SOI: 1.1/TAS DOI: 10.15863/TAS

International Scientific Journal

Theoretical & Applied Science

p-ISSN: 2308-4944 (print) **e-ISSN:** 2409-0085 (online)

Year: 2017 **Issue:** 09 **Volume:** 53

Published: 18.09.2017 **http://T-Science.org**

SECTION 9: Chemistry and chemical technology.

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

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

Aleksandr Nikolaevich Baklanov
Kharkiv V.N. Karazine
National University, PhD, Full Professor of
Chemical Metrology Department,
baklanov_oleksandr@meta.ua

Larysa Vladimirovna Baklanova
Kharkiv V.N. Karazine
National University, PhD, Researcher of
Chemical Metrology Department
baklanovalarisa@ukr.net

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

COMPLEX APPROACH TO THE ANALYSIS OF SODIUM CHLORIDE OF PHARMACOPOEIAL PURITY

Abstract*: A set of techniques for quality control of sodium chloride of pharmacopeia purity is proposed. In comparison with the methods of analysis according to FS 42-25-78-88, the proposed methods have greater expressiveness and better metrological characteristics.*

Key words*: purity control, FS 42-25-78-88, a set of techniques, metrologic characteristics.*

Language*: English*

Citation: Yurchenko OI, Chernozhuk TV, Baklanov AN, Baklanova LV, Kravchenko OA (2017) COMPLEX APPROACH TO THE ANALYSIS OF SODIUM CHLORIDE OF PHARMACOPOEIAL PURITY. ISJ Theoretical & Applied Science, 09 (53): 52-55.

Soi: http://s-o-i.org/1.1/TAS-09-53-8 ***Doi***: https://dx.doi.org/10.15863/TAS.2017.09.53.8

Introduction

To control of sodium chloride of pharmacopeia purity quality according to actual now FS 42-25-78-88, the methods of titrimetry, gravimetry and colorimetry which are characterized by durability and low accuracy are used [1, p 7]. It makes more complete to control quality of prepared substance also to carry out quick control of product quality at process of its manufacture [2, p.44].

We propose to use complex of analytic methodic, includes methods of flame and inflame atomic-absorption spectrometry, turbidimetry, potentiometry to control quality of sodium chloride of pharmacopeia purity. To determine sulphates we recommend to use turbidimetry, pH- potentiometry, potassium-spectral emission; lead, copper, cadmium ,zinc- inflame atomic-absorption, mercury- inflame atomic-absorption of “cold vapor”, arsenic-spectrometry with silver dithyldithiocabaminate with pre-saturating with coprecipitation; chlorine-titrimetry; ammonia-photometry with Nessler reagent. [2,p.46; 3,p.40;4,p.96;5,p.11]. But due to insufficient sensibility of atomic-absorption determination of calcium, magnesium and iron in food salt can not be used to analyze sodium chloride of pharmacopeia purity [1,p.3; 2, p.56]. Moreover, due to FS 42-25-78-88, we should control barium contain [1,p.7].

The purpose of our work is to develop complex of methodic to control sodium chloride of pharmacopeia purity quality.

Experimental

Devices. Determination of calcium, magnesium, iron and barium was done on atomic-absorption spectrometer AAS-3 by analytic lines 422,7, 285,2, 248,3 и 553,6 nm correspondingly. Acetilene-nitrogen (I) oxide to determine barium and acetilene-air to determine another elements was used. Sulphate-ions were determined with use of colorimeter KFK-2 by turbidimetry at wave length 590 nm [2, p.67]. Determination of pH of solutions was done with use pH meter 673 M or ionomer EV – 74 with glass electrode ESP - 14 – 01.

Chemical substances. 8-oxichinoline was purified by recrestallization from ethanol and vacuum sublimation [2, p.78]. Organic solvents was purified by distillation in quartz device, ammonia-by isothermal distillation [2, p.80]. Sodium chloride for spectral analysis was purified by recrystallization into ethanol. Solutions of substances were prepared on bidistillated water, obtained in quartz bidistillator BDK-10. Standard solutions of calcium, magnesium, potassium, iron, barium, sulphate-ions were done on the base on state standatd samples, made in FHI of Bogatsky [5,p.14; 6, p.12;7,p.11].

Determination of lead was done by inflame atomic-absorption method, arsenic by spectrometry with silver dithyldithiocabaminate, pH by potentiometric according to [2,p.77; 5,p.11]. In the vessel perfect volume of calcium, magnesium, iron and barium solutions was put. By addition of ammonia or hydrochloric acid perfect value of pH of the solution was established, put into filter and extract calcium, magnesium, iron by 8-oxichinoline, barium by hexafluoracetylacetone solution during some time. After phase separation, extraction was repeated. Extracts were spread into atomic-absorption spectrometer. An influence of nature of organic solvent and concentration of sodium chlorine on extraction degree of calcium, magnesium, iron and barium, pH of solution, extraction time, number of extractions was studied.

At determination of barium use like solvents isoamileacetate, butilacetate was investigated [2, p.100; 8, p.10].

Results and discussion

We can find out from the experiments that from sodium chloride solution (50 g/dm^3) and correlation between organic and water phases 1:1 and extraction time 15 min maximal analytical signal is getting for calcium at pH 10,5-13,0 at extraction by 8-oxichinoline in n-buthanole, for magnesium at pH 8,5-11,0 at extraction by 8-oxichinoline in methylisobuthilecetone, for iron at pH 4-12, at extraction by 8-oxichinoline in methylisobuthilecetone, for barium at pH 10,5-12,5 in 1M solution of hexafluoracetylacetone in isoamylacetate. At use for barium extraction solutions of 8-oxichinoline in methylisobuthilecetone and n-buthanole extraction degree was not more than 50% (table 1).

At extraction of calcium, magnesium and iron quantitative extraction (extraction degree more than 90%) is possible up to sodium chlorine concentration 200 g/dm^3 and use of 8-oxichinoline solution in n-buthanole, and at use of 8-oxichinoline in methylisobuthilecetone is 50 g/dm^3 (table 1).

At extraction of barium quantitative extraction is possible up to sodium chlorine concentration not more than 50 g/dm^3 and maximal possible extraction degree is 95 % at use of isoamilacetate like organic solvent. (table 1). Optimal proportion of organic and water phases is 1:3 at extraction of calcium and magnesium, 1:3 at extraction of iron and barium. (table 2).

Magnesium, barium and iron are extracted at single extraction, calcium is at double one (table 3).

Extraction time should be not less than 15 minutes at extraction of magnesium and calcium and not less than 3 minutes at extraction of iron and barium (table 4).

So we proposed complex of methodic to analyze sodium chloride of pharmacopeia purity. The proposed complex of methodic was used at penetration of manufacturing sodium chloride of pharmacopeia purity at OOO “Slavyansk salt manufacture company”.

The results of sodium chloride analysis with use of the proposed complex of methodic and FS 42-25-78-88 are in the table 5. We can see that the proposed by us methodic possess better metrological characteristics that FS 42-25-78-88, that supply better control of product quality. Time of analysis of 10 sodium chloride of pharmacopeia purity samples not more than 2 hours, but at use of FS 42-25-78-88 we need 8 hours. An accuracy of the proposed methodic was tested on samples of sodium chloride of pharmacopeia purity from OOO “Slavyansk salt manufacture company”. Determination limit of calcium is $1,6\cdot10^{-5}$ %; magnesium is $0,8*10^{-6}$ %, iron is $8,0\cdot10^{-5}$ %, barium is $4,0\cdot10^{-4}$ %.

Conclusions

The complex of methodic to control quality **of** sodium chloride of pharmacopeia purity was proposed. Compare to FS 42-25-78-88 the proposed methodic possess more expresses and better metrological characteristics.

Methodic of determination of magnesium, calcium and iron. 5,000 g of sodium chloride, dried up to constant mass, is placed in the vessel, dissolved in 30 sm^3 bidistilled water, and add ammonia up to pH 10,5-11,0. The obtained solution put into filter, and extract magnesium, calcium and iron *by* 10 sm^3

0,1 M solution of 8-oxichinoline in n-buthanole. Extraction is repeated. Extracts are united, solutions are made up to 20 sm^3 . *magnesium, calcium and iron are determined by* atomic-absorption method in acetylene-air flame at waves 422,7, 285,2 and 248,3 nm correspondingly. Standard solutions are prepared on the base of twice recrestallizated sodium chloride for spectral analysis. It is recommended to purify sodium chloride solution by extraction of 8-oxichinoline in n-buthanole.

Methodic of determination of barium .5,000 g of sodium chloride, dried up to constant mass, is placed in the vessel, dissolved in 100 sm^3 bidistilled water, and add ammonia up to pH 10,5-12,0. The obtained solution put into the volumetric flack of 200 sm^3 volume. 20 sm^3 1 M of hexafluoreacetilacetone in isoamilacetate in extracted during 3 minutes. After phases distribution bidistilled water is added to increase organic layer and spread it out in acetylene-nitrogen (I) flame. Determination is conducted at wave 553,6 nm. Standard solutions are prepared on the base of twice recrestallizated sodium chloride for spectral analysis.

Table 1

An influence of sodium chloride on extraction degree of calcium, magnesium and iron.

Concentration of sodium chloride, g/dm^3	Extraction degree, %									
	Ca		Mg		Fe		Ba			
	*	**	*	**	*	**			*	**
40	98	98	99	99	97	96	95	94	42	50
50	97	97	98	99	96	93	95	92	36	43
60	94	89	97	88	96	85	86	78	33	38
100	94	54	96	68	94	79	77	71	21	26
200	90	35	91	45	91	64	54	53	17	15
210	75	18	73	29	82	43	40	48	5	9
250	31	12	35	21	25	20	17	38	-	-
260	14	-	18	11	12	7	5	31	-	-

Table 2

An influence of proportion of organic and water phases on extraction degree of calcium, magnesium and iron and barium.

Proportion of organic and water phases	Extraction degree, %			
	Ca	Mg	Fe	Ba
1:1	94	97	94	95
1:2	93	96	94	95
1:3	92	95	94	94
1:4	91	93	94	94
1:6	90	92	94	94
1:8	90	90	90	92
1:10	87	87	84	90
1:15	82	85	72	85

Table 3

An influence of number of extractions on extraction degree of calcium, magnesium and iron and barium.

Number of extractions	Extraction degree, %			
	Ca	Mg	Fe	Ba
3	92	95	94	94
2	90	93	93	93
1	90	91	91	91

Table 4

An influence of time of extractions on extraction degree of calcium, magnesium and iron and barium.

Time of extractions	Extraction degree, %			
	Ca	Mg	Fe	Ba
1	89	89	87	88
3	93	93	91	91
5	94	94	92	92
10	95	95	93	93
15	92	95	94	94
16	93	95	96	94

Table 5

The results of analysis of sodium chloride of pharmacopeia purity.

№	Found out elements, % / Sr (p = 0,95; n = 6)										
	Ca $\cdot 10^4$	Mg $\cdot 10^4$	Fe $\cdot 10^4$	Ba $\cdot 10^4$	K $\cdot 10^4$	$SO_4^{2-} \cdot 10^4$	$NH_4^+ \cdot 10^4$	pH	As	Pb	NaCI
1	3,20/ 0,045	0,42/ 0,065	0,91/ 0,055	4,22/ 0,069	1,43/ 0,035	22,1/ 0,031	2,90/ 0,045	7,0	0,12/ 0,047	0,031/ 0,058	99,8/0,027
1*	5,12/ 0,032	2,37/ 0,054	2,87/ 0,054	6,08/ 0,058	3,38/ 0,041	51,3/ 0,038	5,21/ 0,053	7,0	0,21/ 0,051	0,126/ 0,061	99,7/0,032
2	2,85/ 0,135	--	1,10/ 0,141	3,11/ 0,134	--	--	3,10/ 0,131	Neutral	Less than 0,5	Less than 0,05	Not less than 99,5
2*	5,53/ 0,139	2,32/ 0,127	2,93/ 0,142	2,39/ 0,135	--	--	4,79/ 0,141	Neutral	Less than 0,5	0,08/0,165	Not less than 99,5

References:

1. (2017) FS 42-25-78-88. Sodium chloride pharmacopeia.
2. Baklanov A.N. (2011) Analytichna chimia kuhonnoi soli ta rozsoliv. Kramatorsk, DDMA, 2011, -282 p.
3. Chmilenko F.A., Baklanov A.N. (1991) Izvestia vuzov SSSR.1991, V. 34. pp. 40-44.
4. Chmilenko F.A., Baklanov A.N. (1992) Izvestia vuzov SSSR.1992, V. 35. pp. 95-97.
5. Chmilenko F.A., Baklanov A.N. (2017) GOSTs 26927, 26930-26934
6. (1976) GOST 4212-76 Metody prigotovlenia standartnyh rastvorov.
7. (1984) GOST 13685-84 Sol pishevaa povarennaa. Metody ispytanii.
8. Frumina N.S. (1977) Analytichna chimia baria.Moskva, Nauka, 1977, 199 p.