



Analysis of 14 Trace Elements in Stainless Steel

Joël Sire, Daniel Legain
Serma Technologie
France

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1 Introduction

This Application Note describes a method for the routine analysis of Stainless Steel samples. Fourteen trace elements, with a wide range of concentrations, were determined using one sample preparation method.

2 Principle

2.1 Technique used

The elemental analysis of these samples was undertaken by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES). The sample is nebulized then transferred to an argon plasma. It is decomposed, atomized and ionized whereby the atoms and ions are excited. We measure the intensity of the light emitted when the atoms or ions return to lower levels of energy. Each element emits light at characteristic wavelengths and these lines can be used for quantitative analysis after a calibration.

2.2 Wavelength choice

The choice of the wavelength in a given matrix can be made using the "profile" function, or by using Win-IMAGE, which is rapid semi-quantitative analysis mode using multiple wavelengths. The principle is the same in either case: record the scans of analytes at low concentration, and of the matrix. By superimposing the spectra, we see possible interferences.

3 Sample preparation

3.1 Required solutions

Hydrofluoric acid 40% (d = 1.13),
Nitric acid 68% (d = 1.4),
Hydrochloric acid 36% (1.18),
Ammonium phosphate,
Single element standard solutions at 1 g/L of
Al, Co, Cu, Mn, Mo, Nb, P, Si, Ti, V, W,
Iron powder (Johnson Matthey),
Nickel and Chromium powder (Merck).

3.2 Calibration standards

Use six 250 mL PTFE beakers.

Beaker STD0: Weigh, exactly, 1250 mg of Fe.
Beaker STD1: Weigh, exactly, 1244.6 mg of Fe, 1.25 mg of Cr and Ni.
Beaker STD2: Weigh, exactly, 1225 mg of Fe, 5 mg of Cr and Ni.
Beaker STD3: Weigh, exactly, 1167 mg of Fe, 25 mg of Cr and Ni.
Beaker STD4: Weigh, exactly, 876 mg of Fe, 150 mg of Cr and Ni.
Beaker STD5: Weigh exactly 636.5 mg of Fe, 250 mg of Cr and 200 mg of Ni.
Beaker STD6: Weigh, exactly, 437.5 mg of Fe, 325 mg of Cr and 250 mg of Ni.

In each beaker, add 6.5 mL of HF then, with care, 12.5 mL of HNO₃ and 12.5 mL of HCl. If necessary, heat on sand bath to finish the dissolution and let it cool. Transfer the solution to six 500 mL PTFE flasks and follow the instructions described below.

Flask 1 (STD0):

Dilute to volume with deionized water to obtain a solution of 2500 mg/L to be used as the blank sample.

Flask 2 (STD1):

Add 625 µL of the 1 g/L Mo, W and Mn standard solutions.
Add 1.25 mL of 0.1 g/L Al, Si, Nb, Ti, V, Cu, Co standard solutions.
Add 300 µL of the 0.1 g/L P standard solution.
Dilute to volume with deionized water.

**Flask 3 (STD2):**

Add 2.5 mL of the 1 g/L Mo, W and Mn standard solutions.

Add 1 mL of 1 g/L Al, Si, Nb, Ti, V, Cu, Co standard solutions.

Add 1.20 mL of the 0.1 g/L P standard solution. Dilute to volume with deionized water.

Flask 4 (STD3):

Add 5 mL of the 1 g/L Mo, W and Mn standard solutions.

Add 2.5 mL of 1 g/L Al, Si, Nb, Ti, V, Cu, Co standard solutions.

Add 250 µL of the 1 g/L P standard solution. Dilute to volume with deionized water.

Flask 5 (STD4):

Add 12.5 mL of the 1 g/L Mo, W and Mn standard solutions.

Add 5 mL of 1 g/L Al, Si, Nb, Ti, V, Cu, Co standard solutions.

Add 500 µL of the 1 g/L P standard solution. Dilute to volume with deionized water.

Flask 6 (STD5):

Add 30 mL of the 1 g/L Mo, W and Mn standard solutions.

Add 10 mL of 1 g/L Al, Si, Nb, Ti, V, Cu, Co standard solutions.

Add 1 mL of the 1 g/L P standard solution. Dilute to volume with deionized water.

Flask 7 (STD6):

Add 50 mL of the 1 g/L Mo, W and Mn standard solutions.

Add 15 mL of 1 g/L Al, Si, Nb, Ti, V, Cu, Co standard solutions.

Add 2.5 mL of the 1 g/L P standard solution. Dilute to volume with deionized water.

3.3 Sample preparation

Weigh, exactly, 1g of sample to analyze.

Add 5 mL of HF then add carefully 10 mL HNO₃ and 10 mL of HCl. Heat on a sand bath until the sample is dissolved and transfer to plastic 100 mL flask. Take care that the temperature does not exceed 55 °C to avoid the loss of Si during digestion of SiF₆, which is volatile. Dilute to volume with deionized water. A 10 g/L solution is obtained. Dilute it 4 times to obtain a 2.5 g/L solution, suitable for analysis.

4. Instrument specification

The work was done on a JY 80 instrument and is also applicable to the JY 180 and ULTIMA 2CHR ICP spectrometers. The specifications of this instrument are listed on the following page. The specifications of this instrument are listed in Tables 1 to 3.

Table 1: Specification of spectrometer: monochromator

Parameters	Specifications
Mounting	Czerny Turner
Focal length	1m
Nitrogen purge	Yes
Variable resolution	Yes
Grating number of grooves	3600 gr/mm

Table 2: Specification of spectrometer: polychromator

Parameters	Specifications
Mounting	Paschen Runge
Focal length	1m
Nitrogen purge	Yes
Grating number of grooves	3000 gr/mm

Table 3: Specification of RF Generator

Parameters	Specifications
Type of generator	Solid state
Observation	Radial
Frequency	40.68 MHz
Control of gas flowrate	by computer
Control of pump flow	by computer
Cooling	air



5 Operating conditions

The operating conditions are listed in Table 4 below.

Table 4: Operating conditions

Parameter	Condition
RF Generator power	1000 W
Plasma gas flowrate	12 L/min
Auxiliary gas flowrate	0 L/min
Sheath gas flowrate	0.2 L/min
Nebulizer flowrate	3.2 bars (48 psi)
Sample uptake	1.8 mL/min
Type of nebulizer	Cross Flow
Type of spray chamber	Scott
Argon humidifier	Yes
Injector tube diameter	3.0 mm

6 Wavelength selection and analytical conditions

For each element, the line with the highest sensitivity was used for analysis, because there were no problems with interferences. For all the elements the conditions were the same.

Table 5: Analytical conditions

Element	Slits (μm)	Analysis mode	Integration time (sec)
All elements	40 x 20	Direct peaking	10

Table 6: Wavelengths used on the simultaneous spectrometer

Element	Wavelength (nm)
Co	228.616
Cr	267.716
Cu	324.754
Fe	259.940
Mn	257.610
Mo	202.032
Nb	316.340
Ni	231.604
Si	251.611
Ti	337.279
V	310.230
W	207.911

Table 7: Wavelengths on the sequential spectrometer

Element	Wavelength (nm)
Al	308.215
P	178.226

The normalization or 100% method was used. The sum of the concentrations obtained is considered to be equal to 100%. The calibration curve should be undertaken with great care. This method can be used for the analysis of all types of steel alloys.

7 Results

7.1 Calibration

The standards prepared are shown in Table 8 below.

**Table 8: Standard concentration**

	STD0	STD1	STD2	STD3	STD4	STD5	STD6
Fe	2500 mg/L 100 %	2486.2 mg/l 99.6 %	2450 mg/l 98.0 %	2334 mg/l 93.3 %	1752 mg/l 70.0 %	1273 mg/l 50.9 %	875 mg/l 35.0 %
Cr	0	2.5 mg/l 0.1 %	10 mg/l 0.4 %	50 mg/l 2 %	300 mg/l 12 %	500 mg/l 20 %	600 mg/l 24 %
Ni	0	2.5 mg/l 0.1 %	10 mg/l 0.4 %	50 mg/l 2 %	300 mg/l 12 %	400 mg/l 16 %	500 mg/l 20 %
Mo	0	1.25 mg/l 0.05 %	5 mg/l 0.2 %	10 mg/l 0.4 %	25 mg/l 1 %	60 mg/l 2.4 %	100 mg/l 4 %
Mn	0	1.25 mg/l 0.05 %	5 mg/l 0.2 %	10 mg/l 0.4 %	25 mg/l 1 %	60 mg/l 2.4 %	100 mg/l 4 %
W	0	1.25 mg/l 0.05 %	5 mg/l 0.2 %	10 mg/l 0.4 %	25 mg/l 1 %	60 mg/l 2.4 %	100 mg/l 4 %
Al	0	0.25 mg/l 0.01 %	2 mg/l 0.08 %	5 mg/l 0.2 %	10 mg/l 0.4 %	20 mg/l 0.8 %	30 mg/l 1.2 %
Si	0	0.25 mg/l 0.01 %	2 mg/l 0.08 %	5 mg/l 0.2 %	10 mg/l 0.4 %	20 mg/l 0.8 %	30 mg/l 1.2 %
Nb	0	0.25 mg/l 0.01 %	2 mg/l 0.08 %	5 mg/l 0.2 %	10 mg/l 0.4 %	20 mg/l 0.8 %	30 mg/l 1.2 %
Ti	0	0.25 mg/l 0.01 %	2 mg/l 0.08 %	5 mg/l 0.2 %	10 mg/l 0.4 %	20 mg/l 0.8 %	30 mg/l 1.2 %
V	0	0.25 mg/l 0.01 %	2 mg/l 0.08 %	5 mg/l 0.2 %	10 mg/l 0.4 %	20 mg/l 0.8 %	30 mg/l 1.2 %
Cu	0	0.25 mg/l 0.01 %	2 mg/l 0.08 %	5 mg/l 0.2 %	10 mg/l 0.4 %	20 mg/l 0.8 %	30 mg/l 1.2 %
Co	0	0.25 mg/l 0.01 %	2 mg/l 0.08 %	5 mg/l 0.2 %	10 mg/l 0.4 %	20 mg/l 0.8 %	30 mg/l 1.2 %
P	0	0.06 mg/l 0.002 %	0.20 mg/l 0.008 %	0.5 mg/l 0.02 %	1 mg/l 0.04 %	2 mg/l 0.08 %	5 mg/l 0.2 %



7.2 Accuracy

The accuracy of the method was determined by analyzing a certified reference material (CRM JK27a).

Table 9: Results for accuracy

Element	Measured concentration (%)	Expected Concentration (%)
Cr	16.83	16.76
Ni	12.00	12.04
Mo	2.57	2.53
Mn	1.58	1.59
W	0.023	0.028
Si	0.40	0.41
Ti	0.0006	ND
V	0.041	0.04
Cu	0.19	0.20
Co	0.095	0.09
P	0.022	0.02

7.3 Repeatability

The repeatability is illustrated by three measurements of 5 seconds on a certified reference material BCS 466.1.

Table 10: Results for repeatability

Element	Obtained Concentration (%)	RSD(%)	Expected Concentration (%)
Al	0.044	2.9	ND
Co	0.023	0.92	ND
Cu	0.023	0.65	ND
Mn	0.69	0.67	0.698
Mo	2.21	0.71	2.19
Nb	0.030	1.3	0.029
P	0.026	11	0.020
Si	0.355	1.4	0.50
V	0.025	0.46	ND
W	0.003	12	ND

7.4 Reproducibility

The reproducibility of the method was tested over 2 months. During these two months, six measurements were made (01/6, 01/9, 01/15, 02/16, 02/22) on BCS 466.

Table 11 below summarizes the results.

Table 11: Results for reproducibility

Element	Obtained concentration (%)	RSD (%)	Expected conc. (%)
Al	0.041	13	ND
Co	0.023	2.8	ND
Cu	0.023	2.2	ND
Mn	0.70	1.23	0.698
Mo	2.21	0.85	2.19
Nb	0.031	5.7	0.029
P	0.021	17	0.020
Si	0.35	4.3	0.50
V	0.02	3	ND
W	0.003	20	ND

8 Conclusion

The results presented here show that the accuracy, repeatability and reproducibility are very good for the routine analysis of stainless steel. No particular difficulties were encountered in the analysis of the samples.

info-sci.fr@horiba.com
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France: HORIBA Jobin Yvon S.A.S., 16-18 rue du Canal, 91165 Longjumeau Cedex - Tel: +33 (0)1 64 54 13 00 - Fax: +33 (0)1 69 09 07 21 - Email: info-sci.fr@horiba.com
USA: HORIBA Jobin Yvon Inc., 3880 Park Avenue, Edison, NJ 08820-3012. Toll-free: +1-866-jobinyvon - Tel: +1-732-494-8660 - Fax: +1-732-549-5125
 Email: info-sci.us@horiba.com
Japan: HORIBA Ltd., Scientific Instruments Sales Dept., Alte-Building Higashi-Kanda, 1-7-8 Higashi-Kanda, Chiyoda-ku, 101-0031 Tokyo - Tel: +81 (0)3 3861 8231
 Fax: +81 (0)3 3861 8259 - Email: info-sci.jp@horiba.com
Germany: HORIBA Jobin Yvon GmbH, Hauptstrasse 1, 82008 Unterhaching - Tel: +49 (0)89 46 23 17-0 - Fax: +49 (0)89 46 23 17-99 - Email: info-sci.de@horiba.com
Italy: HORIBA Jobin Yvon Srl, Via Cesare Pavese 35/AB, 20090 Opera (Milano) - Tel: +39 0 2 57 60 30 50 - Fax: +39 0 2 57 60 08 76 - Email: info-sci.it@horiba.com
UK: HORIBA Jobin Yvon Ltd, 2 Dalston Gardens, Stanmore, Middlesex HA7 1BQ - Tel: +44 (0)20 8204 8142 - Fax: +44 (0)20 8204 6142 - Email: info-sci.uk@horiba.com
China: HORIBA Jobin Yvon SAS, Room 1801, Capital Tower No.6, Jianguomenwai Av., Chaoyang District, Beijing 100022 - Tel: +86 (0)10 8567 9966 - Fax: +86 (0)10 8567 9066
 Email: info-sci.cn@horiba.com
Other Countries: Tel: +33 (0)1 64 54 13 00 - Email: info.sci@horiba.com