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SPECTROPHOTOMETRIC DETERMINATION OF CADMIUM USING THIAZOLYLAZO CHROMOGENIC REAGENTS IN THE PRESENCE OF TRITON X-100: APPLICATION IN ENVIRONMENTAL SAMPLES

Alaa S. Amin[a]

[a] Chemistry Department, Faculty of Science, Benha University, Benha, Egypt

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SPECTROMETRY

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Alaa S. Amin

Chemistry Department, Faculty of Science,
Benha University, Benha, Egypt

ABSTRACT

A highly sensitive, selective, and accurate spectrophotometric method for the determination of cadmium using some thiazolylazo (I-V) chromogenic reagents has been developed. In the presence of Triton X-100 and NH_3/NH_4Cl buffer solutions, the optimum conditions for complexation, stoichiometric ratio, and stability constants are studied. Beer's law is obeyed up to at least 4.4 μg/10 ml of cadmium. A limit of detection and quantifications of 0.96 and 3.3 ng ml^{-1} have been estimated and a coefficient of variation of 1.9% at the 3.0 μg level was found. The molar absorptivity and Sandell sensitivity for each reagent are calculated. The only species that interfere seriously are Hg^{2+} and Ni^{2+}. The method has been applied to determine cadmium in water samples, and plant materials, and compared statistically with the AAS method. Interferences were overcome by using mixed

masking agents and preliminary extraction into Aliquat 366/carbon tetrachloride.

Key Words: Thiazolylazo compounds; Cadmium determination; Surfactants; Environmental analysis; Spectrophotometry

INTRODUCTION

Cadmium is one of the most hazardous of elements to human health. For this reason, years of effort have been devoted to the development of more effective, fast, precise, and accurate approaches to the determination of this element in biological and environmental materials using numerous classical and modern analytical methods. Cadmium in aqueous solution has been determined colorimetrically by extraction of its dithizonate complex for more than 60 years. The method has a low limit of detection but is not specific. Selectivity is achieved by stripping and re-extraction of the cadmium. More recently, a number of azolylazo-dyes have been prepared and investigated for the determination of cadmium (1–4). These include bromobenzothiazolylazonaphthol (BrBTAN) (5) and 6-bromobenzothiazolyl (2-azo-2)-4-methyl-phenol (BrBTAC) (6). The reagent 2-(2-benzothiazolylazo)-4,5-dimethylphenol (BTDMP) has been reported as a sensitive and highly selective reagent for cadmium ($\varepsilon = 5.2 \times 10^4$ l mol^{-1} cm^{-1} at 600 nm in o-xylene) (7). The most sensitive reagent for cadmium so far reported is 2-[2-(5-bromopyridyl) azo]-5-dimethylaminophenol (BrPADAP) ($\varepsilon = 1.4 \times 10^5$ l mol^{-1} cm^{-1} at 555 nm in methyl-1-butanol) (8), but it is less selective than BTDMP. The compounds 2-(2-benzothiazolyl-azo)4,6dimethylphenol (BTADMP) (9), 2-[2-(5-bromopyridyl) azo]4,5dimethylphenol (BrPDMP) (10), and 2-[2- benzothiazolylazo)-5-dimethylaminophenol (BTADAP) (10) have also been reported as reagents for cadmium determination. Hence, there is a need to obtain reagents that would be more sensitive and highly selective for the spectrophotometric determination of cadmium.

The present work has led to the development of precise, sensitive, and selective procedures for the determination of Cd^{2+} using some prepared thiazolylazo compounds (11,12). These have been examined as potential reagents for Cd^{2+} and compared with the parent compounds.

EXPERIMENTAL

Apparatus

Absorbance measurements were made with a Perkin-Elmer λ3B UV/Vis spectro-photometer in conjunction with a quartz cuvette of 10 mm path length.

The pH measurements of buffer solutions were carried out using an Orion research model 601A/digital ionalyzer. Conductance measurements were carried out with a YSI model 32 conductance meter of cell constant $k = 1.0$.

Reagents for Syntheses

The starting materials (Aldrich) were used without further purification. 2,3-(I), 1,4- (II), 1,5- (III) and 1,6-naphthalenediol (IV) and 3-hydroxy-2-naphthoic acid (V) were appropriately weighed and dissolved in the least amount of methanol and pyridine and cooled at $0-2°C$. Separate five weights of 2-aminobenzothiazole were dissolved in 10 ml formic acid and 20 ml of concentrated sulphuric acid. To each solution, the appropriate weight of sodium nitrite was dissolved in 30 ml and cooled at $0-2°C$. The diazotization, coupling, and recrystalization process in addition to elemental analysis were made according to the previously recommended method (11,12).

A stock 3×10^{-3} M solution of reagents (I-V) was prepared by dissolving an appropriate weight of the reagent in the minimum amount of pure ethanol and brought to 100 ml in a calibrated flask with the same solvent.

Ammonia–ammonium chloride buffer solutions of pH 8.0–11.5 were prepared as recommended previously (13).

Standard Cadmium Solution

A stock solution (100 μg/ml) was prepared by dissolving hydrated cadmium sulphate in water, with dropwise addition of 2.0 M sulphuric acid until the solution became clear, and standardized by EDTA titration at pH 5.0 using xylenol orange as indicator. Dilute solutions (1, 10 and 25 μg ml^{-1}) were freshly prepared each day from a 100 μg ml^{-1} solution prepared each week from the stock solution.

General Procedure

To 10-ml calibrated flasks containing 0.03–4.4 μg of cadmium(II) were added 2.0 ml of 3×10^{-3} M reagent solution, 1.0 ml ethanol, 4.0 ml of the optimum pH value as recorded in Table 1, and 1.0 ml of 3.0% (v/v) Triton X-100. The volume was then made up to the mark with doubly distilled water. The solution was thoroughly mixed and the mixture allowed to stand for 3.0 min. The absorbance was measured at 616, 653, 670, 648, and 635 nm using reagent I, II, III, IV, and V, respectively, against a reference blank solution prepared in the same manner.

Table 1. Spectral Characteristics of Cd^{2+} Complexes of I-V Reagents

	Reagent				
Parameter	I	II	III	IV	V
pH	9.0	9.5	9.0	10.0	9.5
λ_{max}/ nm	616	653	670	648	635
Stability/h	30	24	24	36	24
Stoichiometric ratio (M:L)	1:2	1:2	1:2	1:2	3:2
Logarithmic stability constant/pK	8.65	9.28	8.90	9.77	9.45
Beer's range/ $\mu g\ ml^{-1}$	0.003–0.40	0.004–0.36	0.004–0.42	0.004–0.38	0.003–0.44
Ringbom range/ $\mu g\ ml^{-1}$	0.004–0.35	0.005–0.32	0.004–0.38	0.005–0.33	0.004–0.40
Molar absorptivity/ $10^5\ 1\ mol^{-1}\ cm^{-1}$	2.14	2.25	2.09	2.34	2.45
Sandell sensitivity/ $ng\ cm^{-2}$	0.53	0.50	0.54	0.48	0.46
Regression equation[a]					
Slope (b)/ A/ $\mu g\ ml^{-1}$	1.90	2.00	1.86	2.08	2.18
Intercept (a)	−0.005	+0.008	+0.01	−0.003	−0.007
Correlation coefficient (r)	0.9990	0.9992	0.9988	0.9994	0.9996
Standard deviation of blank	0.0027	0.0031	0.0033	0.003	0.0024
Detection limit/ng ml^{-1}	1.08	1.24	1.32	1.20	0.96
Determination limit/ng ml^{-1}	3.65	4.15	4.44	4.05	3.30
Relative standard deviation/%	1.70	1.90	1.50	1.20	1.05

[a] $A = a + bC$ (where C is the concentration in /$\mu g\ ml^{-1}$)

The calibration graph was prepared by the same procedure and was linear, passing through the origin.

Determination of Cadmium in Environmental Samples

In aqueous solutions containing sufficient chloride, cadmium exists as the anionic complex $CdCl_4^{2-}$, which can be separated and concentrated with a liquid anion-exchanger. For convenience, a chloride concentration approximating that in seawater ($\cong 0.5$ M) was selected for use. Cadmium solutions (1.0–2.0 ppb [$\mu g/L$]) in 0.5 M sodium chloride were prepared, and 1000-ml portions of these were

extracted with two 5.0-ml portions of 0.1 M Aliquat 336 in carbontetrachloride, as described elsewhere (14). The cadmium was stripped from the combined extracts with 5.0 ml of 0.4 M perchloric acid, and determined with reagents (I-V) as described above. Cadmium was also determined in some natural water samples adjusted to a 0.5-M concentration of sodium chloride. The samples were also analyzed after pre-concentration by flame atomic absorption spectrometry (AAS).

Plant materials (5.0 g) were first mineralized with nitric and hydrochloric acids, according to the procedure of Mullin and Riley (15), modified by omission of the perchloric acid. The resulting solution was adjusted to 50-ml volume with 1.0 M HCl. The Cd^{2+} was extracted, stripped, and determined with reagents (I-V) as described in the above general procedure, and compared with that obtained by AAS.

RESULTS AND DISCUSSION

Absorption Spectra

The absorption spectra of 1-(2-benzothiazolylazo) 2,3-naphthalenediol (I), 8-(2-benzothiazolylazo)1,4-naphthalenediol (II), 8-(2-benzothiazolylazo)1,5-naphthalenediol (III), 8-(2-benzothiazolylazo)1,6-naphthalenediol (IV) and 1-(2-benzothiazolylazo)2-hydroxy–3-naphth-oic acid (V) and their complex with cadmium are shown in Figure 1. It is evident that the maximum absorption of the free reagents are at 491, 560, 566, 533, and 527 nm, while that of the complex is at 616, 653, 670, 648, and 635 nm using reagents I-V, respectively.

Choice of Surfactant and Surfactant Concentration

All reagents are barely soluble in pH 9.5 buffer solution and therefore cadmium does not form a colored complex with these reagents. However, in the presence of a non-ionic surfactant a red complex was formed instanta neously. Anionic and cationic surfactants have no effect, an amphoteric surfactant has some effect, but a non-ionic surfactant is most effective. Among those non-ionic surfactants used are Tween-80, Tween-60, emulgent-OP, Triton X-100, etc.; Triton X-100 was used in subsequent experiments, since they are highly concordant.

The effect of Triton X-100 concentration on the formation of the complex was studied and the results showed that in 10 ml of test solution containing more than 0.5 ml of 3.0% Triton X-100, a colored complex was formed instantaneously and maximum color formation with constant absorbance was attained when 3.0% of Triton X-100 was added in volumes of 0.8–1.3 ml. Therefore, 1.0 ml of Triton X-100 solution (3.0%) was the preferred additive.
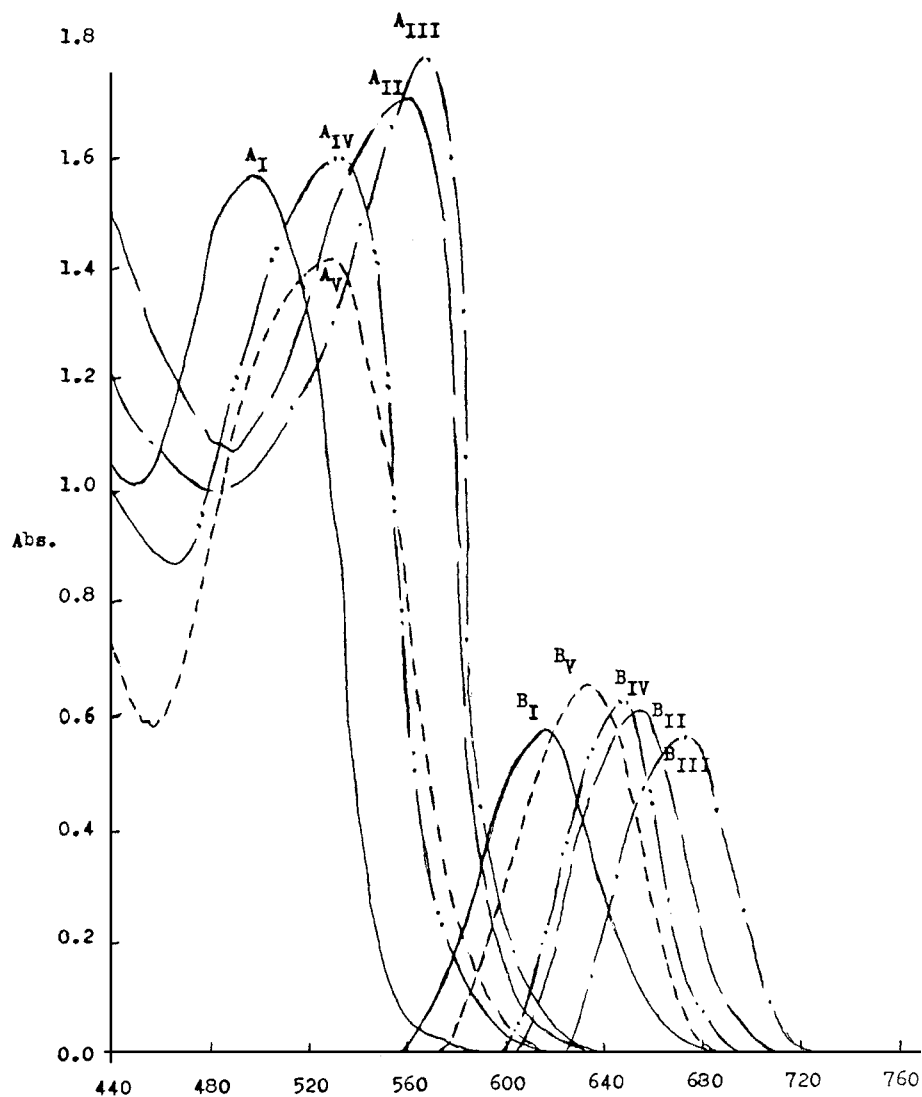
Figure 1. Absorption spectra of A-6 × 10⁻⁴ M regent in optimum pH value for I, II, −III, IV and V, and B-their complexes 3.0 μg per 10 ml Cd²⁺ solution.

Matrix Conditions

Buffer solutions of ammonia–ammonium chloride were used as the best buffer media and their pH values were measured with a pH meter. The maximum intensity of the Cd^{2+}-reagent complexes was achieved over the pH range 8.5–10.5. Therefore, all subsequent studies were carried out at pH 9.0, 9.5, 9.0, 10.0, and 9.5 using reagents I, II, III, IV, and V, respectively, since the results are highly concordant at these pH values. Moreover, with a constant pH for each complex, the amount of ammonia–ammonium chloride buffer solution added was varied from 0.5 to 5.0 ml and the optimum was found to be 3.0–5.0 ml. Therefore, an addition of 4.0 ml was chosen for subsequent work.

Trails were made to increase the sensitivity of the proposed methods. Increasing ethanol ratio increased the absorbance until 25%. With a lot of ethanol, ratio up to 40% (v/v), no change in the absorbance values was observed, while more than 40% (v/v) decreased the sensitivity of the procedures (Fig. 2). Therefore, 30% (v/v) ethanol was used for all further studies.

The absorbance value at the optimum pH value (Tab. 1) of a test solution containing 3.0 μg of Cd^{2+} per 10 ml and increasing amount of 3×10^{-3} M reagent (I-V) solution showed that the absorbance remained constant after the addition of
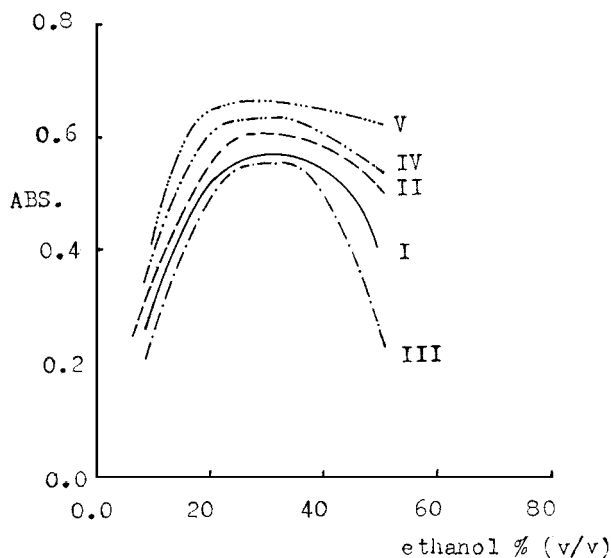
Figure 2. Effect of ethanol ratio on the absorbance of 3.0 μg/10ml Cd^{2+}-complexed with reagents (I-V).
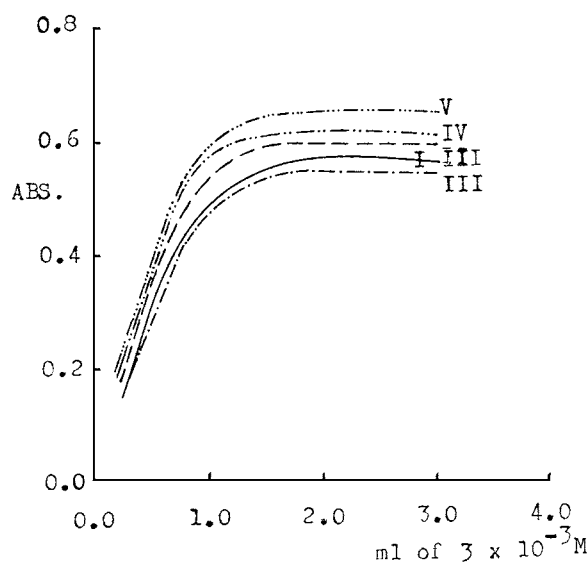
Figure 3. Effect of reagent concentration on the complex formed with 3 μg/10 ml of Cd^{2+}.

1.5–2.5 ml of the reagent (Fig. 3). Hence, 2.0 ml of the reagent was used in subsequent studies.

Order of Reagents with Stability

The effect of the order of adding reagents on the absorbance and stability of the complex was investigated and the results showed that the absorbance and stability were unaffected. Hence, any order can be chosen in which to add reagents.

In the presence of Triton X-100, the formation of the colored complex of Cd^{2+} with reagents (I-V) is instantaneous and the absorbance of the complex remains virtually constant for no less than 24 h.

Composition of the Complex

The molar ratio, continuous variation, and equilibrium shifting methods determined the composition of the complex. A ratio of Cd^{2+} to reagent was found to be 1:2 (Cd^{2+}-R) for all reagent complexes except for reagent V, which forms 3:2 (Cd^{2+}:V). These results were confirmed from conductometric titration of Cd^{2+} with reagents (I-V). The logarithmic stability constants calculated from the data

obtained using the molar ratio and continuous variation method applying Harvey and Manning equation are recorded in Table 1.

Adherence to Beer's Law and Sensitivity

A series of standard cadmium solutions were prepared and the absorbance of each was measured at the λ_{max} corresponding to each complex (Tab. 1) in 10-mm

Table 2. Comparison of the Sensitivity of the Methods for the Spectrophotometric Determination of Cadmium

Reagent	Conditions	λ_{max}	Range μg/10 ml	ε L mol^{-1} cm^{-1}	Ref
5-Cl-DMPAP	pH 8.8–10.7 in ethanol	550	0.0–10	1.02×10^5	17
ADAAS	NH$_3$, NH$_4$Cl, Triton X-100	532	0.0–4.8	2.02×10^5	18
CCDAA	pH 9.8, Na$_2$B$_4$O$_7$-NaOH, Triton X-100	485	0.0–4.0	1.24×10^5	19
DAPAAB	pH 12, Na$_2$B$_4$O$_7$- NaOH, Tween-80	520	0.0–5.0	1.20×10^5	20
MBADAB	pH 12, Na$_2$B$_4$O$_7$- NaOH, Tween-80	505	0.0–4.0	1.20×10^5	21
HDAA	pH 10, Na$_2$B$_4$O$_7$, Triton X-100	520	0.0–4.0	1.97×10^5	22
BTADAP	Pot. Sod. Tartarate-NaOH, Xylene	600	0.0–16	4.9×10^4	10
I	pH 9.0, NH$_3$, NH$_4$Cl, Triton X-100	616	0.03–4.0	2.14×10^5	This work
II	pH 9.5, NH$_3$, NH$_4$Cl, Triton X-100	653	0.04–3.6	2.25×10^5	This work
III	pH 9.0, NH$_3$, NH$_4$Cl, Triton X-100	670	0.34–4.2	2.09×10^5	This work
IV	pH 10.0, NH$_3$, NH$_4$Cl, Triton X-100	648	0.04–3.8	2.34×10^5	This work
V	pH 9.5, NH$_3$, NH$_4$Cl, Triton X-100	635	0.03–4.4	2.45×10^5	This work

5-Cl-DMAP: 2-[2-(5-chloropyridyl)azo]-5-dimethylaminophenol.
ADAAS: p-Azobenzenediazoaminoazobenzene sulphonic acid.
CCDAA: 2-Chlorocarboxylbenzenediazoaminoazobenzene.
DAPAAB: 4,10-Diazoaminophenylarsonic acid azobenzene.
MBADAB: p-Methylbenzeneaminodiazoazobenzene.
HDAA: *O*-Hydroxybenzenediazoaminoazobenzene.
BTADAP: 2-[2-benzothiazolylazo]-5-dimethylaminobenzene.

Table 3.   Tolerance Limits for the Determination of 3.0 μg of Cd^{2+}

Species	Amount of tolerated μg				
	I	II	III	IV	V
$B_4O_7^{2-}$, NH_4^+, CO_3^{2-}	8000	10000	10000	12000	9000
HC_3^-, Cl^-, F^-, I^-	6000	7500	7500	10000	8000
NH_2OH HCl, Br^-	5000	6000	6000	7500	6000
$S_2O_3^{2-}$, $S_2O_8^{2-}$, SO_4^{2-}	3750	4000	4000	5000	4250
Succinate, acetate	3000	3500	3250	3750	3500
Oxalate, benzoate,	2300	2500	2250	3000	2750
Tartrate, nitrate	1800	2000	1900	2250	2000
Phosphate, Thiourea	1200	1400	1250	1500	1300
Citrate, urea	800	900	750	1000	900
CN^-, EDTA	25	25	30	40	30
Na^+, Li^+, K^+	7000	7500	8000	9000	8500
Ca^{2+}, Sr^{2+}, Mg^{2+}	6000	6000	7000	7500	7000
Ba^{2+}, Pb^{2+}, Pd^{2+}	4000	4250	4500	5000	4500
Ge^{4+}, Zr^{4+}, Ti^{4+}	2500	2500	2750	3000	2750
Cr^{6+}, W^{6+}, Mo^{6+}	1500	1500	2000	2000	1600
Th^{4+}, UO_2^{2+}, Te^{4+}	800	850	900	1000	900
Mn^{2+}, Co^{2+}, Cu^{2+}	300	300	250	400	350
Al^{3+}, Fe^{2+}, Fe^{3+}	180	200	200	250	250
Zn^{2+}, Bi^{2+}, Sn^{2+}	150	160	175	175	175
Ni^{2+a}, Hg^{2+a}	25	30	25	30	30

[a] Amount of tolerated by adding masking agent.

quartz cells against a reagent blank prepared in the same manner without Cd^{2+} ions. Beer's law was valid over the concentration range 0.03–4.4 μg of cadmium per 10 ml of final solution. For more accurate analysis, Ringbom optimum concentration range was obtained by applying the transmittance percent against the logarithmic of concentration in μg ml^{-1} (Tab. 1). The apparent molar absorptivity and Sandell sensitivity (based on an absorbance of 0.001) of the complexes were calculated and recorded in Table 1. The standard deviation of the absorbance measurements obtained from a series of 13 blank solutions for each reagent were calculated (Tab. 1). The limits of detection (K = 3) and of determination (K = 10) of the method were established according to the IUPAC definitions ($C_1 = KS_0/s$) where C_1 is the limit of detection, S_0 the standard error of blank determination, s is the slope of the standard curve, and K is the constant related to the confidence interval (16). The calculated values were recorded in Table 1. The relative standard deviations were obtained from a series of 10 standards, each containing 3.0 μg cadmium per 10 ml final solution.

Characteristics of the method are compared with those of similar published spectrophotometric for Cd^{2+} determination. Thiazolylazo compounds under consideration are a group of the most sensitive reagents for cadmium and the proposed method compared favorably with existing methods. The sensitivity of various reagents is compared in Table 2. Comparison shows also that the proposed method is simple, rapid, and selective. It does not require heating, standing time, or extraction with organic solvents.

Effect of Interfering Ions

The criterion of interference was an error of more than three standard deviations. Numerous cations and anions were examined by applying the method to a fixed amount of cadmium in the presence of increasing amounts of the ions being studied. A systematic study of interfering ions in the determination of 3.0 μg Cd^{2+} per 10 ml of final solution has been made and the results are recorded in Table 3. The most serious positive interferents are Ni^{2+} and Hg^{2+}, which compete with cadmium for the reagent. EDTA and cyanide caused some negative interference, which compete with the reagents for cadmium. The

Table 4. Spectrophotometric Determination of Cadmium in Water Samples by Pre Concentration with Aliquat–/–Carbontetrachloride and Complexation with Reagents (I-V) (Sample Volume; n.d.= Not Detectable)

Sample	Cd^{2+} added μg	Cd^{2+} Found[a] μg						t^b value	F^b test
		I	II	III	IV	V	AAS		
Distilled	1.50	1.60	1.58	1.61	1.59	1.62	1.61	1.27	2.66
	3.00	3.11	3.12	3.09	3.13	3.14	3.15	1.53	3.21
River	0.00	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	—	—
Nile	0.50	0.506	0.503	0.499	0.497	5.02	5.05	1.82	4.14
Midtrania	1.00	1.04	0.99	1.02	1.03	1.06	1.10	1.35	2.90
n sea	2.00	1.99	2.03	2.05	1.99	2.00	2.08	1.68	3.47
Benha tap	0.00	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	—	—
water	0.75	0.74	0.76	0.75	0.76	0.75	0.76	1.12	2.51
Waste	0.08	1.00	1.02	1.01	1.00	1.01	1.02	1.74	3.65
water	1.60	1.81	1.79	1.80	1.81	1.79	1.81	1.57	3.22

[a] Average of six determinations.
[b] t- and F-values for reagent V compared with AAS at five degrees of freedom and 95% confidence level. Theoretical values are 2.57 and 5.05, respectively.

Table 5. Spectrophotometric Determination of Cadmium in Some Plant Samples After Digestion with Nitric/Hydrochloric Acids and Separation by Means of Aliquat/Carbon Tetrachloride Extraction

Sample	Weight Taken, g	Cd^{2+} added μg	Cd^{2+} Found[a], μg					
			I	II	III	IV	V	AAS
Kale	3.5	0.00	1.37	1.36	1.35	1.38	1.36	1.35
	7.00	0.00	2.73	2.71	2.72	2.70	2.74	2.75
	3.00	1.00	2.14	2.15	2.13	2.16	2.15	2.17
	6.00	1.00	3.32	3.30	3.31	3.33	3.32	3.32
	t-test[b]		1.22	1.53	1.71	1.08	1.15	
	F-value[b]		2.50	3.22	3.65	2.27	2.44	
Parsni	1.80	0.00	1.27	1.26	1.28	1.28	1.27	1.26
	3.60	0.00	2.53	2.55	2.52	2.55	2.54	2.53
p Peel	2.50	1.00	2.76	2.78	2.77	2.75	2.77	2.76
	4.00	1.00	3.81	3.80	3.82	3.81	3.80	3.80
	t-test[b]		0.97	1.32	1.73	1.46	1.15	
	F-value[b]		2.08	2.81	3.40	3.04	2.35	
Lettuce	2.20	0.00	2.33	2.32	2.34	2.33	2.35	2.35
	4.40	0.00	4.65	4.66	4.67	4.65	4.67	4.70
	2.00	1.00	3.13	3.14	3.12	3.13	3.12	3.15
	4.00	1.00	5.23	5.25	5.24	5.24	5.25	5.20
	t-test[b]		1.56	1.74	1.13	1.05	1.20	
	F-value[b]		3.17	3.82	2.44	2.22	2.61	

[a] Average of six-determinations.
[b] Theoretical values for t- and F-tests at 95% confidence level and five degrees of freedom are $t = 2.57$ and $F = 5.05$.

reaction is relatively free from interference by Zn^{2+}, Pb^{2+}, Fe^{2+}, Mn^{2+}, Bi^{3+}, Al^{3+}, and Fe^{3+}. It was shown that cations and anions that interfere can be masked by adding 1.0% sodium fluoride–sulfocarbamide–sodium citrite solution.

Analytical Applications

The proposed method was used for determining cadmium in water samples. The results in Table 4 are sufficiently accurate and the recoveries of cadmium quantitative at the level examined in reasonable agreement with those obtained by AAS. The performance of the method was assessed by calculation of the t- and F-values compared with the AAS method. Mean values were obtained in a Student's t-and F-test and 95% confidence limits for five degrees of freedom, and

the results showed that the calculated t- and F-values did not exceed the theoretical values.

As a test of the proposed method, cadmium was determined in some plant materials. The results are listed in Table 5. The recovery of cadmium was 98.3–101.5% and R.S.D. $< 1.9\%$. Comparing the results obtained by the proposed method with those of the AAS using the t- and F-values, the calculated values did not exceed the corresponding theoretical values, indicating insignificant differences between the results.

CONCLUSION

A method has been developed for the spectrophotometric determination of Cd^{2+} with thiazolylazo compounds (I-V) as chromogenic reagents, and applied satisfactorily to the determination of Cd^{2+} in water and plant samples. The proposed method has several advantages to warrant its use:

1. The reagents are easily synthesized and purified, which constitutes an advantage over the group of azo compounds, type reagents.
2. An excess amount of reagent in the sample solution does not interfere, which is a considerable advantage over other groups of reagents.
3. The measurements can be carried out rapidly using Triton X-100 in aqueous buffer medium containing 30% (v/v) ethanol without heating, extraction, or pretreatment of sample.
4. Measurements of molar absorptivity and Sandell sensitivity prove it to be more sensitive than previous works (17–22).
5. The method is more selective, especially on using mixed masking agents, than other recommended methods (17–22).

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