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ATOMIC-ABSORPTION DETERMINATION OF LEAD, COPPER AND CADMIUM IN BRINES USING CO-PRECIPIATION CONCENTRATING ON TWO COMPONENT COLLECTOR

Abstract: Coprecipitation of lead, copper and cadmium on magnesium hydroxide was studied. Coprecipitation mechanism was proposed. The use of two-frequency ultrasound for intensification of coprecipitation was investigated. It has been found that dispersing and mixing effects are the determining factors of the US action on coprecipitation concentration. The method of determination of lead, copper and cadmium in natural brines, using coprecipitation and ultrasound intensification has been developed. The content of lead, copper and cadmium in the natural brines of Ukraine was determined. It was shown that the use of double-frequent US allows us to increase the degree of determination lead, copper and cadmium from 92-95% up to 97-98% and also to improve metrological characteristics to determine analytes in natural brines.

Key words: atomic absorption spectrometry, natural brines, ultrasound, coprecipitation, metrologic characteristics.

Language: English

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Introduction

Natural sodium chloride brines are a valuable raw material for the chemical and food industries. Natural sodium chloride brines of Ukraine have a salinity of 10 to 300 g/l. Sodium chloride brines are

used in chemical enterprises to produce baking and technical soda, metallic sodium, and sodium hydroxide. Brine - is a raw material for the production of pool salt (Heroic and Genichesk saltworks), vacuum-evaporating table salt grade "Extra" (Slavic

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salt company). Ukraine is very rich in natural brines, brine deposits are located in the southern and eastern part of the country. Sodium chloride brines consist of sodium chloride - 60-90%, magnesium chloride, calcium and potassium chlorides, sodium sulfate, potassium, magnesium - 10-40%. Also in brines there are micro-impurities of lead, copper, cadmium, iron, boron and others. The first three components are toxic and their content in the finished product - table salt is regulated. According to sanitary-anti-epidemic and sanitary-antitoxicologic rules and norms of SanPiN 42-123-4089 the content of lead, copper and cadmium in table salt should not exceed 2.0; 3.0; 0.1 mg / kg.

Kitchen salt is the most important food product and also a valuable raw material for industry. It is sometimes called "white death". Indeed - if you exceed the recommended intake of 5-6 g per day, it can cause hypertension.

However, it has been statistically proven that complete abstinence from salt leads to a rapid decline in immunity, and as a result - to the emergence of a number of different diseases - from fungal lesions of internal organs to cancer.

In addition, the refusal to use salt leads to blood clotting, a rapid increase in the load on the heart and the formation of blood clots. The life expectancy of volunteers who completely refused to use salt for 5 years or more did not exceed 55 years. At the same time, a survey of long-lived Yakutia and Greece revealed the unexpected fact that they all consumed 10 to 12 grams of salt per day. The presence of lead, copper and cadmium in natural brines leads to the appearance of these toxic trace elements in table salt. The lower limit for the determination of lead, copper and cadmium is 40 $\mu\text{g} / \text{l}$, 0.8 $\mu\text{g} / \text{l}$ and 0.4 $\mu\text{g} / \text{l}$. The management of the Slavic salt mining company insists on essential reduction of the lower limit of definition of microcells in 5 times.

We proposed to use for preconcentration of lead, copper and cadmium from brines the coprecipitation on a collector of magnesium oxide-magnesium carbonate. The choice of the collector is justified by us as follows: on the reservoir of magnesium hydroxide-calcium carbonate quantitative coprecipitation of lead, copper and cadmium from sea water is possible. In addition, magnesium and calcium are macro-impurities of natural brines, which eliminates the need to add them in the form of additional reagents and as a result - reduces the risk of "contamination" of brine samples with impurities from the reagents. [1,p.202; 2,p.39; 3,p.575; 4,p.282, 5,p.261; 6,p.555; 7,p.26; 8,p.131; 9,p.85; 10,p.29; 11,p.664, 12,p.345; 13,p.65; 14,p.157; 15,p.186]

The purpose of the work is to develop a competitive method of atomic absorption determination of lead, copper and cadmium in the natural brines on two component collector with better metrological characteristics.

Experimental

The following is used in the work:

1. Atomic absorption spectrometer AAS-3 (Carl Zeiss Jena, Germany).
2. Personal computer.
3. Electronic scales analytical VLR-200 (Russia).
4. Ultrasonic dispersant UZDN - 1m with magnetostrictive ultrasonic emitters from 18 kHz to 100 kHz.
5. The ultrasonic generator 24 - UZGI - K - 1,2 with piezoelectric ultrasonic radiators from 200 kHz to 10 MHz
6. T 23 D centrifuge (Germany)
7. The device for shaking of AVU 6s.
8. Electronic ionomer pH-meter pH-673.
9. Bidistillator quartz BC
10. Laboratory measuring utensils according to GOST 1770-74.

For the preparation of all types of solutions used double-distilled water obtained in a quartz double-distiller (type BC). For the preparation of standard solutions of Lead, Copper and Cadmium, standard samples of composition of solutions of MSO 1061: 2004 with a concentration of Lead, Copper and Cadmium of 1 mg/ml were used.

Sodium chloride "chemically pure for spectral analysis" TU 6-09-3658-74 was further purified by recrystallization.

Hydrochloric acid, with a density of 1.19 g / cm³, was further purified by isopiestic distillation, while the concentration of hydrochloric acid thus obtained was approximately 6 mol / l. All other reagents were prepared from substances of qualification not lower than chemically pure.

All the experiment on the atomic absorption determination of Lead, Copper and Cadmium was carried out on an atomic absorption spectrometer AAS - 3 (Germany) with a flame atomizer in a flame "acetylene - air".

Results and discussion

It was found that sodium chloride does not affect on the degree of coprecipitation of lead, copper and cadmium to a concentration of 130 g / l, and quantitative coprecipitation (degree of coprecipitation $\geq 90\%$) is possible up to a concentration of 140 g / l for cadmium and up to 150 g / l for copper and Lead. (Pic.1)

The optimal pH of the solution for coprecipitation were as follows: lead 9.5-13.0; Copper 12.0 - 13.0; Cadmium 12.0 - 13.5. (Pic.2)

Increasing the number of microcomponents of lead, copper and cadmium from 5 up to 120 $\mu\text{g} / \text{l}$ cause to increase the degree of coprecipitation of lead (Pic.3). This indicates that absorption of lead, copper and cadmium is by the ion exchange mechanism with the formation of solid solution.

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The amount of collector of simultaneous co-deposition of lead, copper and cadmium must be at least 5 mmol / l (Pic.4)

The optimal ratio in the collector of magnesium hydroxide - calcium carbonate for simultaneous co-deposition of lead, copper and cadmium is 1: (4-6). (Table 1). The higher efficiency of the two-component collector is explained by the fact that according to the literature in the presence of calcium carbonate, the particles of magnesium hydroxide are finer, or the particles of calcium carbonate do not allow the particles of magnesium hydroxide to stick together. (Tables 2-3).

Conclusions

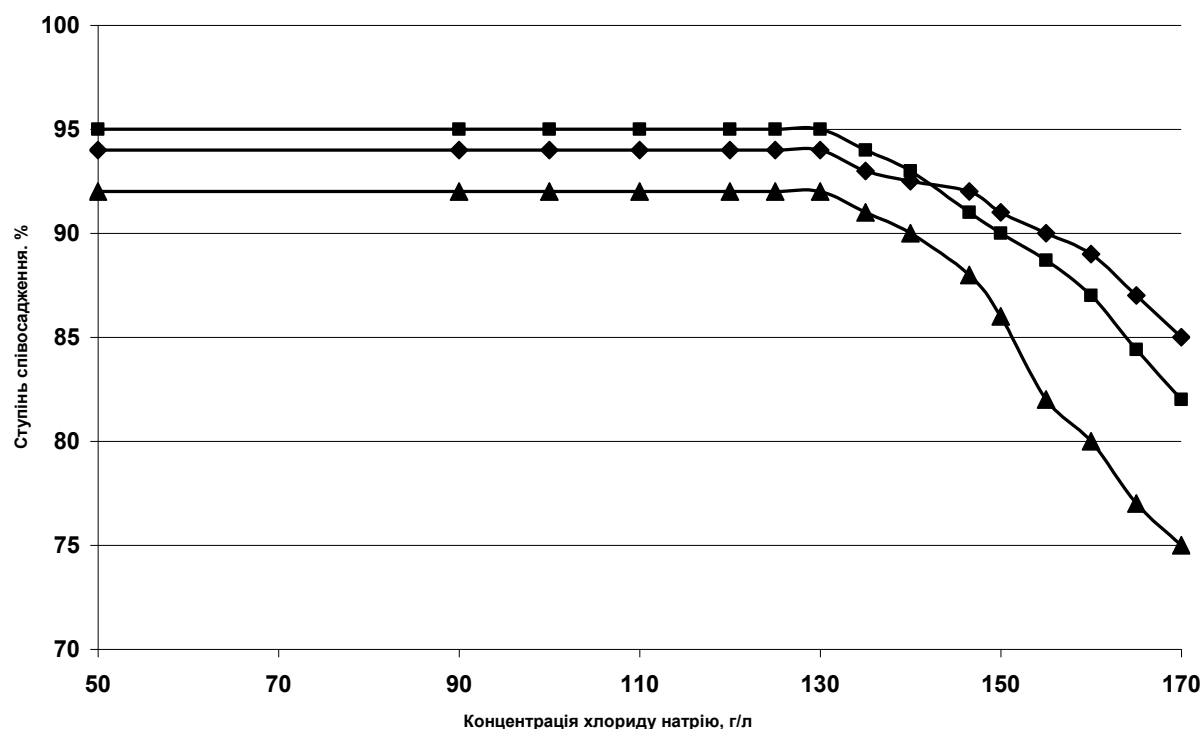
Co-precipitation of lead, copper and cadmium in a two-component reservoir of magnesium hydroxide - calcium carbonate was studied. It is shown that in optimal conditions the degree of coprecipitation of lead, copper and cadmium does not exceed 93, 95 and 92%, respectively. To increase the degree of coprecipitation, it is recommended to use the action of ultrasound.

It was found that more than 60% of lead, copper and cadmium are in brines and salt solutions in forms that are not co-precipitated. It was concluded about

necessity of the process of destruction of organic compounds of lead, copper and cadmium.

The use of ultrasound in the determination of lead, copper and cadmium in highly mineralized waters, brines and kitchen salt has been studied: for the conversion of compounds of lead, copper and cadmium into forms that are to coprecipitate, as well as intensify the concentration of co-precipitation on magnesium hydroxide. It has been experimentally established that the determining factor in the intensifying effect of ultrasound in the conversion of lead, copper and cadmium into coprecipitate forms are sound chemical reactions, and intensification of concentration by coprecipitation is mixing and dispersing effect of ultrasound.

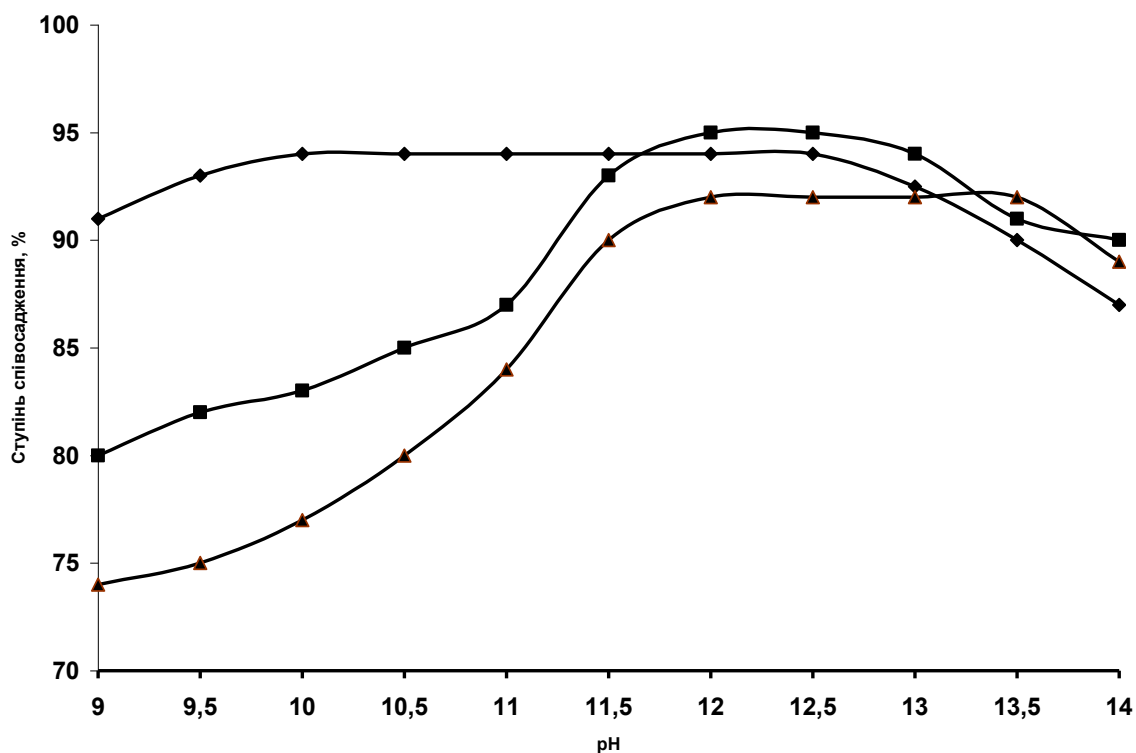
An express methods for determination of lead, copper and cadmium has been developed, which includes destruction of organic impurities of lead, copper and cadmium, coprecipitation of lead, copper and cadmium on calcium magnesium carbonate hydroxide with ultrasonic intensification of the process. In the obtained concentrate, lead, copper and cadmium were determined by the atomic absorption method at a wavelength of 283.3 nm; 324.8 nm and 228.8 nm, respectively, in an acetylene-air flame.



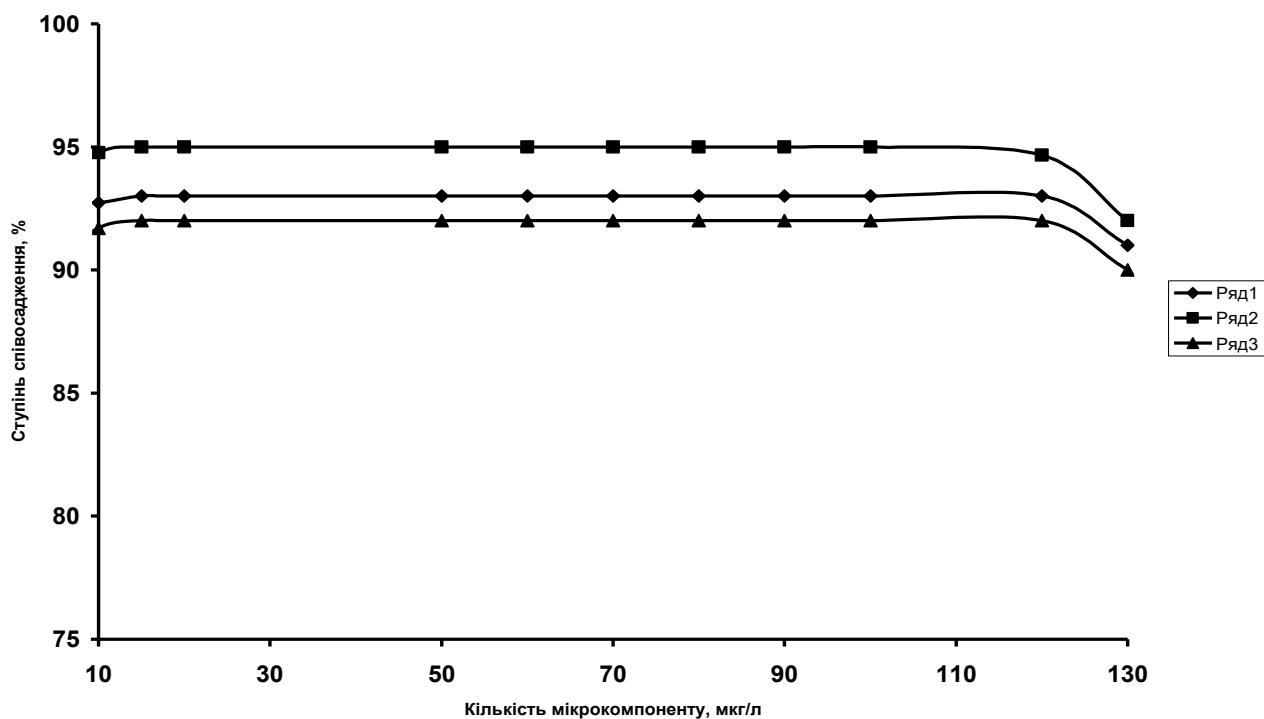
Pic.1 An influence of sodium chloride concentration on the degree of coprecipitation of lead (1), copper (2) and cadmium (3)

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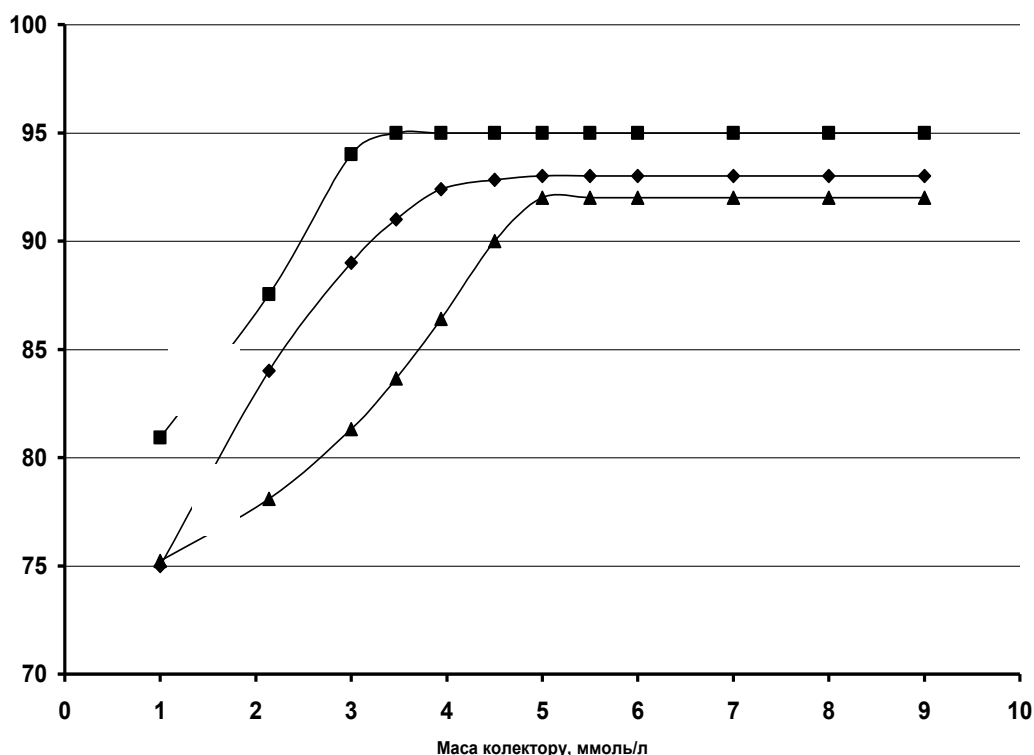
Pic.2 The effect of solution pH on the degree of coprecipitation of lead (1), copper (2) and cadmium (3)



Pic.3 Influence of the amount of microcomponent on the degree of coprecipitation of lead (1), copper (2) and cadmium (3)

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Pic.4 An influence of the amount of collector on the degree of co-deposition of lead (1), copper (2) and cadmium (3)

Table 1 An influence of the time of contact of the precipitate with the solution on the degree of coprecipitation of lead, copper and cadmium (n = 5; p = 0.95)

Time, min.	Lead	Copper	Cadmium
2	52,4	27,0	32,1
4	60,6	41,9	49,3
6	78,5	79,7	81,2
8	91,1	90,2	92,1
10	92,9	95,0	91,9
12	92,8	94,8	91,8
14	93,1	95,2	92,0
16	92,9	94,9	92,0

Table 2 The optimal ratio in the collector

Ratio	Lead	Copper	Cadmium
1:0	35,3	95,0	92,1
2:1	43,6	91,9	92,2
1:1	47,5	86,7	84,6
1:2	85,1	90,3	85,3
1:3	91,4	92,6	90,1
1:4	92,8	95,1	92,1
1:5	93,0	95,0	92,0
1:6	92,7	94,8	92,2
1:7	93,0	90,2	92,0
0:1	91,6	86,5	45,8

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Table 3 Determination of the content of lead, copper and cadmium in brines of Ukrainian deposits. (n=5; p=0,95)

Standard brine sample	10,6±0,72	0,065	10,4±0,83	0,076
Brine of the Slavic deposit, well 1	12,4±0,87	0,067	12,1±0,87	0,075
Brine Heroic deposits	19,4± 1,28	0,063	19,5±1,43	0,070
Brine of the Genichesky deposit	22,1± 1,41	0,061	21,5±1,67	0,074
Copper				
Standard brine sample	3,80 ± 0,279	0,070	3,81±0,320	0,080
Brine of the Slavic deposit, well 1	4,42± 0,329	0,071	4,16± 0,371	0,085
Brine Heroic deposits	8,07± 0,618	0,073	8,01± 0,706	0,084
Brine of the Genichesky deposit	12,16±0,880	0,069	11,99±1,018	0,081
Cadmium				
Standard brine sample	4,90± 0,360	0,070	4,84± 0,406	0,080
Brine of the Slavic deposit, well 1	4,04±0,301	0,071	3,86±0,344	0,085
Brine Heroic deposits	8,11±0,621	0,073	8,07±0,711	0,084
Brine of the Genichesky deposit	11,10±0,838	0,072	11,17±0,949	0,081

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