DEVELOPMENT OF WATER DETRITIATION FACILITY FOR JET

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This paper presents results from the concept evaluation, experimental trials, and design of a water detritiation facility (WDF) for the JET fusion machine. The design is based on the combined electrolysis and catalytic exchange process and will allow construction of the plant and for its integration into the JET tritium plant in three stages.

The first stage includes a liquid phase catalytic exchange column and electrolyzer to concentrate the water into a smaller amount of tritium-enriched water. There would then be three options for dealing with this water: processing off-site, conversion to solid intermediate-level waste for disposal, and further processing on-site for complete tritium recovery. The latter option will require the second stage of implementation to integrate the WDF with the isotope separation system of the tritium plant. The third stage might be desirable to reduce the amount of time that the existing isotope separation system would need to be involved in the recovery of tritium from the WDF.

KEYWORDS: *fusion fuel cycle, tritiated water, water detritiation*

I. INTRODUCTION

JET is the world's largest tokamak and has the capacity to operate with a tritium plasma. Since the intensive experiments with tritium, DTE1 (Ref. 1) in 1997, a continuous flow of tritiated water has been generated from the discharges of air and gases contaminated with tritium to the exhaust detritiation system (EDS). EDS provides the final barrier at JET for protecting the envi-

ronment against tritium release. Tritium, in the form of molecular hydrogen and hydrocarbons, is catalytically oxidized in the EDS. Water vapor produced and already present in the incoming gas stream is adsorbed in dryers filled with molecular sieve, and the detritiated dry air is discharged to the atmosphere. The driers need regular regeneration of the molecular sieve, and this produces tritium-contaminated water at rates and concentrations that vary depending on the scenario and period of time the machine is in operation or shut down for maintenance and enhancement. The tritiated water collected in the EDS is transferred into 200-ℓ stainless steel drums for storage and subsequent processing. The quantity of water and tritium inventory in each drum are recorded and the yearly arising are summarized in Table I.

Batches of drums are sent off-site for processing. A high likelihood for continued operation of the JET machine for several more years and preparation for its decommissioning initiated an evaluation of options for processing tritiated water on-site. Reviews[2–11] of various technologies showed that the combined electrolysis and catalytic exchange (CECE) process is the most effective way of processing tritiated water and therefore has been chosen for a water detritiation facility (WDF) proposed at JET. This technology has also been selected for processing tritiated water in the tritium plant of the next fusion reactor, the International Thermonuclear Experimental Reactor[10,12] (ITER).

A block diagram of a facility for water detritiation based on CECE technology is illustrated in Fig. 1. Tritiated water (HTO feed) is converted to gaseous hydrogen in the electrolyzer (EL). This hydrogen is fed to the liquid phase catalytic exchange (LPCE) column, in which it participates in tritium isotopic exchange with tritium-free water (H_2O feed) fed to the top of the LPCE column. The isotopic exchange reactions result in tritium being transferred from the hydrogen stream to the water stream. The detritiated hydrogen (H_2 discharge) is discharged from the LPCE column. The tritium-enriched water accumulates in the electrolyzer until a predetermined tritium inventory has been reached. It is then withdrawn from the electrolyzer and transferred into storage. For

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TABLE I

Rate of Tritiated Water Collection at JET and Range of Tritium Concentration

Year	Mass (kg)	Recorded Tritium Concentration (GBq/kg)		Total Tritium Activity (TBq)	Average Tritium Concentration* (GBq/kg)
		Minimum	Maximum		
1997	4 972	0.04	16.7	42.55	8.6
1998	9 181	24.7	206.4	771.68	84.6
1999	13 803	8.7	24.9	229.06	16.6
2000	6 219	5.5	16.7	62.46	10.0
2001	12 970	1.5	5.7	37.30	2.9
2002	10 834	1.4	3.8	31.16	2.9
2003	7 869	2.6	3.2	24.43	3.1
2004	20 566	0.6	5.0	41.40	2.0
2005	7 884	0.45	2.09	5.34	0.68

*This value presents an average concentration for the water collected for the whole year.
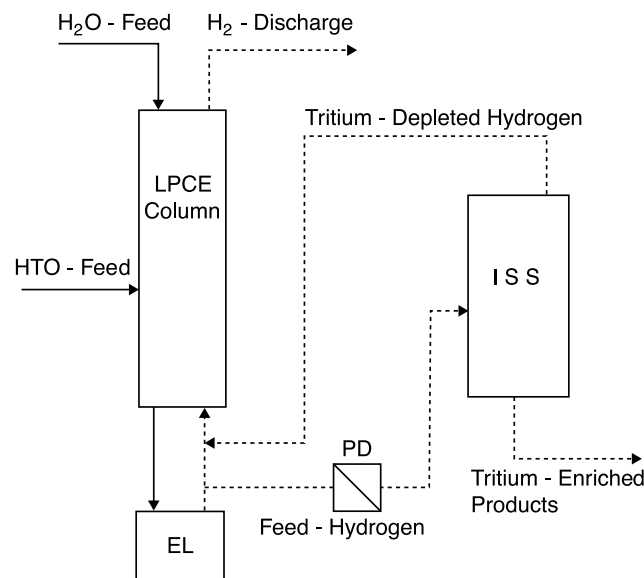

Fig. 1. Logic diagram of CECE WDF (Refs. 2 and 3).

this scenario of operation, the facility provides a reduction in volume of tritiated water but does not recover tritium for reuse.

To recover tritium for reuse, the tritium-enriched hydrogen stream from the electrolyzer can be processed in an isotope separation system (ISS). The most suitable method of separating hydrogen isotopes for employing with the WDF is cryogenic distillation, and the JET active gas handling system (AGHS) is equipped already with a hydrogen cryogenic distillation ISS (CD ISS) facility.[13] Figure 1 shows a portion of the tritium-enriched hydrogen flow produced by the electrolyzer being withdrawn to the ISS for further enrichment. The hydrogen fed to the cryogenic ISS has to be chemically ultrapure and a palladium permeator (PD), which allows only hydrogen to go through the gas separation membrane, is shown placed between the electrolyzer and the ISS feed for this purpose. The hydrogen depleted with tritium in the ISS is returned to the LPCE column.

Since the DTE1 experiment, the JET machine has operated mostly with deuterium plasmas. Tritium, outgassing from in-vessel components, contaminates the tokamak exhaust gases. For a large number of plasma pulses, the concentration of tritium in the tokamak exhaust was too small to process for further recovery in the AGHS, and this, along with deuterium used for first-wall conditioning by glow discharge cleaning (GDC), was discharged to the EDS rather than collected for processing. Additionally, GDC using helium released deuterium from the first wall, which was also sent to the EDS. All these discharges lead to an increase in deuterium concentration in the water collected in the EDS, to a level beyond that in natural water. The WDF therefore needs to handle all three hydrogen isotopes and will recover this deuterium and accumulate it along with tritium.

The development to date of the WDF has been carried out in phases, commencing with concept evaluation and leading to experimental trials to verify the operability of the key elements. This was followed by selecting the operation mode for the facility and, finally, the design of the facility. This paper presents results of all stages of the work.

II. CONCEPT EVALUATION

The concept evaluation included an assessment of the key elements that would be needed (such as the LPCE column, electrolyzer, and ISS), preparation of

the process flow diagram, the carrying out of a numerical evaluation of the facility performance, and a hazard and operability (HAZOP) study. A scoping study[2] has concluded that a throughput of 10 t/yr or larger would be needed to process the water arising listed in Table I.

The key element of the facility is the LPCE column: This determines the detritiation factor that can be achieved. The removal of tritium from a gaseous hydrogen stream in the LPCE column is driven by two sets of isotopic exchange reactions: those between gaseous hydrogen and water vapor and between water vapor and liquid water. Each of the hydrogen isotopes participates in these reactions. Equilibrium separation factors for hydrogen isotopic exchange reactions between gaseous hydrogen and water vapor decrease very noticeably with temperature rise. The temperature dependence of the equilibrium separation factors for reactions between water vapor and liquid water is weak. Therefore, to benefit from a large separation factor, it is desirable to operate the LPCE column at as low a temperature as possible, but still sufficient for providing the required rates of reaction and mass transfer. In CECE technology the operating temperature for LPCE columns lies in the range between 313 and 353 K and pressure at, or slightly above, atmospheric. The reaction between water vapor and gaseous hydrogen needs a catalyst, but operating at temperatures below the boiling point of water makes a hydrophilic catalyst in the LPCE column undesirable. Therefore, special catalysts with hydrophobic properties have been developed in many laboratories for CECE technology, and those based on platinum have proved to be most effective. To promote hydrogen isotopic exchange reactions between liquid water and water vapor, devices to arrange contact between the liquid and vapor phases, such as trays or packing employed in the water distillation column, will be used. For the relatively small throughput and large rate of mass transfer needed for LPCE columns, packed columns with very efficient packing are usually employed.

II.A. Types of Packing for the LPCE Column

Three main types[4–10,14] of packing arrangement specific for LPCE columns have been developed and tested. The first two types are based on using random packing.[5–9] In the first type, to allow all reactions mentioned above to occur at the same time throughout the packed bed, catalyst is mixed with inert packing. In the second type of arrangement,[10] catalyst and inert packing are loaded into modules in which they are separated from each other. Gas and liquid water flow in the countercurrent direction through the layer of inert packing, but only the gas stream (comprised of hydrogen and water vapor) is permitted to flow through the catalyst layer.

The large specific surface area provided by random packing usually results in a high rate of mass transfer. However, mixing of random packing with a hydrophobic catalyst causes reduction in the gas superficial velocity limit for column flooding. For LPCE columns with this type of packing arrangement, the hydrogen flow threshold is usually in the range from 10 to 15 mol/m$^2 \cdot$s (Refs. 5, 8, and 14). The performance of LPCE columns with the first type of packing arrangement has been tested in columns of diameter up to 0.1 m (Refs. 5, 7, and 8). If the WDF should require an LPCE column greater than 0.1-m diameter, then the height of the packing layer will need to be corrected using a scale-up factor; however, such factors are not known for this type of packing arrangement. Flooding of an LPCE column with the second type of packing arrangement occurs at a higher gas velocity than for the first type; this is because there is no direct contact of hydrophobic catalyst with liquid water. The scale-up factors for such arrangements can be obtained from well-proven chemical engineering data for gas-liquid separation processes, such as absorption and distillation, arranged in packed columns.

The third type of packing arrangement uses catalysts embedded in structured packing.[4] Columns with structured packing usually have a much higher gas velocity threshold to the onset of flooding than random packings,[15] although a limited trial[4] demonstrated that the effectiveness of an LPCE column with packing of the third type could be considerably smaller than those of the other two types.

II.B. Electrolyzer

The next key element of the facility is the electrolyzer, which holds water enriched with tritium and therefore presents the largest portion of overall tritium inventory in the WDF. Two main types of electrolyzers available commercially are alkali electrolyte electrolyzers and solid polymer electrolyte (SPE) electrolyzers. The alkali electrolyte type is based on the use of inorganic membranes and an aqueous solution of potassium hydroxide as an electrolyte. Because the presence of KOH in the hydrogen stream is not desirable for the majority of applications, alkali electrolyzers are usually equipped with hydrogen purification devices. The SPE electrolyzers use a proton exchange polymeric membrane as a solid electrolyte, and the water inventory in this type of electrolyzer is considerably smaller than that in the alkali electrolyte type. However, polymeric membranes are also less resistant to degradation from β irradiation by tritium and are more sensitive to the purity of feedwater than alkali electrolyzers. A life expectancy of 30 000 h or more has been demonstrated for both SPE and alkali electrolyzers operated with tritium-free and chemically pure water, although it has been estimated that this may reduce to ~1 yr for an SPE electrolyzer processing water having a tritium concentration of 18 500 GBq/kg (Ref. 12). SPE electrolyzers, however, are free of the disadvantage presented by alkali electrolyzers whereby potassium hydroxide might break through the hydrogen

purification device and poison the catalyst in the LPCE column.[6]

Modern designs of industrial electrolyzers use a "filter press"–type electrolysis cell stack and operate at elevated pressures, usually in the range of 0.4 to 1 MPa. There is the potential for tritiated hydrogen and water to escape between the cells, so the cell stack would need to be placed within secondary containment to provide a barrier against tritium escaping to the environment.

The scoping study[2] showed that an electrolyzer with a hydrogen production rate of 10 Nm^3/h should be sufficient to serve a detritiation facility processing 10 t/yr of tritiated water and will provide hydrogen to the LPCE column at a flow of 446 mol/h.

II.C. Incorporation with Existing AGHS Plant

Using a ratio of tritiated water (HTO) over tritium-free (H$_2$O) feedwater of 1 as a design reference value and with the tritium discharge from the WDF being 10% of the daily discharge limit of 20 GBq adopted for the JET AGHS when fed with water at the maximum tritium concentration listed in Table I, the WDF would be required to provide a decontamination factor (DF) of about 10 000 (Ref. 2). A numerical evaluation[2] showed that an LPCE column of a length equivalent to 25 "theoretical plates"[15] would be capable of providing such a DF.

The LPCE column will discharge 10 Nm^3/h of gaseous hydrogen, and this has been identified as a major hazard in the HAZOP study of the WDF. To reduce the probability of hydrogen explosion at the discharge point, a number of engineering measures can be employed.[10,16] In addition to the engineering measures, catalytic oxidation of hydrogen during its mixing with air for discharge could be employed.[17,18]

The water detritiation facility has to be incorporated into the environment of the JET tritium plant (AGHS) while it is in continuous operation to support the JET experimental program. The construction and operation of the WDF needs to present as small a disruption as possible to the AGHS, although, to reduce capital cost, the WDF needs to use as much as possible of the infrastructure and facilities already available at the AGHS. These requirements make it desirable for the water detritiation facility to be designed and supplied as a turnkey facility with only a minimum of connections to the AGHS.

The nature of the process taking place in the LPCE column requires the facility to operate continuously, 24 h a day and 7 days a week, for long periods of time. This requires the facility to have sufficient reliability and redundancy of key items such as the electrolyzer, which has a service life linked to tritium concentration in the water of the electrolyzer loop. It is desirable to have spare capacity to process amounts of water at rates higher than 10 t/yr to cover for occasional increased demand, such as that seen in 2004 (see Table I). Finally, operation of the water detritiation facility in combination with the

CD ISS of the AGHS must not compromise the availability of the CD ISS for supporting the JET operation. To meet the last requirement, two options were considered:

Option A: Utilize the CD ISS as it becomes available for short-term campaigns to enrich tritium recovered in the WDF. The CD ISS is designed to process ~5 mol/h of hydrogen. This option requires the WDF to operate without tritium transfer to CD ISS between the campaigns.

Option B: Incorporate a new hydrogen cryogenic distillation column (CD column) to enrich the tritium to the level at which it can be processed in the gas chromatographic (GC) ISS of the AGHS (Ref. 1). The CD column will be designed to have a higher throughput than the CD ISS and utilize the spare cooling capacity (~100 W) of the helium refrigerator that serves the AGHS CD ISS. The scoping study[2] showed that with this cooling capacity available, a hydrogen feed flow rate to the new CD column can be as large as 100 mol/h, albeit at a reduced separation capacity. The tritium concentration to be reached in this new CD column was therefore chosen[2] to be 0.5 at.%, which is sufficient purity for AGHS GC ISS feedstock. Option B would obviate the need to utilize the AGHS CD ISS.

These options are now considered by way of examples of processing arising tritiated water for the years 1998, which had the largest tritium inventory (referred to as scenario 1), and 2005, which had the lowest (scenario 2), listed in Table I. Using a specific activity of pure tritium of 1074 TBq/g-at. (358 TBq/g), the following assumptions were made: The maximum allowable tritium inventory of 37 TBq (or 0.0345 g-at. T) allowed in the AGHS safety case for a unit of equipment is contained in an SPE-type electrolyzer holding 20 kg of tritiated water, giving the tritium concentration in the water of the electrolyzer loop to be $X_{el} = 1.85$ TBq/kg = 3.1×10^{-5} g-at. T/mol. The tritium separation factor for water electrolysis in an SPE electrolyzer is $\alpha_{el}(T) = 2$ (Ref. 19; also see Sec. II.E), and so the tritium concentration of the hydrogen produced by the electrolyzer is $Y_{el} = X_{el}/\alpha_{el}(T) = 1.55 \times 10^{-5}$ g-at. T/mol.

The hydrogen production rate for the electrolyzer is 446 mol/h. Taking the ratio of tritiated water feed rate (L_{HTO}) to tritium-free water (L_{H_2O}) of 1 as a reference, the feed rate of tritiated feedwater to the WDF should be 223 mol/h. Tritium concentration in hydrogen discharge (Y_{dis}) from ISS (where ISS is either the CD column or the CD ISS of the AGHS) is ≈0.3 ppm (Ref. 13).

The results of the evaluation are given in Table II for "steady-state" operation of the WDF ISS with a stock of feedwater having a homogeneous tritium concentration. For the tritium concentrations in the feed (Y_{el}) and discharge (Y_{dis}) streams mentioned above, ISS will provide tritium removal fraction $F = (Y_{el} - Y_{dis})/Y_{el}$ above 99%. The rate of tritium transfer from the WDF to the ISS is

TABLE II

Evaluation of the WDF ISS*

Parameter	Scenario 1	Scenario 2
Stock of water to be processed (mol)	5.1E5[a]	4.4E5
Tritium inventory of the stock (g-at. T)	0.72	0.5E−3
Tritium concentration in the stock (g-at. T/mol):		
Minimum	4.1E−7	7.5E−9
Maximum	3.46E−6	3.5E−8
Average	1.42E−6	1.14E−8
Average tritium feed rate to the WDF (g-at. T/h)	3.17E−4	2.53E−6
Time to reach maximum tritium inventory of the electrolyzer (h)	109	13 583
Rate of electrolyzer tritium inventory change[b] (%/h)	Option A: +0.7 (gaining inventory) Option B: −3.6 (losing inventory)	Option A: −0.2 (losing inventory) Option B: −4.5 (losing inventory)
Time of WDF operation to restore electrolyzer tritium inventory[c] (h/% of inventory)	Option A: 2.2 (with no HTO feed to the WDF) Option B: 1.1 (with no tritium transfer to the ISS)	Options A and B: 136 (with no tritium transfer to the ISS)

*Also see Table I.

[a]Read as 5.1×10^5.

[b]For the WDF operating with the ISS and for tritium inventory of electrolyzer of 0.0345 g-at. T.

[c]For the WDF operating either with no feeding HTO water or with no tritium transfer to the ISS. A tritium inventory of the electrolyzer of 0.0345 g-at. T is assumed.

then determined by the expression $G_{ISS}Y_{el}F$, where G_{ISS} is the feed flow rate to the ISS.

The results in Table II demonstrate that for a tritium concentration in the feedwater equal to an average concentration (1.42×10^{-6} g-at. T/mol, 84.6 GBq/kg) for the 1998 stock shown in Table I, the inventory of the electrolyzer will rise when the WDF operates with the CD ISS (option A). This will apply at any concentration above 3.5×10^{-7} g-at. T/mol because the tritium flow to the WDF is larger than the rate at which tritium can be transferred from the electrolyzer to the CD ISS. For tritium concentrations in the feedwater below 3.5×10^{-7} g-at. T/mol, the electrolyzer will lose its inventory as a result of tritium transfer to the CD ISS. For option B, the electrolyzer tritium inventory will fall when processing all the arising listed in Table I.

This evaluation clearly shows that for a feedstock of variable tritium concentration, a steady-state mode of WDF ISS operation, where the rate of tritium feeding to the WDF matches the rate of tritium transfer from the WDF to the ISS, cannot be achieved. The electrolyzer will either lose or gain tritium inventory, depending on tritium concentration in the water fed to the WDF and feed flow to the ISS. To control tritium inventory of the electrolyzer, either feeding of tritiated water to the WDF or tritium transfer from the electrolyzer of the WDF to the ISS would need to be stopped, as required. Changes of tritium inventory in the electrolyzer will cause either a corresponding change in tritium concentration in the hy-

drogen fed to the ISS or a discontinuous feed to the ISS. Operating an ISS under these conditions is undesirable.

To reduce the negative effect of fluctuating composition or flow rate of the feed to the ISS, the WDF could operate first to convert the stock of water of variable tritium concentration to a smaller stock of water enriched with tritium to a predetermined concentration. An option of withdrawing water enriched with tritium from the electrolyzer to a storage tank is considered now. This stock of water, with a smaller volume than that of the original but with higher tritium concentration, can be processed later by feeding to another electrolyzer. The hydrogen generated in this second electrolyzer is fed to the ISS. This approach also makes operation of the WDF independent of ISS availability. Alternatively, the stock of tritium-enriched water can be sent off-site for processing. Table III presents results of an evaluation of this approach for two given tritium concentrations in the water of the storage tank (X_{st}). In case I the concentration, 3.1×10^{-5} g-at. T/mol (1.85 TBq/kg), is determined by a maximum tritium inventory of 37 TBq in an electrolyzer having a 20-kg water inventory. In case II the concentration, 5.5×10^{-6} g-at. T/mol (330 GBq/kg), is a maximum tritium concentration allowed for international transfer of tritiated water.

Table III shows that for an electrolyzer operated at its largest allowable tritium inventory (case I), both options A and B are technically acceptable for processing all arising listed in Table I with complete tritium recovery.

TABLE III

Time of Tritium Transfer from the WDF to the ISS*

Parameter	X_{st} (g-at. T/mol)	
	Case I: 3.1E−5[a]	Case II: 5.5E−6

Volume of tritium-enriched water in the storage tank for processing:
    Water arising in 1998 (scenario 1)
    Water arising in 2005 (scenario 2)
	2.3E4 mol (418 kg)
	162 mol (2.9 kg)
		1.3E5 mol (2338 kg)
		899 mol (16.2 kg)

Time (h) of tritium transfer from the storage tank to the ISS for processing:
    Water stock 1998 (scenario 1)
 	
    Water stock 2005 (scenario 2)
	Option A: 4600
	Option B: 230
	Option A: 32
	Option B: 1.6
		Option A: 26 000
		Option B: 1 300
		Option A: 181
		Option B: 9

*Also see Table I.
[a]Read as 3.1×10^{-5}.

For tritium concentration in an electrolyzer limited to 330 GBq/kg (case II), processing water of large tritium concentration (scenario 1, for water arising in 1998) using option A requires an unacceptably long time. Option A might be technically acceptable for stocks of moderate concentration and is acceptable for stocks of low concentration. Option B is acceptable for processing all stocks listed in Table I.

If required, the time needed for processing a stock of tritium-enriched water can be reduced using the approach described in Ref. 20: Hydrogen produced by the second electrolyzer can be enriched with tritium prior to being fed to the ISS. This will speed up tritium transfer from the storage tank to the ISS. The approach is based on using a vapor phase catalytic exchange (VPCE) reactor and on the property that the tritium concentration in the water of the electrolyzer is always higher than that in the hydrogen produced by electrolysis of this water. The VPCE reactor would be fed with water vapor produced by evaporation of water in the loop of the second electrolyzer and with hydrogen produced by this electrolyzer. The catalyst in the VPCE reactor promotes tritium isotopic exchange between the water vapor and the hydrogen. If the operating temperature of the VPCE reactor is sufficiently high, the isotopic effect of the reaction will be near to 1, causing tritium transfer from the water vapor to the hydrogen. For a water electrolysis tritium separation factor of $\alpha_{el}(T) = 2$ and molar ratio of hydrogen to water vapor flow rates of 0.5, a tritium enrichment factor of 1.6 can be achieved in a VPCE reactor operated at an elevated temperature (>570 K).

II.D. Selection of Tritiated Water Feeding Point

The common practice for operation of industrial separation columns is that the feed should be injected to the point at which its composition matches the profiles established along the column. This is particularly impor-tant for columns designated for separation of isotopes where equilibrium separation factors are usually small. The reason is to avoid damaging the profiles, which would cause considerable reduction in the separation capability of the column. With the range of possible feed compositions as shown in Table I, providing a feed of constant composition to the LPCE column of the WDF would not be possible: The feed point would need to be moved as the composition of the feedwater changes. For feedwater containing both tritium and deuterium at levels exceeding those of natural water, finding a feeding point to the LPCE column at which the water composition matches both the tritium and deuterium profiles in the column is a very difficult task, for two reasons. First, the column will have different profiles for tritium and deuterium. Second, the equilibrium separation factor for the tritium isotopic exchange reaction between hydrogen and water vapor depends on deuterium concentration. This is illustrated in Table IV, which shows that the tritium separation factor decreases drastically, from 5.0 for light water

TABLE IV

Equilibrium Separation Factors for Tritium (α_T) and Deuterium (α_D) Isotopic Exchange Between Water Vapor and Hydrogen for Different Deuterium Concentrations in Water*

Separation Factor[a]	Deuterium Concentration (at.%)				
	0.015	25	50	75	99
α_T α_D	5.0 3.1	4.1 3.0	3.2 3.0	2.3 2.9	1.6 2.7

*From Ref. 21.
[a]Values for separation factors are given as an example for a temperature of 333 K and a tritium concentration ≪1 at.%.

TABLE V

Change of Tritium Inventory of Electrolyzer as
Result of Tritium-Enriched Water Withdrawal

Scenario[a]	Case[b]	Rate[c] (mol/h)	Resulted Concentration[d] (%)
1 (processing water stock 1998)	I	10.2	99
	II	57.6	96
2 (processing water stock 2005)	I	0.08	99.99
	II	0.46	99.99

[a]See Table II.
[b]This is for two nominal tritium inventories of the electrolyzer: 37 TBq for case I and 6.6 TBq for case II (see Table III).
[c]This is the amount of tritium-enriched water to be withdrawn from the electrolyzer every hour.
[d]This is the tritium inventory of the electrolyzer as a result of its replenishment with the feedwater. It is given as a percentage to the inventory prior to the replenishment.

to 1.6 for heavy water. In contrast, the change of the separation factor for deuterium is small, ~10%. For tritiated water containing deuterium at levels exceeding that of natural water, searching for an optimum feeding point to the LPCE column must take into account both tritium and deuterium concentrations in the feed and the profiles of both isotopes in the column. For large separation factors for deuterium and tritium isotopic exchange reactions between water vapor and gaseous hydrogen, as shown in Table IV, the isotopic profile will change very quickly along the LPCE column. The optimization of the feeding point based on three variable parameters, such as isotope profiles in the column and the tritium separation factor, would need further theoretical investigation and experimental validation.

An alternative method of feeding the WDF with tritiated water is directly to the electrolyzer loop, independently of water composition. This mode of feeding will result in the tritium concentration in the electrolyzer decreasing after tritium-enriched water is withdrawn and the electrolyzer replenished with the tritiated feedwater. This effect is illustrated in Table V by way of an example of tritium-enriched water being withdrawn from the electrolyzer every hour.

Table V shows that even for the worse case, tritium inventory of the electrolyzer drops by 4% only. The electrolyzer acts as a buffer tank to smooth the disturbance of the isotopic profiles in the LPCE column created by feeding tritiated water.

II.E. Effect of Deuterium Content in Feedwater

The effect of the increased deuterium content in the feedwater on the performance of the LPCE column can

be illustrated using a McCabe-Thiele diagram[15] that represents steady-state operations. As an example, the McCabe-Thiele diagram in Fig. 2 shows the equilibrium separation factor (α) for the protium-deuterium isotopic mixture (α_D) and operation line (λ) for the LPCE column in the WDF operated with tritiated water fed to the electrolyzer. The presence of tritium in this mixture can be disregarded because its concentration is always on a trace level (in parts per million), which has no effect on the deuterium separation factor. Parameter λ is a molar ratio of gaseous hydrogen flow (G) to water flow (L) in the LPCE column. For equal flow rates of tritiated (L_{HTO}) and tritium-free (L_{H_2O}) waters, $\lambda = G/L \approx (L_{HTO} + L_{H_2O})/L_{H_2O} \approx 2$. For the WDF with tritiated water fed to electrolyzer, the ratio λ is the same for the whole length of the LPCE column because there is no change in water flow along the column. Because the LPCE column removes deuterium from the hydrogen to be discharged, deuterium will accumulate in the electrolyzer until a maximum possible concentration is reached. This maximum concentration is presented by the point at which the operation line (λ) crosses the equilibrium line (α). For $\lambda = 2$ and an operating temperature of the LPCE column of 333 K, this maximum deuterium concentration is ~45 at.%. Figure 2 clearly shows that increase of λ will lead to smaller maximum deuterium concentration at the bottom of the LPCE column, and consequently in the electrolyzer. For example, for $\lambda = 3$, this value will be ~15 at.%.

Once the deuterium concentration at the bottom of the LPCE column has reached its maximum possible
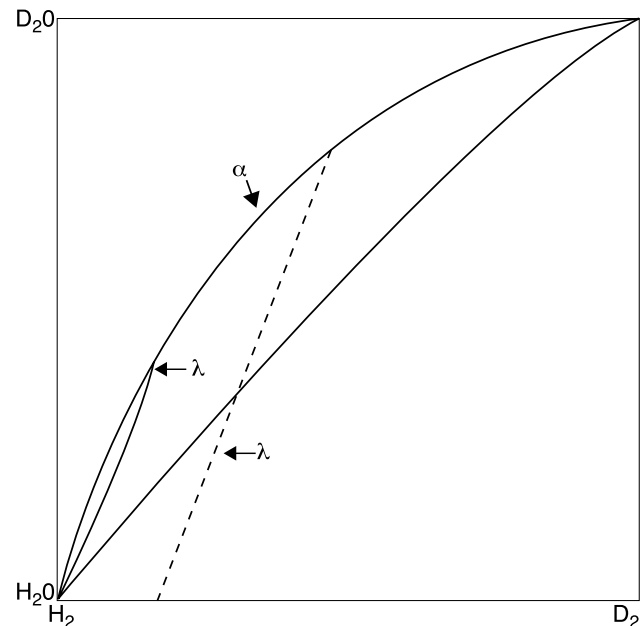
Fig. 2. McCabe-Thiele diagram for separation of protium-deuterium mixture in LPCE column with tritiated water fed to the electrolyzer.

value, further deuterium fed to the WDF will result in an increase in deuterium concentration of the hydrogen discharged at the top of the LPCE column (dashed line in Fig. 2) until the flow of deuterium discharged with the hydrogen from the LPCE column equates with the flow of deuterium fed to the WDF.

The operation of the WDF with tritiated water fed into the LPCE column is presented in Fig. 3 by a more complex McCabe-Thiele diagram. Deuterium concentrations in the water and hydrogen reduce from the bottom to the top of the LPCE. The portion of the LPCE column above the feeding point is usually termed the "stripping column" because it acts to remove heavy isotopes from the hydrogen to be discharged. The operation line of the stripping column is shown in Fig. 3 as λ_S. At the point of tritiated water feed to the LPCE column, water in the column having a high deuterium concentration mixes with the feedwater, which has a lower concentration, leading to a drop in deuterium concentration. The lower portion of the column, usually termed the "enriching column" because it acts to enrich heavy isotopes, operates at ratio λ_E. For this part of the column, $G \approx L$ and the ratio λ_E is always about 1 [$\lambda_E = G/(L_{HTO} + L_{H_2O})$ with $G \approx L_{HTO} + L_{H_2O}$]. Figure 3 shows that feeding tritiated feedwater to the LPCE column allows a much higher deuterium concentration to be reached at the bottom of the LPCE column than in the case of tritiated water fed directly to the electrolyzer.

When the WDF processes tritiated water having deuterium levels exceeding that in natural water, the deute-
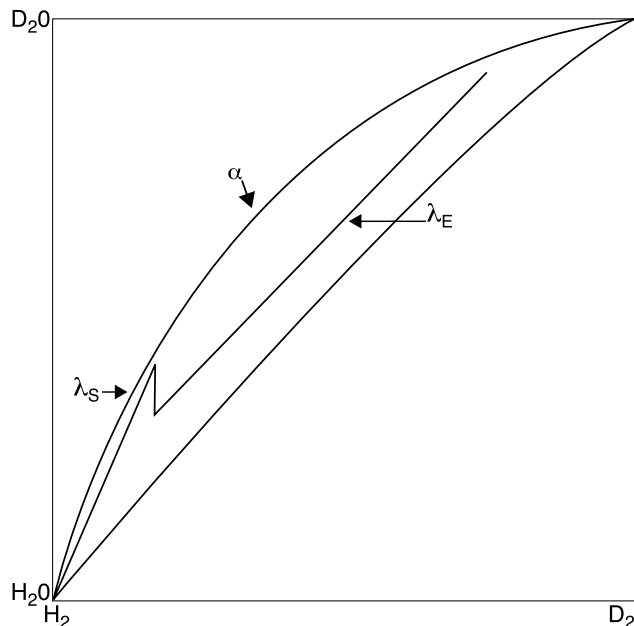
rium will accumulate in the same way tritium does. This will affect the performance of both the LPCE column and the electrolyzer as tritium separation factors for both units decrease with the amount of deuterium accumulated. As illustrated in Figs. 2 and 3, the impact depends mostly on the mode of WDF feeding with tritiated water.

The deuterium profile in the LPCE column depends on the correlation between equilibrium separation factor α_D and ratio λ. This is illustrated by way of a simplified example in Fig. 4, in which the equilibrium line is shown as a straight line. Figure 4a illustrates the case where $\lambda < \alpha_D$, in which deuterium concentration decreases at the bottom of the column faster than at the top. For $\lambda = \alpha_D$ (Fig. 4b), the deuterium concentration changes linearly from the bottom to the top, and for $\lambda > \alpha_D$ (Fig. 4c), the deuterium concentration changes more quickly at the top of the column than at the bottom. This shows that by increasing λ, the part of the column containing water
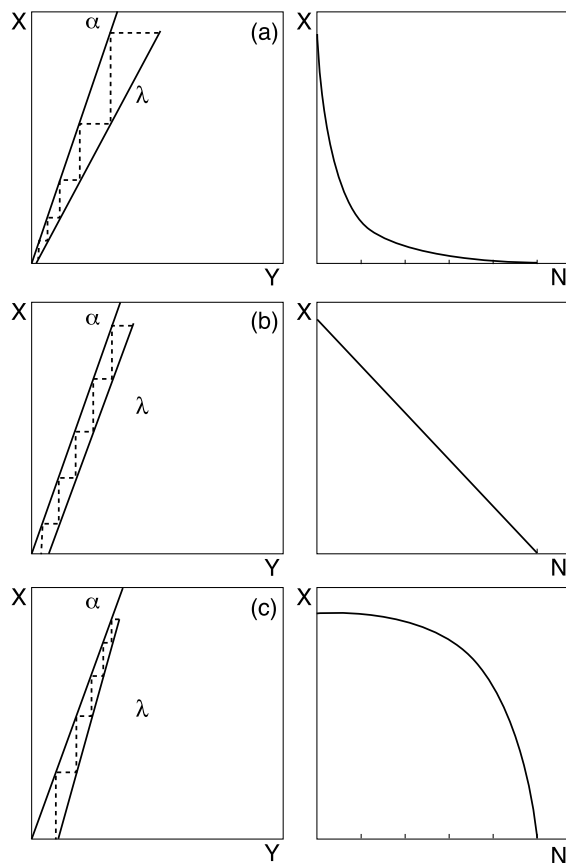

Fig. 4. Simplified illustration of the effect of the ratio between equilibrium separation factor α_D and ratio λ on the deuterium profile in the LPCE column: (a) $\lambda < \alpha_D$, (b) $\lambda = \alpha_D$, and (c) $\lambda > \alpha_D$. X is the deuterium concentration in the water, Y is the deuterium concentration in the hydrogen, and N is the number of theoretical plates counted from the bottom to the top of the LPCE column.

Fig. 3. McCabe-Thiele diagram for separation of protium-deuterium mixture in LPCE column with tritiated water fed to the column.

above the normal level of deuterium concentration will rise, and the impact of this together with increased deuterium concentration in the feedwater will reduce the effectiveness of tritium removal in the LPCE column: The deuterium profile in the column will determine α_T.

A rise of λ increases the rate of tritiated water processing [because $\lambda = G/L_{H_2O} = (L_{H_2O} + L_{HTO})/L_{H_2O} = 1 + L_{HTO}/L_{H_2O}$]. However, the rise in ratio L_{HTO}/L_{H_2O} will give a noticeable decrease in the decontamination factor for the LPCE column.[2] This indicates that optimized operating parameters will differ when processing water of low and high tritium concentrations through the LPCE column.

The concept evaluation presented here identified a need for experimental trials to validate the packing arrangement for the LPCE column, the use of a VPCE reactor for tritium enrichment in hydrogen to be fed to the ISS, and other issues.

III. EXPERIMENTAL TRIALS

The experimental trials included performance testing of various hydrophobic catalysts and packing arrangements for the LPCE column, testing the efficiency of a hydrophilic catalyst to be used in the VPCE reactor, and testing a method for catalytic conversion of hydrogen to water for the purpose of discharge.

III.A. LPCE Column

A noticeable discrepancy between the observed and predicted performances had been reported[4,5] for LPCE columns filled with catalyst mixed with random packing material (packing of the first type referred previously); therefore, the effectiveness of LPCE column operation was studied using catalysts representing two basic types of hydrophobic catalyst. The experimental procedure and

the results are given in Ref. 14. The catalyst of the first type was hydrophilic by nature but was made hydrophobic by additional treatment[3,7]: The catalyst was prepared by coating platinum on charcoal and was made hydrophobic by mixing with polytetrafluoroethylene (PTFE). The mixture was formed into a flat matrix from which cylindrically shaped pellets measuring 2 mm × 2 mm were cut. The catalyst of the second type was hydrophobic by nature[5,22,23] and was prepared by coating platinum onto spherically shaped styrene-divinylbenzene copolymer. Both catalysts were mixed with inert packing in the shape of mini spirals made of stainless steel with the surface acid etched to increase its wetting properties for water. Both tested catalysts performed according to the predictions. For a hydrogen flow of 10 mol/m$^2 \cdot$s, height equivalent to theoretical plate (HETP) was measured to be in the range 0.2 to 0.3 m for λ from 2 to 4 (Ref. 14).

The effect of increased deuterium concentration on LPCE column performance was studied using a catalyst of the second type, based on styrene-divinylbenzene copolymer. Parameters characterizing the rate of mass transfer, such as height of mass transfer unit (HTU) and overall rate of mass transfer (K_G) (Ref. 15), were evaluated together with HETP. The experiments were performed with feedwater containing 74 kBq/kg of tritium and about 10 at.% of deuterium. The experimental procedure is described in Refs. 14 and 24. The results given in Table VI allow the following conclusions to be drawn: At hydrogen flow rates above 6 mol/m$^2 \cdot$s, $K_T \approx K_D$. Decreasing λ at constant G (achieved by raising the water load to the column) led to an increase in K_G for both tritium (K_T) and deuterium (K_D). Increasing G from 10 to 20 mol/m$^2 \cdot$s led to a rise of HETP and HTU; however, the HTU and HETP for tritium and deuterium remained equal. The difference in K_G for tritium and deuterium become more pronounced with hydrogen flow rate decreased below 6 mol/m$^2 \cdot$s; the explanation for these observations is

TABLE VI

Results of Evaluation of HTU, HETP, and K_G*

Hydrogen Flow Rate G (mol/m$^2 \cdot$s)	λ	HETP (m)		HTU (m)		Overall Rate of Mass Transfer K (mol/m$^2 \cdot$s)	
		h_T	h_D	H_T	H_D	K_T	K_D
2.0	2	0.5	0.18	0.33	0.15	6.25	13.4
3.8	2	0.48	0.17	0.32	0.14	12.0	27.2
6.0	2	0.32	0.18	0.21	0.15	29.0	40.2
8.0	4	0.20	0.17	0.18	0.20	44.6	40.2
10.0	1	0.20	0.17	0.10	0.10	100.9	100.9
10.0	4	0.20	0.16	0.18	0.19	55.8	53.1
20.0	4	0.42	0.33	0.38	0.38	53.1	53.1

*From Ref. 24.

given in Ref. 24. The negligible effect of deuterium presence on the rate of tritium mass transfer may be a specific feature of the type of catalyst[5,22,23] tested. Catalysts of other types might behave differently; some of them show a decrease in the tritium mass transfer rate[3,25] in the presence of deuterium.

LPCE column packing based on a mixture of hydrophobic catalyst and inert random packing material exhibited a high separation capability, although the throughput of columns with this type of packing is limited to ~20 mol/m²·s (Ref. 14). Embedding a hydrophobic catalyst into a structured packing material may bring a benefit from its large throughput of structured packing material; such an arrangement was tested with both of the hydrophobic catalysts mentioned previously using Katapak structured packing material manufactured by Sulzer Chemtech, Winterthur, Switzerland, as a matrix for the catalyst. This stainless steel packing, surface treated to improve water wetting and then loaded with the hydrophobic catalyst, allows the LPCE column to operate at a very large gas flow rate without becoming flooded. However, the measured mass transfer rate (Table VII) for this arrangement was found to be about an order of magnitude smaller than that for the same catalyst mixed with a random inert packing material (see Table VI).

III.B. VPCE Reactor

The performance of a VPCE reactor for hydrogen enrichment with tritium has been tested using a commercial platinum catalyst coated on alumina. For a molar ratio of 4 for water vapor over hydrogen flow rates and at a temperature of 573 K, the catalyst demonstrated the same rate constant of tritium isotopic exchange of $4.4 \pm 0.15 \ s^{-1}$ in the range of gas velocity 0.06 to 0.6 m/s. This allows evaluation of the length of catalyst bed needed to provide a given approach to isotopic equilibrium between hydrogen and water vapor. For example, to achieve a 95% approach to the isotopic equilibrium, the VPCE reactor should be designed to provide reaction time of 0.675 s. It is also worth noting that at the same operation conditions, the rate constant for deuterium isotopic exchange of $6.4 \pm 0.2 \ s^{-1}$ was observed, which is greater than that for tritium.

III.C. Oxidation of Hydrogen to be Discharged

The hydrogen, depleted with tritium in the LPCE column, can be discharged to atmosphere. Because hydrogen can form an explosive mixture with air, the discharge must be performed employing engineering solutions that prevent explosion. The simplest way to do this is by fast mixing with air to reduce the hydrogen concentration to below the low explosion limit of 4 vol%. However, the mixing procedure still presents a possibil-

ity for explosion, so flame arresters of various types are usually added for preventing hydrogen explosion in WDFs (Refs. 10 and 12). Flame arresters constrain flame propagation back to the point of hydrogen generation and limit the extent of potential damage to the relevant equipment, but they do not eliminate the risk of damage. A possible solution for improving the safety of hydrogen discharge is to use catalytic conversion to water vapor in reaction with either oxygen or air. This conversion can be performed using either a hydrophilic[17] or hydrophobic[18] catalyst. Hydrogen oxidation on a hydrophilic catalyst is well-proven technology in the chemical industry, such as that employed in hydrogen and oxygen purification devices of electrolyzers. Oxidation of hydrogen in reaction with air will cause an air temperature rise of ~85 K per each volume percent of hydrogen in the air. Some preheating of the catalyst might be needed at the beginning of the process for the reaction to start. For air at a temperature of 293 K and having 50% relative humidity, oxidation of 1 vol% hydrogen would result in the air being almost saturated with water vapor. If hydrogen is mixed with air at concentrations above 1 vol%, measures have to be taken to prevent vapor condensation on the way to the discharge point.

Hydrogen oxidation in reaction with oxygen on a hydrophobic catalyst (the same as for the LPCE column) was demonstrated[18] to be very efficient. Experimental tests[26] with hydrophobic catalyst of the second type and random inert material loaded into a compact reactor show that hydrogen can be oxidized using a compact catalytic reactor to a residual content below 2 ppm. A rate constant

TABLE VII

Mass Transfer Rate of Tritium for Structured Packing Katapak with Embedded Hydrophobic Catalyst

T (K)	G (mol/m²·s)	λ	HETP (m)	HTU (m)	K_G (mol/m³·s)
Catalyst of First Type					
335	8.2	2.6	2.2	1.6	5.1
335	8.2	7.9	1.3	1.7	4.8
335	8.2	9.5	1.8	2.5	3.3
333	14.5	5.12	5.3	5.3	2.7
333	20.9	5.8	5.9	6.2	3.3
335	20.9	6.8	6.2	7.2	2.8
Catalyst of Second Type					
335	8.2	4.2	1.1	1.0	8.2
335	8.2	7.2	1.0	1.2	6.8
333	14.5	5.1	1.6	1.6	8.9
335	20.9	3.7	4.5	4.8	4.9
303	8.2	3.1	3.2	2.3	3.6
314	20.9	3.7	5.0	4.0	5.1

for hydrogen oxidation reaction of 16 ± 2 s^{-1} was observed for this reactor. The catalyst needs to be prevented from overheating, which is usually arranged through a contact with liquid water loaded through the top of the reactor.

IV. DESIGN OF THE FACILITY

Evaluation of concept and the experimental trials have allowed detailed design of a WDF for JET to be completed as an option for processing on-site. The design package included supply and purification of tritiated and tritium-free waters, electrolyzer, LPCE column, and hydrogen recombiner for its discharge. Connections between the WDF and the ISS were incorporated for the transfer of tritium to the ISS and the return of tritium-depleted hydrogen from the ISS to the LPCE column.

Several commercial alkali and SPE electrolyzers were evaluated, and an SPE electrolyzer was identified as a preferred option since there is no possibility of poisoning the catalyst in the LPCE column by KOH and it also has a small water inventory. One manufacturer offers the cell stack immersed in a container filled with pressurized water and acting as a secondary containment for tritiated gaseous hydrogen and water, eliminating completely the possibility for tritium to be released from the cell stack to the environment. Leakage can be detected through monitoring the tritium concentration in the water of the container. Electrolyzers from other manufacturers would need either a purpose-built secondary containment around the cell stack or placement of the stack into a glovebox.

The selection of packing arrangement was based on the results of the experimental trials. The structured packing with embedded hydrophobic catalyst exhibited an unacceptably low rate of mass transfer. Therefore, selection was to be made between a mixture of the catalyst with random packing and catalyst and packing loaded separately into modules. Both arrangements demonstrated similar rates for mass transfer.[10,14] An electrolyzer with a hydrogen production rate of 10 Nm^3/h (446 mol/h) selected for the JET WDF would require an LPCE column of 0.1-m diameter operated at a gas flow of 15 mol/m$^2 \cdot$s. Columns of such diameter with random packing of the first type were already successfully operated in WDFs (Refs. 5, 7, and 8). This type of packing arrangement has been selected, therefore, because it is easy to manufacture and load the column. The type of hydrophobic catalyst and inert packing material have not been identified at this stage. For the design of the LPCE column, it was assumed that an HETP will be ~0.4 m (this value is larger than the 0.3 m observed in the trials) and independent of deuterium concentration in the water.

An important parameter that affects the operation of the LPCE column, the electrolyzer, and the ISS is the mode of tritiated water feeding to the WDF. As mentioned, the changing isotopic composition of the tritiated feedwater would require moving the feeding point along the LPCE column to reduce disturbance of the isotope profiles. In the column packed with a mixture of catalyst and random packing, this could be arranged by splitting the column into sections and providing means of water feeding between sections. This limits changing feed points to large steps only. The presence of deuterium in the JET water makes optimized selection of the feeding point difficult. Software programs and engineering means for doing this would have to be developed and experimentally validated. Therefore, feeding of the tritiated water directly to the electrolyzer loop has been selected. This option has an additional benefit described in Sec. II.E. Determination of the length of the packed bed for the LPCE column was based on a referred ratio of feed rate of tritiated water to tritium-free water (L_{HTO}/L_{H_2O}) of 1. This ratio determines the rate of tritiated water processing, the decontamination factor achievable in the LPCE column, and the maximum deuterium concentration in the hydrogen to be fed to the ISS. The ratio L_{HTO}/L_{H_2O} can be changed during the course of the WDF operation to optimize its performance together with that of the ISS.

To be reused at JET, tritium recovered from the water waste in the WDF would need to be enriched to isotopic concentration of $\geq 98\%$. Two options for tritium final enrichment have been described previously. Option A, based on employing the CD ISS of the AGHS, has been chosen for the JET WDF. Rationale for this was that this option covers processing all stocks listed in Table I (see case I in Table III) without investing in building a new CD column. In addition, it minimizes disruption to the AGHS operation, which would be unavoidable for construction of the new column and its connection to the AGHS.

The block diagram of a candidate WDF for JET is depicted in Fig. 5. Tritiated feedwater is supplied from 200-ℓ drums to the feedwater purification system. Purified tritiated water is then fed to the electrolyzer (EL1) loop. Operation of the electrolyzer together with the LPCE column leads to the accumulation of tritium and deuterium in the electrolyzer loop. It is intended that the electrolyzer will operate with tritium concentrations up to 1850 GBq/kg. The LPCE column is designed for a nominal ratio $L_{HTO}/L_{H_2O} = 1$ (equivalent to $\lambda = 2$); however, during the time tritium is accumulating in the electrolyzer and the tritium concentration is much below 1850 GBq/kg, the LPCE column can operate with a smaller DF, and the ratio λ can be maintained greater than 2. This allows a larger flow of tritiated water to be processed and gives the WDF a spare throughput capacity. The maximum deuterium concentration in the water flowing from the LPCE column to the electrolyzer would be limited to ~45 at.% for $\lambda = 2$, or smaller for $\lambda > 2$ (see Sec. II.E). Design parameters for the LPCE column are listed in Table VIII. The selected type of packed bed requires
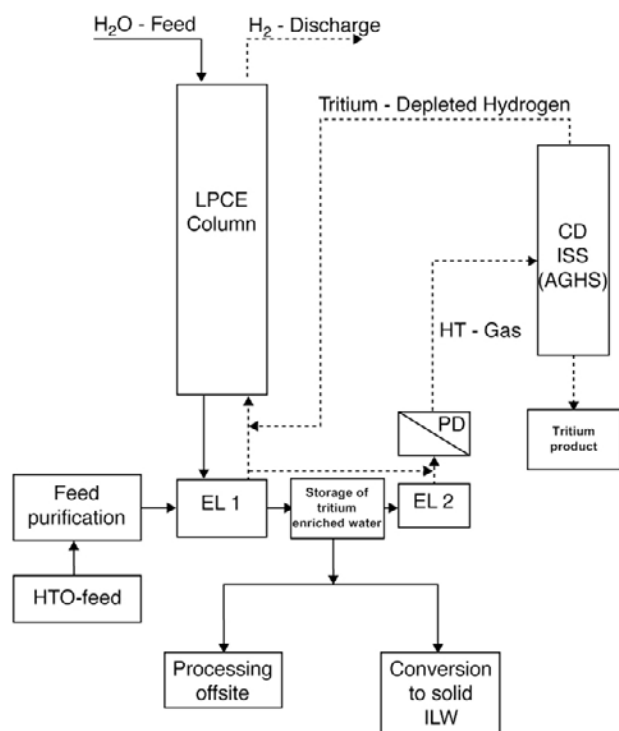
Fig. 5. Block diagram of the WDF for JET.

TABLE VIII

Design Parameters of LPCE Column

Parameter	Value
Total height of packed bed[a] (m)	7.6
Number of sections	6
Height of packed bed per section (m)	1.3
Column diameter (m)	0.13
Total volume of packed bed (dm^3)	100
Total volume of catalyst[b] (dm^3)	25 ÷ 33
Total volume of inert packing (dm^3)	67 ÷ 75
Hydrogen flow rate ($mol/m^2 \cdot s$)	10

[a]The height of the packed bed was evaluated for a maximum allowable tritium concentration of 3.1×10^{-5} g-at. T/mol in the electrolyzer and a concentration of 1.7×10^{-14} g-at. T/mol in the hydrogen discharge. The latter concentration allows the conversion of this hydrogen to water, with its free release from the JET site.

[b]A ratio of the inert packing volume to the catalyst volume was assumed in the range from 2:1 to 3:1.

water to be redistributed on regular intervals along the length of the column, and this has been accommodated by dividing the column into six sections connected in series. Each section will have access to the column internals through the top flange. This enables the catalyst

and packing in any section to be withdrawn and replaced, if that becomes necessary during the many years the LPCE column is to be in service, without having to dismantle the whole column.

Once a predetermined tritium concentration has been reached in the electrolyzer, either feeding of tritium-enriched hydrogen from the electrolyzer to the CD ISS can be started or tritium-enriched water can be withdrawn to the storage tank. For the CD ISS operation together with the WDF, the isotopic composition of the hydrogen feed to the ISS will vary. Withdrawing tritium-enriched water from the electrolyzer to the storage tank makes the WDF and CD ISS independent of each other and gives three possible options for dealing with this water:

1. Process it off-site.

2. Convert it to solid intermediate-level waste for disposal (case II in Table III illustrates the range of water quantities that would need to be treated).

3. Continue processing it on-site for complete tritium recovery.

The selection among options will be based on availability and cost effectiveness. While the first two are straightforward, the third involves transferring tritium-enriched water to the CD ISS via a second electrolyzer (EL2). This electrolyzer should be of the SPE type with a nominal hydrogen production rate of ~10 mol/h. The flow rate and isotopic composition of the hydrogen fed to the CD ISS will then be constant.

A possible upgrade (not shown in Fig. 5) is to incorporate a VPCE reactor and boiler into the water loop and hydrogen supply line of the second electrolyzer.[20] The VPCE reactor will then supply hydrogen to the palladium permeator and return condensed water to the electrolyzer water loop. This will reduce either treatment time or load on the CD ISS.

V. CONCLUSIONS

A design for a WDF for JET has been developed that allows the processing of water having a wide range of isotopic compositions in respect to both tritium and deuterium. The selected method of feeding tritiated water to the WDF is simple and does not require analysis of water waste isotopic composition and evaluation of isotopic profiles in the LPCE column as a part of the feeding procedure. This mode of feeding also allows control of the negative effect on the LPCE column, electrolyzer, and ISS connected to the WDF caused by the presence of deuterium in the tritiated feedwater. The WDF can be manufactured and upgraded in steps, depending on the requirements for final processing. The first step is the WDF itself, which allows the water feed stock to be

reduced to a small volume of tritium-enriched water. There would then be three possible options for dealing with this water; selection will be dictated by availability and cost effectiveness. The option of complete tritium recovery on-site would need the WDF to be upgraded and connected to the CD ISS of the JET AGHS. Operations of the WDF and ISS can be separated in time by withdrawing tritium-enriched water product into a storage tank and using a second electrolyzer of a small scale to convert this water to hydrogen to be fed to the ISS. This makes the WDF and the ISS operations almost independent of each other. For stocks of water having a low tritium concentration, the WDF would operate most of the time on its own without the ISS being involved. The time needed by the ISS to process tritium-enriched water can be reduced by enriching hydrogen produced by the second electrolyzer with tritium in a VPCE reactor.

ACKNOWLEDGMENT

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