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AIR DETRITIATION DRYERS**

CFFTP-G-8902 (AECL-9900)  
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CFFTP-03420

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## TRITIUM ISOTOPIC EXCHANGE IN AIR DETRITIATION DRYERS

Report No. CFFTP-G-8902  
Cross Reference No. AECL-9900

1989 February

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## 1.0 EXECUTIVE SUMMARY

Air detritiation dryers at fusion facilities require high detritiation factors to minimize environmental releases. The technique of eluting the residual adsorbed tritiated water on a molecular sieve bed by H<sub>2</sub>O steam washing the bed after thermal regeneration has been suggested by Dombra [1] as a method of increasing the detritiation factor (DF). The method relies on additional detritiation of the air stream through isotopic exchange between the tritiated water vapor passing through a dryer bed and previously adsorbed non-tritiated water. This report presents data from two experimental programs studying isotopic exchange in molecular sieve beds, a laboratory scale test of isotopic exchange and an industrial scale demonstration of an air detritiation dryer using the steam washing technique. The validity of Dombra's isotopic exchange model is also assessed through comparisons of simulated and observed data.

Laboratory scale experiments were performed at comparatively humid conditions (+6°C dew point) and at varying temperatures. The purpose of the tests was to demonstrate isotopic exchange in a well-controlled environment, and compare the observed rate of isotopic exchange with previously reported data [4]. The tests were successful in clearly demonstrating the isotopic exchange effect. The rate of isotopic exchange at test conditions was shown to be lower than previously reported values.

The industrial scale demonstration of isotopic exchange was carried out at NPD (a small 25 MW CANDU generating station located near CRNL) using the boiler room air dryer system. Dryer inlet conditions were set at 54°C, a dew point of 6°C and tritium concentrations of 12-14 Ci<sup>1</sup>/kg water vapor. The purpose of the tests was to demonstrate the process of steam washing tritium from the sieve bed after thermal regeneration and the enhanced DF achieved as a result of H<sub>2</sub>O steam washing.

The air dryer system was put through four cycles of adsorption and regeneration with H<sub>2</sub>O steam washing. A DF of 12 600 was demonstrated. This is much higher than a DF calculated for humidity reduction only (430). Analysis of the data suggests that one order of magnitude in the observed detritiation performance can be attributed to isotopic exchange in the unsaturated zone of the bed (the portion of the bed believed to offer the most detritiation through isotopic exchange). A control test designed to illustrate a conventional dryer's detritiation performance yielded unusual results and was inconclusive.

The technique of eluting the residual tritium from an industrial-sized bed by H<sub>2</sub>O washing at high temperature, high humidity and low bed loading was demonstrated to be a fast and effective way of removing tritium from a molecular sieve bed during regeneration.

The isotopic exchange model accurately predicted the exchange between tritiated and non-tritiated water species in a molecular sieve bed where there is no net adsorption or desorption. The model's prediction of the tritium breakthrough trend observed in the NPD tests was poor. However, if the exchange rates were manipulated from those reported in reference 4, a forced fit could be achieved. More experimental data are needed to determine the tritium exchange rates in the mass transfer zone and the unsaturated zones of a dryer bed.

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<sup>1</sup> 1 Ci = 37 GBq

## 2.0 INTRODUCTION

Air detritiation systems for fusion facilities will require high performance molecular sieve dryers capable of achieving a high detritiation factor. To increase the detritiation performance of an air dryer system, above that achieved through adsorption of water vapor alone, Dombra [1] has proposed utilizing the process of isotopic exchange between tritiated water vapor passing through the bed and previously adsorbed tritium-free water. It is postulated that the dynamic equilibrium established between adsorbed water on the sieve and vapor over the sieve will further detritiate the air stream through exchange of tritiated for non-tritiated species of water if the bed initially contains tritium-free water

To effect an advantageous isotopic exchange, the molecular sieve must be pretreated to reduce the tritium concentration on the sieve. This is achieved by extending the conventional thermal regeneration process for a further 4-6 hours and passing tritium-free steam through the hot bed until the small residual quantity of adsorbed tritiated water on the sieve has been displaced by tritium-free water.

Pretreating a molecular sieve bed with tritium-free water offers enhanced detritiation of the air stream and, more importantly in a system where the influent tritium concentration can vary widely, eliminates any detrimental effects from previously adsorbed, highly tritiated water. These benefits are achieved at a moderate expense of increased complexity and time requirements of the regeneration cycle (a 4-6 hour steam wash) and increased volumes of tritiated waste water (50% increase in the demonstration dryer system).

Other alternatives for increasing the DF obtained on molecular sieve dryers in air detritiation systems have been considered. Tanaka [2] reports on experimental work investigating both the effect of adding H<sub>2</sub>O to the tritium contaminated feed and the effect of preloading the sieve bed with H<sub>2</sub>O (both techniques commonly referred to as isotopic swamping). He concludes there is little increase in the DF using either technique. The steam washing technique described in this report differs from the techniques studied by Tanaka in that the bed is not preloaded with H<sub>2</sub>O to "swamp" the incoming water vapor nor is the inlet process air stream diluted with H<sub>2</sub>O but, simply, tritium is thoroughly eluted from the small quantity of residual adsorbed water on the bed in a separate regeneration step after the bed has been thermally regenerated.

To study isotopic exchange in molecular sieve beds, two types of experiments were performed. Laboratory scale tests investigated isotopic exchange in a desiccant bed under static conditions (no net vapor adsorption or desorption) and an industrial scale demonstration of an operational air detritiation dryer using a pretreated (detritiated) molecular sieve bed was performed at NPD. Results from the experimental test programs are described in this report. Additionally, the ability of the isotopic exchange model, developed by Dombra and incorporated into an air detritiation dryer simulation [3], to fit the experimental results is discussed in this report.

## 3.0 LABORATORY EXPERIMENTAL PROGRAM

The purpose of the tests was, first to demonstrate the process of isotopic exchange in a well-controlled situation (i.e., no net water adsorption or desorption) and, secondly, to compare the observed isotopic exchange rates at different temperatures with those reported by Ono et al. [4], to assess the reported data. Observed isotopic exchange rates were determined by varying the model's exchange rate until the simulated results matched the experimental results, assuming the model accurately predicts the experimental data.

### 3.1 Apparatus and Method - Laboratory Tests

The experimental apparatus, shown in Figure 1, consisted of a 0.1 m diameter, 0.3 m high molecular sieve bed (Linde AW-500), a 206 L/min (STP) dry air supply, a constant water injection pump (1.5 g/min D<sub>2</sub>O), a 600 W heater for vaporization of the water and heating of the inlet air stream and the associated hygrometers, bubblers and ion chambers for the measurement of inlet and outlet tritium concentration.

To generate isotopic exchange on a sieve bed and eliminate interference from adsorption/desorption of water the following procedure was used:

- (a) Air, humidified with tritium-free water to a dew point of 6°C and heated to the prescribed temperature, was passed through the bed at a rate of 206 L/min (STP) for 4-6 hours until the bed outlet conditions matched the inlet conditions, thus leaving the bed uniformly loaded with tritium-free water and in equilibrium with the inlet conditions.
- (b) Maintaining the same inlet conditions (air flow rate, air temperature and water injection rate), but now using tritiated water to humidify the inlet air, the test was continued for a further 4-6 hours.

The tritium content of the inlet and outlet air streams was measured using bubblers and ion chambers. Tritium concentrations were converted from Ci/m<sup>3</sup> to Ci/kg vapor for consistency with the NPD dryer results. Vapor tritium concentrations, as Ci/kg vapor (C<sub>v</sub>), were calculated in the following manner.

$$C_v = \text{ICR or BR} \cdot \frac{1}{\text{SMV}} \cdot \frac{P_T}{P_o} \cdot \frac{1}{M_{wt}}$$

where ICR or BR is the ion chamber/bubbler response as Ci/m<sup>3</sup> at process conditions, SMV is the standard molar volume of 1 m<sup>3</sup> at process conditions assuming ideal gas behavior, P<sub>o</sub> is the equilibrium water vapor pressure at the observed dew point, P<sub>T</sub> is the ion chamber/bubbler total pressure at process conditions and M<sub>wt</sub> is the vapor molecular weight (kg/mol).

This "once through" experimental arrangement was chosen after an unsuccessful attempt to perform the experiment in a glovebox using an air blower to circulate the tritiated glovebox atmosphere through the bed. After several attempts it was conceded that the adsorption/desorption of tritiated water vapor on the multitude of surfaces in the glovebox was adding an unacceptable complication to the experiment.

### 3.2 Results - Laboratory Tests

Three laboratory experiments were performed to determine the isotopic exchange rate as a function of temperature in molecular sieve beds operating at a high humidity (dew point of 6°C). These conditions are characteristic of the saturated section in an operating room air dryer system.

Figures 2, 3 and 4 show the results from the three laboratory tests performed at temperatures of 38, 62 and 46°C, respectively. In each experiment, the results indicate that isotopic exchange has occurred between the tritiated water vapor passing through the bed and the tritium-free water adsorbed on the bed.



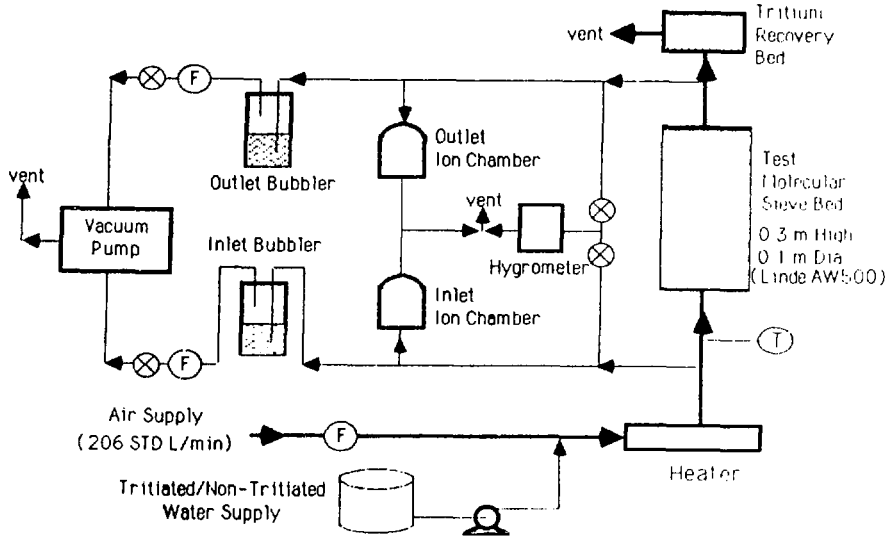


Figure 1 : Laboratory Apparatus for Isotopic Exchange Investigation

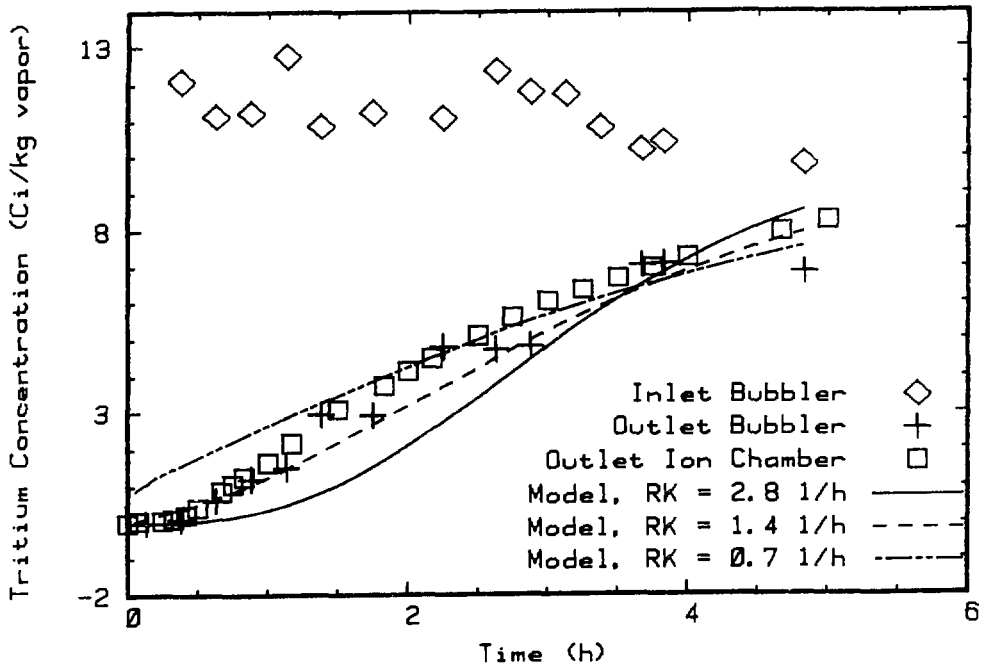


Figure 2 : Bed Outlet Tritium Concentration Trend  
Laboratory Isotopic Exchange Test #1 (Temp = 38°C)

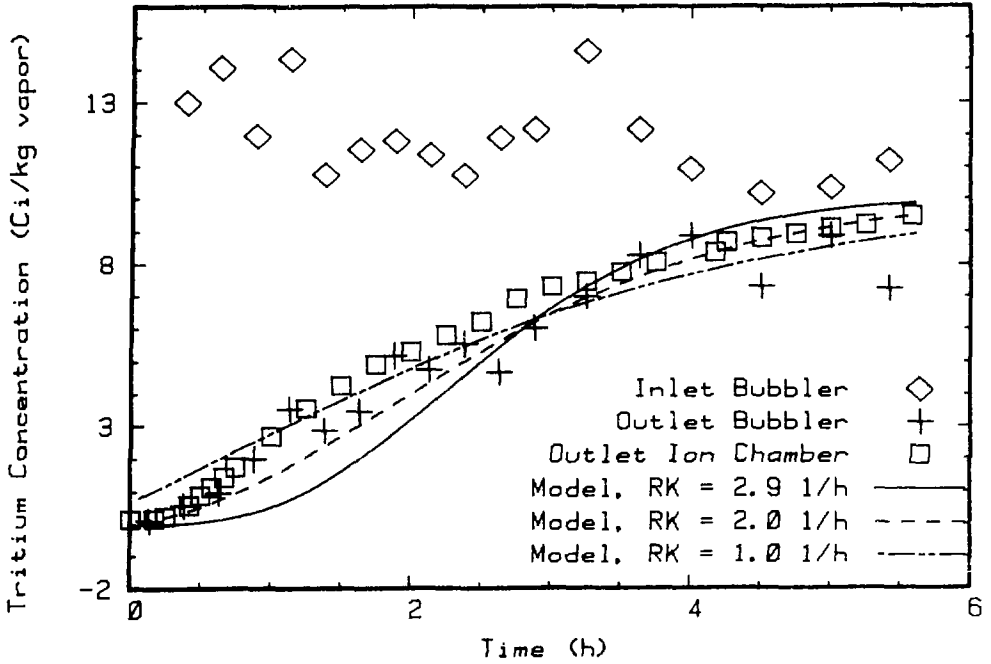


Figure 3 : Bed Outlet Tritium Concentration Trend  
Laboratory Isotopic Exchange Test #2 (Temp = 62°C)

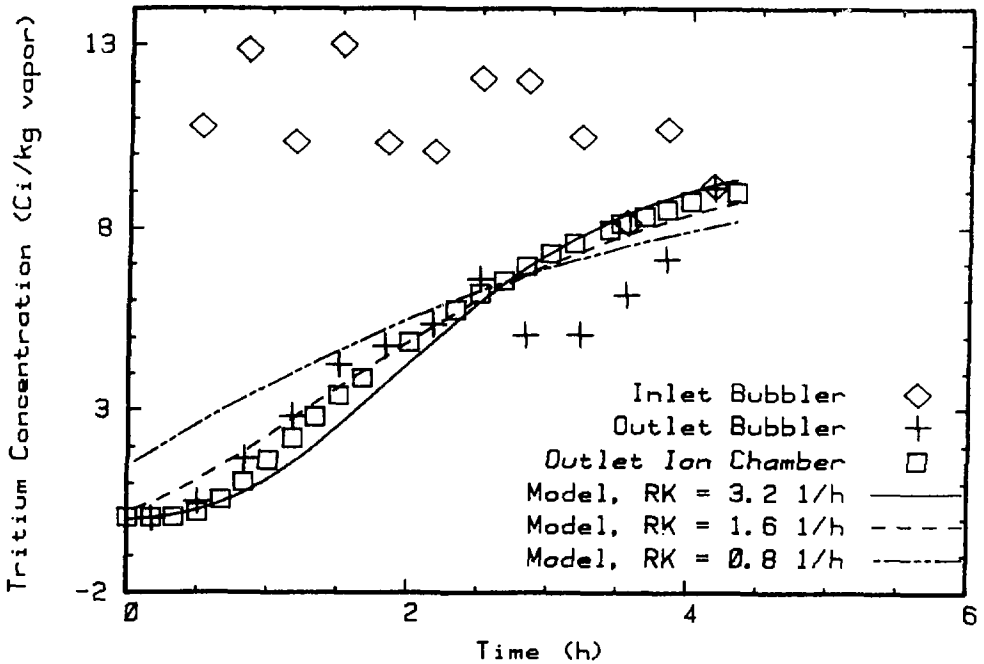


Figure 4 : Bed Outlet Tritium Concentration Trend  
Laboratory Isotopic Exchange Test #3 (Temp = 46°C)

The modelled results, generated from the isotopic exchange model/simulation, show good agreement with the observed tritium breakthrough trend. Modelled results have been generated using three arbitrary rate constants (RK) to illustrate the effect of changing the rate constant. Table 1 summarizes the process conditions and the comparison between the rate constant reported elsewhere [4] and the observed rate constant (inferred from the modelled fit) for the three tests.

**Table 1 - Comparison of Reported and Observed Rate Constants**

Test #	Temperature (°C)	Reported* RK (h <sup>-1</sup> )	Observed RK (h <sup>-1</sup> )	Observed RK (% of Reported)
1	38	2.74	1.5	55
3	46	2.81	2.4	85
2	62	3.20	2.0	63

\* Values for tests 2 and 3 were extrapolated from reported data.

In each experiment, the reported rate constant gives a modelled result that shows a larger isotopic exchange effect than was observed. Although three tests are insufficient to draw any conclusions about the actual tritium transfer rate constant, one could conservatively state for the purposes of design that the tritium transfer rate constant is 50% of the reported values.

Further laboratory experiments are planned to investigate the degree of isotopic exchange occurring at dry, unsaturated conditions.

#### 4.0 NPD DRYER TESTS

The industrial scale demonstration of an operational air dryer system employing the H<sub>2</sub>O steam washing technique during regeneration was carried out at NPD. Preparations were started in 1987 December and the tests performed in 1988 February/March, during the decommissioning of the station. The decommissioning process imposed a very tight schedule for the tests and allowed for only five runs before the dryer system was disabled. The tests were to demonstrate the process of eluting the residual tritium from a dryer sieve bed with tritium-free water (steam washing) and the enhanced detritiation factors expected during the adsorption cycle as a result of the steam washing procedure. The process conditions were chosen to duplicate conditions expected at JET, thus demonstrating that the steam washing technique would be of use in the proposed JET Exhaust Detritiation System.

##### 4.1 Apparatus and Method - NPD Dryer Tests

The air dryer system used was dryer VR6, part of the NPD boiler room air dryer/detritiation system. The regeneration circuit comprised a short (10 m) recirculating circuit containing a gas cooler and blower on the dryer outlet and a 120 kW heater on the dryer inlet. The adsorption circuit comprised the same gas cooler and blower on the dryer outlet and approximately 300 m of a 0.36 m diameter closed-loop recirculating air duct containing, at a point 100 m before the dryer bed, a 75 kW air heater and a water injection port (spray nozzle). The dryer bed, 1.82 m diameter and 0.76 m high, contained a Baylith W-894 molecular sieve. A schematic of the dryer system and the associated experimental apparatus is shown in Figure 5.

To simulate dryer inlet conditions during the water adsorption cycle, tritiated heavy water (12-16 Ci/kg) was sprayed into the air stream at the 75 kW heater. An air flow of 4 470 m<sup>3</sup>/h and water injection rates of 24-31 kg/h yielded dryer inlet temperatures of ~54°C and inlet dew points of 2-6°C. Dryer outlet conditions were typically 75°C with dew points of -50 to -60°C. Tritium content of the dryer inlet and outlet streams was measured by both ion chamber and gas scrubbers (bubblers). Outlet dew points were measured using a capacitance-type hygrometer calibrated for dry conditions (dew points of -60°C to -35°C) and a cooled-mirror type hygrometer at wet conditions (dew points greater than -35°C). Vapor tritium concentration in the dryer inlet and outlet air stream, expressed as Ci/kg vapor, was calculated as in the laboratory tests.

Regeneration of the bed for the next adsorption cycle involved a two stage process: thermal regeneration of the bed and elution of the residual tritium on the sieve. Thermal regeneration was accomplished by recirculating 1 200 m<sup>3</sup>/h of air through the bed, heating the inlet air to 280°C and cooling the outlet air stream to 10°C. Elution of tritium from the bed was accomplished by spraying 20 kg/h of tritium-free water into the dryer inlet air stream immediately after thermal regeneration, and while maintaining thermal regeneration conditions. After the initial elution attempt, the method was modified to include a short cooling-down period (inlet heaters off for 30 minutes) prior to starting the flow of tritium-free water. This was necessary to prevent the rapid release of heat, which occurs when initially adding water at a high rate (20 kg/h) to a sieve bed, from setting off the bed high temperature heater trip.

The progress of the elution step was monitored by measuring the tritium concentration of the cooler condensate throughout the elution. Determination of the bed outlet tritium concentration trend by sampling the cooler condensate did not smooth the trend data in any significant way. The estimated volume of the condenser and sampling line was 1-2 litres, thus giving a liquid residence time in the sampling system of 6 minutes.

#### 4.2 Results and Discussions - NPD Dryer Tests

The dryer was put through five cycles of adsorption and regeneration. Each adsorption cycle lasted 5-6 hours. Typically, vapor breakthrough was reached after 4 hours of operation. From 4-6 hours the outlet humidity increased, reaching 10-15% of the inlet value. The water adsorption characteristics of the dryer are illustrated by Figures 6 and 7. Figure 6 illustrates a typical dryer outlet humidity trend observed during an adsorption cycle. Figure 7 shows simulated bed loading profiles at three intervals during an adsorption cycle. That portion of the bed where water adsorption is occurring, where the bed loading changes from 20 to 2 wt%, is referred to as the mass transfer zone (MTZ). The simulated bed loading profile was confirmed by the water vapor breakthrough trend.

The observed dryer outlet tritium concentration trends (Ci/kg vapor) for adsorption cycles 2-5, as measured by ion chamber and bubblers, are shown in Figures 8-11, respectively. Adsorption cycles 2, 3 and 4 are each a demonstration of an operating air detritiation dryer utilizing the steam washing technique. In each preceding regeneration cycle, the bed was dried and steam washed to elute the tritium from the residual adsorbed water to a level less than 5% of its initial value. Adsorption cycle 5 was a control test, where the bed was dried but not steam washed. This adsorption cycle also differed in that the regenerated (dried) bed was left on cold (24°C) recirculation for eight hours prior to the start of the adsorption cycle.

Bubbler data collected during the NPD tests were generally in poor agreement with the ion chamber data. Ion chamber data were a function of temperature, pressure and humidity. Bubbler data were a function of temperature, pressure, humidity and gas flow rate through the bubbler. Much care was given to maintaining a constant bubbler sampling flow rate; however, large

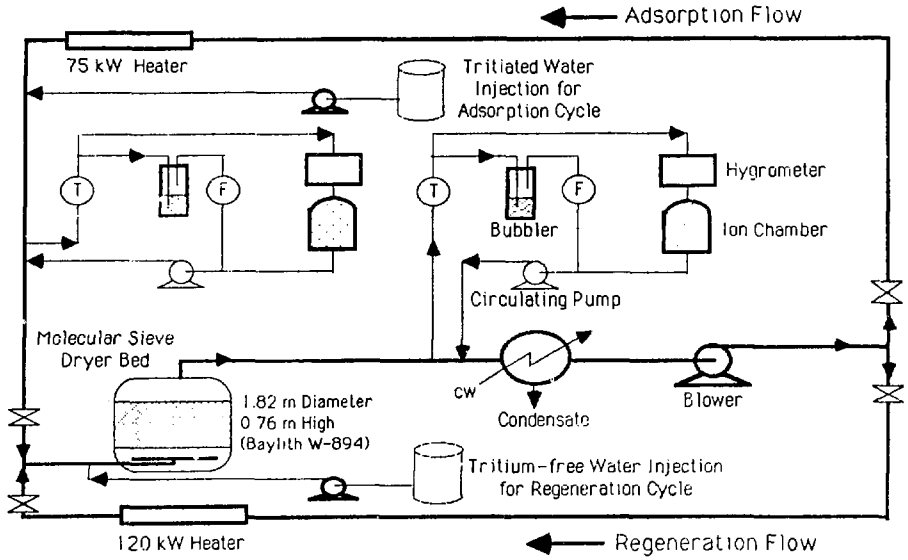


Figure 5: NPD Dryer System and Sampling Apparatus

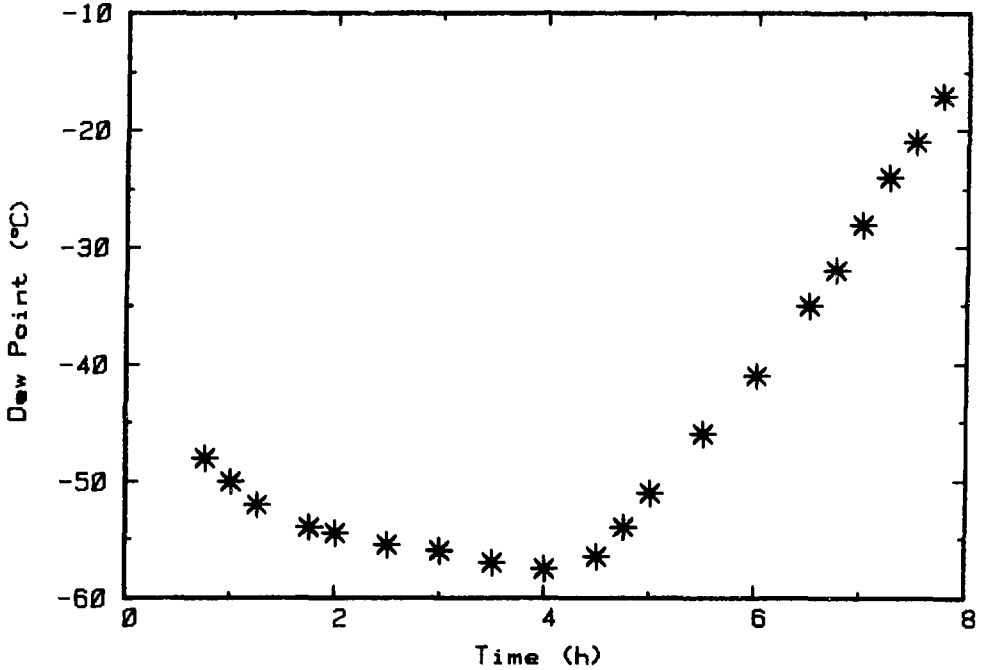


Figure 6: Typical Dew Point Trend  
NPD Dryer Test #3

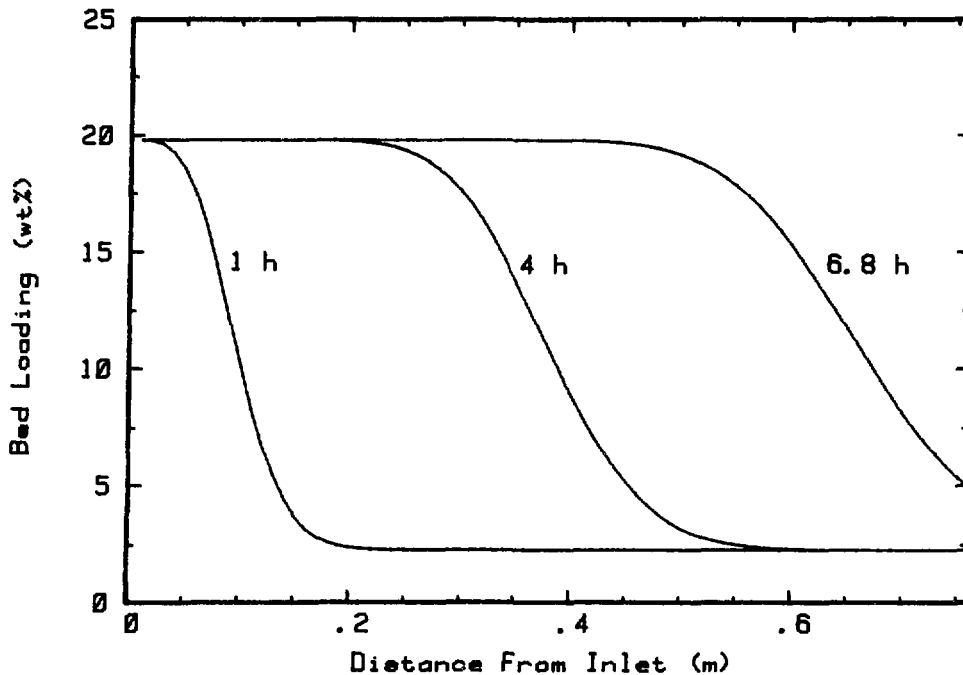


Figure 7: Simulated Bed Loading Profiles  
NPD Dryer Test #3

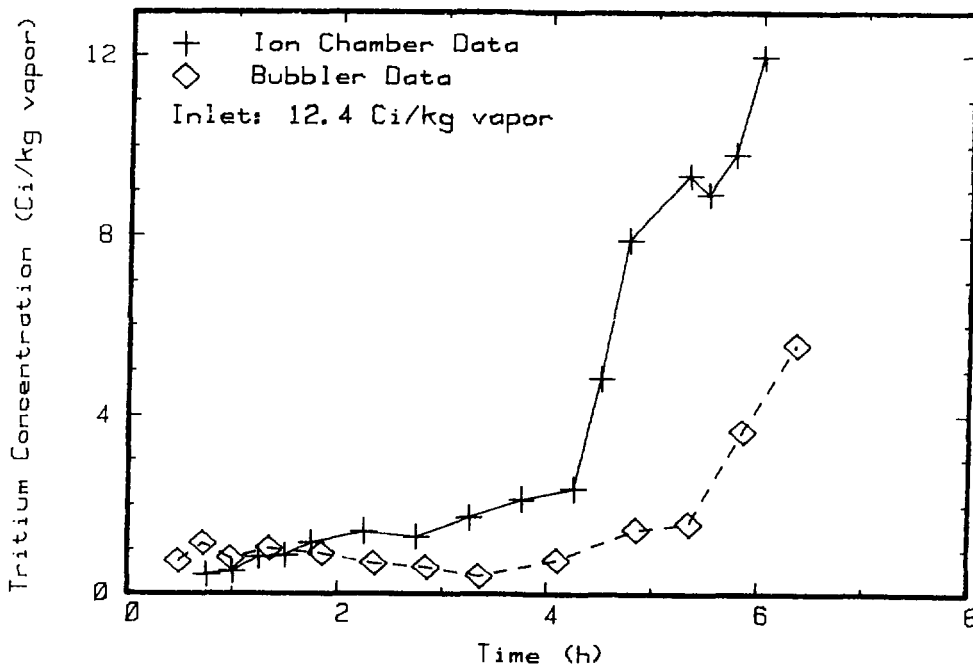


Figure 8: Dryer Outlet Tritium Concentration Trend  
NPD Dryer - Adsorption Cycle 2

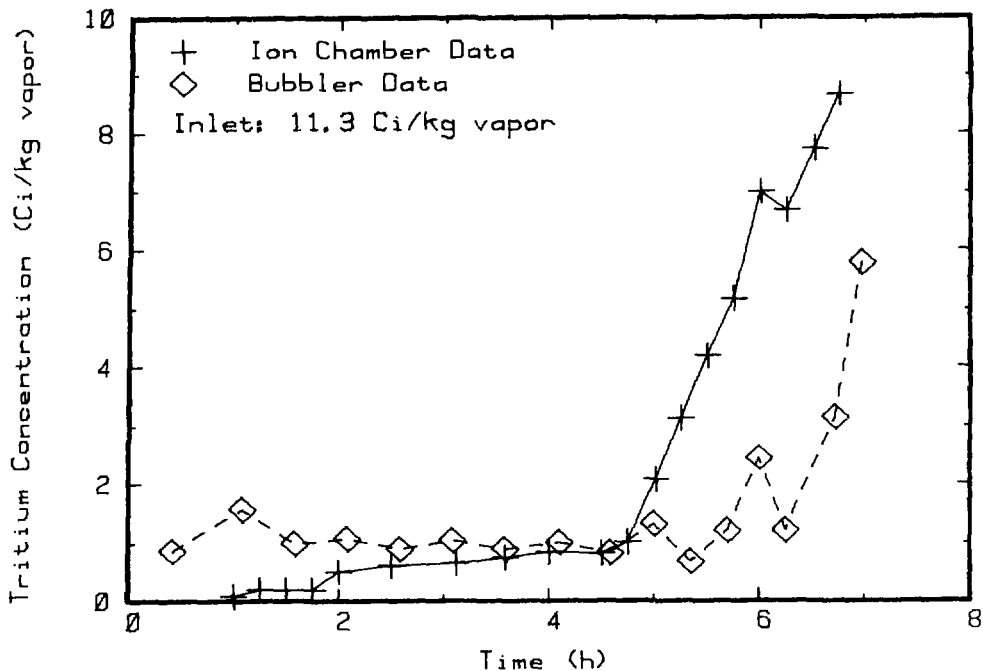


Figure 9: Dryer Outlet Tritium Concentration Trend  
NPD Dryer - Adsorption Cycle 3

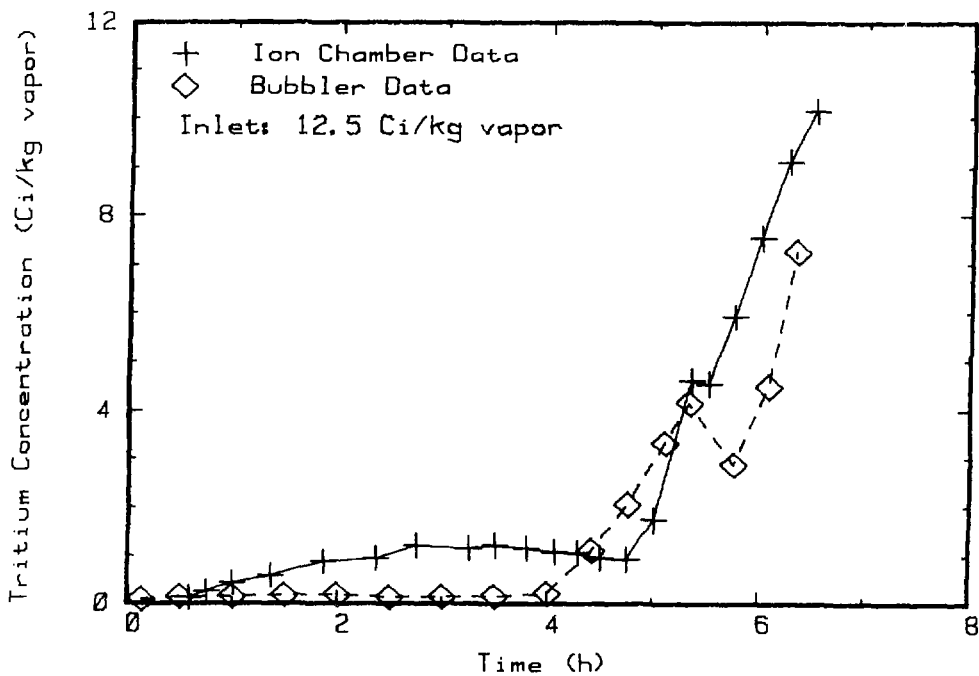


Figure 10: Dryer Outlet Tritium Concentration Trend  
NPD Dryer - Adsorption Cycle 4

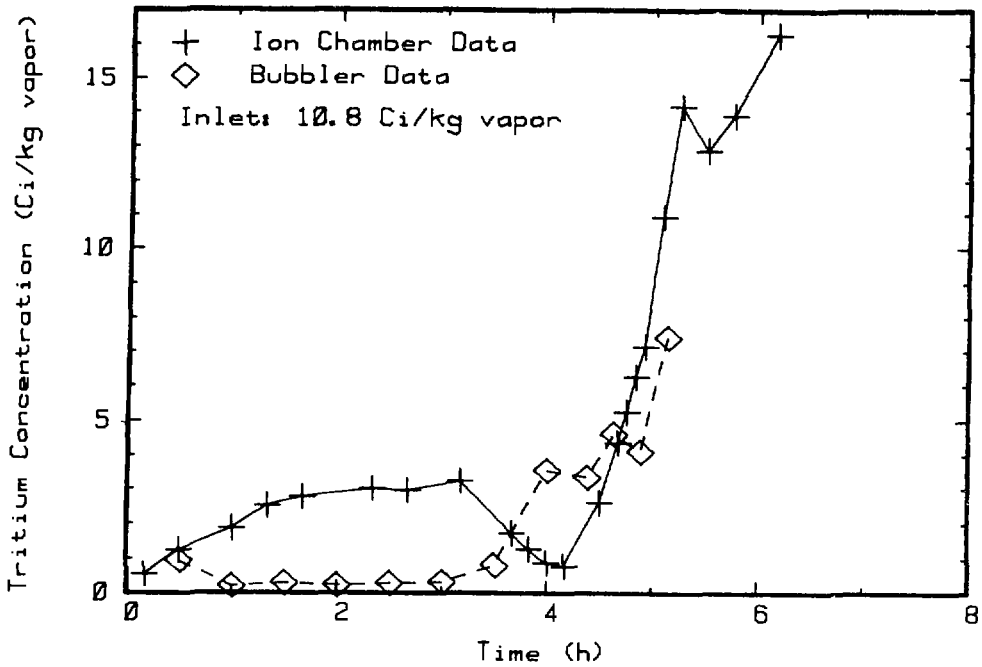


Figure 11 : Dryer Outlet Tritium Concentration Trend  
NPD Dryer - Adsorption Cycle 5

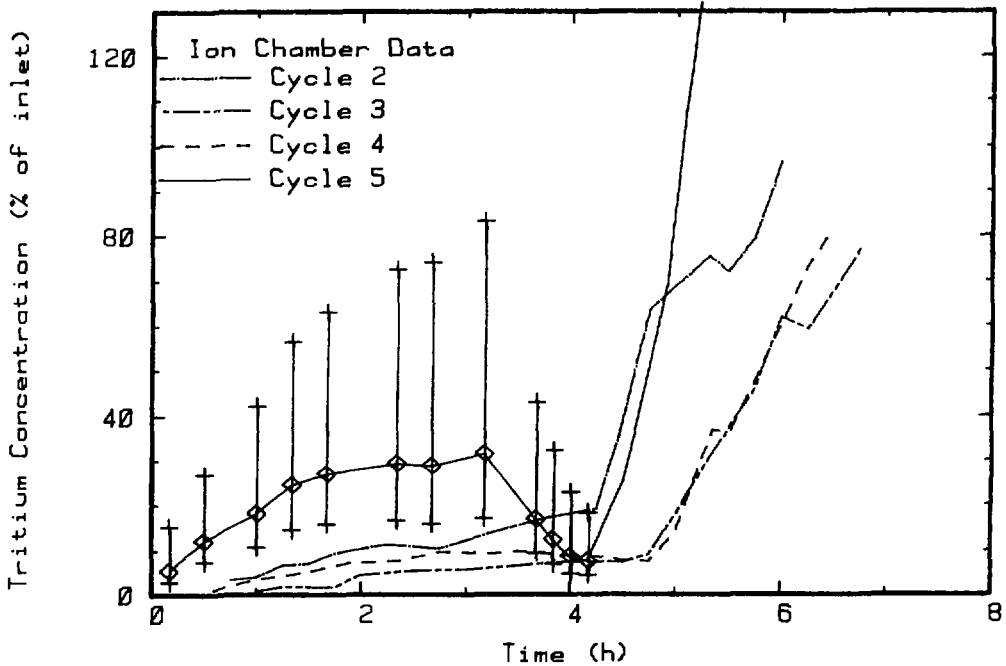


Figure 12: Comparison of Outlet Tritium Concentration Trends  
NPD Dryer - Adsorption Cycles 2-5



fluctuations in the flow rate persisted ( $\pm 20\%$ ) and a final recalibration of the bubbler flow meters in the latter part of the experiment gave markedly different results from the initial values. Bubbler data were considered unreliable, but are shown as they confirm the basic trends in the data.

A comparison of the outlet tritium concentration trends for adsorption cycles 2-5 (ion chamber data) is shown in Figure 12. The detritiation factors for runs 2-4 as calculated from the inlet and outlet ion chamber response ( $\text{Ci}/\text{m}^3$ ), which includes the humidity reduction effect, typically ranged from an initial value of 12 600 to 4 600 just before breakthrough. Detritiation factor expressed in terms of water vapor tritium concentration ( $\text{Ci}/\text{kg}$  vapor), or the ratio of inlet to outlet vapor tritium concentration, typically ranged from an initial value of 100 to 10 at vapor breakthrough.

The outlet tritium concentration traces for adsorption cycles 2-4 show two separate trends, the gradual rise in tritium concentration from the start of a run to approximately 4.5 hours and the rapid rise in concentration from 4.5 hours to the end of the run. The rise in tritium concentration at 4.5 hours was coincident with start of vapor breakthrough and demonstrates that there was a tritium concentration gradient across the MTZ. It is believed that desorption was also occurring in the MTZ and that the varying rates of adsorption/desorption for the different vapor species in the MTZ gave an overall preferential adsorption for tritiated species. One could interpret the observed outlet tritium concentration breakthrough trends as such: the slow rise in outlet tritium concentration up to four hours was a result of the diminishing potential for isotopic exchange in the unsaturated zone (the unsaturated zone becomes shorter with time and gradually loaded with tritium). The increase in tritium concentration (or rise to near inlet levels) after four hours was due to the breakthrough of the MTZ or the lack of preferential adsorption of tritiated vapor in the MTZ. From this interpretation and from the observation that the DF expressed in terms of tritium vapor concentration decreased from an initial value of 100 to 10 at vapor breakthrough, one order of magnitude reduction in tritium concentration can be attributed to the isotopic exchange process occurring in the unsaturated zone. It should be noted, however, that the NPD dryer bed was very short (0.76 m) and operated at a high humidity ( $+6^\circ\text{C}$  dew point), therefore having a comparatively long MTZ and short unsaturated zone (see Figure 7). A dryer bed operating with a longer unsaturated zone could be expected to achieve and maintain higher tritium concentration reductions.

The attempt to demonstrate the anticipated lower DF when not using the steam washing procedure to elute tritium from the bed (the control test, adsorption cycle 5) proved inconclusive (see Figure 12). Adsorption cycles 2-4 all consistently demonstrated the DF expected. The response from adsorption cycle 5 was unexpected. Adsorption cycle 5 results show a varying tritium concentration reduction over the first four hours (although there is significantly less reduction than previous cycles) and a concentration increase during breakthrough of 1.3 times the inlet value. After adsorption cycle 5 the calibrations of all instruments were rechecked. The error bars shown in Figure 12 for cycle 5 represent the maximum possible error in calculated ion chamber data, a  $\pm 2$  K error in dew point, a  $15 \text{ uCi}/\text{m}^3$  (15%) over-estimate in ion chamber background signal and a  $\pm 5\%$  error in ion chamber signal.

Due to this unusual response, no conclusions are made about the outlet tritium concentration trend in an untreated bed. One possible explanation is that during the 8-hour cold recirculation of air through the dry bed (3 wt% water), a partitioning occurred between the tritiated and the non-tritiated species, setting up a non-uniform tritium concentration profile along the length of the bed. The decommissioning schedule of the station prevented repeating this test. Further laboratory tests at NPD conditions are planned to study adsorption cycle 5 results.

Figure 13 compares the observed and simulated outlet tritium concentration trend for adsorption cycle 4. The simulated result is from the isotopic exchange model/simulation and was calculated using the tritium transfer rate constants reported in reference 4. The isotopic exchange model has been shown in the laboratory experiments to accurately simulate isotopic exchange under static conditions (no net adsorption or desorption). However, the agreement between the modelled and the observed NPD data is poor. The model is not simulating the tritium reduction processes occurring in the MTZ. By adjusting the rate constants in the unsaturated zone and the MTZ one can force the model to fit the data; however, there is currently no justification for doing this. More experimental work is needed to determine the relative rates of tritium exchange occurring in the three zones of a dryer bed.

A second objective of the NPD tests was to demonstrate the elution of adsorbed tritium from an unsaturated bed (3-4 wt% water loading) at elevated temperatures (280°C) using tritium-free water. These conditions are thought to represent the optimum conditions to elute tritium from the bed, a high H<sub>2</sub>O vapor pressure, a high temperature (high transfer rate) and a minimum bed loading.

Figure 14 illustrates the observed bed outlet tritium concentration trend during the elution step of regeneration cycle 3. Note that the zero point on the time axis of the observed data coincides with the first arrival of cooler condensate and not the start of water injection, as there is a time delay while injected water is adsorbed (bed loading typically increases from 2.9 to 3.5 wt%). The results of the elution process indicate that, at the prescribed physical conditions, tritium can be readily eluted from a large molecular sieve bed. The elution process took 4-6 hours and required a water injection equivalent to 50% of the adsorption cycle capacity.

Also shown in Figure 14 are three modelled outlet tritium concentration trends using three arbitrarily chosen rate constants. In this static system (no net adsorption or desorption after a steady state is achieved) the modelled results show a good fit to the observed results. Of the three traces shown in Figure 14, the best fit to the observed results appears to be those modelled using a tritium transfer rate constant of 9 h<sup>-1</sup>. The trace showing the sawtooth wave form is characteristic of a recirculating system and a high transfer rate; successive waves of less concentrated tritiated water pass through the bed.

## 5.0 SUMMARY

Isotopic exchange between tritiated and non-tritiated water species in a molecular sieve bed has been demonstrated. At high humidities (+6°C dew point) the rate of tritium isotopic exchange in a 2.4 L molecular sieve bed has been demonstrated to be at least 50% of published exchange rates.

In an industrial-sized air detritiation dryer, utilizing the pretreatment technique of H<sub>2</sub>O steam washing to elute the residual tritium, a DF of 12 600 has been demonstrated when operating at an inlet vapor tritium concentration of 14 Ci/kg and at inlet and outlet dew points of 4.8 and -54°C, respectively. In the NPD dryer bed studied, which was not optimally designed for full benefit from isotopic exchange, at least one order of magnitude in additional detritiation is attributed to isotopic exchange in the unsaturated zone.

The technique of eluting the residual tritium from an industrial sized bed by H<sub>2</sub>O washing at high temperature, high humidity and low bed loading has been demonstrated to be a fast and effective way of removing tritium from a molecular sieve bed during regeneration.

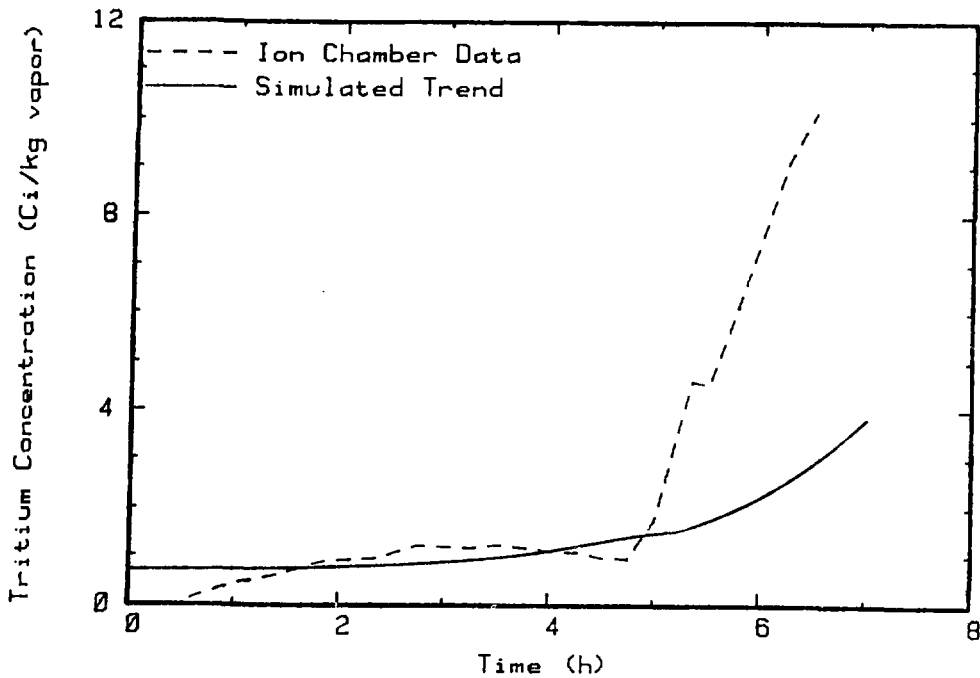


Figure 13 : Simulated and Observed Dryer Outlet Tritium Concentration Trend  
NPD Dryer - Adsorption Cycle 4

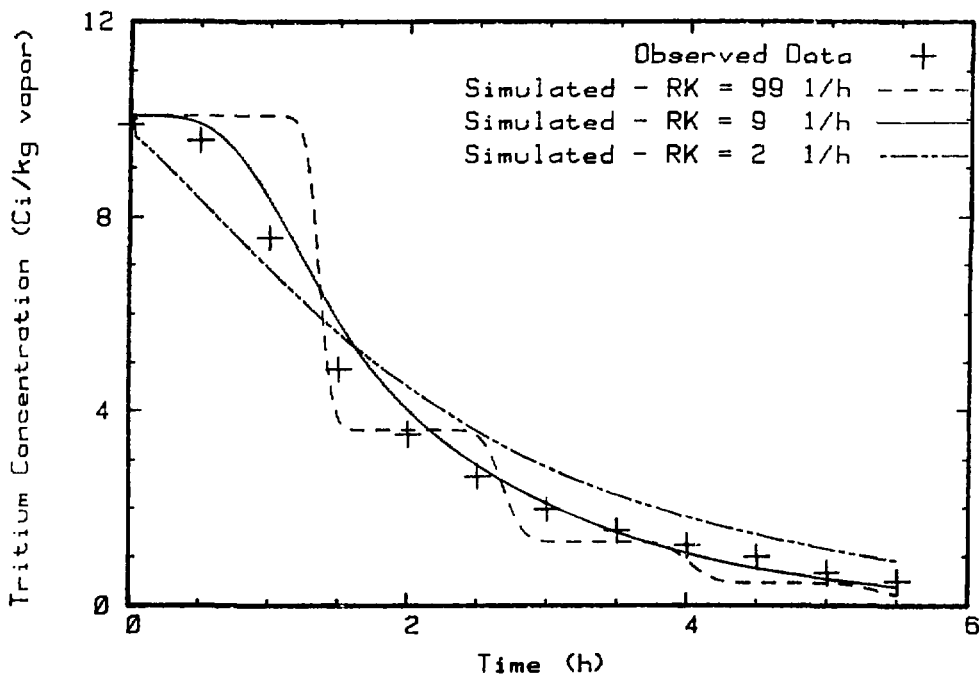


Figure 14 : Bed Outlet Tritium Concentration Trend  
Tritium Elution for Regeneration Cycle 3 - NPD Tests

The isotopic exchange model accurately predicted the exchange between tritiated and non-tritiated water species in a molecular sieve bed where there is no net adsorption or desorption. The model's prediction of the tritium breakthrough trend observed in the NPD tests was poor; however, a forced fit can be achieved if the exchange rates in the MTZ and the unsaturated zone are manipulated. More experiments are needed to determine the relative rates of tritium exchange in the saturated, mass transfer, and unsaturated zones of a dryer bed.

## 6.0 ACKNOWLEDGMENTS

The cooperation and interest shown by the NPD staff was appreciated. Particular thanks and appreciation to D.Nicks and R. Archer for their efforts in quickly arranging solutions to problems and performing the tests.

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