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Substoichiometric Radiochemical Determination of Selenium by Sodium-isopropyl Xanthate as a Reagent

K.V. MURALIKRISHNA†, B. POLAIAH and B. RANGAMANNAR*
Radiochemical Laboratories, Sri Venkateswara University
Tirupati-517 502, India

A simple selective and sensitive method for the determination of selenium has been investigated. Sodium isopropyl xanthate was used as a reagent and chloroform as an extractant. The complex was extracted into chloroform from pH 4 acetate buffer. The influence of various interferences ions on the extraction was studied. 3–10 μg selenium was determinted with a precision of 1.42%. The method developed was utilized to determine the selenium content in Jaggery and wheat samples.

INTRODUCTION

Xanthates are familiar analytical reagents and well utilized for the extraction of a number of metals[1]. Among these classes of reagents, only a few reagents are utilised for substoichiometric isolation. In the present investigation, an attempt has been made to introduce sodium isopropyl xanthate as a substoichiometric radioanalytical reagent for the determination of selenium.

EXPERIMENTAL

The inactive stock solution of selenium containing 1 mg of Se/mL was prepared by dissolving 0.3334 g of $Na_2SeO_3 \cdot 5H_2O$ (Merck GR). Tracer Se was obtained from Board of Radiation and Isotope Technology, Mumbai.

20 μL tracer solution was added to 1 mL of stock solution and diluted to 100 mL with distilled water which served as 0.01 mg/mL selenium. The reagent sodium isopropyl xanthate was prepared and purified by the method described by Keskyula[2]. 5.1×10^{-4} M reagent was prepared in distilled methanol. Acetate buffer solutions were prepared in the pH range of 3.5 to 6.5 using solutions of acetic acid and sodium acetate. Activity measurements were performed with a well type NaI(Tl) scintillation counter connected to a single channel analyser. Chloroform employed as extractant was purified before use[3].

Extraction Studies: A series of solutions consisting of 1 mL of tracer selenium was adjusted in the range of 3.5 to 6.5 acetate buffer. 0.85 mL of reagent (5.1×10^{-4} M) was added and equilibrated for 5 minutes. The selenium : isopropyl xanthate complex formed was extracted into chloroform. 1 mL of the chloroform was withdrawn and its activity was measured in each instance. The results were recorded as a function of pH (Fig. 1).

Reproducibility of Substoichiometric Extractions: Tracer solutions contain-

†Computer Centre, Sri Venkateswara University, Tirupati-517 502, India

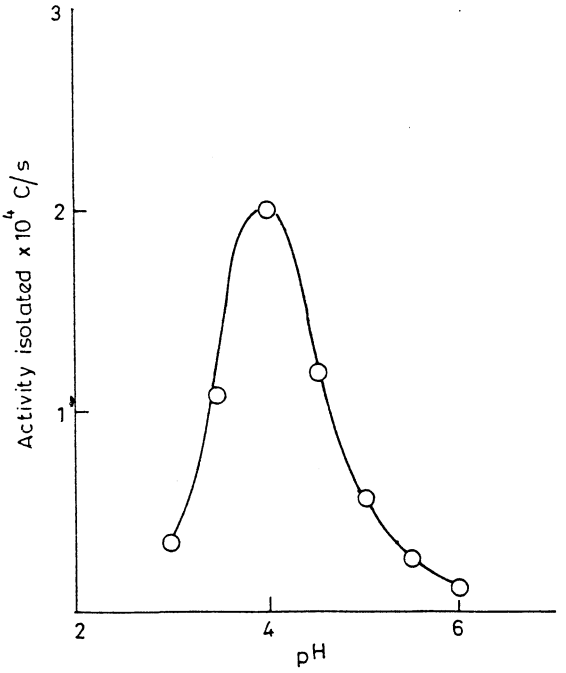

Fig. 1 Extraction of behaviour of selenium isopropyl xanthate

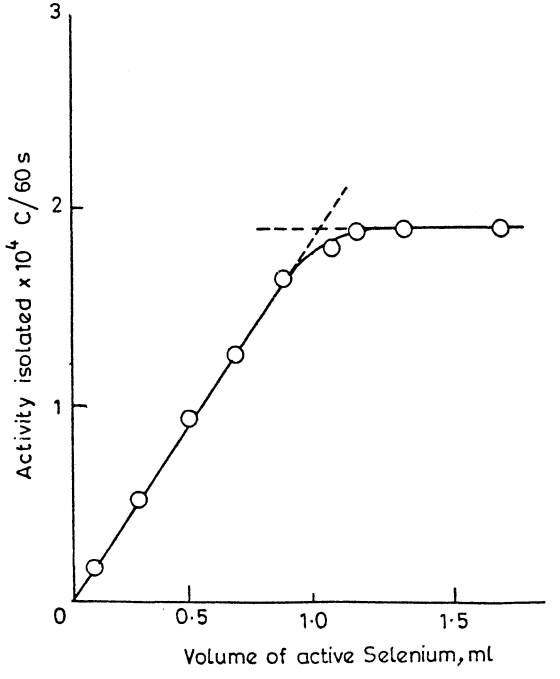

Fig. 2 Reproduction of substoichiometric extraction

ing 1–18 μg of selenium were prepared. The pH of the solution was adjusted to 4.0 with acetate buffers. A uniform and constant amount of the reagent, 1.0 mL 5.1×10^{-4} M was added. The contents of the solutions were equilibrated for 5 minutes. The contents of all tubes were extracted into 2 mL of chloroform. The activity of 1 mL of the organic phase was measured. The results were incorporated graphically (Fig. 2) as a function of tracer selenium.

Calibration: Solutions containing inactive selenium in the range of 3–10 μg were prepared. A constant amount of labelled selenium was added and the pH of the solutions was adjusted to 4. Substoichiometric amount of reagent (0.5 mL, 5.1×10^{-4} M) was added and the complex formed was extracted into 2 mL of chloroform. The activity (a) of 1 mL of the organic extract was measured in each instance. Similarly the activity (a_s) of the tracer solution was obtained without the addition of active selenium. The values a_s/a were computed and plotted against the amount of inactive selenium (Fig. 3).

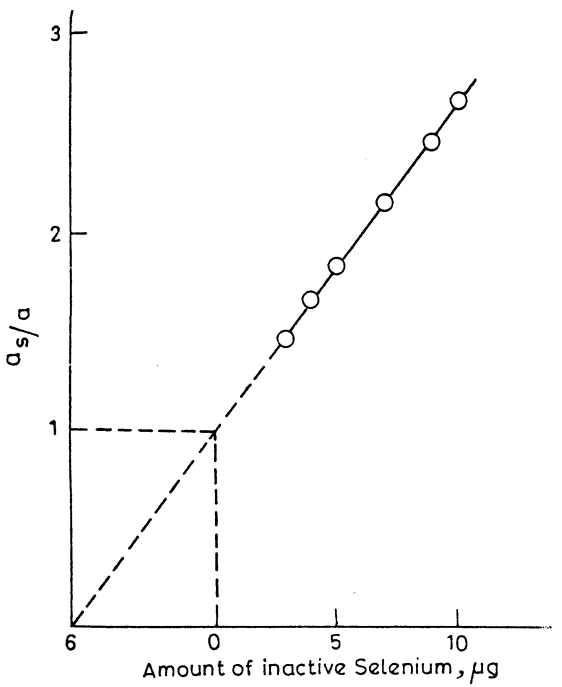

Fig. 3 Calibration plot

RESULTS AND DISCUSSION

The extraction of selenium : isopropyl xanthate complex is maximum at pH 4 in acetate buffers. Hence, the pH 4.0 was considered for further studies, *i.e.*, reproducibility and calibration.

The substoichiometric reproducibility was good with chloroform for the extraction of the Se : isopropyl xanthate complex. Equivalence point of the titration curve indicates the formation of the 1:4 complex, *i.e.*, Se : $(IPX)_4$.

The calibration curve is linear in the range of 3–10 μg of Se. Selenium could be determined successfully with a precision of 1.42%. Each 8 μg of Tl(I), Ag(I), Co(II), Cu(II), Cd(II), Pd(II), Zn(II), Hg(II), Pb(II), Tl(III), Sb(III), In(III), Te(IV), EDTA, citrate and F^- on the substoichiometric extraction of 4 μg of selenium is negligible.

The samples of jaggery and wheat collected from Tirupati, Andhra Pradesh (India) were processed. The selenium content of the samples was determined by the present method and the values are in close agreement with those obtained by standard method[4]. The values of the present investigation are represented in Table-1.

TABLE-I

(a) DETERMINATION OF SELENIUM CONTENT IN TEST SOLUTION

Amount of selenium taken (μg)	Amount of selenium found (μg)
4.03	4.14
6.04	6.02
8.05	7.99
10.07	9.96
Synthetic mixture (each 6 μg of Co(II), Cd(II), Hg(II), Ag(I), Pb(II), Zn(II), Te(IV) and Se 5 μg)	4.92

Average error +1.42%

(b) DETERMINATION OF SELENIUM CONTENT IN SAMPLE SOLUTIONS

Sample	Amount of Se determined (μg/g)	
	Present method*	Standard method
Jaggery	0.594 + 0.05	0.532
Wheat powder	0.644 + 0.06	0.691

*Average of four determinations.

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