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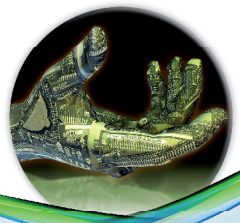
Application Note



Material Science

ICP

ICP63

Trace analysis in Nd Fe B Samples for Magnetic Materials using ICP-OES

Li Zhaofen, HORIBA Scientific China, Shanghai

Dr. Matthieu Chausseau, HORIBA Jobin Yvon, 16-18 rue du canal, 91165 Longjumeau Cedex, France,

As applications of Nd Fe B magnetic materials become more and more crucial, the quality of materials is more and more important. Not only has the quality of raw material to be considered but also the quality of intermediate and final products. Quality of products can be highly improved if analysis of raw materials can be performed as well as intermediate and final products. The production phase can also be shortened and thus its cost reduced.

High resolution ICP-OES is well suited for such application as it will help to solve all spectral interferences occurring to the line-rich spectrum emitted by Nd and Fe all over the spectral range. This application describes the analysis of trace elements in Nd Fe B matrix with evaluation of stability and accuracy.

Sample preparation

0.1g of sample was dissolved in 5 ml H_2O and 5 ml HNO_3 then volume was made up to 100 mL. All standard solutions were prepared using Spex CertiPrep single element standard solution. For improved accuracy, the standard addition technique was used with addition of 0.5, 1 and 1.5 mg/L of Al, Ba, Bi, Ca, Co, La, Mn, Ni, S, Si and Zn.

Operating conditions

The characteristics of the ULTIMA 2 High Resolution spectrometer used for this study are given in Table 1. Note that this instrument is equipped with the dual grating system that improves resolution for Rare Earth Elements based applications.

Figure 1: ULTIMA 2 High Resolution ICP-OES

Table 1. Characteristics of ULTIMA 2 ICP-OES Spectrometer

Optical mounting	Czerny-Turner
Focal length	1 meter
Gratings	Back-to-back gratings used in the 1st order 4320 g/mm for 160 - 430 nm 2400 g/mm for 430 - 800 nm
Resolution	< 5 pm for 120 - 430 nm < 10 pm for 430 - 800 nm
Thermoregulation	32 ± 0.1°C
RF Generator	40.68 MHz solid state, water cooled
Torch	Vertical with Radial Viewing and Total plasma View*

* Total Plasma View: Measurement of the whole Normal Analytical Zone for enhanced sensitivity and reduced matrix effects

A concentric glass nebulizer and a cyclonic spray chamber were used to get the highest sensitivity. All details on the introduction system are given in Table 2.

Table 2. Specification of the sample introduction system

Nebulizer	Glass concentric
Spray chamber	Glass cyclonic
Sample uptake	1 mL/min
Injector tube inner diameter	3 mm
Pump tubing	Black-black pump tubing for sample Grey-grey pump tubing for drain

All plasma parameters were optimized for sensitivity and robustness and are given in Table 3.

Table 3. Operating conditions

Power	1000 W
Plasma gas	12 L/min
Auxiliary gas	0 L/min
Sheath gas	0.3 L/min
Nebulizer flow	0.8 L/min (2.9 bars)
Pump speed	15 rpm

Acquisition was done using 3 replicates with Max mode and 2s integration time for analyte. A 20 µm / 15 µm slit combination was used for all lines.

Results

Line selection

Lines used are given in Table 4. Selection of lines was done after performing profiles on samples to identify potential spectral interferences.

Table 4: List of lines used for the analysis

Element	Wavelength (nm)	Element	Wavelength (nm)
Al	308.215	Mn	257.610
Ba	455.403	Ni	221.647
Bi	223.061	S	181.978
Ca	317.933	Si	251.611
Co	228.616	Zn	213.856
La	333.749		

Different profiles are shown in Figures 1 to 5. These profiles correspond to lines free from spectral interferences. Some other lines were suffering from spectral interferences.

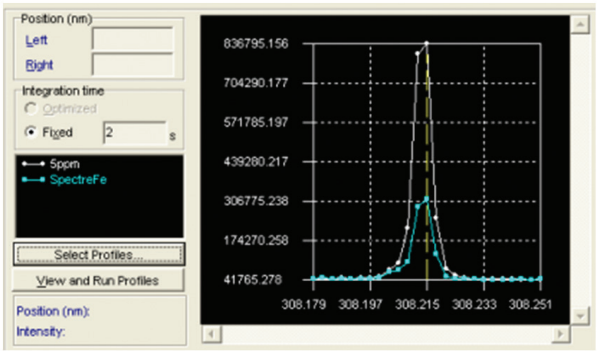


Figure 1: Profile for Al 308.215 nm.

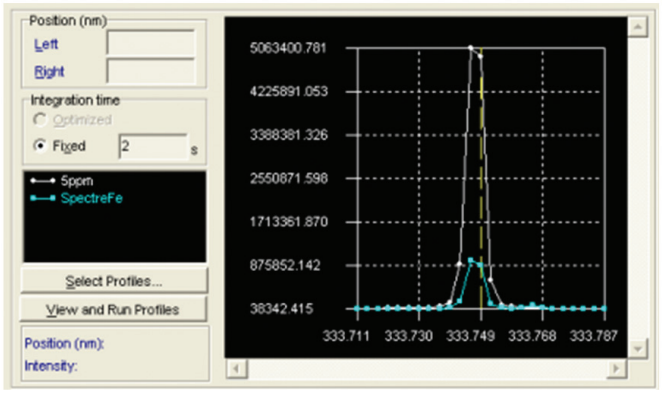


Figure 2: Profile for La 333.749 nm.

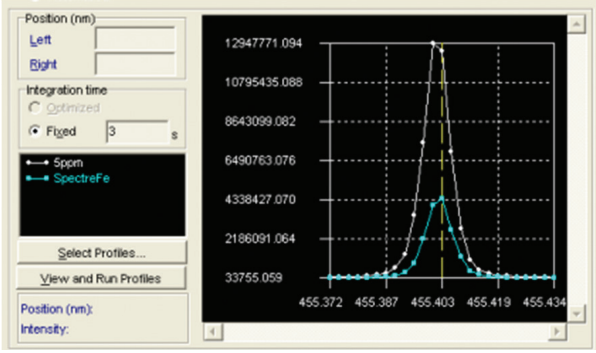


Figure 3: Profile for Ba 455.403 nm.

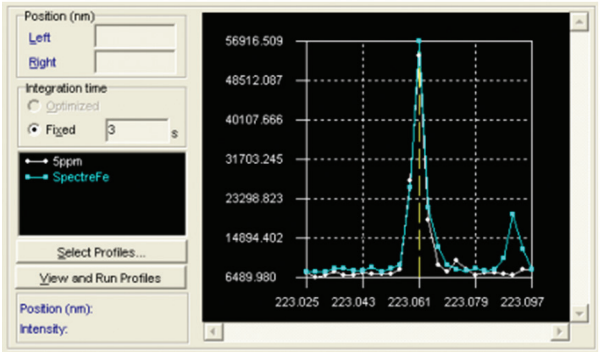


Figure 4: Profile for Bi 223.061 nm.

Calibration

The standard addition technique was used. Additions of 0.5, 1 and 1.5 mg/L were done for all elements. All calibration curves were validated with correlation coefficient r>0.999. Some calibration curves are displayed in Figures 6 to 8.

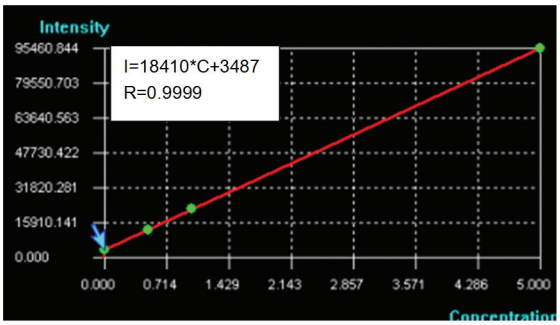


Figure 6: Calibration curve for Al 308.215 nm.

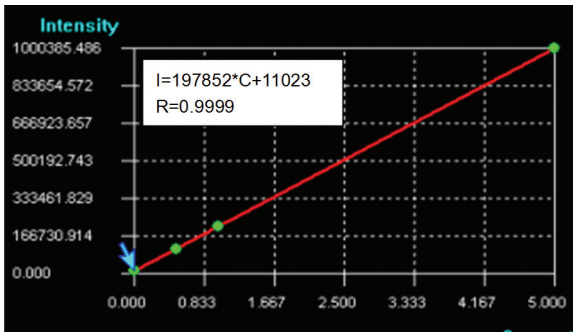


Figure 7: Calibration curve for La 333.749 nm.

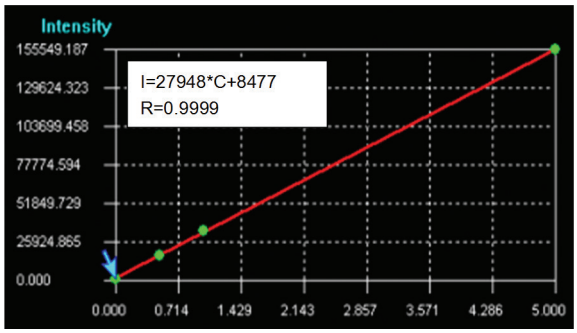


Figure 8: Calibration curve for Ni 221.647 nm.

Results on samples

Concentrations measured on 3 samples are given in Table 5. The concentration is given for the prepared sample and the content in the original sample is also given.

Table 5: Results obtained on samples

		Sample 4030	
Elt	**Wavelength (nm)**	**Measured Conc. (mg/L)**	**Content in raw sample (%)**
Al	308.215	1.19	0.11
Ba	455.403	0.90	0.086
Bi	223.061	< 215 ng/L	< 0.206 mg/kg
Ca	317.933	5.46	0.52
Co	228.616	0.42	0.040
La	333.749	0.95	0.091
Mn	257.610	2.28	0.22
Ni	221.647	0.14	0.013
S	181.978	0.69	0.066
Si	251.611	2.79	0.27
Zn	213.856	0.058	0.0055

		Sample 4240 (Powder)		Sample 4240 (Solid)	
Elt	**Wavelength (nm)**	**Measured Conc. (mg/L)**	**Content in raw sample (%)**	**Measured Conc. (mg/L)**	**Content in raw sample (%)**
Al	308.215	0.10	0.0098	0.21	0.021
Ba	455.403	1.26	0.12	0.98	0.096
Bi	223.061	< 215 ng/L	< 0.211 mg/kg	0.15	0.015
Ca	317.933	1.97	0.19	1.73	0.17
Co	228.616	4.83	0.47	5.16	0.50
La	333.749	12.2	1.20	12.4	1.21
Mn	257.610	1.51	0.15	1.26	0.12
Ni	221.647	0.02	0.0020	0.0098	0.00098
S	181.978	0.60	0.059	3.15	0.31
Si	251.611	2.54	0.25	0.055	0.0049
Zn	213.856	0.0053	0.00052	0.017	0.0017

Recovery test

To evaluate the accuracy of the measurement, the sample 4030 was spiked and the spiked sample was analyzed to check for the recovery. Recoveries obtained are given in Table 6.

Table 6: Recoveries obtained on sample 4030

Elt	Wavelength (nm)	Measured Conc. on unspiked sample (mg/L)	Spiked concentration (mg/L)	Measured Conc. on spiked sample (mg/L)	Recovery (%)
Al	308.215	1.17	1	2.16	99
Ba	455.403	0.89	1	1.94	105
Ca	317.933	5.47	5	10.7	105
Co	328.616	0.42	0.5	0.90	96
La	333.749	0.94	1	1.99	105
Mn	257.610	2.27	2	4.22	97
Ni	231.064	0.14	0.5	0.63	98
S	181.978	0.96	1	1.99	103
Si	251.611	2.73	2	4.79	103

Elt	Wavelength (nm)	Measured Conc. on unspiked sample (mg/L)	Spiked concentration (mg/L)	Measured Conc. on spiked sample (mg/L)	Recovery (%)
Ba	455.403	1.26	1	2.31	105
Ca	317.933	1.98	2	3.89	95
Mn	257.610	1.50	2	3.41	96
S	181.978	0.58	0.5	1.06	96
Si	251.611	2.59	2	4.49	95

All recoveries obtained are in the range 95-105% that is excellent and proves the accuracy of results measured with the instrument.

Stability test

Stability was evaluated on sample 4030 on Ba 455.403 nm line. The sample was measured every 5 minutes and the stability was evaluated by calculating the RSD of the measurements.
Results of the stability test are given in Table 7.

Table 7: Stability test on Ba 455.403 nm

Time (Minutes)	Measured Concentration (mg/L)
0	0.88
5	0.89
10	0.90
15	0.88
20	0.89
25	0.90
30	0.91
35	0.88
40	0.89
45	0.88
50	0.90
Average Value	**0.89**
RSD %	**1.2**

RSD obtained is less than 1.5%, proving the excellent stability of the ULTIMA 2 ICP-OES spectrometer for this application.

Conclusion

Results obtained on the determination of trace elements in a Nd Fe B matrix show that the ULTIMA 2 ICP-OES spectrometer offers accurate results and provides stability over the time. This makes the ULTIMA 2 High Resolution ICP-OES spectrometer the perfect instrument as this is the only instrument able to measure samples for Nd Fe B magnetic applications from raw material to final product.

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JOBIN YVON Technology
info.sci@horiba.com
www.horiba.com/scientific

HORIBA Scientific

USA: +1 732 494 8660
UK: +44 (0)20 8204 8142
China: +86 (0)21 6289 6060
France: +33 (0)1 69 74 72 00
Italy: +39 2 5760 3050
Brazil: +55 (0)11 5545 1500
Germany: +49 (0)89 4623 17-0
Japan: +81 (0)3 6206 4721
Other: +33 (0)1 69 74 72 00