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Substoichiometric Radiochemical Determination of Cadmium with Sodium Isopropyl Xanthate

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The present paper brings out a rapid and sensitive substoichiometric radiochemical method in determining the quantity of cadmium by utilising sodium isopropyl xanthate as a reagent. The estimation could be done up to the microgram quantity. Cd complex formed with the reagent was extracted into chloroform by monitoring the value of pH 8 with the ammonia buffers. In the present investigation 2–10 μg of cadmium has been determined from the sample, test solutions with an average error of ±1.76%.

INTRODUCTION

Cadmium is toxic in both plants and animals. The reagent namely sodium isopropyl xanthate has its applications in a wide variety of areas[1, 2]. Since this reagent has not previously been used in a radiochemical technique, we have chosen this reagent to develop the substoichiometric radiochemical method for the evaluation of cadmium in water samples.

EXPERIMENTAL

Cadmium stock solution was prepared by dissolving about 0.2282 g of $CdSO_4 \cdot 8H_2O$ (BDH, AnalaR) into 100 mL of double distilled water to form a solution containing 1 mg/mL cadmium and standardised[3].

Tracer cadmium-115m ($t_{1/2} = 44d$) was supplied by Board of Radiation and Isotope Technology, Bombay.

Cadmium(II) active solution was prepared by adding 20 μL of tracer to 1 mL. stock solution and diluted to 100 mL to yield a solution containing 0.01mL of Cd/mL.

Activity measurements were performed by using GM Counter (I-1030 coupled to the scaling system CGS-13).

Water samples each one litre were collected from different areas surrounding the town of Tirupati. These samples, after filtration, were concentrated to 15 mL by gentle distillation after treatment with a few drops of concentrated nitric acid and sulphuric acid. The preconcentrated solution was filtered again and made up to 25 mL standard flask.

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Development of the method

Effect of pH: The extraction behaviour of cadimum isopropyl xanthate complex was carried out in the pH range of 8.5 to 11 ammonia buffers. Solutions containing 5 μg of cadmium were taken in a series of stoppered tubes and the pH was adjusted to the required value. 0.3 mL (1.8E–04M) sodium isopropyl xanthate was added and equilibrated for 5 minutes. The complex thus formed was extracted into 2 mL of chloroform. The activity of one mL of organic layer was measured in each case.

Reproducibility of substoichiometric isolation: The reproducibility of substoichiometric extraction was tested by radiometric titration. Solutions containing tracer cadmium in the range of 1–11 μg were prepared. The pH of each solution was adjusted to 9.5. Equal amounts (0.4 mL 1.8E–04M) of sodium isopropyl xanthate was added and equilibrated for 5 minutes. The contents were extracted with 2 ml of chloroform. The activity of one mL of the organic layer was measured in each case and plotted against the volume of tracer cadmium.

Calibration: Calibration plot was prepared by utilising carrier amount variation technique. Solutions containing inactive cadmium in the range of 1–10 μg were prepared. A fixed quantity of tracer (5 μg) was added and the pH of the solution was adjusted to 9.5 with the ammonia buffers. Equal but substoichiometric amount of sodium isopropyl xanthate was added and the complexes formed were extracted with 2 mL of chloroform. The activity (a) of one mL of the organic extracts was measured in each case. Similarly, the activity (a_s) of the tracer solution was obtained without the addition of inactive cadmium. The values of (a_s/a) are computed and plotted against the amount of inactive cadmium.

RESULTS AND DISCUSSION

The complex forming ability of the cadmium isopropyl xanthate was found to be maximum when the sample solution pH value reaches to 9.5. Hence pH 9.5 was selected for further studies (Fig. 1).

The reproducibility curve was found to be satisfactory and also appeared as a perfect radiometric titration curve (Fig. 2) and indicates the formation of 1 : 2 complex.

A linear calibration curve was obtained in the range of 1–10 μg and the extrapolated portion indicates the quantity of the tracer cadmium added. About 2–10 μg of cadmium has been determined by the present method with an average error of ±1.76%.

The metals such as 5 μg of Pb(II), Te(IV), Se(IV), Tl(III), Al(III), Cr(III), Fe(III), Co(II), Zn(II), Pd(II), Sn(II), Mn(II), Hg(II), Ni(II), Bi(III) and VO_3^-, CN^-, F^-, citrate, tartrate and EDTA do not interfere on the substoichiometric extraction of 7 μg of cadmium. The interference due to Cu(II) and Ag(I) can be masked out with CN^-.

The present developed method of substoichiometric analysis becomes quite a convenient technique for the determination of cadmium content of different water

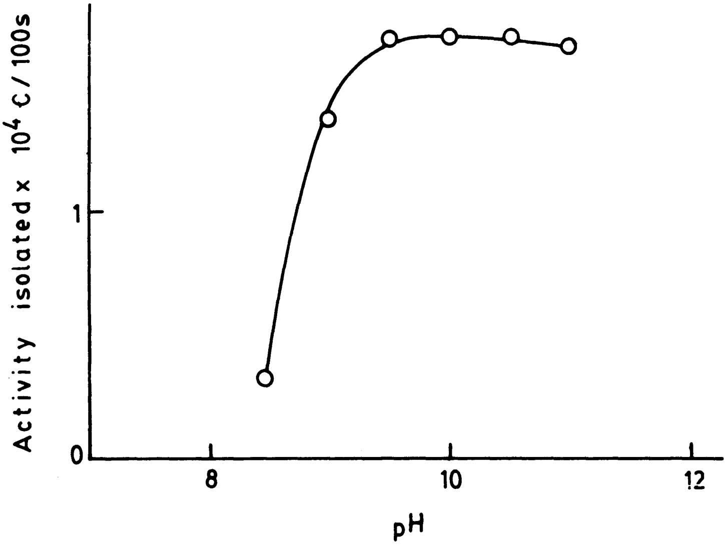

Fig. 1 Extraction behaviour of cadmium isopropyl xanthate

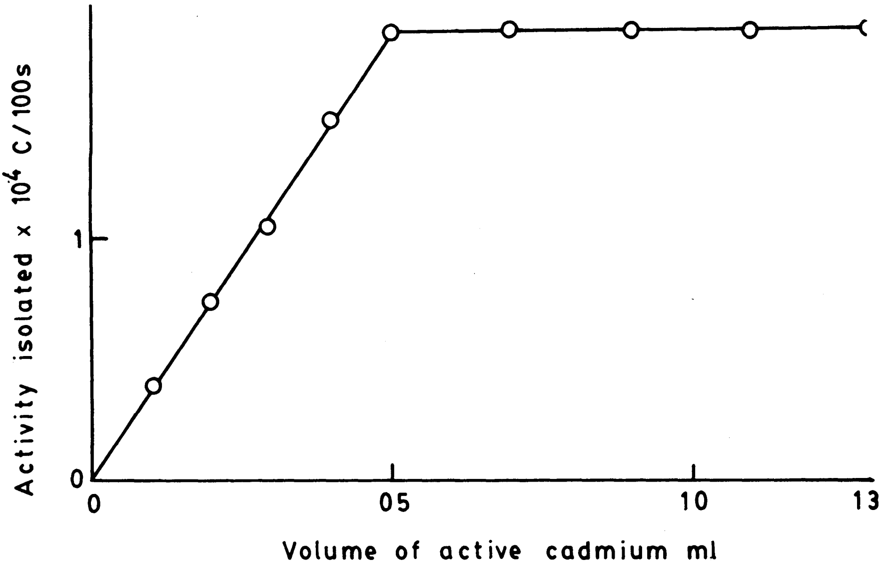

Fig. 2 Reproductibility of substoichiometric extraction

samples of *Pushkarini* (temple tanks of Tirupati and Tirumala) and also other public residential areas (Table 1). The values obtained were compared well with the values obtained by the standard method[4].

TABLE 1(a)
DETERMINATION OF Cd IN TEXT SOLUTION

Amount of Cd taken (μg)	Amount of Cd found (μg)
2.24	2.14
4.48	4.43
5.60	5.56
7.84	7.65
10.08	10.11
Synthetic mixture: 5. 6 μg of Cd + 4 μg of each Co(II), Fe(III), Zn(II), Ca(II), Pb(III), Ni(II)	5.62

Average error ±1.76%.

TABLE 1(b)
DETERMINATION OF Cd CONTENT IN SAMPLE SOLUTIONS

Sample	Amound of Cd determined (μg/1)	
	Present Method	Standard method [4]
Kapilatheertham water	*2.47 ± 0.17	2.48
Tirumala pushkarini water	*2.62 ± 0.14	2.63
Tap water	*6.94 ± 0.12	6.39
Bore well water (University campus)	*6.36 ± 0.16	6.47

*Average of four determinations

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