Analysis of Metals in High Alloy Steel by ICP-OES

Application Note

Inductively Coupled Plasma-Optical Emission Spectrometers

Author

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Introduction

The analysis of steel is important not only for manufacturers but also for the large number of steel consumers in the engineering and metallurgical industries. The information from an analysis is useful for various purposes, such as the inspection of raw materials, intermediate product and end product; environmental assessment in the factory; process control in iron and steel manufacturing; quality control and product research. However, most of the routine analyses involve Spark Emission Spectroscopy or occasionally X-Ray Fluorescence. With these techniques, the analytical signal is highly dependent on the matrix composition, and precise matching of sample and standard matrices is required for accurate results. However, such techniques lack the detection limit sometimes required.

Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-OES) is a well established analytical technique which offers better detection limits for trace element analysis, large linear dynamic range, high precision, good accuracy, and relative freedom from chemical interferences. It can be automated for high speed multi-element analysis of major and minor elements in high alloy steel.

Agilent Technologies

Experimental

Instrumental

An Agilent Liberty 200 ICP spectrometer was used for all measurements. The characteristics of the system and the operating parameters are listed in Table 1.

Table 1. Instrument Characteristics and Operating Conditions

Vacuum spectrometer		
Type	Czerny-Turner	
Focal length	0.75 m	
Grating	1800 grooves/mm	
Wavelength range	160-900 nm	
Resolution	0.018 nm	1st order
	0.009 nm	2nd order
	0.007 nm	3rd order
	0.006 nm	4th order
RF generator		
Frequency	40.68 MHz	
Oscillator	Crystal controlled	
Torch type	Standard one piece quartz torch with 1.4 mm id injector tube	
Nebulizer		
Type	Glass concentric	
Pressure	160 kPa	
Plasma power	1.2 kW	
Argon flow rate		
Plasma	12 L/min	
Auxiliary	0.75 L/min	
Sample uptake rate	1.4 mL/min	
Integration time	3 seconds	
Viewing height	Optimised on SBR	
PMT voltage	650 V	
Grating order	Default	
Filter position	Default	

Sample Preparation [1]

Half a gram of the sample was dissolved in a mixture of 15 mL of HCl and 5 mL of HNO_3. After the reaction subsided, the mixture was heated on a hot plate for 20 minutes. It was then transferred to a 100 mL volumetric flask and made up to the mark with deionized distilled water.

Standard Preparation

High purity 10 000 µg/mL ICP standards were used for the preparation of multi-element working standards. Working standards and blank were prepared with matching acid content and iron content as for the sample solution.

The working standards were prepared as follows:

Standard 1	Cr, Cu, Mn, Ni.
Standard 2	Al, Co, Mo, Ti, V.
Standard 3	Si.

Results and Discussion

Analytical Wavelength Selection

Because of the high level of Fe, Ni and Cr present in the sample solution, all analytical wavelengths were selected primarily based on freedom from Fe, Ni and Cr interferences.

The Al 396.152 nm line was selected in favor of the Al 167.081 nm line because of the interference of Fe on the 167 line, as shown in Figure 1.

Wavelength scans of the effect of 1 000 µg/mL Fe and 1000 µg/mL Cr on 1 µg/mL V show wing overlap of Fe and Cr on the V 282.402 nm line, as illustrated in Figure 2. But the V 292.464 nm line is free from Fe interference. An elevated background caused by Mn can be treated by background correction, as shown in Figure 3.

An example of the spectral scan of the sample solution and a 100 µg/mL Mo standard solution at the Mo 202.030 nm line is shown in Figure 4.

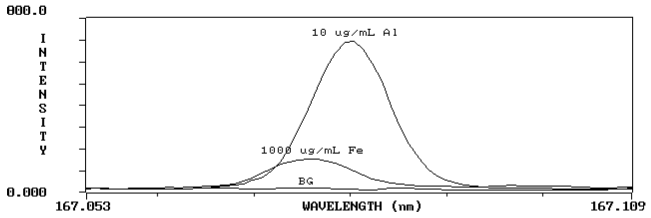

Figure 1. Spectral scan of 1000 µg/mL Fe and 10 µg/mL Al at the Al 167.081 nm line.

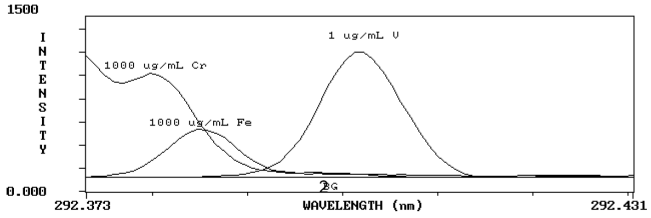

Figure 2. Spectral scan of 1000 µg/mL Cr, 1000 µg/mL Fe and 1 µg/mL V at the V 292.402 nm line.

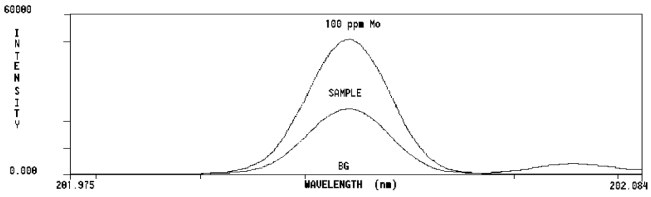

Figure 3. Spectral scan of 1000 µg/mL Mn and 1 µg/mL V at the V 292.464 nm line.

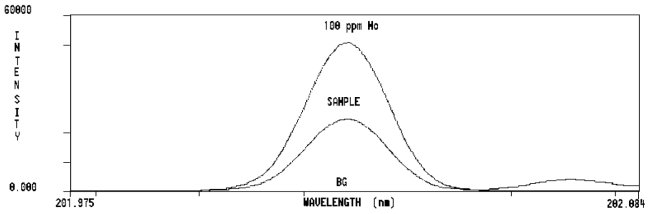

Figure 4. Spectral scan of the steel sample solution and 100 mg/mL Mo at the Mo 202.030 nm line.

The detection limits for the analytical wavelengths used are listed in Table 2.

Table 2. Detection Limits in 0.5 wt% Fe Solution

Element	Wavelength (nm)	Detection limit (ng/mL)
Ni	231.604	14
Cr	267.716	6
Ti	334.941	1
Mo	202.030	8
Mn	293.306	6
Si	288.158	12
Al	396.152	5
V	292.464	20
Co	228.616	6
Cu	324.754	3

Results

The analysis of an NIST SRM high alloy steel sample was performed with the operating conditions listed in Table 1. The results of the analysis are listed in Table 3. The precision of the measurements ranged from 0.1 to 1 %RSD. The measured values are in good agreement with the certified values.

Table 3. Results of NIST SRM High Alloy Steel 348a Analysis

Element	Measured value (wt%)	Certified value (wt%)
Ni	23.9	24.2 ± 0.1
Cr	15.0	14.8 ± 0.1
Ti	2.12	2.12 ± 0.07
Mo	1.18	1.18 ± 0.03
Mn	0.64	0.64 ± 0.02
Si	0.41	0.43 ± 0.01
Al	0.25	0.24 ± 0.01
V	0.22	0.23 ± 0.01
Co	0.15	0.15 ± 0.01
Cu	0.15	0.14 ± 0.01

Conclusion

The determination of major and minor elements in high alloy steel by ICP-OES has been described. The values found compare well with the certified values. The precision of the measurements ranged from 0.1 to 1 %RSD.

References

1. A. F. Ward, L. F. Marciello,"Analysis of metal alloys by Inductively Coupled Argon Plasma Optical Emission Spectrometry", Anal. Chem., 1979, 51, 2264-2272.

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Printed in the USA
November 1, 2010
ICPES-6

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