Spectrophotometric Determination of Tellurium (IV) with Chrome Azurol S: Application to Sea Water and Synthetic Alloys

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ABSTRACT

The detailed investigation of a sensitive spectrophotometric method for the determination of trace amounts of tellurium (IV) is described. The method is based on the reaction of tellurium (IV) with chrome azurol S in the presence of cetylpyridinium chloride hydrate surfactant at a final pH of 3.1 to form a pink coloured chelate absorbing maximum at 525 nm. Beer's law is obeyed over the range (5-50) μ g/25 ml, with a molar absorptivity of (2.5 ×10⁴) 1.mol⁻¹.cm⁻¹ and a coefficient of determination (r²) of 0.9997. Sandell's sensitivity index is (5.104×10⁻³) μ g.cm⁻², a relative error of (0.8-0.2%) and a relative standard deviation of (0.2 to ±1.8) %, depending on the concentration levels in the calibration curve. The method has been applied successfully to determine Te (IV) in sea water and some synthetic alloys.

Keywords: Te (IV) determination, Chrome azurol S reagent, Cetyl pyridinium chloride hydrate, Spectrophotometry.

-Chrome Azurol S

3.1	Cetylpyridinium ch	Chrome Azurol S	
	525		
	25	(50-5)	
	0.9997	1- 1-	$(10^4 \times 2.5)$
	%(0.8-0.2)	2-	(10 ⁻³ ×5.104)
			%(.18± 0.2±)

INTRODUCTION

Tellurium came from the Latin word tellus that means earth, it can be found in dioxide form, the properties of tellurium included a silvery-gray colour, boiling point of 1810° F, melting point of 841.12° F. In air, tellurium burns in a greenish-blue flame forming the dioxide, TeO₂ is highly insoluble in water and soluble in concentrated sulfuric acid. It reacts with acids to make tellurium salts and bases to make tellurites (Mediawlki, 2012).

Tellurium's conductivity can also increase if exposed to light. Tellurium can be used in ceramics. It can be added to lead to prevent corrosion and strength (Whinnie,1994).

The impact of tellurium supply on cadmium telluride photovoltaics modules have become the lowest-cost producer of solar electricity, despite working at lower efficiency than crystalline silicon cell (Ken, 2010).

A spectrophotometric method for the determination of palladium, iron and tellurium from nitric acid media after the extraction of their p-[4-(3,5-dimethyl soxazol-yl) azophenylazo]calyx(4) arene complexes has been developed with possible synergistic effects (Kumar *et al.*, 2008)

Atomic absorption spectrometric determination of trace tellurium after hydride trapping on platinum-coated tungsten coil has been worked out. This method was applied to the determination of tellurium in several geological standard reference materials, by hyphenation of electrically heated quartz tube atomic absorption spectrometry and tellurium hydride trapping on platinum-coated tungsten coil. With a mixture of Ar and H₂, tellurium hydride was transported to tungsten coil for trapping at 390°C and releasing at 1200°C (Maoyang *et al.*, 2010).

Te (IV) and Te (VI) were determined by employing dispersive liquid-liquid microextraction combined with electrothermal atomic absorption spectrometry using palladium as a permanent modifier. Under acidic condition (pH1), only Te (IV) formed a complex with ammonium pyrrolidine dithiocarbamate (APDC) and therefore Te (IV) was determined in the sedimented organic phase while Te (VI) remained in the aqueous phase (Najafi, *et al.*, 2010).

Determination of bismuth, selenium and tellurium in nickel-based alloys and pure copper by flow-injection hydride generation atomic absorption spectrometry with ascorbic acid prereoduction and cupferron chelation extraction was performed in phosphate buffer. For H₂Te, the linear range for the calibration curve was 0.5-12 ng/ ml (Hui-Ming *et al.*, 2002).

The reagent used in this investigation chrome azurol S (CAS) is trisodium salt of sulphodichlorohydroxy dimethyl fuchson dicarboxylic acid. The structural formula of the reagent is:



Chrome Azurol S (MW = 605.28 g/ mole)

The above reagent has been used to determine indium (Poledniok and Buhl, 2006), vanadium (IV) (Starczewska, 2004) and cobalt (Mushran *et al.*, 2004) as ternary (in the presence of surfactant) or as binary complexes.

The present study involves the determination of Te (IV) using the reagent CAS. To the best of our knowledge, such investigation has not yet been touched.

EXPERIMENTAL

Apparatus

Spectral and absorbance measurements were carried out using CECIL CE7200 Aquarius spectrophotometer with 1-cm matched quartz cells.

The pH measurements were carried out using professional Beuchtop pH meter Bp3001.

Reagents

All chemicals used were of analytical reagent grade.

Stock Tellurium (IV) (100 µg/ ml) solution.

This solution was prepared by dissolving 0.0125 g of tellurium dioxide (Hopkin and Williams) in 2 ml concentrated HCl solution and the volume was completed to 100- ml with distilled water in a volumetric flask.

Working Tellurium (IV) (50 µg/ ml) solution.

This solution was prepared by diluting 50 ml of the above stock tellurium (IV) solution to 100 ml with distilled water in a volumetric flask.

Chrome Azurol S (CAS) reagent $(1 \times 10^{-4}M)$ solution.

It was prepared by dissolving 0.00605 g of Chrome Azurol S (Merck) in distilled water and the solution is diluted to 100 ml in a volumetric flask and then transferred to a dark bottle, where it was stable for at least seven days.

Buffer solution (pH10)

This solution was prepared by mixing 17.41 ml of 0.02 M sodium bicarbonate solution and 21.89 ml of 0.01 M sodium carbonate and the volume was diluted to 100 ml with distilled water in a volumetric flask (Perrin and Dempsey, 1974).

Cetylpyridinium chloride hydrate (CPC) $(1 \times 10^{-3}M)$ solution

This solution was prepared by dissolving 0.0358 g of cetylpyridinium chloride hydrate (CPC)(BDH) in distilled water and the volume was completed to 100 ml in a volumetric flask.

Interfering ion solutions.

A 100 μ g/ ml solution of each ion tested was prepared in distilled water.

Synthetic sea water.

It was prepared as described (Herkinson, 1965) by dissolving 2.2 g NaCl, 9.7 g $MgCl_2.6H_2O$, 3.7 g Na_2SO_4 , 1 g $CaCl_2$, 0.65 g KCl, 0.17 g $NaHCO_3$ and 0.023 g H_3BO_3 in about 500 ml of distilled water, then the solution was diluted to 1 l with distilled water in a volumetric flask.

RESULTS AND DISCUSSION

Preliminary Investigations

For subsequent investigations, 50 μ g of Te (IV) were taken in a 25 ml final volumes. Upon the addition of CAS reagent to Te (IV) solution, an orange-red species was formed against the corresponding yellow blank solution.

Optimization of Experimental Conditions

The effect of various factors that may affect the analytical characteristics of the coloured species has been investigated and optimal experimental conditions are selected.

Effect of pH

The effect of pH of the final reaction solutions is studied by adding different volumes of (0.01) M HCl or NaOH to the mixture containing 50 μ g of tellurium (IV) and 5 ml of $1 \times 10^{-4}M$ CAS. The spectrum was scanned for each coloured solution against its corresponding reagent blank, blank against distilled water and the final pH was measured. The results obtained were given in Table (1).

ml of acid or base	Absorbance / electrolyte added											
(0.01 M) solution added]	NaOH									
auucu	A ^S _B	λ_{max} , nm	A^{B}_{w}	рН	A ^S _B	λ _{max} , nm	A ^B _w	$\lambda_{max,}$ nm	рН			
0.0	0.073	486.9	0.248	2.4	0.073	486.9	0.248	434	2.49			
1	0.006	_	0.123	2.47	0.151	487.1	0.271	431	2.50			
3	-0.001	_	0.121	2.40	0.173	501	0.286	425.3	2.53			
4	-0.001	_	0.118	_	0.180	501	0.292	425.3	2.59			
5	-0.010	_	0.123	_	0.192	501.5	0.275	425.3	2.6			
7	-0.017	_	0.127	_	0.201	502	0.266	425.3	2.69			
8	_	_	-	_	0.208	502	0.264	425.3	2.81			
9	_	_	_	_	0.220	502.5	0.264	425.3	2.86			
10	_	_			0.294	505.3	0.257	425.3	2.96			
11	_	_	_	_	0.300	505	0.227	425.3	3.1			
12	_		-	_	0.294	502.5	0.219	425.3	3.25			
13	_		_	_	0.290	500	0.210	425.3	3.48			
14	_		_	_	0.273	497	0.204	425.3	4.07			
15	-		—	-	0.065	424.1	0.200	425.3	9.35			

Table 1: Effect of pH on the absorbance of Te solution

From the results in Table (1), it can be shown that pH values (2.96-3.48) were favorable for the formation of the coloured compound. Furthermore, the wavelengths of maximum absorption of both standard and blank were constant. The pH was 3.1 although 11 ml of 0.01M NaOH solution was added because the element was originally dissolved in some strong acid medium.

At the optimum amount of NaOH (11 ml of 0.01M) solution, buffers of pH10 (sodium carbonate + sodium bicarbonate) and pH12 (NaOH, KCl buffer) (Perrin and Dempsey, 1974) have been tried. pH10 gives more useful results than pH12 and, therefore, were incorporated in the subsequent experiments. Also, pH10 gave more useful results in terms of colour contrast while NaOH solution gave lower blank value. The experimental results were represented in Table (2).

mL of		Absorbance / mL of buffer added							
buffer	pН	pH10 at λ 515 nm*			pH12				
solution	A ^S _B	A ^B _W	pH _{formation}	A ^S _B	A ^B _W	λnm	pH _{formation}		
1	0.087	0.038	2.88	0.195	0.039	519	2.67		
2	0.098	0.039	3.01	0.208	0.041	516	2.72		
3	0.100	0.040	3.06	0.204	0.044	516	2.88		
4	0.122	0.042	3.09	0.197	0.050	516	3.13		
5	0.155	0.044	3.10	0.057	0.050	495	5.80		
6	0.193	0.045	3.15	0.015	0.055	448	9.48		
7	0.181	0.046	3.32	0.011	0.056	432	10.74		
8	0.169	0.046	4.53	0.009	0.062	422	11.98		
9	0.153	0.047	5.15	0.007	0.074	420	11.05		
10	0.148	0.047	6.09	0.005	0.150	420	11.14		
11	0.140	0.048	6.52	Turbid					
12	0.109	0.048	6.60	Turbid					
13	0.083	0.052	6.83		Tu	ırbid			

Table 2: Effect of buffers on absorbance

*With buffers, λ_{max} shifts from 506 nm to 515 nm a bicarbonate carbonate buffer.

According to the results in Table 2, 6 ml of pH10 buffer solution was chosen for the subsequent work, since it gave a good absorbance for the standard and lower blank value.

For the final pH of 3.1, pH10 and pH12 have been selected for the examination. These higher pH values buffers were chosen because the Te element was initially dissolved in acid solution (see experimental). The effect of the amount of buffer solutions on the absorbance of the final coloured solutions was shown in Table (2).

Effect of Reagent Amount and The Concentration of Tellurium (IV)

The formation of the coloured complex has been completed using 7 ml of $1 \times 10^{-4} M$ CAS, the results were shown in Table (3).

 Table 3: Effect of the reagent amount and concentration of tellurium

ML of CAS reagent		Absorbance / µg of Tellurium added					
	5	25	40	50	75*	100*	\mathbf{R}^2
1	0.011	0.050	0.075	0.093	0.078	0.050	0.9988
3	0.017	0.125	0.173	0.205	0.185	0.163	0.9803
5	0.025	0.135	0.204	0.245	0.191	0.111	0.9960
7	0.045	0.130	0.191	0.230	0.205	0.190	0.9997
9	0.012	0.144	0.245	0.295	0.179	0.156	0.9955

*75 μ g and 100 μ g were not considered when calculating R².

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The coefficient of determination correlates that 7 ml of $1 \times 10^{-4}M$ CAS was the optimum amount for the determination of (5-50) µg Te (IV) ion.

Effect of Surfactants

The effect of different types of surfactants (cationic, anionic and non-ionic) with different concentrations on the intensity of the coloured complex with different orders of addition has been examined and the results were shown in Table (4).

	Surfactant	Order	Variable		А	bsorbanc	e*/ ml of	surfactar	nt solutior	1	
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $	solution used			0.0	1.0	2	3	4	5	λ_{max} nm	pН
$ \begin{array}{ c c c c c c c } (1 \times 10^3 M) & 1 & A^{B}_{u} & 0.070 & 0.055 & 0.052 & 0.050 & 0.050 & 0.050 \\ \hline H & A^{S}_{B} & 0.269 & 0.315 & 0.320 & 0.327 & 0.315 & 0.310 \\ \hline A^{B}_{u} & 0.070 & 0.055 & 0.052 & 0.050 & 0.050 & 0.050 \\ \hline H & A^{S}_{B} & 0.269 & 0.311 & 0.143 & 0.069 & 0.065 & 0.063 \\ \hline A^{B}_{u} & 0.065 & 0.052 & 0.050 & 0.050 & 0.050 & 0.050 \\ \hline A^{B}_{u} & 0.065 & 0.052 & 0.050 & 0.050 & 0.050 & 0.050 \\ \hline A^{B}_{u} & 0.065 & 0.052 & 0.050 & 0.050 & 0.050 & 0.050 \\ \hline A^{B}_{u} & 0.070 & 0.044 & 0.032 & 0.026 & 0.025 & 0.025 \\ \hline H & A^{S}_{B} & 0.269 & 0.382 & 0.380 & 0.370 & 0.358 & 0.356 \\ \hline A^{B}_{u} & 0.070 & 0.040 & 0.035 & 0.028 & 0.025 & 0.025 \\ \hline H & A^{S}_{B} & 0.269 & 0.395 & 0.380 & 0.360 & 0.358 & 0.357 \\ \hline A^{B}_{u} & 0.070 & 0.060 & 0.052 & 0.051 & 0.049 & 0.049 \\ \hline H & A^{S}_{B} & 0.269 & 0.235 & 0.280 & 0.255 & 0.025 \\ \hline H & A^{S}_{B} & 0.269 & 0.247 & 0.253 & 0.258 & 0.253 & 0.245 \\ \hline A^{B}_{u} & 0.070 & 0.055 & 0.052 & 0.052 & 0.052 \\ \hline H & A^{S}_{B} & 0.269 & 0.291 & 0.298 & 0.310 & 0.312 & 0.304 \\ \hline H & A^{S}_{B} & 0.269 & 0.201 & 0.049 & 0.049 & 0.049 \\ \hline H & A^{S}_{B} & 0.269 & 0.230 & 0.233 & 0.258 & 0.250 & 0.051 \\ \hline H & A^{S}_{B} & 0.269 & 0.201 & 0.052 & 0.052 & 0.052 \\ \hline H & A^{S}_{B} & 0.269 & 0.201 & 0.029 & 0.010 & 0.012 & 0.004 \\ \hline H & A^{S}_{B} & 0.269 & 0.201 & 0.049 & 0.049 & 0.048 & 0.048 \\ \hline H & A^{S}_{B} & 0.269 & 0.230 & 0.233 & 0.258 & 0.240 & 0.231 \\ \hline H & A^{S}_{B} & 0.269 & 0.239 & 0.23 & 0.258 & 0.205 & 0.051 \\ \hline H & A^{S}_{B} & 0.269 & 0.239 & 0.23 & 0.258 & 0.240 & 0.231 \\ \hline H & A^{S}_{B} & 0.269 & 0.239 & 0.23 & 0.258 & 0.240 & 0.231 \\ \hline H & A^{S}_{B} & 0.269 & 0.239 & 0.23 & 0.258 & 0.240 & 0.231 \\ \hline H & A^{S}_{B} & 0.269 & 0.239 & 0.23 & 0.258 & 0.240 & 0.231 \\ \hline H & A^{S}_{B} & 0.269 & 0.239 & 0.23 & 0.258 & 0.240 & 0.231 \\ \hline H & A^{S}_{B} & 0.269 & 0.239 & 0.23 & 0.258 & 0.240 & 0.241 \\ \hline H & A^{S}_{B} & 0.269 & 0.239 & 0.23 & 0.258 & 0.240 & 0.241 \\ \hline H & A^{S}_{B} & 0.269 & 0.239 & 0.23 & 0.258 & 0.240 & 0.241 \\ \hline H & A^{S}_{B} & 0.269 & 0.239 & 0.23$	СТАВ		A ^S _B	0.269	0.350	0.376	0.372	0.365	0.360		
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $	$(1 \times 10^{-3} M)$	Ι	A ^B _w	0.070	0.055	0.052	0.050	0.050	0.050		
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $		II	A ^S _B	0.269	0.315	0.320	0.327	0.315	0.310		
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $											
$ \begin{array}{c c c c c c c c c } & A^8_B & 0.269 & 0.311 & 0.143 & 0.069 & 0.065 & 0.063 \\ \hline A^8_w & 0.065 & 0.052 & 0.050 & 0.050 & 0.050 \\ \hline A^8_w & 0.070 & 0.044 & 0.032 & 0.026 & 0.025 & 0.025 \\ \hline A^8_w & 0.070 & 0.044 & 0.032 & 0.026 & 0.025 & 0.025 \\ \hline A^8_w & 0.070 & 0.040 & 0.035 & 0.028 & 0.025 & 0.025 \\ \hline A^8_w & 0.070 & 0.040 & 0.035 & 0.028 & 0.025 & 0.025 \\ \hline A^8_w & 0.070 & 0.040 & 0.035 & 0.028 & 0.025 & 0.025 \\ \hline A^8_w & 0.070 & 0.040 & 0.035 & 0.028 & 0.025 & 0.025 \\ \hline A^8_w & 0.070 & 0.037 & 0.035 & 0.028 & 0.025 & 0.025 \\ \hline A^8_w & 0.070 & 0.037 & 0.035 & 0.028 & 0.025 & 0.025 \\ \hline A^8_w & 0.070 & 0.060 & 0.052 & 0.051 & 0.049 & 0.049 \\ \hline A^8_w & 0.070 & 0.055 & 0.052 & 0.052 & 0.050 \\ \hline A^8_w & 0.070 & 0.055 & 0.052 & 0.052 & 0.050 \\ \hline A^8_w & 0.070 & 0.055 & 0.052 & 0.052 & 0.050 \\ \hline A^8_w & 0.070 & 0.055 & 0.052 & 0.052 & 0.050 \\ \hline A^8_w & 0.070 & 0.055 & 0.052 & 0.052 & 0.050 \\ \hline A^8_w & 0.070 & 0.055 & 0.052 & 0.052 & 0.050 \\ \hline A^8_w & 0.070 & 0.055 & 0.052 & 0.052 & 0.050 \\ \hline A^8_w & 0.070 & 0.055 & 0.052 & 0.050 & 0.049 \\ \hline A^8_w & 0.070 & 0.055 & 0.052 & 0.052 & 0.050 & 0.049 \\ \hline A^8_w & 0.070 & 0.055 & 0.054 & 0.052 & 0.050 & 0.049 \\ \hline A^8_w & 0.070 & 0.055 & 0.054 & 0.052 & 0.050 & 0.049 \\ \hline A^8_w & 0.070 & 0.055 & 0.054 & 0.052 & 0.050 & 0.049 \\ \hline A^8_w & 0.070 & 0.055 & 0.054 & 0.052 & 0.050 & 0.049 \\ \hline A^8_w & 0.070 & 0.055 & 0.054 & 0.052 & 0.050 & 0.049 \\ \hline A^8_w & 0.070 & 0.055 & 0.054 & 0.052 & 0.050 & 0.049 \\ \hline A^8_w & 0.070 & 0.051 & 0.049 & 0.048 & 0.048 & 0.048 \\ \hline A^8_w & 0.070 & 0.051 & 0.049 & 0.048 & 0.048 & 0.048 \\ \hline A^8_w & 0.070 & 0.051 & 0.049 & 0.048 & 0.048 & 0.048 \\ \hline A^8_w & 0.070 & 0.050 & 0.049 & 0.048 & 0.047 & 0.047 \\ \hline A^8_w & 0.070 & 0.050 & 0.049 & 0.048 & 0.047 & 0.047 \\ \hline A^8_w & 0.070 & 0.050 & 0.049 & 0.048 & 0.047 & 0.047 \\ \hline A^8_w & 0.070 & 0.050 & 0.049 & 0.048 & 0.047 & 0.047 \\ \hline A^8_w & 0.070 & 0.050 & 0.049 & 0.048 & 0.047 & 0.047 \\ \hline A^8_w & 0.070 & 0.050 & 0.049 & 0.048 & 0.047 & 0.047 \\ \hline A^8_w & 0.070 & 0.050 & 0.049 & 0.048 & 0.047 & 0.047 \\ \hline A^8$			A^{B}_{w}	0.070	0.055	0.052	0.050	0.050	0.050	524.5	3.02
$ \begin{array}{ c c c c c c c c c } \hline Hi & I & I & I & I & I & I & I & I & I & $			A ^S _B	0.269	0.311	0.143	0.069	0.065	0.063		
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $		III									
$ \begin{array}{c c c c c c c c c } \hline CPC & & & & & & & & & & & & & & & & & &$			A^{B}_{w}	0.065	0.052	0.050	0.050	0.050	0.050		
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $			e.								
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	CPC		A ^s _B	0.269	0.380	0.376	0.365	0.360	0.359		
$ \frac{ A^8_w 0.070 0.044 0.032 0.026 0.025 0.025 0.025 }{A^8_w 0.070 0.040 0.038 0.380 0.370 0.358 0.356 0.356 0.255 0.255 0.265 0.2$	(1×10 ⁻³ M)	I	. P		0.044		0.000	0.0 0.7			
$ \frac{11}{10^{4}} + \frac{A^{8}_{B}}{A^{8}_{W}} + 0.269}{A^{8}_{W}} + 0.070 + 0.040 + 0.035 + 0.028 + 0.025$			$\mathbf{A}_{\mathbf{w}}^{\mathbf{b}}$	0.070	0.044	0.032	0.026	0.025	0.025		
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $			A ^S _P	0.269	0.382	0.380	0.370	0.358	0.356	525	2 1 4
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $		II	T B							525	5.14
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $			A ^B _w	0.070	0.040	0.035	0.028	0.025	0.025		
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $		III	A ^S _B	0.269	0.395	0.380	0.360	0.358	0.357		
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $			Б								
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $			A ^B _w	0.070	0.037	0.035	0.028	0.025	0.025		
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	an a		. 5	0.0(0)	0.0.17	0.050	0.050	0.0.50	0.045		
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $	SDS		$\mathbf{A}_{\mathbf{B}}$	0.269	0.247	0.253	0.258	0.253	0.245		
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $	$(1 \times 10^{-1} \mathrm{M})$	1	▲ B	0.070	0.0(0	0.052	0.051	0.040	0.040		
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$			A_{W}^{-}	0.070	0.060	0.052	0.051	0.049	0.049		
$\begin{array}{c c c c c c c c c c c c c c c c c c c $		п	AB	0.269	0.235	0.280	0.295	0.297	0.286	511	3 21
$\begin{array}{c c c c c c c c c c c c c c c c c c c $		11	A B	0.070	0.055	0.052	0.052	0.052	0.050	511	5.21
$\begin{array}{c c c c c c c c c c c c c c c c c c c $				0.070	0.055	0.032	0.032	0.032	0.030		
$\begin{array}{c c c c c c c c c c c c c c c c c c c $		ш	A B	0.209	0.291	0.298	0.310	0.312	0.304		
$\begin{array}{c c c c c c c c c c c c c c c c c c c $		111	AB	0.070	0.055	0.054	0.052	0.050	0.049		
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	Triton V_			0.070	0.033	0.034	0.052	0.030	0.049		
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	100(1%)		LA B	0.207	0.220	0.255	0.230	0.240	0.231		
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	100(170)	I	AB	0.070	0.051	0.049	0.049	0.048	0.048		
$\begin{array}{ c c c c c c c c c c c c c c c c c c c$		-	A ^S _p	0.269	0.031	0.013	0.225	0.220	0.219		
$\begin{tabular}{ c c c c c c c c c c c c c c c c c c c$		Π	- B	0.209	0.209	0.20	0.220	0.220	0.21)	504.5	3.64
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$			A ^B _w	0.070	0.050	0.049	0.048	0.047	0.047		
$\left \begin{array}{c c} \mathbf{H} & \mathbf{B} \\ \mathbf{H} \\ \mathbf{H}$			A ^S _D	0.269	0.230	0.228	0.226	0.223			
		ш	τ , Β	0.207	0.230	0.220	0.220	0.225			
$\mathbf{A}^{\mathbf{B}}_{\mathbf{w}}$ 0.070 0.050 0.050 0.050 0.050			A ^B _w	0.070	0.050	0.050	0.050	0.050			

Table 4: Effect of surfactant amounts

 $\lambda_{\text{max}} = 515 \text{ nm}$

Order number I: (M+B+R+S), II: (M+B+S+R), III: (M+S+B+R)

The results shown in Table (4) show that the maximum absorbance has been achieved using 1 ml of $1 \times 10^{-3}M$ cetylpyridinium chloride hydrate (CPC) with the order (tellurium (IV) ion+ CPC + pH 10 + CAS reagent).

The presence of CPC surfactant afforded both hyperchromic effect and bathochromic shift (515 nm to 525 nm) which are both admired from the analytical point of view.

Effect of time and CPC Surfactant

The effect of time on the colour intensity of the Te (IV)-CAS chelate produced from different amounts of tellurium (IV) ion has been explored. The maximum formation of the coloured complex took place after 5 min, and remained stable for about 60 min at least in the presence of CPC, while in the absence of CPC lower absorbencies were observed and less efficient stability was attained. The results are shown in Table (5)

Time	Absorba	nce / µg Te (IV) added					
Min.	CPC abs	sent			CPC pro	esent		
	5	25	40	50	5	25	40	50
5	0.012	0.128	0.188	0.220	0.025	0.345	0.365	0.378
10	0.014	0.125	0.194	0.220	0.026	0.340	0.362	0.375
20	0.016	0.125	0.194	0.210	0.026	0.315	0.362	0.370
30	0.015	0.124	0.194	0.208	0.026	0.314	0.362	0.370
40	0.015	0.124	0.194	0.208	0.026	0.314	0.362	0.370
50	0.015	0.124	0.186	0.208	0.026	0.314	0.362	0.370
60	0.015	0.12	0.186	0.205	0.025	0.314	0.362	0.370

Table 5: Effect of time and CPC surfactant on absorbance and stability

Final Absorption Spectra

The absorption spectra have been obtained on solution containing 50 µg Te (IV), 1 ml of $1 \times 10^{-3}M$ CPC, 6 ml pH10, 7 ml of $1 \times 10^{-4}M$ CAS, and the absorption spectra of the coloured complex are shown in Fig.1 at 525 nm, while the reagent blank gives maximum absorption at 423 nm.



Wavelength, nm

Fig. 1: Absorption spectra of 50 µg Te (IV) treated as under optimum experimental conditions and measured (A): against blank, (B): against distilled water and (C): blank measured against distilled water.

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Recommended procedure and calibration graph

Under the optimum established conditions, described before, a calibration graph has been constructed as follows: increasing volumes of 50 μ g/ ml solution were transferred into 25-ml volumetric flasks to cover the range (5-75) μ g/25 ml. Beer's law was obeyed over the range of (5-50) μ g Te (IV) Fig. (2). The average molar absorptivity of the coloured complex was calculated from the calibration graph and found to be (2.5×10^4) 1.mol⁻¹.cm⁻¹, Sandell's sensitivity was (5.104×10^{-3}), while the limit of detection (LOD) and the limit of quantization (LOQ) (Valcarcel, 2000) were found to be (61.8×10^{-3}) μ g/ ml and (84.6×10^{-3}) μ g/ ml, (n= 30) respectively. At higher concentrations (> 50 μ g/ 25 ml) of Te (IV), a negative deviation was observed.



Concentration, µg/ 25ml

Fig. 2: Calibration graph for tellurium (IV) determination with chromeazurol S reagent.

Accuracy and precision

To check the accuracy and precision of the calibration graph, tellurium (IV) was determined at three different concentrations.

Table 6:	Accuracy	and	Precision
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Te(IV) taken, μg	Recovery, % *	Relative Standard
		Deviation, %
25	98.2	± 1.8
40	100.2	±/0.2
50	100.8	± 0.8

*For six determinations

The data given in Table 6 indicate that the results are satisfactory.

Nature of the complex

Job's method of continuous variations and mole-ratio method (Hargis, 1988) was used to evaluate the combining ratio between Te (IV) ion and CAS chelating agent. The results revealed that the ratio of tellurium (IV) to CAS is 1:2. Also, Job's method of continuous variations was used to determine the proportion of CPC in the complex. The results revealed that the components react in a ratio of 1: 4 Te to CPC. The structural formula of the chelate may be postulated as follows:

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Spectrophotometric Determination.....



The stability constant of the postulated chelate is $3.6 \times 10^{10} M^{-2}$.

Effect of organic solvents

Different organic solvents were used to examine their effects on the Te (IV)-CAS coloured complex. The results are shown in Table (7) and (Fig. 3). H_3C

Table 7 : Effect of solvents on the optical properties of Te (IV) - CAS

Solvent	Absorba	nce	λ , max _{nm}	ε, l.mol ⁻¹ cm ⁻¹	
	A ^S _B				
Ethanol	Turbid				1
n-Propanol	0.153	0.086	479	9.7×10^{3}]
Formic acid			Turbid		
Acetone			Turbid		
DMF	0.213	0.063	520	$1.4 \times Te^4$ -Chrome A	zurol
Dioxane	0.124	0.102	487	7.9×10^{3}	
Tetrahydrofuran	0.343	0.128	485	2.2×10^4	
Water	0.382	0.025	525	$2.4 imes 10^4$	
0.4 0.3 0.2 0.1 Pro	F	- A		Water Dioxane	

Fig. 3: Effect of organic solvents on the spectral properties of Te (IV) - CAS chelate.

As shown in Table (7), water seems to be still the most favorable solvent from the analytical point of view.

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С

Ο

Ο

OCPC

Study of interferences

In order to assess the analytical applications of the proposed method, the effects of foreign ions were examined by adding known amounts of each foreign ion to a solution containing 50 μ g of tellurium (IV), and then determining the latter using the recommended procedure. The results obtained are summarized in Table (8).

	v		
Foreign ion	Form added	Amount added, ug	Interferences, %
D;3+		50	0.7
DI	BI (1403)3.51120	100	-9.7
p1 2+	N (NO)	100	-10.5
Pb ²	Pb(NO_3) ₂	50	-12.9
4.		100	-35.9
Se ⁴⁺	SeO ₂	50	-63.5
		100	-64.9
Ce ³⁺	CeCl ₃ .7H ₂ O	50	0.0
		100	0.0
Th ⁴⁺	Th(NO ₃) ₄ .6H ₂ O	50	-47.0
	(5)4 2-	100	-45.4
Li ¹⁺	Li ₂ SO ₄ H ₂ O	50	-0.3
2.	212004.1120	100	-1.4
Cd^{2+}	Cd(CH,COO), 2H,O	50	62
Cu	Cu(CII3COO)2.2II2O	100	-0.2
G.,2+	S-CL (ILO	100	-0.2
Sr	SICI2.0H2O	50	14.5
a 2+	a. a.	100	14.7
Ca ²	CaCl ₂	50	+0.8
1.		100	-3.5
Na ¹⁺	NaCl	50	-2.2
		100	-1.9
Cu ²⁺	CuSO ₄	50	10.0
		100	23.2
Hg ²⁺	HgCl ₂	50	-4.3
č	0 2	100	-4.1
V ⁵⁺	NH4VO2	50	+2.7
	1114,003	100	-10.8
A 1 ³⁺	AICL 6H.O	50	+0.7
ЛІ	AIC13.01120	100	+15.0
NT:2+	Nicl (ILO	100	+13.9
INI	NICI ₂ .0H ₂ O	50	-0.9
- 2+		100	+8.1
Fe ³⁺	$Fe(NH_4)(SO_4)_2.6H_2O$	50	-2.9
		100	-5.5
Cr ³⁺	CrCl ₃ .6H ₂ O	50	-7.5
		100	-4.2
Sn ²⁺	SnCl ₂ .2H ₂ O	50	+0.9
		100	-4.5
Mn ²⁺	MnCl ₂ .4H ₂ O	50	-19.5
		100	-18.8
Mg ²⁺	MgSO4.7H2O	50	-2.3
0	0	100	-16.9
W ⁶⁺	Na ₂ WO ₄ 2H ₂ O	50	-3.2
	11421104.21120	100	+21.4
Mo ⁶⁺	$(NH_{\rm e})$, Mo $(0.14H_{\rm e})$	50	13
IVIO	(14114)6.14107024.41120	100	-1.5
D 2+	D CL ML O	100	-23.1
Ba	BaCl ₂ .2H ₂ O	50	+8.5
a. a. 2		100	+16.1
$S_2O_3^{2^2}$	$Na_2S_2O_3.5H_2O$	50	+3.0
		100	+6.5
HCO ₃ ¹⁻	NaHCO ₃	50	-3.1
		100	+7.6
CO_{3}^{2}	Na ₂ CO ₃	50	-8.5
		100	+10.8
CrO ₄ ²⁻	K ₂ CrO ₄	50	+2.5
		100	+5.8
BO ₃ ³⁻	H ₃ BO ₃	50	-1.2
- 5	5-5	100	+3.1
Br ¹⁻	KBr	50	+0.4
51	15.01	100	+0.8
SO 2-	N2.80	50	1.0
504	1102504	100	-1.0
CONI-	KOON	100	+7.1
SUN	KSUN	50	+/.1
H DO -	N H DO AVI O	100	-10.3
H_2PO_4	NaH ₂ PO ₄ .2H ₂ O	50	+1.5
1	1	100	+4.6

 Table 8: Study of interference

The results in the above table indicate that a high negative and positive errors were observed in the case of Al (III), Bi (III), Th (IV), Ba (II), Mn (II), Pb (II), Se (IV), Cu (II), SCN⁻ and $CO_3(^-$ II) due to the competition of these ions with Te (IV) for the reagent (appearance of blue colour) there by depleting the reagent available for the Te (IV) ion. **Application of the method**

The method was applied to the determination of tellurium ion in synthetic composite mixture first and in sea water second. The results are listed below:

1. Determination of tellurium (IV) in synthetic composite mixture

Mixtures containing cations and anions and 50 μ g of tellurium (IV) were analyzed for tellurium. The results are given in Table (9).

Ion added	Mixture	Amount added, µg mixture	Recovery, %
	numbers		
$Sn^{+2}, Ce^{+3}, Ni^{+2}, SO_4^{-2}, S_2O_3^{-2}$	(I)	50	91.5
		100	104.5
$Ca^{+2}, Mg^{+2}, Ce^{+3}, BO_3^{-3},$	(II)	50	97.5
Br , SO_4^{-2}		100	131.6
$Ca^{+2}, Mg^{+2}, Ni^{+2}, Bi^{+3},$	(III)	50	88.3
$H_2PO_4^-, S_2O_3^{-2}$		100	88.0
$H_2PO_4^-, Ce^{+3}, W^{+6}, Mg^{+2},$	(IV)	50	104.0
SO_4^{-2}, BO_3^{-3}		100	108.8
Ni ⁺² , Mg ⁺² , W ⁺⁶ , Ca ⁺² ,	(V)	50	103.1
$S_2O_3^{-2}, Br^{-1}$		100	106.3

Table 9: Recovery of Tellurium (IV) in synthetic mixture

Other composite mixtures were tried but results were unsuccessful. It can be observed Table (9) that the method gives a successful recovery of tellurium (IV), and applicable for all cases except for mixture (III)

2. Determination of Tellurium (IV) in sea water

The following Table shows the results obtained for the determination of Te (IV) in sea water which was prepared in the laboratory (Henriksen, 1965).

Table 10: Determination of Te (IV) in sea water sample

Sample of sea water (mL)	Te (IV) added, μg	Recovery* %
0.5	25	96.3
	40	100.2
	50	98.5
1.0	25	95.1
	40	102.9
	50	93.31
1.5	25	89.7
	40	103.2
	50	90.2

*Average of three determinations.

The results in Table (10) were satisfactory to some extent and recovery ranged from 89.7 to 103.2%.

Comparison of the method

Table (11) shows the comparison between the analytical variables of the present method with another method (Marczenko, 2000).

Table 11: Comparison of the method

Analytical parameters	Present method	Literature method (Marczenko, 2000)
Reagent	Chrome azurol S	bismuthiol (II)
λ_{max} (nm)	525	330
Surfactant used	CPC	
Beer's Law range (ppm)	0.2-2	
pH	10	8
Sandell's index (µg.cm ⁻²)	5.104×10 ⁻³	1.595×10^{-2}
Nature of Complex	1:2	1:4
Molar absorptivity (l.mol ⁻¹ .cm ⁻¹)	2.5×10^4	8×10^3
$K, (M^{-2})$	3.6×10^{10}	

The present method differs from the literature method in two main things:

- (a) The introduction of CPC surfactant, gives more sensitive reaction with Te (IV) ion than the literature method.
- (b) The detailed optimization of experimental conditions has not been established yet.

REFERENCES

- Fluka, Riedel-Dehean, Sigma-Aldrich. (2007-2008). "Labrotary Chemical, and Analytical Reagents". 526 p.
- Hargis, L.G. (1988). "Analytical Chemistry Principles and Techniques". PrenticHall International Ltd., London, pp. 424-426.
- Henriksen, A. (1965). An automatic method for determining nitrate and nitrite in fresh and saline waters. *Analyst.* **90**, 38-88
- Hui-Ming, L.; Shi-Yang, C.; Pin-Husan, C.; Suh-Jen, J.T. (2002). Determination of bismuth, selenium and tellurium in nicke alloys and pure copper by flow-injection hydride generation atomic absorption spectrometry-with ascorbic acid prereduction and cupferron chelation-extraction. *Anal. Chim. Acta*, **459**, 161-168.
- Ken, Z. (2010). The impact of tellurium supply on cadmium telluride photovoltaics. *Science*, **328**(5979), 699-701.
- Kumar, A.; Sharma, P.; Chandel, L.K. (2008). Synergistic extraction and spectrophotometric determination of palladium (II), iron (III), and tellurium (IV) at trace level by newly synthesized p-[4-(3,5-dimethylisoxazolyl) azophenylazo] calyx (4). J. Incl. Phenom. Macrocycl. Chem. 61, 335-342.
- Maoyang, Xi.; Rui, I.; Peng, Wu.; Kailai, Xu.; Xiandeng, H.; Yi, L. (2010). Atomic absorption spectrometric determination of trace tellurium after hydride trapping on platinum-coated tungsten coil. *Microchem. J.*, **95**(2), 320-325.

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- Marczenko, Z.; Balcerzak, M. (2000). "Analytical Spectroscopy Library Separation, Preconcentration and Spectrophotometry in Inorganic Analysis". EllisHorwood Ltd, Chichester, Elsevier (CD- ROM). pp. 415-419.
- Mushran, S.P.; Prakash, O.; Sanyal, P. (2004). Spectrophotometric study of Co (II)- chrome azurol s chelate. J. Fur. Prak. Chem., **383** (4), 125-129.
- N.M.: Tavakoli. Н.; Alizadeh, R.; Seidi, S. (2010). Speciation and Najafi, determination ultra trace amounts inorganic tellurium of of in environmental water samples by dispersive liquid-liquid microextraction and electrothermal atomic absorption spectrometry. Anal. Chim. Acta, 670, (1-2), 18-23.
- Perrin, D.D.; Dempsey, B. (1974). "Buffers for pH and Metal Ion Control". Chapman and Halld, London, 47, 152 p.
- Poledniok, J.; Buhl, F. (2006). Application of chrome azurol s and benzyldodecyldimethylammonium bromide for determination of indium in zinc and its compounds. *Chem. Anal.*, **51**(4), 631.
- Starczewska, B. (2004). Spectrophotometric determination of vanadium (IV) by ternary complexes of chrome azurol S with imipramine and chlorprothixene. *J. Trace and Microprobe Techn.*, **20**(3), 377-384.
- Valcarcel, M. (2000). "Principles of Analytical Chemistry". Spring-Verlag, Berlin, 68 p.

Mediawlki, (2012). Wikipedia, in encyclopedia, at 09: 25, pp.1-5.

Whinnie, W.R.Mc. (1995). "Tellurium Inorganic Chemistry Encyclopedia of Inorganic Chemistry". Ed. R. Bruce King (1994). Jon Wiley and Sons.