

Radiochemical & Decommissioning Solutions

A new bomb-combustion system for ³H extraction

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Outline

- ³H extraction
- Analytical requirements
- Bomb combustion
- The HBO₂ system
- User case studies



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Options for ³H extraction

- Techniques for ³H sample preparation typically include combustion furnaces and sample oxidisers
 - → Versatile (sample type, throughput)
 - ↔ Well suited to inorganic matrices
- Organic matrices can be problematic
 - → Incomplete combustion
 - ← Limited sample mass / LOD

Appropriate analytical techniques limited





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Analytical requirements

Instances where extraction of ³H from organicrich samples is beneficial:

- Environmental monitoring
 - → Often organic rich + low LOD requirements
- Nuclear decommissioning
 - ← Orphan wastes e.g. oils, rubbers, plastics
- Fusion reactor operational support
 - → Heterogeneous soft wastes

Bomb Combustion

- Enable ³H extraction via complete oxidation in an excess oxygen environment.
- Few commercial systems available for ³H extraction, typically a Parr 1121 used

e.g. Moghissi et al., (1974)

- Limited sample size (< 10 g)</p>
- Manual operation
- Incomplete oxidation (quenching)



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The Raddec Hyperbaric Oxidiser (HBO₂)

- Designed specifically for ³H extraction
- High capacity combustion vessel
- Operates at pressures ≤ 100 bar
- Optimised for organic-rich matrices
- Large samples can be combusted (typically up to 30 g)



System components (I)



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Combustion Procedure



Sample pelletised or cut to size



Sample loaded into disposable silica crucible

Measurement by LSC



Combustion water recovered under vacuum



Sample combusted

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System components (II)



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Case Study 1

Environmental ³H Measurement

Canadian Nuclear Safety Commission (CNSC), Ottawa



All data & images courtesy of Nadereh St-Amant, CNSC, Ottawa.

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Background

- Discharges from Canada's nuclear industry regulated by CNSC
- Principal releases from D₂O moderated
 CANDU reactors e.g. Bruce, Darlington etc.
- Also ³H processing, removal and research facilities e.g. Chalk River, SRB Technologies etc.

Independent discharge and environmental monitoring – compliance and reassurance

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³H analysis

HTO in water, HT/HTO in air and <u>OBT</u> in foodstuffs



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System comparison (I)

Parr 1211

- Max sample size 10 g
- Combustion process not visible and often incomplete
- Manual (incomplete) combustion water recovery
- Cloudy / coloured combustion water
- Requires purification prior to counting by LSC



- Max sample size 30+ g
- Combustion progress is visible
- Integrated vacuum collection of combustion water
- Direct measurement of combustion water is possible

System comparison (II)

Parr 1211

tSIE % difference typically 12-24%





tSIE % difference typically 0-1%

tSIE % difference values measured for wheat samples, relative to distilled water quench values.

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OBT Environmental data

1.5 to 60 Bq/kg fresh weight

System	HBO ₂						Parr 1121	
Foodstuff	Fruits and Berries	Vegetables	Potatoes	Beef	Chicken	Pork	Milk	Fish
Typical LOD (10 g sample)	2 Bq/kg (0.5 - 1 for 20-30 g sample)						3 Bq/kg	
Tritium Processing Facility	11.8 - 17.5	1.5	1.5	N/A	N/A	N/A	1.5	N/A
NPP1	1.5 - 2	1.5 - 2	2	2 - 56.5	1.5	2 - 21	2	1.8 - 2
NPP2	1.5	1.5	1.6 - 2.1	N/A	1.5 - 10.5	N/A	1.5	N/A
NPP3	1.5 - 2.8	1.5 - 1.6	N/A	N/A	N/A	N/A	1.5	1.5 - 15.9

All results are Bq/kg fresh weight

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Case Study 2

Heterogeneous Soft Waste Culham Centre for Fusion Energy (CCFE), Oxfordshire, UK



All data & images courtesy of Natasha Cooper, CCFE, Didcot.

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Background

- Operation of the Joint European Torus (JET) generation of various tritiated waste streams
- Diverse mix of materials:

PVC, cardboard, housekeeping (mix of gloves, tissues, paper, plastic, cotton)

- Heterogeneous activity & organic rich
- Significant ³H activity Bq to kBq / gram
- HBO₂ operated in a controlled environment

Sample preparation experience

- Cellulose powder used to assist binding of housekeeping materials
- Samples pelleted in non-PVC cling-film
- Reduces incomplete combustion
- Vessel combustion chamber can be readily cleaned and re-polished







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HBO₂ – Pyrolyser validation

Sample	HBO ₂	Pyrolyser	HBO ₂ memory (%)
Cardboard	11284	11202 ± 2240	0.62
Cardboard	5885	7526 ± 1505	0.51
"Housekeeping"	614	3072 ± 614	0.33
"Housekeeping"	76	2674 ± 535	1.32
PVC	2973	2648 ± 530	N/A

- Good agreement for Cardboard and PVC samples
- Minimal observed memory
- Discrepancy between "Housekeeping" data are associated with the highly heterogeneous nature of the samples

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Conclusions

- The HBO₂ enables rapid and efficient recovery of ³H from organic rich samples up to 30 g
- The system is applicable to a wide range of sample types including biota and soft-waste
- Also applicable to orphan wastes such as oils and sludge's
- Offers improved LODs compared to thermal oxidisers (Pyrolysis/sample oxidisers).

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OBT Wheat Intercomparison



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