

Welcome to the Autumn 2014 Raddec newsletter

In this issue, we introduce the new Hyperbaric Oxidiser and describe exciting innovations in Pyrolyser furnace system controls and logging functions now installed on the latest range of furnaces. We have also included a technical section on the measurement of ^3H in concretes and analysis of ^{36}Cl and ^{129}I . We are also pleased to announce the launch of a new look website in 2014 which will provide additional information and support for our customers.

The Raddec range

Raddec Ltd was established in 2003 to supply and develop technologies for the analysis of ^3H and ^{14}C in support of environmental, bioassay, decommissioning and waste sentencing analysis. Since then, Raddec and Raddec International Ltd have supplied over 70 furnaces and associated consumables around the world. In partnership with our agents (Triskem International Ltd, NOKI td and Clovertch), Raddec has installed furnaces in the UK, France, Sweden, Germany, Switzerland, Russia, Romania, South Korea, India, China and the USA. In addition to the furnace technologies, Raddec Ltd delivered the first Hyperbaric Oxidiser to a UK customer in 2014. Raddec has also extended the Pyrolyser's analytical range to include other volatile radionuclides.



New Hyperbaric oxidiser released

The HBO is an integrated system designed to safely and quantitatively oxidize combustible materials and liberate ^3H using an excess of oxygen in a closed pressure vessel. The liberated ^3H is subsequently recovered via a cryo-trapping system. It has been rigorously tested over 5 years by running a broad variety of sample types (several hundred samples). The system has been demonstrated to quantitatively oxidise rubber, oil, plastic, cellulosic materials and fish. The system incorporates:

- 5 Litre stainless steel chamber (with removable sacrificial liner) pressure rated to 90 Bar
- Easy to operate door opening and closure mechanism with viewing port to enable real-time monitoring of combustion.
- Reliable integral resistance-wire sample ignition system
- Novel low thermal mass sample holder and crucibles suitable for combustion of up to 40g of material (dependent on sample type)
- Manual gas handling system to control filling of oxygen and subsequent release of combustion gases
- Safety system that includes a pressure relief valve, burst disk and 4 safety interlocks to inhibit firing unless all interlocks are correctly engaged
- Real-time monitoring sensors comprising 2 pressure transducers and 2 thermocouples with outputs to digital displays and a National Instruments DAQ permitting system control and data logging
- Integrated software (Labview) for monitoring chamber conditions and controlling gas release via a mass flow controller.



Hyperbaric oxidiser with 5L chamber

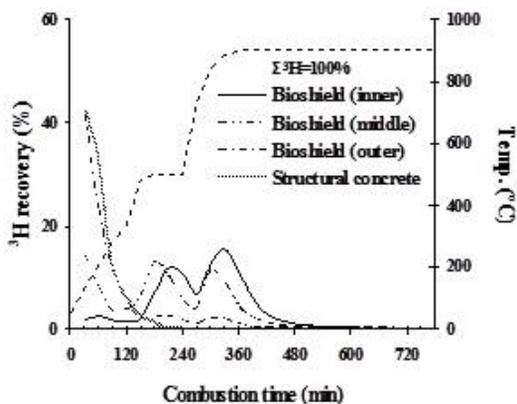
Datalogging and gas control features on the Pyrolyser-Trio

For the past 12 years, Raddec Ltd has supplied purpose-designed multi-tube furnaces and consumables for efficient ^3H and ^{14}C extraction. The next generation of Pyrolyser furnaces have been recently released incorporating the latest control technologies. All new Pyrolyser systems are now fitted with Eurotherm 3504 controllers with 10 user-definable programs controlling the sample and mid-zones. Programs can be easily set up and modified directly through the controller panel or off-line via PC and ethernet connection. Programs can also be backed-up via the PC. The 3504 controller is also used to automatically control air / oxygen supply throughout the run. The Pyrolyser system also incorporates the latest in Eurotherm Nanodac datalogging. As well as controlling the catalyst zone, the Nanodac displays and logs sample, mid zone and catalyst zone temperatures, sample zone set points and air / oxygen status at 1 second intervals throughout the run. Up to 12 months' data can be stored. Data are downloaded to USB memory sticks or via an Ethernet connection. These innovations improve the overall functionality of the Pyrolyser system whilst meeting the demands for demonstrable quality control.



Technical brief - Tritium in concretes

Characterisation of construction materials for radionuclide content is vital in nuclear facility decommissioning and now forms a significant part of the workload for many radioanalytical laboratories. The Pyrolyser system is widely used for routine analysis of ^3H and ^{14}C in a diverse range of matrices and has also been used in studies investigating the form of ^3H in such materials. One such material that has proven particularly interesting is concrete which is widely used in the construction of nuclear facilities. Concrete consists of a number of mineral phases which could potentially become contaminated with ^3H and which could impact on the way ^3H is released from the matrix during analysis. Studies by Kim *et al* (2008) of concretes operationally exposed to tritiated water vapour over decades of operation (referred to as structural concrete) indicated that the ^3H was predominantly present in a weakly-bound form which can be easily released at temperatures below 200°C . However, bioshield concretes, exposed to neutrons, contain a much more strongly bound form of ^3H that requires significantly higher temperatures to achieve its release. These findings have a significant implication to the analytical techniques for analysis of ^3H in these materials and in the long term stability of ^3H in wastes. Studies of ^3H thermal desorption profiles using the Pyrolyser furnace have also been extended to a wide range of materials typically encountered in decommissioning wastes (Warwick *et al*, 2010) and in metals (Croudace *et al*, 2014) providing further insight into the association of ^3H in such materials.



Kim D-J, Warwick PE, Croudace IW (2008) Anal Chem **80**, 5476.
 Warwick PE, Kim D-J, Croudace IW, Oh J-S. (2010) Anal Chim Acta **676**, 93.
 Croudace IW, Warwick PE & Kim D-J (2014) Anal Chem (in press).

Measurement of ^{36}Cl & ^{129}I in addition to ^3H & ^{14}C

Raddec Pyrolyser systems have traditionally been used for the extraction of ^3H and ^{14}C . However, the furnace systems are also suitable for the extraction of other radionuclides such as ^{36}Cl and ^{129}I . Iodine-129 extraction using the Pyrolyser system is established, with two Pyrolyser systems being used by the Xi-an AMS facility in China for ^{129}I extraction. Initial validation has shown that, using Raddec's purpose-designed glassware and running conditions, the furnace can be used to quantitatively extract both ^{36}Cl and ^{129}I from a range of matrices. Raddec Ltd has also worked with Triskem International Ltd to develop post combustion separation techniques for the separation of co-trapped ^{36}Cl and ^{129}I and to provide purified fractions ideally suited for liquid scintillation analysis.

Reference materials for ^3H

One problem faced by radioanalytical laboratories is the lack of reference materials for radioanalytical method validation. Raddec is working to address this by developing a range of reference materials for ^3H , ^{14}C , ^{36}Cl and ^{129}I . The first reference material to be produced is a ^3H -labelled sediment containing ^3H between 100 -200 Bq/kg. The ^3H is present as organically-bound ^3H improving the stability of the standard. The sediment has been incorporated into the 2014 OBT intercomparison exercise and distributed to a number of laboratories for analysis. Initial results will be presented at the OBT 2014 meeting in Ottawa, Canada. Once these data have been assessed and published, the reference material will be made available to laboratories to aid method development and validation.

Events Autumn 2014

Triskem Users Group Meeting, Bath, UK, 16th Sept 2014
 ERA12, 2014, Bath, UK, 17 – 19th Sept 2014
 3rd OBT workshop, 15 – 18th Sept. 2014, Ottawa, Canada

Further information on Raddec Ltd and details of the Raddec range of analytical equipment can be found on our website www.raddec.com. Alternatively please feel free to contact us at sales@raddec.com for any specific queries.

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