# AUTHOR'S PUBLISHED WORK

1. Extraction, spectrophotometric and atomic absorption spectrophotometric determination of selenium with N-phenylbenzohydroxamic acid, Analy. Lett. 19(1&2), 25 (1986).
2. Extraction, spectrophotometric and atomic absorption spectrophotometric determination of tellurium with N-phenylbenzohydroxamic acid, Analy. Lett., 20(1), 11 (1977).
3. Preparation and properties of substituted $\alpha$-phenyl cinnamohydroxamic acids, J. Chem. Eng. Data (Communicated).
4. The thermodynamic proton-ligand stability constants of some substituted $\alpha$-phenyl cinnamohydroxamic acids in water-dioxan media. Aust. J. Chem. (Communicated).
5. Extraction and spectrophotometric determination of vanadium in environment, Bull. Soc. Chem. Belgs. (Communicated).
6. Extraction and spectrophotometric determination of bismuth in pharmaceuticals. Analyst (Communicated).
7. Extraction and spectrophotometric determination of titanium in high purity grade metals and alloys, Analyst (Communicated).

ANALYTICAL LETTERS, 19(1&2), 25-36 (1986)

# EXTRACTION, SPECTROPHOTOMETRIC, AND ATOMIC ABSORPTION SPECTROPHOTOMETRIC DETERMINATION OF SELENIUM WITH N-PHENYLBENZOHYDROXAMIC ACID

Keywords: Selenium determination, Spectrophotometric, Atomic absorption spectrophotometric, Hydroxamic acid, Separation from tellurium

Yadvendra K. Agrawal and Kirit H. Desai
Analytical Laboratories, Pharmacy Department,
Faculty of Tech. & Engg., M.S. University of
Baroda, Kalabhavan, Baroda-390 001, INDIA.

## ABSTRACT

Solvent extraction spectrophotometric and atomic absorption spectrophotometric methods for the determination of selenium(IV) in microgram quantities are described. The selenium(IV) forms yellow colored complex with N-phenylbenzohydroxamic acid (PBHA) extractable into chloroform from 7 M $HClO_4$. Se-PBHA complex has maximum absorbance at 345 nm with a molar absorptivity $1.5 \times 10^5$ l $mol^{-1}$ $cm^{-1}$ and Sandell's sensitivity 0.000526 $\mu g/cm^2$. Effect of molarity,

0003-2719/86/1901-0025$3.50/0

reagent concentration, diverse ions on the extraction of selenium complex were studied. The selenium is determined in presence of tellurium.

## INTRODUCTION

Selenium and its compounds have been used commercially in the production of rectifiers, photocells and pigments. Selenium has long been regarded as a toxic metal, because of the wellknown selenium poisoning of cattle[1]("alkali disease and blind staggers") caused by consumption of selenium bearing plants. Selenium and its compounds are toxic and affect various organs. Very small amounts of selenium and selenium derivatives irritate the respiratory tract and cause colds and headache.

Considerable attention has been devoted to trace analysis of selenium in environmental[2] geological[3] and biological[4] samples by various techniques[5-16]. These techniques are time consuming and suffering from interference of various ions or require sophisticated instrumentation.

Diaminobenzidine[5], 4-dimethylamino-1, 2-phenelene diamine and 4-methylthio-1, 2-phenelene diamine[6] reagents were used for the determination of Se(IV) in the absence of oxidizing ions.

Hydroxamic acids have received a great deal of attention as gravimetric, titrimetric and colorimetric reagents for several metal ions[17-19]. In the present investigation a new, rapid solvent extraction, and spectrophotometric method for microgram determination of selenium in presence of tellurium is described.

## EXPERIMENTAL

### Apparatus

The spectrophotometric measurements were made on a Bausch and Lomb, spectronic-20-spectrophotometer, and a GBC 901 atomic absorption spectrophotometer equipped with GBC HG-900 vapor generation accessary. The selenium line at 196 nm with argon gas was used.

### Chemical Reagents

All the chemicals used in this study were of AnalaR and Guaranteed Reagents of British Drug House London or E. Merck, respectively, unless otherwise specified.

The N-phenylbenzohydroxamic acid (PBHA) was freshly synthesised as described elsewhere[20]. Purity was checked by melting point, thin layer chromatography, infrared and ultra violet spectra; and its 0.1% solution was prepared in chloroform.

Metallic selenium (99.99% pure) was dissolved in nitric acid and the solution evaporated to dryness on a waterbath. Perchloric acid was added and the mixture heated on a waterbath, filtered, and diluted to 100 ml with doublely distilled water. The selenium concentration was determined colorimetrically[5].

### Procedure

2.0 ml of selenium solution (3.73 $\mu g$) was transfered into 60-ml separatory funnel followed by 15 ml $HClO_4$ (11.6 M) and 8 ml water (to keep final molarity of $HClO_4$ at 7.0 M). Then 10 ml of chloroform solution of the reagent was added, and the contents were shaken for 5 min. The yellow colored chloroform extract was separated, dried over anhydrous sodium sulfate, and collected into a 25-ml volumetric flask. The extraction was repeated with 2 ml of reagent solution to check the complete extraction of selenium. The anhydrous sodium sulfate was washed with chloroform, and the washings were collected. The extracted layer was diluted by adding chloroform upto the mark, and the absorbance was measured at 345 nm vs the blank which was prepared in the absence of selenium.

## RESULTS AND DISCUSSION

### Absorption Spectra

The yellow colored Se-PBHA complex has a maximum absorbance at 345 nm. The blank has an insignificant absorbance at 345 nm.

### Effect of Molarity

The extraction of selenium was carried out in the 3-9 M $HClO_4$ range and results shown in Table 1. An

TABLE 1

Effect of Molarity of Perchloric acid on the Extraction of Se-PBHA Complex in Chloroform

Se(IV) : 0.149 ppm
PBHA : 0.1%, 10 ml in chloroform

Solvent : Chloroform
Color : Yellow
$\lambda$max : 345 nm

| Molarity of $HClO_4$ | % Extraction | Molar absorptivity $l\ mol^{-1}\ cm^{-1}$ |
|---|---|---|
| 3.0 | 0 | 0 |
| 4.0 | 0 | 0 |
| 5.0 | 0 | 0 |
| 6.0 | 31 | $4.7 \times 10^4$ |
| 7.0 | 100 | $1.5 \times 10^5$ |
| 8.0 | 54 | $8.0 \times 10^4$ |
| 9.0 | 48 | $7.3 \times 10^4$ |

optimum (100%) extraction was obtained at 7.0 M $HClO_4$ with higher or lower acidity tending to decrease the % extraction.

Effect of Solvent

The selenium was extracted with various solvents including chloroform, toluene, and benzene. Chloroform

TABLE 2

Effect of Solvents on the Extraction of Selwnium with N-Phenylbenzohydroxamic acid

Se(IV) : 0.149 ppm
PBHA : 10 ml (0.1%) in chloroform
Molarity : 7.0 M $HClO_4$
Solvent : Chloroform
Color : Yellow
$\lambda_{max}$ : 345 nm

| Solvents | % Extraction | Molar absorptivity $l\ mol^{-1}\ cm^{-1}$ |
|---|---|---|
| Chloroform | 100 | $1.5 \times 10^5$ |
| Toluene | 66.6 | $1.0 \times 10^5$ |
| Benzene | NE | NE |

NE = Not extracted

was found the most suitable solvent for extraction of selenium (Table 2).

Selenium was extracted at 7 M $HClO_4$ while varying the reagent concentrations and results shown in Table 3. The results showed that 10 ml of 0.1% N-phenylbenzohydroxamic acid (PBHA) was adequate for the quantitative extraction of selenium. Lower concentration gave

TABLE 3

Effect of PBHA Concentration

Se(IV) : 0.149 ppm
PBHA : 10 ml in chloroform
Molarity : 7.0 M $HClO_4$

Solvent : Chloroform
Color : Yellow
$\lambda$max : 345 nm

| N-PBHA added 0.1% (ml) | Absorbance | Molar absorptivity $l\ mol^{-1}\ cm^{-1}$ |
|---|---|---|
| 2.5 | 0.16 | $8.0 \times 10^4$ |
| 5.0 | 0.23 | $1.2 \times 10^5$ |
| 7.5 | 0.29 | $1.5 \times 10^5$ |
| 10.0 | 0.29 | $1.5 \times 10^5$ |
| 15.0 | 0.29 | $1.5 \times 10^5$ |

incomplete extraction. Hence all the extractions were done with 10 ml of 0.1% reagent.

Effect of Shaking time and Stability of the Color

It was confirmed that manual shaking time of 5-10 min was adequate for the complete extraction of selenium. However, a longer shaking time had no effect on the equilibrium system. The complex was stable under optimum conditions and the absorbance remained unchanged for several hours.

Validity of the Beer's Law

Beer's law was obeyed in the range of 0.015-0.6 ppm of selenium. The molar absorptivity was $1.5 \times 10^5$ $l\ mol^{-1}\ cm^{-1}$ at 345 nm. The Sandell's sensitivity is 0.000526 $\mu g/cm^2$.

Effect of Diverse Ions

Different amounts of diverse ions were added to a fixed amount of selenium and extracted according to the procedure. Most of the metal ions associated with selenium do not interfere. The interference of the large amount of $Te^{4+}$ can be tolerated by masking with thioglycolic acid. The data are shown in Table 4.

Determination of Selenium in the Presence of Tellurium

Selenium and tellurium have similar analytical and chemical properties and usually occur together. Hence, separation of selenium from tellurium presents some importance, particularly as tellurium causes an interference in the measurement of selenium.

Tellurium forms a reddish-yellow complex with PBHA extractable into $CHCl_3$ from highly acidic media together with selenium. However, tellurium can be selectively masked with thioglycolic acid, the resulting Te-thioglycolic acid complex was

TABLE 4

Effect of Diverse Ions in the Determination of Selenium(IV) with N-Phenylbenzohydroxamic acid

Selenium : 0.149 ppm    PBHA : 0.1%, 10 ml in chloroform    Solvent :Chloroform

Color : Yellow    Absorbance: 0.29    $\lambda_{max}$ : 345 nm

| Ions | Added as | Amount added mg | Absorbance | Ions | Added as | Amount added mg | Absorbance |
|---|---|---|---|---|---|---|---|
| $Al^{3+}$ | $Al(NO_3)_2$ | (60) | 0.29 | $Nb^{5+}$ | $Nb_2O_5$ | (60) | 0.30 |
| $As^{3+}$ | $AsF_3$ | (60) | 0.28 | $Ni^{2+}$ | $NiCl_2$ | (60) | 0.29 |
| $Ba^{2+}$ | $BaCl_2$ | (60) | 0.29 | $Pb^{2+}$ | $Pb(C_2H_3O_2)_2 \cdot 4H_2O$ | (40) | 0.29 |
| $Be^{2+}$ | $BeSO_4$ | (80) | 0.29 | | | | |
| $Bi^{3+}$ | $BiONO_3$ | (60) | 0.28 | $Sb^{3+}$ | $SbCl_3$ | (60) | 0.29 |
| $Ca^{2+}$ | $Ca(NO_3)_2$ | (80) | 0.30 | $Sn^{2+}$ | $SnCl_2$ | (60) | 0.29 |
| $Cd^{2+}$ | $CdSO_4$ | (60) | 0.29 | $Ta^{5+}$ | $Ta_2O_5$ | (60) | 0.30 |
| $Co^{2+}$ | $Co(C_2H_3O_2)_2 \cdot 4H_2O$ | (60) | 0.28 | $Te^{4+a}$ | $Te(ClO_4)_4$ | (40) | 0.29 |
| $Cr^{3+}$ | $CrO_3$ | (40) | 0.29 | $Th^{4+}$ | $Th(NO_3)_4$ | (60) | 0.29 |
| $Cu^{2+}$ | $CuSO_4$ | (60) | 0.30 | $Ti^{4+}$ | $TiCl_4$ | (60) | 0.28 |
| $Ga^{3+}$ | $GaI_3$ | (40) | 0.30 | $V^{5+b}$ | $NH_4VO_3$ | (40) | 0.28 |
| $Mg^{2+}$ | $Mg(NO_3)_2$ | (60) | 0.29 | $Zn^{2+}$ | $ZnCl_2$ | (40) | 0.28 |
| $Mn^{2+}$ | $Mn(C_2H_3C_2)_2 \cdot 4H_2O$ | (60) | 0.29 | $Zr^{4+}$ | $Zr(NO_3)_4 \cdot 5H_2O$ | (40) | 0.28 |

a = masked with thioglycolic acid; b = masked with ascorbic acid.

TABLE 5

Determination of Selenium in presence of Tellurium

| Selenium taken (ppm) | Tellurium added (mg) | Selenium found (ppm)** | | Relative % error |
|---|---|---|---|---|
| | | Present* Method | AAS | |
| 0.20 | 40 | 0.20 | 0.19 | 0.0 |
| 0.50 | 40 | 0.49 | 0.50 | 2.0 |
| 1.00 | 60 | 1.01 | 1.00 | 1.0 |
| 1.50 | 60 | 1.50 | 1.51 | 0.0 |
| 2.00 | 60 | 2.00 | 2.00 | 0.0 |
| 2.50 | 70 | 2.49 | 2.49 | 0.4 |

* Six determinations

** The regression equation for the two methods is

$S = 0.996\ A + 0.005;\quad r = 0.999$

Where S = Selenium+Spectrophotometric, A = Selenium by atomic absorption spectrophotometry and r = Correlation coefficient.

soluble in water and selenium can be extracted as Se-PBHA complex. The results are shown in Table 5.

Stoichiometry of the Se-PBHA Complex

The stoichiometry of the Se-PBHA complex was determined by taking a fixed amount of metal and gradually increasing the amount of reagent. The slope of the plot of log $D_M$ (the logarithm of the distribution coefficient of the metal) against the log C (the logarithm of the ligand concentration) was found to be 2.0, confirms that the selenium to PBHA ratio is 1:2.

The probable reaction based on the above results is as follows:

$$Se(ClO_4)_4 + 2\ HA \rightleftharpoons Se(A)_2\ (ClO_4)_2 + 2HClO_4$$

Where HA = PBHA

## ACKNOWLEDGEMENT

The authors are grateful to CSIR, New Delhi for awarding the research project and providing financial assistance.

## REFERENCES

1. E.A. Cerwenka Jr., and W.C. Cooper, Arch. Environ. Health, 3, 71, (1961).
2. Y. Hashimoto and J.W. Winchester, Environ. Sci. Technol, 1, 338, (1967).
3. V. Lavrakas, T.J. Golembeski, G. Pappas, J.E. Gregory and H.L. Wedlick, Anal. Chem. 46, 952 (1974).
4. D.C. Washington, "Selenium in Nutrition", National Academy of Sciences, (1971).

5. J. Hoste, Anal. Chim. Acta. 2, 402 (1948).
6. E. Sawicki, Anal. Chem. 29, 1376 (1957).
7. O.E. Olson, J. Assoc. Office. Anal. Chem. 52, 627 (1969).
8. O.J. Kronborg and E. Steinnes, Analyst, 100, 835 (1975).
9. Y. Shimoishi, Analyst, 101, 298 (1976).
10. P.N. Vijan. and G.R. Wood, Talanta, 1, 22 (1976).
11. M.W. Balades, J.A. Dalziel and C.M. Elson, J. Assoc. Office. Anal. Chem. 59, 1234 (1967).
12. G.P. Bound and S. Forbes, Analyst, 103, 176 (1978).
13. R.W. Andrews and D.C. Johnson, Anal. Chem. 47, 294 (1975).
14. J.C. Chambers and B.E. McCleilan, Anal. Chem. 48, 2061 (1976).
15. S. Ooba and S. Ueno, Talanta, 22, 51 (1975).
16. J.M. Watkinson, Anal. Chem. 38, 92 (1966).
17. Y.K. Agrawal and S.A. Patel, Revs. Anal. Chem. 4, 237 (1981).
18. Y.K. Agrawal, Revs. Anal. Chem. 5, 3 (1980).
19. Y.K. Agrawal and R.K. Jain, Revs. Anal. Chem. 6, 49 (1982).
20. U. Pryadarshini and S.G. Tandon, J. Chem. Eng. Data, 12, 143 (1967).

Received August 26, 1985
Accepted November 5, 1985

ANALYTICAL LETTERS, 20(1), 11-22 (1987)

# EXTRACTION, SPECTROPHOTOMETRIC AND ATOMIC ABSORPTION SPECTROPHOTOMETRIC DETERMINATION OF TELLURIUM WITH N-PHENYLBENZOHYDROXAMIC ACID

Key words: Tellurium determination, Spectrophotometry, Atomic absorption spectrophotometry, Hydroxamic acid, Separation from selenium.

Kirit H. Desai and Yadvendra K. Agrawal

Analytical Laboratories, Pharmacy Department,
Faculty of Technology and Engineering,
M.S. University of Baroda, Baroda-390 001,
INDIA.

## ABSTRACT

Solvent extraction, spectrophotometric and atomic absorption spectrophotometric determination of tellurium(IV) in nanogram levels are described. The tellurium(IV) forms yellow colored complex with N-phenylbenzohydroxamic acid (PBHA) which is extractable into chloroform from 7 M $HClO_4$. Te-PBHA complex has a maximum absorbance at 345 nm with the molar absorptivity $3.5 \times 10^4$ l $mol^{-1}$ $cm^{-1}$ and Sandell's sensitivity 0.00365 $\mu g/cm^2$. The tellurium(IV) is also determined in the range of 0.01 - 0.08 ppm by flameless atomic absorption spectrophotometry using GBC HG 900 vapor generation accessary. Effect of molarity,

reagent concentrations, diverse ions on the extraction of tellurium complex were studied. The tellurium was determined in presence of selenium and also in standard samples.

## INTRODUCTION

In recent years tellurium steels have been widely used in automation, radioengineering and semiconductor technology. This has increased the interest in tellurium, and led to large scale studies on the inorganic and analytical chemistry of tellurium.

Numerous methods have been proposed for the spectrophotometric determination of traces of tellurium. Several reagents like bromopyrogallol[1], rhodamine 6G[2], malachite green[3], bismuthol II[4], thiourea[5], sodium diethyl dithiocarbomate[6,7] were used for the determinations of tellurium(IV).

In recent years more sophisticated techniques such as neutron activation[8], isotope dilution mass spectrophotometry[9] and atomic absorption spectrophotometry (AAS) have been employed to analyse tellurium. The major difficulty in the determination of tellurium by atomic absorption spectrophotometry is that the limits are high and the major elements constituting the silicate matrix must be separated before estimation.

Hydroxamic acids have received a great deal of attention as spectrophotometric gravimetric, titrimetric and colorimetric reagents for several metal ions[10-13]. In the present investigation a new rapid solvent extraction, and spectrophotometric method for nanogram levels of tellurium in presence of selenium is described. A sensitive flameless atomic absorption spectrophotometric method is also described.

## EXPERIMENTAL

### Apparatus

The spectrophotometric measurements were made on a Bausch and Lomb, Spectronic 20, spectrophotometer, GBC 901 atomic absorption spectrophotometer (AAS) with GBC HG 900 vapor generation accessary is used for AAS determination of tellurium. The tellurium line at 214.3 nm with lamp current 7 mA and argon gas was used.

### Chemical Reagents

All the chemicals used in this study were of AnalaR and Guaranteed Reagents of British Drug House, London or E. Merck, respectively, unless otherwise specified.

The N-phenylbenzohydroxamic acid (PBHA) was freshly synthesised as described elsewhere[14]. Its purity was checked by mp, TLC, infrared and ultraviolet spectra, and its 0.2% solution was prepared in chloroform.

0.5 gm of tellurium metal (99.99) was dissolved in 20 ml of con. HCl and $HNO_3$ (1:3) and diluted to 500 ml with doubly distilled water. This solution contains 1000 ppm of tellurium.

### Extraction with PBHA

2.0 ml of tellurium solution (20 ppm) was transferred into 60-ml separatory funnel followed by 15 ml

$HClO_4$ (11.6 M) and 8 ml water (to keep final molarity of $HClO_4$ to 7.0 M). Then 10 ml of chloroform solution of the reagent (PBHA) was added, and the contents were shaken for 10 min. The yellow colored chloroform extract was separated, dried over anhydrous sodium sulfate, and collected into a 25-ml volumetric flask. The extraction was repeated with 2.0 ml of reagent solution to check the complete extraction of tellurium. The anhydrous sodium sulfate was washed with chloroform, and the washings were collected. The extracted layer was diluted with chloroform upto the mark, and the absorbance was measured at 345 nm against the blank which was prepared in the similar manner in absence of tellurium.

### Atomic Absorption Spectrophotometric Determination

For the AAS measurements the tellurium was back extracted with 0.1 M HCl from the chloroform extract and estimated using a GBC HG 900 vapor generation accessary.

## RESULTS AND DISCUSSION

### Absorption Spectra

The yellow colored Te-PBHA complex has a maximum absorbance at 345 nm. The blank does not absorb at this wavelength. The molar absorptivity is $3.5 \times 10^4$ $l\ mol^{-1}\ cm^{-1}$. The data obtained by the present method are in good agreement with those obtained by AAS and the correlation coefficient is 0.998.

TABLE 1

Effect of Molarity of Perchloric acid on the Extraction of Te-PBHA Complex in Chloroform.

Te(IV) : 0.80 ppm
**PBHA** : 10 ml (0.2%) in chloroform.

Solvent : Chloroform
Color : Yellow
$\lambda$max : 345 nm

| $HClO_4$ (M) | % Extraction | Molar absorptivity 1 mol$^{-1}$ cm$^{-1}$ |
|---|---|---|
| 5.0 | 0 | 0 |
| 5.5 | 0 | 0 |
| 6.0 | 18 | $6.3 \times 10^3$ |
| 6.5 | 46 | $1.6 \times 10^4$ |
| 7.0 | 100 | $3.5 \times 10^4$ |
| 7.5 | 69 | $2.4 \times 10^4$ |
| 8.0 | 58 | $2.0 \times 10^4$ |

Effect of Molarity

The extraction of tellurium was carried out in the range of 6-8 M $HClO_4$. The results given in Table 1 indicate that the 50% extraction is obtained at 6.5 M $HClO_4$ and maximum (100%) extraction is at 7.0 M $HClO_4$. Above 7.0 M the extraction decreases and hence all the ex tions were carried out at 7.0 M $HClO_4$.

TABLE 2

Effect of Solvents on the Extraction of Tellurium with N-phenylbenzohydroxamic acid.

Te(IV) : 0.80 ppm
PBHA : 10 ml (0.2%) in chloroform
$HClO_4$ : 7.0 M
Solvent : Chloroform
Color : Yellow
λmax : 345 nm

| Solvents | % Extraction | Molar absorptivity l $mol^{-1}$ $cm^{-1}$ |
|---|---|---|
| Chloroform | 100 | $3.5 \times 10^4$ |
| Toluene | 54 | $1.9 \times 10^4$ |
| Isoamyl alcohol | NE | NE |
| Methylisobutyl ketone | NE | NE |

NE = Not Extracted

## Effect of Solvent

The tellurium was extracted with various solvents including chloroform, toluene, isoamyl alcohol and methyl isobutyl ketone. Chloroform was found the most suitable solvent for extraction of tellurium (Table 2).

## Effect of Reagent Concentration

Tellurium was extracted at 7 M $HClO_4$ with varying the reagent concentrations. The results given in

TABLE 3

Effect of PBHA Concentration

Te(IV) : 0.80 ppm
$HClO_4$ : 7.0 M

Solvent: Chloroform
Color : Yellow
λmax : 345 nm

| PBHA 0.2% added ml. | Absorbance | Molar absorptivity $l\ mol^{-1}\ cm^{-1}$ |
|---|---|---|
| 2.5 | 0.12 | $1.9 \times 10^4$ |
| 5.0 | 0.16 | $2.5 \times 10^4$ |
| 7.5 | 0.22 | $3.5 \times 10^4$ |
| 10.0 | 0.22 | $3.5 \times 10^4$ |
| 15.0 | 0.22 | $3.5 \times 10^4$ |

Table 3 show that 10 ml of 0.2% N-phenylbenzohydroxamic acid (PBHA) is adequate for the quantitative extraction of tellurium. Lower PBHA concentration gave incomplete extraction. Hence all the extractions were carried out with 10 ml of 0.2% PBHA. Higher PBHA concentration has no adverse effect on the extraction of tellurium.

Effect of Shaking time and Stability of the Color

It was confirmed that manual shaking time of 5-10 min was adequate for the complete extraction of tellurium. However, a longer shaking time had no adverse effect on the equilibrium system. The complex is stable under optimum conditions and the absorbance remained unchanged for several hrs.

### Validity of the Beer's Law

Beer's law is obeyed in the range of 0.10 to 5.0 ppm of tellurium. The Sandell's sensitivity is 0.00365 $\mu g/cm^2$. The Beer's law limit for the flameless atomic absorption spectrophotometric determination of tellurium is 0.01 - 0.08 ppm.

### Effect of Diverse Ions

Different amounts of diverse ions were added to a fixed amount of tellurium and extracted according to the procedure. Most of the metal ions associated with tellurium do not interfere. The interference of large amount of Se(IV) can be tolerated by masking it with the mixture of citric acid, sodium tartarate and EDTA. The data are shown in Table 4.

### Determination of Tellurium in the presence of Selenium

Tellurium and selenium have similar analytical and chemical properties and usually occur together. Hence, separation of tellurium from selenium is very difficult.

Selenium forms reddish yellow complex with PBHA extractable into chloroform from highly acidic media together with tellurium. However, selenium can be selectively masked with the mixture of citric acid, sodium tartrate and EDTA, the resulting complex was soluble in water

### TABLE 4

### Effect of Diverse Ions in the Determination of Tellurium with N-phenylbenzohydroxamic acid.

Tellurium : 0.80 ppm; PBHA: 10 ml (0.2%) in Chloroform; Solvent: Chloroform

$HClO_4$: 7.0 M; Color: Yellow; Absorbance: 0.22; $\lambda$max: 345 nm

| Ions | Added as | Amount added mg | Absorbance | Ions | Added as | Amount added mg | Absorbance |
|---|---|---|---|---|---|---|---|
| $Ag^{+}$ | $AgNO_3$ | (40) | 0.22 | $La^{3+}$ | $La(NO_3)_3\ 6H_2O$ | (40) | 0.21 |
| $As^{3+}$ | $AsF_3$ | (40) | [illegible] | [illegible] | [illegible] | (50) | 0.22 |
| $Ba^{2+}$ | $BaCl_2$ | (40) | 0.21 | $Nd^{3+}$ | $Nd(NO_3)_3$ | (40) | 0.22 |
| $Be^{2+}$ | $BeSO_4$ | (50) | 0.22 | $Ni^{2+}$ | $NiCl_2$ | (40) | 0.22 |
| $Ca^{2+}$ | $Ca(NO_3)_2$ | (40) | 0.22 | $Sn^{2+}$ | $SnCl_2$ | (40) | 0.22 |
| $Cd^{2+}$ | $CdSO_4$ | (50) | 0.21 | $Se^{4+}$ | $Se(ClO_4)_4$ | (40)[a] | 0.22 |
| $Co^{2+}$ | $Co(C_2H_3O_2)_2\ 4H_2O$ | (40) | 0.22 | $Ti^{4+}$ | $TiCl_4$ | (50) | 0.22 |
| $Cu^{2+}$ | $CuSO_4$ | (40) | 0.22 | $Th^{4+}$ | $Th(NO_3)_4$ | (40) | 0.21 |
| $Ga^{3+}$ | $GaI_3$ | (40) | 0.22 | $V^{5+}$ | $NH_4VO_3$ | (40)[b] | 0.22 |
| $In^{3+}$ | $InCl_3$ | (40) | 0.22 | $Zn^{2+}$ | $ZnCl_2$ | (40) | 0.22 |

a = masked with mixture of citric acid + Na-tartrate + EDTA,

b = masked with ascorbic acid.

TABLE 5

Determination of Tellurium in presence of Selenium

| Tellurium taken (ppm) | Selenium added (mg) | Tellurium found (ppm)** | | Coefficient variation |
|---|---|---|---|---|
| | | Present method* | AAS | % |
| 1.0 | 50 | 1.00 | 0.99 | 1.00 |
| 3.0 | 50 | 3.01 | 3.00 | 0.66 |
| 5.0 | 60 | 5.01 | 3.00 | 0.20 |
| 7.0 | 60 | 6.99 | 7.00 | 0.28 |
| 9.0 | 70 | 9.00 | 9.00 | 0.22 |
| 11.0 | 70 | 10.99 | 10.99 | 0.27 |

* Seven determinations

** The regression equation for the two methods is
$T = 0.998\,A +$ [illegible] 1.000
Where T = Tellurium by spectrophotometry, A = Tellurium by AAS and r = Correlation coefficient.

and tellurium can be extracted as Te-PBHA complex. The results are shown in Table 5.

### Stoichiometry of the Te:PBHA Complex

The stoichiometry of the Te-PBHA complex was determined by taking a fixed amount of metal and gradually increasing the amount of reagent. The slope of the plot of log $D_M$ (the logarithm of the distribution coefficient of the metal) against the log C (the logarithm of the ligand concen-

TABLE 6

Determination of Tellurium in NBS Cartridge Brasses

| Sample | Certified Te % | Te found % | Coefficient of variation % |
|---|---|---|---|
| NBS - 1100 | 0.0035 | 0.0034 | 5.88 |
| NBS - 1101 | 0.0015 | 0.0013 | 7.69 |
| NBS - 1102 | 0.0003 | 0.0003 | 16.66 |

tration)[15] was found to be 2.2 confirms that the tel ium to PBHA ratio is 1:2.

The probable reaction based on the above results is as follows:

$$Te(ClO_4)_4 + 2\ HA \rightleftharpoons Te(A)_2(ClO_4)_2 + 2HClO_4$$

where HA = PBHA.

Determination of Tellurium in NBS Cartridge brasses

The method was also applied to three standard reference cartridge brass samples. The results of these analyses are shown in Table 6.

ACKNOWLEDGEMENT

One of the author (KHD) is grateful to CSIR, New Delhi for awarding the Fellowship.

REFERENCES

1. G.G. Shitreva, E.K. Poluektova and V.A. Nazarenko, Zh. Anal. Khim., 30(6), 1166 (1975).

2. V.G. Skripchuk and V.I. Murashova, Zh. Anal. Khim. 29(9), 1823 (1974).

3. Xu. Hong and Liu Song Jun, Kao Teng Hsueh Hsiao Hua Hsueh Hsueh Pao, 2(2), 261 (1981).

4. V.I. Buser and L.N. Simonova, Zh. Akh., 22, 1850 (1967).

5. V.M. Tarayan and A.A. Sarkisyan, Zh. Nkh., 10, 2684 (1965).

6. H. Bode, Z. Anal. Chem., 143, 182 (1954).

7. H. Bode and H. Rosenthin, Z. Anal. Chem., 164, 232 (1958).

8. E.P. Mignonsin and I. Roelandts, Chem. Geol., 16, 137 (1975).

9. C.L. Smith, J.R. DeLaeter and K.J.R. Rosman, Geochim. Cosmochim. Acta, 41, 676 (1977).

10. Y.K. Agrawal and K.H. Desai, Anal. Letter., 19(1-2), 25 (1986).

11. Y.K. Agrawal and S.A. Patel, Revs Anal. Chem., 4, 237 (1981).

12. Y.K. Agrawal, Revs. Anal. Chem., 5, 3 (1980)

13. Y.K. Agrawal and R.K. Jain, Revs. Anal. Chem., 6, 49 (1982).

14. U. Pryadarshini and S.G. Tandon, J. Chem. Eng. Data, 12, 143 (1967).

15. B. Branko Tomazic and W. Jerome O'Laughlin, Anal. Chem., 45, 1519 (1973).

Received August 25, 1986
Accepted October 14, 1986