Forschungszentrum Karlsruhe
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(Subtask WP-B 6.1.1 of the European Blanket Program 1996)

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Abstract

Ten different methods for the extraction of tritium from the purge gas of a ceramic blanket are described and evaluated with respect to their applicability for ITER and DEMO. The methods are based on the conditions that the purge gas is composed of helium with an addition of up to 0.1 % of H_2 or O_2 and H_2O to facilitate the release of tritium, and that tritium occurs in the purge gas in two main chemical forms, i.e. HT and HTO. Individual process steps of many methods are identical; in particular, the application of cold traps, molecular sieve beds, and diffusors are proposed in several cases. Differences between the methods arise mainly from the ways in which various process steps are combined and from the operating conditions which are chosen with respect to temperature and pressure. Up to now, none of the methods has been demonstrated to be reliably applicable for the purge gas conditions foreseen for the operation of an ITER blanket test module (or larger ceramic blanket designs such as for DEMO). These conditions are characterized by very high gas flow rates and extremely low concentrations of HT and HTO. Therefore, a proposal has been made (FZK concept) which is expected to have the best potential for applicability to ITER and DEMO and to incorporate the smallest development risk. In this concept, the extraction of tritium and excess hydrogen is accomplished by using a cold trap for freezing out HTO/H_2O and a 5A molecular sieve bed for the adsorption of HT/H_2.

Kurzfassung

Verfahren zur Tritium Extraktion aus einem Feststoff-Brüterblanket

Es werden zehn verschiedene Verfahren zur Tritium-Extraktion aus dem Spülgas eines keramischen Brüterblankets beschrieben und bzgl. ihrer Anwendbarkeit für ITER und DEMO diskutiert. Bei allen beschriebenen Verfahren besteht das Spülgas aus Helium, und es enthält Zusätze von bis zu 0.1% H_2 oder O_2 + H_2O, um die Freisetzung des Tritiums zu verbessern. Dieses liegt dann hauptsächlich in Form von HT und HTO im Spülgas vor. Einzelne Verfahrensschritte vieler Methoden sind identisch; besonders die Anwendung von Kaltfallen, Molekularsieb-Betten und Permeatoren wird in mehreren Verfahren vorgeschlagen. Die Unterschiede ergeben sich vor allem aus der Kombination der einzelnen Verfahrensschritte und aus der Wahl der Randbedingungen für Temperatur und Druck. Eine zuverlässige Anwendbarkeit für die Betriebs-Bedingungen eines ITER Blanket Testmoduls (oder für noch größere Blanket-Designs wie z.B. bei DEMO) konnte bis jetzt noch für kein Verfahren demonstriert werden. Diese Bedingungen sind nämlich durch sehr hohe Gasdurchsätze und extrem niedrige Konzentrationen an HT und HTO gekennzeichnet. Es wurde daher ein weiteres Verfahren vorgeschlagen (FZK-Konzept), von dem ein sehr gutes Anwendungspotential für ITER und DEMO zu erwarten ist und das gleichzeitig ein sehr geringes Entwicklungs-Risiko beinhaltet. In diesem Verfahren werden eine Kaltfalle zum Ausfrieren von HTO/ H_2O und ein 5A-Molekularsieb-Bett zur Adsorption von HT bzw. H_2 eingesetzt.

Table of Contents

Summary

Introduction

The tritium extraction system is an essential part of the fuel cycle of each fusion machine equipped with a solid or liquid breeder blanket. Tritium is produced in the blanket by the nuclear reaction of neutrons emitted from the plasma vessel with the lithium atoms (^6Li) contained in the breeder material. The case considered here is a solid breeder blanket consisting e. g. of Li_4SiO_4 pebbles, where the tritium is purged out by a helium gas stream containing up to 0.1 % of H_2 (or an equivalent addition of O_2 and H_2O) to facilitate the release by isotopic exchange.

It is then the task of the purge gas loop (this term is equivalent to "tritium extraction system") to

- separate tritium from the helium purge gas,
- to remove any solid or gaseous impurities, and
- to recondition the purified gas for further use as purge gas.

As tritium is expected to occur in two chemical forms, i.e. as HT and HTO, it is also necessary to carry out a chemical decomposition prior to sending H_2/HT to the Isotope Separation System (ISS).

In principle, the extraction of tritium from the purge gas can be achieved in two ways:

I By removal of HT and HTO in two separate steps with subsequent chemical reduction of HTO to HT, or

II By oxidation of all HT to HTO, followed by its removal from the purge gas and chemical reduction.

The first way appears to be more straightforward and economically effective because any additional production of HTO is avoided. The second way is relevant if it is intended to minimize the permeation of tritium into the blanket coolant by adding oxygen and water to the purge gas instead of hydrogen. In this case, however, the amount of tritiated water to be processed is about 100 times larger than in the first case.

Considerable technical difficulties are involved in the extraction procedure, because the projected purge gas flow rates are quite large (ITER blanket test module: ≥ 20 Nm³/h, DEMO blanket : 12 000 Nm³/h), and because the concentrations of the tritium species are of the order of a few ppm which corresponds to partial pressures less than 1 Pa.

The present report gives a description and evaluation of the technical proposals for the tritium extraction published during the past 10 years. As a conclusion of this evaluation, an a proposal is made (FZK concept) which is expected to have the best potential to be reliably applicable for an ITER test blanket test module as well as later on for a DEMO solid breeder blanket.

1. PSA Method

Principle /1,2/:

Separation of hydrogen from helium is achieved by taking advantage of the different adsorption behavior of both gases on a 5A molecular sieve (MS) bed at 77 K and by performing a fast loading / unloading cycle with adsorption (loading) under high pressure and desorption (unloading) under low pressure.

Two PSA columns containing the MS are operated simultaneously: the feed goes to one column while the other column is being evacuated for regeneration.

Process Description :

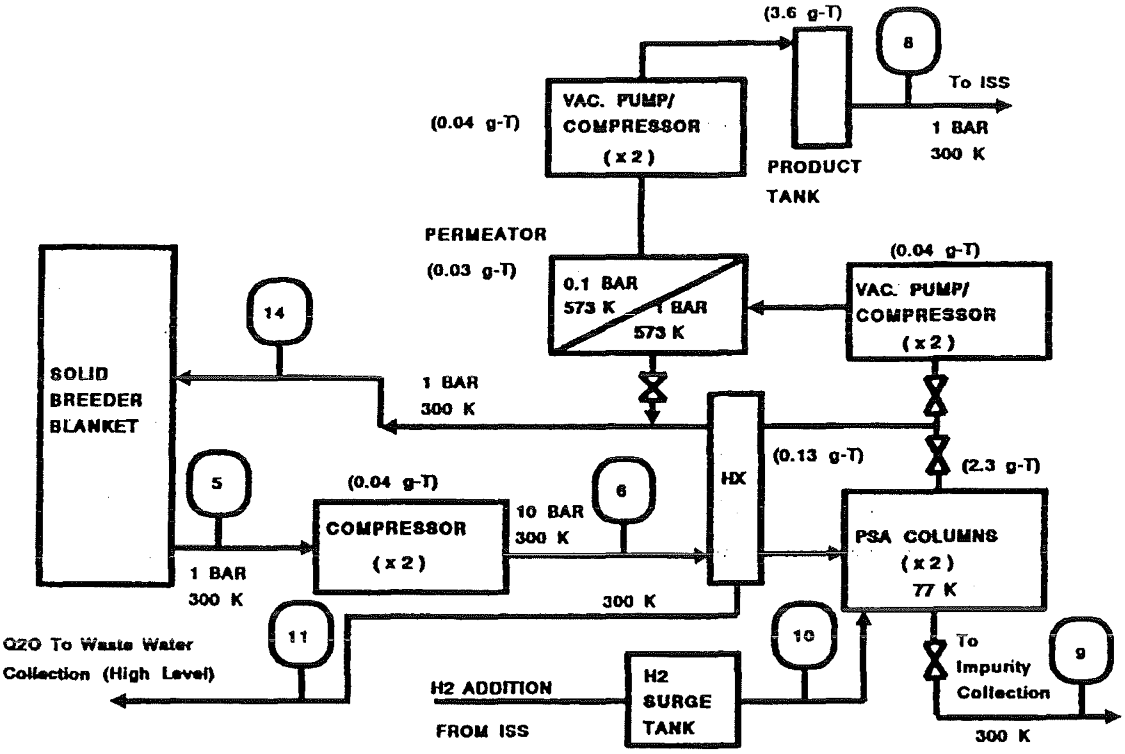

Fig. 1 : Block Diagram for the Blanket Tritium Recovery System /2/

1. Step: Gas compression (to 10 bar) and removal of moisture (precooler HX); the tritiated water is sent to the High Level Waste Water Collection System.
2. Step: Adsorption on the first PSA column at 10 bar pressure until complete break through; carry over of 45 % HT is allowed.
3. Step: First phase of depressurization / desorption by use of a vacuum pump; the pump exhaust containing mainly co-sorbed helium is recycled to the blanket.
4. Step: Second phase of desorption: evacuation to a pressure of 7.5 mbar; HT is pumped to the ISS via a compressor and a permeator to ensure that no impurities are carried over to the ISS; the bleed of the permeator is recycled to the blanket.

5. Step: Pure H_2 is introduced into the column to raise the hydrogen partial pressure to about 0.2 bar and to purge out any residual tritium from the bed.

6. Step: Repressurization of the column to 10 bar.

Characteristic Data of the Layout /2/:

a) Considered Processing Requirements:

Tritium breeding rate	479 g/d
Swamping ratio He : H_2	1000 : 1
H : T	20 : 1
Impuritiy concentration (N_2, CO, Ar, ...)	0.46 ppm (0.032 mol/ h)
Moisture (Q_2O)	9.94 ppm (0.7 mol/ h)
He flow rate	$6.99 \cdot 10^4$ mol / h

b) PSA Performance Data:

Increased He flow rate	$1.26 \cdot 10^5$ mol/h
Pumping speed for Step 3	775 Nm^3 /h
Amount of H_2 added in Step 5	986 mol/ h
Single path hydrogen recovery	55 %
No intermediate storage of Q_2	
PSA cycle time	10 min

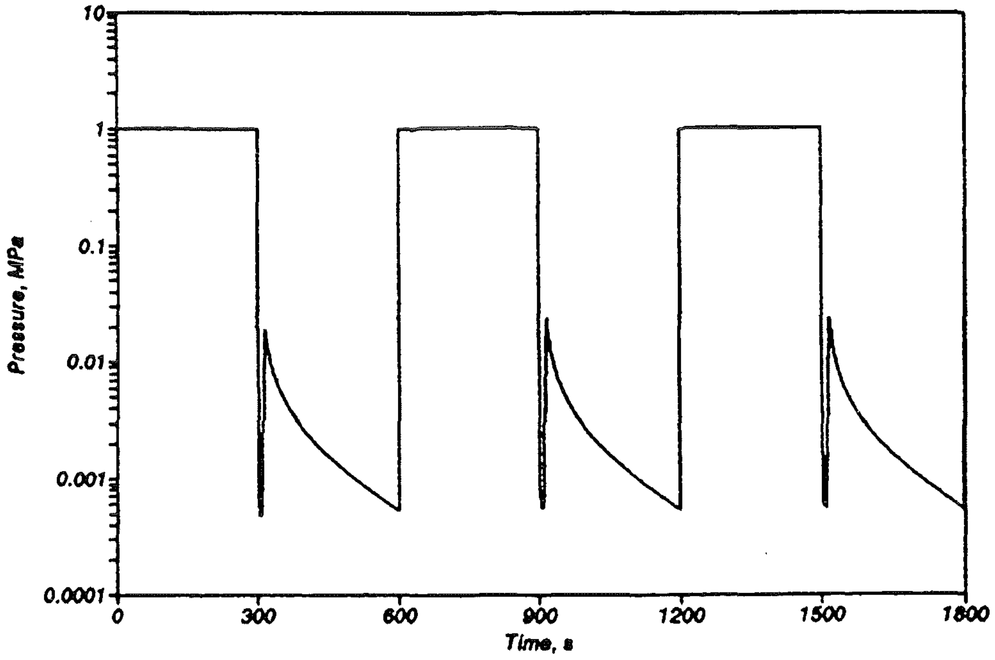

Fig. 2: PSA column pressure variation with time (one column) for 1 MPa feed pressure and 300 s adsorption time /1/

Advantages of the PSA-Method:

- Small tritium inventory because of a short cycle time (10 min),
- smaller molecular sieve beds than in comparable cryogenic methods in which a break through is not allowed.

Disadvantages:

- Increase of helium flow rate by 80 %,
- addition of 22 Nm^3 (986 mol / h) H_2; the amount of Q_2 to be processed by the ISS is thereby increased by a factor of 14,
- about 3.6 Nm^3 of almost pure hydrogen must be warmed up and sent through the permeator within 10 min (4. Step); this may be combined with techical and safety problems,
- no procedure is offered for the processing of tritiated water,
- complete regeneration of the molecular sieves including desorption of impurities will probably not be possible within the 10 min operation cycle.

2. TCPSA-Process

Principle: The Thermally Coupled Pressure Swing Adsorption (TCPSA) process is is a method for enriching a gas component (e.g. molecular hydrogen isotopes) in a carrier gas. It is similar to the Pressure Swing Adsorption (PSA) gas separation process, but uses a mechanism in analogy to Stirling cycle heat engines for generating the gas pressure swings with recovery of expansion energy.

A gas mixture flowing over an adsorbent column in one direction at high pressure will be depleted in its more readily adsorbed component, but flowing in the opposite direction at reduced pressure will be enriched in that component.

Process Description /3,4/

The compound separator is shown in Fig. 3 in a double-acting configuration with identical left and right sides operating 180° out of phase. On each side, a series of adsorbent columns (stepped in size) are connected in series along a flow path with the left and right working spaces of several cylinders. The pistons in these cylinders oscillate at the same frequency; the phase shift between the pistons at the upper and the lower ends of the figure generates a cyclically reversing flow through the adsorbent columns.

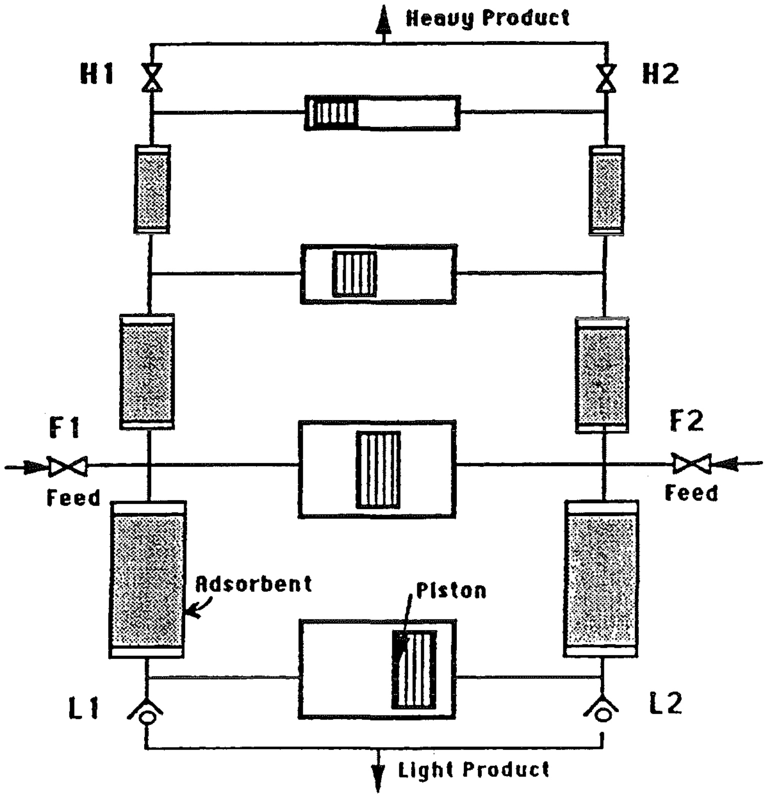

Fig. 3: TCPSA Compound Separator

A feed gas (e.g. helium as a light carrier gas with hydrogen isotopes and impurities as the "heavy" components) is admitted by valves F1 and F2. The piston at the bottom of the figure is reciprocated in leading phase relative to the smaller pistons towards the top of the figure, so that the gas depleted in the heavy component flows downward during higher pressure intervals, while the gas enriched in the heavy component flows upward during low pressure intervals. The purified carrier gas is delivered through valves L1 and L2, while the concentrated heavy component is delivered through valves H1 and H2.

To provide an hermetic enclosure for the tritium bearing gas species, the pistons of the compound separator must be driven by an external mechanical or hydraulic reciprocating drive transmitted through the cylinder walls by permanent magnet couplings. Linear electric motors, and mechnical or hydraulic drives isolated from the working space by metal bellows may be also considered.

Suitable adsorbent materials for different applications include zeolite molecular sieves, activated charcoal, or alumina gel. The gas separation may be carried out either at cryogenic or elevated temperatures.

A complete procedure for tritium recovery from blanket purge gas using the TCPSA method as one process step is described in /4/ and shown in Fig. 4.

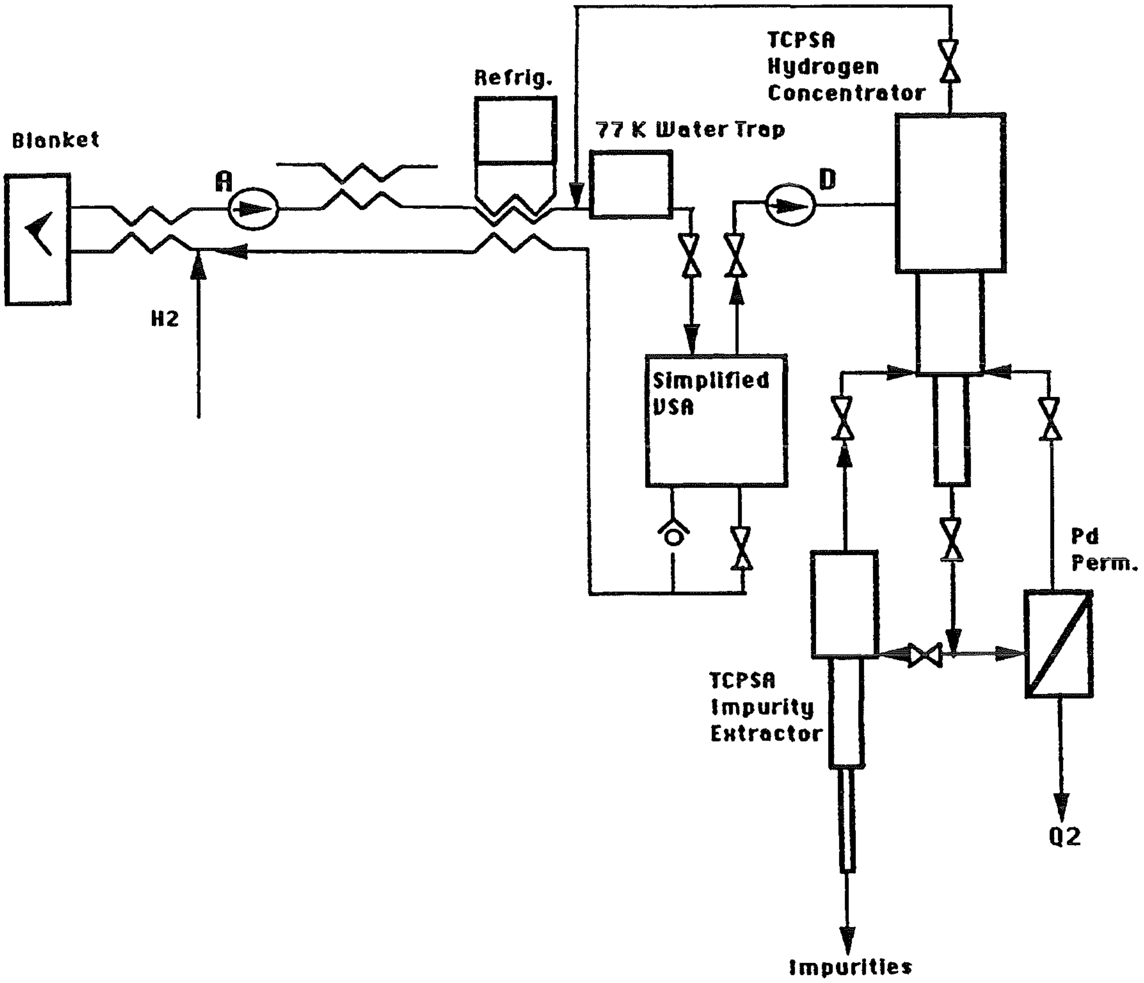

Fig. 4 : Blanket Tritium Recovery System using VSA, TCPSA, and Pd Diffusion

The procedure includes the following steps:

1. Cold trap removal of water,
2. Initial concentration of hydrogen by vacuum swing adsorption (VSA) at 77 K,
3. Removal of helium and impurities by TCPSA at 150 K,
4. Final purification by palladium diffusion prior to isotope separation.

The VSA system comprises multiple10X or 5A molecular sieve beds operated with an input pressure of 2 bar. Since 90 % Q_2 extraction from the purge gas appears to be sufficient, the VSA system is deliberately operated with some break through of the Q_2 mass transfer zone concentration wavefront into the helium product stream; nevertheless, removal of impurities and water vapour will still be complete.

VSA depressurization is carried out by using the vacuum pump D and a warm helium stream to enable displacement of the hydrogen isotopes from the adsorbent beds. A volume of 100 l is proposed for the adsorber bed along with a cycle time of 5 min (!) for a helium gas flow of 117,430 mol/h containing 0.1% Q_2. This condition leads to a tritium inventory of the whole process of only 1 g.

The feed stream to the TCPSA module includes helium and hydrogen from the VSA module as well as the recycle bleed gas from the Pd diffusor. Impurities contained in this bleed gas may be stripped by an additional TCPSA impurity extractor operating at about 300 K. The Q_2 depleted fraction of the main TCPSA module is retured to the VSA feed; the hydogen isotopes separated by the diffusor are transferred to the ISS.

Advantages of the TCPSA-Process:

- When high cycle speeds (>10 rpm) are used the TCPSA adsorbent columns and hence the tritium inventory can be relatively small,
- no need for hydrogen purging of adsorbent columns like in the PSA process,
- applicable to gas separation at various temperature levels,
- no large diffusor units required.

Disadvantages:

- Process needs to be optimized and tested with tritium,
- experience is limited to small scale laboratory experiments and cannot be considered as a technically proven process for ITER / DEMO,
- very frequent valve switching operations will be required as a consequence of the short cycle times of the VSA module and, in particular, of the TCPSA module.

Additional Comments:

- Cycle times and size of the VSA adsorber beds may be too small for large purge gas streams; as a temperature cycling is also involved in the operation of the adsorber beds, the questions of heat capacity, of heating and cooling, etc. should be addressed to define the most appropriate cycle times.
- If the TCPSA step is abandoned, the whole pocedure is simplified; however, the gas flow rates to the Pd diffusor will be larger, and an additional molecular sieve bed will be needed to collect the impurities.

3. ANL Tritium Recovery System

Principle:

Procession of two separate helium purge gas streams:

- one stream from the LiO_2 zones containing 0.1 % H_2 and
- one stream from the Be zones of the blanket containing no additional H_2;

The main components of the system are:

- cold traps (-100°C),
- 5A molecular sieves (-196°C and 25 °C),
- Pd/Ag permeator,
- solid oxide electrolysis cells.

Process Description /5/:

The concept has been developed for the U.S. LiO_2/Be water cooled ITER blanket. This blanket consists of a series of zones parallel to the first wall which contain water, LiO_2, and Be. Since the Be zones are immediately adjacent to coolant zones, an in-leakage of water into the beryllium appears to be probable. Thus, a steady state water partial pressure is assumed for the Be zones.

The main parameters for the tritium recovery system are:

H/T ratio at LiO_2 outlet	100		
Tritium flow rate, full power, g/day	150		
Protium flow rate, full power, g/day	5000		
Helium flow rate at LiO_2 outlet, g/day	10^7	=	2330 m^3 (NTP)/h
Helium flow rate at Be outlet, g/day	$1.33 \cdot 10^5$	=	31 m^3 (NTP)/h

Units to process the Gas from the LiO_2 Zones (see upper part of Fig. 5) :

- A set of cold traps to separate water (0.47 kg/d) and some CO_2 from He,
- Two circulators and a storage system to handle the helium gas,
- A small solid electrolysis unit to decompose the water,
- A set of 5A molecular sieves at -196°C (termed X1) to remove the hydrogen isotopes and all remaining impurities,
- A set of 5A molecular sieves at 25°C (termed Y1) to recover water, CO_2, and other impurities from the stream leaving the X1 molecular sieve during regeneration, and to remove entrained water from the hydrogen stream leaving the electrolysis cell,
- A Pd/Ag diffusor to separate inerts from the hydrogen isotopes prior to their transfer to the cryogenic distillation unit (ISS),
- A SAES getter bed held at 350°C to prevent active impurities from entering the ISS,
- Possibility to recycle the bleed gas of the diffusor by feed back to the cold molecular sieve X1.

Units to process the Gas from the Beryllium Zones:

- A set of cold traps to separate water from helium (the leak rate from the blanket coolant is assumed to be up to 10 kg water/d),
- A circulator and a storage system to handle the helium gas,
- Several linked solid oxide electrolysis units to decompose water,
- A set of 5A molecular sieves at -196°C (termed X2) to remove tritium and impurities,
- A set of molecular sieves at 25°C (termed Y2) to recover water, CO_2, and other impurities from the stream leaving the X2 molecular sieve during regeneration, and to remove entrained water from the hydrogen stream leaving the electrolysis units. The exit hydrogen species from the Y2 molecular sieves are routed to the Y1 sieve for recycle. The waste products are routed to the common waste recovery unit in which tritium is recovered as tritiated water while the non-tritiated waste is passed to the stack.

Operational Data of the Main Components

1. Warmup frequency of the cold traps:
   a) LiO_2 stream: every 2 hours
   b) Be stream without large water leakage: every 2 days
   Be stream with large water leakage: every 2 hours

2. Regeneration of molecular sieve beds
   a) Beds X1: 3 temperature steps
   -100°C / every 2 hours,
   25°C / once a week,
   250°C / once a month,
   + purging with H_2 to enhance desorption of HT and impurities;
   impurities are transferred to MS Y1,
   H_2 and HT are passed through Y1 to the diffusor.
   b) Beds X2: slow heatup to 250°C once a day,
   + purging with H_2,
   impurities are transferred to MS Y2,
   HT is passed to Y1.
   c) Beds Y1/Y2: heatup to 250°C every 12 hours,
   + purging with He,
   impurities are transferred to the Waste Disposal System.

3. Diffusor: The bleed gas is cycled back to MS X1 to be reprocessed;
   In the case of a membrane rupture, fast acting valves installed at the hydrogen exit of the diffusor will minimize the transport of helium to the cryodestillation unit.

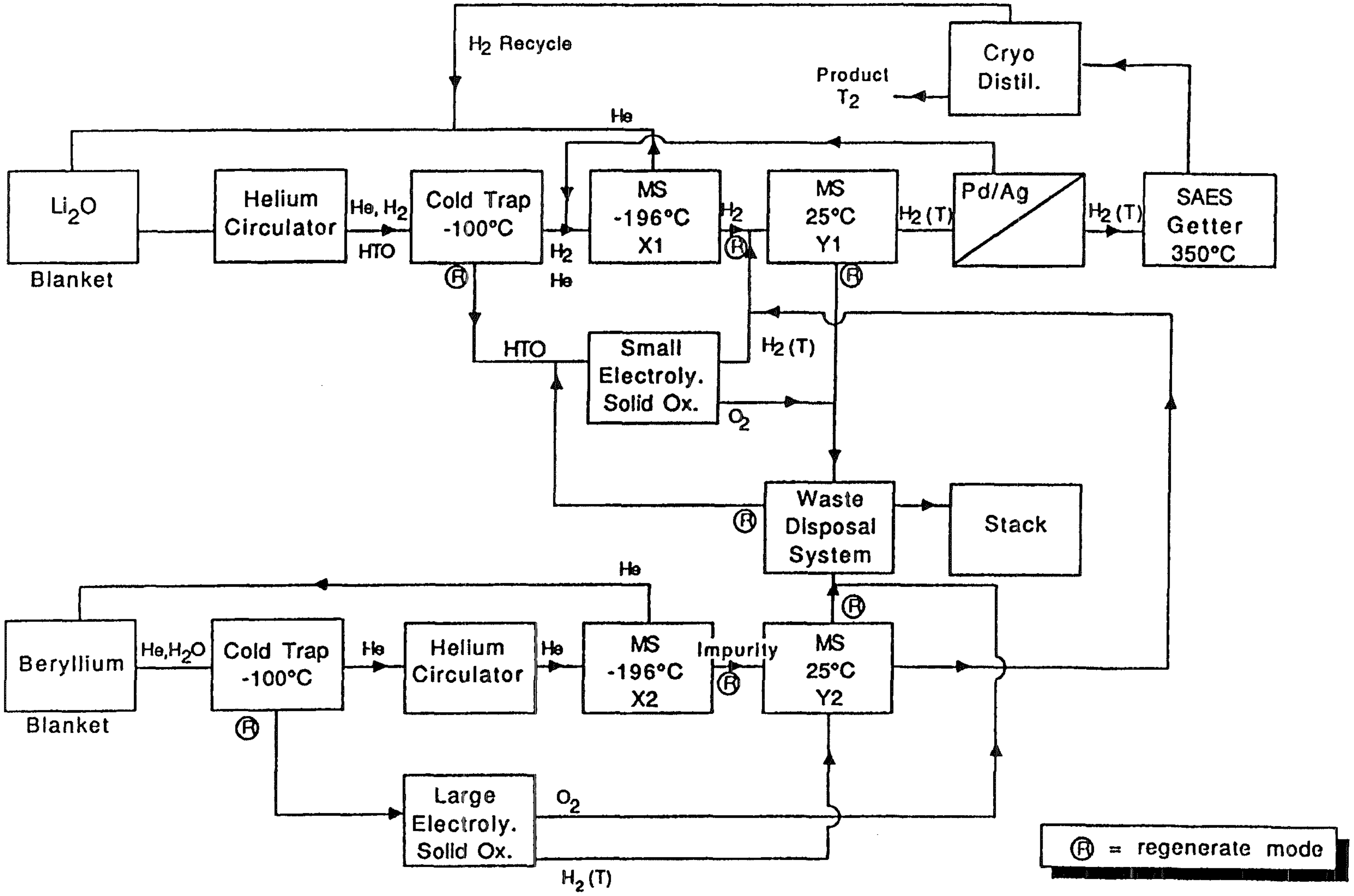

Fig. 5: Block Diagram for the ANL Tritium Recovery System

Comments

1. In the paper mentioned above /5/ several details of general importance for the lay out of a tritium recovery system are discussed. These items are summarized here along with the main conclusions:

   a) Specification and transport of impurities:

   Most of the impurities released from LiO_2 or Be are deposited in elemental form within the blanket. The remaining particulates are removed by filter units, which are cooled to 25°C and located in front of the helium circulators. It s recommended to use two parallel set of filters to collect medium size particles (10....100 μm) as well as very small particles ($\approx$ 1 μm).

   The species which pass the filters are CO_2, H_2O, and the radioactive species $S^{35}O_2$, Ar-39, Kr-85, Kr-87, Xe-133, and Xe-135.

   b) Massive water leak into the Be zone:

   The maximum in-leakage rate is assumed to be 10 kg water per day. This water will be tritium contaminated because of the tritium release from Be and isotope exchange. The expected activity of the water is $\leq$ 270 Ci/kg.

   c) Electrolysis cells:

   Three types of electrolysis cells, conventional wet electrolysis cells, solid polymer cells, and high temperature solid oxide cells were examined. Solid oxide cells were found to be the preferable option, since they have no liquid water inventory, they are not damaged by radiation, leave minimal waste, operate at high temperature (which is more efficient for electrolysis), and can completely seperate hydrogen and oxygen.

   Their main disadvantage is insufficient long-time experience for the operation of prototype cells with tritium.

2. Apart from the use of electrolysis cells and the two separate purge gas streams, the ANL procedure is very similar to the FZK concept.

3. A minor comment refers to the dimension of the molecular sieve beds X1: Complete removal of H_2 / HT during a 2-hour adsorption step cannot be obtained with 192 kg of 5A sieve material; this would require a capacity of 23.8 L (STP) per kg sieve material which is even higher than the H_2 saturation load at 1 mbar H_2 partial pressure /6/.

4. JAERI Methods

Option A: Molecular Sieve - Diffuser
Option B: Oxidizer - Molecular Sieve

Principle of Option A /7/:

Three removal steps are carried out in the primary loop:

- removal of water by a molecular sieve bed at 300 K,
- removal of hydrogen isotopes by several Pd diffusors,
- removal of impurities by a molecular sieve bed at 100 K.

Tritium recovery from water is obtained in a secondary loop by using either an electrolytic cell or a water gas shift reactor.

Basic Design Parameters:

Flow Rates	
He	10^4 mol/h
HT	2.08 mol/h
HTO	0.02 mol/h
H_2O	1.04 mol/h
H_2	105 mol/h
Impurities	0.6 mol/h (max.)
Tritium Breeding Rate	150 g/d
H : T	100
Isotopic Exchange Reactions:	
$T_2O + H_2 \rightarrow HT + HTO$	
$HTO + H_2 \rightarrow HT + H_2O$	

Process Description:

The procedure is shown in a simplified form in Fig. 6. Gas circulation in the primary loop is realized with two blowers of type Normetex 600. Two molecular sieve beds, alternately operated at 300 K remove the water content of the purge gas by a factor of 100. The beds containing 673 liters of zeolite 5A are unloaded (regenerated) at 572 K with a maximum cycle time of 50 days. The released water is then decomposed by two electrolizers which are operated at 972 K and subjected to a sweep gas flow rate of 1010 mol/h. The expected water reduction efficiency is 90%.

Seven Pd diffusors operating at 722 K and 0.9 bar, with a hydrogen recovery rate of 90% are used to extract the hydrogen isotopes which are directly sent to the ISS.

Impurity removal is achieved with a molecular sieve bed operated at 100 K with a maximum regeneration time of 25 days.

Additional technical specifications concerning the size of the components, local flow rates, gas temperatures, power of heater and cooler units, etc. are listed in /7/.

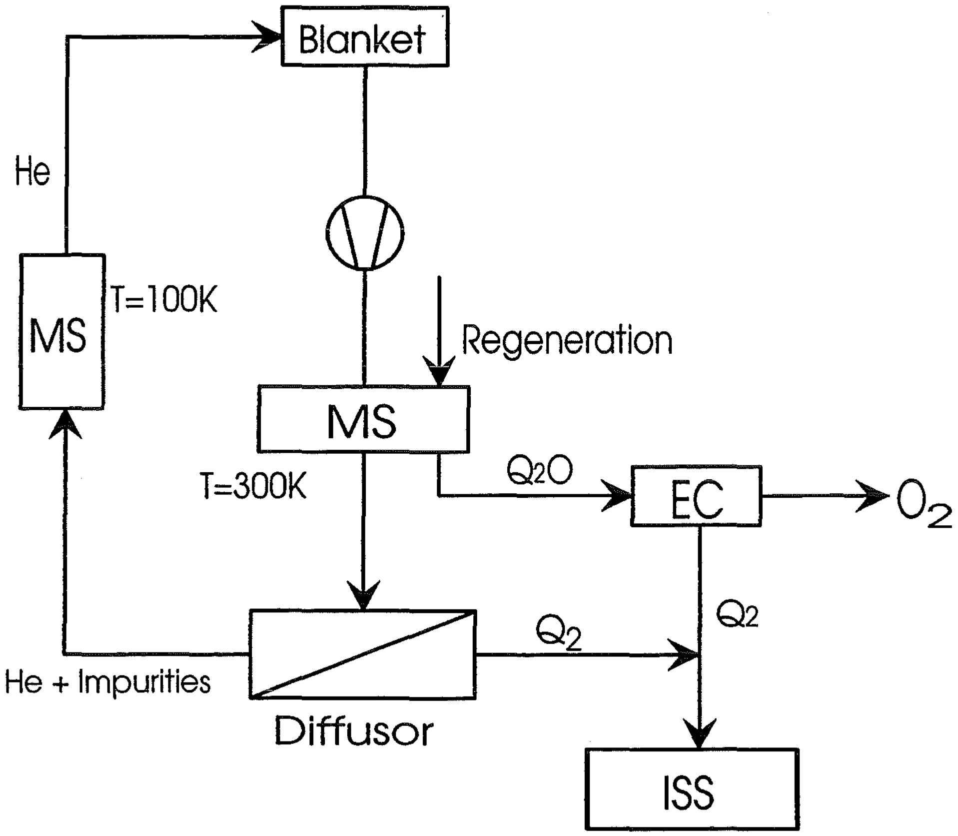

Fig. 6: Simplified Flow Diagram of the JAERI Option A; a water gas shift reactor is foreseen as a backup solution for the decomposition of water.
(MS = Molecular Sieve Bed, EC = Electrolytic Cell, ISS = Isotope Separation System)

Advantages of Option A:

Use of molecular sieves for removal of water and impurities represents a proven technology; the proposed temperatures are appropriate and can be realized with moderate energy consumption.

Disadvantages:

- Many diffuser units are needed for the Q_2 separation step due to the high gas flow rate and the fact that sufficiently large permeators are not commercially available; this aspect is even more severe for the larger purge gas flow rates of DEMO;
- Insufficient long-time experience with electrolytic cells; high operation temperature may cause tritium diffusion losses; preference should be given to the proposed backup solution (water gas shift reactor).

Principle of Option B /8/:

Hydrogen isotopes and impurities are oxidized prior to adsorption on a moleculear sieve bed; the primary purge gas is then directly retured to the blanket, see Fig. 7.

The moisture released from the MS during regeneration is reduced by using the water gas shift reaction. Finally, the hydrogen isotopes are separated by employing a Pd/Ag permeator.

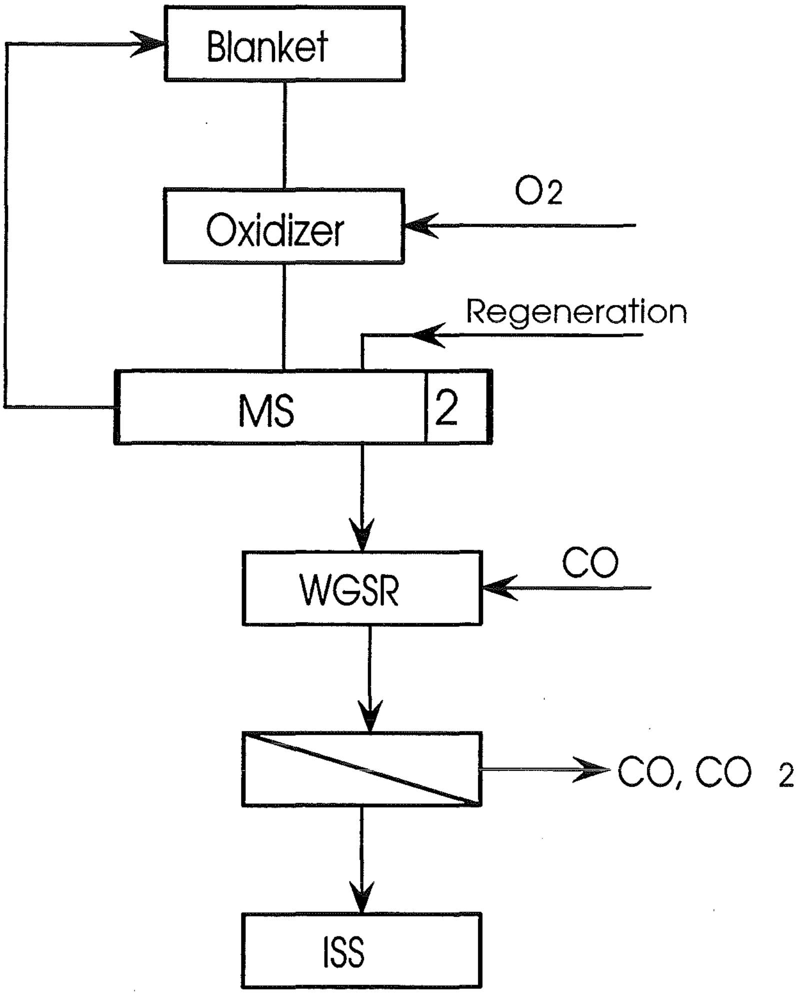

Fig. 7: Flow Diagram of JAERI Option B (MS = Molecular Sieve Bed, WGSR = Water Gas Shift Reactor, ISS = Isotope Separation System)

Comments:

Option B does not exhibit the severe disadvantages of Option A; it is equivalent to the ENEA Option A described in more detail in the next chapter.

5. ENEA Methods

Principle of Option A/B /9/:

All hydrogen isotopes of the purge gas are oxidized to water and separated from the gas by freezing out in a cryotrap or by adsorption on a molecular sieve. Cracking of the water is then achieved by using the water gas shift reaction; finally, the hydrogen isotopes are separated by means of a Pd/Ag diffusor.

The description given in /9/ starts with the processing of tritiated water. The water is contained in one of two alternately operated water tanks; one of them recieves condensed water from the oxidation section of the plant while the other feeds water to the water gas shift reactor (WGSR).

Process Description of Option A (see Fig. 8):

The water in the water tank is slowly evaporated, mixed with a pressurized CO stream (5 bar, excess CO), and then sent to the WGSR. The outlet stream from this unit contains the hydrogen isotopes, unreacted water, excess corbon monoxide, and the produced carbon dioxide. The hydrogen isotopes are extracted by means of a Pd/Ag diffusor and sent to the ISS.

The unpermeated stream is fed to a catalytic oxidizer to convert CO to CO_2, and Q_2 to Q_2O. The stream is then sent to a molecular sieve bed to remove the moisture. As the HTO concentration in the outlet gas is about 0.05 ppm, additional treatment is required before the gas can be released into the waste gas system. So the stream is sent to a second molecular sieve bed with water added for isotopic dilution, resulting in an outlet stream with negligible tritium concentration.

The molecular sieve beds are regenerated with warm CO gas which is later on transferred to the water tank and then used for the shift reaction.

Process Description of Option B (see Fig. 9):

The gas stream leaving the WGSR is cooled and dryed in a molecular sieve bed. It is then sent to a CO_2 adsorber (containing molecular sieve of a modified Y-modernite) to remove the carbon dioxide. In both cases, two sets of molecular sieve beds are needed because of the discontinuous operation (adsorption / regeneration mode). The last step is the separation of the hydrogen isotopes by a Pd/Ag diffusor.

The regeneration of the moisture adsorption beds is carried out with fresh CO gas which is added to the diffusor bleed gas. After leaving the adsorption bed, this gas is transferred to the water tank (like in Option A). The CO_2 adsorber bed is regenerated by heating and flushing with inert gas.

Remarks:

1. Two operation cycles are used in both concepts: In the first, CO gas is fed continuously to the WGSR to promote the shift reaction; in the second, the gas is recirculated through the diffusor until complete detritiation is obtained.
2. The main advantage of Option B is the CO_2 removal upstream of the diffusor; this leads to a smaller gas flow rate at the diffusor inlet and, hence, to a reduced size of the required permeation membrane.
3. The requirements of the purge gas oxidizer have not been discussed. In the case of DEMO, the conversion of $4.8 \cdot 10^3$ mol Q_2 into Q_2O requires about 50 m^3 O_2/day.

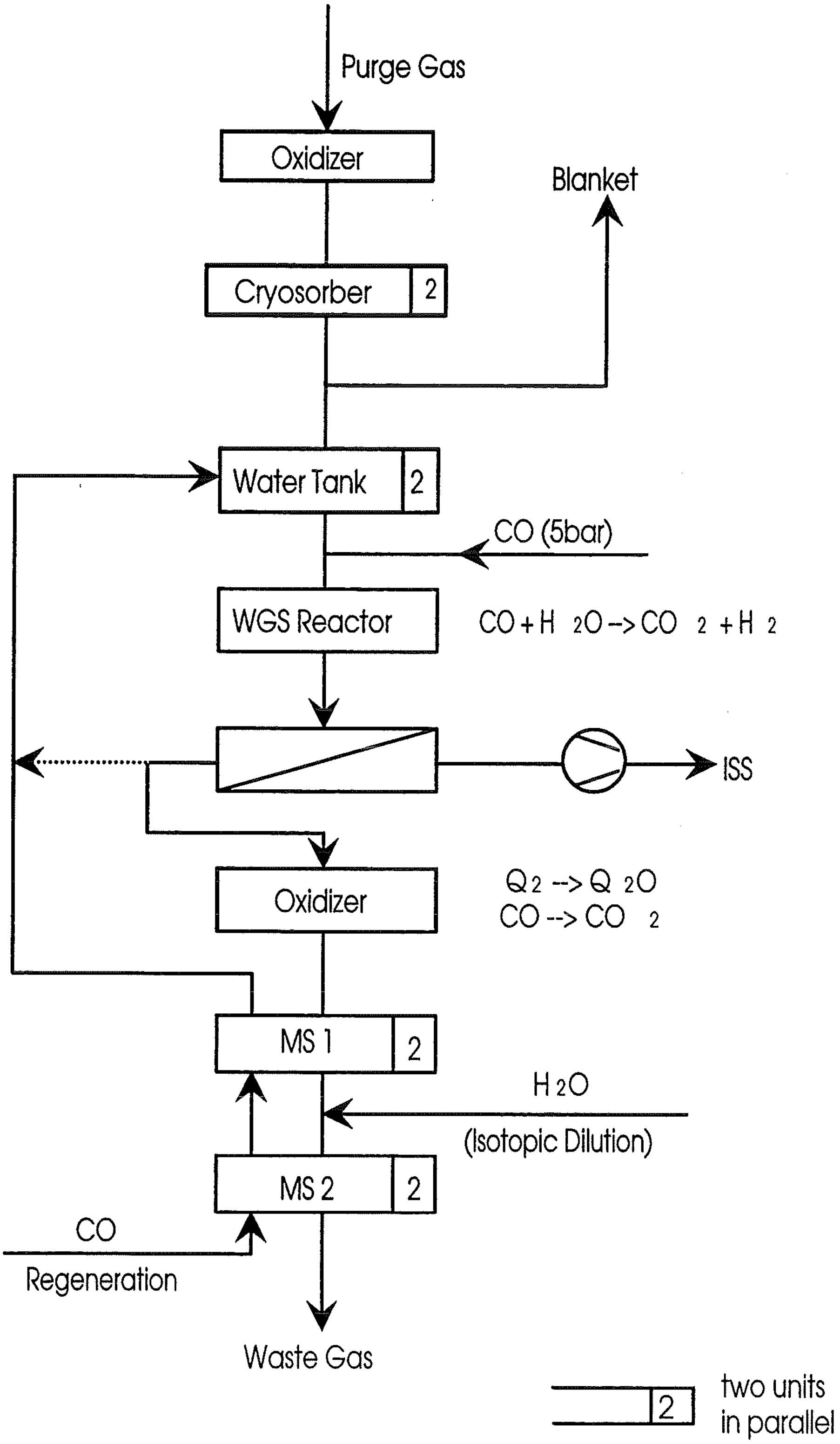

Fig. 8: ENEA Option A

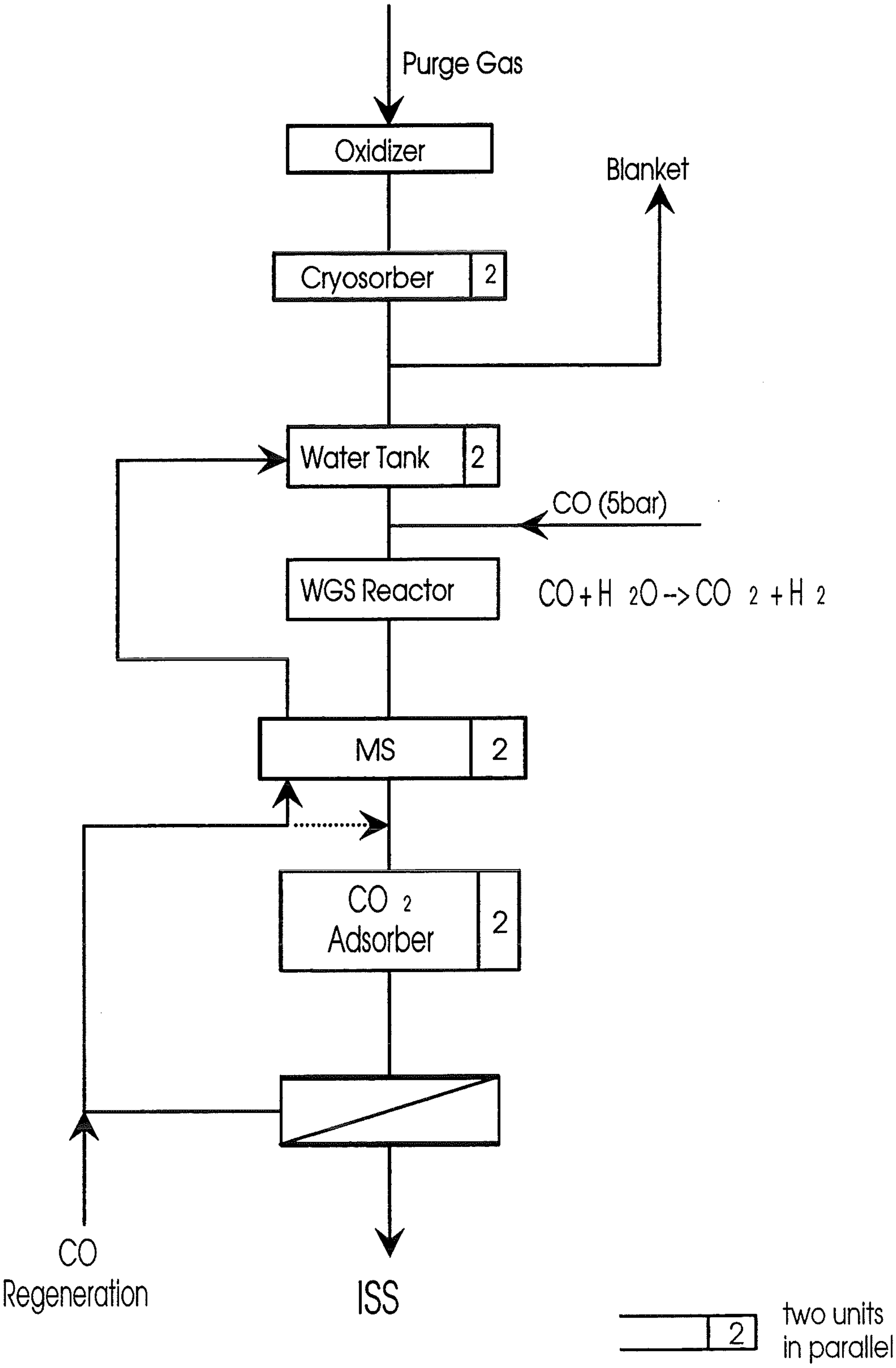

Fig. 9: ENEA Option B

Principle of Option C /10/:

Option C is also based on the idea to oxidize all tritium and tritiated compounds in the purge gas. This is achieved by adding an excess amount of oxygen (and water instead of hydrogen) to the sweep gas and by using a catalytic recombiner to ensure a complete conversion to water. The water is then separated from the sweep gas in a cryotrap and decomposed in a water gas shift reactor (WGSR). Finally, molecular hydrogen is separated by using a membrane permeator. A main feature of the process is the reconversion of CO_2 generated by the WGSR to CO.

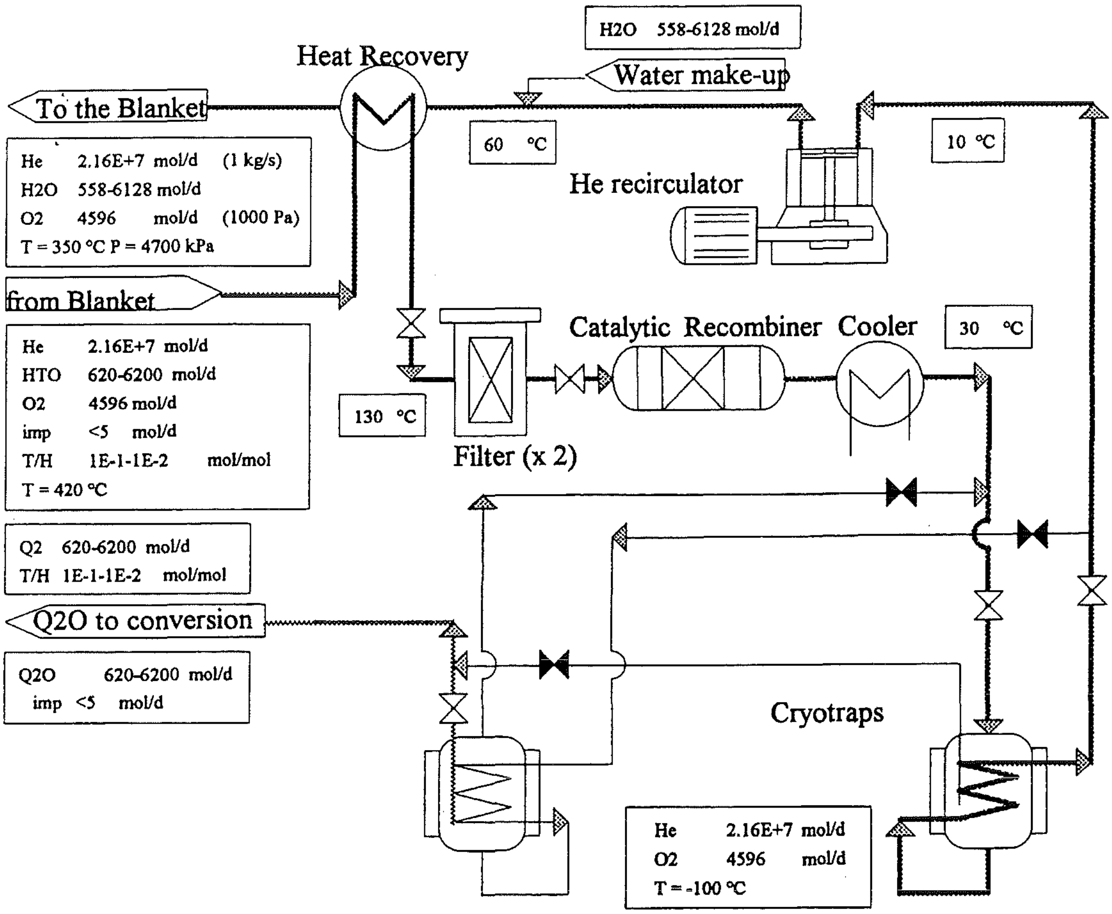

Fig. 10: ENEA Option C, Sweep Gas Loop /10/

Process Description of Option C (Fig. 10):

The composition of the blanket sweep gas is shown in Fig. 10 for the case of a DEMO blanket. The helium flow rate is 1 kg/s at a pressure of 4.7 MPa. The amount of water to be extracted is expected to range between 11.2 and 112 kg/day. The main components of the sweep gas loop are:

- Regenerative heat exchanger (T_{in} / T_{out} = 350 / 130°C)
- Two filters for removal of particulate impurities
- Catalytic recombiner (T = ?)
- Cooler (T_{out} = 30°C)
- Two cryotraps, alternately operated
- Helium circulator and a unit for water / hydrogen addition.

Each croytrap is operated for two hours and then warmed up for Q_2O extraction. The amount of water sent to the Q_2O conversion system is 1 - 10 l (every 2 hours) with a content of 31 g tritium.

Water Conversion:

The chemical processing for tritium recovery from water is carried out in a secondary loop which is shown in Fig. 11.

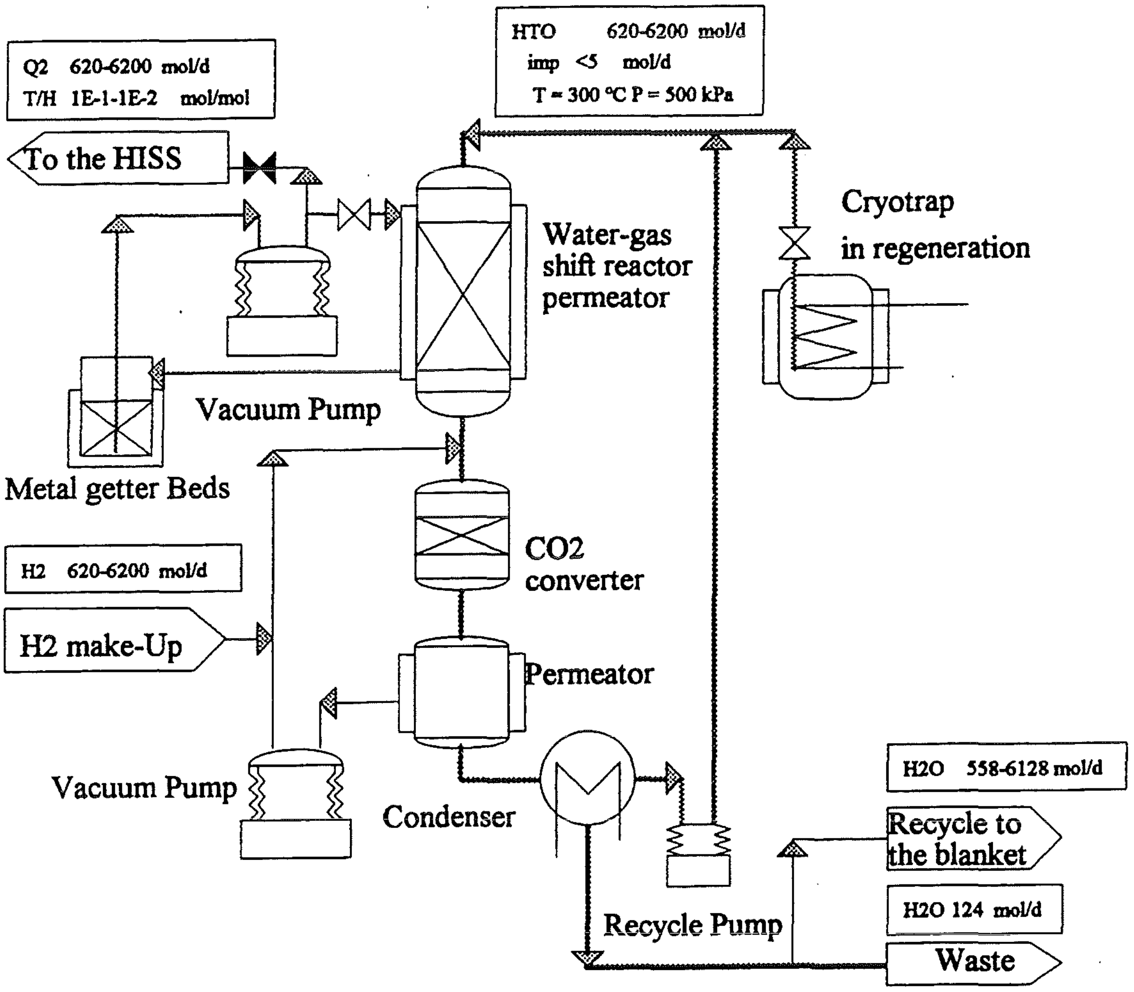

Fig. 11: Process Flow Sheet for Tritium Recovery from Water

The process is based on the use of a palladium membrane water gas shift reactor. The reactor consists of a bundle of ceramic tubes which are covered by Pd/Ag hydrogen permeating membranes. The tubes are filled with a catalyst promoting the reaction the reaction of water with carbon monoxide to produce hydrogen and carbon dioxide ($CO + Q_2O = CO_2 + Q_2$).

The mixture of residual helium and water vapor extracted from the cryotrap during regeneration is further warmed up to 300°C and enriched by an excess amount of CO before the gas enters the WGSR. The hydrogen isotopes formed by the reaction permeate through the membrane; they are transferred to a metal getter bed for interim storage and later on to the Isotope Separation System (ISS).

The main effluent stream from the WGSR contains CO_2, some residual CO, trace amounts of non-reacted water, and the non-permeated hydrogen isotopes. This gas is processed in a second catalytic reactor in which the water gas shift reaction is reversed to reconvert CO_2 into CO ($CO_2 + H_2 = CO + H_2O$). For this aim, additional hydrogen is added to the gas. The converter is operated at T = 500°C. Excess hydrogen is then extracted by a Pd/Ag permeator. Finally, the water is removed from the gas in a condenser (at T = 20°C) while the remaining CO is transferred to the beginning of the process. Most of the removed water (still slightly tritiated) is partly used as an addition to the blanket sweep gas; a smaller fraction is sent to the waste water system.

Comments:

1. Option C is an advanced proposal in comparison to the Options A / B. The design of the components of the sweep gas loop (i.e. circulator, cryotrap, catalytic recombiner, etc.) has not been discussed; apparently, because no specific development needs are seen.
2. Two general advantages of Option A / B / C result from the fact, that the hydrogen isotopes occur in the sweep gas loop essentially in one chemical form, i.e. Q_2O. Compared to all other procedures without the oxidation step, this leads to a reduced tritium permeation into the blanket coolant. In addition, it is not required to install molecular sieve beds (or equivalent components) for the removal of molecular hydrogen isotopes (Q_2).
3. On the other hand, the catalytic recombiner (oxidizer) is not needed in the other cases. As this component will be operated at a temperature of at least 400°C and with a gas flow rate of $2 \cdot 10^4$ Nm^3/h, the size and complexity of the whole sweep gas loop will not become much less than in other concepts.
4. Problems could be connected with the Q_2O processing in the WGSR loop. At a throughput of 620 mole Q_2O/ day (see Fig.11), the H/T ratio is about 10. It is known that tritium concentrations of this order lead to radiochemical reactions with CO that will cause severe poisoning of the permeator. In addition, organic compounds like methane and / or formaldehyde are formed from which tritium cannot be recovered by the described procedure. Thus, the feasibility of Option C appears very questionable under these conditions.
   At a throughput of 6200 mole Q_2O/ day, these problems will be less severe; however, a detailed discussion of the radiochemical reactions is indispensible.
   In addition, it will be quite difficult to realize the warm-up of the cryotrap, the vaporization of 6200 mole Q_2O, and the processing of 140 Nm^3 gas within two hours.

6. Sulzer Methods

Option A: Cryogenic Freezer
Option B: Combined Recombiner-Catalytic Exchange Process

Principle of Option A /11,12/:

The constituents of the blanket purge gas are removed in 3 steps:

1. In a cold trap at 173 K (water and some of the high boiling gases, mainly CO_2),
2. In an adsorber at 75 K (O_2, N_2, C_xQ_y, etc., and some hydrogen),
3. In a freezer at 10 K (hydrogen isotopes).

After the third step, clean helium is returned to the blanket through several countercurrent heat exchangers. The hydrogen isotopes are released after warmup of the freezer to 26 K and directly fed into the Isotope Separation System (ISS). Hydrogen recovery from water is obtained by using an electrolyzer.

An adsorber operated at 30 K instead of the freezer is used in a modification of this option.

Process Description (Fig. 12)

The temperature of the purge gas is reduced by a countercurrent heat exchanger and a cooler to room temperature. The gas is then compressed to 10 bar; after removal of the compression heat it enters a cold trap where most of the water and some CO_2 are frozen out at 173 K (first step).

In the second step, low boiling gases (O_2, N_2, C_xQ_y, ... and some Q_2) are adsorbed an a molecular sieve at approx. 75 K. In the last step, the hydrogen isotopes are frozen out in a cryogenic freezer at 8 - 10 K. The remaining clean helium is sent to a buffer vessel and finally returned to the blanket.

Every 24 hours, the removed moisture is recovered from the freezer panels by warmup. The liquid is degassed and electrolyzed, whereby the gases Q_2 and O_2 are both fed back into the process line without further purification.

Regeneration of the adsorber (every two weeks) is performed in 3 steps:

- by evacuation to remove coadsorbed helium and some Q_2 (both are refed into the process),
- by slight warmup to remove the majority of the Q_2 which is refed as well,
- by warmup to 150 - 200°C to remove the remaining components; as these will include tritiated species, such as HTO and C_xQ_y, additional treatment is needed, e.g. by sendig the gas to a catalytic recombiner to convert all hydrogen species into water. The remaining gas is then delivered into the waste gas system, while the water is transferred to a water treatment unit.

The freezer is regenerated first by evacuation to minimize the helium flow to the ISS; the pumped gas (mainly helium) is refed into the process. The freezer panels are then warmed up to 26 K which is sufficient to evaporate the hydrogen and to generate a pressure of about 4 bar to enable the transfer to the ISS without additional pumping or compressing. The regeneration cycle time is 24 hours.

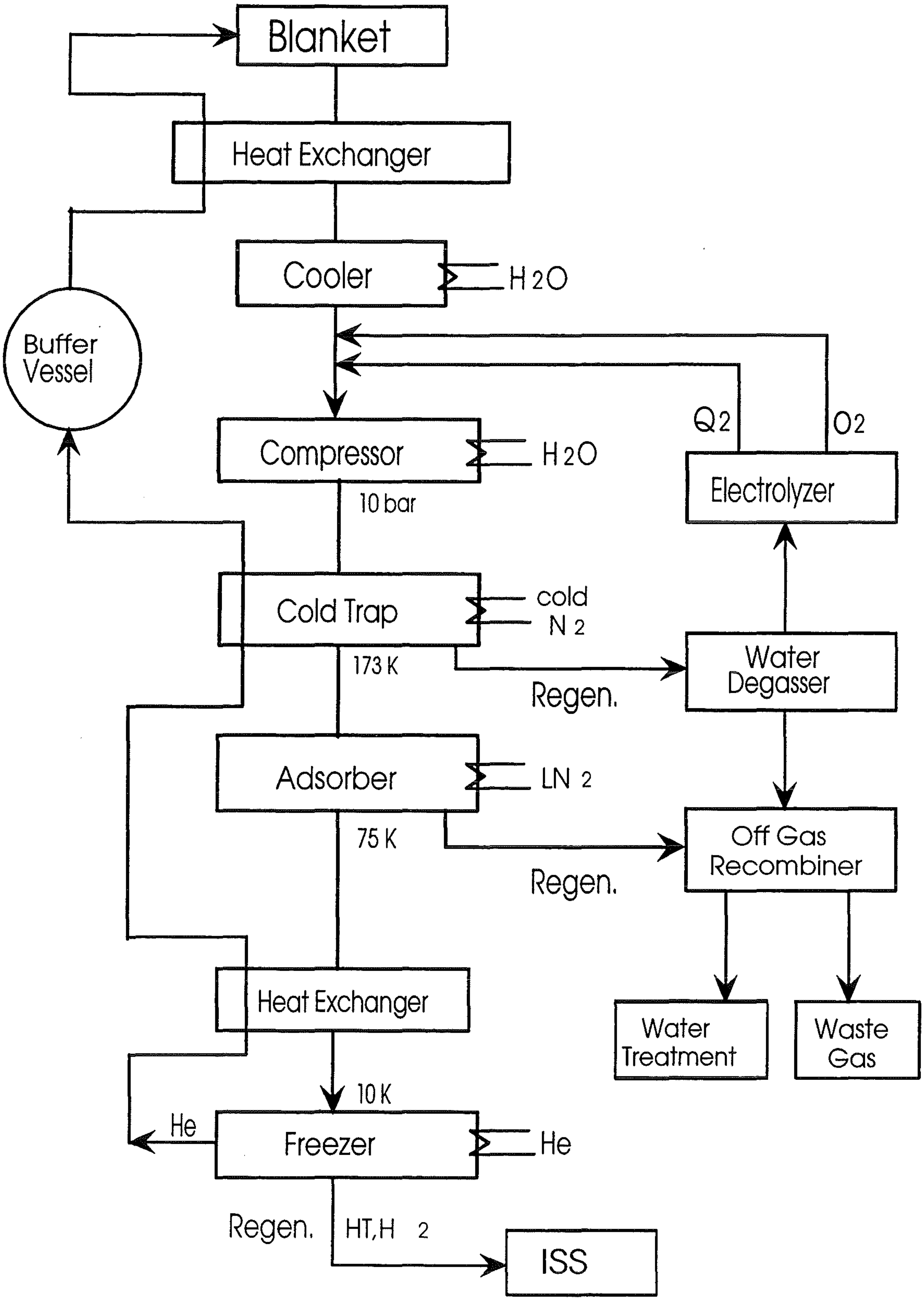

Fig. 12: Sulzer Option A: Cryogenic Freezer

An equipment design was carried out in /11/ for the ITER case #2 which includes the following conditions (see also Table A1 in /12/):

Tritium generation:	106 g/d
Helium flow rate:	$2{\cdot}10^6$ mol/d
H/T swamping ratio:	101
Purge gas pressure	0.12 MPa (at blanket outlet)
Impurities	< 1.2 mol/d („ „)

Advantages of the Option A

- Compression of the gas to 10 bar leads to an increased efficiency of the cold trap and reduces the size of the succeeding components,
- The size of the adsorber (molecular sieve bed) is uncritical because there is no demand for complete hydrogen retention; the bed is only used to remove the low boiling gases; thus, a regeneration time of two weeks is sufficient;
- Most of the gas processing is carried out at very low temperatures; this is advantegeous with respect to tritium permeation losses;
- There is no need for the use of a large Pd/Ag diffusor;
- It is not neccessary to purify the gases produced by the electrolyzer from entrained water, since they are returned into the process.

Disadvantages

- The use of electrolyzers is connected with various problems, such as relatively high leak rates, tritium diffusion losses, high explosion potential, and insufficient long time experience with tritiated water;
- The amount of hydrogen retained on the adsorber (molecular sieve bed) will be considerable; this means that extensive gas processing is needed after regeneration of the adsorber;
- A high cooling power is needed for the operation of the freezer at 8 -10K when the high purge gas flow rate projected for DEMO is considered.

Principle of Option B /11,13,14/:

The purge gas is processed in the following steps:

1. The molecular hydrogen isotopes are oxidized by use of a recombiner;
2. The tritiated water is removed by use of a 4A molecular sieve dryer;
3. The helium leaving the dryer is purified and fed back to the blanket;
4. The moisture recovered from the dryer is fed to a multi-stage LPCE/ VPCE system; in this system, the tritium is transferred into a hydrogen stream which is directly connected with the ISS.

This option is a modification of the JAERI Option B where a water gas shift reactor is used instead of the catalytic exchange.

Process Description (Fig. 13)

The purge gas coming from the breeder passes through a counter current heat exchanger where it is cooled to approx. 400°C. At the outlet of the exchanger, a filter is installed to remove eventual dust from the breeder. After addition of a slightly substochiometric amount of oxygen (to avoid an additional oxygen removal step further downstream) the gas enters the recombiner. The free hydrogen content is reduced to a few ppm, whereby the relatively high temperature of the gas is useful with respect to the reaction speed and the size of the catalyst bed. The latter is an inconel 800H vessel filled with platinum or palladium containing aluminium silicate pellets.

After leaving the recombiner, the gas is cooled down in two steps to about 30°C and then compressed to 6 bar. This pressure allows easy operation of the two molecular sieve dryers (MSD) which follow and reduces their size to reasonable dimensions. The dryer beds are filled with 4A molecular sieve pellets. At the inlet of the dryer, slightly tritiated water from the water distillation unit (see below) is added to increase the dew point and to dilute the tritium concentration. While one bed is drying, the second bed is heated and regenerated with a slip stream. This stream is cooled in a condenser where the removed water is separated and then sent to a storage tank. A time of 24 hours is forseen for a complete drying and regeneration cycle.

The dryed gas is sent to a filter to remove eventual debris and dust from the dryer; a fraction of the gas is then passed to a purification system (installed in a bypass) to remove other impurities than oxygen and hydrogen. Finally, the gas is sent back to the breeder via the two counter current heat exchangers mentioned above.

The moisture recovered from the dryer is sent to an installation containing a combination of LPCE and VPCE columns operating on a multi-stage basis. The tritiated water and hydrogen gas are flowing in countercurrent through each stage. The exchange, i.e. the transfer of tritium from the water into the hydrogen stream according to $HTO + H_2 \leftrightarrow H_2O + HT$ occurs with the help of a hydrophobic catalyst /15,16/. The tritium is removed from the hydrogen by the ISS (cryogenic distillation), and the hydrogen is returned to LPCE/ VPCE system.

Comments:

The process described above was proposed by Sulzer Brothers/ Winterthur on the basis of the experience gained with a Tritium Removal System (TRS) at Grenoble/France and a second, much lager TRS facility designed to process 350 kg tritiated wate per hour by the same company for Ontario Hydro at Pickering/ Canada /14/. The tritium concentration at the inlet of these facilities was about 10 Ci per kg of tritiated water and a tritium depletion factor (DF) in the range of DF = 12 appeared to be sufficient.

In the case of the DEMO purge gas processing with H : T = 100 and a tritium production of 15 g/h, however, one will get 4550 g Q_2O/ h with a specific activity of 32 kCi/ kg. Thus, a depletion factor of 10^5 and several thousand catalytic exchange stages would be needed to arrive at a residual activity of 0.3 Ci/ kg.
It is concluded, therefore, that this approach is an unrealistic proposal for the DEMO purge gas - a judgement, which is in principal agreement with the results of a “Comparison of practical applications for tritium separation processes“ reported in Table IX of /17/.

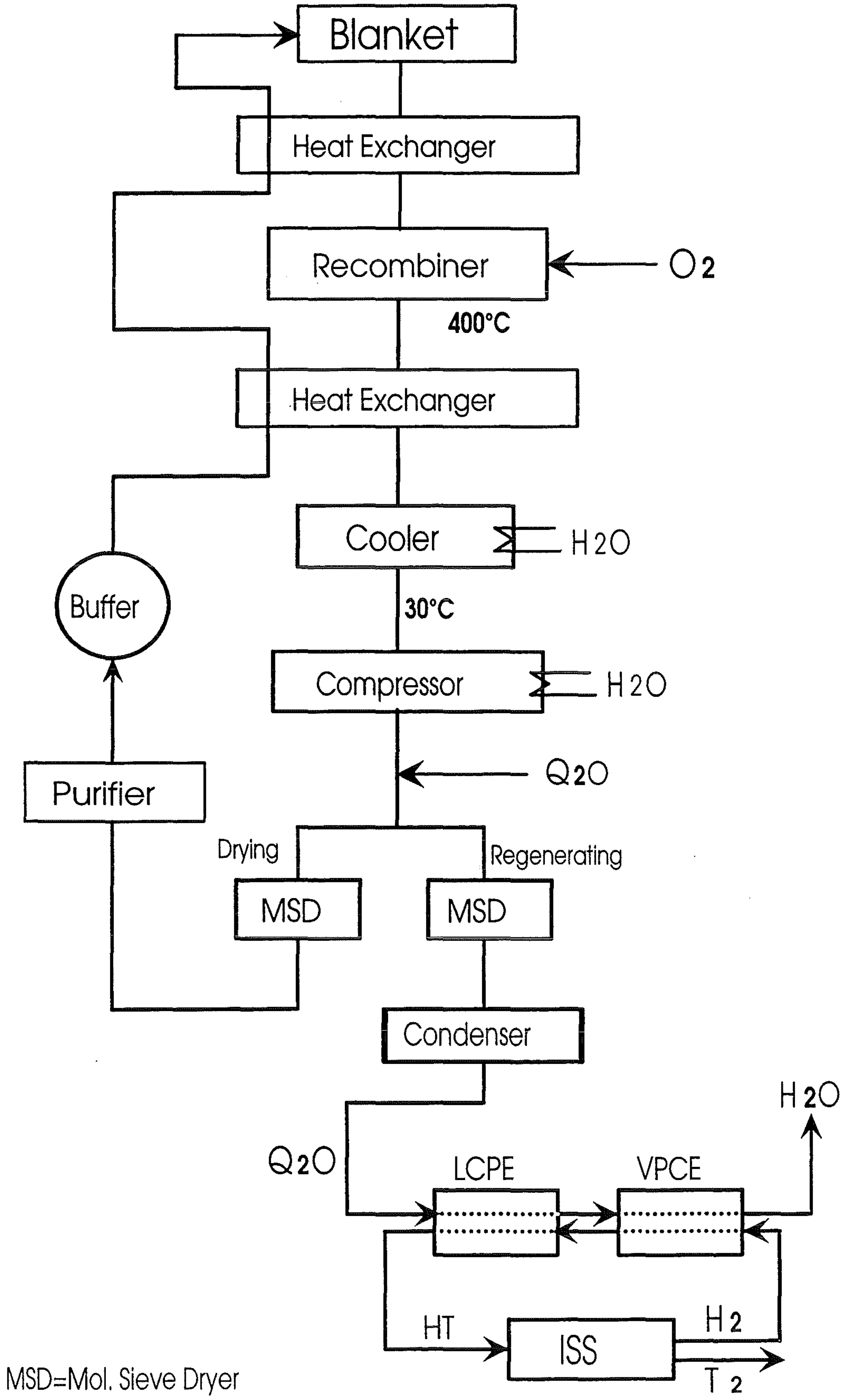

Fig. 13: Sulzer Option B: Recombiner/ Catalytic Exchange

7. Conclusions: FZK Concept

As a conclusion of the current evaluation, an additional concept is proposed (FZK Concept) which comprises / modifies various process steps of the methods described above. It is expected to have the best potential for applicability to ITER and DEMO and to incorporate the smallest development risk. The most characteristic aspects of the concept are:

- It is especially capabable to cope with large purge gas streams; only a few components (compressor, cold traps, and molecular sieve beds) are exposed to the gas flow of the main purge gas loop. All additional gas processing and separation work is carried out in secondary loops where the flow rates and hence the size of the components can be much smaller.
- The concept uses well known methods of technical and chemical engineering; the chemical steps carried out in the Q_2O-Loop are being demonstrated with tritium in a technical scale by the CAPRICE tests in the Tritium Laboratory Karlsruhe (TLK). In addition, the technical feasibility of the concept has been investigated with a positive result by an engineering company (Linde AG), who also provided the layout of the primary TES loop shown in Fig.14 /25/.

Principle /18/:

Since tritium will occur in the purge gas in two main chemical forms (HT and HTO), two specific process steps are used for its recovery in the main TES loop:

- Freezing out of the water form in a cold trap at 173 K and
- Adsorption of the molecular form on a 5 A molecular sieve bed at 80 K.

Additonal processing, i.e water cracking and separation of the hydrogen isotopes from impurities and remaining helium is carried out in secondary loops, where much smaller gas flow rates are employed than in the main TES loop. The main procedure in the secondary loop is the application of a water gas shift reactor in combination with a Pd/Ag diffuser.

Process Description

The main requirements and design parameters of the tritium extraction system are summarized in the following tables:

Tritium Production Rate	370 g/d = 62 mole T_2/day
Mass Flow of Helium Purge Gas	0.6 kg/s = $5.4 \cdot 10^5$ mole/h
Average Helium Pressure	p(He) = 110 kPa
Swamping Ratio	$He : H_2$ = 1000 : 1[a)]

a) this corresponds to H_2 / HT = 108

Purge Gas Component	Mass Flows and Concentrations TES Inlet	 TES Outlet
He	$13 \cdot 10^6$ mole/d	$13 \cdot 10^6$ mole/d
H_2	$13 \cdot 10^3$ mole/d	1300 mole/d
HT	120 mole/d[a)]	< 12 mole/d
HTO + H_2O	124 mole/d	< 12 mole/d
Impurities (N_2, CO, CH_4,...)	< 5 mole/d	< 1 mole/d[b)]

a) see text
b) some removal is necessary to avoid the accumulation of impurities

It is assumed that tritium is released from the breeder material in the form of gaseous HTO; the release rate is 124 mol HTO/day. At the beginning of the reactor operation, some of the tritiated water will be chemically reduced at the steel walls of the blanket canisters; this fraction becomes smaller when a thin oxide layer has been formed; it can be neglected furtheron because - due to the relatively high H_2 concentration in the purge gas - an oxidation / reduction equilibrium will be reached.

Most of the HTO will be subjected to isotopic exchange ($HTO + H_2 \leftrightarrow H_2O + HT$) which leads to a very small amount (0.9 %) of remaining HTO, if the same HT/H_2 ratio is obtained for the molecular form (Q_2) as for the water form (Q_2O). However, as we do not know if this equilibrium is actually reached, we make the assumption that we will get 4 mole HTO + 120 mole H_2O + 120 mole HT per day as shown above in the table. In this case, the remaining HTO fraction is 3.2 %.

Primary Loop (Fig. 14)

The main components of the primary loop are:

- a gas circulator,
- 2 cold traps for the removal of tritiated water (E100 A/B),
- 3 molecular sieve beds for the removal of molecular hydrogen isotopes (A100 A/B/C),
- a precooler and 2 recuperative heat exchangers (E200, D100).

The operation temperature of the cold traps (173 K) is low enough to freeze out the Q_2O content of the purge gas almost completely (the residual concentration at the exit is less than 0.015 ppm). The required capacity of the trap is 2.5 kg/day.

While the first trap is in the freezing mode, the second is warmed up to about 300 K to liquefy the water which is then drained into an evacuated water collector (not shown in Fig. 14). Finally the cold trap is recooled to 173 K. It is expected that a time span of 12 hours will be sufficient for this procedure; an operation time of 24 hours, however, would be preferable.

Recovery of tritium from the water molecules is carried out in a secondary loop (Q_2O-Loop) which is described in the next paragraph.

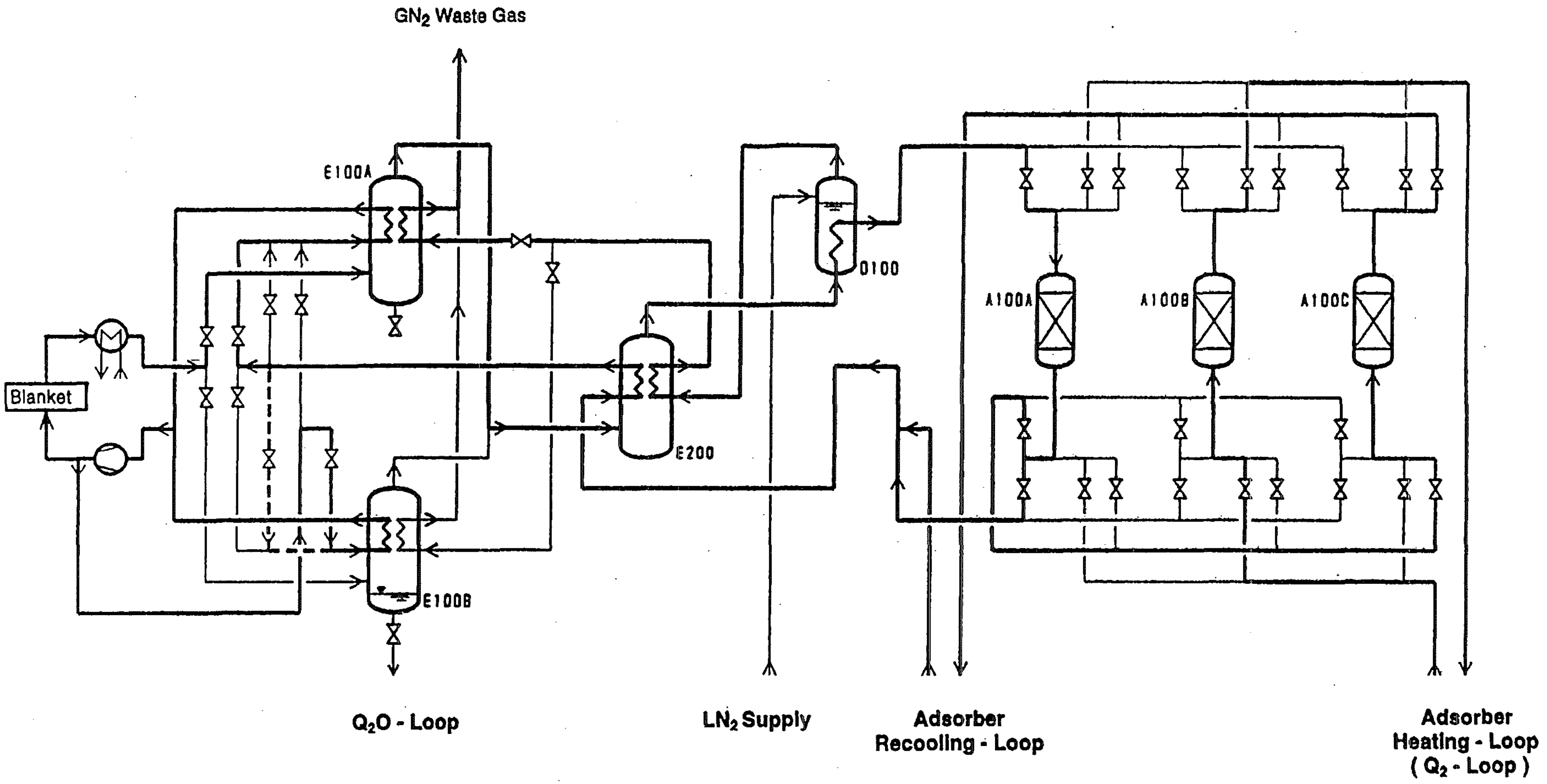

Fig. 14: Primary Loop of the FZK Tritium Extraction System (TES)

Further processing of the primary purge gas is carried out by three simultaneously operated 5A molecular sieve beds. The operation includes 3 steps:

1. Adsorption of hydrogen isotopes and gaseous impurities at 80 K (MS1),
2. Desorption of hydrogen isotopes and co-sorbed helium at ≤ 160K (MS2, which has been in the adsorption mode before),
3. Re-cooling to 80 K (MS3, which has been loaded and deloaded before).

To enable step 2, the molecular sieve bed A100B is connected to the “Asorber Heating Loop“ or “Q_2-Loop“ which is shown in detail in Figure 15.

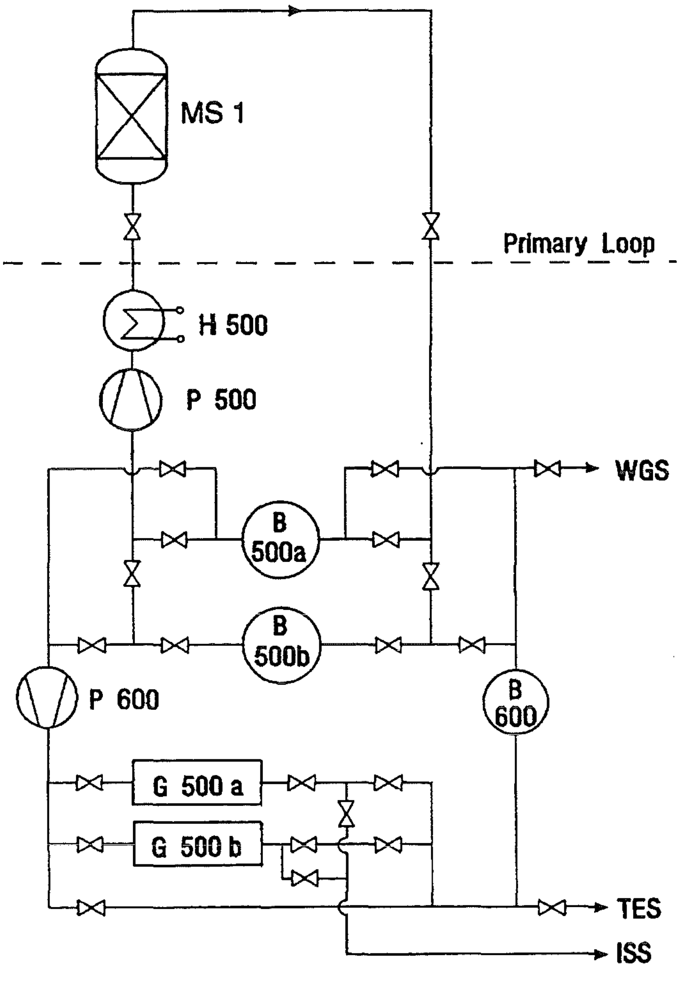

Fig. 15: Q_2-Loop

The upper part of the loop includes an electrical heater H500, the circulation pump P500 and two buffer vessels B 500a/b. At the beginning of a warmup cycle, the vessel B 500a is filled with about 0.3 bar of pure helium which is pumped through the cold adsorber until a temperature of 160 K is reached. With increasing temperature, the co-adsorbed helium and the hydrogen isotopes are released, while the impurities (N_2, CO, CQ_4 ...) are almost completely retained on the molecular sieve. At the end of the deloading phase, the next adsorber bed is connected with the Q_2-Loop, and another warmup phase is started with 0.3 bar helium contained in vessel B500b.

With the help of pump P600, the He-Q_2-mixture contained in vessel B500a is sent through an uranium getter bed which is operated at room temperature to ensure a selective and effective sorption of the hydrogen isotopes. The maximum amount of uranium needed to store all H_2/HT of a 6 hour operation cycle is about 500 kg. The first fraction of the hydrogen-free gas is fed into the purified gas at the TES exit, the remaining gas is collected in the buffer vessel B600 and lateron pumped back into vessel B500a.

The second getter bed is used when the first one is unloaded by heating. The released hydrogen isotopes are transferred directly into the Isotope Separation System (ISS).

If a molecular sieve bed is to be completely regenerated, the first step will be the desorption and storage of the hydrogen isotopes as described above. The bed is then further heated to about 550 K. The desorbing moisture and impurities are then sent to the Waste Gas System (WGS).

Q_2O-Loop (Fig. 16)

The Q_2O collected in the cold trap is liquified by warmup to about 300 K and then drained into an evacuated water collector. This container is also used to reduce the gas pressure in the cold trap which increases during warmup.

It is equipped with an electrical heater allowing a slow evaporation of the water. The gas is then transferred within a closed loop to a water gas shift reactor (WGSR) where it is converted to CO_2 and Q_2 by addition of CO and by the use of a copper chromite catalyst (T = 200°C, conversion rate > 99.5% /19/).

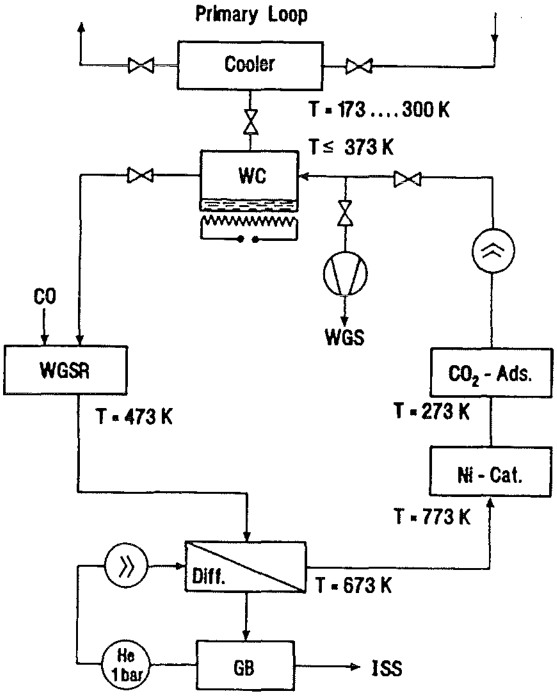

Fig. 16: Q_2O - Loop (WC = Water Collector, WGS = Waste Gas System, WGSR = Water Gas Shift Reactor)

The hydrogen isotopes are removed from the gas stream by a Pd/Ag diffusor. The back pressure side of the diffusor is integrated into a small helium loop (diffusor loop) which serves to continuously transport the permeated hydrogen isotopes to a metal getter bed for storage. The purity of these isotopes is sufficiently high to allow a direct transfer to the Isotope Separation System (ISS) after desorption from the getter bed. The diffusor loop is operated with a second identical storage bed during the unloading step of the first bed.

The next component of the Q_2O-Loop is a nickel catalyst bed (T = 500°C) which has the task to crack tritiated methane that is eventually formed with a small yield by the reaction $CO + 3Q_2 \rightarrow CQ_4 + Q_2O$. The CO_2 adsorber (e.g. modernite /20/) downstream of the nickel catalyst is used to remove the CO_2 formed by the water gas shift reaction.

All chemical process steps carried out in the Q_2O-Loop have been verified in laboratory tests with hydrogen and tritium /21/. In addition, the feasibility of the complete loop is being demonstrated in a technical scale by the CAPRICE tests /22, 23/ in the Tritium Laboratory Karlsruhe (TLK).

Comments

1. The use of large adsorber beds (containing about 10 tons of molecular sieve material) or of large cold traps is not unusual in the chemical industry /24/.
2. A minor disadvantage connected with the operation of the molecular sieve beds is the fact that they strongly adsorb water /26/ and therefore steadily collect the residual humidity of the gas. This water has to be removed in time intervals of 3 - 6 months by heating the bed to 550K and by purging with dry helium.
3. An interesting alternative to the tritium extraction procedure described above would be possible if H_2O and / or O_2 instead of H_2 is added to the purge gas to facilitate the tritium release from the breeder material /27/. This case leads to the conditions decribed in the ENEA Option C, i.e.
   - the concentration of H_2 and HT in the purge gas is very small,
   - the tritium extraction is predominantly carried out by the cold trap and the succeeding water processing in the Q_2O-Loop,
   - the task of the molecular sieve beds will be essentially reduced to the removal of impurities; the beds can be much smaller and may be operated, therefore, in a bypass with a reduced gas flow rate.

   On the other hand, one has to reconsider the application of the water gas shift reaction for tritium recovery from water because in this case one would have to process about 115 kg Q_2O/ day (under the assumption that the water addition to the purge gas leads to H : T = 100).

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