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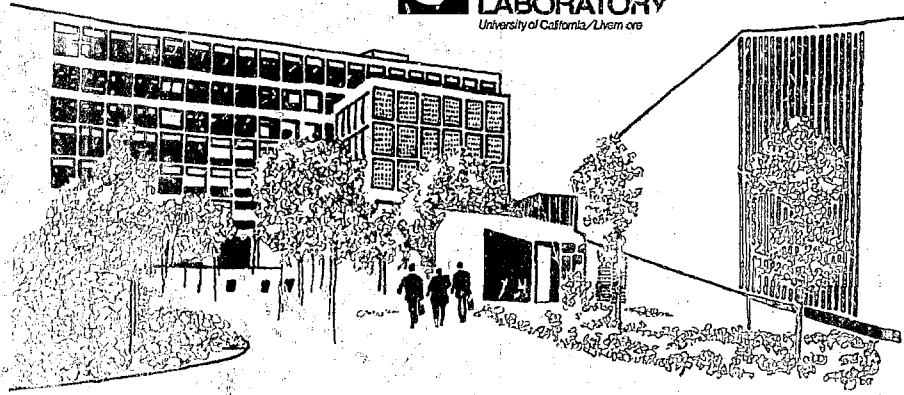
# REDUCTION OF WEIGHING ERRORS CAUSED BY TRITIUM DECAY HEATING

MASTER

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January 10, 1978

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# REDUCTION OF WEIGHING ERRORS CAUSED BY TRITIUM DECAY HEATING

## ABSTRACT

The deuterium-tritium source gas mixture for laser targets is formulated by weight. Experiments show that the maximum weighing error caused by tritium decay heating is 0.2% for a 104-cm<sup>3</sup> mix vessel. Air cooling the vessel reduces the weighing error by 90%.

## DETERMINING THE WEIGHING ERROR

In this study I determined the magnitude of the apparent weight loss when weighing heated pressure vessels, and I investigated a means of reducing the weight loss. A study of this type is necessarily empirical, since there are many variables and we can control only a few. Container shape and surface area influence both the temperature increase and the apparent weight loss. To show this, I experimented with two types of pressure vessels: one with a small length-to-diameter ratio and one with a large ratio. I used electricity rather than tritium as a heat source until the very end of the cooling studies. Electrical heating made it easy to change the effective tritium amount, although it caused a difference in the spatial temperature distribution.

During the experiments, I measured the temperature increase above ambient attained by each container at several simulated tritium loads. To determine the temperature drop during weighing, I measured the cooling rate. After analyzing the preliminary data, I measured the apparent weight of each container at several simulated loads.

### Equipment

The equipment included a Voland\* 25-kg balance with an A-500 electronic null indicator. Table I shows the balance performance measurements.

The first pressure vessel was a 316 stainless steel cylinder 229 mm long with an outside diameter (o.d.) of 102 mm and an inside diameter (i.d.) of 25 mm. The overall surface area of the cylinder, including attachments at both ends but excluding the mounting fixture, was approximately 0.0994 m<sup>2</sup>,

and the volume was 104 cm<sup>3</sup>. A 600- $\Omega$  resistance heater was introduced into the vessel by means of an O-ring-seated, threaded flange (see Fig. 1). This heater occupied 80% of the length of the vessel interior. A glassbeaded electrical feedthrough enabled the vessel to maintain a vacuum (Fig. 2). The vessel was evacuated and backfilled to 1 atm of helium-4.

The second pressure vessel was a 316 stainless steel coiled tube 9.4 m long with an o.d. of 14 mm, an i.d. of 7 mm, a surface area (excluding the mounting fixture) of 0.411 m<sup>2</sup>, and a volume of 400 cm<sup>3</sup>. Both ends had been cut off and replaced with an electrical feedthrough (Fig. 3). Heater resistance was 418  $\Omega$ .

Copper/copper constantan thermocouples were used to monitor the experiments. The contacts were parallel and were made intrinsic by means of spot-welding to the vessel wall. The reference thermocouple was intrinsic to a 10-kg block of stainless steel. A six-place digital voltmeter provided readout of the thermocouples. Two power sources provided 100 V of controlled direct current to the heater.

### Procedure

Vessels were heated electrically with a controlled direct current. Heater resistances (R) equaled 609  $\Omega$

Table I. Balance performance.<sup>a</sup>

Range, kg	Sensitivity, mg	Repeatability, mg
0	1.4	0.2
5	1.4	1.2
10	1.4	3.0
15	1.4	1.4
20	1.4	1.4

<sup>a</sup>Class S weights were used.

\*Reference to a company or product name does not imply approval or recommendation of the product by the University of California or the U.S. Department of Energy to the exclusion of others that may be suitable.

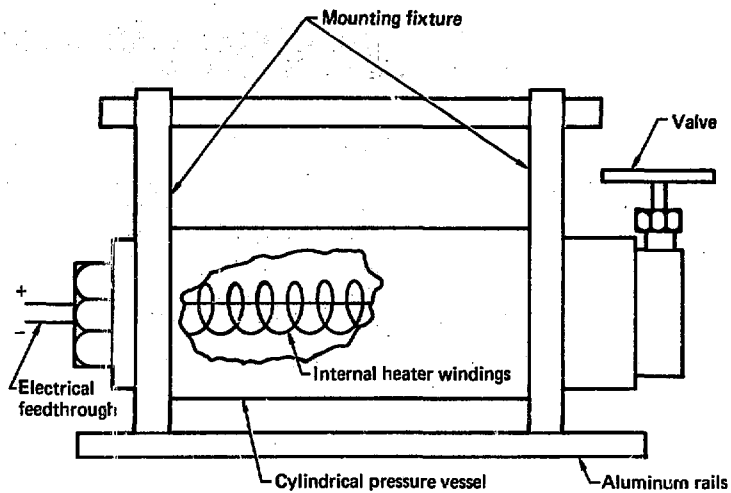


Fig. 1. 104-cm<sup>3</sup> cylindrical pressure vessel.

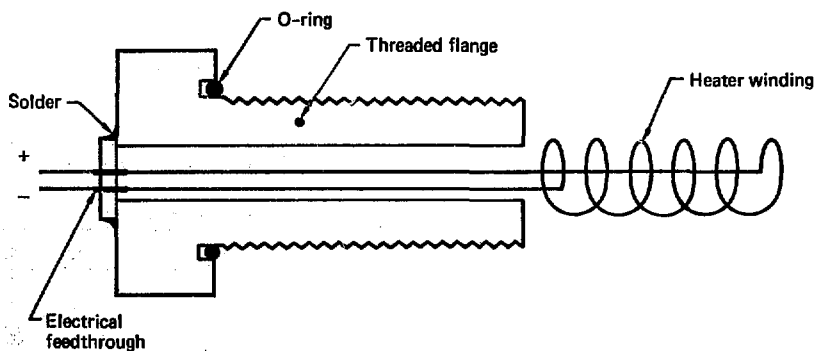


Fig. 2. Electrical heater for cylindrical pressure vessel.

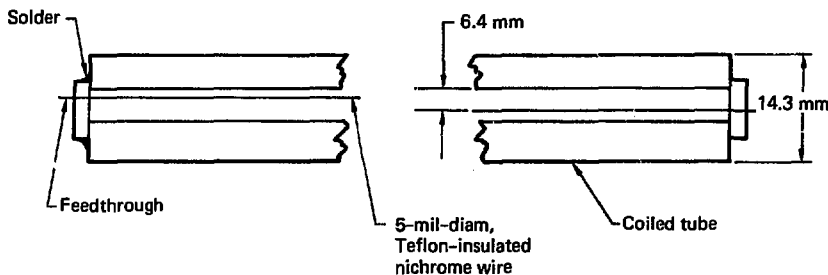


Fig. 3. 400-cm<sup>3</sup> coil pressure vessel.

for the cylinder and 418  $\Omega$  for the coil. The voltage used ( $E$ ) is given by

$$E = \sqrt{(gT_2)(0.324 \text{ W/g})(r)},$$

where

$$\begin{aligned} gT_2 &= \text{grams of tritium,} \\ W/g &= \text{watts per gram of tritium,} \\ r &= \text{resistance in ohms.} \end{aligned}$$

Temperature increments were obtained for the heat equivalent of 1.4, 2.0, 2.5, 3.5, and 5.0 mol of tritium-2. Cooling rates for 3 min showed no loss greater than 0.2 K. After removing the thermocouples, I heated the vessel at a preselected voltage equivalent to the desired tritium concentration. Equilibrium periods were 5 h for the cylinder and 2 1/2 h for the coil. Before weighing each vessel I kept it at a level temperature overnight to be certain that equilibrium had been established.

Working quickly, I disconnected the power leads and placed the vessel on the balance pan. A built-in spirit level on the pan showed when the vessel was positioned properly. Balance weights were not removed from these pans between weighings. After recording the rest point, I shifted the rider 10 mg to determine the balance sensitivity. Then I found and recorded a new rest point. Next I removed the vessel from the balance and reconnected the power leads to the vessel. It took less than 3 min to disconnect the leads, weigh the vessel, and reconnect the leads to the vessel. The vessel was reheated for at least 15 min between weighings. A minimum of ten succes-

sive weighings were made at each tritium concentration. Two tares were taken, one before and one after the series of tritium weighings.

## Conclusions

The results of my measurements are summarized in Figs. 4 and 5. In Fig. 4, the temperature above ambient is plotted with respect to the simulated tritium load. A straight line has been drawn through the set of points for each vessel. At a given heat input, the temperature increase in the coil vessel is about 1 K lower than in the cylindrical vessel. This difference is expected, since the coil vessel has over four times the surface area of the cylindrical vessel.

The temperature increments were measured while the vessels were on the table and some air was moving due to the room air conditioning. Different conditions of air flow would change the results. The results show the relative effects of loading on these vessels and indicate the magnitude of the temperature increase.

Apparent weight loss vs tritium load is plotted in Fig. 5. The temperature decrease during weighing was small, so the curves are not adjusted for this effect. The weight loss for the coil is almost twice that of the cylinder.

Based on these data, the weight loss correction is a maximum of 0.3% for the coil and a maximum of 0.2% for the cylinder. This error is so large that a correction must be used for accurate work. An alternative would be to force air-cool a tritium-loaded vessel before weighing to minimize the temperature above the ambient and thus the weight loss.

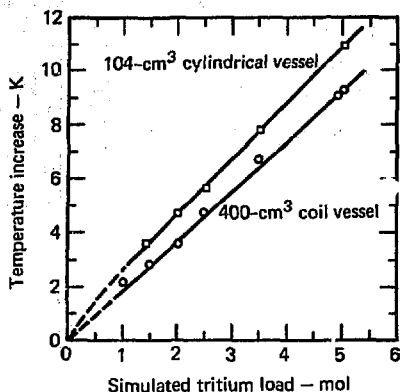


Fig. 4. Temperature above ambient as a function of simulated tritium load.

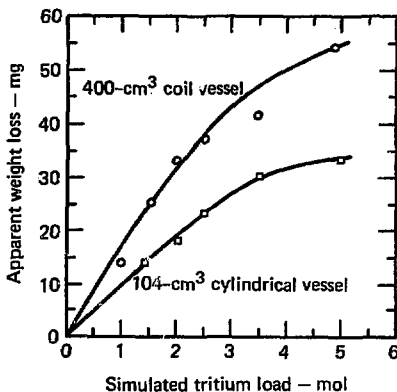


Fig. 5. Apparent weight loss as a function of simulated tritium load.

## REDUCING THE WEIGHING ERROR

In the first part of this paper I have shown that a vessel treated by tritium decay can have an apparent weight loss as high as 40 mg. Here I show that forced air-cooling a tritium-loaded vessel to near the ambient temperature in the balance room is a practical way to eliminate most of these weight losses and, equally important, to reduce weighing errors considerably. This method obviates a corrective factor. Forced room air cools less efficiently than a liquid heat-transfer medium, and, unlike chilled air, it cannot eliminate temperature increases entirely. These disadvantages are offset by its cleanliness and ease of application, however.

### Equipment

The air-cooling apparatus is a stainless steel tube 71 cm long with an o.d. of 20.635 cm and an i.d. of 20 cm. A Rotron vane fan is bolted to one end. The entire assembly is bolted in a horizontal position to a large table in our balance room. A Teflon mat lines the cooling chamber floor to prevent scuffing the aluminum support rails on the vessel whenever it is slid in and out. The electronic measuring equipment, power supplies, electrically heated vessels, and thermocouples are the same as were used during the weight loss studies. For these cooling studies, I used a 200-cm<sup>3</sup> cylindrical pressure vessel loaded with 2.88 mol tritium and a new thermocouple for the vessel. Instead of being spotwelded to

the vessel surface, the junction was inserted into a thermowell in the vessel wall.

### Simulated Tritium Loading

To determine the effectiveness of the cooling chamber, I again heated the two pressure vessels to preselected tritium concentrations. When temperature equilibrium was reached and with the vessel's electrical power still on, I placed the vessel inside the cooling chamber, turned on the fan, and recorded temperature vs time. Average air velocities measured at the chamber exit were:

- 104-cm<sup>3</sup> cylindrical vessel, 60 liters/s;
- 200-cm<sup>3</sup> cylindrical vessel filled with tritium, 56 liters/s;
- 400-cm<sup>3</sup> coil vessel, 70 liters/s.

Once the vessel had cooled to its lowest temperature increase, I removed it from the chamber and measured its reheating rate while the vessel was on the balance pan and the balance case door was shut. Since no weighings were to be made, the balance beam was left in its arrested position. For uniformity between measurements, zero time and the baseline temperatures are those recorded the moment before the vessel was removed from the cooling chamber. This explains the slight shift in the slope at the start of the warming rate curve. The time spent to carry the vessel from the chamber and place it on the balance pan varied from 1 to 1 1/2 min.

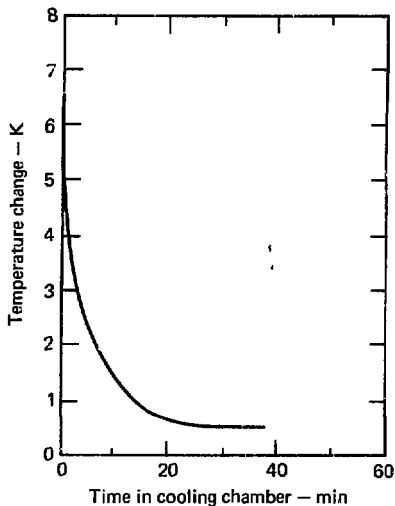


Fig. 6. Cooling rate for 400-cm<sup>3</sup> coil vessel. Simulated tritium load = 5 mol. Cooling air flow = 150 ft<sup>3</sup>/min.

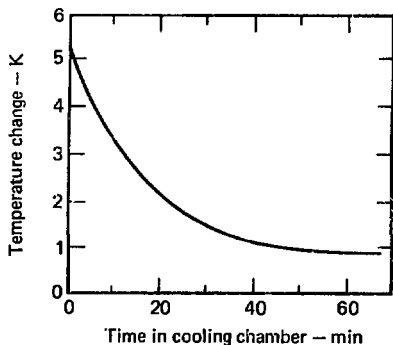


Fig. 7. Cooling rate for 104-cm<sup>3</sup> cylindrical vessel. Simulated tritium load = 2.5 mol. Cooling air flow = 130 ft<sup>3</sup>/min.

The 104-cm<sup>3</sup> cylindrical vessel was heated to a simulated tritium concentration of 2.5 mol, equivalent to the maximum allowable working pressure of the vessel. The voltage available from the regulated direct current power supply determined the size of the simulated tritium loading of the coil vessel, 4.0 and 5.0 mol.

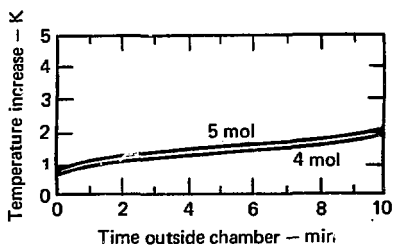


Fig. 8. Reheating rate for 400-cm<sup>3</sup> coil vessel. Simulated tritium load = 4 and 5 mol.

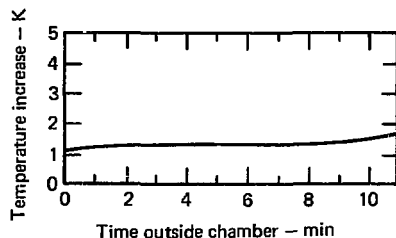


Fig. 9. Reheating rate for 104-cm<sup>3</sup> cylindrical vessel. Simulated tritium load = 2.5 mol.

Cooling rate curves show that the electrically heated vessels can be cooled to a temperature increase of 2°C or less (Figs. 6 and 7). The coil vessel with its greater surface area can be cooled to a temperature slightly less than 1°C above the original temperature (Fig. 6), whereas the 104-cm<sup>3</sup> cylinder can be cooled to a temperature slightly more than 1°C above the original (Fig. 7). The lower mass, higher loading, and larger surface area of the coil vessel allow it to warm on the balance faster than the thick-walled cylindrical vessel (see Figs. 5 and 6). Since a reasonably accurate weighing takes less than 5 min, the temperature remains within 1.5°C of the original during weighings and the weight error is less than 0.1% (Figs. 8 and 9).

### Actual Tritium Loading

The procedure for studying the 200-cm<sup>3</sup> source vessel loaded with 2.88 mol tritium was somewhat different from that used to study the electrically heated vessel. After it was warmed to some equilibrium temperature (Fig. 10), I placed the source vessel on the balance pan and made five consecutive



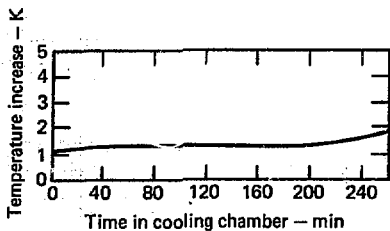


Fig. 10. Reheating rate for 200-cm<sup>3</sup> cylindrical vessel. Actual tritium load = 2.9 mol.

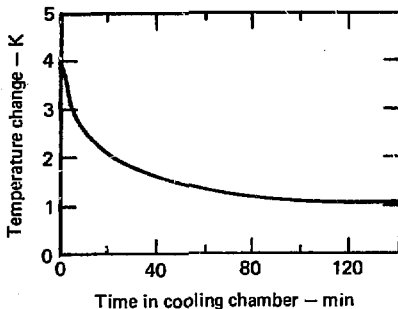


Fig. 11. Cooling rate for 200-cm<sup>3</sup> cylindrical vessel. Actual tritium load = 2.9 mol. Cooling air flow = 120 ft<sup>3</sup>/min.

weighings. The average of these five was used as the gross weight. Next I removed the vessel from the balance and placed it inside the cooling chamber. Once the vessel had reached its minimum temperature (Fig. 11), I removed it from the cooling chamber, placed it on the balance pan, and reweighed it. The average of five consecutive weighings was the tare from which I calculated the reported weight shifts (Fig. 12).

Equilibrium temperatures ranging from 3 to 7°C were obtained by allowing the vessel to equilibrate under various laboratory storage conditions—on a work bench, in a fume hood, and inside the balance case itself. The weight shifts in Fig. 10 confirm those I found during the earlier part of this study, using electrically simulated tritium decay heat. This part of the study shows that applying a corrective factor would be difficult because the equilibrium temperature depends so strongly on the storage conditions.

In Fig. 10, the plot of apparent weight loss vs temperature change, the scatter between the tares taken on the warm vessel has a standard deviation

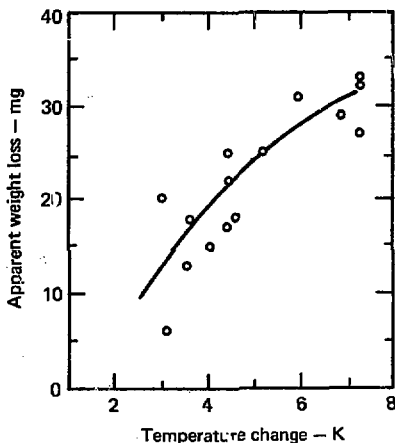


Fig. 12. Measured weight loss for 200-cm<sup>3</sup> cylindrical vessel. Actual tritium load = 2.9 mol.

of  $\pm 14$  mg. Not shown in this figure, since the Y-axis points were derived from the difference between the warm mass and the cool mass, are the tares taken on the vessel after it was cooled. These had a standard deviation of  $\pm 7$  mg. Yet the repeatability of the balance in the 20-kg weight range is approximately 3 mg.

To find some clear reason for this discrepancy, I again air-cooled the vessel several times and measured its minimum temperature equilibrium by a potentiometer more sensitive than the one used earlier. Here I found that, for the same cooling period, the minimum temperature the vessel reached varied by  $\pm 0.15^\circ\text{C}$ . This scatter is characteristic of the cooling method itself, more than likely coming from temperature variations in the air that enters the cooling chamber and changes in the fan motor's speed due to line voltage fluctuations. Cooling to a uniform temperature before each weighing reduced the deviation from  $\pm 7$  mg to  $\pm 4$  mg.

## Conclusions

Cooling the vessel before weighing has two positive, related effects:

- The negative weight shift is all but eliminated,
- The weighing error band is narrowed close to that which is normal for the balance (measured as  $\pm 3$  mg).

The significance of this error reduction can better be seen in a gas loading where the amount of tritium approximates a real life situation. Thus, when the tritium loading is 20 g and the temperature increase for the vessel is 5°C, the mean weighing error for the warm vessel is 0.125%. However, after being cooled

to near 1°C above the original temperature, the same vessel has a mean weighing error of 0.025%. By measuring carefully the final temperature increment and cooling to this same temperature before each weighing, the error can be reduced a bit more—to 0.020%.