

Rapid determination of tellurium(IV) by ultraviolet spectrophotometry using *o*-methylphenyl thiourea as a new chromogenic ligand

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O-Methylphenyl thiourea (OMPT) coordinates with tellurium(IV) as a 1:1 (tellurium(IV)-OMPT) complex in hydrochloric acid medium (7.0 mol L⁻¹). The novelty of the proposed method is the instant complex formation at room temperature with no need of heating or standing. Method is applicable over a wide Beer's range (up to 70 µg ml⁻¹). A low reagent concentration is required (2 ml, 0.018 mol L⁻¹ in methanol). The complex exhibits maximum absorption at a wavelength of 280 nm. The molar absorptivity is 1.98×10⁴ L mol⁻¹ cm⁻¹, Sandell's sensitivity is 0.00641 µg of tellurium(IV) cm⁻². The proposed method was successfully applied for analysis of a real sample.

Keywords: Tellurium(IV); UV-spectrophotometry; Analysis, Real sample.

INTRODUCTION

Abundance of tellurium in the earth's crust is 0.001 ppm. Its compounds are used in metallurgy, mostly in making steel and non-ferrous alloys [1]. It is used as a semiconductor material. Tellurium and its compounds are widely used in thin films, rechargeable batteries and charge transfer systems. Compounds like hydrogen telluride are highly toxic in nature. Tellurium exposure results into garlic-like breath. Tellurium aerosol irritates the eyes and the respiratory track. Tellurium compounds may affect liver and central nervous system. It causes abdominal pain, constipation and vomiting. It is a potential toxic environmental pollutant [2]. Addition of tellurium to lead prevents corrosion [3]. Cadmium telluride photovoltaic modules have become the lowest-cost producer of solar electricity [4]. Trace abundance, application in metallurgy, solar and semiconductors, environmental toxicity and health hazards support the necessity and demand for the development of a simple, sensitive method for determination of tellurium and monitoring trace tellurium concentrations in various sample matrices.

Many analytical techniques have been studied and methods for determination of tellurium have been reported such as voltammetry [5], stripping voltammetry [6,7], inductively coupled plasma atomic emission spectroscopy (ICP-AES) [8,9], inductively coupled plasma mass spectrometry

(ICP-MS) [10-12], atomic absorption spectrometry (AAS) [13,14] and hydride generation atomic fluorescence spectrometry [15]. These methods, based on different instrumental techniques, have positive merits like determination at trace level, low limit of detection, minimum interferences, analysis of various sample matrices and fast determination. However, practical application of these techniques has serious drawbacks and it requires sophisticated instrumentation. Spectrophotometric molecular absorption methods involve less expensive instrumentation, and are simple to operate with high sensitivity.

Recently, very few reagents and a limited number of methods are reported for spectrophotometric determination of tellurium. According to the review of literature for spectrophotometric determination of tellurium, the methods are based on catalytic kinetic determination [16-19], synergic extraction [20], direct spectrophotometric determination [21-25], solvent extraction spectrophotometric determination [26-31], determination after extraction using molten naphthalene [32-34] and determination by ion-association complex formation [35]. These methods are sensitive, but have the drawback that catalytic kinetic methods need controlled conditions. Most direct determination methods suffer from interferences from associated metal ions and extraction spectrophotometric determination methods require costly and environmentally hazardous organic solvents.

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In our laboratory, work has been carried on for extraction spectrophotometric determination of platinum group metals using OMPT [36-41], ruthenium & osmium [42], selenium [43], palladium [44] and cerium(IV) [45] using OMePT. In extension of this work, solvent-free direct spectrophotometric determination of tellurium with the sensitive chromogenic chelating ligand *o*-methylphenyl thiourea (OMPT) was developed in this work.

EXPERIMENTAL

Apparatus

Elico digital spectrophotometer model SL-159 with 1 cm quartz cells and Contech electronic balance model CA -123 were used for absorption measurements and weighing. Glassware was cleaned by soaking in acidified solution of potassium dichromate followed by washing with soap water and rinsing twice with distilled water.

Standard tellurium(IV) solution

A stock solution of tellurium was prepared by dissolving 0.250 g solid tellurium metal in a nitrating mixture, HCl:HNO₃ (1:3) and diluting up to the mark in a 250 mL standard volumetric flask. A working standard solution (25 µg mL⁻¹) was prepared by diluting an aliquot of the stock solution with distilled water.

o-Methylphenyl thiourea solution

O-Methylphenyl thiourea (OMPT) was synthesized as reported by Frank and Smith [42]. ¹H NMR spectrum of *o*-methylphenyl thiourea is given in Fig 1. A 0.01 mol L⁻¹ methanolic solution was prepared by dissolving 0.149 g of OMPT in 20 mL of methanol and diluted up to the mark with methanol in a 50 mL calibrated volumetric flask.

Solutions of foreign ions

Standard solutions of different metal ions used to study the effect of foreign ions were prepared by dissolving weighed quantities of their salts in water or dilute hydrochloric acid in a calibrated volumetric flask. Solutions of anions were prepared after dissolving their respective alkali metal salts in water in a calibrated volumetric flask. Distilled water was used throughout the study.

Recommended procedure

An aliquot of a solution containing 25 µg of tellurium(IV), hydrochloric acid (6.2 mL) and 2 mL of 0.018 mol L⁻¹ OMPT in methanol was transferred to a 10 mL volumetric flask and diluted up to the mark with water. The absorbance of the

tellurium-OMPT complex was measured in the ultraviolet region at 280 nm against reagent blank prepared in a similar manner.

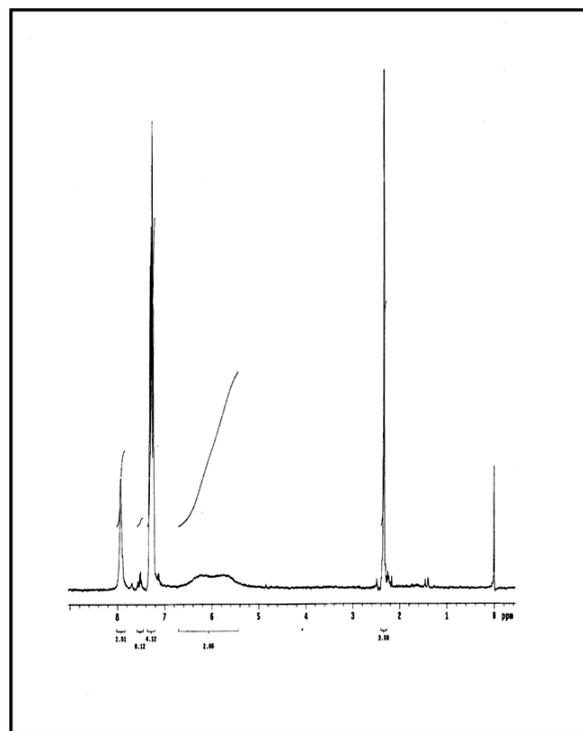


Fig. 1. ¹H NMR spectrum of *o*-methylphenyl thiourea (OMPT).

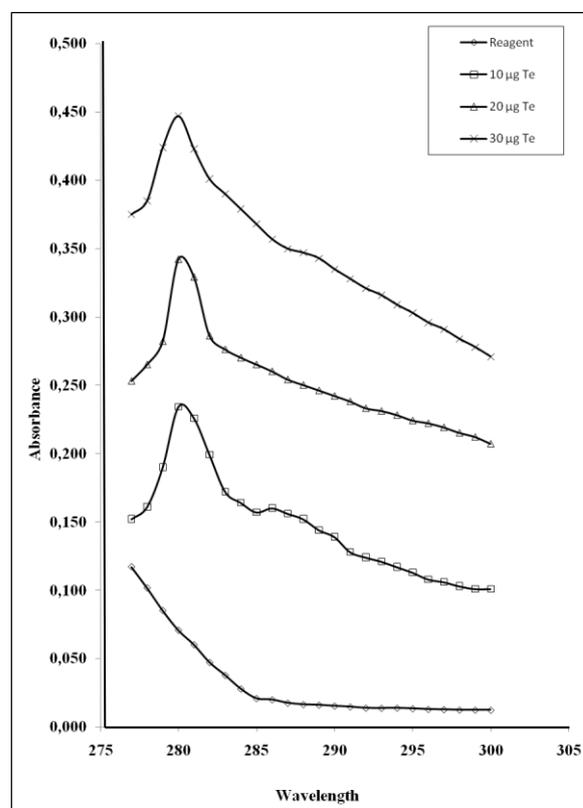


Fig. 2. Absorption spectrum of tellurium(IV)-OMPT-chloride complex vs OMPT reagent blank.

RESULTS AND DISCUSSION

Spectral characteristics

Tellurium(IV) formed 1:1 (tellurium:OMPT) complex in 7.0 mol L⁻¹ hydrochloric acid medium. The complex showed maximum absorption at 280 nm. The optimum conditions for determination of tellurium were established by studying the hydrochloric acid concentration, OMPT concentration and interferences by various foreign ions. The proposed method, when compared with other extraction spectrophotometric methods, (Table 1) offers advantages such as reliability, easy reproducibility, and simple operation for determination of tellurium (IV). The spectral and physico-chemical characteristics along with the precision data are reported in Table 2.

Absorption spectra

The absorption spectrum of the tellurium-OMPT complex showed maximum absorbance in the ultraviolet region at 280 nm. Thus, all further spectral measurements of the complex were made at a wavelength of 280 nm (Fig.2).

Effect of hydrochloric acid concentration

Tellurium(IV)-OMPT complex formation was studied in hydrochloric acid, nitric acid, sulphuric acid and perchloric acid. Amongst the acids studied, the tellurium(IV)-OMPT complex formation took place in presence of hydrochloric acid but not in any other acid studied. Maximum absorbance was registered in 7.0 mol L⁻¹ hydrochloric acid. Hence, all further measurements were performed in 7.0 mol L⁻¹ hydrochloric acid (Fig. 3).

Effect of reagent and extraction solvent

The reagents studied were methanol, dimethylsulphoxide (DMSO), dimethylformamide (DMF) and 1,4-dioxan. There was no complex formation in the presence of DMSO. Complete complexation with maximum absorbance was achieved in methanol. Hence, methanol was chosen for further study. Various extraction solvents were studied, viz., chloroform, benzene, toluene, xylene, isoamyl alcohol and butanol, but none of them was effective for the extraction of the Te-OMPT complex.

Effect of OMPT concentration

The concentration of OMPT in methanol (2.0 mL) was varied in the range of 0.005 - 0.02 mol L⁻¹. It was observed that 2 mL of 0.018 mol L⁻¹ reagent

was sufficient to ensure complete complexation, (Fig. 4). The excess reagent has no adverse effect on the determination of tellurium(IV).

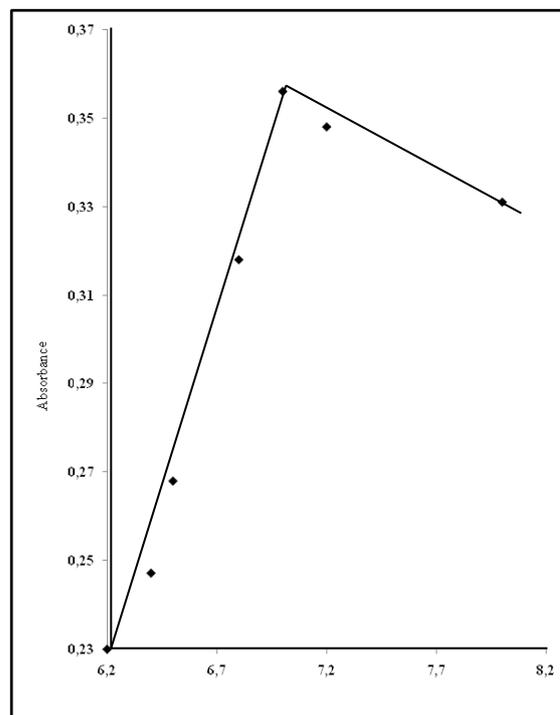


Fig. 3. Effect of hydrochloric acid concentration. Te(IV) 25 µg; OMPT 2.0 ml, 0.01 mol L⁻¹; λ_{max} 280 nm

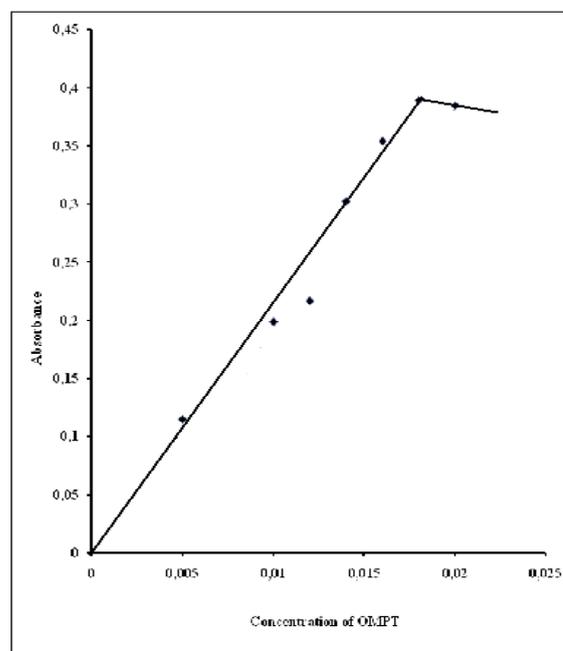


Fig. 4. Effect of *o*-methylphenyl thiourea (OMPT) concentration. Te(IV) 25 µg; HCl 7.0 mol L⁻¹; λ_{max} 280 nm.

Table 1. Comparison of the present method with spectrophotometric determination methods of tellurium.

Reagents	λ_{\max} (nm)	Condition	Beer's Law validity range, ($\mu\text{g mL}^{-1}$)	Solvent	Molar Absorptivity, ($\text{L mol}^{-1}\text{cm}^{-1}$)	Remark	Ref
Gallocyaine	618	pH/5.0 acetate buffer	2.0–200 $\mu\text{g mL}^{-1}$	water	NR	Interfering ions need removal after passing through cation exchange resin	16
Cetyl trimethyl ammoniumbromide	600	pH/4.0 acetate buffer	0.6–500 $\mu\text{g mL}^{-1}$	water	NR	30 min standing at 35 °C before initiation reaction	17
Leuco methylene Green	650	pH/3.0 acetate buffer	0.2–2.5 $\mu\text{g mL}^{-1}$	water	4.9×10^4	Interference study of Se, Bi, Po not studied	18
Toluidine blue	630	pH/7.2 phosphate buffer	0.01–0.08 $\mu\text{g mL}^{-1}$	water	NR	Controlled 25 °C essential for completion of reaction. Lengthy analysis time 100 sec.	19
p-[4-(3,5-dimethylisoxazolyl)azophenylazo]calix[4]arene	425	3.0 M HNO ₃	1.0 to 14.0 $\mu\text{g mL}^{-1}$	1,2 dichloroethane	1.67×10^4	1.0 hour centrifugation and 10 min standing to separate phases	20
Nile blue	580	6.0 M H ₂ SO ₄	0.004–0.006 $\mu\text{g mL}^{-1}$	water	3.33×10^5	10 min standing before adding reagent and 5 min standing after adding reagent	21
4-bromophenylhydrazine	550	4.0 M NaOH	1.0–2.5 $\mu\text{g mL}^{-1}$	water	1.0×10^5	5.0 min standing, test volume restricted to 1.0 ml	22
Chrome azurol S	525	pH/3.1	Up to 2.0 $\mu\text{g mL}^{-1}$	water	2.5×10^4	Method is sensitive to order of addition of reagents, 5 min standing, many cations interfere	23
N,N-di(acetoxyethyl)indocarbocyanine	542	4.0–5.5 M H ₂ SO ₄	0.04–15.0 $\mu\text{g mL}^{-1}$	Toluene	4.3 to 11.2 $\times 10^4$	Hg(II) interferes, No applications studied	26
Hexabromide-diantipyrylmethane	336	2.0 M H ₂ SO ₄	NR	chloroform	1.82×10^3	Standing 15 min to ensure complexation, lengthy procedure with more number of chemicals required	27
1-(2',3'-dichlorophenyl)-4- β -trimethyl-(1H,4H)pyrimidine-2-thiol	430	HCl	2.5–12.5 $\mu\text{g mL}^{-1}$	chloroform	7.56×10^3	No real samples analyzed	28
1-(4-Bromophenyl)-4- β -trimethyl-1,4-dihydropyrimidine-2-thiol	440	HCl	1.0–15.0 $\mu\text{g mL}^{-1}$	chloroform	8.1×10^3	No real samples analyzed	29
Morpholine-4-carbodiimide	415	pH/3.5–7.0	0.5–12.5 $\mu\text{g mL}^{-1}$	chloroform	1.07×10^4	Heating at 60 °C, complex stable on for 3.4 h on chloroform	32
o-Methylphenylthiourea (OMPT)	280	7.0 M HCl	0.5–12.5 $\mu\text{g mL}^{-1}$	water	1.99×10^4	Minimum reagent required. No need of organic solvent. Higher molar absorptivity. Wide beer's range	PM

NR : Not reported, PM : Present method.

Table 2. Spectral and physico-chemical characteristics along with precision data of the tellurium-OMPT complex.

Spectral characteristics and precision	Parameters
Hydrochloric acid concentration	7.0 mol l ⁻¹
Reagent solvent	methanol
Reagent concentration	2 ml, 0.018 mol l ⁻¹
λ_{\max}	280 nm
Molar absorptivity	$1.99 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$
Sandell's sensitivity	0.00641 $\mu\text{g cm}^{-2}$
Beer's law range	up to 70.0 $\mu\text{g mL}^{-1}$
Ringbom's optimum range	13.0 to 70.0 $\mu\text{g mL}^{-1}$
Limit of detection	0.77 $\mu\text{g mL}^{-1}$
Relative standard deviation	0.004%
Stoichiometry of the complex	1:1 (Te:OMPT)
Stability of complex	1.0 h
Correlation coefficient	0.99

Stability of the complex

The stability of the complex was studied by measuring the absorbance at intervals of 10 min each. Absorbance of the complex was stable for a period of 1.0 h.

Beer's law and sensitivity

Beer's law was obeyed over the concentration range up to 70 $\mu\text{g mL}^{-1}$ (Fig. 5). Ringbom's plot was of sigmoid shape with a linear segment at intermediate absorbance values of 13.0 to 70.0 $\mu\text{g mL}^{-1}$ and with a slope value of 0.576 (Fig. 6). The ratio between the relative error in the concentration and the photometric error was found to be 3.99. The sensitivity of the method as defined by Sandell was 0.00641 $\mu\text{g cm}^{-2}$ and the molar absorptivity was $1.98 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$. The correlation coefficient value of the tellurium-OMPT complex with concentration in $\mu\text{g mL}^{-1}$ as independent variable and absorbance as dependent variable was found to be 0.99. The standard deviation calculated from 10 determinations of a solution containing 25 μg tellurium was 0.004.

Stoichiometry of the complex

The composition of the tellurium(IV):OMPT complex was ascertained using the slope ratio method by plotting the graph of $\log D_{(\text{Te})}$ against

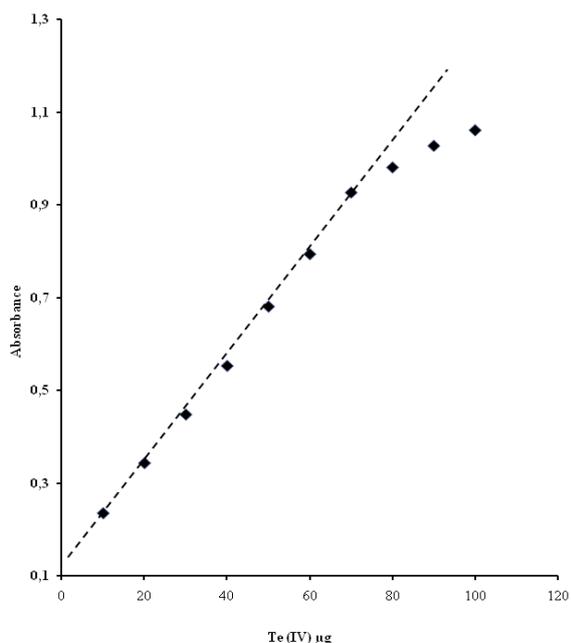


Fig. 5. Beer's law. Te(IV) 25 to 100 μg ; HCl 7.0 mol L^{-1} ; OMPT 2.0 ml, 0.01 mol L^{-1} ; λ_{max} 280 nm

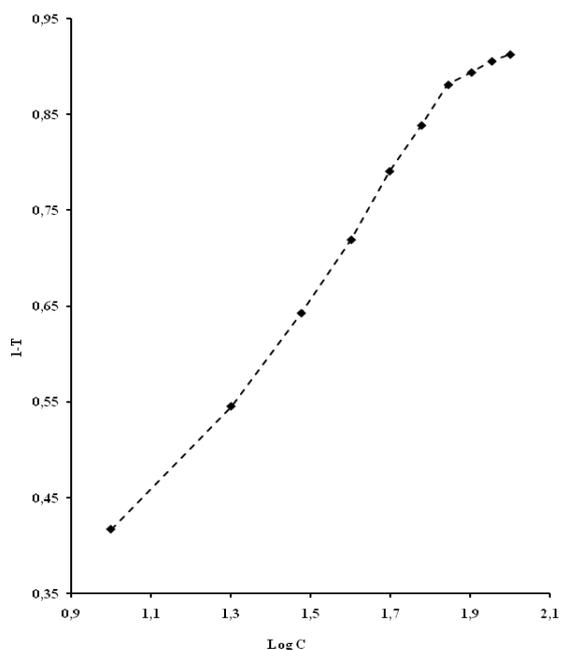


Fig. 6. Ringbom's plot. Te(IV) 25 to 100 μg ; HCl 7.0 mol L^{-1} ; OMPT 2.0 ml, 0.01 mol L^{-1} ; λ_{max} 280 nm.

$\log C_{(\text{OMPT})}$ at 1.0 mol L^{-1} and 3.0 mol L^{-1} hydrochloric acid concentration. These graphs were linear with slope values of 0.89 and 0.98, respectively (Fig. 7). Hence, the probable composition of the extracted species was calculated to be 1:1 (tellurium(IV):OMPT). The composition of the complex was also confirmed by the mole ratio method (Fig. 8) which supported the stoichiometry as 1:1 (tellurium:OMPT). OMPT acts

as a multidentate ligand, sulphur from the thio group ($-\text{C} = \text{S}$) and nitrogen from the amine group ($-\text{NH}_2$) coordinating with tellurium to form a 1:1 (tellurium:OMPT) complex. Based on this investigation the probable structure recommended for the complex is given in Fig. 9.

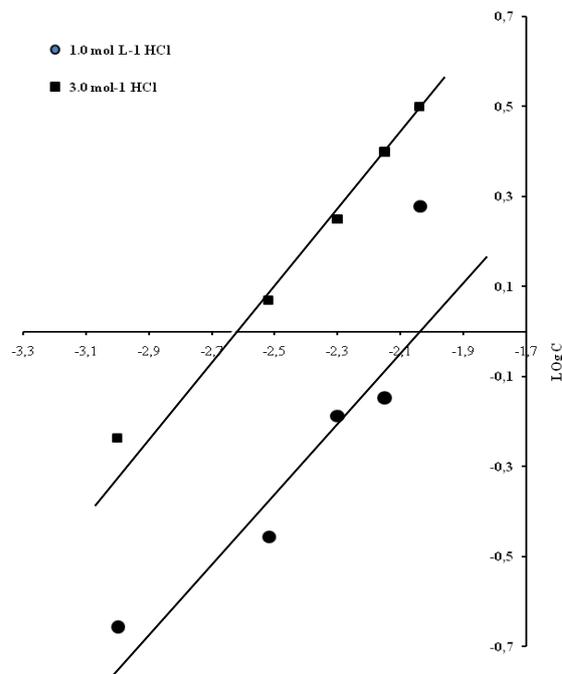


Fig. 7. Stoichiometry by the slope ratio method - $\log D_{(\text{Te})}$ against $\log C_{(\text{OMPT})}$

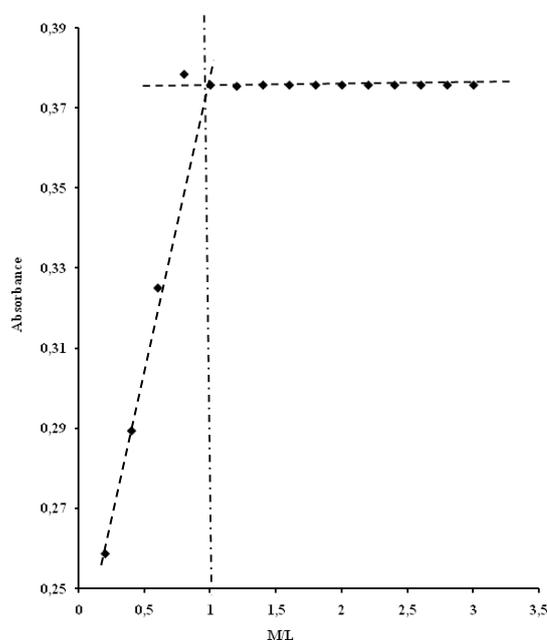
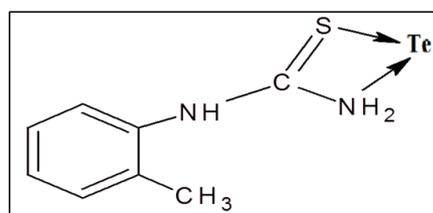


Fig. 8. Mole ratio method.

Te(IV) 25 μg ; HCl 1.0 mol L^{-1} , 3.0 mol L^{-1} and 7.0 mol L^{-1} ; OMPT 2.0 ml (0.001 mol L^{-1} to 0.01 mol L^{-1}); λ_{max} 280 nm.

Table 3. Effect of foreign ions. Te (IV) 25 µg ; HCl 7.0 mol L⁻¹ ; OMPT 2.0 ml, 0.01 mol L⁻¹; λ_{max} 280 nm.

Foreign ion	Added as	Tolerance limit (mg)	Foreign ion	Added as	Tolerance limit (mg)
Mn(II)	MnCl ₂ .6H ₂ O	0.25	Ca(II)	CaCl ₂ .2H ₂ O	0.50
Ce(IV)	Ce(SO ₄) ₂ .4H ₂ O	1.00	Tl(III)	Tl ₂ O ₃	0.02
Co(II)	CoCl ₂ .6H ₂ O	1.00	In(III)	InCl ₃ .4H ₂ O	0.50
Bi(III)	BiCl ₃	0.25	Os(VIII)	OsO ₄	0.13
Ni(II)	NiCl ₂ .6H ₂ O	1.00	Ba(II)	BaCl ₂ .6H ₂ O	10.0
Se(II)	SeO ₂	0.10	Ir(III)	IrCl ₃	0.25
Al(III)	AlCl ₃ .6H ₂ O	0.80	Os(IV)	OsO ₄	0.13
La(III)	LaCl ₃ .7H ₂ O	3.00	Zr(IV)	ZrOCl ₂ .8H ₂ O	0.10
Li(I)	LiCl	0.10	As (III)	As ₂ O ₃	0.50
Ti(III)	(Ti ₂ SO ₄) ₃	0.25	W(VI)	Na ₂ WO ₄ .2H ₂ O	0.10
Mg(II)	MgCl ₂ .6H ₂ O	1.00	Zn(II)	ZnSO ₄ .7H ₂ O	50.0
Sn(II)	SnCl ₂ .2H ₂ O	0.10	Be(II)	BeSO ₄ .2H ₂ O	0.10
Ga(III)	GaCl ₃	1.00	Sr(III)	Sr(NO ₃) ₂	2.50
Au(III)	HAuClO ₄ .H ₂ O	1.10	Sulphate	K ₂ SO ₄	0.50
Mo(VI)	(NH ₄) ₆ MO ₇ O ₂₄ .2H ₂ O	0.25	Succinate	(CH ₃ COONa) ₂ .6H ₂ O	0.25
Sb(III)	Sb ₂ O ₃	1.00	Citrate	C ₆ H ₈ O ₇ .H ₂ O	0.50
V(V)	V ₂ O ₅	0.25	Malonate	CH ₂ (COONa) ₂	1.00
Ce(IV)	Ce(SO ₄) ₂ .4H ₂ O	0.25	Acetate	CH ₃ COONa.3H ₂ O	10.0
U(VI)	UO ₂ (CH ₃ COO) ₂ .2H ₂ O	0.25	E.D.T.A	Na ₂ EDTA	0.75

**Fig. 9.** Probable structure of the tellurium-OMPT (1:1) complex.

Effect of foreign ions

Various foreign ions were tested to determine their tolerance limits in the determination of tellurium(IV) (Table 3). The tolerance limit was defined as the amount of the ion, which does not cause deviations more than $\pm 2\%$ in the absorbance of the tellurium-OMPT complex. A large number of foreign ions do not interfere in the method except chromium(VI), lead(II), rhodium(III), copper(II), cadmium(II) and mercury(II).

APPLICATIONS

Analysis of a solar glass plate

The method developed was applied for determination of the tellurium content of solar glass plates present in solar calculators. Known weight of a solar plate containing a thin layer of tellurium below the transparent glass was treated with *aqua regia* to dissolve the tellurium. It was washed with water and the washings were collected in the same container. The clean glass plate was dried and weighed to get the weight of the tellurium coating. The solution containing tellurium, *aqua regia* and

washings was evaporated to moist dryness and cooled. The residue was dissolved in water and made up to the mark in a 10 ml volumetric flask. 1.0 ml of this mixture was used for determination of tellurium by the recommended procedure. The results obtained are in good agreement with those obtained using a standard method (Table 4).

Table 4. Determination of tellurium from a solar glass plate. HCl 7.0 mol L⁻¹, OMPT 2.0 ml, 0.01 mol L⁻¹; λ_{max} 280 nm.

Weight of tellurium coating	Tellurium present*	Tellurium content by the recommended method	% Tellurium*
0.0035 g	0.1155	0.1153	99.82

*Six determinations

CONCLUSIONS

O-methylphenyl thiourea (OMPT) is a highly sensitive reagent for determination of tellurium when compared with other spectrophotometric determination methods (Table 1). It was successfully applied to the determination of tellurium in a real sample.

Salient features of the proposed novel method are:

1. Low reagent concentration required for spectrophotometric determination of tellurium.
2. Method is free from interferences from a large number of foreign ions associated with tellurium in its natural occurrence.
3. The method is applicable for analysis of real samples.

4. The proposed method is simple, rapid and reproducible with quantitative recovery of tellurium at a trace level.

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БЪРЗО ОПРЕДЕЛЯНЕ НА ТЕЛУР(IV) ЧРЕЗ УЛТРАВИОЛЕТОВА СПЕКТРОСКОПИЯ С ПОМОЩТА НА *o*-МЕТИЛФЕНИЛТИОКАРБАМИД КАТО НОВ ХРОМОГЕНЕН ЛИГАНД

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(Резюме)

o-Метилфенилтиокарбамидът (ОМРТ) образува с телур (IV) комплекс в моларно съотношение 1:1 (tellurium(IV)-ОМРТ) в солно-кисела среда (7.0 mol L^{-1}). Новостта на предложения метод е незабавното образуване на комплекса при стайна температура без нужда от нагряване или престой. Методът е приложим за широк обхват на закона на Beer (до $70 \mu\text{g ml}^{-1}$). Изискват се ниски концентрации на реагента ($2 \text{ ml}, 0.018 \text{ mol L}^{-1}$ в метанол). Комплексът има максимална абсорбция при дължина на вълната 280 nm . Моларната абсорбция е $1.98 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$, а чувствителността по Sandell е $0.00641 \mu\text{g}$ за телур (IV) cm^{-2} . Предложеният метод бе успешно приложен за анализа на реални проби.