

CONF-800427-10

TITLE: Performance and Improvements of the Tritium Handling Facility at the Los Alamos Scientific Laboratory\*

AUTHOR(S): J. E. Nason **MASTER**

SUBMITTED TO: American Nuclear Society Topical Meeting Tritium Technology in Fission, Fusion, and Isotopic Applications

DISCLAIMER

University of California

\*Work performed under the auspices of the U.S. Department of Energy

By acceptance of this article, the publisher recognizes that the U.S. Government retains a nonexclusive, royalty free license to publish or reproduce the published form of this contribution, or to allow others to do so, for U.S. Government purposes.

The Los Alamos Scientific Laboratory requests that the publisher identify this article as work performed under the auspices of the U.S. Department of Energy.



LOS ALAMOS SCIENTIFIC LABORATORY

Post Office Box 1663 Los Alamos, New Mexico 87545

An Affirmative Action/Equal Opportunity Employer

DISTRIBUTION OF THIS REPORT

PERFORMANCE AND IMPROVEMENTS OF THE TRITIUM HANDLING FACILITY  
AT THE LOS ALAMOS SCIENTIFIC LABORATORY

J. E. Nasise

University of California  
Los Alamos Scientific Laboratory  
Los Alamos, New Mexico 87545

ABSTRACT

A major problem facing today's nuclear energy industry is the release of radioactive waste products to the atmosphere. The DP-East Tritium Handling Facility at the Los Alamos Scientific Laboratory (LASL), activated December 5, 1974 has processed  $3.8 \times 10^6$  curies of tritium with a total stack release of 704.5 curies as of December 12, 1979. This averages only 11.7 Ci/month which to our knowledge is the lowest stack release any major tritium facility has achieved. The facility includes an 11.5 m<sup>3</sup> dry box with associated gas purification system (GPS) and an effluent treatment system (ETS). The system performance, problems, and improvements are discussed with special emphasis given to the ETS, the new dry box waste disposal system, and the new automated logic control system, all of which contribute significantly to the low level tritium release at this facility.

INTRODUCTION

The DP-East Tritium Handling Facility was activated December 5, 1974, to support the LASL weapons program. The facility has processed  $3.8 \times 10^6$  curies of tritium with a total stack release of 704.5 curies as of December 5, 1979. This averages 11.7 Ci/month, which to our knowledge is the lowest stack release any major tritium facility has achieved.

The facility includes an 11.5 m<sup>3</sup> dry box with associated gas purification systems (GPS) and an effluent treatment system (ETS). All effluents generated from the facility, except the room air, are processed through the ETS prior to release to the atmosphere.

The improvements, problems, and performance of this facility are discussed with special emphasis given to the ETS, dry box waste disposal system, and the automated logic control system. A brief description of the components will be given, but it will be assumed that the reader has a prior understanding of the system (Ref. 1).

Work performed under the auspices of the U.S. Department of Energy.

COMPONENT PERFORMANCE

A. Dry Box

The main tritium facility at DP-East consists of a large dry box system connected to a gas purification system (GPS) for maintaining an inert atmosphere. The dry box is 11.5 m<sup>3</sup> in volume and the GPS has a 5.7 m<sup>3</sup>/min capacity. Thus, every two minutes there is a complete turnover of dry box gas. Both the dry box and the GPS are interfaced with an effluent treatment system (ETS) designed to remove tritium from all effluents prior to release of these effluents to the environment.

The dry box is designed for handling highly reactive metal tritides, in particular Li(D,T) salt. The dry box is maintained at a slightly positive pressure of 1/2 inch of H<sub>2</sub>O by two photohelic gauges, one on the GPS and the other on the nitrogen removal system. The photohelic gauges control the addition of fresh helium and the removal of excess dry box gas to the ETS. An oil bubbler is provided to protect the dry box from large pressure differentials in the event the photohelic system fails. The bubbler consists of a metal U-tube filled with oil to the correct level that will release when the dry box internal pressure exceeds  $\pm 10$  inches of H<sub>2</sub>O. This simple device protects the dry box gloves and windows from failure due to excessive pressure. The only drawback is that the bubbler is vented into a fume hood where the exhaust from the hood goes directly to the atmosphere via the stack. This will be corrected in the near future.

Fifty-six Hypalon (Chloro-Sulfonated Polyethylene) gloves are used on the tritium dry box and none has needed to be changed from high stress deterioration. This type of deterioration is from ozone attack and is commonly seen on neoprene gloves. When the gloves are not in use, plastic covers with vacuum fittings are inserted in the glove port openings to reduce the permeation of water and oxygen through the gloves. The dry box is kept at such a low level of moisture ( $\sim 0.5$  ppm) that one gram of water a day may permeate through an uncovered glove.

1. DT Gas Handling System. A tritium gas handling system is located inside the dry box (Fig. 1). The system was designed for three main purposes: 1) to prepare Li(D,T) compounds, 2) to

carry out work on tritiated chemical systems, and 3) to prepare and fill laser targets for fusion experiments.

Fig. 1. Tritium gas handling system inside dry box

The manifold system is constructed primarily of 304 SS components, joined by welding where possible, and utilizing 316 SS Hoke Valves with Teflon seats (Ref. 2). The valve seats have seen continuous tritium service. Only one has developed a measurable leak across its seat thus far. It has not been determined if the leak was caused by tritium reacting with Teflon and forming HF, as some believe happens with extended contact between Teflon and tritium, or mechanical failure (Ref. 3). Where weld joints are not possible or practical the parts are joined using Cajon compression couplings with nickel-gasketed seals. Nickel baskets are used instead of aluminum ones because they do not creep as fast as the aluminum gaskets, offering a longer sealing time.

The initial 30-liter uranium bed has been replaced by a larger 75-liter capacity bed for storage and purification of tritium. The initial 25g capacity metal tritide resistance-heated synthesis reactor was replaced by a 300g capacity reactor (Fig. 1) that is inductively heated by a 5KW-RF generator. The system has performed reasonably well with the exception of the double-vane-transfer pump, also used as a vacuum pump to evacuate connecting apparatus. The pumping pressure differential of the vane pump is typically 50 millitorr to 760 torr. Porous stainless steel met 1 filters had to be installed on the pump's intake and exhaust lines to prevent particulates from entering the pump and reducing its efficiency. The main pitfall with these types of pumps is that they require a low vapor pressure lubricant resistant to radiation damage. The oil used at this facility is ether 4-ring polyphenyl 110 made by the Monsanto Research Corp.

2. Waste Disposal System. Prior to 1977, tritium contaminated dry box waste had to be removed from the dry box through one of the three pass boxes located in fume hoods. The exhaust from these hoods goes directly to the stack due to the limited capacity of the ETS of only 7 liters/sec. Therefore, removing tritium contaminated waste through any pass box always resulted in the release of tritium to the atmosphere ranging from 1 to 3 curies and was a potential hazard to the personnel involved. The dry box trash was packaged in containers within the dry box. Personnel wearing protective clothing would remove the packaged trash and place it into a 30-gal. drum, which was in turn placed into an asphalt-lined 55-gal. drum. An absorbent (vermiculite) was poured into the interstices of the asphalt-lined 55-gal. drum, the drum was sealed, and the unit was taken to the burial site (Fig. 2).

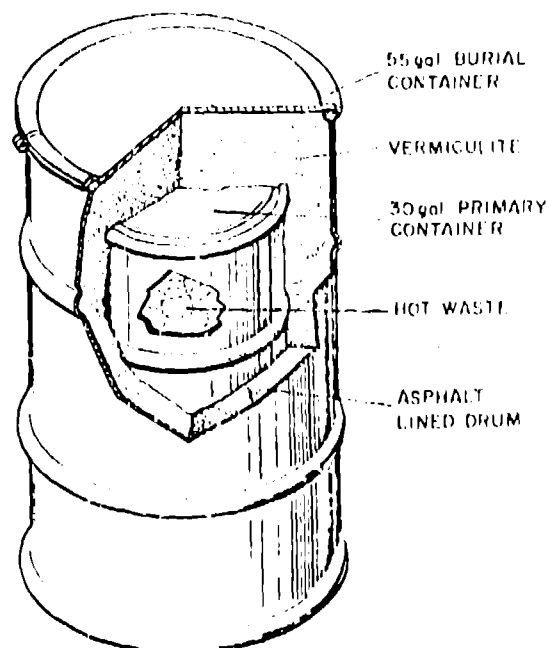


Fig. 2. Past and present burial container

The radiation transfer hazard, dry box packaging storage problems, and the small volume of trash after packaging that could be placed into the 30-gal. primary trash container, were the reasons why a waste disposal system was designed and fabricated for this facility. The requirements of the system included: 1) Sealing the primary container to the dry box floor, 2) Removing the air within the primary container by evacuation and replacing it with inert gas, 3) Sealing the primary container once it is filled within the dry box, and 4) Removing the container without the release of tritium contamination. The problem was solved by utilizing a vacuum closet capable of evacuating both the primary container (PC) and the space between the lid of the PC and the dry box (Fig. 3). The 30 gal. drums were modified by welding a steel flange

near the top of the opening providing an O-ring sealing surface for an aluminum lid that is then bolted to the flange from within the dry box when the PC is full and ready for burial. The lip of the drum is sealed with a neoprene gasket by the force of the screw jack. The PC is now evacuated and backfilled with helium by the ports shown in Fig. 4. The door connecting the PC and the dry box is now opened, the contaminated waste loaded, the lid sealed, the door closed, and the space between the PC and the door evacuated and backfilled with helium. The PC is now ready for removal, the closet door is opened and the drum is removed by lowering the screw jack.

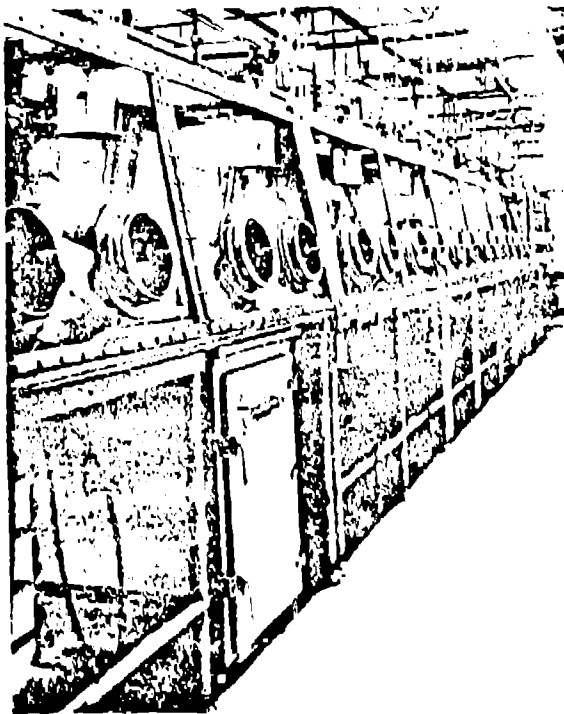


Fig. 3. Waste disposal system under dry box

The new system eliminates the problems encountered during the waste packaging operation in the past, namely the release of tritium gas into the atmosphere, the high risk of personal exposure, and the decontamination and handling problems during the transfer operations. The waste disposal system permits the trash to be buried in an inert atmosphere eliminating air and moisture that may eventually react with any metal tritides in the burial container. This reaction ( $\text{LiT} + \text{H}_2\text{O} \rightarrow \text{LiOH} + \text{HT}$ ) could increase the tritium gas pressure within the burial container increasing the likelihood of a future release of tritium contamination after burial.

#### B. Gas Purification System (GPS)

The GPS consists of six drying towers each containing 2.3 Kg of 5A molecular sieve for water removal and 3.6 Kg of Dow Q-1 for oxygen removal (finely divided copper on an alumina substrate).

The copper reacts with oxygen forming copper oxide, reducing the oxygen concentration in the dry box gas. The molecular sieve removes water from the gas, giving an inert helium atmosphere. The towers will, under normal conditions, maintain the dry box water level below 5 ppm. The gas from the dry box is circulated through the GPS by two 2.8 m<sup>3</sup>/min, Cyclonaire CH-6 blowers operating in parallel. The blowers are enclosed in a sealed stainless steel box which isolate them from the room air.

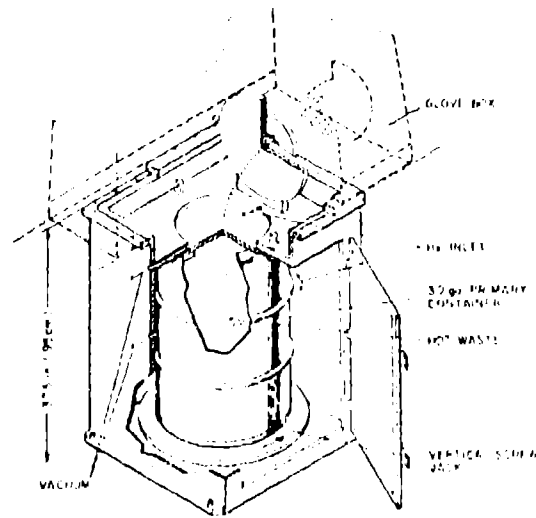


Fig. 4. Schematic of waste disposal system

The two blowers ran continuously for 2.5 years in the dry box atmosphere before they developed bearing problems from the dry atmosphere. The blower box, designed to be removed as a unit, was replaced in less than 2 days. Due to the shutdown the dry box tritium activity, oxygen, and water levels rose to 11.8 Ci/m<sup>3</sup>, 400 ppm, and 70 ppm, respectively. A vacuum line had to be added in order to evacuate the tritium from the old blower box and the air from the new one.

The towers are regenerated in pairs on the average of twice a year by heating them to 570 K and then passing 200 liters of a 94% Ar - 6% H<sub>2</sub> gas mixture through each pair to reduce the copper oxide, followed by a 270 liter helium purge to drive the water into a common 30-gal. collection container. The purged gas after leaving the collection container enters the ETS where it is scrubbed for tritium prior to release to the environment. The average amount of water collected from all six towers after regeneration has been 5.2 liters.

The GPS contains a heated titanium bed for removing nitrogen from the dry box gas. The bed has a separate 16 liter/sec blower that pulls a drag stream off the main circulation system.

This bed can getter tritium, if operated at 780 K, or nitrogen when operated at 1100 K. The bed is continuously kept at 1100 K to remove nitrogen but, at this temperature, it will also remove oxygen. The original nitrogen removal system failed after three years of continuous use and was replaced by newer Stainless Equipment model GPU-20X Inert Gas Purifier. Once the titanium is saturated the bed must be replaced, as the titanium nitride-oxide cannot be regenerated.

### C. Effluent Treatment System (ETS)

The ETS removes tritium by the catalytic reaction of hydrogen isotopes with oxygen, forming water which is then absorbed on a drying tower (Fig. 5). The ETS effectively reduces tritium levels in the process stream to one ppm. The capacity of the ETS is 15 ft<sup>3</sup>/min, but the ETS is designed to operate intermittently depending on the effluent load. The gas from the ETS is dumped into the hood exhaust system where a flow of 17,000 ft<sup>3</sup>/min reduces the tritium concentration to 1 part in 10<sup>9</sup>.

Fig. 5. High pressure half of ETS showing 30-gal. H<sub>2</sub>O collection drum

All components, except the low pressure receiver, are redundant. One is in use and an identical component is on-line in the event of a component failure or during periods of scheduled maintenance. Virtually every component of the ETS can be isolated by valves, and evacuated and back filled with helium. This reduces the probability of exposing personnel to high tritium level gases during maintenance of the ETS. All effluents produced by vacuum systems within the tritium facility and all purge gases used for regeneration of drying towers are processed by the ETS.

All effluents produced by vacuum systems first pass through a series of mist eliminators. These are baffled traps designed to condense oil mist from the vacuum pumps. After passing the

gas through the mist eliminators the effluents pass through an activated charcoal trap to reduce the organics to a minimum. The mist eliminators and the two charcoal traps can be valved off and replaced, but this has not been necessary yet.

Precautions to eliminate hydrocarbons must be taken to avoid certain substances which will permanently poison the catalyst within the recombiner. These substances are acids, chlorides, sulfur compounds, oil, vapors of some organic solvents, and base metals (Ref. 4).

From the charcoal trap the gas is dumped into a low-pressure receiver (LPR), a large 1.9 m<sup>3</sup> storage and ballast tank. The pressure within the LPR is sensed by mercoid switches. At a pressure of 7 psia the mercoid switch activates the Corken Model D390 reciprocating compressor which pulls gas from the LPR through the ETS until the pressure reaches 2 psia and the compressor is deactivated. This requires approximately 5 minutes operating time. The LPR is equipped with a pressure relief valve so if pressure starts to build in the LPR at a rate faster than the compressor can handle, the pressure relief valve will open at 13.5 psia venting the gas directly into the stack and preventing non-processed gas from entering the room. The T<sub>2</sub> concentration of the gas in the LPR depends on the operations performed that day. Concentrations vary from a fraction of a Ci/m<sup>3</sup> to several thousand Ci/m<sup>3</sup>.

From the LPR the process gas enters the recombiner where the gas temperature is increased to 900°F by a gas-to-gas heat exchanger and an electric preheater. The hydrogen isotopes and hydrocarbons are catalytically converted to water vapor and carbon dioxide by reacting with oxygen, and are then cooled by a gas-to-water heat exchanger to 70°F. If only hydrogen isotopes are to be converted to water vapor in the catalytic reactor the gas preheater may be operated at temperatures as low as 350°F. To be sure that all tritiated hydrocarbons are converted to carbon dioxide the gas preheater is maintained at a temperature of 900°F.

Following the recombiner, the gas is pressurized by the Corken compressor to 30 psig in the on-line high pressure receiver (HPR). This pressure is controlled by down stream Fairchild-Hiller back pressure regulating valves set at 30 psig. The spare HPR is always kept at a vacuum in case more storage volume is needed. These tanks can store gas at a pressure of 120 psig. Pressure relief valves are on both the HPR tanks and the compressor, each of which vent back to the LPR if the down stream back-pressure regulating valves fail.

From the HPR the gas flows through one of two 13X molecular sieve filled towers. The other standby tower has been regenerated by heating to 350°F and purging with helium gas. When one tower is brought on-line the other tower starts regenerating automatically if the controls are in the automatic mode. The tower can be regenerated

at a more convenient time by placing the controls in the manual mode. The gas, after passing through the 13X tower, is monitored for water content and tritium concentration. If low water and low tritium concentrations exist the gas is stacked, which is usually the case. High water concentrations  $>50$  ppm result in stack discharges of 1 MPC of tritium ( $5 \mu\text{Ci}/\text{m}^3$ ). Before this concentration of tritium is reached, the gas is automatically routed through a similar set of towers filled with 4A molecular sieve to further reduce the tritiated water content prior to release to the stack. If this fails, the ETS automatically goes into the recycle mode. In this mode the gas is continuously processed through the ETS in a closed loop until a preset activity level has been reached. The activity levels indicated by both the 13X and 4A ion chambers are fed into the logic control and warning system which determines at what activity level the gas is stacked.

1. Logic control and warning system. The main components of the system is a Panametrics Model 2000 6 channel moisture analyzer, a Teledyne Model 326A oxygen analyzer, and a 4-channel tritium activity recording and logic control system (Fig. 6). The moisture analyzer measures the water level in the 13X process stream under normal ETS running conditions, but can be switched to measure the moisture content in the LPR, both HPRs, 4A process stream, and the regeneration gas from the GPS. If the analyzer senses a moisture content  $>50$  ppm exiting from the 13X drying tower and a concentration  $>0.30 \text{ Ci}/\text{m}^3$  from the 13X ion chamber, the effluent gas stream will be routed through the 4A drying tower. On exiting the 4A tower, the tritium concentration is measured again if it is  $<0.30 \text{ Ci}/\text{m}^3$ , the gas is stacked. If it is still high the gas is returned to the LPR for further processing.

At this facility a gas swamping technique is used to improve the effectiveness of the ETS. Prior to 1978, 94% Ar - 6%  $\text{H}_2$  gas was manually introduced into the LPR if tritium levels in the ETS were expected to be high. By increasing the hydrogen concentration before the effluent gas enters the recombiner more tritium in the process stream can be converted to water. To further reduce the tritium concentration in the ETS a metal bellows pump was manually activated that continuously pulled gas from the LPR through the recombiner and back into the LPR. This provided a better cleanup factor than can be achieved with a single pass through the recombiner.

At the present these operations are done automatically by the tritium activity logic control system. When the activity in the 13X ion chamber reaches  $0.12 \text{ Ci}/\text{m}^3$ , 94% Ar - 6%  $\text{H}_2$  is introduced from a 200  $\text{ft}^3$  capacity high pressure tank to raise the internal pressure in the low pressure half of the ETS by two inches of Hg, adding  $\sim 6$  liters of hydrogen to the 2  $\text{m}^3$  volume. Also, the metal bellows pump is automatically activated at this activity level. If the tritium concentration reaches  $0.30 \text{ Ci}/\text{m}^3$  the ETS is put into the recycle mode and warning lights are activated on the console and in the outside hallway. The warning light lets the operator know that a high tritium charge was admitted to the ETS. Once a warning light goes on, it can only be turned off by a reset button. If the ETS activity continues to rise to  $0.54 \text{ Ci}/\text{m}^3$ , indicating tritium is being added to the ETS faster than it can process it, an emergency alarm will sound. When the tritium concentration falls below  $0.54 \text{ Ci}/\text{m}^3$  the audio alarm will cease, but the ETS will remain in recycle until activity drops to  $0.18 \text{ Ci}/\text{m}^3$ . Once the ETS is in recycle, the tritium activity drops rapidly. Typically, it takes 15 minutes for the activity to go from  $0.60 \text{ Ci}/\text{m}^3$  to  $0.18 \text{ Ci}/\text{m}^3$ . From this level the metal bellows pump continues to process the gas until  $0.10 \text{ Ci}/\text{m}^3$  has been reached.

#### D. Conclusion

In the first year of operation of this facility, the total stack release was 341.6 curies, this compares to 69.7 curies in 1979. These low stack releases result from a well designed ETS and the improvements made to it and to the facility. The two main contributing factors are the dry box waste disposal system and the new ETS logic control system.

Factors reflecting low room activity averages of  $1.6 \times 10^{-7} \mu\text{Ci}/\text{ml}$  and floor swipes of  $\sim 500 \text{ dpm}/100 \text{ cm}^2$  can be attributed to the following techniques (which should be required at all future tritium handling facilities) used at DP-Plant: 1) All vacuum pumps that are tritium contaminated should exhaust to sub-atmospheric pressure to prevent contamination from entering the room through faulty vacuum pump seals. 2) Vacuum pump oil and other tritium contaminated liquid waste should be removed by evacuation into absorbent filled containers to prevent personnel

Fig. 6. Logic control and warning system

exposure. Tritium saturation levels in vacuum pump oil can be as high as 30 Ci/liter. 3) Glove ports should be covered when not in use to prevent tritium from permeating through the gloves and into the room. Also, these covers should permit the gloves to be evacuated prior to use.

All tritiated liquid waste from the ETS and GPS is collected in a common 30-gal. collection drum; the same drum design used for the waste disposal system and for all tritiated organic liquids. At the present, the drum is filled with vermiculite to absorb the water, but this material will be changed to 5A molecular sieve in the future to retain more water within the drum. A total of 61.23 Kg of water has been collected and buried with an average tritium concentration of 622 Ci/liter. The collection drum is suspended by a load cell where the weight and rate of water uptake can be easily recorded.

One major weakness present tritium effluent treatment systems have in common is the lack of good methods to determine tritium levels within the components of the system and in the final tritium waste collection container for inventory reasons. Due to the size of most waste containers, calorimetry would be awkward but not impossible for the final collection container.

#### REFERENCES

1. J. L. Anderson, F. A. Damiano, and J. E. Nasise, "Tritium Handling Facilities at the Los Alamos Scientific Laboratory," Proc. of 23rd Conference on Remote Systems Technology, 77 (1970).
2. Dean H. W. Carstens, "A Gas Handling System for Studies of Tritium-Containing Materials," Proc. of 23rd Conference on Remote Systems Technology, 69 (1975).
3. J. G. Monahan, "Materials Compatibility Study of 316 Stainless Steel at the LLNL Tritium Facility," Proc. of 3rd Topical Meeting on the Technology of Controlled Nuclear Fusion, Santa Fe, NM, May 9-11, 1978, Paper P/809, Vol. 2, pp 809-816.
4. Engelhard Industries Div., Engelhard Minerals & Chemicals Corp., "Installation, Operation & Maintenance Instructions Engelhard Catalytic Recombiner Systems," East Newark, NJ (1973).