Karlsruhe Institute of Technology

30 Years TLK: Development & Operation of the TLK Tritium Loops

Handschuhkastenreihe nach dem
Zusammenbau (ohne Scheiben)

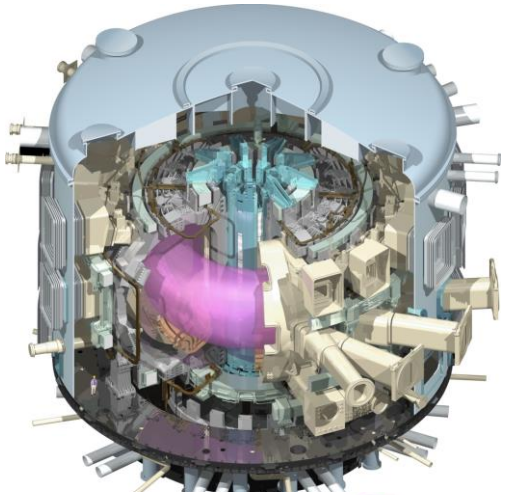
20.12.89

Chris Komorowisky (LAB) Dr. Ken Tarr (LAB) Martin Carney (CFFTP)

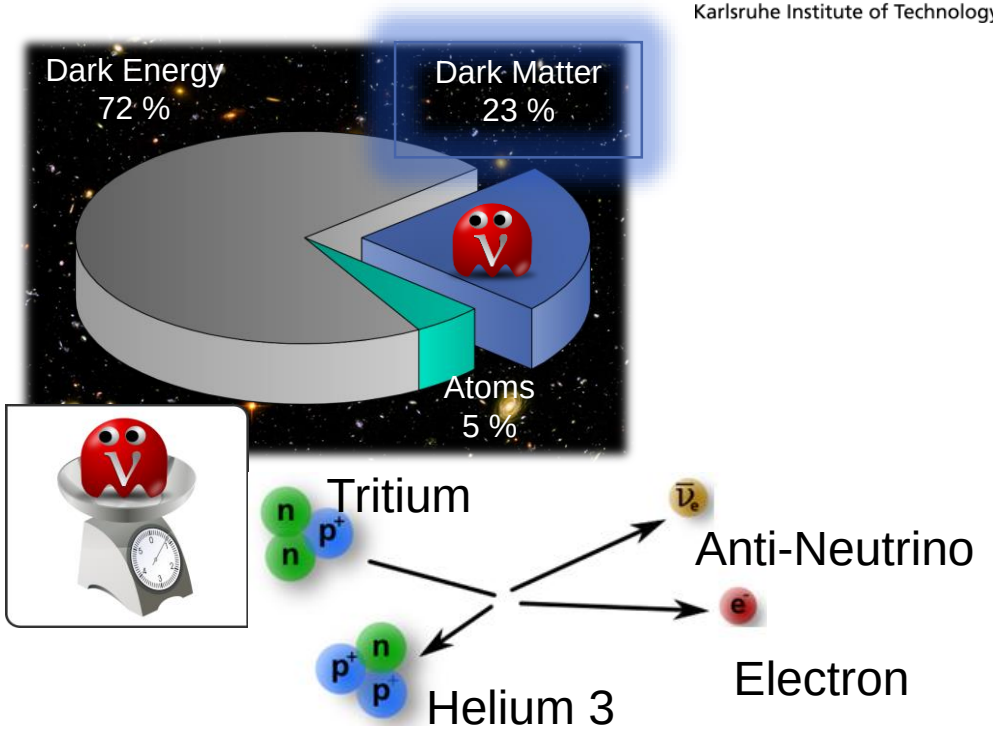
EG & G Leiterin zur Inspektion nach Montageende in der KATRIN-Fertigungshalle

Vor dem TLK-Gebäude

The Missions



Development of Fusion Fuel Cycle

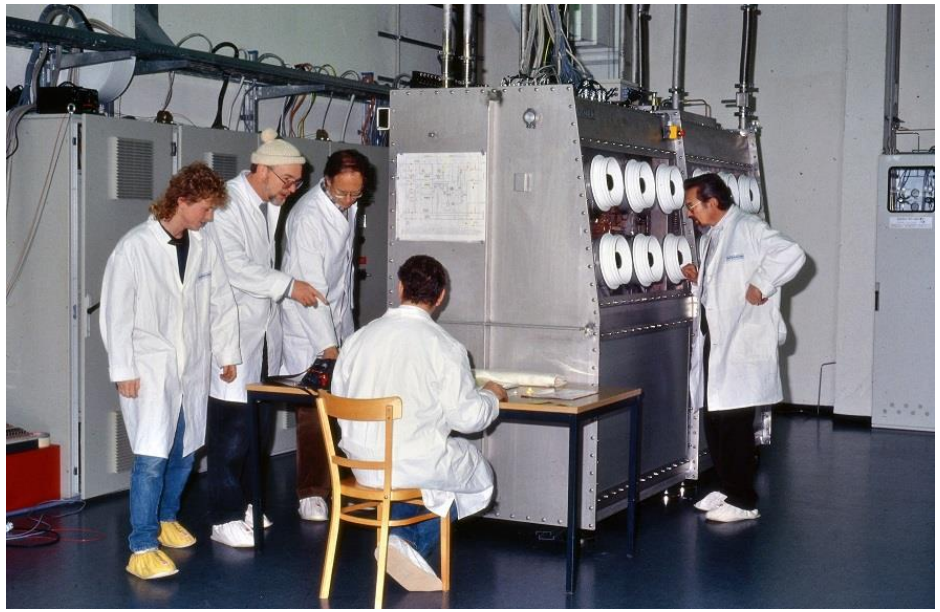


KATRIN: Answer to a fundamental question in Astroparticle-Physics

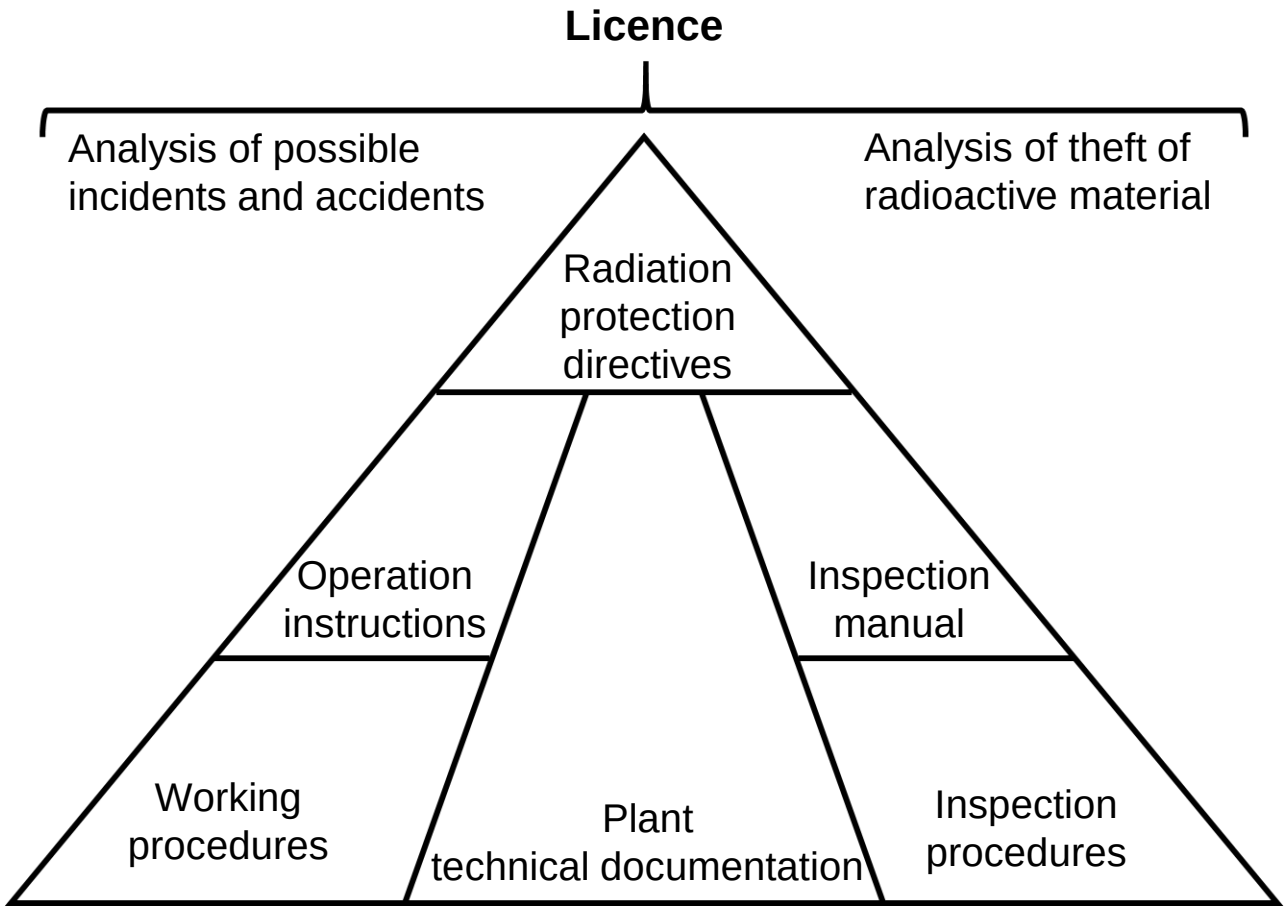
Both missions necessitate reliable handling of substantial amounts of Tritium in a closed loop

The Licence for Operation of TLK: Past - Present

License (§12 StrlSchG)	T1/93	T6/07 (12/2019)
H-3 (g)	10	40
H-3 (Bq)	≈3.5 E15	≈1.4 E16
H-3 fumehood	-	1.0 E10
U-238 (kg)	100	100
Rb-83 (Bq)	-	1.0 E10
Rb-84 (Bq)	-	5.0 E9
Covering	Original installations (expired)	TLK/KATRIN (valid)



The Licence for Operation of TLK: Administrative Frame



Administrative rules:

Implementation of safety framework into daily work

- Addressing § 45 StrlSchVO (radiation protection directives)
- Organisation
- Radiation protection & handling of radioactive substances
- Formal procedures for operation & experiments

Technical rules:

Implementation of design, construction and quality requirements

- Technical Terms of Delivery and Acceptance (TLA)
- Technical Editing process

Safety Framework:
Set of rules summarising the administrative and technical requirements to comply with tritium license

The Licence for Operation of TLK: Technical Standards

- TLK operation experience
- TLK developments

Technical terms of delivery & acceptance for

Devices and Vessels

for the
Karlsruhe Tritium Laboratory
TLA No. 01 / 2007

1st edition dated August 14, 1997
2nd edition dated March 03, 2004
3rd edition dated March 03, 2007

Requirements for containments:

- Design, Planning & Manufacturing
  - Welding
  - Tritium facing materials
  - Leak rates
  - Type of Fittings
  - Type of Surfaces
- QA requirements:
  - Radiography
  - Leak test
  - Cleanliness

Technical terms of delivery & acceptance:
Implementation of DIN-, EN-, ISO- Norms, AD2000 and TLK experience as a TLK Technical Standard

The Licence for Operation of TLK: Tritium Technical Requirements

Requirements:

Primary containment:

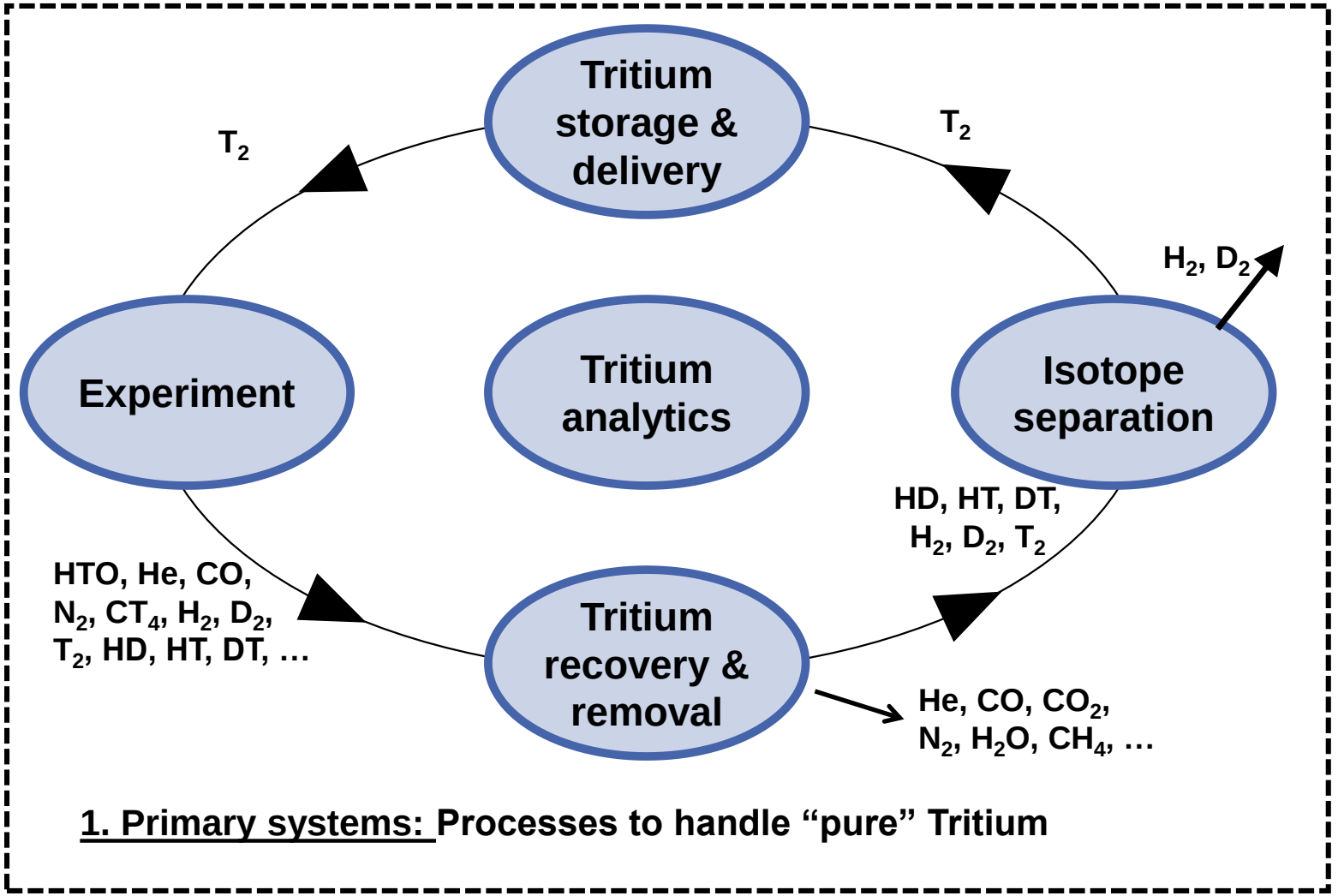
- $<1 \cdot 10^{-8}$ mbar l s^{-1}
- Limited choice for tritium facing materials (metallic or ceramic)
- Very strict QA requirements

Glove box (2. containment):

- 0.1 $\%_{vol}$ h^{-1}
- Polymer allowed
- Less strict QA requirements

Technical standardisation in administration, design requirements and implementation

Tritium Confinement: Basics



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Implementation of Multi Barrier Philosophy provides reliable tritium confinement

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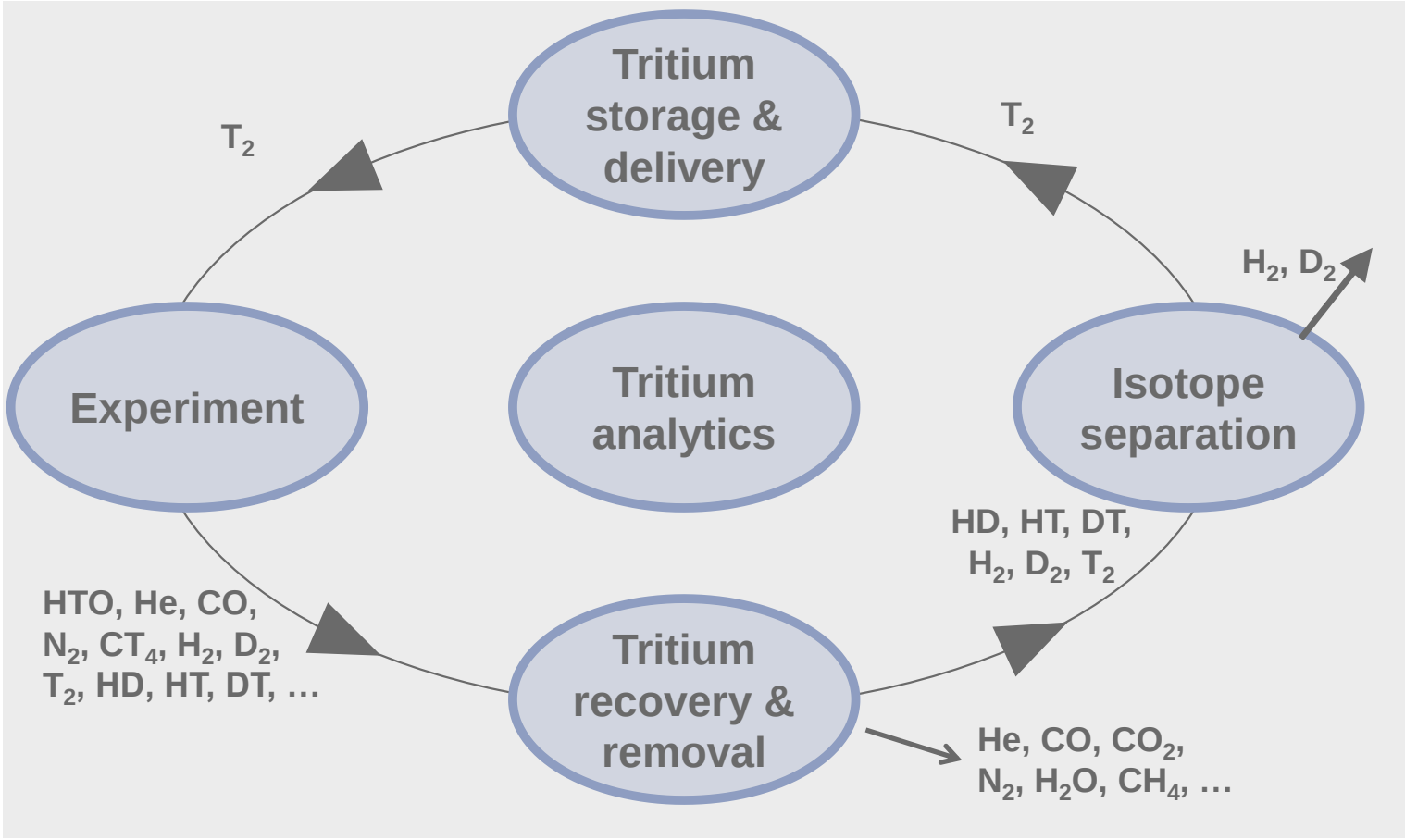

1. Primary systems: Processes to handle "pure" Tritium

2. Containment (Glove Boxes, Tritium Retention systems)

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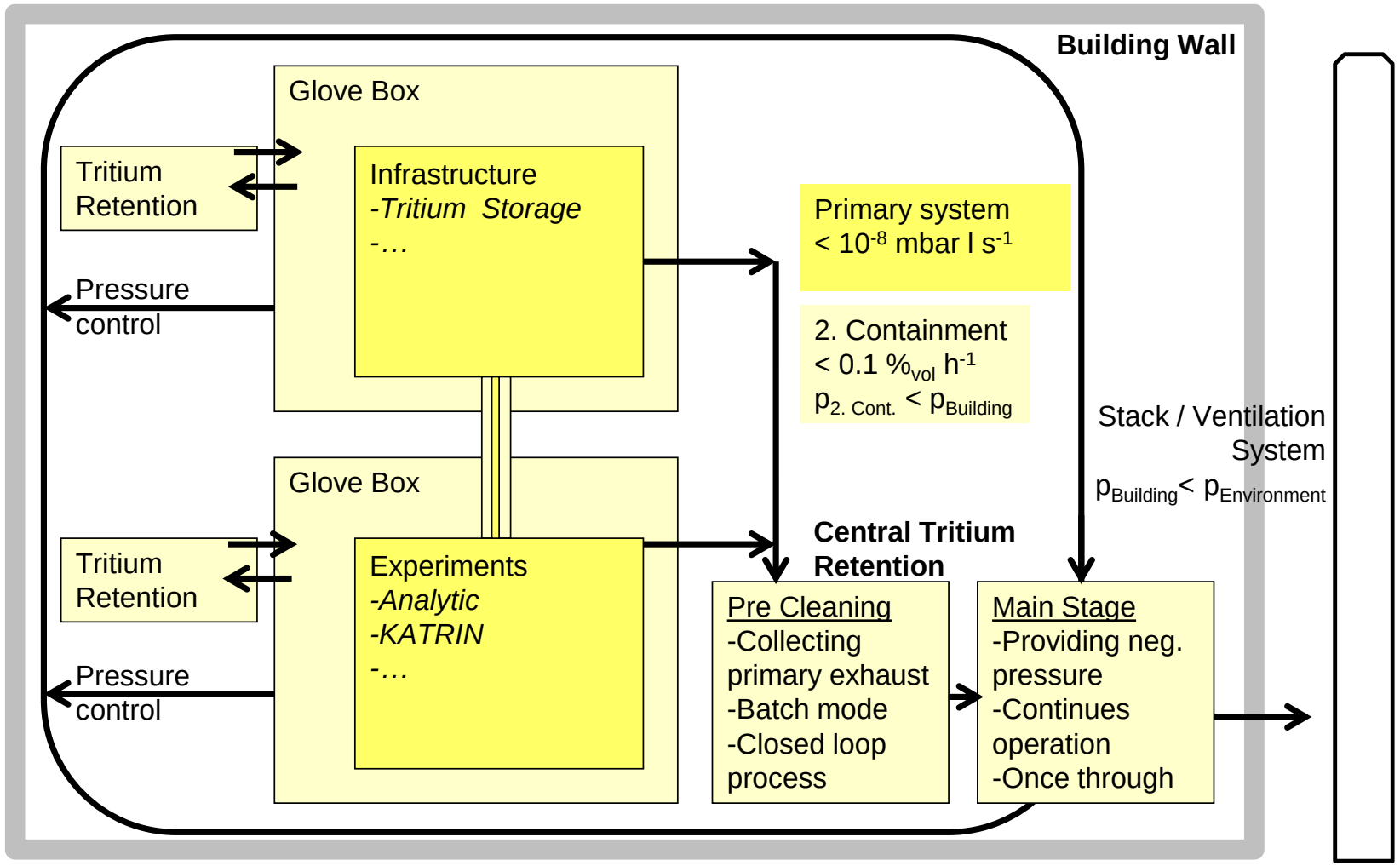
Barrier concept: 2. Containment

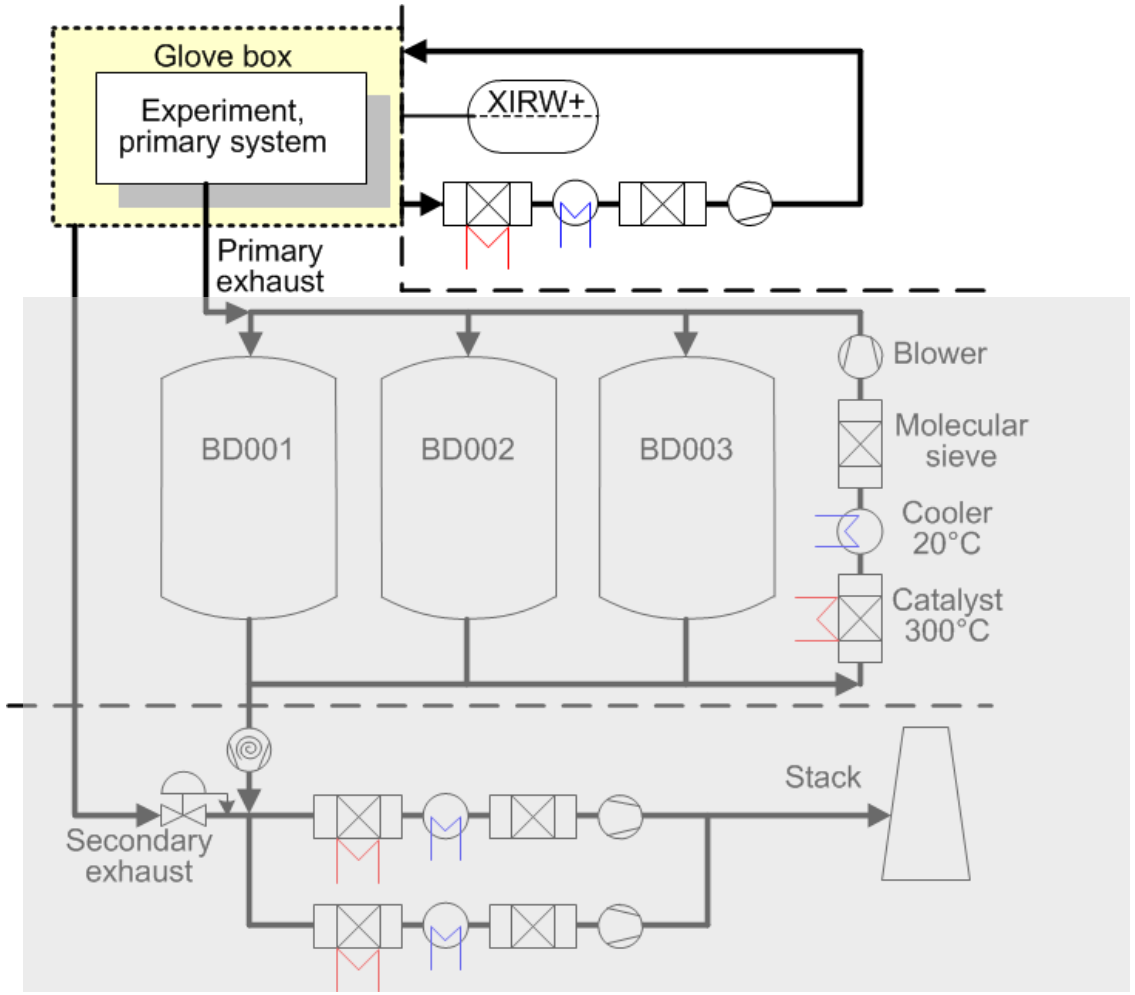


2. Containment :

- Glove Box
- Double wall tubing connecting systems
- Tritium retention systems (central and individual systems)
- Below atmospheric pressure control

Implementation of Multi Barrier Philosophy provides reliable tritium confinement

2. Containment: Tritium Retention Systems



Reliable detritiation before exhaust

Individual for glove box

- Closed loop for one glove box
- Blower, catalyst & mol sieve number related to box volume
- Glove box atmosphere only

Central tritium retention

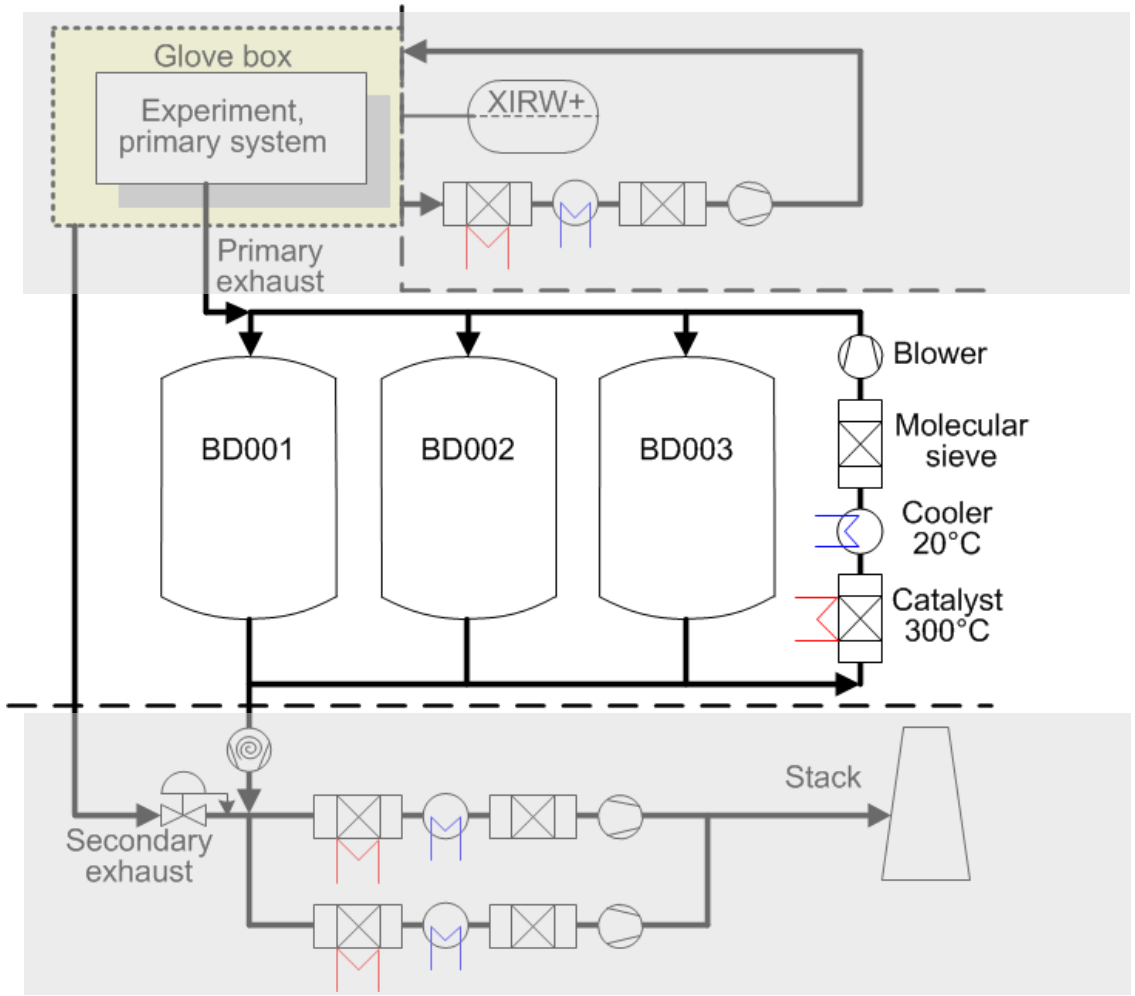
Pre cleaning stage

- Batch mode process
- Collecting primary exhaust

Main cleaning stage

- Once through continuous process
- All exhaust from pre cleaning and glove boxes
- Double redundancy
- Last clean-up stage before stack

2. Containment: Tritium Retention Systems



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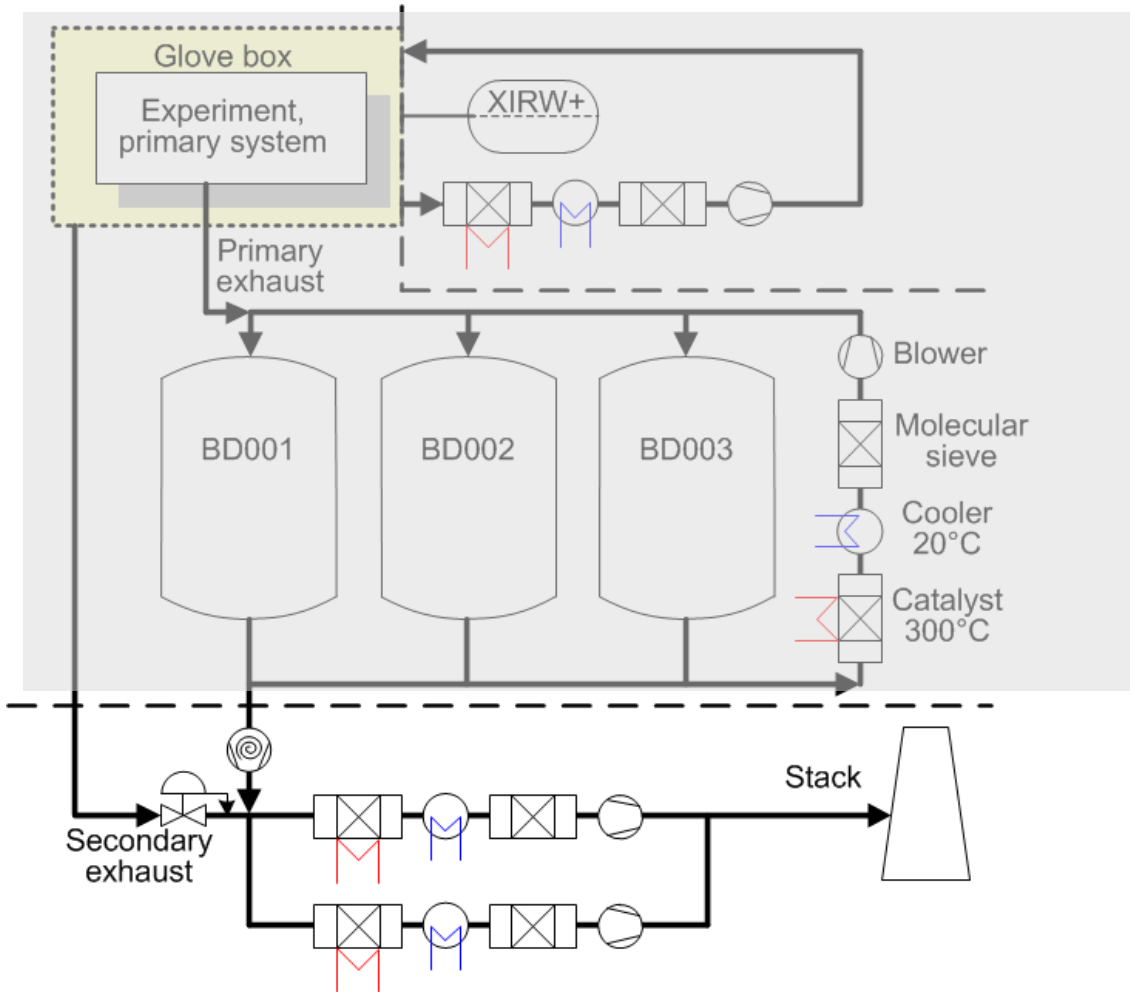

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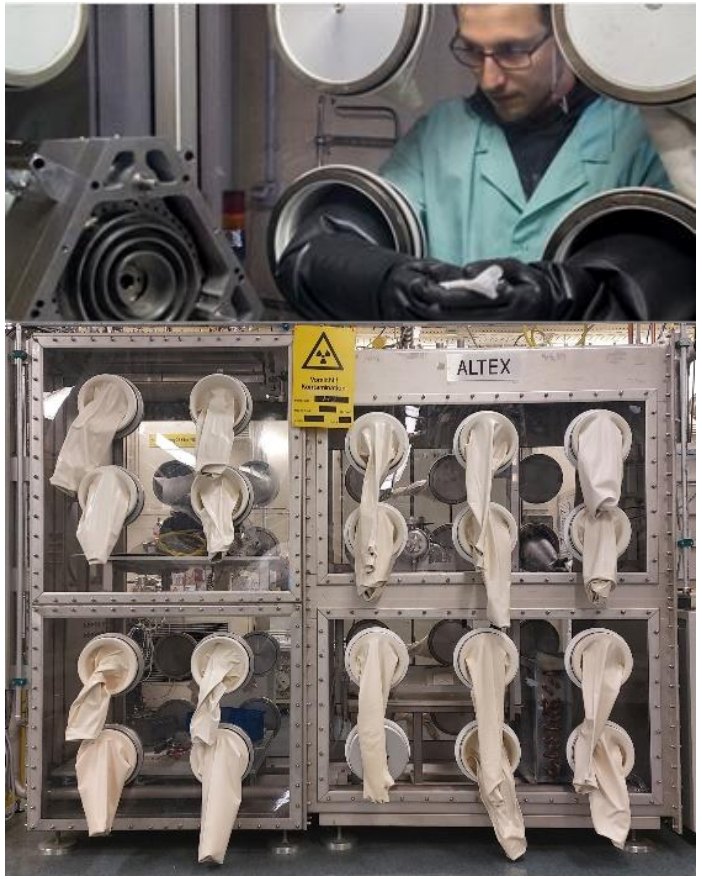
2. Containment: Glove Box



Glove box working conditions

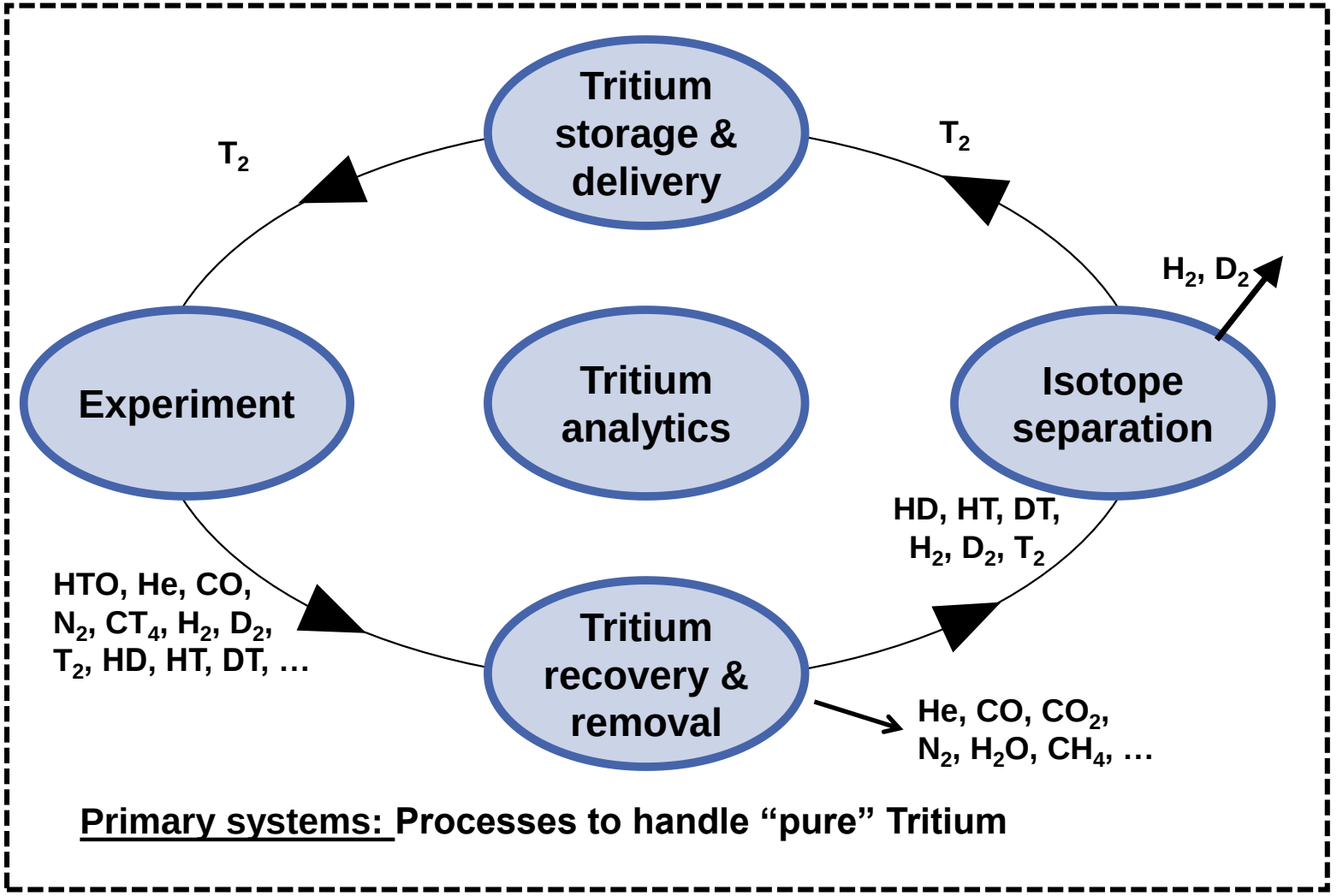
- Below atmospheric pressure control at -3 mbar to -6 mbar
- Leak rate 0.1 $\%_{vol}$ h^{-1}
- Geometry as standardised as possible
- Smoke detectors
- Redundant activity measurement (IC)
- Humidity measurement
- Oxygen control (optional)
- Temperature monitoring
- Mostly standardised components
- Constant monitoring

M. Braun Inertgas-Systeme GmbH
Dieselstraße 31, 85748 Garching

GS GLOVEBOX Systemtechnik GmbH
Daimlerstraße 29-31, 76316 Malsch

Working safe, with a maximum of accessibility & flexibility on potentially highly contaminated components

Tritium Processing: Basics



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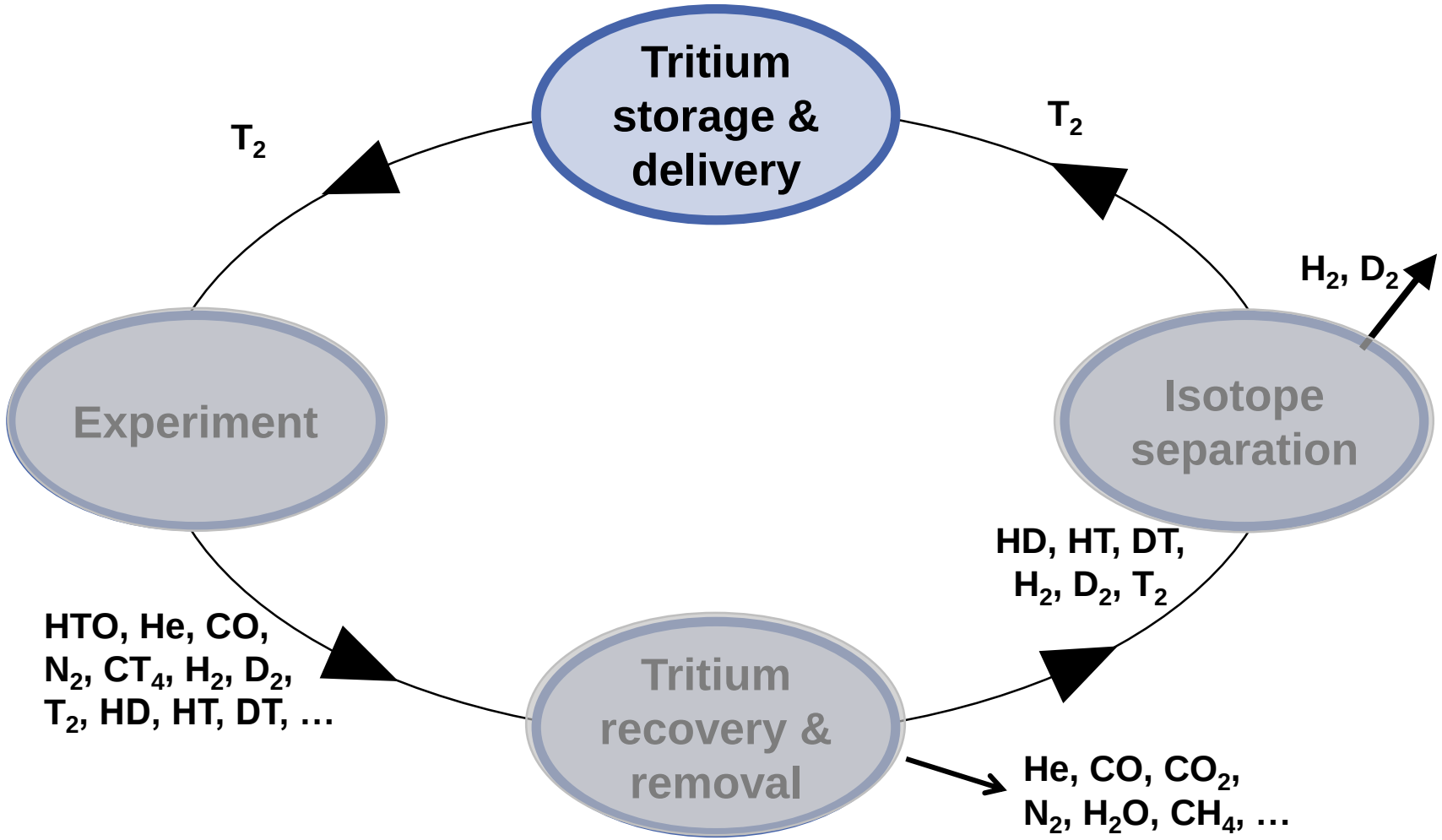
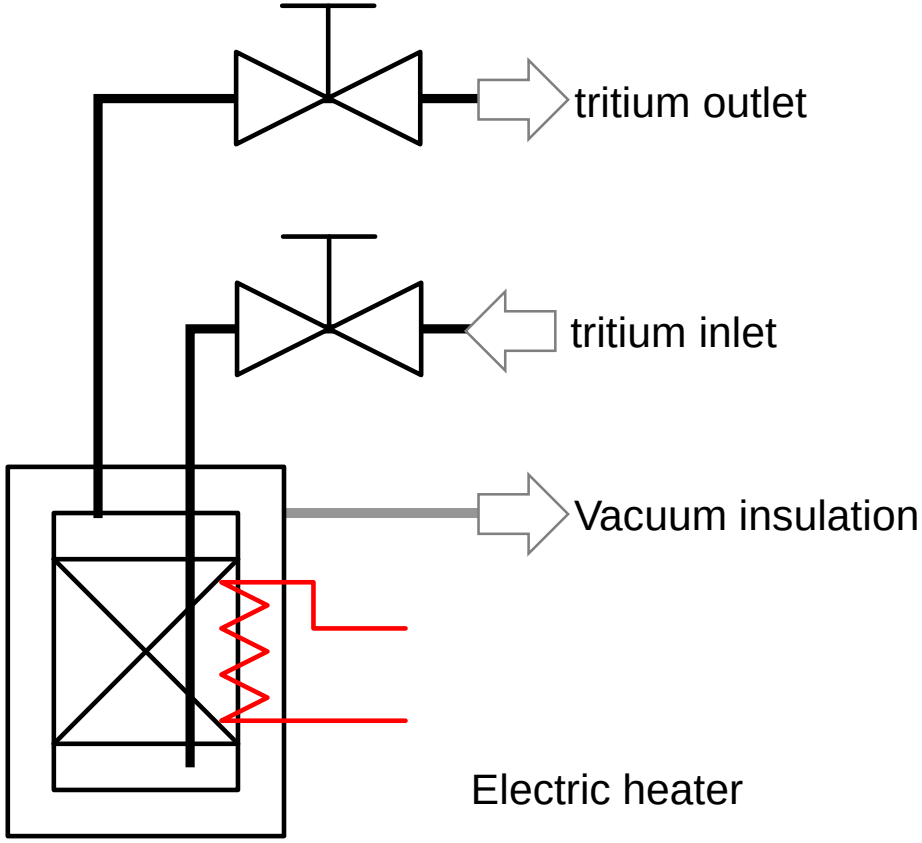
MHG Anlagentechnik GmbH

Implementation of Closed Loop Philosophy provides efficient tritium handling

Tritium Processing:



Tritium Processing: T_2 Storage



Tritium storage as MeT_x:

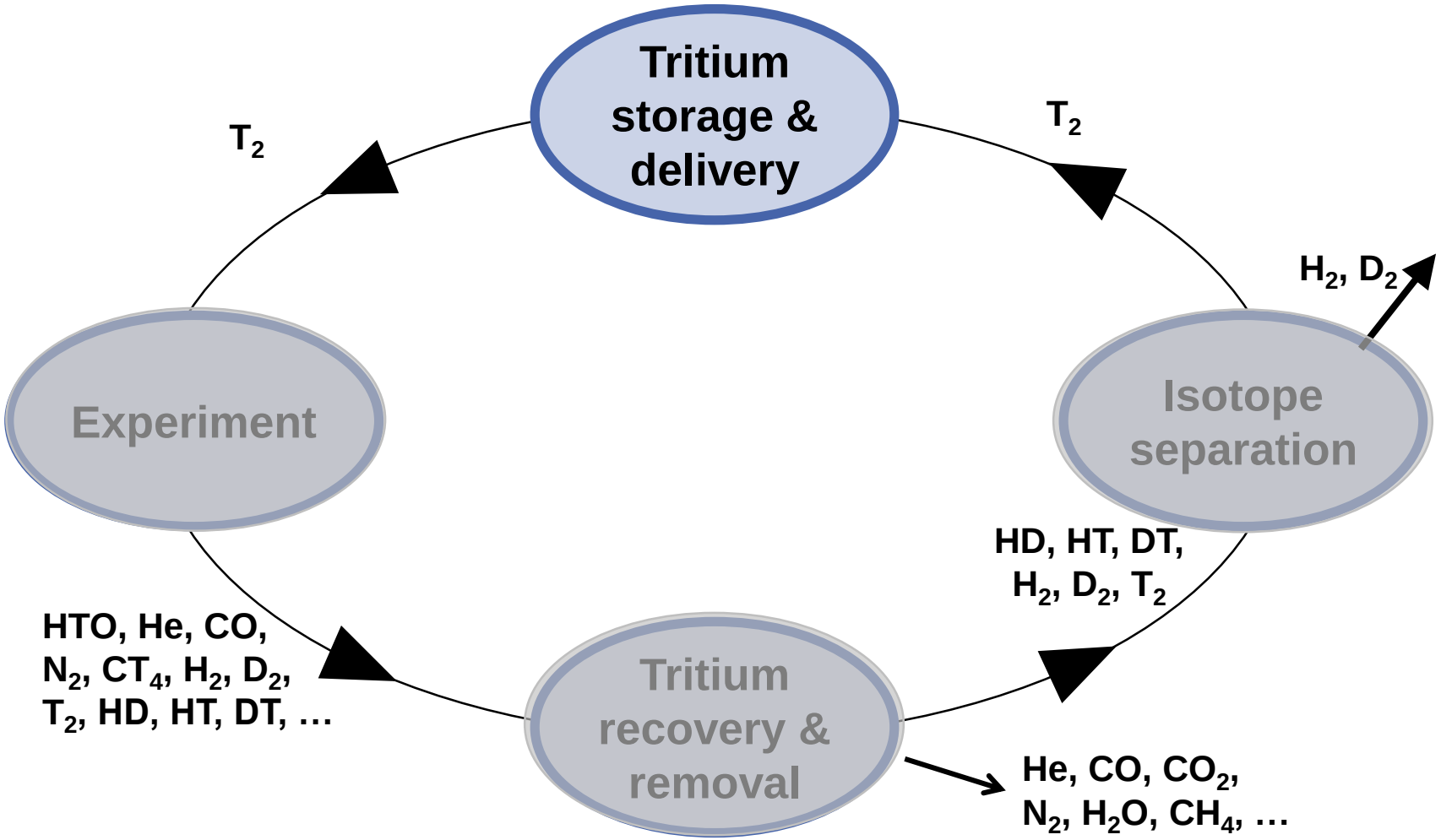
$2\ U + 3\ Q_2 \leftrightarrow 2\ UQ_3$

- 9 uranium beds
- 1 ZrCo bed
- ½ mol capacity / bed

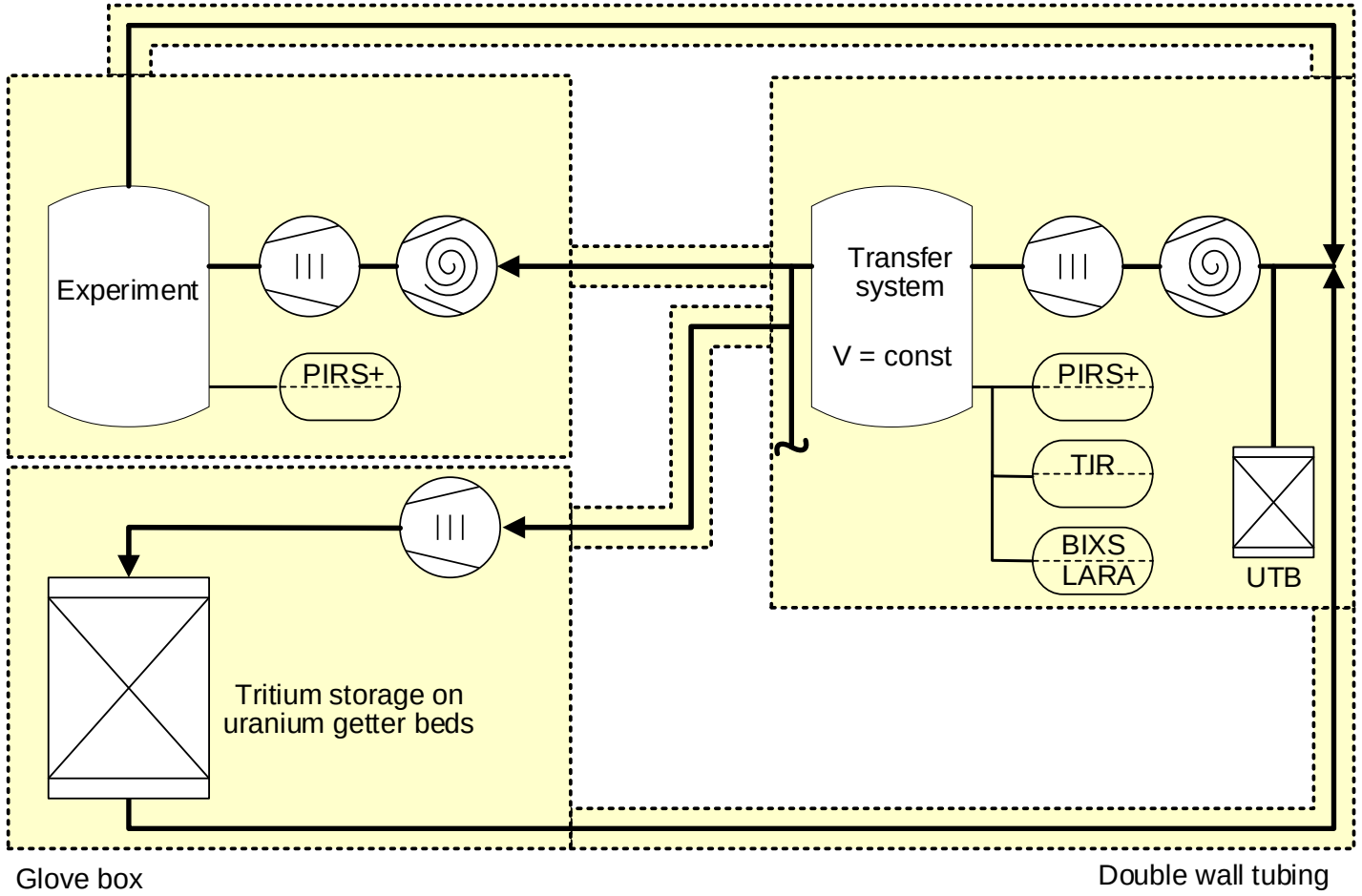
$Q_2 = T_2$, DT, HT,
D_2, HD, H_2

Secure storage of tritium in the solid phase

Tritium Processing:



Tritium Processing: T_2 Transfer System & accountancy

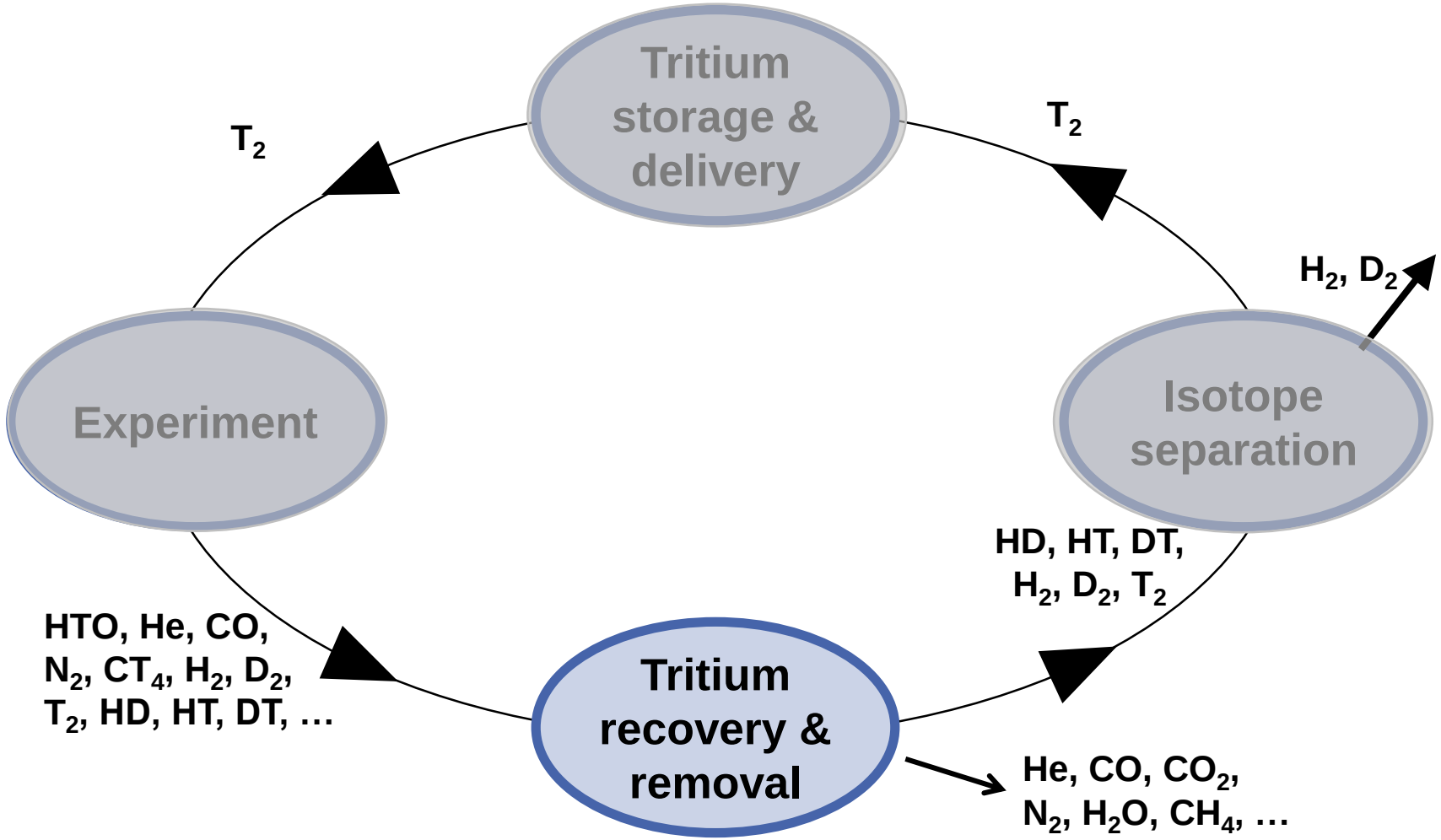


Tritium transfer system:

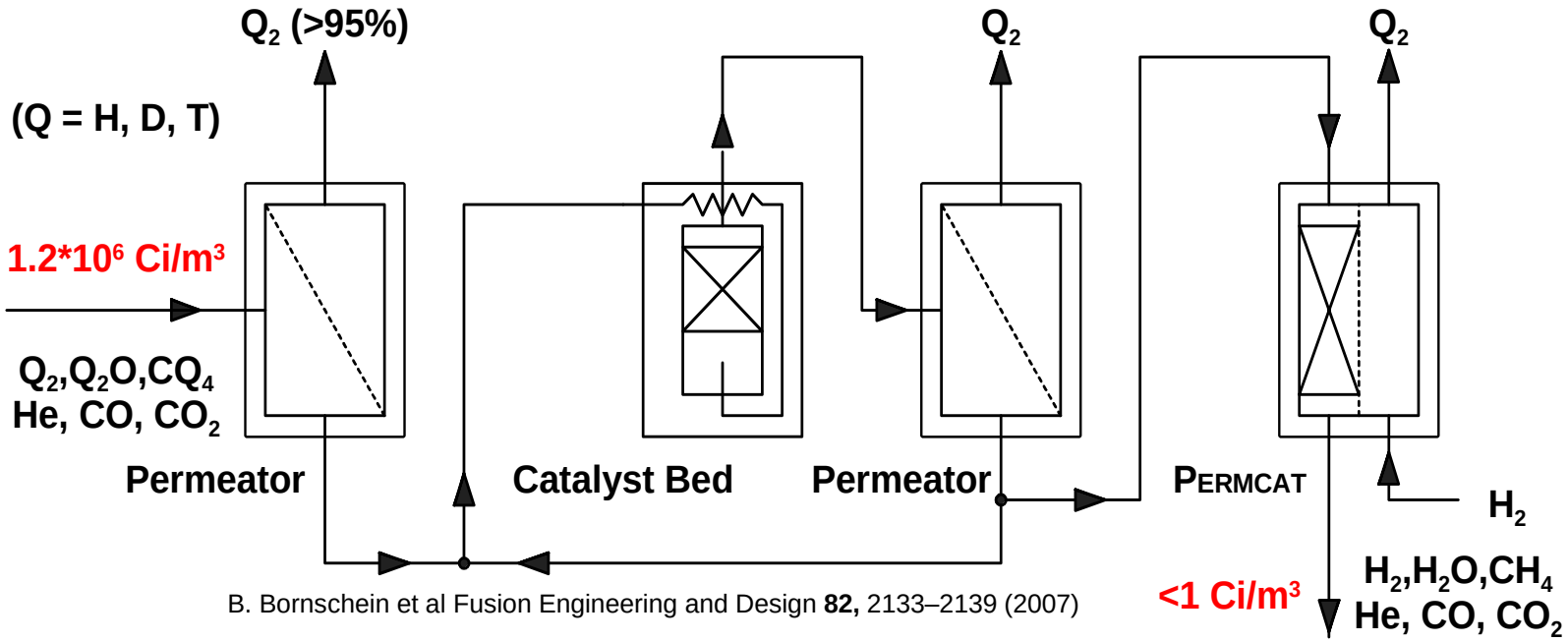
- Distribution hub for tritium from storage & external supply
- Accepting tritium from external (UTB)
- Precise measurement of volume
- Precise measurement of pressure
- Precise measurement of temperature
- → pVT for determination of gas amount
- LARA, BIXS & calorimeter for analysis
- Tritium accountancy

Secure distribution of tritium and reliable accountancy

Tritium Processing:



Tritium Processing: Tritium Purification in the CAPER System



B. Bornschein et al Fusion Engineering and Design **82,** 2133–2139 (2007)

3 stage process having DF > 10^5

- Permeator: Quantitative removal of Q_2 trough Pd/Ag membrane
- Liberation of Q_2 from molecules (shifting equilibrium by recovery through Pd/Ag membrane)
- Counter current isotopic swamping: Simultaneous isotopic exchange and Q_2 recovery

CAPER: CAtalytic PuRifiCation Experiment / PERMCAT; T_2 recovery

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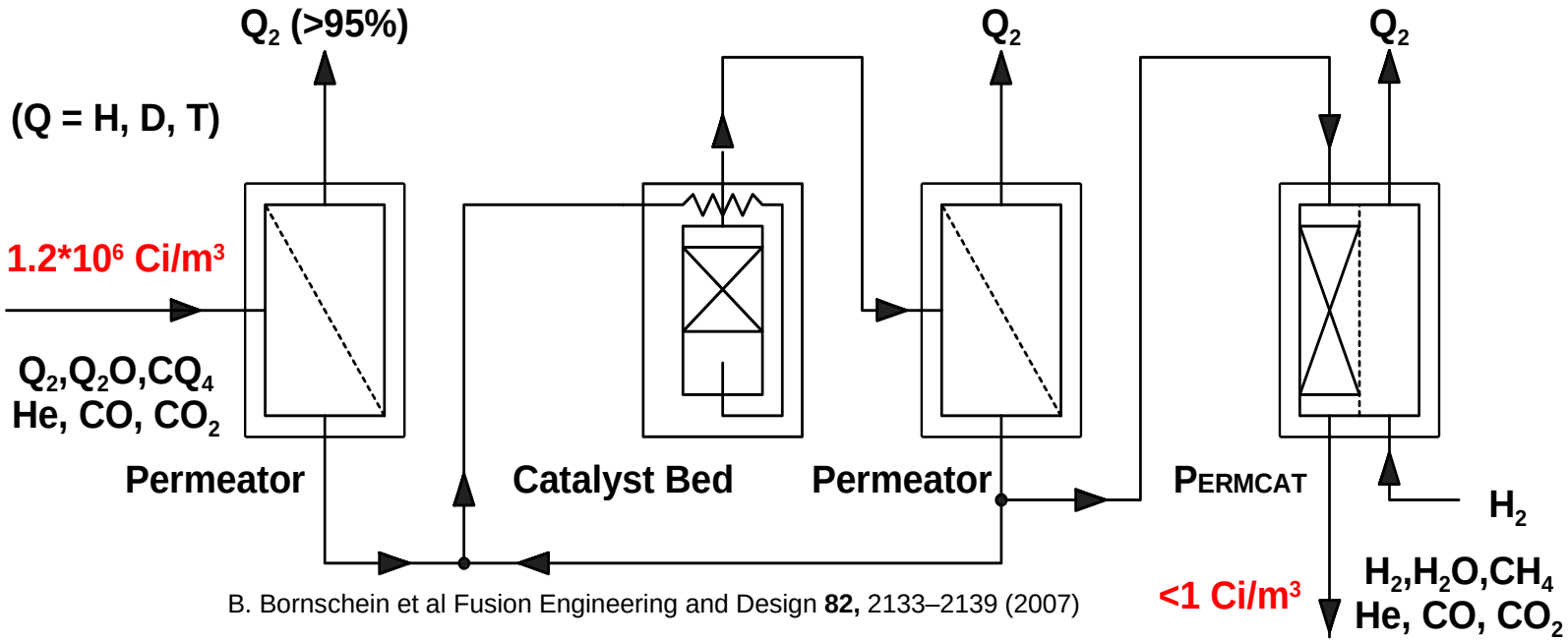

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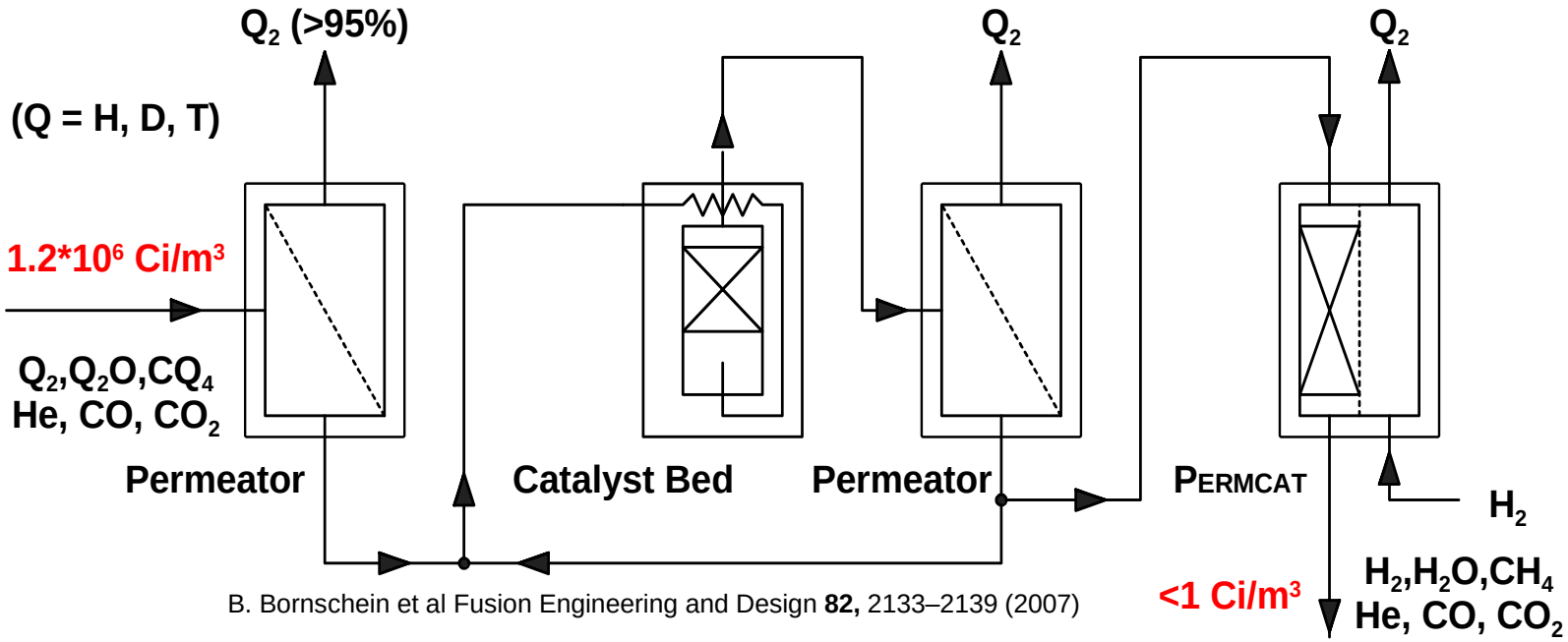

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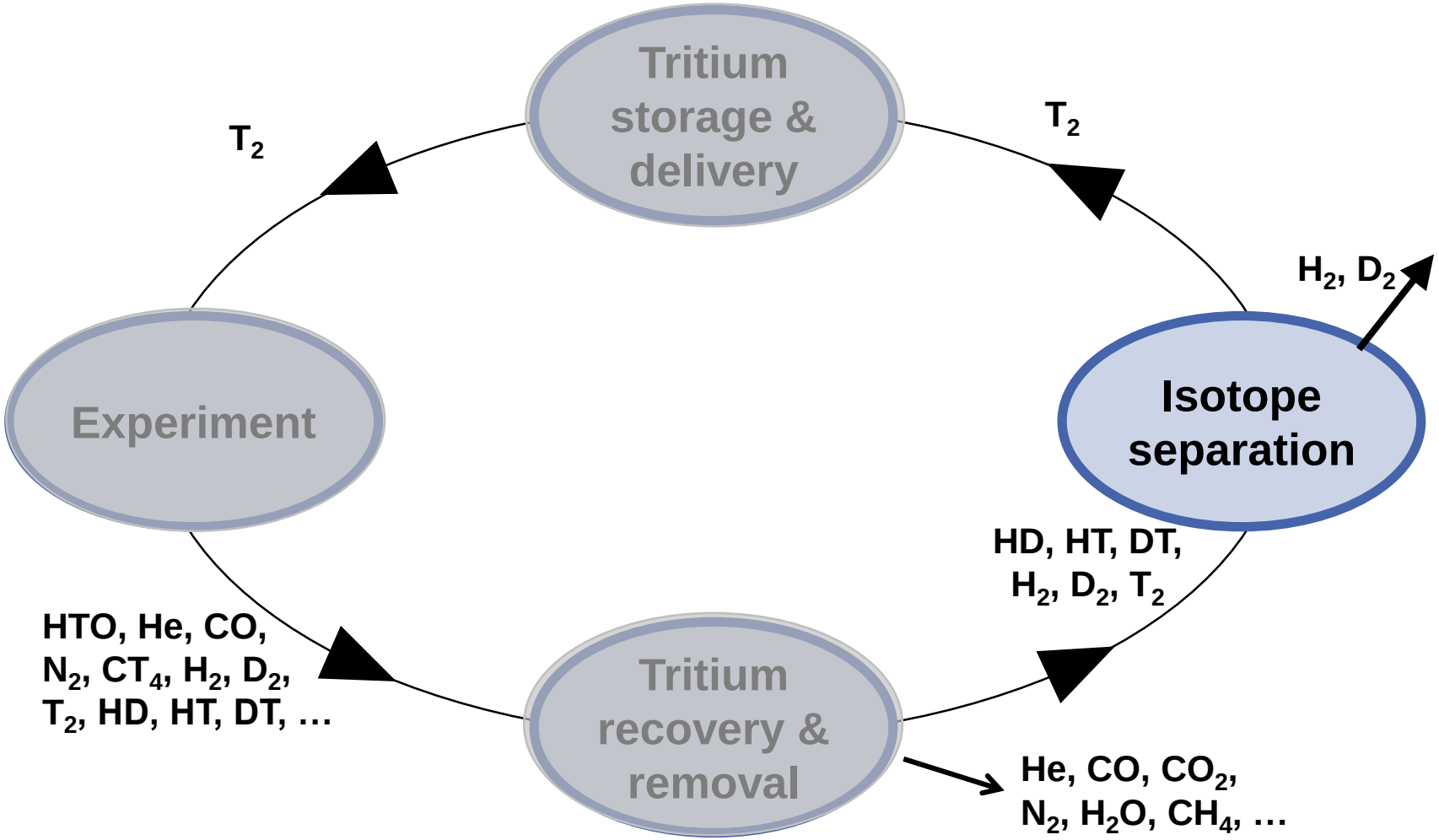
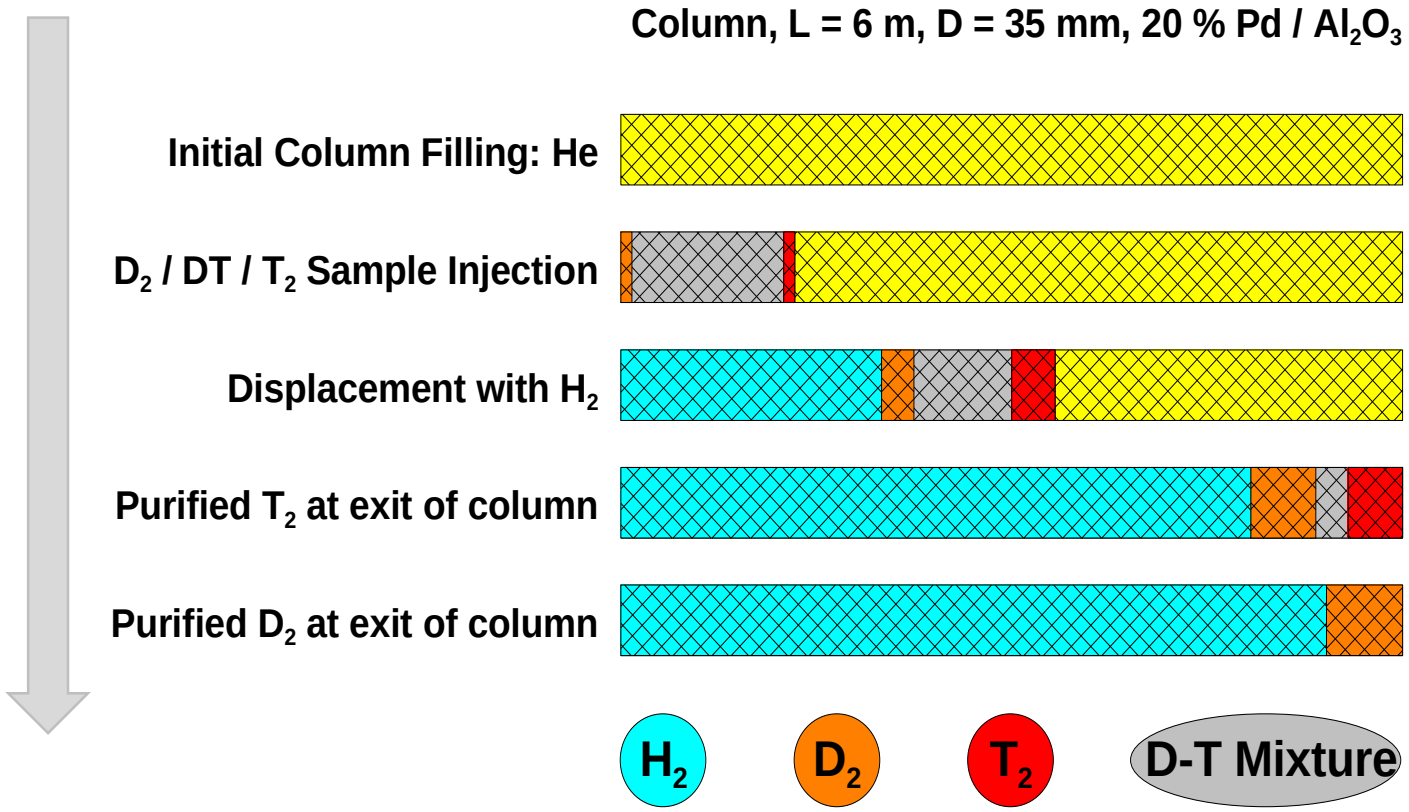
Tritium Processing: Isotope-separation



Tritium Processing: Isotope Separation System

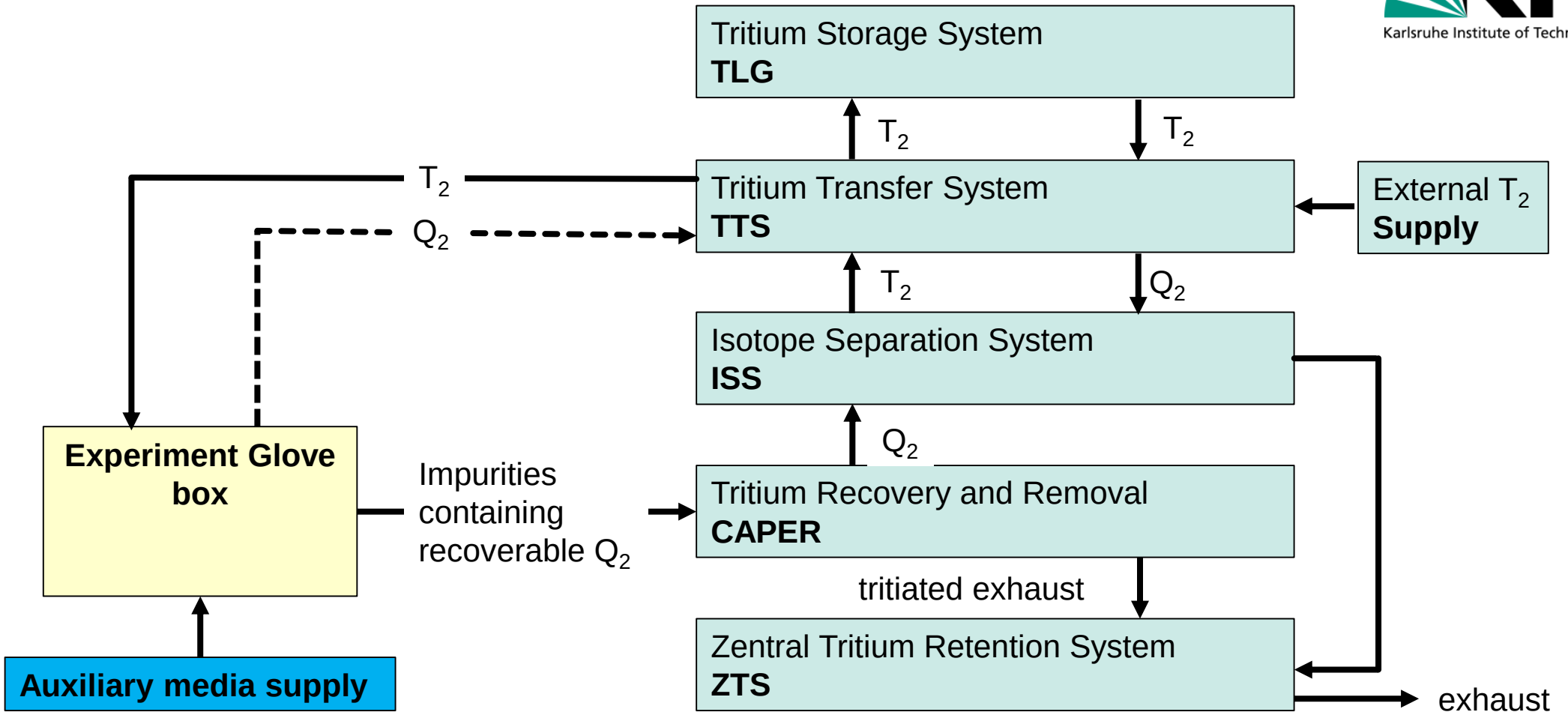


Displacement gas chromatography:

- Pd forms palladium-hydride
- Strong isotopic effect
- Lighter hydrogen isotope transferred to solid phase
- Heavier isotope remains preferably in the gas phase
- Operation at ambient conditions
- Detection of different fractions: TCD / IC

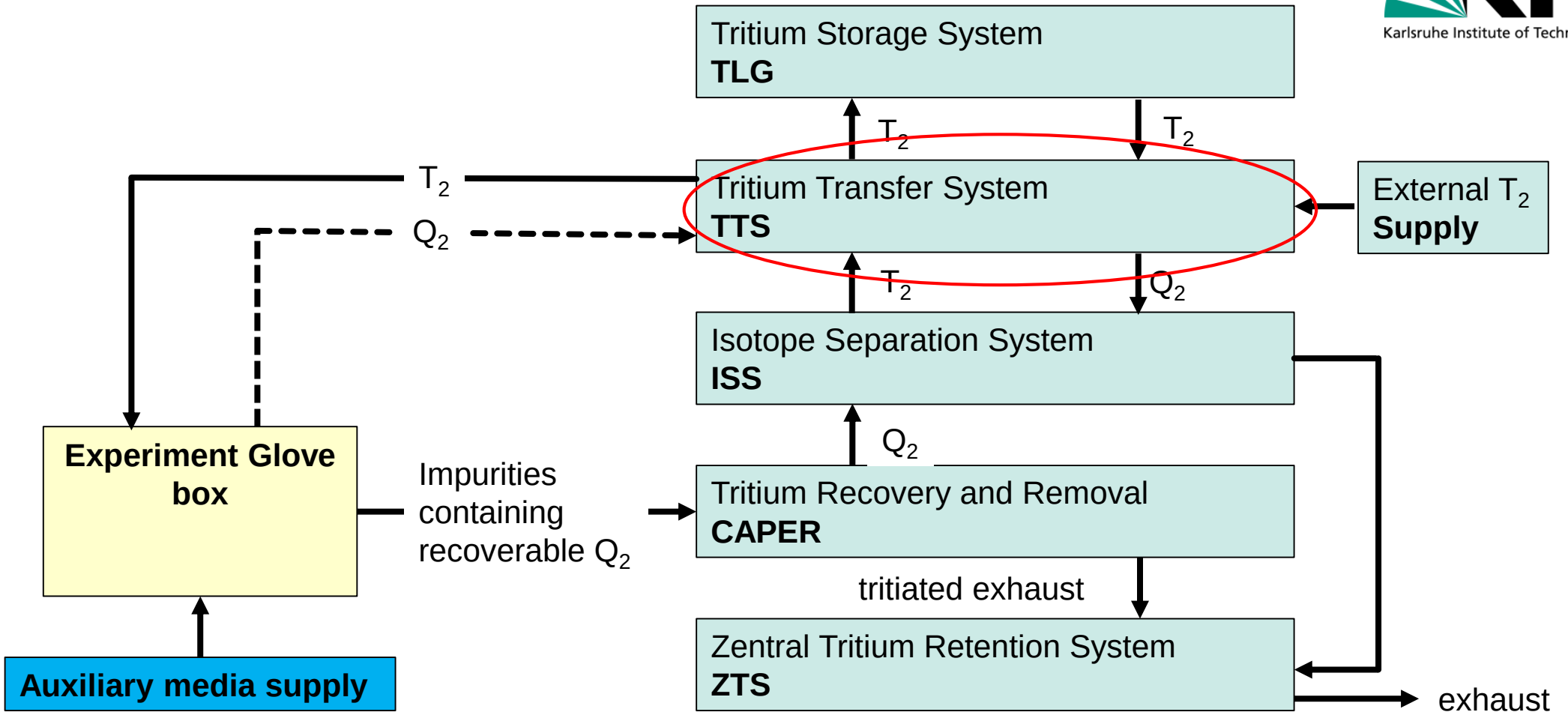
Pure tritium without interfraction

The actual TLK Tritium „LOOP“



Implementation of Tritium Loop: TTS is the “Kraken” distributing tritium with many arms

The actual TLK Tritium „LOOP“



Implementation of Tritium Loop: TTS is the “Kraken” distributing tritium with many arms

Updates in the TLK Tritium-Infrastructure over many years

Incomplete List of upgrades:

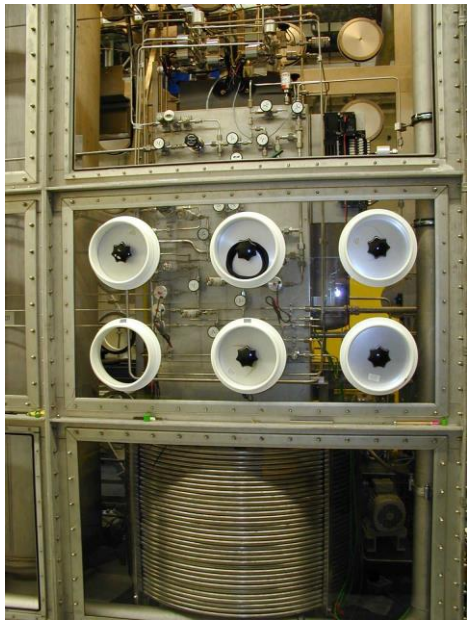
- ISS: From carrier gas chromatography to displacement gas chromatography separator
- TLG / TTS from grab sample to LARA to µRA & BIXS
- ISS-DGC from grab sample to in line measurement
- CAPER from experiment to infrastructure
- LARA / BIXS / µRA: From science to tool of daily use

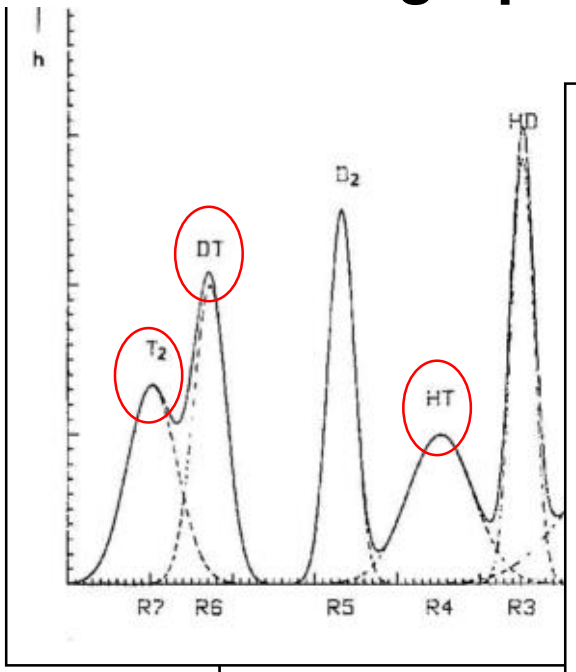
Future Infrastructure upgrades being set up:

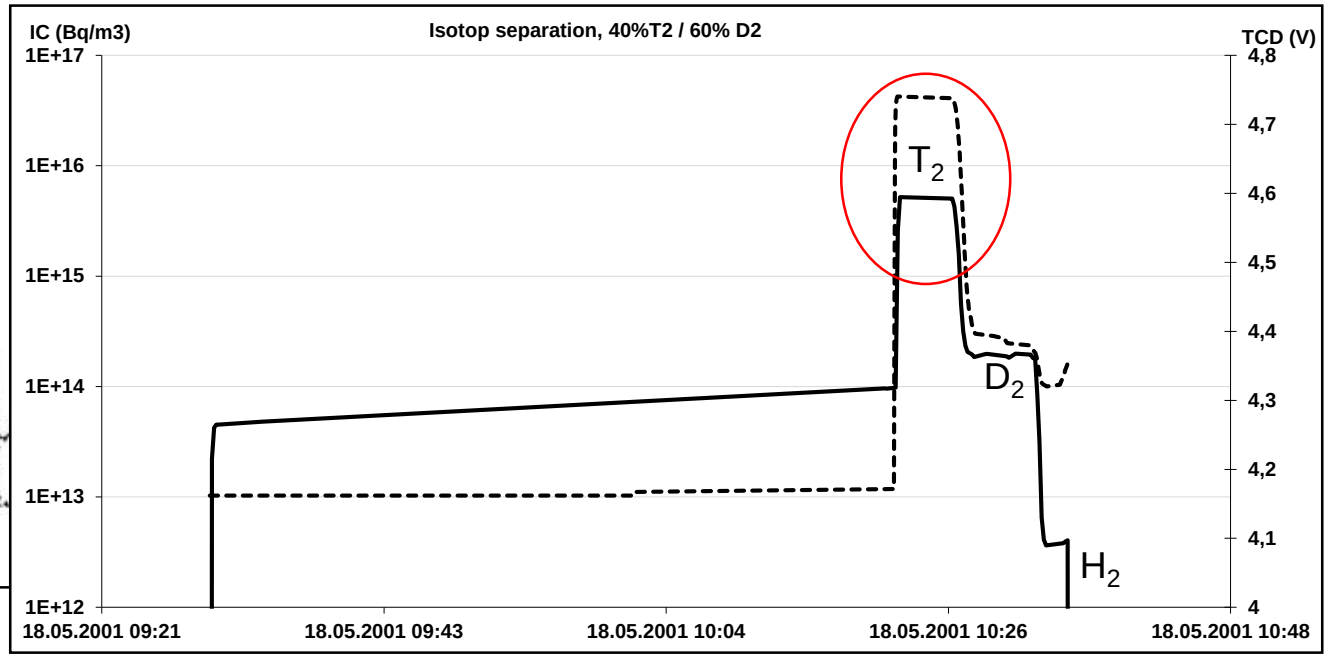
- Purpose build isotope recovery
- Infrastructure extension for waste handling

Implementation of experience & adaptation of infrastructure to meet new challenges

ISS / DGC: From Preparative Carrier Gas based separator to Displacement Gas Chromatography



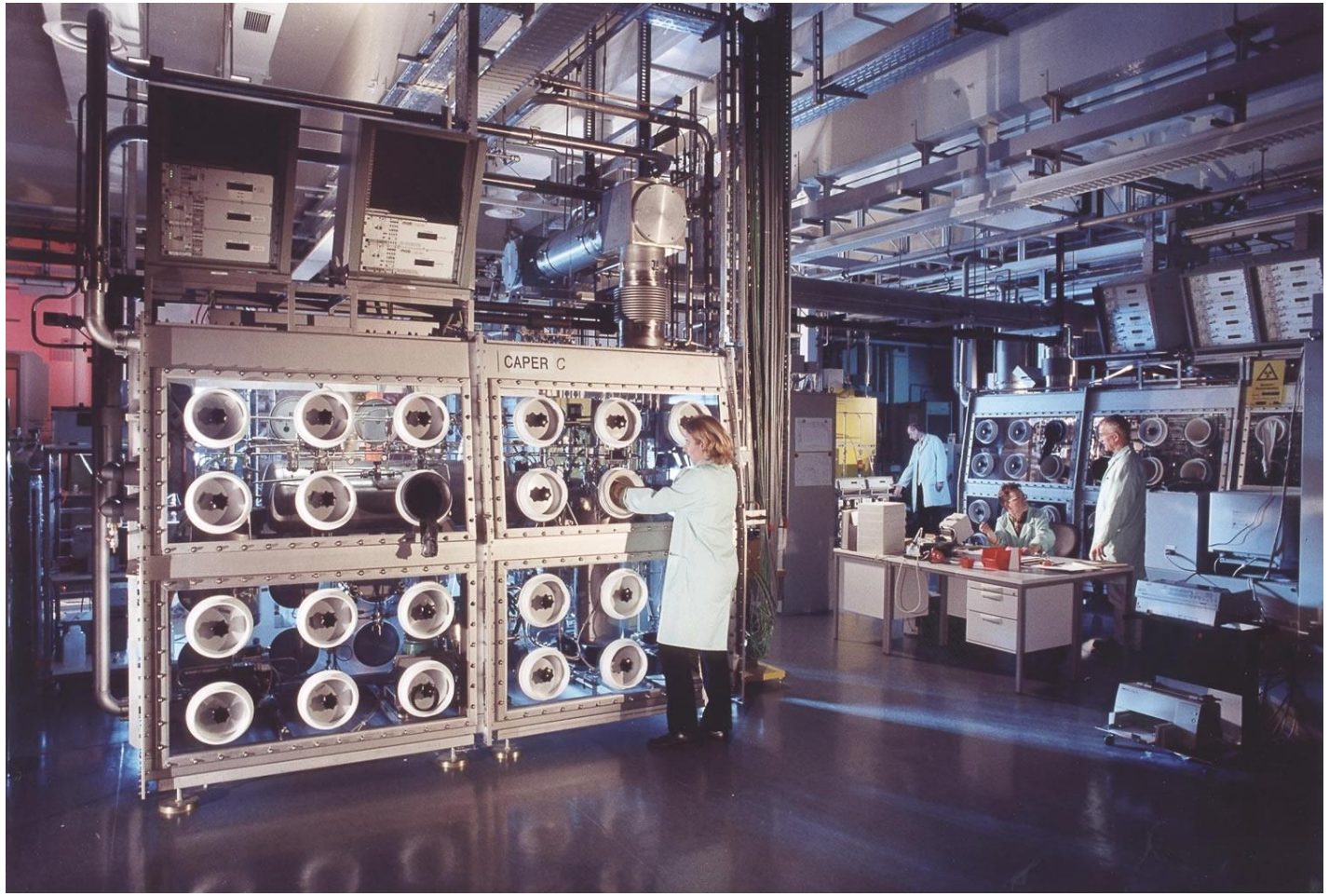




Parameter	Carrier Gas ISS	DGC
Batch size	≈1.3 mol	≈1.3 mol
Operation Temp.	LN_2	ambient
Operation Pressure	4 bar	0.9- 1.4 bar
Time / Batch	>33 h	4 h
Product Fractions	H_2, HD, HT, D_2, DT, T_2	H_2, D_2, T_2
Additional Media	He carrier-gas LN2-coolant	H_2 eluent He purge gas

- **Faster operation cycles**
- **Better separation**
- **Higher gas quality**
- **Less demanding conditions**

CAPER Facility: From Experiment to daily Service Provider



From experiment to a daily service provider:

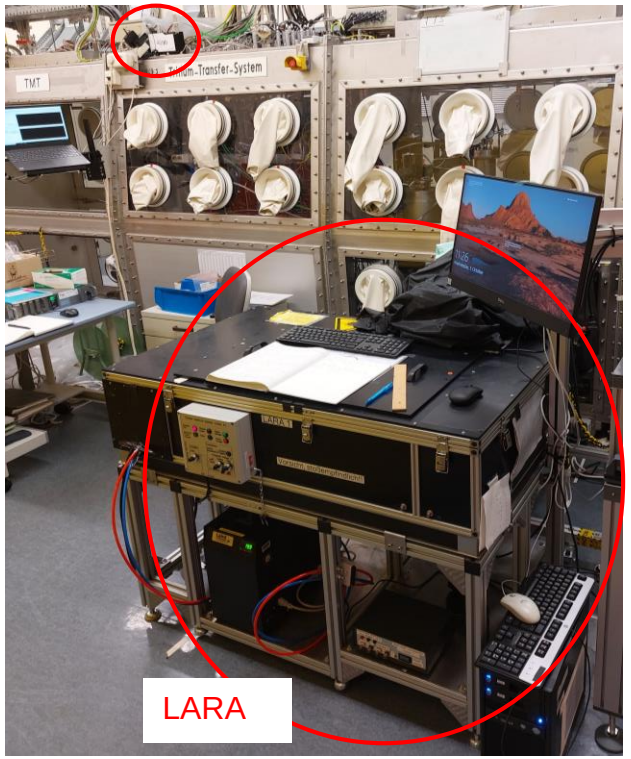
- On demand isotope recovery
- Filter between experiments & ISS
- Still available for experiments other then KATRIN
- Providing training for operators
- Analytical section updated
- Glove box space needs to be optimized
- Piping needs to be optimized

Valuable facility for lab operation, science and expanding operational experience

Updates in the TLK Tritium-Infrastructure: From Grab Sampling to Online Measurement in TTS, ISS/DGC, CAPER

Advantages:

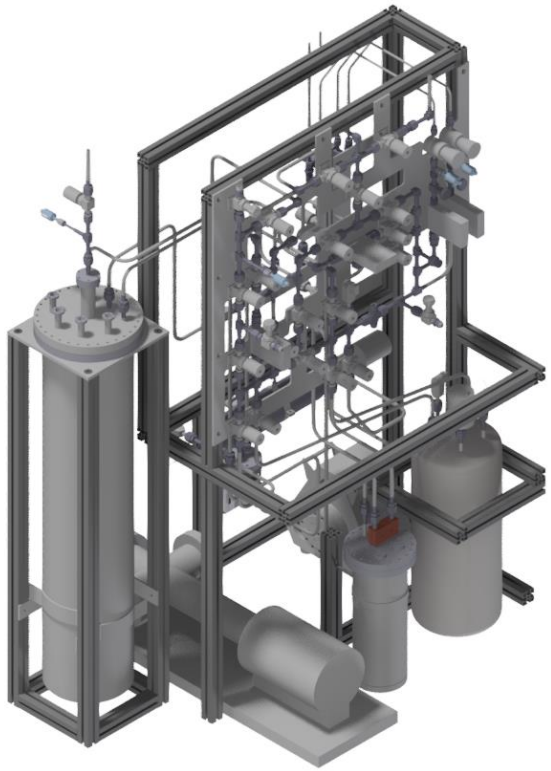
- Information online and nearly immediately in the different processes
- Less sampling time
- No tritium loss from sampling
- No samples moved between boxes, no risk of contamination
- Easy to operate

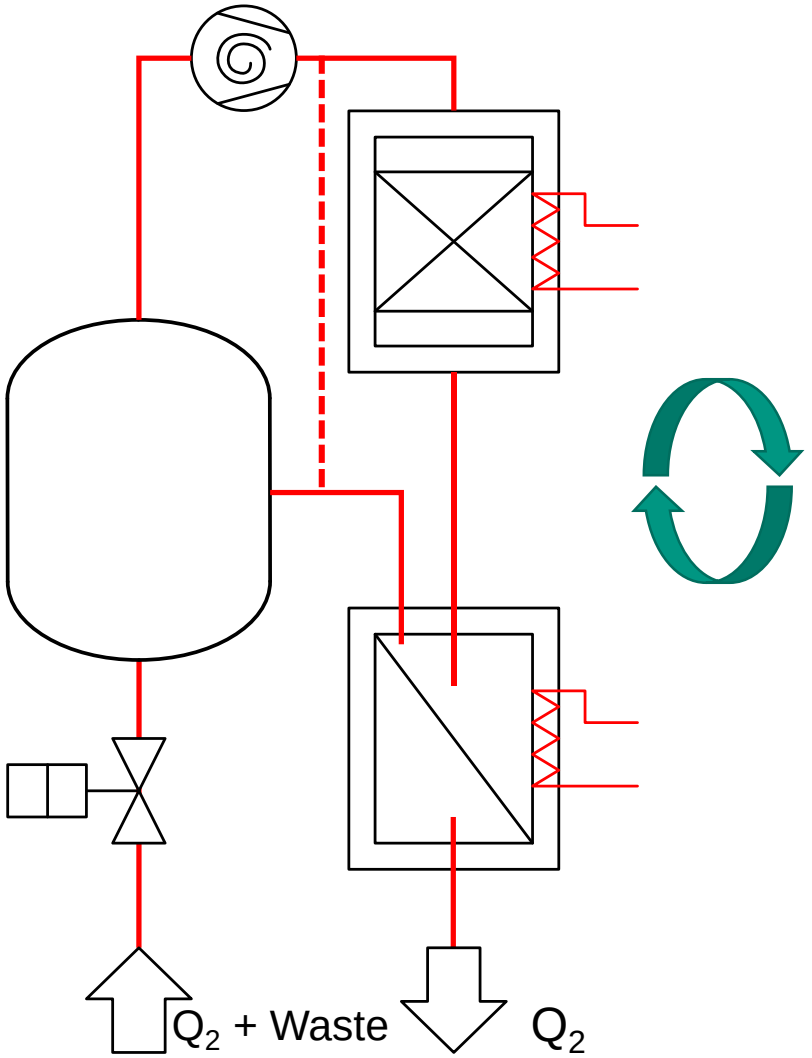


Development with industry of a tritium compatible interface flange, now commercially available

Enabling operator decision making during tritium processing regarding next process steps

Infrastructure development: Down scaled, purpose build Isotope Recovery

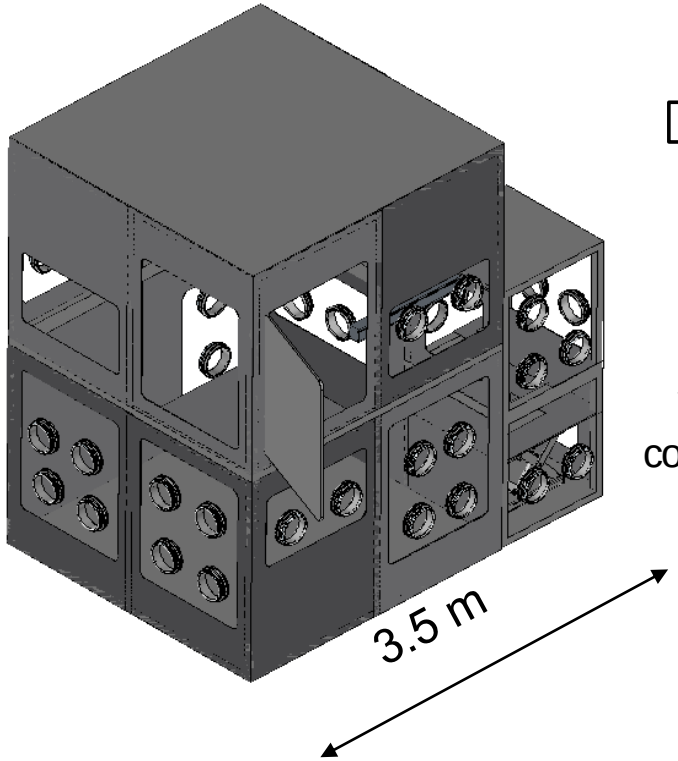


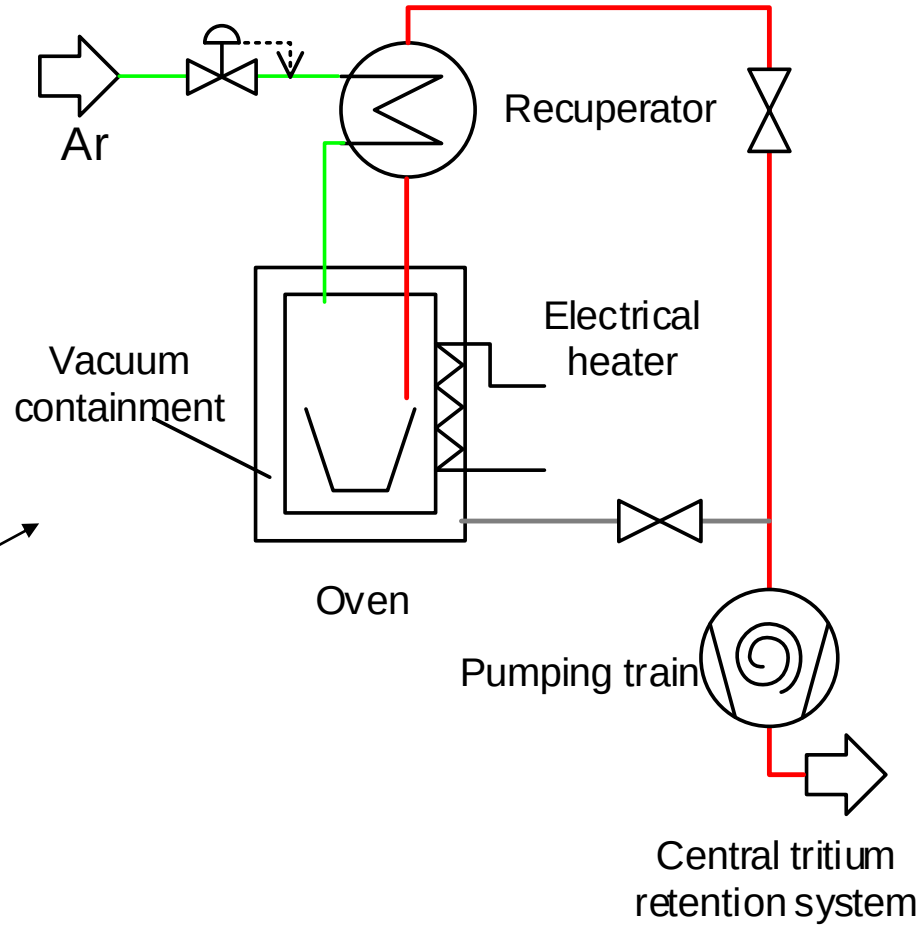


- Efficient facility to separate hydrogen isotopes from waste
- Reduction of components by ≈70% to previous facility
- Easier to maintain
- Simpler to operate
- CAPER facility free to address scientific tasks

Infrastructure development for Waste Handling: Thermal Decontamination System

Copyright @Gerolite

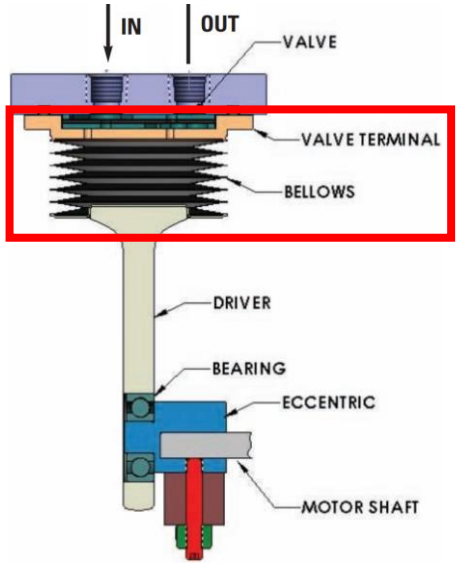




- **Decontamination of stainless steel waste > 1.9 E11 Bq**
- **Melting at 1600°C**
- **Volume reduction**
- **Disposal of waste simplified**

We break highly contaminated equipment & and have to repair it

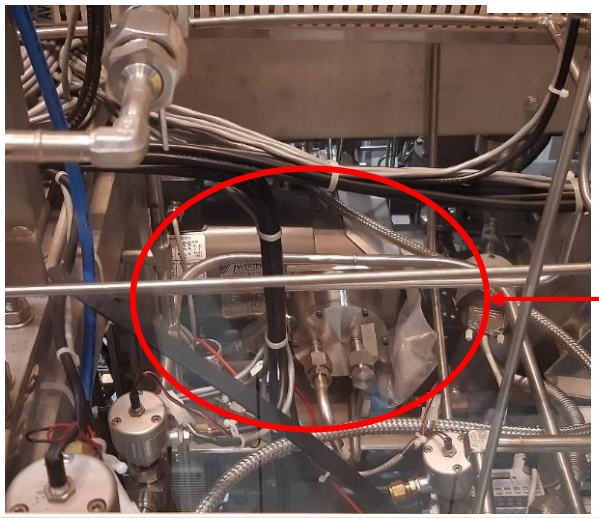
- Failing pump bellows



- Failing scroll pump

- Failing valves

- Failing measuring equipment

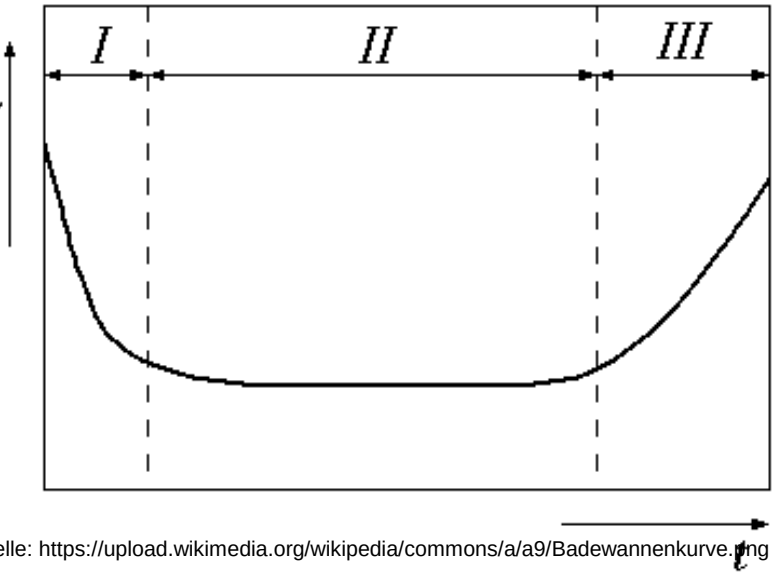


- **Specialised equipment of primary system**
- **Highly contaminated environment**
- **Access is usually not perfect**

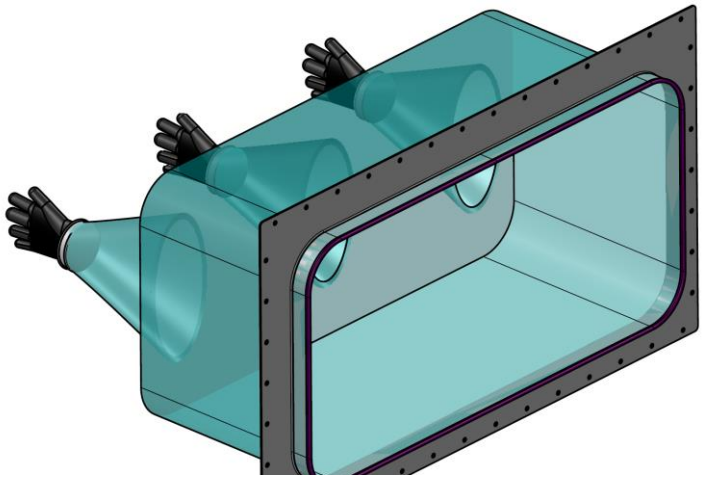
We break highly contaminated equipment & and have to repair it

The operators

Accessibility?



Quelle: https://upload.wikimedia.org/wikipedia/commons/a/a9/Badewannenkurve.png



Intervention tool

- Make reliability & accessibility part of the design
- Choosing components: Do lifetime comparison with planed experiment
- Gain experience how long tritium facing components live
- Do a thorough HD commissioning, testing reliability & accessibility
- Design intervention tools to provide accessibility
- Get a bunch of guys experienced with tritium

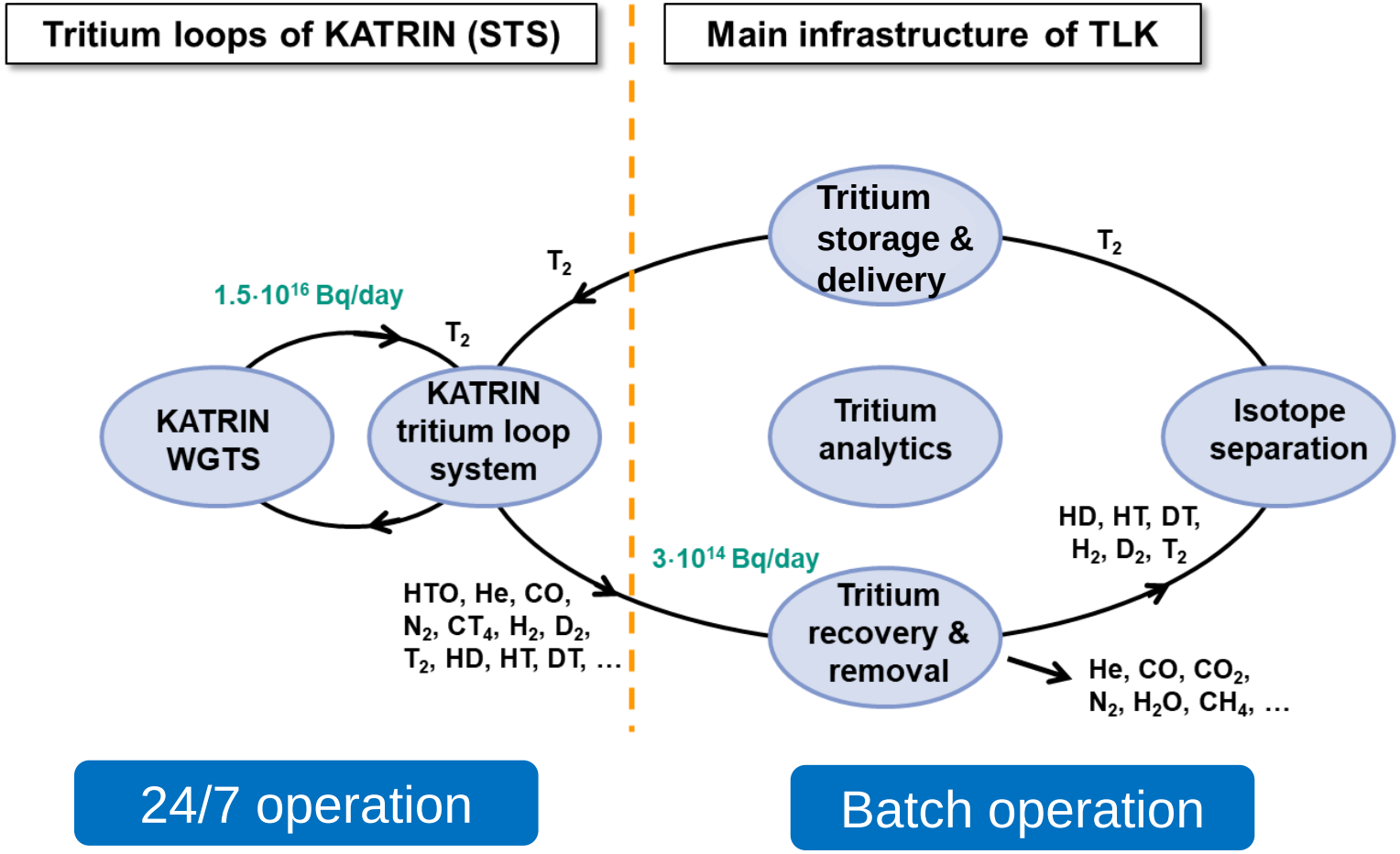
Supporting Science: Tritium Experiments supported

An incomplete list of experiments & tasks supported:

- KATRIN (Karlsruhe Tritium Neutrino Experiment)
- PETRA (Long-term permeator experiment)
- CAPER / CAPRICE (Catalytic Purification Experiment)
- HITEX (High-temperature process for recovering tritium)
- TRENTA (CECE / CD –WDS system)
- Different Membrane reactor developments: Permeators, PERMCAT, zeolite-membranes for tritium recovery)
- TRANSAT (Tritium barrier development)
- Analytics development
- Decommissioning and disposal of tritium experiments (ALTEX / OMEGATRON…)
- Atomic Tritium Source
- …

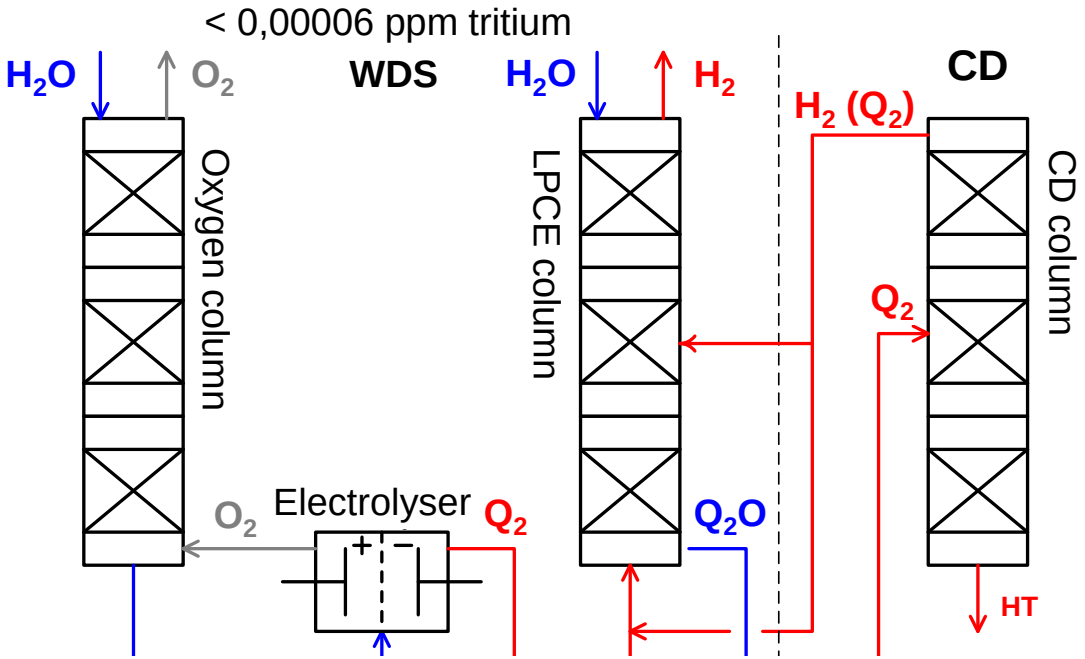
- **Support in design, planning & ensuring licencing conformity**
- **Manufacturing & assembly supervision**
- **Support in preparation & performance of experiments**
- **Tritium supply, purification, enrichment & accountancy**
- **Recurring inspections**
- **Decommissioning and disposal**

Supporting Science: Status of Tritium Loops operations & performance



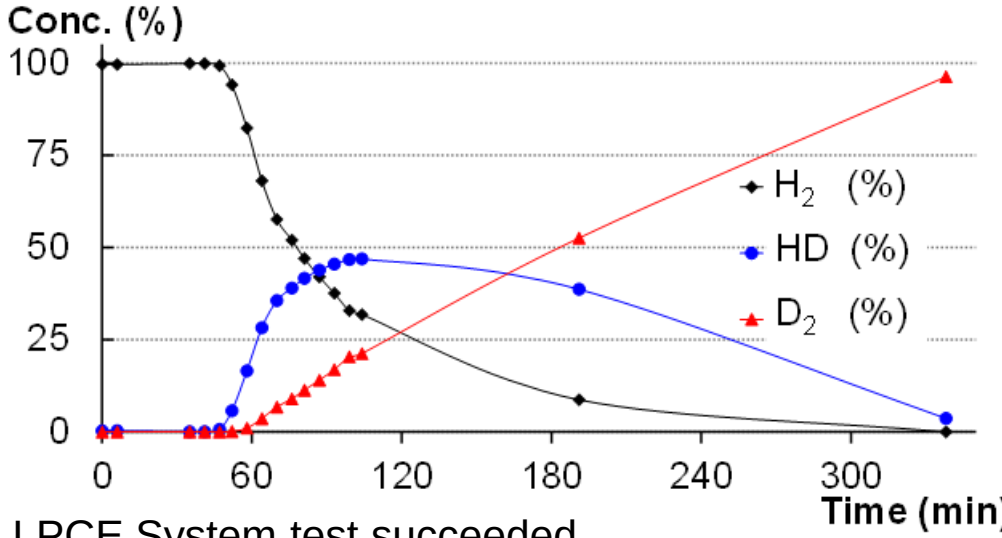
	KATRIN Tritium Loops throughput /kg	TLK infrastructure throughput /kg
2019	3	0.511
2020	4.7	0.367
2021	7.8	0.483
2022	2.6	0.261
2023	4.15	0.233
Σ	22.25	1.86

Supporting Science: TRENTA CECE WDS system

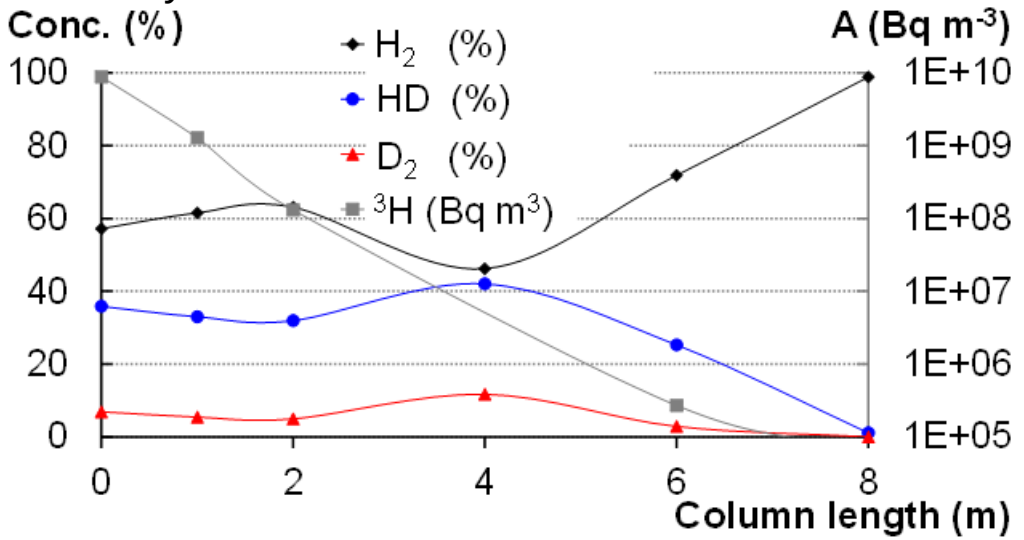


- Building LPCE column and facility
- Modifying electrolyser to be tritium compatible
- Building CD column and system
- Running experiments
- Demonstrating combined operation of LPCE and CD system
- System performs as expected in combined operation

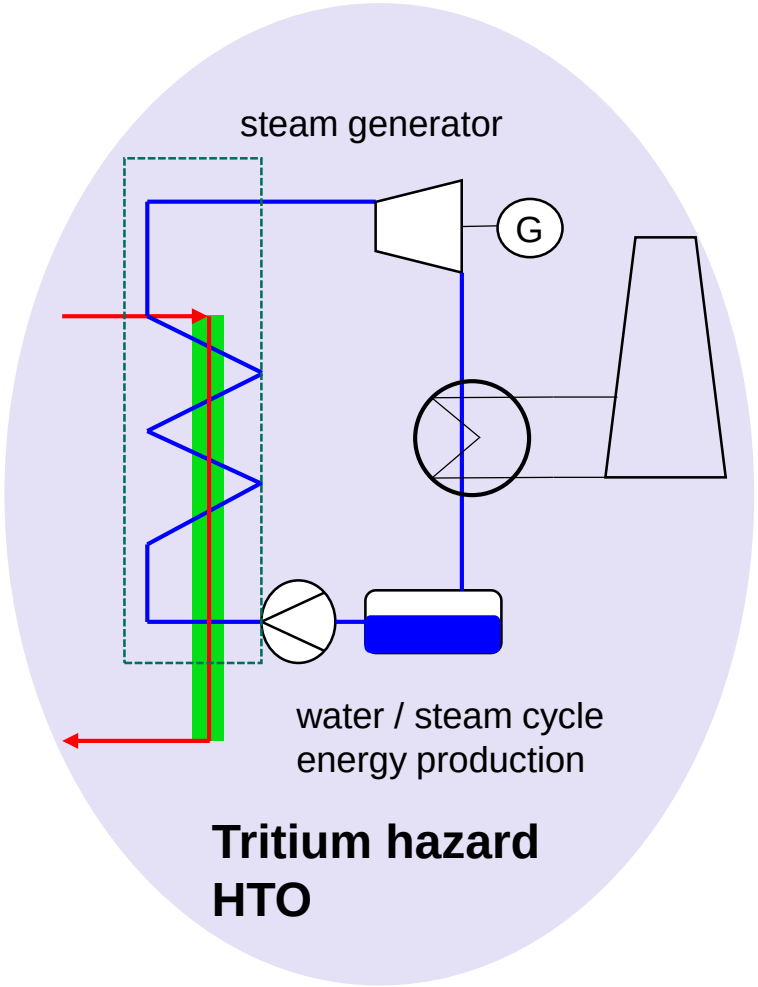
CD System test succeeded



LPCE System test succeeded

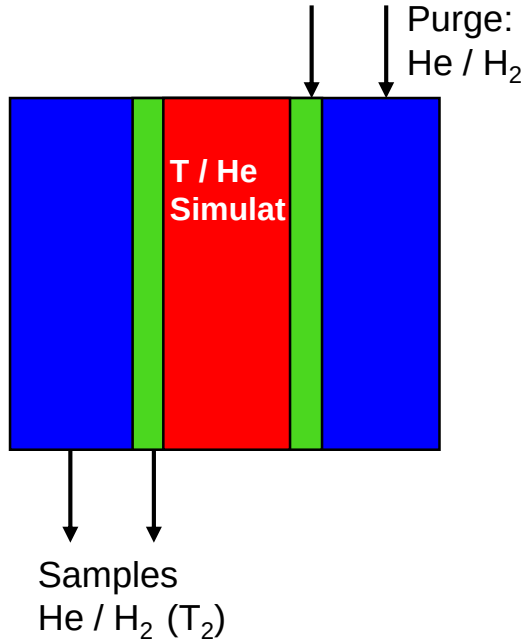


Supporting Science: Tritium Permeation Barrier



Tritium hazard
HTO

Central chamber = Tritium sample
Purged gap = Barrier
Jacket = Goal: Tritium mitigation



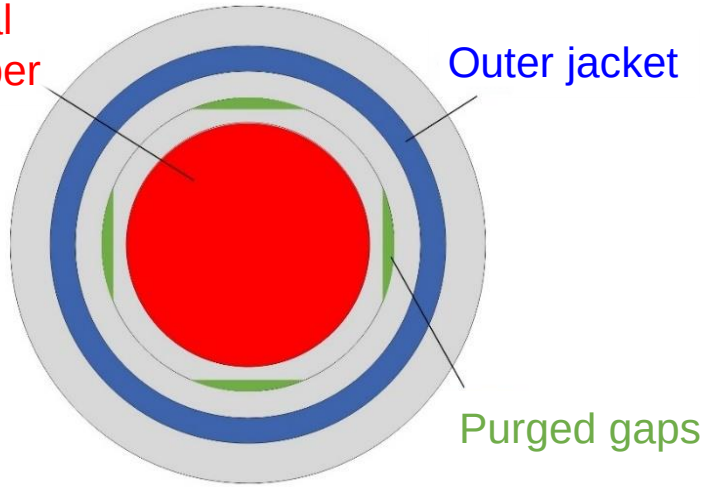
Proof of principal for active barrier

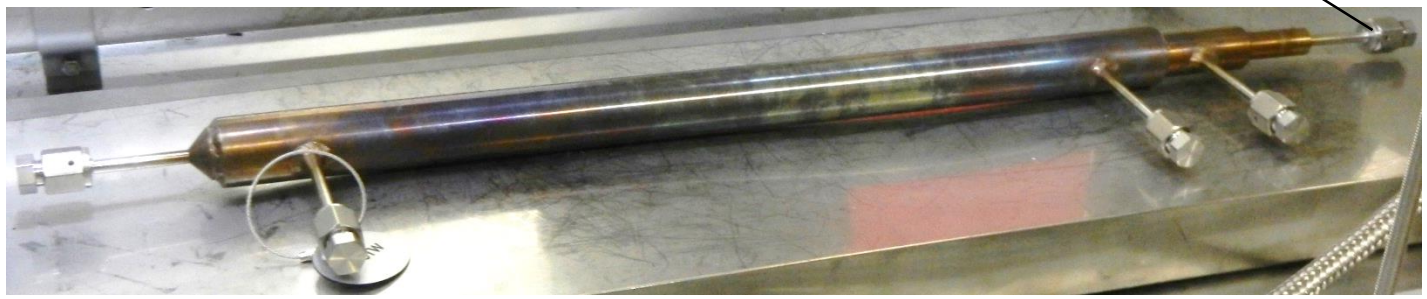
Basic experimental parameters:

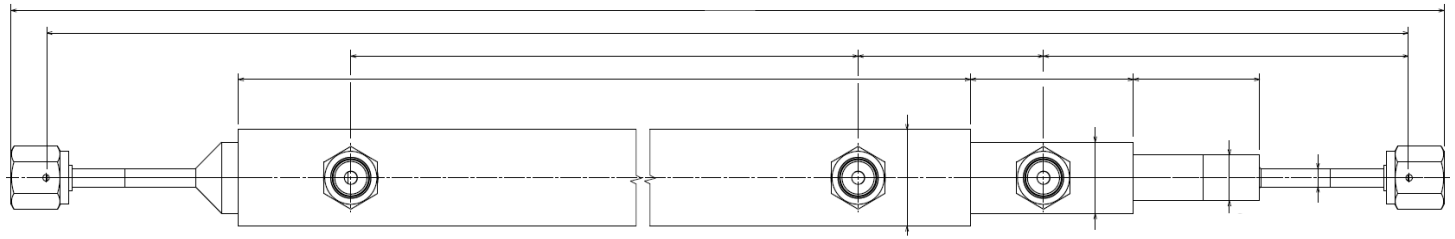
-Temperature: 450 °C
-Pressure: 70 bar
-T_2 activity: 1E13 Bq

Supporting Science: Tritium Permeation Barrier

- Triple coaxial pipe in pipe configuration
- Purged gaps remove permeating tritium (= active barrier)







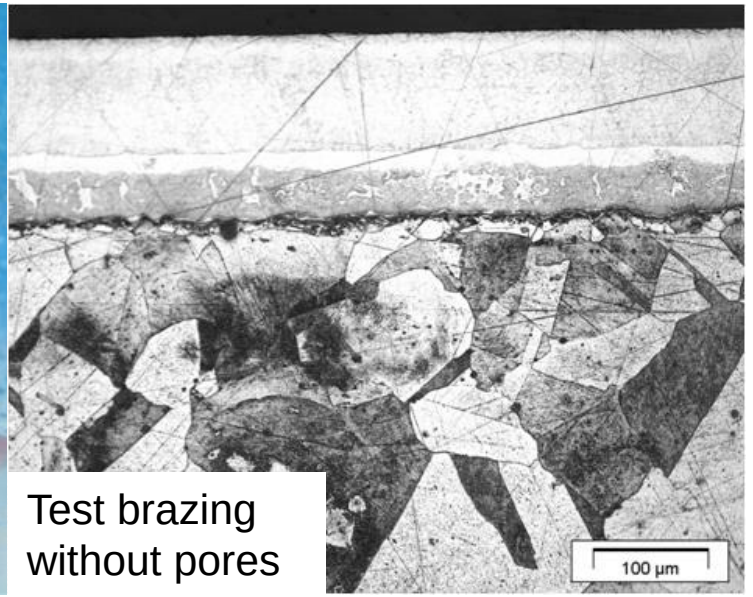
B&C Tech Solution Timisoara, Romania

- **Manufactured according to TLK technical terms of delivery and acceptance & Pressure Equipment Directive**
- **Proof of principal for active barrier**

Supporting Science: Permeator & PERMCAT Developement

Brazing cup

Membrane bundle



Test brazing without pores

- **Designing and building different evolutions types of reactors**
- **Experimental validation of design & manufacturing methods for different applications**

30 Years of TLK: Tritium-infrastructure Summary

- Gained lots of experience in operation
- We train and maintain experienced operators, engineers and scientists
- TLK developed a solid and comprehensive concept for administration and operation
- A solid framework of administrative & technical rules gives lots of room for operation and also making mistakes without serious consequences
- The barrier concept has been validated many times, providing both safety and flexibility
- The TLK tritium loop concept has been validated many times, providing both safety, efficiency and flexibility
- Standardisation in as many sectors of tritium operation, specialised solution when necessary
- We have a modular concept to be adaptable

TLK can serve as a blueprint for tritium / isotope handling facilities

30 Years of TLK: Thank you very much

- To all the colleagues at TLK
- To reliable Industry