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# Solvent Extraction of Selenium(IV) with 2-Thenoyltrifluoroacetone

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## NOTE

# Solvent Extraction of Selenium(IV) with 2-Thenoyltrifluoroacetone

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### Summary

Selenium(IV) can be quantitatively extracted at trace concentrations with 2-thenoyltrifluoroacetone-xylene (0.03 $M$) at pH ~ 1.0–4.5. It is determined in the organic phase photometrically by complexation with 3,3′-diaminobenzidine. The system conforms to Beer's law in the concentration range of 2.2–13.2 μg/ml of selenium. The complex is stable. Salting-out agents have no effect on extraction. Selenium can be extracted and determined in one operation in the presence of several elements.

2-Thenoyltrifluoroacetone (TTA) was used for the extraction and direct spectrophotometric determination of several elements. Further studies revealed that it is possible to quantitatively extract microgram concentrations of selenium at pH 2.0 with 0.030 $M$ TTA–xylene. Selenium in the organic phase is directly determined photometrically as its complex with 3,3′-diaminobenzidine at 420 nm. The methods for the solvent extraction of selenium are summarized by De, Khopkar, and Chalmers (*1*) in their recent monograph. However, these methods are not practical as they involve critical control of different factors such as temperature, acidity, and time of equilibration. The proposed method is simple, rapid, and applicable at trace concentration. It is also selective and affords clean-cut separation and determination of selenium in one operation.

## EXPERIMENTAL

### Apparatus and Reagents

SF-8 Recording Spectrophotometer, 0 3KH-57 photometer, wrist action flask shaker, and Cambridge pH meter.

A stock solution of selenium was prepared by dissolving about 0.25 g of sodium selenite in a liter of distilled water. The solution on standardization gravimetrically (*2*) contained 110 $\mu$g/ml of selenium.

2-Thenoyltrifluoroacetone (B.D.H. AnalaR) solution in xylene (0.030 *M*).

3,3′-Diaminobenzidine (Fluka A.S.) 0.1% in 0.1 *N* hydrochloric acid.

### Buffer Solution

Buffer solution was prepared by dissolving 280 g of sodium acetate in water, acidifying with acetic acid to pH 6.5, and diluting to 1 liter.

## GENERAL PROCEDURE

An aliquot of the solution containing 66 $\mu$g of selenium was taken. The pH of this solution was adjusted to pH 2.0 with 0.01 *N* sodium hydroxide and 0.01 *N* hydrochloric acid to a total volume of 25 ml. Then 10 ml of 0.030 *M* TTA–xylene was added, and the solution was shaken in a separating funnel for 10 min. The phases were allowed to settle and separate, and then the aqueous phase was carefully withdrawn. The organic phase was mixed with 10 ml of buffer solution, 5 ml of 3,3′-diaminobenzidine solution, and it was once again shaken for about 10 min. The two phases were allowed to settle and separate. The organic phase was transferred to a 10-ml volumetric flask. The absorbance of the yellow colored complex was measured at 420 nm. The amount of selenium was computed from the calibration curve (*3*).

## RESULTS AND DISCUSSION

### Absorption Spectrum

The absorption spectrum of Se(IV)–TTA complex (Se $= 8.36 \times 10^{-5}$ *M*) shows strong absorbance at 420 nm (Fig. 1). The reagent blank shows no absorbance at this wavelength. All absorbance meas-

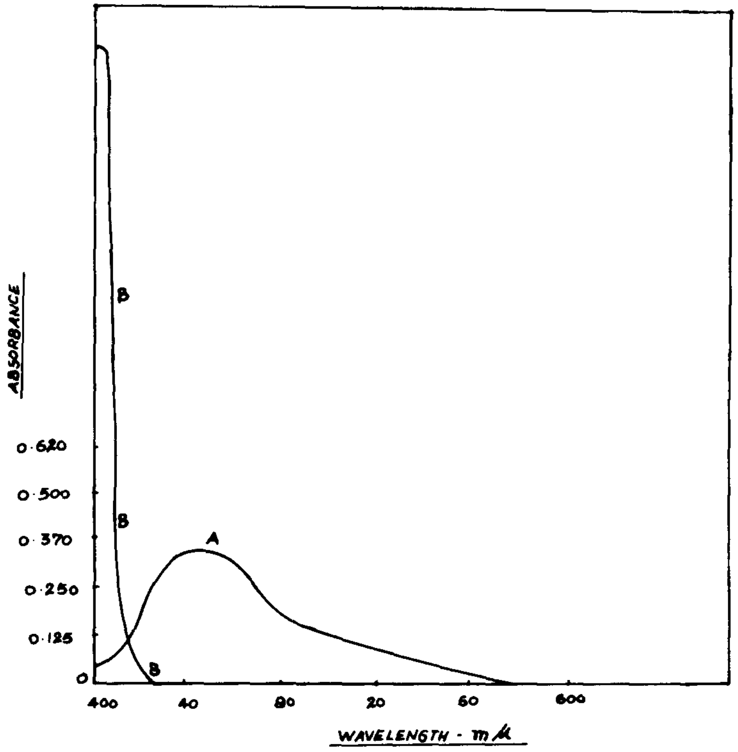

**FIG. 1.** Absorption spectra of Se(IV)-diaminobenzidine in TTA-xylene. A: Absorption spectra of Se(IV)-diaminobenzidine in TTA-xylene vs. reagent blank. B: Reagent blank vs. xylene; Se(IV) $= 8.36 \times 10^{-5} M$; TTA $= 3.0 \times 10^{-2} M$.

urements were, therefore, taken at 420 nm. The molar absorptivity is $4.3 \times 10^3$ at 420 nm on the basis of selenium contents.

## Extraction as a Function of pH

The extraction of selenium(IV) was carried out from pH 0.1–8.0 with TTA (Fig. 2). The results showed that the extraction was quantitative in the pH region of 0.5–4.5.

## Beer's Law

Different amounts of selenium(IV) (1.1–13.2 μg/ml) were extracted at pH 2.0 and their absorbance measured at 420, 460, and 510 nm.

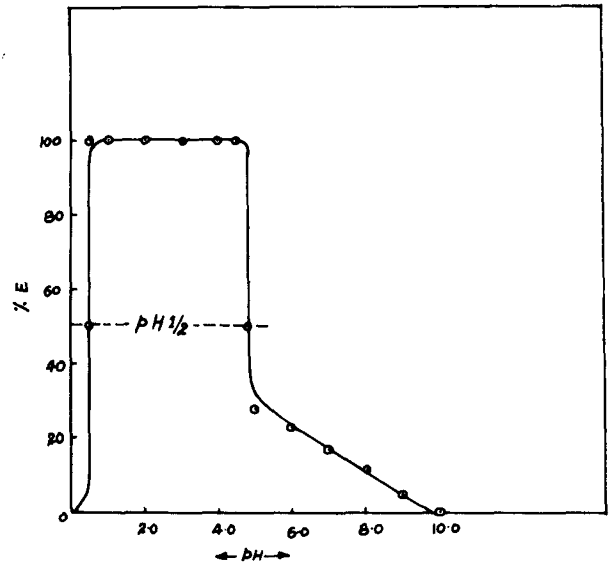

**FIG. 2.** Extraction as a function of pH for Se(IV)–TTA–xylene.

It was seen that the system conforms to Beers law at pH 2.0–4.5 over the concentration range of 0.22–13.2 μg/ml of selenium at 420 nm.

## Effect of Reagent Concentration

The selenium was extracted at pH 2.0 with varying volume and concentration of the reagent (Table 1). The results indicated that

**TABLE 1**

Effect of Reagent Concentration[a]

| TTA concn *M*, in xylene | Vol of TTA (ml) | Absorbance (420 nm) | Per cent extraction, *E* | Distribution ratio, *D* |
|---|---|---|---|---|
| 0.030 | 5 | 0.225 | 62.5 | 4.141 |
| 0.030 | 7.5 | 0.350 | 97.2 | 86.780 |
| 0.030 | 10 | 0.360 | 100 | ∞ |
| 0.030 | 20 | 0.360 | 100 | ∞ |
| 0.005 | 10 | 0.230 | 63.8 | 4.407 |
| 0.010 | 10 | 0.240 | 66.7 | 5.009 |
| 0.015 | 10 | 0.280 | 77.8 | 8.800 |
| 0.030 | 10 | 0.360 | 100 | ∞ |

[a] Se(IV) = 66 μg; pH = 2.0.

10 ml of 0.030 *M* TTA is quite adequate for quantitative extraction of selenium, while extraction is incomplete with 0.005, 0.010, and 0.015 *M* of TTA.

### Stability of Color

The absorbance of the colored complex was measured at intervals of 30 min. The complex was stable for at least 24 hr. It is therefore recommended that the solution be measured within 12 hr of complexation.

### Period of Shaking

Variation from 3 to 30 min in the shaking period revealed that 94.5% extraction occurs in 5 min whereas beyond 8 min quantitative extraction occurs. Hence, for quantitative extraction the solution should be shaken for at least 10 min.

### Effect of Salting-Out Agents

The chlorides of lithium, ammonium, and calcium (0.5–3.0 *M*) were used as the salting-out agents. The results showed that they have no effect on the extraction.

### Effect of Diverse Ions

The effect of the presence of several ions on the extraction behavior was studied (Table 2). The tolerance limit was calculated as the

**TABLE 2**

Effect of Other Ions[a]

| Tolerance limit (μg) | Other ions present |
|---|---|
| $5.0 \times 10$ | $WO_4^{2-}$, $CN^-$, $SO_3^{2-}$ |
| $1.0 \times 10^2$ | $Ir^{3+}$, $Sn^{2+}$, $Os^{8+}$, $U^{6+}$, $EDTA^{4-}$, $S_2O_3^{2-}$, Tartrate$^{3-}$, Malonate$^{2-}$ |
| $1.5 \times 10^2$ | $F^-$, $SCN^-$ |
| $2.0 \times 10^2$ | $Hg^{2+}$, $Tl^+$, $Bi^{3+}$, $Pd^{2+}$, $Be^{2+}$, $Mg^{2+}$, $CrO_4^{2-}$, $S^{2-}$, $AsO_4^{3-}$, $VO_3^-$, Ascorbate$^-$ |
| $2.5 \times 10^2$ | $Pt^{4+}$, $Al^{3+}$ |
| $5.0 \times 10^2$ | $Au^{3+}$, $Li^+$ |
| $2.5 \times 10^3$ | $ReO_4^{2-}$ |
| $4.5 \times 10^3$ | $Pb^{2+}$, $Cd^{2+}$, $Zn^{2+}$, $Mn^{2+}$, $Ca^{2+}$, $Sr^{2+}$, $Ba^{2+}$, $Rb^+$, $Cs^+$, $Rh^{3+}$, $CH_3COO^-$, $PO_4^{3-}$, $C_2O_4^{2-}$, Citrate$^{3-}$ |

[a] Se(IV) = 22 μg; pH = 2.0; 0.030 *M* TTA–xylene.

amount of ion which can cause ± 2% error in total recovery of selenium. The results showed that ions such as alkali and alkaline earths, lead, rhodium, manganese, oxalate, citrate, acetate, and phosphate were tolerated in a 1:200 ratio. All other ions were tolerated in a 1:10 ratio. The ions showing strong interferences were tellurium, copper, thorium, iron, nickel, cerium, and ruthenium. The interference of these ions can be eliminated by complexation with suitable sequestering agents.

Twenty determinations showed the absorbance of the solution to be 0.360 ± 0.010. The standard deviation is ± 1.10%. Sandell's sensitivity is 0.022 $\mu$g/cm$^2$ of selenium/ml. Thus the method is selective and sensitive.

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