Application Brief

Geochemistry, Mining and Metals

Agilent

Trusted Answers

Measuring Multiple Elements in Steel Samples Using FAAS With Automated Standard Preparation and Sample Dilution

Ensuring the quality of steel

Steel manufacturers test the quality of their products by measuring several elements in the steel to ensure the grade and performance of their final product. Different grades of steel have different specifications for elemental content.

Laboratories are looking beyond whether an instrument can 'do the job' to whether a specific instrument can improve their sample throughput, lower their costs, simplify sample preparation and instrument operation, and deliver reliable results throughout the analysis of a large batch of samples.

Complex matrices, such as steel samples, introduce a lot of chemical or physical interferences to flame atomic absorption spectroscopy measurements. Manual dilution of samples is the conventional way to prevent these interferences causing incorrect results. Unfortunately, such dilutions can be complex and tedious. Manually diluting samples can also cause sample contamination.

Standard addition is a calibration method that can replace the conventional standard curve calibration method to help eliminate chemical and physical interferences. Manually preparing standard additions is time-consuming and laborious due to the need to prepare multiple solutions for each sample, with variable amounts of known concentrations of the analyte.

The Agilent Sample Introduction Pump System (SIPS) accessory provides automation for flame AA analysis, increasing sample throughput, simplifying sample preparation and instrument operation. The accessory is available in a single pump version (SIPS 10) and a dual pump version (SIPS 20). The SIPS 10 enables automatic preparation of standard solutions from a single standard solution and auto dilution of over-range samples. The SIPS 20 also automates the preparation of standard additions and spikes (for spike recovery measurements) and can add matrix modifiers and internal standards.

Table 1. The instrument settings for the analysis.

Element	Wavelength (nm)	Slit Width (nm)	Lamp Current (mA)	Oxidant gas/gas	Oxidant flow (L/min)	Acetylene flow (L/min)
Co	240.7	0.2	7.0	Air/Acetylene	13.5	2.0
Mn	403.1	0.2	5.0	Air/Acetylene	13.5	2.0
Ni	352.5	0.2	4.0	Air/Acetylene	13.5	2.0
Cr	429.0	0.5	7.0	Nitrous oxide/ Acetylene	11.0	6.6
Mo	313.3	0.5	7.0	Nitrous oxide/ Acetylene	11.0	7.6

The SIPS 20 is ideal for the analysis of steel samples with complex matrices, as it automates and simplifies the standard addition calibration method. An operator needs to prepare just one bulk standard solution and present it to the right pump of the SIPS 20. The desired additions amounts are entered into the SpectrAA software and the right pump on the SIPS 20 automatically adds the standard to the samples as they are presented. It significantly reduces the manual labour of preparing large batches of samples and improves the analysis efficiency and increases the sample throughput.

Sample preparation

0.1000 g of two reference steel samples; NIST-348a and BCS-336 was accurately weighed into a digestion vessel. 2 mL of HCl and 1 mL of HNO_3 was added, and the reaction proceeded to take place. After approximately 10 minutes, when the reaction was complete, 2 mL of perchloric acid was added and the sample heated on a hot plate until white smoke was generated. The solution was then diluted to 100 mL with distilled water.

Molybdenum can experience interferences from the high concentration of Iron (Fe) in steels, it was therefore necessary to add 0.5% $AlCl_3$ to remove the interference.

Instrument setup

Table 1 lists the instrument operation parameters. The analysis used the default gas flows as provided in the SpectrAA instrument software.

Result accuracy

Recoveries of the measured values were within ±10% of the certified values, as shown in Table 2. Compared with the conventional standard addition method, the SIPS accessory removed most of the solution preparation work, improving analytical efficiency.

In this example, the use of the SIPS 20 accessory enabled automated inline standard addition, saving manual dilution and other tedious work required by the conventional standard addition method, resulting in a much higher analytical efficiency.

Table 2. Measured concentration versus the certified concentration of two of reference steel samples.

Element	NIST-348a Measured value (%)	NIST-348a Certified value (%)	Recovery (%)	BCS-336 Measured value (%)	BCS-336 Certified value (%)	Recovery (%)
Co	0.15	0.15	100%	0.069	0.063	109%
Mn	0.62	0.64	97%	0.77	0.81	95%
Ni	23.2	24.2	96%	9.36	9.48	99%
Cr	14.0	14.8	95%	17.0	17.6	96%
Mo	1.15	1.18	97%	2.25	2.43	93%

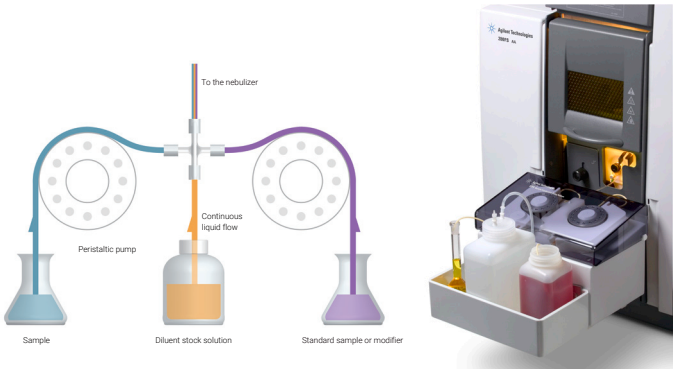

Figure 1. How the SIPS accessory works (left) and the accessory mounted on the front of an AA instrument (right).

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