



ICP Figures of Merit

Odile Hirsch
 Jobin Yvon S.A.S., Horiba Group
 Longjumeau, France

Keywords: General

1 Introduction

ICP-AES is a multi-element analytical technique, which is well accepted. As a way to differentiate the performances of various ICP-AES instruments, J.M. Mermet a renowned spectroscopist published an article in Applied Spectroscopy, Vol. 49, No.10 (1995) entitled:

ICP Emission Spectrometers: 1995 Analytical Figures of Merit.

After defining the analytical figures of merit, he gives a way of measuring them and scales of evaluation. This Application Note summarizes the publication and gives the results for JY instruments.

2 Analytical figures of merit

2.1 Analytical figures of merit

The main analytical figures of merit are listed below.

- number of elements
- selectivity
- repeatability
- long-term stability
- robustness
- limits of detection
- accuracy

2.2 Evaluation of the analytical figures of merit

In the following table is the information on the experiments to characterize the figures of merit.

Table 1: Figures of merit

Figures of merit	Diagnosis	Line, element	Measurement
Selectivity	Resolution	Ba II 230.424 nm	Line profile
Repeatability	RSD of the signal	Mg I 285.213 nm	RSD
Long-term stability	Warm-up time	Ar I 404.442 nm	Time
		Ba II 455.403 nm	
		Zn II 206.200 nm	
	Stability	Ar I 404.442 nm	RSD
		Ba II 455.403 nm	
		Zn II 206.200 nm	
Robustness	Mg II/ MgI	Mg I 285.213 nm	Net line intensity ratio
		Mg II 280.270 nm	
Limits of detection	SBR	Ni II 231.604 nm	SBR
	RSD	Background at 230.400 nm	RSD with optimized integration time

**Notes:****Repeatability:** 15 replicates of Mg I 285 nm.**Conditions:** default values for repeatability, long-term stability, and robustness.**LOD:** made after optimization of conditions (observation height, integration time...).

Integration time can be up to 40 s.

For optimal accuracy, the following procedures should be provided in the software:

- selection of the number of standards kept for the calibration.
- rejection of outliers
- weighting the measurements
- standard addition method
- no blank

2.3 Scale

The scale of performance is from 1 to 5 with 5 being the best result.

Table 2: Scale of mark

Mark	Figure of merit
5	Outstanding
4	Excellent
3	Good
2	Acceptable
1	Needs improvement

Table 3 defines the range of values that correspond to the marks

Table 3: Range of values

Mark	5	4	3	2	1
Wavelength range (nm)	120 - 770	165 - 770	165 - 450	190 - 770	190 - 450
Resolution	< 5 pm	5 - 7 pm	7 - 11 pm	11 - 16 pm	> 16 pm
RSD signal	< 0.2 %	0.2 - 0.5 %	0.5 - 0.8 %	0.8 - 1.2 %	> 1.2 %
Warm-up time	< 15 min	15 - 30 min	30 - 60 min	60 - 90 min	> 90 min
Long term RSD	< 1 %	1 - 2 %	2 - 4 %	4 - 10 %	> 10 %
Mg II/ Mg I	> 10	10 - 8	8 - 6	6 - 4	< 4
SBR Ni 1 ppm	> 30	30 - 20	20 - 10	10 - 2	< 2
RSD bkgd	< 0.3 %	0.3 - 0.6 %	0.6 - 1.0 %	1.0 - 1.5 %	> 1.5 %
Calibration	4 +	3 +	2 +	1 +	conventional
	Standard Addition	Weighting	No blank	Nbr of std	Rejection of outliers



3 Sample preparation

A solution of 5 mg/L of Zn, Mg, Ba, and Ni in 5% HNO₃ was obtained by dilution of 1 g/L stock solutions

4 Instrument specification

The work was done on a JY ULTIMA. The specifications of this instrument are listed below.

Table 4: Specification of spectrometer

Parameters	Specifications
Mounting	Czerny-Turner
Focal length	1 m
Thermoregulation	Yes
Variable resolution	Yes
Nitrogen purge	Yes
Grating number of grooves	2400 gr/mm
1st order resolution	0.010 nm
2nd order resolution	0.005 nm
Order	2nd order

Table 5: Specification of RF Generator

Parameters	Specifications
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 the table below.

Table 6: 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 gas flowrate	0.7 L/min
Nebulizer flowrate	3.4 bars (51 psi)
Sample uptake	1 mL/min
Type of nebulizer	Concentric
Type of spray chamber	Cyclonic
Argon humidifier	No
Injector tube diameter	3.0 mm

6 How to undertake the test

All elements are measured with a background correction.

Selectivity: perform an autosearch of the Ba 230 nm line, with narrow slits, for example 20 μm x 15 μm . Calculate the width at half height.

Repeatability: take 15 replicates of the Mg 285 nm line. The integration time is usually 4 to 8 s, slits 20 μm x 80 μm .

Long term stability: the warm up time and stability test can be run at the same time. Perform an analysis of 3 replicates of the Ba 455 nm, Zn 206 nm and Ar 404 nm over several hours, for example 4 hours. No calibration should be made.

All elements in direct peaking mode, 4 s integration time, and slits 20 μm x 80 μm .

Robustness: The two lines of Mg should have the same high voltage (522 V for example) and gain. Undertake an analysis of the two lines. Mode direct peaking, 4 s integration time.

Limits of detection: the SBR and the RSD₀ of Ni are measured with slits 20 μm x 15 μm , 16 s integration time, direct peaking mode. Use the Print option in order to calculate the RSD₀.



7 Results

These tests have been performed many times and the following table shows what can be expected from the JY ULTIMA

Table 7 : ULTIMA results

Figure of merit	Result For Grade "5"	ULTIMA
Wavelength	120- 800 nm	120- 800 nm
Resolution	< 5 pm	< 5 pm
RSD signal	< 0.2 %	< 0.2 %
Warm-up time	< 15 min	< 10 min
Long term RSD	RSD < 1 %	RSD < 2 %
Mg II/Mg I	> 10	> 10
SBR Ni 1 ppm	>30	>30
RSD bkgd	< 0.3 %	< 0.3 %
Calibration	Standard addition	Yes
	Weighting	Yes
	No blank	Yes
	Nbr of standards	Yes
	Rejection of outliers	Yes

8 Summary

The overall performance of the JY ULTIMA consistently gives the highest rating according to Mermet's criteria of "Robustness". This gives the user of JY instruments high performance with respect to limits of detection, stability and accuracy. The high efficiency RF system ensures top performance where difficult samples are being analyzed, e.g., methanol.



In the USA:
Jobin Yvon Inc.
3880 Park Avenue
Edison, NJ 08820
Tel: 1-732-494-8660
Fax: 1-732-494-8796
E-mail:
emission@jyhoriba.com

In France:
Jobin Yvon S.A.S.
16-18, rue du Canal
91165 Longjumeau Cedex
Tel: (33) 1/64 54 13 00
Fax: (33) 1/69 09 90 88

In Japan:
Horiba Ltd.
2 Miyanohigashi, Kisshoin
Minami-ku, Kyoto 601-8510
TEL: (81) 75 313 8121
FAX: (81) 75 321 5725
www.horiba.com

Germany: (49) 89/46 23 17-0
Italy: (39) 2/57 60 56 90
U.K.: (44) 20/82 04 81 42

China: (86) 10/6836 6542
Spain: (34) 91/724 16 57
Other Countries: Contact JY S.A.S.

1-877-JYHORIBA

www.jyhoriba.com



HORIBAGROUP

EMISSION • FLUORESCENCE • FORENSICS • GRATINGS & OEM • RAMAN • OPTICAL SPECTROSCOPY • THIN FILM