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SIMPLE AND RAPID SPECTROPHOTOMETRIC DETERMINATION METHOD FOR TRACE LEVEL OF SILVER USING 2,2' -DI (2,3,4-TRIHIDROKSIFENILAZO)BIFENIL

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Abstract. A very simple, sensitive, highly selective and non-extractive spectrophotometric method for the determination of trace amounts silver(I) has been developed. 2,2'-di (2,3,4-trihidroksifenilazo)bifenil (DTFAB) has been proposed as a new analytical reagent for the direct non-extractive spectrophotometric determination of silver(I). In the water media DTFAB reacts with silver to give a highly absorbent greenish yellow chelate with a molar ratio 2:1(Ag:DTFAB) The reaction is instantaneous and the maximum absorption was obtained at 540 nm and remains stable for 24 h. The average molar absorptivity and Sandell's sensitivity were found to be 4.3×10^4Lmol^{-1} cm^{-1} and 5.0 µg cm^{-2} of silver(I), respectively. Linear calibration graphs were obtained for 0.1–30 µg mL^{-1}of silver(I). A large excess of over 30 cations, anions and complexing agents do not interfere in the determination. The method is highly selective for silver and was successfully applied to synthetic mixtures and a number of drink and tap water samples. The method has high precision and accuracy (S = ± 0.01 for 0.5 µg mL^{-1}).

Keywords: *spectrophotometry, silver, 2,2'-di (2,3,4-trihidroksifenilazo)bifenil, determination, drink water.*

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1. Introduction

Silver as a useful element has been widely used in industry, also in currency and as an investment medium, silver is used in solar panels, water filtration, jewellery, ornaments, high-value tableware and utensils (hence the term silverware), in electrical contacts and conductors, in specialized mirrors, window coatings, in catalysis of chemical reactions, as a colorant in stained glass and in specialized confectionery. Its compounds are used in photographic and X-ray film. Silver compounds and alloys have been widely used in dental and pharmaceutical preparations because of their marked antibacterial properties (Silver, n.d.). According to World Health Organization average silver concentrations in natural waters are 0.2–0.3 µg/litre, in drinking water in the USA that had not been treated with silver for disinfection purposes varied between “non-detectable” and 5 µg/litre(WHO,*2003*).Silver is both vital and toxic for many biological systems and its content in drink and tap water samples is increased with the increasing use of silver compounds and silver containing products in industry and in medicine. Thus, separation, preconcentration and sensitive determination of silver ion is of increasing interest.

1,5-Diphenylthiocarbazone is one of the most widely used photometric reagents and forms colored water-insoluble complexes with silver ions. Silver-dithizonecomplexes are water insoluble and thus their determination requires a prior solvent extraction step into $CHCl_3$ or CCl_4, followed by spectrophotometric

determinations (Pyatnitsky & Sukhan, 1973). Since these methods involve solvent extraction are lengthy and time-consuming and lack selectivity due to much interference, $CHCl_3$ and CCl_4 have been listed as toxic. This problem has been overcome in recent years by has been proposed a new analytical reagent for the direct non-extractive spectrophotometric determination of silver(I). The azocompounds on the base of pyroghallol had widely been appliedfor the determination of noble metal ions, this type of reagent has higher sensitivity and high selectivity. In the search for more sensitive azocompounds on the base of pyroghallol reagent, in this work, a reagent2,2'-di (2,3,4-trihidroksifenilazo)bifenil (DTFAB) was synthesized and a color reaction of DTFAB with Ag(I) in aqueous media was carefully studied.

Jamaluddin Ahmed et al. determined trace level silver using 1, 5-diphenylthiocarbazone in aqueous micellarsolutions at λ_{max} =495 nm (Jamaluddin & Syeda, 2014). Reemused meloxicam for the spectrophotometric micro determination of silver. The molar absorptivity of the formed complex was calculated to be 1.124 x 10^4 L mol^{-1} cm^{-1} at 412 nm (Reem, 2016). Hung et al. determined silver with 2-(3,5-dibromo-2-pyridylazo)-5-diethyl-aminophenol in the presence of anionic surfactant. The molar absorptivity is 7.7×10^4 $l\cdot mole^{-1}\cdot cm^{-1}$ at 570nm (Hung *et al.*, 1982).

Although many sophisticated techniques, such as electrothermal AAS (Manzoori *et al.*, 2007, Rahman *et al.*,2004), flame AAS (Chakrapani *et al.*, 2001, Tunceli & Turker, 2000), graphite furnace AAS (Resano *et al.*, 2006), liquid chromatography (Hu *et al.*, 2003), electrophoresis (Aguilar *et al.*,1993) are available for the determination of silver at trace levels in numerous complex materials, factors such as the low cost of the instrument, easy handling, portable, lack of any requirement for consumables and almost no maintenance, have caused spectrophotometry to remain a popular technique, particularly in laboratories of developing countries with limited budgets.

The aim of the present study is to develop a simpler direct spectrophotometric method for the trace determination of silver(I) with DTFAB in aqueous solutions. The method described here has recorded for the first time the non-extractive direct spectrophotometric determination of silver(I) in aqueous media without the recourse of any “clean-up” step. This method is far more selective, sensitive, non-extractive, simple and rapid than all of the existing spectrophotometric methods (Fujimura *et al.*, 2011; George *et al.*, 1997; Ghaedi *et al.*, 2007; Guo *et al.*, 2002; Hung *et al.*, 1982; Jamaluddin & Syeda, 2014; Kassem, 2015; Nagaraja *et al.*, 2002; Salinas *et al.*, 1995). This method is very reliable and a concentration in the µg mL^{-1} range in an aqueous medium at room temperature can be measured in a very simple and rapid way.

2. Experimental section

Instrumentation

The absorbance of solutions was measured with a Perkin Elmer (United States) (Model: Lambda-40) double-beam UV/VIS spectrophotometer and with a KFK-2 photoelectrocolorimeter (Russia), with 1 cm matched quartz cells. The pH values of solutions was controlled on the ionomer İ-121 with glass electrode customized by standard buffer solutions.

Chemicals and Reagents

All of the chemicals used were of analytical reagent grade or the highest purity available. Distilled deionized water, which is non-absorbent under ultraviolet radiation, was used throughout. Glass vessels were cleaned by soaking in acidified solution of

$KMnO_4$ or $K_2Cr_2O_7$ followed by washing with concentrated HNO_3 and rinsed several times with deionized water. Stock solutions and environmental water samples (1000 ml each) were kept in polypropylene bottles containing 1ml of concentrated HNO_3. More rigorous contamination control was applied when the silver levels in the specimens were low.

2,2'-di (2,3,4-trihidroksifenilazo)bifenil (2×10^{-3} M)

The reagent was synthesized according to the known method (Pyatnitsky & Sukhan, 1973). The solution was prepared by dissolving the requisite amount of 2,2'-di (2,3,4-trihidroksifenilazo)bifenilin a known volume of absolute ethanol. More dilute solutions of the reagent were prepared as required.

Standard silver solution (1×10^{-2} M)

A stock solution 1×10^{-2} M, 100 ml of silver(I) was prepared by dissolving 0.1575 g of silver nitratein 100 ml of distilled deionized water and added 0.1 ml con. HNO_3. The working standard of silver solution was prepared by suitable dilutions of this stock solution.

EDTA solution

A 100-mL stock solution of EDTA (0.01%) was prepared by dissolving 10 mg of A.C.S.grade (≥90%) ethylenediaminetetraacetic acid, dissodium salt dehydrate in (100-ml)deionized water.

Tartrate solution

A 100ml stock solution of tartrate (0.01%) was prepared by dissolving 10 mg of A.C.S. grade(99%) potassium sodium tartrate tetrahydrate in (100ml) deionized water.

Dilute ammonium hydroxide solution

All solutions of ammoniumhydroxide was prepared by diluting15 mlof 10^{-1}N NH_4OH (28-30% A.C.S. grade) to1 l with deionized water. The solution was stored in a glass bottle.

Other solutions

Solutions of a large number of inorganic ions and complexing agents were prepared from their analytical grade or equivalent grade, water soluble salts. In the case of insoluble substances, special dissolution methods were adopted.

General Procedure

To 0.1-1.0 ml of a slightly acidic solution containing 2×10^{-3} M of silver(I) in a 25-ml volumetric flask was mixed with 1.0-4.0 ml (preferably 2.0 ml) of 1×10^{-3} Mbis-(2,3,4-trihidroksifenilazo) benzidine solution (preferably 2.0 ml). The mixture was diluted up to the mark with pH solution to attain the necessary acidity, acetate ammonia buffer solutions (pH 3-11) and H_2SO_4 (pH 0-2) were used. After 5 minutes the absorbance was measured at540 nm against a corresponding reagent blank. The silver content in an unknown sample was determined using a concurrently prepared calibration graph.

3. Results and discussions

Absorption spectra

The absorption spectra of greenish yellow color of the silver - DTFAB system in presence of pH 8 solution were recorded using a spectrophotometer. The absorption spectra of the silver - DTFAB is a symmetric curve with maximum absorbance at 540 nm and an average molar absorptivity of 4.3 x 10^4 l mol^{-1} cm^{-1} (Fig. 1). The reagent blank having maximum absorbance wavelength at430 nm. In all instances,

measurements were made at 540 nm against a corresponding reagent blank. The reaction mechanism of the present method is as reported earlier.

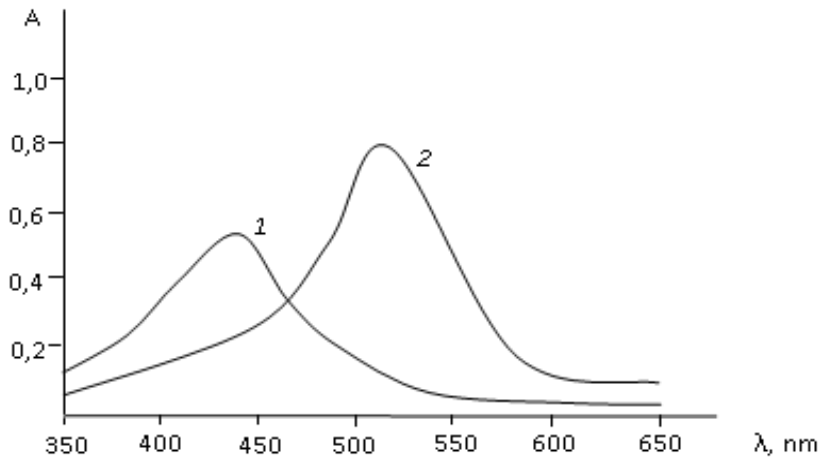

Figure 1. Absorption spectra of the reagent blank(1) and the Ag- DTFAB system (2).C_{DTFAB}=1,7×10^{-4} M, C_{Ag}=2×10^{-5} M

Effect of acidity

Of the various pH 0-10 of the solution studied, pH 7-9 was found to be the optimal for the silver - DTFAB system. The maximum and constant absorbance of the silver - DTFAB system was obtained in the presence of pH 8 at room temperature (25±0.5^{0}C). The absorbance of the reagent solution and the silver - DTFAB system depends on the medium pH; therefore, the absorption spectra are studied relative to a blank experiment (DTFAB).

Effect of time

The reaction is fast. Constant maximum absorbance was obtained just after 5 min of the dilution to volume at room temperature (25 ±0.5^{o}C), and remained strictly unaltered for 24 h.

Effect of temperature

The absorbance at different temperatures, 0–80°C, of a 25 ml solution of silver - DTFAB was measured according to the standard procedure. The absorbance was found to be strictly unaltered throughout the temperature range of 10–40°C. Therefore, all measurements were performed at room temperature (25 ±0.5^{o}C).

Effect of the reagent concentration

Different molar excesses of DTFAB were added to a fixed metal-ion concentration and the absorbance was measured according to the standard procedure. It was observed that a 1 μg $mL^{-1}$of silver metal (optical path length 1 cm), the reagent molar ratios of 1:10 to 1:50 produced a constant absorbance of Ag - DTFAB system. A greater excess of the reagent was not studied. For all subsequent measurements, 2 ml of 2 ×10^{-4} M DTFAB reagent was added.

Stoichiometry. The component ratio in the complexes was found using the isomolar series method, the relative yield method by Starik and Barbanel', and the equilibrium shift method (Hung *et al.,* 1982).All the methods showed that the component ratio was 2:1 in the the silver - DTFAB system. The number of protons displaced upon complexation was determined by the Astakhov method, and the

indicated component ratio in the complexes was confirmed (Jamaluddin & Syeda, 2014).

Analytical performance of the method

Calibration curve

The effect of silver(I) concentration was studied over 0.01–100 μg mL^{-1}, distributed in four different sets (0.01–0.1, 0.1–1, 1–10, 10–100 μg mL^{-1}) for convenience of the measurement. The absorbance was linear for 0.1–30 g mL^{-1} of silver(I) in aqueous media. From the slope of the calibration graph, the average molar absorption coefficient was found to be 4.3×10^4L mol^{-1} cm^{-1} in aqueous media. Of the four calibration graphs, the one showing the limit of the linearity range is given in Fig. 5; the next two were straight-line graphs passing through the origin (R_2 = 0.9986). The selected analytical parameters obtained with the optimization experiments are summarized in Table 1.

Table 1. Selected analytical parameters obtained by optimization experiments.

Parameters	Studied range	Selected value
Wavelength / λ_{max} (nm)	200-800	540
pH	0 - 11	8
Time/h	1 - 24h	5 - 10 min
Temperature /^oC	0 – 80^oC	25 ± 0.5^oC
Reagent(fold molar excess, M:R)	1:10 - 1:50	1:20
Molar absorption coefficient / l mol^{-1} cm^{-1}	1.5×10^4- 5.8×10^4	4.3×10^4
Linear range/ μg mL^{-1}	0.001-100	0.1-30
Detection limit / μg mL^{-1}	0.001	0.001
Sandell's sensitivity / μg cm^{-2}	0.1 - 100	5
Relative Standard	0 -2	0 -2
Regression Coefficient	0.998 - 0.9999	0.999

Effect of foreign ions

The effect of over 30 cations, anions and complexing agents on the determination of only 1 μg mL^{-1} of silver was studied. The criterion for interference (Kassem, 2015) was an absorbance value varying by more than 5% from the expected value for silver(I) alone. There was no interference from the following 1500-fold amount of EDTA and tartrate, a 100-fold amount. EDTA prevented the interference of a 40-fold excess of Al(III), 145-fold excess of Cr (VI), 120-fold excess of Pb (II), 140-fold excess of Na or a 40-fold excess of Sn (IV) and tartrate prevented the interference of a 30- fold excess of V (V) or Au(III) . During interference studies, if a precipitate was formed, it was removed by centrifugation. The quantities of these diverse ions mentioned were the actual amounts studied but not the tolerance limit. However, for those ions whose tolerance limit has been studied, their tolerance ratios are mentioned in Table 2.

Table 2. Tolerance limits of foreign ions, tolerance ratio [Species(x)]/Ag (w/w)

Species x	Tolerance ratio [Species (x) /Ag (w/w)]	Reference (Jamaluddin & Syeda, 2014)	Species x	Tolerance ratio [Species (x) /Ag (w/w)]	Reference (Jamaluddin & Syeda, 2014)
Na(I)	140	100	Au(III)	30	25
K(I)	140	10	Ga(III)	130	100
Mg(II)	170	100	La(III)	140	100
Ca(II)	160	20	Ta(IV)	150	100
Cr(III)	145	200	Ni(II)	165	100
Fe(III)	40	25	Pb(II)	120	100
Cu(II)	70	50	Cl^-	135	20
Cd(II)	120	50	HCO_3^-	170	100
Hg(II)	135	50	$C_2O_4^{2-}$	200	100
Mo(II)	145	100	EDTA	1500	1000
Co(III)	150	100	tartarat	1500	1000
Zn(II)	155	10	CH_3COO^-	1300	1000
Be(III)	140	50	Sr(II)	45	20
Sn(IV)	40	10	phosphate	130	100
Mn(II)	150	100	cyanide	140	50
V(V)	30	10	Al(III)	40	10

Precision and accuracy

The precision of the present method was evaluated by determining different concentrations of silver(I) (each analyzed at least five times). The relative standard deviation (n = 5) was 2%–0%, for 0.1–30 g of Ag(I) in 25 ml, indicating that this method is highly precise and reproducible. The detection limit (3s of the blank) and Sandell's sensitivity (concentration for 0.001 absorbance unit) for Ag(I) were found to be 1 μg mL^{-1}, 5 μg cm^{-2}, respectively. The reliability of our Ag-chelate procedure was tested by recovery studies. Regression analysis of Beer's law plots at 540 nm revealed a good correlation (R_2 = 0.999). The method was also tested by analyzing several synthetic mixtures containing silver and diverse ions (Table 3). The results for silver recovery were in good agreement with added values. The average percentage recovery obtained for the addition of silver spike to some drink and tap water samples were quantitative, as shown in (Table 4). Hence, the precision and accuracy of the method were found to be excellent.

Applications

The present method was successfully applied to the determination of silver in series of synthetic mixtures of various compositions and also in a number of drink water samples.

Determination of silver in synthetic mixtures

Several synthetic mixtures of varying compositions containing silver(I) and diverse ions of known concentrations were determined by the present method using EDTA as a masking agent and the results were found to be highly reproducible. The results are shown in Table 3. The accurate recoveries were achieved in all solutions.

Table 3. Determination of silver(I) in synthetic mixtures.

	Composition of mixture (mg mL^{-1})	Silver(I) (mg/ml)		Recovery ± S_b (%)
		Added	Found[a]	
A	Ag^+	1.5 2.0	1.49 1.98	99± 0.3 98± 0.4
B	As in A + Mg^{2+} (25)+Fe^{3+}(25)	1.5 2.0	1.48 1.98	98± 0.6 98 ± 0.2
C	As in B +Mn^{2+} (25)+Co^{3+} (25)	1.5 2.0	1.51 2.02	101 ± 0.2 102 ± 0.2
D	As in C +Cr^{3+}(25)+ Ca^{2+} (25)	1.5 2.0	1.52 2.04	102 ± 0.3 104 ± 0.2
E	As in D +Cu^{2+}(25)+Ni^{2+}25)	1.5 2.0	1.52 2.03	102 ± 0.4 101± 0.5

[a]Average of five analysis of each sample
[b]The measure of precision is the standard deviation (s).

Determination of silver in drink water samples

Each drink water samples (1000 ml) was mixed with 10 ml of concentrated HNO_3 and 2ml of concentrated H_2SO_4 in a 2000 ml distillation flask. The sample was digested in the presence of an excess potassium permanganate solution following a method recommended by Greenberg et al.(Greenberg *et al.*,1992). The solution was then cooled and neutralized with dilute NH_4OH solution. The resulting solution was then filtered and quantitatively transferred into 50 ml calibrated flask and made up to the mark with deionized water. An aliquot (1-2 ml) of this solution preconcentrated drink water was pipetted into a 25 ml calibrated flask and the silver content was determined as described under the general procedure using EDTA or tartrate as a masking agent. Analyzed drink and tap water samples were taken from markets of Baku city and Baku State University (BSU), respectively. The results of analyses of environmental water samples from various sources for silver are given in Table 4.

Table 4. Determination of silver(I) in some drink water samples

Sample	Silver(I) (µg mL^{-1})		Recovery ± S_b (%)	Sr^b (%)
	Added	Found[a]		
BonAqua	50 100	52 103	102 ± 0.4 103 ± 0.2	0.15 0.18
Vita1000	50 100	48 0.98	97 ± 0.3 98 ± 0.2	0.20 0.28
Shahdag	50 100	53 101	103 ± 0.2 101± 0.2	0.16 0.29
Damla	50 100	52 104	102 ± 0.3 104 ± 0.2	0.33 0.17
Slavyanka	50 100	47 102	97 ± 0.3 102 ± 0.2	0.21 0.23
Sirab	50 100	51 102	101 ± 0.2 102 ± 0.2	0.15 0.16
Badamli	50 100	49 101	98 ± 0.2 101 ± 0.2	0.22 0.18
Tap water (BSU)	50 100	55 103	105 ± 0.2 103 ± 0.2	0.25 0.28

[a]Average of five analysis of each sample
[b]The measure of precision is the standard deviation (s).

4. Conclusion

It is approach and alternative standard method for silver(I). In the present work a simple sensitive, selective non-extractive and inexpensive method with silver - DTFAB system was developed for the determination of silver(I) in drink water samples for continuous monitoring to establish trace level of silver(I) in difficult sample matrices. The method also offers a very efficient procedure for speciation analysis. Therefore, this method will be successfully applied to the monitoring of trace amounts of silver(I) in drink water samples.

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