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**Technology Developments for Improved
Tritium Management**

**Progrès réalisés en techniques pour l'amélioration
de la gestion du tritium**

J.M. Miller, W.T. Shmayda, S.K. Sood, D.A. Spagnolo

Presented at the International Atomic Energy Agency Technical
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AECL Research

TECHNOLOGY DEVELOPMENTS FOR IMPROVED TRITIUM MANAGEMENT

by

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RÉSUMÉ

Les progrès réalisés en techniques du tritium font partie intégrante de l'amélioration de la technique des réacteurs CANDU. La compréhension du comportement du tritium dans les systèmes d'eau lourde conduit à l'amélioration des procédés de récupération du tritium, des techniques de mesure du tritium et de la gestion générale du tritium. On met en application la technique de détritiation dans le cadre des pratiques de gestion de l'eau lourde et du tritium. On résume les progrès réalisés en ces techniques.

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ABSTRACT

Tritium technology developments have been an integral part of the advancement of CANDU reactor technology. An understanding of tritium behaviour within the heavy-water systems has led to improvements in tritium recovery processes, tritium measurement techniques and overall tritium control. Detritiation technology has been put in place as part of heavy water and tritium management practices. The advances made in these technologies are summarized.

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TABLE OF CONTENTS

Page

1.	INTRODUCTION	1
2.	DETRITIATION TECHNOLOGY	2
2.1	Technology Options and Strategies.....	2
2.2	Operation of the Darlington Tritium Removal Facility (DTRF)	5
2.2.1	Plant Performance.....	5
2.2.2	Tritium Management	6
3.	R&D PROGRAMS	6
3.1	Program Direction.....	6
3.2	Tritium Removal from Air Atmospheres	7
3.3	Tritium Measurement.....	8
3.4	Tritium and Materials Interactions	9
4.	SUMMARY.....	10
5.	ACKNOWLEDGMENTS	10
6.	REFERENCES.....	10

1. INTRODUCTION

The presence of significant amounts of tritium is a unique and characteristic feature of heavy-water reactors. In the development of CANDU® (Canadian Deuterium Uranium) reactor technology, tritium management activities have, and continue to be, an integral part of the developments carried out by design, operations and research personnel. The focus has been on improving tritium containment, tritium removal and tritium measurement, to mitigate man-rem exposures and emissions to the environment.

While sophisticated tritium control measures are included in operating CANDU stations, occupational dose from tritium typically ranges from 30-50% of the total dose. In particular, tritium is a significant contributor to maintenance dose and, hence, overall maintenance costs. The suggestion of the International Commission on Radiation Protection (ICRP) to limit worker dose to an average 20 mSv per year over five years, and the relatively high contribution of tritium to the overall total dose, provides incentive to develop improvements in tritium-control technology for operating stations and advanced designs.

In the heavy-water-moderated and -cooled CANDU reactors, the rate of tritium production is directly proportional to the neutron flux and to the time the heavy water is exposed to this flux. With the moderator and coolant of CANDUs on separate circuits, tritium formation in the moderator dominates, since the residence time of the coolant in the reactor core is significantly shorter. Annual tritium production for a CANDU is typically 1.4-1.5 kCi*/MW(e). Of this, only 2.5-3.0% is generated in the coolant, but because of its higher operating pressure and temperature, tritium releases from the coolant contribute approximately half of the tritium dose. Great care is taken to keep both coolant and moderator circuits separate, to minimize potential cross contamination and to limit the coolant tritium concentration to below 2 Ci/kg (preferably <1 Ci/kg). An increase in the tritium concentration in the moderator is of less concern, with present concentrations of some CANDU 6s approaching 40 Ci/kg. Without detritiation or heavy-water replacement, the tritium concentration in the moderator would equilibrate at about 80 Ci/kg.

Tritium removal or detritiation of the heavy-water systems is a technology already in place at Ontario Hydro, and is under consideration by other CANDU operators and designers. This extends tritium management practices from that required for a few parts per million tritium in the reactor system to that necessary for the containment, measurement and handling of a very high specific-activity tritium product, T₂. Although there is additional handling at higher concentrations of tritium, the relative toxicity of the T₂ product is ~25 000 times less than that for tritiated water^[1] making overall dose reduction possible. Tritium-handling laboratories at Ontario Hydro Research^[2] and AECL Research^[3] have the capability of handling significant inventories of T₂, allowing R&D work to be carried out in experimental and pilot-scale facilities, to circumvent costly operational or design changes within the operating facilities. The operating experience gained from these laboratories also allows for improvements in tritium measurement, decontamination, and general handling procedures used in plant operations.

An overview of detritiation technology, R&D results and their impact on tritium management practices is presented.

*Ci = 3.7 x 10¹⁰ Bq

2. DETRITIATION TECHNOLOGY

2.1 Technology Options and Strategies

Ontario Hydro is the only CANDU utility that has committed to detritiating its heavy water. This is done at a centralized site at the Darlington Nuclear Generating Station (DNFS). All other CANDU utilities have so far limited their efforts to reduce tritium by simply replacing circuit losses with low-tritium heavy water. However, interest by utilities in larger scale detritiation is steadily increasing. Future CANDU targets could require moderator and coolant concentrations as low as 5 Ci/kg and 0.2 Ci/kg, respectively.

Continuous detritiation of the moderator and coolant can be tackled in two ways:

- (1) Detritiate the moderator and coolant in separate campaigns.
- (2) Detritiate the moderator and return the detritiated product to the coolant, thus displacing coolant (after upgrading) to the moderator.

The latter is used at Ontario Hydro and is preferred, provided the tritium extraction efficiency of the detritiation plant is sufficient to produce a detritiated product with a lower tritium concentration than exists in the coolant.

The process of detritiating water consists of three main steps:

- (1) A front-end that handles the tritium in the aqueous phase and exchanges it to the less toxic hydrogen phase. This can be done either through chemical exchange in the presence of a platinum-based catalyst, $\text{DTO} + \text{D}_2 = \text{D}_2\text{O} + \text{DT}$, or through the decomposition of water, $\text{DTO} \rightarrow \text{DT} + \text{O}_2$.
- (2) A back-end that concentrates the tritium in the hydrogen phase. Cryogenic hydrogen distillation (C-D) at ~ 23 K is usually preferred for this service, since other potential candidates, such as gas chromatographic methods, do not scale-up economically.
- (3) A means of immobilizing the concentrated tritium and storing it safely. Uranium metal is used if storage is temporary; titanium has been selected for permanent storage.

Several technology options exist for the front-end step. The most industrially proven process is the Vapour Phase Catalytic Exchange (VPCE), which was developed and patented by the Commissariat à l'Énergie Atomique (CEA) of France and first used to detritiate the high-flux research reactor in Grenoble.^[4] This plant was built by Sulzer under licence from the CEA. This same technology was later used on a much larger scale by Sulzer Canada for Ontario Hydro at the Darlington Tritium Removal Facility.^[5]

The VPCE is schematically illustrated in Figure 1. The process normally comprises between three to eight stages, each stage consisting of a water evaporator, superheater, catalyst bed and condenser/separator. Since conventional platinized catalyst is used, superheating is required to minimize steam condensation within the catalyst pores, which would inhibit isotopic exchange. Heavy water is fed to the first VPCE stage, where it is evaporated and mixed with cold deuterium gas returning from the condenser/separator of the subsequent stage. This gas/vapour mixture is superheated to 200°C prior to entering the catalyst bed. Here tritium is exchanged and equilibrated. The deuterium gas, somewhat enriched in tritium, is separated from water in the condenser and fed to the back-end C-D system. The tritium-depleted water flows to the evaporator of the subsequent stage. Detritiated water from the last-stage condenser is fed back to the reactor as product. Deuterium gas feeding the last stage comes from the C-D system, where it has been depleted of its tritium.

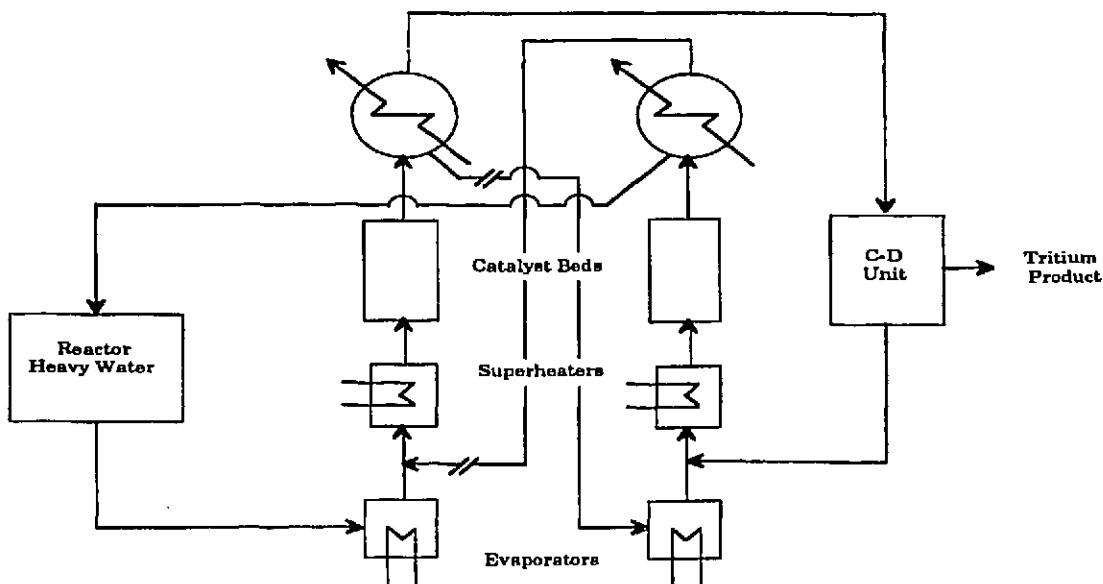


Figure 1 - VPCE Process

With the development of a wetproofed catalyst by AECL,^[6] this catalytic isotopic exchange process can be greatly simplified to a packed catalyst column, with liquid entering from the top and trickling down the column in counter-current contact with deuterium gas that is flowing up. Wetproofing allows the catalyst to operate in liquid or very humid environments. This process, which operates at 50°C, is referred to as Liquid Phase Catalytic Exchange (LPCE), and is illustrated in Figure 2. The Chalk River Laboratories (CRL) Tritium Extraction Plant (TEP)^[7] was based on this process. However, because of the shut down and subsequent decommissioning of all AECL's prototype heavy-water reactors, need for the TEP was no longer justified and thus the plant has not been commissioned. This process and catalyst is patented by AECL, with Sulzer Canada as a licensee.

A third option for the front-end is Combined Electrolysis and Catalytic Exchange (CECE). This process has also been patented by AECL, with Sulzer Canada as a licensee for tritium extraction/upgrading. Unlike the other options, this process does some pre-enrichment of the tritium stream before sending the deuterium to the C-D. As the name implies, and as illustrated in Figure 3, this process consists of LPCE columns and electrolytic cells. The catalyst column is divided into stripping and enriching sections. Tritiated water is fed to the top of the enriching section, in which it picks up tritium from the electrolytic deuterium produced from the water leaving the bottom of the column. (Electrolysis causes a further — generally unexploited — tritium enrichment in the liquid phase, because its separation factor approaches 2.) A side stream of tritium-enriched deuterium gas is taken from the electrolysis cell as feed to the C-D system, and is returned detritiated higher up in the exchange column. The deuterium gas flowing through the stripping section is stripped of tritium by the detritiated reflux water formed by catalytic recombination with oxygen produced in the electrolytic cells. The detritiated product water is also drawn from the recombiner.

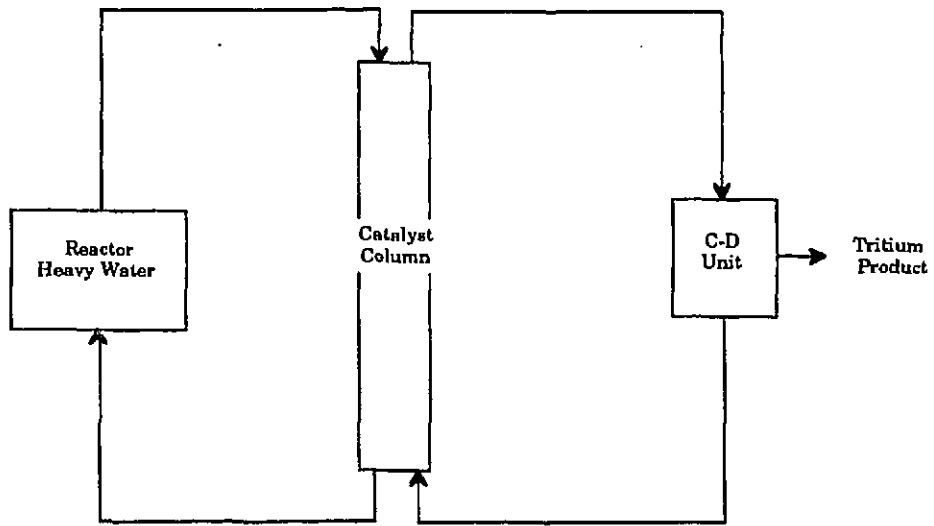


Figure 2 - LPCE Process

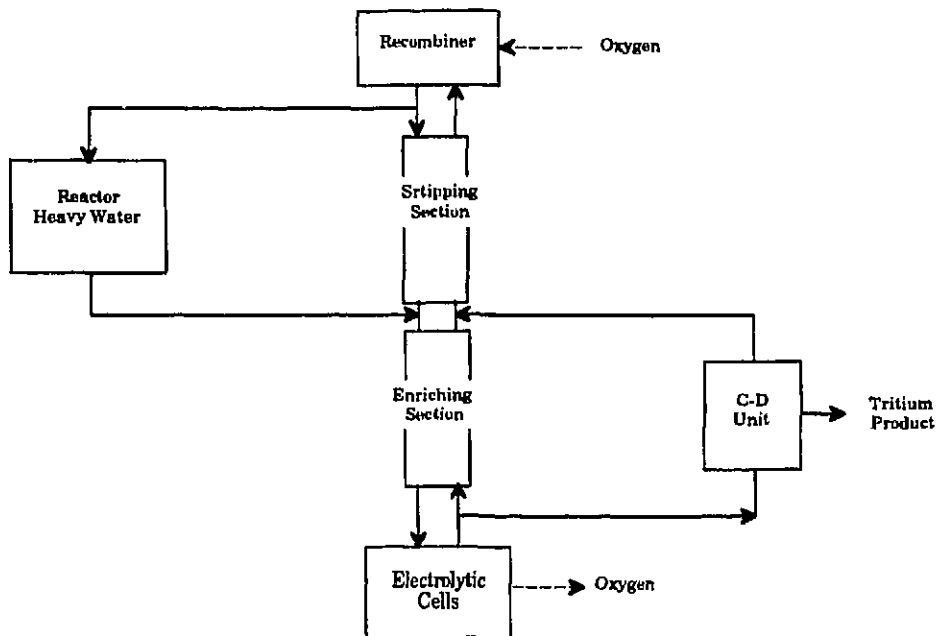


Figure 3 - CECE Process

A small-scale CECE was built and operated by Monsanto Research Corp. at the Mound Laboratories,^[8] in collaboration with AECL, for tritium recovery from light water. AECL is presently investigating various schemes, based on CECE, to detritiate CANDU reactors. A version of the CECE, which will not only detritiate but also upgrade heavy water and detritiate light water wastes, has been chosen as the reference design concept for the Advanced Neutron Source (ANS) reactor at Oak Ridge (US). A similar scheme could be applied to future CANDUs, with CECE providing the upgrading function, replacing the water distillation (DW) technology presently used.

The last technology option that has frequently been considered for the front-end is electrolysis, in which the heavy water is completely electrolyzed to deuterium and oxygen. The deuterium gas is fed directly to the C-D system, where it is detritiated, and then sent to a catalytic or flame-type recombiner to reform with the oxygen from the electrolytic cells, to produce the detritiated water product. This option is power-intensive and expensive. The same could be said for the CECE, which also employs even more electrolysis, except that the CECE can pre-enrich the feed stream substantially and concomitantly reduce the size and refrigeration power requirements of the back-end C-D system.

Other options for tritium pre-enrichment and/or phase conversion have at times been mentioned, such as water distillation, laser isotope separation and chemical decomposition, but for various reasons none have been considered as serious contenders.

Significant progress has been made in the development of low-inventory cryogenic distillation systems at Ontario Hydro. This has allowed the design of C-D systems that are compact and have very low tritium inventory. On this basis, Ontario Hydro has designed a C-D system for the Princeton Plasma Physics Laboratory, which is expected to produce pure tritium, but with a working inventory of less than one gram of tritium. This reduction in inventory can help to reduce the potential hazards of CANDU detritiation systems, and also make the licensing of such facilities much easier.

2.2 Operation of the Darlington Tritium Removal Facility (DTRF)

Ontario Hydro owns and operates 20 CANDU reactors, with a rated capacity of 14 160 MWe. About 60% of Ontario Hydro's total generating capacity is nuclear-based. The Darlington TRF is intended to service all Ontario Hydro's nuclear reactors, and has been in operation since 1989, removing tritium primarily from the heavy-water moderator systems of the reactors. The DTRF was originally constructed to help reduce the occupational tritium hazard and environmental tritium emissions from Ontario Hydro's reactors. The DTRF has become a vital part of the D₂O management system, reducing the demand for virgin D₂O by processing tritiated heavy water that had been stored, thus freeing it for re-use.

The DTRF process is based on a VPCE system for exchanging tritium from heavy water into recirculating deuterium gas, and a C-D system for concentrating the extracted tritium.

2.2.1 Plant Performance

A total of about 4300 Mg of heavy water with an average concentration of 20 Ci/kg has been processed by the facility, at a rate of 360 kg/h. As a result, approximately 57.4 MCi of tritium gas, with an atomic purity >97%, has been immobilized as titanium tritide, and is stored at the DTRF.

Several problems were encountered during commissioning and early operation of the DTRF, typical of a complex and prototypical engineered facility. Leakage was experienced in the VPCE

evaporator tubes, caused by stress-corrosion cracking of stainless steel due to residual chlorides migrating from the VPCE catalyst. The stainless-steel tube bundles in all nine evaporators were replaced with tube bundles made of Inconel 625. No further cracking has been observed since the bundles were replaced.

To establish stable C-D operation, the Pt-on-charcoal-based catalyst in the equilibrator, located on the vapour exit line from the final distillation column, was replaced with a Pt-on-alumina catalyst. Some line sizes around the distillation columns were increased. This involved decontaminating, cutting and welding highly contaminated lines. These operations were completed with minimal worker dose.^[9]

Some other problems involved the reliability of valves around molecular-sieve dryers, reliability of liquid-ring compressors and blockage of filters in the refrigeration system. All of these problems were successfully overcome by early 1992, and the DTRF has been operating at or above its rated capacity since then.

2.2.2 Tritium Management

The DTRF has treated water from the Pickering and Bruce reactors, as well as tritiated water that had been stored as part of Ontario Hydro's tritium displacement program. In this manner, 1200 Mg of D₂O was detritiated to provide heavy water for filling the moderator systems of the Darlington reactors.

Heavy water from Pickering and Bruce is transported to the DTRF on flat-bed trucks equipped with two type B(U) containers, each with a capacity of 5 Mg. At Pickering and Bruce, systems installed for on-line moderator upgraders are used to fill and empty the transportation containers. One truckload of heavy water is equivalent to a day's operation of the DTRF.

The DTRF was used initially to reduce moderator concentrations at Pickering "A" and "B" stations. During 1989 to 1991, moderator concentrations were reduced from about 18-20 Ci/kg to about 10 Ci/kg. In 1991, the DTRF started processing water from the Bruce "A" and "B" reactors. The moderator concentrations at Pickering, therefore, have since climbed slowly back to an average of about 14 Ci/kg. In the meantime, Bruce "A" moderators, which were as high as 34 Ci/kg, have been reduced to about 24 Ci/kg, and Bruce "B" moderators have been reduced from about 30 Ci/kg to about 26 Ci/kg. The tritium levels in the Bruce "A" heat transport systems have also been lowered from 2.2 Ci/kg to 1.9 Ci/kg. This has been accomplished by using low-curie D₂O, mainly from the DTRF, to displace the water in the heat-transport systems. These reductions in concentration have had a positive impact on worker safety and tritium emissions.

3. R&D PROGRAMS

3.1 Program Direction

R&D programs are in place to help the reactor operators and designers achieve effective tritium control. Various technologies employed in the DTRF and CRL's TEP were developed and demonstrated via internal R&D programs. Considerable advances have been made in understanding tritium behaviour within reactor and processing systems and worker environments. Tritium recovery from atmospheres and exhaust streams, decontamination techniques, tritium measurement, detritiation methods, and tritium storage are areas of special interest. Recent developments in some of these program areas are highlighted below.

3.2 Tritium Removal from Air Atmospheres

Desiccant dryers employed in vapour-recovery dryer systems (VRDs) and air detritiation dryers (ADDs) are used as the final barriers to control airborne tritium emissions from CANDU stations and tritium extraction facilities, respectively. The VRDs have a dual role: to minimize heavy-water losses and to minimize tritium levels within the reactor building that are emitted to the environment. R&D work at CRL has shown that changes to the methods of operating and regenerating these dryers can reduce tritium levels in their exhaust.^[10] It has been demonstrated that the concentration of tritiated water exiting an ADD/VRD during adsorption is a strong function of the residual HTO remaining on the bed from the previous regeneration cycle. This means that tritium (HTO) and heavy-water control are not identical to moisture control.

Figure 4 illustrates the effect of residual HTO on the outlet tritium concentration from a pilot-scale dryer. The humidity and tritium concentration of the feed air to the two dryer beds were kept constant, but one bed was regenerated with tritium-free water while the other was regenerated with 17 Ci/kg water. The outlet tritium concentration from the latter "used" bed was significantly higher, as shown Figure 4. This result demonstrates that the dryer exhaust is not necessarily a reflection of the current airborne tritium concentrations, particularly if the specific activity of the water in the room or building environment has changed over the cycle time of the dryer system. This has implications for the monitoring techniques used to track the dryer performance (requiring moisture (hygrometer) and tritium measurement), and for the use of the VRD outlet activity to identify leaks, as it will lag the inlet conditions by one operating cycle. Thus, regeneration techniques play a major role in determining the performance of the dryer unit, and must be well-defined and controlled. An experimental program is currently examining the kinetics and effect on desiccant life of "steam-washing", a technique in which non-tritiated steam is used to wash the residual tritium from the dryer bed during the regeneration cycle, so that in theory a dryer could operate with no tritium or heavy water in its exhaust (100% efficient).

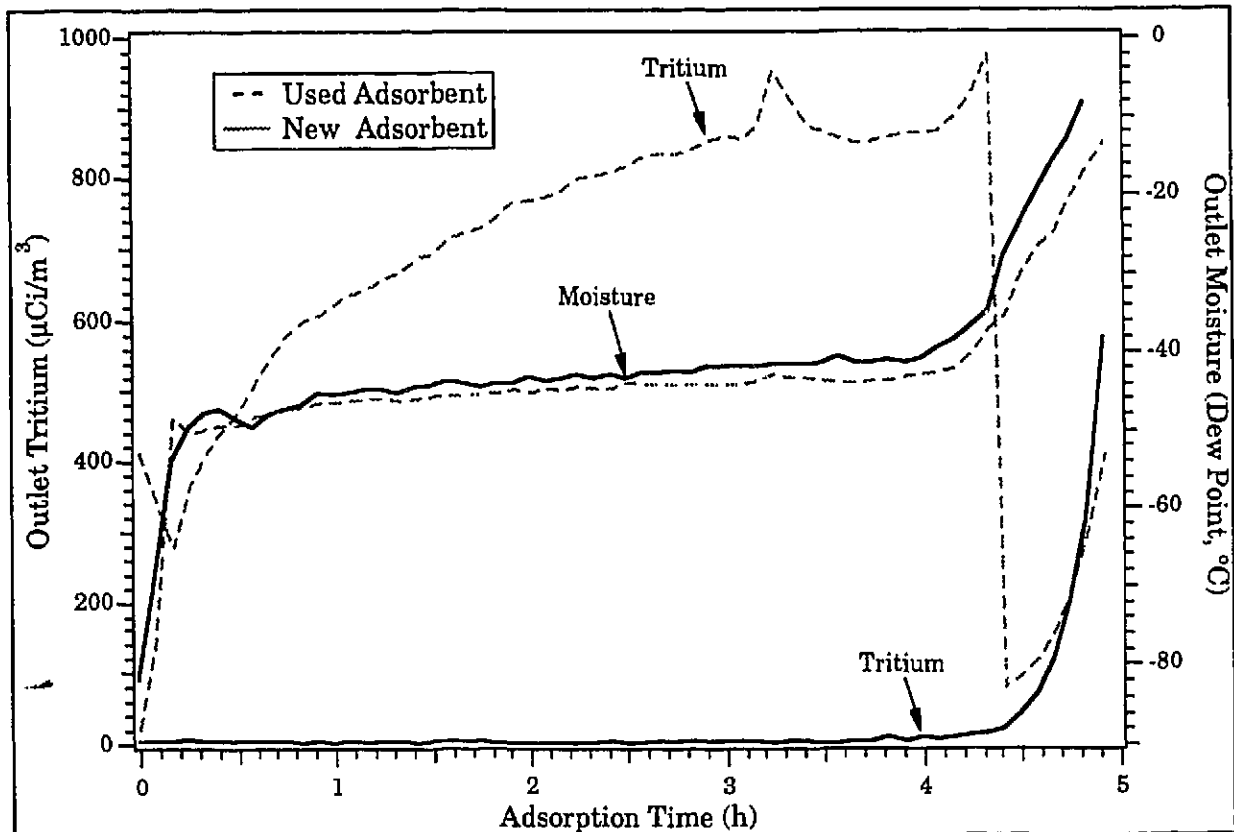


Figure 4 - Effect of the Residual Tritium of the Adsorbent Bed on Outlet Humidity and Tritium Concentration

A dynamic dryer simulation, incorporating experimentally obtained physical-property data and treating the bed as a multi-component adsorber, is being developed for use as a diagnostic tool. This will assist with the analysis of existing dryer systems, and act as a design tool for predicting the effects of various system changes. Such a tool will help improve failure analysis, limit the resources required to diagnose and maintain dryers within operating systems, and help ensure that the desired performance in new designs will be achieved.

3.3 Tritium Measurement

A key element of demonstrating tritium containment within reactor systems or tritium extraction facilities is to have available accurate tritium measurement techniques. Tritium monitoring programs for health-physics purposes and compliance with emission limits have been integral parts of the development of CANDU technology. However, to allow more accurate and timely assessments of process performance and operational upsets, real-time monitoring of work environments, process streams, and effluent or exhaust streams would provide a definite advantage. This would also be of particular help in characterizing man-rem exposures, to help achieve the lower targets in advanced designs. For this broader scope and the more restrictive operation, the development of measurement techniques for a wide range of concentrations, tritiated sources, and process conditions is required.

To help improve tritium-release control, selected commercial tritium monitors are being evaluated for their ability to measure tritium accurately on-line in the low-humidity atmosphere of an ADD or VRD exhaust, or in stack exhaust gases. Erroneous readings due to the presence of gamma fields are well known, but the dry atmosphere can also be a concern for the commonly used ionization chamber-type monitor. Contamination of the interior surfaces of the tritium monitor by sorbed tritiated water can result in significantly higher readings than the true value.^[11] This is commonly referred to as the "memory effect". Humidifying the carrier stream for measurement is a solution that has been proven, but this technique has some practical limitations in the reactor environment. Improvements in construction material selection and chamber design are being pursued. Along this line, Ontario Hydro Research have designed and tested a simple and compact ionization chamber that is bakeable to 300°C.^[12] This heating feature can limit memory-effect problems and allow rapid recovery from a high-background contamination following exposure to high specific-activity tritium. On-line liquid effluent monitoring systems are also being evaluated, to determine whether they can provide the necessary compliance monitoring of liquid emissions in a timely manner. Station evaluation of commercial radio-chromatography detection units has recently commenced.

The operation of a detritiation facility such as the DTRF provides many new requirements for tritium measurement, particularly in the concentration and packaging processes, where tritium is concentrated from a few ppm DT to pure T₂. Specially designed ionization chamber-type process tritium monitors^[13] have been assessed over a wide range of operating conditions and concentration levels. The effects of system pressure, gas composition, tritium concentration, and surface contamination on the calibration of these instruments have been determined, and operating limits defined.^[14] Figure 5 shows the excellent reproducibility and linearity achievable in measuring 95% T₂ from 40 to 100 kPa with a "cross-type" monitor design. Tritium losses to the walls are responsible for the non-linearity below 50 kPa in this design. Based on the test results, these monitors can be used in various locations in tritium extraction facilities as process diagnostic tools, to help ensure safe and reliable operation of the process.

Mass spectrometry is used to measure the purity of the T₂ product stream at the DTRF. Hydrogen isotope gas chromatography has also been developed for this purpose, and is used for compositional analysis of the various tritium sources in CRL's Tritium Laboratory.^[15] In the DTRF, the T₂ product is immobilized on titanium metal for secure, long-term storage of this

strategic material. Accountability of the T_2 product may become a concern, and the use of calorimetry to verify the tritium content of the titanium storage package has been investigated.

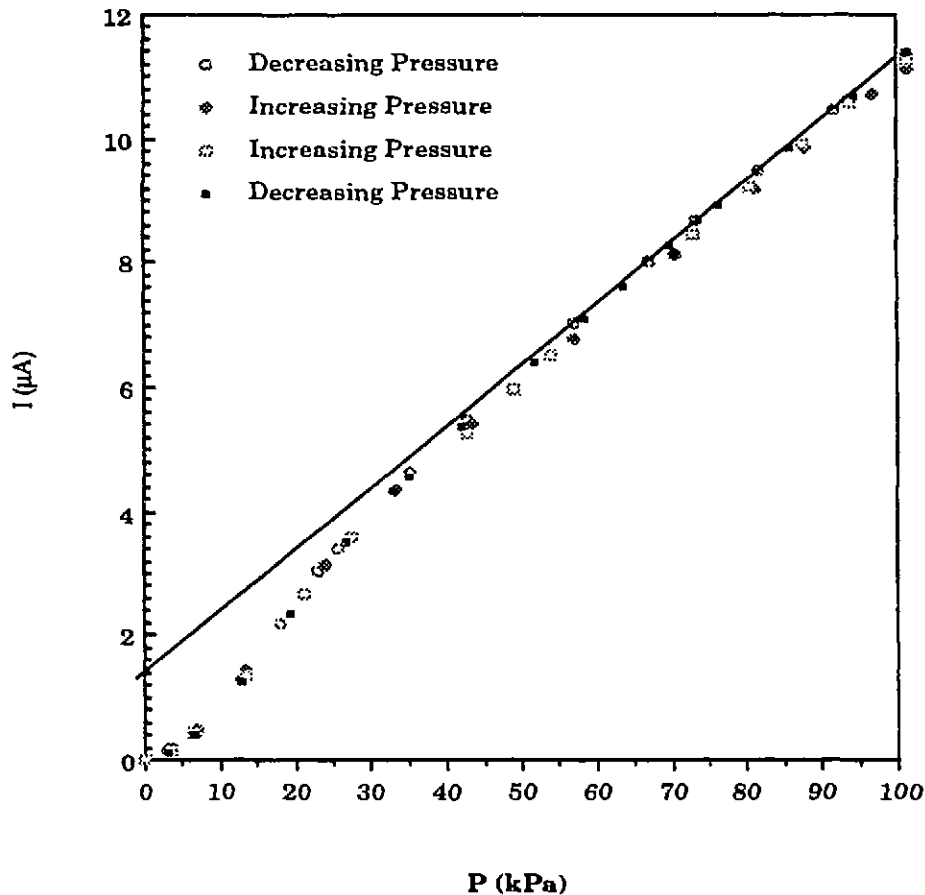


Figure 5 - Performance of Process Monitor in Measuring 95% T_2 , Over Pressure Range to 101 kPa

3.4 Tritium and Materials Interactions

Tritium, as elemental hydrogen or water vapour, is readily adsorbed by, and penetrates into, most materials. It can also be incorporated into the structure of organic compounds, such as those that constitute pump oils and valve seats, compromising their material properties. Once adsorbed, it will subsequently outgas, giving rise to chronic airborne contamination.

Understanding tritium materials interactions is necessary to ensure tritium containment within the intended process system, and to identify possible tritiated sources: contaminated components, waste products, and building or room atmospheres. Contaminated components can include something as simple as tools or measuring instruments, or much more complex process equipment that must be maintained or removed from service.

Various studies have been undertaken to identify the tritiated compounds present on metal surfaces through the analysis of thermal desorption spectra,^[16] in an effort to assist in the development of tritium dosimetry models. Analysis of stainless-steel surfaces exposed to pure tritium gas has indicated that the tritium is desorbed largely as HTO and that a small fraction is

also present in more complex forms. Methods to determine their composition, suspected to be tritiated methane and tritiated ammonia, are being developed.

A number of techniques are used to decontaminate tritium-containing systems or components, including a combination of washing, purging, thermal desorption and isotopic exchange.^[17] As mentioned in Section 2.2, a combination of these techniques has been successfully used for the necessary modifications to the DTRF after operation and production of pure T₂ within the process system. Plasma-driven cleaning has been developed by Ontario Hydro Research as a decontamination technique, which avoids production of other low-activity tritiated wastes.^[18] It has been demonstrated as a successful method for decontaminating uranium shipping beds used for the sale of tritium, and a decontamination workstation including this technique has been designed for the DTRF.

Metal getter technology—the use of metals and metal alloys to remove tritium and form a metal tritide—has been developed for the safe storage of the recovered T₂ product from detritiation facilities,^[19] and also for the purification of inert gas streams, such as glove-box atmospheres or purge gases.^[20] Storage and purification beds of titanium, uranium, and metal alloys of Zr/Fe and Zr/Fe/V (SAES Getters S.p.A., Italy) have been designed, and are in use in the research laboratories and in the DTRF for tritium storage and tritium release control.

4. **SUMMARY**

Tritium containment, tritium measurement and tritium removal have been identified as important technology areas for effective, proactive tritium management within the CANDU reactor system. R&D work, operating procedures, and design advances made in the tritium technology area contribute to the goals of improved operational efficiency, worker safety, and emission reduction for the CANDU reactor.

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