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R. T. Tsugawa, D. Fearon, P. C. Souers, R. G. Hickman, and P. E. Roberts

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A SECONDARY CONTAINMENT SYSTEM FOR A
HIGH TRITIUM RESEARCH CRYOSTAT

R. T. Tsugawa, D. Fearon, P. C. Souers,
R. G. Hickman, and P. E. Roberts*

Lawrence Livermore Laboratory, University of California
Livermore, California 94550

ABSTRACT

A 4.2- to 300-K liquid helium cryostat has been constructed for cryogenic samples of D-T containing up to 4×10^{14} dis/s (10,000 Ci) of tritium radioactivity. The cryostat is enclosed in a secondary box, which acts as the ultimate container in case of a tritium release. Dry argon is flushed through the box, and the box atmosphere is monitored for tritium, oxygen, and water vapor. A rupture disk and abort tank protect the box atmosphere in case the sample cell breaks. If tritium breaks into the box, a powdered uranium getter trap reduces the 4×10^{14} dis/s (10,000 Ci) to 4×10^9 dis/s (0.1 Ci) in 24 h. A backup palladium-zeolite getter system goes into operation if an overabundance of oxygen contaminates the uranium getter.

INTRODUCTION

There is considerable interest in controlled hydrogen fusion by lasers, electron beams, or magnetic confinement. The fusion fuel mixture at the lowest temperature is the three-component heavy hydrogen mixture, D_2 -DT- T_2 , commonly referred to as D-T. Because D-T can be used in liquid or solid form, the physical properties at cryogenic temperatures become of extreme interest.

* This work was performed under the auspices of the U.S. Energy Research & Development Administration.

In companion articles we reported on the phase diagram and other physical properties expected for D-T.^{1,2} To measure some of these properties, we constructed a liquid helium research cryostat designed to hold up to 4×10^{14} dis/s (10,000 Ci) tritium, which is equivalent to only $7.5 \times 10^{-6} \text{ m}^3$ of liquid D-T. Most of our design and construction effort went into the tritium containment system. This system is built in accordance with modern "zero release" standards, which have been adopted for protection of personnel and the environment. The method used is secondary containment, in which the entire experimental apparatus is housed in a gastight box. Although chances of a rupture in the cryostat are slight, any leaked tritium is again contained by the box.

THE D-T CRYOSTAT SYSTEM

The cryostat system is composed of the cryostat itself, liquid helium transfer lines, D-T gas pressure bottles and input lines, a rupture disk and abort tank, a quadrupole mass spectrometer, a helium leak detector, and two pumps.

The cryostat is a commercial, liquid helium unit (Air Products*) modified for tritium use by replacing all elastomer O-rings by metal (tritium radiation degrades elastomers). It is a small unit (1.5 W at 4.2 K, 8 W at 20 K), requiring no liquid nitrogen and containing only $2 \times 10^{-6} \text{ m}^3$ liquid helium at any time. This liquid can evaporate into a large exhaust volume that passes out of the box. Therefore, no helium enters the box atmosphere; the entire path of the helium is gastight. The cryostat is insulated by a $1.3 \times 10^{-6} \text{ Pa}$ (10^{-8} Torr) vacuum, provided by a $0.175 \text{ m}^3/\text{s}$ oil diffusion pump (with mechanical roughing pump). During assembly, leaks may be monitored by a helium leak detector (Varian), sensitive to $1.3 \times 10^{-10} \text{ Pa}$ (10^{-12} Torr) air, on the vacuum line.

* 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. Energy Research & Development Administration to the exclusion of others that may be suitable.

The $2 \cdot 10^{-6} \text{ m}^3$ liquid helium reservoir is inside a cold block, wrapped with heater wire to yield continuous temperatures from 4.2 to 300 K. The sample cell is attached to the cold block by a C-shaped copper connector. Varnish (General Electric 7031) is used for good thermal contact. A germanium temperature sensor, calibrated with liquid helium and the vapor pressure of D_2 ,¹ is glued to the sample cell. A stainless steel tube (3.18-mm o.d., 2.67-mm i.d.) runs the length of the cryostat to the sample cell and is pinned to the cold block to reduce thermal loss. This tube carries the D-T gas from an input pressure bottle and also removes the D-T to a small uranium getter trap outside the containment box. The D-T vapor may be analyzed by a quadrupole mass spectrometer (Uthe Technology International), sensitive to $1.3 \cdot 10^{-12} \text{ Pa}$ (10^{-14} Torr) N_2 . The cryostat has been tested by the successful freezing of D_2 .

The sample cell can contain up to $3 \cdot 10^{-6} \text{ m}^3$ liquid or solid D-T. This amount, combined with that in the lines and pressure bottle, totals $4 \times 10^{14} \text{ dis/s}$ (10,000 Ci). Also, the thermal capacity of the cryostat is small to avoid large amounts of cryogenic liquid that might vaporize. Nevertheless, the possibility of a sudden breakage of even this small sample cell must be recognized. This is especially critical in optical experiments with glass sample tubes and viewports.

If the sample cell breaks, the tritium is contained in the cryostat vacuum jacket, because the vacuum pump to this space is closed off before loading the D-T. However, the pressure buildup in the vacuum space of the cryostat could reach 1 MPa (150 psi). To prevent the blowing out of optical windows or gaskets (and the tritium entering the box), a protective disk ruptures at $3.5 \times 10^4 \text{ Pa}$ (5 psi) over atmospheric pressure, and the gas passes into a 0.04-m^3 abort tank. Hence, the secondary tritium containment system is not required in this case.

THE TRITIUM CONTAINMENT SYSTEM

The main component of the tritium containment system (shown schematically in Fig. 1) is the argon flush box, inside of which are the cryostat and the tritium recovery equipment. A dry argon atmosphere

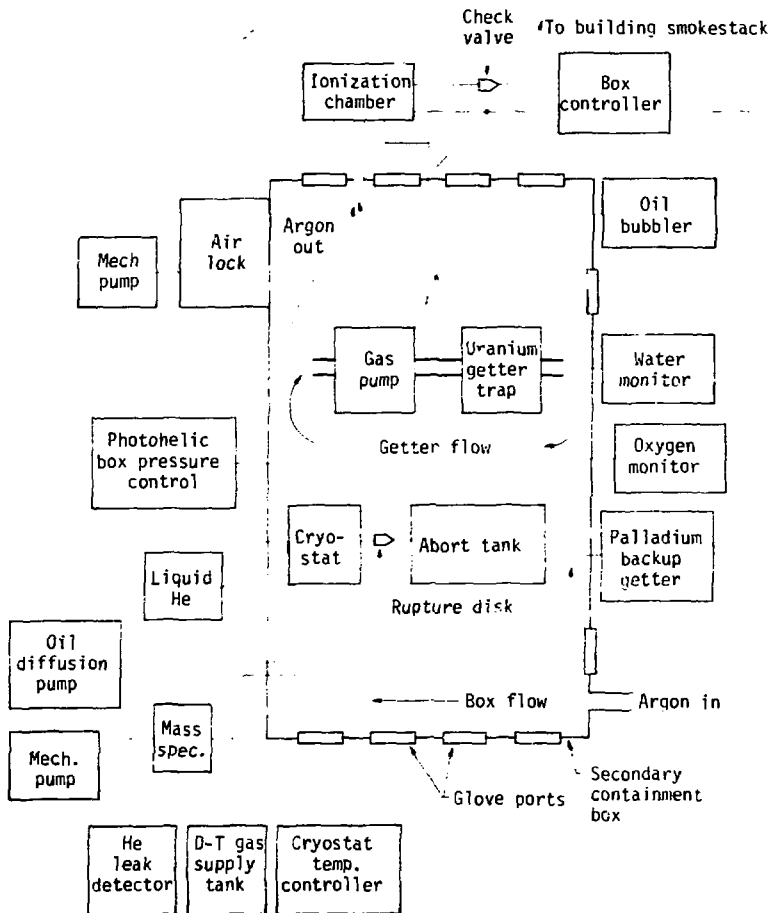


Fig. 1. Schematic of Tritium Cryostat Containment.

is maintained in the box to avoid conversion of tritium to the more dangerous tritiated water and to avoid contamination of the tritium recovery system. A large assortment of monitors and control valves are interfaced with the box. The completed containment system, including the enclosed cryostat, is shown in Fig. 2.

The box (Vacuum Atmosphere) is made of aluminum with Pyrex windows tightened into rubber gaskets and has an internal volume of 2 m^3 . The cryostat is attached by O-ring seals to the box roof. There are 10 ports for rubber gloves along the sides, and air and water vapor slowly permeate through these gloves while in use. To purge these contaminants, a steady flow of dry argon (H_2O , O_2 , and N_2 , less than 1 ppm) is passed through the box at a $1.5 \times 10^{-2} \text{ m}^3/\text{min}$ rate. The argon passes through a one-way check valve and exhausts into the building smokestack. If a rubber glove is punctured, all operations must cease until the glove is replaced and the atmosphere reestablished. (It requires one week of argon purge at a high flow rate to achieve the equilibrium low-contaminant atmosphere.) When not in use, the glove ports are covered with aluminum plates seated against flat rubber gaskets. The space between the plates and the rubber gloves is pumped to 1.3 Pa (10^{-2} Torr) with the second $0.25\text{-m}^3/\text{s}$ mechanical pump. Articles may be brought into the box through an air lock connected to this mechanical pump. Dry argon is admitted to bring the air lock to atmospheric pressure.

The box argon atmosphere is controlled to $101 \pm 0.3 \text{ kPa}$ ($1 \text{ atm} \pm 0.3\%$) by a photohelic rubber diaphragm switch (Dwyer Instruments), which electrically opens the mechanical pump valve if the box pressure is too high or opens the argon inlet valve if it is too low. As a backup system, in the event of photohelic control failure, the box is connected to the outside world via an oil bubbler. If the box pressure is too high, the excess gas will be forced out through the oil and into the stack. If the box pressure is too low, stack air will be sucked into the box, destroying the integrity of the atmosphere but saving the box from implosion.

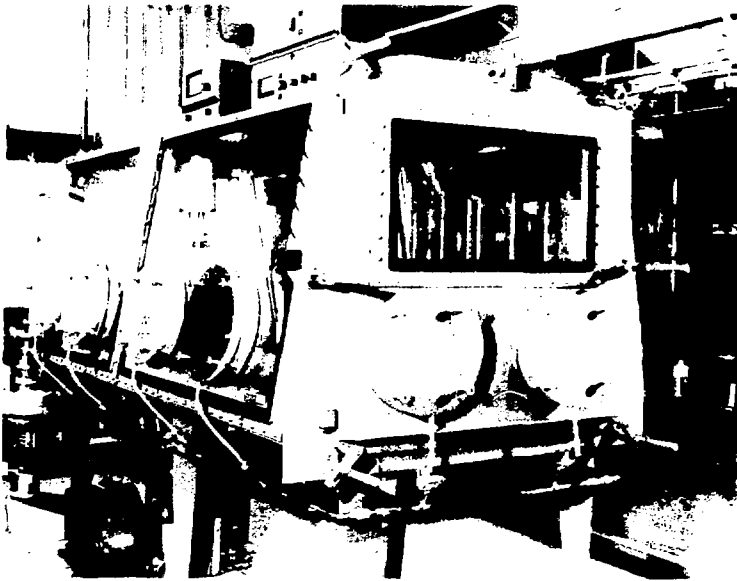


Fig. 2. Containment System for Crystal.

The tritium concentration in the system is monitored by an ionization chamber in the argon outlet pipe. The ionization chamber is an all-metal unit, bakeable to 470 C, designed especially for low-level tritium work.¹ Its lowest sensitivity is about 1.1×10^7 dis/m³·s (0.01 Ci/l). The control system can be set to operate at activity levels as low as 1×10^7 dis/m³ (1 Ci/l) in the control box (1.9×10^{10} dis/s or 500 Ci/l) in the ionization chamber. If the measured radioactivity rises above this value, the control system closes the argon inlet and argon exhaust, opens the uranium getter, and starts the active circulating pump. As a final precaution, the gas return inlet to the exhaust system is shut off. Tritium concentration in the system drops to a trigger at 1.1×10^7 dis/m³·s (50 Ci/m³).

Water is not allowed to enter the system for several reasons. First, tritium molecules can exchange with water to form tritiated water, which is readily absorbed into the metal. Second, water, even at a thousand times its diffusivity as helium gas, would poison the argon and oxygen getters with the uranium getter that it carries, which cannot be removed by washing. This has a potential tritium-absorbing capacity from the getter. Last, the uranium powder of the getter is pyrophoric in the presence of large quantities of oxygen. For these reasons, the water vapor in the box is monitored to 1 ppm by a impedance changes across an Al₂O₃ disk (Panagotri, S). The oxygen is monitored to 1 ppm (proprietary method, Telcom). The systems fire alarms at concentration of 5 (water) and 10 (oxygen) ppm.

If the box fills with tritium because of a cell rupture, the radioactivity will be removed by the uranium getter trap. This getter, shown schematically in Fig. 5, is the most advanced design to date.^{11,12} A metal bellows pump of 0.132-m³/min capacity circulates the box atmosphere through the getter. The getter assembly is basically two concentric cylinders. The inner cylinder holds six trays of "activated" uranium, activated by reacting uranium chips to hydride and then thermally decomposing the compound. The result is a uranium powder of micrometer size and large surface area. The uranium powder is prevented from

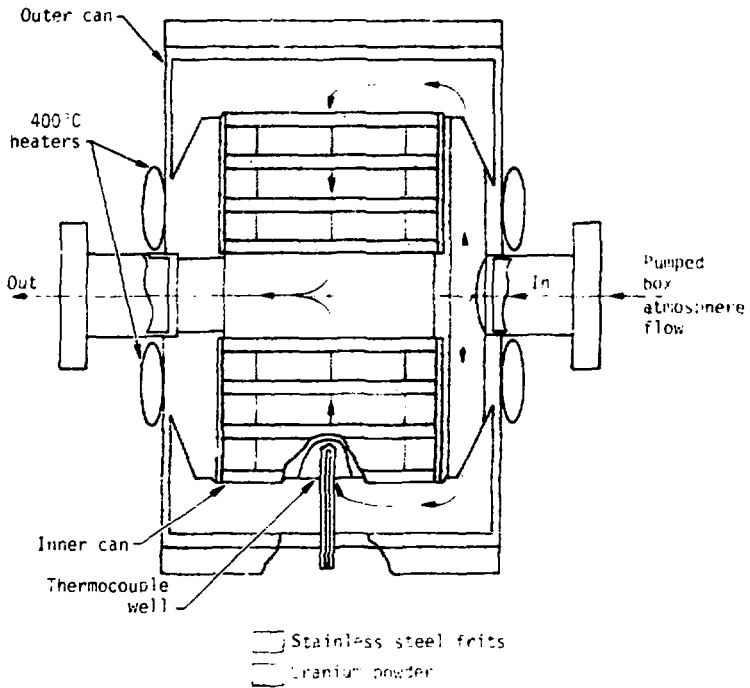


FIG. 3. Schematic of Heated Uranium Getter Trap.

spilling out by beam stainless steel frits through which the box atmosphere is passed. The outer cylindrical can serves to further enclose the uranium and also permits maintenance of an expected 1.0×10^{-4} Pa (0.001 psi) pressure differential across the uranium beds. The uranium reacts with hydrogen at room temperature. Four band heaters surround the outer cylinder and can heat the uranium hydride to 400°C. At this point, the hydride decomposes, and the tritium may be collected in a separate vessel.

The getter contains 100 grams of uranium, and only 1.7% of its capacity will be needed to hold 1.0×10^{17} DIS (0.1 Ci) of tritium.

The getterline times for 1.0×10^{17} DIS (0.1 Ci) tritium are estimated to be: after 10 min, 1.0×10^{17} DIS (0.1 Ci); after 8 h, 5×10^{15} DIS (0.005 Ci); and after 10 days, 1.0×10^{14} DIS (0.001 Ci). The getter can, therefore, remove 99,999% of the total tritium in an accident of maximum size.

Finally, provision is made for a backup system for the uranium getter trap. Should the D-T gas from an accident be mixed with excess oxygen from a leak, a portable palladium-zeolite molecular sieve may be attached to the box. The palladium causes the tritium and oxygen to form water that is retained on the zeolite in a form not easily recoverable.

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