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ATOMIC ABSORPTION DETERMINATION OF IRON AND MAGNESIUM IN PHARMACEUTICALS

Abstract: *The modern sample preparation of pharmaceuticals by decomposing an organic matrix by ultrasound and Triton X-100 was done. The influence of surfactant concentration and ultrasound treatment time on the sensitivity of Iron and Magnesium determination was studied. It was established that the most significant increase in the sensitivity of the atomic absorption determination of analytes was achieved at ω (Triton X-100) = 3% and ultrasound treatment time of 15 min.*

It was found out that the sensitivity enhancement for the determination of Magnesium increases by 1.5 and Iron by 1.8 times.

The content of Iron and Magnesium in such as medicaments "Analgin," "Caffeine," "Paracetamol" was determined by the atomic absorption method. The correctness of the results of atomic absorption determination of analytes by the "inserted-found" method was checked. It was shown that the systematic error is not significant. The detection limit of Iron ($C_{min} = 0{,}014 mg\ /\ l$) was estimated by the atomic absorption method. It appeared that limit is even lower than that presented in the literature data.

Key words: *Iron, Magnesium, atomic-absorption spectroscopy, sample preparation, ultrasound, Triton X-100, metrologic characteristics.*

Language: *English*

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Introduction

The pharmaceutical industry occupies one of the leading positions in the chemical industry of Ukraine. The pharmaceutical industry is developing rapidly. However, the public health issues, product quality, and the direct impact of pharmaceuticals on the body's vital functions are still poorly understood. The medical industry is characterized by a wide range of medicines. Among them, considerable attention is paid to those drugs that contain metals. The pharmacopoeial specifications require the definition of analytes in the structure of drugs. The pharmaceutical analysis includes control at all production stages: from the control of the raw materials to the standardization of the target products. This analysis creates an opportunity to study not only

individual medicaments but also mixtures containing many components. Pharmaceutical analysis has several areas depending on the analysis's objectives: pharmacopoeial analysis, dose formulation of particular drug components analysis, step-by-step control of drug production, rapid analysis in pharmacies, and biopharmaceutical analysis. For those issues, different physical, physicochemical, and biological methods are used. [1,p.24; 2,p.16; 3,p.11; 4,p.720; 5,p50; 6,p.39; 7,p.52; 8,p.38; 9,p.48; 10,p.5; 11,p.17; 12,p.9; 13,p.51; 14,p.55; 15,p.84]

Experimental

An atomic absorption spectrometer and CE333500 (flame version, hollow cathode lamps, acetylene-air flame) were used, Iron was determined at **λ** = 248.3 nm, and Magnesium at **λ** = 285.4 nm. The current used for both elements was I = 5mA, photoelectric pickup = 1.15 kV; the slit width of the monochromator was 0.1 nm. Laboratory weight scales ONAUS 64 (65 / 0.0001 g). Ultrasonic bath, model PS - 20, power - 120 W, frequency - 40 kHz. Triton X-100, $C_{14}H_{22}O$ $(C_2H_4O)_n$, where n = 9-10, Mr = 631 g/mol, CCM = $2.9 \cdot 10^{-4}$ mol/l. The initial concentration of metal solutions for the preparation of the calibration solutions is 0.1 g / l. The used distilled water and chemical reagents qualification not lower than analytically pure.

Results and discussions

We choose the surfactant concentration to increase the sensitivity of the atomic absorption determination of analytes.

As shown in Table 1, the most considerable value of the analytical signal was achieved at a concentration of Triton X-100 w = 5%.

As shown in Table 2, the most considerable value of the analytical signal was achieved at a concentration of Triton X-100 w = 5%.

As shown in Table 2.3, the most considerable value of the analytical signal was achieved at a concentration of Triton X-100 w = 5%.

As shown in Table 4, the most considerable value of the analytical signal was achieved by ultrasound treatment for 15 minutes

As shown in Table 5, the most considerable value of the analytical signal was achieved by ultrasound treatment for 15 minutes

As shown in Table 6, the most considerable value of the analytical signal was achieved by ultrasound treatment for 15 minutes

We constructed the dependence of the analytical signal of the analyte on its concentration. (Fig.1, 2)

The sensitivity factor is a numerical characteristic of sensitivity. If the graduated line is linear, then the sensitivity factor is defined as the tangent of the angle of inclination of the graduated curve. The sensitivity of the method is determined by the slope of the linear part of the graduated curve.

$$S = tg\alpha = \frac{dA}{dC} \quad (1)$$

$$\Delta S = \frac{tg\alpha_2}{tg\alpha_1}, \quad (2)$$

where

S - sensitivity,

ΔS – increasing of sensitivity,

$tg\alpha_1$ is the tangent of the angle of inclination of the graduated function of aqueous solutions,

$tg\ \alpha_2$ is the tangent of the angle of inclination of the graduated function with the modifier.

The value of the sensitivity coefficients according to formula 2.1. For Iron $\frac{tg\alpha_2}{tg\alpha_1}$ = 1.4, so the sensitivity increased by 1.2 times; for Magnesium $\frac{tg\alpha_2}{tg\alpha_1}$ = 1.5, so the sensitivity increased by 1.5 times.

The results of the atomic absorption determination of analytes by in samples of pharmaceutical substances

Firstly, the value of atomic absorption of samples should be determined. Next, the concentration of analytes g / l should be established from the graph. The following formula to calculate the concentration of metals in mg / kg is used:

$$C(mg/kg) = \frac{C(\frac{g}{l}) * V_f * 10^3}{m_{quant}}, \quad (3)$$

where

C $(\frac{g}{l})$ - the found concentration of the analyzed sample from the graduated graph,

V_f is the volume of the flask into which the sample was filtered,

m_{quant} - mass of the quantity of the sample.

Checking the correctness of the results of the atomic absorption determination of analytes by the "injected-found out"method.

The standard addition method is used in the analysis of samples with a complex chemical composition or in checking the correctness of the results of chemical analysis. The chemical and physical properties of the graduated solutions may differ from the properties of the samples that cause a systematic error.

Means of reducing the detection limit:

-usage of the maximum brightness of illumination of a source;

- the maximum possible width of a crack which does not lead to overlapping with other close lines;

-minimization of non-selective absorption;

-general optimization of the signal-to-noise ratio.

Multiple measurements of the absorption signal of the zero solution. Measures of 15-20 values of the digital recording device should be estimated, then the value of the standard deviation of the background by formula (4) should be calculated:

$$S_0 = \sqrt{\frac{\Sigma(\bar{A}-A)^2}{n-1}} \quad (4)$$

Calculation of the detection limit should be evaluated by the following formula (5):

$$C_{min} = \frac{3S_0}{S} \quad (5)$$

Thus, the obtained value of the detection limit of Iron is less than the one presented in the literature data.

Conclusions

1. The content of Iron and Magnesium in the substances of pharmaceuticals was determined by the atomic absorption method.
2. The influence of the concentration of Triton X-100 and ultrasonic treatment time on the signal value at atomic absorption determination of Iron and Magnesium were studied. It is shown that the maximum analytical signal for analytes is achieved by using Triton X-100 with a mass fraction of 5% and an ultrasonic treatment time of 15 minutes;
3. The most significant increase in sensitivity was determined at the addition of Triton X-100 (w = 5%), for Iron - 1.4 times, for Magnesium - 1.5 times.
4. The correctness of the results of atomic absorption determination of analytes by the "inserted-found" method was checked out. It is shown that the systematic error is not significant.
5. The limit of the detection of Iron (C_{min} = 0.014 mg / l) is lower than the one represented in the literature.

Table 1. Choice of concentration of Triton X-100 for increasing of the sensitivity of atomic absorption determination of Iron and Magnesium for the sample "Paracetamol" (n = 5, P = 0.95).

w(Triton X-100),%	C(Fe)mg/kg $C \pm \frac{t_p, S}{\sqrt{n}}$	S_r	C(Mg)mg/kg $C \pm \frac{t_p, S}{\sqrt{n}}$	S_r
0	106±1	0,01	859±11	0,01
3	127±2	0,01	3007±37	0,01
4	128±2	0,01	3283±41	0,01
5	149±2	0,01	3446±43	0,01
6	148±2	0,01	3444±43	0,01

Table 2. Selection of the concentration of Triton X-100 for increasing the sensitivity of the atomic absorption determination of Iron and Magnesium for the sample "caffeine" (n = 5, P = 0.95).

w(Triton X-100),%	C(Fe)mg/kg $C \pm \frac{t_p, S}{\sqrt{n}}$	S_r	C(Mg)mg/kg $C \pm \frac{t_p, S}{\sqrt{n}}$	S_r
0	56,7±0,7	0,01	656±8	0,01
3	67,5±0,8	0,01	2295±29	0,01
4	68,0±0,8	0,01	2495±31	0,01
5	79,4±0,9	0,01	2629±32	0,01
6	79,0±0,9	0,01	2627±33	0,01

Table 3. Selection of Triton X-100 concentration for increasing of the sensitivity of atomic absorption determination of Iron and Magnesium for the sample "Analgin" (n = 5, P = 0.95).

w(Triton X-100),%	C(Fe)mg/kg $C \pm \frac{t_p, S}{\sqrt{n}}$	S_r	C(Mg)mg/kg $C \pm \frac{t_p, S}{\sqrt{n}}$	S_r
0	61,1±0,8	0,01	1749±22	0,01
3	72,7±0,9	0,01	6121±76	0,01
4	73,3±0,9	0,01	6454±80	0,01
5	85,5±1,1	0,01	7013±87	0,01
6	85,3±1,1	0,01	7010±87	0,01

Table 4. Selection of ultrasound treatment time in the atomic absorption determination of iron and magnesium for the sample "paracetamol" (n = 5, P = 0.95).

US,min.	C(Fe)mg/kg $C \pm \frac{t_p, S}{\sqrt{n}}$	S_r	C(Mg)mg/kg $C \pm \frac{t_p, S}{\sqrt{n}}$	S_r
10	155±2	0,01	3470±43	0,01
15	159±2	0,01	3523±44	0,01
20	157±2	0,01	3508±44	0,01
25	155±2	0,01	3511±44	0,01
30	155±2	0,01	3518±44	0,01

Table 5. The choice of ultrasound treatment time in the atomic absorption determination of Iron and magnesium for the sample "caffeine" (n = 5, P = 0.95).

US,min.	C(Fe)mg/kg $C \pm \frac{t_p, S}{\sqrt{n}}$	S_r	C(Mg)mg/kg $C \pm \frac{t_p, S}{\sqrt{n}}$	S_r
10	82,7±1,0	0,01	2687±33	0,01
15	84,6±1,1	0,01	2745±34	0,01
20	80,0±0,9	0,01	2720±34	0,01
25	82,4±1,0	0,01	2734±34	0,01
30	83,9±1,0	0,01	2740±34	0,01

Table 6. Selection of ultrasound treatment time in the atomic absorption determination of Iron and Magnesium for the sample "Analgin" (n = 5, P = 0.95).

US,min.	C(Fe)mg/kg $C \pm \frac{t_p, S}{\sqrt{n}}$	S_r	C(Mg)mg/kg $C \pm \frac{t_p, S}{\sqrt{n}}$	S_r
10	89,1±1,1	0,01	7133±89	0,01
15	91,2±1,1	0,01	7195±89	0,01
20	89,2±1,1	0,01	7167±89	0,01
25	88,8±1,1	0,01	7174±89	0,01
30	90,5±1,1	0,01	7187±89	0,01

Table 7. Values of analytical signals of aqueous solutions of Iron and solutions of Iron with a modifier, treated with ultrasound (n = 5, P = 0.95).

C(Fe) g/l	Analytical signals of aqueous solutions of Iron	Analytical signals of aqueous solutions of Iron with a modifier, treated with ultrasound
0,0001	5	6
0,0003	14	17
0,0005	24	29
0,0007	34	41
0,001	50	60

Table 8. Values of analytical signals of aqueous solutions of Magnesium and solutions of Magnesium with a modifier treated with ultrasound (n = 5, P = 0.95).

C(Mg) g/l	Analytical signals of aqueous solutions of Magnesium	Analytical signals of aqueous solutions of Magnesium with a modifier, treated with ultrasound
0,0001	20	24
0,0003	57	71
0,0005	100	121
0,0007	143	166
0,001	200	242

Table 9. The results of atomic absorption determination of Iron using Triton X-100 (w = 5%), stabilized by ultrasound (treatment time 15 min) (n = 5, P = 0.95).

Medicine	Iron content, mg/kg $C \pm \frac{t_p, S}{\sqrt{n}}$	S_r
Paracetamol	159±2	0,01
Coffein	84,6±1,1	0,01
Analgin	91,2±1,1	0,01

Table 10. The results of atomic absorption determination of Magnesium using Triton X-100 (w = 5%), stabilized by ultrasound (treatment time 15 min) (n = 5, P = 0.95).

Medicine	Magnesium content, mg/kg $C \pm \frac{t_p, S}{\sqrt{n}}$	S_r
Paracetamol	3523±44	0,01
Coffein	2745±34	0,01
Analgin	7195±89	0,01

Table 11. Validation by "injected-found out " method of for Iron (n = 5, P = 0.95).

Medicine	Iron content, mg/kg	Iron injection, mg/kg	Iron found out, mg/kg	S_r
Paracetamol	159±2	150	310±4	0,01
Coffein	159±2	80	238±3	0,01
Analgin	84,6±1,1	80	165±2	0,01
	84,6±1,1	40	126±2	0,01
	91,2±1,1	90	180±2	0,01
	91,2±1,1	45	137±2	0,01

Table 12. Validation by " injected-found out " method of for Magnesium (n = 5, P = 0.95).

Medicine	Magnesium content, mg/kg	Magnesium injection, mg/kg	Magnesium found out, mg/kg	S_r
Paracetamol	3523±44	3500	7020±87	0,01
	3523±44	1750	5270±65	0,01
Coffein	2745±34	2700	5442±68	0,01
	2745±34	1350	4093±51	0,01
Analgin	7195±89	7000	14192±176	0,01
	7195±89	3500	10690±133	0,01

Table 13. Estimation of the limit of detection for Iron

№	A_1	A_2	A_3	A_4	A_5	A_6	$\bar{A}$	S_0	C_{min}(mg/l)
1	2	4	3	2	1	2	2,3	0,280	0,014
2	3	1	2	1	3	2	2,0		
3	2	3	2	3	2	3	2,5		
4	1	2	3	2	1	2	1,8		
5	2	4	3	2	1	2	2,3		
6	3	1	2	1	2	3	2,0		C_{lit}=0,015

7	2	3	2	3	2	3	2,5		
8	2	1	2	3	1	2	1,8		
9	4	2	3	2	1	2	2,3		
10	1	3	1	2	3	2	2,0		
11	3	2	3	2	3	2	2,5		
12	2	1	3	2	1	2	1,8		
13	1	3	1	2	3	2	2,0		
14	2	4	2	3	1	2	2,3		
15	2	3	2	3	2	3	2,5		
16	2	3	2	1	2	1	1,8		
17	3	2	1	2	1	2	1,8		
18	3	2	3	2	3	2	2,5		
19	2	1	3	2	4	2	2,3		
20	2	3	2	1	3	1	2,0		

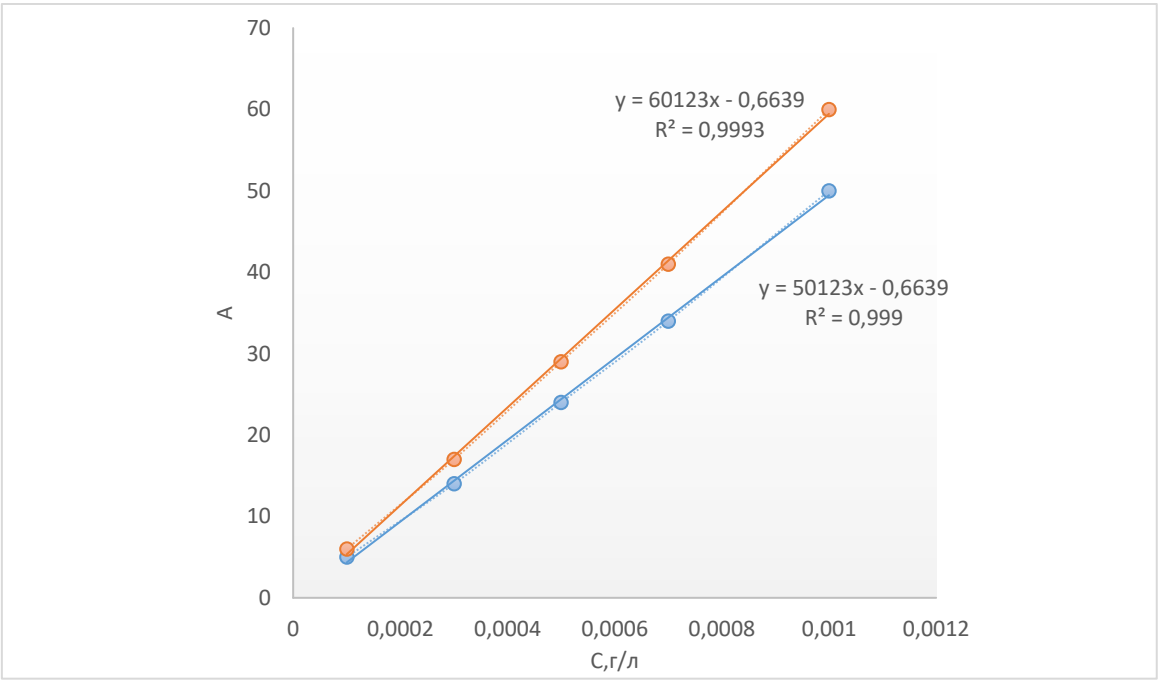

Fig. 1 Dependence on the concentration of Iron of the analytical signal of an aqueous solution of Iron and a solution of Iron with a modifier treated with ultrasound

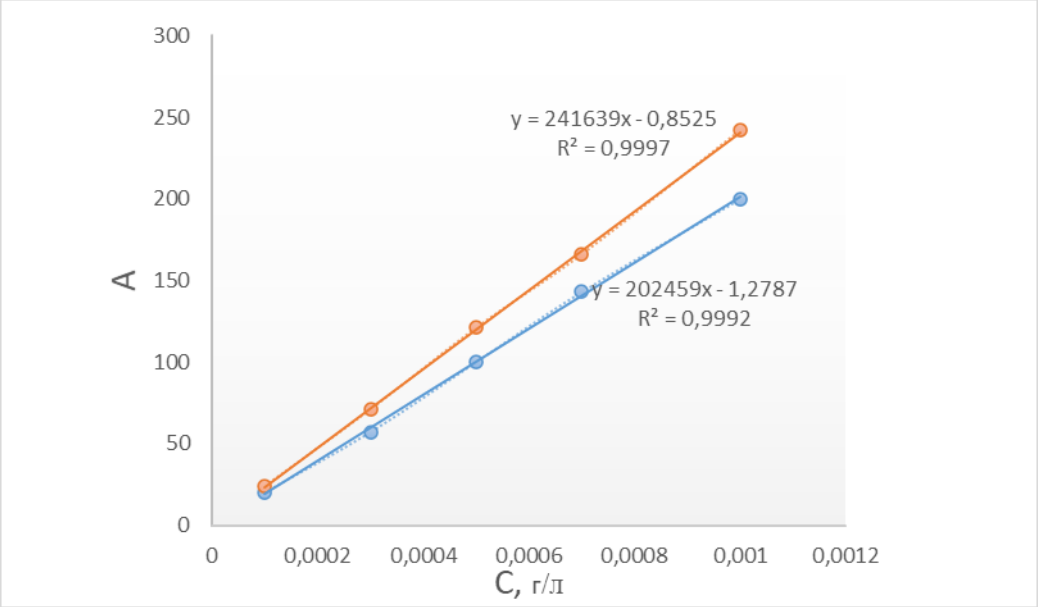

Fig. 2 Dependence on the concentration of Magnesium of the analytical signal of an aqueous solution of Magnesium and Magnesium solution with a modifier, under ultrasound treatment.

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