

CONTINUOUS FLOW PYROLYSIS TECHNIQUES FOR THE ISOTOPIC MEASUREMENTS OF OXYGEN-DEUTERIUM IN WATERS, ORGANIC AND INORGANIC COMPOUNDS

John Morrison¹ and [Andy Mower](#)²

1 Micromass UK Ltd., Wythenshawe, Manchester, M23 9LZ, UK

2 Micromass Australia Pty Ltd., 26 Glendale Place, Jannali, Australia, AU-NSW2226

This paper describes a new EA technique for the measurement of hydrogen isotopes in water, in a continuous flow carrier stream of helium. The system is totally carbon free and is based on the use of chromium (patented) as the active reactor material. Water injected into this system is reduced resulting in the quantitative release of hydrogen gas, which is then carried by the helium flow to the mass spectrometer and analysed for dD. This technique exhibits remarkably low memory effects, excellent precision and accuracy and addresses very small sample sizes down to 50 nano litres. In addition, it allows a very high sample throughput with individual sample analysis time of 3 minutes.

Instrumentation and conditions: The system consisted of a EuroVector EA configured with a chromium-packed reactor at a temperature of 1050 °C and fitted with a 1.5m molecular sieve packed GC column. Samples were dispensed into 1.5 ml septa-sealed vials and placed on the carousel of an automated liquid autosampler device (EuroVector LAS2000). A sequence of 3 wash cycles was carried out on each sample prior to injection into the reactor through a heated septa-sealed injector port. A sample size of 0.5 µl of water was chosen for the analysis. The liquid autosampler has the ability to measure up to 110 individual samples; each sample can be analysed up to 144 times. Hydrogen generated in the reactor is passed through the GC column and transported *via* an open split capillary into the source of a Micromass IsoPrime stable isotope ratio mass spectrometer.

In addition to water, this new technique has been applied to the measurement of hydrogen isotopes in chlorinated hydrocarbons such as tri-chloroethane and tri-chloroethylene. We will demonstrate ¹⁸O analyses of both organic and inorganic samples using a range of reactor configurations and temperatures.
