Analysis of ^{3}H & ^{14}C using the Raddec Pyrolyser

Ian Croudace & Phil Warwick

Geosciences
Advisory
Unit

RADDEC LTD
Radiochemical and Decommissioning Solutions Ltd

Introduction

- The Pyrolyser series© of furnaces allow the simultaneous decomposition of up to 6 samples and the efficient oxidation of the liberated ^{3}H and ^{14}C species
- The furnace design has been thoroughly tested and proven over many years.
- Continued R&D ensures that upgrades become incorporated into the system design

Features

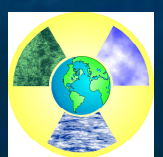
- 4 or 6 samples decomposed simultaneously in ~4 hours using a programmable thermal ramp.
- 10g Pt-alumina catalyst per tube allowing 20+ determinations.
- Rapid cool-down of sample zone enabling good cycle times between runs.
- Bubbler-traps (20mls) have >95% efficiency.
- The Pyrolysers have been in regular use for extraction of ^{3}H and ^{14}C from a wide range of materials (vegetation, fish, soil, sediment, concrete, metal etc).
- The design and proven effectiveness follows several years of testing of samples from intercomparison exercises, environmental studies and nuclear site decommissioning programmes.

Number of H-3/C-14 analysis

- 2002 H-3 (242)/C-14 (79)
- 2003 H-3 (376)/C-14 (127)
- 2004 H-3 (847)/C-14 (565)
- 2005 H-3 (996)/C-14 (153)

Max. number of samples that can be run using a single 6-tube furnace is 1352

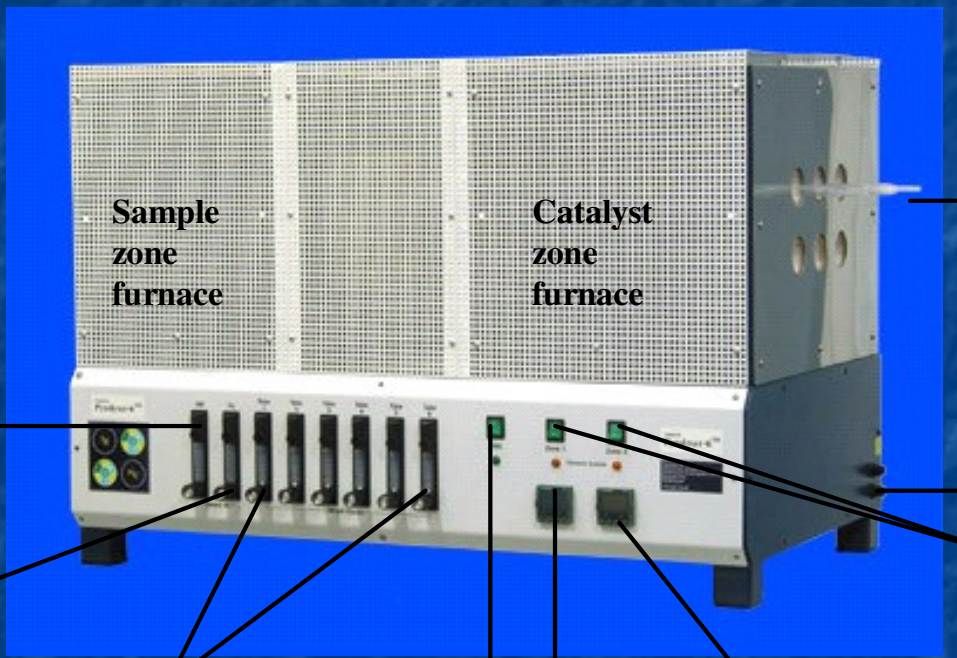
(52 x 24= 1352 p.a.)



Geosciences
Advisory
Unit

RADDEC LTD
Radiochemical and Decommissioning Solutions Ltd

Purpose-built tube furnace for H-3/C-14 analysis (Pyrolyser 6™)



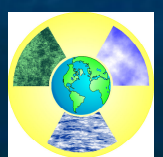
Geosciences Advisory Unit

RADDEC LTD
Radiochemical and Decommissioning Solutions Ltd

Sample types

- Environmental samples
  - Soil/Sediment, Fruit, Water, Grass, Milk, Fish, Sludge etc.

- Decommissioning samples
  - Concrete, Brick, Asbestos, MMMF, Metal, Plastic, Desiccants, Paper, Electric wire, Sewage sludge, Graphite, Paint etc.



Geosciences
Advisory
Unit

RADDEC LTD
Radiochemical and Decommissioning Solutions Ltd

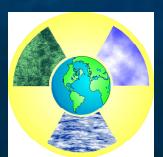
Sample sizes

- Samples with low organic contents ;

  Soil/Sediment, concrete, brick, metal etc. (1 - 10g)

- Samples with high organic contents ;

  Biota, plastics, organic rich sediment/soil etc. (Normally 0.5 - 1g but can be increased with a slow ramp)



Geosciences
Advisory
Unit

RADDEC LTD
Radiochemical and Decommissioning Solutions Ltd

Counting efficiency and furnace recovery for H-3/C-14

- Technical information

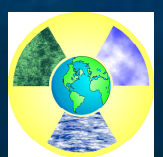
	SQPE*	Counting efficiency	Furnace recovery**	Limit of detection***
H-3	719 - 729	18 – 20 %	> 90 %	0.020 Bq/g
C-14	720 - 750	66 – 73 %	> 95 %	0.015 Bq/g

* Typical SQPE of concrete samples

** Average value of 70 measurements using an organic H-3/C-14 standard

*** Using 5g sample size and 2 hour counting time on Quantulus

$$L_D(Bq/g) = \frac{2.71 + 4.65\sqrt{C}}{t} \times \frac{100}{E} \times \frac{100}{R} \times \frac{1}{M_g}$$



Geosciences Advisory Unit

RADDEC LTD
Radiochemical and Decommissioning Solutions Ltd

Intercomparison results*

Sample	H-3/C-14 type	Measured value Bq/g ± 1σ	Reference value Bq/g ± 1σ
NPL (2002)	Tritiated water	20 ± 10	20.04 ± 0.18
NPL (2004)	Tritiated water	0.536 ± 0.042	0.539 ± 0.006
NPL (2004)	C-14 carbonate	23.4 ± 1.6	24.4 ± 0.5
FSA	H-3 thymidine (Milk)	4.72 ± 0.66	Mean = 4.04 0.18 – 4.93
FSA	H-3 thymidine (Plaice)	4.42 ± 0.30	Mean = 4.67 2.7 – 8.3

* There are no commercially available certified reference material for H-3/C-14 activities

Geosciences Advisory Unit

RADDEC LTD
Radiochemical and Decommissioning Solutions Ltd

LSC+ -Plus

Enhanced LSC data processing software

..... better and faster

Results for the H-3 in water (8ml + 12ml Gold Star™)

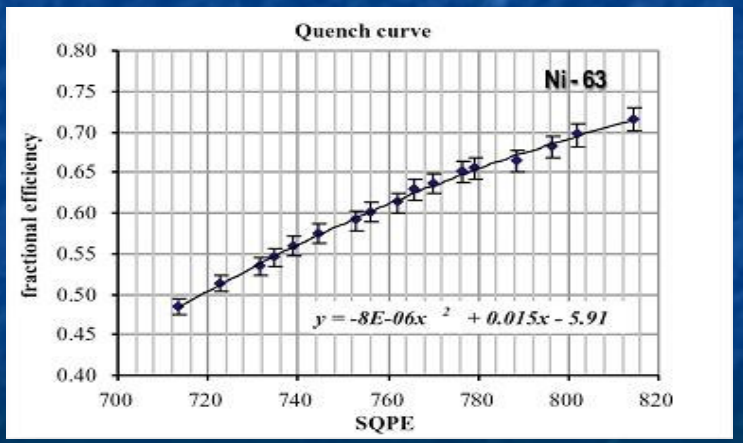
Report date : 7-Nov-2003
Customer : Raddec Ltd
Job reference : Raddec 372
Date samples received : 6-Nov-2003

Date of analysis : 7-Nov-2003
Working instruction number: - Raddec/RC/2022
Calibration report number: - Raddec/CAL/16

	Counter S/N	Laboratory S/N	Reference date	H-3 Bq/ml	2 s.d.
1	INST STD		7-Nov-2003	23.224	2.548
2	6-169-1	372-1	7-Nov-2003	< 0.006	
3	6-169-2	372-2	7-Nov-2003	< 0.007	
4	6-169-3	372-3	7-Nov-2003	< 0.006	
5	6-169-4 STD		7-Nov-2003	0.115	0.014

All results are in Bq/ml and are decay corrected to the reference date (12.30 Years half life)
"< values" are limits of detection as defined by Currie, 1968
Uncertainties are at the 2 s.d. confidence level and are based on propagated method uncertainties

Analyst : A Other



- Calculates activities directly from counter files
- Eliminate transcription errors
- Calculates LODs (Currie)
- Built in Quality Control
- Statistical analysis of results
- Full diagnostic report
- Range of input file formats for all LSC counters

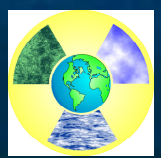
Geosciences Advisory Unit

RADDEC LTD
Radiochemical and Decommissioning Solutions Ltd

Organisations currently using the Raddec Pyrolyser

- GAU (Geosciences Advisory Unit): 3 furnaces
- AMEC-NNC (NIRAS) Ltd.: 5 furnaces
- Scientifics Ltd.: 1 furnace
- HPA (Health Protection Agency): 1 furnace (Formerly known as NRPB)
- AWE plc. (Atomic Weapons Establishment): 1 furnace
- Babcock BES : 1 furnace
- DSTL (Defence Science & Technology Laboratory): 1 furnace
- KAERI (South Korea) : 1 furnace
- 4 installations pending

- 14 Pyrolyser furnaces are being used for both commercial and research purposes in the UK.
- The Pyrolyser was invented, developed and commercialised by Raddec. The Pyrolyser design is copyright protected.



Geosciences Advisory Unit

RADDEC LTD
Radiochemical and Decommissioning Solutions Ltd

Raddec Ltd
PO Box 1210
Southampton
SO17 1ZQ
U.K.
Tel: (+44) 07739 898344
Tel & Fax: (+44) 02380 559367
Email: sales@raddec.com
www.raddec.com