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Oleg Ivanovych Yurchenko
Kharkiv V.N. Karazin National University
PhD, Full Professor of Chemical Metrology Department,
yurchenko@karazin.ua

Tetyana Vasylivna Chernozhuk
Kharkiv V.N. Karazin National University
PhD, Associate Professor of Inorganic Chemistry Department,
tanya.chernozhuk@gmail.com

Alexandr Nikolaevych Baklanov
Kharkiv V.N. National University
PhD, Full Professor of Chemical Metrology Department,
baklanov_oleksandr@meta.ua

Oleksii Andriovych Kravchenko
Kharkiv V.N. Karazin National University
PhD, Associate Professor of Chemical Metrology Department
alekseykravch@ukr.net

ATOMIC ABSORPTION DETERMINATION OF CHROMIUM IN KITCHEN SALT USING MACROCOMPONENT EXTRACTION

Abstract: *The use of ultrasound (US) at determination of chromium in kitchen salt using macrocomponent extraction was investigated. It has been found that US using let us to increase the degree of chromium extraction and improve , metrologic characteristics of the obtained results at kitchen salt analysis. It was shown that the use of double-frequent US allows us to increase solubility of sodium chloride in hydrogen peroxide from 42 up to 98 % and to increase the degree of chromium extraction from 94% up to 98% .dispersing and mixing effects are the determining factors of the US action on co-precipitation concentration. The method of atomic absorption determination of chromium in kitchen salt using macrocomponent extraction has been developed.*

Key words: *atomic absorption spectrophotometry, Chromium, kitchen salt, ultrasound, metrologic characteristics.*

Language: *English*

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Introduction

Chromium is toxic biologically active element, The toxicity of Chromium depends on the forms of its existence. Hexavalent Chromium is the most toxic. However, according to the sanitary-anti-epidemic and sanitary-anti-toxicological rules, the norm is normalized in the kitchen only in general. But even now there are no standard methodic to determine Chromium in kitchen salt, mineral waters brines. There are methodic to determine Chromium by extraction with hydrochloric acid and determination of the content of Chromium by atomic-absoption method. In this case, for the formation of

acetylacetonate Chromium, the solution is boiled for 1 hour [1,p.14;2,p.120;3,p.117;4,p.47].

The purpose of this work is to develop methodics for of atomic absorption determination of chromium in kitchen salt with improved metrological characteristics.

Experimental
We used the next substances and equipment to conduct the experiment

1. AAS-3 atomic absorption spectrometer (Capl Zeitz Jena company, Germany).

2. Personal computer.

3. Libra analytical VLP-200 (Russia).

4. Ultrasonic disperser UZDN - 1m with magnetic ultrasonic emitters from 18 kHz to 100 kHz.

5. Ultrasonic generator 24 – UZGI – K – 1,2 with piezoelectric ultrasonic emitters from 200 kHz to 2,5 MHz

4. Ash-free ash filters according to TU 2642-001-42624157-98

5. Laboratory glassware for DCT 1770-74.

6. Water distilled for DCT 6709-72.

7. Argon and helium

8. Chromium solution - 1, containing - 1 mg of Chromium in 1 ml. Prepared with the use of state standard samples.

9. Chromium, solution - 2, which contains 10.0 mcg of Chromium in 1 ml. Prepare by sampling 10 ml of solution-1 in a flask with a capacity of 1000 ml and bringing the volume of the solution in the flask with distilled water to the scale.

10. Chromium, solution-4, which contains 0.10 µg of nickel in 1 ml. Prepare by sampling 10 ml of solution-3 in a flask with a capacity of 100 ml and bringing the volume of the solution in the flask with distilled water to the scale.

11. AIU 6c shaker.

12. pH-meter pH-121.

14. NaCl, TU 6-09-3658-74

When analyzing only some of the substances for concentrating micro-impurities, the method of base formation can be used. Such substances include sodium chloride. It is known that sodium chloride dissolves in hydrogen peroxide at low temperatures (in the range from -5 to -10 ° C). To increase the solubility of sodium chloride in hydrogen peroxide and increase the speed of the process, we used the effect of ultrasound from the moderated ultrasonic dispensers of the dispenser. The atomic-observational value of Chromium was performed on the atomic-observational spectrometer AAS-3 (Germany). It was used distilled water, sodium chloride for spectral analysis, hydrogen peroxide 90%. The technique of the experiment was as follows.

The analyzed samples were put in flaks on 1000 ml, cooled and treated by ultrasound of 14 ... 47 kHz frequency for 20-25 sec. Then the concentrate was dissolved in hydrochloric acid (1: 1), quantitatively

transferred to a test tube and the volume of the solution was reduced to 10 ml. The concentrate was sprayed into the flame of the spectrometer burner. Chromium was determined by the flame atomic-absorption method in the reduction flame of acetylene-air at a wavelength of 357.9 nm at a monotonic gap width of 0.2x mm.

The optimal parameters of the influence of ultrasound on the causes: intensity and time were selected experimentally, changing each of them until the appearance of the maximum possible. For the process of dissolving sodium chloride in hydrogen peroxide, these parameters were taken: frequency 18 ... 44 kHz, power 0.5 ... 0.8 W, time 20 ... 25 s. The maximum possible solubility of sodium chloride in hydrogen peroxide (up to 35 g / 100 ml) is reached at a temperature of -20 to -25 C (Table 1). Due to the low rate of dissolution of sodium chloride in hydrogen peroxide, large volumes of solvent were used.

At ultrasonic intensity less than 0.5 W, there is a slight increase in the solubility of sodium chloride. Under the influence of ultrasound less than 20 s of complete dissolution of sodium chloride, sodium does not occur, and the influence of more than 25 s leads to burning of the solution and, as a result, to a decrease in the solubility of Chromium. Analysis of impurities in sodium chloride was carried out before and after treatment with hydrogen peroxide. [5,p563;6,p.7;7,p.179;8,p.100;9,p.12;10,p.124; 11,p572;12,p.260;13,p.3324;14,p.139; 15,p.110;]

The correctness of the technique is determined by the "introduced-found" method (Table 2).

The method of determination of impurities in the kitchen salt is characterized by high sensitivity and accuracy (Table 3).

The use of US of two frequencies for the dissolution of sodium chloride in hydrogen peroxide is due to the higher efficiency of two-frequency ultrasound in comparison with ultrasound. Its due to liquidation of small cavitation bubbles in our solutions.

The change in the frequency of low-frequency ultrasound during the treatment of the mixture from 18 to 100 kHz on the solubility of sodium chloride in hydrogen peroxide did not occur (Table 4). Comparison of the results obtained with the use (Table 5). Comparison of the results obtained with the use of high-frequency ultrasound with a frequency of 0.5… 3.0 MHz showed that the best results were obtained with the use of 2.0. At the same time, the intensity of low-frequency ultrasound should be 0.15… 0.25 W, and high-frequency - 0.25… 0.50 W (Table 5).

As shown in Table 5, the use of two-frequency ultrasound leads to an increase in the solubility of sodium chloride in a hydrogen peroxide.

Thus, the use of two-frequency ultrasound in comparison with the use of ultrasound of the same frequency, allows us to increase the solubility of sodium chloride in the experiment from 42 up to 47

g/100 ml, also to increase the degree of extraction of the introduced part of Chromium from 94 to 98%, to improve the metrological characteristics of the results of the analysis of Chromium.

Conclusions

The use of ultrasound (US) in the determination of Chromium in the kitchen salt with the use of extraction of the macrocomponent was studied.

Ultrasound was used to intensify the process of extraction of macrocomponent - sodium chloride. The use of ultrasound allows to increase the sensitivity and to improve the metrological characteristics of the analysis results.

The methods are intended for accreditation of chemical laboratories, for laboratories of state bodies of standardization.

Table 1. Solubility of NaCl in hydrogen peroxide

Temperature, ºC	Solubility of NaCI, g / 100 ml of solvent	
	Without US	With US
-10	12,7	15,4
-15	18,9	26,7
-20	33,7	41,9
-25	34,8	42,0
-30	34,8	41,9

Table 2. An influence time of solubility on the degree of Chromium extraction

Time, min	Degree of Chromium extraction, %
10	72
15	79
20	85
25	85
30	85

Table 3. The results of Chromium determination in kitchen salt

	Found out, mg/kg / S_r (n = 6, p = 0,95)			
	Kitchen salt «Extra»		Kitchen salt «Artemsil»	
	Injected,mg/kg			
	0	0,06	0	0,06
Without US	0,025±0,010 0,10	0,089±0,022 0,10	0,036±0,009 0,09	0,089±0,010 0,09
One frequency US	0,030±0,003 0,04	0,092±0,007 0,05	0,043±0,004 0,03	0,102±0,007 0,02
Two frequency US	0,033±0,002 0,02	0,093±0,002 0,02	0,045±0,002 0,02	0,104±0,002 0,02

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Table 4. Comparison of well - known and proposed by us methods of Chromium determination in kitchen salt

Parameters	Ways to obtain concentrate	
	Without US	With US
Limit of Chromium determination, mg/kg	0,001	0,001/0,001
S_r	0,08	0,04/0,01
Time of analysis	45…50 min.	20…25 c / 20…25 c
Degree of extraction, %	85	94 /98

Table 5. An influence of low frequency US on solubility of kitchen salt

Characteristics	US frequency, кHz %							
	17	18	22	50	60	80	100	110
Solubility of NaCl in hydrogen peroxide, g/100 ml	43	47	47	48	47	47	47	45

Table 6. An influence of high frequency US on solubility of kitchen salt

Characteristics	US frequency, MHz					
	0,5	1	1,5	2	2,5	3
Solubility of NaCl in hydrogen peroxide, g/100 ml	40	47	47	48	44	36

Table 7. An influence of intensity of US on solubility of kitchen salt

US intensity, W/sm^2	*Solubility of NaCl in hydrogen peroxide*, g/100 ml					
0,10	34	38	40	41	43	40
0,15	43	46	47	46	46	43
0,20	42	47	46	47	45	43
0,25	41	47	47	46	47	44
0,30	40	42	42	49	43	40

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