WMT-Carbolite Ashing Furnace for ^{3}H and ^{14}C Analysis.

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28/11/06

wmt
waste management technology

Tritium and Carbon-14 can contaminate a material either by:

^{13}C (n,g) > ^{14}C

^{14}N (n,p) > ^{14}C

^{17}O (n, a) > ^{14}C

^{14}N (n) > ^{3}H + ^{12}C

^{16}O (n) > ^{3}H + ^{14}N

^{6}Li (n) > ^{3}H + ^{4}He

^{238}U (n) > tertiary fission products + ^{3}H

- Direct material activation
- Contamination from the fission process
- Contamination from association with tritiated material

The industry requires the analysis of waste materials for tritium and carbon-14, where the specific activity ranges from environmental to ILW levels of activity.

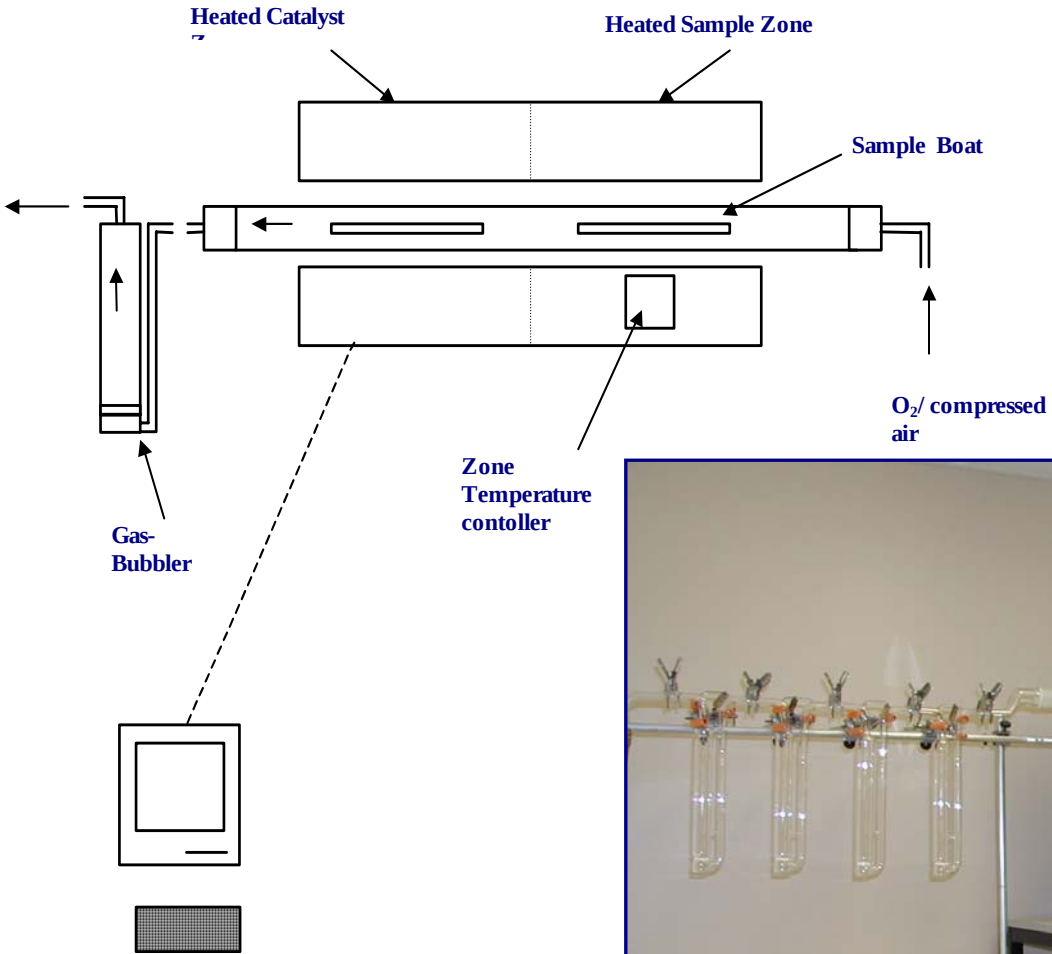
A combustion methodology where the hydrogen and carbon species are converted to water and carbon dioxide for selective trapping and assessment for H-3 and C-14 respectively is the only sensible solution.

But how to realise this?

System Requirements:

- **Flexible Design**
- **Precise control of sample combustion**
- **Low potential for Contamination/Memory Effects**
- **Efficient**
- **Cost Effective**
- **Large Sample Loading**
- **Rapid Throughput**

So we developed:


Heated Catalyst
Heated Sample Zone
Sample Boat
O_2/ compressed air
Zone Temperature contoller
Gas-Bubbler


FURNACE 8

WMT/Carbolite Combustion Apparatus

- All glass design.
  - minimises contamination
  - cost effective
  - easily replaced/replicated
- Efficient gas-bubbler design.
- PC control of sample combustion for single or multiple units enabling remote operation for hazardous/ILW materials.
- High sample loadings (e.g., 30-40g soft wastes)
- Rapid cooling - enables fast throughput

(e.g., 2-3 “burns” a day per unit depending on the material)

WMT/Carbolite Combustion Apparatus

Rugged, Reliable Furnace performance

– furnaces have been in continuous use without breakdown for 10 years.

-Single tube design means that if one furnace does fail there is no “one-out all-out” quandary for the user as is the case for “multi-tube” furnaces.

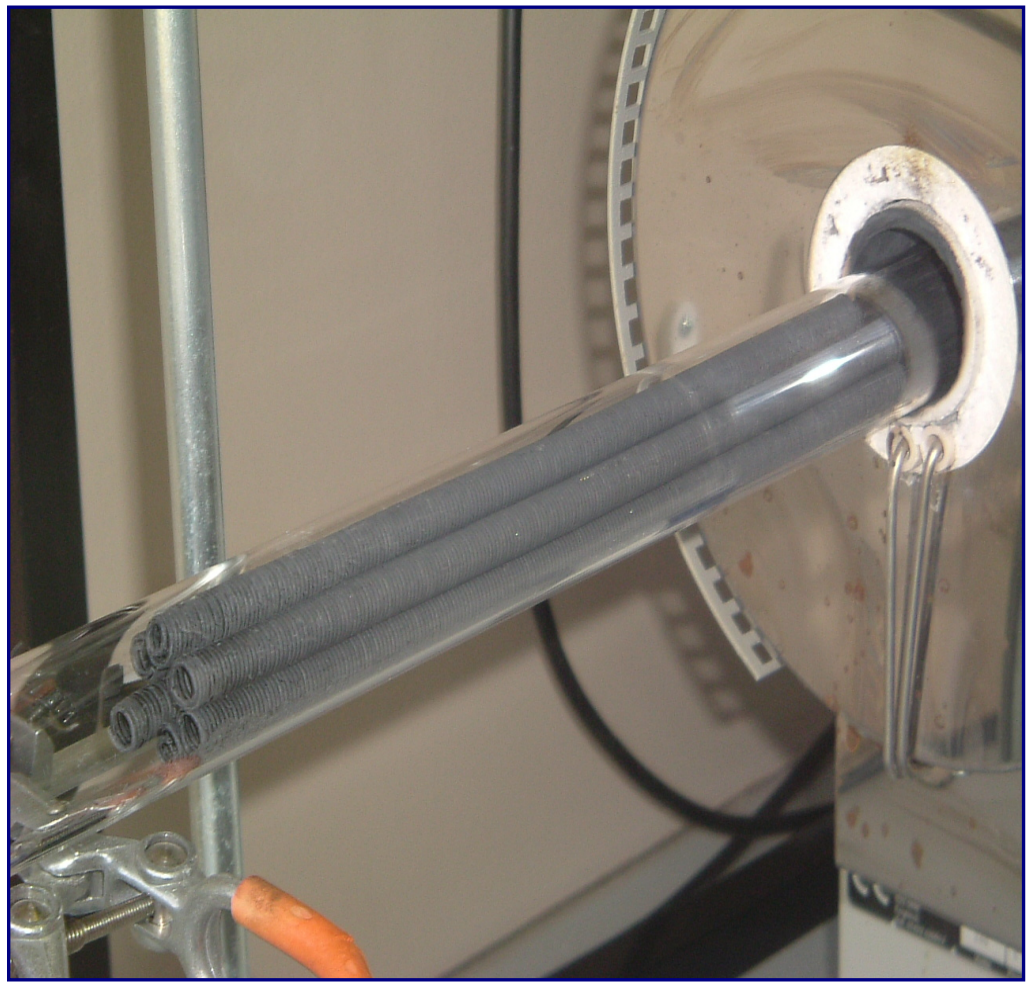
-Full repair service available.

WMT/Carbolite Furnace

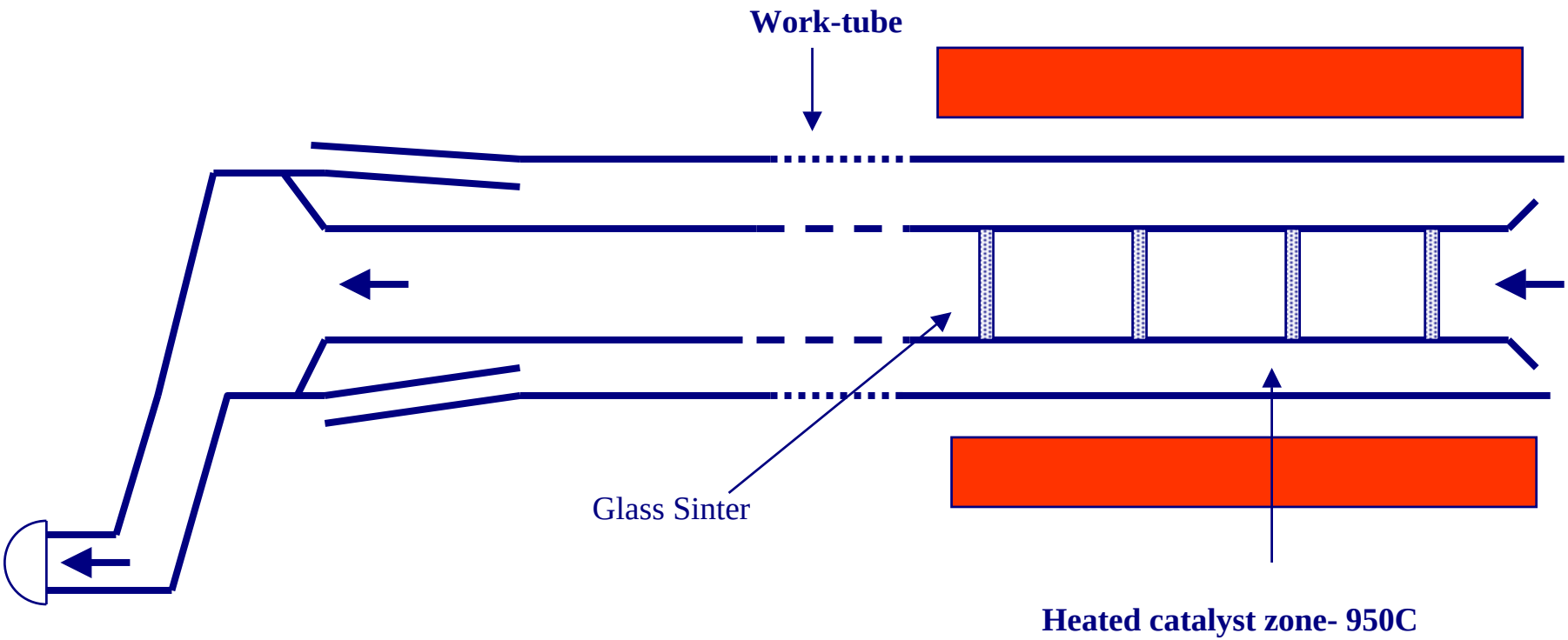
Shown by kind permission of Carbolite UK

Exhaust Manifold

Copper Oxide Catalyst
– copper wire sintered in O_2 at 850°C



New Exhaust Manifold Design- Under Test



Typical Combustion Protocol

- Stage One
  - Set Catalyst zone temperature to 850°C
- Stage Two
  - Set main carrier gas-flow to 200ml/min
- Stage Three
  - Raise sample to 350°C, dwell to ensure pyrolization of the sample
  - Raise temperature to 750-900°C (dependent on material); change carrier gas to O_2 at ~600°C
  - Dwell at final temperature for sixty minutes; cease heating; allow to cool

Materials Analysed (1).

Environmental:

Terrestrial: Herbage - Foodstuffs - Biota - Soils - Silts

Marine: Seaweed - Seafoods - Sediment

Materials Analysed (2).

Operational and Decommissioning Wastes:

Constructional:	Wood - Metals - Plastics - Rubber - Concrete – Brick – Asbestos cements - Roof Felt – Road Macadam
Decorative:	Paints - Wall Coverings - Carpet - Linoleum - Fabric - Ceiling Tiles
Novel Materials:	Reactor Graphite - Oils – Pond Skip Debris- Mixed Soft Wastes - Smelted Metals - Resins - Desiccant - PWR Reactor Coolant - Raffinates –FED-Supercompactor Leachate - Effluents

Typical Decommissioning Materials

Materials Analysed (3).

Health Physics Surveillance:

- Area Swabs or Smears

- Decontamination Liquors ($^{3}H/^{14}C$)

Performance - Reproducibility

Run No	FP	Soil	Grass
1	94	98	92
2	92	97	91
3	92	96	94
4	96	98	90
5	94	95	89
% Mean Rec.	94	97	91
Std. Dev	1.7	1.2	1.9

Source: Recent Developments in the Analysis of Tritium, Carbon-14, and Sulphur-35 Using a Combustion Technique, Wickenden D.A., "Environmental Radiochemical Analysis", Proceedings of the 8th International Symposium on Environmental Radiochemical Analysis, September 1998

Performance - Accuracy

Run No	FP	Soil	Grass
	Calc/Actual (% Error)	Calc/Actual (% Error)	Calc/Actual (% Error)
1	0.98 (1.2)	0.99 (0.9)	0.98 (1.2)
2	0.99 (1.2)	1.00 (1.1)	0.99 (1.2)
3	0.98 (1.2)	0.98 (1.2)	1.00 (1.2)
4	0.99 (1.1)	0.99 (1.2)	1.00 (1.2)

Source: Recent Developments in the Analysis of Tritium, Carbon-14, and Sulphur-35 Using a Combustion Technique, Wickenden D.A., "Environmental Radiochemical Analysis", Proceedings of the 8th International Symposium on Environmental Radiochemical Analysis, September 1998

Typical Lower Limits of Detection

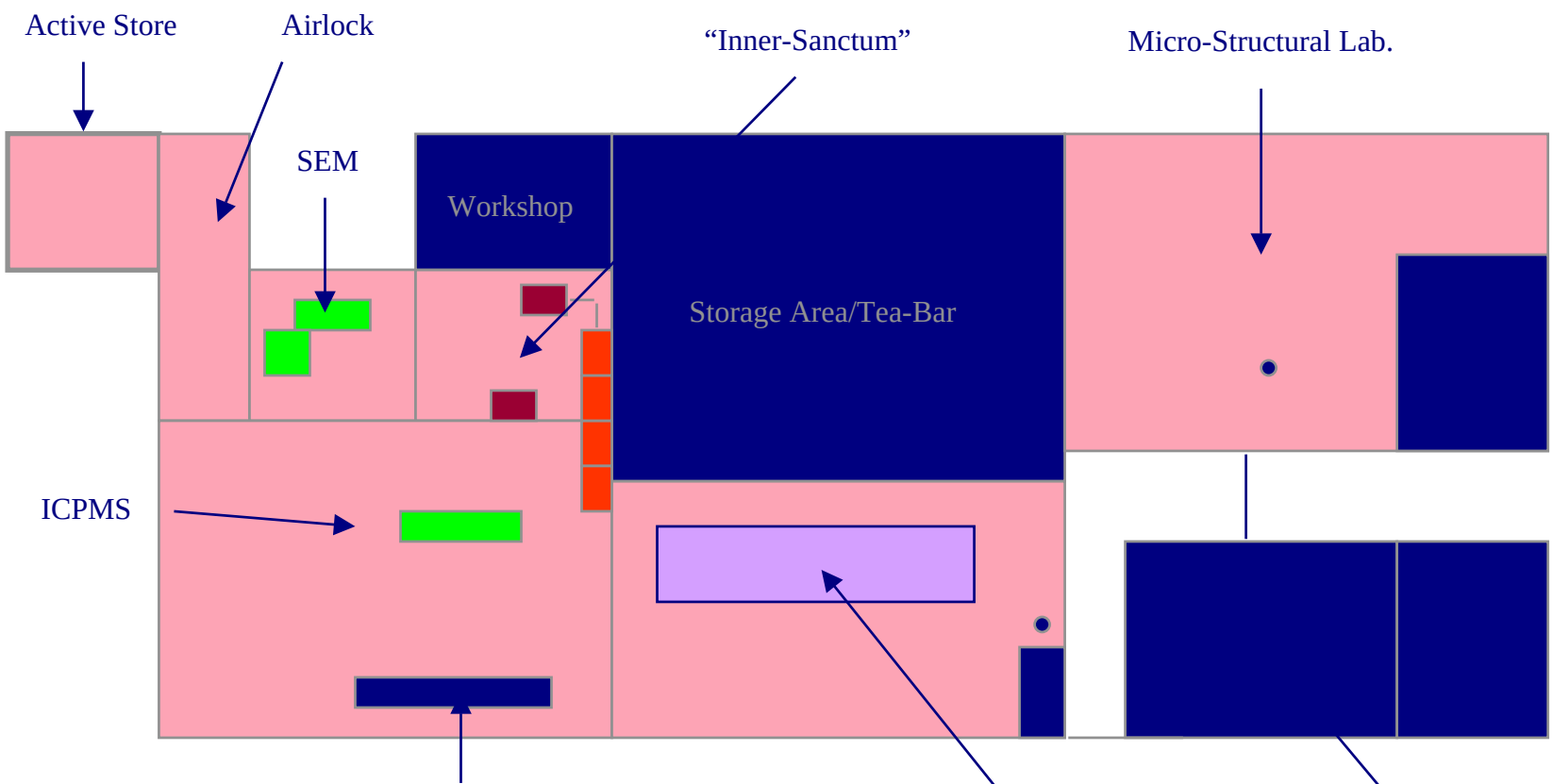
Sample Type	Soil	Grass	Plastic	Concrete	Graphite
Sample Wt (g)	30	20	2	40	2
^{14}C LOD ($Bq.g^{-1}$)	0.02	0.03	0.30	0.02	0.30
^{3}H LOD ($Bq.g^{-1}$)	0.04	0.06	0.61	0.03	0.61

Source: Recent Developments in the Analysis of Tritium, Carbon-14, and Sulphur-35 Using a Combustion Technique, Wickenden D.A.,
"Environmental Radiochemical Analysis", Proceedings of the 8th International Symposium on Environmental Radiochemical Analysis, September 1998

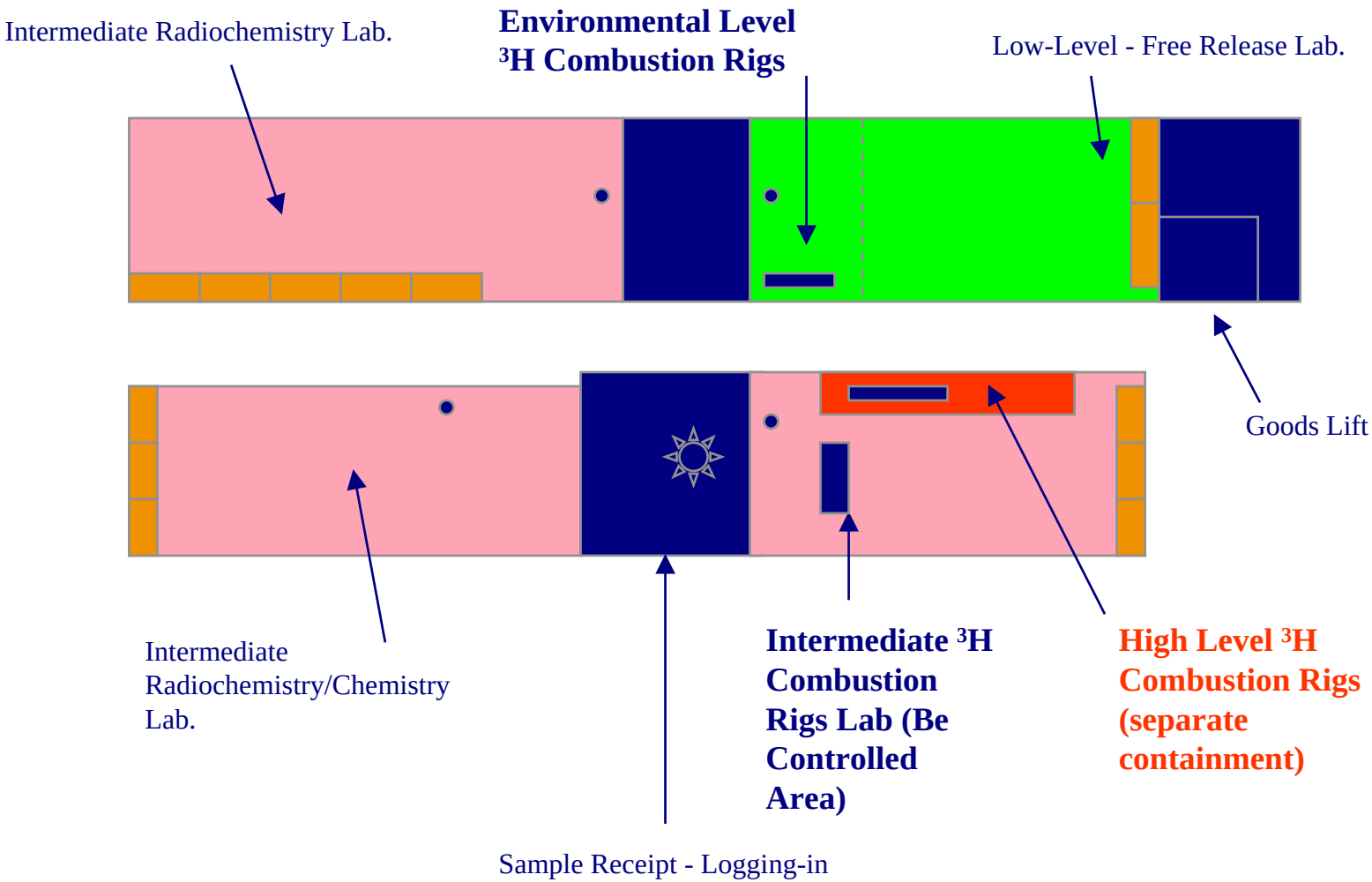
WMT Segregated Tritium Analysis Facility

- Low-level Environmental ($mBq.g^{-1}$)
- Free-release to Low-Intermediate level ($Bq – KBq.g^{-1}$)
- High Intermediate to High level ($KBq – MBq.g^{-1}$)
- High Level ($MBq.g^{-1} – GBq.g^{-1}$)
- Very High level ($>GBq.g^{-1}$ → TBq)

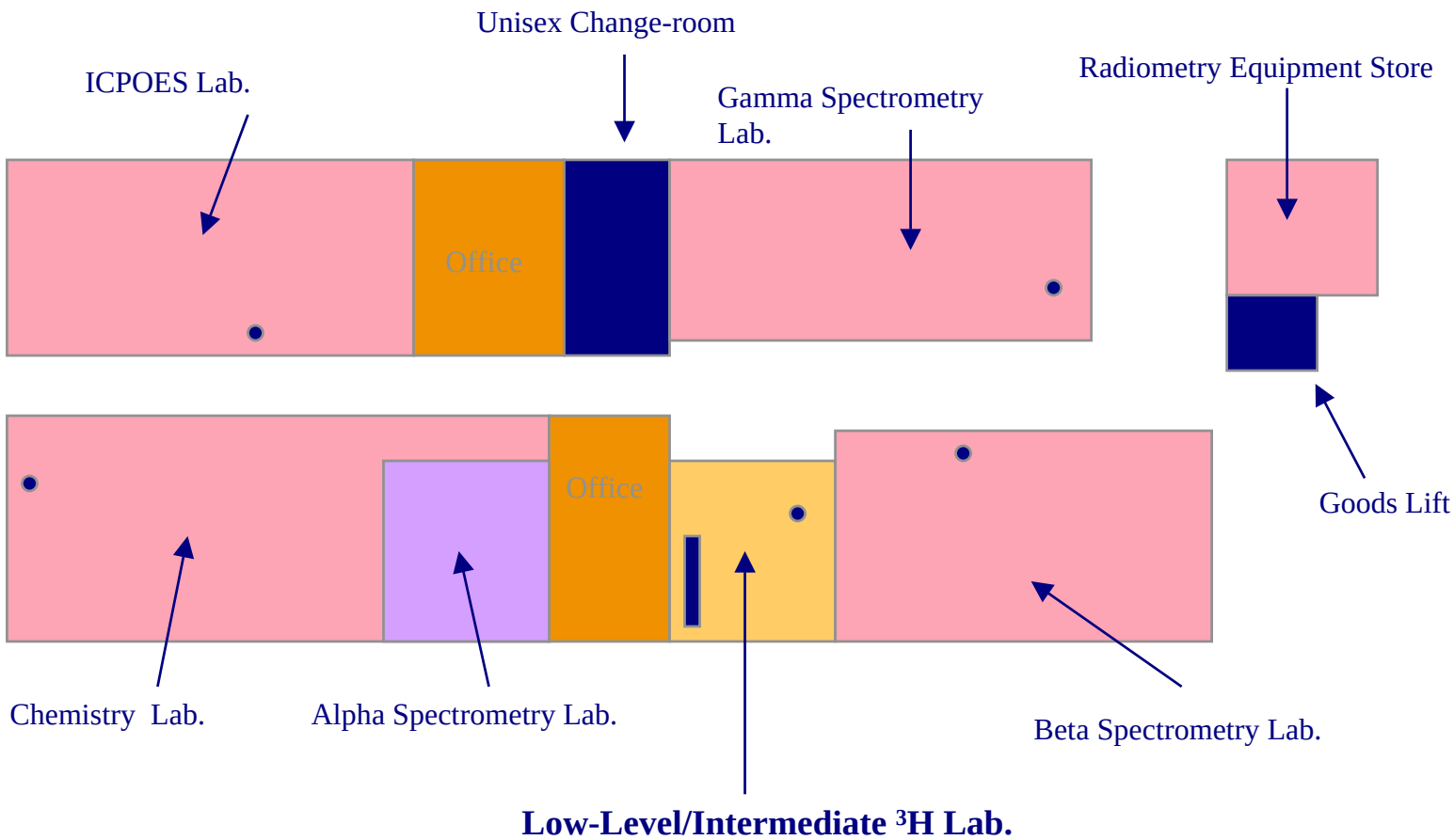
A51 Controlled Facility



A50 Supervised Facilities -



A50 Supervised Facilities: Ground Floor



LIMS workstation

Liquid Beta Scintillation Counters

LKB-Wallac 1220 Quantulus

LKB-Wallac 1411

Principle Interferences?

Volatile Radionuclides:

Technetium-99 as volatile Tc_2O_7

Ruthenium-106 as volatile RuO_4

(Sulphur-35 as SO_x)

Solutions:

Tc and Ru easily removed by performing an alkaline distillation of the primary bubbler trapping agent.

$S^{35}O_x$ can be prevented from reaching the gas-bubblers by placing a sacrificial silver catalyst, i.e., a ball of fine silver wire, just before or after the copper oxide catalyst bed.

The separation of tritium from waste material as tritiated water using a combustion methodology is not definitive.

The analysis must be performed in the light of all available analytical data such as that given by gamma spectrometry, which could indicate the presence of Ru-106.

Always interrogate the LSC beta-spectrum.

Patience is a virtue. Leave the analysis for H-3 (C-14) until last?

WMT/Carbolite Furnace

Shown by kind permission of Carbolite UK

So what do you get for your money?

Carbolite Type 12/38/850 Dual Zone Tube Furnace:

- 8 Step controller for Sample Zone
- Three Quartz Glass Work-Tubes
- Three Complete Sets of Glass Gas-Bubblers (12 total)
- All work-tube connectors/flow-controllers (inc. molecular sieve waste aerosol trap)
- Copper Wire Catalyst (3 “charges”)
- Comprehensive Instruction Manual

Price Ca. £6-7K (under revision)

Optional Extras:

Computer Control (comms and software)

16 Step Sample Zone Controller

Over-temperature control on either or both temperature zones

For further details:

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