

Application News

No.J112

Inductively Coupled Plasma Atomic Emission Spectrometry

Simultaneous Analysis of Trace and Major Elements in Iron and Steel by ICPE-9820

■ Introduction

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 diverse characteristics – such as strength, and heat and corrosion resistance – both process and quality control is extremely important, as these characteristics depend on the types and amounts of elements present. 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 capable of high sensitivity coupled with a wide dynamic range.

We performed elemental analysis of iron and steel standard substances using the Shimadzu ICPE-9820 multi-type ICP atomic emission spectrometer. The ICPE-9820, with its dual (axial and radial) observation, permits simultaneous analysis of elements present at the micro level to the principle component element level.

■ 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) A sample weighing 1 g was heated and dissolved in aqua regia, and the solution was then passed through a filter.
- (2) The residue was melted using an alkali flux mixture (sodium carbonate, sodium tetraborate = 2:1), and the melt product was dissolved in hydrochloric acid.
- (3) The solution was then combined with the filtrate of step (1), tartaric acid was added to reach a concentration of 5.4 % (w/v) in solution, and the volume was adjusted to 200 mL. This was used as the analytical sample. Here, yttrium (Y) was added to the sample solution for use as an internal standard at a concentration of 100 mg/L.

■ Standard Solutions for Calibration Curve

High purity iron (99.99 % or greater) was dissolved in the same manner as the samples, and using this as a base, standard solutions of analytical elements were added in stepwise fashion to prepare calibration curve samples.

■ Instrument and Analytical Conditions

For measurement, the Shimadzu ICPE-9820 multi-type ICP atomic emission spectrometer was used. The measurement conditions are shown in Table 1. The ICPE-9820 can conduct measurements while automatically switching between the two observation directions: axial direction for high sensitivity and radial direction for high-concentration analysis, thus providing

an extremely wide dynamic range. In addition, the vertical orientation of the plasma torch reduces any potential memory effects. Not only do these features permit measurement of trace-level elements to high-concentration principle component elements in the same sequence, but even in cases where the same element is present at widely different concentrations; in the case of low-alloy steel and stainless steel, analysis of both of these can be conducted at the same time.

Table 1 Analytical Conditions

Instrument	: ICPE-9820
Radio frequency power	: 1.2 kW
Plasma gas Flowrate	: 14 L/min
Auxiliary gas Flowrate	: 1.2 L/min
Carrier gas Flowrate	: 0.7 L/min
Sample introduction	: Nebulizer 10
Misting chamber	: Cyclone chamber
Plasma torch	: Torch for high salt concentration
Observation	: Axial (AX) / Radial (RD)
Measurement time	: 3 min. and 40 sec. / sample (Including rinse time)

■ Analysis

Quantitation was performed by the calibration curve method, with interferences corrected by use of an internal standard. Elements which indicated no certified or reference values were cross-checked by high-resolution type ICP (Shimadzu sequential-type model ICPS-8100) to confirm the quantitation value.

■ Analytical Results

Table 2 shows the blank sample values obtained directly following measurement of the high-concentration sample. From this, it is apparent that internal instrument contamination (memory effect) is quite low. Even when measuring a low-alloy steel sample, with low concentrations of Ni and Cr, directly after measurement of a stainless steel sample containing high concentrations of Ni and Cr, there was no evidence of analytical error due to the memory effect.

Fig. 1 shows the calibration curves for Ni and Cr. Calibration curves for the same element can be generated over different concentration ranges by changing the plasma observation direction and measurement wavelengths. This makes it possible to conduct quantitation over a wide concentration range without having to change samples or dilution factors. Calibration curve selection is conducted automatically by the software based on concentration, thereby shortening the time required to evaluate the data after completion of analysis, as well as the ancillary benefit of reducing the risk of human error in evaluation of the results.

The analytical results are shown in Table 3. For most of the elements, good results consistent with the certified value (or the confirmation results obtained using the ICPS-8100) were obtained.

■ Conclusion

Use of the ICPE-9820 permits efficient, accurate analysis of various types of iron and steel samples.

[References]

- JIS G1258-3:2014 (Iron and Steel – ICP Atomic Emission Spectrometric Method – Part 3: Determination of Various Elements – Decomposition with Acids and Fusion with Sodium Carbonate)

Table 2 Blank Sample Results Following Injection of High-Concentration Sample (Memory Effect Values) (Unit: mg/L)

	Ni	Cr
High-concentration sample solutions	1000 (20 wt%)	2000 (40 wt%)
Blank sample values obtained directly after injection of high-concentration sample	0.003 (0.00006 wt%)	0.016 (0.00032 wt%)

Note: Values in parentheses are solid conversion values.

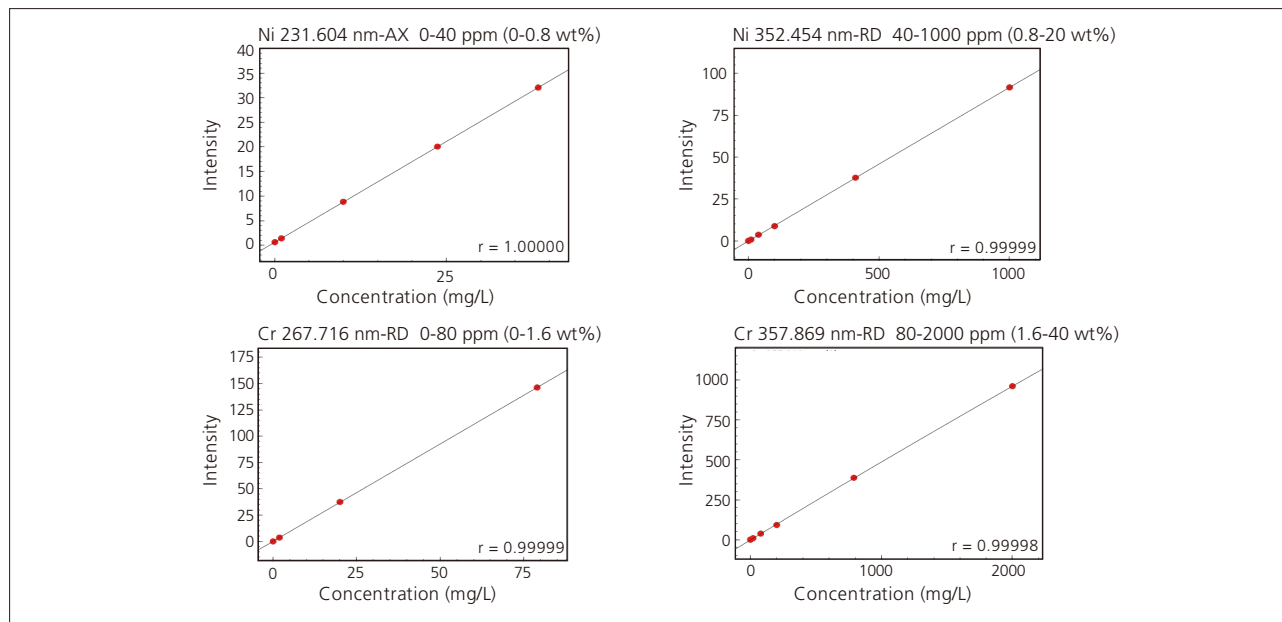


Fig. 1 Calibration Curves of Ni and Cr

Table 3 Analytical Results for Iron and Steel Certified Reference Materials (Unit: wt%)

Element	Sample name Detection Limit	JSS154-12			JSS650-5			JSS651-12		
		Quantitation Value	Standard deviation	Certified Value	Quantitation Value	Standard deviation	Certified Value	Quantitation Value	Standard deviation	Certified Value
Si	0.0002	0.623	0.002	0.61	0.325	0.002	0.32	0.671	0.002	0.69
Mn	0.00002	1.156	0.003	1.16	0.443	0.001	0.44	1.322	0.003	1.33
P	0.001	0.004	0.001	0.0039	0.022	0.0007	0.024	0.027	0.002	0.026
S *	0.002	0.006	0.0003	0.004 (0.006)	0.004	0.0003	0.0035	0.008	0.0003	0.0052 (0.009)
Cu	0.00004	0.203	0.002	0.20	0.0263	0.0001	0.026	0.0781	0.0002	0.076
Ni	0.0001	0.515	0.0004	0.51	0.207	0.0003	0.21	9.05	0.008	9.03
Cr	0.0002	1.94	0.007	1.93	16.3	0.03	16.18	18.3	0.05	18.26
Mo	0.0001	0.384	0.0004	0.38	0.0075	0.00005	0.008	0.055	0.0003	0.054
Ti	0.00001	0.0002	0.00002	(0.0002)	<		(<)	0.00113	0.00002	(0.00124)
V	0.00001	0.305	0.0003	0.30	0.0297	0.00004	(0.0302)	0.0679	0.0001	(0.0690)
Nb	0.00004	0.0005	0.00003	(0.0004)	0.0013	0.00005	(0.0016)	0.0031	0.0002	(0.0033)
Al	0.0001	0.0013	0.0001	0.001	0.0045	0.0001	0.005	0.0027	0.0002	0.003
Co	0.00003	0.0037	0.00004	(0.0034)	0.0332	0.0002	(0.032)	0.216	0.0003	0.22
Sn	0.0002	0.001	0.0001	(0.001)	0.002	0.0003	(0.002)	0.004	0.0003	(0.003)
Zn	0.00007	<		(0.0001)	0.0008	0.0002	(0.001)	0.001	0.0002	(0.001)
Zr	0.00004	0.0001	0.00002	(0.0001)	0.0002	0.00003	(0.0001)	<		(<)

Detection limit: 3 times the concentration of the standard deviation obtained from 10 repeated measurements of the calibration curve blank × Dilution factor (200)

<: Below the detection limit

SD: Standard deviation with respect to n = 2 repeat measurements

(): 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.