

## EFDA JET TECHNOLOGY WORKPROGRAMME 2004

## FT-FUSION TECHNOLOGY

**TASK: JW4-FT-2.20**

***Endurance test catalyst – packing mixture , proposed for water detritiation system at JET, using SCK-CEN mixture***

***Deliverable: Catalyst- packing mixture preparation and delivering (after 3, 6, 9 months of exposure to tritiated and water-vapour). Characterization of the water from the Liquid Phase Catalytic Exchange (LPCE) column during the endurance test. Development of a regeneration procedure for catalyst/ packing mixture. Contribution to the definition of selection criteria of the catalyst/ packing and how to get these data.***

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## **1. Introduction**

At JET, a Water Detritiation System (WDS), based on electrolysis combined with catalytic isotopic exchange in liquid water (LPCE) was designed and now is under testing [1]. In the ITER a similar process for water detritiation is envisaged and the tritium concentration profile along the LPCE column is in the same range as for JET column. The operation conditions are similar for both facilities.

Before a full-scale water detritiation plant for JET and ITER can be designed, laboratory experiments to investigate the separation performances of different catalysts/packing mixture were carried out. These studies lead to select two different catalytic mixed packing: SCK-CEN packing, based on M01254 catalyst [2] and FZK packing based on ICIT-FZK catalyst with DX\* SULZER packing [3]. The main characteristics of both packing are presented in Table 1. Both mixed packings are based on Pt/C/PTFE catalyst and have similar performances but their catalytic stability at exposure to  $\beta$ -tritium radiation have to be very well known. The separation performances, assessed with the height equivalent of theoretical plates (HETP), are highly dependent on the ration G/L between the specific molar flow rates

of hydrogen and water. The two catalytic mixed packing, which have both good technological behavior and separation performances have to be investigated for their stability at tritium radiation exposure. The present report presents nine months endurance test results for the SCK-CEN packing in dynamic and static regimes.

## 2. Background of Research

The JET option for the WDS is the Combined Electrolysis Catalytic Exchange (CECE) process, a combination of electrolysis and catalytic exchange of hydrogen isotopes in a LPCE column, followed by cryogenic distillation (CD). In this process, part of the incoming tritiated water is directly electrolyzed in a commercial electrolyser. Afterwards the tritiated hydrogen is fed to the bottom of LPCE column, which is filled with a catalytic mixed packing and flows to the top in counter-current with water. In such a reactor the chemical exchange of tritium with hydrogen and water vapor and the physical exchange of tritium between water vapor and liquid water are timely and locally connected. This results in a simple and small exchange installation. Unfortunately, the chemical exchange between hydrogen and water is very slow at ambient temperature and requires high temperatures or a catalyst. Conventional hydrophilic catalysts lose their activity in contact with liquid water and water vapor because of low solubility and thus also the low diffusion rate of hydrogen in liquid water.

*Table 1. Characteristics of the SCK and FZK catalyst mixed packings*

|                                          | <b>SCK-CEN Packing</b>                                | <b>FZK Packing</b>                                        |
|------------------------------------------|-------------------------------------------------------|-----------------------------------------------------------|
| <b>Catalyst</b>                          | <b>Pt: 1%w</b><br><b>C: 19%w</b><br><b>PTFE: 80%w</b> | <b>Pt: 1.5%w</b><br><b>C: 15%w</b><br><b>PTFE: 83.5%w</b> |
|                                          | <b>Pellets</b>                                        | <b>Pellets</b>                                            |
|                                          | <b>Ø=1.9mm x2mm</b>                                   | <b>Ø=2mm x 4mm</b>                                        |
| <b>Packing</b>                           | <b>Minispirals</b>                                    | <b>Wire mesh</b>                                          |
|                                          | <b>Stainless steel</b>                                | <b>Stainless steel</b>                                    |
|                                          | <b>Ø=2mm x 2mm</b>                                    |                                                           |
| <b>Filling ratio (catalyst/packing)</b>  | <b>1:2</b><br><b>unordered - homogeneous</b>          | <b>1:2,5</b><br><b>ordered - sandwich method</b>          |
| <b>Temperature</b>                       | <b>40°C</b>                                           | <b>60-80°C</b>                                            |
| <b>Column diameter</b>                   | <b>20 mm</b>                                          | <b>38 mm</b>                                              |
| <b>[T] in H<sub>2</sub> inlet stream</b> | <b>100 –400 kBq/mol</b>                               | <b>-</b>                                                  |
| <b>[D] in H<sub>2</sub> inlet stream</b> | <b>50 ppm</b>                                         | <b>1-2 %</b>                                              |
| <b>G/L</b>                               | <b>3.51</b>                                           | <b>3.35</b>                                               |
| <b>HETP (cm)</b>                         | <b>11.38</b>                                          | <b>26</b>                                                 |

Hydrophobic catalysts have a good catalytic activity and high stability and could avoid such disadvantages. For this purpose, over 100 hydrophobic catalyst types have been prepared and tested [4]. From reviewed references it is concluded that platinum is the most active catalytic metal and TEFLON the most hydrophobic material [4].

Around 1980, SCK-CEN in collaboration with Johnson Matthey Company, patented a hydrophobic catalyst [5], for the exchange of tritium between hydrogen and water.

In June 2002 a new batch of catalyst, containing 1 % (w/w) platinum, 19 % (w/w) activated carbon and 80 % (w/w) PTFE (polytetrafluoroethylene) has been prepared. The cylindrical particles of 1.9 mm diameter and 2 mm height were used in a 1:2 volumetric ratio with etched stainless steel spirals of about the same dimensions. This new type of catalytic mixed packing, called SCK-CEN packing have been selected beside FZK-ICIT packing and proposed to be used in LPCE column for the WDS at JET. In order to select the most appropriate catalytic mixed packing to equip the LPCE column of the WDS, 3, 6 and 9 months endurance tests are necessary to know the influence of  $\beta$ -tritium radiation and the impurities from process water on catalytic activity and stability of the components of the two packings.

### 3. Experimental

#### 3.1 Description of installation for endurance test

Endurance tests for SCK-CEN catalytic mixed packing, exposed to beta-tritium radiation, in two different exposure modes (dynamic and static regimes) have been performed in order to get a complete data base concerning the influence of  $\beta$ -tritium radiation and of impurities on activity and stability of the catalyst. In order to perform endurance test in a dynamic regime an experimental installation has been established.

The installation is schematically shown in Figure 1, and mainly consists of: a) liquid phase catalytic exchange (LPCE) column; b) trickle bed reactor (TBR); c) apparatus for measurement and checking of operation parameters and tritium activity; d) water and hydrogen circuit; e) catalytic burner and f) condensers, phase splitters and traps with molecular sieves for drying hydrogen before analysis and before feeding the LPCE column. The LPCE column plays a main role to perform the endurance test and to determine the performances of the SCK-CEN catalytic mixed packing before and after exposure to tritiated water. The LPCE column is made of glass having a 20mm diameter and 2m height and is filled with the SCK-CEN catalytic mixed packing. The SCK-CEN catalytic mixed packing consist of Belgian MO 01254 hydrophobic catalyst and stainless steel spirals as the hydrophilic component in ratio 1:2. The active zone is 160 cm. To keep a constant testing temperature the LPCE column is foreseen with a heating jacket.

The trickle bed reactor (TBR) is a column with 30 mm diameter and 115 cm height and is filled up with a packing consisting of Romanian Pt/C/PTFE catalyst [6] and stainless steel rings in a ratio 1:1. On top and beneath this zone there is a height of 20 cm filled with packing material. This is necessary to uniformly distribute the incoming water and to heat it to the working temperature (zone on top) and to evaporate the water and to heat the incoming gas (zone beneath).

The trickle bed reactor for the preparation of tritiated hydrogen is fed with natural hydrogen from a battery of gas cylinders and the flow rate is adjusted to 5 l/min. Before feeding the TBR, the hydrogen is saturated with tritiated water vapor by bubbling through a saturating vessel that is filled with the hydrophylic packing and heated to 65°C. By means of a heating jacket the trickle bed reactor is kept at a constant temperature of 60°C. The main function

of the TBR is to produce tritiated hydrogen by isotopic exchange between tritiated water and the hydrogen LPCE column.

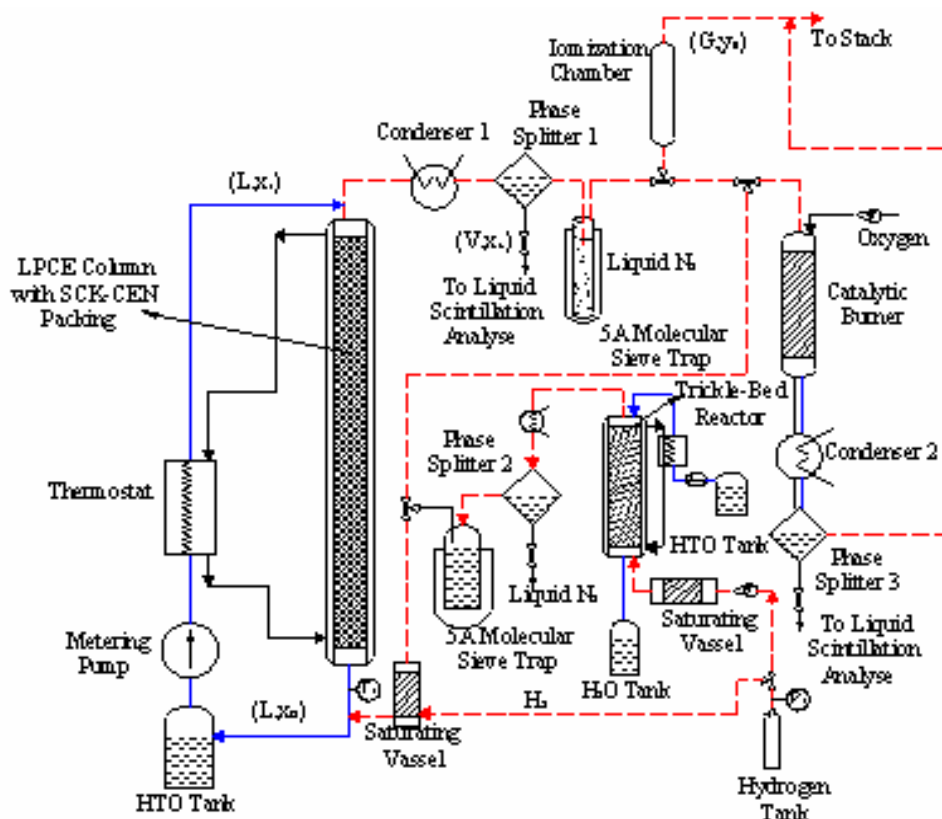


Figure 1. Layout of endurance facility of SCK-CEN packing

Saturated hydrogen flows from the bottom to the top of the TBR in counter-current with tritiated water (1-3 Ci/l). After the TBR, the main fraction of the water vapor present in the hydrogen stream is removed by cooling in condenser 2 and then separated and evacuated by phase splitter 2. Before feeding the LPCE column the tritiated water vapor traces are adsorbed on 5 A molecular sieve trap cooled by liquid nitrogen.

The installation includes also two metering pumps and two thermostats for heating of the feeding water of the LPCE column and an ionization chamber to determine the tritium activity of hydrogen at input and output of the LPCE column. A drawing of the experimental installation for the endurance test is shown in Figure 2. A double checking of tritium activity in hydrogen will be done by combustion of tritiated hydrogen in a catalytic burner (filled out Pd/alumina catalyst). Taking into consideration the use of radiotoxic tritium and flammable hydrogen, we provided ample safety measures and we obtained the necessary approvals

### 3.2 Testing Principles

The main purpose of research is to create a complete data base concerning:

- influence of tritium  $\beta$ -radiation and of impurities on the SCK-CEN catalytic packing mixtures' performances as in the LPCE process;

- influence of tritium  $\beta$ -radiation and of impurities on physic-structural parameters of materials from SCK-CEN mixed catalytic packing



*Figure 2. View of experimental installation and ionization chamber*

- stability in time of SCK-CEN catalytic mixed packings' performances;
- procedure for regeneration of catalyst and the activation of the SCK-CEN packing;
- erosion and mechanical resistance of the SCK-CEN packing in the LPCE process;
- identification of all potential poisons for a Pt-hydrophobic catalyst in hydrogen-water LPCE process;
- selection criteria of catalytic mixed packings and their application in the LPCE process.

The endurance test in a dynamic regime is performed in the LPCE column by continuous exposure of the SCK-CEN catalytic mixed packing to tritiated water for 3, 6 and 9 months. The tests are carried out at a given constant temperature (40°C) and constant pressure (normally atmospheric) in the LPCE column filled with the SCK-CEN packing. The SCK packing consist of Pt-catalyst particles and etched stainless steel spirals of about the same size, uniformly mixed in a volumetric ratio of 1: 2. During the day, for 8 hours by means of metering pump, tritiated water of 1 Ci/l heated at 40°C flow up to down through the column with 1ml/min flow in closed circuit. Afternoon and overnight the SCK-CEN packing from the LPCE column is flooded with tritiated water in a static regime. The tests are carried out at a specific molar flow rate  $G$  (typically 10 mole s<sup>-1</sup> m<sup>-2</sup>) of hydrogen and at a ratio  $G/L$  (typically 3 to 4) between the specific molar flow rates of hydrogen and water. In order to evaluate the separation performances of the SCK packing before and after exposure to tritiated water, the LPCE column is fed at the top with tritium-free water (distilled water) and at the bottom with tritiated hydrogen produced in the TBR. Before feeding

the LPCE column, the tritiated water vapor from tritiated hydrogen, is condensed and separated, and the traces of HTO vapor are adsorbed on the 5A molecular sieve trap, cooled with liquid nitrogen. To increase the water vapor contribution, before to feed the LPCE column, the hydrogen is bubbled in a saturating vessel (Figure 1), where becomes saturated with natural water vapor. The tritium concentrations in hydrogen (y) and in water (x) are measured at the top of the LPCE column, where water (tritium free for our experiments) is fed, ( $x_{inlet}$  and  $y_{outlet}$ ) and at the bottom of the column, where the (tritiated) hydrogen enters ( $y_{inlet}$  and  $x_{outlet}$ ). The tritium concentration in hydrogen are measured by means of an ionization chamber after total removal of water vapor, and by liquid scintillation method after conversion of the tritiated hydrogen to liquid water by catalytic combustion.

For the endurance test in a static regime 300 cm<sup>3</sup> of the SCK-CEN packing have been immersed in tritiated water (1 Ci/l) and after decontamination of Pt-catalyst, its physic-structural parameters after 3, 5 and 9 months from immersion will be measured and compared with initial values.

#### **4. Results and Discussions**

In the framework of endurance test the follow experimental works have been done:

- **commissioning** of the experimental installation static and dynamic exposure
- **exposure** of the SCK-CEN packing, continuously for 9 months at tritiated 1 Ci / L water in static and dynamic regimes
- **investigation** of physico-structural parameters of MO 1254 catalyst from the SCK packing, **before and after** 9 months exposure.
- **investigation** of process water quality.
- **evaluation** of separation performances of the SCK-CEN packing before and after 9 months exposure to  $\beta$ -tritium radiation

After each period of three months of continuous exposure to tritiated water, the physic-structural parameters of the Pt- catalyst, the separation performances of the SCK packing and the quality of process water are measured and compared to initial value. Before and after exposure of the SCK-CEN packing at 1Ci/l tritiated water, the physic-structural parameters of MO 1254 catalyst are determined by conventional methods. In Table 2, the main physic-structural parameters and the method to test for fresh MO1254 catalyst, are shown.

Comparable with conventional catalysts, the fresh MO 1254catalyst has a good porosity and Pt-particle size, a high metallic area and platinum dispersion. The same parameters, determined by the same methods for MO1254 catalyst sampled from the SCK-CEN packing exposed for 3 months to tritiated water are shown in the same table. The supplier of the MO1254 catalyst didn't give complete information on the parameters of interest. The fresh catalyst has a good porosity and Pt-particle size, a high metallic area and platinum

dispersion, comparable with conventional catalysts. In comparison with the initial values for a fresh catalyst, the physic-structural parameters for the catalyst after 3 months exposure to tritiated water are slightly lower but with no significant modifications.



Figure 3. SCK-CEN packing

Table 2. Physic-structural parameters of MO 1254 catalyst before and after 3 and 6 months exposure at  $\beta$ -tritium radiation

| Property □                                              | Analysis test method         | Value           |                         |                         |
|---------------------------------------------------------|------------------------------|-----------------|-------------------------|-------------------------|
|                                                         |                              | Before exposure | After 3 months exposure | After 6 months exposure |
| Platinum Content, (%wt)                                 | INCERP METHOD                | 0.916           | 0.914                   | 0.920                   |
| Macropore Volume (cm <sup>3</sup> /g)                   | ASTMD 4284-92                | 0.1467          | 0.1397                  | 0.1495                  |
| Micropore Volume, (cm <sup>3</sup> /g)                  | ASTMD 4222-91                | 0.1005          | 0.0413                  | 0.090                   |
| Pore Average Radius (Å)                                 | ASTMD 3663-92+□ASTMD 4365-95 | 42.15           | 57.76                   | 48.50                   |
| Specific Area (m <sup>2</sup> /g)                       |                              | 117.3235        | 98.677                  | 102                     |
| Adsorbed H <sub>2</sub> Volume (cm <sup>3</sup> /g cat) | ASTMD 3908-88 (93)           | 0.58            | 0.47                    | 0.51                    |
| H/Pt Dispersion (atom H/atom Pt)                        | ASTMD 3908-88 (93)           | 1.06            | 0.89                    | 0.94                    |
| Platinum Active Surface, (m <sup>2</sup> /g Pt Surface) | ASTMD 3908-88 (93)           | 290             | 274                     | 281                     |
| Platinum Particle Size Pt, Å                            |                              | 8.1             | 9.6                     | 9.0                     |

Metal content, macropore volume, platinum particle sizes are similar while the micropore volume, metallic active area and surface area, decreases slightly after exposure to tritiated water. A distribution calculation by pore radius confirms this trend and the phenomenon is probably due to a blocking of the macropores during the isotopic exchange process.

Another important parameter is the quality of tritiated water after direct contact with the SCK packing for long period of time. The conductivity, pH,  $\text{KMnO}_4$  demand, chlorine, fluorine and total iron content of water have been measured by conventional chemical methods, before and weekly after starting endurance test. All these values for fresh water and process water are shown in Table 3.

*Table 3. Quality of fresh water before and after continuous contact with the SCK – CEN packing*

| Type of sample        | pH          | Conductivity<br>$\mu\text{S}/\text{cm}$ | $\text{KMnO}_4$<br>demand,<br>$\text{mg/l}$ | Total Fe<br>$\text{mg/l}$ | $\text{F}^-$ ,<br>$\text{mg/l}$ | $\text{Cl}^-$ , $\text{mg/l}$ |
|-----------------------|-------------|-----------------------------------------|---------------------------------------------|---------------------------|---------------------------------|-------------------------------|
| <b>Fresh water</b>    | <b>6.47</b> | <b>1.72</b>                             | <b>2.2</b>                                  | <b>&lt; 0.01</b>          | <b>&lt; 0.02</b>                | <b>&lt; 0.1</b>               |
| After 2 weeks         | 6.16        | 54.4                                    | 26.5                                        | < 0.01                    | 0.31                            | 14.5                          |
| After 3 weeks         | 6.2         | 36.4                                    | 11.6                                        | < 0.01                    | 0.29                            | 8.3                           |
| After 4 weeks         | 6,08        | 24.59                                   | 4.7                                         | 0.03                      | 0.27                            | 3.6                           |
| After 5 weeks         | 6.0         | 18.69                                   | 3.8                                         | < 0.01                    | < 0.18                          | 1.3                           |
| After 6 weeks         | 6.24        | 6.94                                    | 4.3                                         | < 0.01                    | < 0.11                          | 0.23                          |
| After 7 weeks         | 5.95        | 9.08                                    | 4.8                                         | < 0.01                    | < 0.05                          | 0.20                          |
| After 8 weeks         | 6.15        | 8.16                                    | 6.3                                         | < 0.01                    | < 0.02                          | 0.18                          |
| After 11 weeks        | 6.4         | 7.99                                    | 4.1                                         | < 0.01                    | < 0.02                          | < 0.1                         |
| <b>After 3 months</b> | <b>6.35</b> | <b>8.51</b>                             | <b>5.6</b>                                  | <b>&lt; 0.01</b>          | <b>&lt; 0.02</b>                | <b>&lt; 0.1</b>               |
| <b>After 4 months</b> | <b>6.30</b> | <b>8.02</b>                             | <b>3.96</b>                                 | <b>&lt; 0.01</b>          | <b>&lt; 0.02</b>                | <b>&lt; 0.1</b>               |
| <b>After 6 months</b> | <b>6.38</b> | <b>7.97</b>                             | <b>4.5</b>                                  | <b>&lt; 0.01</b>          | <b>&lt; 0.02</b>                | <b>&lt; 0.1</b>               |
| <b>After 7 months</b> | <b>6.41</b> | <b>8.01</b>                             | <b>4.20</b>                                 | <b>&lt; 0.01</b>          | <b>&lt; 0.02</b>                | <b>&lt; 0.1</b>               |
| <b>After 8 months</b> | <b>6.35</b> | <b>8.10</b>                             | <b>4.86</b>                                 | <b>&lt; 0.01</b>          | <b>&lt; 0.02</b>                | <b>&lt; 0.1</b>               |
| <b>After 9 months</b> | <b>6.43</b> | <b>7.89</b>                             | <b>5.10</b>                                 | <b>&lt; 0.01</b>          | <b>&lt; 0.02</b>                | <b>&lt; 0.1</b>               |

The conductivity, pH,  $\text{KMnO}_4$  demand, chlorine, fluorine and total iron content of water have been measured by conventional chemical methods, before and periodically after starting the endurance test.



At the beginning of the endurance test **all parameters** of process water are **much higher in comparison to fresh water** (distilled tritiated water) but after repeated washings of the SCK-CEN packing, the values for all parameters significantly decrease.

Some amounts of **chlorine and fluorine have been identified** in process water but with time the contents continuously decrease till they reach the initial value for fresh water. Their presence could be due to some chlorine and fluorine traces adsorbed during preparation of the catalyst or during chemical treatment of the stainless steel spirals. Their presence in larger amounts could reduce the catalytic activity.

Iron and copper have not been identified in fresh or process water.

**After a month** of continuously testing, **the majority of physic-chemical parameters of process water reach the initial values of fresh water.**

The conductivity and oxidability of process water is also increased at the beginning of the endurance test and then gradually decreases without reaching the value of fresh water.

Therefore we recommend washing very well the packing before using and to check the quality of drained water. Iron has not been identified in fresh or process water. The conductivity and oxidability of process water is also increased at the beginning of the endurance test and then gradually decreases without reaching the value of fresh water. The initial separation performances of the fresh SCK-CEN packing have been evaluated by the height of unit transfer and of theoretical plate, the transport constants and their values for old and new batches are presented in Table 4.

*Table 4. Performances of the SCK-CEN Packing before and after exposure to tritiated water*

| Time of exposure                       | G/L  | H <sub>(OG)</sub> cm | HETP, cm | K (mole/ m <sup>3</sup> *s) |
|----------------------------------------|------|----------------------|----------|-----------------------------|
| <b>Fresh Packing (Before exposure)</b> | 3.25 | 11.18                | 15.74    | 105                         |
| After 3 months                         | 3.20 | 12.33                | 17.43    | 96                          |
| After 6 months                         | 3.15 | 11.10                | 16       | 106                         |
| After 9 months                         | 3.31 | 11.7                 | 16.5     | 101                         |
| Old SCK Packing*                       | 3.51 | 9                    | 11.4     | 123                         |
| <b>New SCK Packing*</b>                | 3.84 | 9.4                  | 11.6     | 118                         |

\* Values delivered and determined by SCK-CEN Mol before the endurance test

After 9 months endurance test performed in the above-mentioned conditions of continuously exposure of 1Ci/l tritiated water no significant modifications of separation performances have been observed. Decontamination factors between 1100-1300 on tritiated hydrogen have been obtained.

Evaluation of separation performances for SCK-CEN packing has confirmed the good performances reported by SCK-CEN in the previous report. The difference between the separation performances of the SCK packing determined by ICIT and SCK-CEN is around 15%. This difference could be due to isotopic mass balance errors.

After 9 months endurance test performed in above-mentioned conditions of continuous exposure to 1 Ci/ l tritiated water in a dynamic regime, no significant modifications of separation performances have been observed.

The slight modification of some physic-structural parameters of catalyst, and of the quality of water during the endurance test, in the above-mentioned conditions, don't significantly affect the performances of the SCK-CEN packing.

### Conclusions

The influence of  $\beta$ - tritium radiation and of impurities on the MO 1254 catalyst and on the separation performances of the SCK-CEN packing in the detritiation water process by isotopic exchange in the liquid phase has been investigated in dynamic and static regime, **for 9 months.**

**The initial separation performances of the SCK-CEN packing remain almost constant after 9 months exposure to 1Ci/l tritiated water in the dynamic and static regimes.**

The physic-structural parameters of the MO 1254 catalyst from the composition of the SCK-CEN catalyst are comparable to a conventional Pt-catalyst. After 6 months continuous exposure to 1Ci/l tritiated water, no significant modifications of the catalyst's physic-structural parameters have been observed. Only the micropore volume, metallic active area and surface area tend to slightly decrease after exposure.

Some amounts of chlorine and fluorine have been identified in process water but with time, their contents continuously decrease till they reach the initial values of the feeding water. On the other hand, at the beginning of the endurance test, the conductivity and KMnO<sub>4</sub> demand are also increased and gradually decrease without reaching the value of fresh water.

### References:

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