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**In-Line Tritium Monitoring in the GCFR  
Vented Irradiation Capsule GB-10**

M. E. Pruitt  
A. W. Longest

**OAK RIDGE NATIONAL LABORATORY**  
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IN-LINE TRITIUM MONITORING IN THE GCFR  
VENTED IRRADIATION CAPSULE GB-10

M. E. Pruitt  
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IN-LINE TRITIUM MONITORING IN THE GCFR VENTED  
IRRADIATION CAPSULE GB-10

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ABSTRACT

An in-line tritium monitoring system was designed, tested, and operated on the sweep gas from the Gas-Cooled Fast Reactor irradiation capsule GB-10. Cooled charcoal traps removed fission and activation products and the HTO form of tritium from the gas stream but allowed the HT form to pass through. The tritium-bearing gas stream then passed through an ionization chamber containing a vibrating-reed electrometer which recorded the tritium response on a strip-chart recorder. Total tritium (HT + HTO) determination was attempted by passing the sweep gas through an HTO converter (magnesium at 500°C) that quantitatively converted HTO to HT when the gas stream contained hydrogen carrier. An alternate and independent tritium detection system was included downstream from the ionization chamber. The gas stream could be valved through an HT converter (CuO at 500°C), which quantitatively converted HT to HTO, and then valved through heated lines to molecular-sieve traps. Laboratory procedures were necessary to release the tritium and to determine the amount adsorbed by the molecular-sieve traps.

Fifteen tritium monitoring experiments were completed with this system. Data obtained from the electrometer and molecular-sieve samples were in excellent agreement. Molecular-sieve samples were necessary on some experiments due to low tritium levels and the unexpected presence of  $^{24}\text{Ne}$  in the gas stream. The low-level  $^{24}\text{Ne}$  was apparently produced in the fuel region of the capsule and was not retained by the charcoal traps. Monitored tritium was significantly lower than the calculated birth rate. Areas exposed to fission product deposition retained tritium from high-purity helium and released tritium when the gas stream contained hydrogen carrier. Temperature-dependent tritium release was observed while sweeping the heated charcoal trap region of the GB-10 fuel rod.

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1. INTRODUCTION

Potential tritium release pathways from a Gas-Cooled Fast Reactor (GCFR) should be known to assist in selection of design and of construction

materials. Tritium (T) can be more easily controlled by knowing how it is transported and the ratio of HT to HTO in which it appears.\* Capsule GB-10 contained a GCFR-type fuel rod that was vented and pressure equalized. The capsule design included five sweep gas lines which permitted sweeping across the top of the rod (normal venting mode) or sweeping through the fuel, blanket, and charcoal trap regions independently or in series. Capsule GB-10 was the only facility available for direct measurement of released fission products from fast breeder reactor fuel, including tritium.

A continuous tritium monitoring system was designed to measure the tritium concentration in the GB-10 effluent sweep gas. Tritium release data as a function of flow rate, capsule power, and flow mode would provide information on tritium production, release, and cladding permeation. Determination of the HT to HTO ratio would provide information on fission product chemistry and transport mechanisms. This system was tested, installed, and operated on the sweep gas from capsule GB-10.

The capsule had attained a burnup of approximately 75 MWd/kg of heavy metal when the first series of experiments was conducted using high-purity helium as the capsule sweep gas. The second series of experiments utilized hydrogen carrier in the capsule sweep gas and was conducted as the capsule reached the revised burnup goal of 100 MWd/kg.

The original monitoring schedule was revised periodically to reflect operational constraints and data obtained. Each tritium monitoring experiment, consisting of one or more steps, required 20 to

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\* Throughout this report the amounts of  $T_2$  and  $T_2O$  are assumed to be minuscule and are included in the HT and HTO respectively.

32 h of continuous operation. In some cases the step sequence was altered during the experiment due to time limitations. Fifteen tritium monitoring experiments were completed prior to removing the capsule for postirradiation examination.

## 2. DESCRIPTION OF THE TRITIUM MONITORING SYSTEM

The tritium monitoring system was designed to measure tritium (HT and HT + HTO) in the GB-10 effluent gas stream in a versatile manner.<sup>1</sup> Tritium pathways and release mechanisms could be characterized by varying the flow rate, capsule power, and flow mode. Four basic design criteria were included in the system:

1. On-line and continuous tritium monitoring.
2. Alternate and independent tritium measurement systems.
3. HT and HTO measurement capability.
4. In situ calibration and cleanup.

A schematic of the tritium monitoring system is shown in Fig. 1. In addition to the auxiliary components normally associated with an in-pile experiment, the system consists of six discrete components: (1) an ionization chamber, (2) charcoal traps, (3) an HTO converter, (4) an HT converter, (5) molecular-sieve traps, and (6) a calibration gas header.<sup>2</sup>

Helium sweep gas entered the tritium monitoring system from either the GB-10 effluent sweep line or the calibration gas supply header. The first active component was the HTO converter. When the gas stream bypassed the HTO converter, the HTO form of tritium was retained on the charcoal traps, and only the HT form was measured. When the gas stream

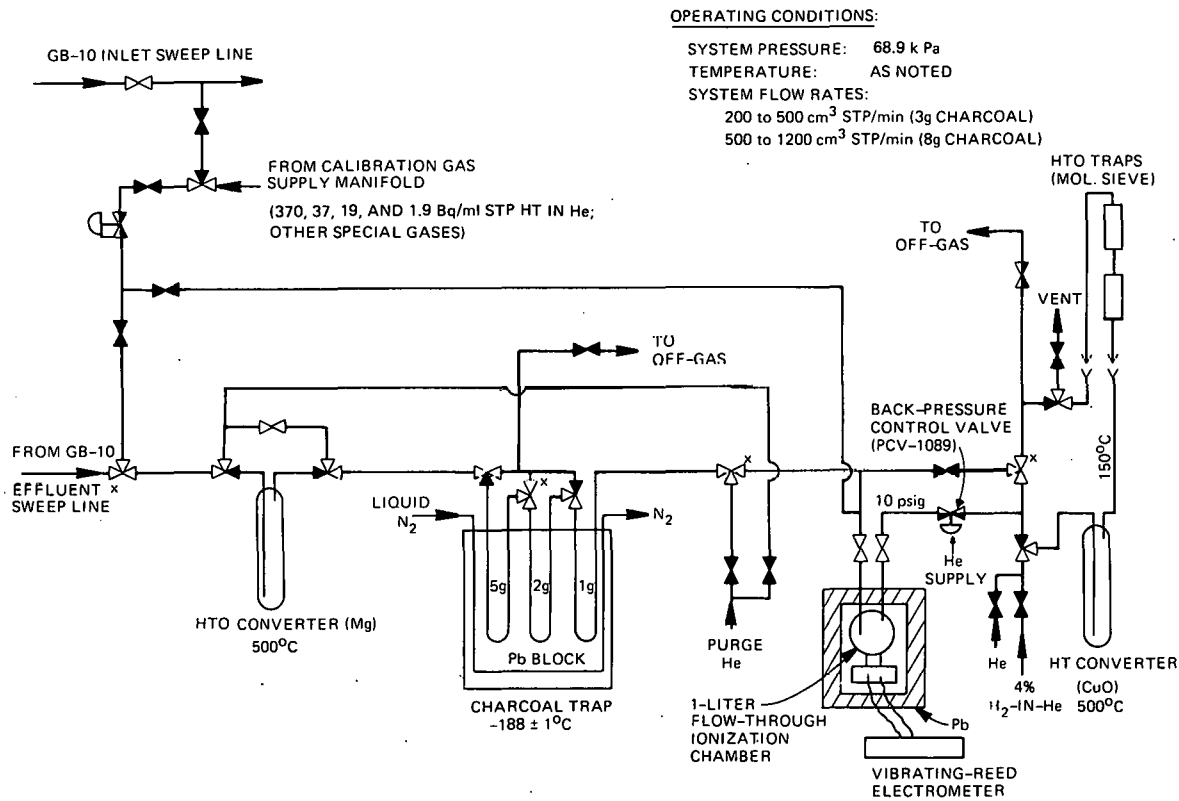


Fig. 1. Schematic of capsule GB-10 Tritium Monitoring System.

passed through the HTO converter, HTO was expected to be converted to HT, and measurements were expected to be obtained for the combined HT and HTO. The second active components were the charcoal traps, which retained the HTO form of tritium, activation products, and fission products while allowing the HT form of tritium to break through after a short retention time.

The purified gas stream containing tritium (HT) then entered an ionization chamber which was monitored by a vibrating-reed electrometer and output to a strip-chart recorder. After the ionization chamber response to the tritium was obtained, the gas stream was normally vented

to off-gas. However, if alternate or verification analyses were desired, the gas stream was valved through the HT converter, which converted HT to HTO. The gas stream carrying HTO passed through heated lines to molecular-sieve traps. After a timed collection period, the traps were transferred to the laboratory, where the tritium was subsequently determined by liquid scintillation beta counting.

## 2.1 Ionization Chamber

A 1-liter flow-through ionization chamber was connected to a Cary model 401 vibrating-reed electrometer for continuous tritium measurements. The electrometer response was output to a Hewlett-Packard Model 7100B strip-chart recorder. When tritium in high-purity helium was passed directly to the ionization chamber, the electrometer response was characterized by a peak and then a slow decrease to steady state (Fig. 2).

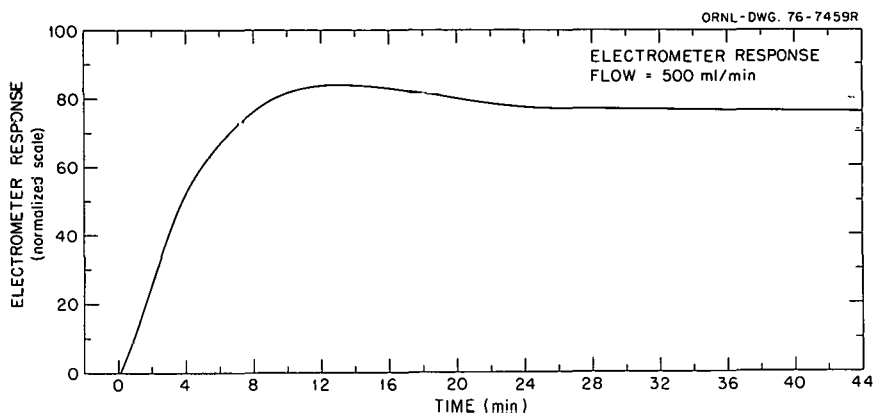


Fig. 2. Electrometer response to tritium in high-purity helium flowing directly to ionization chamber.

The ionization chamber response was enhanced by the presence of hydrogen in the gas stream. A flow dependence observed in laboratory testing was eliminated by the addition of hydrogen to the carrier gas. The peaking effect observed when using high-purity helium was also eliminated with the addition of hydrogen to the carrier gas; the dashed line in Fig. 3 is the valid calibration curve for tritium in helium containing 1% hydrogen. The detection limit for tritium with the ionization chamber was approximately 0.03 Bq/ml at standard temperature and pressure (STP). (The becquerel is equivalent to one disintegration per second).

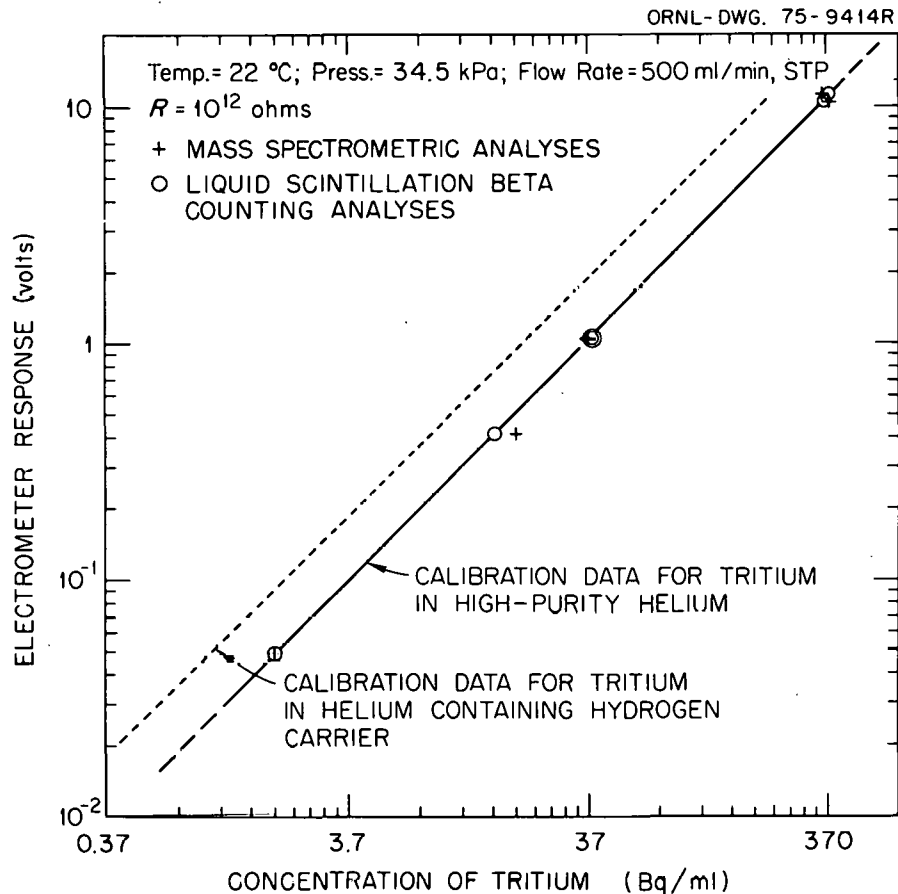


Fig. 3. Tritium calibration curves for 1-liter ionization chamber.

## 2.2 Charcoal Traps

The charcoal traps were designed to retain fission products and induced activities for several days while allowing tritium (HT) to break through within a few minutes. This was accomplished by utilizing charcoal traps cooled to approximately liquid nitrogen temperature ( $-188 \pm 1^\circ\text{C}$ ). The possibility of production and accumulation of ozone in the radiation field was precluded by using a continuous flowing stream of liquid nitrogen to the traps. In a typical graph of tritium breakthrough (Fig. 4), the sine wave response was a result of, and closely followed, the  $2^\circ\text{C}$  temperature cycle of the trap. However, the average sine wave value was in good agreement with other calibrations.

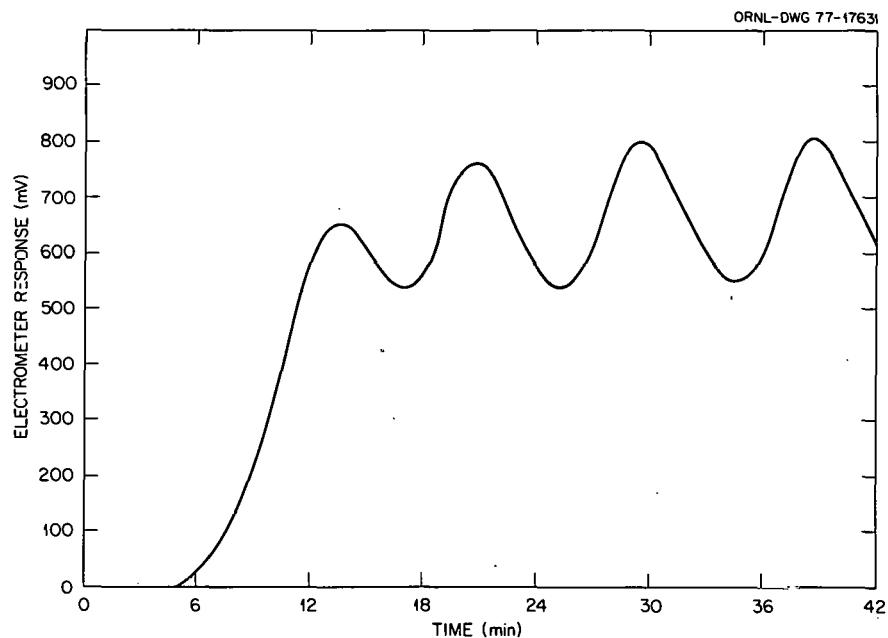


Fig. 4. Typical electrometer response to tritium breaking through 8 g of charcoal at  $-188^\circ \pm 1^\circ\text{C}$  (flow rate = 1200 ml/min STP).

The traps were designed, loaded, and valved to permit the use of 1, 3, or 8 g of activated coconut charcoal type MI-8558.\* Under normal operating conditions, 3 g of charcoal was used for flow rates of 200 to 500 ml/min STP. For flow rates of 600 to 1200 ml/min STP, 8 g of charcoal was used. During laboratory testing,  $^{37}\text{Ar}$  was retained on 1 g of charcoal for 29 h with no indication of breakthrough. The time required for tritium breakthrough was a function of charcoal trap valving, flow rate, and hydrogen concentration. Under the conditions described in this report, breakthrough time varied between 12 and 32 min.

### 2.3 HTO Converter

The HTO converter contained 153 g of 40- to 60- $\mu\text{m}$  magnesium maintained at 500°C. When a gas stream was directed through the HTO converter, hot magnesium was expected to reduce HTO to HT, and a response was expected for the combined HT and HTO. When the HTO converter was bypassed, HTO was retained in the nitrogen-cooled charcoal traps, and only HT was present in the gas stream for measurement. The HT/HTO ratio could then be obtained by flowing through and bypassing the HTO converter.

Laboratory models of the HT converter and molecular-sieve traps were used for the laboratory evaluation of the HTO conversion process. Since hydrogen carrier was routinely used to quantitatively transfer HTO from the HT converter to the molecular-sieve traps, it was also used in the laboratory evaluation of the HTO converter. The rapid exchange in the  $\text{H}_2$ , HT, HTO, and  $\text{H}_2\text{O}$  system was not considered during laboratory

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\* Barnebey Cheney, Columbus, Ohio.



testing. Due to these considerations, the laboratory evaluation of the HTO converter was inadequate.

Experiment 8b demonstrated that granular magnesium at 500°C quantitatively retained tritium from a high-purity helium system. Experiment 9a demonstrated that the HTO converter did not retain tritium when hydrogen was present in the gas stream. Therefore, valid experimental data were not obtained for HT plus HTO in a high-purity helium system. Experimental data obtained with the HTO converter using hydrogen carrier should be valid. Even if the HTO converter had retained tritium, exchange between H<sub>2</sub> and retained HT and/or HTO would have released an equivalent concentration of HT to the gas stream.

#### 2.4 HT Converter

The HT converter contained 452 g of <60- $\mu$ m CuO at 500°C. The HT form of tritium passing through the CuO was oxidized to HTO, which then passed through heated (150°C) lines to tandem 4A molecular-sieve traps. For all laboratory and experimental tests, hydrogen carrier was used in this portion of the system, and the H<sub>2</sub>O produced served as a carrier to quantitatively transfer HTO from the converter to the molecular-sieve traps. Tritium retained on the molecular sieve was released by reflux with distilled water and subsequently determined by liquid scintillation beta counting. The final analyses were obtained by the standard addition technique using tritiated water certified by the National Bureau of Standards (NBS). Complete recovery was obtained in all experiments.

## 2.5 Molecular-Sieve Traps

Two tandem stainless steel cylinders containing approximately 60 cm<sup>3</sup> of 4A molecular sieve were used to absorb the HTO form of tritium produced by the HT converter. The second trap was designed as a backup to collect tritium that passed through the first trap. Since tritium was not detected in the second trap, quantitative retention of tritium by the first trap could be assumed.

Samples were collected by valving the sweep gas through the HT converter and molecular-sieve traps. Sample collection times were 60 to 120 min. Auxiliary flow of helium containing 1% hydrogen through the HT converter carried the tritium from the converter into the traps.

The molecular sieve was refluxed with water for 120 min in the laboratory. The aqueous solution was then doubly distilled, and the tritium was determined by liquid scintillation beta counting. The detection limit for a flow rate of 500 ml/min STP and a sample collection time of 120 min was approximately 0.3 mBq/ml STP.

## 2.6 Calibration Gas Header

The eight-cylinder calibration gas header (Fig. 5) was valved so that any gas could be directly supplied to the ionization chamber, the inlet to the tritium monitoring system, or the GB-10 sweep gas supply line. Cross contamination of gases was minimized by low volume design and by use of stringent evacuation and backfill procedures. During the first series of experiments, the header was loaded with the five calibration gases, Matheson high-purity helium, and individually analyzed

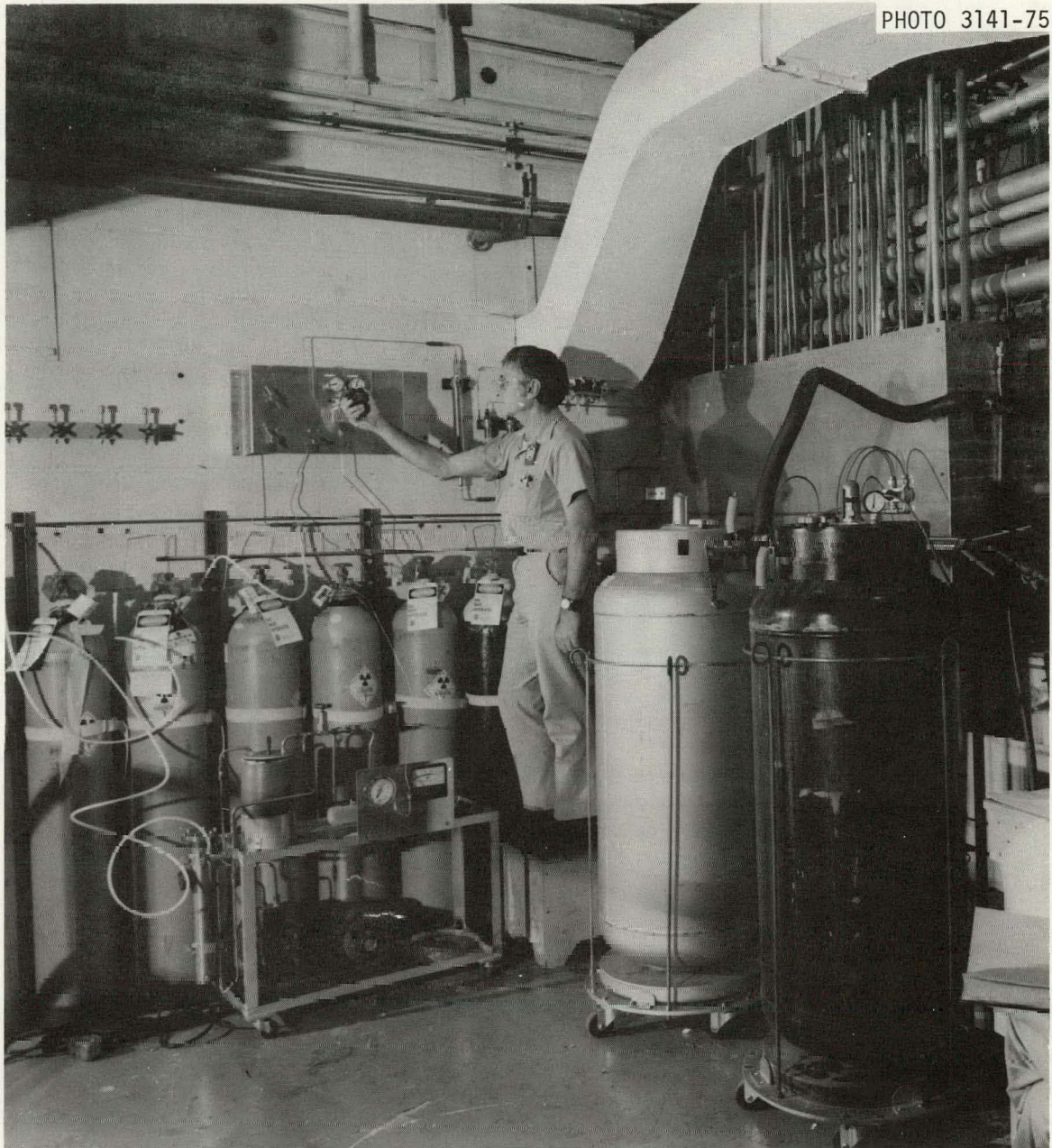


Fig. 5. Calibration gas header and liquid nitrogen supply tanks in the GB-10 tritium monitoring system.



Bureau of Mines helium. During the second series, calibration gases, helium, and helium containing 1% hydrogen were loaded on the header. The calibration gas supply line was designed with a molecular-sieve trap to retain water vapor that might be present in the gases. All gases from the calibration header passed through this trap.

### 3. DESCRIPTION OF CAPSULE GB-10

The GB-10 fuel rod (Fig. 6) was a shortened prototype of the GCFR rod with a 0.23-m-long (9-in.) region of sol-gel-derived (U,Pu)O<sub>2</sub> solid pellet fuel, a 51-mm-long (2-in.) upper blanket region of depleted UO<sub>2</sub> pellets, and a 25.4-mm-long (1-in.) charcoal trap. The two UO<sub>2</sub> half-pellets at each end of the mixed-oxide column were included to suppress power peaking at the ends of the test fuel. Initial oxygen-to-metal ratios (O/M) were 1.98 in the (U,Pu)O<sub>2</sub> pellets, 2.004 in the UO<sub>2</sub> half-pellets, and 2.002 in the depleted UO<sub>2</sub> blanket pellets. Fuel column smear density was 84.2% of theoretical. The charcoal trap contained 0.461 g of activated coconut charcoal, type MI-6736.\*

The NaK-filled capsule was equipped with an electrical heater formed into a coil over the upper blanket and trap regions of the fuel rod. The charcoal trap temperature could be controlled over the range 200 to 400°C by adjusting the output of the heater. The fuel region power level was controlled by adjusting the capsule position in the Oak Ridge Research Reactor (ORR) poolside facility (i.e., by adjusting the distance from the reactor core). Additional details of the capsule and fuel rod design were reported previously.<sup>3,4</sup>

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\* Barnebey Cheney, Columbus, Ohio.



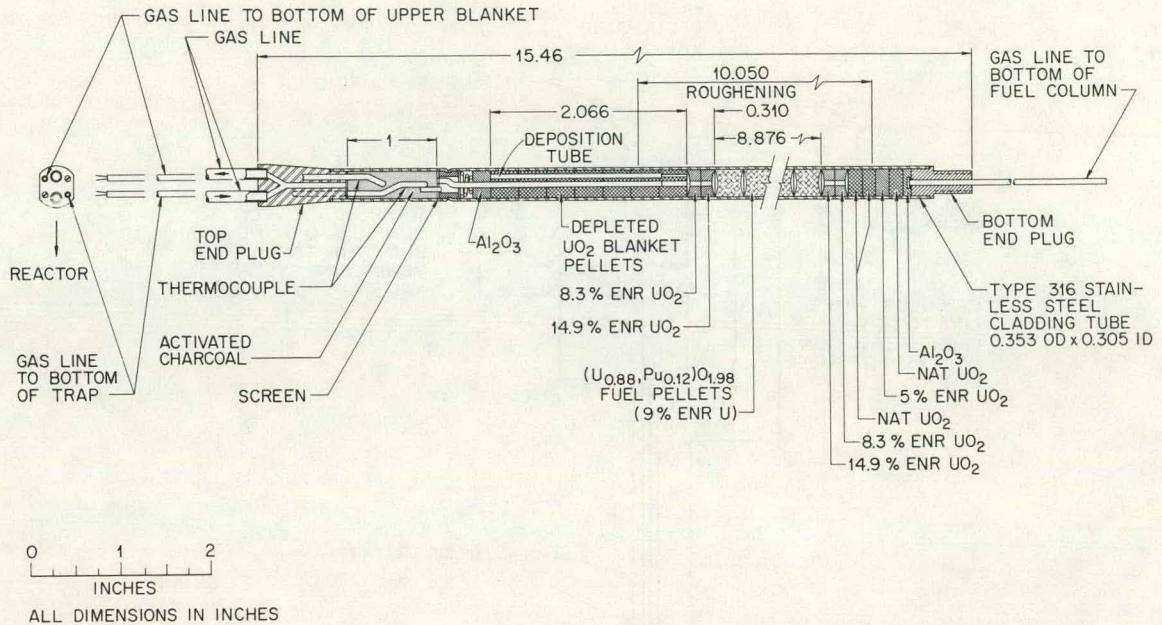


Fig. 6. Fuel rod specimen for irradiation in GCFR-ORR capsule GB-10. To convert dimensions to millimeters, multiply by 25.4.

The GB-10 arrangement of sweep lines and valves (Fig. 7) permitted flow across the top of the fuel rod, which was the normal sweep flow mode, or flow through the three main regions of the rod (trap, blanket, and fuel), either individually or in series. As shown in Fig. 7, the sweep lines were designated as follows: BF = line to bottom of fuel, TT = lines to top of charcoal trap, BT = line to bottom of trap, and BB = line to bottom of upper blanket. Specific sweep flow modes were designated by two letters indicating the entrance point, followed by two more letters indicating the exit point from the fuel rod. When the sweep flow was directed across the top of the rod, the flow mode was TT-TT, and when the sweep flow was directed into the bottom and out of the top of the rod, the flow mode was BF-TT. The latter flow mode simulated a leak in the cladding of a GCFR fuel rod. Two lines (Fig. 7),



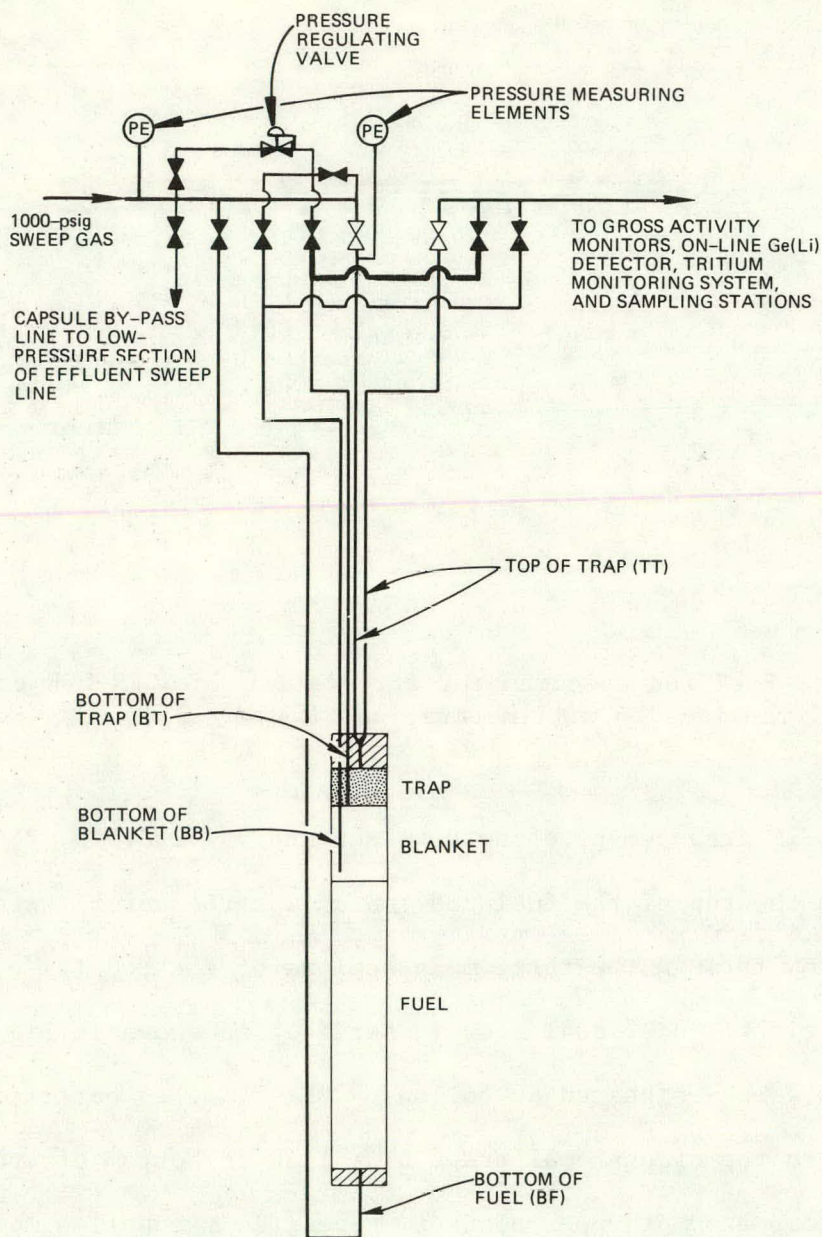


Fig. 7. Schematic of GCFR-ORR capsule GC-10, showing sweep gas lines. Heavy line indicates the BT-BT bypass flow mode.

one with the pressure regulating valve and one bypassing the capsule, were added to the system to aid in making fission-gas release measurements at very low flow rates through the fuel region of the rod.

The sweep and cladding external gas systems for capsule GB-10 are shown in Fig. 8. Flow through the sweep system (150 to 1300 ml/min STP)



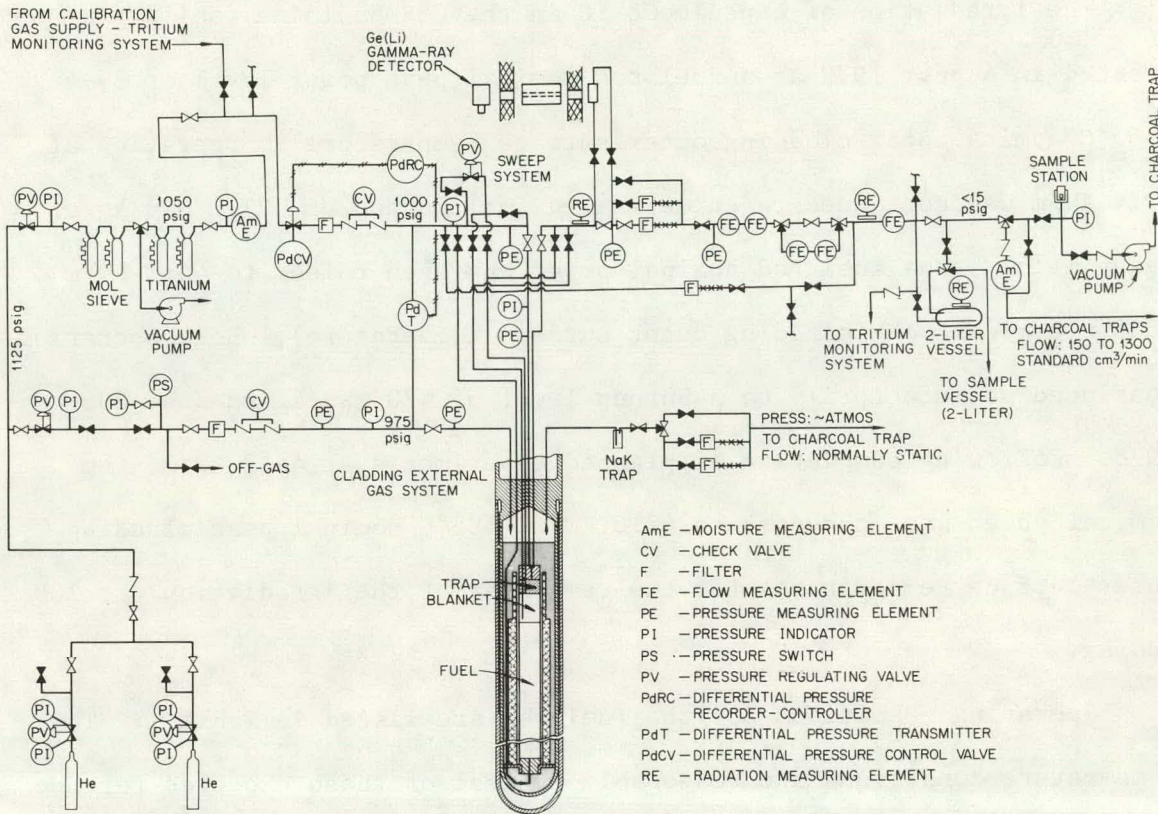


Fig. 8. GCFR-ORR capsule GB-10 sweep and cladding-external gas systems.

was regulated by adjusting the downstream flow resistance with a needle valve. Flow restrictors were employed immediately upstream of the needle valve to limit the flow rate out of the system to a maximum value. An automatic pressure differential control valve adjusted the inlet sweep flow and maintained the sweep pressure at  $172.4 \pm 0.7$  kPa above the pressure in the normally static cladding external gas system. Analyzed helium was used as the sweep gas and was passed through room-temperature molecular-sieve traps and then through  $625^{\circ}\text{C}$  titanium-sponge traps before going to the capsule.



The irradiation of capsule GB-10 in the ORR poolside facility was started in August 1972 at a fuel rod nominal peak power level of 39.4 kW/m (565°C nominal peak cladding outer surface temperature). Operation at 39.4 kW/m was continued to an estimated fuel burnup of  $\sim 27.1$  MWd/kg of heavy metal. The fuel rod nominal power was then raised to 44.3 kW/m (630°C nominal peak cladding outer surface temperature), where operation continued satisfactorily to a burnup level of  $\sim 70$  MWd/kg on June 25, 1975. Following completion of planned experiments at 44.3 kW/m, the nominal power was increased to 48.6 kW/m (685°C nominal peak cladding outer surface temperature) for the remainder of the irradiation<sup>4</sup> to 100 MWd/kg.

Operating conditions for the fuel rod are listed in Table 1. The temperature conditions are reasonably typical of those expected for the GCFR fuel rod, but the low fast neutron exposure was atypical. Sweep flow was maintained whenever the capsule was at power so that fission gases would be free to diffuse out of the fuel rod as in the case of the GCFR rod. The irradiation was interrupted occasionally for short periods for reactor refueling shutdowns (a few hours once every two weeks) and reactor end-of-cycle shutdowns (a few days every two months). Other interruptions of a few weeks occurred for installation of new equipment and maintenance work. A complete description of capsule GB-10 irradiation and operation to postirradiation examination is presented in a separate report.<sup>5</sup>



Table 1. Capsule operating conditions

Fuel-rod operating power levels, <sup>a</sup> kW/m	39.4	44.3	48.6
Cladding outer surface temperature, <sup>a</sup> °C			
Fuel region, peak	565	630	685
Charcoal trap	300	300	300
Cladding pressure, MPa gage (psig)			
External			6.7 (975)
Internal			6.9 (1000)
Time at power, days			972
Fuel burnup goal, <sup>a</sup> MWd/kg of heavy metal			100
Fast-neutron exposure (E < 0.18 MeV), neutrons/cm <sup>2</sup>			$\sim 1 \times 10^{20}$

<sup>a</sup>Nominal values.

#### 4. LABORATORY CALIBRATION

Before they were installed in the GB-10 system, the components (or laboratory models) of the tritium monitoring system were tested and calibrated in the laboratory. Five cylinders of Matheson certified standard tritium in high-purity helium were used for testing and calibration. The tritium concentration covered the range from 1.8 to 377 Bq/ml STP. A 1-liter calibrated sample flask, manometer, and barometer were used to quantitatively collect and transfer calibration gas samples in the laboratory. This approach was selected to circumvent the installation and calibration of high-quality flow meters.

The ionization chamber was calibrated by first flowing the lowest concentration of calibration gas (cylinder 43372) through the ionization chamber at 200 ml/min and 34.5 kPa. The effluent stream from the ionization chamber was valved through the 1-liter gas sampling flask. In these



tests, the electrometer response was characterized by a peak followed by a slow decrease to steady state. The steady-state values were used as calibration values. Sixty minutes after steady state, the electrometer response was recorded and the sample flask isolated at atmospheric pressure.

The contents of the sample flask was then valved in-line through the HT converter and heated transfer lines to 4A molecular-sieve traps. Fifteen sample volumes of helium containing 1% hydrogen were passed through the flask to quantitatively transfer the sample. After collection, the molecular-sieve sample was transferred to a round-bottom flask and refluxed with 100 ml of distilled water for 120 min. The liquid was decanted and doubly distilled prior to tritium determination by liquid scintillation beta counting.

This process was repeated for each of the five calibration gas cylinders (Table 2). The electrometer responses were plotted vs liquid scintillation counting data to obtain the solid calibration curve shown in Fig. 3. Results from the molecular-sieve samples were in good agreement with the electrometer measurements for all calibration gases. In two cylinders, the tritium analysis obtained from molecular-sieve samples was lower than the mass spectrometer analysis quoted by Matheson. Later experiments demonstrated that the HT concentration was decreasing with time.

After the ionization chamber was calibrated, calibration gases were valved through the charcoal traps and the ionization chamber. A characteristic tritium breakthrough (Fig. 4) was obtained in all cases. The average value of the sine wave response was in good agreement with



Table 2. Calibration gas concentrations

Cylinder	Bq/ml STP	
	Matheson	Liquid scintillation
43372	1.82	1.83
13690T	18.2	14.9
15417P	35.8	38.0
10693T	377	358
66204	352	377

steady-state values obtained with direct flow to the ionization chamber. Flow rates of 200, 500, and 1000 ml/min were used to test 1, 3, and 8 g of charcoal respectively. A small flow dependence was observed but was insignificant for this experimental program.

Oxidation of HT to HTO was evaluated with a laboratory model HT converter. Three 1-liter samples from separate calibration gas cylinders were tested. The calibration gas was swept through the HT converter and heated lines to molecular-sieve traps. Fifteen sample volumes of 1% hydrogen in helium were used as sweep gas. Sample analysis by liquid scintillation beta counting gave results that were in excellent agreement with the known concentration. Hydrogen carrier was used for these tests to transport the HTO form of tritium.

After evaluation of the HT converter, a laboratory model HTO converter was connected between the HT converter and molecular-sieve traps with heated lines. The HTO converter was evaluated with the procedure used for the HT converter. Less than 3% of the sampled tritium was retained on the molecular sieve for each of three tests. At that time,



these tests were thought to be adequate evaluation of the HTO converter. The low tritium concentrations found on the molecular sieves are now considered to be the result of HT exchange with small concentrations of atmospheric water on the molecular sieve.

## 5. EXPERIMENTAL RESULTS

Tritium monitoring, which began in June 1975, was conducted in two series. The first series used high-purity helium (the normal capsule sweep gas) and was conducted as the capsule attained a burnup of  $\sim 75$  MWd/kg of heavy metal. At that time, flow resistance through the GB-10 fuel region had increased to such an extent that full flow rates could not be obtained through the fuel region.

The second series of tritium monitoring experiments was conducted as the fuel attained a burnup of approximately 100 MWd/kg of heavy metal. Flow resistance through fuel region of the GB-10 rod increased with fuel burnup during the latter part of the irradiation. The second series of tritium experiments utilized 1% hydrogen in helium as sweep gas. Priority was placed on preserving the fuel region flow restriction for postirradiation examination. Therefore, flow was not directed through the fuel region during the second experimental series.

A table summarizing the 15 tritium monitoring experiments is presented in Appendix I. Results of the molecular-sieve samples are presented in Appendix II. Because several unexpected phenomena occurred (see Sect. 6.1), the tritium monitoring schedule was continuously revised to conform with new information and constraints.



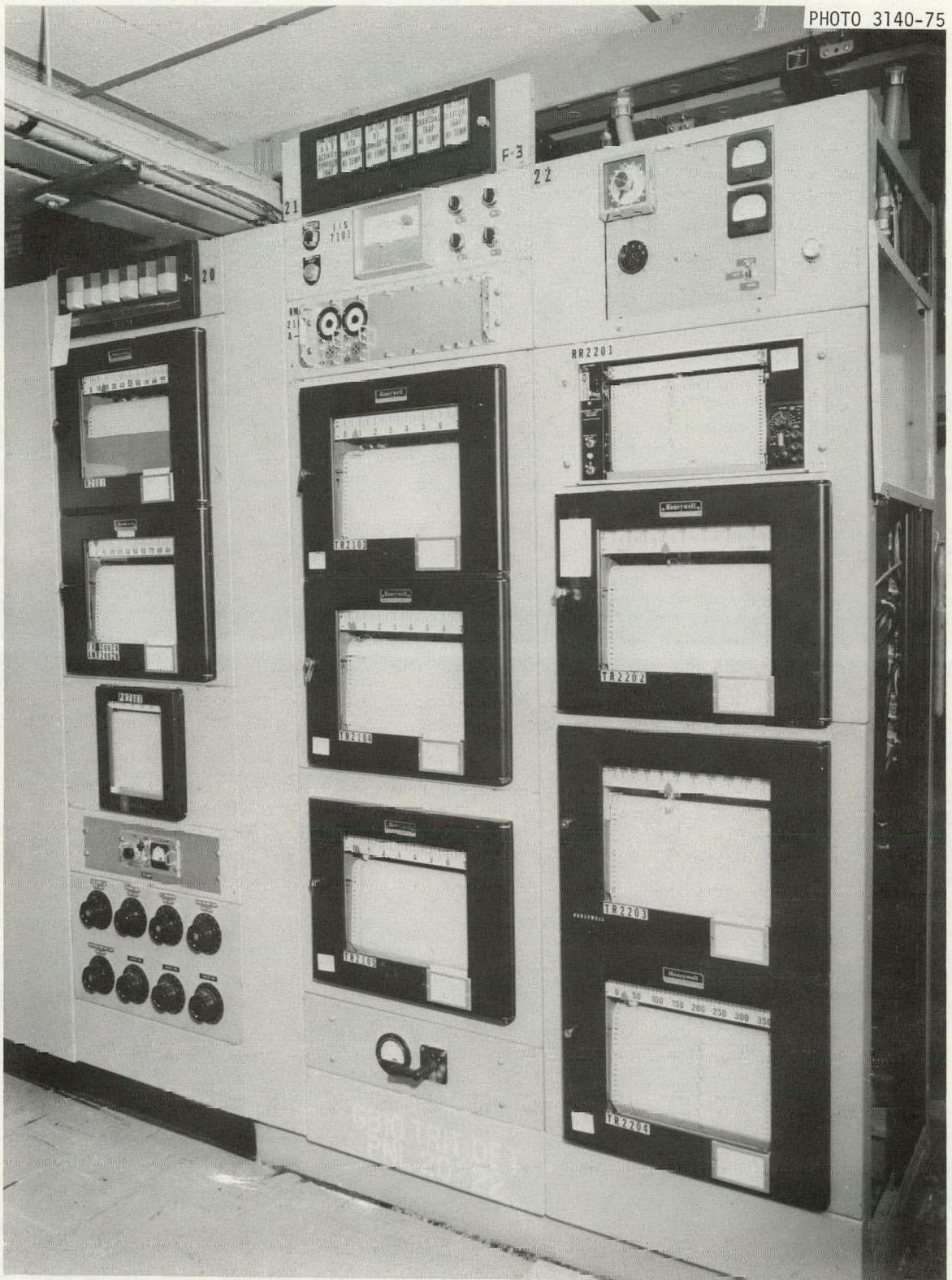


Fig. 9. Control room instrumentation for tritium monitoring system in ORR facility.



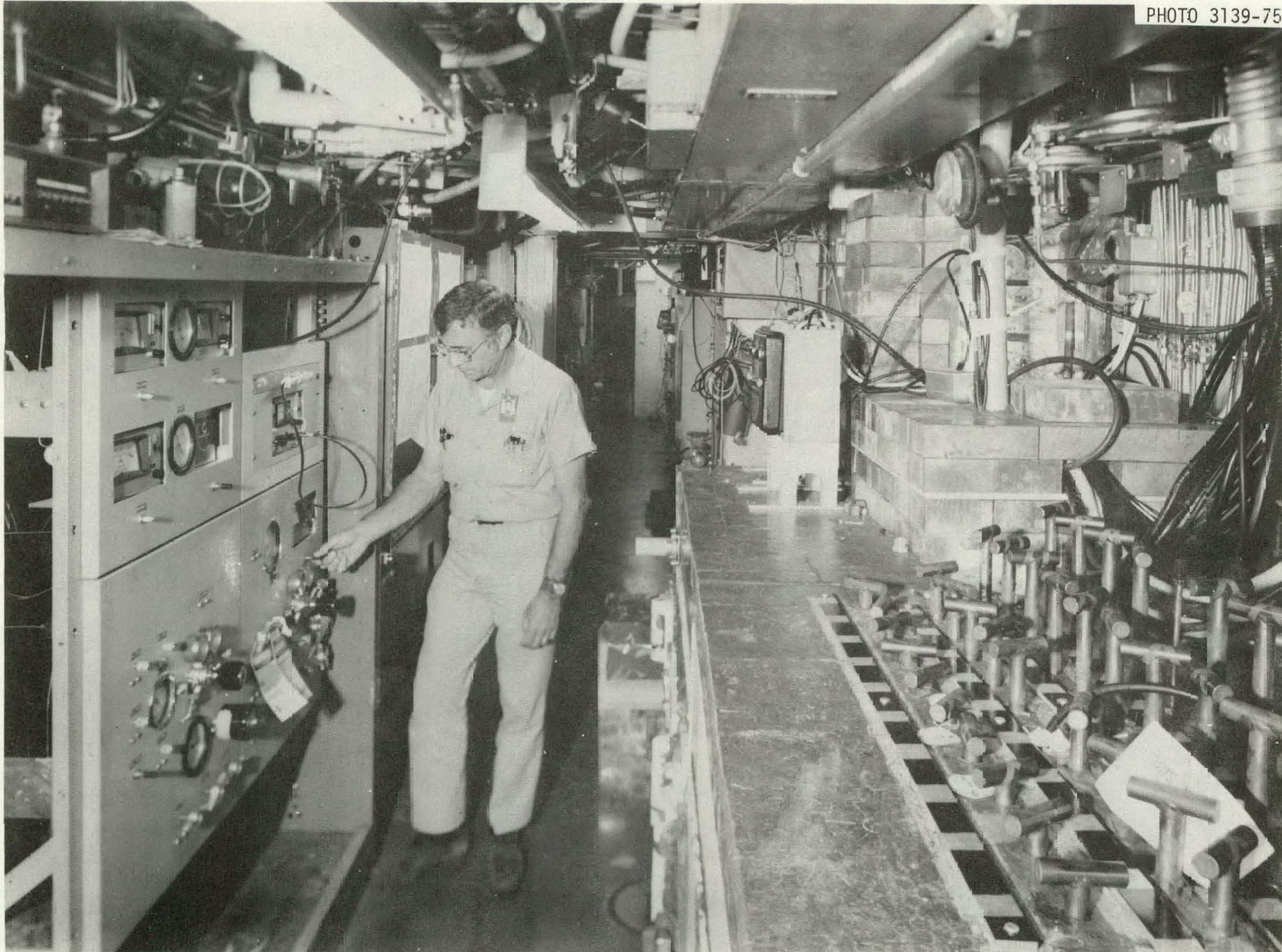


Fig. 1C. Valve box and controls for tritium monitoring system in ORR facility.

Control room instrumentation for the tritium monitoring system is shown in Fig. 9. The work area in the vicinity of valve box is shown in Fig. 10.

## 5.1 Experiments, First Series: July 12 to October 1, 1975

### 5.1.1 In-line calibration

Experiment 1. The objective of experiment 1 was to determine the ionization chamber baseline response in the ORR environment. The baseline established in the laboratory was 0.60 mV, unshielded. In the ORR, the chamber was installed within a 0.1-m-thick lead shield to reduce the effect of external low-level radiation. By valving high-purity helium from the calibration gas manifold directly to the ionization chamber, the flow rate was maintained at 200 ml/min at a pressure of 34.5 kPa for several hours. A 2 MBq source of  $^{137}\text{Cs}$  was used to test the ionization chamber response to an external radiation source. The ionization chamber response remained stable at 0.5 mV during the test and did not respond to the  $^{137}\text{Cs}$  source.

Experiment 2. The objective of experiment 2a was to verify the ionization chamber calibration after installation in the ORR facility. Calibration gas was passed directly to the ionization chamber. The system was operated at steady state for several hours with a flow rate of 200 ml/min at 34.5 kPa. The tritium concentration obtained from a molecular-sieve trap sample collected in this flow mode was in good agreement with the calibrated electrometer response. However, these values were lower than the laboratory calibration under similar conditions.

Experiment 2b was designed to investigate the tritium retention characteristics of the neoprene diaphragm in the back-pressure control valve downstream from the ionization chamber. The flow path was altered to bring the incoming calibration gas in contact with the neoprene diaphragm before entering the ionization chamber. The electrometer response remained constant and was in good agreement with a molecular-sieve sample. Therefore, tritium absorption on the neoprene diaphragm was not significant. The "sweep-down" with helium was monitored after valving out the calibration gas. The electrometer followed the expected sweep-down curve to a baseline of 0.5 mV. Thus memory effects in the monitoring system were of no significance. Experiment 2b demonstrated that the ionization chamber, HT converter, and molecular-sieve traps were operational. Results for molecular-sieve samples collected in 2a and 2b are presented in Appendix II. This experiment also demonstrated that the baseline could be reproduced after passing calibration gas through the system.

Experiment 3. Experiment 3 was designed to evaluate the tritium monitoring system with capsule sweep gas. Experiment 3a was planned to evaluate the performance of the charcoal traps. Calibration gas was valved through 3 g of charcoal at a flow rate of 200 ml/min at 34.5 kPa. Tritium breakthrough was normal, and the electrometer response was in agreement with the value obtained in the previous experiment. However, constant pressure over a wide flow range could not be obtained at 34.5 kPa due to the pressure drop across the downstream lines that included check valves. The tritium system pressure was increased to 69 kPa with excellent stability and was regulated to a nominal 69 kPa for all subsequent experiments.



After the system pressure stabilized at 69 kPa, a molecular-sieve sample collection began. During this collection period, a temporary loss of liquid nitrogen to the charcoal traps allowed the trap temperature to increase about 5°C before sample collection ended. Sufficient tritium was released from the charcoal traps to invalidate the sample. Since the electrometer had responded as expected, the molecular-sieve sample was not repeated.

Experiment 3b was designed to evaluate the HTO converter. About 1 h after regaining temperature control of the charcoal traps at  $-188 \pm 1^\circ\text{C}$ , the HTO converter was valved in-line. The experiment was aborted 63 min later due to a loss of charcoal-trap coolant. Normal tritium breakthrough was not observed while the HTO converter was valved in-line. Integration of the electrometer response indicated that 23 kBq of tritium passed to the system had not been released when the experiment was terminated. After normal cleanup procedures, a baseline of 0.5 mV was obtained.

Temperature regulation problems were attributed to excessive liquid-nitrogen flow to the charcoal traps. A constant supply of liquid nitrogen was achieved by regulating the overpressure on the supply Dewars at a nominal pressure of 62 kPa. Perturbations on Dewar change-over were eliminated by making the changes about midway on the cooling cycle.

#### 5.1.2 Experiments using helium as a sweep gas

Experiment 4. The objective of experiment 4a was to determine tritium (HT) in the GB-10 sweep gas flowing in the TT-TT mode. With the capsule at 44.3 kW/m, capsule gas flow of 200 ml/min STP was valved to

the tritium monitoring system, bypassing the HTO converter. The electrometer baseline increased in about 1 min from 0.5 mV to 1.1 mV and remained stable. After sufficient time for breakthrough and steady-state operation, a molecular-sieve sample was collected which gave a tritium release of 1.25 Bq/min. Tritium breakthrough had not been observed.

The objective of experiment 4b was to determine total tritium (HT + HTO) flowing in the TT-TT mode. The run was continued by valving the HTO converter in-line. About 45 min later, a small peak (9 mV max) was observed which slowly decreased to near baseline. While the system was operating under steady-state conditions, a molecular-sieve sample was collected but was later shown to be invalid due to tritium retention by the HTO converter (see Sect. 6.1.1).

Experiment 5. By the time experiment 5 was conducted, flow resistance through the fuel region of GB-10 was appreciable.<sup>6</sup> Following several days of stable operation at 44.3 kW/m, a flow of 23 ml/min STP was obtained in the BF-TT mode. To obtain a faster monitoring system response time, this stream was merged with 177 ml/min STP of capsule supply helium from the capsule bypass line (Fig. 7) to give 200 ml/min STP to the tritium monitoring system. Since the HTO converter had previously shown anomalous behavior, it was bypassed, and the electrometer response immediately increased from a baseline of 0.6 mV to 1.1 mV and remained stable. Tritium breakthrough was not observed during 2 h of stable operation. The HTO converter was then valved in-line. About 45 min later, tritium breakthrough with an electrometer response of 3.3 mV was observed. The response decreased slowly to near baseline, and a

molecular-sieve sample was collected but was considered invalid due to retention of tritium by the HTO converter. However, this experiment demonstrated that the system could be operated with low flow rates from the capsule.

Experiment 6. In experiment 6, a tritium sample was obtained while flowing high-purity helium through the HTO converter. The behavior of the HTO converter in experiments 4 and 5 suggested that some monitored tritium was released from the converter. High-purity helium (200 ml/min) was valved through the tritium monitoring system, including the HTO converter. An apparent breakthrough of approximately 2 mV was observed. As in previous experiments, the electrometer response slowly decreased to near baseline. After 4 h of stable operation, a molecular-sieve sample gave a tritium concentration of 15.3 mBq/ml STP. This concentration was indeed low, but sufficiently high to negate the experimental data obtained by flowing through the HTO converter in experiments 4 and 5. Thus the same general response had been obtained by flowing high-purity helium through the HTO converter and tritium monitoring system as was obtained on the previous capsule gas experiments by utilizing the same flow path.

Experiment 7. The purpose of experiment 7a was to determine the extent of contamination of the tritium monitoring system when the HTO converter was bypassed. The data obtained in experiment 6 dictated that this experiment be inserted in the monitoring schedule. High-purity helium (200 ml/min) was valved through the tritium monitoring system, bypassing the HTO converter. Tritium breakthrough was not observed during 3.5 h of stable operation. A molecular-sieve trap sample

collected in this flow mode gave a detection limit of 0.4 mBq/ml. Thus the tritium monitoring system was not contaminated when the HTO converter was bypassed.

Experiment 7b was conducted to determine tritium (HT) release in the BF-TT flow mode at a high flow rate. By reducing the capsule power from 44.3 kW/m to 39.4 kW/m, the flow resistance<sup>6</sup> was partially relieved, and a flow rate of 745 ml/min STP was obtained in the BF-TT flow mode. This gas stream was then valved to the tritium monitoring system, bypassing the HTO converter. An immediate ( $<1$  min) high-level electrometer response (430 mV) was observed. After the electrometer stabilized, the ionization chamber was isolated and the decay followed for 18 min. This activity was later shown to be  $^{24}\text{Ne}$  (see Sect. 6.1.2). The ionization chamber was then valved in-line to observe tritium breakthrough. However, tritium breakthrough was not observed during 3 h of stable operation. A molecular-sieve sample collected to determine tritium in the presence of  $^{24}\text{Ne}$  gave a release rate of 36.7 Bq/min. This release rate was approximately 1% of the calculated production rate based on a 0.0001 fission yield.

Experiment 7c was conducted to observe the response when the HTO converter was valved in-line. The BF-TT sweep gas then was valved through the HTO converter for about 2.5 h. During this time the electrometer response increased slowly ( $^{24}\text{Na}$  buildup) with no change attributed to the HTO converter. The experiment was then terminated. After normal cleanup, the electrometer response was 125 mV. The ionization chamber was again isolated and the decay followed for 72 h. One week later, the electrometer response had returned to the normal baseline value of 0.5 mV.

Experiment 8. The purpose of experiment 8 was to calibrate and reevaluate the tritium monitoring system in the ORR. Two problem areas needed clarification. First, the ionization chamber and molecular-sieve traps were in good agreement, but the values obtained for the calibration gases were lower than the laboratory values and the concentrations quoted by Matheson. Second, anomalous behavior had been observed with the HTO converter. This was of particular significance since the HTO converter had not been adequately tested in the ORR.

Experiment 8a was a repetition of experiment 2a calibration. Calibration gas (cylinder 43372) was valved to the tritium monitoring system, bypassing the HTO converter. The flow rate was 200 ml/min STP. Breakthrough was observed 86 min later followed by a slow, steady increase. In 4 h the steady-state value of 51.3 mV was reached. This slow approach to steady state was attributed to interchange (HT, H<sub>2</sub>, HTO, H<sub>2</sub>O) on the room-temperature molecular-sieve trap located on the calibration gas header. This problem was observed in other experiments (see Sect. 6.1.3). After steady state was reached, a molecular-sieve sample agreed well with the electrometer but indicated that the HT form of tritium in the cylinder was decreasing with time.

At the beginning of experiment 8b, calibration gas continued to flow through the tritium monitoring system at steady state. The system had been at steady state for several hours with calibration gas flowing at 200 ml/min. The HTO converter was then valved in-line with an electrometer response at 51.5 mV. The electrometer response was then characterized by a maximum of 73 mV about 20 min after breakthrough. Thirty minutes later the response had decreased to 10.5 mV. The

electrometer response continued to decrease and was reading 0.9 mV when the HTO converter was valved out after 3.5 h of operation. Thus the general response of this experiment was similar to that of previous capsule experiments. After the experiment, cleanup of the system using high-purity helium from the calibration gas header was slower than normal. Apparently, tritium was being released from the molecular-sieve trap on the calibration gas manifold.

This experiment demonstrated that the HTO converter was quantitatively retaining tritium from high-purity helium. The laboratory-model HTO converter was laboratory tested in conjunction with the laboratory-model HT converters and molecular-sieve traps. Hydrogen carrier was routinely used to quantitatively transfer the HTO form of tritium. Thus, proper operation of the HTO converter could be expected if hydrogen carrier were present in the gas stream. The next experiment tested this hypothesis.

#### 5.1.3 Experiments using helium as a sweep gas with hydrogen added in the tritium monitoring system

Experiment 9. Experiment 9a released the tritium inventory from the HTO converter by passing hydrogen carrier through the monitoring system. Helium containing 1% hydrogen was valved through the HTO converter at a flow rate of 200 ml/min STP. Tritium breakthrough was observed in 32 min. The electrometer response increased from 0.5 mV to a maximum of 450 mV about 25 min after breakthrough. Twenty minutes later the response had decreased to 150 mV. After 2.5 h the response had decreased to 1.3 mV. Integration of released tritium indicated that the total inventory from previous experiments had been released.

Experiment 9b was carried out to demonstrate that complete transmission through the HTO converter could be obtained when hydrogen was present in the gas stream. Calibration gas (cylinder 13690T) with an assigned tritium concentration of 18.3 Bq/ml STP was merged with helium containing 1% hydrogen upstream from the HTO converter. The calibration gas flow rate was 400 ml/min, and the flow of helium containing 1% hydrogen was 100 ml/min, to give a total flow of 500 ml/min with a hydrogen concentration of 0.2%. Tritium breakthrough was observed 32 min later. Two hours later the electrometer response was 195 mV and increasing very slowly; the response calculated from the original calibration was 400 mV.

Since it was suspected that the hydrogen concentration was too low for complete transmission, the merged gas stream was switched to 100 ml/min STP of calibration gas and 400 ml/min of 1% hydrogen in helium. This increased the hydrogen carrier from 0.2 to 0.8%. The electrometer response was characterized by a maximum which readily decreased to 53 mV. A 100-mV response was expected from the original concentration of cylinder 13690T. A molecular-sieve sample was collected after the system had operated at steady state for about 1 h. After valving out the calibration gas, sweep-down of the HTO converter with 1% hydrogen in helium indicated that tritium retention was insignificant.

Experiment 9c was the investigation of the apparent slow approach to steady state in experiments 9a and 9b. Calibration gas (cylinder 13690T) flowing at 400 ml/min was merged with 100 ml/min of helium containing 1% hydrogen. The combined stream of 500 ml/min STP containing 0.2% hydrogen was valved to the HTO converter and the tritium monitoring

system. Sharp tritium breakthrough was observed 15 min later, and the response increased rapidly to a steady-state value of 55 mV with no indication of peaking. With 0.2% hydrogen in the gas stream, the HTO converter appeared to be operating as predicted. These data suggest that the previous slow approach to steady state was due to the establishment of equilibrium between HT, H<sub>2</sub>, HTO, and H<sub>2</sub>O on the molecular-sieve trap located on the calibration gas header. Therefore the slow response obtained in 9b cannot be attributed to insufficient hydrogen (0.2%) in the gas stream.

Experiment 9d was the investigation of the low electrometer responses obtained with the calibration gas. Electrometer responses obtained in 9b and 9c indicated that the calibration gas was a factor of 2 lower than the previously assigned value. To eliminate the possibility of tritium holdup in the system, 100 ml/min STP of calibration gas and 400 ml/min of 1% hydrogen in helium were valved directly to the ionization chamber. The ionization chamber response increased rapidly and stabilized at 51 mV after 20 min. In this experiment the peaking effect was not observed. Therefore, possible tritium holdup in the system was eliminated when 0.8% hydrogen was present in the gas stream.

Experiment 9e was direct flow recalibration with calibration gas (cylinder 13690T) in high-purity helium. This experiment was considered necessary because the peaking effect observed in the laboratory was not observed in experiment 9d. Calibration gas was valved directly to the ionization chamber at the rate of 495 ml/min STP. An initial peak of 228 mV was observed, which decreased slowly to 213 mV. This behavior was similar to that of laboratory experiments utilizing high-purity



helium. Thirty-eight minutes after breakthrough, 50 ml/min STP of 1% hydrogen in helium was merged with the calibration gas. Instead of decreasing by the dilution effect, the ionization chamber response increased from 213 mV to 228 mV and remained steady (Fig. 11). When the 228-mV response was corrected for dilution, excellent agreement was obtained with the previous value of 51 mV. Also, the results were in good agreement with the molecular-sieve samples collected in 9b. Clean-up again suggested that tritium had been retained on the molecular-sieve trap located on the calibration gas manifold.

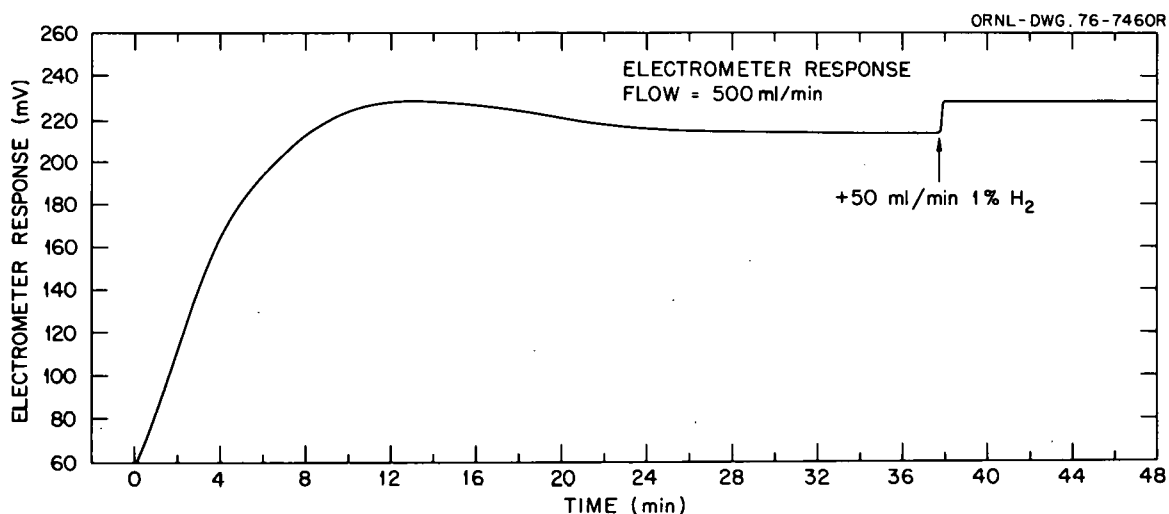


Fig. 11. Electrometer response when hydrogen was added to gas stream of tritium in high-purity helium.

The lower HT concentration observed in cylinder 13690T probably resulted from an equilibrium (including interchange) process within the cylinder. The decrease in HT concentration for this cylinder was first observed during calibration and continued to decrease with time. The other calibration gas cylinder (43372) that was frequently used also decreased with time. These data are included in Appendix II.

This experiment demonstrated that the tritium monitoring system met design criteria when hydrogen was present in the gas stream. The slow approach to steady state was probably of equilibrium origin and associated with the molecular-sieve trap on the calibration gas header. Therefore, pertinent information was not obtained on the necessary or optimum hydrogen carrier concentration. The improved performance of the ionization chamber gave a new calibration curve (Fig. 3). The presence of hydrogen produced steady-state responses in a shorter time period; therefore, more conditions could be evaluated during an experiment. Further, the faster response permitted more detailed observations.

Experiment 10. The purpose of experiment 10 was to determine the molecular species of tritium (HT or HTO) released at high flow rates in the BF-BT flow mode. To obtain a high flow rate, the capsule power was reduced from 44.3 kW/m to 39.4 kW/m. At the reduced power, a flow of 745 ml/min STP was obtained through the fuel region of the capsule.

Experiment 10a was conducted to determine the HT form of tritium. Capsule gas flowing at 745 ml/min was merged with 155 ml/min of helium from the capsule bypass line (see Fig. 7) and 300 ml/min of 4% hydrogen in helium at the tritium monitoring system. This gas stream was first valved to the tritium monitoring system, bypassing the HTO converter. As expected, an immediate high-level response due to  $^{24}\text{Ne}$  was observed. Since observation of tritium breakthrough was not expected, a molecular-sieve sample was collected after sufficient time had elapsed for steady-state operation. This sample gave a release rate of 382 Bq/min.

For experiment 10b, the gas stream was valved through the HTO converter to obtain total tritium (HT + HTO). At that point the response

due to  $^{24}\text{Ne}$  and  $^{24}\text{Na}$  was about 525 mV, which precluded observation of tritium breakthrough with the ionization chamber. After sufficient time for steady state was allowed, a molecular-sieve sample was collected. However, an error in collection technique invalidated the sample. Thus another attempt to determine the HT/HTO ratio was thwarted.

Experiment 11. At this point, only one more tritium experiment could be performed due to funding limitations. Tritium injection was selected for this experiment to eliminate the time-consuming molecular-sieve samples and thereby obtain data for more flow modes. Up to this point we had deliberately avoided injecting tritium into the complex capsule system because some of the tritium could have been trapped in various stagnant areas, thus precluding further low-level release measurements. The GB-10 capsule power was reduced to 39.4 kW/m to permit flow through the fuel region. The gas stream for the entire run consisted of 900 ml/min of calibration gas from cylinder 15417P (originally containing 38 Bq/ml of tritium) merged with 300 ml/min of helium containing 4% hydrogen upon entry to the tritium monitoring system. Hydrogen was not added to the capsule sweep gases. Since hydrogen was present in the tritium monitoring system, the HTO converter was valved in-line throughout the experiment.

Experiment 11a was conducted to determine the tritium (HT) concentration in cylinder 15417P. After determining that the tritium (HT) concentration in the calibration gas cylinders was decreasing with time, this step was included in each experiment which used calibration gas. The gas stream (900 ml/min STP of calibration gas and 300 ml/min of helium containing 4% hydrogen) was valved directly to the ionization

chamber. The approach to steady state was slower than theoretically predicted, but stable operation was observed at 1000 mV. This was approximately 50% of the original calibration value.

Experiment 11b consisted of valving calibration gas through the capsule TT-TT flow mode and then to the tritium monitoring system. Tritium breakthrough time was normal for this mode. After breakthrough, the electrometer increased rapidly to 21 mV and then continued to increase slowly. The experiment was continued in this flow mode with stable operation for 3.5 h with the electrometer increasing slowly to 95 mV. The TT-TT flow was then terminated to search for a possible explanation of the low transmission.

Experiment 11c was conducted to evaluate the transmission of tritium in high-purity helium through the long stainless steel lines, the valves, and the fittings. In the previous experiment, carrier-free calibration gas was passed to and from the GB-10 capsule in the TT-TT mode. The hydrogen carrier was merged with the calibration gas just prior to entering the tritium monitoring system. Valving the calibration gas through the BT-BT fuel rod bypass mode (see Fig. 7) would require the calibration gas to travel through about 50 m of stainless steel lines. Supply and return lines were the same lines used in experiment 11b and shown in Figs. 1 and 8.

Calibration gas from cylinder 15417P was valved to the tritium monitoring system through the BT-BT bypass mode. The flow (900 ml/min calibration gas and 300 ml/min STP of helium containing 4% hydrogen) to the tritium monitoring system was the same as in experiment 11b. Breakthrough was normal, and the response readily stabilized at 1000 mV.

Complete recovery had been obtained after passing tritium in high-purity helium through about 50 m of stainless steel lines, valves, and fittings. Therefore the tritium losses in the TT-TT mode were not due to permeation of or retention by lines, valves, and fittings.

For experiment 11d, calibration gas was valved to the tritium monitoring system through the BT-TT flow mode. The neon signal appeared immediately and gave an average reading of 16 mV. Tritium breakthrough occurred 12 min later and was characterized by a net maximum of 13 mV, which slowly decreased to 7 mV. The final net response of 7 mV was equivalent to a transmission rate of less than 1%.

For experiment 11e, calibration gas was valved through the TT-BT flow mode. In this mode, the sweep gas travels the same pathway in the capsule as in experiment 11d. However, the gas exits the capsule through the BT line rather than through the TT line. The BT line was used occasionally, whereas under normal operating conditions the capsule was vented through the exit TT line. Therefore, fission product deposition should be greater on the exit TT line than on the BT line.

Breakthrough was observed 11 min after valving the calibration gas through the TT-BT flow mode. Following breakthrough, the response was characterized by a maximum of 137 mV, decreasing to 41 mV during 3 h of stable operation. If the final response was all due to transmission, the maximum transmission was 4.1%. Since transmission could not be obtained through the top of the capsule, the possibility of obtaining meaningful data from flow through the fuel was extremely remote. Therefore, the planned flow of calibration gas through the fuel region was omitted to preserve the fuel integrity for future tritium monitoring and

postirradiation examination. Normal cleanup procedures were followed. Cleanup following the higher levels of tritium used in this experiment strongly suggested an equilibrium (HT, H<sub>2</sub>, HTO, H<sub>2</sub>O) process on the molecular-sieve trap located on the calibration gas header.

## 5.2 Experiments, Second Series: June 29 to July 14, 1976

A second series of experiments began in late June 1976. Several constraints existed for this series of experiments. Since the capsule had attained and perhaps exceeded, the burnup goal of 100 MWd/kg and was scheduled for postirradiation examination, time precluded the installation of controlled hydrogen and water injection equipment. Earlier experiments had demonstrated the desirability, if not in fact the necessity, of using hydrogen carrier in the sweep gas. Since the optimum hydrogen concentration had not been determined, a decision was reached to use sweep and calibration gas containing 1% hydrogen. Satisfactory results had been obtained in laboratory tests and previous capsule experiments with 1% hydrogen. A cylinder (29333) of Matheson certified standard tritium in helium containing 1% hydrogen was obtained for calibration and injection. A cylinder (94649) of helium containing 1% hydrogen was obtained for sweep gas. These cylinders were placed on the calibration gas header in place of the original calibration gases.

During the last year of capsule operation, flow restriction in the fuel region increased. Therefore, primary emphasis was placed on the fuel constriction and its preservation for postirradiation examination. Thus a serious limitation was placed on tritium monitoring when permission was not obtained to flow hydrogen carrier through the fuel region.

One recalibration and three tritium monitoring experiments were conducted in this series.

### 5.2.1 Recalibration

Experiment 12. Experiment 12a was devoted to system checkout and calibration since the tritium monitoring system had been on standby for several months. Calibration gas containing 1% hydrogen was valved directly to the ionization chamber at a flow rate of 1200 ml/min STP. The approach to steady state was slow, an effect attributed to the establishment of a new equilibrium on the molecular-sieve trap located on the calibration gas header. Steady-state operation at 2000 mV was attained with no indication of peaking. The observed response was in good agreement with the tritium value of 39.7 Bq/ml STP reported by Matheson for cylinder 29333.

Experiment 12b was conducted to check the flow dependence when hydrogen was present in the gas stream. The calibration gas flow of experiment 12a was continued for 1 h of stable operation. Then the flow was decreased to 300 ml/min STP, and operation continued for 1 h. The ionization chamber did not exhibit flow dependence in the presence of 1% hydrogen.

Experiment 12c was conducted to evaluate the tritium monitoring system. Calibration gas from cylinder 29333 was valved to the tritium monitoring system including the HTO converter at a flow rate of 1200 ml/min STP. Tritium breakthrough and the approach to steady state were normal. A molecular-sieve sample collected after 1 h of stable operation gave a

tritium concentration of 40 Bq/ml STP. Thus the tritium system appeared to be operational and the original calibration valid. After system cleanup, the electrometer returned to the original baseline value of 0.5 mV.

### 5.2.2 Experiments using helium with 1% hydrogen as sweep gas

Experiment 13. Experiment 13a was conducted to release and measure the tritium inventory in the lines and top portion of the fuel rod, using helium containing 1% hydrogen as sweep gas, and then measure the steady-state response. When the sweep gas was valved through the TT-TT capsule flow mode, breakthrough was characterized by a maximum of 36 mV, which readily decreased to 20 mV. The response then increased slowly to approximately 95 mV over the next 3.5 h of stable operation. During this time the HTO converter was valved out for 1 h. No significant change was observed when the HTO converter was valved out or valved back in-line. Isolation of the ionization chamber indicated that  $^{24}\text{Ne}$  did not significantly contribute to the ionization chamber response in the TT-TT flow mode.

For experiment 13b, the flow was valved through the TT-BT mode, since steady state could not be attained in a reasonable time in the TT-TT flow mode. An immediate ( $\leq 1$  min) breakthrough of  $^{24}\text{Ne}$  was observed which stabilized at 16 mV. After tritium breakthrough, an 11,000-mV maximum was observed which decreased to 950 mV 1 h later. Again, no significant difference was observed when the HTO converter was valved out for 1 h. The ionization chamber response was 430 mV and decreasing when the TT-BT flow was terminated after 3.5 h of operation.



Experiment 14. Experiment 14a continued experiment 13b since steady state had not been attained in the previous run. Helium containing 1% hydrogen and flowing at 1200 ml/min STP was valved to the tritium monitoring system through the TT-BT flow mode. Tritium breakthrough was normal, giving a maximum response of 1800 mV, which decreased to 495 mV 1 h later. As the response continued to decrease, the HTO converter was valved out for 1 h with no significant change in response. Six hours after breakthrough, steady state was being approached at 196 mV. A molecular-sieve sample collected at this point gave a tritium release rate of 3650 Bq/min, which was in good agreement with the ionization chamber.

For experiment 14b, tritium release at zero power was measured. While we continued to monitor tritium in the TT-BT flow mode, the capsule was retracted (reducing the power from 48.6 kW/m to near zero). During retraction, the charcoal-trap temperature dropped from 300°C to 250°C. The ionization chamber response decreased to 26 mV and stabilized. With the response stable, the ionization chamber was isolated, and the  $^{24}\text{Ne}$  contribution was determined to be insignificant. Tritium monitoring continued for 2.5 h with the capsule retracted and the charcoal trap controlling at 250°C. The ionization chamber response remained stable at 26 mV during this time period. Sweep-down following this experiment was normal.

Experiment 15. Injection of calibration gas containing 1% hydrogen was selected as the last tritium monitoring experiment for capsule GB-10. This experiment was conducted to determine the transmission rate through various flow modes. After having determined that transmission

was complete in the TT-BT flow mode, temperature-dependent tritium release was investigated.

In experiment 15a, tritium concentration in cylinder 29333 was determined by valving calibration gas directly to the ionization chamber at 1200 ml/min STP. Again, the approach to steady state was slower than expected, and the peaking effect was not observed. The steady-state response demonstrated that the tritium (HT) concentration of the calibration gas had not changed.

Experiment 15b repeated the flow dependence test. Calibration gas had been flowing directly to the ionization chamber at 1200 ml/min for about 1 h when the flow was reduced to 300 ml/min. About 1 h later the flow was increased to 1200 ml/min STP. No detectable flow dependence was observed. Therefore the ionization chamber was not flow dependent when hydrogen was present in the system.

Experiment 15c was planned to determine tritium transmission through the TT-BT flow mode with 1% hydrogen as a carrier. Calibration gas was valved to the tritium monitoring system at 1200 ml/min STP, using the TT-BT flow mode. Tritium breakthrough was normal, exhibiting a peak and then decreasing to the expected value for complete transmission (calibration gas plus capsule tritium). Again, the HTO converter was valved out for 1 h with no significant change in response. Several hours of stable operation indicated that complete transmission was being observed. Therefore, complete transmission could be expected in the other flow modes. In this experiment the capsule release data were hard to interpret since the calibration gas was at least an order of magnitude higher than the expected and observed capsule release. The calibration gas was valved out for the remainder of the run.

Experiment 15d repeated the steady-state response obtained in 14a. Sweep gas containing 1% hydrogen was valved through the TT-BT flow mode at 1200 ml/min STP. The ionization chamber response stabilized at a steady-state level of 200 mV. The fuel rod charcoal-trap heater was operating at 307°C prior to reduction of capsule power. The higher charcoal-trap temperature may have been responsible for the slightly higher steady-state response. Therefore the steady-state response observed earlier could be reproduced.

Experiment 15e was the observation of the electrometer response as a function of capsule power. With the fuel rod power at 48.6 kW/m, the fuel rod charcoal trap at 307°C, and the ionization chamber stable at 200 mV, the capsule power was reduced by 50% while adjusting the charcoal trap to 300°C. The ionization chamber response decreased to 100 mV and remained steady for 1 h. The fuel rod power was then increased to 48.6 kW/m while holding the capsule trap at 300°C. The ionization chamber response increased to 154 mV and remained steady for 1 h. With the ionization chamber steady at 154 mV, the capsule was retracted (near zero power) and the charcoal trap controlled at 300°C. Under these conditions the ionization chamber gave a steady-state response of 77 mV.

In experiment 15f, electrometer response was measured as a function of charcoal-trap temperature. After about 1 h of stable operation at zero power, the charcoal-trap heaters were adjusted from a control point of 300°C to 250°C. The electrometer response readily stabilized at 25 mV, the same value obtained under similar conditions in experiment 14b. After 1 h of stable operation, power to the charcoal-trap heaters was reduced to zero. The electrometer decreased to 11 mV and remained

stable until the experiment was terminated. The rapid response of the ionization chamber to charcoal-trap temperature and fuel rod power changes indicated that temperature-dependent tritium release was occurring.

Electrometer values reported above as a function of power and temperature are gross readings which include tritium,  $^{24}\text{Ne}$ ,  $^{24}\text{Na}$ , and normal baseline. At full power, the  $^{24}\text{Ne}$  contribution was approximately 16 mV and was insignificant at zero power. The  $^{24}\text{Na}$  contribution was approximately 4 mV and constant. The baseline response was typically 0.5 mV. Electrical heating was used to control the temperature of the charcoal trap and the blanket region of the capsule. Fuel rod power contributed to the heating process, but the effect on tritium release at zero power was probably minimal since the electrometer responded rapidly to charcoal-trap temperature changes. When the electrical heaters were reduced to zero power, the maximum tritium release was 6.5 mV. At that time, the capsule thermocouples (Te-711, Te-712, Te-713)<sup>4</sup> were indicating 50°C.

## 6. DISCUSSION OF RESULTS

### 6.1 Unexpected Phenomena

#### 6.1.1 Retention of tritium by the HTO converter in high-purity helium

Unexpected behavior was observed in the first two capsule experiments (4b, 5) when the HTO converter was used. After valving the HTO converter in line in experiment 4b, a maximum of 9 mV was observed, which slowly decreased to near baseline. On successive use of the HTO converter in experiment 4b and 5, the general response was similar, but the magnitude

of the initial response decreased. Before experiment 4b was started, approximately 23 kBq of tritium had been passed to the HTO converter. However, thorough checkout of the converter's behavior had not been possible. The unexpected behavior of the HTO converter in experiment 4b and 5 together with literature data pertaining to magnesium hydride formation<sup>7</sup> led to the conclusion that tritium was being retained by the HTO converter from high-purity helium.

Experiments 6 and 7a were planned to determine the effect of tritium retention in the HTO converter on previous capsule experiments. Experiment 6 consisted of flowing high-purity helium through the HTO converter and collecting a molecular-sieve sample. Analysis gave 15 Bq/ml STP, which was indeed low, but sufficiently high to negate the experimental data obtained while using the HTO converter in experiments 4b and 5. Experiment 7a consisted of flowing high-purity helium around (bypassing) the HTO converter and collecting a molecular-sieve sample. The detection limit of 0.4 mBq/ml demonstrated that the system was clean and that capsule data were valid when the HTO converter was bypassed.

Due to operational difficulties in experiment 3b, the HTO converter had not been adequately tested. Therefore, experiment 8 was conducted to evaluate the converter. Experiment 8a demonstrated that the tritium monitoring system was operating satisfactorily when the HTO converter was bypassed. In experiment 8b, calibration gas from cylinder 43372 was passed through the HTO converter. The electrometer response was characterized by a maximum which decreased to near baseline after 200 min of operation. The response obtained in this experiment was typical of previous experiments when the HTO converter was valved in-line. This experiment was then terminated with the anomalous behavior explained.

Essentially all the tritium introduced to the HTO converter in high-purity helium was retained. The HTO conversion was laboratory tested in conjunction with the HT converter and molecular-sieve traps. However, hydrogen carrier was routinely used to quantitatively transfer HTO and was used in the laboratory evaluation of HTO conversion. Thus, proper operation of the HTO converter could be expected if hydrogen were present in the gas stream. The next logical step was to check that this hypothesis was true for the in situ HTO converter.

Experiment 9 was scheduled to evaluate the HTO converter using hydrogen carrier. Tritium retained by the HTO converter was quantitatively released when the converter was swept with helium containing 1% hydrogen. Complete transmission of calibration gas was observed when the gas stream contained 1% hydrogen. The sharp breakthroughs indicated that the HTO converter memory effect was insignificant. This was further evidenced during cleanup since tritium tailing was not observed. Therefore, satisfactory operation of the HTO converter could be expected for all experiments when hydrogen was present as a carrier.

Use of hydrogen carrier also provided other beneficial effects. Charcoal-trap breakthroughs were faster and better defined. The electrometer showed improved sensitivity and was not flow dependent. Figure 11 shows the peaking effect in high-purity helium and the response when hydrogen carrier was added to the gas stream. The peaking effect was not observed when hydrogen carrier was present in the system. These data strongly suggest that hydrogen carrier should be utilized for tritium monitoring.

### 6.1.2 Observation of $^{24}\text{Ne}$ in capsule sweep gas

In experiment 4a, when TT-TT sweep gas was valved to the tritium monitoring system, the electrometer response promptly increased ( $\leq 1$  min) from a baseline of 0.6 mV to 1.1 mV. The response remained stable throughout the run; the tritium level was low and was not observed on the electrometer. After the experiment and routine system cleanup, the baseline remained at 0.8 mV. This was the first time the baseline did not return to the minimal value of 0.5 mV.

In experiment 7b, instant breakthrough ( $\leq 1$  min) was observed when BF-TT sweep gas was valved to the tritium monitoring system. The response rapidly increased to 415 mV and stabilized. In an effort to determine the nature and origin of the activity, the ionization chamber was isolated, and the decay of this activity was followed by the electrometer.

Results of the decay study are shown in Fig. 12. The dashed line represents  $^{24}\text{Na}$  deposited in the chamber before isolation. The triangles show the ingrowth of  $^{24}\text{Na}$  during the decay study. When the raw data are corrected by subtracting the  $^{24}\text{Na}$  contribution, the closed circles are obtained, giving a half-life of 3.4 min, in excellent agreement with the published half-life of  $^{24}\text{Ne}$ . After the decay data were obtained, the ionization chamber was valved on-line and the experiment continued. Molecular-sieve samples were collected to obtain the tritium concentration.

The electrometer response increased slowly during the experiment. After the experiment and routine system cleanup, the electrometer was stable at 125 mV. Again, the ionization chamber was isolated and a

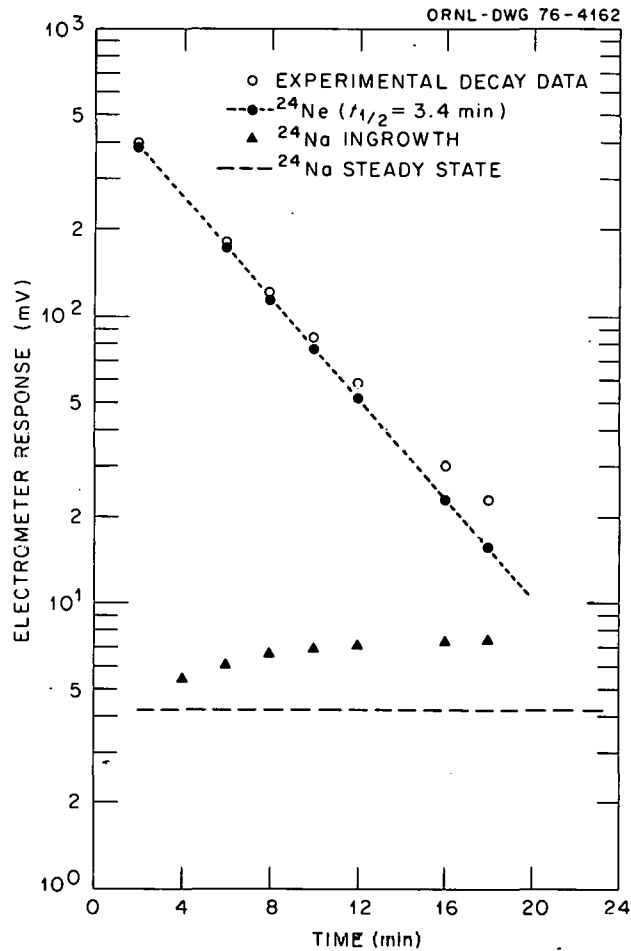


Fig. 12. Decay data for instant breakthrough activity.

decay study initiated. Figure 13 shows the results of this study. An experimental half-life of 15.0 h was obtained, which is the published half-life of  $^{24}\text{Na}$ . The presence of  $^{24}\text{Na}$  was further confirmed by gamma spectrometry on the molecular-sieve sample from experiment 10a.

Thus  $^{24}\text{Ne}$  was present in the capsule sweep gas and was not significantly detained by the charcoal traps. As  $^{24}\text{Ne}$  passed through the system, the daughter product  $^{24}\text{Na}$  deposited on the lines and in the ionization chamber. Since the actual concentration was quite low, several nuclear reactions could be postulated for the production of



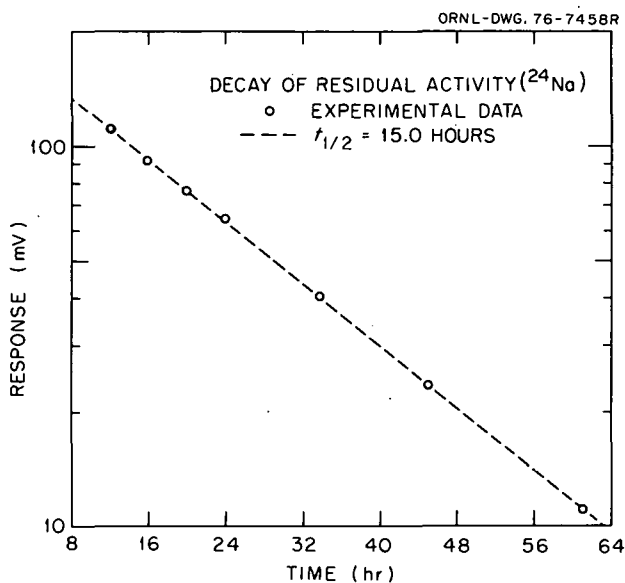


Fig. 13. Decay data for activity deposited in ionization chamber.

$^{24}\text{Ne}$ . Available data indicate that  $^{24}\text{Ne}$  was produced in the fuel region, but no large effort was made to determine the specific nuclear reactions contributing to its production.

The level of  $^{24}\text{Ne}$  in the sweep gas was a function of both flow mode and flow rate. The highest initial response was 420 mV observed in experiment 7b when the flow rate was 745 ml/min through the BF-TT flow mode. The highest post experiment response was 125 mV, also observed in experiment 7. The lowest initial response was approximately 0.5 mV, observed in experiment 4a with 200 ml/min flowing in the TT-TT flow mode. The presence of  $^{24}\text{Ne}$  in the sweep gas did not alter the experimental schedule; however, the use of molecular-sieve samples was necessary for some experiments.

### 6.1.3 Conversion of tritium (HT to HTO) in calibration gas cylinders

A slow approach to steady state when directing calibration gas to the tritium monitoring system was first observed in experiment 8a when about 4 h was required to reach steady state. Following the experiment, modified cleanup procedures demonstrated that tritium had been retained on the room-temperature molecular-sieve trap located on the calibration gas manifold. The trap had been installed to remove any water vapor ( $H_2O$  or  $HTO$ ) from the gas stream before directing the gas to either the GB-10 capsule or the tritium monitoring system. Experiment 8a also confirmed an earlier suspicion that the tritium (HT) concentration in the calibration gas cylinders had decreased. Data for the calibration gases are presented in Table 2. Appendix II gives a summary of the molecular-sieve samples.

Tritium (HT) concentration in cylinder 43372 had decreased by the time of experiment 2 to 74.5% of the original concentration and by experiment 8a to 60.1%. In experiment 9b, the tritium (HT) concentration in cylinder 13690T had decreased to 40.1% of the original value. Thus the tritium (HT) concentration in the calibration gas cylinders was decreasing with time. No attempt was made to determine the concentration of  $HTO$  in the calibration gas cylinders.

The conversion of HT to  $HTO$  within the cylinders could have been enhanced by impurities in the gas or cylinder wall effects. Extensive analyses were requested for all gases associated with the GB-10 capsule and tritium monitoring system. Detection limits from 1 to 10  $\mu l/liter$  were reported for gaseous impurities. However, these limits are much

too high to permit discussion of reaction or interchange mechanisms. Likewise, the unknown history of the cylinders precludes discussion of nonvolatile impurities and wall effects.

After experiment 8, each time a different calibration gas was valved to the tritium monitoring system the slow approach to steady state was observed. This delay was attributed to  $H_2$ , HT,  $H_2O$ , and HTO establishing an equilibrium on the molecular-sieve trap. The calibration gas in cylinder 29333 contained 1% hydrogen and did not decrease with time. However, the slow approach to steady state was observed with this cylinder. This response is consistent with the equilibrium process since the molecular-sieve trap contained an inventory of  $H_2O$  and HTO. After equilibrium had been established, stable operation could be expected throughout the experiment. No attempt was made to elucidate the species involved in the equilibrium. After the nature of this phenomenon was understood, its effect on the tritium monitoring program was insignificant.

## 6.2. Tritium Monitoring in High-Purity Helium

Tritium monitoring experiments utilizing high-purity helium sweep gas were conducted to study the effects of capsule power, flow rate, and flow mode on tritium production, release, and transport, and to attempt to determine the ratio of HT to HTO. However, several attempts to determine the HTO form of tritium were unsuccessful, and all quantitative data in this section pertain to the HT form of tritium. Quantitative tritium release data in high-purity helium were obtained only from molecular-sieve samples. In some experiments the release rate was below

the detection limit of the electrometer, and in other experiments the electrometer response was influenced by  $^{24}\text{Ne}$ .

Data from experiments 4a, 7b, and 10a (Table 3) gave representative release rates for the TT-TT, BF-TT, and BF-BT flow modes respectively. Tritium release in the TT-TT flow mode was quite low and should be representative of vented tritium (HT) from the GB-10 capsule. Experiments 7b and 10a showed the tritium release rate for the BF-BT flow mode to be ten times the release rate for the BF-TT flow mode, but even the relatively high release rate of experiment 10a represented only 10% of the calculated production rate based on a 0.01% fission yield. During three years of operation, the BT line was used only occasionally for experimental purposes, while the TT exit line was used almost all the time. Therefore the TT exit line may have been exposed to more fission product deposition than was the BT line. The charcoal should not retain tritium; however, the charcoal trap should be an area of high fission product deposition. If tritium was retained in areas exposed to fission product deposition, the relative release rates for the BF-BT and BF-TT flow modes can be easily explained. Tritium retention by fission products could be expected since they are routinely used as tritium getters.<sup>8,9</sup>

Table 3. Tritium release in high-purity helium vs flow mode

Experiment	Fuel rod power level (kW/m)	Flow mode	Capsule sweep flow rate (ml/min STP)	Tritium release (Bq/min)	Tritium release rate <sup>a</sup> as percent of calculated production rate (%)
4a	44.3	TT-TT	200	1.25	0.03
7b	44.3	BF-TT	745	36.7	1.1
10a	39.4	BF-BT	745	382	9.6

<sup>a</sup>Based on 0.001 fission yield.

For the last experiment (11) in this series, tritium injection was selected. Since the TT-TT flow mode was expected to yield the highest transmission, it was evaluated first. Representing flow to and from the fuel rod, this mode allows some diffusion to and from the charcoal trap (Fig. 14). After several hours of operation in the TT-TT flow mode, transmission had increased to only 9.4% of the predicted value. The BT-BT bypass flow mode was then evaluated to determine the transmission rate through long stainless steel lines. Since complete transmission was readily attained in this flow mode, tritium permeation of or retention on the stainless steel lines was insignificant.

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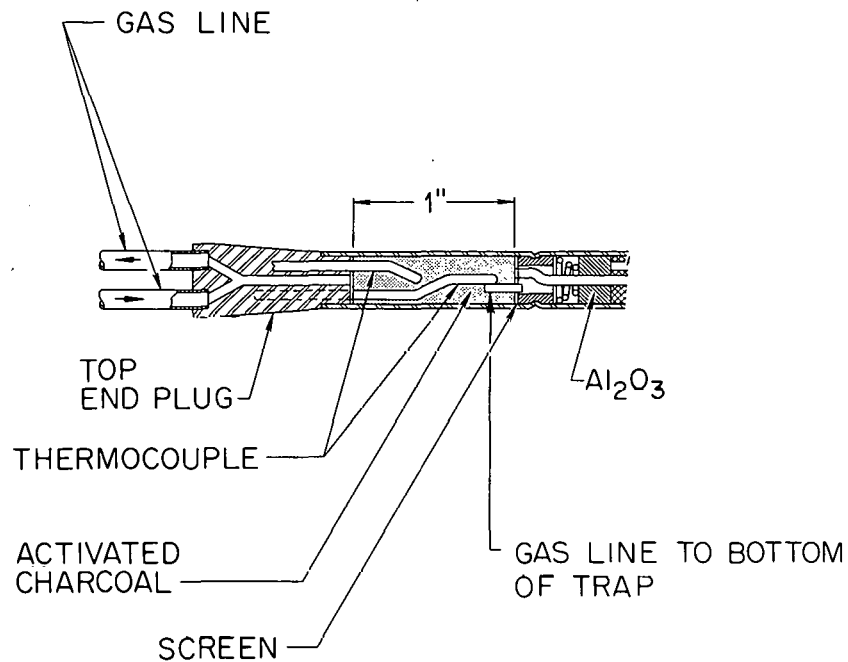


Fig. 14. Expanded view of charcoal trap region of irradiation capsule GB-10.



Transmission of tritium in high-purity helium for several flow modes is presented in Table 4. The maximum transmission obtained in the BT-TT flow mode was 0.75%. Flow through this mode required the gas to travel upward through the charcoal trap and exit through the TT line. In an effort to determine the area of retention, the gas was valved through the TT-BT flow mode. In this flow mode the gas traveled downward through the charcoal trap and exited through the BT line. A maximum transmission of 4.2% was observed. Thus the transmission difference between the TT-BT and the BT-TT flow modes appears to be the exit line. Due to the operational history, more fission product deposition was expected in the exit TT line than in the BT line. Tritium injection experiments indicated that tritium in high-purity helium is quantitatively transmitted through clean lines but retained in areas exposed to fission product deposition. If tritium monitoring in high-purity helium were necessary and were initiated on a new capsule with clean lines, tritium retention as a function of time and fuel burnup could be determined. Then satisfactory tritium monitoring on a capsule such as GB-10 would be feasible.

Table 4. Tritium transmission in high-purity helium vs flow mode

Flow mode	Percent transmission
BT-BT <sup>a</sup>	100
TT-TT	9.4
BT-TT	0.75
TT-BT	4.2

<sup>a</sup>BT-BT bypass mode (see Fig. 7).

### 6.3 Tritium Monitoring Using Hydrogen Carrier

For the second series of tritium monitoring experiments, 1% hydrogen in helium was selected as sweep and calibration gas. Experiment 12, recalibration, showed that when calibration gas was passed through the tritium monitoring system, the approach to steady state was slower than predicted by laboratory tests. The slow approach to steady state was also observed in experiment 15 and was attributed to the establishment of a new equilibrium on the molecular-sieve trap located on the calibration gas header. A similar response had been observed with high-purity helium as a sweep gas.

When the hydrogen-bearing calibration gas had reached steady state, a molecular-sieve sample gave good agreement with the electrometer response and the certified tritium concentration quoted by Matheson. A flow dependence check in this experiment and in experiment 15 demonstrated that the ionization chamber response was not flow dependent when hydrogen was present as a carrier. So the calibration check indicated that the calibration was valid and that the tritium monitoring system was operating satisfactorily.

In experiment 13 the electrometer response obtained by passing 1% hydrogen in helium through the TT-TT flow mode was nearly identical to the response obtained by passing tritium in high-purity helium through the same flow mode. A comparison of these data is presented in Table 5, where the time scale for experiment 13 was shifted by 16 min to eliminate the peaking effect observed on all hydrogen carrier experiments. The results of these two experiments show that tritium was being retained from high-purity helium and released when 1% hydrogen was present in the

Table 5. Tritium injection and release response

	Exp. 11 (10-1-75)	Exp. 13 (7-2-76)
Sweep gas	HT in He (36.7 Bq/ml)	H <sub>2</sub> in He (1.01%)
Flow mode	TT-TT	TT-TT
Elapsed time (min)	(mV)	
0	21	20
40	50	39
100	72	57
160	85	76
220	95	94

gas stream. Since the response with time was similar, the same reversible reaction between tritium and some active species was suggested for both experiments. Although gamma spectrometry has shown that cesium was one of the principal gamma-emitting fission products on GB-10 effluent lines, this work has not established a relationship between cesium deposition and tritium retention.

Since steady state had not been attained in the TT-BT flow mode during experiment 13, the run was continued as experiment 14. A molecular-sieve sample collected during experiment 14 gave a tritium release rate of 3650 Bq/min. While continuing to monitor tritium in the TT-BT flow mode, the capsule was retracted to near zero power, which also lowered the charcoal-trap temperature from 300°C to 250°C. The electrometer response readily decreased to 26 mV and stabilized.

In experiment 15, an initial check demonstrated that the tritium (HT) concentration of the calibration gas had not changed during experiments 12 through 15. When calibration gas was valved through the TT-BT flow mode, tritium breakthrough was normal. A maximum was observed

which slowly decreased to the expected value for complete transmission (calibration gas plus capsule tritium). The calibration gas was valved out and helium containing 1% hydrogen was valved in the TT-BT flow mode during the remainder of the experiment. The steady-state response at full power obtained in the previous experiment was readily duplicated. Then tritium release as a function of capsule power and charcoal-trap temperature was investigated. The experiment was terminated by reproducing the tritium release at zero power observed in the previous experiment.

The HT/HTO ratio could not be determined in the presence of hydrogen carrier. Further, this program did not either establish the presence of HTO in the gas stream or ascertain that HTO could be passed through the capsule and/or stainless steel lines exposed to fission product deposition. Due to limitations imposed in this program, it was not possible to measure direct tritium release rates from the fuel in the presence of hydrogen carrier. Temperature-dependent tritium release was observed in experiments 14 and 15 when the fuel rod power and charcoal-trap temperature were reduced. Since  $H_2$  and HTO exchange readily, the observed phenomena were at least partially due to capsule tritium; detailed analysis of experiments 11 through 15 shows that more tritium was released than was injected.

In all hydrogen injection experiments, tritium release data were characterized by a maximum decreasing through an inflection point and approaching steady state. This indicates that two or more processes were responsible for the observed tritium release. It was likely that the original maximum was due to an exchange process with loosely bound

tritium, possibly a hydride. The secondary response could be an exchange reaction with chemically bound tritium such as an oxide. If tritium were present as a nonvolatile oxide, an exchange reaction would release HT to the gas stream. Such a compound could have a temperature-dependent first-order reaction rate. An extensive program would be necessary to adequately characterize the capsule chemistry and tritium transport mechanism for a GB-10 type of capsule.

## 7. CONCLUSIONS

The tritium monitoring system operated satisfactorily when hydrogen was present as a carrier in the sweep gas. The HTO converter retained tritium from high-purity helium but quantitatively transmitted HT without holdup when the gas stream contained 1% hydrogen. Reduction of HTO was not adequately tested in the laboratory; however, there was no reason to believe the HTO converter was not quantitatively converting HTO to HT in experiments using hydrogen carrier.

Measured tritium release from the GB-10 fuel rod using high-purity helium as the sweep gas was very low compared to the predicted production rate, but the relative release rates for the various flow modes are valid and are in general confirmed by the relative transmission rates obtained under similar conditions. The experimental program demonstrated that tritium in high-purity helium is retained in areas exposed to fission product deposition. When helium containing 1% hydrogen was passed through these areas, the tritium was released. Tritium was quantitatively transferred through the top of the fuel rod when hydrogen was present in the gas stream.

Due to flow restriction in the fuel region and the desire to preserve the existing fuel condition for postirradiation examination, permission was not obtained to pass hydrogen carrier through the fuel region. Therefore the tritium production rate was not determined.

Sufficient experimental data are not available to characterize the observed tritium release as a function of capsule power and charcoal-trap temperature. Fuel rod power-dependent and charcoal-trap temperature-dependent tritium release was observed when sweep gas containing hydrogen carrier was passed through the charcoal trap region of the capsule. The observed tritium release was probably due to interchange between hydrogen and tritium. Most likely, the release was predominately temperature dependent. Highly localized temperature gradients could have existed in areas exposed to fission product deposition. Significant diffusion does not appear likely since the response to power and temperature changes were rapid and sharp.

Substantial information was obtained on tritium transport and monitoring in the GB-10 system. This information provides a solid basis for design of future experiments in which tritium production, transport, release, and cladding permeation could be determined as a function of sweep gas composition, flow rate, fuel-rod power, and flow mode. However, experience with tritium monitoring in the GB-10 system strongly indicates that such experiments would require a large effort and that tritium monitoring should be the main objective. Under these conditions, compromise with other considerations could be minimized.



## 8. RECOMMENDATIONS

Experiments showed that adding hydrogen to the sweep gas eliminated the problem of tritium retention on the HTO converter. Since the hydrogen concentration of 1% was arbitrarily chosen for the laboratory experiments and worked well, it was also used for the capsule experiments. However, the optimum level of hydrogen concentration should be determined. Hydrogen concentration should be sufficient to eliminate tritium retention on the HTO converter, to provide sharp breakthrough from the charcoal traps, and to eliminate flow dependence and peaking effects characteristic of the ionization chamber in high-purity helium. Granular magnesium appears to be an acceptable reducing agent. However, further laboratory evaluation is strongly suggested. Also the mass of magnesium in the HTO converter was excessive and should be optimized for future experiments.

The origin of neon should be determined. If neon is produced by activation of fuel impurities, the problem can be minimized by more stringent fuel specifications.

Tritium monitoring for a capsule such as GB-10 should be initiated simultaneously with capsule irradiation. Provision should be included for controlled water and hydrogen content in the capsule sweep gas. Since measured tritium release rates were much less than predicted, the species responsible for tritium retention should be identified. Because capsule conditions cannot be duplicated in the laboratory, the best source of information on capsule fuel chemistry and fission product transport would be another irradiation capsule experiment similar to GB-10.

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## Appendix I

## SUMMARY OF TRITIUM MONITORING EXPERIMENTS IN HIGH-PURITY HELIUM

Run	Date	Fuel-rod power (kW/m)	Flow rates (ml/min STP)		Tritium monitoring system	Sweep gas	Flow mode	Hydrogen in trit. mon. sys.	HTO converter	Molecular-seive sample	Description of experiment
			Capsule	Capsule bypass							
1	6-12-75	NA <sup>a</sup>			200	He <sup>b</sup>	DT <sup>c</sup>	No <sup>d</sup>			Determine baseline in ORR
2a	6-17-75	NA			200	HT + He	DT	No		CG-1	In situ calibration
2b		NA			200	HT + He	DT	No		CG-2	Effect of neoprene gasket
3a	6-26-75	NA			200	HT + He	TM <sup>e</sup>	No	Out	CG-3	Evaluate tritium monitoring system
3b					200	HT + He	TM	No	In	No	Evaluate HTO converter - run aborted
4a	7-1-75	44.3	200		200	Capsule	TT-TT	No	Out	TT-TT-4	Determine tritium (HT) in capsule sweep gas
4b		44.3	200		200	Capsule	TT-TT	No	In	TE-TT-5	Determine tritium (HT + HTO) in capsule sweep gas
5	7-3-75	44.3	23	177	200	Capsule	BF-TT	No	In	BF-TT-6	Determine tritium (HT + HTO) in capsule sweep gas
6	7-23-75	NA			200	He	TM	No	In	He-7	Determine residual tritium in HTO converter
7a	7-31-75	NA			200	He	TM	No	Out	He-8	Determine tritium system background
7b		39.4	745		745	Capsule	BF-TT	No	Out	BF-TT-9	Determine tritium (HT) in capsule sweep gas
7c		39.4	745		745	Capsule	BF-TT	No	In	No	Observe electrometer response when HTO converter valved in
8a	9-3-75				200	HT + He	TM	No	Out	CG-10	Repeat calibration of experiment 2a
8b					200	HT + He	TM	No	In	No	Repeat calibration with HTO converter in-line
9a	9-11-75				506	He	TM	Yes	In	No	Observe tritium release from HTO converter with hydrogen

## Appendix I (continued)

Run	Date	Fuel-rod power (kW/m)	Flow rates (ml/min STP)		Tritium monitoring system	Sweep gas	Flow mode	Hydrogen in trit. mon. sys.	HTO converter	Molecular-seive sample	Description of experiment
			Capsule	Capsule bypass							
9b					506	HT + He	TM	Yes	In	CG-11	Determine tritium transmission through HTO converter with hydrogen
9c					506	HT + He	TM	Yes	In	No	Perform cleanup and repeat 9b
9d					500	HT + He	DT	Yes		No	Verify electrometer response observed in 9b and 9c
9e1					495	HT + He	DT	No		No	Repeat 9d in high-purity helium
9e2					545	HT + He	DT	Yes		No	Observe electrometer response during hydrogen addition
10a	9-17-75	39.4	745	155	1200	Capsule	BF-BT	Yes	Out	BF-BT-12	Determine tritium (HT) in capsule sweep gas
10b		39.4	745	155	1200	Capsule	BF-BT	Yes	In	BF-BT-13	Determine tritium (HT + HTO) in capsule sweep gas
11a	10-1-75				1200	HT + He	DT	Yes	In	No	Determine tritium concentration in cylinder 15417P
11b		39.4	900		1200	HT + He	TT-TT	Yes	In	No	Determine tritium transmission through TT-TT flow mode
11c		39.4	900		1200	HT + He	BT-BT <sup>f</sup>	Yes	In	No	Determine tritium transmission through stainless steel lines
11d		39.4	900		1200	HT + He	BT-TT	Yes	In	No	Determine tritium transmission through BT-TT flow mode
11e		39.4	900		1200	HT + He	TT-BT	Yes	In	No	Determine tritium transmission through TT-BT flow mode



## Appendix I (continued)

Run	Date	Fuel-rod power (kW/m)	Flow rates (ml/min STP)			Sweep gas	Flow mode	Hydrogen in trit. mon. sys.	HTO converter	Molecular-seive sample	Description of experiment
			Capsule	Capsule bypass	Tritium monitoring system						
12a	6-29-76	NA			1200	HT + H <sub>2</sub> + He	DT	Yes			Determine tritium concentration in cylinder 29333
12b		NA			300	HT + H <sub>2</sub> + He	DT	Yes			Determine electrometer response vs flow rate
12c		NA			1200	HT + H <sub>2</sub> + He	TM	Yes	In	CG-14	Determine tritium concentration in cylinder 29333
13a	7-2-76	48.6	1248		1248	H <sub>2</sub> + He	TT-TT	Yes	In-out-in	No	Observe tritium release in TT-TT flow mode with carrier
13b		48.6	1248		1248	H <sub>2</sub> + He	TT-BT	Yes	In-cut	No	Observe tritium release in TT-BT flow mode with carrier
14a	7-9-76	48.6	1200		1200	H <sub>2</sub> + He	TT-BT	Yes	In-out-in	TT-BT-15	Flow TT-BT to steady state and determine tritium (HT + HTO)
14b		0.0	1200		1200	H <sub>2</sub> + He	TT-BT	Yes	In	No	Observe tritium release at zero power
15a	7-14-76	NA			1200	HT + H <sub>2</sub> + He	DT	Yes			Determine tritium concentration in cylinder 29333
15b		NA			300	HT + H <sub>2</sub> + He	DT	Yes			Determine electrometer response vs flow rate
15c		48.6	1200		1200	HT + H <sub>2</sub> + He	TT-BT	Yes	In-out-in	No	Determine tritium transmission flowing TT-BT
15d		48.6	1200		1200	H <sub>2</sub> + He	TT-BT	Yes	In	No	Repeat steady-state response of experiment 14
15e		48.6/24.3/48.6	1200		1200	H <sub>2</sub> + He	TT-BT	Yes	In	No	Determine electrometer response vs capsule power
15f		0.0	1200		1200	H <sub>2</sub> + He	TT-BT	Yes	In	No	Determine electrometer response vs charcoal-trap temperature

<sup>a</sup>Not applicable.<sup>b</sup>Individual analyzed helium (H<sub>2</sub> ≤ 4 μg/ml STP).<sup>c</sup>Direct flow from calibration gas header to ionization chamber.<sup>d</sup>No hydrogen added - analysis of gases is (H<sub>2</sub> ≤ 4 μg/ml STP).<sup>e</sup>Tritium monitoring system only.<sup>f</sup>BT-BT fuel-rod bypass mode.

Appendix II  
SUMMARY OF MOLECULAR-SIEVE SAMPLES

Exp.	Date	Sample code	Flow mode	Sweep gas	Cylinder	Conc. (Bq/ml)	Percent of original calibration	Release rate (Bq/min)
2a	6-17-75	CG-1	DT <sup>a</sup>	HT + He	43372	1.35	73.6	
2b	6-17-75	CG-2	DT	HT + He	43372	1.39	75.5	
3a	6-26-75	CG-3	TM <sup>b</sup>	HT + He	43372	<i>c</i>		
4a	7-1-75	TT-TT-4	TT-TT	Capsule		0.00637		1.25
4b	7-1-75	TT-TT-5	TT-TT	Capsule		<i>d</i>		
5	7-3-75	BF-TT-6	BF-TT	Capsule		<i>d</i>		
6	7-23-75	He-7	TM	He		0.0153		
7a	7-31-75	He-8	TM	He		0.000548		
7b	7-31-75	BF-TT-9	BF-TT	Capsule		0.0512		36.7
8a	9-3-75	CG-10	TM	HT + He	43372	1.21	60.1	
9b	9-11-75	CG-11	TM	HT + He	13690T	5.97	40.1	
10a	9-17-75	BF-BT-12	BF-BT	Capsule		0.318		380
10b	9-17-75	BF-BT-13	BF-BT	Capsule		<i>e</i>		
12c	6-29-76	CG-14	TM	HT + H <sub>2</sub> + He	29333	39.9	101	
14a	7-9-76	TT-BT-15	TT-BT	Capsule + H <sub>2</sub>		3.04		3650

<sup>a</sup>Direct flow from calibration gas header to ionization chamber.

<sup>b</sup>Tritium monitoring system only.

<sup>c</sup>Value high due to temporary loss of charcoal-trap temperature control.

<sup>d</sup>Results invalid due to tritium release and retention on HTO converter.

<sup>e</sup>Results questionable due to operational error.

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