CRC LEME

OPEN FILE REPORT 185

The measurement of δ^{34} S in environmental samples by continuous flow mass spectrometry (CFMS)

John Dighton, Richard Cresswell, Andrew Herczeg and Steve Welsh Completed February 2005, published October 2006

Executive Summary

In our attempts to describe, characterise and understand the regolith we take for granted the production of thousands of geochemical analyses that we manipulate to provide clues on the formation of minerals, the dispersion of metals, the origins of salts, and other processes. Increasingly, isotopes are being used to help constrain models of the evolution and reaction pathways of elements in the regolith and hydrosphere. Key amongst these are the isotopes of sulphur, which show significant fractionation effects (up to 100‰) as sulphur-bearing minerals are dissolved, precipitated and re-dissolved; transformed from reduced to oxidised species, and undergo bacterial reduction from sulphate to sulphide, all depending on the prevailing redox potential, the pH and elemental concentrations in the fluids in their environment. Sulphur is present in nearly all natural environments and occurs in a number of oxidation states, from sulphides to elemental sulphur, to sulphates. It is obviously of great interest in studies of the regolith.

Behind all these analyses, however, extreme care needs to be exercised by the providers of the numbers. Precision and accuracy are vital if the conclusions gleaned from models are to be believed. So, we have an arena where we require increasing numbers of analyses to populate our models, balanced by a requirement for increased accuracy and precision as we attempt to disentangle the sometimes complex histories these models suggest.

The highly reactive nature of sulphur and the use of oxidised species (SO_2) for analyses, however, has meant that, until recently, large amounts of sample and stringent cleaning procedures were required to prevent severe fractionation effects occurring during the analyses themselves.

The development of continuous flow mass spectrometry (CFMS) in the 1990s has alleviated these problems, but is generally performed on purpose-built spectrometers. This report describes a novel, but generic, CFMS procedure that can routinely, relatively cheaply, determine the sulphur isotopic ratio for dissolved sulphates from water samples and from sulphur-bearing minerals with an accuracy and precision of 0.3‰. By combining an elemental analyser with a standard mass spectrometer we can now also resolve the different combustion products produced during preparation, an important aid in determining potential sources of analytical error.

Sulphur isotope analyses have never been so easy, nor so useful!