High-Precision Iron Isotopic Analysis of IAEA-B5 basalt and rock Reference Materials

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Paolo Di Giuseppe, Simone Vezzoni, Stefano Iannini Lelarge, Andrea Rielli, Samuele Agostini, Andrea Dini

Consiglio Nazionale delle Ricerche, Istituto di Geoscienze e Georisorse, Via Moruzzi, 1, 56124 Pisa, Italy

1. Licence

2. Citation

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3. Data Description

We report the results of high throughput, robust, and sensitive method for the precise analysis of $^{56}Fe/^{54}Fe$ and $^{57}Fe/^{54}Fe$, performed using Multi-Collector-Inductively Coupled Plasma-Mass Spectrometer (MC-ICP-MS), Thermo Scientific Neptune PlusTM. We measured the Fe isotope compositions of widely used standard reference materials ranging from basaltic to rhyolitic compositions (JB-2, BHVO-2, BE-N, AGV-1, and RGM-1). We also propose a new iron isotope reference material, IAEA-B5, a basalt from Mount Etna already commonly used as a reference for B and Li isotopes.

3.1. Analytical procedure

Iron isotope compositions of Standard IRMM-524b and other geological reference materials were measured using a Thermo Scientific High Resolution (HR)-MC-ICP-MS Neptune Plus in the "*Laboratorio Isotopi Radiogenici e Stabili Non-Convenzionali (Radiogenic and Non-Conventional Stable Isotopes Laboratory)*" at the Istituto di Geoscienze e Georisorse of Consiglio Nazionale delle Ricerche (IGG-CNR) of Pisa, Italy. Major element analyses of IAEA-B5 were carried out by X-Ray Fluorescence (XRF) at the Dipartimento di Scienze della Terra "Ardito Desio", Università degli Studi di Milano, whereas trace element data are available in the literature (Tonarini et al., 2003).

Rock powders were dissolved using a mixture of HF and HNO_3. After drying, the samples were re-fluxed with HNO_3 and, following evaporation, HCl was added. Iron was purified in HCl solution through ion-exchange chromatography columns. Fe eluates were dried, re-fluxed with 1mL concentrated 14M HNO3 and put again on hot plate until dryness to eliminate any chloride complexes. Subsequently, 1mL 2% HNO3 was added, and the vials were placed in an ultrasonic bath and dried again. This step ensures that the acid molarity is perfectly matched across the samples, standard, and blank solutions, which is essential for achieving accurate isotopic analyses. Finally, the dried Fe residues were dissolved in 8mL 2% HNO3 to obtain a sample concentration of ~5 µg/g, ready for measurement by MC-ICP-MS

A protocol consisting in Standard-Sample Bracketing + Ni doping was performed for data measurements. In this protocol, both standard and sample solutions were spiked using Ni standard SRM NIST 986 with a Fe:Ni ratio of 1:1, following Chen et al. (2017), who reported no significant Fe isotope variation when Fe:Ni ratio ranged between 0.5 and 2.0. ^{54}Fe, ^{56}Fe, ^{57}Fe, $^{58}(Fe+Ni)$, ^{60}Ni, and ^{62}Ni were measured, respectively at Faraday Cups Low 4 (L4), Low 3 (L3), Low 1 (L1), Central (C), High 1 (H1) and High 2 (H2), equipped with $10^{12}\Omega$, and $10^{11}\Omega$ resistors with ^{56}Fe intensity comprised between 10 and 11 V using 5µg/g concentrated solutions of IRMM-524b Fe.

To assess reproducibility, accuracy, time efficiency and solution consumption, sample analyses were performed as single, duplicate, triplicate, and quintuplet measurements in the bracketing procedure. Sample results were corrected for the consecutive standard mean, from which the blank mean was subtracted. When replicates were measured in sequence, blanks consisted of 25 cycles (5 blocks of 5 cycles), while standards and samples had 60 or 90 cycles (6 blocks of 10 cycles or 9 blocks of 10 cycles) with 8.389 seconds integration time and 3 seconds' idle time. For single measurements, blanks, samples, and standards all used 25 cycles with three measurements averaged for the result. A 2% HNO_3 washing solution was used for washing before and after each blank, standard, and sample measurement. An instrumental baseline correction, which consisted of 100 cycles with integration times of 1.02s, was performed before every sample and standard.

3.2. Data processing

The $^{62}Ni/^{60}Ni$ ratio of 0.138599 was applied for mass bias correction using the exponential law, $r=R(1+\Delta M/M)^{\beta}$, where r is the measured isotopic ratio, R is the true ratio, $\Delta M/M$ represents the relative mass difference, and β is the mass fractionation factor (Russell et al. 1978, Maréchal et al. 1999).

4. File description

4.1. File inventory

The data are provides as the following three tables:

i. 2025-040_Di-Giuseppe-et-al_Table1_SRM_FeIsotopeResults
ii. 2025-040_Di-Giuseppe-et-al_Table2_IAEA-B5_FeIsotopeResults;
iii. 2025-040_Di-Giuseppe-et-al_Table3_IAEA-B5_MajorTraceElements

4.2. Description of data tables

2025-040_Di-Giuseppe-et-al_Table1_SRM_FeIsotopeResults

File "2025-040_Di-Giuseppe-et-al_Table1_SRM_FeIsotopeResults" contains the values of Fe isotope compositions (56Fe/54Fe and 57Fe/54Fe) of Reference Standard Materials JB-2, BHVO-2, BE-N, AGV-1, and RGM-1. Data are organised in 11 columns:

Standard	**Name of the Standard Reference Material**
Date/Average	Day/Month/Year (Italian format) of performed analysis/Average value of "n" analysis
$\delta^{56}Fe$	Isotopic ratio between ^{56}Fe and ^{54}Fe expressed using Delta notation
2SD	2 x Standard Deviation (referred to $\delta^{56}Fe$)
2SE	2 x Standard Error (referred to $\delta^{56}Fe$)
2SE*	* indicates 2 Standard Error (2SE) calculated using 2SD and the number of analyses "n" (referred to $\delta^{56}Fe$)
$\delta^{57}Fe$	Isotopic ratio between ^{57}Fe and ^{54}Fe expressed using Delta notation
2SD	2 x Standard Deviation (referred to $\delta^{57}Fe$)
2SE	2 x Standard Error (referred to $\delta^{57}Fe$)
2SE*	* indicates 2 Standard Error (2SE) calculated using 2SD and the number of analyses "n" (referred to $\delta^{57}Fe$)
n.	Number of measurements during the bracketing sequence

2025-040_Di-Giuseppe-et-al_Table2_IAEA-B5_FeIsotopeResults

File "2025-040_Di-Giuseppe-et-al_Table2_IAEA-B5_FeIsotopeResults" contains the values of Fe isotope compositions (56Fe/54Fe and 57Fe/54Fe) of Reference Standard Materials IAEA-B5. Data are organised in 11 columns.

Standard	**Name of the Standard Reference Material IAEA-B5**
Date/Average	Day/Month/Year (Italian format) of performed analysis/Average value of "n" analysis
$\delta^{56}Fe$	Isotopic ratio between ^{56}Fe and ^{54}Fe expressed using Delta notation
2SD	2 x Standard Deviation (referred to $\delta^{56}Fe$)
2SE	2 x Standard Error (referred to $\delta^{56}Fe$)
2SE*	* indicates 2 Standard Error (2SE) calculated using 2SD and the number of analyses "n" (referred to $\delta^{56}Fe$)
$\delta^{57}Fe$	Isotopic ratio between ^{57}Fe and ^{54}Fe expressed using Delta notation
2SD	2 x Standard Deviation (referred to $\delta^{57}Fe$)
2SE	2 x Standard Error (referred to $\delta^{57}Fe$)
2SE*	* indicates 2 Standard Error (2SE) calculated using 2SD and the number of analyses "n" (referred to $\delta^{57}Fe$)
n.	Number of measurements during the bracketing sequence

2025-040_Di-Giuseppe-et-al_Table3_IAEA-B5_MajorTraceElements

File "2025-040_Di-Giuseppe-et-al_Table3_IAEA-B5_MajorTraceElements" contains the values of major and trace elements compositions of IAEA-B5 standard. Data are organised in five columns. Data are organised in 5 columns for major elements and three columns for trace elements.

Major elements (reported in weight per cent)	
Oxides and Sum	Reported the major element expressed in oxides and the total sum of their values
Average	Average composition of each oxide
Std. Dev.	Standard Deviation
n	Number of measurements during the bracketing sequence
Reference	Bibliographic source from which the indicated values were derived
Trace elements (expressed in µg/g)	
Trace element	Each analysed trace element is reported
Reference	Bibliographic source from which the indicated values were derived

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6. References

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