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**Heavy Water: A Distinctive and Essential
Component of CANDU****L'eau lourde : un élément distinctif
et essentiel du réacteur CANDU**

A.I. Miller, H.M. van Alstyne

Presented at the International Atomic Energy Agency Technical Committee Meeting,
Toronto, Ontario, 1993 June 7-10

AECL Research

Heavy Water: A Distinctive and Essential Component of CANDU

by

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EACL Recherche

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Résumé

Les propriétés exceptionnelles de l'eau lourde comme modérateur de neutrons constituent l'un des éléments distinctifs des réacteurs CANDU. Bien que la plupart des propriétés chimiques et physiques du deutérium et protium (hydrogène de nombre de masse 1) sont nettement différentes, la faible abondance du deutérium dans la terre fait de la séparation de l'eau lourde un procédé relativement cher et donc d'une importance considérable pour le système CANDU.

Le stock mondial d'eau lourde provient actuellement du procédé Girdler-Sulphide ou des procédés basés sur l'échange entre l'ammoniaque et l'hydrogène. En raison des facteurs coût et risque, il faudra des nouveaux procédés de production de l'eau lourde dans la prochaine décennie et au-delà de celle-ci. Grâce à la mise au point et à l'amélioration, par EACL, de catalyseurs hydrofuges pour l'échange d'isotopes d'hydrogène, on s'attend à ce qu'on utilise une famille de nouveaux procédés. Deux procédés monothermiques, CECE (Combined Electrolysis and Catalytic Exchange) (Électrolyse et échange catalytique combinés) à transformation de l'eau en hydrogène par électrolyse et CIRCE (Combined Industrially Reformed Hydrogen and Catalytic Exchange) (Hydrogène réformé industriellement et échange catalytique combinés) basés sur le réformage des hydrocarbures à la vapeur, sont les plus avancés. En plus de servir à la production de l'eau lourde, le procédé CECE est une technique très efficace pour l'amélioration de l'eau lourde et la séparation du tritium de l'eau lourde (ou ordinaire).

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Abstract

The exceptional properties of heavy water as a neutron moderator provide one of the distinctive features of CANDU reactors. Although most of the chemical and physical properties of deuterium and protium (mass 1 hydrogen) are appreciably different, the low terrestrial abundance of deuterium makes the separation of heavy water a relatively costly process, and so of considerable importance to the CANDU system

World heavy-water supplies are currently provided by the Girdler-Sulphide process or processes based on ammonia-hydrogen exchange. Due to cost and hazard considerations, new processes will be required for the production of heavy water in and beyond the next decade. Through AECL's development and refinement of wetproofed catalysts for the exchange of hydrogen isotopes between water and hydrogen, a family of new processes is expected to be deployed. Two monothermal processes, CECE (Combined Electrolysis and Catalytic Exchange, using water-to-hydrogen conversion by electrolysis) and CIRCE (Combined Industrially Reformed hydrogen and Catalytic Exchange, based on steam reforming of hydrocarbons), are furthest advanced. Besides its use for heavy-water production, the CECE process is a highly effective technology for heavy-water upgrading and for tritium separation from heavy (or light) water.

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1. HEAVY-WATER REACTORS

Today's nuclear reactors all depend on the absorption of a neutron to induce fission in a fissile nuclide, such as ^{235}U , followed by the release of further neutrons from that fission, and the survival of enough of these next generation of neutrons that an average of at least one new fission can be induced — a chain reaction. In the predominant, thermal form of today's nuclear reactors, between fission-induced release and fission-inducing recapture, neutrons must be "moderated"; i.e., give up a large part of their initial kinetic energy. Light hydrogen (mass 1 or ^1H) is an unsurpassed substance for this moderation and can conveniently be provided as water, since oxygen isotopes collectively interact to only a minor degree with neutrons. Inconveniently, however, ^1H itself can capture neutrons and will compete so effectively with fissile nuclides that a chain reaction cannot be sustained unless fissile nuclei are more than normally abundant. So, where light water is used as a moderator, uranium fuel must be enriched with the fissile ^{235}U isotope, typically by a factor of four above the natural 0.7 mole % abundance.

The alternative approach, used in CANDU[®] (Canada Deuterium Uranium) reactors, is to provide moderation with the heavier hydrogen isotope, deuterium (D or ^2H), again in the oxide form, D_2O , commonly known as "heavy" water. Heavy water's much smaller ability to capture neutrons, compared to natural water, allows the use of uranium with the natural 0.7% abundance of ^{235}U .

Hence, there is a choice of reactor systems and both require isotopic enrichment: one can use ordinary (predominantly light) water and modest enrichment of all of its uranium fuel, or one can use natural uranium with almost pure heavy water. Each approach has its advantages and disadvantages: (1) light-water-moderated reactors require ^{235}U isotope enrichment continuously, and the differences in properties between uranium isotopes are small, though ^{235}U is relatively abundant; (2) the differences in properties between hydrogen isotopes are often very large, but the terrestrial natural abundance of deuterium is low (averaging ~0.0155 mole %); (3) heavy-water-moderated reactors require appreciable quantities of isotope separation before they can begin to operate, but — at least in theory — most of that heavy water is available for re-use at the end of reactor life. Actually, a blending of the two approaches offers much of the advantages of both, and CANDU reactors using slightly enriched uranium are drawing increasing attention. However, CANDU's commitment to heavy-water moderation remains, and so Canada retains a strong commitment to developing efficient heavy-water production processes.

2. EXISTING HEAVY-WATER PRODUCTION

Heavy water is currently produced in Canada, India, and China. Production facilities are being commissioned this year in Romania and Argentina. The largest installed capacity among these producers utilizes the Girdler-Sulphide (GS) process, which is a cascaded series of dual-temperature, mass-transfer columns circulating large quantities of hydrogen sulphide gas countercurrently to natural water feed. A number of small-capacity plants using the hydrogen-ammonia catalytic exchange process are in service in India, and a 250 Mg/a plant, also using ammonia-hydrogen exchange, is in its start-up phase in Argentina.

Both of these production processes require large capital expenditures. The ammonia process has size limitations, and the GS process consumes large amounts of energy and utilizes very hazardous hydrogen sulphide, though after initial technical problems had been resolved, GS technology has performed extremely well in Canada. The history of GS technology in Canada has been reviewed by Rae.¹

Forecasts of the cost of heavy water produced in new plants using GS and ammonia-based processes are very high, high enough to affect the economic attractiveness of a heavy-water reactor such as CANDU.

Installed Canadian capacity for heavy-water production is forecast to be adequate throughout the present decade. However, in the next decade and beyond, new processes that have lower production costs will be required.

3. SEPARATION METHODS

The difference in any property on which an isotope separation process can be based is usually expressed as a separation factor, α :

$$\alpha = \frac{[DX] \cdot [HY]}{[DY] \cdot [HX]}$$

where [DX] is the deuterium concentration in one chemical or physical form, [DY] is the deuterium concentration in the other chemical or physical form, and [HX] and [HY] are the corresponding protium or light hydrogen concentrations.

Note that at low concentrations of deuterium, the equation tends toward:

$$\alpha = \frac{[DX]}{[DY]}$$

Numerous reviews, some of which are cited,² have appeared on the merits of diverse processes based on divergences between physical and chemical processes that could be used to separate the isotopes of hydrogen, and particularly to produce heavy water. It is not intended in this paper to review the myriad possibilities. However, a practical process must meet three exacting criteria: (1) because the natural abundance of deuterium is so low, the feedstock must be abundant; (2) because of this low abundance, large quantities of material must be processed, and so processing must be kept relatively simple and should use a minimum of energy; and (3) the separation factor pertaining to the properties of deuterated and protiated molecules, α , should be as large as possible, to reduce the number of protium atoms that will separate with the deuterium-rich stream and to reduce the number of deuterium atoms that will remain with the deuterium-depleted stream.

4. PROCESSES BASED ON CHEMICAL EXCHANGE

A small group of chemical exchange processes meets these criteria for economic viability, and they have dominated and appear likely to continue to dominate heavy-water production. They all involve transfer of hydrogen isotopes between two hydrogen-containing chemical species. They can be described, in the most general way, as:



Exploitation of chemical exchange can be based either on exchange at one (low) temperature or on exchange in opposite directions at two different temperatures. These are shown schematically in Figures 1 and 2, using water and hydrogen as typical examples of working substances. In principle, a monothermal process is advantageous for two reasons: (1) its intrinsic capacity to recover deuterium is higher (limited to $1-1/\alpha_c$ rather than $1-\alpha_h/\alpha_c$ for a bithermal process, where the subscripts refer to the cold and the hot tower conditions*), and (2) one exchange column is

* To say that a process is limited to a particular recovery is not strictly correct, since exchange sections can be extended to provide stripping to any degree of the process feed, but this approach does not reduce the internal flows of the process, and is useful only where there is limited availability of feedstock.

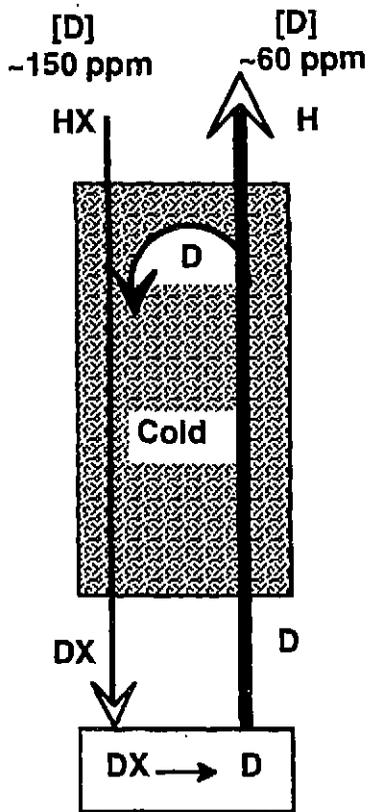


Figure 1: Schematic of a Monothermal Process

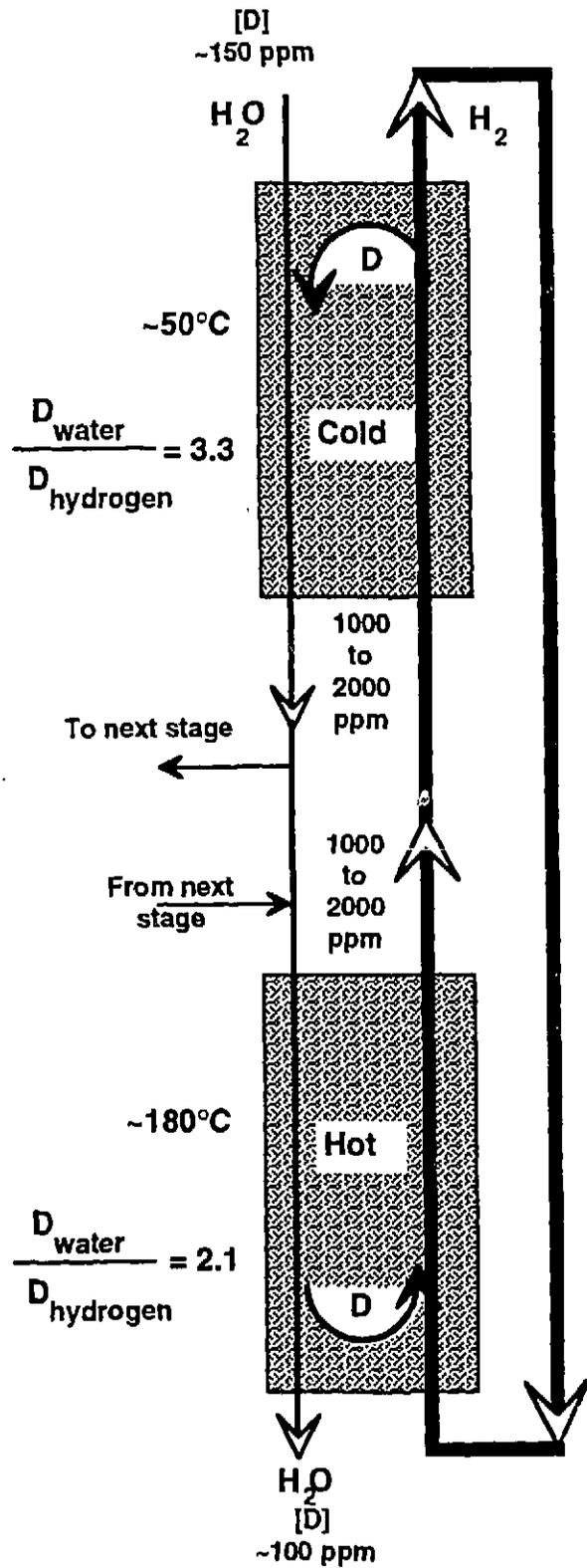


Figure 2: Schematic of a Bithermal Process

eliminated and the volume of the other is substantially reduced. However, monothermal processes must have a means of converting the deuterium-enriched chemical species into the other chemical species. This is often impractical and, even when practical, usually uneconomic.

A number of conditions influence the choices for such a system. None are irrevocably essential, but their combination is very important. Obviously, the separation factor for the reaction should favour one or the other side of the reaction fairly strongly, and it would be helpful if this separation factor was also strongly temperature-dependent. It would be useful if one of the species was abundantly available. The molecules should be relatively light and inexpensive. One should be a gas at high pressure and the other a liquid.

In practice, only three systems plus one variant have been found to be economically attractive. They are water-hydrogen sulphide, water-hydrogen, ammonia-hydrogen, and the latter's variant, aminomethane-hydrogen. (In the case of the aminomethane (CH_3NH_2) system, it is the amino-hydrogens that are active, though the methyl-hydrogens are not completely inert.) Only the water-hydrogen and the ammonia-hydrogen systems can permit the chemical conversions to hydrogen needed for monothermal processes.

While Canadian heavy-water production to date has relied exclusively on the bithermal water-hydrogen sulphide process (the GS process), capital costs for a new GS plant coupled with the process's high energy costs would result in an undesirably high heavy-water cost, and so AECL's development is now focused almost entirely on the water-hydrogen system.

5. PROCESSES BASED ON HYDROGEN-WATER EXCHANGE

The attractiveness to the Canadian program of processes based on hydrogen-water exchange has been discussed elsewhere.³ The crux of that superiority is revealed in Figure 3, which shows that the separation factor for the water-hydrogen system is much larger and varies much more strongly with temperature than that for the water-hydrogen sulphide system.

However, while thermodynamics favour the water-hydrogen system, successful processes must also have acceptable kinetics. Exchange between water and hydrogen sulphide is a fast, ionic reaction that does not require catalysis; exchange in the hydrogen-water system is effectively non-existent, unless a catalyst is provided, and the only really effective catalysts are on solid surfaces.

The superiority of the water-hydrogen system and of platinum as the most active catalyst were recognized during the Manhattan Project.⁴ The system was exploited by the Manhattan District at Trail in British Columbia, taking advantage of the electrolytic hydrogen that was generated there. Because of the severely adverse effect of liquid water on the activity of the catalyst, an elaborate system was devised with alternating contacts between hydrogen containing superheated water vapour with a catalyst (to transfer deuterium from the hydrogen to the water vapour), and between liquid water and the same water vapour after it had been cooled to saturation. This was not intrinsically an economic route to heavy-water production. Despite considerable efforts to devise a heterogeneous catalyst system able to operate in liquid water,^{5,6} further economic exploitation of the hydrogen-water system remained blocked, entirely for lack of a suitable catalyst able to operate in the presence of liquid water. That situation has been changed by 25 years of Canadian development of wetproofed catalysts.

The seminal insight came in 1968 from W.H. Stevens, of AECL Research, who conceived and demonstrated the concept of a "wetproofed" catalyst, where a porous, hydrophobic coating over a platinum catalyst left the platinum accessible to hydrogen and water vapour, but protected it from direct contact with liquid water.⁷ Refinement and improvement of Stevens's concept has continued at AECL Research over the last 25 years. The concept demonstrated by Stevens has had to be developed in a number of very significant ways. Butler et al.⁸ summarized early progress in

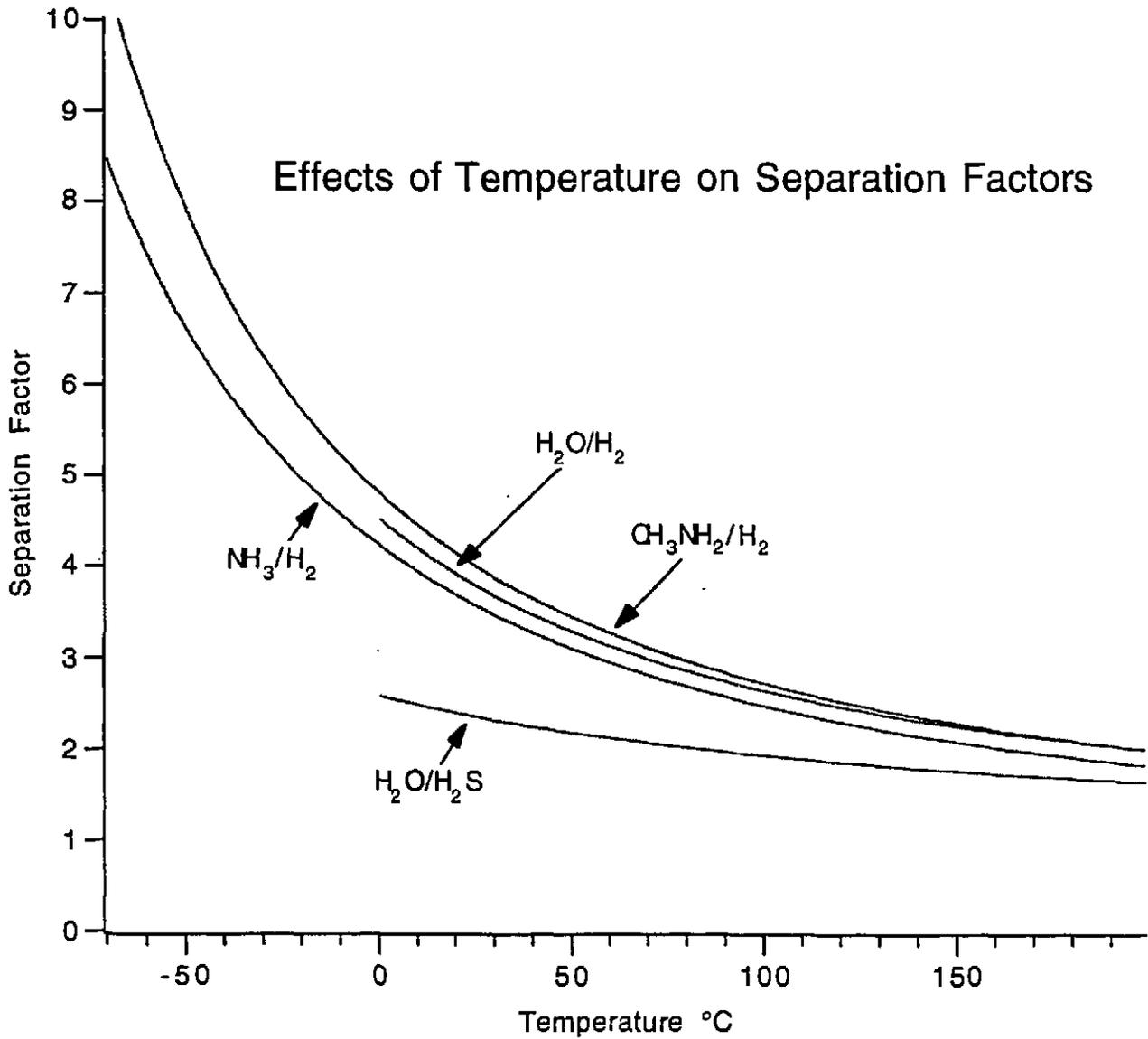


Figure 3: Separation Factors for the Main Chemical Exchange Systems as a Function of Temperature

Canada. It was quickly appreciated that mixing approximately 50% of an inert hydrophilic with 50% of hydrophobic catalyst was advantageous, since the former promoted the complementary exchange between water vapour and liquid water. One of the most significant advances was the concept of a structured or ordered bed.⁹ This concept uses alternating layers of catalytic hydrophobic metal gauze and hydrophilic material, with one layer being finely corrugated to give a large fraction of open area, and hence an exceptionally low pressure drop. Shimizu et al.¹⁰ have provided a broad review of wetproofed catalyst development work in Canada, Japan, Belgium, and Germany.

Japan moved away from the simple trickle-bed concept to a separated bed concept, with alternating contacts between hydrophobic catalyst and hydrophilic packing. This appears to have been influenced by difficulties in maintaining catalyst activity over even short trickle beds.

One can summarize the main thrusts of catalyst development in the AECL program: (1) much increased activity was needed over that initially demonstrated by Stevens; (2) low pressure drop through the catalyst bed was seen as a desirable characteristic; (3) an operating lifetime of five years was set; (4) scale-up had to be assured both for long beds (tens of metres) and large diameters (a few metres); (5) catalyst cost had to be optimized; and (6) mechanisms that could cause poisoning of the catalyst had to be understood and solutions identified.

There is no single set of targets for these parameters, because the catalyst can be applied to a number of process configurations and some are much more demanding than others. Excellent catalyst activities have been demonstrated with values at standard conditions (1 m/s superficial velocity, 25°C, and 101 kPa) as high as 700 mol(D)·m³·s, though sustained values above 400 mol(D)·m³·s have not yet been achieved. Pressure drop with the ordered-bed configuration is excellent. The desired lifetime seems to be attainable, though it has yet to be demonstrated for the most recent, very high activity catalysts. Contrary to the Japanese experience of rapidly falling catalyst activity as trickle beds went from a few centimetres to one metre, Figure 4 shows an AECL example of the maintenance of catalyst activity. (This study was done on a relatively low activity catalyst, but the important point is that it demonstrates the principle that activity is being fully maintained.) AECL has also successfully carried out two commercial contracts, where deuterium was almost completely transferred from a gas form to a water stream. These required many metres of catalyst packing. Attention to initial water distribution and subsequent retention within the bed are both extremely important to maintaining activity with increasing column length. Large-diameter scale-up has still to be demonstrated, but is not expected to present any special problem. Catalyst cost is still being reduced, but is already within an acceptable range. Provided the catalyst is not poisoned by a select group of permanent poisons (copper and sulphur compounds being the commonest), brief treatment with hot air recovers completely any loss of activity caused by "temporary" poisons, such as carbon monoxide or non-volatile organics. A special problem associated with carbon monoxide poisoning is discussed further below.

6. PROCESS CONFIGURATIONS

By far the simplest approach for harnessing hydrogen-water exchange is Combined Electrolysis and Catalytic Exchange (CECE). This is illustrated in Figure 5. It is a monothermal process, with conversion of water into hydrogen achieved by electrolysis. Unfortunately, because the entire feed stream must be electrolysed, the cost of electrolysis would result in a prohibitively expensive process for heavy-water extraction. However, where large-scale (certainly above 50 MW) electrolysis is already performed for other reasons, this process has the capability of producing heavy water very economically on a small scale (100 MW \approx -12 Mg/a D₂O production). A very small demonstration CECE plant was recently committed by the Egyptian Atomic Energy Authority. It is expected to lead to the commitment of a CECE production plant at Kima, near Aswan, where 180 MW of electrolysis currently is in operation. The CECE process also has considerable potential for upgrading of downgraded reactor heavy water, and for the separation of tritium from light or heavy water.

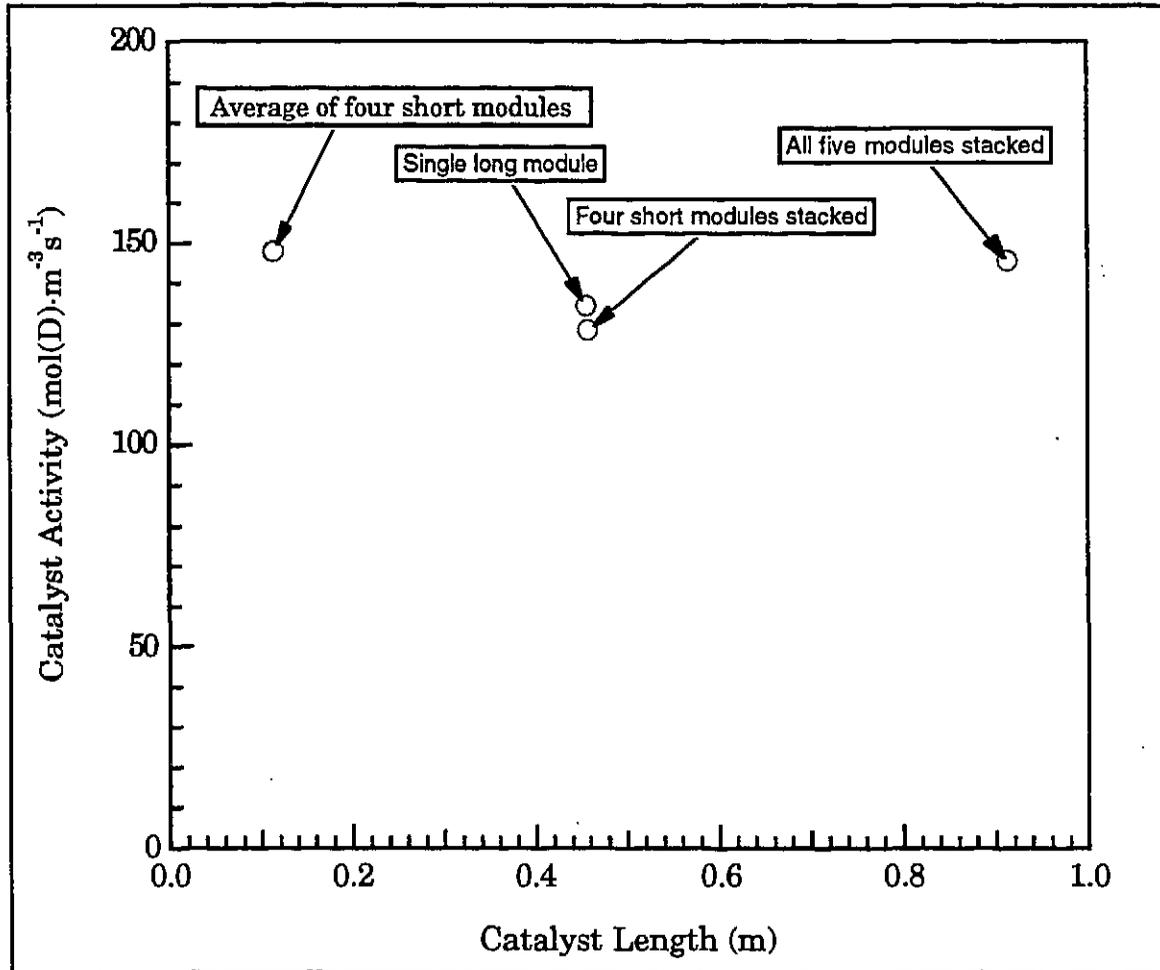


Figure 4: Example of Maintenance of Catalyst Activity with Increasing Packed Column Height

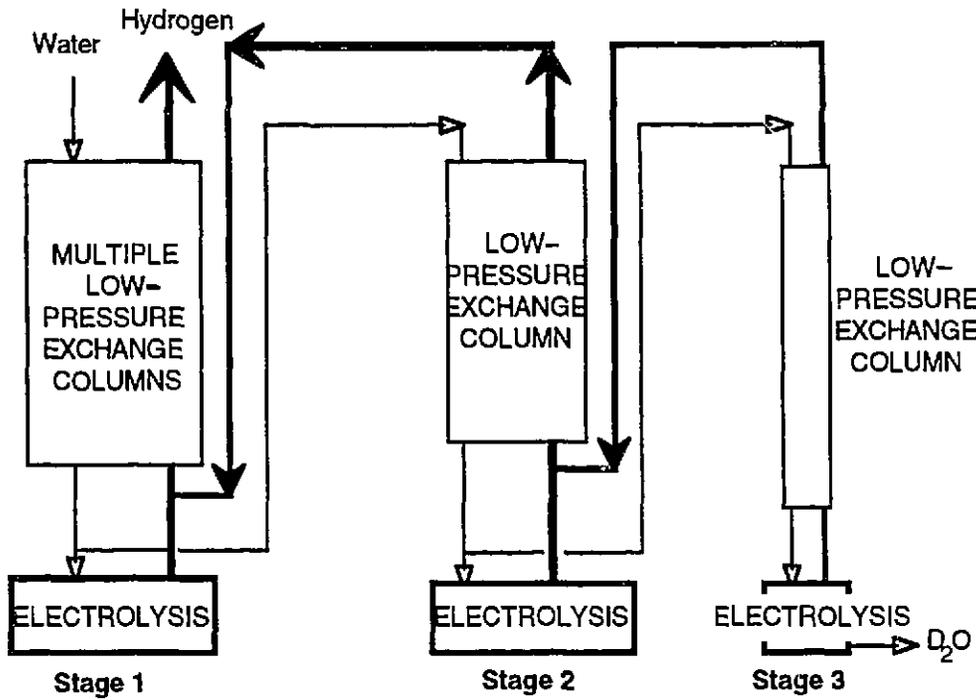


Figure 5: Simplified Schematic of a CECE Process

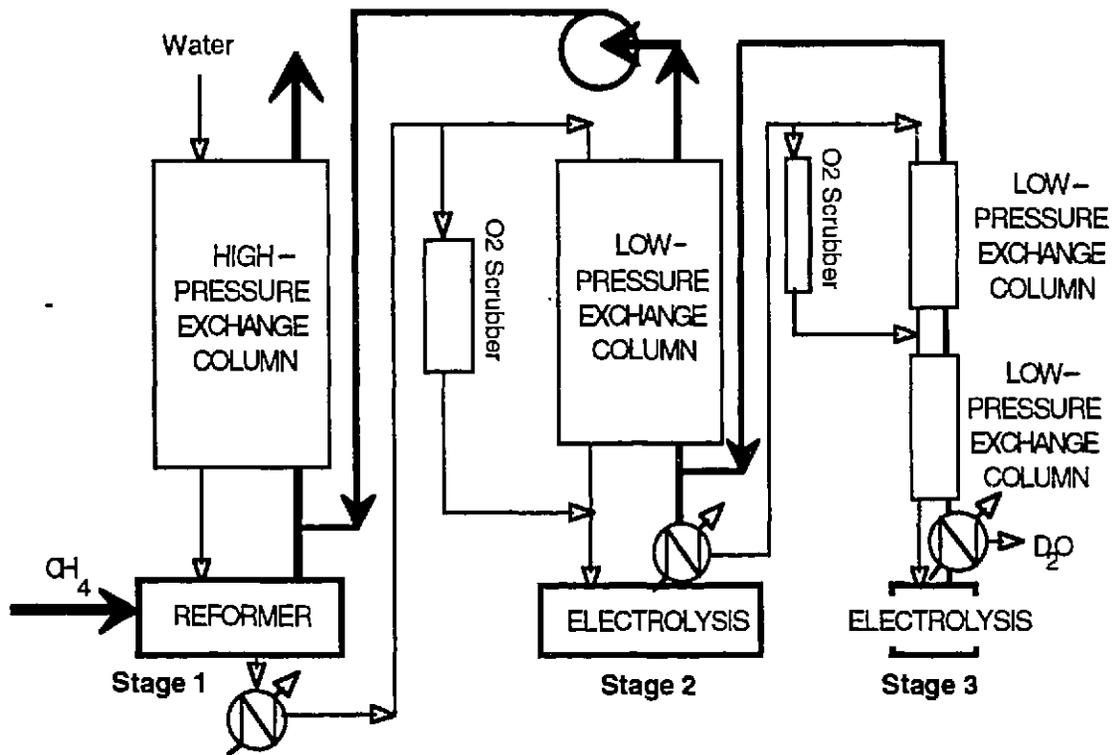


Figure 6: Simplified Schematic of a CIRCE Process

A second parasitic process, Combined Industrially Reformed hydrogen and Catalytic Exchange (CIRCE), harnesses hydrogen-water exchange to hydrogen production by steam reforming. This is illustrated in Figure 6. Note that the second and higher stages actually use the CECE process. The first stage in CIRCE is also a monothermal process, but conversion of hydrogen to water is done by steam reforming, and this introduces many complications compared to the CECE process. The complications are summarized in Table I.

Table I: Factors of Relative Complexity for the CIRCE Process

	Complication	Consequences
1	Hydrogen isotope exchange must take place at the high pressure of the reformer (typically 2 to 3.5 MPa).	The activity of wetproofed catalysts is found to decline as approximately the square root of pressure: volume of catalyst increases 4.5- to 6-fold.
2	Water flow provides only half the hydrogen generated in the reformer.	Water must extract twice as much deuterium from the hydrogen stream as the water flow in the CECE process, which means weaker driving forces and longer columns.
3	The reformer becomes the equivalent of the electrolysis cell of the CECE process.	The reformer contains elevated levels of deuterium, which means that losses of any deuterated species (H_2 , H_2O , and CH_4) must be maintained at very low levels. CH_4 is particularly difficult, since it will pass through the water-hydrogen exchange column unaltered.
4	Steam-reformed hydrogen usually contains traces of CO, and Pt catalysts are poisoned by CO.	CO must either be removed or be shift-converted to CH_4 (but this would cause unacceptable losses of deuterium, unless CO levels are low), or be tolerated by a specially formulated catalyst, or be absorbed on platinum on a guard-bed ahead of the main exchange column.

The first two complications are unavoidable. The third is plant-specific. (Figure 7 shows the major changes that would be needed to adapt CIRCE to one design of steam reformer using pressure swing absorption for hydrogen purification.) The fourth complication can be dealt with generically and, on it, recent work by AECL Research has produced preliminary but encouraging results for catalysts able to methanate CO at low temperatures and for water-hydrogen exchange catalysts resistant to CO.

Although it is obviously more complex than the CECE process, the main attraction of the CIRCE process is the widespread availability of relatively large plants producing hydrogen by steam reforming. Each 1000 Mg/d ammonia plant, for example, has the capacity to produce over 50 Mg/a of heavy water, and the production cost should be very competitive with production from an existing G-S plant.

The final process to harness water-hydrogen exchange is the Bithermal Hydrogen Water (BHW) process, which is illustrated in Figure 2. This is very similar to the G-S process, but with the advantages of much superior separation factors, lower energy consumption, and non-toxic and non-corrosive process fluids. Offsetting these is the disadvantage of requiring exchange catalyst in both

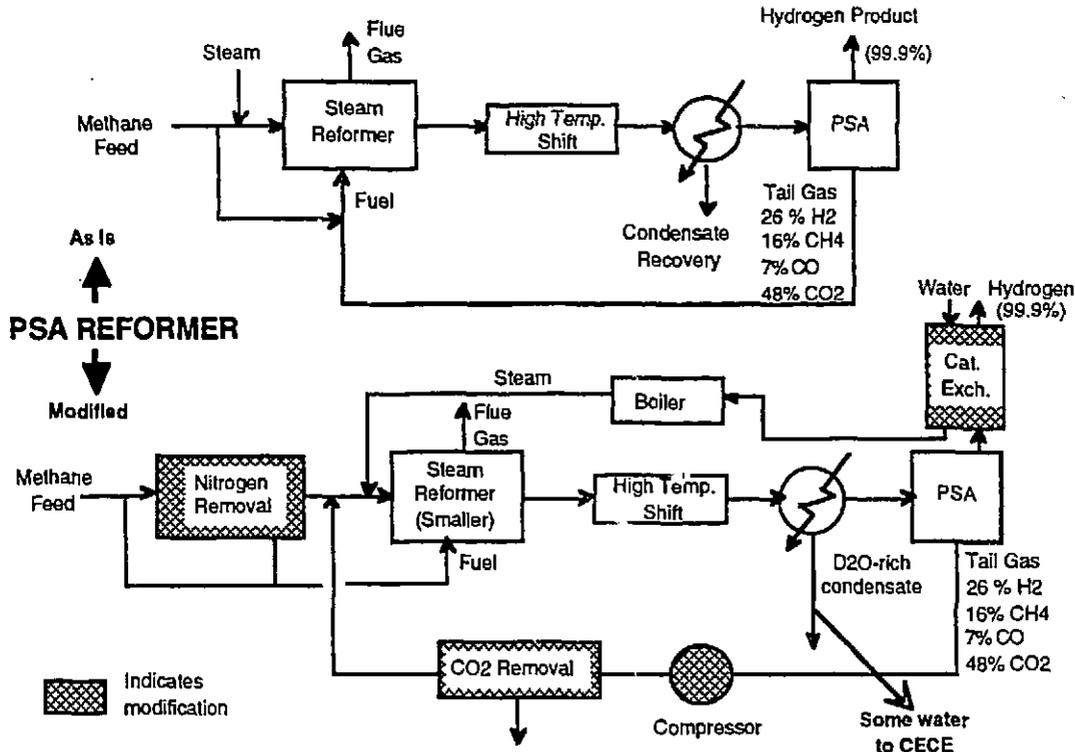


Figure 7: Schematics of a Steam Reformer using Pressure Swing Absorption both Unmodified and Modified to Accommodate a CIRCE Plant

towers. However, vis-a-vis a new G-S plant, BHW is considered to have clearly superior economics, even after making allowance for a recent observation in AECL's program that Teflon-based wetproofed catalyst may degrade very slowly at temperatures close to 200°C, thus putting a limit on the temperature of the hot towers.

7. SUMMATION

AECL is planning to shift its heavy-water production from G-S to water-hydrogen based processes using our proprietary wetproofed catalyst. Work on the catalyst has progressed well. It is already amply capable of supporting the CECE process, and is coming close to meeting the required performance for an economical CIRCE process.

AECL's immediate emphasis is on the monothermal CIRCE process, supplemented wherever the opportunity arises by the CECE process. A small amount of effort continues to be directed to the BHW process. By deploying both CIRCE and CECE processes worldwide, this program will assure continuing supplies of heavy water for CANDU reactors that can meet any conceivable demand for heavy-water production at a cost that maintains the competitiveness of CANDU reactors.

8. ACKNOWLEDGEMENTS

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