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Storage And Assay Of Tritium In STAR

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STORAGE AND ASSAY OF TRITIUM IN STAR

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The Safety and Tritium Applied Research (STAR) facility at the Idaho National Engineering and Environmental Laboratory (INEEL) is currently being commissioned to investigate tritium-related safety questions for fusion and other technologies. The tritium inventory for the STAR facility will be maintained below 1.5 g to avoid the need for STAR to be classified as a Category 3 nuclear facility. A key capability in successful operation of the STAR facility is the ability to receive, inventory, and dispense tritium to the various experiments underway there. The system central to that function is the Tritium Storage and Assay System (SAS).

The SAS has four major functions: (1) receiving and holding tritium, (2) assaying, (3) dispensing, and (4) purifying hydrogen isotopes from non-hydrogen species.

This paper describes the design and operation of the STAR SAS and the procedures used for tritium accountancy in the STAR facility.

I. STORAGE AND ASSAY SYSTEM DESCRIPTION

The SAS is the system in the STAR facility in which tritium handling and accountancy centers. It performs four major functions essential to tritium activity in STAR.

1. Receiving and holding tritium from shipping containers brought into the STAR facility.
2. Assaying the amount of tritium in the SAS using pressure-volume-temperature (PVT) techniques.
3. Dispensing tritium to secondary beds or containers used for transferring it to the experimental systems in the STAR facility[a].
4. Purifying hydrogen isotopes from non-hydrogen species.

To that may be added a fifth, optional function: isotopic separation of hydrogen isotopes using bed-to-bed transfer techniques.

I.A. System Configuration

The SAS is installed in a glovebox in the STAR facility, as shown in Fig. 1. Fig. 2 shows a schematic of the SAS configuration. The basic elements of the system include a vacuum manifold equipped with electro-pneumatic and manual isolation valves, a pair of uranium storage beds, two calibrated volumes, pressure and temperature sensors, a metal bellows pump, a dry vacuum pump system, a beta-scintillation detector, and a quadrupole mass spectrometer. The system is operated and controlled through a computerized data acquisition and control system using LabView [1] software and a battery of FieldPoint [2] modules that interface SAS control and sensor components to the computer. Fig. 3 is a schematic illustration of the SAS control and data system.

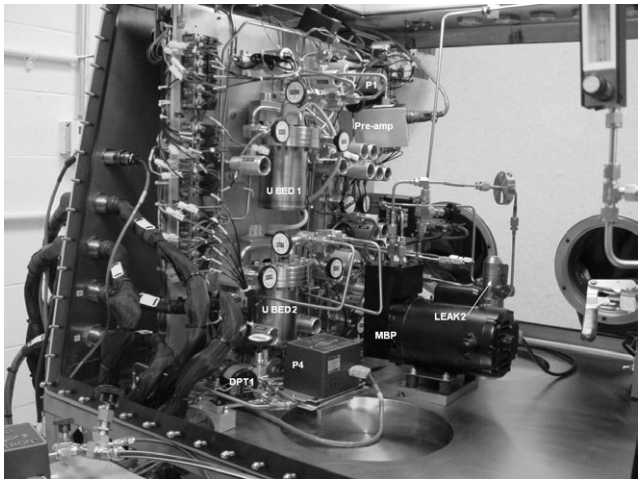

Fig. 1. SAS installed in its glovebox; windows have been removed for clarity.

In Fig. 2, valves represented with circles are electro-pneumatic valves. Those represented with boxes and handles are manual valves. There are two leak valves. LEAK1 admits manifold gas to the beta scintillation detector. LEAK 2 admits gas to a residual gas analyzer for total gas composition measurement. The different shades for the various loop sections identify connected segments and represent contiguous volumes used in the PVT assay of tritium in the system.

The uranium beds were custom fabricated by Tyne Engineering, Inc. of Oakton, Ontario, Canada. Each bed contains 47 g of depleted uranium (DU). Each bed is of double wall construction with the uranium in a copper-lined stainless steel inner chamber at whose ends are 2-μm

[a] No recovery of tritium from experiments is planned. Once used, tritium in experiments will go to the Tritium Cleanup System for disposition.

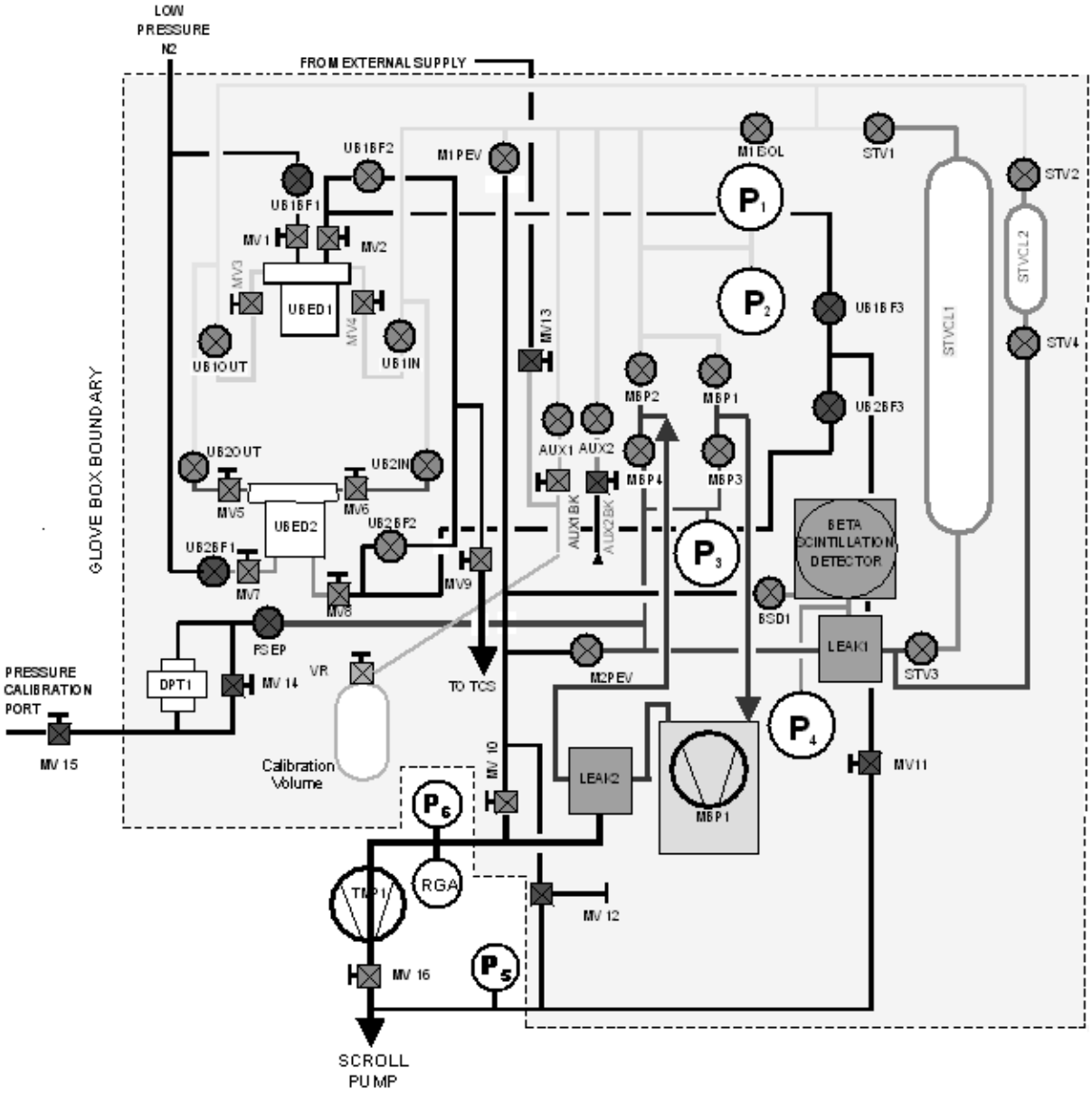

Fig. 2. STAR Tritium Storage and Assay System functional layout.

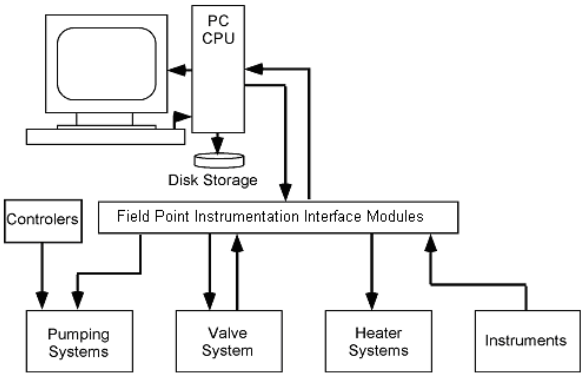

Fig. 3. Main elements of the SAS control and data system.

stainless steel frits. The uranium chamber is heated on the outside with a 375-W Watlow cold-leads heater (125 CH47A3X). Stainless steel tubing brings gas into the bottom of the chamber and carries it from the top. Gases are flowed upward through the bed to tend toward fluidizing the bed and ensuring good particle contact with the gases. Two thermocouples attached to the outside of the uranium chamber provide measurement of the chamber temperature. One of those on each bed is connected to an Omega N1462 temperature controller that provides a preset heating rate to a preset temperature and can hold it for a predetermined interval. The beds have demonstrated capacity of 0.3 moles of H_2 each.

I.B. SAS Instrumentation

Pressure measurements are made with capacitance manometers. Those designated P_1 and P_2 in Fig. 2 are MKS Baratron 615A capacitance manometers with full-scale ranges of 1,000 torr and 10 torr, respectively. P_3, an identical instrument to P_1, provides manifold pressure and can serve as a second reading for the pressures indicated by P_3. P_4 is an MKS Model 590 Baratron, used with the beta scintillation detector (BSD1 in Fig. 2) to determine tritium activity in the manifold. The beta scintillation detector, Model BSD1001-U1000-BC6, was furnished by Femto-Tech, Inc., Miamisburg, OH. Operating at pressures in the sub-torr range, the detector has a T_2 resolution precision of 386 Ci/m^3 (10^{-4} torr). For measurements, gas circulating through the standard volume will be leaked into the scintillation detector at low pressure.

A manometer calibration capability is built into the SAS. A pressure standard can be connected to the Pressure Calibration Port, which in turn is coupled to the SAS manifold through a high-precision differential pressure manometer, DPT1. This approach provides a means to correlate SAS manifold pressure sensor measurements to pressure measurements by the calibration standard sensor that is outside the glovebox. Gas pressure can be adjusted in the SAS manifold until the reading on DPT1 vanishes. Then, the actual pressure in the manifold is directly traceable to the pressure measured by the standard sensor connected to the calibration port, permitting calibration adjustments to be made for the manifold sensors. Externally applied pressure is measured with a high-precision MKS Model 590 Baratron with calibration traceable to the National Institute for Standards and Technology (NIST).

Dual Type-K thermocouples provide indications of temperatures within the uranium beds. Both are on the outside of the uranium chamber, one near the mid-plane of the container and the other near the upper end. Additional Type-K thermocouples are mounted on the standard volumes. One volume is nominally 3 L and the other is nominally 0.15 L.

A MetBel Model 610 metal bellows pump (MBP1) provides the motive force for gas circulation when the uranium beds are out of the loop, and basic evacuation capability is provided by a Pfeiffer TMU 071P turbo-molecular pump. That pump is backed by an Edwards Model ESDP 12 scroll pump, which can also be connected to the SAS manifolds independently. These serve to evacuate the manifolds and other system components and transport their contents to the Tritium Cleanup System.

I.C. SAS Control and Data System

The computerized data collection and control system operates in one of 35 predetermined valve configurations or in full manual valve control mode. Preset configurations are established for each of the standard operations performed with the SAS and are executed by selecting the desired activity in the SAS control system and clicking an "execute valving" button on the screen. Typical preset valve configurations include: load UBED1 from AUX1, Assay UBED1 using STV1, etc. This feature enables accurate and easy valve configuration control, while maintaining the integrity of the operation. Software and hardware inter-locks prevent heating the uranium beds in configurations where pressure rises could damage the beds or ducts and in the manual valve control mode. Screen displays provide the electronic equivalent of strip chart records for either the suite of temperatures, the various pressures, or any arbitrarily selected combination of any of the 27 state variables recorded. A digital record is kept of those variables as well as the open/closed status of each of the pneumatically operated valves at arbitrarily selected time intervals longer than 3 seconds.

II. OPERATIONS

Tritium is handled primarily as elemental gas (T_2, HT) for research activities in STAR. Typically, it will be received in DOT approved containers either as a gas or on a metal storage bed. Upon receipt, the shipping vessel is transferred into the SAS glovebox for direct connection to an AUX port or it is connected to an external port for unloading of tritium gas to the SAS. After assay certification measurements, the tritium is stored on a DU bed. Similarly, dispensing of tritium to various experiments entails unloading tritium from a storage bed, assaying the tritium quantity in the SAS manifold and associated standard reservoir, and transferring the tritium to an experiment storage vessel connected to an AUX port. Gaseous effluent from the SAS is routed to the Tritium Cleanup System.[3]

Our plans have implemented the SAS in four principal stages of operations.

1. System calibration–Measuring the volumes of each manifold segment and component and verifying or calibrating the performance of all pressure transducers, thermocouples, and the beta scintillation detector.
2. System conditioning–Performing system bake-out and evacuation followed by uranium bed conditioning by cycling hydrogen on and off the beds.
3. System functional performance–The operational phase where tritium and other hydrogen isotopes will be stored, measured, and dispensed.
4. System cleanup–Removing unwanted tritium or other contaminants from the system during periodic maintenance or at end of life.

The first two of these have been completed, and the remaining ones are service phases.

II.A. SYSTEM CALIBRATION

SAS manometers were calibrated at several pressures using the approach described in the previous section. For manometers P1 and P3, with one atmosphere full range, calibration was performed at 11 pressures between vacuum and 1 atmosphere. Measured pressures were compared with the pressure indicated by the calibration manometer. Agreement between the manometer readings and the calibration manometer averaged 0.04% over the range of measurements for both manometers with 1-σ standard deviations of 0.12% for P1 and 0.14% for P3. The calibration manometer agreed with site atmospheric pressure, as measured by the National Oceanographic and Atmospheric Administration (NOAA), within 0.025%.

For manometers P2 and P4, where the full range is only 10 torr, calibration was performed using the same technique but with only 7 points over the pressure range. Agreement with the calibration manometer averaged 0.68% with standard deviation of 0.66% for manometer P2, and 0.24% with standard deviation 0.77% for P4.

All segments of the SAS manifold were calibrated for volume by filling calibration volume attached at AUX1 to a known pressure and then expanding the gas into successively larger regions of the system at constant temperature. The calibration volume was known to a 1-σ precision of 0.1 mL. At least five repetitions of the expansion tests were conducted to give a measure of the statistical certainty of the measurements. Combining the uncertainties in the calibration volume and in the pressure measurements, the manifold segment volume uncertainties were less than 0.9%.

Thermocouple calibration is indirect. Evaluation is made by comparing an individual thermocouple reading with others reading essentially the same temperature. Experience has shown this provides acceptable accuracy. Using this technique, our assessment is that temperature readings for the SAS will be accurate to within 0.5 K or 0.17% at ambient temperature.

The beta scintillation detector was calibrated electronically at the manufacturer's plant with a 1-σ uncertainty of ±1% over equivalent T_2 partial pressures from 1.3 Pa to 390 Pa (0.01 to 3 torr) for both pure T_2 and 10% N_2 impurity. This instrument will be recalibrated annually using a standard tritium gas mixture.

Based on the-uncertainties for pressure measurements, manifold segment volumes and the beta scintillation detector, we expect that the 1-σ uncertainty for the number of moles of tritium in the SAS standard volumes will be no more than 1.4%. The smallest quantity That could be measured is 17 mCi, based on 0.5 L (small standard volume plus manifolds) at 1.3 Pa and 298°K. The largest measurable quantity is set by the volume of the combined standard volumes (~4.2 L) and the desire to keep pressure in the system under local atmospheric pressure. If pure T_2, that would be 8,000 Ci or half of the STAR allowed inventory.

II.B. SYSTEM PERFORMANCE

The SAS has been operationally tested in all of its major functions short of actually introducing tritium. Of particular interest are performance tests in storage and retrieval of H_2 from the uranium beds. One such test is described in the following paragraphs.

U-Bed 1 was annealed at 450°C for one hour under vacuum of 10^{-6} torr to remove residual hydrogen. Then 5.127E-03 mole of research grade (99.999% pure) H_2 was introduced to a calibration volume (at AUX1 in Fig. 2). When that volume was connected to UBed 1 by opening the isolation valve between them, the H_2 pressure in the manifold and calibration volume vanished in less than one minimum data cycle time of 3 seconds. The partially filled DU bed was subsequently connected to standard volume STV2 through manifolds that included capacitance manometers P1, P2, P3, and P4 and the metal bellows pump. It was then heated to release the hydrogen to the SAS manifold and volume STV2. Pressure and temperature measurements were made during the DU bed unloading. The results are shown in Fig. 4.

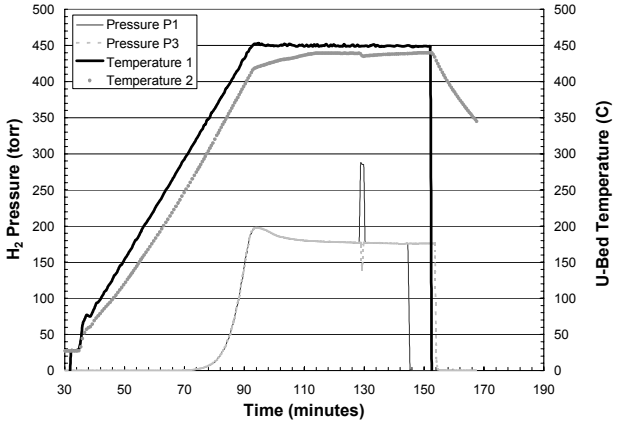

Fig. 4. Results of heating to recover H_2 from U-Bed 1.

Several points are worth noting in interpreting these results. First, two thermocouples are on each uranium bed. One, designated Temperature 1in Fig. 4, serves as the control response function to the temperature controller, and the second is at a different location on the bed. In this case, the Temperature 1 thermocouple is near the heater element, and the Temperature 2 thermocouple is located near the mechanical support for the inner chamber containing the DU. Because these thermocouples are at a finite distance from the DU, it is possible for some regions in the DU Bed container to get hotter than indicated by the thermocouple data. Temperature 2 obviously lags Temperature 1 in time, but they come closer over a period of many minutes as the bed thermally equilibrates. After approximately 154 minutes, the DU

bed heater controller was turned off, resulting in termination of the signal for Temperature 1.

The observed H_2 pressure rises to a peak of ~200 torr and then declines to an asymptotic pressure of ~176 torr. Full hydrogen recovery from the bed would correspond to a measured pressure of 208.3 torr. The finite equilibrium pressure of H_2 over UH_x for low x [4] implies full return should not be expected from a previously empty bed except at vacuum equal to that at which the bed was emptied. The asymptotic pressure of 176 torr in Fig. 4 suggests that as much as 15% returned to or remained on the bed after 94.7% was initially driven off. The initially high return is a consequence of the uranium being temporarily hotter than the measurement points on the housing during the heating transient. The pressure spike and corresponding drop in Temperature 2 at 130 minutes was the result of starting the metal bellows circulation pump momentarily to examine its effect on loop pressure.

These results highlight the need for a thorough characterization of the uranium bed performance and properties when determining their total tritium contents.

III. CONCLUSION

We have developed and tested a system for storing and assaying tritium. Initial testing was done with hydrogen, and testing with tritium is planned to calibrate the beta-scintillation detector in-situ and to demonstrate system performance with tritium. The SAS is meeting its design expectations and should be fully capable of performing the storage and assay function for the STAR facility.

ACKNOWLEDGEMENT

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