



Rare Earth Analysis by ICP-OES

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Services

Updated: February 2017

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Inductively Coupled Plasma-Optical Emission Spectrometry method for the determination of trace-level rare earth elements (REE) in geological materials

As defined by the International Union of Pure and Applied Chemistry (IUPAC), rare earth elements (REE) are a set of 17 chemical elements including the 15 lanthanides plus scandium and yttrium. The 17 metallic elements have similar chemical properties and occur together in similar geologic settings. The rare earths were discovered beginning in the late 18th century as oxidized minerals-hence “earths”. They are referred to as “rare” because it is uncommon to find them in commercially viable concentrations. Economically exploitable REE concentrations are generally found in rare types of igneous rocks such as carbonatites and alkaline rocks. Less important sources are secondary deposits which form when rare earth and heavy minerals are concentrated from primary mineralization by weathering and physical processes. REEs generally fall into one of two categories based on varying ionic radii and different physical properties. Light rare earths (LREE), which accounted for 66.8% of global demand in 2010, are Lanthanum (La), Cerium (Ce), Praseodymium (Pr), Neodymium (Nd), and Samarium (Sm). Heavy rare earths (HREE) are less common and more valuable, include Europium (Er), Gadolinium (Gd), Terbium (Tb), Dysprosium (Dy), Holmium (Ho), Erbium (Er), Thulium (Tm), Ytterbium (Yb), Lutetium (Lu) and Yttrium (Y). REE mineral deposits are usually rich in either LREE or HREE, but rarely contain both in significant quantities. With the ongoing technological advances in electronics, vehicles, and national defense systems, the demand for rare earth elements has increased exponentially.

The total grade of a rare earth deposit is expressed as total rare earth oxide percentage (%TREO). This can be subdivided into light rare earth grade (%LREO) and heavy rare earth grade (%HREO). The ratio of HREO to TREO expressed as a percentage is the “heavy rare earth enrichment”. For example if TREO = 5% and HREO = 0.5% then the degree of heavy rare earth enrichment is 10%. When the percentage is greater than 10% the deposit is considered to be “enriched in heavies”.

Florin Analytical Services (FAS) developed a rare earth assay for the quantitative determination of the REE concentrations in one of a kind deep sea cores of very limited volume. Samples are carefully dried and pulverized in a dedicated ring and puck bowl with carefully cleaning between samples. After pulverization the hydrophilic samples are stored in a desiccator. A half gram of pulverized ore is placed into a 100 mL Teflon beaker. A 4-acid digestion is performed by adding concentrated hydrochloric and hydrofluoric acids to the ore and heating to dryness. 17.5% hydrogen peroxide and concentrated nitric acid are added. After the reaction subsides concentrated hydrochloric acid is added and heated on a hot plate until the solution is between 3mL to 6mL. The solution is brought to a final volume of 50mL with 25% hydrochloric acid in a class A volumetric flask. The solutions are analyzed by ICP-OES. An external comparison of select samples using ICP-MS shows a strong correlation to determinations of REE by ICP-OES from FAS.

FAS incorporates an extensive quality assurance/quality control (QA/QC) program to ensure accurate results. Replicate samples are run to insure data integrity (typically, 1 in 10 samples submitted are run in duplicate). Certified Reference Materials (CRM's) as well as reagent blanks are also included with each batch of 20 samples. Additional elements are available on request.

Element	Ce	Dy	Er	Eu	Gd	Ho	La	Lu	Nd	Pr	Sc	Sm	Tb	Tm	Y	Yb
FAS LDL	10	5	10	5	5	5	5	5	5	5	5	5	10	5	5	5
CANMET Till-4	78		3.2	<1.0			41	0.5	30		10	6.1	1.1		33	3.4
CANMET SY-4	122	18.2	14.2	2	14	4.3	58	2.1	57	15	1.1	12.7	2.6	2.3	119	14.8
NIST 2702	123						73.5		56		26	10.8				
CANMET REE-1	3960	847	701	23.5	433	208	1661	92	1456	435	8	381	106	106	5480	678
OREAS 604	38.1	1.60	0.62	0.74	2.72	0.25	19.4	0.081	16.5	4.55	4.81	3.08	0.33	0.081	7.16	0.2

Table 1 Recommended data for CRM's used for QA/QC of FAS REE package in mg/kg. Results in blue are provisional.