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Inductively Coupled Plasma Atomic Emission Spectrometer

Application News

Analysis of Trace and Major Elements in Steel by ICP-AES

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User Benefits

- The ICPE-9820 enables analysis across a wide concentration range, from trace to major elements in steel.
- Using a torch for high-salt samples and a water bubbler can suppress salt deposition when analyzing high-salt samples.
- The use of a vacuum spectrometer enables rapid startup and quick analysis in the vacuum ultraviolet region (wavelengths below 190 nm).

■ Introduction

Steel materials are widely used in a diverse range of industries, including construction, transportation, and machinery manufacturing, and are indispensable in modern society. Steel contains various elements, and the types and concentrations of these elements significantly influence its properties. Accordingly, elemental analysis is essential for quality assurance and performance control.

The elements in steel range from trace constituents to major components, with concentrations spanning a broad range from mg/L levels to percentage levels. Inductively coupled plasma atomic emission spectrometry (ICP-AES) is well suited to the analysis of elements across such a wide concentration range. It enables highly accurate quantitative analysis from trace elements to major components.

This Application News presents an analysis example of a stainless-steel certified reference material using the Shimadzu ICPE-9820 multi-type ICP atomic emission spectrometer (Fig. 1).

Fig. 1 ICPE-9820

■ Sample

Stainless-steel certified reference material NSTM SUS570-1
(Nippon Steel Technology Co., Ltd.)

■ Sample Pretreatment

One gram of the sample was weighed and dissolved by heating in 60 mL of mixed acid (hydrochloric acid: nitric acid: water = 3 : 1 : 4). The resulting solution was made up to 100 mL and used as the stock solution for measurement.

As a parallel preparation test, the same pretreatment procedure was repeated three times.

Sample for Major Element Analysis

The stock solution obtained during sample preparation was diluted 25-fold with dilute hydrochloric acid solution. Yttrium (Y) was added as an internal standard element to achieve a concentration of 20 mg/L in the measurement solution.

Sample for Trace Element Analysis

The stock solution obtained during sample preparation was diluted twofold with purified water.

During dilution, the elements to be analyzed were added to prepare standard addition sample solutions.

■ Instrument Configuration

Measurements were performed using the Shimadzu ICPE-9820. For the analysis of trace elements at low dilution ratios, a torch for high-concentration salt solution samples and a water bubbler were used to prevent sensitivity loss from salt deposition. In addition, the plasma torch is positioned vertically in the ICPE-9820, which helps reduce potential memory effects (Application News No. J112).

The chamber was equipped with a Twister spray chamber (Fig. 2), which enables finer particle-size selection and improves analytical accuracy for high-salt samples.

Details of the instrument configuration are provided in Table 1.

Fig. 2 Twister Spray Chamber

Table 1 Instrument Configuration

Instrument:	ICPE-9820
Nebulizer:	Nebulizer, 10UES
Chamber:	Twister Spray Chamber, HE
Torch:	Torch for High-concentration Salt Solution Samples
Auto Sampler:	AS-10
Other Accessories:	Water Bubbler

■ Analysis Conditions

The ICPE-9820 enables simultaneous analysis using two view orientations: axial view (AX), which offers higher sensitivity, and radial view (RD), which is suitable for high-concentration analysis.

For major element analysis, quantification was performed using the calibration curve method with the internal standard method.

For trace element analysis, quantification was performed using the standard addition method.

The analysis conditions are listed in Table 2.

Table 2 Analysis Conditions

RF Power:	1.40 kW
Plasma Gas Flowrate:	15.0 L/min
Auxiliary Gas Flowrate:	1.20 L/min
Carrier Gas Flowrate:	0.70 L/min (0.65 L/min*)
Observation Mode:	AX, RD

* Carrier gas flowrate used for trace element analysis

■ Calibration Standards

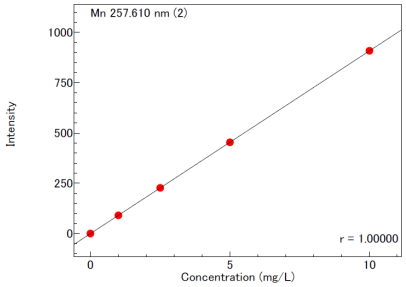
For Major Element Analysis

Commercial single-element standard solutions were diluted and mixed to prepare calibration standards. The concentrations of the analytes contained in the calibration standards for major element analysis are listed in Table 3.

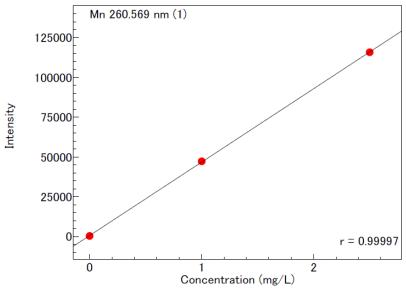
Table 3 Calibration Standards for Major Elements

Elem.	STD1 (mg/L)	STD2 (mg/L)	STD3 (mg/L)	STD4 (mg/L)	STD5 (mg/L)
Cr	0	10	25	50	100
Mn	0	1	2.5	5	10
Mo	0	2	5	10	20
Ni	0	5	12.5	25	50
Y	20 mg/L				
Fe	250 mg/L				
Nitric Acid	0.3 v/v%				
Hydrochloric Acid	2.0 v/v%				

Fig. 3 shows an example of manganese (Mn) calibration curves. For the same element, calibration curves covering different concentration ranges can be prepared by changing the view orientation or measurement wavelength. This allows the software to automatically select the appropriate calibration curve for each sample according to the element concentration, thereby reducing post-analysis data evaluation time and minimizing evaluation errors.



(a) Mn 257.610nm RD (0.625–2.5 wt%)



(b) Mn 260.569nm AX (0–0.625 wt%)

Fig. 3 Mn Calibration Curves

For Trace Element Analysis

Standard addition samples were prepared as shown in Table 4.

Table 4 Standard Addition Samples for Trace Elements

Elem.	STD1 (mg/L)	STD2 (mg/L)	STD3 (mg/L)	STD4 (mg/L)
Cu	0	5	10	-
P	0	5	10	-
Sn	0	1	2	-
Si	0	-	-	50

■ Results

For the quantitative analysis, samples prepared in triplicate during sample pretreatment were analyzed. The three sample solutions prepared during pretreatment were each measured three times within a single analytical run (N = 3), yielding a total of nine analytical results. Table 5 shows the mean solid concentration (Solid Conc.), standard deviation (SD), and repeatability (Rep.) for each element.

Quantitative results for the major elements are presented in Table 5, and those for the trace elements are presented in Table 6.

For many elements, good agreement with the certified values (Cert. Values) was obtained. The parallel tests also demonstrated good reproducibility.

Table 5 Quantitative Results for Major Elements

Elem.	Wavelength (nm)	View	Solid Conc. (%)	SD σ (%)	Rep. (%)	Cert. Values (%)
Cr	267.716	RD	18.17	0.02	0.1	18.14 ± 0.04
Mn	257.610	RD	1.96	0.002	0.1	1.95 ± 0.01
Mo	202.030	RD	2.58	0.004	0.2	2.59 ± 0.02
Ni	231.604	RD	8.21	0.007	0.08	8.21 ± 0.02

Table 6 Quantitative Results for Trace Elements

Elem.	Wavelength (nm)	View	Solid DL (%)*1	Solid Conc. (%)	SD σ (%)	Rep. (%)	Cert. Values (%)
Cu	324.754	AX	0.0002	0.0509	0.0003	0.6	0.0515 ± 0.0008
P	178.287	AX	0.0005	0.0421	0.0004	0.9	0.0423 ± 0.0005
Sn	189.989	AX	0.0001	0.0097	0.0001	1.0	0.00976 ± 0.00024
Si*2	251.611	AX	0.009	0.530	0.005	1.0	0.515 ± 0.005

*1 Solid Detection Limit (Solid DL): 3 × σ(standard deviation of STD-1) × slope of calibration curve × dilution factor

*2 Si was subjected to pretreatment with mixed acid; however, due to its limited long-term stability, it is listed here as a reference value.

■ Conclusion

In this Application News, trace and major elements in a stainless-steel certified reference material were analyzed using the ICPE-9820. The measured values showed good agreement with the certified values.

The sample introduction system, consisting of a torch for high-concentration salt solution samples and a water bubbler, effectively prevented sensitivity loss from salt deposition. At the same time, the use of a Twister spray chamber enabled finer particle-size selection, thereby improving analytical accuracy for high-salt samples.

In addition, the ICPE-9820 employs a vacuum spectrometer, enabling rapid startup and fast analysis in the vacuum ultraviolet region (wavelengths below 190 nm).

Related Applications

1. Simultaneous Analysis of Trace and Major Elements in Iron and Steel by ICPE-9820, Application News No. J112
2. Trace Element Analysis of Steel Using ICP-MS, Application News No. 01-01207-en

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