Geophysical Research Abstracts
Vol. 15, EGU2013-13881, 2013
EGU General Assembly 2013

Inconel crucibles – an alternative to quartz glass crucibles when analysing problematic samples by EA-IRMS

Wolfram Meier-Augenstein (1,2), Helen Kemp (1), and Ken Neal (3)

(1) Stable Isotope Forensic Lab, The James Hutton Institute, Dundee, UK, (2) Environmental & Forensic Science Group, Robert Gordon University, Aberdeen, UK, (3) OEA Labs Ltd, Kelly Bray, Callington, UK

Anecdotal evidence as well as observations made in our own stable isotope laboratory suggest for samples with either a high halogen content (such as marine samples) or a high carbon and oxygen content (such as carbohydrates) to result in flash combustion temperatures exceeding temperature or burn time or both of flash combustion under typical conditions. Whatever the exact circumstances during combustion of such samples, they weaken the wall of the quartz glass reactors in the combustion zone and ultimately lead to pin-prick holes being formed through which carrier gas escapes thus resulting in a dramatic loss of carrier gas flow. Occasionally these pin-prick holes get plugged or "sealed" by molten tin thus restoring carrier gas flow but at the other end of the spectrum these pin-prick holes can become so wide for molten tin being able to pass through and to run down the outside of the reactor tube. In the latter event, a catastrophic failure of the reactor tube is inevitable with carrier gas flow downstream of the holes dropping to almost zero.

While pin-prick holes (going unnoticed during an autosampler run of a large batch of samples) typically result in the loss of 3 or 4 samples until the hole/s "self-sealed" with molten tin, in a worst case scenario a catastrophic failure of the reactor tube can result in the loss of 40 or more samples (depending on number of samples in a batch run and when the failure occurred).

Here we present examples of combustion reactor failure as well as observations made with crucibles made of quartz glass, stainless steel or inconel alloy during experiments to see if crucible design can mitigate against the effects of problematic samples.