

# Determination of Arsenic and Antimony in Ferrotungsten by Inductively Coupled Plasma Atomic Emission Spectrometry

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**Abstract**—The conditions for the coprecipitation of arsenic and antimony with trace amounts of tungsten and iron in the form of  $\text{PbWO}_4$  and  $\text{Na}_3\text{FeF}_6$  are found. The introduction of hydrofluoric acid in the deposition of macrocomponents results in the formation of precipitates with the lowest specific surface area and porosity, which contributes to the inhibition of the coprecipitation of the analytes (As and Sb). We prepared certified reference materials of ferrotungsten composition according to the developed procedure for the determination of arsenic and antimony by inductively coupled plasma atomic emission spectrometry (ICP–AES). The difference between the found and certified concentrations of analytes does not exceed the values specified by *GOSTs*. The method of simultaneous ICP–AES determination of arsenic and antimony with preliminary separation of the main components is applicable to the analysis of materials and alloys with high contents of iron and tungsten.

**Keywords:** arsenic, antimony, coprecipitation, adsorption isotherms, inductively coupled plasma–atomic emission spectrometry, precipitation separation of tungsten and iron

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Ferrotungsten is one of the essential alloying components in the production of specialized shear, structural, and high-speed steels. The addition of ferrotungsten into steel in combination with other metals (for example, chromium, vanadium, molybdenum) increases its hardenability and resistance to tempering and ensures its insensitivity to hot cracking [1]. The presence of small amounts of antimony and arsenic in steels leads to their embrittlement and rapid aging [2]; therefore, the concentration of these elements in steel and ferrotungsten is strictly regulated by current state standards [3, 4]. The *GOSTs* (State Standards) recommend a spectrophotometric method for the determination of arsenic and antimony in ferrotungsten [5, 6]. The procedures approved by these standards are long and laborious; they require the preliminary separation of antimony and arsenic from the main components of samples using thioacetamide and do not ensure the simultaneous determination of the analytes. It is advisable to develop a more straightforward procedure for determining the concentration of arsenic and antimony in ferrotungsten simultaneous using modern analytical equipment.

ICP–AES is characterized by rapid measurements, ease of calibration, a wide linear range of concentrations to be determined, and the possibility of simulta-

neous multielement analysis. These advantages led to the introduction of ICP–AES into the practice of many analytical laboratories, including for testing materials and products of the ferrous metallurgy [7].

Earlier, we proposed a procedure for direct ICP–AES determination of tungsten in ferrotungsten [8]. The development of a procedure for determining small amounts of arsenic and antimony is a rather difficult task, in particular, because these elements have high potentials of the excitation of the spectral lines [9, 10].

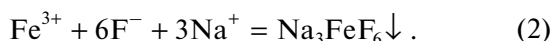
Yang et al. [11] showed that accurate ICP–AES determination of arsenic and antimony in tungsten-containing materials is difficult due to spectral interference from tungsten and the inability to perform the conventional “out-of-peak background correction” procedure. Using the multicomponent spectral fitting (MSF), that is, the method for mathematical spectral noise correction [12], the error in the ICP–AES determination of the arsenic and antimony concentration in the tungsten-containing standard sample was found to be 27.9 and 5.16% of the initial concentration, respectively. Probably, information about the procedures of direct ICP–AES determination of arsenic and antimony concentration in such objects is practically absent precisely because of the spectral noise from tungsten and iron. In laboratory practice, ana-

lytes are either preliminarily isolated by the generation of their hydrides [13, 14] or separated from the matrix using tributyl phosphate [15]. However, the presence of trace amounts of iron and tungsten in the test materials adversely affects the formation of hydrides of trace analytes; it is required to inhibit the interference from the matrix to obtain accurate results [16, 17]. The procedure proposed in [15] for separating the iron-containing matrix from arsenic with tributyl phosphate is laborious. Thus, the development of a simple, rapid, and economical method for the preliminary separation of arsenic and antimony from the main components of iron and tungsten remains an urgent task. Its solution yields the simultaneous ICP–AES determination of small quantities of analytes with high accuracy.

We found earlier [18, 19] that iron in trace amounts is precipitated from acidic solutions by sodium fluoride with the formation of a  $\text{Na}_3\text{FeF}_6$  crystalline precipitate. The coprecipitation of arsenic and antimony could be inhibited by introducing a complex-forming agent, for example, hydrofluoric acid, in a molar ratio of  $\text{NaF} : \text{HF} \approx 1 : 1$ . Under these conditions, a large-crystalline precipitate is formed with lower specific surface area and porosity than that obtained without using hydrofluoric acid. The use of this procedure with the aim of separating macroscopic amounts of tungsten leads only to its partial precipitation as  $\text{Na}_2\text{WF}_8$ . As a result, tungsten remains in large quantities in the test solution, and the accurate determination of the concentration of analytes becomes impossible due to spectral interference.

A possibility of the complete precipitation of trace amounts of tungsten using lead acetate  $\text{Pb}(\text{CH}_3\text{COO})_2$  as a  $\text{PbWO}_4$  precipitate was mentioned in [20]; however, arsenic is coprecipitated under these conditions [21]. Antimony behaves similarly.

We believe that the introduction of a certain amount of hydrofluoric acid with the simultaneous separation of iron and tungsten in the form of a  $\text{Na}_3\text{FeF}_6$ – $\text{PbWO}_4$  precipitate helps eliminate the coprecipitation of analytes, that is,



It should be noted that the introduction of excess HF into the solution can lead to the dissolution of the  $\text{PbWO}_4$  precipitate and the transition of tungsten to the filtrate in the form of a stable complex compound



It was necessary to study the process of coprecipitation of arsenic and antimony with  $\text{Na}_3\text{FeF}_6$ – $\text{PbWO}_4$ . Understanding the mechanism of the process will enable achieving the desired effect, that is, a

decrease or complete inhibition of the coprecipitation of analytes.

The goal of this work was to study the mechanism of the coprecipitation of arsenic and antimony with  $\text{Na}_3\text{FeF}_6$ – $\text{PbWO}_4$  and a possibility of its inhibition upon the introduction of a complexing agent, hydrofluoric acid; to evaluate the effectiveness of the proposed procedure by preparing certified reference materials of the composition of ferrotungsten for the ICP–AES determination of arsenic and antimony.

## EXPERIMENTAL

**Preparation of solutions.** Solutions of ions W(VI), Fe(III), As(III), and Sb(III) were placed in a heat-resistant fluoroplastic glasses in amounts corresponding to the composition of the material (wt %): W, 70.0; Fe, 30.0; As, 0.002; and Sb, 0.002. Twenty milliliters of aqua regia and a specific volume of hydrofluoric acid (40 wt %, from 5 to 15 mL) were added. The mixtures were heated on an electric hot plate until the solutions start boiling (75°C). Then, 40 mL of  $\text{CH}_3\text{COOH}$  (95 wt %) and 10 mL of a  $\text{Pb}(\text{CH}_3\text{COO})_2$  solution with a concentration of 50 g/L were added under constant stirring to create a molar excess of the precipitant. The pH was adjusted to 4.3 using indicator paper (pH 4.0–7.0) by pouring a 2 M NaOH solution in portions under constant stirring. The precipitate formed was kept for 10 min at the boiling point of the solution, filtered through a white ribbon filter, and washed with concentrated acetic acid and distilled water. The precipitates thus obtained were discarded or retained for further study. The filtrate was transferred to a polypropylene volumetric flask, diluted to the mark with distilled water, and stirred. If necessary, the dilution procedure was used. The resulting solutions were analyzed for the concentration of arsenic, antimony, iron, and tungsten by ICP–AES.

A blank solution containing all components except W(VI), Fe(III), As(III), and Sb(III) ions was prepared simultaneously with the concentrations similar to those used in the preparation of test solutions.

**Precipitation.** Precipitates of the main components of iron and tungsten, obtained in the presence of different volumes of hydrofluoric acid (40 wt %, 5–15 mL) (see preparation of solutions), were dried in air for 24 h. Then they were used to determine the parameters of specific surface area and porosity and to study their morphology using a scanning electron microscope.

**Conditions for measuring atomic emission of W, Fe, As, and Sb.** An Optima 2100 DV ICP–AES spectrometer (Perkin Elmer) with a quartz burner was used. The operating parameters of the spectrometer were as follows: high-frequency power, 1500 W; sample-supplying argon flow, 0.8 dm<sup>3</sup>/min; auxiliary argon flow, 0.2 dm<sup>3</sup>/min; plasma-forming argon flow, 15.0 dm<sup>3</sup>/min; radial observation of plasma; solution

flow rate, 0.9 mL/min; sample nebulization time, 40 s; the number of measurements for a single sample, 2. A spray system resistant to the aggressive effect of hydrofluoric acid was used. The following analytical spectral lines were used: As I 189.042, Sb I 206.836, Fe II 358.119, and W II 207.912 nm.

**Calibration of the spectrometer.** Solutions for the calibration of the spectrometer were prepared by diluting certified reference materials of the composition of solutions of As, Sb, W, and Fe ions. Aliquot portions of a blank solution were added to them so that the concentrations of acids, lead acetate, and sodium ions, which are contained in the blank solution, corresponded to their levels in the test solutions. The concentrations of the elements to be determined in the calibration solutions are presented in Table 1.

**X-ray powder diffraction analysis.** X-ray powder diffraction analysis (qualitative and quantitative) was performed using an XRD-7000 X-ray diffractometer. The recording modes of the diffractometer were as follows: graphite monochromator;  $\text{CuK}\alpha$  radiation; tube voltage  $U = 40.0$  kV; current  $I = 30.0$  mA; angular range  $15^\circ$ – $80^\circ$  with a step of  $0.02^\circ$ ; time at a point 1.5 s; silicon powder external standard. The primary processing of the experimental data (background subtraction, separation, and subtraction of the  $K\alpha_2$  line) was performed using the Shimadzu software; the ORIGIN program was used to determine the integrated intensities and peak positions. The phases were identified using the PDF-2 database of the International Center for Diffraction Data (ICDD). The quantitative determination of crystalline phases was performed by a comparative evaluation of the intensities of diffraction maxima in a powder diffraction pattern.

**Microscopic studies and electron probe X-ray microanalysis (EPMA)** of the precipitates were performed using an EVO 40 scanning electron microscope (Carl Zeiss) equipped with an INCA X-Act SDD spectrometer (Oxford Instruments) and an INCA Energy SEM system for energy dispersive microanalysis. Thin layers of predried precipitate powders were applied on conductive double-sided tape and thoroughly blown with compressed air to remove particles that did not stick to the surface of the substrate. The preparations of the powders were placed in the microscope chamber, evacuated to a residual pressure of  $\sim 1.5 \times 10^{-3}$  Pa, and the images were taken at an accelerating potential difference of 20 kV and a probe current of 710 pA. The photographs of powder particles were recorded at various magnifications using a backscattered electron detector. Then, EPMA of representative powder particles was performed to determine their chemical composition.

**Determination of surface and porosity parameters.** The specific surface area and porosity of precipitates were determined by low-temperature nitrogen adsorption–desorption using a NOVA 1200e instrument (Quantachrome). The precipitation powders were

**Table 1.** Concentrations (mg/L) of As, Sb, W, and Fe ions in calibration solutions

Element	No. of solution for calibrating the spectrometer		
	1	2	3
Trace components			
As	1.00	0.50	0.10
Sb	0.50	0.10	1.00
Macrocomponents			
W	10.0	50.0	100.0
Fe	50.0	100.0	10.0

preliminarily calcined in air at  $500^\circ\text{C}$  for 5 h to remove residual moisture.

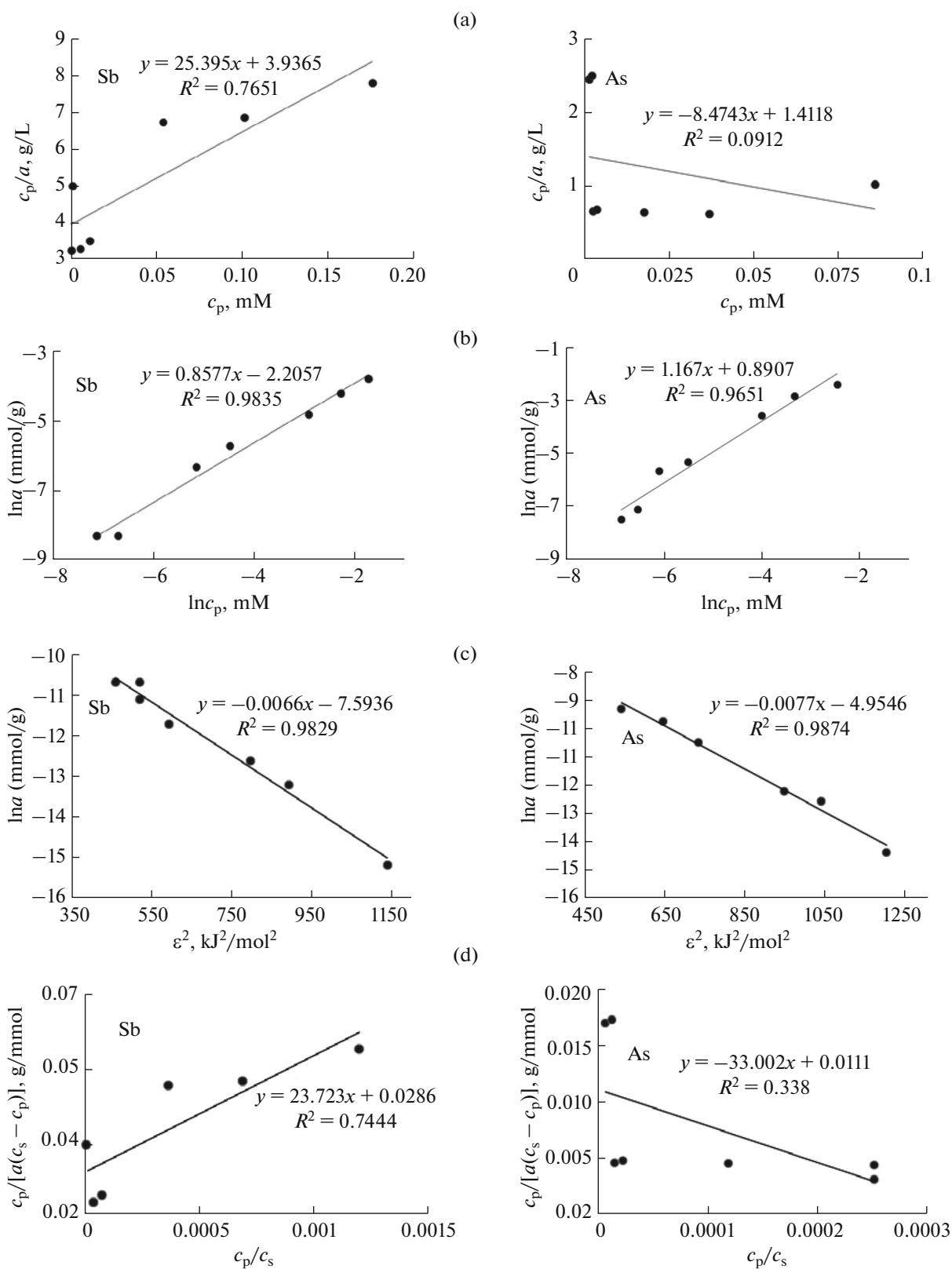
## RESULTS AND DISCUSSION

**Adsorption isotherm models in the description of the coprecipitation of arsenic and antimony with  $\text{Na}_3\text{FeF}_6$ – $\text{PbWO}_4$ .** It was previously found [18, 21] that the precipitation of macroscopic amounts of iron and tungsten using sodium fluoride and lead acetate gave precipitates of the composition of  $\text{Na}_3\text{FeF}_6$ – $\text{PbWO}_4$ , and the matrix material can be successively separated; however, trace analytes were lost from the test solution. We used various models of equilibrium adsorption isotherms (Figs. 1a–1d) to determine the coprecipitation mechanism of arsenic and antimony. A detailed description of the models used is given in [14]. The calculated values of the adsorption parameters are listed in Table 2. The Dubinin–Radushkevich model (the highest value of  $R^2$ ) is best suited for describing the coprecipitation of arsenic and antimony with the precipitate of  $\text{Na}_3\text{FeF}_6$ – $\text{PbWO}_4$ . Freundlich's model describes the process less accurately. The Langmuir model has the smallest  $R^2$  value for both analytes (As and Sb); therefore, according to the theory, there are few active adsorption centers with the same energy on the surface of the precipitate. The results indirectly prove that the surface of  $\text{Na}_3\text{FeF}_6$ – $\text{PbWO}_4$  samples is heterogeneous; the active centers have different energies. Thus, the coprecipitation of arsenic and antimony can be considered as the process of filling up the micropore volume of the precipitate containing trace amounts of tungsten and iron.

The Dubinin–Radushkevich model can be used to calculate the average free adsorption energy [22] as

$$E = (-2k)^{-0.5}. \quad (4)$$

Using the numerical value of  $E$  in the Dubinin–Radushkevich isotherm equation, one can judge the nature of the interaction forces between arsenic, antimony, and active centers on the precipitate surface and determine whether the fixation of ions is a physi-



**Fig. 1.** Adsorption isotherms of arsenic and antimony on  $\text{Na}_3\text{FeF}_6\text{-PbWO}_4$  precipitate in the coordinates of the linear equation of the (a) Langmuir, (b) Freundlich, (c) Dubinin–Radushkevich, and (d) BET models. precipitation conditions:  $V_{\text{HF}} = 5$  mL (40 wt %); concentration of precipitant  $\text{Pb}^{2+}$  2.41 mM; 40 mL of  $\text{CH}_3\text{COOH}$  (95 wt %); pH 4.3; 75°C;  $t = 10$  min.

**Table 2.** Calculated values of the adsorption parameters of the Langmuir, Freundlich, Dubinin–Radushkevich, and Brunauer–Emmett–Teller (BET) models

Model	Model parameter	Sb	As
Langmuir	$K_L$ , L/mmol	0.254	0.708
	$a_m$ , mmol/g	6.94	6.000
	$R^2$	0.7651	0.0912
Freundlich	$K_F$ , (mmol/g)(L/mmol) <sup>1/n</sup>	0.110	2.44
	1/n	0.858	1.18
	$R^2$	0.9835	0.9651
Dubinin–Radushkevich	$k$ , mol <sup>2</sup> /kJ <sup>2</sup>	0.0066	0.0077
	$E$ , kJ/mol	8.7	8.1
	$R^2$	0.9829	0.9874
BET	$K_{BET}$ , g/mmol	830.5	2972.1
	$a_m$ , mmol/g	0.042	0.030
	$R^2$	0.7444	0.338

cal process or it is chemical in nature. The values of  $E$  calculated for antimony and arsenic are 8.7 and 8.1 kJ/mol, respectively, and they are in the range of 8–16 kJ/mol for both analytes. In this case, according to the theory [23], arsenic and antimony are fixed in the micropores of the precipitate as a result of a chemical (ion-exchange) reaction.

**Effect of hydrofluoric acid on the coprecipitation of arsenic and antimony in the separation of macroscopic amounts of iron and tungsten.** As the fixation of arsenic and antimony on the precipitations of macroscopic quantities of iron and tungsten is due to chemical adsorption by the ion-exchange mechanism, the procedures of dilution, mixing, or increasing the solution temperature (which are useful in the case of physical adsorption) do not inhibit the coprecipitation process. To prevent the chemisorption fixation of arsenic and antimony in micropores, it is necessary to lower the solution supersaturation during the crystallization process, that is, to increase the solubility of the precipitates (in this case, Na<sub>3</sub>FeF<sub>6</sub>–PbWO<sub>4</sub>) and at the same time to decrease the concentration of precipitated ions (Fe and W) in solution [24]. A very effective way to minimize the concentration of precipitated ions is to bind them into medium-strength complex compounds. In this case, hydrofluoric acid is the complexing agent.

The effect of the added amount of hydrofluoric acid during the deposition of iron and tungsten on the coprecipitation of arsenic and antimony was investigated. We determined other precipitation conditions (pH, temperature, time) (see Experimental section) in the preliminary experiments. Table 3 shows the results of X-ray powder diffraction analysis of the precipitates of macrocomponents, depending on the volume of hydrofluoric acid introduced during the precipitation

process. It is found that tungsten presents in the composition of precipitation as a PbWO<sub>4</sub> phase, and iron is represented by three compounds: Na<sub>3</sub>FeF<sub>6</sub>, Na<sub>5</sub>Fe<sub>3</sub>F<sub>14</sub>, and Na<sub>4</sub>FeO<sub>3</sub>. An increase in the amount of hydrofluoric acid leads to an increase in the concentration of the NaF phase and a decrease in PbWO<sub>4</sub>. The most significant changes are observed with an increase in HF volume from 10 to 15 mL (corresponding to the HF concentration from 0.224 to 0.336 M), which is apparently due to the partial dissolution of the PbWO<sub>4</sub> precipitate and the transition of tungsten to the filtrate according to Eq. (3). The concentration of Na<sub>3</sub>FeF<sub>6</sub>, Na<sub>5</sub>Fe<sub>3</sub>F<sub>14</sub>, and Na<sub>4</sub>FeO<sub>3</sub> varies slightly with varying amount of added hydrofluoric acid.

The micrographs of the resulting tungsten- and iron-containing precipitates and the results of determining their composition using the EPMA method are presented in Figs. 2a–2c. It is seen that an increased amount of hydrofluoric acid added during the precipitation of macrocomponents leads to a decrease in the size of the resulting chisel-shaped tungsten-containing and spherical iron-containing crystals. An increase in the concentration and particle size of the NaF phase is also noted. The most significant changes in the composition of precipitates are observed with an increase in the HF volume from 10 to 15 mL (from 0.224 to 0.336 M). In this case, no arsenic and antimony compounds were found in the precipitates.

Changes in the specific surface area and porosity of iron- and tungsten-containing precipitates are shown in Figs. 3 and 4. All samples are characterized by a relatively low level of specific surface area and porosity. By the nature of the hysteresis loops on the adsorption and desorption isotherms, it is determined that the precipitates of macrocomponents have open porosity;

**Table 3.** Results of determination of the phase composition of precipitates by X-ray powder diffraction analysis

$V$ (mL)/ $c$ (M) HF	Phase, wt %				
	PbWO <sub>4</sub>	NaF	Na <sub>3</sub> FeF <sub>6</sub>	Na <sub>3</sub> Fe <sub>3</sub> F <sub>14</sub>	Na <sub>4</sub> FeO <sub>3</sub>
5.0/0.112	83.3	11.5	1.3	3.5	0.5
7.5/0.168	74.0	19.0	1.6	4.8	0.8
10/0.224	74.7	21.9	1.7	2.8	0.7
15/0.336	58.0	37.9	1.3	2.8	0.6

**Table 4.** Results of determination of the specific surface area and porosity of the precipitates of macrocomponents by low-temperature nitrogen adsorption–desorption

Parameters	$V$ (mL)/ $c$ (M) HF			
	5/0.112	7.5/0.168	10/0.224	15/0.336
Specific surface area (BET), m <sup>2</sup> /g	3.78	3.34	2.73	2.56
Specific surface area of micropores ( $t$ -method), m <sup>2</sup> /g	0	0	0	0
Specific surface area of mesopores ( $t$ -method), m <sup>2</sup> /g	3.78	3.34	2.73	2.56
Total pore volume (in the range from 0 to 154 nm), mL/g	0.020	0.015	0.013	0.012
Average pore size, nm	21.2	18.0	18.8	13.4
Dominant pore size, nm	3.95	3.95	3.71	3.71
Pore structure (hysteresis loop analysis)	Open, slit	Open, slit	Open, slit	Open, slit

the pores are slit-shaped [25]. There are two types of pores in all the samples: 4 and 35 nm in size. With the volume of hydrofluoric acid increased from 5 to 15 mL (corresponding to a concentration from 0.112 to 0.336 M), the specific surface area of the precipitates decreases 1.4 times, and the average pore size decreases 1.6 times. There is a simultaneous decrease in the pore volume of both the first and second types by 1.7 times. The most significant changes are observed with an increase in the HF volume from 7.5 to 10.0 mL (from 0.168 to 0.224 M). A further increase in the HF volume to 15 mL does not lead to a significant change in the pore size or volume.

Thus, the deposition of trace amounts of iron and tungsten in the presence of 10 and 15 mL of hydrofluoric acid (40 wt %) results in precipitations with the smallest specific surface area and porosity, and, apparently, coprecipitation of trace analytes under these conditions is minimal.

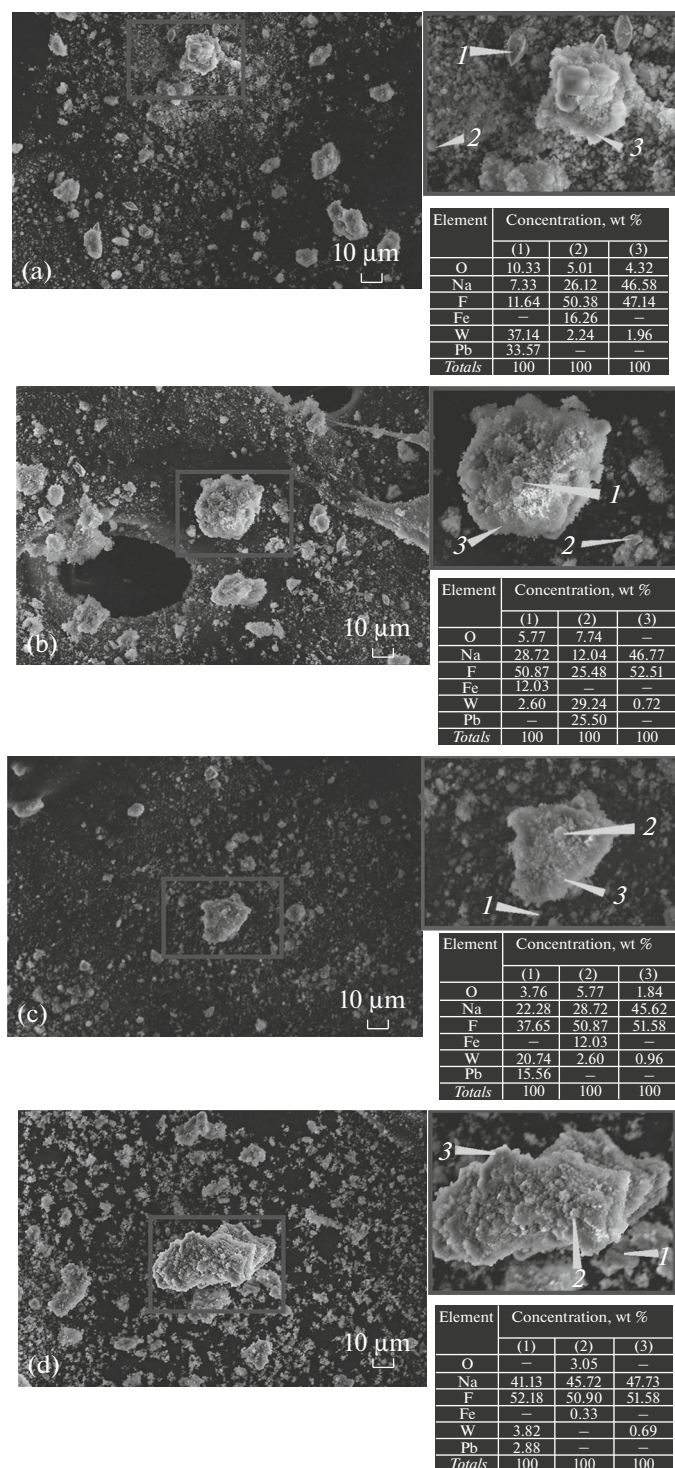
The results of the ICP–AES analysis of the filtrates obtained after the separation of the macrocomponents in the presence of different volumes of hydrofluoric acid are shown in Fig. 4. An increase in the HF volume from 5 to 10 mL under the experimental conditions (10 mL corresponds to a molar excess of HF : W(VI) > 100 : 1) inhibits significantly the coprecipitation of the analytes and makes possible their separation from the trace amounts of iron and tungsten (the relative concentration of the analytes in the filtrate is >90%). As expected, the subsequent increase in the HF volume from 10 to 15 mL (corresponding to a molar excess of

HF : W(VI) > 150 : 1) leads to a partial dissolution of the precipitate and the transition of tungsten into the test solution. The presence of a small amount of tungsten in the filtrate (up to 150 mg/mL) causes spectral interference, which deteriorates the accuracy of the ICP–AES determination of arsenic and antimony; the concentration of arsenic and antimony is higher than that added (see Fig. 4, the values at  $V$ (HF) = 15 mL).

Thus, the coprecipitation of arsenic and antimony depends on the specific surface area and porosity of the precipitates obtained, and a necessary condition for the inhibition of this process is strict adherence to the molar ratio of HF : W(VI)  $\approx$  100–150.

**Simultaneous ICP–AES determination of arsenic and antimony in ferrotungsten.** We used certified reference materials of the composition of ferrotungsten for the experimental verification of the effectiveness of the found conditions for inhibiting the process of coprecipitation of arsenic and antimony in the separation of iron and tungsten.

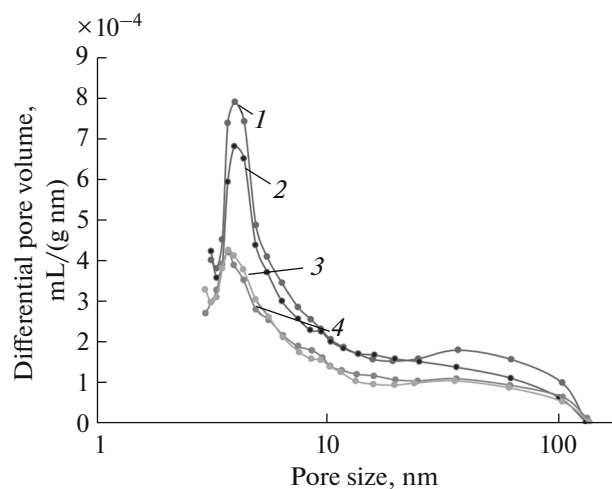
The sample preparation of standard samples for the ICP–AES determination of arsenic and antimony consisted of two stages: (1) dissolution and (2) precipitation and separation of the material matrix as described in the Experimental section. The samples were dissolved in a mixture of concentrated acids, consisting of hydrofluoric acid (HF : W(VI)  $\approx$  100–150) and aqua regia. The analytes were separated from iron and tungsten under the following conditions: molar excess of Pb<sup>2+</sup> : W<sup>6+</sup> > 1.1; 40 mL of CH<sub>3</sub>COOH



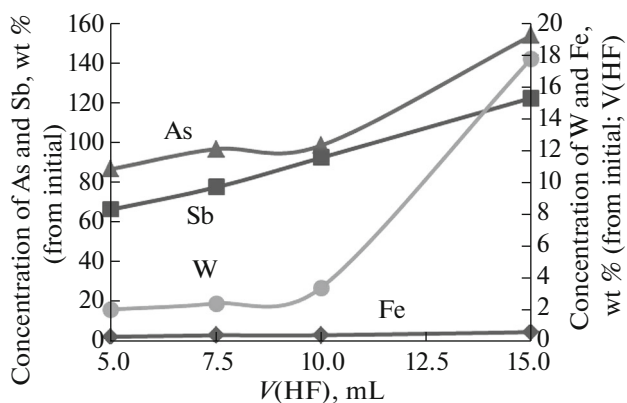
**Fig. 2.** Micrographs of iron- and tungsten-containing precipitates obtained in the presence of different volumes of HF (40 wt %): (a) 5.0, (b) 7.5, (c) 10.0, and (d) 15.0 mL. The chemical composition of representative particles of the precipitates is determined by X-ray microanalysis at positions (1–3). The other conditions of deposition as in the caption to Fig. 1.

(95 wt %); 2 M NaOH solution; pH 4.3; 75°C; the precipitate was kept at the specified temperature for 10 min.

The results of the simultaneous ICP–AES determination of arsenic and antimony in the standard samples of composition are given in Table 5. The anal-



**Fig. 3.** Results of the determination of the porosity of precipitates of macrocomponents, depending on the volume of conc. HF (40 wt %): (1) 5.0, (2) 7.5, (3) 10.0, and (4) 15.0 mL. The other conditions of deposition as in the caption to Fig. 1.



**Fig. 4.** Concentration of Fe, W, As, and Sb ions (wt % from initial) in the filtrate after the precipitation of macrocomponents depending on the added volume of HF (40 wt %). The other conditions of deposition as in the caption to Fig. 1.

**Table 5.** Results ( $c \times 10^2$ , wt %) of determination of arsenic and antimony in the certified reference materials of composition by ICP–AES ( $n = 5$ ,  $P = 0.95$ )

Standard sample	As		Sb	
	certified	found	certified	found
GSO 765-92P (F18b) Ferrotungsten	$2.8 \pm 0.1$	$2.5 \pm 0.3$	$0.69 \pm 0.06$	$0.59 \pm 0.07$
GSO 10223-2013 (F48) Ferrotungsten	$3.7 \pm 0.1$	$3.5 \pm 0.2$	$1.4 \pm 0.1$	$1.3 \pm 0.1$
GSO 2853-84 (F33a) Ferrotungsten	$0.20 \pm 0.02$	$0.18 \pm 0.01$	$0.06 \pm 0.02$	$0.06 \pm 0.01$

ysis error was calculated as  $t_{(0.95; 4)}s/\sqrt{n}$ , where  $t$  is the Student's  $t$ -value (for a two-sided distribution) of 2.78 with the number of measurements  $n = 5$  and the confidence level  $P = 0.95$ , and  $s$  is the standard deviation.

The certified and found values of the mass fraction of arsenic and antimony in almost all cases converge within the limits of random error (Table 5). The difference between the certified and found values does not exceed the standards given by *GOSTs* [5, 6]. The results prove the effectiveness of the proposed procedure for the determination of arsenic and antimony in their separation from macroscopic quantities of iron and tungsten.

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