DEVELOPMENT OF THE METHOD FOR QUANTITATIVE DETERMINATION OF THE COMPOSITION OF Ag@Fe₃O₄ MAGNETIC NANOCOMPOSITE

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The method for quantitative determination of the components of the Ag@Fe₃O₄ nanocomposite has been developed; it allows simultaneously determining silver and iron in one sample without the stage of taking the aliquot for individual determinations of these components. The method proposed comprises: the use of a considerably smaller quantity of the substance in the test sample; elimination of the need to prepare and standardize the solution of the indicator for silver determination; reduces the labour intensity of the process by saving time and expensive reagents; eliminates the stage of separation of the mixture components, etc. The basis of the method proposed is two conjugated detection procedures –Ag determination by Volhard’s method and Fe (III) determination in magnetite by the method of iodometry. It has been shown that this method allows determining silver without adding the indicator since the second component is magnetite containing bivalent and trivalent iron. The experiment is performed by the action of nitric acid on the sample of Ag@Fe₃O₄ powder. The acid, in its turn, helps silver to pass into solution and to oxidize Fe²⁺ ions to Fe³⁺ being an indicator in this determination. With the simultaneous presence of silver and iron in one sample at first silver is quantified, a pale pink colour of the solution above the precipitate appears only after all the silver has been titrated, that means the completeness of its precipitation. The experiment is completed with determination of Fe (III) by iodometry. To assess the validity of determinations the results have been confirmed by instrumental methods that are consistent with the results of the titrimetric method developed for quantitative determination of components in magnetically controlled nanocomposites. The relative error of the titrimetric determination does not exceed 0.1-0.2%.

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Magnetic nanoparticles and composites on their basis are the most studied and widely used nanoscale materials for medicine and pharmacy, and it is associated with their remote control by applying an external magnetic field [1]. Appropriate sizes, low toxicity and stable magnetic characteristics [2, 3] make it possible to use them for biomedical research at the cellular level [4, 5].

The world science and industries are constantly working to improve new methods for the synthesis of nanosized particles of silver and create a great number of products for their application in various fields [6]. Physicochemical and biomedical properties of silver nanoparticles allow including them virtually in any materials without the loss of valuable features. Combining several materials with useful properties into one system creates new opportunities when developing drugs of a new generation. Applying silver to the magnetite surface in the form of islets [8] leads to formation of the composite of the "core-shell" type [7], and silver as a modifying agent gives the system the ability to be controlled by the external magnetic field with the simultaneous antibacterial effect. This multifunctional system is one of the layered magnetic composites [9], which in recent years are profoundly studied for using in biomedical and pharmaceutical purposes.

When studying composites of the given composition the question of quantitative determination of the system components using expedient, economical, and rapid methods arises.

To determine the qualitative and quantitative composition the classical volumetric and gravimetric methods of analysis are used [10]. The disadvantage of gravimetric methods is a significant time of the research conducting (from several hours to several days). Being inferior in accuracy to gravimetry, the titrimetric methods differ by rapidity and are characterized as kinetic methods for the quantitative determination of the composition that are the most common in analytical practice. Currently classic methods are gradually giving way to highly sensitive instrumental ones, but they, in turn, have a number of disadvantages when determining ultra-small amounts of components. However, titrimetry and gravimetry remain unsurpassed in accuracy: the relative error of the titrimetric determination rarely exceeds 0.1-0.2%, while the error of many instrumental methods is 2.5%. Therefore, classical methods remain to be standard for quantifying the reliability of determinations.

The study of nanoscale objects, improvement of the quality of the existing objects and creation of the new ones involves improvement of the instrumental base, the use of modern express methods of research.

Based on the analysis of classical and modern sources of literature data [11-13] on the use of the methods for quantitative determination of silver and iron with their simultaneous presence in the same sample by express gravimetric and titrimetric methods are almost unknown. Modern instrumental methods of determining the quantitative content of the components in the objects of study (X-ray diffraction analysis (XDA) [12], atomic absorption spectrometry (AAS) [13], etc.) are not always available since they require the use of specific equipment and skills, the purchase of expensive reagents, the use of special software for processing results, etc.

Currently, quantitative determination of silver and iron in one sample is carried out separately by suitable methods [14, 15].

The aim of the work is to develop an express and simple method for quantitative determination of the composition of Ag@Fe₃O₄ magnetic nanocomposite.

According to the method developed for quantitative determination of the composition of Ag@Fe₃O₄ nanocomposite it has the following essential advantages. They are: determination of silver and iron in one sample; determination of silver does not require the indicator since iron is a part of magnetite; preparation of the titrants does not take much time, they are prepared from the titration standard with the known concentrations; titration takes place in the acidic medium to prevent hydrolysis of iron (III) with formation of coloured products of hydrolysis; when determining iron (III) the colour becomes visible already in the concentration of 6.4×10⁻⁶ mol/L [17], and the indicator is quite sensitive in relation to thiocyanate ions.

The method proposed is performed according to the experimental procedure developed: place 0.0200 g (accurate weight) of Ag@Fe₃O₄ into a conical flask with glass stopper, add 10 ml of 6 M nitric acid, and boil to complete removal of a brown gas. Cool the solution, add 50 ml of deionized water and mix thoroughly. Titrate with 0.01M solution of KSCN, it is accompanied by formation of a white precipitate AgSCN (Ksp = 1.1×10⁻¹² mol/L – practically insoluble [18]) until a pink colour of the solution appears (formation of insoluble iron complexes) and record the end point.
After that add 1 ml of concentrated hydrochloric acid and 0.3 g of potassium iodide to the test solution, mix thoroughly, close with a glass stopper and leave for 10-15 min in a dark place. Then wash the stopper on the flask with deionized water and titrate the liberated iodine with 0.05М sodium thiosulphate solution until a pale yellow colour of the solution appears. After that add 2-3 drops of starch solution (the solution becomes blue), and the titration is continued with vigorous stirring to discoloration of the solution above the precipitate. Record the end point.

The results of determination of the correction factors to the titres of 0.1 М solutions of potassium thiocyanate and sodium thiosulphate are given in Tab. 1 and 2.

As can be seen from Tab. 1 and 2, both titrated solutions meet the requirements of monograph 4.2.2. “Titrated solutions” of the SPhU, ed. 1 [14], as well as the requirements for precision of the molarity specified. It allowed obtaining correct and accurate results.

Determination of the quantitative content of silver and iron in Ag@Fe$_3$O$_4$ magnetic composite was performed in 5 parallel tests, for which homogeneity of the sample of the results for quantitative determination of the components in the mixture was checked. The calculated values $Q_1$, $Q_5$ for both components in separate determinations (Tab. 3) and components in one sample (Tab. 4) do not exceed the tabular values $Q(P_{1,n})$ [14]. It has been found that the results of the experiment are clear of gross error.

### Table 1

| Parameters                                | Quantitative determination of silver | Quantitative determination of iron |
|-------------------------------------------|-------------------------------------|------------------------------------|
| $x_i,\%$                                  | 1 2 3 4 5                           | 1 2 3 4 5                          |
| $R$                                       | 0.13                                | 2.98                               |
| $Q_1$                                     | 0.38                                | 0.16                               |
| $Q_5$                                     | 0.23                                | 0.33                               |
| $Q(P_{1,n})$                              | 0.64                                | 0.64                               |

### Table 2

| Parameters                                | Quantitative determination of KSCN | Quantitative determination of Na$_2$S$_2$O$_3$ |
|-------------------------------------------|-----------------------------------|-----------------------------------------------|
| $x_i,\%$                                  | 20.00                             | 19.95 0.9930                                  |
| $R$                                       | Mean $R$ 0.9922                   | Mean $R$ 1.0430                              |
| $SD_x$                                    | 0.0008                            | 0.0006                                        |
| $RSD_x$ %                                 | 0.08                              | 0.06                                          |
| $\Delta x$ (%)                           | 0.18                              | 0.11                                          |
| Compliance with the requirements of the SPhU $\Delta x\leq0.2\%$ | 0.18$\leq0.2$                    | 0.11$\leq0.2$                                |

### Table 3

| Parameters                                | Quantitative determination of silver | Quantitative determination of iron |
|-------------------------------------------|-------------------------------------|------------------------------------|
| $x_i,\%$                                  | 37.09 37.14 37.17 37.19 37.22       | 52.16 52.63 54.15 54.17 55.14      |
| $R$                                       | 0.13                                | 2.98                               |
| $Q_1$                                     | 0.38                                | 0.16                               |
| $Q_5$                                     | 0.23                                | 0.33                               |
| $Q(P_{1,n})$                              | 0.64                                | 0.64                               |

### Table 4

| Parameters                                | Quantitative determination of silver | Quantitative determination of iron |
|-------------------------------------------|-------------------------------------|------------------------------------|
| $x_i,\%$                                  | 36.72 36.97 37.48 37.48 37.49       | 53.10 53.88 54.53 54.64 55.31      |
| $R$                                       | 0.77                                | 2.21                              |
| $Q_1$                                     | 0.32                                | 0.35                              |
| $Q_5$                                     | 0.01                                | 0.30                              |
| $Q(P_{1,n})$                              | 0.64                                | 0.64                              |
Metrological characteristics of the average result of quantitative determination of silver by thiocyanometry

| n  | Sample weight | The volume of KSCN 0.01 M titrant | The silver content in % (Xi, %) |
|----|---------------|----------------------------------|--------------------------------|
| 1  | 0.0200        | 6.90                             | 37.14                          |
| 2  | 0.0199        | 6.85                             | 37.22                          |
| 3  | 0.0199        | 6.85                             | 37.19                          |
| 4  | 0.0200        | 6.85                             | 37.09                          |
| 5  | 0.0200        | 6.90                             | 37.17                          |
| Mean, Xi (%) |                         |                                  | 37.16                          |
| Standard deviation, s |                          |                                  | 0.0497                         |
| Dispersion, s² |                          |                                  | 0.0081                         |
| Relative standard deviation (in relation to the average result), sr |                          |                                  | 0.0013                         |
| Relative standard deviation, RSD₀ (%) |                          |                                  | 0.13                           |
| Standard deviation of the average result |                          |                                  | 0.0222                         |
| Relative standard deviation of the average result |                          |                                  | 0.0006                         |
| Relative standard deviation of the average result in %, RSDₓ (%) |                          |                                  | 0.05981                        |
| The numerical value of Student's coefficient t (95%, 4) |                          |                                  | 2.7764                         |
| The half-width of the confidence interval of the average result Δx |                          |                                  | 0.06                           |
| The half-width of the relative confidence interval of the average result Δₓ,r |                          |                                  | 0.0017                         |
| The relative uncertainty of the average result, % |                          |                                  | 0.17                           |

Metrological characteristics of the average results are presented in Tab. 5 (for silver), 6 (for iron) and 7 (for silver and iron in one sample); it has been shown that the method gives reproducible results.

To compare the methods for the simultaneous and separate quantitative determination of silver and iron (III) Fischer test $F$ was calculated. Its comparison with the tabular value of the distribution percentage point $F(\nu_1, \nu_2)$ [14] shows that the discrepancy between the values $S^2_1$ and $S^2_2$ is not significant in both cases – for silver and iron, respectively ($S^2_1$ and $S^2_2$ – are dispersions of separate and simultaneous determinations), the reproducibility of both series are the same (dispersions are homogeneous).

The data of the results of the study of Ag@Fe₃O₄ nanocomposite obtained earlier by the methods of XDA and AAS [19] were compared with the results of quantitative determination of silver and iron by titrimetry. Analysis of the results of the content of magnetite in the sample took into account the gravimetric form (magnetite) and the analyte (iron III), for this purpose the analytical factor – 1.37 was determined [20].

Tab. 8 gives the comparative characteristics of the results obtained by different methods. The discrepancy between the percentage of the main components in the composite does not exceed 7-10% for magnetite and 10-20% for silver.

The results of determination of the quantitative content of Ag@Fe₃O₄ nanocomposite by the method proposed confirm the given core/shell molar ratio of 1 : 0.5 previously determined [21]. Therefore, this method can be recommended for use when developing the quality control methods for the substance mentioned above.

**Experimental Part**

The quantitative determination of silver and iron in the Ag@Fe₃O₄ nanocomposite under research by
the kinetic method was performed using two approaches: 1) separately, in individual samples; 2) simultaneously in one sample.

Silver was determined using thiocyanometry by Volhard’s method [16]. The indicator in this method is Fe$^{3+}$ ions, which forms soluble, red complex ions ([Fe(SCN)]$^{4-}$, [Fe(SCN)]$^{3-}$, [Fe(SCN)]$^{2+}$, [Fe(SCN)]$^{2+}$) with SCN$^-$ ions in the end point of titration. The thiocyanate complexes of iron (II) are not formed to the end point since thiocyanate ions, first of all, bind to AgSCN.

Before starting the experiment the control experiments were carried out. The first experiment for determination of silver – the sample contained magnetite nanoparticles and other necessary components except silver. The second experiment for determination of iron – the sample contained silver nitrate and the necessary components except iron.

Determination of the composition of nanoparticles was started with quantitative determination of silver. The accurately weighed quantity of the sample

### Table 7

Metrological characteristics of the average result of quantitative determination in the simultaneous presence of silver and iron (III) in one sample

| n | Sample weight | The volume of KSCN 0.01 M titrant | The volume of Na$_2$S$_2$O$_3$ 0.05 M titrant | The silver content in % (Xi, %) | The content of iron (III) in % (Xi, %) |
|---|--------------|-------------------------------|---------------------------------|---------------------------------|-------------------------------------|
| 1 | 0.0201       | 6.95                          | 3.85                            | 37.48                            | 55.31                               |
| 2 | 0.0203       | 6.75                          | 3.90                            | 37.37                            | 54.53                               |
| 3 | 0.0199       | 6.80                          | 3.90                            | 37.15                            | 54.64                               |
| 4 | 0.0200       | 6.95                          | 3.85                            | 37.49                            | 53.88                               |
| 5 | 0.0202       | 6.95                          | 3.80                            | 37.48                            | 53.10                               |

Mean, Xi (%) 37.39 54.29

Standard deviation, $s$ 0.1450 0.8374

Dispersion, $s^2$ 0.0210 0.7012

The numerical value of Fisher test $F$ (99%, 5) 15.98

Fisher test, $F$ 2.5963 2.1397

Relative standard deviation (in relation to the average result), $sr$ 0.0039 0.0154

Relative standard deviation, RSD$_s$ (%) 0.39 1.54

Standard deviation of the average result 0.0644 0.3745

Relative standard deviation of the average result 0.0017 0.0069

Relative standard deviation of the average result in %, RSD$_x$ (%) 0.1734 0.6897

Confidence probability, $P$ 95%

The numerical value of Student’s coefficient $t$ (95%, 4) 2.7764

The half-width of the confidence interval of the average result $Δx$ 0.18 1.04

The half-width of the relative confidence interval of the average result $Δx_r$ 0.0048 0.0192

The relative uncertainty of the average result, % 0.48 1.92

### Table 8

The comparison of the results of the quantitative content of silver and iron (III) in Ag@Fe$_3$O$_4$ nanocomposite

| Method | Analytical signal | The content of components, % | Accuracy of measurements, % |
|-------|-------------------|------------------------------|----------------------------|
| XDA   | The phase composition$^1$ Fe$_3$O$_4$, JCPDS # 88-315 Ag JCPDS # 87-717 | The phase content, w % 67 33 | ±2.5% |
| AAS   | Analytical lines$^2$ Fe # 248.3 nm, calculated with reference to Fe$_3$O$_4$ Ag # 328.1 nm | Content, % 51±2 69.87±2 29.6±1.5 | 3-5% |
| Titrimetry | The end point$^3$ Fe – discoloration of the solution, calculated with reference to Fe$_3$O$_4$ Ag – precipitate AgSCN | Content, % 54.29±2.0 74.38±2.0 37.17±0.3 | 0.01%±0.02% |

Notes: 1. The number of substance according to the X ray card-file in the JCPDS (the Joint Committee of Powder Diffraction Standards) database of powder diffraction standards, PDF2. 2. Resonance lines (absorption signal) in the spectral range that is characteristic for the element. 3. The colour change of solutions, precipitation.
A white precipitate Ag\textsubscript{SCN} was formed. The titration was stopped when a stable pale pink colour of the solution appeared; it indicated the complete precipitation of silver ions. To the resulting solution 1 ml of the concentrated hydrochloric acid was added by a micropipette, then potassium iodide was added, thoroughly mixed, closed with a glass stopper and left for 10-15 min in a dark place. Then the stopper on the flask was washed with deionized water, and the liberated iodine was added. The molar ratio of the core/shell.

The affordable, simple, efficient, sensitive and reliable method for quantitative determination of the composition of the magnetic nanocomposite of \text{Ag@Fe}_3\text{O}_4 type has been developed. Using it silver and iron (III) can be identified in a single sample; and it can be recommended for use when developing the quality control methods for \text{Ag@Fe}_3\text{O}_4. The content of silver (37.17±0.3) and iron (III) (calculated with reference to \text{Fe}_3\text{O}_4 – 74.38±2.0) confirms the given molar ratio of the core/shell.

**Conclusions**

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