

Analysis of Some Low Silicon Content Alloys by Inductively Coupled Plasma Atomic Emission Spectrometry*

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Inductively coupled plasma atomic emission spectrometry procedures have been developed for the analysis of some low silicon content alloys such as low-alloy steels, nickel metal, FeMo, high-alloy steel, cobalt alloy, FeNb and FeV. In most cases, the special emphasis has been placed on the determination of silicon together with the other analyte elements.

Keywords: Inductively coupled plasma atomic emission spectrometry; multi-element analysis; silicon; alloys; sample decomposition; phosphoric acid

For the determination of low silicon contents of up to about 0.3% Si (sample mass of 0.500 g), it is possible to replace molecular absorption spectrometry (MAS), originally used, *i.e.*, UV/VIS spectroscopy, with inductively coupled plasma atomic emission spectrometry (ICP-AES) or flame atomic absorption spectrometry (FAAS) for some metallic materials. The sample decomposition procedure (sample dissolution with hydrochloric acid, nitric acid or their mixture) is sufficient for the dissolution of silicon and some other elements. The dissolution conditions, *i.e.*, heating at a temperature of about 60°C does not result in the exclusion of silicon from the sample solution. In this way, it is possible to dissolve metallic samples with matrices containing largely iron, nickel, chromium or manganese. The silicon determination in FeMo however, results in incorrect values of silicon content because molybdenum silicides such as MoSi, MoSi₂ and possibly Mo₃Si₂ are not decomposed with hydrochloric and nitric acids. It was found that sufficient decomposition of molybdenum silicides is ensured by dissolving them with a mixture of sulfuric, phosphoric and, if necessary, nitric acids. The sample solution must be evaporated until white fumes of sulfur trioxide appear. Therefore, ferroalloys containing low silicon content (FeV and FeNb), Co alloys (Real) and steel Poldi AKRB samples were also decomposed with a mixture of sulfuric and phosphoric acids in a study of the hardly soluble components.

Experimental

Instrumentation

An ARL 33000 LA sequential atomic emission spectrometer with an inductively coupled argon plasma was used for the determination of analyte elements under compromise instrumental conditions, *i.e.*, at an observation height of 18 mm and an argon carrier gas flow rate of about 1.2 l min⁻¹. A Perkin-Elmer 503 flame atomic absorption spectrometer was used for comparative analysis by FAAS.

Sample Preparation

The average chemical composition of some alloys containing low and medium silicon contents is presented in Table 1. Table 2 summarizes the sample dissolution procedures. Essentially, the alloy samples in chip (alloys and steels) or the fine powder form (ferroalloys, especially the FeNb particle sizes should be less than 0.05 mm without separating the various size fractions) are dissolved with phosphoric and sulfuric acids (except for low-alloy steels and nickel metal) in polytetrafluoroethylene (PTFE) vessels at a temperature of between 150 and 200°C without the addition of hydrofluoric acid. The decomposition time has to be prolonged for about 10–15 min after the sample dissolution. In some instances, it is appropriate to start the sample decomposition with hydrochloric and nitric acids. The low-alloy steel and nickel metal samples are dissolved only with hydrochloric and nitric acids in glass beakers. In this way the alloy samples are quantitatively converted into a soluble form. If the FeNb samples are analysed, the sample solutions have to be stabilized with the addition of oxalic acid. In the analysis of FeV it was found that the aluminium content values obtained are lower. Therefore, the residual aluminium content was determined by FAAS after fusing insoluble residues with potassium disulfate, see Table 2. The sample decomposition of some alloys was also performed in a microwave oven using practically the same procedures as in PTFE beakers, under atmospheric pressure. The decomposition time was reduced from 2–3 h to about 10–15 min for all the samples analysed.

Blank and synthetic samples were used for calibration and were prepared by the same procedures as the real alloy samples. The matrix element composition was simulated using high-purity metals. The synthetic calibration samples also contained known additions of the analyte elements in the concentration range to be considered. For practical purposes, the matrix composition of the calibration samples can also be simulated using suitable certified or internal reference materials with known additions of some analyte elements.

Table 1 Chemical composition of some low and medium silicon content alloys (%)

Alloy	Al	Co	Cr	Fe	Mo	Nb	Ni	Si	V	W
Low-alloy steel	—	—	—	99	—	—	—	0.5	—	—
Ni metal	—	—	—	—	—	—	99	—	—	—
FeMo	—	—	—	30	70	—	—	1	—	—
High-alloy steel AKRB	2	—	15	55	2	—	30	1	—	3
Cobalt alloy Real	—	70	15	2	1†	—	2†	2	—	5
FeNb	—	—	—	25	—	65	—	0–10	—	—
FeV	2	—	—	50/20	—	—	—	2	40/80	—

† Some cobalt alloy Real types also contain Mo and Ni.

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Table 2 Sample preparation

Alloy	Sample mass/g	Dissolution	Evaporation to white fumes	Final volume/ml
Low-alloy steel	1.000	10 ml HNO ₃ conc. 10 ml HCl (1 + 1)	—	100
Ni metal	1.000	10 ml HNO ₃ conc. 10 ml HCl (1 + 1)	—	100
FeMo	0.500	10 ml HNO ₃ conc. 10 ml HCl (1 + 1)	10 ml H ₂ SO ₄ (1 + 1) 25 ml H ₃ PO ₄ conc.	250
High-alloy steel AKRB	0.500	10 ml HNO ₃ (1 + 1) 10 ml HCl (1 + 1)	10 ml H ₂ SO ₄ (1 + 1) 25 ml H ₃ PO ₄ conc.	250
Co-alloy Real	0.500	20 ml HCl (1 + 1)	10 ml HNO ₃ conc. 10 ml H ₂ SO ₄ (1 + 1) 25 ml H ₃ PO ₄ conc.	250
FeNb*	0.200	—	10 ml H ₂ SO ₄ (1 + 1) 25 ml H ₃ PO ₄ conc.	250*
FeV†	0.500	10 ml HCl conc. 5 ml HNO ₃ (1 + 1)	10 ml H ₂ SO ₄ (1 + 1) 25 ml H ₃ PO ₄ conc. 2 g NH ₂ OH·HCl	250†

* FeNb sample solution is stabilized with addition of 2.5 g of oxalic acid (COOH)₂·2H₂O.

† Aluminium contained in FeV sample was not quantitatively converted into a solution by the described procedure. Insoluble residues were analysed after filtration of a sample solution, washed with warm HCl (1 + 9) and water, ashed and fused with 2 g of potassium disulfate in a quartz crucible. After dissolution of the melt in water and dilution to 100 ml with water in a calibrated flask, the determination of aluminium was performed by flame atomic absorption spectrometry using the spectral line 309.3 nm in the fuel rich N₂O–C₂H₂ flame. The total aluminium content in FeV is given by the sum of aluminium contents found by ICP-AES and FAAS.

Results and Discussion

The analyte elements were determined using the spectral lines recommended by the manufacturer, see Table 3, except for cobalt, tantalum, titanium and tungsten. The original Co II 238.892 nm line was used only for the analysis of nickel metal, where a low iron content was found, because the line was severely affected by the nearby Fe II 238.863 nm line. In the

Table 3 Analyte elements and analytical spectral lines for ICP-AES

Element and line	Wavelength/nm	Secondary slit-width/μm
Al I	394.401	75
B I	249.678	50
Cd II	226.502	75
Co II	238.892	75
Cr I	360.533	75
Cu I	324.754	75
Fe II	259.940	75
Mn II	257.610	75
Mo I	317.035	75
Ni II	231.604	75
Si I	251.611	75
Ta	265.327	75
Ti I	363.546	75
V II	311.071	75
W I	400.875	50
Zn II	202.548	75

Table 4 Interferences from added elements at a concentration of 1% for different Ni lines (nm)

Interfering element	Background equivalent concentration (% Ni)		
	Ni II 221.647 nm	Ni I 232.003 nm	Ni II 231.604 nm
Al	—	—	<0.00005
Co	0.0018	0.005	<0.005
Cr	0.0004	0.047	<0.0005
Cu	—	—	<0.0005
Fe	0.00007	0.0009	0.0001
Mn	0.00016	0.0011	0.0002
Mo	0.0004	0.007	0.0002
Si	0.044	0.0001	0.0002
Ti	—	—	0.0002
V	—	—	0.00005
W	0.0085	0.0013	0.0004

Table 5 Interferences from added elements at a concentration of 1% for different tungsten lines (nm)

Interfering element	Background equivalent concentration (% W)				
	W II 207.911	W II 224.875	W II 218.936	W II 239.709	W I 400.875
Al	0.0017	—	—	—	<0.0002
Co	0.00018	—	—	—	0.0002
Cr	0.00027	—	—	—	0.0001
Cu	0.0012	—	—	—	<0.001
Fe	0.00012	0.0037	0.0012	0.0042	0.00025
Mn	0.00014	—	—	—	0.0003
Mo	0.00075	—	—	—	0.001
Ni	0.00012	—	—	—	0.00005
Ti	0.0003	—	—	—	0.09
V	0.0012	—	—	—	0.0004

Table 6 Interferences from added elements at a concentration of %

Interfering element	Background equivalent concentration (%)	Note
Co	<0.005	Ni
Cr	0.00013	Co
	0.008	Zn
Cu	0.01	Zn
Fe	0.0005	Cd
	0.03	Co
	0.005	Cr
	<0.0005	Si
	0.0015	Ta
	0.0004	Zn
Mo	0.0004	Co
	0.0075	Si
	<0.0015	V
Nb	0.0007	Al
	0.0055	Cu
	0.0005	Si
	0.0005	Ta
	0.007	Ti
Ni	0.001	Al
	0.0005	Co
	0.001	Ti
Ti	0.011	V
V	0.0003	Cr
W	0.00014	Co
	0.0016	Si

other instances, the determination of cobalt was performed using the Co I 350.228 nm line at an observation height of 26 mm. Similarly, the determination of tantalum in FeNb was performed using the Ta II 240.063 nm line at an observation height of 18 mm instead of the Ta 265.327 nm line of low intensity, which is severely influenced by the presence of chromium. For the determination of titanium in steel Poldi AKRB, the Ti II 323.452 nm line at an observation height of 18 mm was selected instead of the Ti I 363.546 nm line which is affected by the presence of molybdenum. For the determination of nickel and tungsten in the cobalt alloy Real, more suitable spectral lines were investigated. Besides the original Ni II 231.604 nm line, the Ni II 221.647 and Ni I 232.003 nm lines were examined. For comparison, the spectral interferences found at the optimal observation height of 18 mm are presented in Table 4. Finally, the determination of nickel was performed using the Ni II 231.604 nm line because it was the most significant spectral line. The determination of tungsten was originally performed using the W I 400.875 nm line of very low intensity. Therefore, the W II 207.911, W II 224.875, W II 218.936 and W II 239.709 nm spectral lines were studied. The

W II 207.911 nm line appeared to be the most advantageous spectral line compared with the original line at an observation height of 18 mm. The most important interferences from accompanying elements found for tungsten are shown in Table 5. The spectral interferences of the matrix and the other accompanying elements of the spectral line intensities of the analyte elements are reported in Table 6. The contingent interferences were corrected for the increase or decrease in the spectral background, due to differences in the sample matrix composition, if necessary.

The proposed procedures were verified by means of Czechoslovakian, British and German certified reference materials. For comparison and verification of accuracy, the samples were also analysed by other analytical methods such as gravimetry, titrimetry, MAS and FAAS. The following tables present results obtained for some metallic materials analysed by ICP-AES. Comparison of the ICP-AES and MAS results for the low silicon contents in low-alloy steels is given in Table 7. Table 8 presents the analytical results of ICP-AES and FAAS and certified values for nickel metal. Similarly, the analytical results obtained by ICP-AES, FAAS and other analytical methods are shown in Tables 9–13 for ferromolybdenum, high-alloy steel Poldi AKRB, cobalt alloy Real, FeNb and FeV, respectively.

The sample decomposition of FeNb with sulfuric and phosphoric acids was also used for the determination of nitrogen by titrimetry after distillation. The results for the nitrogen contents in FeNb are compared with those found by vacuum extraction (analyser, Balzers EAN 202) are given in Table 14.

The precision of the determination of analyte elements characterized by means of the basic statistical data is presented in Tables 15–17 for FeMo, FeNb and FeV, respectively.

The limits of determination, defined as ten times the standard deviation of the background noise, were mostly within the range 0.002–0.02% for a sample mass of 0.500 g in 250 ml of solution, i.e., within the range 40–400 ng ml⁻¹ for the analyte elements. The limit of determination achieved also depends on the sample matrix composition, the other accompanying elements and their mutual spectral influence.

Table 7 Comparison of results for silicon content (%) in real and certified reference materials (CRMs) low-alloy steels

Sample	Certificate value	MAS*	ICP-AES
871-CRM	0.0095	0.013	0.0105
BCS-CRM 149	0.0025	<0.002	0.0015
BCS-CRM 270	0.050†	0.055	0.056
R 426/6	—	0.052	0.056
R 427/6	—	0.043	0.051
R 428/6	—	0.053	0.057
R 429/6	—	0.057	0.064
R 430/6	—	0.045	0.051
R 431/6	—	0.046	0.053

* MAS, Molecular absorption spectrometry.

† Correlation coefficient for determination of silicon (MAS and ICP-AES) was found to be 0.993.

Table 8 Comparison of results (%) for CRMs of nickel metal

Sample	Method	Cd	Co	Cr	Cu	Fe	Mn	Si	Zn
ČSAN-CRM 5-15-015	Certificate	0.0003	0.078	—	0.011	0.016	0.105	0.035	0.0057
	FAAS	0.0004	0.064	<0.005	0.011	0.015	0.106	—	0.005
	ICP-AES	0.001	0.064	<0.005	0.012	0.014	0.109	0.035	0.006
ČSAN-CRM 5-51-016	Certificate	0.002	0.21	—	0.079	0.101	0.0138	0.163	0.019
	FAAS	0.0017	0.193	<0.005	0.083	0.108	0.014	—	—
	ICP-AES	0.0015	0.195	0.005	0.084	0.107	0.014	0.155	0.0195

Table 9 Comparison of results (%) for real and CRM samples of ferromolybdenum

Sample	Method	Al	Cr	Cu	Mn	Ni	Si	V
02	FAAS, others	—	<0.02	0.24	0.01	0.04	0.03*	<0.01
	ICP-AES	<0.02	<0.02	0.26	<0.01	0.05	0.25	<0.01
03	FAAS, others	—	<0.02	0.49	0.01	0.04	0.04*	<0.01
	ICP-AES	<0.02	<0.02	0.53	<0.01	0.05	0.03	<0.01
41	FAAS, others	—	<0.02	0.18	0.01	0.07	0.03*	<0.01
	ICP-AES	<0.02	<0.02	0.185	<0.01	0.06	0.03	<0.01
42	FAAS, others	—	<0.02	0.15	0.01	0.05	0.02*	<0.01
	ICP-AES	<0.02	0.02	0.15	<0.01	0.05	0.035	<0.01
43	FAAS, others	—	<0.02	0.20	0.01	0.05	0.04*	—
	ICP-AES	<0.02	0.02	0.205	<0.01	0.06	0.03	—
90	FAAS, others	—	0.07	0.36	0.08	0.05	0.60*	<0.01
	ICP-AES	<0.02	0.04	0.37	0.08	0.05	0.63	<0.01
ČSAN-CRM 4-4-02	Certificate	—	—	0.52	—	—	0.091	—
	FAAS	—	0.02	0.495	<0.01	0.045	0.10	<0.01
211J16A	ICP-AES	<0.02	<0.02	0.50	<0.01	0.04	0.095	<0.01
	Certificate	—	—	0.192	0.004	—	0.226	—
	ICP-AES	<0.02	<0.02	0.02	<0.01	0.02	0.22	0.01

* Gravimetry.

Table 10 Comparison of results (%) for real samples of high-alloy steel AKRB. Correlation coefficients for determination of manganese, molybdenum, silicon, titanium and tungsten (chemical methods and ICP-AES) were found to be 0.999, 0.999, 0.999, 0.998 and 0.999, respectively

Sample	Method	Al	B*	B†	Co	Cr	Cu	Mn	Mo	Si	Ti	V	W
1608 P1	Chemical	—	—	—	—	12.69‡	—	0.27‡	0.515‡	0.10§	0.525§	—	1.975§
	FAAS	2.01	—	—	0.001	—	0.039	0.235	0.55	—	0.52	0.003	—
	ICP-AES	2.00	0.065	0.063	0.002	12.53	0.037	0.245	0.52	0.08	0.535	0.002	1.955
1608 P2	Chemical	—	—	—	—	13.29‡	—	0.30‡	0.75‡	0.26§	0.73§	—	2.21*
	FAAS	2.35	—	—	0.0015	—	0.090	0.255	0.79	—	0.72	0.004	—
	ICP-AES	2.31	0.05	0.55	0.002	13.04	0.087	0.26	0.76	0.24	0.735	0.003	2.22
1608 P3	Chemical	—	—	—	—	13.74‡	—	0.44‡	1.03‡	0.41	0.89§	—	2.61§
	FAAS	—	—	—	0.0015	—	0.040	0.405	—	—	—	0.005	—
	ICP-AES	2.61	0.085	0.090	0.0025	13.66	0.037	0.42	1.04	0.385	0.90	0.007	2.62
1608 P4	Chemical	—	—	—	—	14.82‡	—	0.58‡	1.28‡	0.50§	1.04§	—	2.99§
	FAAS	—	—	—	0.002	—	0.042	0.54	—	—	—	0.006	—
	ICP-AES	2.87	0.10	0.095	0.0025	14.47	0.038	0.56	1.295	0.46	1.09	0.005	3.03
1608 P5	Chemical	—	—	—	—	15.84‡	—	0.68‡	1.56‡	0.64§	1.17§	—	3.32§
	FAAS	—	—	—	0.002	—	0.043	0.64	—	—	—	0.006	—
	ICP-AES	3.18	0.12	0.113	0.005	15.22	0.041	0.66	1.56	0.61	1.195	0.006	3.41
3102 X1	Chemical	—	—	—	—	14.40‡	—	0.31‡	1.19‡	0.19§	0.80§	—	2.87§
	FAAS	—	—	—	0.002	—	0.031	0.28	—	—	—	0.003	—
	ICP-AES	2.71	0.02	0.018	0.002	14.10	0.030	0.29	1.22	0.18	0.805	0.003	2.95
3102 X	Chemical	—	—	—	—	15.12‡	—	0.32‡	1.22‡	0.22§	0.99§	—	2.80§
	FAAS	—	—	—	0.0015	—	0.032	0.28	—	—	—	0.004	—
	ICP-AES	3.16	0.09	0.095	0.003	14.76	0.030	0.29	1.20	0.21	1.04	0.004	2.88

* Boron content determined directly without separation. ‡ Titrimetry.
† Boron content determined after hydroxide separation. § MAS.

Table 11 Comparison of results (%) for real samples of cobalt alloy Real

Sample	Method	Al	Cu	Fe	Mn	Mo	Ni	Si	V	W
1638 P	FAAS and others	0.01	0.01	2.22*	0.20†	—	—	1.93§	0.005	4.85*
	ICP-AES‡	0.01	0.01	2.13	0.175	—	—	1.925	0.005	4.90
1640 P	FAAS and others	0.01	0.01	3.04*	0.20†	—	—	1.90§	0.005	4.72*
	ICP-AES	0.01	0.01	2.85	0.17	—	—	1.85	0.005	4.77
1641 P	FAAS and others	0.01	0.01	2.18*	0.21†	—	—	1.92§	0.005	4.60*
	ICP-AES	0.01	0.01	2.00	0.175	—	—	1.925	0.005	4.71
1642 P	FAAS and others	0.01	0.01	2.22*	0.19†	—	—	1.89§	0.005	4.79*
	ICP-AES	0.01	0.01	2.07	0.15	—	—	1.87	0.005	4.88
1643 P	FAAS and others	0.01	0.01	2.10*	0.20†	—	—	1.94§	0.005	4.68*
	ICP-AES	0.01	0.01	1.98	0.16	—	—	1.91	0.005	4.81
1734 P1	FAAS and others	0.01	—	0.33*	0.49†	0.015*	—	1.02§	0.005	4.35*
	ICP-AES	0.01	—	0.32	0.50	0.02	0.05	0.97	0.005	4.33
1734 P	FAAS and others	0.01	—	0.38*	0.50†	0.70*	2.19	1.16§	0.005	4.78*
	ICP-AES	0.01	—	0.37	0.50	0.70	2.20	1.17	0.005	4.65
1735 P1	FAAS and others	0.01	—	0.15*	0.52†	—	—	1.27§	0.005	4.35*
	ICP-AES	0.01	—	0.14	0.54	0.025	0.075	1.26	0.005	4.35
1735 P	FAAS and others	0.01	—	0.18*	0.50†	0.74*	1.89	1.12§	0.005	4.54*
	ICP-AES	0.01	—	0.18	0.485	0.73	1.93	1.16	0.005	4.63

* MAS. ‡ Sample decomposition for ICP-AES was performed by means of a microwave oven.
† Titrimetry. § Gravimetry.

Table 12 Comparison of results (%) for real and CRM samples of ferroniobium

Sample	Method	Al	Cr	Cu	Mn	Mo	Ni	Si	Ta	Ti	V	W
5	FAAS and others	—	0.04	0.02	1.71	0.03	0.06	—	4.98	0.23	0.05	—
	ICP-AES	0.96	0.04	0.02	1.67	0.03	0.04	0.285	5.02	0.25	0.04	0.24
S	FAAS and others	—	0.01	0.02	5.46	0.02	0.02	—	0.40	0.63	0.03	—
	ICP-AES	1.59	<0.02	0.03	5.20	<0.02	<0.02	0.97	0.32	0.63	0.025	<0.02
NHKG	FAAS and others	—	0.14	0.01	0.27	0.02	0.02	10.77*	1.04†	4.3	0.06	—
	ICP-AES	1.00	0.14	<0.02	0.34	0.02	<0.02	10.6	1.03	4.76	0.085	<0.02
BCS-CRM 362	Certificate value	1.70	—	—	—	—	—	0.72	4.9	0.47	—	0.24
	FAAS and others	—	0.195	0.01	1.88	0.01	0.01	0.73*	4.75†	0.44	0.04	—
CRM 576-1	ICP-AES	1.605	0.195	<0.02	1.81	<0.02	<0.02	0.73	4.76	0.465	0.045	0.24
	Certificate value	2.53	—	—	—	—	—	1.79	0.306	1.32	—	—
CRM 579-1	ICP-AES	2.42	0.20	0.055	0.275	—	0.045	1.86	0.30	1.12	0.075	—
	Certificate value	1.86	—	—	—	—	—	1.03	3.85	0.567	—	—
	ICP-AES	1.765	0.19	0.055	1.45	—	0.055	1.055	3.29	0.515	0.355	—

* Titrimetry (after separation and hydrolysis of K₂SiF₆).
† MAS (with Malachite Green).

Table 13 Comparison of results (%) for real and CRM samples of FeV

Sample	Method	Al total	Cr	Cu	Mn	Ni	Si
58	FAAS and others	1.80	0.15	0.01	0.18	0.06	0.70†
	ICP-AES	1.77	0.11	0.01	0.17	0.05	0.66
D 4087	FAAS and others	1.76	0.08	0.025	0.18	0.065	—
	ICP-AES	1.70	0.08	0.025	0.175	0.075	0.71
D 4088	FAAS and others	1.88	0.135	0.025	0.17	0.10	—
	ICP-AES	1.85	0.14	0.025	0.17	0.105	0.76
D 4089	FAAS and others	1.66	0.10	0.025	0.16	0.07	—
	ICP-AES	1.58	0.10	0.025	0.16	0.075	0.73
D 4130	FAAS and others	1.75	0.12	0.03	0.16	0.05	0.72†
	ICP-AES	1.74	0.12	0.03	0.15	0.06	0.72
D 4131	FAAS and others	1.80	0.10	0.05	0.16	0.05	0.72†
	ICP-AES	1.70	0.11	0.05	0.16	0.05	0.74
D 4132	FAAS and others	1.10	0.15	0.04	0.20	0.04	0.83†
	ICP-AES	0.97	0.14	0.04	0.19	0.05	0.82
D 4133	FAAS and others	1.60	0.11	0.12	0.17	0.11	0.70†
	ICP-AES	1.69	0.11	0.12	0.17	0.11	0.62
BCS-CRM 205/2	Certificate value	2.0*					1.02*
	FAAS	1.95	0.19	0.247	0.25	<0.02	1.03
	ICP-AES	1.92	0.205	0.258	0.255	0.045	1.005
BAM-CRM 531-1	Certificate value	1.59		0.019	0.15	—	0.69
	ICP-AES	1.61	0.365	0.023	0.14	0.02	0.685

* Information value, not certified.
† Gravimetry.

Table 14 Comparison of results for nitrogen content (%) in real samples of FeNb

Sample	Vacuum extraction (Balzers, Ni-bath)	Distillation*	
S	0.010	0.006	0.011
45	0.163	0.168	0.162
59	0.112	0.106	0.112
72	0.123	0.123	0.118

* Parallel determinations.

Table 15 Precision of determination of analyte elements (%) in FeMo ČSAN-CRM 4-4-02 samples

Parameter	Al	Cr	Cu	Mn	Ni	Si	V
n*	10	8	15	13	13	16	8
Average	<0.02	<0.02	0.50	<0.01	0.04	0.095	<0.01
SD†	—	—	0.012	—	0.004	0.012	—
RSD‡	—	—	2.4	—	10.0	12.6	—

* n = Number of analyses.
† SD = Standard deviation.
‡ RSD = Relative standard deviation.

Table 16 Precision of determination of analyte elements (%) in FeNb BCS-CRM 362 samples

Parameter	Al	Cr	Cu	Mn	Mo	Ni	Si	Ta	Ti	V	W
n	5	5	5	4	4	4	6	6	6	4	4
Average	1.605	0.195	<0.02	1.81	<0.02	<0.02	0.73	4.76	0.465	0.045	0.24
SD	0.026	0.021	—	0.080	—	—	0.030	0.089	0.025	0.011	0.022
RSD	1.6	10.8	—	4.4	—	—	4.1	1.9	5.3	24.4	9.2

Table 17 Precision of determination of analyte elements (%) in real and CRM samples of FeV

Sample	Parameter	Al total	Cr	Cu	Mn	Ni	Si
FeV 80	<i>n</i>	33	33	34	34	33	33
	Average	2.715	0.19	0.0065	0.21	0.075	0.685
	SD	0.069	0.012	0.0011	0.005	0.005	0.017
	RSD	2.5	6.3	16.9	2.2	6.5	2.5
BAM-CRM 531-1	<i>n</i>	25	24	25	25	25	24
	Average	1.61	0.365	0.023	0.14	0.02	0.685
	SD	0.038	0.013	0.0024	0.004	0.003	0.019
	RSD	2.4	3.6	10.4	2.7	13.7	2.8

Conclusion

It was found that the described ICP-AES procedures can be used successfully in metallurgical laboratories for the multi-element analysis of some low silicon content alloys. Silicon can be determined together with the other analyte elements without the application of hydrofluoric acid. In some instances, the microwave oven was used to reduce the digestion time.

The described ICP-AES procedures have been used for the multi-element analysis of high silicon content ferroalloys in everyday laboratory practice for the past 7 years.

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