# AUTHOR'S PUBLISHED WORK

- Extraction, spectrophotometric and atomic absorption spectrophotometric determination of selenium with N-phenylbenzohydroxamic acid, Analy. Lett. <u>19(1&2)</u>, 25 (1986).
- Extraction, spectrophotometric and atomic absorption spectrophotometric determination of tellurium with N-phenylbenzohydroxamic acid, Analy. Lett., <u>20(1)</u>, 11 (1977).
- 3. Preparation and properties of substituted dephenyl cinnamohydroxamic acids, J. Chem. Eng. Data (Communicated).
- 4. The thermodynamic proton-ligand stability constants of some substituted ∝-phenyl cinnamohydroxamic acids in water-dioxan media. Aust. J. Chem. (Communicated).
- 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

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### 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-phenylbenzotydroxamic acid (PBHA) extractable into chloroform from 7 M HClO<sub>4</sub>. Se-PBHA complex has maximum absorbance at 345 nm with a molar absorptivity 1.5 x  $10^5$  l mol<sup>-1</sup> cm<sup>-1</sup> and Sandell's sensitivity 0.000526 / 4g / cm<sup>2</sup>. Effect of molarity,

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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<sup>1</sup>("alkali disease and blind staggers") caused by connumption 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<sup>2</sup> geological<sup>3</sup> and biological<sup>4</sup> samples by various techniques<sup>5-16</sup>. These techniques are time consuming and suffering from interference of various ions or require sophisticated instrumentation.

Diaminobenzidine<sup>5</sup>, 4-dimethylamino-1, 2-phenelene diamine and 4-methylthio-1, 2-phenelene diamine<sup>6</sup> 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

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reagents for several metal ions<sup>17-19</sup>. 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<sup>20</sup>. 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. Perchlogic 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<sup>5</sup>.

### Procedure

2.0 ml of selenium solution  $(3.73 \ \mu g)_{Was}$ transfered into 60-ml separatory funnel followed by 15 ml HClO<sub>4</sub> (11.6 M) and 8 ml water (to keep final molarity of HClO<sub>4</sub> at 7.0 M). Then 10 ml of chloroform solution of the reagent was adaed, 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 SOLUTER to check the complete extraction of selenium. The and difference 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<sub>L</sub> range and results shown in Table 1. An

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- 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		: Chloroform : Yellow : 345 nm
Molarity of HClO <sub>4</sub>	% Extraction	Molar absorptivity 1 mol <sup>-1</sup> cm <sup>-1</sup>
3.0	0	0 ′
4.0	0	Ο,
5.0	0	0
6.0	31	$4.7 \times 10^4$ 1.5 x 10 <sup>5</sup>
7.0	100	$1.5 \times 10^5$
8.0	54	$8.0 \times 10^4$
9.0	48	7.3 x 10 <sup>4</sup>

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

# Effect of Solvent

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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

So(IV)	:	0.149 ppm	Solvent	:	Chloroform
PBHA	1	10 ml (0.1%) in	Color	:	Yellow
		chloroform	$\lambda_{\max}$	:	345 nm
Molari ty	:	7.0 м нс104			

Solvents	% Extraction	Molar absorptivity 1 mol <sup>-1</sup> cm <sup>-1</sup>
('h].oroform	100	1.5 x 10 <sup>5</sup>
loluene	66.6	1.0 x 10 <sup>5</sup>
henzone	NE	NE

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

Selenium was extracted at 7 M HClO<sub>4</sub> 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

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## TABLE 3

Effect of FBHA Concentration

Se(IV)	: 0.149 ppm	Solvent : Chloroform
PBHA	: 10 ml in chloroform	Color : Yellow
Molarity	: 7.0 M HClo4	$\lambda_{\max}$ : 345 nm

N-PBHA added 0.1% (ml)	Absorbance	Molar absorptivity l mol <sup>-1</sup> cm <sup>-1</sup>
2.5	0.16	8.0 x 10 <sup>4</sup>
5.0	0.23	$1.2 \times 10^5$
7.5	0.29	$1.5 - 10^5$ $1.5 \times 10^{3}$
10.0	0,29	1.5 x 10 <sup>5</sup>
15.0	0,29	1.5 x 10 <sup>5</sup>

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  $1.5 \times 10^5$  1 mol<sup>-1</sup> cm<sup>-1</sup> at 345 nm. The Sandell's sensitivity is 0.000526 /  $Mg/cm^2$ .

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 FBHA extractable into CHCl<sub>3</sub> from highly acidic media together with selenium. However, tellurium can be selectively masked with thioglycolic acid, the resulting Te-thioglycolic acid complex was

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TABLE	

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

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hydroxamic acid

Selenium	: 0.149 ppm	PBHA : 0.1%, 10 ml in chloroform	10 ml in chl	orofor		Solvent :Chloroform	Corm
Color	: Yellow	Absorbance: 0.29			Amax	: 345 nm	٩
Ions	Added as	Amount added mg	Absorbance	Ions	Add <b>ed</b> as	Amount added mg	Amount Absorbance dded mg
A1 <sup>3+</sup>	A1 (NO, ),	(90)	0+29	<sup>Rb</sup> <sup>5</sup> +	Mb <sub>2</sub> 0 <del>5</del>	(99)	0*30
As <sup>3+</sup>	ASFa	(09)	0.28	hi <sup>2+</sup>	NICL	(09)	0.29
Ba <sup>2+</sup>	Baci	(09)	0.29	Pb <sup>2+</sup>	$Pb(c_{2}H_{3}O_{2})_{2}$ .	(97)	0.29
Be <sup>2+</sup>	$Besc_{h}$	(80)	0.29		4H20		
B1 <sup>3+</sup>	BIONOz	(09)	0.28	sb <sup>5+</sup>	sbcl <sub>3</sub>	( 60 )	0.29
ca <sup>2+</sup>	ca(NO <sub>x</sub> ),	(80)	0.30	sn <sup>2+</sup>	snc1 <sub>2</sub>	(09)	0.29
cd <sup>2+</sup>	$cdso_{h}$	(09)	0.29	Ta <sup>2+</sup>	Ta205	(60)	0.30
co <sup>2+</sup>	Co(C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> ) <sub>2</sub> .4H <sub>2</sub> O	<b>,0 (</b> 60)	0.28	Tetta	Te(Clo <sub>4</sub> )4	(91)	0.29
ۍ م	Cr0, Cr0,	(07)	0.29	$_{\rm Th}^{4+}$	$Th(NO_3)_4$	(09)	0.29
cu <sup>2+</sup>	cuso,	(09)	0.30	11 ++	Ticl4	(60)	0.28
Ga <sup>3+</sup>	Galz	(077)	0.30	q+cv	NH4V03	(140)	0.28
	Mg(NO <sub>x</sub> ),	(09)	0.29	+2,42	ZnCL	(07)	0.28
	Mn(c <sub>2</sub> H <sub>3</sub> c <sub>2</sub> ) <sub>2</sub> .4H <sub>2</sub> 0	( 09 )	0.29	2r.#	Zr(NO <sub>3</sub> )4.5H <sub>2</sub> 0		0.28

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

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### TABLE 5

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Gelenium	Tellurium	Selenium fou	ind (ppm)*	Relative
taken (j.pm)	ndded (mg)	Present <sup>*</sup> Method	AAS	% error
0,20	140	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

Delermination of Selenium in presence of Tellurium

\* Six determinations

\*\* The regression equation for the two methods is
S = 0.996 A + 0.005; r = 0.999
where S = Selenium+Spectrophotometric, A = Selenium
by atomic absorption spectrophotometry and
r = Correlation coefficient.

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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

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the amount of reagent. The slope of the plot of  $\log D_{\rm M}$  (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<sub>4</sub>)<sub>4</sub> + 2 HA 
$$=$$
 Se(A)<sub>2</sub> (ClO<sub>4</sub>)<sub>2</sub> + 2HClO<sub>4</sub>

Where HA = PBHA

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# EXTRACTION, SPECTROPHOTOMETRIC AND ATOMIC ADSORPTION SPECTROPHOTOMETRIC DETERMINATION OF TELLURIUM WITH N-PHENYLBERZOHYDROXAFIC ACID

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

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### ABSTRACT

Solvent extraction, spectrophotometric and domic 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 extra table into chloroform from 7 K HClO<sub>4</sub>. Te-PBHA complex has a maximum absorbance at 045 nm with the molar absorptivity 3.5 x 10<sup>4</sup> 1 mol<sup>-1</sup> cm<sup>-1</sup> and Sandell's sensitivity 0.00365 /<sup>1</sup>g/cm<sup>2</sup>. 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,

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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; ai i led to large scale studies on the inorganic and analytical chemistry of tellurium.

Numerous methods have been proposed for the spectrophotom tric determination of traces of tellurium. Several rengents like bromopyrogallol<sup>1</sup>, rhodamine  $6G^2$ , malachite (reen<sup>3</sup>, bismuthol II<sup>4</sup>, thiourea<sup>5</sup>, sodium diethyl dithio arbomate<sup>6,7</sup> were used for the determinations of tellurium(IV).

In recent years more sophisticated techniques such as new ron activation<sup>8</sup>, isotope dilution mass spectrophoto-Métry<sup>9</sup> and atomic absorption spectrophotometry (AAS) have been e ployed to analyse tellurium. The major difficulty in the determination of tellurium by atomic absorption spectr photometry is that the limits are high and the major lements constituting the silicate matrix must be separa ed before estimation.

H droxamic acids have received a great deal of attention a spectrophotometric gravimetric, titrimetric and colori etric reagents for several metal ions $^{10-13}$ . In the

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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<sup>14</sup>. 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<sub>11</sub> (11.6 M) and 8 ml water (to keep final molarity of H lO<sub>4</sub> to 7.0 M). Then 10 ml of chloroform solution of t e reagent (FBHA) was added, and the contents were shak n for 10 min. The yellow colored chloroform extr ct was separated, dried over anhydrous sodium sulf te, 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 HNNydFGus sodium sulfate was washed with chloroform, and the Washings were collected. The extracted layer was lituted with chloroform upto the mark, and the abso bonce 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

Abscrption Spectra

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

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# TABLE 1 Effect of holarity of Perchloric acid on the Extrition of Te-PBHA Complex in Chloroform. Te(IV) : 0.80 ppm Solvent : Chloroform PBHA : 10 ml (0.2%) in Color : Yellow chloroform. Xmax : 345 nm

HC104 (M)	% Extraction	Noler absorptivity l mol <sup>-1</sup> cm <sup>-1</sup>
5.0	0	0
5.5	0	0
6.0	18	6.3 x 10 <sup>3</sup>
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 x 10 <sup>4</sup>

# Effect of Molarity

The extraction of tellurium was carried out in the range of 6-8 M HClO<sub>4</sub>. The results given in Table 1 indicate that the 50% extraction is obtained at 6.5 M HClO<sub>4</sub> and maximum (100%) extraction is at 7.0 M HClO<sub>4</sub>. Above: 7.0 M the extraction decreases and hence all the ex.

				TABL	E 2				ŧ
Elfec	ct c	of Sol	Lvents	on t	he	Extract	tion	of	Tellurium
ì		wit	th N-pr	lenyl	ben	zohydro	oxan	dc a	acid.
Te(IÝ)	: (	0.80 j	ppm			Solver	it :	Chl	loroform
FilHA	: 1	10 m1	(0.2%)	l		Color	:	Ye.	llow
	1	In ch	Lorofor	rm		λmax	1	34	5 nm
EI1.'1.0	1 1	7.0 H							
50.ve	its		9	6 Ext	rac	t.I.on			absorptivit
Chloroid			9	6 Ext	rac 100			. mo:	-
			9	6 Ext				. mo: 3	1 <sup>-1</sup> cm <sup>-1</sup>
Chloroid	) <b>r</b> m		9	6 Ext	100			. mo: 3	$1^{-1}$ cm <sup>-1</sup> .5 x 10 <sup>4</sup>
Chlorofo Toluthe	orm alo	cohol		6 Ext	100 54			. mo: 3	$1^{-1}$ cm <sup>-1</sup> .5 x 10 <sup>4</sup> .9 x 10 <sup>4</sup>

# 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).

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# Effect of Reagent Concentration

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

DETERMINATION OF TELLURIUM

	+	TABLE 3			
	Effec	t of PBHA Conc	entra	ation	
Te(IV)	: 0.80 ppm	Solv	ent:	Chloroform	a stress and
HCLO4	: 7.0 M	Colo	r ;	Yellow	ine F
		λmax	κ :	345 nm	
PBHA O. added m	•	Absorbance		lar absorptivi L mol <sup>-1</sup> cm <sup>-1</sup>	lty
2.5		0.12		1.9 x 10 <sup>4</sup>	
5.0		0.16		$2.5 \times 10^4$	8
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.

Validit of the Béer's Law

Be r's law is obeyed in the range of 0.10 to 5.0 ppm of tellurium. The Sandell's sensitivity is 0.00365  $/^{1}$  g/cm<sup>2</sup>. The Beer's law limit for the flameless atomic absorption spectrophotometric determination of tellurium is 0.01 - 0.08 ppm.

Eligat of Diverse Ions

If  $f: \partial H$  dominants of diverse ions were added to a fined 6 66 of  $\partial f$  tellurium and extracted according to the protector host 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 chemice properties and usually occur together. Hence, Separation of tellurium from selenium is very difficult.

Selenium forms reddiah 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 ED/A, the resulting complex was soluble in water

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N-phenylberzohydroxamic acid.         N-phenylberzohydroxamic acid.         Fiellurium : 0.80 ppm; F3HA: 10 ml (0728\$) in Chloroform; Solvent; Chloroform         HCD04; 7.0 k; color: Yellow; Absorbance: 0.22; Amex: 545 rm       Added as Amount added Absorbance i.e.s i.e. i.e. i.e. i.e. i.e. i.e. i.	N=phenylberzohydroxamic sold.Ellurium : 0.80 ppm; P3HA: 10 ml (072%) in Chloroform: Solvent: ChloroformEllurium : 0.80 ppm; P3HA: 10 ml (072%) in Chloroform: Solvent: ChloroformEllurium : 0.80 ppm; P3HA: 10 ml (072%) in Chloroform: Solvent: ChloroformEllurium : 0.80 ppm; P3HA: 10 ml (072%) in Chloroform: Solvent: ChloroformAdded as Amount added Absorbance Lons Added as AmountAdded as Amount added Absorbance Lons (40)Beso <sub>4</sub> Calio <sub>3</sub> )2Calio <sub>3</sub> )2Calio <sub>3</sub> )2AH20Calio <sub>3</sub> )2AH20Calio <sub>3</sub> )2AH20Calio <sub>4</sub> Coloc (213 <sub>5</sub> 0 <sub>2</sub> )2AH20Calio <sub>4</sub> Coloc (213 <sub>5</sub> 0 <sub>2</sub> )2AH20Calio <sub>4</sub> Coloc (213 <sub>5</sub> 0 <sub>2</sub> )2AH2Calio <sub>4</sub> AddedCalio <sub>4</sub> AddedAddedAddedBeso <sub>4</sub> Calio <sub>2</sub> Calio <sub>2</sub> AddedCalio <sub>2</sub> Calio <sub>2</sub> AddedAddedAddedBeso <sub>4</sub> AddedAddedBeso <sub>4</sub> AddedAdded		Effect of	Effect of Diverse Ions in the Determination of Tellurium with	n the Deteradn	letion of	Tellurium with	e	
III.04: 7.0 I; Color: Yellow; Absorbance: 0.22: Nuer: 345 mu         Added as       Amount added Absorbance I.ons       Added as       Amount added Absorbance I.ons       Added as         AsNo <sub>3</sub> (40)       0.22       Le <sup>3+</sup> La(10 <sub>5</sub> )) $= 61_{12}0$ (40)         AsNo <sub>3</sub> (40)       0.22       Le <sup>3+</sup> La(10 <sub>5</sub> )) $= 61_{12}0$ (40)         AsNo <sub>4</sub> (40)       0.22       Le <sup>3+</sup> La(10 <sub>5</sub> )) $= 61_{12}0$ (40)         Beso <sub>4</sub> (40)       0.22       N:1 <sup>2+</sup> N:1Cl2       (40)         Ca(:10 <sub>5</sub> )2       (40)       0.22       SnCl2       (40)         Ca(:10 <sub>5</sub> )2       (40)       0.22       SnCl2       (40)         Ca(so <sub>4</sub> (50)       0.22       Tr <sup>4+</sup> Tr(Cl <sub>4</sub> (50)         Cuso <sub>4</sub> (40)       0.22       Tr <sup>4+</sup> Tr(Cl <sub>4</sub> (50)         Cuso <sub>4</sub> (40)       0.22       Tr <sup>4+</sup> Tr(Cl <sub>4</sub> (50)         Cuso <sub>4</sub> (40)       0.22       Tr <sup>4+</sup> Tr(Cl <sub>4</sub> (40)         Cuso <sub>4</sub> (40)       0.22       Tr <sup>4+</sup> Tr(Cl <sub>4</sub> (40)         Cuso <sub>4</sub> (40)       0.22       Tr <sup>4+</sup> Tr(Cl <sub>4</sub> <t< td=""><td><math>IO_4</math>; 7.0 k; color: Yellow; Absorbance: 0.22; Amer: 343 FB       Added as Amount added Absorbance Ions (40)         ASNO<sub>3</sub>       (40)       0.21       Nd<sup>3</sup>+       Nd(NO<sub>3</sub>)<sub>3</sub>       (40)         BesO<sub>4</sub>       (50)       0.21       Nd<sup>3</sup>+       Nd(NO<sub>3</sub>)<sub>3</sub>       (40)         Callo<sub>2</sub>       (40)       0.21       Se<sup>4+</sup>       Se(CIO<sub>4</sub>)<sub>4</sub>       (40)         Calso<sub>4</sub>       (40)       0.22       Th<sup>4+</sup>       TiCI<sub>2</sub>       (40)         Calso<sub>4</sub>       (40)       0.22</td><td>-</td><td></td><td>N-phenylt</td><td>en zohydroxand</td><td>c acid.</td><td>tono [d] . throw [d</td><td></td><td></td></t<>	$IO_4$ ; 7.0 k; color: Yellow; Absorbance: 0.22; Amer: 343 FB       Added as Amount added Absorbance Ions (40)         ASNO <sub>3</sub> (40)       0.21       Nd <sup>3</sup> +       Nd(NO <sub>3</sub> ) <sub>3</sub> (40)         BesO <sub>4</sub> (50)       0.21       Nd <sup>3</sup> +       Nd(NO <sub>3</sub> ) <sub>3</sub> (40)         Callo <sub>2</sub> (40)       0.21       Se <sup>4+</sup> Se(CIO <sub>4</sub> ) <sub>4</sub> (40)         Calso <sub>4</sub> (40)       0.22       Th <sup>4+</sup> TiCI <sub>2</sub> (40)         Calso <sub>4</sub> (40)       0.22       Th <sup>4+</sup> TiCI <sub>2</sub> (40)         Calso <sub>4</sub> (40)       0.22       Th <sup>4+</sup> TiCI <sub>2</sub> (40)         Calso <sub>4</sub> (40)       0.22       Th <sup>4+</sup> TiCI <sub>2</sub> (40)         Calso <sub>4</sub> (40)       0.22       Th <sup>4+</sup> TiCI <sub>2</sub> (40)         Calso <sub>4</sub> (40)       0.22	-		N-phenylt	en zohydroxand	c acid.	tono [d] . throw [d		
Added as       Amount added       Absorbance       Lors       Added as       Amount added       Absorbance       Lat(103)       Gillo       (40)         ASNO3       (40)       0.22       Le <sup>3+</sup> La(103)       Gillo       (40)         AsNO3       (40)       0.21       Na <sup>3+</sup> Nd(NO <sub>3</sub> )       (40)       0.21       Nd <sup>3+</sup> Nd(NO <sub>3</sub> )       (40)         AsNO3       (40)       0.21       Nd <sup>3+</sup> Nd(NO <sub>3</sub> )       (40)       (40)         BesO4       (50)       0.22       N1 <sup>2</sup> (40)       (40)       (40)         Ca(::0 <sub>3</sub> )2       (H <sub>2</sub> )       0.22       Sn <sup>2+</sup> SnCl2       (40)       (40)         Ca(::0 <sub>3</sub> )2       (H <sub>2</sub> )       0.22       Sn <sup>2+</sup> SnCl2       (40)       (40)         Ca(SO4       (50)       0.22       T1 <sup>4+</sup> T1Cl4       (40)       (40)         Cuso4       (40)       0.22       T1 <sup>4+</sup> T1Cl2       (40)       (40)         GaI <sub>3</sub> (40)       0.22       T1 <sup>4+</sup> T1Cl4       (40)       (40)         GaI <sub>3</sub> (40)       0.22       T1 <sup>4+</sup> T1Cl4       (40)       (40)       (40)       (40)       (40) <td>Added as       Amount added       Absorbance:       C.22       Amer:       Added as       Amount added         AgNO<sub>3</sub>       (40)       0.22       Le<sup>3+</sup>       Le(10<sub>5</sub>)<sup>3</sup>       Eli20       (40)         AsF<sub>3</sub>       (40)       0.21       Ka<sup>3+</sup>       Nd(NO<sub>3</sub>)<sup>3</sup>       Eli20       (40)         BesO<sub>4</sub>       (50)       0.21       Nd<sup>3+</sup>       Nd(NO<sub>3</sub>)<sup>3</sup>       (40)       (40)         BesO<sub>4</sub>       (50)       0.21       Nd<sup>3+</sup>       Nd(NO<sub>3</sub>)<sup>3</sup>       (40)       (40)         BesO<sub>4</sub>       (50)       0.22       Sn<sup>2+</sup>       SnCl<sub>2</sub>       (40)       (40)         BesO<sub>4</sub>       (50)       0.22       Sn<sup>2+</sup>       SnCl<sub>2</sub>       (40)       (40)         BesO<sub>4</sub>       (40)       0.22       Sn<sup>2+</sup>       SnCl<sub>2</sub>       (40)       (40)         CalSO<sub>4</sub>       (40)       0.22       Tn<sup>4+</sup>       TnCl<sub>4</sub>       (50)       (40)         CalSO<sub>4</sub>       (40)       0.22       Tn<sup>4+</sup>       TnCl<sub>2</sub>       (40)       (40)         CalSO<sub>4</sub>       (40)       0.22       Tn<sup>4+</sup>       TnCl<sub>2</sub>       (40)       (40)         CalSO<sub>4</sub>       (40)       0.22       Tn<sup>4+</sup>       TnCl<sub>2</sub>       (40)</td> <td>••</td> <td>udd 08°0 : untintTai</td> <td>) TH OL : VHEA :</td> <td>iono ut feiz.</td> <td></td> <td>INJOTUN : LUTOLO</td> <td>, muo</td> <td>÷</td>	Added as       Amount added       Absorbance:       C.22       Amer:       Added as       Amount added         AgNO <sub>3</sub> (40)       0.22       Le <sup>3+</sup> Le(10 <sub>5</sub> ) <sup>3</sup> Eli20       (40)         AsF <sub>3</sub> (40)       0.21       Ka <sup>3+</sup> Nd(NO <sub>3</sub> ) <sup>3</sup> Eli20       (40)         BesO <sub>4</sub> (50)       0.21       Nd <sup>3+</sup> Nd(NO <sub>3</sub> ) <sup>3</sup> (40)       (40)         BesO <sub>4</sub> (50)       0.21       Nd <sup>3+</sup> Nd(NO <sub>3</sub> ) <sup>3</sup> (40)       (40)         BesO <sub>4</sub> (50)       0.22       Sn <sup>2+</sup> SnCl <sub>2</sub> (40)       (40)         BesO <sub>4</sub> (50)       0.22       Sn <sup>2+</sup> SnCl <sub>2</sub> (40)       (40)         BesO <sub>4</sub> (40)       0.22       Sn <sup>2+</sup> SnCl <sub>2</sub> (40)       (40)         CalSO <sub>4</sub> (40)       0.22       Tn <sup>4+</sup> TnCl <sub>4</sub> (50)       (40)         CalSO <sub>4</sub> (40)       0.22       Tn <sup>4+</sup> TnCl <sub>2</sub> (40)       (40)         CalSO <sub>4</sub> (40)       0.22       Tn <sup>4+</sup> TnCl <sub>2</sub> (40)       (40)         CalSO <sub>4</sub> (40)       0.22       Tn <sup>4+</sup> TnCl <sub>2</sub> (40)	••	udd 08°0 : untintTai	) TH OL : VHEA :	iono ut feiz.		INJOTUN : LUTOLO	, muo	÷
Added as         Amount added         Absorbance         Ions         Added as         Amount added         Amount added         Absorbance         Ions         Added as         Amount added         Absorbance         Ions         Added as         Amount added         Amount added         As         Added as         Amount added         Amount added <th>Added es         Amount added         Absorbance         Ions         Added as         Amount added         Asount added         Amount added         <tha< th=""><th>~</th><th>HClO4: 7.0 E; Color:</th><th>Yellow; Absorbs</th><th>nce: 0.22; &gt;</th><th>CFC 1120</th><th>Ē</th><th></th><th></th></tha<></th>	Added es         Amount added         Absorbance         Ions         Added as         Amount added         Asount added         Amount added <tha< th=""><th>~</th><th>HClO4: 7.0 E; Color:</th><th>Yellow; Absorbs</th><th>nce: 0.22; &gt;</th><th>CFC 1120</th><th>Ē</th><th></th><th></th></tha<>	~	HClO4: 7.0 E; Color:	Yellow; Absorbs	nce: 0.22; >	CFC 1120	Ē		
$A_{GNO_3}$ $(40)$ $0.22$ $Le^{3+}$ $La(HO_3) = GH_2 0$ $(40)$ $AsF_3$ $(4c)$ $0.22$ $Le^{3+}$ $La(HO_3) = GH_2 0$ $(40)$ $BeSO_4$ $(4c)$ $0.21$ $Nd^{3+}$ $Nd(NO_3) = GH_2 0$ $(40)$ $BeSO_4$ $(50)$ $0.22$ $N1^{2+}$ $N1CI_2$ $(40)$ $BeSO_4$ $(50)$ $0.22$ $N1^{2+}$ $N1CI_2$ $(40)$ $Ca(iO_3)_2$ $(4H_2)$ $(50)$ $0.22$ $Sn^{2+}$ $SnCI_2$ $(40)^{-1}$ $CalsO_4$ $(50)$ $0.22$ $Sn^{2+}$ $SnCI_2$ $(40)^{-1}$ $CulsO_4$ $(40)$ $0.22$ $T1^{4+}$ $TiCI_4$ $(50)$ $CulsO_4$ $(40)$ $0.22$ $Tn^{4+}$ $TiCI_4$ $(50)$ $CulsO_4$ $(40)$ $0.22$ $Tn^{4+}$ $TiCI_4$ $(40)^{-1}$ $CalsO_4$ $(40)$ $0.22$ $Tn^{4+}$ $TiCI_4$ $(40)^{-1}$ $CalsO_4$ $(40)$ $0.22$ $Tn^{4+}$ $TiCI_4$ $(40)^{-1}$ $(40)^{-1}$ <	AGNO3       AGNO3       (40)       0.22       Le <sup>3+</sup> La(HO <sub>3</sub> )       GH20       (40)         AsF3       (40)       0.21       Nd <sup>3+</sup> Nd(NO <sub>3</sub> )       (40)       (50)         BesO4       (40)       0.21       Nd <sup>3+</sup> Nd(NO <sub>3</sub> )       (40)         BesO4       (50)       0.22       Sn <sup>2+</sup> SnCl <sub>2</sub> (40)         Ca(HO <sub>3</sub> )2       (40)       0.22       Sn <sup>2+</sup> SnCl <sub>2</sub> (40)         Ca(HO <sub>3</sub> )2       (40)       0.22       Sn <sup>2+</sup> SnCl <sub>2</sub> (40)         Ca(HO <sub>2</sub> )2       (412)       0.22       Sn <sup>2+</sup> SnCl <sub>2</sub> (40)         Ca(SO <sub>4</sub> (50)       0.22       Th <sup>4+</sup> Th(H <sub>2</sub> O <sub>3</sub> ) <sub>4</sub> (40)         Co(C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> )2       (412)       0.22       Th <sup>4+</sup> Th(H <sub>2</sub> O <sub>3</sub> ) <sub>4</sub> (40)         CasO <sub>4</sub> (40)       0.22       Th <sup>4+</sup> Th(H <sub>2</sub> O <sub>3</sub> ) <sub>4</sub> (40)         CasI <sub>3</sub> 0.22       Th <sup>4+</sup> Th(H <sub>2</sub> O <sub>3</sub> ) <sub>4</sub> (40)         CasI <sub>3</sub> 0.22       Th <sup>4+</sup> Th(H <sub>2</sub> O <sub>3</sub> ) <sub>4</sub> (40)         CasI <sub>3</sub> 0.22       Th <sup>4+</sup> Th(H <sub>2</sub> O <sub>3</sub> ) <sub>4</sub> (40)         CasI <sub>3</sub> 0.22	Ions	Added es	Amount added mg	Absorbance	Ions	Added as	Amount added mg	Absor- bance
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	AsF <sub>3</sub> (4c) $2 = \frac{2}{10}$ $4 = \frac{2}{10}$ $4 = \frac{2}{10}$ $4 = \frac{2}{10}$ $4 = \frac{2}{10}$ $5 = \frac{2}{10}$	+ - 	AGNO3		0.22	, +Call	La(110 <sub>3</sub> ) <del>5 G</del> 120		0.21
$ \begin{array}{llllllllllllllllllllllllllllllllllll$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	A53+		I		}}		(20)	0.22
$\begin{array}{llllllllllllllllllllllllllllllllllll$	$ \begin{array}{llllllllllllllllllllllllllllllllllll$	3e <sup>2+</sup>	Bach	(07)	0.21	Nd <sup>3+</sup>	E(CON)PN	(07)	0.22
$ \begin{array}{llllllllllllllllllllllllllllllllllll$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	ве <sup>2+</sup>	Beso4	(20)	0.22	к1 <sup>2+</sup>	NICL	(07) 	0.22
$cdso_4$ (50)       0.21 $se^{4+}$ $se(clo_4)_4$ (40) <sup>B</sup> $co(c_2!I_3o_2)_2$ $4H_2o$ (40)       0.22 $T1^{4+}$ $TiCl_4$ (50) $cuso_4$ (40)       0.22 $T1^{4+}$ $TiCl_4$ (40) $cuso_4$ (40)       0.22 $Tn^{4+}$ $Ti(io_3)_4$ (40) $cal_3$ (40)       0.22 $V^{5+}$ $iH_4Vo_3$ (40) <sup>b</sup> $Indl_3$ (40)       0.22 $V^{5+}$ $iH_4Vo_3$ (40) <sup>b</sup> $Indl_3$ (40)       0.22 $V^{5+}$ $2n^2^2$ (40) <sup>b</sup>	CdSO4(50)0.21 $se^{4+}$ $se(ClO_4)_4$ $(40)^8$ CdSO4(40)0.22 $T1^{4+}$ $TiCl_4$ (50)CdSO4(40)0.22 $T1^{4+}$ $TiCl_4$ (50)CdSO4(40)0.22 $Tn^{4+}$ $TiCl_3)_4$ (40)CdS13(40)0.22 $V^{5+}$ $iH_4VO_3$ (40)CdS13(40)0.22 $2n^{2+}$ $2nCl_2$ (40)CdS13(40)0.22 $2n^{2+}$ $2nCl_2$ (40)LnU13(40)0.22 $2n^{2+}$ $2nCl_2$ (40) $s = mesked with mixture of citric acid + Na-tartrate+ HMA,.$	Ca <sup>2+</sup>		( ( ( ( ( )	0,22	sn <sup>2+</sup>	SnC1 <sub>2</sub>	(07)-	
$ \begin{array}{llllllllllllllllllllllllllllllllllll$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	cd <sup>2+</sup>		(20)	0.21	Se <sup>4+</sup>	Se(Cl04)4	(07) <sup>a</sup>	0.22
$\frac{1}{\text{Cuso}_4} \qquad (40) \qquad \cdot \ 0.22 \qquad \text{Th}^{4+} \ \text{Th}(\text{RO}_3)_4 \qquad (40) \\ \frac{1}{\text{Cal}_3} \qquad (40) \qquad 0.22 \qquad \text{V}^{5+} \ \text{RH}_4 \text{VO}_3 \qquad (40)^{\text{b}} \\ \text{InCl}_3 \qquad (40) \qquad 0.22 \qquad \text{Zn}^{2+} \ \text{Zn}_2 \qquad (40)^{\text{b}} \\ \frac{1}{\text{Cal}_3} \qquad (40) \qquad 0.22 \qquad \text{Cal}^{2+} \ \text{Zn}_2 \qquad (40)^{\text{b}} \\ \text{Cal}_3 \qquad (40) \qquad 0.22 \qquad \text{Cal}^{2+} \ \text{Zn}_2 \qquad (40)^{\text{b}} \\ \frac{1}{\text{Cal}_3} \qquad (40) \qquad 0.22 \qquad \text{Cal}^{2+} \ \text{Zn}_2 \qquad \text{Cal}^{2+} \qquad (40)^{\text{c}} \\ \frac{1}{\text{Cal}_3} \qquad (40)^{\text{c}} \\ \frac{1}$	$\begin{aligned} \cos 0_4 & (40) & 0.22 & Th^{4+} Th(KO_3)_4 & (40) \\ GaI_3 & (40) & 0.22 & V^{5+} & iH_4VO_3 & (40)^b \\ InOl_3 & (40) & 0.22 & Zn^{2+} & ZnCl_2 & (40) \\ a = mesked with mixture of citric acid + Na-tartrate + EDVA, \end{aligned}$	Co <sup>2+</sup>		(07)	0.22	T1 <sup>4+</sup>	rici4	(05)	0.22
$\frac{\sigma_{\text{BL}_{3}}}{1000} = \frac{1}{2} \left( \frac{1}{100} \right) = \frac{1}{0.22} \left( \frac{1}{2} \right) \left( \frac{1}{100} \right) \left( \frac{1}{100} \right) = \frac{1}{0} \left( \frac{1}{100} \right) \left( \frac{1}{100} \right) = \frac{1}{100} \left( 1$	GaI <sub>3</sub> (40) 0.22 $V^{5+}$ $iH_4VO_3$ (40) <sup>b</sup> InUl <sub>3</sub> (40) 0.22 $2n^{2+}$ $2nCl_2$ (40) a = m sked with mixture of citric acid + Na-tartrate + EDYA,	cu <sup>2+</sup>		(07)	. 0.22	Th <sup>4+</sup>	Th(1:03)4	(07)	c.21
Incl <sub>3</sub> (40) $0.22$ $2n^{2+}$ $2ncl_2$ (40)	Incl <sub>3</sub> (40) $0.22$ $2n^2 + 2ncl_2$ (40) a = mesked with mixture of citric acid + Na-tartrate + EUYA, 2	00 <b>3</b> +		(07)	0,22	۰+2 <sup>N</sup>	ith4v03	q(07)	0.22
	ric acid + Na-tartrate + EDVA,	1 <sup>2+</sup>		(07)	0,22	+2 <sup>-15</sup>	ma <sub>2</sub>	(07)	0.22

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# DETERMINATION OF TELLURIUM

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TABLE 4

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TABLE	5
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Hetermination of Tellurium in presence of Selenium

Tell/Flum taken (ppm)		elenium ied (mg)	Tellurium (ppm)		Coefficient variation
			Present method	BAA	%
1.0		50	1.00	0.99	1.00
3)	1	50	3″.01	3.00	0.66
5.)	'	6Ò	5.01	3,00	0.20
7.)		60	6.99	7.00	0.28
9.0		70	9.00	9.00	0.22
11.)		70	10.99	10.99	0.27

\* Seven doterminations

\*\* The regression equation for the two methods is  $1 = 0.990 \text{ A} + 1^{-1.000}$ (here T = Telluming by 1 Ctrophotometry, A = Tellurium  $1 \neq AAS$  and r = Correlation coefficient.

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

Stoi hiometry 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 emount 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-

DETERMINATION OF TELLURIUM

# TABLE 6

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Determination of Tellurium in NBS Cartridge Brasses

Sample	Certified Te %	<b>Te</b> 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)<sup>15</sup> was found to be 2.2 confirms that the tel ium to PBHA ratio is  $1:\frac{1}{2}$ .

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

 $Te(ClO_4)_4 + 2 HA \xrightarrow{TR} Te(A)_2(ClO_4)_2 + 2HClO_4$ where HA = PBHA.

Determinetion of Tellurium in NBS Cartridge brasses

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

#### ACKNOWLEDGENENT

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