The Association of Exploration Geochemists - AEG
Sociedade Brasileira de Geoquímica - SBGq

XIII International Geochemical Exploration Symposium

RIO 89 II Brazilian Geochemical Congress

Abstracts

Rio de Janeiro, Brazil
October 1-6, 1989

DETERMINATION OF RARE EARTH ELEMENTS IN GEOLOGICAL SAMPLES BY ISOTOPE DILUTION MASS SPECTROMETRY

Noemia M.P. Moraes
S.S. Iyer
Laboratory of Nuclear and Isotope Geology
Instituto de Pesquisas Energéticas e Nucleares
Departamento de Processos Especiais, São Paulo
K. Sato
Universidade de São Paulo, Instituto de Geociências

Among the many raw materials that are being used in modern industry, rare earth elements are playing an increasingly important role. Since their early commercial exploration, great changes have taken place in the type of deposits explored, as well as their industrial application.

New advances in the development of analytical techniques, established by geochemists, are helping in the characterization and quality control in the fields of rare earth element application, permitting the precise and accurate determination of these elements, even at very

low concentrations.

Considering the importance of precision (and accuracy) in the analytical data, the objective of the present study was to establish the mass spectrometric isotope dilution technique for the analysis of the rare earth elements, as this technique possesses high sensitivity, precision (and accuracy). Within this general objective, chemical separation methods involving ion exchange chromatography were established for the group and individual separation of the rare earth elements.

The method was established for the international rock standards AGV-1, GSP-1, G-2 and PCC-1 and the results obtained compared with the literature values. The analytical procedure established involved the preparation and calibration of the tracers by isotope dilution mass spectrometry, sample digestion, chemical separation and isotope analyses using a fully automated thermoionic mass spectrometer micromass VG isotope model 354. The sample dissolution was carried out in a teflon vessel under pressure. The material (samples + tracer) was digested using a mixture of concentrated HNO_3 and 40% HF in the pressure vessel at 160°C for a period of 18 hours. The sample solution was evaporated and, later, a mixture of concentrated $HClO_4$ and HNO_3 was added and evaporated again. Finally, the residue was dissolved in 2N HCl and taken for chemical separation. The elaborate dissolution procedure was necessary as the rare earth elements are generally present in the accessory, resistant minerals.

The chemical separation was carried out in two steps in cationic resin columns. In the first column, the total rare earth elements were separated from other elements using 2N HCl and 8N HNO_3 as eluiting agents. In the second column, using 0,25N α-HIBA (pH = 4.6) as an eluiting agent, rare earth elements were separated into individual fractions (La, Ce and Nd) and sub groups (Sm-Eu-Gd and Yb-Er-Dy). Nine elements (La, Ce, Nd, Sm, Eu, Gd, Dy, Er and Yb), with concentrations in the range 0.5ppm to 400ppm, were determined with a total precision of 1 to 2% and an accuracy of 5%. The rare earth element concentrations, determined in the rock standard PCC-1, demonstrated the low blank levels in our analysis.

The results obtained in the present study have shown that the analytical procedures developed and employed are satisfactory, as indicated by the high precision and accuracy obtained for the elemental concentrations values. Further, the great advantage of the technique, as shown by the present study, is the non-dependence of the precision and accuracy on the concentration levels of the elements in the samples. The study has also shown that α-HIBA is an efficient eluiting agent for the separation of individual rare earth elements.