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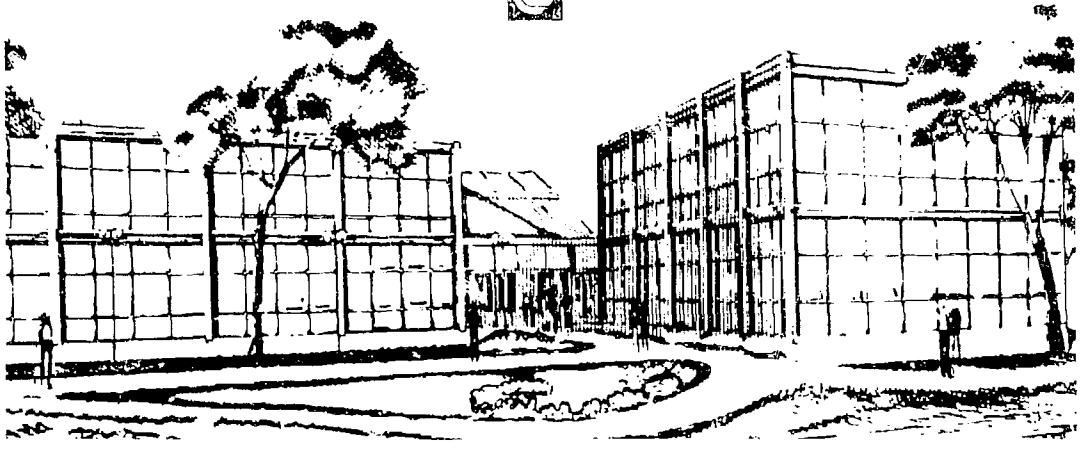

DECOMMISSIONING A TRITIUM GLOVE-BOX FACILITY*

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ABSTRACT

A large glove-box facility for handling reactive metal tritides was decommissioned. Major sections of the glove box were decontaminated and disassembled for reuse at another tritium facility. To achieve the desired results, decontamination required repeated washing, first with organic liquids, then with water and detergents. Worker protection was provided by simple ventilation, which was combined with careful monitoring of the work areas and employees. Several innovative techniques are described.

INTRODUCTION

A major tritium research facility at Lawrence Livermore Laboratory (LLL) was obsolete for current programs and was decommissioned. This facility included:

- A nine-section glove box with a volume of about 8 m^3.
- An enclosed 500-ton hydraulic press.

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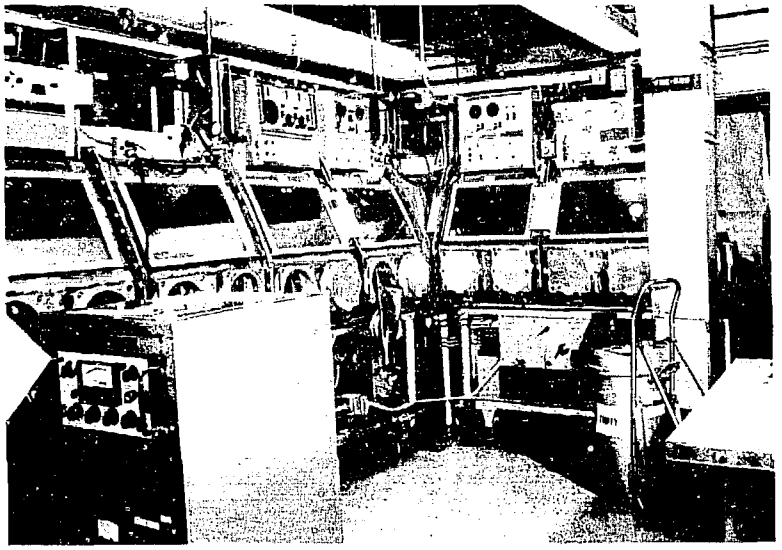

Fig. 1. Glove box for handling reactive metal tritides in an inert atmosphere.

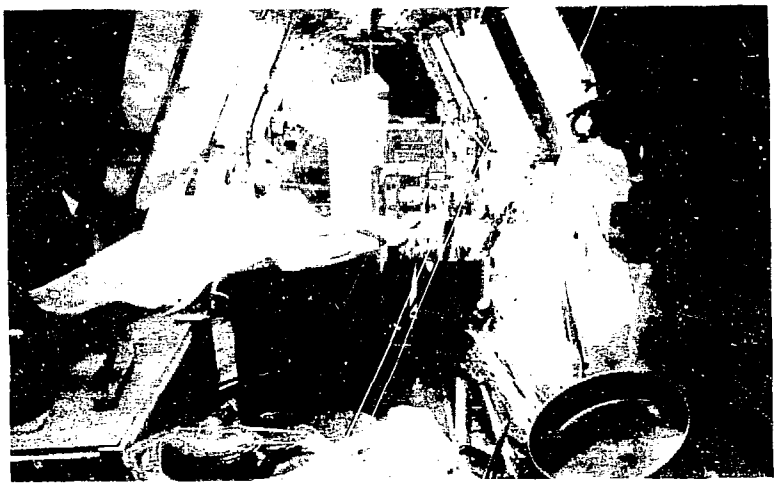

Fig. 2. Interior view of glove box, showing typical equipment to be disposed of as waste.

system included a Pd-catalyst bed and a 140-kg bed of 5A molecular sieve, with a blower, vacuum pumps, piping, and monitoring equipment. We estimated the molecular-sieve bed held about 9 g of tritium as HTO.

The molecular-sieve bed had been last regenerated nearly 12 years previously. The original equipment for regeneration was inadequate, and all temporary equipment was removed at that time. Even now the purification system maintained the glove-box atmosphere at about 1 to 2 ppm water (containing all hydrogen isotopes) and about 0.2 Ci/m^3 (combined HT and HTO). However, without the ability to regenerate the molecular-sieve bed, any large additions of water during decontamination would partially displace the existing HTO, thereby increasing tritium in the glove-box atmosphere and thus increasing worker exposures as well as effluent releases to the atmosphere. This greatly influenced the decontamination processes we used.

We knew that tritium-contaminated facilities at several laboratories, including ours, had been decommissioned, but we were unaware of any reports on the procedures used or the amount of effort needed for specified goals. After making repairs on a similar glove box, Folkers and Johnson[1] reported decontamination of nearby areas with methyl alcohol and trichloroethylene and an effective use of local ventilation for worker protection. Harris, Kokenge and Marsh[2] and Gilbert, Wright and Madding[3] reported that procedures for decontaminating facilities for ^{238}Pu and ^{210}Po, respectively, required repeated washing with detergents to get the desired results.

Because of this, part of its operation, we had to develop innovative techniques for tritium decontamination while minimizing worker exposures and atmospheric releases. This report describes the methods used and our results.

PLANNING

The two key issues identified[4] were:

- To adopt standards for residual tritium contamination that are acceptable for subsequent use of recovered equipment.
- To minimize the radiation dose to workers and the public during the operation.

Normal procedures for operation of our tritium facility provided the basis for most of our planning.[5]

A safety assessment[6] provided guidance for the entire operation, especially for unusual or high risk operations. Key recommendations included:

- Use of protective clothing while handling contaminated equipment.
- Provision for adequate ventilation (0.75 m/s) while working on open contaminated equipment.
- Monitoring of workers by taking daily bioassay samples during high hazard operations and at least weekly samples at other times.

The principal tritium-exposure pathways were associated with the initial breaking of tritium lines, decontamination efforts, and the handling of tritium-contaminated vacuum-pump oil.

Primary protection for the workers consisted of maintaining a positive flow of air away from personnel and the wearing of protective clothing, that is, 5-mil polyvinyl chloride gloves and cotton lab coats. While working in the glove box, 1.75-mil shoulder-length polyethylene gloves were worn to prevent skin contact with the rubber gloves on the box; these polyethylene gloves were replaced four times per hour. The bioassay program recorded actual exposures.

The room containing the glove box was continuously monitored for airborne tritium using two, Overhoff Beta-Tech*, 2-litre,

*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.

gamma-compensated, ion chambers. The average tritium concentration in the room was <10 $\mu Ci/m^3$ throughout the decommissioning project. On two separate occasions, the tritium room concentration approached 350 $\mu Ci/m^3$. Normal room ventilation reduced the level in about 10 min.

Maximum allowable surface contamination levels are shown in Table 1. As we expected to ship the glove-box sections to Sandia Laboratory Livermore (SLL) for use in their tritium facility, special contamination levels were adopted for this purpose. Surface-contamination levels were assessed, using standard swiping techniques and a liquid scintillation unit located in the same building.

The maximum off-site exposure from a postulated accidental release of 1 g of tritium as HTO was estimated[6] to be less than 30 mrem. This was unlikely to occur since most of the tritium here was bound on the molecular-sieve bed.

Table 1. Maximum Levels of Tritium Surface Contamination Allowed for Various Uses.

Item	Maximum Allowable Level ($dis/s \cdot cm^2$)
Equipment for unrestricted use, including general public[a]	1
Equipment or furniture, use limited to offices within our tritium facility[a]	2
Equipment or furniture, use limited to laboratories within our tritium facility[a]	20
External surfaces of equipment to be sent to SLL for use in their tritium facility	2
Internal surfaces of equipment to be sent to SLL for use in their tritium facility	2000[b,c]

[a] Taken from Ref. 5.

[b] Each glove-box section also limited to total (absorbed plus swipable) tritium of 1×10^{12} dis/s (~25 Ci).

[c] Enclosed during shipment; required continuous ventilation except during shipment; required protective clothing during work.

Both HT and HTO outgas continuously from contaminated surfaces, even after decontamination. All steps were planned to retain the gas purification system or to flow air continuously, so that unexpected concentrations of tritium would not build up in enclosed spaces or piping.

A preliminary survey was made of the glove box, its contents, and related equipment. We anticipated the hazard for various operations from the data in Table 2. For example, we know from experience that surface contamination levels of <20 dis/s·cm^2 can be handled in the open by workers appropriately monitored, while levels up to about 8×10^5 dis/s·cm^2 can be safely handled with plastic or rubber gloves inside a flush hood. Vacuum-pump oil with at least 30 mCi/ℓ of tritium contamination and no water content can be handled with plastic gloves and modest ventilation. The hazard is severe when the oil also contains water.

Table 2. Tritium Contamination Levels in a Research Glove-Box Facility After Exposure of Glove Box to Solid Tritium Compounds for Over 11 Years.

Item	Measured Contamination Level
Molecular-sieve bed, 140 kg type 5A, tritium stored as HTO	~9×10^4 Ci (estimated)
Equipment and surfaces inside glove box	0.3 to 30×10^7 dis/s·cm^2 (1 to 100 Ci/m^2)
Equipment and surfaces inside flush hoods	0.7 to 800×10^3 dis/s·cm^2 (0.2 to 200 mCi/m^2)
External surfaces of glove box, purification system, nearby walls, floor, and furniture	0.4 to 8 dis/s·cm^2 (0.1 to 2 μCi/m^2)
Vacuum pump oil, pump used to recover hydrogen isotopes	~150 mCi/ℓ
Vacuum pump oil, pump used for glove-box atmosphere	1 to 30 mCi/ℓ
Argon atmosphere inside glove box	~0.2 Ci/m^3

DECONTAMINATION AND DISASSEMBLY PROCEDURES

Some metal tritides react spontaneously with H_2O or O_2 to liberate tritium. For example:

$$LiT\ (s) + H_2O\ (\ell \text{ or } g) \rightarrow LiOH\ (s) + HT\ (g) \quad .$$

Other tritides such as TiT_2 are relatively stable and as contaminants must be physically removed. Water-based detergents used to remove stable particulates would react with less stable materials, giving excessive HT in the glove-box atmosphere and thus increase worker exposures. Use of water was also limited by the capacity of our molecular-sieve bed, as described earlier. Therefore, our procedures used mineral oil and kerosene to remove "loose" contamination before using any water.

Glove Box Contents

All quantities of metal tritides were removed, including dust and debris vacuumed from the glove box. Equipment inside the glove box was packaged as contaminated waste. The following procedures were generally followed:

- Tritium contamination was estimated from past experience.
- Equipment was placed in "lard" cans (~45-ℓ volume). Pieces too large to fit were disassembled, broken, or crushed.
- Outsides of the lard cans were wiped off with mineral oil, and closed cans were moved from the glove box to the flush hood.
- Lids were sealed with silastic sealant and taped.
- Cans were sealed in plastic bags and placed in 55-gal barrels. These also had to be promptly sealed or some contamination from the lard cans would permeate the plastic bags.

Glove Box Decontamination

Decontamination of the inside surfaces of the glove box was as follows:

- Washed with mineral oil.
- Washed with kerosene, followed by kerosene with detergent.

- Water spray to decompose reactive tritides.
- Washed twice with water and detergents.
- Converted the glove box from argon atmosphere to once-through air flush, followed by two water rinses.
- Removed windows and gloves, washed adjacent areas with water-based detergent.
- Washed entire glove box with water-based detergent.
- Separated glove-box sections and washed flanges with water-based detergent.

The air locks were cleaned by a modified procedure, which eliminated the use of kerosene with detergents.

Results of the decontamination procedures for the glove box are shown in Fig. 3. Repeated scrubbings were clearly necessary to achieve the desired decontamination, even though the final contamination levels were well below the target level of 2000 dis/s·cm^2.

When the glove-box interior was washed or rinsed, this included spraying hard-to-reach corners with a pressurized nozzle and an external source of fluid. Dishwashing swabs with extended handles provided scrubbing action. Fluids and dirt were wiped up with paper products and sealed in paint cans.

The first use of water as a "spray" was by a hand-held spray bottle similar to those used for household detergents. Reaction with tritide salts caused an effervescence, increasing the tritium in the glove-box atmosphere. When the level exceeded 1 to 2 Ci/m^3, we stopped spraying until the level came back down so as to minimize worker exposures. Joints were saturated repeatedly to decompose everything possible before converting to once-through air flow. This decomposition was much slower than expected--this was true even on flat surfaces after repeated applications of water, as evidenced by increasing tritium in the glove-box atmosphere. We later estimated that decomposition of salts liberated about 2 to 12 Ci/m^2 of surface as HT, although these surfaces were swiping only a few percent of this just before the water spray (see Fig. 3).

In order to use water, we valved out our large molecular-sieve bed and installed an in-situ dryer in the glove box. The regular purification-system blower and catalyst were maintained so that HT would be converted to HTO; while the glove-box atmosphere was still basically argon, it contained several hundred ppm O_2 for this purpose. This effectively gave us an unlimited capacity for water. The in-situ dryer is described elsewhere.

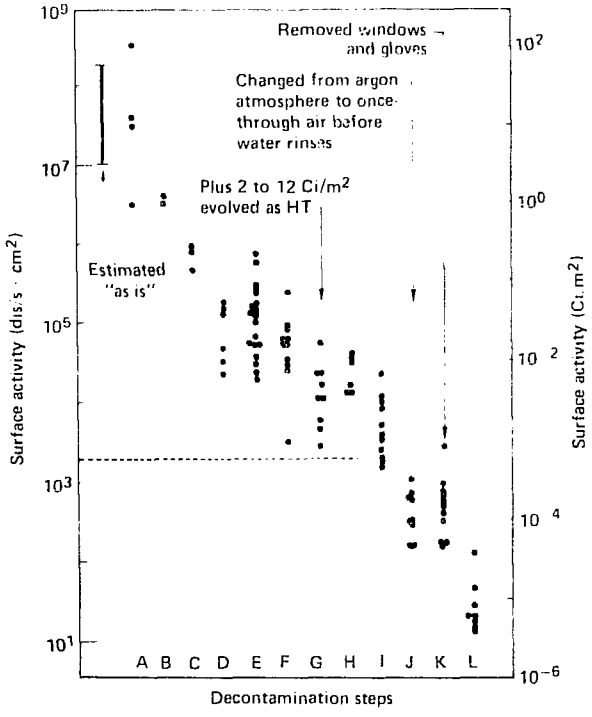

Fig. 3. Glove-box internal-surface contamination as reduced by decontamination procedures. Area below dashed line shows target level of activity for shipment off site. The decontamination steps are:

A	measured "as is"	F	2% Triton CF-10 with kerosene
B	mineral oil	G	water spray
C	kerosene	H, I	2% X-100 with water
D	kerosene	J	water rinses (2)
E	various detergents with kerosene	K	air flush 30 days
		L	swish; water and methanol rinse

We tried using several detergents in kerosene to lift off visible grime. These were anhydrous nonionic surface active agents, one example being a 2-vol% solution of Triton CF-10 (trademark of Rohm and Haas Co.), a benzyl ether of octyl phenol. Detergents in kerosene did not appear to be effective beyond the ability of kerosene to wipe away loose particulates, as shown in Fig. 3.

With water, we used a general purpose, nonionic surface active agent as a 2-vol% solution of Triton X-100 (trademark of Rohm and

Haas Co.), a type of alkylaryl polyether alcohol (see Fig. 3). Another detergent we used effectively was Swish (trademark of Haviland Products Co.), a proprietary water-base amphoteric surfactant containing alkaline silicates, which is packaged in a spray can. This product foams upon application, and after standing it was rinsed off with water or methyl alcohol (see Fig. 3). At this point, we conclude that any good detergent in water would be effective.

After most of the glove box decontamination was done, the box was purged with room air for about 30 days. The windows and gloves were removed during this time. We then swiped the inside surfaces again to see if there was any tritium accumulation from outgassing. Results are noted in Fig. 3. There was no increase of tritium activity inside the glove box and only a modest increase on the air-lock surfaces.

Windows, Gloves, and Fittings

After contamination levels dropped below 2000 dis/s·cm^2, we removed windows and gloves. We used a simple, portable, acrylic plastic hood with an exhaust blower over the work area and with the glove box under negative pressure so that air would flow into it. The hood is illustrated in Fig. 4.

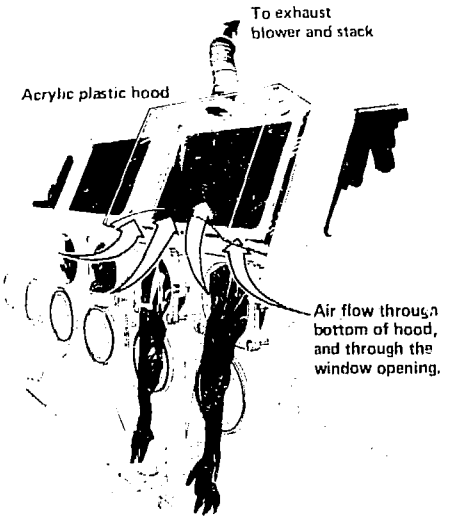

Fig. 4. Portable hood to provide ventilation while removing windows and cleaning adjacent areas.

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In-Situ Packed-Bed Dryer

We used an inexpensive, temporar[illegible] operated inside the glove box, eliminating [illegible] purification-system molecular-sieve bed. This bed [illegible] by simply pouring out the expended desiccant and [illegible] fresh desiccant.

The in-situ dryer was made from a lard can, [illegible] a wire screen, as shown in Fig. [illegible]. Joints were [illegible] adhesive. The blower provided about [illegible] a bed of 5A molecular sieve about [illegible] cm deep.

A molecular-sieve charge of 2.3 kg gave a maximum [illegible] about 460 g of water. A fresh charge of molecular sieve [illegible]

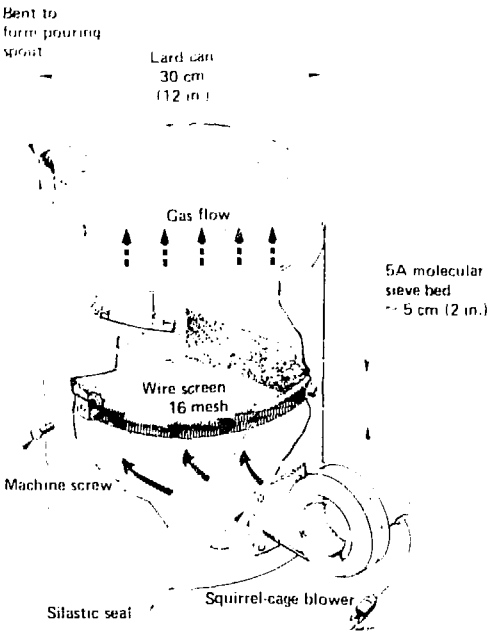

Fig. 5. Construction features of in-situ pack-bed dryer for scavenging water containing some HTO.

dryer could maintain the glove-box atmosphere at 250 ppm H_2O, with HTO levels typically 0.2 to 0.3 Ci/m^3. It could also reduce the glove-box atmosphere from about 30,000 to 2000 ppm H_2O in less than 2 h. This performance was adequate for our purposes.

Bioassay Program

Workers submitted urine samples daily during heavy work loads and at least weekly at other times. The typical range of tritium concentration was 1 to 5 μCi/ℓ, and the highest value was 8 μCi/ℓ. The maximum worker exposure was 110 mrem, and the total integrated dose for all workers was 420 mrem for the 6-month period to date. These values are within our guidelines.

Rubber Gaskets

We recovered several gaskets from air-lock doors and other locations to verify their condition after more than 11-years service

hardness.

Analyses of tritium concentrations in the gasket materials ranged up to 2 mCi/g. There was some indication that Buna N has hardened, but this could be because of other factors as well as tritium exposure. We judged the gaskets to be generally suitable for further service.

WASTE DISPOSAL

Requirements of our waste disposal program were largely limited to packaging waste in containers that conformed to current regulations[7]. Ultimate disposal was handled by the LLL Waste Disposal Group, and commercial burial sites were used.

Bagged or canned waste was loaded into containers, and the tritium content was estimated; when appropriate, the item was weighed. A log of these data were kept. From this point on, the Waste Disposal Group processed the containers.

CONCLUSIONS

A major tritium research facility for handling reactive metal tritides was decommissioned. A large glove box was decontaminated and disassembled for reuse at another tritium facility. Tritium contamination inside the glove box was reduced by six orders of magnitude to a level of about 10 to 100 dis/s·cm^2. Decontamination required repeated washing, first with organic liquids and then with water and detergents, to achieve the desired results. Special swipe techniques were used to monitor progress inside the glove box. Careful planning before and during our operations played a major role in our success. Adequate worker protection was provided by the use of simple protective clothing in conjunction with continuous monitoring of the room atmosphere and regular bioassays of the workers. Adequate local ventilation gave protection to workers while opening contaminated equipment or breaking contaminated plumbing lines. Radiation exposures were well within our guidelines; the highest individual exposure being 110 mrem for a 6-month period.

An inexpensive in-situ packed-bed dryer was developed for temporary service during decontamination operations. This effectively gave us unlimited capacity to adsorb water containing some HTO.

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[illegible] of the authors. [illegible] helped us, especially [illegible] his intimate understanding of [illegible] for his many useful suggestions; R. H. [illegible] of rubber gaskets; E. J. [illegible]; and F. M. Alire for [illegible] manuscript.

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