TRITIUM TECHNOLOGIES FOR FUSION RESEARCH

NATIONAL R&D INSTITUTE FOR CRYOGENICS AND ISOTOPES TECHNOLOGIES
ICIT RM. VALCEA
Research activities/facilities for Fusion Technologies at ICIT Rm. Valcea

At the Institute of Cryogenics & Isotope Technologies, Rm. Valcea research concerning tritium separation is underway. In the last few years, the research activities for water detritiation were dedicated to develop the technology for heavy water detritiation by catalytic isotope exchange between heavy water and deuterium and cryogenic distillation of hydrogen and the experiments was mostly performed in the “Experimental Pilot Plant for Tritium and Deuterium Separation" facility.

**Potential combination of WDS and ISS at ITER**

- H₂ – HR – Helium refrigerator
- H₂O, HT – Condenser
- Electrolyser
- O₂ Purifier
- LPCE Column
- Boiler
- Condenser
- HR – Cryogenic distillation
- CD1, CD2 – Cryogenic distillation
- Boiler
- LCP

**ICIT PILOT PLANT**

Catalytic isotopic exchange tritiated heavy water – deuterium, followed by cryogenic distillation

**Links between experimental modules for Tritium and Deuterium Separation Pilot Plant**

**WDS / ITER:**
- LPCE ELECTROLYSES (PERMEATION)
- CRYOGENIC DISTILLATION

**WDS / ICIT**
- LPCE PURIFICATION
- CRYOGENIC DISTILLATION
GENERAL PRESENTATION OF ICIT

MAIN ACTIVITIES:

- Equilibrium research and isotopes separation (tritium, deuterium), including industrials pilot plants level;

- Equilibrium research and gases separation process, purification technologies and highly recuperative system;

- Cryogenic installation and technologies for separation, purification and gases liquefaction (argon, helium, hydrogen, carbon dioxide, nitrogen, oxygen);
GENERAL PRESENTATION OF ICIT

MAIN ACTIVITIES:

- Technologies for heavy water separation, technical analysis and tritium analysis;
- Turbo molecular pumps, vacuum accessories, membranes compressors, cryogenic pumps;
- Heavy water etalons, depleted water;
- Risk studies for chemical and petrochemical plants;
- Static and dynamic equilibration analysis.
EURATOM PROGRAM

SEPARATION TECHNOLOGIES FOR HYDROGEN ISOTOPES. SYSTEM FOR TRITIUM EXTRACTION FROM TRITIATED HEAVY WATER

(TWO-TRIT/REM)

(2000-2001)

NATIONAL R&D INSTITUTE FOR CRYOGENICS AND ISOTOPES TECHNOLOGIES
ICIT RM. VALCEA
OBJECTIVES:

- Comparative study of the catalysts used in isotopic catalyst exchange system;
- Develop analysis method for hydrogen isotopes;
- Study the species distribution of tritium in multicomponent mixtures;
- Security architecture for tritium processing plants.
Performed Tasks

- Experimental facility "System for Tritium Removal from Water" by catalytic isotope exchange water-hydrogen and/or deuterium and cryogenic distillation of hydrogen and its isotopes;

- Software for designing and simulation the behavior of a system for catalytic isotope exchange tritiated water - hydrogen and deuterium;

- Comparison of isotope exchange catalysts manufactured at FZK Karlsruhe and ICIT Rm. Valcea;

- Comparatively determination of efficiency and pressure drop for ordered and disordered packages.
SEPARATION TECHNOLOGIES FOR HYDROGEN ISOTOPES.
SYSTEM FOR TRITIUM EXTRACTION
FROM TRITIATED HEAVY WATER
(TWO-TRIT/REM)

Experimental facility
- LPCE column
- Condenser
- Boiler
- Equilibrator H-D
OPTIMISATION OF THE RATIO PACKAGE/CATALYST FOR THE SIMULTANEOUS TRANSFER OF TRITIUM AND DEUTERIUM IN A WATER DETRITIATION FACILITY

(JW0-FT-2.1)

OBJECTIVES:

- To manufacture ordered packages with different size and catalysts with different size and composition with higher surface area for gas-vapor-liquid exchange;
- To establish the influence of deuterium transport on tritium transport when the two processes take place simultaneously;
- The model related to deuterium transport will be developed to include also the tritium transport, i.e. the transfer all three hydrogen isotopes will be followed;
- To establish the correlation between the required ratio package to catalyst in different working conditions: flow rates of gas, vapors and liquid in order to maximize the isotopic transfer for a specified height of column.
The influence of geometrical characteristics of ordered package/catalyst on the separation performances for two diameters of isotopic exchange column: 40 mm and 100 mm;

The separation performances were verified for specified working conditions of temperature, flow rates of gas, vapor and liquid, as well as the ratio of package/catalyst;

Influence of deuterium transport on tritium transport when the two processes take place simultaneously in the catalytic isotopic exchange column.
ENDURANCE TEST FOR THE CATALYST-PACKING MIXTURE PROPOSED FOR WATER DETRITIATION SYSTEM AT JET USING SCK-CEN MIXTURE

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National Institute for Physics and Nuclear Engineering (IFIN H-H) - Bucharest
**WATER DETRITIATION SYSTEM (WDS)**

* The Water Detritiation System (WDS) is based on an enrichment of the tritiated water inside a Liquid Phase Catalytic Exchange (LPCE) column, then a dissociation of oxygen and hydrogen isotopes in an electrolyser and the separation of the hydrogen isotopes inside a cryogenic distillation (CD) column and Gas Chromatography (GC)

*The main requirements for the design of a WDS for JET are:
- to process **10 tons tritiated water of ~ 1Ci.kg⁻¹** during one operation campaign of **2-3 months**,  
- to achieve a decontamination factor of **10⁴ along the stripping section of the LPCE column** to deliver a tritium content in bottom product of **CD column in the range of 0.5-1% atomic ratio** to allow further separation by the GC systems.
LIQUID PHASE CATALYTIC EXCHANGE (LPCE)

* The key point of WDS is LPCE column, based on isotopic exchange process between hydrogen and water. At ambient temperature isotope transfer is very low and need a catalyst. In practice a mixture of hydrophobic catalyst and hydrophilic packing promotes the process.

* $\text{HT} + \text{H}_2\text{O} (\text{v}) \leftrightarrow \text{H}_2 + \text{HTO(v)}$
* $\text{H}_2\text{O} (\text{v}) + \text{HTO (l)} \leftrightarrow \text{H}_2\text{O (l)} + \text{HTO (v)}$
* $\text{HT} + \text{HTO (l)} \leftrightarrow \text{H}_2 + \text{H}_2\text{O}$

* Conventional hydrophilic catalysts lose their activity in contact with liquid water and water vapor because of the low solubility of hydrogen in liquid water. Hydrophobic catalysts have high catalytic activity and high stability and could avoid such disadvantages. For this purpose, over 100 hydrophobic catalyst types have been prepared and tested.
THE GOAL OF THE PROJECT

The main purpose of project is to study:

* influence of tritium β-radiation and of impurities on SCK-CEN catalyst/packing mixture’ performances in LPCE process;
* influence of tritium β-radiation and of impurities on physico-structural parameters of materials from SCK-CEN mixed catalytic packing;
* stability in time of SCK-CEN catalytic mixed packing’ performances;
* procedure for regeneration of catalyst and the activation of SCK-CEN packing;
* erosion and mechanical resistance of SCK-CEN packing in LPCE process;
* identification of potential poisons for Pt-hydrophobic catalyst in hydrogen-water LPCE process.
EXPERIMENTAL PROTOCOL

* Endurance tests for SCK – CEN packing was performed for 3, 6 and 9 months in two modes:
(A). Static regime;
(B). Dynamic regime.

(A) STATIC REGIME:
- Immersion of 300 cm$^3$ of SCK-CEN packing in tritiated water (1Ci/l);
- Decontamination of Pt-catalyst and its physico-structural characterisation after 3, 6 and 9 months from immersion;
- Determination of physical and chemical parameters of process water before and after exposure to $\beta$-tritium radiation.
ENDURANCE TEST IN DYNAMIC REGIME

* SCK-CEN packing will equip isotopic exchange column and it will be daily exposed to (1 Ci/l) tritiated water (in closed circuit) and water vapour (see figure of experimental installation)
* Experimental conditions: temperature (40° C), atmospheric pressure, tritiaded water, natural hydrogen( 45-50 ppm deuterium)
* Decontamination of Pt-catalyst and its physico-structural characterisation after 3, 6 and 9 months from immersion.
* Evaluation of separation performances after 3, 6 and 9 month of exposure in the following conditions:
  - temperature: 40° C
  - atmospheric pressure
  - flow rates: 5L/min for H₂ and 1 ml/min. for tritiated water
  - gas/liquid ratio:3.5; (G=10 mole / m².s)
  - tritium free water
  - tritiated hydrogen (.100 MBq/mole)
PERMEABILITY OF TRITIUM INTO DIFFERENT MATERIALS AS A FUNCTION OF PARTIAL PRESSURE, TEMPERATURE AND CONCENTRATION

National Institute for Cryogenics and Isotope Technologies, Rm. Valcea

S. Brad, I. Stefanescu, L. Stefan, M. Zamfirache, A. Bornea, A. Lazar, F. Vasut, N. Bidica

Collaboration with:
Association EURATOM - FZK, Karlsruhe, Germany
Toughness stand with specific polystyrene foam like a thermal protection envelope.

Industrial plants who use cryogenic process like plants for liquid hydrogen distillation impose to use material with high tenacity in order to assure safety in working. In order to verify materials and equipment that work at cryogenic temperatures, it was necessary to realize a testing stand to determine fracture energy of different materials at cryogenic levels temperature. The experimental set-up developed allows to verify fracture energy for stainless steels which, after that, were used in the construction of equipment from "Experimental Plant for Tritium and Deuterium Separation" from ICIT Rm.Valcea.

The main element of the older stand was a Dewar recipient with liquid hydrogen which assure the temperatures level of 20 K permanently, connected with a cryogenerator. Constructively, the set-up was realized assembling three insulated components: liquid hydrogen storage vessel of 0.7 l capacity, the vacuum box and the insulation screen with liquid nitrogen. More than that, in order to decrease the evaporation rate, between the compartments where placed radiation screen made it from thin copper plate. During the testing procedure, the hydrogen vapors and the environment are separated by helium injection in order to eliminate the explosion dangers.
STAND MODIFICATION

a) The experimental stand (the new version)
   a) cryogenic liquid transfer line (between Cold-Box and Charpy device)
   b) acquisition system for temperature and toughness values
   c) cryogenerator type PPH
   d) Charpy F040/ S testing machine
Study of Hydrogen isotopes permeation into various materials as a function of gas composition, partial pressure and temperature

The first objective was to design and build an experimental stand dedicated to the hydrogen isotopes permeability investigation for metallic samples, used generally as materials for fusion facilities. Few general requirements has been considered when we started to design this new facility. The plate samples were thin discs with dimension between 20 and 25 mm diameter and 0.1-0.5 mm thickness. The membrane is squeezed by two cooper gaskets between knife-edge-sealed vacuum flanges. The assembly is connected to a vacuum system and before each experiment the stand was pump all by several hours. All the samples was cleaned with sequential washes of detergent, distilled water, acetone, and distilled water again.

For determination of hydrogen and deuterium isotopes permeation through membrane, the stand was connected with a gas-chromatograph. For aluminium 99.99% purity the results are shown in the following table

Table 1

<table>
<thead>
<tr>
<th>Material</th>
<th>Diffusivity $D_0(\text{m}^2/\text{s})$</th>
<th>Solubility $K_s(\text{mol/m}^3\text{Pa}^{1/2})$</th>
<th>Temperature $T(\text{K})$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$H_2$-Al</td>
<td>$1.8 \times 10^{-5}$</td>
<td>7.9</td>
<td>700</td>
</tr>
<tr>
<td>$H_2$-Al</td>
<td>$0.5 \times 10^{-5}$</td>
<td>6.9</td>
<td>770</td>
</tr>
<tr>
<td>$D_2$-Al</td>
<td>$1.1 \times 10^{-7}$</td>
<td>1.9</td>
<td>700</td>
</tr>
</tbody>
</table>

The measured permeability of $H_2$ and $D_2$ through materials tested, was quantified and agreed well with previously reported values ($P=D*K_s$).
For tritium permeation the experiments was performed at same concentration of tritium in a HT gas. The permeation tests were made at IFIN Bucuresti for radioactive protection rules reasons, inside of a special glove-box, at different tritium concentration values. The variation of the concentration of tritium and of course of the partial pressures was done in a decreasing mode by adding hydrogen gas, in the hydrogen/ tritium stocking vessel. The program of investigation is shown in the following table. The volume of HT gas for each sample was 140mL with a total concentration 585, 43 µCi/ mL. The time period for each test was set at 100h, and after that, the permeated gas was burned to water in a catalyst burner system. The volume of tritiated water, measured by LSC, was 3.5mL. All the samples were protected in order to not have a contact with air because even small amounts of oxygen at the surface can change the permeation rate. Oxide layers at the surface are usually used for to reduce protium, deuterium or tritium permeation. Also the permeation of hydrogen through metals is influenced by trapping at internal defects.

Table 2

<table>
<thead>
<tr>
<th>Sample</th>
<th>Thickness (mm)</th>
<th>Temperature (K)</th>
<th>Pressure before the membrane (Pa)</th>
<th>Total activity (on the burned water) (Bq)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al 99.99%</td>
<td>0.25</td>
<td>500K</td>
<td>1.9x10^5</td>
<td>425.912</td>
</tr>
<tr>
<td>Al 99.99%</td>
<td>0.13</td>
<td>500K</td>
<td>1.9x10^5</td>
<td>486.817</td>
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<tr>
<td>Al 99.99%</td>
<td>0.13</td>
<td>500K</td>
<td>2.2x10^5</td>
<td>498.124</td>
</tr>
<tr>
<td>Al 99.99%</td>
<td>0.13</td>
<td>550K</td>
<td>1.9x10^5</td>
<td>521.567</td>
</tr>
</tbody>
</table>
Design of a "Tritium concentrator" to be upgraded to water detritiation system for JET

Deliverable 2

- Objectives:
The design of the cold-box within cryogenic distillation column and other cryogenic equipments are accommodated and the hard shell confinement for the equipment which realized the interfaces with the CECE process will be provided.

For the cold-box design the following issues will be developed:

• mechanical analysis of strength and stability for the cold-box and its internals in specific operation conditions (high vacuum, different stresses determined by low temperatures and cold-box size);
• design of the support elements for the cold-box and its internals according to the specific location at JET AGHS.
• the vacuum train will be established.
• the arrangement of vacuum components for the specific space constrains at JET will be developed.
• the P&ID for the vacuum system, including the control and interlock requirements will be provided as well.
• 3-D layout of the hard shell confinement for the components working at room temperature and the safety issues according to general safety philosophy implemented at JET AGHS will be provided and implemented in the existing P&ID from JW3-FT-2.11 task.

- How they will be performed:
The design of the CD cold-box will be based on the available data for the CD column and other components specified in the JW3-FT-2.11. CATIA V5 and/or AUTOCAD will be used to produce drawings. The design of the cold-box will be realized according to ASME 8 standard for pressurized vessels/or other standard if may be required by JET.
Components data sheets for all components designed during the Deliverable 2, related to the CD cold-box and hard shell confinement will be produced.

Deliverable 3

- Objectives:
To provide the cost estimation and the time schedule for the construction and installation of the components related to the CD cold-box and hard shell confinement.
A PRM (Project Resource Management) and Standard Parts Catalogues in CATIA V5 for Tritium-containing System and Components are required. The CATIA V5 Equipment and Systems is a workbench chosen to perform design and integration of the electrical, fluid and mechanical systems within ITER. The wide range of applications such as Piping & Instrumentation Diagrams (P&ID), Piping Design, Tubing Design and Equipment Arrangement provides the ability for the upstream design process from the functional 2D design (P&ID) to the 3D detailed design. The Project Resource Management (PRM) is a framework of the project which on the one hand gives a tool to customize the working environment, on the other hand it organizes the design process to ensure design compliance with established standards, specifications, standardization, industrial conventions, terminology and practice. Feature Dictionaries, Catalogues for components as well as for standards and specifications are main parts of the CATIA V5 PRM.

The Project Resource Management system enables to:
- Control all resources of the Equipment and Systems (E&C) workbench in CATIA V5 for a certain project (ITER)
- Enables to set and follow standards and design rules for a project

Allows to
- Set up the necessary Feature Dictionaries for the applications of E&S
- Define different object classes in the different Feature Dictionaries
- Set proper attributes on the object classes
- Logically relate the functional 2D design with the detailed 3D design
- Build up and manage 2D symbol catalogues
- Build up and manage 3D parts catalogues
- Manage parametric parts
- Implement general and specific design rules
- Place parts and groups of parts automatically
- Offer only proper parts to the designer (e.g. correct size, correct material)
- Create reports
- Implement the project numbering system
- Producing a logical design using 2D tools such as P&ID Diagrams, HVAC Diagrams, Tubing Diagrams and so on.