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SHIMADZU

Application News

Inductively Coupled Plasma Atomic Emission Spectrometry No.J90

Iron and Steel Analysis Using ICPE-9000

Iron and steel comprise one of the most typical classes of materials used in all fields, including various industrial products as well as everyday commodities. Due to the many types of iron and steel with their different characteristics, such as heat and corrosion resistance, etc., control of processing and quality is very important because these characteristics depend on the types and amounts of elements in these products. The concentration range of these elements ranges widely from the ppm level to several tens of percent, so analysis of the content levels requires an analytical instrument that possesses high sensitivity as well as a wide linear dynamic range.

Here we introduce the analysis of iron and steel standard substances using the Shimadzu ICPE-9000 multi-type ICP emission spectrometer.
The ICPE-9000 is a simultaneous type ICP-AES which utilizes a CCD for the detector. High-sensitivity axial observation is equipped as standard, and since radial observation can be selected as an option, everything from trace elements such as phosphorus and sulfur to high-concentration elements such as nickel and chrome in stainless steel can be analyzed simultaneously. Moreover, samples such as low alloy steel and stainless steel, which may contain widely differing concentrations of the same elements, can be analyzed simultaneously in a single batch.

■ Analytical Conditions

Instrument	: ICPE-9000
RF output	: 1.2 (kW)
Plasma gas flow rate	: 14 (L/min)
Auxiliary gas flow rate	: 1.2 (L/min)
Carrier gas flow rate	: 0.70 (L/min)
Sample introduction	: Coaxial nebulizer
Sample intake rate	: 1.0 (mL/min)
Spray chamber	: Cyclone chamber
Plasma torch	: Torch for high salt content
Observation method	: Axial / radial

■ Samples

Standard materials certified by the Japan Iron and Steel Federation were used.

- JSS154-12 (low alloy steel)
- JSS650-5 (SUS430, ferritic stainless steel)
- JSS651-12 (SUS304, austenitic stainless steel)

■ Sample Preparation

(1) Weigh out 1 g of sample into a beaker, add 10 mL nitric acid + 30 mL hydrochloric acid, and heat to dissolve.
(2) Filter the solution.
(3) After ashing the undissolved solids, melt using 2 g of mixed flux (sodium carbonate: sodium tetraborate = 2:1). Dissolve the melted product in a small amount of warm water and 5 mL of 6M hydrochloric acid.
(4) Combine the solution from step (3) with the filtrate of step (2), add 30 mL of 5.4% tartaric acid (w/v), add 2 mL of 10 mg/mL yttrium, bring to 200 mL, and use this as the analytical sample.

*The samples and preparation processing were provided by JFE Steel Corporation Steel Research Laboratory.

■ Standard Solutions for Calibration Curve

Dissolve high purity iron (99.99 % or greater, sponge iron) in the same way as for the samples, and using this as a base, add the analysis element standard solution in stepwise fashion to generate a plot.

■ Analysis

Quantitation of each element was conducted by the calibration curve method using yttrium as the internal standard element.
Elements which indicated no certification value or reference value were cross-checked by high-resolution type ICP (Shimadzu sequential type model ICPS-8100) to confirm the quantitation value.

■ Results

Table 1 shows the quantitation results and the detection limit of this analysis.
For most of the elements, the quantitative results agreed with certified values or with ICPS-8100 results.

Table 1 Results for Iron and Steel Certified Standard Substances (Unit: %)

	Sample Name	JSS154-12			JSS650-5			JSS651-12		
Element Name	DL	Quantitation Value	SD	Certified Value	Quantitation Value	SD	Certified Value	Quantitation Value	SD	Certified Value
Si	0.0002	0.628	0.002	0.61	0.324	0.001	0.32	0.668	0.003	0.69
Mn	0.00001	1.157	0.002	1.16	0.441	0.001	0.44	1.320	0.002	1.33
P	0.001	0.004	0.001	0.0039	0.023	0.001	0.024	0.026	0.001	0.026
S*	0.001	0.006	0.0002	0.004 (0.006)	0.003	0.0002	0.0035	0.009	0.0003	0.0052 (0.009)
Cu	0.00005	0.205	0.001	0.20	0.0265	0.0001	0.026	0.0785	0.0003	0.076
Ni	0.0001	0.514	0.0006	0.51	0.206	0.0003	0.21	9.02	0.01	9.03
Cr	0.0001	1.93	0.008	1.93	16.3	0.04	16.18	18.3	0.04	18.26
Mo	0.0001	0.383	0.0005	0.38	0.0074	0.00006	0.008	0.056	0.0002	0.054
Ti	0.00001	0.0002	0.00001	(0.0002)	ND		(ND)	0.00114	0.00001	(0.00124)
V	0.00002	0.304	0.0004	0.30	0.0294	0.00005	(0.0302)	0.0677	0.0001	(0.0690)
Nb	0.00005	0.0006	0.00002	(0.0004)	0.0012	0.00004	(0.0016)	0.0030	0.0001	(0.0033)
Al	0.0001	0.0012	0.0001	0.001	0.0044	0.0001	0.005	0.0013	0.0001	0.003 (0.001)
Co	0.00005	0.0038	0.00003	(0.0034)	0.0337	0.0001	(0.032)	0.215	0.0003	0.22
As	0.0004	0.002	0.0003	(0.001)	0.006	0.0003	(0.005)	0.004	0.0003	(0.003)
Sn	0.0001	0.001	0.0001	(0.001)	0.003	0.0002	(0.002)	0.004	0.0002	(0.003)
Ce	0.00004	ND	—	(ND)	ND	—	(ND)	ND	—	(ND)
La	0.00001	ND	—	(ND)	ND	—	(ND)	ND	—	(ND)
Pb	0.0005	ND	—	(ND)	ND	—	(ND)	ND	—	(ND)
Sb	0.0007	ND	—	(ND)	ND	—	(ND)	ND	—	(ND)
Zn	0.00006	ND	—	(0.0001)	0.001	0.0001	(0.001)	0.001	0.0001	(0.001)
Zr	0.00003	0.0001	0.00002	(0.0001)	0.0002	0.00002	(0.0001)	ND		(ND)

DL : Detection limit (3s) obtained from the standard deviation of N=10 repeat measurements using a calibration curve blank
SD : Standard deviation with respect to N=10 repeat measurements
ND : Not detected
( ) : Results obtained using ICPS-8100
S* : Since sample preparation did not take into account sulfur analysis, this is a reference value used for the sulfur quantitation result.

NOTES:
*This Application News has been produced and edited using information that was available when the data was acquired for each article. This Application News is subject to revision without prior notice.

SHIMADZU

SHIMADZU CORPORATION. International Marketing Division
3. Kanda-Nishikicho 1-chome, Chiyoda-ku, Tokyo 101-8448, Japan Phone: 81(3)3219-5641 Fax. 81(3)3219-5710
Cable Add.:SHIMADZU TOKYO

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