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# CONTROLLING PARTICULATES, TEMPERATURE, AND TRITIUM IN AN INERT GLOVEBOX FOR A WEAPONS PROGRAM

**Abstract:** A glovebox is described in which several environmental parameters are controlled and monitored. Included in these are particulate, tritium, water vapor, oxygen and temperature. The paper details the design rationale and process and describes the glovebox, presently in use for neutron generator production.

## INTRODUCTION

The production of neutron generators has been performed at a Department of Energy (DOE) contractor-operated facility, the Pinellas Plant, in Largo, Florida since the 1940's. Because of the new world order and the reduced number of nuclear weapons DOE chose to close the Pinellas Plant and transfer their reduced-build function to Sandia National Laboratory (SNL) in Albuquerque, and Los Alamos National Laboratory (LANL) in Los Alamos, New Mexico. The transfer was conducted under the DOE's non nuclear reconfiguration project.

The building of neutron tubes and neutron generators requires a target that is reacted (loaded) with tritium to form a solid compound, erbium tritide. The target is approximately one inch in diameter and is composed of a 10 mil thick molybdenum substrate under a 5000 Å thick evaporated layer of metallic erbium. The target is placed into a loader in which temperatures and pressures are adjusted to cause the reaction of the erbium with tritium gas, creating erbium tritide.

The tritium reaction process to obtain a functional target was considered to be the most critical and most difficult step in the neutron generator production business. Because of chronic deadline requirements, the Pinellas Plant did not optimize the chemical process and consequent conditions for target loading. The DOE decided to separate the target loading function from the rest of the neutron generator business and LANL to work with SNL to produce working generators. This project was a good fit to existing expertise and facilities at LANL and allowed SNL to concentrate on other critical aspects of neutron generator construction.

The loader employed at the Pinellas Plant had a large capacity and was not located within a glovebox. Although the process worked, the part reject rate and waste product was significant due to dust contaminants and excessive oxidation. The project transfer period gave LANL an opportunity to improve the hardware and safety aspects of production. The primary decisions were that:

1) the loader would be downsized and more recent technology would be used, and
2) the loader would be located within a glovebox to control the atmosphere, reduce particulates, and isolate workers from direct tritium exposure.

Although use of the specified tritium gas processing equipment is not new, the circumstances and combination of technologies used for the neutron target loading may be considered novel. For this paper discussion will focus on:

1) improvements in loader hardware which leads to glovebox restraints
2) glovebox design accounting for particulate control, moisture and oxygen control, temperature control, and tritium control and abatement.

A guiding principle behind the achievement of these functional goals was the consideration of the work environment in order to make the process less burdensome.

## PINELLAS PROCESS AND DESIRED CHANGES DURING RECONFIGURATION

The following sections describe the improvements made by LANL to the Pinellas operations of loading tritium onto the targets.

### Increase the Containment Security of the Process Equipment

The general approach at Pinellas to tritium handling was to use fume hoods or to have a ventilating duct near the loader to carry away tritium released during load-in/load-out operations. These practices potentially expose the worker to tritium. This approach is also not consistent with current practices at LANL, so the decision to contain the loader and associated equipment in a monitored glovebox was integral to the LANL design from inception. This was true, in spite of the fact that tritium inventory could be maintained at less than 1000 curies without impacting production rates. Capping tritium inventory at 1000 curies allows the process to be conducted in a radiological area, a much less regulated work environment than a Category II facility. ALARA principles drove the design of the glovebox and operating procedures such that reaching the best clean-up achievable was a prerequisite to opening the load chamber.

### Increase Oxygen and Humidity Control

The Pinellas experience pointed to the desirability of maintaining impurities (oxygen and moisture) to an absolute minimum as a way of improving product quality. The significance of oxygen and water in the system is due to the propensity of erbium to form an oxide film; this slows the tritiding process and necessitates higher activation and loading temperatures. The Pinellas process relied on getters to purify process gas during loading. It was felt that the impurities that drove the process at Pinellas to use high temperatures and purifying getters were introduced during load-in/load-out. Therefore, it was determined to control the atmosphere in the vicinity of the loader in order to reduce oxygen and water vapor contamination of the interior of the loader during transfer operations.

### Introduce Particulate Control System

At Pinellas it was discovered that a major cause of component failure was particulate contamination on the target surface. Particles of 5 *micron* size and larger on the surface were found to cause a high voltage breakdown of the component

Although clean rooms were used at the plant for various manufacturing processes, the environment where target loading took place was not controlled for particulates. At Pinellas, the particulate control consisted of cleaning off the targets with blow-off guns prior to further processing. It was determined that the design of the Los Alamos glovebox should incorporate a "clean" work zone in which targets could be loaded onto fixturing and then loaded into the vacuum system and vice versa. This zone was defined as a Class 10 (no more than 10 *0.5 micron* particles per cubic foot) for airborne particulates. The addition of this controlled environment inside the glovebox would help increase the acceptance rate of processed components.

### Change the High Vacuum Pump to Reduce Tritium Waste

Ion pumps were used extensively on load chambers at Pinellas to achieve oil-free high vacuum prior to processing targets as a means of impurity control. At the conclusion of a tritium load the bulk of the unused tritium was pumped back to the uranium beds and this was followed by several purge/rough-pump cycles to clean up the loader prior to unloading targets. Nevertheless, the tritium pumped by the high vacuum pumps eventually led to pump inventories on the order of 2000 curies. This retention necessitated special recovery procedures to be performed at the end of the pump's life. Based on the Pinellas experience, a design goal of replacing the ion pumps with a pump that did not hold-up tritium was adopted. A related goal was to find a pump that was more compact and simpler to operate than an ion pump.

## IMPLEMENTATION AT LOS ALAMOS & EFFECT ON GLOVEBOX DESIGN

The following sections describe the rationale behind the LANL implementation of a glovebox approach to target loading.

### Glovebox Considerations

The primary functions of the glovebox were to provide secondary containment for the loader system and incorporate all the essential equipment and systems necessary for the processing of targets. This would include equipment for the handling, inspection, and cleaning of targets, hardware for operating the loader system, and equipment necessary for maintaining the desired environment.

The glovebox design had to incorporate all the manual, mechanical, electrical, and gas interface components necessary for interfacing with the loader system, the support systems, facilities, and human operators. It was also essential that the glovebox provide the features needed for the reliable operation of the loader system and efficient processing of targets.

### Glovebox Atmosphere Monitoring and Control

Whereas waste tritium from load operations at Pinellas was stacked, the philosophy at LANL was to contain any waste tritium within the glovebox until the level rose sufficiently to warrant a dry-gas purge. When purging the glovebox to reduce tritium concentration, free tritium and tritiated water vapor should be stripped from the purge gas to microcurie levels before exhausting the purge gas to the atmosphere. Therefore, the design objective for the new facility at Los Alamos was to reduce losses of tritium during production to an absolute minimum, contain the tritium within a glovebox and then, when a glovebox purge was required, to reduce waste tritium releases to the atmosphere to negligible levels.

It was determined that the atmosphere in the glovebox would be nitrogen and that both the oxygen and moisture levels should be maintained under 100 ppm. Tritium containment safety usually requires a negative pressure in the glovebox, but product purity requirements suggested that the glovebox be maintained with a positive pressure. In selecting the operating pressure of the glovebox, these two conflicting requirements were considered. Because the glovebox had a very low leakage rate, it was possible to operate at a negative pressure without compromising particulate control. It was also necessary to constantly monitor the oxygen and moisture levels inside the glovebox.

A system was needed that could perform the following functions:

1) Maintain glovebox pressure,
2) Monitor atmosphere for tritium, oxygen, and moisture,
3) Maintain oxygen and moisture levels below 100 ppm, and
4) Purge glovebox with nitrogen when necessary.

**Particulate Control System**

Two different approaches were examined for implementing a "clean" zone inside the glovebox. One approach was to purchase a HEPA filtered glovebox. This would provide downward laminar flow into a grated floor and recirculated with internal blowers to roof mounted HEPA units via ducting built into the glovebox. The alternative approach was to install a 2'x2' horizontal flow HEPA Integrated Blower Filter (IBF) unit inside the glovebox directly in front of the desired work zone.

The advantages of the filtered glovebox were that the cleanliness level of the entire glovebox would be improved and the filters would not use any floor space within the glovebox. The disadvantages were the cost of the glovebox itself, minor incompatibilities with the vacuum system the glovebox would be housing, the extended delivery time, and the cost of clean up in the event of severe tritium contamination inside the glovebox.

Because components would only be handled in the designated zone within the glovebox, it was determined that the independent horizontal IBF unit would be a superior solution to the problem. However, testing would be necessary in order to design and incorporate a system that would provide the largest clean zone.

It was determined that it would be necessary to monitor the work zone for airborne particulates; it would be beneficial to monitor other locations in the glovebox as well. The HEPA IBF unit would need to be monitored for performance with a velocity sensor in order to detect clogging. The sensors that would be utilized to perform the monitoring functions would have to be compatible with the various equipment located inside the glovebox. Sensor size was a critical issue since space inside a glovebox is limited.

A user-friendly system was desired that could continuously monitor all sensors, display real-time data, and archive the necessary data. These features would allow us to see which processes and actions performed in the glovebox result in increased airborne particulate activity that could possibly cause contamination problems. The system also needed to be expandable for monitoring up to three gloveboxes.

**Temperature Control Within the Glovebox**

Containing the Loader components within a static pressure, impurity and particulate controlled, inert atmosphere glovebox raised concerns about heat generation within the box and it's effect on component reliability and upon operating personnel. It was recognized that the comfort of the operating personnel should be a primary concern. Comfort during extended operations while wearing smocks and Pylox gloves with arms and hands working within glovebox gloves was important because the amount of tritium absorbed through the skin is strongly influenced by perspiration. Also, some of the operations involved in target load-in/load-out are tedious and require special care to prevent particulate contamination of the targets. Reducing both the time required for loading and the temperature of the environment was felt to offer a greater payback in the form of lowered rejection rates than the initial outlay. The goal of controlling temperature was integrated into the design of the new Loader as a consequence of the design goal of glovebox containment.

### Maintenance and Accessibility

Containing the loader system inside a glovebox added a level of complexity to the efficient operation of the system from a production perspective. It was therefore necessary to design features into the loader system and glovebox support systems that would facilitate accessibility and ease of operation and maintenance for system operators. It was also desirable to automate system features where feasible so that operators could activate equipment from outside the glovebox.

## LOS ALAMOS GLOVEBOX SYSTEM DESIGN

The following sections show how the particular technology used in the target loading operations was integrated into the design specifics of the glovebox layout. The sections show how a better environment for loaded target production was achieved at LANL.

### Glovebox Sizing, Configuration, and Features

One of the driving forces behind the size of the glovebox was to keep it as standard as possible in order to minimize the price and delivery time. AutoCAD was used to spatially relate the loader components and then to give a required envelope surrounding the parts so that glovebox procurement drawings could be prepared. It was soon apparent that an extra wide standard glovebox would not quite work, mainly because of a pneumatic actuator on the Loader chamber valve. Rather than adopt a non-standard glovebox width to accommodate the valve, a window frame extender was added to the window adjacent to the actuator. This provided the four extra inches needed without departing from a cross section which is, itself, at the limit for glove access to the entire box volume.

The length of the Loader was found to be longer than two standard sections but three sections were too long for the designated location in the tritium facility. Ten feet was selected as the compromise length and did not greatly affect fabrication cost. Another deviation from a standard glovebox was the addition of a full-length six inch ceiling "tophat" extension. This was necessitated by the need to locate the HEPA filter return duct as well as to find a location to route cables and gas piping. Although it wasn't considered standard, the ceiling extension had been previously incorporated into gloveboxes provided by the manufacturer, and drawings were readily available.

A "working" side was designated to the glovebox, where it was determined that the almost all processing would take place. This included transferring targets in and out of the glovebox, inspection of targets, loading of targets onto fixturing, transferring targets in and out of loader system, and taking gas samples. The glovebox design was configured to include features that would be driven by this working side.

Three windows were designed into the roof of the glovebox directly over the working side. Fluorescent light fixtures were placed over these windows to provide the necessary lighting in the glovebox. The roof also incorporated 22 penetrations with external KF flanges to serve as gas and electrical interfaces between the glovebox, loader system, and support systems. These penetrations were located towards the "non-working" side of the glovebox in order to keep any gas and electrical lines clear of the working area. Two electrical socket feedthroughs were also placed in the roof to provide power to equipment inside the glovebox.

Both a 15" and a 6" pass-thru were placed at the end of the glovebox closest to the working area to facilitate the easy transfer of components and tools from outside the glovebox into the work area and vice versa. A window with two gloveports was placed at the opposite end of the glovebox. Also located at the far end away from the work area were 12 penetrations containing hermetically-sealed electrical cable feedthroughs. These provide the electrical interface between the loader system and support systems inside the glovebox and their control systems located on the outside.

The features of the glovebox included 1/4-20 x 3/4" threaded studs on a uniformly spaced grid that were welded to five of the six internal surfaces of the glovebox. These provided the means for the mounting and fastening of various equipment within the glovebox. Racks for cables and gas lines were mounted in the extended roof section of the glovebox and located on the non-working side of the glovebox. Penetrations were made in the glovebox floor in the necessary locations with flanges to provide the interfaces for the loader system and shipping containers (see Section on Glovebox System Design).

Gloveport placement was standard on most of the Lexan windows except for the window adjacent to the work area. Some windows incorporated four ports to aid in maintenance accessibility. In the window adjacent to the work area, an extended six foot window without gloveports was ordered. The ports for this window were located only after the Loader had been installed and trial operations performed to find the optimum hand positions. 15 mil Hypalon gloves were used to combine both dexterity and tritium safety. The gloves were ordered specially cleaned from the manufacturer to remove surface particulates and prevent contamination within the glovebox. Ambidextrous gloves were used at all non-production specific gloveports.

In order to minimize the tritium exposure to operators when removing components from the glovebox, an exhaust hood was connected to the 15" pass-thru. This hood incorporates a door/window with a gloveport for accessing the pass-thru and a 6" diameter exhaust port in its roof. Finally, the material selected for the glovebox was stainless steel. Although not optimum for tritium retention, stainless is easier to clean and maintain than aluminum.

Transfer of the Loader system into the glovebox consisted of the following. The Loader and it's support equipment were assembled and operated in a portable clean room, using deuterium in place of tritium, prior to being installed in the glovebox. To facilitate the transfer without complete disassembly, the Loader was designed in two units: load chamber, pumps, piping and instrumentation were mounted on a pallet and the valves and uranium beds were installed on a vertical panel. Assembly in the clean room was spatially identical to that in the glovebox. When the transfer was effected, rollers were added to the pallet and the assembly was rolled into the glovebox from the open end. The end used for the transfer had the flanges rolled outward to provide a maximum, smooth, opening and most feedthroughs were located in the ceiling to keep the floor as smooth as possible.

### Glovebox Atmosphere Monitoring and Control

As previously stated, control of oxygen and water vapor was identified as one of the key factors in producing a high quality product. A commercially available Dri-Train system (mfg. by Vacuum Atmospheres Inc.) offered control of these two contaminants to ppb levels. These units also incorporate a glovebox pressure control unit as well as oxygen and moisture sensors needed for monitoring the glovebox.

An additional system was needed that could monitor the glovebox tritium level and purge the glovebox with nitrogen when needed. This led to the design of the Gas Purge and Monitoring System (GPMS). The system circulates glovebox gas through a sensing loop and back to the glovebox using a metal bellows pump. The sensing loop incorporates an Ion Chamber for tritium detection, an oxygen sensor, and a moisture sensor.

A purge loop was designed into the GPMS that can purge the glovebox with nitrogen and also provide an exhaust path to the facility Effluent Treatment System. A purge can be initiated either manually or automatically through the use of a Programmable Logic Controller. The system also incorporates a glovebox pressure controller for when the Dri-Train is not being functioned.

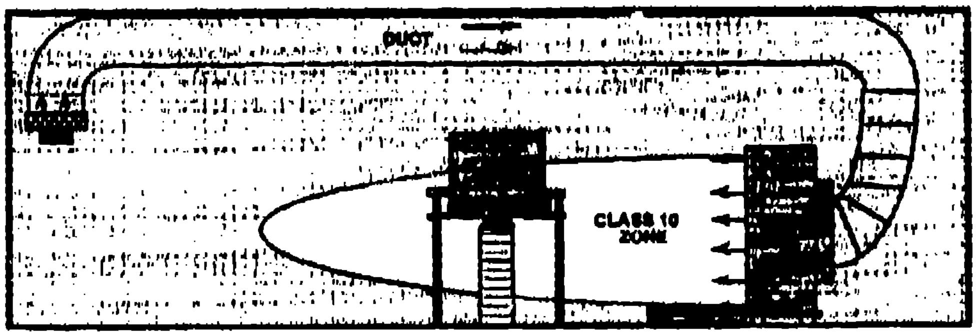
### Particulate Control System

Testing for the clean zone was performed using an IBF unit inside a glovebox in the following configurations:

1) Stand alone unit (no recirculation)
2) Ducting assisted recirculation
3) Ducting with additional blower assisted recirculation.

The largest clean zone was obtained using ducting assisted recirculation. This approach resulted in a design utilizing the IBF unit along with a 9 ft. long 6" diameter recirculation duct running the length of the glovebox. The schematic of this solution in Figure 1 shows the extent of the Class 10 zone.

FIGURE 1
WORKING SIDE VIEW OF NTTL GLOVEBOX

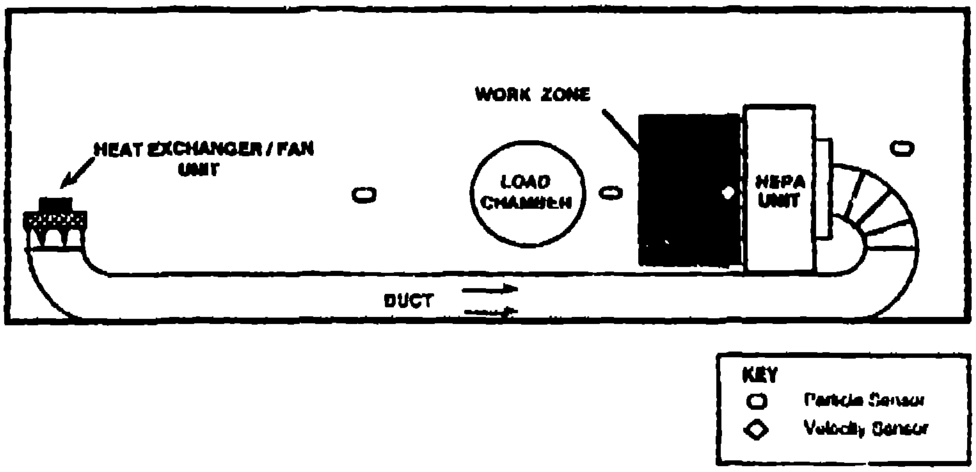


This duct mounts in the overhead tophat section of the glovebox so that it does not interfere with gloves and equipment and does not block any glovebox windows. A flexible duct connects the main duct to an adapter mounted on the rear of the IBF unit. The adapter incorporates a pre-filter that is easily accessible for replacing. The IBF unit is mounted on a rolling shelf that, along with the flex duct, allows for mobility of the unit inside the glovebox.

For the monitoring system, it was decided to use three particle sensors inside the glovebox--one in front of the work zone, one downstream of the load chamber, and one behind the IBF unit near the glovebox pass-thrus. The sensors chosen were Met One Model R4700 Remote Airborne Particle Sensors. The choice of the sensor is sensitive to moisture content becuase some particulate monitoring devices cannot operate in dry environments. The R4700's compact size (3.7" x 2.4" x 1.5"), two size channels (*0.5 and 5.0 micron*), and low cost made the sensor an excellent choice for our glovebox. The velocity sensor chosen was a Sierra Instruments Series 600 Accu-Flo that mounts from the glovebox ceiling at the face of the HEPA filter. The layout of the sensors within the glovebox can be seen in Figure 2.

The monitoring system chosen was a Lighthouse Associates "Lighthouse Monitoring System" (LMS). This is a completely automated system that works with sensors from all major vendors. It utilizes an Engineering Control Station (ECS) which is a finely tuned microcomputer that runs LMS software in Windows to perform various configurations. These include displaying a glovebox map with sensor locations and real-time conditions as well as displaying data charts, graphs, and tables with real-time and historical data.

FIGURE 2
TOP VIEW OF NTTL GLOVEBOX




The system utilizes a System Interface Unit (SIU) which controls the data collection interface to the sensors. The SIU is located in the glovebox so only one RS-232 cable is needed to run outside the glovebox through an electrical feedthrough to the ECS. The system's modular architecture makes it easily expandable for additional gloveboxes and clean rooms.

### Oil-free Pumps With no Tritium Hold-up

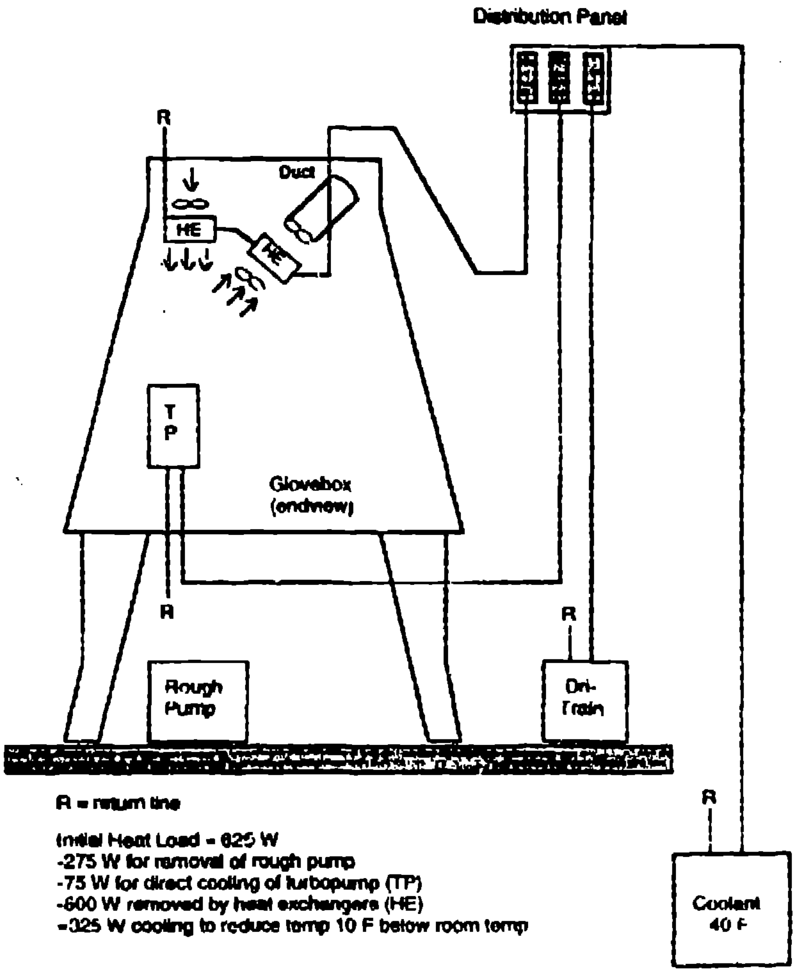
Ion pumps were used for final pump-out of the load chambers at Pinellas because of the desire to control contaminants with an oil-free system. Since the Pinellas plant went into production, other pump types have been developed, notably magnetic-bearing turbomolecular pumps that are completely dry and simple to operate. The turbomolecular pump satisfied the desired glovebox design parameters because it was small, lightweight, easy to operate, clean, and does not hold-up tritium.

### Glovebox Temperature Control

As stated previously, a high temperature glovebox may cause greater skin absorption of tritium due to perspiration on hands and arms and increases the likelihood of operator error. A measured and estimated heat load within the glovebox was first obtained in order to size the cooling needed. The glovebox, with major heat producing components operating, was allowed to equilibrate for several days. At the end of the period, the average box atmosphere temperature was measured to be 90°F. With the temperature difference between the box atmosphere and the room known, the total heat load within the glovebox was calculated to be 625 watts. Next, the manufacturer's rating or the actual current draw of the component was used to estimate the heat generation of the major components. This total was 475 watts; the difference between the measured and calculated heat load was 150 watts and this amount was attributed to all other sources within the box. The goal for removal from the box to achieve room temperature (70°F) became 625 watts.

Several steps were taken to achieve this 625 watt reduction. First, a roughing pump generating 275 watts was removed from the glovebox; this was possible because only trace amounts of tritium were pumped. Second, a turbomolecular pump that generated 75 watts was water-cooled directly thus eliminating that heat load. Finally, two fan-forced tube fin heat exchangers manufactured by Thermatron Engineering, Inc. were installed in the glovebox. Each heat exchanger measures about 6"x 6"x3" and, when supplied with cooled water at 40°F, is capable of 300 watts of cooling. The heat exchangers are located to not interfere with the particle free zone. One heat exchanger is positioned to cool the HEPA filter return gas as it enters the return duct; the second is positioned between the valve bulkhead and the glovebox end opposite the specimen handling zone. The second heat exchanger pulls warm nitrogen from the ceiling of the glovebox and directs the cooled gas downward. The net result of the above changes was a cooling surplus of 325 watts which was sufficient to bring the glovebox down about 10°F below room temperature. Chilled water for the heat exchangers is from a remotely located 2.4 kW chiller. Cooled water is brought to a distribution panel near the glovebox where it is apportioned to the heat exchangers, turbopump and dry-train. Figure 3 shows the layout of this plan.

FIGURE 3
GLOVEBOX TEMPERATURE CONTROL

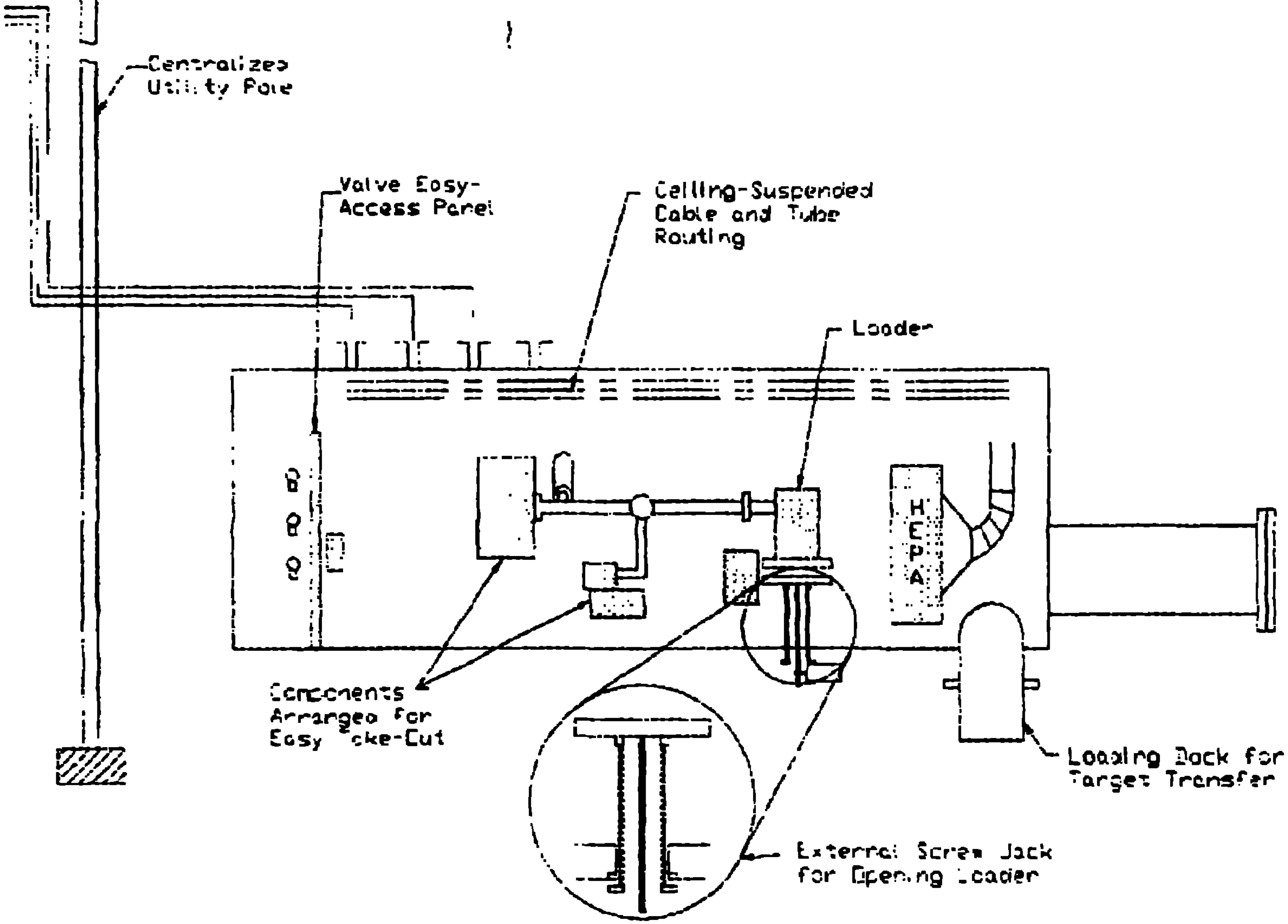



## Maintenance and Accessibility

Several items in the loader glovebox were assumed to require periodic attention and were located for easy inspection and replacement. Over 20 metal sealed pneumatic bellows valves are used to support the loader process. These valves all utilize solenoid valves and are actuated from a control panel located outside the glovebox. This helps minimize "hands in the box" time for operators. Most of these valves and all solenoid valves are placed on a vertical panel parallel to the end closure opposite the specimen handling zone. The end closure is a window and is fitted with gloves which are within easy reach of all valves.

The loader chamber is also easily accessed by gloves and features a two bolt chain clamp for easy opening and closing of the eight inch flange. The chain clamp is supported by a special fixture that carries the clamp weight and centers the clamp around the flange. When the clamp is clear of the flange, the chamber is opened by lowering the flange that supports the target fixture and heater. This lowering and raising of the loader lower flange is effected by a screw jack external to the glovebox.

FIGURE 4
MAINTENANCE AND ACCESSIBILITY



The seal between the glovebox and the screw jack is a metal bellows which seals the glovebox yet allows the lower flange 12 inches of vertical travel.

All utilities supporting the glovebox are mounted on a single "utility tree" at one corner of the glovebox and are routed from there to appropriate penetrations in the floor and ceiling of the box. A special passbox was placed in the floor of the box near the loader and sized for the target carrying fixture. This permits target transfer to and from the loader chamber without exposure to room air. The outer target transfer container mounts to a flange at this passbox and is not subject to external tritium contamination. Major overhaul of loader components will be performed in an adjacent fume hood after the component has been exchanged for a spare. A schematic of the general layout related to maintenance and accessibility can be found in Figure 4.

## CONCLUSIONS

Transferring Neutron Tube Target Loading operations to LANL resulted in superior tritium loading of targets. Instead of using a fume hood during load-in/load-out operations, all of the operations involving tritium were enclosed in a glovebox. The glovebox minimized worker and environmental tritium exposure and its design allowed for the efficient operation of the loader system and processing of targets.

The environment that the glovebox offered for loading was one with oxygen and moisture controlled to ppb levels by a dry-train. The combination of a very clean glovebox atmosphere plus passivated vacuum hardware offered the possibility of refining the loading parameters to improve the product; this eventually proved correct. Maintaining this atmosphere also precluded the need for vacuum baking the loader vacuum components after each transfer which, itself, might adversely affect the targets.

Particulates were controlled to a Class 10 level in the work zone with a horizontal flow HEPA Integrated Blower Filter and recirculation system. A user-friendly modular monitoring system was also incorporated in the glovebox to monitor for airborne particulate and velocity.

A state of the art turbomolecular pump was used in the glovebox instead of an ion pump previously used at Pinellas such that pump inventories of tritium were reduced substantially. In addition, space was optimized by using the turbopump because it was smaller than the ion pump.

A final parameter controlled in the glovebox was its temperature. Arrangement of components required for loading and the use of heat exchangers led to a decrease in temperature inside the glovebox from 90°F to 10°F below room temperature.

The combination of these technologies as a state of the art upgrade for a reduced volume production of weapons components resulted in an improved tritium loading scenario. Safety of the process, state of the working environment, and quality of the product were all enhanced.