudhar Industrial Areas of Jodhpur.

EXPERIMENTAL

Instrumentation

A pulse polarograph (Model CL-90) in combination with x-y recorder (Model LR-108), by Elico Hyderabad, India was used to record anodic stripping voltammograms. A gold disc electrode (1.6 mm diameter) by Bioanalytical systems Inc (BAS), West Lafayette, U.S.A. was used as the working electrode. The surface of the gold electrode was polished with diamond polishing compound supplied by BAS to attain a fresh mirror like surface. Then the electrode was electrochemically activated by cycling between −0.25 V and +1.7 V several times, before each application. [7]

Cyclic voltammetry was performed on a cyclic voltammograph (Model, CV-27) by BAS.

All potentials were measured against a saturated calomel electrode (SCE) and a platinum wire was used as an auxiliary electrode throughout.

Sample Collection and Preparation

Glassware and polyethylene sample containers were soaked in 2 M nitric acid for at least one week and washed with doubly distilled water prior to use. Industrial waste water samples were collected from different sites of Marudhar and Basni Industrial Areas of Jodhpur. Samples were filtered to separate particulate matter and these were acidified with hydrochloric acid to pH 2 for storage purposes. A 100 ml aliquot was digested with an oxidising mixture of nitric acid and hydrochloric acid and heated till the solution fumed. The contents were transferred to a volumetric flask and made up to the volume.

All experiments were carried out in an airconditioned laboratory where temperature was maintained 25 ± 1°C. The solutions were deaerated by bubbling with purified nitrogen for 20 min. Chemicals used were of reagent grade purity. Stock solution of Hg(II) was prepared from mercuric chloride by Sarabhai M.

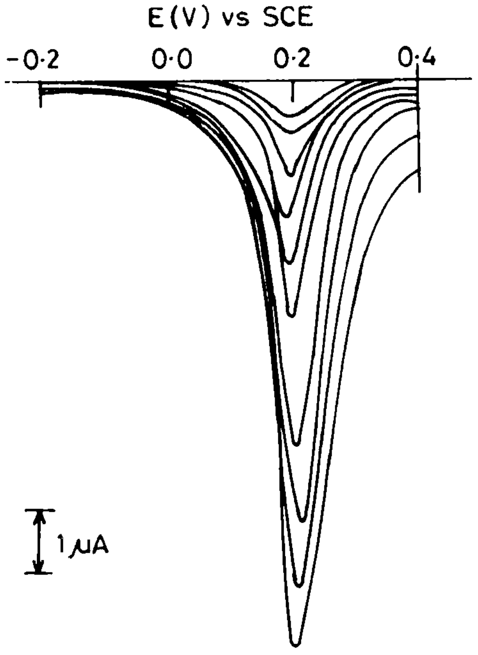

FIGURE 1 Differential pulse anodic stripping voltammograms of Hg(II) at different concentrations in 0.5 HCl + 0.5 M H_2SO_4. Modulation amplitude, 50 mV, pulse duration 57 ms, td, 5 min, scan rate, 5 mv/sec, W.E., gold disc electrode.

Chemicals, Baroda, India. Test solutions were prepared each day by diluting 0.01 M stock solution. Supporting electrolyte was used 0.5 M HCl + 0.5 M H_2SO_4.

RESULTS AND DISCUSSION

A well defined dc polarographic wave of Hg(II) in 0.5 M HCl + 0.5 M H_2SO_4 is observed at a potential of +0.17 V. The reversibility of the electrode reaction was evaluated by performing cyclic voltammetry of Hg(II) in 0.5 HCl + 0.5 H_2SO_4 at a gold disc electrode. Both cathodic and anodic peaks (Epc, 0.18 V and Epa, 0.20 V Vs Ag/AgCl) were noticed, suggesting a reversible system.

Hg(II) in 0.5 M HCl + 0.5 M H_2SO_4 also showed a sharp differential pulse peak (Ep, +0.18 V Vs SCE). The parameters used in DPP were as follows: modulation amplitude 50 mV, pulse duration 57 ms, clock time of pulse 1 sec and scan rate 5 mV/sec. Higher sensitivity was achieved by differential pulse anodic stripping voltammetry where mercury was deposited on the surface of the gold disc electrode for a period of 5 min at the potential of −0.20 V. Mercury peak

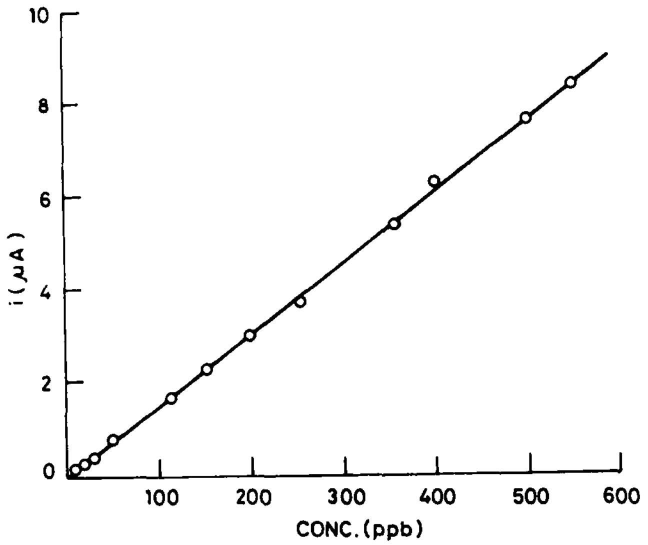

FIGURE 2 The plot of calibration curve, concentration (in μg/L) Vs peak current. Slope = 1.045 ± 7.64×10^{-3}, coefficient of correlation = 0.9996, intercept = $-2.72 \times 10^{2} \pm 5.06 \times 10^{-2}$.

TABLE I Industrial waste water analysis for mercury contents by anodic stripping voltammetry.

Water sample		Conc. of Hg in μg/L.	
		Marudhar*	Basni*
1.		12.2	8.0
2.		12.8	12.3
3.		8.4	9.2
4.		10.5	12.5
5.		13.1	N.D
	Average	11.4	10.5
	S.D. ±	1.9	2.2
	R.S.D.	17%	21%

* = Industrial Areas N.D. = Not detected n = 3, no. of determination

was noticed at +0.19 V. Figure 1 shows the anodic stripping voltammograms of mercury at varied concentrations. The peak current was found to be proportional to the concentration of mercury in the range 1.0 to 545 μg/L. A calibration curve showing mercury concentration versus peak current is shown in Figure 2.

The stripping voltammetric determination of mercury was evaluated for its precision and average standard deviation of ±3.6 was obtained at a concentration of 10 μg/L (number of replicates 3). The limit of determination was 1.0 μg/L.

Determination of mercury in industrial waste waters

The digested samples were taken into the polarographic cell dissolved in 0.5 M HCl + 0.5 M H_2SO_4 and anodic stripping voltammograms were recorded in the potential range of -0.2 to $+0.4$ V. The peak currents were measured at $+0.19$ V after making blank corrections.

Quantitation in all observations was made by standard addition method.[11] The results of mercury determination in industrial waste waters are listed in Table I.

Acknowledgements

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References

[1] J. J. Dulka and T. H. Risbey, *Anal. Chem.*, **45**, 640A-653A (1976).

[2] S. E. Manahan, *Environmental Chemistry* (Lewis Publishers, Inc. Michigan, 1991) pp. 150-152.

[3] I. Ioneci, I. Tanase and C. Luca, *Anal. Letters*, **18**, 929-945 (1985).

[4] J. R. Pretty, E. A. Blubaugh, J. A. Caruso and T. M. Davidson *Anal. Chem.*, **66**, 1540-1547 (1994).

[5] A. M. Bond, *Modern Polarographic Methods in Analytical Chemistry* (Marcel Dekker, Inc, New York 1980) pp. 439-448.

[6] R. Fukai and L. Huynh-Ngoc, *Anal. Chim. Acta*; **83**, 375-379 (1976).

[7] L. Sipos, J.Golimowski, P.Valenta, H. W.Nurnberg and Z.Fresenius, *Anal. Chem.*, **298**, 1-8 (1979).

[8] A. J. Bard and L. R. Faulkner, *Electrochemical methods, Fundamental and Applications* (John Wiley & Sons, New York, 1980) pp. 413-420.

[9] R. E. Allen and D. C. Johnson, *Talanta*, **20**, 799-809 (1973).

[10] R. W. Andrew, J. H. Larochelle and D. C.Johnson, *Anal. Chem.*, **48**, 212-214 (1976).

[11] H. Willard, L. Merrit and J. Dean, *Instrumental Methods of Analysis*, 5th Edn., (D. Van Nostrand, New York, 1974), pp. 659-660.