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TRITIUM IN-BED ACCOUNTABILITY FOR A PASSIVELY COOLED, ELECTRICALLY HEATED HYDRIDE (PACE) BED

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A Passively Cooled, Electrically heated hydride (PACE) Bed has been deployed into tritium service in the Savannah River Site (SRS) Tritium Facilities. The bed design, absorption and desorption performance, and cold (non-radioactive) in-bed accountability (IBA) results have been reported previously. Six PACE Beds were fitted with instrumentation to perform the steady-state, flowing gas calorimetric inventory method. An IBA inventory calibration curve, flowing gas temperature rise (ΔT) versus simulated or actual tritium loading, was generated for each bed. Results for non-radioactive ("cold") tests using the internal electric heaters and tritium calibration results are presented.

Changes in vacuum jacket pressure significantly impact measured IBA ΔT values. Higher jacket pressures produce lower IBA ΔT values which underestimate bed tritium inventories. The exhaust pressure of the IBA gas flow through the bed's U-tube has little influence on measured IBA ΔT values, but larger gas flows reduce the time to reach steady-state conditions and produce smaller tritium measurement uncertainties.

I. INTRODUCTION

The Savannah River Site (SRS) Tritium Facilities use a variety of metal hydride storage beds for process gas absorption, storage, and desorption. The 1st generation (Gen1) process storage beds contain up to 12.6 kg of $\text{LaNi}_{4.25}\text{Al}_{0.75}$ ("LANA0.75") metal hydride in a 7.62 cm (3 inch) pipe process vessel surrounded by a 10.2 cm (4 inch) pipe jacket. The beds desorb and absorb hydrogen isotopes by thermal-swing supplied by large flows of hot and cold nitrogen through an insulated jacket.¹ The in-bed accountability (IBA) technique has been developed and deployed for tritium accountability (inventory) measurements on these Gen1 storage beds without removal of tritium from the bed^{2,3,4} for over 15 years.

A 2nd generation (Gen2) metal hydride bed has been deployed into tritium service which does not use hot and cold nitrogen to supply the thermal swing for gas desorption and absorption.⁵ This Gen2 bed, developed as

a Passively-Cooled, Electrically heated hydride (PACE) Bed, utilizes electric heaters for desorption and much lower forced atmosphere cooling flow rates through the bed's jacket than the Gen1 beds for gas absorption. These beds are sometimes referred to as Forced-Atmosphere (glove box nitrogen) Cooled, Electrically heated (FACE) Beds or PACE Beds operating in FACE mode. Gen2 beds contain the same mass of LANA0.75 as Gen1 beds.

The Gen2 beds were put into tritium service in 2004. Electrical (cold) IBA calibrations were performed on the six Gen2 beds and the inventory results were presented before the results of tritium calibration data were available.⁶ The purpose of this paper is to present and compare IBA results obtained for the Gen2 storage beds using simulated and actual tritium. The impact of jacket pressure on IBA results will also be discussed.

II. BACKGROUND

The Gen2 bed design included the need to measure the tritium within the bed. In an attempt to reduce decay heat losses through the jacket of the Gen1 design, the Gen2 bed jacket is evacuated to supply thermal insulation and the IBA gas flow directed through a "U-tube" internal to the bed - a concept similar to the internal coil of a ZrCo storage bed.⁷ Higher accuracy tritium measurement designs have been proposed for ITER metal hydride storage beds,⁸ but simpler bed designs were desired for SRS process beds.

Cold test results showed smaller IBA inventory measurement errors were obtained for the IBA gas flow through the U-tube compared to IBA jacket flow.⁶ Unless stated otherwise, reference to all flowing gas temperature rises are for flows through the U-tube and denoted as ΔT .

III. EXPERIMENTAL

The Gen2 production beds were similar in design to the previously described prototype bed PB1,⁵ but had the large ConFlat[®] flanges replaced with 2.54 cm (one inch)

tubing and VCR[®] fittings. The placement of the thermocouples (TCs) for IBA gas temperature measurements were the same for the Gen2 process beds as the prototype bed: the U-tube TC tips were inserted 11.4 cm (4.5 inches) into each leg of the U-tube to correspond to the position of the bed's 1st divider plate.

For cold testing, tritium decay heat was simulated by adjusting the voltage to the bed's two 400 Watt electric heaters and the power was measured by a power meter connected to a data acquisition system. The bed was initially evacuated and back-filled with a nominal 101 kPa (760 torr) of helium. An IBA gas flow of 45 SLPM (standard conditions of 101 kPa and 0°C) nitrogen was used. The jacket of the bed was evacuated using a Varian Triscroll pump to less than 133 Pa (1 torr) and monitored using a pressure transducer. When the pressure exceeded a nominal 133 Pa, the valve between the bed's jacket and the vacuum pump header was opened to evacuate the jacket and then closed.

Two, six-point cold test IBA data sets were collected to generate IBA calibration curves. A zero-point ΔT test, without heater power or tritium, was run to determine the temperature off-set of the installed system and components. A single zero-point run was used for the two cold calibration data sets. A separate zero-point run was performed for the tritium IBA calibration data.

For the 1st cold test IBA calibration curve (Run A), data were collected at nominal heater power levels corresponding to 0, 20%, 40%, 60%, 80%, and 100% of full bed capacity: a full bed capacity is defined as 0.6 T/M where T/M is the tritium-to-metal atom ratio. For the 2nd cold test IBA calibration curve (Run B), data were collected at nominal heater power levels corresponding to 15%, 35%, 55%, 75%, and 95% of full bed capacity.

For tritium testing, the bed was evacuated and tritium loaded from a calibrated volume onto the bed to "100%" full bed capacity for the first, non-zero, calibration run. The same 45 SLPM IBA gas flow was used, but the valve between the bed's vacuum jacket and the vacuum pump header was left open during ΔT measurements. At the end of each IBA run, tritium was removed to reach each of the nominal 80%, 60%, 40%, and 20% bed loadings for construction of the IBA calibration curve.

Linear and quadratic regression models, with and without zero intercept, were fit to the ΔT versus T/M data to obtain regression coefficients and other statistics parameters. The previously described method⁴ was used to select the "best" regression model at the 95% confidence level and calculate the inverse regression standard deviation, σ_{inv} . The uncertainty of the IBA technique was calculated as the product of the statistical

student's t-test value and σ_{inv} : $t^* \sigma_{inv}$. $t^* \sigma_{inv}$ varies as a function of T/M and the values at 0.6 T/M, the largest values, will be reported.

IV. RESULTS

IBA calibration data for the two cold data sets and the tritium data set are shown in Figure 1 for the "100" bed. After cold data collection, a review of the power analyzer digital-to-analog calibration data found an off-set in the logged data. The raw data shown in Figure 1 were off-set corrected to produce the cold data for Run A and Run B shown in Figure 1. Figure 2 shows the tritium IBA calibration data for all six beds.

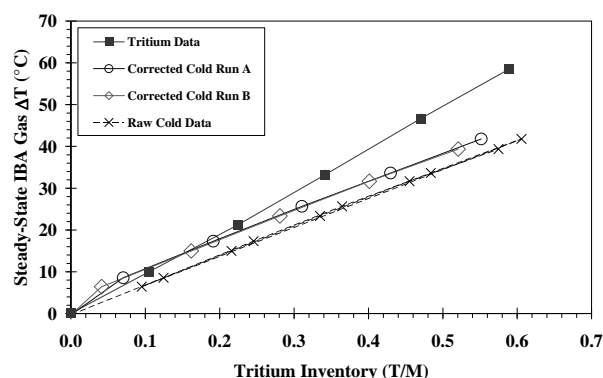


Fig. 1. IBA Calibration Data for Bed "100".

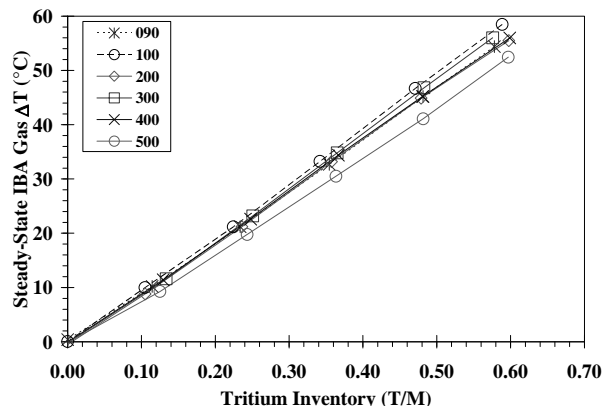


Fig. 2. Tritium IBA Calibration Curves for All Beds.

Figure 3 shows the IBA inverse regression results, expressed as $t^* \sigma_{inv}$, as a function of IBA gas flow rate for the PB1 prototype bed⁶ and the six process PACE Beds for both cold test and tritium data. Figure 3 off-sets the plotted $t^* \sigma_{inv}$ values for the process bed around the 45 SLPM flow rate to aid in differentiating the results for the

three runs. The results at 70 SLPM are for three different PACE beds and will be discussed later.

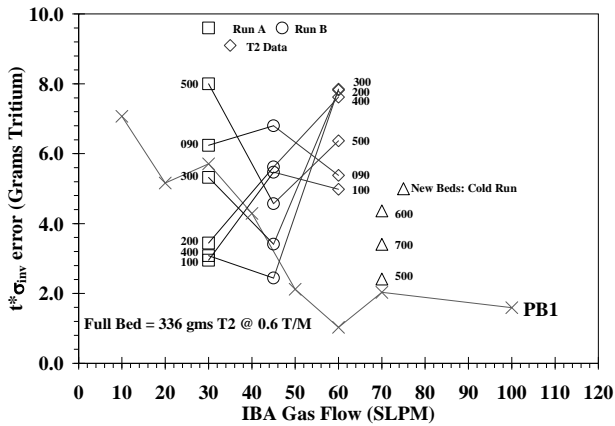


Fig. 3. PACE Bed IBA Inventory Errors.

V. DISCUSSION

Figure 1 shows a shift in cold test results due to the off-set power meter values. It was not clear when the instrument drifted so the post-test, as-found calibration data were used to adjust the raw data. Due to the facility start-up schedule, the cold runs were not repeated since tritium calibrations had been scheduled for the beds.

The impact of vacuum jacket pressure on the steady-state IBA ΔT was not appreciated until initial tritium IBA calibrations were in progress during start-up testing. The IBA gas flows had been established and a seemingly long time, over 2 days, did not produce steady-state results. While examining the slow variations in ΔT , bed temperature, and bed pressure versus time, a very sudden increase in bed temperature was observed and the ΔT values departed from their previous trend. Speculation that the temperature increase was due to an inadvertent addition of tritium to the bed or a change in glove box atmosphere flow around the bed was unfounded.

The sudden change in bed temperature was traced to the opening of the valve between the bed's vacuum jacket and the vacuum pump header. To eliminate this type of rapid change in bed temperature during IBA runs, the vacuum jacket valves for all the beds were left open to the vacuum pump header to maintain a constant jacket pressure. This procedure was used for all the tritium IBA calibrations of the beds. It is not clear if bed jacket pressure variations alone explain the difference between the cold and tritium results shown in Figure 1.

Lower PACE bed jacket pressures during IBA measurements increased the bed temperature and the measured ΔT values. It took longer for the beds to achieve steady-state conditions at higher temperatures since the criteria for steady-state, variations of a few tenths of degrees Celsius, did not change. The time needed to achieve steady-state conditions was reduced for three PACE beds in another facility when using a 70 SLPM IBA gas flow during cold IBA calibrations. The beds achieved steady-state faster than those using a 45 SLPM flow and produced inventory measurement uncertainties smaller than those using the slower flow rate as shown in Figure 3.

During one tritium inventory period, a large facility discrepancy in tritium inventory was discovered. Several beds had their gas inventories desorbed to tanks and the residual tritium inventory on the beds measured using the IBA technique. This process allowed closure of the tritium inventory discrepancy.

Post-inventory analysis of the PACE bed IBA system showed jacket pressures higher than during bed calibrations. Figure 4 shows the pressure history of the jacket pressure for three beds and the system vacuum pump with the higher pressure values recorded during the inventory discrepancy. The system vacuum pump was replaced and the system pressure returned to previous vacuum levels as shown in Figure 4.

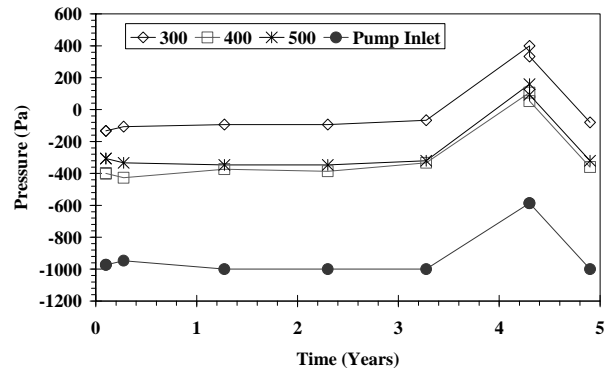


Fig. 4. Vacuum Jacket System Pressures During IBA

The following tests were performed on six beds to determine the impact of vacuum jacket pressure and U-tube flowing gas pressure on the IBA ΔT measurement. An initial loading of tritium was placed onto one of the PACE beds. IBA ΔT measurements were made under two sets of conditions. The baseline conditions were with the bed's jacket valve open to the vacuum pump header and the exhaust pressure of the IBA gas flow at 160kPa (1200 torr) – the exhaust header pressure when IBA was

performed on all beds simultaneously. A second IBA test was performed with the jacket pressure changed to be between approximately 270 Pa (2 torr) to 800 Pa (6 torr). For the 400 bed, a third IBA test was performed with the IBA gas flow exhaust pressure reduced to 107 kPa (800 torr) with the bed jacket open to the pump header.

The results for the 400 bed are shown graphically in Figure 5. The baseline test produced a ΔT value of 49.5°C which corresponds to be a bed loading of 0.530 T/M or 296 grams of tritium. With the jacket pressure increased to between 270 and 800 Pa, the measured ΔT value decreased to 40.2°C which corresponds to be a bed loading of 0.430 T/M or 240 grams of tritium: a difference of 56 grams of tritium!

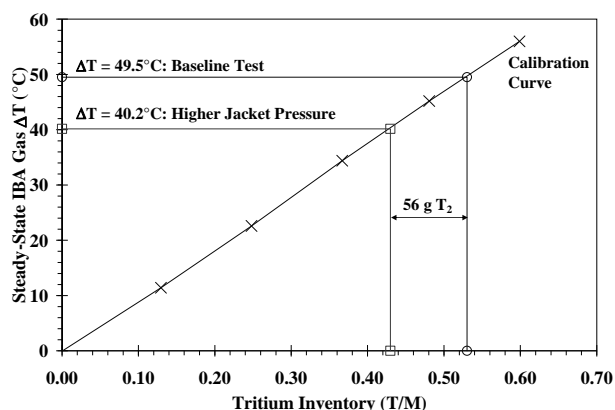


Fig. 5. Impact of Jacket Pressure on 400 Bed IBA

The tests showed changes in the U-tube pressure did not significantly impact the measured IBA ΔT : 0.03°C. However, the relatively small increase in jacket pressure significantly decreased the measured IBA ΔT and thus tritium inventory that would be calculated on the bed. The reduction in IBA ΔT at higher jacket pressures was attributed to a higher heat flux from the IBA gas to the vacuum jacket, which dissipates heat to the glove box, and thus produced a lower ΔT measurement.

VI. CONCLUSIONS

Slight variations in the positive pressure of the IBA gas flow through the U-tube did not produce a significant change on the measured tritium in PACE beds. Variations in PACE bed vacuum jacket pressures produce significant changes in bed temperatures, and thus IBA measurements. Higher jacket pressures than those used during bed IBA calibrations will underestimate the tritium

inventory on a bed due to a lower measured ΔT due to increased heat transfer from the bed.

Not understanding the impact of seemingly small changes in vacuum jacket pressure on IBA did not allow direct comparison of cold/simulated and tritium IBA calibrations. Increasing the IBA gas flow rate through the PACE Bed U-tube allowed faster equilibration times and reduced tritium measurement uncertainties.

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