CAMPOVERDE

WAYS TO BE SAFE

DATA SHEET
Pyrolyser Trio

DA OLTRE ***35 ANNI*** TRACCIAMO
PERCORSI PER LA ***SICUREZZA***

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

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The PYROLYSER series

- Easy to use system designed by scientists / analysts.
- Simultaneous decomposition of up to 6 samples.
- Efficient oxidation of the liberated ^{3}H and ^{14}C species (and ^{36}Cl and ^{129}I).
- The furnace design has been thoroughly tested and proven over many years. The system has been in use commercially since 2003.
- Continued R&D programme ensures that the system and procedures are based on the latest research and best available technologies.

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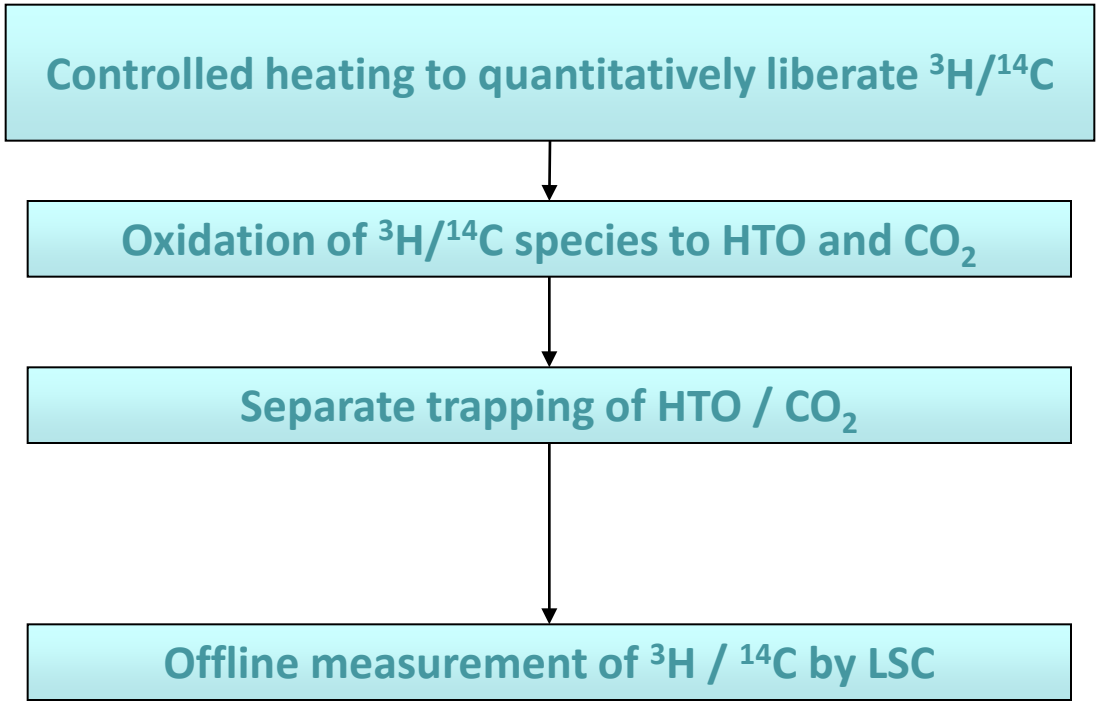
Principle of $^{3}H/^{14}C$ extraction



The Pyrolyser system

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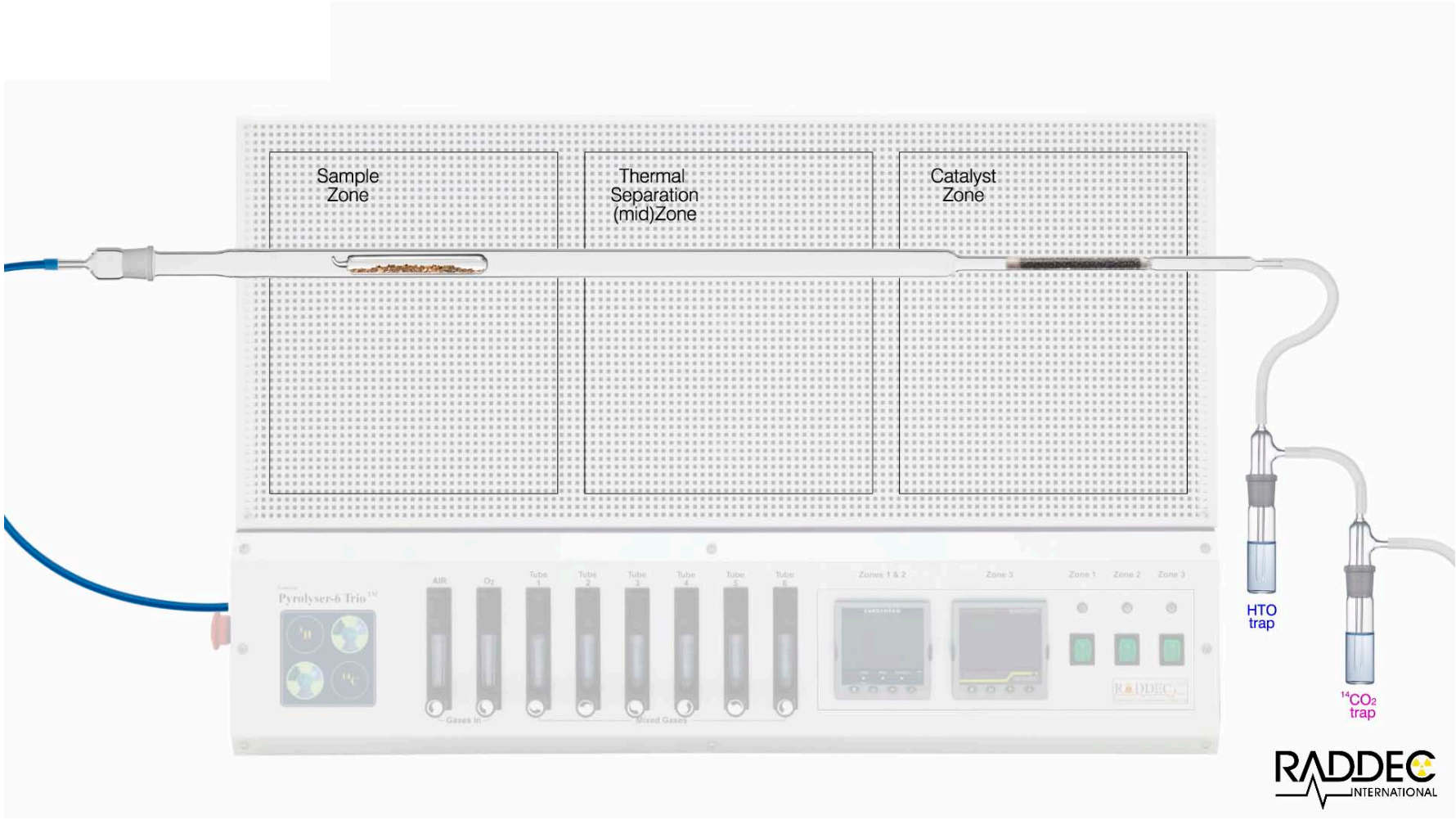
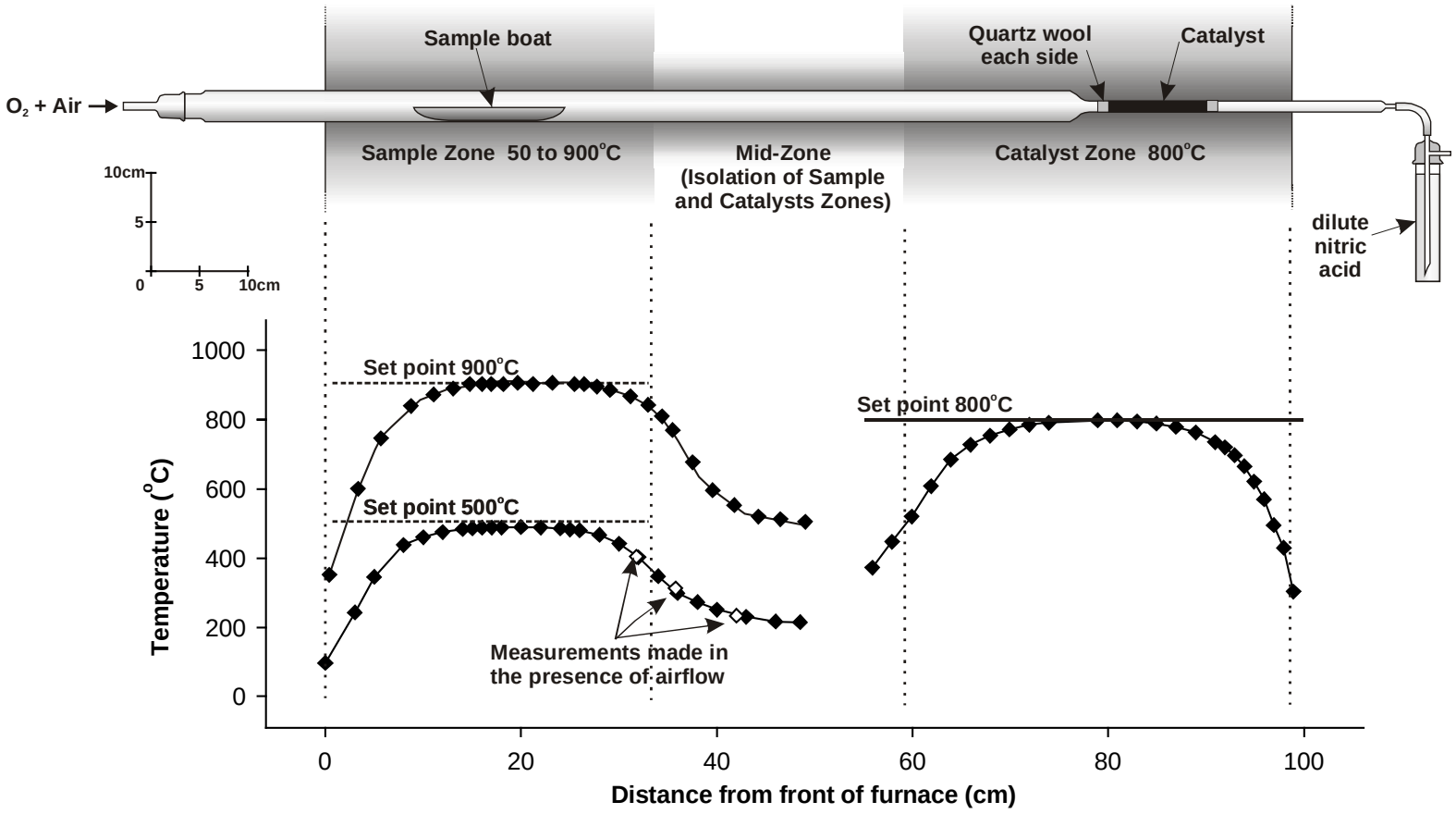
Purpose-built tube furnace for $^{3}H/^{14}C$ analysis

Also available as Pyrolyser 2 and 4 variants

Configuration of the system



Furnace temperature profiles



Sample boats

Silica boats

Aluminium
boats (< 600°C)

Bubblers

22ml borosilicate
glass bubblers

Features

- 6 samples decomposed simultaneously within a single furnace system in ~2-4 hours (depending on sample type) using a programmable thermal ramp.
- The multi-tube, small footprint design, permits high sample throughput without occupying significant laboratory space (unlike with multiple single-tube furnaces).
- 10g Pt-alumina catalyst per tube lasting 20+ determinations.
- Sample zone can operate up to 900°C permitting efficient ^{3}H extraction from concrete and graphite

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Features

- Rapid cool-down of sample zone enabling good cycle times between runs.
- Incorporation of a mid-zone furnace to prevent condensate problems whilst maintaining thermal separation of the sample and catalyst zones.
- 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.

Recent developments

- Over-temperature protection on all furnaces
- Fan cooling of electronics compartment
- Automated Gas Control to activate air and oxygen flows (AGS system)
- Improved furnace control and data logging functionality using Eurotherm 3504 & Nanodac controllers
- Glassware development for ^{36}Cl & ^{129}I extraction

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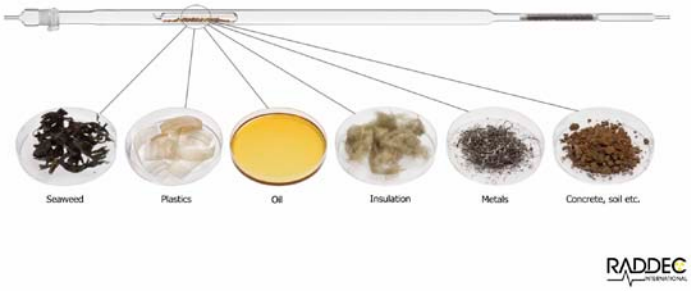
Sample types routinely run

- Environmental samples

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

- Decommissioning samples

  - Concrete, Brick, Asbestos, MMMF, Metal, Plastic, Desiccants, Paper, Electrical wire, Sewage sludge, Graphite, Paint, Oil etc.



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Sample sizes

- Samples with low organic contents

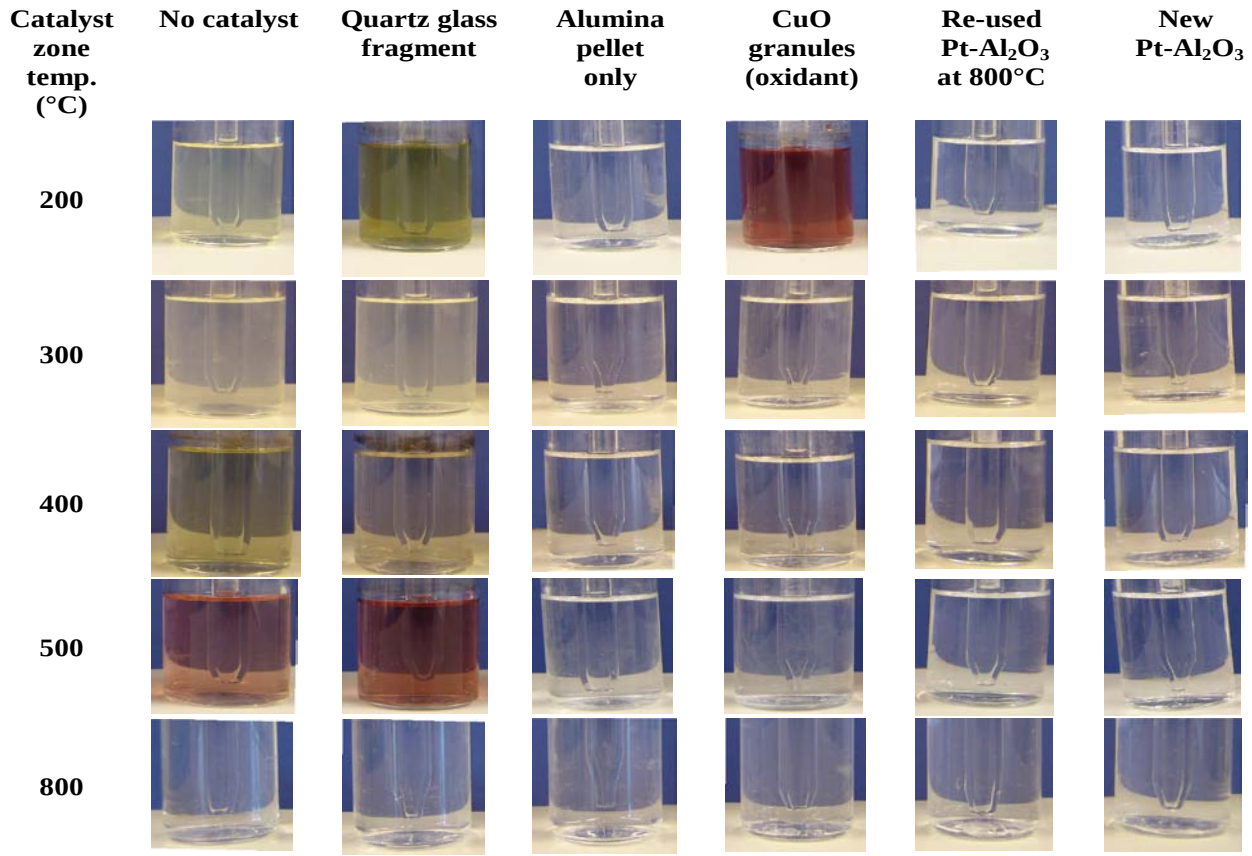
  Soil/Sediment, concrete, brick, metal etc. (1 - 30g)

- Samples with high organic contents

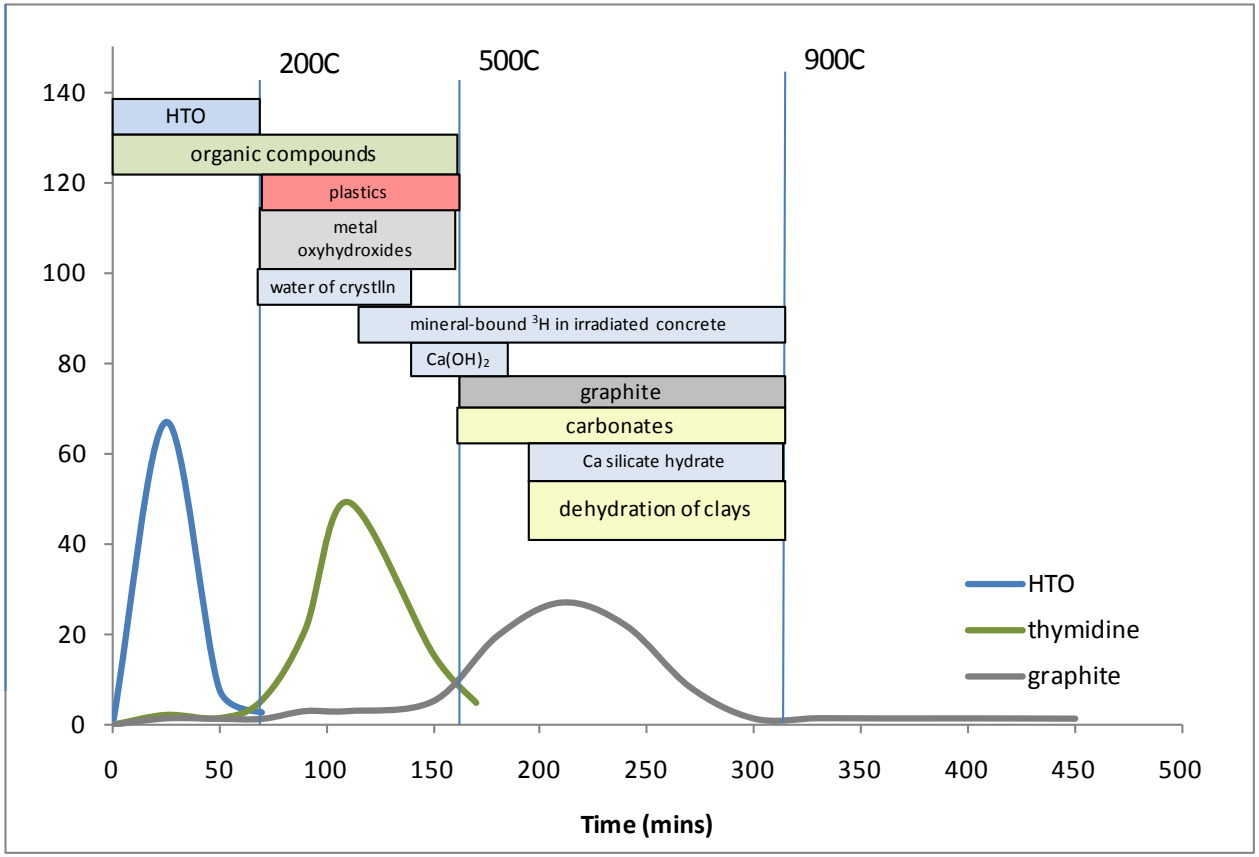
  Biota, plastics, organic rich sediment/soil etc. (Normally 0.5 - 1g if ^{14}C is being determined).

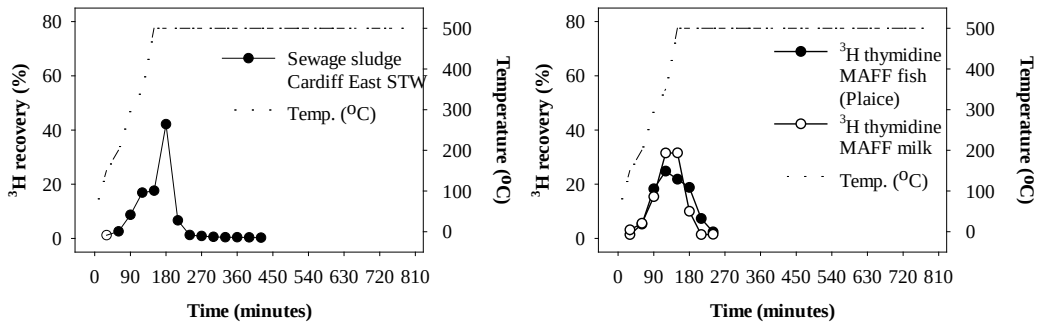
  Samples up to 5g can be run if only tritium is being measured.

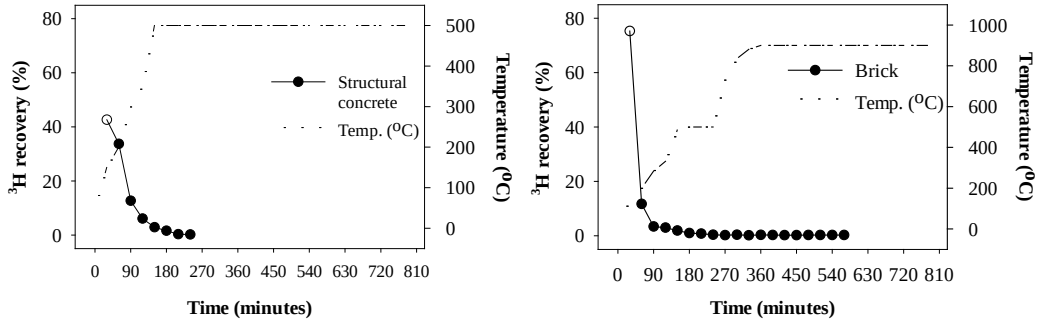
Catalyst performance

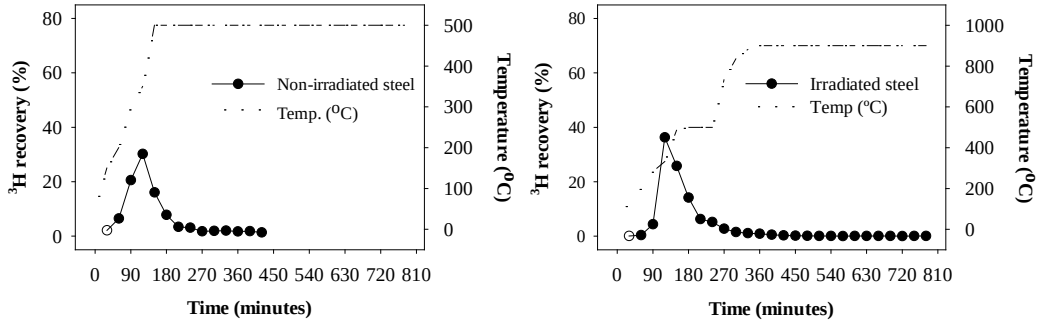


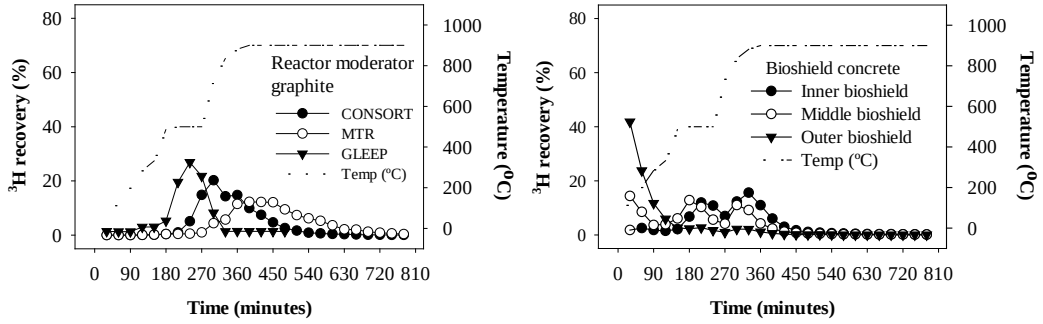
^{3}H desorption profiles











Thermal Evolution profiles

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Counting efficiency and furnace recovery for $^{3}H/^{14}C$

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

* Typical SQPE of concrete samples

** Average of 70 measurements using an organic $^{3}H/^{14}C$ standard

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

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Intercomparison results for ^{3}H

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	0.54 ± 0.04	0.539 ± 0.006
NPL (2009)	Tritiated water	1.69 ± 0.28 1.35 ± 0.22	1.688 ± 0.024 1.389 ± 0.030
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

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Intercomparison results for ^{14}C

Sample	Composition	Measured value Bq/g ± 1σ	Reference value Bq/g ± 1σ
NPL (2004)	C-14 carbonate in solution	23 ± 2	24.4 ± 0.5
NPL (2004)	C-14 carbonate in solution	0.9 ± 0.1	0.905 ± 0.006
IAEA C2	Carbonate	< 0.02	0.0112
IAEA C6	Sucrose	0.13 ±0.01	0.143
IAEA C7	Oxalic acids	0.02 ± 0.01	0.021

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^{36}Cl / ^{129}I analysis set

Bubbler solution 6 mM Na_2CO_3

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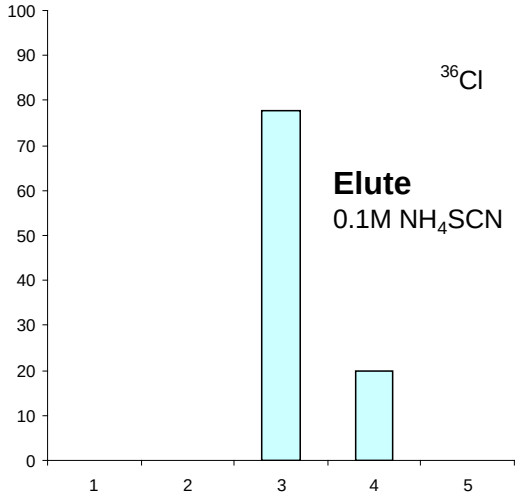
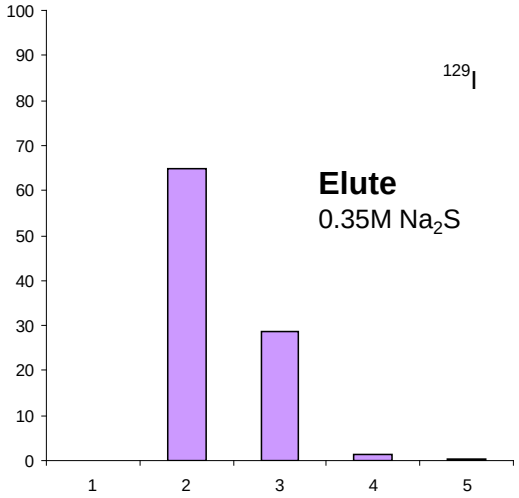
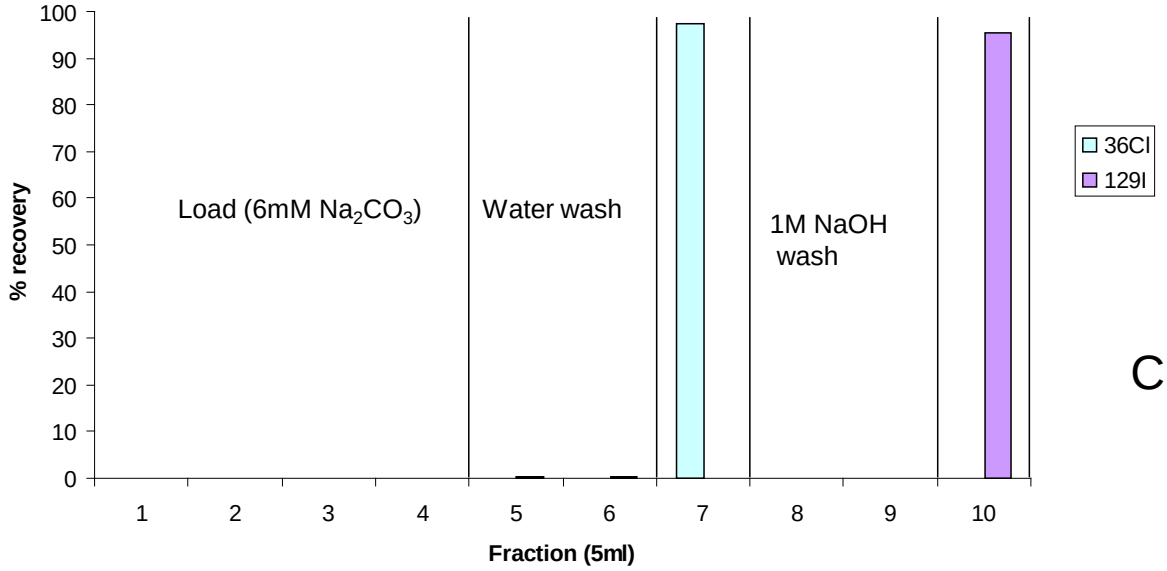
Separation using Cl resin

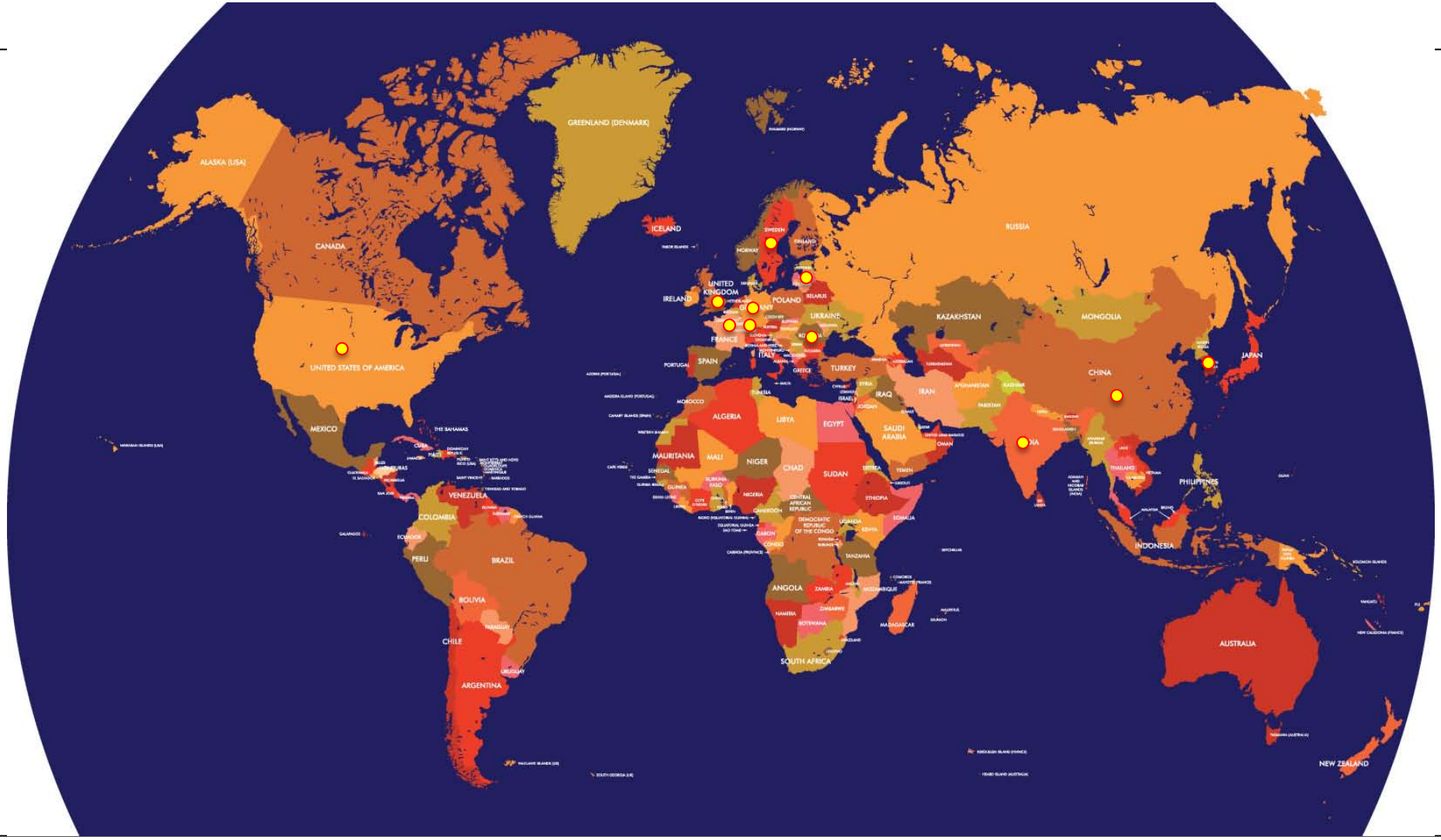






Cl recovery 98%

Pyrolysers worldwide



TOTAL = 63 worldwide (2014)

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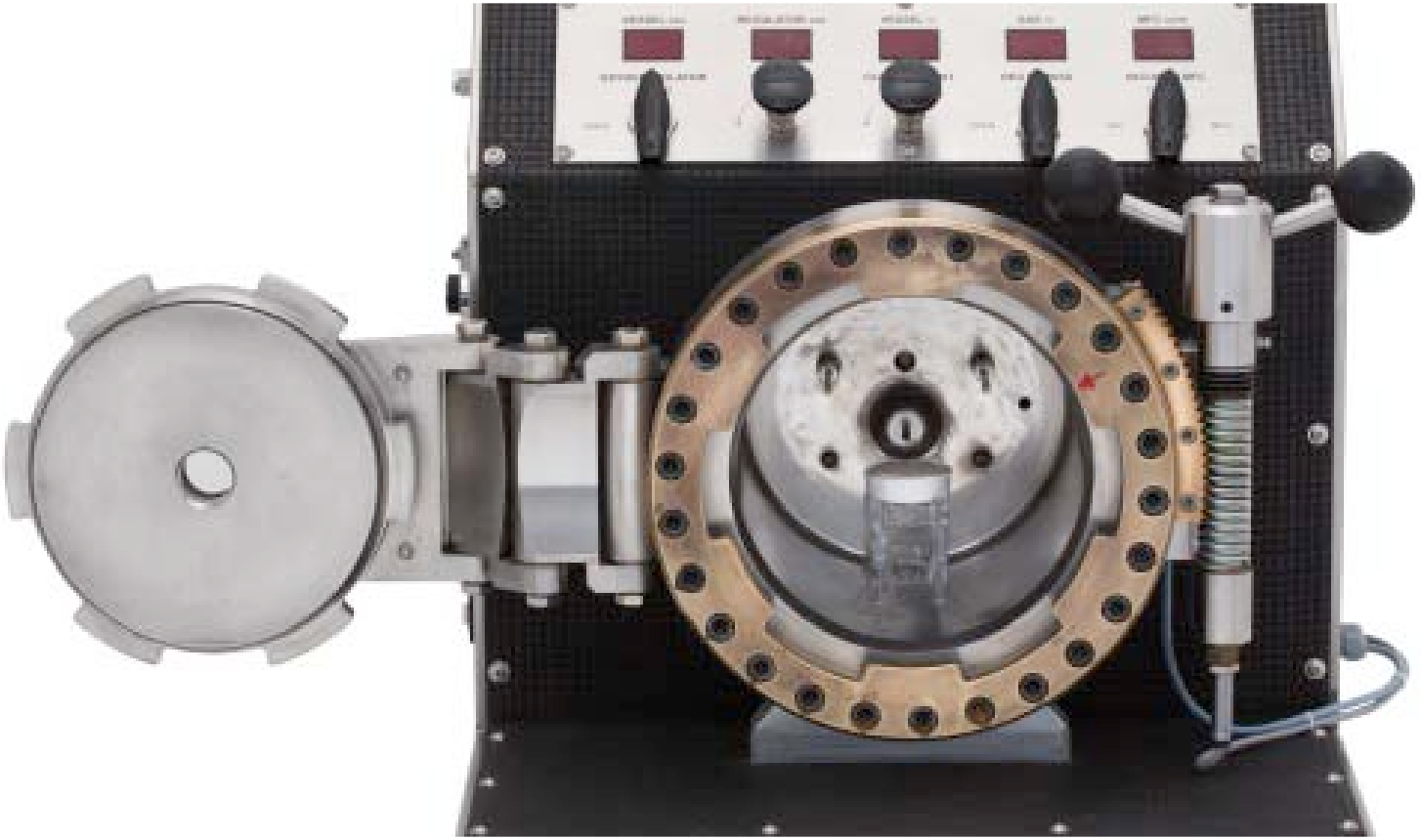
The new HBO system



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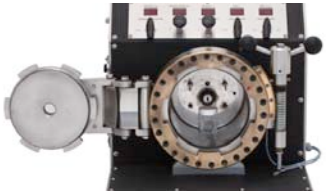
Advantages of HBO



- A high capacity sample oxidiser (RADDEC Ltd)
- Quantitative combustion in an excess oxygen environment
- Operates at pressures ≤100 bar
- Large samples (≤30 g) can be combusted
- Wide range of sample matrices maybe combusted:

  Cellulose (e.g. vegetation)

  Environmental samples (biota / veg.)

  Vacuum pump oil

  Nitrile rubber (e.g. lab waste)

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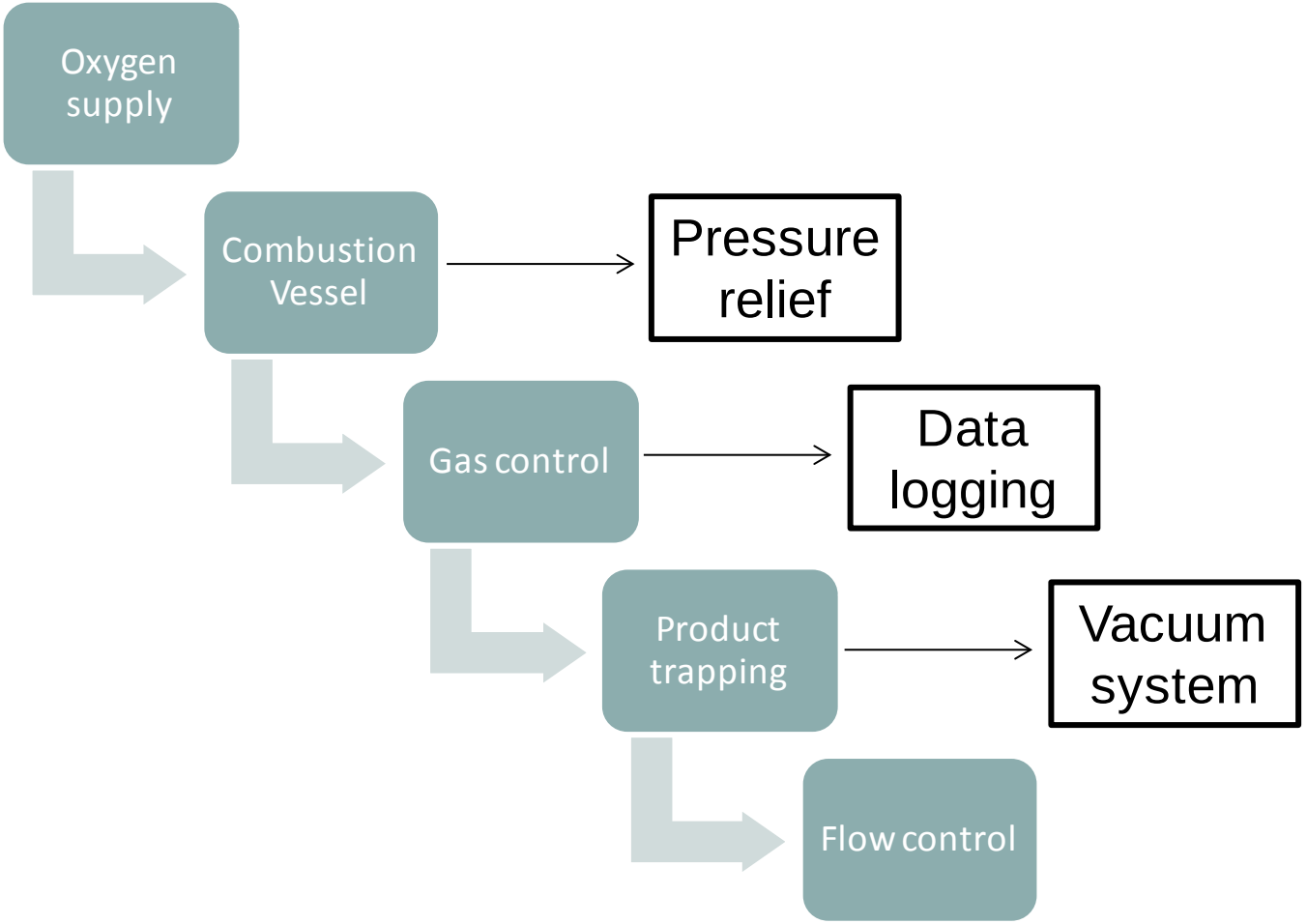
Instrument schematic



Combustion Procedure

Sample pelletised or cut to size

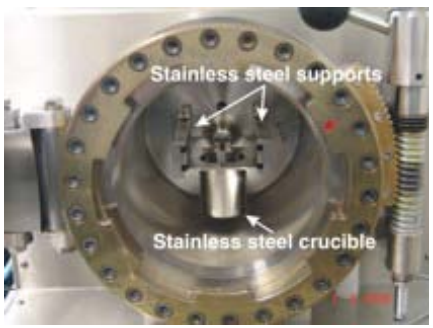
Sample loaded into disposable silica crucible

Chamber filled with either 10, 20 or 30 bar pure O_2

Sample combusted

Measurement by LSC

HTO / H_2O trapped from exhaust gas

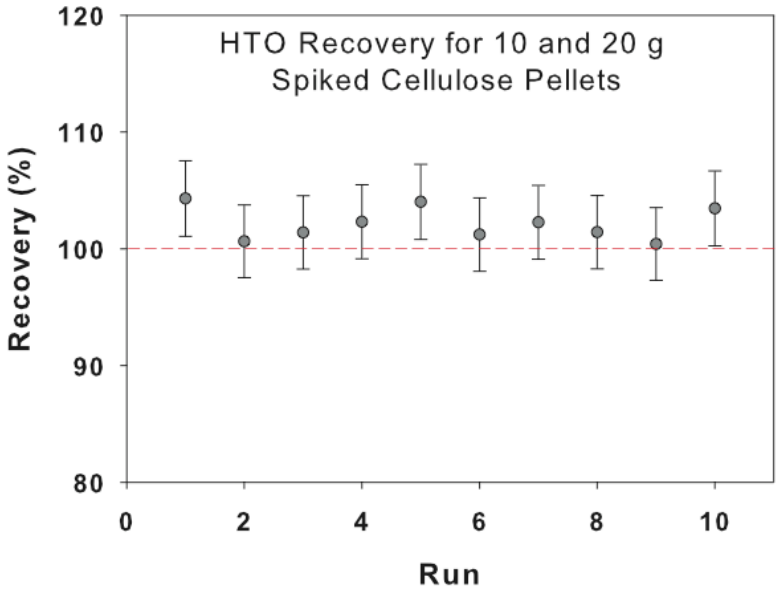


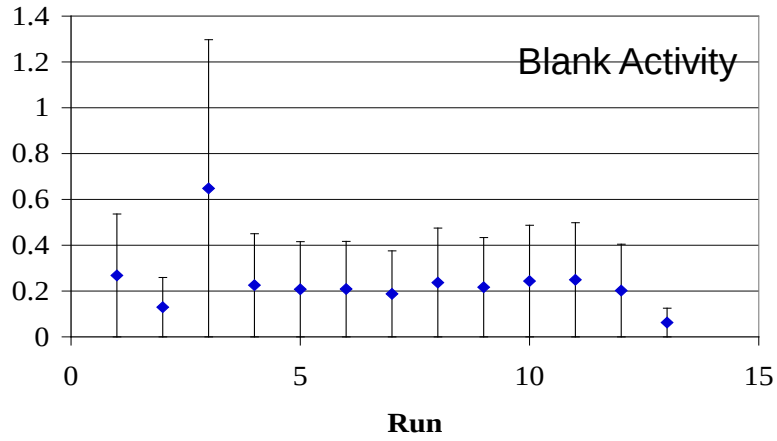
Original steel sample cup abandoned due to combustion quench

Instrument Evaluation

HTO activity recovery:

- Ability to recover HTO activity from within the HBO assessed
- Cellulose pellets spiked with HTO (~100 Bq)
- Recovered activity compared to spike activity
- Very small memory effect ≤0.7%

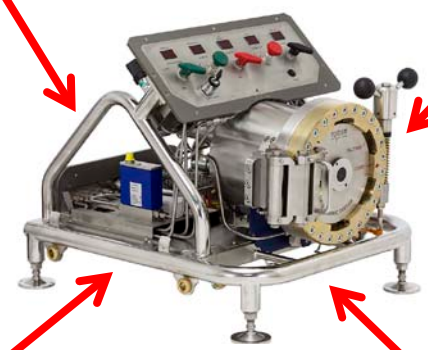


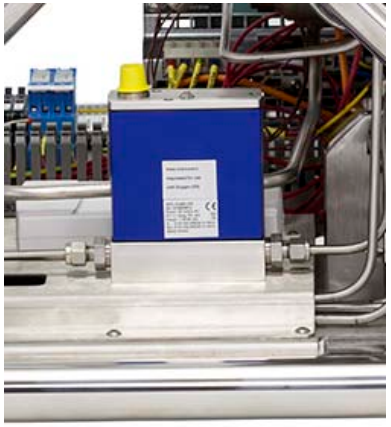


HBO_2 Mk2 developments

High volume pressure relief valve

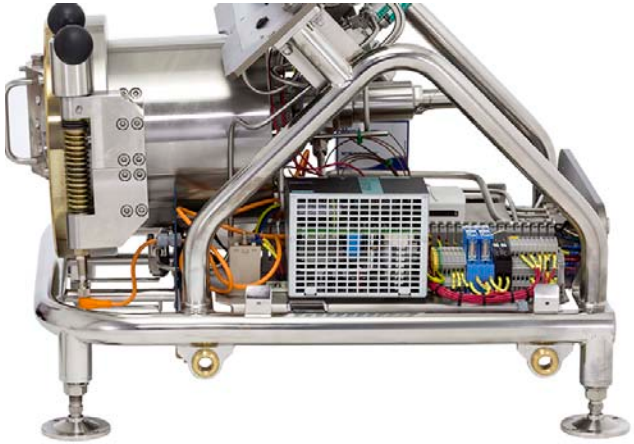
Improved pressure vessel with resistance wire ignition





Digital flow control and pressure regulation

Lightweight yet robust SS frame and lifting system



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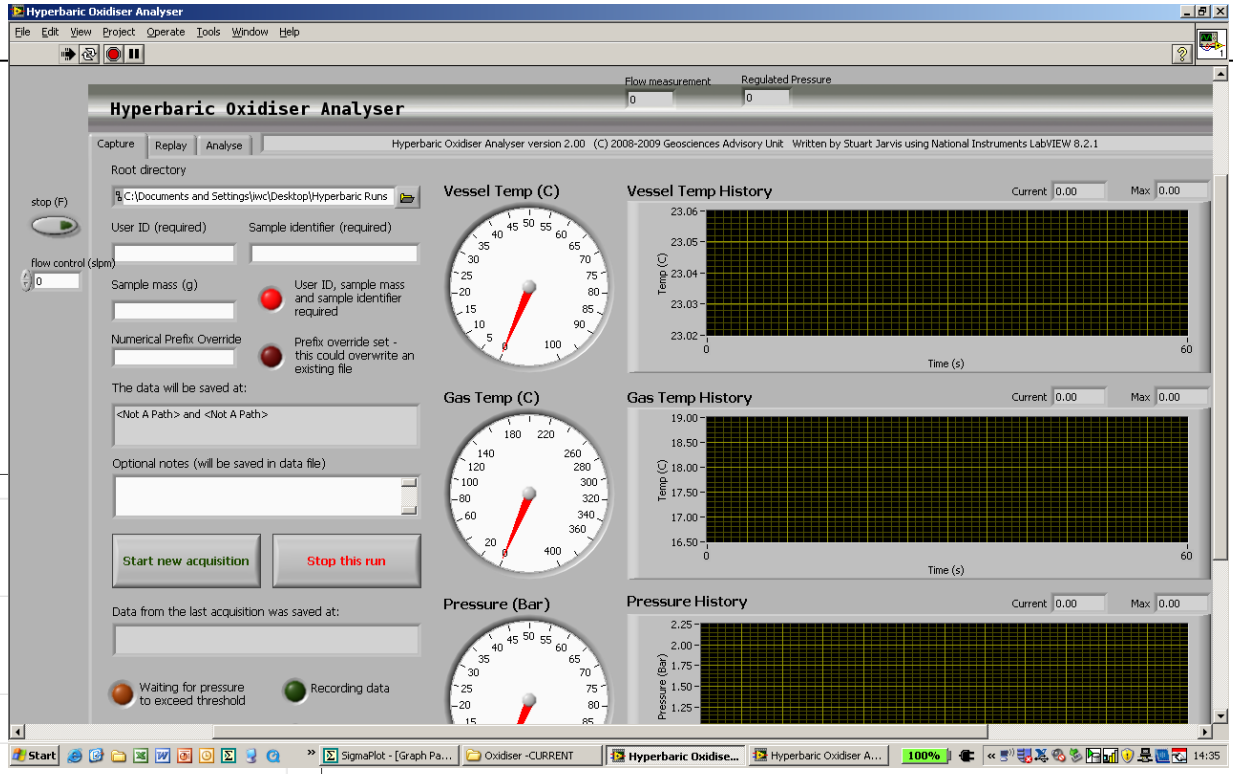
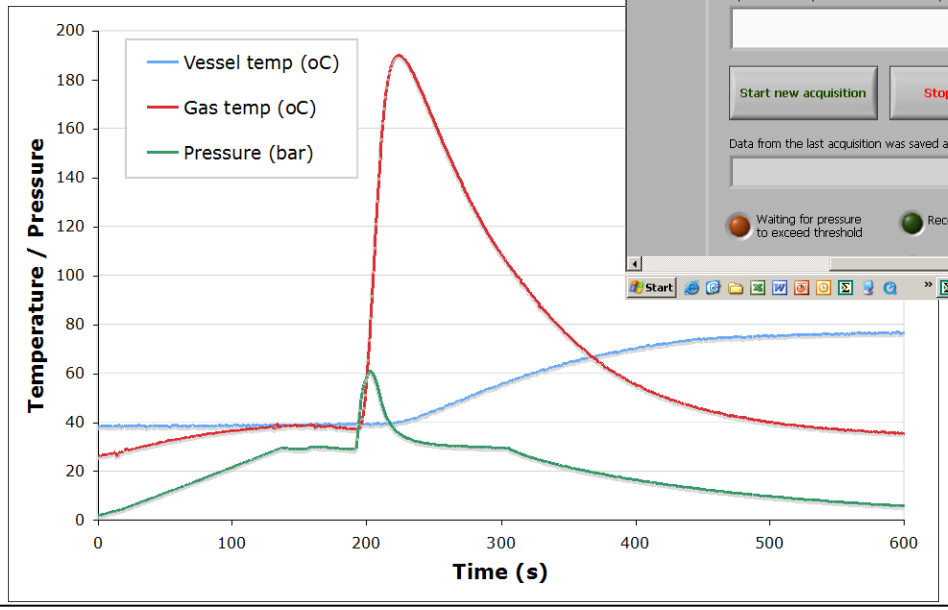
Real time data output

LabVIEW interface allows continuous data output and process control





Software development

LSC+

Liquid scintillation data processing

Raddec LIMS

Laboratory information management system

Itrax plot

Data visualisation software for XRF core loggers

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LSC+ Data processing software

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

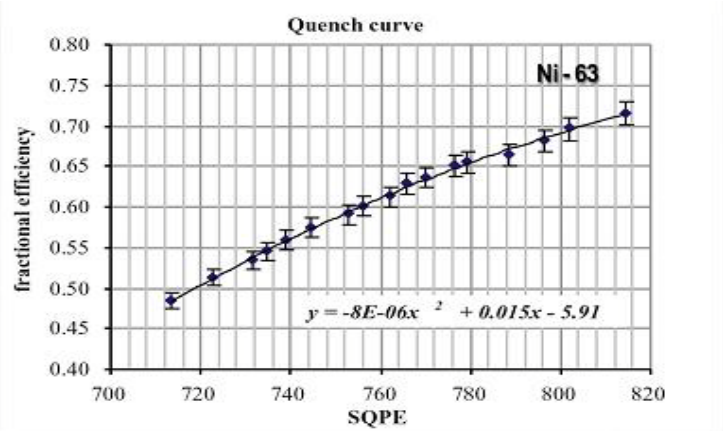
Report date : 7-Nov-2003
Customer : Raddec Ltd
Job reference : Raddec 372
Date samples received : 6-Nov-2003

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
- Eliminates transcription errors
- Calculates LODs (Currie)
- Quality Control Built-in
- Statistical analysis of results
- Full diagnostic report
- Range of input file formats for all LSC counters

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