Application Note

Materials

Agilent
Trusted Answers

Trace Impurity Elemental Analysis of Magnet-Grade Neodymium–Iron–Boron by ICP-OES

Overcoming rare earth element spectral interferences using an Agilent 5800 ICP-OES with FACT technology

Authors

Zhang Ping, Zhao Zhifei,
Song Juan'e,
Agilent Technologies (China) Co., Ltd.
Ruby Bradford,
Agilent Technologies, Inc.

Introduction

Neodymium (NdFeB) magnets are high-performance permanent magnetic materials that are widely used in electric vehicles, wind power, electronics, aerospace, and other industries. NdFeB is a key foundational material supporting energy conservation, environmental protection, advanced manufacturing, and many other emerging industries. Its performance directly affects the energy efficiency and value of final products.

The main magnetic phase of NdFeB is $Nd_2Fe_{14}B$. Minor elements and impurities, including rare earth elements (REEs) such as praseodymium (Pr), samarium (Sm), dysprosium (Dy), and gadolinium (Gd) can disrupt the crystal structure of the main phase through atomic substitution, resulting in possible degradation of magnetic performance. Fluctuations in impurity element content can also interfere with critical processes such as melting and sintering, leading to a sharp decline in product yield. Accurate quantification of REE impurities in NdFeB is therefore essential.

China has published seven standardized chemical analysis methods for NdFeB alloys, which cover REEs and impurities. Part II of the *Chemical Analysis Methods for Neodymium–Iron–Boron Alloys* (XB/T 617)[1] series uses inductively coupled plasma optical emission spectrometry (ICP-OES). However, during testing by ICP-OES, REEs produce numerous emission lines at closely spaced wavelengths, making them highly susceptible to spectral interference. If left unchecked, these interferences would compromise the accuracy of the quantitative results for REEs in NdFeB.

This study presents a method for the Agilent 5800 ICP-OES that uses proprietary Fast Automated Curve-fitting Technique (FACT) background correction to eliminate spectral interferences between REEs.[2] This method improves the accuracy and detection limits for 13 REEs in NdFeB, providing efficient quality control (QC) of NdFeB materials.

Experimental

Reagents, standards, and samples

High-purity deionized water was prepared freshly each day using a Millipore Milli-Q ultrapure water system. Calibration standards and spiked samples were prepared using an Agilent mixed REE standard solution comprising Ce, Dy, Er, Eu, Gd, Ho, La, Lu, Nd, Pr, Sc, Sm, Tb, Th, Tm, Y, and Yb. Single element Agilent standards for Fe, Nd, and Pr were used for FACT interferent correction (Figure 2). An NdFeB sample was procured from a supplier in China.

Sample preparation

Approximately 0.1 g of the NdFeB sample was accurately weighed into a digestion vessel and 5 mL of freshly prepared aqua regia (nitric acid to hydrochloric acid = 1:3, v/v) was added. The mixture was heated on a hot plate at 150 °C until the sample had dissolved completely. After cooling, the solution was diluted to 50 mL with ultrapure water, ready for analysis. Spiked solutions were prepared by adding known spike volumes to the digested samples before analysis. The spike concentrations used are shown in Table 3.

Instrumentation

An Agilent 5800 ICP-OES equipped with a SeaSpray nebulizer, double-pass cyclonic spray chamber, and a fully demountable torch assembly with a 1.8 mm inner diameter injector was used. The instrument was controlled using Agilent ICP Expert software (version 7.7.3). Instrument operating parameters are summarized in Table 1. The software includes FACT, comprehensive QC, and IntelliQuant semiquantitative analysis that provides five-star spectral line recommendations, interference diagnostics, and data screening functions (Figure 1).

Table 1. Agilent 5800 ICP-OES operating parameters.

Parameter	Value
Viewing Mode	Axial
Read Time (s)	10
Number of Replicates	3
Uptake Delay (s)	13
Stabilization Time (s)	12
Pump Speed (rpm)	12
RF Power (W)	1300
Plasma Gas Flow (L/min)	14
Auxiliary Gas Flow (L/min)	1.0
Nebulizer Gas Flow (L/min)	0.7
Sample Pump Tubing	PVC white/white
Waste Tubing	PVC blue/blue
Background Correction	FACT, fitted

Selection of analytical wavelengths

The IntelliQuant function in the ICP Expert software offers powerful analytical and intelligent decision-making capabilities.[3] It accurately determines elemental composition ratios, approximate concentrations, and visually presents elemental information using distinct colors for rapid identification. IntelliQuant helps users refine the quantitative method by identifying elements present in the sample, guiding the selection of additional wavelengths, and highlighting potential spectral interferences that can be corrected using FACT. In this study, FACT was applied to 11 elements and fitted background correction (FBC) was used for two elements.

Based on the sample's composition, IntelliQuant recommends optimal analytical wavelengths for each element, effectively avoiding interference from high concentration matrix elements. This automated system eliminates tedious manual screening, significantly improving analytical efficiency and accuracy while making the workflow more convenient for the analyst. In this study, the primary analytical line for Tb, Tb 367.636 nm, was not selected for the quantitative method due to an interference from Fe (Figure 1). IntelliQuant suggested alternative lines with a higher star rating such as Tb 332.440.

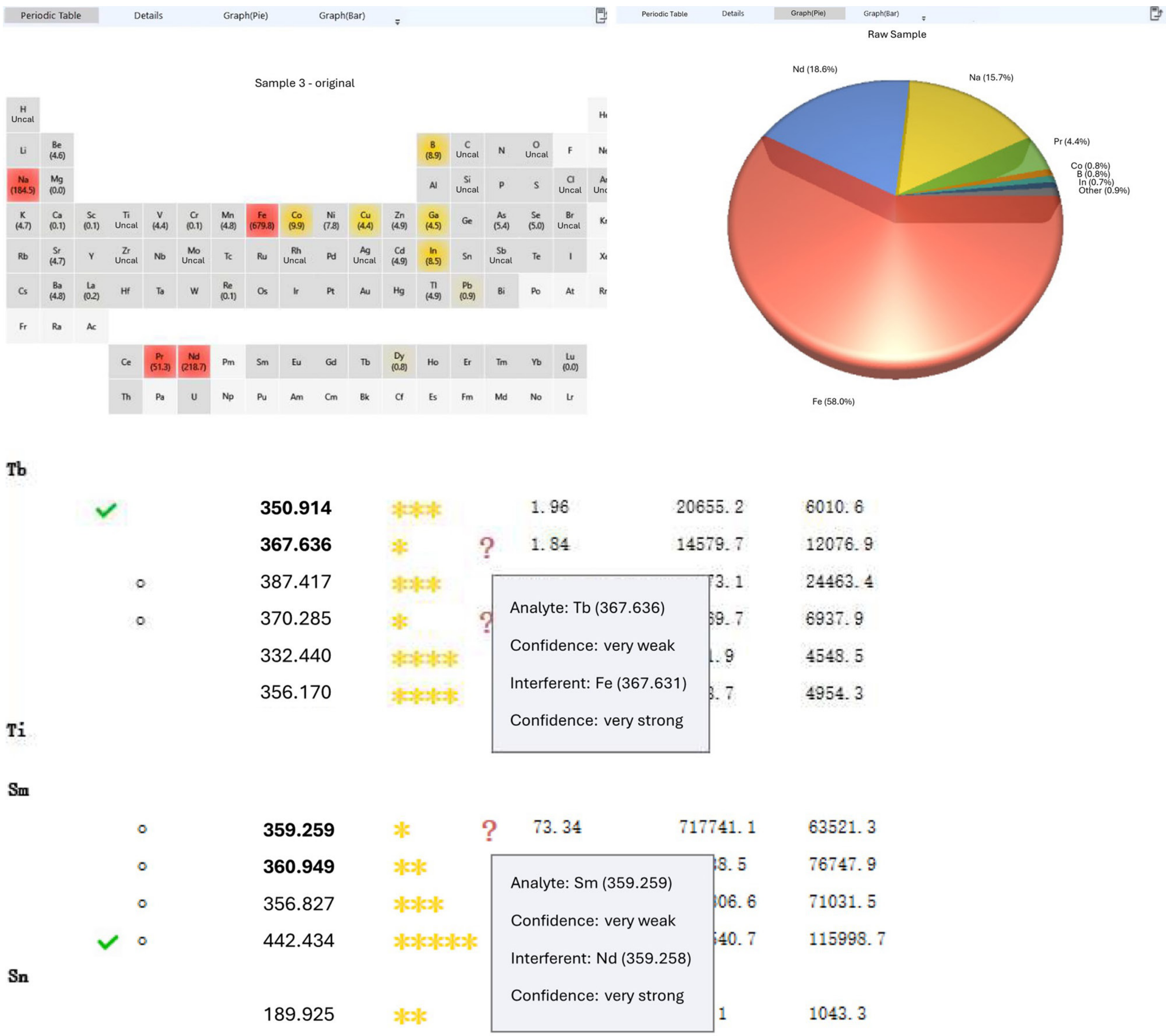

Figure 1. Top: IntelliQuant screening revealed the elemental composition of the REE magnets with Fe, Nd, Dy, Pr, and B occurring at the highest concentrations, among other detected elements (displayed in the periodic table and pie chart). Bottom: IntelliQuant wavelength 'star rating' recommendation system.

FACT spectral deconvolution

FACT background correction uses advanced spectral modeling technology to provide real-time spectral correction.[2] It mathematically resolves (separates) analyte signals from the original spectrum by modeling expected components and their individual responses.

For example, when determining La 408.671 nm in NdFeB, an interference occurs from the Nd line at 408.680 nm. By establishing a FACT model using a blank, a La single-element standard, and an Nd single-element standard, the correct analyte signal can be separated, ensuring analytical accuracy (see Figure 2).

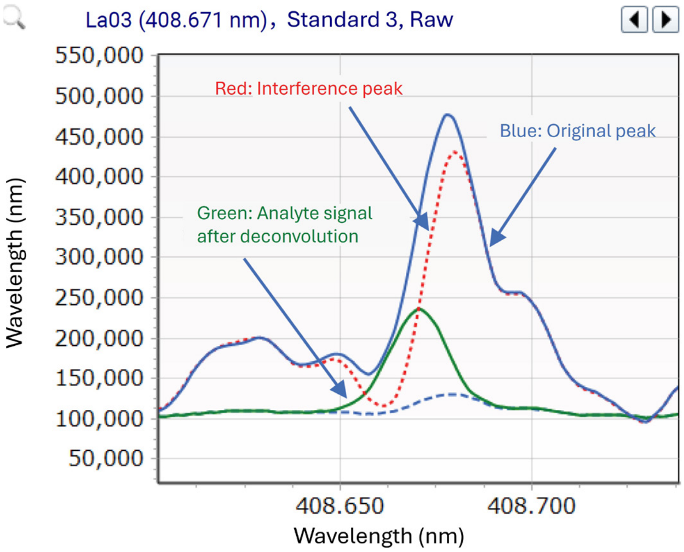

Figure 2. FACT deconvolution effect enabling the accurate measurement of the La 408.671 nm line (green line), free of interference by Nd 408.680 nm (red line).

Results and discussion

Calibration range and linearity

Calibration solutions were prepared as described in Part II: XB/T 617 of the *Chemical Analysis Methods for Neodymium–Iron–Boron Alloys* standard methods. The selected wavelengths, calibration concentration ranges, and background correction modes are shown in Table 2.

All 13 rare-earth impurity elements exhibited calibration curve correlation coefficients greater than 0.999, demonstrating excellent linearity across the concentration range (Table 2).

Detection limits and spike recoveries

The 10% aqua regia blanks were analyzed to calculate instrument detection limits (IDLs) and method detection limits (MDLs). Using the method conditions outlined in Table 1, the blanks were measured 11 times. The IDL was defined as three times the standard deviation of the 11 blank measurements, and the MDL was calculated by multiplying the IDL by the dilution factor from the sample preparation (Table 3).

Spike recovery experiments were conducted and all 13 rare-earth impurity elements showed recoveries between 90 and 111%, demonstrating excellent method accuracy.

Quantitative results for the NdFeB sample digest are also shown in Table 3.

Table 2. Analytical wavelengths, background correction, and linear analytical ranges.

Element	Recommended Wavelength (nm)	Background Correction	Calibration Range (mg/L)	Correlation Coefficient
Ce	446.021	FACT	0.05–0.5	0.99943
Dy	357.625	FACT	0.05–0.5	0.99997
Er	369.265	FACT	0.05–0.5	0.99999
Eu	420.504	FACT	0.05–0.5	0.99999
Gd	342.246	FACT	0.05–0.5	1.00000
Ho	341.644	FACT	0.05–0.5	0.99997
La	408.671	FACT	0.05–0.5	0.99995
Lu	261.541	Fitted	0.05–0.5	0.99999
Sm	442.434	FACT	0.05–0.5	0.99965
Tb	332.440	FACT	0.05–0.5	0.99995
Tm	329.100	FACT	0.05–0.5	1.00000
Y	371.029	FACT	0.05–0.5	0.99999
Yb	328.937	Fitted	0.05–0.5	1.00000

Table 3. Detection limits, spike recoveries, and quantitative results for the NdFeB sample digest. MDLs and sample content (mg/kg) have been adjusted for the dilution factor.

Element	IDL (µg/L)	MDL (mg/kg)	Spike Level (mg/L)	Recovery (%)	Sample Content (mg/kg)
Ce	4.5	2.25	0.20	90	157
Dy	3.0	1.5	0.20	111	180
Er	1.0	0.5	0.01	108	3.1
Eu	1.0	0.5	0.01	108	<MDL
Gd	2.5	1.25	0.02	108	25.0
Ho	3.0	1.5	0.01	93	3.1
La	0.5	0.25	0.02	102	14.1
Lu	0.5	0.25	0.01	100	1.4
Sm	4.8	2.4	0.02	102	21.1
Tb	4.5	2.25	0.01	103	4.6
Tm	3.3	1.65	0.01	99	<MDL
Y	0.3	0.15	0.01	101	0.7
Yb	0.2	0.1	0.01	98	0.3

Conclusion

An Agilent 5800 ICP-OES combined with Agilent Fast Automated Curve-fitting Technique (FACT) background correction was used successfully to analyze 13 rare earth element (REE) impurity elements in an NdFeB sample. Instrument detection limits for all elements were below 5 ppb, demonstrating the high sensitivity of the 5800 ICP-OES. Despite the complexity of rare-earth impurity analysis in NdFeB materials, FACT effectively eliminated spectral interferences between the REEs, enabling spike recoveries of 90 to 111% for all 13 elements. These results confirm that the method provides excellent sensitivity and accuracy, making it well suited for the quantitative determination of REE impurities in NdFeB materials.

References

1. Part II: XB/T 617 – Chemical Analysis Methods for Neodymium–Iron–Boron Alloys, https://www.chinesestandard.net/PDF/English.aspx/XBT617.2-2014
2. Real-Time Spectral Correction of Complex Samples Using FACT Spectral Deconvolution Software, 5991-4837EN
3. Agilent IntelliQuant Software: For greater sample insight and simplified method development, Agilent publication, 5994-1516EN

Products used in this application

Agilent products

Easy-fit 1.8 mm semi-demountable torch for 5000 series VDV/SVDV ICP-OES

Double-pass spray chamber, glass cyclonic design with ball joint socket and UniFit drain outlet

SeaSpray concentric glass nebulizer for 5000 series ICP-OES

Peristaltic pump tubing, white/white, 12/pack

Peristaltic pump tubing, blue/blue, 12/pack

Multi-element calibration standard (10 µg/mL):

Ce, Dy, Er, Eu, Gd, Ho, La, Lu, Nd, Pr, Sc, Sm, Tb, Th, Tm, Y, Yb in 5% HNO_3, 100 mL

Iron (Fe) standard, 10,000 µg/mL in 5% HNO_3, 100 mL

Neodymium (Nd) standard, 10,000 µg/mL in 5% HNO_3, 500 mL

Praseodymium (Pr) standard, 1,000 µg/mL in 5% HNO_3, 100 mL

www.agilent.com/chem/5800icp-oes

DE-016409

This information is subject to change without notice.

© Agilent Technologies, Inc. 2026
Published in the USA, August 13, 2026
5994-9376EN

Agilent
Trusted Answers