Tritium Loading of Pinellas U-Bed No. 874

by

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Aiken, South Carolina 29808

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Rev 1.

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Date:

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Reviewing Official J.S. Holder, MTF Mgr
(Name and Title)
Date: 5-5-99

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Kirk L. Shanahan

Abstract

Pinellas U-Bed No. 874 was loaded with approximately 955 Ci of 98% purity tritium on the ETM manifold in the MTF in Building 232-H.

Introduction

The DOE-Richland office has requested WSRC to supply PNNL with a Pinellas U-bed loaded with tritium for permeation experiments. It is desired to have less than 1000 Ci tritium in the bed to allow shipping without excessive packaging requirements.

The Chemical and Hydrogen Technology Section of the Savannah River Technology Center maintains its R&D tritium handling capability in Building 232-H, known as the MTF (Materials Test Facility). In the MTF is a tritium handling manifold known as the Experimental Tritium Manifold (ETM). The Pinellas U-bed was loaded on the ETM on January 20, 1999 with approximately 955 curies of tritium.

Procedures

The Pinellas U-bed used was designated as number 874 by an impressed tag hanging from the valve handle and by a scribed "874" on the valve handle itself. Pinellas had previously characterized this bed as a deuterium-only bed. It had been prepared on the ETM for another use at PNNL by exchange with deuterium gas and vacuum bakeout to fully unload it. It had in fact already been shipped to PNNL with an Ar blanket inside and was returned to the MTF for T2 loading. The bed consists of a sample cell of ~1" diameter and ~6" length welded to 6" piece of a 1/2" tubing, in turn welded into a metal bellows valve. The inlet connection was a 2-3/4" Conflat-type flange. The bed was mounted on the ETM by using an adapter flange that converted from the Conflat flange to a 1/4" VCR connection. The sample cell holds a ~50g pellet of depleted U.

The bed was mounted to the ETM using a second Nupro valve for double isolation and a 'tee' connector. The other leg of the 'tee' had a gas sample bottle attached to it. A second gas sample bottle was attached to another ETM port. Manifold and interconnection volumes were determined by Ar expansion from a calibrated section of the ETM. The interior of the bed was evacuated to less than 250 millitorr before loading. Prior volume determinations on the bed gave an approximate internal volume of 50 milliliters.

The ETM was pressurized to an appropriate level with the T2 that was to be loaded on the U-bed. Small samples of the gas were taken by bleeding into the two gas sample bombs. The pressures were not equilibrated since this would have overloaded the bottles. After the U-bed loading was completed, these bottles were removed and sent off for mass spectroscopic analysis (see attached results).

After gas samples were taken, the U-bed was exposed to the tritium. Loading was quick and easy, indicating that the shipment under Ar back and forth from PNNL had not deactivated the bed. The bed was allowed to equilibrate for 2 hours, at which point it was sealed off and the residual gas evacuated. This residual gas remains on the U-bed and is the only blanket present. It is expected to be approximately 100% ^3He . The U-bed was dismounted and the Conflat adapter was replaced with a blank Conflat flange. Thus the space between the bellows seal and the flange is filled with air.

It should be noted that the part of the bed volume between the Conflat flange and the bellows valve seat showed residual pressure of about 9 torr in the ETM during initial evacuation procedures. This was eventually pumped away, which indicates that it was likely adsorbed water. This water probably contains tritium so appropriate caution should be taken when mounting the U-bed at other locations, as tritiated water may well have formed again from the interaction with atmospheric oxygen introduced during removal of the bed from the ETM. The likely culprit for this is the large bellows assembly of the U-bed valve.

Computations

Volume determination results are shown in Table 1. There was some indication that the volume determined for the space between the U-bed's valve and the added Nupro valve was pressure dependent, producing a slightly higher standard deviation. It may be that the bellows was compressing with pressure. As this volume was not critical to the computation no further determination was done.

Table 1. Volume Calibration Results (1 standard deviation shown).

A-manifold (initial charge volume)	92.45 +/- .07
Port to gas bottle (#1) outer valve ("tee")	13.84 +/- .02
Between #1's valves	1.42 +/- .02
Port to gas bottle (#2) outer valve	11.33 +/- .02
Between #2's valves	1.45 +/- .01
U-Bed between valve space	39.41 +/- .23

The final charge volume used to calculate the U-bed loading was $92.45 + 13.84 + 11.33 + 1.42 + 1.45 = 120.49$ cubic centimeters, as the innermost gas bottle valves were used to isolate the bottle from the charge volume. The final

pressure just before loading was 2589.1 torr as measured by a calibrated 10,000 torr MKS Baratron pressure sensor. Manifold temperature was measured at 21.6 °C with a type K thermocouple taped to the manifold. Analytical results on the gas bottles showed %T = 96.96%, %inerts=1.141%, and %³He=0.662%. The actual results sheets are attached. Assuming the inerts will not absorb on the bed, a residual pressure of 29.54 torr could be expected. If only ³He was left, a residual pressure of 17.1 torr would be expected based on the analytical results. The final pressure measured before closing off the U-bed was 14.3 torr at 21.4 °C.

Using the Ideal Gas Law to estimate the number of moles of gas initially present gives:

$$N = 2589.1 \text{ torr} * 120.49 \text{ cc} / (62.361 \text{ torr-cc/mmole/K} * 294.75 \text{ K}) = 16.97 \text{ mmoles}$$

Since the residual pressure is about that expected for the residual He, we will assume all T in the charge was loaded, thus mmoles T2 loaded = $16.97 * 96.96\%$ = 16.46 mmoles.

Assuming a molecular weight of 238 for depleted U, the mmoles of U present are approximately $50,000/238 = 210.1$ mmoles. Thus the T/U ratio of the bed is $2*16.46/210.1 = 0.16$, implying the bed is quite far from its theoretical maximum of 3 and the as-received condition of D/U=2.0. We routinely prepare U-beds here for T use by baking at 430°C. Higher temperatures are avoided to prevent T permeation through the stainless steel. However the low loading level may require higher temperatures to achieve appreciable pressure.

With 6.032 milligrams/mmole and 9619 Ci/g = 9.619 Ci/mg, the curie load on the bed can be calculated as $16.46 * 6.032 * 9.619 = 955.0$ Ci. Thus the desired goal of close to but not exceeding 1000 Ci has been achieved.

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~~SECRET-RE~~LAB RESULTS
(SRTC Copy)

DESCRIPTION : SRTC
LOG NUMBER : 409
LEAK NUMBER : N/A
BUILDING : 232-H
ANALYST : BYM
TANK NUMBER : MTF #3

MASS RANGE SCANNED: 2 - 44
SAMPLE TYPE : ROUTINE
TANK PRESSURE : 300 TORR
MASS SPEC ID : MASS SPEC 24
CALCULATION DATE : 4 FEB 1999 (12:10)

PEAK	MASS	PART.PRES. [microns]	MOLE PERCENT WITH WATERS	MOLE PERCENT WITHOUT WATERS
1	2 H2	.016	.026	.026
2	3 H ₂	.386	.642	.644
3	3 H D	.009	.015	.015
4	4 H T	1.710	2.840	2.850
5	4 D2	.003	.004	.004
6	5 D T	.530	.879	.882
7	6 T2	57.007	94.663	94.992
8	18 H2 O	0.000	0.000	0.000
9	19 H D O	0.000	0.000	0.000
10	20 H T O	.002	.003	0.000
11	20 D2 O	.002	.004	0.000
12	21 D T O	.192	.318	0.000
13	22 T2 O	.012	.021	0.000
14	24 C T4	.205	.341	.342
15	26 N2	.002	.003	.003
16	32 O2	0.000	0.000	0.000
17	40 Ar	.146	.242	.243
		60.221	100.000	100.000

EXCLUDING WATERS

TOTAL H :	1.459 %	2 :	.026 %
TOTAL D :	.453 %	3 :	.644 %
TOTAL T :	96.858 %	40 :	.243 %
H+D+T :	98.769 %	44 :	0.000 %
3He / T :	.0066	INERTS :	1.231 %
D / T :	.0047	KDT :	1.9624

ANALYZED BY BARBARA T. MUE, PAYROLL # 3884

THIS ANALYSIS IS A RESULT OF A SCAN OF THE FOLLOWING MASS TO CHARGE RATIOS: 2,3,4,5,6,14,15,16,18,19,20,21,22,24,28,32,36,40,44. IF ANY MASS TO CHARGE RATIO IS NOT REPORTED ON THIS FORM, ITS VALUE IS 0.00 %. ALL PEAKS IN THE 18-22 MASS REGION ARE REPORTED AS MOISTURE; THE POSSIBLE CONTAMINANTS IN 18-22 ARE MOISTURE, HYDROCARBONS, AND AMMONIA.

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~~SECRET-RO~~LAB RESULTS
(SRTC Copy)

DESCRIPTION : SRTC
 LOG NUMBER : 410
 LEAK NUMBER : NA
 BUILDING : 232-W
 ANALYST : PP
 TANK NUMBER : MTF 5N

MASS RANGE SCANNED: 2 - 44
 SAMPLE TYPE : ROUTINE
 TANK PRESSURE : 532 TORR
 MASS SPEC ID : MASS SPEC 24
 CALCULATION DATE : 4 FEB 1999 (13:05)

PEAK	MASS	PART. PRES. [microns]	MOLE PERCENT WITH WATERS	MOLE PERCENT WITHOUT WATERS
1	2 H2	.010	.025	.025
2	3 3He	.266	.681	.684
3	3 H D	.008	.020	.020
4	4 H T	.980	2.506	2.515
5	4 D2	.002	.006	.006
6	5 D T	.453	1.159	1.163
7	6 T2	37.111	94.878	95.221
8	18 H2 O	0.000	0.000	0.000
9	19 H D O	0.000	0.000	0.000
10	20 D2 O	.002	.005	0.000
11	21 D T O	.133	.339	0.000
12	22 T2 O	.006	.016	0.000
13	24 C T4	.142	.364	.365
14	28 N2	.001	.002	.002
15	32 O2	0.000	0.000	0.000
		39.115	100.000	100.000

EXCLUDING WATERS

TOTAL H :	1.292 %	2 :	.025 %
TOTAL D :	.597 %	3 :	.684 %
TOTAL T :	97.060 %	40 :	0.000 %
H+D+T :	98.949 %	44 :	0.000 %
3He / T :	.0070	INERTS :	1.051 %
D / T :	.0062	KDT :	2.4691

ANALYZED BY Pam Pearson, PAYROLL # 5868

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