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EXTRACTION OF NON-FERROUS METALS FROM AQUEOUS AMMONIUM SOLUTIONS

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Abstract

The work is devoted to the extraction of cadmium from ammonia aqueous solutions. The interaction of cadmium with 2-hydroxy-5-alkylbenzylethylenediamine in a wide pH range was studied. The composition of the extracted compounds was also determined. It has been shown that the state of cadmium in ammonia solutions depends on the pH value of the medium, as well as on the concentration of ammonium salt ions. It was found that cadmium in the reactions carried out is in amine forms – $[\text{Cd}(\text{NH}_3)_4]^{2+}$. In this case, the extractant coordinates with the cadmium ion through the oxygen atoms of the phenolic OH group and the amine nitrogen group. This is confirmed by the fact that cadmium interacts with the reagent as a result of the exchange of the proton of the phenolic OH group and coordination by molecules of the NH group can be provided by the results of an IR spectroscopic study of the reagent and the extracted complex in CCl_4 , taken in the range of $700\text{-}4000\text{ cm}^{-1}$, characteristic of the appearance of ν -vibrations of hydroxyl and NH bonds in the molecules of the extracted compound. The IR spectra of the reagent and the extracted cadmium compound show that the characteristic absorption band of the OH group in the phenol molecule at a wavelength of 3590 cm^{-1} sharply decreases, which indicates the replacement of the hydroxyl group proton with the extracted metal ion and the formation of a Cd-O bond. In addition, the absorption band of the NH bond at 1640 cm^{-1} is shift into the short-wavelength region of the spectrum in the complex, which also indicates the participation of the NH group in the formation of the complex. These analyzes helped establish that indeed the cadmium ion in complex compounds is hydrogen bonded to molecules and forms a tetrahedral geometry.

Keywords: liquid-phase extraction; extraction; extraction system; extract; re-extraction.

ВИЛУЧЕННЯ КОЛЬОРОВИХ МЕТАЛІВ ІЗ ВОДНИХ АМОНІЙНИХ РОЗЧИНІВ

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Анотація

Робота присвячена вилученню кадмію з аміачних водних розчинів. Досліджено взаємодію кадмію з 2-гідрокси-5-алкілбензилетилендіаміном у широкому інтервалі рН. Визначено склад екстрагованих сполук. Показано, що стан кадмію в аміачних розчинах залежить від значення рН середовища, а також від концентрації іонів амонійної солі. Встановлено, що кадмій в проведених реакціях знаходиться в аміачних формах – $[\text{Cd}(\text{NH}_3)_4]^{2+}$. У цьому випадку екстрагент координується з іоном кадмію через атоми кисню фенольної ОН-групи та аміної групи азоту. Підтвердженням взаємодії кадмію з реагентом в результаті обміну протоном фенольної групи ОН та координації молекулами групи NH, можуть слугувати результати ІЧ-спектроскопічного дослідження реагенту та екстрагованого комплексу в CCl_4 , зняті в області $700\text{-}4000\text{ cm}^{-1}$, характерні для появи ν -коливань гідроксильних та NH-зв'язків в молекулах екстрагованої сполуки. ІЧ-спектри реагенту та екстрагованої сполуки кадмію показують, що характерна смуга поглинання ОН-групи в молекулі фенолу за довжини хвилі 3590 cm^{-1} різко зменшується, що свідчить про заміщення протона гідроксильної групи на іон екстрагованого металу та утворення зв'язку Cd-O. Крім того, смуга поглинання NH-зв'язку за 1640 cm^{-1} в комплексі зміщується в короткохвильову область спектра, що також свідчить про участь NH-групи в утворенні комплексу. Ці аналізи допомогли встановити, що іон кадмію в комплексних сполуках дійсно зв'язаний водневим зв'язком з молекулами і утворює тетраедричну геометрію.

Ключові слова: рідиннофазна екстракція; екстракція; екстракційна система; екстракт; реекстракція.

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Introduction

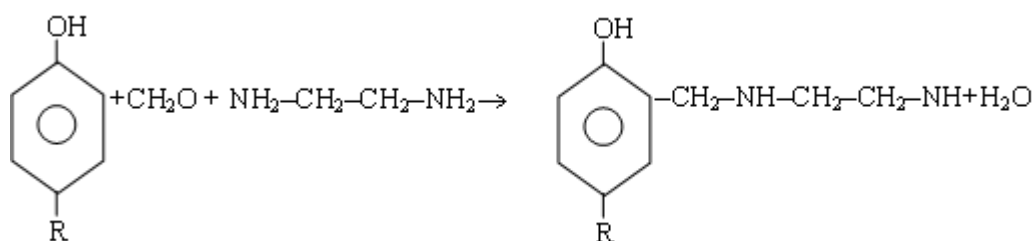
The 20th century was a century of rapid development of extraction. At this time, with the help of extraction, important scientific and technical problems of nuclear energy were solved, in particular: the issues of reprocessing nuclear fuel, purification and isolation of many radioactive elements. These advances have caused a significant increase in research on the theory and practice of extraction processes [1–10]. From this point of view, cadmium extraction is also of great interest. For this reason, in recent years, many extraction processes have also been carried out to extract cadmium from various solutions [11–18].

Cadmium is characterized by the formation of complexes, in particular ammonia complexes, containing from 1 to 6 ammonia molecules. Thus, its hydroxide $\text{Cd}(\text{OH})_2$ reacts with a solution of NH_3 to form the complex $[\text{Cd}(\text{NH}_3)_4]^{2+}$. Cadmium has unique properties and is considered a relatively rare element. Most often used when coating other metals to protect against rust formation, but can also be used in other areas of activity; production of solar panels, semiconductors, stabilizers for PVC, phosphors.

Alloys with cadmium are ductile and wear resistant. They are used to make wires for power lines, bearings for stuffing sea and air liners. Alloys are used for soldering glass, metal, and in fire extinguishers. Good thermal conductivity at extremely low temperatures has made the metal indispensable in cryotechnology. The beneficial property of the substance to accumulate by cancer cells is used in antineoplastic therapy.

In this work, the extraction of cadmium from ammonia solutions using the extractant 2-hydroxy-5-alkyl (C_8 – C_9) benzylethylenediamine in hexane was studied. The state of 2-hydroxy-5-alkyl (C_8 – C_9) benzylethylenediamine in hexane and the resulting complex was previously studied using cryoscopy, chemical analysis and IR spectroscopy.

The extractant 2-hydroxy-5-alkyl (C_8 – C_9) benzylethylenediamine is of great interest for the



where R – is an alkyl radical (C_8 – C_9).

hydrometallurgy of nonferrous metals, in particular copper, cobalt and zinc. This extractant is capable of effectively extracting nonferrous metals from leaching solutions of various compositions of oxidized nickel ores, deadcobalt-containing catalysts, and ocean concretions.

Experimental part

Extraction was carried out by manually mixing the phases at a temperature of 21 ± 0.5 °C for 1–3 minutes in separating funnels with a volume ratio of aqueous (V_w) and organic (V_o) phases of 2 : 1 from an aqueous solution of cadmium (II) with a concentration of 10^{-3} mol/l. A 0.02 M hexane solution of 2-hydroxy-5-alkylbenzylethylenediamine was used as an extractant. The required pH values of the medium (~7 and 8) were created using a 1M NH_4OH solution. The pH of the aqueous solution was measured using an “EV-74” universal ion meter. The ammonia content in the aqueous phase was determined by titration with a 0.1 N HCl solution, and in extracts using the Kjeldahl method [19]. The cadmium content in aqueous phases was determined titrimetrically using complexone-III (EDTA) [20]. 2-hydroxy-5-alkyl (C_8 – C_9) benzylethylenediamine purified according to the method [21] was used in this work. Mineral salts and acids were used with chemical grade “reagent grade” or “analytical grade”, hexane – “high-purity grade”.

IR spectra of the extractant and extracts were carried out on an IR-Specord-72IR spectrophotometer in the range of 700–4000 cm^{-1} . Liquid samples and solutions of the reagent in CCl_4 were taken in cuvettes with windows made of KRS-5 glass with a thickness of 0.1–0.002 mm. Solid samples were pressed together with KBr according to the generally accepted procedure. Electronic spectra were recorded on a Specord-71 spectrophotometer.

2-hydroxy-5-alkyl (C_8 – C_9) benzylethylenediamine was synthesized by the Mannich reaction, respectively, from the industrial fraction of diamine and alkylphenol in the presence of formaldehyde:

The reaction occurs at a temperature of 75–80 °C without a catalyst. The process is monitored by the refractive indices of the final product and the reaction is completed at $n_D^{20} = 1.3210\text{--}1.3250$. The industrial product, in addition to the main substance, contains unreacted alkylphenol, formaldehyde, etc. Therefore, we performed additional purification of the reagent. For this purpose, the product is diluted in aliphatic hydrocarbons and treated with distilled water. The released reagent is dried at a temperature of 80–90 °C. The product thus obtained is a gray, odorless, solid mass with a molecular weight of 282. Solubility in water is very low – 2 mg/l. After additional purification, the industrial product is diluted in hexane or other aliphatic hydrocarbons.

Results and its discussion

When extracting cadmium, experiments were carried out with a 0.02 M hexane solution of 2-hydroxy-5-alkylbenzylethylenediamine in 100 ml separating funnels, in the ratio of organic ($V_{org.}$) and aqueous ($V_{aq.}$) phases $V_o: V_a = 1:2$. After 2 minutes of stirring and phase separation, the organic layer was separated from the aqueous layer, the pH of the medium was measured, and the equilibrium concentration of cadmium remaining in the aqueous phase was determined. By the difference between the phases, the content of metal extracted into the organic phase was calculated. The results obtained are shown in Fig. 1.

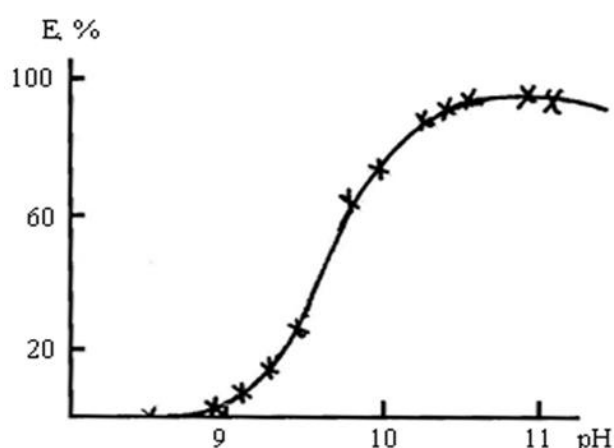


Fig. 1. Extraction of cadmium with a 0.02 M hexane solution of 2-hydroxy-5-alkyl benzylethylenediamine depending on the pH of the medium. $C_{Cd} = 0.85$ g/l; $V_{org}:V_{aq.}=1:2$; $\tau=3$ min

As can be seen from the figure, the extraction process begins at a much higher pH value of the environment (pH = 8). By increasing the pH of the

medium, the transition of cadmium into the organic phase increases and at pH = 10.6, extraction reaches a maximum. Further increase in pH values decreases cadmium extraction. In our opinion, this is due to the form of the compound under existing conditions. In the indicated pH range, the slope tangent ($tg\alpha$) of the dependence of the cadmium distribution coefficient on pH, $lgD\text{--}f(pH)$ is a straight line close to two (Fig. 2).

This type of curve proves that cadmium is extracted from ammonia solutions in the form of a doubly charged cation by separating an equivalent amount of hydrogen ions from the extractant molecule. In other words, the extraction process proceeds through the cation exchange mechanism with the formation of a complex compound in the form of $[Cd(NH_3)_4R_2 \cdot 2HR]$.

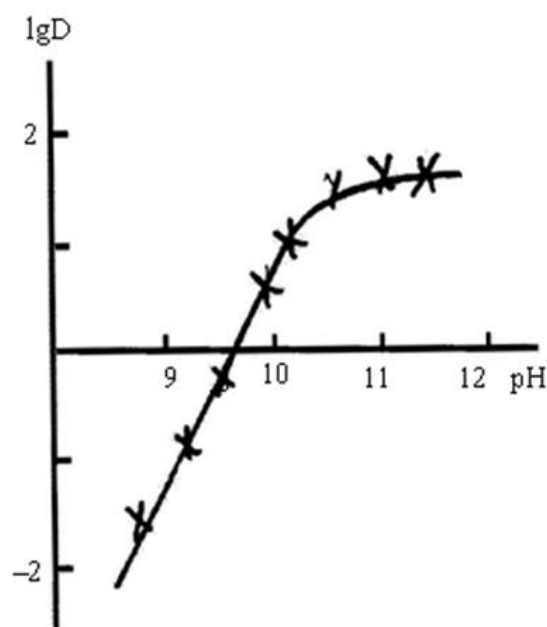


Fig. 2. Dependence of the logarithm of the cadmium distribution coefficient (lgD) on the pH of the aqueous solution

To determine the solvate number, the dependence of the cadmium distribution coefficient on the concentration of the extractant was studied. For this purpose, we extract cadmium under conditions of constant metal concentration and variable extractant concentration (0.01; 0.02; 0.04; 0.06; 0.08 M) at the same pH value. The results obtained are presented in Fig. 3. As can be seen from the presented data, the distribution coefficient of cadmium increases to 10.6 from the pH value of the studied reagent concentrations, the graphical dependence ($lgD\text{--}pH$) in this area for different concentrations of the extractant is parallel to

each other, the slope coefficient ($\text{tg}\alpha$) is constant and approximately equal to two.

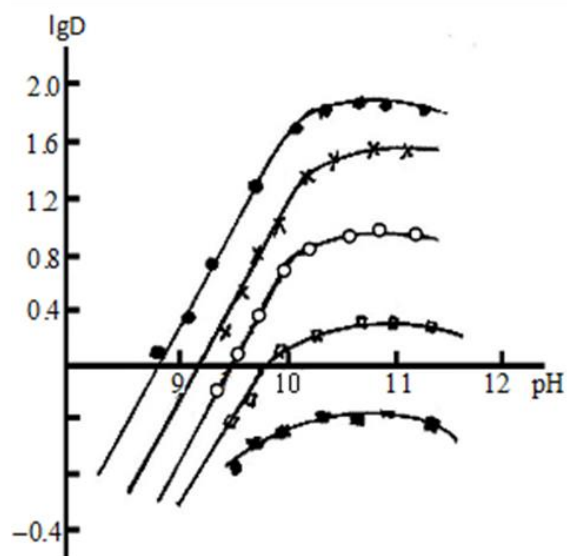


Fig. 3. Dependence of the logarithm of the cadmium distribution coefficient (lgD) on the pH of the aqueous medium during extraction with different extractant concentrations. ■ - 0.01M; □ - 0.02M; ○ - 0.04 M; x - 0.06M; ● - 0.08M.

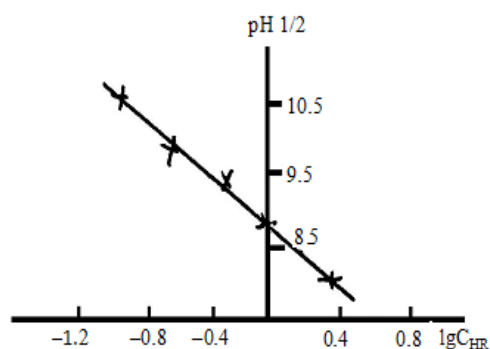


Fig. 4. Dependence of 1/2 cadmium extraction on the studied reagent concentration

This nature of the extraction curves indicates that, firstly, regardless of the concentration of the extractant in the system, cadmium passes into the organic phase in the form of a doubly charged cation, which is typical for extraction systems. Secondly, the extracted complex, regardless of the excess of the extractant, is in the same form, in other words, the composition of the compound of the resulting reagent with cadmium is constant and the Me:HR ratio is close to 1:2.

In this case, the slope angle ($\text{tg}\alpha$) of the dependence of $\frac{1}{2}$ cadmium extraction on the studied concentrations of 2-hydroxy-5-alkyl benzylethylenediamine is a straight line close to unity (Fig. 4.). This type of curve indirectly confirms the composition of the above complex.

Note that under identical conditions, iron is not extracted from a solution of 2-hydroxy-5-alkyl benzylethylenediamine. This difference in the extraction of the studied metals makes it possible to extract them from a solution containing iron, first with cadmium, and then by changing the pH of the medium separately.

To obtain additional data on the composition of the extracted compounds during the extraction of cadmium from ammonia solutions, the extract was subjected to chemical analysis for ammonia and metal content using the Kjeldahl method [19].

Chemical analysis of the organic phase obtained by extracting cadmium from saturated ammonium chloride solutions at different pH values showed that the ratio of ammonia and cadmium in the compound (Table 1) is $\text{NH}_3:\text{Cd}=4:1$, that is, cadmium is extracted during extraction in the form of ammonia complex- $[\text{Cd}(\text{NH}_3)_4]^{2+}$.

Table 1

Chemical analysis of cadmium complexes with a solution of 2-hydroxy-5-alkylbenzylethylenediamine at different pH values of the aqueous medium

pH equilibrium	Contents NH_3 and Cd in the complex, mol/l		Ammonia to cadmium ratio $\text{NH}_3:\text{Cd}$
	NH_3	Cd	
9.95	0.81	0.40	2:1
10.05	2.05	1.08	2:1
10.16	4.63	2.21	2.1:1
10.50	7.50	2.40	3.1:1
10.65	5.18	1.36	4.3:1
10.80	5.20	1.25	4.16:1

In order to clarify the state of the 2-hydroxy-5-alkylbenzylethylenediamine extractant in the

organic phase, its molecular weight was determined by cryoscopy [22] (Table 2).

Table 2

Determination of the molecular weight of 2-hydroxy-5-alkylbenzylethylenediamine by cryoscopy ($\Delta t_{\text{benz}}^0 = 0.55^\circ\text{C}$)

Reagent concentration, M.	Reagent mass, g.	Volume of benzene, ml.	Mass of benzene, g.	Depression Δt^0	Molecular mass of the reagent	
					Theoretical calculation	Defined
0.0012	0.0066	25	21.975	0.003	282	512.12

Continuation of the table 2						
0.0025	0.1320	25	21.975	0.006	282	519.84
0.0050	0.0264	25	21.975	0.012	282	519.85
0.0100	0.0528	25	21.975	0.022	282	563.12
0.0500	0.2640	25	21.975	0.110	282	563.20
0.1000	0.5280	25	21.975	0.220	282	563.19
0.2000	0.0560	25	21.975	0.440	282	563.19

The table data shows that in the experiments performed, the reagent in all concentrations was present in the organic phase in a dimerized form.

At the same time, the molecular mass of the extracted complex was determined. The results obtained are shown in Table 3.

Table 3

Determination of the molecular mass of the cadmium complex with a solution of 2-hydroxy-5-alkylbenzylethylenediamine ($\Delta t_{benz}^0 = 0.55^\circ\text{C}$)

Reagent concentration, M	Mass of the complex, g	Volume of benzene, ml.	Mass of benzene, g	Depression Δt^0	Molecular mass of the reagent	
					Theoretical calculation	Defined
0.0025	0.160	25	21.975	0.052	744.40	718.1
0.0050	0.340	25	21.975	0.110	744.40	725.0
0.0100	0.510	25	21.975	0.158	744.40	752.3

As can be seen, the resulting complex with cadmium also confirms that under extraction conditions the reagent remains in a dimerized form.

To clarify the composition and structure of the extracted complex, i.e. to confirm the fact that cadmium is extracted as a result of the exchange

of the proton of the phenolic OH group and coordination with the NH group, the IR spectra of 2-hydroxy-5-alkyl($\text{C}_8\text{-C}_9$) benzylethylenediamine and the resulting complex in the region of 700–4000 cm^{-1} were studied on a Specord -72 IR device in a KBr cuvette 0.06 cm thick (Fig. 5).

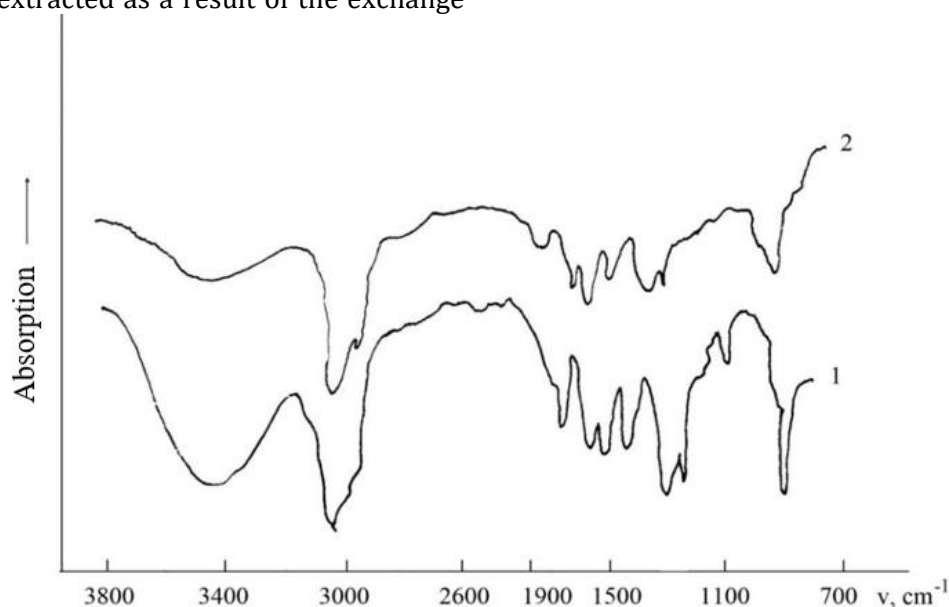


Fig.5. IR spectra of the extractant (1) and the extracted complex (2) of cadmium

As can be seen from the IR spectrum data of the extractant and cadmium extract (Fig. 5), the intensity of the absorption band of the OH group in the phenol molecule at 3590 cm^{-1} sharply decreases in cadmium extracts, which indicates the replacement of the hydroxyl group proton with the extracted cadmium ion and the formation it contains Cd-O bonds [23]. This also confirms the shift in the absorption spectrum of the C-O bond from 1245 cm^{-1} to 1310 cm^{-1} due to the formation of the Me-O valence bond. This is also evidenced by the electronic spectra (Fig. 6).

As can be seen, two bands are observed in the UV region of the spectrum, which correspond to benzene absorption at 46000 and 36000 cm^{-1} for the extractant, 44000 and 32000 cm^{-1} for the extract [24]. As is clear from Fig. 6, during complex formation, a long-wave shift of absorption bands occurs, which is characteristic of the formation of metalphenolates.

In addition, the disappearance of the absorption band associated with deformation vibrations of the NH group in the region of 1640 cm^{-1} of the spectrum in the complex

indicates that the compound is ion-associated and at the same time shows the creation of a coordination bond between the cation and amino groups.

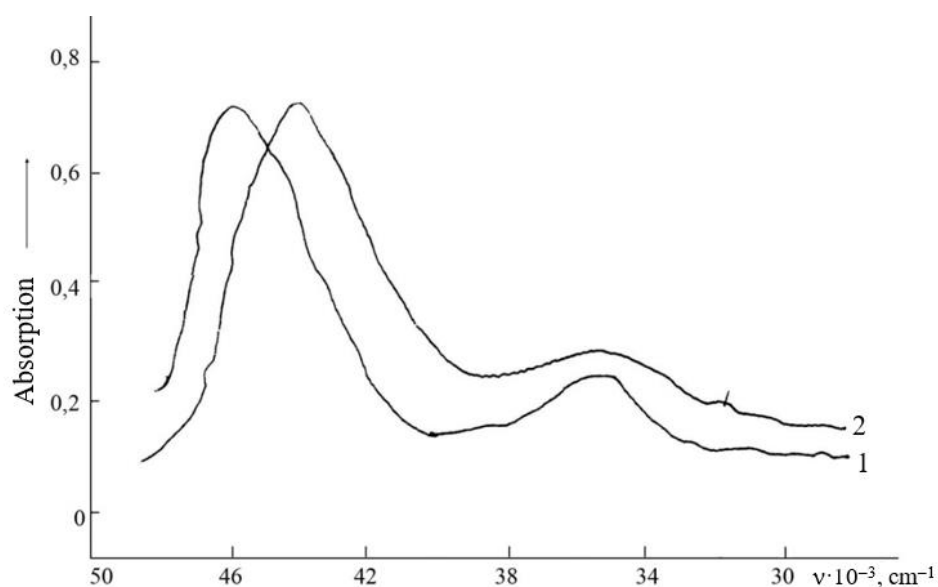
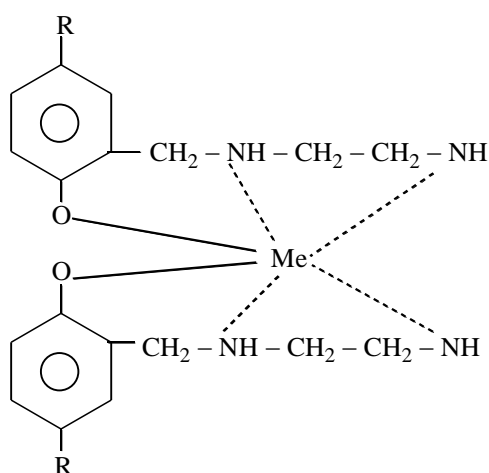


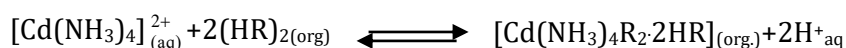
Fig.6. Electronic spectra of the extractant (1) and the extracted complex (2) of cadmium in the UV region

Based on the data obtained, the structure of the cadmium complex can be represented as follows:



Thus, based on chemical analysis data (Table 1), IR spectroscopy, electronic spectrum, extraction process curves, as well as the state of 2-hydroxy-5-alkylbenzylethylenediamine in the

organic phase, the extraction of cadmium from ammonia solutions can be written by the following equation:



with extraction constant: $K_{\text{ex}} = \frac{[\text{Cd}(\text{NH}_3)_4\text{R}_2\cdot 2\text{HR}]_{(\text{org})} [\text{H}^+]_{(\text{aq})}^2}{[\text{Cd}(\text{NH}_3)_4]_{(\text{aq})}^{2+} [(\text{HR})_2]_{(\text{org})}^2}$.

The reextraction of cadmium from the organic phase was also studied. It has been established that cadmium is easily re-extracted with weak solutions of mineral acids. For example, an extract containing 5 g/l cadmium, stirred for 5-

10 minutes with an equal volume of 0.3-0.4 N solution of sulfuric (or hydrochloric) acid completely passes into the aqueous phase (Table 4).

Re-extraction of cadmium from the organic phase depending on the mixing time of the phases and the concentration of mineral acid, $V_{org}:V_{aq}=1:1$

Cd ²⁺ g/l.	H ₂ SO ₄ solution 0,3n.	τ , min.	Reextraction from organic phases, g/l		Reextraction, %
			organic phase	Aqueousphase	
5.0	0.025	40	4.5	0.5	10.0
5.0	0.05	40	4.0	1.0	20.0
5.0	0.05	30	3.25	1.75	35.0
5.0	0.10	30	2.5	2.5	50.0
5.0	0.15	20	2.0	3.0	60.0
5.0	0.20	20	1.25	3.75	75.0
5.0	0.30	10	0.11	4.89	97.8
5.0	0.30	15	-	5.0	100.0
5.0	0.35	10	-	5.0	100.0

Conclusions

Thus, the results of laboratory studies on the extraction of cadmium from ammonia solutions using a hexane solution of 2-hydroxy-5-alkyl

(C₈-C₉) benzylethylenediamine showed that this reagent is an effective cadmium extractant and can be successfully used for the extraction of non-ferrous metals from ammonia solutions.

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