Selective determination of selenium(IV) from environmental samples by UV-visible spectrophotometry using O-methoxyphenyl thiourea as a chelating ligand

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A selective extraction–spectrophotometric method has been developed for determination of selenium(IV) using O-methoxyphenyl thiourea (OMePT) as a chelating agent. The basis of the proposed method is the spectrophotometric determination of selenium(IV)–OMePT complex obtained after extraction of selenium(IV) from 3.5 M hydrochloric acid media using OMePT in chloroform solvent. The complex shows maximum absorbance at 350 nm against the reagent blank. The Beer’s law was obeyed over the concentration range 5–60 µg mL$^{-1}$ of selenium(IV). The optimum concentration range was 20–50 µg mL$^{-1}$ as evaluated from Ringbom’s plot. The molar absorptivity and Sandell’s sensitivity of the selenium(IV)–OMePT complex in chloroform were $3.312 \times 10^2$ L mol$^{-1}$ cm$^{-1}$ and 0.2384 µg cm$^{-2}$, respectively. The composition of selenium(IV)–OMePT complex was 1:2 established from slope ratio method, mole ratio method and Job’s continuous variation method. The complex was stable for more than 72 h. The interfering effect of various foreign ions was studied and suitable masking agents were used wherever necessary to enhance the selectivity of the developed method. The proposed method was successfully applied for the determination of selenium(IV) from real samples, viz. pharmaceutical formulations, shampoo, vegetable sample, synthetic mixtures and environmental samples. Repetition of the method was checked by finding the relative standard deviation (RSD) for 10 determinations which was 0.35%.

Keywords: O-methoxyphenyl thiourea; pharmaceutical samples; environmental samples; selenium; solvent extraction; spectrophotometry

1. Introduction

Selenium is an essential trace element. It has importance in human biology and human health. It is essential for the prevention of a variety of diseases. The selenium content in the human body is widely distributed in all the tissues in which it is bound to proteins. Selenium deficiency causes several reproductive and obstetric complications, including male and female infertility, miscarriage, pre-eclampsia, foetal growth restriction, preterm labour, gestational diabetes and obstetric cholestasis [1]. Recent studies showed that supplemental selenium in human diets may reduce cancer risk [2]. Selenium supplementation for patients undergoing different pathologies is necessary as selenium is a structural component of the active centre of the enzyme glutathione peroxidase (GSH-Px) connected with a protective activity against free radicals [3]. In the agriculture organo-selenium compounds are reported as bactericides, fungicides and herbicides [4–7].

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Though selenium is essential to life, but its excess quantity is toxic. Dermatologic effects, such as nail and hair loss and dermatitis, occur after exposure to high levels of environmental selenium [8]. An excess of selenium in food or pharmaceutical preparations is hazardous to human health. Selenium and its compounds are listed as the Basel convention on the control of transboundary movements of hazardous waste and their disposal [9]. Trace abundance, human health applications, bactericide and herbicidal applications create a background for a need of development of selective method for the determination of selenium at trace level for its quantitative determinations.

Different methods are available for determination of Se(IV) including neutron activation analysis [10], atomic absorption spectroscopy (AAS) [11], high performance liquid chromatography [12], capillary electrophoresis [13], stripping voltammetry [14] and catalytic kinetic spectrophotometry [15]. These methods have limitations, viz. it requires more time for sample preparation, less sensitivity, instrumental set-up, maintenance of instruments, etc.

In routine analysis, spectrophotometric method is versatile and economical. Spectrophotometric methods have received considerable attention because of their significant advantages in the determination of various components at trace levels. Many methods and large variety of reagents are reported for spectrophotometric determination of selenium. A solid phase extraction multi-syringe flow injection system was used for the spectrophotometric determination of selenium with 2,3-diaminonaphthalene [16]. However, this method involves the number of steps with the laborious and lengthy procedure. A spectrophotometric method was developed for the determination of selenium in cosmetic and pharmaceutical preparations after pre-concentration with cloud point extraction [17]; the method suffers from drawbacks, viz. it requires standing time of 15 min for complete colour development, further heating is required for 10 min at 40°C and phase separation was accelerated by centrifuging in the test tube at 3500 rpm for 15 min. Methdilazine hydrochloride was reported as a reagent for the spectrophotometric determination of selenium [18]. The method lacks with extended 10 minutes for colour development and higher hydrochloric acid concentration. 3,3′-Diaminobenzidine hydrochloride was used as chromogene for spectrophotometric determination of selenium, it requires 20 min heating at 70°C [19]. A large number of methods are reported for the determination of selenium(IV). The comparison of the present method with reported methods for spectrophotometric determination of selenium(IV) is presented in Table 1 [18,20–26].

The proposed method offers several advantages over the reported methods, viz. simple and precise complex formation at room temperature, high Beer’s range, low reagent concentration (0.012 mol L⁻¹) and single extraction. The proposed method was applied for the determination of selenium in synthetic mixtures, pharmaceutical and toilet preparations and environmental sample.

2. Experimental

2.1. Apparatus

A double beam UV-VIS spectrophotometer (Elico, model SL-191) with matching 10 mm quartz cells was used for absorbance measurements. An electronic balance (Contech, model CA-123) was used for weighing purposes. Calibrated glassware were used and were cleaned by soaking in dilute nitric acid followed by washing with soap water and rinsed two times with water.

2.2. Reagents

2.2.1. Standard selenium(IV) solution

All the reagents used were of analytical reagent grade unless otherwise stated. A standard stock solution of 1 mg mL⁻¹ selenium(IV) was prepared by dissolving 1.404 g of selenium dioxide
| Reagents                                                                 | \( \lambda_{\text{max}} \) (nm) | Condition                                                                 | \( \text{Beer's law validity range} \) (µg mL\(^{-1}\)) | Solvent       | Molar absorptivity (L mol\(^{-1}\) cm\(^{-1}\)) | M:L | Remark                                                                                     | Ref.  |
|------------------------------------------------------------------------|-------------------------------|---------------------------------------------------------------------------|---------------------------------|--------------|-----------------------------------------------|-----|--------------------------------------------------------------------------------------------|------|
| J-acid (6-amino-1-naphthol-3-sulphonic acid)                          | 520                           | 5 mL of cone sulphuric acid                                                | 0.03–0.3                        | Butanol      | \(1.8 \times 10^3\)                         | 1:2 | Initially low pH (1–1.5) with concentrated sulphuric acid Beyond 1.7 \times 10^{-5} mol L\(^{-1}\) CPC, co-precipitation takes place. Limited application. | [20] |
| Cetylpyridinium chloride (CPC)                                         | 510                           | pH 2.5 acetate buffer                                                     | 50–1000 ng mL\(^{-1}\)          | Aqueous      | \(1.8 \times 10^4\)                         | NM | Beyond 1.7 \times 10^{-5} mol L\(^{-1}\) CPC, co-precipitation takes place. Limited application. | [21] |
| 2',4'-Dichlorophenylfluorone (p -CPF)                                  | 480                           | Potassium bromate, HNO\(_3\) heating –10 min                            | 0.4–15                          | Aqueous      | NM                                            | NM | High heating time at 80°C for 10 min                                                      | [22] |
| 1,3,3-Trimethyl-2-[3-(1,3,3-trimethyl-1,3-dihydroindol-2yldene)propenyl]-3 H-indolium chloride Methdilazine hydrochloride | 556                           | 2.2 mol dm\(^{-3}\) H\(_2\)SO\(_4\) pH 7                               | 0.01 to 3.84                    | toluene      | \(2.4 \times 10^5\)                         | NM | Limited applications                                                                     | [23] |
| Methdilazine hydrochloride                                             | 513                           | 10 M HCl (20 mL)                                                         | 0.1–2.3                         | Aqueous      | \(9.32 \times 10^4\)                        | NM | High acid concentration, Low Beer’s range 10 min standing time required                  | [18] |
| 2,4-Dinitrophenyl hydrazine hydrochloride (2,4-DNPH)                   | 520                           | 5 mL conc HCl                                                           | 0.03–3.5                        | Aqueous      | \(3.10 \times 10^4\)                        | NM | Long shaking time of 7 min                                                                | [24] |
| Furfuraldehyde thiocarboxydrazone (FATCH)                             | 400                           | 0.5 M HCl                                                                | 5–25                            | Dichloromethane (DCM) | \(4.026 \times 10^3\) | 1:1 | High heating time in two steps at 50°C for 80 min and at 50°C for 15 min                 | [25] |
| N-1-naphthylethlenediamine dihydrochloride (NEDA)                     | 545                           | Hydroxylamine hydrochloride, \(\rho\)-nitroaniline                     | 0.01 to 2.50                    | Aqueous      | \(2.85 \times 10^4\)                        | NM | High heating time in two steps at 50°C for 80 min and at 50°C for 15 min                 | [26] |
| O-Methoxyphenylthiourea (OMePT)                                       | 350                           | 3.5 M HCl                                                                | 5–60                            | Chloroform   | \(3.312 \times 10^2\)                       | 1:2 | Simple and precise, complex formation at room temperature, large Beer’s range, low reagent concentration (0.012 M L\(^{-1}\)), single extraction | PM   |

Note: NM, not mentioned; PM, proposed method.
(Fluka) in concentrated hydrochloric acid (11.1 mL) and diluted to 1000 mL with water. This solution was standardised by the reported method [27]. The working standard solution of selenium(IV) was prepared after diluting the standard stock solution with water.

2.2.2. Solution of foreign ions
Standard solutions of different metal ions used for interference study were prepared after dissolving exactly weighed quantity of their respective salts in distilled water or dilute hydrochloric acid. Standard solutions of anions were prepared after dissolving their respective alkaline metal salts in distilled water. Different synthetic mixtures were prepared by combining their definite compositions. Double distilled water was used throughout the experimental study.

2.2.3. O-methoxyphenyl thiourea solution
OMePT was synthesised as per the method reported by Frank and Smith [28]. The 0.012 mol L$^{-1}$ solution of OMePT was prepared after dissolving 0.219 g OMePT in 25 mL chloroform and diluted to the mark with chloroform in a 100 mL calibrated volumetric flask.

2.3. Recommended procedure
Hydrochloric acid was added to an aliquot of solution containing 400 µg selenium(IV) in a 25 mL volumetric flask, to maintain the acidity 3.5 mol L$^{-1}$ on dilution up to mark with distilled water. This solution was equilibrated with 10.0 mL, 0.012 mol L$^{-1}$ OMePT in chloroform for 3 min in a 125 mL separatory funnel. The two phases were allowed to separate, and the organic phase containing selenium(IV)–OMePT complex was dried over anhydrous sodium sulphate. It was transferred in a 10 mL volumetric flask and volume was adjusted to the mark with chloroform. Absorbance of selenium(IV)–OMePT complex in organic phase was measured at 350 nm against the reagent blank.

3. Results and discussion
3.1. Absorption spectra
The absorption spectra of the selenium(IV)–OMePT complex in chloroform shows maximum absorbance at 350 nm. The reagent blank has a negligible absorbance at the maximum absorbance wavelength of selenium(IV)–OMePT complex (Figure 1). Thus, all further absorption measurements of the complex were made at 350 nm.

3.2. Effect of acid type and concentration
Selenium(IV)–OMePT complex formation takes place in mineral acid media like hydrochloric acid, sulphuric acid and perchloric acid (Figure 2). Though selenium(IV)–OMePT complex formation takes place in various mineral acids, it was observed that the maximum absorbance is obtained in 3.5 mol L$^{-1}$ hydrochloric acid concentration. Therefore, 3.5 mol L$^{-1}$ hydrochloric acid concentration was used for this work.
3.3. Effect of OMePT concentration

Different molar concentrations of OMePT in chloroform (10 mL) in a range of 0.001 to 0.2 mol L\(^{-1}\) were varied using a fixed selenium(IV) concentration (40.0 µg mL\(^{-1}\)) and absorbance measurements were performed as per the recommended method. A 10 mL, 0.012 mol L\(^{-1}\) OMePT was sufficient for complete complex formation with selenium(IV). Absorbance increases up to 0.012 M OMePT and further remains constant. The excess of reagent does not have any adverse effect (Figure 3).

3.4. Effect of extraction solvent

Various extraction solvents, viz. toluene, xylene, benzene, isoamyl alcohol, MIBK and chloroform, were studied for quantitative extraction of selenium(IV)–OMePT complex. Amongst the extraction solvents studied, quantitative extraction with maximum absorbance was obtained in chloroform as a solvent.
3.5. Effect of equilibration time and stability of complex

The study of change in absorbance with variation in equilibration time was carried out over 30 s to 30 min. It was observed that extraction of selenium(IV) was complete in 3 min and there was no any adverse effect of prolonged equilibration on extraction of selenium(IV) up to 30 min. Hence, 3 min equilibration time was fixed for further study.

The absorbance of the complex remained stable and constant for more than 72 h. The spectral and physico-chemical characteristic of the selenium(IV)–OMePT complex is given in Table 2.

4. Analytical figures of merit

4.1. Validity of Beer’s law

The Beer’s law was obeyed over the concentration range of 5–60 µg mL$^{-1}$ selenium(IV) (Figure 4). The Ringbom’s plot was sigmoid shape with linear segment at intermediate absorbance values of 20–50 µg mL$^{-1}$ (Figure 5) [29].
Figure 3. Effect of reagent concentration Se(IV)–OMePT complex.

Table 2. Spectral and physico-chemical characteristics along with precision data of selenium(IV)–OMePT complex.

| Spectral characteristics and precision | Parameters |
|---------------------------------------|------------|
| Hydrochloric acid concentration       | 3.5 mol L⁻¹ |
| Reagent concentration                | 10.0 mL, 0.012 mol L⁻¹ |
| Extraction solvent                   | Chloroform |
| Equilibration time                   | 3 min |
| λ max                                | 350 nm |
| Molar absorptivity                   | 3.312 × 10² L mol⁻¹ cm⁻¹ |
| Sandell’s sensitivity                | 0.2384 µg cm⁻² |
| Beer’s law range                     | 5 to 60 µg mL⁻¹ |
| Ringbom’s optimum                    | 20 to 50 µg mL⁻¹ |
| Limit of detection                   | 0.6121 µg mL⁻¹ |
| Relative standard deviation          | 0.35% |
| Stoichiometry of the complex         | 1:2 (Se(IV):OMePT) |
| Stability of complex                 | >72 h |
| Correlation coefficient              | 0.99 |
4.2. Precision accuracy and detection limit

The molar absorptivity and Sandell’s sensitivity of the selenium(IV)–OMePT complex were found to be $3.312 \times 10^2$ L mol$^{-1}$ cm$^{-1}$ and 0.2384 µg cm$^{-2}$, respectively. The optimum conditions and other analytical parameters were evaluated. The ratio of relative error to photometric error in concentration was found to be 4.34. The limit of detection for the developed method was 0.61 µg mL$^{-1}$ in terms of thrice the standard deviation of the blank value. The correlation coefficient value of selenium(IV)–OMePT complex with an independent variable as concentration in µg mL$^{-1}$ and a dependent variable as absorbance was found to be 0.99, it indicates a clear linearity between these two variables. The slope value and intercept for the best-fitted line were 0.0036 and 0.018, respectively. The content of selenium(IV) in real samples can be determined using the straight line equation $Y = 0.0036X + 0.018$.

4.3. Stoichiometry of selenium(IV)–OMePT complex

The composition of selenium(IV)–OMePT complex was ascertained using the slope ratio method in which the graph of log($D_{Se(IV)}$) against log($C_{OMePT}$) at 1.0, 2.0 and 3.0 mol L$^{-1}$ hydrochloric acid concentrations were plotted. The plots were linear, having slope value 1.90,
1.93 and 1.99, respectively (Figure 6). Hence, the probable composition of the extracted species was 1:2 (Se(IV):OMePT). This composition of the complex was confirmed by the mole ratio method (Figure 7) and Job’s continuous variation method (Figure 8).

OMePT acts as a multidentate ligand; sulphur from thio group (–C=S) and nitrogen from the amino group (–NH₂) coordinate with selenium(IV) to form a 1:2 (Se(IV):OMePT) complex. Based on this investigation, probable structure recommended to complex is reported (Figure 9).

4.4. Effect of interfering ions

The selectivity of the proposed method was checked for the determination of selenium(IV) (40 µg mL⁻¹) in the presence of high concentration of various foreign ions. The tolerance limit was fixed for the ions which do not cause deviation more than ±2% in the absorbance of yellow coloured selenium(IV)–OMePT complex. The interference of cations was removed by using suitable masking agents. Tolerance limit for various interfering ions is reported in Table 3.
5. Applications

5.1. Separation and determination of selenium(IV) from binary synthetic mixtures

The proposed method was applied for separation and determination of selenium(IV) from different metal ions, viz. Ni(II), Au(III), Bi(III), Al(III) and Sb(III). After quantitative extraction of selenium(IV), the aqueous phase was evaporated to moist dryness followed by 3.0 mL concentrated hydrochloric acid. The residue obtained was cooled, dissolved in water and added metal ions were determined by reported methods [30]. To enhance the extraction of selenium(IV) in the presence of Ni(II) it was masked with EDTA. It was de-masked after treatment with 3.0 mL nitric acid, evaporated to moist dryness, followed by 3.0 mL hydrochloric acid. The residue obtained was cooled, dissolved in water and Ni(II) was determined spectrophotometrically as per the reported method [30] (Table 4).
Determination of selenium(IV) from pharmaceutical samples

The proposed method was also applied for separation and determination of selenium(IV) from pharmaceutical samples, viz. Menopace ISO, Cardio-Vit plus, Betared, Lyco-First, EC-350, Casera and toilet preparation Selsun shampoo. The pharmaceutical sample (5 to 15 tablets or capsules) was heated with the minimum amount of concentrated hydrochloric acid followed by the addition of 1 mL of concentrated nitric acid. The organic matter was destroyed by treatment with 5 mL of 60% perchloric acid. The solution was slowly evaporated to moist dryness. The residue obtained was dissolved in hot dilute hydrochloric acid and made up to 25 mL volume with distilled water. An aliquot (5 mL) of this solution was extracted and selenium(IV) was determined by the proposed method. The results obtained were precise and accurate with that of reported data by manufacturer (Table 5; supplemental data).

Figure 7. Mole ratio method for Se(IV)-OMePT complex. Se(IV) = OMePT: $3.0 \times 10^{-3}$ mol L$^{-1}$ and $4.0 \times 10^{-3}$ mol L$^{-1}$; HCl concentration: $3.5$ mol L$^{-1}$; $\lambda_{\text{max}}$: 350 nm.

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5.3. Analysis of selenium(IV) in environmental sample

5.3.1. Vegetable

A 25.0 g finely chopped fresh cabbage (Brassica oleracea var. capitata) sample, from local village in Ahmednagar district, was placed in a 250 mL beaker. It was digested with 10–20 mL concentrated nitric acid for 20 min. After cooling 0.5 mL perchloric acid was added and heating was continued for another 10 min. The residue was cooled and 10 mL water and 5 mL of concentrated hydrochloric acid were added. This mixture was boiled for 10 min to convert...
selenium (VI) into selenium (IV). The solution was evaporated to moist dryness and the residue was dissolved in hot dilute hydrochloric acid and diluted to 50 mL with distilled water. An aliquot (10 mL) of this solution was analysed for selenium(IV) according to the recommended method (Table 6). A Systronics 8130 atomic absorption spectrometer equipped with a hydride generator was used for comparative purposes.

Table 3. Effect of foreign ions.

| Foreign ions | Added as | Tolerance limit (mg) | Foreign ions | Added as | Tolerance limit (mg) |
|--------------|----------|----------------------|--------------|----------|----------------------|
| Mn(II)       | MnCl2.6H2O | 12.0                  | Ca(II)       | CaCl2.2H2O | 50.0                |
| Cd(II)       | CdCl2.2H2O | 12.0                  | Ti(III)      | Ti2O3     | 2.00                |
| Fe(III)      | (NH4)2Fe(SO4)2.12H2O | 13.0        | In(III)      | InCl3.4H2O | 4.00                |
| Hg(II)       | HgCl2    | 1.00                  | Rh(III)      | RhCl3     | 1.00                |
| Bi(III)      | BiCl3    | 11.0                  | Pt(IV)       | H2PtCl6   | 1.50                |
| Ni(II)a      | NiCl2.6H2O | 3.00                  | Ir(III)b     | IrCl3     | 0.40                |
| Cu(II)a      | CuSO4.5H2O | 1.80                  | Os(IV)       | OsO4      | 0.05                |
| Al(III)      | AlCl3.6H2O | 12.0                  | Ru(III)      | RuCl3.3H2O | 0.60               |
| La(III)      | LaCl3.7H2O | 1.00                  | Pd (II)c     | PdCl2     | 0.125               |
| Li(I)        | LiCl     | 15.0                  | Zr(IV)       | ZrOCl2.8H2O | 18.0        |
| Ti(III)      | (Ti2SO4)3 | 12.0                  | As (III)     | As2O3     | 4.00                |
| Mg(II)       | MgCl2.6H2O | 18.0                  | W(VI)        | Na2WO4.2H2O | 15.0         |
| Sn(II)       | SnCl2.2H2O | 2.00                  | Zn(II)       | ZnSO4.7H2O | 50.0               |
| Ga(III)      | GaCl3    | 2.00                  | Be(II)       | BeSO4.2H2O | 18.0               |
| Au(III)      | HAuCl4.3H2O | 1.00                 | Fluoride     | NaF       | 100                 |
| Mo(VI)       | (NH4)2Mo7O24.2H2O | 12.0     | Phosphate    | Na3PO4    | 100                 |
| Sb(III)      | Sb2O3    | 3.00                  | Sulphate     | K2SO4     | 100                 |
| V(V)         | V2O5     | 25.0                  | Succinate    | (CH3COONa)2.6H2O | 100    |
| Ce(IV)       | Ce(SO4)3.4H2O | 0.50                      | Citrate      | C8H12O7.5H2O | 100     |
| Pb(II)       | PbCl2    | 7.00                  | Malonate     | CH2(COONa)2 | 100     |
| U(IV)        | UO2(CH3COO)2.2H2O | 16.0                | Tartrate     | CHO3:COOH | 100     |
| Co(II)       | CoCl2.6H2O | 10.0                  | Acetate      | CH3COONa.3H2O | 100    |
| Ba(II)       | BaCl2.6H2O | 50.0                  | Oxalate      | Na2C2O4.2H2O | 100   |
| Sr(III)      | Sr(NO3)2 | 50.0                  | E.D.T.A      | Na2EDTA   | 100                 |

Note: a Masked with 100 mg EDTA. b Prior extraction of Ir(III). c Prior extraction of Pd (II).

Table 4. Separation of selenium(IV) from binary synthetic mixtures.

| Mixture | Amount taken (µg) | Recovery (%)a | RSD (%) | Chromogenic ligand | Ref. |
|---------|-------------------|---------------|---------|--------------------|------|
| Se(IV)  | 400               | 99.33         | 0.44    | OMePT              | –    |
| Ni(II)b | 100               | 99.13         | 0.52    | DMG                | [30] |
| Se(IV)  | 400               | 99.65         | 0.44    | OMePT              | –    |
| Au(III) | 200               | 99.51         | 0.62    | Rhodamine-B        | [30] |
| Se(IV)  | 400               | 99.62         | 0.27    | OMePT              | –    |
| Bi(III) | 300               | 99.76         | 0.13    | Iodide (KI)        | [30] |
| Se(IV)  | 400               | 99.60         | 0.21    | OMePT              | –    |
| Al(III) | 50                | 99.51         | 0.39    | 8-Hydroxy quinoline| [30] |
| Se(IV)  | 400               | 99.54         | 0.24    | OMePT              | –    |
| Sb(III) | 300               | 99.77         | 0.22    | Iodide (KI)        | [30] |

Note: a Average of six determinations.  
bMasked with 100 mg EDTA.
Table 5. Analysis of pharmaceutical and toilet preparation samples.

| Sample            | Composition                                                                                                      | Certified value of Se(IV) (µg/Tab) | Amount of Se(IV)* found (µg/Tab) | Amount of Se(IV) (µg g⁻¹) | RSD (%) |
|-------------------|------------------------------------------------------------------------------------------------------------------|------------------------------------|---------------------------------|--------------------------|---------|
| Menopace ISO      | Nicotinamide 10 mg, Vit-C 75 mg, Vit-E 20 mg, iron 5 mg, selenium 100 µg                                        | 100                                | 98.87                           | 36.06                    | 0.40    |
| Cardio-Vit plus   | Pyridoxine hydrochloride 3 mg, nicotinamide 100 mg, cyanocobalmin 15 µg, folic acid 1.5 mg, chromium picolinate 250 µg, selenium 100 µg, zinc sulphate monohydrate 61.8 mg | 100                                | 98.82                           | 28.41                    | 0.44    |
| Betared           | Vit-C 100 mg, Vit-E 25 IU, manganese 1.5 mg, beta carotene 10.33 mg, selenium 75 µg                               | 75                                 | 74.11                           | 45.49                    | 0.10    |
| Lyco-First        | Lycopene 6% 5000 µg, Vit-A 2500 IU, Vit-E 10 IU, Vit-C 50 mg, selenium 70 µg                                       | 70                                 | 69.76                           | 23.91                    | 0.33    |
| EC-350            | Vit-C 150 mg, Vit-E 25 mg, alpha lipoic acid 100 mg, selenium 75 µg                                              | 75                                 | 73.79                           | 28.03                    | 0.33    |
| Casera            | Vit-A 2500 IU, Vit-C 100 mg, alphatocopheryl acetate 25 IU, beta carotene 6 mg, selenium 55 µg, zinc 7.5 mg, molybdenum 25 µg | 55                                 | 54.29                           | 17.42                    | 0.68    |
| Selsun shampoo    | Selenium sulphide 2.5% w/v                                                                                       | 275b                               | 273.39 b                        |                          | 0.19    |

Note: *Average of six determinations. bµg of Se(IV) per 2 mL of diluted solution.
5.3.2. Soil

A known amount of selenium was mixed with 20 g of soil sample, and extracted with hydrochloric acid and nitric acid mixture three times. The filtrate of the extract was evaporated to moist dryness and the residue was treated with 20 mL of 10 mol L$^{-1}$ hydrochloric acid and then heated to convert all selenium into selenium(IV). The solution was further diluted with water to give a suitable concentration of selenium. The selenium content was determined from an aliquot of this solution following the recommended method. Results are in good agreement with that of the analysis by AAS.

5.3.3. Water from Bhandardara Dam

One litre of water from Bhandardara Dam (Maharashtra) was filtered through Whatman filter paper and concentrated to about 60 mL by heating on a hot plate. Concentrated nitric acid, 10 mL, was added in this solution. The mixture was heated on a hotplate and evaporated to moist dryness. The residue was dissolved in 10 mL hot dilute hydrochloric acid and the solution was boiled to convert all selenium into selenium(IV); after cooling the solution was transferred into a 25 mL calibrated flask, and analysed by the proposed method. Results are in good agreement with that of the analysis by AAS.

6. Conclusion

O-Methoxyphenyl thiourea (OMePT) is a sensitive and selective reagent for the spectrophotometric determination of selenium(IV). Low concentration (0.012 mol L$^{-1}$) of the OMePT is required for quantitative extraction of selenium(IV). The selenium(IV)–OMePT complex is highly stable. Rapid determination of selenium(IV) at room temperature with low interference of foreign ions is achieved by the recommended method.

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