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ARTICLES

DETERMINATION OF GALLIUM BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY IN NICKEL- AND IRON-BASED ALLOYS WITH PRELIMINARY SEPARATION FROM THE MATRIX

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Abstract. A procedure is proposed for the separation of gallium micro-component and macro-components Cr, Mo, W, Ni and Co for the determination of gallium by inductively coupled plasma atomic emission spectrometry in nickel and iron-based alloys (precision nickel alloys, alloy and high-alloy steel). Sodium fluoride was used as a precipitator. Optimal conditions for the co-deposition of gallium on the sediment during separation from the macro components under consideration have been determined. The detection limit of gallium upon separation from macro quantities of Cr, Mo, W, Ni, and Co was 5×10^{-4} wt.%.

Keywords: *inductively coupled plasma atomic emission spectrometry, coprecipitation, gallium*

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Gallium is a rare element and belongs to the scattered ones, since it practically does not form its own mineral phases, with the exception of some very rare minerals, and is found in nature mainly in the form of impurities in rocks and minerals [1]. Gallium is in demand in high-tech industries such as electronics, energy, laser, and medical technologies [2, 3].

Nickel and iron-based alloys (precision nickel alloys, alloy and high-alloy steel), due to their excellent high-temperature properties combined with corrosion resistance, are widely used in jet and gas turbine engines where high strength at high temperatures is required [4]. The quality of these alloys depends to a large extent on the amount of trace elements present, as they can seriously affect the mechanical and physical properties of the material. Thus, the presence of gallium and its compounds can lead to premature destruction of alloys based on iron and nickel [5].

In most cases, gallium is contained in alloys as a trace element with a content of from thousandths to hundredths of a percent (by weight), but there are alloys with a gallium content of ten thousandths of a percent.

Currently, the determination of gallium content in alloys and concentrates is regulated by state standards (GOST standards) [6, 7], which use methods of spectrophotometry and atomic absorption spectrometry for analysis. The proposed spectrophotometric methods for determining the gallium content are laborious and involve the use of organic solvents such as butyl acetate and benzene.

Spectral analysis methods such as atomic absorption spectrometry [8, 9], inductively coupled plasma mass spectrometry (**ICP-MS**) [10-12], and inductively coupled plasma atomic emission spectrometry (**ICP-AES**) are used to determine gallium in natural waters, coals, rocks, and alloys [13, 14].

One of the sensitive methods for determining gallium is ICP-MS. In [10], the content of Ga, Ge, As, and Se in certified standard samples of nickel alloys microalloyed with rare earth metals was determined using the ICP-MS method. The detection limit of gallium was 2×10^{-6} wt.%. However, the authors note that the results of the determination of micro-impurities can be influenced by spectral interferences, which in this paper it is proposed to eliminate using mathematical correction equations. It is worth noting that the use of ICP-MS is not always possible, including due to the high cost of equipment.

Earlier, we found that the analytical spectral lines of gallium Ga I 294.364, Ga I 403.298, Ga I 417.206 are subject to the least spectral matrix influence from macro components (Fe, Cr, Mo, W, Ni, and Co) during the ICP-AES determination of gallium in metallurgical materials [15]. In Table 1, the wavelengths of the analytical spectral

Table 1. Comparison of the wavelengths of analytical gallium lines and lines of macro components

Analytical spectral line of gallium, nm	Relative intensity	Background correction (on the left), nm	Analytical spectral line of the interfering element (on the left), nm	Background correction (on the right), nm	Analytical spectral line of the interfering element (on the right), nm
Ga I 287.424 [16]	50 [16]	287.390	Cr II 287.406 [17] Fe I 287.417 [17] Co I 287.388 [17]	287.449	Fe I 287.430 [18]
Ga I 294.364 [16]	50 [16]	294.324	W I 294.333 [17] Mo II 294.338 [17] Co I 294.348 [17] Cr II 294.364 [18]	294.404	Ni I 294.391 [16] W I 294.396 [17] Mo II 294.399 [18]
Ga I 294.418 [16]	50 [16]	294.321	W I 294.333 [18] Mo II 294.481 [16] Co I 294.348 [16] Cr II 294.364 [18] Ni I 294.391 [16] W I 294.396 [17] Mo II 294.399 [18]	294.351	Mo I 294.421 [17] W I 294.439 [17] Fe II 294.440 [17] Mo II 294. 482 [17] Fe I 294.505 [17] Cr I 294.511 [17] Fe II 294.526 [17]
Ga I 403.298 [16]	100 [16]	403.199	W I 403. 238 [17] Fe I 403.246 [17] Mo I 403.250 [18] Fe I 403.263 [17]	403.349	Cr I 403.326 [17]
Ga I 417.206 [16]	100 [16]	417.025	W I 417.054 [16] Co I 417.090 [18] Fe I 417.091 [17] Mo I 417.107 [18] W I 417.052 [17] Cr I 417.168 [17] Fe I 417.104 [17]	417.303	Fe I 417.212 [17] Fe I 417.264 [17] Fe I 417.274 [17] Cr I 417.277 [17]

lines of gallium and the macro components under consideration are compared [16-18].

To eliminate matrix influences and reduce the detection limits of gallium in various objects, separation and concentration methods are usually used – liquid-liquid extraction [8, 19], turbidity point extraction [20], solid-phase extraction [21], co-precipitation [22].

Thus, despite the progress in equipping analytical laboratories with instruments (ICP-AES-, ICP-MS-, AAS-spectrometers), there is a need to develop a universal, simple, rapid and economical procedure for the preliminary separation of gallium from the main components of metallurgical materials (precision nickel alloys, alloy and high-alloy steel).

Previously, we proposed the ICP-AES methods for the determination of As, Sb, Se, and Te microcomponents in metallurgical materials with pre-deposition of the matrix with various precipitators [23-26]. This approach can be used in the preparation of metallurgical materials for ICP-AES-gallium determination.

The purpose of this work *is* to develop a procedure for the deposition and separation of macro components, which makes it possible to carry out AES–ICP-determination of gallium content in precision nickel alloys, alloyed and high-alloy steels.

METHODS AND MATERIALS

Reagents and objects of research. Salts $FeCl_3{\cdot}6H_2O$, $CrCl_3{\cdot}6H_2O$, $Na_2MoO_4{\cdot}2H_2O$, $Na_2WO_4{\cdot}2H_2O$, $NiCl_2{\cdot}6H_2O$, $CoCl_3{\cdot}6H_2O$ AR grade were used to prepare the solutions. Solutions for calibration of the ICP-AES spectrometer were prepared by diluting standard samples of the composition of solutions of Ga(III) ions (1000 micrograms/ml), background – 7% HNO_3, Fe(III) CRM 7766-2000, Cr(III) CRM 7257, Mo(VI) CRM 8086-94, W(VI) MSO 0542:2003, Ni(II) CRM 7265 and Co(II) CRM 7268, Na(I) CRM 7474, F(I) CRM 7261. The state reference samples (**RS**) of nickel-based alloys of types KhN62MVKY (N13g), KhN65MV (N12v), and high-alloy steel of type PXN28MDT (ISO C66) were used.

Preparation of solutions. Solutions for the development of a method for precipitation and separation of macro components were prepared according *to procedure 1*: 15 ml of a solution of Fe(III), Cr(III), Mo(VI), W(VI), Ni(II), Co(II) ions with a concentration of 10,000 mg/l and 5 ml were added to heat-resistant fluoroplastic cups solution of Ga(III) ions with a concentration of 50 mg/l. 20 ml of conc. HCl (5 ml, 37 wt.%), HNO_3 (15 ml, 95 wt.%) and HF (from 1 to 15 ml, 37.5 wt.%) were added. The resulting solutions were heated to boiling (T = 75°C). To separate the matrix at pH 0-1.8 on indicator paper, the pH was first adjusted to a value in the range of 0-2.0, adding 2 M NaOH solution in portions with constant stirring, then 0 to 15 g of NaF was added to the resulting solution in portions with constant stirring, and held for 10 minutes. The precipitate was filtered out through a “white ribbon" filter. The filtrate was transferred to a 250 ml polypropylene measuring flask. The precipitate was dissolved on a filter using hydrochloric acid (8 ml, 37 wt.%) and nitric acid (17 ml, 95 wt.%%) and hydrofluoric acid (8 ml, 37.5 wt.%) when heated (T = 75 °C). All experiments were carried out in five parallels. The obtained solutions were analyzed for the content of the components by the ICP-AES method.

The CO solutions were prepared according *to procedure 2*: a 0.5 g bulk of the material was placed in heat-resistant fluoroplastic cups and dissolved upon heating (in five parallels) in a mixture of conc. HCl (5 ml, 37 wt.%), HNO_3 (15 ml, 95 wt.%) and HF (1 ml, 37.5 wt.%), additives of Ga(III) solution of known concentration were previously introduced. The solutions were cooled. An additional aliquot of 11 ml of iron Fe(III) solution (12 g/l) was added to standard nickel alloy samples, and the pH value was adjusted to 0-0.3, adding 2 M NaOH solution in portions with constant stirring. Then, 10 g of NaF was added to the resulting solution in portions with constant stirring, and kept for 10 minutes. Next, the precipitate was filtered out through a “white ribbon" filter. The precipitates were dissolved on a filter using nitric acid (16 ml, 95 wt.%), hydrochloric acid (8 ml, 37 wt.%), and hydrofluoric acid (8 ml, 37.5 wt.%) and a 250 ml solution was obtained. The obtained solutions were analyzed for gallium content by the ICP-AES method. To determine the elements in the filtrate and in the solution from the precipitate, the expression for the relative content of c_r/c_0 was used, where c_r is the concentration of the element after co–precipitation on the precipitate from the solution; c_0 is the initial concentration of the element in the solution.

The error of the analysis was calculated by the equation:

$$t_{(0{,}95;4)} = \frac{s}{\sqrt{n}}, \qquad (1)$$

where t is Student's coefficient (for a two–way distribution) equal to 2.78 for n = 5 and P = 0.95, s is the standard deviation.

Blank solutions containing all components in the same concentrations, except Ga(III) ions, were prepared simultaneously with the studied solutions.

The effect of sodium and fluorine on the analytical spectral lines of gallium was studied in model solutions. The solutions were prepared according to *procedure 3*: 5, 10, 15, 20, 25 ml of solutions of Na^+ or F^- ions with a concentration of 1000 mg/l and 1 ml of Ga(III) solution with a concentration of 50 mg/l were placed in a 50 ml measuring flask. The volume was adjusted to the mark with distilled water.

The manifestation of the matrix effect of sodium and fluorine on the intensity of the spectral lines of gallium was calculated by the equation:

$$\gamma = \frac{(I_i - I_0)}{I_0} \cdot 100\%,$$

where I_0 is the intensity of the spectral line of gallium in an aqueous solution, I_i is the intensity of the spectral line of gallium in the presence of sodium or fluorine.

Conditions for measuring atomic emissions of Fe, Cr, Mo, W, Ni, Co, Ga. The Optima 2100 DV ICP-AES spectrometer (Perkin Elmer) with a quartz burner was used. Operational parameters of the spectrometer: high–frequency power – 1500 W, argon sampling flow – 0.75 l/min, auxiliary argon flow – 0.2 l/min, plasma–forming argon flow – 15.0 l/min, plasma observation method – radial, observation height - 15 mm, solution feed rate - 0.9 ml/min, spray time the time of the sample is 40 seconds, the number of measurements with the device of one sample is 3. A spray system resistant to the aggressive effects of hydrofluoric acid was used. Analytical spectral lines: Ga I 294.364, Fe I 302.107, Cr II 206.158, W II 207.912, Mo II 202.031, Ni II 231.604, Co II 228.616 and Cu I 327.393 nm, Fe II 238.204 nm, Cr II 267.716 nm, Mo II 202.031 nm, W II 207.912 nm, Ni II 231.604 nm, Co II 228.616 nm.

Calibration of the spectrometer. Solutions for calibration of the spectrometer were prepared by diluting state standard samples of the composition of solutions of ions Ga(III), Fe(III), Cr(III), Mo(VI), W(VI), Ni(II), Co(II). Aliquots of the blank solution were added to them so that the salt composition corresponded to the composition of the analyzed solutions.

X-ray phase analysis. The precipitation obtained was examined by X-ray diffraction analysis (**XDA**). RDA was performed using a Shimadzu XRD-7000 diffractometer (Japan) in Cu*K*a radiation using a graphite monochromator. X–ray tube power is 2 kW, voltage $U = 40.0$ kV, current $I = 30.0$ mA, focus size 1.0 × 10 mm, external standard is silicon powder. The diffraction spectrum was recorded in the angular range of $2\Theta = 2\text{-}80°$ in stepwise mode with a scanning step of $\Delta\Theta = 0.03°$ and a pulse accumulation duration of 2 s.

For qualitative and quantitative analysis, the international ICDD (The International Center for Diffraction Data) file, PDF–2 version, was used, which includes a software package for primary processing of diffractograms, automatic phase search and quantitative analysis. The built-in semi-quantitative analysis program uses the corundum number method.

Microscopic examination. Microscopic studies – scanning electron microscopy (**SEM**) and electron probe X–ray spectral microanalysis (**XMA**) of precipitation were performed on a Carl Zeiss EVO 40 scanning electron microscope with an INCA X-Act energy dispersion spectrometer (Oxford Instruments, Great Britain). For quantitative elemental analysis, tablets were prepared from the initial precipitates in a mold with a diameter of 1 cm and a pressure of 40 kN/cm^2. The surface of the tablets under study was sanded with fine sandpaper before measurements.

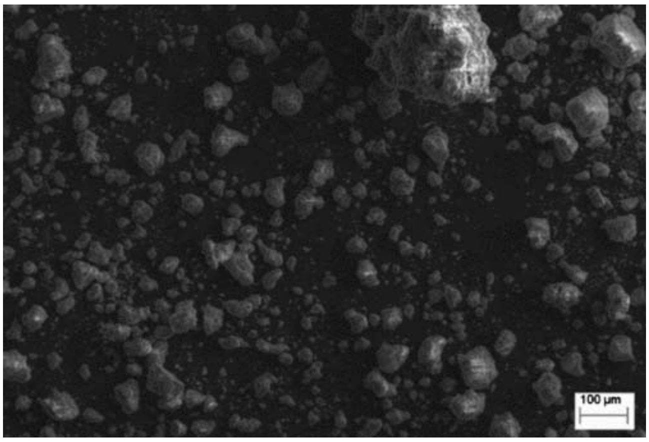

Fig. 1. Images of sediment obtained on a scanning scanning microscope using the Everhart–Thornley detector. Precipitation conditions: pH 0.3, 10 g NaF, 1 ml HF (37.5 wt%).

DISCUSSION OF RESULTS

Procedure of deposition and separation of macro components Cr, Mo, W, Ni, Co from Ga. In the studies, sodium fluoride (NaF) was used as a precipitator, which precipitates iron under certain conditions [23-26]:

$$Fe^{3+} + 6NaF = Na_3FeF_6\downarrow + 3Na^+. \tag{2}$$

According to procedure 1 at pH 0.3 using 10 g of NaF and HF (1 ml, 37.5 wt.%) (see Experimental Part) was obtained by precipitation, which was studied by X-ray diffraction and microscopy.

Table 2 shows the data of the quantitative phase analysis of the sediment obtained using procedure 1. According to the results of the RDA, it was established that the main precipitation phases are $NaHF_2$, Na_3FeF_6, NaF.

Table 2. Phase composition of the precipitate according to X-ray diffraction analysis (deposition conditions: pH 0.3, 10 g NaF, 1 ml HF (37.5 wt%))

Phase and corresponding reference	Content, wt. %
$NaHF_2$, PDF – 00-006-0479	56.46
Na_3FeF_6, PDF – 01-072-1916	5.83
NaF, PDF – 00-071-4667-1455	36.95
$Na_2Fe_2F_7$, PDF – 00-024-1098	0.76

The SEM results showed that the studied sediments

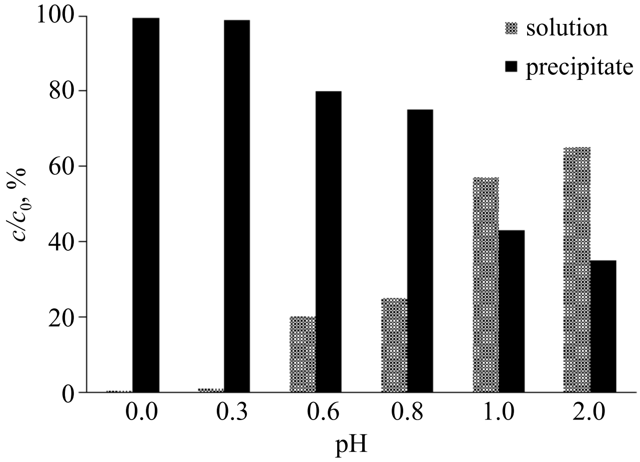

Fig. 2. Relative gallium content in the filtrate and in the sediment solution with varying pH. Conditions: $c_{\mathrm{Fe(III)}}$ = 500 mg/L, $c_{\mathrm{Ga(III)}}$ = 1.0 mg/L, n_{NaF} = 0.25 mol, n_{HF} = 0.45 mmol, 25 °C, V_{sol} = 250 ml.

consist of agglomerates measuring 5-200 microns (Fig. 1). There is an accumulation of charge on the surface of the agglomerates in the form of white dots and stripes due to the poor electrical conductivity of the compounds under study.

Using the XRF method at a point when focusing an electron beam on the surface of various agglomerates, it was possible to establish the elemental composition at a qualitative level. Intense reflexes related to Na, F, and Fe are observed on the energy spectra, as well as W, Mo, Cr, and Ga.

The results of the quantitative analysis showed that the samples are a mixture of at least three phases. The elemental composition of these phases was determined by the XRF method (Table 3). The sediment composition according to the XRF data is consistent with the XRD results.

Optimal conditions for the co-deposition of gallium on the sediment. The effect of pH on the co-precipitation of gallium was investigated. Figure 2 shows the relative contents of gallium in filtrates and in sediment solutions, depending on the pH after the dissolution procedure (hereafter, before the ICP-AES analysis, all sediments were preliminarily converted into solution by dissolving in a mixture of nitric, hydrochloric and hydrofluoric acids (3 :1 :1), see Experimental Part).

No iron was found in the filtrate after the co-precipitation procedure, therefore, all the iron is consumed to form a Na_3FEF_6 precipitate. The upper limit of the pH range was chosen taking into account the pH of precipitation of iron(III) and gallium(III) hydroxides (2.0 and 1.6, respectively). With an increase in pH in the range from 0.6 to 2.0, the degree of co-precipitation of gallium on the sediment decreases. In this case, all the macro components under consideration (Mo, W, Ni, Co), except chromium, pass into the filtrate in the pH range from 0 to 2. Chromium at low pH values

Table 3. Elemental composition (wt.%) of sediment phases (deposition conditions: pH 0.3, 10 g NaF, 1 ml HF (37.5 wt.%))

Sediment (procedure 1)	F	Na	Cr	Fe	Mo	W	Ga
Phase 1	54.20	25.00	1.10	19.30	0.10	0.20	0.10
Phase 2	54.20	45.80	-	-	-	-	-
Phase 3	66.00	33.60	-	0.40	-	-	-

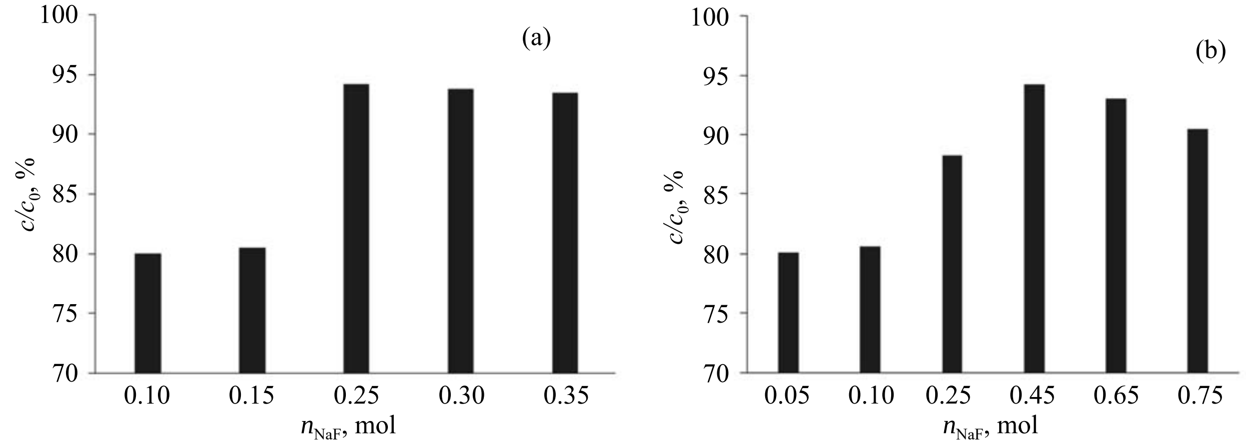

Fig. 3. Relative content (% of the initial) of gallium in the precipitate solution depends on the amount of precipitator NaF (a) and the amount of HF (b). Other conditions: $c_{\mathrm{Fe(III)}}$ = 500 mg/L, pH 0.25 °C, V_{sol} = 250 ml.

Table 4. Results (c_{Ga}, wt.%) ICP-AES-determination of gallium in nickel alloys and high-alloy steel with preliminary separation using certified mixtures and procedures injected–found ($n = 5$, $P = 0.95$)

Reference standard	Injected, $c_{Ga} \times 10^3$	Found, $c_{Ga} \times 10^3$
CRM 1479-91P N13g	2.5	2.60 ± 0.06
	5.0	5.5 ± 0.1
	10.0	11.0 ± 0.3
	15.0	16.0 ± 0.40
	25.0	22.0 ± 0.7
CRM 1519-90P N12v	2.5	2.40 ± 0.05
	5.0	4.70 ± 0.1
	10.0	12.00 ± 0.2
	15.0	17.00 ± 0.4
	25.0	26.00 ± 0.6
CRM 10214-2013 ISO C66	2.5	2.70 ± 0.05
	5.0	5.7 ± 0.1
	10.0	11.0 ± 0.3
	15.0	16.0 ± 0.5
	25.0	29.0 ± 0.7

from 0 to 0.6 is almost entirely contained in the filtrate (95 wt.% of the initial value), and with an increase in the pH value to 2, it partially becomes a precipitate (content of 60 wt.% of the original value). Thus, for the gallium co-precipitation procedure, the recommended optimal pH range is 0-0.3 (the gallium content in the filtrate is less than 1 wt.% of the original value).

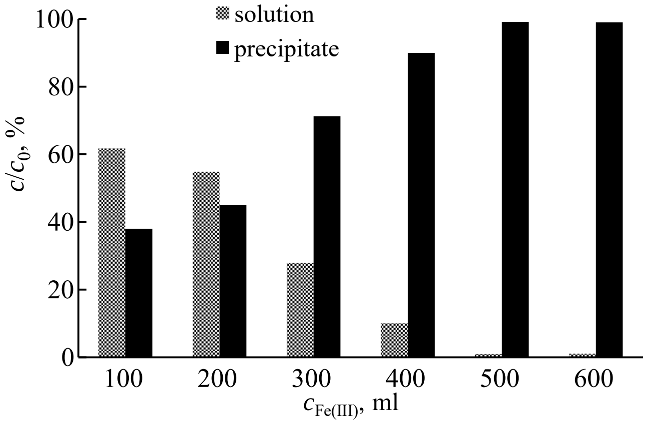

Fig. 4. Relative content (% of the initial) of gallium in the filtrate and in the precipitate (after dissolution) depends on the concentration of the co-precipitator Fe(III). $c_{Ga(III)}$ = 1.0 mg/l, n_{NaF} = 0.25 mol, n_{HF} = 0.45, pH 0.25 °C, V_{sol} = 250 ml.

The effect of the amounts of sodium fluoride and hydrofluoric acid on the process of co-precipitation of gallium on a precipitate during the separation of Cr, Mo, W, Ni and Co was investigated. The results of the ICP-AES analysis of sediment solutions obtained after the separation of macro components are shown in Fig. 3.

It was found that at concentrations of n_{NaF} from 0.1 to 0.25 mol and n_{HF} from 0.05 to 0.45 mmol (here n is the amount of substance), the co–precipitation of gallium is incomplete due to a lack of precipitator components (see Equation (2)). Probably, the collector for the co-deposition of gallium is Na_3FeF_6, which is related to the proximity of the ion radii of gallium and iron (0.076 and 0.075 nm, respectively) [27]. Thus, under the selected precipitation conditions (n_{NaF} = 0.25 mol, $n_{HF =}$ 0.45 mmol), gallium co-precipitates on a precipitate of the composition Na_3FeF_6 (the content in solution after dissolution of the precipitate is more than 95 wt.%).

The effect of the concentration of iron(III) on the process of co-precipitation of gallium has been studied. It can be seen from Fig. 4 that with an increase in the iron content from 100 to 500 mg/l, complete co-precipitation of gallium from the initial solution is observed.

Since the analyzed solution contains sodium and fluorine, which are part of the precipitate during the separation of macro components and gallium, the effect of these matrix elements on the analytical signal of gallium was studied. Solutions were prepared according to the procedure described in the Experimental Part (see procedure 3). It was found that at different concentrations of sodium and fluorine, γ is no more than 5%, i.e., the change in the intensity of the spectral lines of gallium is insignificant. To account for the background effect, sodium and fluorine must be added to the solutions for plotting calibration graphs in the amount contained in the analyzed solutions (see calibration of the spectrometer).

Thus, the following optimal conditions are recommended for the separation of macro components Mo, W, Ni, Co, Cr and gallium: pH 0-0.3, $n_{\mathrm{NaF}} = 0.25$ mol, $n_{\mathrm{HF}} = 0.45$ mmol, $c_{\mathrm{Fe(III)}} = 500$ mg/l. To dissolve the precipitate, it is necessary to use a mixture of nitric, hydrochloric and hydrofluoric acids (3 : 1 :1).

ICP-AES-determination of gallium in metallurgical materials. To experimentally verify the effectiveness of the application of the found optimal conditions for the co-deposition of gallium on sediment during separation from macro components, state standard samples of the composition of precision nickel alloys and high-alloy steel were used (Table 4). Sample preparation of standard samples for ICP-AES-gallium determination is described in Experimental Part (see procedure 2). It should be noted that certified mixtures and the introduced—found procedure were used to confirm the effectiveness of the developed procedure.

The correctness of the obtained results was evaluated according to the Student's criterion at $P = 0.95$ and $n = 5$ and it was found that there were no significant discrepancies between the analysis results obtained by the ICP-AES method and the introduced amounts of gallium. It can be concluded that the proposed procedure for the separation of gallium and macro components is effective and can be recommended for its ICP-AES determination. The advantage of the developed procedure is its simplicity, as well as the ability to use it for the analysis of a wide range of metallurgical materials (precision nickel alloys, alloy and high-alloy steel) with a high content of Cr, Mo, W, Ni and Co.

The detection limit ($c_{\min}$) was estimated by the $3s$ criterion; $c_{\min}$, gallium, when separated from macro quantities of Cr, Mo, W, Ni, Co, was 5×10^{-4} wt.%.

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CONFLICT OF INTERESTS

The authors of this paper declare that they have no conflict of interest.

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