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Application News

Trace Elements Analysis in Steel by ICP-MS

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User Benefits

- Even high-matrix metal samples can be analyzed using the Aerosol Dilution System.
- Trace elements in steel can be analyzed simultaneously under a single He collision mode, reducing analysis time.
- Operating costs can be reduced by using a mini torch, which reduces argon consumption.

■ Introduction

In high-performance metallic materials such as steel, material properties are strongly influenced by trace element concentrations, making their proper control extremely important. Conventionally, inductively coupled plasma atomic emission spectrometry (ICP-AES) has been widely used for trace element analysis in metallic materials. In Japan, steel samples have been controlled and analyzed based on JIS G 1201[1)] and JIS G 1258.[2)] Recently, however, as metallic materials have become increasingly sophisticated, there has been a growing need to control elements at concentrations below those that can be addressed by conventional methods.

Inductively coupled plasma mass spectrometry (ICP-MS) is known as a highly sensitive technique for trace element analysis, but in the past it was not always suitable for analyzing high-matrix samples such as metals. In recent years, however, improvements in sample introduction systems, including gas dilution technology, have made it possible to apply ICP-MS to high-matrix samples as well.

This article describes the trace element analysis of certified reference materials for steel using the Aerosol Dilution System installed as standard in the ICPMS-2050 (Fig. 1). By reducing the amount of sample introduced into the plasma through gas dilution, matrix-induced decreases in signal intensity and signal drift could be effectively suppressed, enabling stable analytical results. The results are presented below.

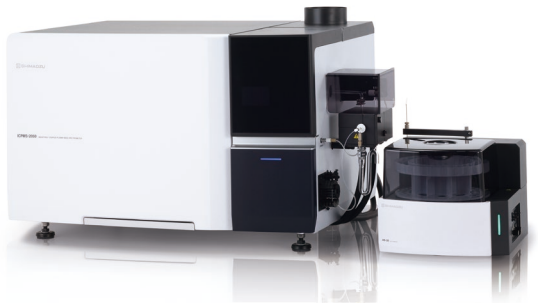

Fig. 1 ICPMS-2040/2050 and AS-20

Table 1 Concentrations in Calibration Standards

Elements	STD1 (µg/L)	STD2 (µg/L)	STD3 (µg/L)	STD4 (µg/L)
P, Cu, Sn	0	50	250	1000
Sb, Te, Pb, Bi	0	2	10	40
Nitric acid	1.5 mL / 50 mL			
Hydrochloric acid	1.5 mL / 50 mL			

■ Sample

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

■ Sample Preparation

1. Approximately 0.5 g of the sample was weighed into a fluororesin vessel, and 15 mL of a mixed acid consisting of hydrochloric acid and nitric acid in a 1:1 (v/v) ratio was added.
2. After heating on a hot plate at 80 °C for 90 minutes, the temperature was raised to 150 °C, and heating was continued for an additional 90 minutes.
3. After cooling to room temperature, the digested solution was transferred and diluted to a final volume of 50 mL with pure water.
4. 10 mL of the solution prepared in step 3 was taken, 3 mL of the mixed acid was added, and the solution was diluted to a final volume of 50 mL with pure water.

These operations were performed three times simultaneously using the same certified reference material, and triplicate sample solutions were prepared.

■ Standard Preparation

- **Calibration Standards**

Calibration standards were prepared by mixing commercially available single-element standard solutions and adding nitric acid and hydrochloric acid. The concentrations of the target elements in each calibration standard are shown in Table 1.

- **Internal Standards**

An internal standard solution was prepared by mixing single-element standard solutions of In and Tl, followed by the addition of nitric acid and hydrochloric acid to match the acid composition of the calibration standards. The concentration of each internal standard element was 1000 µg/L. During analysis, the internal standard solution was introduced using the automatic internal standard addition kit.

■ Configurations and Analytical Conditions

Table 2 lists the instrument configurations. To reduce operating costs, the analysis was performed using a mini torch, which consumes less argon than a conventional torch. In this study, only helium (He) was used to remove spectral interferences. Although ICPMS-2050 was used for this analysis, a similar analysis can also be performed with ICPMS-2040, which is dedicated to collision mode. All target elements in this study can be analyzed under a single set of cell conditions using He as the collision gas, eliminating the need to change cell conditions depending on the element and thereby shortening the analysis time.

In addition, because a 0.2 % stainless steel solution was introduced directly, each parameter was optimized to maintain a robust plasma in the presence of the matrix. The analytical conditions are shown in Table 3.

Table 2 ICP-MS System Configurations

Instrument:	ICPMS-2050
Nebulizer:	Nebulizer DC04
Chamber:	Cyclone Chamber
Torch:	Mini Torch
Sampling Cone:	Nickel
Skimmer Cone:	Nickel
Internal Standard Addition:	Automatic addition using the internal standard addition kit (sample: internal standard solution ≈ 9:1)

Table 3 Analytical Conditions

RF Power	:	1.20 kW
Sampling Depth	:	10.0 mm
Plasma Gas Flowrate	:	9.0 L/min
Auxiliary Gas Flowrate	:	1.10 L/min
Carrier Gas Flowrate	:	0.45 L/min
Dilution Gas Flowrate	:	0.40 L/min
Pump Rotation Speed	:	10 r.p.m.
Cell Gas	:	He
Cell Gas Flowrate (mL/min)	:	7.0
Cell Voltage (V)	:	-40
Energy Filter (V)	:	7.0

Table 4 Detection Limits for Each Element

Elements	Cell Gas	Internal Standard	IDL in Solution (µg/L)	IDL in Solid Sample
^{31}P	He	^{115}In	9	0.0004 (%)
^{63}Cu	He	^{115}In	0.07	4×10^{-6} (%)
^{118}Sn	He	^{115}In	0.08	4×10^{-6} (%)
^{121}Sb	He	^{115}In	0.07	0.04 (µg/g)
^{125}Te	He	^{115}In	0.09	0.05 (µg/g)
^{208}Pb	He	^{205}Tl	0.008	0.004 (µg/g)
^{209}Bi	He	^{205}Tl	0.02	0.009 (µg/g)

■ Detection Limits

Table 4 shows the limits of detection in solution and in the solid sample for each element. The limits of detection were calculated from the standard deviation (σ) of the calibration blank.

■ Analysis and Quantitative Results

In this analysis, sample preparation was carried out in triplicate, and variability within a single analytical batch was evaluated. The three sample solutions prepared during sample pretreatment were each analyzed three times within a single analytical batch (N = 3), yielding nine analytical values. Table 5 shows the concentration of each element in the solid sample (mean value), standard deviation, repeatability, and recovery.

These results demonstrated that the measured values for most elements agreed well with the certified values, and the repeatability was also excellent, confirming that the analysis was stable and exhibited low variability.

■ Conclusion

In this application news article, trace elements in a certified reference material for steel were analyzed using the ICPMS-2040/2050. Agreement with the certified values confirmed the accuracy of the analytical results. In addition, for the elements analyzed in this study, simultaneous analysis under a single condition was possible without changing the cell conditions, thereby reducing the analysis time.

By using a mini torch, the ICPMS-2040/2050 reduces argon gas consumption while enabling stable analysis of high-matrix metal samples such as steel.

References

1) JIS G 1201
2) JIS G 1258

Related Applications

1. Analysis of Trace and Major Elements in Steel by ICP-AES, Application News No. 01-01072-en

Table 5 Quantitative Results

Elements	Mean Concentration	Standard deviation, σ	Repeatability (%)	Certified Value	Recovery (%)
^{31}P	0.0424 (%)	0.0012 (%)	2.8	0.0423 ± 0.0005 (%)	100
^{63}Cu	0.0509 (%)	0.0004 (%)	0.9	0.0515 ± 0.0008 (%)	99
^{118}Sn	0.00962 (%)	0.0001 (%)	1.1	0.00976 ± 0.00024 (%)	99
^{121}Sb	6.12 (µg/g)	0.08 (µg/g)	1.4	5.83 ± 0.15 (µg/g)	105
^{125}Te	6.98 (µg/g)	0.27 (µg/g)	3.8	7.08 ± 0.39 (µg/g)	99
^{208}Pb	3.83 (µg/g)	0.04 (µg/g)	1.0	3.78 ± 0.11 (µg/g)	101
^{209}Bi	2.81 (µg/g)	0.03 (µg/g)	1.0	2.83 ± 0.04 (µg/g)	99

01-01207-EN First Edition: Sep. 2026

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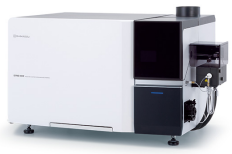

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