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Talanta 64 (2004) 906–911

Talanta

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Study of the concentration and separation of cadmium with microcrystalline phenolphthalein modified by crystal violet

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Received 6 January 2004; received in revised form 22 March 2004; accepted 5 April 2004

Abstract

A new method for cadmium separation and concentration with microcrystalline phenolphthalein modified by crystal violet (CV) was developed in the paper. In the presence of potassium iodide (KI) and CV, cadmium are quantitatively absorbed on microcrystalline phenolphthalein in the pH range 1.0–6.0 as the forms of water-insoluble ion-associated complexes $(CdI_3^-)\cdot(CV^+)$ and $(CdI_4^{2-})\cdot(CV^+)_2$. Effect of different parameters such as phenolphthalein amount, stirring time, the concentration of CV and KI, various salts and metal ions was studied in detail. During the present study, a significant enhancement of the extraction of cadmium was observed. Cd(II) can be completely separated from Zn(II), Fe(II), Co(II), Ni(II), Mn(II), Cr(III) and Al(III) in this microcrystalline system and well concentrated without the interference of these metal ions at high level. The possible reactive mechanism of cadmium concentration has been discussed. Analytical results obtained by this new method were very gratifying.

Keywords: Microcrystalline phenolphthalein; Ion-associated complex; Crystal violet; Separation and concentration; Cadmium

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1. Introduction

Cadmium is a heavy metal that is toxic for humans, animals and plants, and is one of the widespread trace pollutants with a long biological half-life [1]. It is extremely dangerous for its easy absorption into blood and gives a rise to accumulation in essential organs in the body, especially in kidney and liver [2]. Long and continuous exposure to cadmium can cause biochemical and functional changes in some critical organs [3] leading to cancers [4,5], arterioselerosis, high blood pressure [6], etc. Therefore, the study of the separation and determination of trace cadmium has assumed great importance and significance. Online solvent extraction is generally one of the effective techniques for pre-concentration of trace cadmium from aqueous samples, but it suffers from several drawbacks such as complicated instrumentation, environmental pollution because of the leak of organic solvent and induction of blank value owing to the poor purity of organic solvent [7]. Membrane technique is commonly used to cadmium separation. However, the membrane processes generally present some inconveniences such as higher cost, long-term instability, and an insufficient selectivity for the separation [8]. Although considerable work has been performed for the liquid–solid extraction of cadmium with microcrystalline naphthalene [9–11], studies concerning the use of phenolphthalein as an extractant for cadmium extraction are not reported at present. Moreover, naphthalene is toxic, harmful and pollutes environment. Long exposure to naphthalene can cause adverse health effects [12,13]. The present work examines the possibility of using microcrystalline phthenolphthalein to extract Cd(II) from aqueous solution. The aim of the method is to enhance the concentration of Cd(II) at trace level in aqueous samples. Results show that Cd(II) is more efficiently extracted with microcrystalline phenolphthalein. Furthermore, a strong absorption between phenolphthalein and CV was observed during

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doi:10.1016/j.talanta.2004.04.027

the experiment. With comparison to the aforesaid methods, the developed method appears to be simple, non-toxic and effective for cadmium separation and concentration. It overcomes the difficulties described above.

2. Experimental

2.1. Reagents and instruments

A stock of standard solution of 0.10 M potassium iodide (KI) was prepared by dissolving 16.6 g (Tianjin Chemical Plant, Tianjin, China) in distilled water and diluting it to 1.0 L. A 1.0×10^{-3} M of aqueous solution of crystal violet (CV) (A.R. Peking Chemical Plant, Peking, China) was prepared by dissolving 0.409 g in 1.0 L of distilled water. A 1.0×10^{-3} M of 4-(2-pyridylaxo) resorcinol (PAR) (A.R. Shanghai Reagents Plant, Shanghai, China) solution were prepared by dissolving 0.215 g in ethanol. A 15% (m/v) solution of phenolphthalein (A.R. The British Drug Houses Ltd. B.D.H. Laboratory Chemicals Group Poole, England) was prepared in ethanol. A standard cadmium solution was prepared by dissolving 1.00 g high pure metal cadmium in small amount of 1:1 HCl and then diluted to 1000 mL with 1% (v/v) HCl in a standard flask. [14a] Buffer solution of pH 1.0–2.0 were prepared by mixing 48.50 and 11.90 mL 0.20 M hydrochloric acid and 25.00 and 88.10 mL 0.20 M potassium chloride into a 200 mL standard flask, respectively and diluted to the mark with deionized water, and 20.40 mL hydrochloric acid was prepared for pH 3.0 and diluted to the same volume. That of pH 4.0–6.0 were prepared from mixtures of 36.00, 141.0 and 196 mL 0.20 M sodium acetate and 164.0, 59.00 and 4.00 mL 0.20 M acetic acid. [14b] Other reagents were analytical grade.

A model 722 spectrophotometer (Xiamen Analytical Instrument Plant, Xiamen, China) was employed for photometric measurements. A Shanghai model pHs-2pH meter was used for pH measurements. Polarograms were recorded with a three-electrode polarographic analyzer (JP-20, Chengdu Instrument Plant, Chengdu, China).

3. Procedures

3.1. Test of separation conditions

An aliquot of solution (50 μg) was placed to a 50 mL beaker. A given amount of 0.10 M KI and 1.0×10^{-3} M CV solution were added to it and dilute to 10 mL with distilled water. The mixture was shaken well and allowed to stand for a few seconds. Then 0.30 mL of 15% solution of phenolphthalein in ethanol was added to it with continuous stirring. A 1.0 mL of supernatant liquid was taken to a 25 mL grad flask and dilute to the mark. The absorbency is measured at 495 nm (495 nm proved to be the maximum absorption wavelength of the complex by determining its absorbency at different wavelengths) against the reagent blank prepared in the same way. Then the extraction rate of Cd(II) (E/%) is calculated.

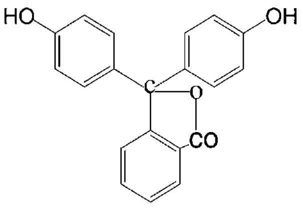
3.2. Test of Cd(II) pre-concentration

Four milliliters of 15% solution of phenolphthalein in ethanol was added to a 50 mL beaker containing 4.0 mL of 1.0×10^{-3} M CV. Dilute the mixture to 30 mL with distilled water. After stirring for 30 min at room temperature, the phenolphthalein well co-precipitated with CV, and the supernatant liquid was filtered out with G4 crucible. The residue was washed several times with a small volume of distilled water. Thus, the absorbent consisting of microcrystalline phenolphthalein and CV was remained in G4 crucible and covered with fiberglass for subsequent use.

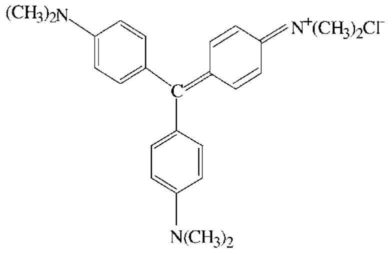
A given volume of sample solution was passed through G4 crucible with the absorbent inside at a flow rate of 2–3 mL min^{-1}. The metal complex anions CdI_3^- and CdI_4^{2-} in the solution reacted with CV^+ absorbed on microcrystalline phenolphthalein to form ion-associated complexes $(CdI_3^-)\cdot(CV^+)$ and $(CdI_4^{2-})\cdot(CV^+)_2$ which were quantitatively retained on microcrystalline phenolphthalein. Then the ion-associated complexes were dissolved with a small volume of hot 0.10 M HCl. A suitable amount of sodium nitrite ($NaNO_2$) was used to convert $CdI_3^-$and CdI_4^{2-} into Cd^{2+}. The excess of $NaNO_2$ was decomposed by carbamide. The pH was adjust to 3.0–5.0 with 0.10 M ammonia ($NH_3\cdot H_2O$). Filtered if needed, and diluted it to 10 mL with distilled water. Then take 5.0 mL of the solution and transfer it to the polargrapbic cell. After the addition of 3.0 mL of 10% (m/v) KI, 1.0 mL of 3% (m/v) Vitamin C (Vc) and 1.0 mL of 0.4% (m/v) Tween 80, the polarograms is record and the extraction rate of Cd(II) (E/%) is calculated.

3.3. Test of absorbent stability

In the present study, a stable absorption between phenolphthalein and CV was observed. The CV could hardly be disabsorbed from microcrystalline phenolphthalein after the absorbent produced according to the procedure above was boiled twice with 1.0–3.0 M HCl for 30 min. It is likely that, in the molecular structural perspective, there is a similarity in the structure between phenolphthalein and CV. As they both belong to the derivatives of thriphenylmehtahne, their similar conjugated system enable them to lap well over each other. Therefore, a strong inter-molecular action strength occurs leading to a considerable absorbability between them.



phenolphthalein



crystal violet

4. Results and discussion

4.1. Effect of CV amount on the extraction rate of Cd(II)

The effect of the amount of CV on the extraction rate of Cd(II) was studied in the system of 2.0 mL of 0.10 M KI, 50 μg of Cd(II), and stirring time 20 min with a total volume 10 mL (Fig. 1). Fig. 1 shows that there was no extraction of Cd(II) without CV, and an increase in the extraction rate of Cd(II) was observed with an increment in CV amount. Cd(II) was completely extracted in the presence of 1.50 mL of CV. Furthermore, the extraction rate of Cd(II) did not change with further increase in CV amount. Hence, the use of 2.0 mL of CV was recommended for a thorough extraction of Cd(II).

4.2. Effect of KI amount on the extraction rate of Cd(II)

Keeping other variables constant and varying KI amount, we studied the effect of KI amount on the extraction rate of Cd(II). No extraction of Cd(II) was found in the absence of KI, and the extraction was improved regularly with increasing amount of KI. When the volume of KI increased to 1.5 mL, the extraction rate of Cd(II) was 100%, and did not change with more KI used. Thus, 2.0 mL of KI was selected to ensure that Cd(II) is concentrated completely.

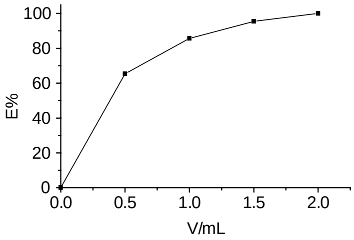
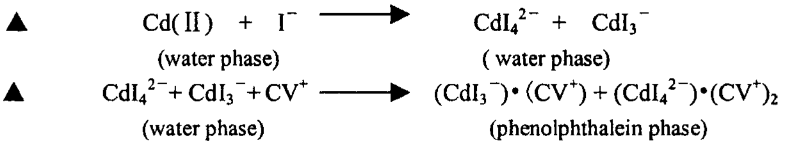


Fig. 1. Effect of CV amount on the concentration rate of Cd(II). Cd(II) 50 μg; KI (0.10 M) 2.0 mL; CV 1.0 × 10^{-3}M; total volume: 10 mL.

4.3. Reactive mechanism of extraction of Cd(II)

As known from the above experimental results, Cd(II) can not extracted at all when there is no CV or KI in the system. This is explained mostly for two reasons. Firstly, Cd(II) can combine with I^- into metal complex anion CdX_n^{2-n}, mainly into CdI_3^- and CdI_4^{2-} with the cumulative stabilization constant β_3, β_4 of $10^{4.34}$, $10^{5.32}$ respectively [15a]. Secondly, CV exists in aqueous solution in the form of cationic crystal violet (CV^+), which can associate with CdI_3^- and CdI_4^{2-} to form water-insoluble ion-associated complexes and be precipitated. In addition, according to the quantitative complex principle, if Cd(II) and I^- form a metal complex anion with a mole ratio of 1:1, the cumulative stabilization constant must be 10^8 or more. Moreover, for a larger mole ratio of the metal complex anion, the larger cumulative stabilization constant is required [16]. Since the stability of CdX_n^{2-n} depends on their cumulative stabilization constant in aqueous solution. Accordingly, it can be deduced that $CdI_3^-$and CdI_4^{2-} have lower stability. Under optimum experimental conditions, 50 μg Cd(II) (0.45 μmol) and 2.0 mL of 0.10 M KI (200 μmol) were used. Obviously, I^- is in much excess to Cd(II). This explains that the lower the stability of CdX_n^{2-n} is, the more I^- is needed, otherwise, Cd(II) can not be extracted quantitatively. Therefore, only in the simultaneous presence of KI and CV, can Cd(II) associate with them and form water-insoluble ion-associated complexes which can be absorbed on microcrystalline phenolphthalein.

From the aforementioned discuss, the precipitates retained on microcrystalline phenolphthalein should be ion-associated complexes $(CdI_3^-)\cdot(CV^+)$ and $(CdI_4^{2-})\cdot(CV^+)_2$. Thus, the following reactive mechanism has been considered.



4.4. Effect of phenolphthalein amount and stirring time on the extraction rate of Cd(II)

The phenolphthalein amount and stirring time play an important role in the thorough extraction of Cd(II). Under the selected experimental conditions, the effect of phenolphthalein amount and various stirring time on the extraction rate of Cd(II) were examined (Figs. 2 and 3). Fig. 2 shows the extraction rate of Cd(II) was approximately 76% while no phenolphthalein was used, and the extraction of Cd(II) was obviously enhanced with an increase in the volume of 15% phenolphthalein in ethanol. A volume of 0.30 mL made the extraction rate of Cd(II) reach 100%. Further increase in phenolphthalein amount had no effect on the extraction rate of Cd(II). It may be that after the addition of phenolphthalein to the reaction system, the formation of

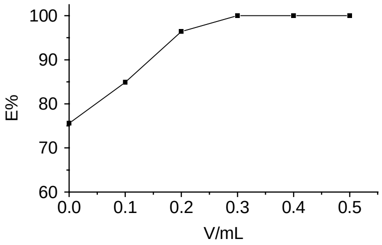


Fig. 2. Effect of phenolphthalein amount on the concentration rate of Cd(II) Cd(II): 50 μg; KI (0.10 M): 2.0 mL; CV (1.0×10^{-3} M): 2.0 mL; total volume: 10 mL.

microcrystalline phenolphthalein occurs. As a result, high concentration of cationic crystal violet (CV^+) was absorbed on microcrystalline phenolphthalein, which makes CV^+ in sufficient contact with CdI_3^- and CdI_4^{2-} hard to associate with CV^+ due to their low concentration in the reaction system. Thus, Cd(II) was extracted in phenolphthalein phase in the forms of $(CdI_3^-)\cdot(CV^+)$ and $(CdI_4^{2-})\cdot(CV^+)_2$. In order to ensure that Cd(II) was extracted completely as well as to save reagents, 0.30 mL of 15% phenolphthalein in ethanol is adequate to achieve a complete extraction of Cd(II). The same trend was observed in Fig. 3 on the effect of different stirring time upon the extraction rate of Cd(II). It is found that there is a distinct improvement in the extraction rate of Cd(II) with increasing stirring time. Over 15 min, the extraction rate kept 100% and unchanged with stirring time prolonged. In addition, the relation curve between the stirring time and the amount of CV^+ not absorbed on microcrystalline phenolphthalein in the system without Cd(II) was studied. The results were in good agreement with that presented in Fig. 2, which provides the information that the concentration of CV^+ retained on microcrystalline phenolphthalein increases when more phenolphthalein and CV were employed. Consequently, it is more useful to improve the concentration and separation of Cd(II). Twenty minutes were selected for further studies.

4.5. *Effect of various salts on the extraction rate of Cd(II)*

In order to check the effect of salts ($NaNO_3$, Na_2SO_4, $NaClO_4$, KCl, KSCN, and KBr) on the extraction rate of Cd(II), a concentration variation of these salts from 0.05 to 0.20 M was examined. As seen from Fig. 4, Na_2SO_4 exerted no influence on the extraction rate of Cd(II). Whereas, the effect of other salts differed and was presented as the following order $NaClO_4$ > KCl > $NaNO_3$ > KSCN > KBr > $(C_6H_5)_4BNa$.

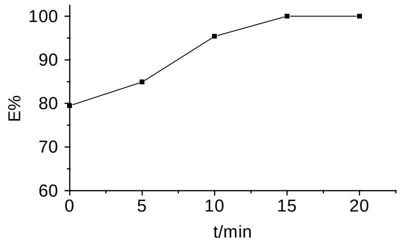


Fig. 3. Effect of stirring time on the concentration rate of Cd(II) Cd(II): 50 μg; KI (0.10 M): 2.0 mL; CV (1.0×10^{-3} M): 2.0 mL; total volume: 10 mL.

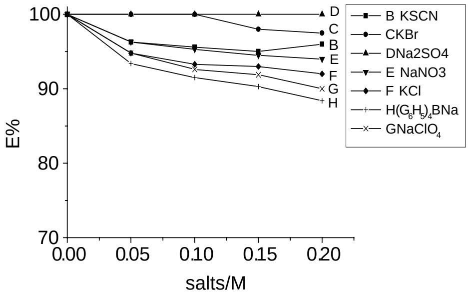


Fig. 4. Effect of various salts on the concentration rate of Cd(II) Cd(II): 50 μg; KI (0.10 M): 2.0 mL; CV (1.0×10^{-3} M): 2.0 mL; total volume: 10 mL.

It has been reported that univalent anions such as Cl^-, SCN^-, Br^-, ClO_4^- and NO_3^- can associate with CV^+ to form their corresponding ion-associated complexes [17], which, to some extent, leads to an decrease in CV^+ concentration expected to associate with CdI_3^- and CdI_4^{2-}. Moreover, the larger the amount of these univalent anions, the more severe the side reaction and the greater the decease in the extraction rate of Cd(II). It can be observed from the figure that the most remarkable effect on the extraction rate of Cd(II) was caused by ClO_4^-. It presumably is because that ClO_4^- belongs to large anions and has a stronger association ability with CV^+ than other univalent anions examined. Similar to ClO_4^-, SO_4^{2-} is also a relatively large anion. But its hydration radius is 4 Å larger than that of ClO_4^- (3.5 Å), which proves that SO_4^{2-} hardly associates with CV^+ because of its strong hydrophilic ability. This can be regarded as the reason that SO_4^{2-} did not influence the extraction rate of Cd(II). Furthermore, Cl^-, Br^- and SCN^- have the same hydration radius of 3 Å, which also proves that the smaller the hydration radius of anion is, the greater the decrease in the extraction rate of Cd(II) is. As for $(C_6H_5)_4B^-$, being a much larger anion than other anions examined, a highest effect on the cadmium extraction was caused. In the same way, Cl^-, Br^- and SCN^- can combine with Cd(II) into CdX_4^{2-}($X = Cl^-$, SCN^- and Br^-), and their cumulative stabilization constant are $10^{1.65}$, $10^{2.93}$, $10^{2.91}$, respectively [15b]. Accordingly, the sequence of anionic hydrophobic ability is $Br^- > SCN^- > Cl^-$ [18], which supports that the order of the hydrophobic ability of CdX_4^{2-}($X = Cl^-$, $SCN^-$and Br^-) $CdBr_4^{2-}$ > Cd $(SCN_4)^{2-}$ > $CdCl_4^{2-}$. It can be deduced that their ability to associate with CV^+ decreases one by one. In the presence of Cl^-, $SCN^-$and Br^-, (CdX_4^{2-}) $(CV^+)_2$ ($X = Cl^-$, $SCN^-$and Br^-) are formed and absorbed on microcrystalline phenolphthalein. Nevertheless, the effect of Cl^-on the extraction rate of Cd(II) was found to be greater than $SCN^-$and Br^-, which results from a weaker association

Table 1
Separation and determination results of binary mixed ions (pH 4.0)

Mixed ions	Added/μg		Metal ions found in water phase/μg		E (%)	
	Cd(II)	Me	Cd(II)	Me	Cd(II)	Me
Cd(II)–Zn(II)	50	50	0	49.83	100	0.34
Cd(II)–Fe(II)	50	50	0	51.27	100	−2.54
Cd(II)–Co(II)	50	50	0	51.08	100	−2.16
Cd(II)–Ni(II)	50	50	0	51.10	100	−2.20
Cd(II)–Mn(II)	50	50	0	49.55	100	1.10
Cd(II)–Al(III)	50	50	0	48.74	100	3.48
Cd(II)–Cr(III)	50	50	0	50.36	100	−0.72

Me refers the metal ions except Cd(II). Reaction conditions, KI(0.10 M): 2.0 mL; CV (1.0×10^{-3} M): 2.0 mL; total volume: 10 mL; stirring time: 20 min; phenolphthalein (15%): 0.30 mL.

Table 2
Determination results of Cd(II) in multiple ions mixtures (pH 4.0)

Initial amount of each co-ions(Fe(II), Ni(II), Co(II), Al(III), Mn(II), Zn(II), Cr(III)/mg mL^{-1}	The extraction rate of Cd(II)[a] (E/%)	R.S.D. (%)
0.4	99.7	1.4
1.0	99.0	1.1
2.0	98.8	0.9

Initial amount of Cd(II): 0.04 μg mL^{-1}; the volume of multiple ions mixtures: 250 mL.

[a] Mean for five determinations.

between $CdCl_4^{2-}$ and CV^+. For NO_3^-, its high effect on cadmium extraction could be explained that, apart from owing more isolated electron pairs, it suffers from less spatial block effect due to its unsymmetrical hybrid of three-dimensional structure, and there is no possibility of forming metal anion with Cd(II), which makes the formation of $CV^+{\cdot}NO_3$ easier. As a consequence, the concentration of CV^+ expected to react with CdI_3^- and $CdI_4^{2-}$is consumed resulting in an higher effect on the extraction rate of Cd(II) than SCN^- and Br^-. However, although Cd(II) extraction was effected by above salts, the degree of effect was so little that the extraction rate of Cd(II) was no less than 90%. It indicates that the microcrystalline system offers a fairly effective concentration behavior of Cd(II) at trace level.

4.6. Separation experiments

An investigation of the effect of pH on the extraction rate of different metal ions was conducted under experimental conditions. It is found that, in the range of pH 1.0–6.0, Cd(II) was thoroughly extracted while other common metal ions such as Zn(II), Fe(II), Co(II), Ni(II), Mn(II) and Cr(III) could not be extracted in the microcrystalline system. It is likely that these metal ions do not combine with I^- into metal complex ions so that no ion-associated complexes are formed. A small portion of extraction of Al(III) above pH 4.0 was observed. With regards to the phenomenon, an formation of aluminium hydroxide [$Al(OH)_3$] was taken into account due to hydrolyzation of Al(III). $Al(OH)_3$ an water-insoluble compound can also be absorbed on microcrystalline phenolphthalein. So Cd(II) can be separated from Al(III) by controlling pH 1.0–4.0.

At pH 4.0, the separation of Cd(II) from other common metal ions in binary ions mixtures and multiple ions mixtures were performed respectively. The measured data were summarized in Tables 1 and 2. (Note that the separation of Cd(II) in binary ions was performed according to separation procedure and that in multiple ions mixtures according to pre-concentration procedure). As seen from the measured data in Tables 1 and 2, a satisfactory separation of Cd(II) from Zn(II), Fe(II), Co(II), Ni(II), Mn(II), Cr(III) and Al(III) was achieved with microcrystalline phenolphthalein. And the concentration behavior of each metal ion existing individually is almost similar to that of metal ions in multiple ions mixtures. Upon that we can estimate the concentration behavior of metal ions in multiple ions mixtures according to that of individual ion.

4.7. Determination of cadmium in $NiSO_4{\cdot}7H_2O$

The proposed method was applied to the analysis of $NiSO_4{\cdot}7H_2O$ to evaluate its accuracy and applicability. Individually dissolve 0.50, 1.0 and 1.2 g to 100 mL with deionezed water in a standard flask. The pre-concentration and determination of cadmium in $NiSO_4{\cdot}7H_2O$ subjected to the recommended procedures were performed by standard addition method. The results in Table 3 show that

Table 3
Determination of cadmium in $NiSO_4{\cdot}7H_2O$

$NiSO_4{\cdot}7H_2O$ (g)	Found[a]/μg	Added/μg	Total amount[a]/μg	Recovery[a]/%	R.S.D./%
0.50[b]	1.62	5.0	6.64	101	1.2
1.0[b]	3.22	5.0	8.20	99.4	1.0
1.2[b]	3.90	5.0	8.90	100	0.8
0.50[c]	2.19	5.0	7.18	99.7	1.2
1.0[c]	4.35	5.0	9.31	99.1	1.1
1.2[c]	5.24	5.0	10.18	98.9	1.4

[a] Mean for five determinations.

[b] Refers to two different samples.

[c] Refers to two different samples.

high recovery, selectivity and freedom from matrix interference make this method suitable for the concentration and determination of cadmium in $NiSO_4 \cdot 7H_2O$.

5. Conclusion

Extraction of Cd(II) was carried out by liquid–solid extraction system using microcrystalline phenolphthalein as an extractant. Following conclusions may be drawn from the results in Tables that, the proposed method is accurate and reliable for the complete separation of Cd(II) from Zn(II), Fe(II), Co(II), Ni(II), Mn(II), Cr(III) and Al(III). What is more, Cd(II) can be well concentrated and separated without matrix interference from Ni(II) at high level. Therefore, it is fairly significant to establish such a method for trace cadmium separation and concentration in multiple ions mixtures and real samples.

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