FLOTATION WITH IRON(III) HEXAMETHYLENEDITHIOCARBAMATE AS A PRECONCENTRATION METHOD FOR LEAD DETERMINATION USING ELECTROTHERMAL ATOMIC ABSORPTION SPECTROMETRY

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Hexamethyleneammonium hexamethylenedithiocarbamate (HMA-HMDTC) was used as a new flotation reagent for the preconcentration of lead. HMA-HMDTC added to the first precipitate collector of hydrated iron(III) oxide ($Fe_2O_3 \cdot xH_2O$) gives the second precipitate collector of iron(III) hexamethylenedithiocarbamate ($Fe(HMDTC)_3$). All important parameters (concentration of collectors, pH, type of tensides, ionic strength, etc.) necessary for the successful lead flotation with $Fe_2O_3 \cdot xH_2O$ and $Fe(HMDTC)_3$ were optimized. At selected pH (6.0) and ionic strength (0.02 mol/l), with the addition of sodium dodecylsulfate (NaDDS) as a foaming reagent, lead can be separated quantitatively with 10 mg Fe(III) and $3 \cdot 10^{-4}$ mol $HMDTC^-$ added to 1 liter of water sample. Zeeman electrothermal atomic absorption spectrometry (ZETAAS) was applied as the method for determining lead in tap and spring water samples. The results were compared with those obtained by inductively coupled plasma-atomic emission spectrometry (ICP-AES). The limit of detection for lead using this method is 0.06 μg/l.

Key words: lead; determination; colloid precipitate flotation; collectors; hydrated iron(III) oxide; iron(III) hexamethylenedithiocarbamate; water; electrothermal atomic absorption spectrometry

INTRODUCTION

There are several papers about the preconcentration of lead by colloid precipitate flotation using hydrated metal oxide as $Fe_2O_3 \cdot xH_2O$ [1–7], $Al_2O_3 \cdot xH_2O$ [8], $In_2O_3 \cdot xH_2O$ [9] or ion flotation [10]. Our experience has shown that the combination of hydrated iron(III) oxide $Fe_2O_3 \cdot xH_2O$ and $Fe(TMDTC)_3$ is very useful for the flotation of many metals (colligends) [11–14] as well for lead [15]. Here we present an attempt at applying HMA-HMDTC as a new dithiocarbamate reagent for the precipitate flotation of lead. HMA-HMDTC is a well-know reagent for extraction, coprecipitation or sorption .before atomic absorption spectrometry

(AAS) in determination of elements [16–25]. During the reaction between HMA-HMDTC and $Fe_2O_3 \cdot xH_2O$ the product $Fe(HMDTC)_3$ was obtained [26], and these collectors were successfully applied for the zinc flotation [27]. The scope of this work is to consider the possibility of its application for lead colloid precipitate flotation. $Fe(HMDTC)_3$ is an insoluble precipitate with significant hydrophobic characteristics that are the most important criterion for successful precipitate determining flotation. ZETAAS is used as a method for determining the analyte after its separation by flotation.

EXPERIMENTAL

Apparatus

A Zeeman atomic absorption spectrophotometer Varian 640Z was applied for measurements. A lead Varian hollow cathode lamp was used as a

primary source. Instrumental parameters for ZETAAS are shown in Table 1. All pH measurements were carried out with a digital Iskra pH-Meter MA 5705 using a combined glass electrode (Iskra Model 0101). The flotation cell used to carry out the pre-

concentration was a glass cylinder (4 x 105 cm) with a sintered glass disc (porosity No. 4) at the bottom to generate air bubbling. For the determination of the electrokinetic (ζ) potential of the collector particle surfaces a device analogous to Chaĭkovskiĭ's equipment was applied [28–30]. Inductively coupled plasma-atomic emission spectrometric measurements (ICP-AES) were performed by Varian spectrometer Model Liberty 110.

Table 1

Optimal instrumental parameters for ZETAAS determination of lead

Parameter	
Wavelength	283.3 nm
Spectral slit	0.5 nm
Lamp current	10 mA
Graphite furnace	
Drying	95 °C, 40 s; 120 °C, 10 s
Charring	400 °C, 8 s
Atomizing	2100 °C, 3 s
Cleaning	2300 °C

Reagents and standards

The reagents used for the preparations of solutions were of analytical-reagent grade except surfactants sodium dodecylsulfate (NaDDS), sodium oleate (NaOL), sodium palmitate (NaPL) and sodium stearate (NaST). All aqueous solutions were prepared in deionized redistilled water. By dissolving $Pb(NO_3)_2$ (Merck), lead stock solution (1 mg/ml) was prepared. Before each investigation lead standard solutions were freshly prepared by diluting this stock solution. The concentration of iron stock solution was prepared by dissolving high-purity iron metal (Merck) in conc. HNO_3 as 30 mg/ml Fe(III). After diluting this Fe(III) stock solution, a series of standards with the concentration of Fe ranging from 2.5 to 100 mg/l were obtained. The solution of HMA-HMDTC was made as 0.1 mol/l in 96 % ethanol. Tensides solutions (0.5 %) were obtained by dissolving appropriate amount of NaDDS or NaOL in 95 % ethanol and NaPL or NaST in 99.7 % 2-propanol. The pH of solutions was regulated by addition of HNO_3 solution (0.1 mol/l) or solu-

tions of KOH (2.5 % and 10 %). Ionic strength (I_c) was adjusted by addition of a saturated solution of KNO_3 (c = 2.78 mol/l). 0.1 mol/l solution of NH_4NO_3 served to transfer quantitatively the content of the beaker into the flotation cell.

Procedure recommended for the flotation step

The basic procedure was applied for uncontaminated water samples (spring and tap waters from neighborhoods in the cites Skopje and Kavadarci in Macedonia). The samples were collected in polyethylene containers (previously treated by conc. HNO_3, washed by deionized redistilled water and then with a portion of the water sample) without filtration. To prevent the possible hydrolytic precipitation of some mineral salts, a few milliliters of conc. HNO_3 was added to 1 liter of natural water. The pH had to be *ca.* 2.8–3.

An acidified water sample (1 liter) was placed in a 1 liter beaker. A combined glass electrode was immersed in the solution. After adding 6 ml of saturated KNO_3 solution, 1 ml of 10 mg/ml Fe(III) solution was added into the beaker. The pH of the solutions was from 2.6 to 2.7. The pH of the medium was carefully adjusted to 5.5 by the addition of 10 % and 2.5 % KOH. The yellow-brown precipitate of $Fe_2O_3 \cdot xH_2O$ was stirred 5 minutes (first induction time – τ_1). Then, 3 ml of HMA-HMDTC solution was added. The precipitate gradually changed color from yellow-brown to black – the color of $Fe(HMDTC)_3$. After the second induction time – τ_2 (15 min of stirring), 1 ml of NaDDS alcoholic solution was added and the content of the beaker was transferred quantitatively into the flotation cell with the small portion (3–4 ml) of 0.1 mol/l NH_4NO_3. Air (50 ml/min) bubbles appeared from the perforated bottom of the cell for 1–2 min. A stream of bubbles raised the precipitate flakes to the water surface. There a black scum formed and the water in the cell became completely clear and cleaned of the collector. Then the glass pipette-tube was immersed into the cell through the foam layer and the water phase was sucked off. A hot 65% HNO_3 solution (2.5 ml) was added to the cell to destroy the scum. When the black sublate together with the tenside foam had decomposed and dissolved, the liquor in the cell become clear yellow. The solution was sucked off by vacuum through the bottom of the cell and collected in a 25 ml volumetric flask. The cell and the pipette-tube were washed

with 4 mol/l HNO_3 solution. The flask was filled up to the mark with the 4 mol/l HNO_3 and the sample was ready for AAS measurements.

To obtain the optimal experimental parameters for lead (collector mass, medium pH, type of surfactant, ionic strength, induction time), a series of standard solutions of lead were treated by the flotation procedure and then were tested by AAS.

Because of the possibility of iron interferences on lead absorbance, the experimental data necessary for construction of the calibration curve were obtained by the proposed flotation method.

RESULTS AND DISCUSSION

Influence of iron mass on lead flotation efficiency

Since iron(III) is a constitutive element of the two collectors used, the investigation of its influence on lead flotation recoveries was performed. The effect of iron mass on lead coprecipitation by $Fe_2O_3 \cdot xH_2O$ was studied previously [7, 15]. The investigation of the influence of iron mass on lead incorporation in $Fe(HMDTC)_3$ particles was performed on a floating series working solutions (1 liter) containing 25 and 50 µg lead at constant pH (5.5) and ionic strength (0.02 mol/l). To each solution a different mass of iron (2.5–100 mg) was added, but the amount of the chelating reagent $HMDTC^-$ was kept constant ($2 \cdot 10^{-4}$ mol). The data of these investigations are presented in Table 2.

T a b l e 2

Lead flotation recovery dependence on iron mass,
coprecipitating with $Fe(HMDTC)_3$
(pH = 5.5, I_c = 0.02 mol/l, c($HMDTC^-$) = $2 \cdot 10^{-4}$ mol/l)

	R (%)	
γ (Fe) mg/l	γ (Pb)* 1 µg/ml	γ (Pb)* 2 µg/ml
2.5	84.6	72.2
5.0	85.7	74.5
10.0	88.5	91.5
20.0	94.8	97.1
30.0	99.0	97.7
40.0	100.0	97.8
60.0	100.0	97.9
80.0	100.0	98.0
100.0	100.0	98.5

*Concentration in the final solutions

Our previous results [7, 15] on lead flotation with $Fe_2O_3 \cdot xH_2O$, used as a single collector, have shown that lead floated partially and insufficiently at pH 5.5 with 10 mg/l (72.4–81.0 %) or 20 mg/l of iron (80.3–87.5 %). The satisfactory lead flotation recoveries – R (95.2–97.1 %) were obtained by addition of 60 mg of iron to 1 liter of solution.

The data of flotation investigations when 2 ml of 0.1 mol/l HMA-HMDTC were added (Table 2), show that lead coprecipitated satisfactorily (94.8–97.1 %) with 20 mg/l iron. This indicates that the contribution of $Fe(HMDTC)_3$, as an additional collector, is significant.

Comparing the influence of iron mass on lead flotation recoveries using the combination $Fe_2O_3 \cdot xH_2O / Fe(HMDTC)_3$ and the combination $Fe_2O_3 \cdot xH_2O / Fe(TMDTC)_3$ [15], at the same conditions (pH, amount of dithiocarbamate and I_c), it can be concluded that the same mass of iron is necessary for quantitative flotation of lead.

Determination of optimal pH range

The effect of medium pH on lead flotability (R) was studied at the different pH of solution (containing 25 and 50 µg/l of lead) at the constant I_c (0.02 mol/l), mass of iron (20 mg) and amount of $HMDTC^-$ ($2 \cdot 10^{-4}$ mol). The pH influence was investigated within the interval of 3.5 to 6.5 (Fig. 1). At pH higher than 6.5, the collector $Fe(HMDTC)_3$ begins to hydrolyze, that is evident since the precipitate gradually changes color from black to black-brown to brown – the color of $Fe_2O_3 \cdot xH_2O$.

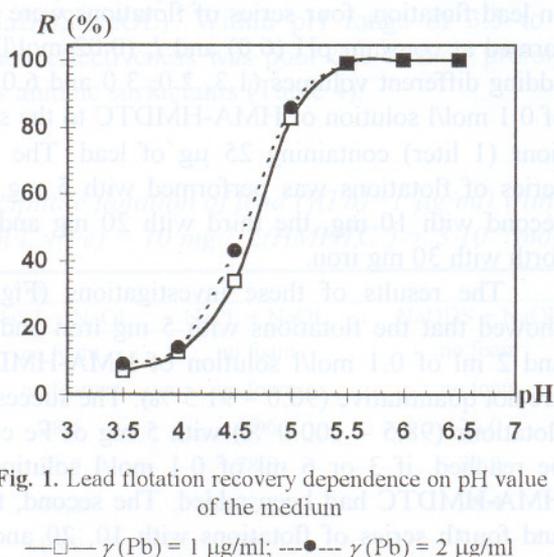**Fig. 1.** Lead flotation recovery dependence on pH value of the mediun

——□—— γ (Pb) = 1 µg/ml; ---●--- γ (Pb) = 2 µg/ml

The *R*/pH curves (Fig. 1) illustrate the significant effect of pH on lead recoveries (99.1–100 %) as a consequence of pH influence on NaDDS as compound. NaDDS is an anionic surfactant and at lower pH its protolysis occurs. Therefore, NaDDS is not able to perform its function as surfactant because it is not in the anionic form. Thus, lower pH values decrease NaDDS efficiency, it foams poorly and lead recoveries obtained within pH range of 3.5 to 4.5 are extremely poor. Within pH range of 4.5 to 5.5 the foaming is better but lead recoveries are still not quantitative (53.1–93.6 %). The best flotation is obtained within pH of 5.5–6.5 where lead recoveries are from 99.0–100.0%. Thus, the pH value 6 was chosen for the procedure. The *R*/pH curves show that different masses of lead had no appreciable effect on flotation efficiency.

Comparing the influence of pH on lead flotation recoveries using the combination of $Fe_2O_3 \cdot xH_2O/Fe(HMDTC)_3$ and the combination of $Fe_2O_3 \cdot xH_2O/Fe(TMDTC)_3$ [15], under the same conditions (20 mg Fe, $2 \cdot 10^{-4}$ mol HMDTC⁻ and $I_c = 0.02$ mol/l), it can be concluded that HMDTC⁻ has the advantage. Namely, the pH range is from 5.5–6.0 using TMDTC⁻, while in the case of addition of HMDTC⁻ flotation can be successfully performed within the extended pH range (5.5–6.5).

Influence of the amount of HMDTC on lead flotation efficiency

To investigate the influence of n(HMDTC⁻) on lead flotation, four series of flotations were performed at constant pH (6.0) and I_c (0.02 mol/l) by adding different volumes (1.3, 2.0, 3.0 and 6.0 ml) of 0.1 mol/l solution of HMA-HMDTC to the solutions (1 liter) containing 25 μg of lead. The first series of flotations was performed with 5 mg, the second with 10 mg, the third with 20 mg and the forth with 30 mg iron.

The results of these investigations (Fig. 2) showed that the flotations with 5 mg iron and 1.3 and 2 ml of 0.1 mol/l solution of HMA-HMDTC are not quantitative (90.0 – 91.5 %). The successful flotations (98.5 – 100.0 %) with 5 mg of Fe could be reached, if 3 or 6 ml of 0.1 mol/l solution of HMA-HMDTC had been added. The second, third and fourth series of flotations with 10, 20 and 30 mg Fe would reach a quantitative separation of lead

(95.2–97.1 %) with adding 2 ml of 0.1 mol/l solution of HMA-HMDTC.

This proves that the amount of HMDTC influences lead collection, but colligend recoveries depend on iron mass too. If smaller quantities of iron were used, the amount of HMDTC should be larger. For the proposed procedure 10 mg of Fe and 3 ml of 0.1 mol/l solution of HMA-HMDTC were chosen as the most appropriate for flotation of lead.
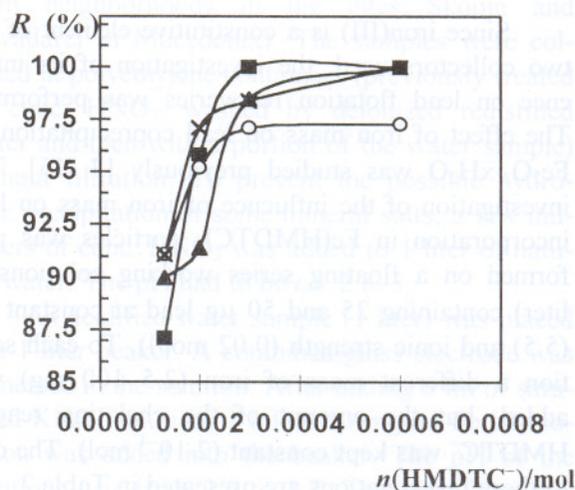

Fig. 2. Influence of n(HMDTC) on lead flotation recoveries (*R*) –▲– 5 mg Fe; –■– 10 mg Fe; –O– 20 mg Fe; –×– 30 mg Fe

Ionic strength

The effect of ionic strength (I_c) on lead flotation recoveries was investigated from the aspect of the proper coagulation of the collectors. For these purposes, three standards of varying volumes (250, 500 and 1000 ml) were floated at pH 6 with both collectors (10 mg Fe and $3 \cdot 10^{-4}$ mol HMDTC) without adding any ionic strength adjuster. Each standard solution contained 25 μg lead, so that the concentration of the final concentrated solutions (25 ml) was 1 μg/ml for 100 % recoveries. To each solution 10 mg Fe was added. The ionic strength of the first, second and third solution, were 0.0043 mol/l, 0.0022 mol/l and 0.0011 mol/l, respectively (Table 3). After the first and the second induction time with the two collectors used, the solutions were floated and lead was determined by AAS. The values for lead flotation recoveries of the first and the second solution was 100 %, while of the third was 92.9 %.

Table 3

Dependence of lead flotation recoveries on the ionic strength (I_c) of the solution

$m(Fe) / V$		R (%)
10 mg Fe / 250 ml		γ(Pb)
$c(Fe^{3+}) = 0.0007162$ mol/l	I_c / mol·l^{-1}	1 μg / ml
$c(NO_3^-) = 0.0021487$ mol/l	0.0043	100.0
10 mg Fe / 500 ml		γ(Pb)
$c(Fe^{3+}) = 0.0003581$ mol/l	I_c / mol·l^{-1}	1 μg / ml
$c(NO_3^-) = 0.0010743$ mol/l	0.0022	100.0
10 mg Fe / 1000 ml		γ(Pb)
$c(Fe^{3+}) = 0.0001791$ mol/l	I_c / mol·l^{-1}	1 μg / ml
$c(NO_3^-) = 0.0005372$ mol/l	0.0011	92.9

As can be seen, the lower ionic strength (I_c = 0.0011 mol/l) is insufficient to induce a proper co-agulation of the collector precipitates under previously established conditions (Table 3). During the treatment of the first and the second solution, the co-agulation of the system is complete (100 %). If 3 ml solution of KNO_3 (c = 2.78 mol/l) was added to 500 ml of the working solution *i.e.* 6 ml of the same solution to 1000 ml of the test solution, the ionic strength would become 0.0167 mol/l. This value of the ionic strength is much higher than the minimal established value of ionic strength (I_c = 0.0022 mol/l) and provides a proper coagulation. Therefore 0.02 mol/l was chosen as an optimal value for the ionic strength of the working solutions.

ζ potential of the collector flocs

A very important parameter, which can determine the choice of tenside, is the electrokinetic or ζ potential of the collector particle surfaces. The ζ potential values of the Fe(HMDTC)$_3$ flocs had been obtained for the pH of 3.5 to 5.35. Within that pH range Fe(HMDTC)$_3$ flocs have positive ζ potentials of 68.0–69.0 mV. ζ potential of the first collector Fe$_2$O$_3$·xH$_2$O within the same pH range was 25.0 to 28.0 mV. The positive charge of the ζ potentials of two collectors used, signifies that it is necessary to use anionic surfactant.

Comparing the value of ζ potential of Fe(HMDTC)$_3$ and ζ potential of Fe(TMDTC)$_3$, which is about 46 mV [15], it can be seen that Fe(HMDTC)$_3$ is the more positive value of ζ potential. This indicates that the micelles obtained by association of Fe(HMDTC)$_3$ with suitable anionic tenside/tensides would be more stable. Consequently, it could be expected that the froth layer obtained on the water surface in the flotation cell would be better fixed at the gas/liquid interface, be less exposed on the reflux with standing and its separation from the processed water phase be easier. These expectations were proved by following investigations.

Choice of tenside

To select the most effective anionic surfactant, a series of flotations of solutions (1 liter) containing 25 μg lead with a constant mass of Fe (10 mg) and amount of HMDTC$^-$ (3.0·10^{-4} mol) at constant I_c (0.02 mol/l) were performed with 1 ml of 0.5 % solution of surfactant within the pH range of 3.5 to 6.5 (Table 4). Surfactants were investigated indivi-dualy and in pair (NaST/NaOL, NaPL/NaOL and NaDDS/NaOL). Within pH range of 3.5 to 5.0 their effectiveness was poor due to their protolysis as anionic surfactants (Table 4).

Table 4

Recovery for different foaming reagents for colloid precipitate flotation of lead (γ(Pb)=1 μg/ml) with Fe$_2$O$_3$·xH$_2$O and Fe(HMDTC)$_3$ as collectors (I_c = 0.02 mol/l, γ(Fe) = 10 mg/l, c(HMDTC$^-$) = 3·10^{-4} mol/l)

pH	R (%)						
	NaDDS	NaOL	NaST	NaPL	NaST + NaOL	NaPL + NaOL	NaDDS + NaOL
3.5	no foam	no foam	no foam	no foam	no foam	no foam	no foam
4.0	no foam	no foam	no foam	no foam	no foam	no foam	no foam
4.5	34.0	64.8	no foam	no foam	28.5	45.6	26.0
5.0	82.6	79.1	68.3	53.6	71.9	78.0	84.0
5.5	99.0	98.1	75.3	78.0	79.3	92.8	98.4
6.0	100.0	98.1	79.1	82.0	98.0	100.0	98.4
6.5	100.0	98.1	86.1	89.6	98.0	100.0	98.4

The data in Table 4 show that NaST and NaPL individualy, as well as the pair NaST/NaOL and NaPL/NaOL were less useful. Among anionic surfactants, NaDDS was shown as the most appropriate and it was chosen for flotation with a collector $Fe(HMDTC)_3$. NaOL was also effective, but its calcium and magnesium salts are insoluble and it would be less effective in hard waters. The pair NaDDS/NaOL was very good, but this combination of surfactants gave unnecessarily a too copious scum which was very difficult to destroy. Because of that, much more conc. HNO_3 could be used to destroy the froth layer.

Induction time

The induction times from the results of the previous papers [7, 11–15] were applied and adjusted. The first induction time of 5 minutes and the second induction time of 15 minutes were enough for quantitative incorporation of lead in the collector precipitates under optimized conditions.

Detection limit

The detection limit of the method was estimated as three values of the standard deviation ($s =$ 0.02 µg/l). For this purpose, ten blank measurements were made. The detection limit ($L_d = 0.06$ µg/l) was estimated as three values of the standard deviation. The relative standard deviation was 3.8 %.

Application of the method

The proposed flotation method can be applied for analyses of natural waters with different hardness. Using a calibration curve, as well the standard additions technique, lead was determined in six samples of fresh water. After their flotation in accordance with the proposed procedure, they were 40-fold concentrated and then were measured by ZETAAS. Both ZETAAS measurements (by the calibration curve and by standard additions) seem to be equally valid. The recoveries of 94.7 to 101.3 % show that the preconcentration and separation of lead is satisfactory using the proposed method of flotation. The result obtained by ZETAAS were compared by the ICP-AES results. The water samples for ICP-AES measurements were prepared by evaporation from 1000 ml to 25 ml (Table 5).

Table 5

Results of ZETAAS determination of lead in fresh water (obtained by a calibration curve and a technique of standard additions) compared by ICP-AES results (obtained by a calibration curve)

Sample of water	ZETAAS				ICP-AES*
	Added µg l^{-1} Pb	Estimated µg l^{-1} Pb	Found µg l^{-1} Pb	R %	Found µg l^{-1} Pb
Pantelejmon 15.05 DH°** pH = 7.84	0.00	–	0.25 ± 0.015	–	<0.5
	1.25	1.50	1.45	96.7	
	2.50	2.75	2.70	98.2	
Sreden Izvor 17.65 DH° pH = 7.36	0.00	–	0.37 ± 0.021	–	<0.5
	1.25	1.62	1.59	98.1	
	2.50	2.87	2.75	95.8	
Kapi{tec 23.36 DH°** pH = 7.50	0.00	–	0.25 ± 0.02	–	<0.5
	1.25	1.50	1.42	94.7	
	2.50	2.75	2.63	95.6	
Radu{a 25.57 DH° pH = 8.50	0.00	–	0.26 ± 0.015	–	–
	1.25	1.51	1.45	96.0	
	2.50	2.75	2.70	98.1	
Kavadarci 5.71 DH° pH = 7.58	0.00	–	0.23 ± 0.012	–	0.22
	1.25	1.48	1.50	101.3	
	2.50	2.73	2.65	97.1	
Ra{~e 16.49 DH° pH = 7.18	0.00	–	10.6 ± 0.48	–	10.7
	1.25	11.85	12.00	101.3	
	2.50	13.1	12.40	94.7	

* Results of comparative ICP-AES determination of lead (samples enriched by evaporation)
** DH (*Deutsche Härte*) German degree of water hardness

CONCLUSION

The present paper has proved that lead can be floated successfully with iron(III) hexamethylene-dithiocarbamate. Zeeman atomic absorption spectrometry was applied as the instrumental method. The experimental conditions for the colloid precipitate flotation preconcentration of lead with two collectors have been established. The first induction time for a coprecipitation step of flotation with hydrated iron(III) oxide ($Fe_2O_3 \cdot xH_2O$) as the first collector is required. Adding $Fe(HMDTC)_3$ as a second collector is necessary for a quantitative lead sublatation. Hexamethylenedithiocarbamate has been shown as a very appropriate reagent for flotation purposes. Added to the working solutions, it increases the hydrophobility of the sublate which is the most important criterion for successful flotation. The investigations have shown that the reagent $Fe(HMDTC)_3$, is beter than $Fe_2O_3 \cdot xH_2O$ and $Fe(TMDTC)_3$ [11–15]. When the collector combination $Fe_2O_3 \cdot xH_2O/Fe(TMDTC)_3$ was used the flotation needs two surfactants (NaDDS and NaOL), while the collector combination $Fe_2O_3 \cdot xH_2O/Fe(HMDTC)_3$ needs only one foaming reagent (NaDDS). This means that $Fe(HMDTC)_3$ as a compound is more hydrophobic than $Fe(TMDTC)_3$ and therefore more effective as a collector for colloid precipitate flotation. The greater hydrophobility of $Fe(HMDTC)_3$ can be illustrated with more positive value of its ζ potential than the ζ potential of $Fe(TMDTC)_3$. The proposed procedure with $Fe(HMDTC)_3$ has the advantage because the use of only one surfactant needs less amount of conc. HNO_3 (2.5 ml than 10 ml conc. HNO_3 [15]). The recommended method extends the concentration range of the conventional atomic absorption determination of lead and can be applied for trace analyses of this colligend in large volumes of dilute aqueous solutions. The precision of the method as a relative standard deviation was found to be 3.8 %, standard deviation is 0.02 µg/l and the detection limit was estimated as three values of the standard deviation (L_d = 0.16 µg/l). The comparison of the detection limit obtained, as well as the relative standard deviation in relation to the method with TMDTC (0.3 µg/l and 5.32 %) [15], show the advantage of the procedure with HMDTC.

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ФЛОТАЦИЈА СО ЖЕЛЕЗО(III)ХЕКСАМЕТИЛЕНДИТИОКАРБАМАТ КАКО МЕТОД ЗА КОНЦЕНТРИРАЊЕ НА ОЛОВО И НЕГОВО ОПРЕДЕЛУВАЊЕ СО ЕЛЕКТРОТЕРМИЧКА АТОМСКА АПСОРПЦИОНА СПЕКТРОМЕТРИЈА

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Клучни зборови: олово; определување; колоидна таложна флотација; колектори; хидратизиран железо(III)оксид; железо(III)хексаметилендитиокарбамат; вода; електротермичка атомска апсорпциона спектрометрија

Хексаметиленамониум-хексаметилендитиокарбамат (HMA-HMDTC) е применет како нов флотационен реагенс за концентрирање на олово. HMA-HMDTC додаден на првиот таложен колектор хидратизиран железо(III)оксид ($Fe_2O_3 \cdot xH_2O$) го дава вториот таложен колектор железо(III)хексаметилендитиокарбамат ($Fe(HMDTC)_3$). Оптимирани се сите важни параметри (концентрација на колекторите, pH, видот на тензид, јонска јачина и др.) неопходни за успешна флотација на оловото со комбинацијата на колекторите $Fe_2O_3 \cdot xH_2O/Fe(HMDTC)_3$. При утврдени вредности на pH (6,0) и јонска јачина (0,02 mol/l), со додавање на NaDDS како тензид, оловото може квантитативно да се сепарира со 10 mg Fe(III) и $3 \cdot 10^{-4}$ mol HMDTC⁻ додадени на еден литар примерок од вода. Zeeman-ова електротермичка атомска апсорпциона спектрометрија (ZETAAS) е применета за определување на оловото во слатки води. Резултатите се споредени со оние добиени со атомска емисиона спектрометрија со индуктивно спрегната плазма (ICP-AES). Границата на детекција за определување на олово со овој метод изнесува 0,06 µg/l.