Application Note

Geology, mining and metals

Routine Analysis of Rare Earth Elements in Basalt using ICP-MS

Accurate, precise, and stable results in a basalt sample matrix with the Agilent 7850 ICP-MS in helium collision mode

Authors

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Introduction

Rare Earth Elements (REEs) play a vital role in numerous industrial applications, including advanced materials, electronics, renewable energy technologies, and geochemical research.[1] Precise and routine quantification of REEs in geological matrices such as basalt is essential for understanding magmatic processes, resource assessment, and environmental monitoring. ICP-MS is suitable for the analysis of trace REEs in basalt. However, the sample poses significant analytical challenges due to its complex matrix, high concentrations of major elements, and the risk of polyatomic interferences such as oxides and hydroxides of barium (Ba) and REEs that can compromise accuracy.[1–3] Because it contains high levels of alumina, silica, and refractory metals, basalt can also compromise plasma robustness due to matrix-induced signal suppression.

Various techniques have been used to measure REEs in geological samples, including multicollector (MC-) ICP-MS detection following α-hydroxyisobutyric acid (HIBA) separation.[4] Instrumental Neutron Activation Analysis (INAA) offers excellent sensitivity for many lanthanides (and long lived actinides like thorium and uranium) but is constrained by long irradiation times and the need to access nuclear reactors.[5] Thermal Ionization Mass Spectrometry (TIMS) provides highly precise isotopic measurements; however, it requires complex sample preparation and is unsuitable for routine multi-element analysis.[6] Laser Ablation ICP-MS (LA-ICP-MS) enables direct, spatially resolved measurements of geological samples but demands careful matrix matching of standards and is sensitive to variations in ablation behavior.[7] High-Resolution ICP-MS (HR-ICP-MS) improves spectral resolution, yet its high cost, lower throughput, and operational complexity limit its widespread use. While these techniques remain valuable for specialized applications, they often fall short when laboratories require rapid, routine, and interference-free determination of lanthanides and actinides in rock matrices.

ICP-MS has become the preferred technique for routine REE analysis due to its exceptional sensitivity, wide linear dynamic range, and multi-element capability. In this study, REE measurements were performed using an Agilent 7850 ICP-MS with an Agilent SPS 4 autosampler and controlled using Agilent ICP-MS MassHunter software. A preset method enabled the accurate and precise determination of 16 lanthanide and actinide elements in a single run, supporting high sample throughput. The 7850 includes Octopole Reaction System (ORS^4) cell technology and optimized helium (He) collision mode to effectively remove polyatomic ion interferences using Kinetic Energy Discrimination (KED). Common interferences can be eliminated by the 7850 using default He KED tuning conditions, ensuring reliable analysis in complex matrices.[8,9]

As polyatomic ions pass through the ORS^4 cell with an optimized helium flow, their larger collisional cross sections cause them to lose more kinetic energy via frequent collisions. Upon exiting the cell, these lower energy polyatomic ions are filtered out by the KED barrier, while analyte ions retain enough energy to pass through the quadrupole for detection by the electron multiplier detector. This process enables accurate multi element analysis using a single gas mode.

Operating the ORS^4 in He KED mode ensures:

- Effective control of common spectral interferences
- Multi element analysis across diverse sample types
- Simplified handling of unknown sample matrices
- Fast screening and semiquantitative analysis of samples using IntelliQuant. IntelliQuant provides rapid matrix assessment, including total matrix solid (TMS) content, and identification of any unexpected elements.[10]

This study presents an end-to-end analytical workflow for the routine analysis of 16 elements in a basalt matrix certified reference material (CRM) MBL-1, CGL-007 (Central Geological Laboratory, Mongolia)[11] using the 7850 ICP-MS in He KED mode. The analytes included 14 lanthanides (La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, and Lu) and two actinides (Th and U). Results show that the 7850 ICP-MS method provides accurate, precise, and stable measurements, effectively overcoming the limitations of some of the traditional analytical techniques and ensuring reliable performance in complex geological matrices.

Experimental

Instrumentation

The Agilent 7850 ICP-MS equipped with the ORS^4 cell and an Agilent SPS 4 autosampler was used for all the measurements of the basalt sample. (The same 7850 ICP-MS setup is also installed at Geochemistry and Geochronology facility of the Institute of Seismological Research, India.)

Because ammonium fluoride (NH_4F) was used during sample preparation, the 7850 ICP-MS was fitted with the optional PFA kit and platinum sampling and skimmer cones. All 16 elements were measured using one set of He KED collision cell mode conditions. He KED ensures effective control of most background and matrix-based polyatomic interferences, such as ArO, ArCl, and BaO.

The instrument was controlled using ICP-MS MassHunter software. Typical instrument operating parameters are listed in Table 1. The highlighted parameters were predefined by selecting the General Purpose preset method, while lens voltages were automatically tuned for optimal performance using the software's autotune feature.

Table 1. Agilent 7850 ICP-MS operating parameters.

ICP-MS Parameters	Value
Plasma Mode	General Purpose
RF Power (W)	1550
Spray Chamber Temp (°C)	2.0
Sampling Depth (mm)	10.0
Nebulizer Gas Flow (L/min)	1.07
Lens Tune	Autotune
Helium Flow Rate (mL/min)	4.3
KED (V)	3

Calibration standards

Calibration standards were prepared from Agilent standard solutions, including multi-element calibration standards 2A and 1, containing 10.0 µg/mL of the target elements. The standards were prepared freshly in 5% HNO_3 (v/v). All elements were calibrated from 0.01 to 40.0 µg/L. A 1.00 mg/L internal standard (ISTD) solution of rhodium (Rh) was mixed with the sample online using a tee connector.

Sample preparation

Twelve replicates of the MBL-1 basalt CRM were prepared separately using microwave digestion. Around 0.10 ± 0.0001 g of the CRM sample was accurately weighed into a polytetrafluoroethylene (PTFE) microwave digestion vessel. Each sample was predigested in 2 mL of saturated NH_4F solution, 6 mL HNO_3, and 0.1 mL HCl before microwave digestion (Milestone, Italy) using the parameters shown in Table 2. These acid volumes and ratios were optimized during method development. Once cooled to room temperature, the digested solutions were transferred to a 10 mL autosampler tube and made up to volume using de-ionized water. All the replicates and method blanks were prepared using the same procedure (Figure 1). To monitor drift throughout the analytical sequence, two quality control (QC) check solutions were prepared at low (0.10 µg/L) and high (1.0 µg/L) concentrations.

Table 2. Microwave digestion program.

Step	Time (min)		Power (W)	Temperature (°C)
	Digestion	Hold		
1	10	5	1800	160
2	5	10		190
3	5	20		230
Cool				Room temp

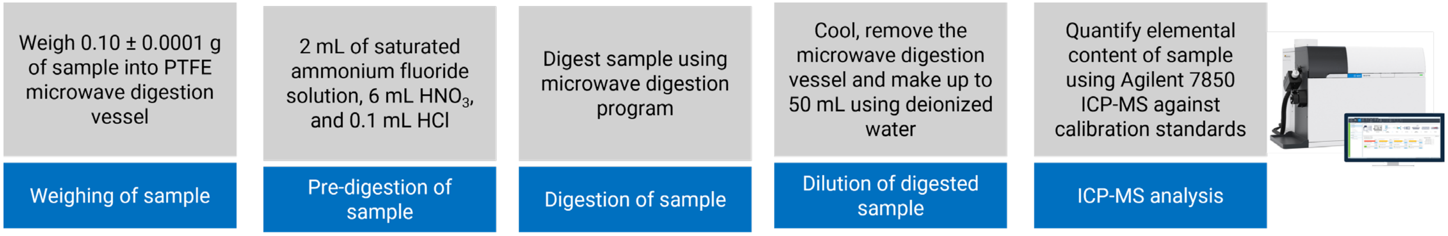

Figure 1. Analytical workflow used for the analysis of the MBL-1 (CGL-007) basalt matrix sample by the Agilent 7850 ICP-MS.

Results and discussion

Detection limits

Instrument Detection Limits (IDLs) were calculated from the calibration graphs (3 x standard deviation (SD) of the concentration of the calibration blank, n=10), while the dilution-adjusted DLs take account of the dilution factor of 500x. The DLs reported in Table 3 are well below the Certified Reference Values (CRVs) specified in the certificate of analysis of the MBL-1 basalt CRM (CGL-007). The IDLs demonstrate the suitability of the 7850 ICP-MS for the determination of the 16 analytes in the CRM.

Internal standard stability

Rh was used as the ISTD as it was not present in the basalt CRM, unlike other potential ISTD elements such as Li and Sc. The analytical sequence comprising the digested CRM samples, method blanks, and QC checks was analyzed repeatedly (70 runs) over three hours. As shown in Figure 2, all the ISTD recovery measurements were within the ±20% limits (indicated by the red dotted lines). The recovery test shows that the 7850 ICP-MS system's robust plasma (as indicated by a low CeO/Ce) was able to decompose the sample matrix effectively, enabling excellent stability to be maintained over the long run. Also, no significant matrix deposition occurred on the interface during the sequence.

Table 3. Calibration coefficient (R), IDLs, and dilution-adjusted IDLs for lanthanides and actinides using the Agilent 7850 ICP-MS.

Elements	R	IDL (µg/ L)	IDLs x 500 (µg/ L)	CRVs of Basalt Matrix CRM MBL-1 (CGL-007), mg/kg		
				Certified Value (CV)	Confidence Interval (±ΔA) at 95% level	Number of Datasets (N)
139 La	0.9999	0.00027	0.1358	55.99	0.94	34
140 Ce	0.9999	0.00066	0.3289	103	2.0	35
141 Pr	0.9999	0.00009	0.0463	11.9	0.24	28
146 Nd	1.0000	0.00001	0.0050	46.62	1.02	35
147 Sm	0.9999	0.00057	0.2843	8.72	0.15	28
153 Eu	0.9999	0.00015	0.0733	2.62	0.06	28
157 Gd	0.9999	0.00062	0.3107	7.17	0.14	26
159 Tb	0.9999	0.00001	0.0050	0.95	0.03	28
163 Dy	0.9999	0.00001	0.0050	4.67	0.1	28
165 Ho	0.9999	0.00001	0.0050	0.78	0.02	27
166 Er	0.9998	0.00001	0.0050	1.84	0.05	27
169 Tm	0.9999	0.00001	0.0050	0.23	0.006	24
172 Yb	0.9999	0.00001	0.0050	1.34	0.02	28
175 Lu	0.9999	0.00017	0.0861	0.19	0.01	26
232 Th	0.9999	0.00001	0.0050	6.95	0.24	29
238 U	0.9999	0.00000	0.0000	1.64	0.09	26

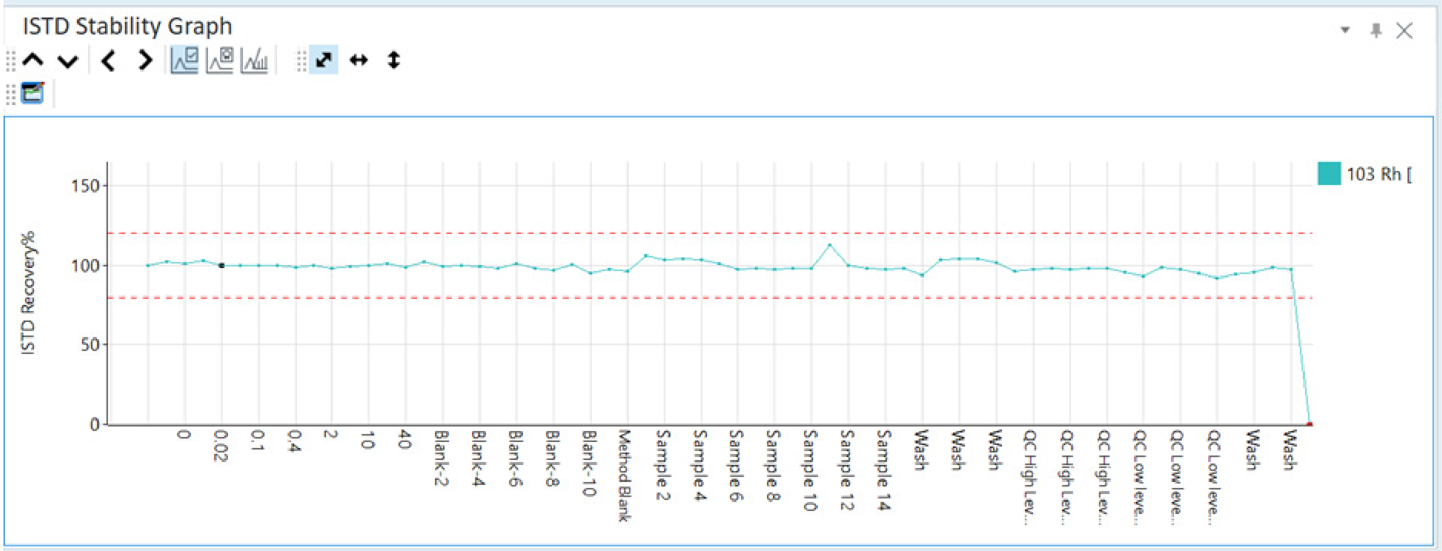

Figure 2. Recovery of the ISTD measured over three hours (70 analyses) using the Agilent 7850 ICP-MS. Due to limited space, not all sample names are shown.

Accurate analysis of basalt

The basalt CRM was analyzed 14 times during the sequence and mean concentrations and recoveries were calculated for each analyte (Table 4). All certified lanthanide and actinide elements recovered within 100 ± 10%, demonstrating the accuracy of the 7850 ICP-MS method.

Quality Control check

The two QC check solutions (0.10 and 1.0 µg/L) were each measured six times during the analytical sequence. The drift (% difference) between the measurements was < 4% for all the elements, demonstrating the stability and robustness of the 7850 ICP-MS after three hours of runtime (Table 5).

Table 4. Recovery of certified values for the basalt CRM after correction for weight and dilution factor (mg/L).

Elements	CRM Concentration	Mean Measured Concentration (n=14)	Recovery (%)	%RSD
139 La	55.99	53.09	95	0.67
140 Ce	103.00	101.77	99	0.71
141 Pr	11.90	11.48	96	1.28
146 Nd	46.62	43.92	94	0.88
147 Sm	8.72	8.19	94	1.24
153 Eu	2.62	2.56	98	1.17
157 Gd	7.17	7.00	98	1.61
159 Tb	0.95	0.88	93	2.39
163 Dy	4.67	4.41	94	2.20
165 Ho	0.78	0.74	95	1.82
166 Er	1.84	1.79	97	2.57
169 Tm	0.23	0.22	95	3.52
172 Yb	1.34	1.26	94	2.83
175 Lu	0.19	0.17	91	4.54
232 Th	6.95	6.36	91	2.63
238 U	1.64	1.66	101	3.37

Table 5. QC check solution measured before and after the three-hour run using the Agilent 7850 ICP-MS.

QC Level 1: Low concentration 0.10 µg/ L										
Elements	**Run 1**	**Run 2**	**Run 3**	**Run 4**	**Run 5**	**Run 6**	**Average (n=6)**	**SD**	**%RSD**	**% Drift**
139 La	0.105	0.103	0.105	0.103	0.103	0.104	0.104	0.001	0.084	104
140 Ce	0.104	0.103	0.102	0.105	0.102	0.099	0.102	0.002	0.194	102
141 Pr	0.101	0.103	0.100	0.101	0.099	0.101	0.101	0.001	0.144	101
146 Nd	0.099	0.104	0.095	0.099	0.099	0.097	0.099	0.003	0.317	99
147 Sm	0.100	0.102	0.101	0.096	0.100	0.102	0.100	0.003	0.252	100
153 Eu	0.102	0.102	0.101	0.101	0.099	0.102	0.101	0.001	0.130	101
157 Gd	0.095	0.104	0.096	0.104	0.098	0.101	0.101	0.003	0.337	101
159 Tb	0.340	0.101	0.101	0.100	0.099	0.099	0.100	0.001	0.078	100
163 Dy	0.101	0.097	0.098	0.097	0.098	0.099	0.098	0.001	0.066	98
165 Ho	0.101	0.098	0.099	0.099	0.101	0.101	0.099	0.001	0.137	99
166 Er	0.100	0.100	0.098	0.100	0.099	0.103	0.100	0.002	0.177	100
169 Tm	0.100	0.101	0.100	0.099	0.100	0.101	0.100	0.001	0.117	100
172 Yb	0.100	0.102	0.100	0.098	0.101	0.097	0.100	0.002	0.217	99
175 Lu	0.100	0.099	0.099	0.096	0.099	0.098	0.098	0.001	0.121	98
232 Th	0.101	0.101	0.103	0.102	0.100	0.101	0.101	0.001	0.136	101
238 U	0.099	0.100	0.101	0.100	0.099	0.098	0.100	0.001	0.117	100

Table 5 continued. QC check solution measured before and after the three-hour run using the Agilent 7850 ICP-MS.

QC Level 2: High concentration 1.0 µg/ L										
Elements	**Run 1**	**Run 2**	**Run 3**	**Run 4**	**Run 5**	**Run 6**	**Average (n=6)**	**SD**	**%RSD**	**% Drift**
139 La	1.020	1.020	1.019	1.018	1.024	1.024	1.021	0.003	0.260	102
140 Ce	1.002	1.007	1.007	0.999	1.010	1.014	1.006	0.005	0.535	101
141 Pr	0.987	0.991	0.996	0.992	0.990	1.003	0.993	0.006	0.568	99
146 Nd	0.976	0.975	0.974	0.985	0.979	0.991	0.980	0.007	0.676	98
147 Sm	0.980	0.976	1.001	0.986	0.974	0.984	0.983	0.010	0.957	98
153 Eu	0.980	0.986	0.991	0.996	0.997	0.986	0.989	0.007	0.651	99
157 Gd	1.001	0.980	0.978	0.980	0.996	0.974	0.985	0.011	1.080	98
159 Tb	0.984	0.987	0.989	0.990	0.986	0.996	0.989	0.004	0.413	99
163 Dy	0.966	0.973	0.985	0.967	0.993	0.981	0.978	0.010	1.048	98
165 Ho	0.979	0.994	0.988	0.994	0.994	0.990	0.990	0.006	0.586	99
166 Er	0.992	0.995	0.995	0.997	0.993	0.997	0.995	0.002	0.221	99
169 Tm	0.986	0.994	0.996	0.995	0.999	0.989	0.993	0.005	0.472	99
172 Yb	0.986	0.987	0.992	1.001	0.989	0.977	0.989	0.008	0.778	99
175 Lu	0.985	0.986	0.989	0.992	0.996	0.984	0.989	0.005	0.467	99
232 Th	0.994	0.996	0.999	1.002	0.995	0.994	0.997	0.003	0.325	100
238 U	0.980	0.986	0.991	0.991	0.989	0.990	0.988	0.004	0.443	99

Conclusion

The Agilent 7850 ICP-MS successfully quantified all 16 lanthanides and actinide elements in the basalt CRM using a single helium KED cell method. The use of a General Purpose preset method and automated lens tuning routine within the Agilent ICP-MS MassHunter software streamlined the analytical workflow, minimizing instrument setup time and ensuring consistent instrument performance. These ease of use features simplified instrument operation while maintaining accurate, interference free results.

Internal standard stability over three hours of continuous operation demonstrated the robustness of the 7850 under routine, high throughput conditions. This level of instrument stability is important for maintaining productivity, as it reduces drift, QC failures, sample reanalysis, and instrument maintenance.

The 7850 ICP-MS method supports laboratories conducting routine, high-throughput multi-element analyses of demanding geological matrices.

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Products used in this application

Agilent products

PFA inert kit with 2.5 mm inner diameter sapphire injector

Platinum sampling cone

Platinum skimmer cone

Multi-element calibration standard 2A

Multi-element calibration standard 1

Rhodium (Rh) single element standard

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