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**Modification and Testing of the Sandia
Laboratories Livermore Tritium
Decontamination Systems**

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Sandia Laboratories

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MODIFICATION AND TESTING OF THE SANDIA LABORATORIES LIVERMORE TRITIUM DECONTAMINATION SYSTEMS

Introduction

Sandia Laboratories, Livermore, has put into operation a new facility, the Tritium Research Laboratory (TRL). The laboratory (Figure 1), designed for a wide range of experiments using multi-gram amounts of tritium, entered its start-up phase on October 1, 1977. In addition to the usual pressure-gradient-controlled once-through ventilation system, the Tritium Research Laboratory provides for both personnel safety and environmental protection by employing a glove box secondary containment system connected to the two central decontamination systems, the Gas Purification System (GPS) and the Vacuum Effluent Recovery System (VERS).

This paper describes the glove boxes and operating principles of the GPS and VERS. It gives their performance specifications, and details Sandia's modifications to the manufacturer's design to enhance the safety of maintenance operations. Performance tests, preoperational and with tritium, are also discussed.

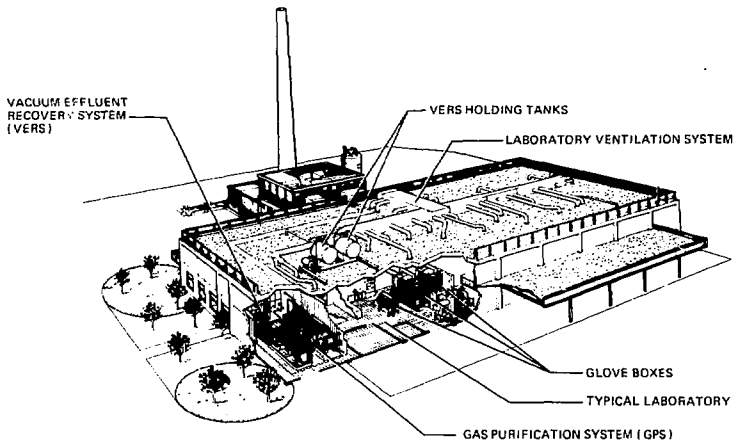


Figure 1 The TRL building at Livermore, showing the arrangement of some of the major systems and a typical experimental station.

Containment

All experiments are secondarily contained in sealed stainless steel glove boxes of welded construction (Figure 2). Each box is equipped with glove ports, viewing windows, and an air lock pass-through; and on each end there are removable panels for the installation of large items. Both regular utility and emergency electrical power are provided to the box. Also, there are feed-through provisions for instrumentation and inert gas pressure connections, and a cooling system to remove the heat generated by experiments. Both tritium concentration and humidity control are maintained by processing the glove box atmosphere through the GPS. Normally, the glove boxes are operated with dry nitrogen maintained by the box pressure control system at a pressure of -0.25 to -1.0 kPa with respect to the room. However, the box can be operated with an argon or air atmosphere if desired.

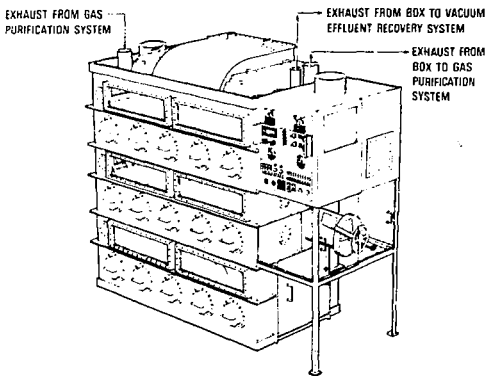


Figure 2 Sealed glove box.

Decontamination

Decontamination is accomplished by two centralized systems. (1) The first of these, the Gas Purification System (GPS), removes tritium and tritiated water vapor from the glove box atmospheres. The second, the Vacuum Effluent Recovery System (VERS), removes tritium, tritiated water vapor, and tritiated hydrocarbons from gases exhausted from the glove box pressure control systems and laboratory vacuum systems before the decontaminated residue gases are vented to the stack. The original systems were manufactured to a Sandia performance specification by Engelhard Industries Systems Department, Union, New Jersey. Both systems were designed to reduce tritium concentrations to a few parts per billion. The tritium removed by the decontamination systems is contained, either for recovery or for disposal, as solid waste on Type 4A molecular sieves.

Gas Purification System

Design and Operation

The Gas Purification System (GPS), conceptually illustrated in Figure 3, removes tritium from the glove box atmosphere in the event of either an accidental release or a slow buildup of background concentration.

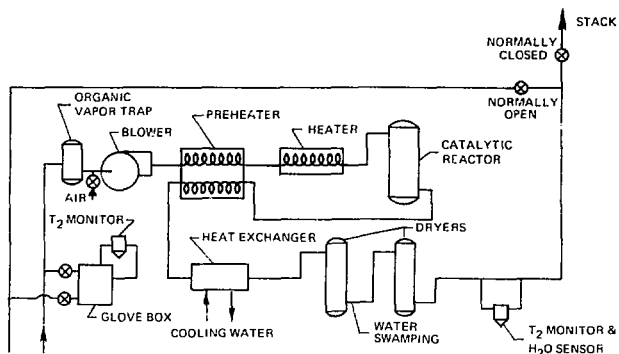


Figure 3 Gas purification system schematic.

The GPS consists of a central manifold connected to each of the laboratory glove boxes, a catalytic reactor to oxidize the tritium, two molecular sieve dryers in series to collect the tritiated water, a blower to circulate the glove box atmosphere through the system, and a control and diagnostics system to provide both automatic and manual control and to assess operational status. Redundant prime function components, i. e., blowers, heaters, and catalyst and dryer beds are provided for reliability. A dryer regeneration system provides for removal of water from a loaded dryer while the GPS is operating with the use of the other two dryers. Entry and exit nozzles allow safe removal and replacement of both catalyst and sieve material while the system remains operational by the use of a redundant flow path. Major system performance criteria are summarized in Table I.

The GPS can be operated either in a recirculation mode or in a stacking mode. In the recirculation mode, the normal method of operation, the glove box gases are pumped from the box, through the GPS, and back to the box until tritium contamination is reduced to an acceptable level. The stacking mode is similar to the recirculation mode with the exception that the glove box atmosphere is not recirculated but stacked after passing through the GPS. This is accomplished by drawing clean box atmosphere gas into the manifold ahead of the glove box and venting the GPS effluent to the ventilation exhaust. This mode of operation may

Table I GPS performance criteria.

Processing Capacity	340 m ³ hr ⁻¹
Catalytic Reactor	
Operating Temperature	783 K
Standby Temperature	783 K
Catalyst	Engelhard Minerals and Chemicals No. A-16648
Tritium Concentration Reduction Factor*	1000 per pass for concentrations from 20,000 ppm to 1 ppm.
Molecular Sieve Dryer	
Capacity	37 moles of water while maintaining less than 1 ppm water at exit
Regeneration Time	6 hours
Regeneration Temperature	589 K
Molecular Sieve	Type 4A
Startup Operating Time	30 s
Glove Box Pressure Control	-0.25 to -1.0 kPa relative to the laboratory
System Leak Rate	1 x 10 ⁻¹⁰ m ³ (STP)s ⁻¹ maximum helium at 98 kPa differential
*Ratio of inlet to outlet tritium concentrations.	

be used to backfill the glove box with dry nitrogen or argon when an inert atmosphere is required. It can also be used to drive the glove box concentration to low levels when it is necessary to clean up a box for reentry.

GPS Modifications

The GPS as originally received from the manufacturer is shown in Figures 4 and 5.

As a result of experience gained during preoperational testing of the GPS in the TRL, it became apparent that system changes were required primarily to increase the safety of maintenance operations. Modifications were necessary to reduce to a minimum the volume of the GPS opened up to the room during such operations as valve seat, heater, catalyst, and sieve removal and replacement. The diagnostics and control systems were also modified at this time. To achieve

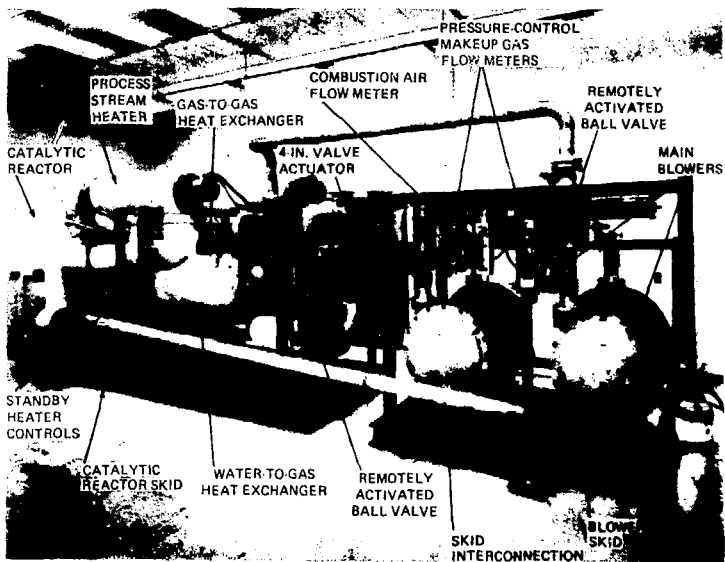
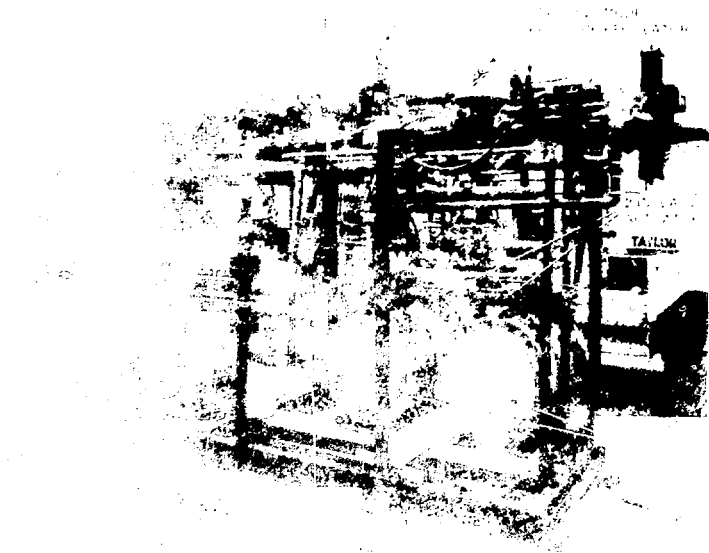


Figure 4 GPS blower and catalytic reactor skids, as received from manufacturer.



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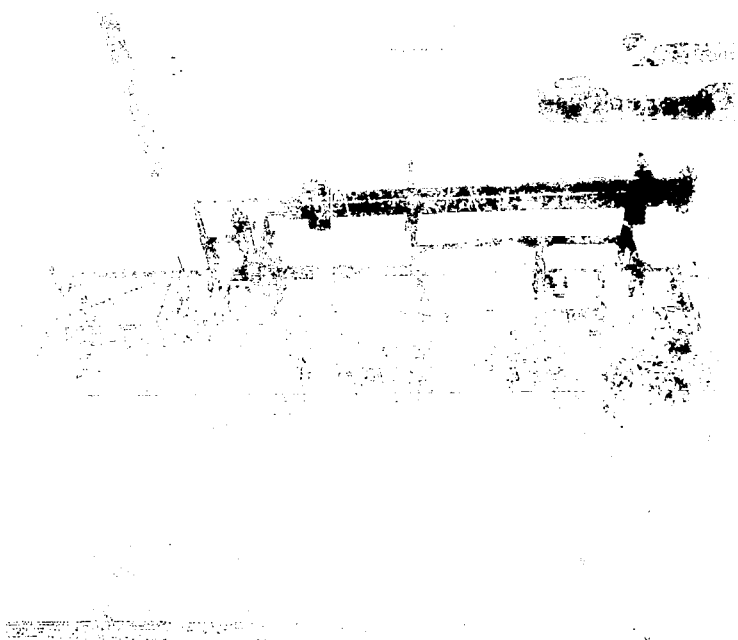
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Figure 7 Modified GPS catalytic reactor skid.



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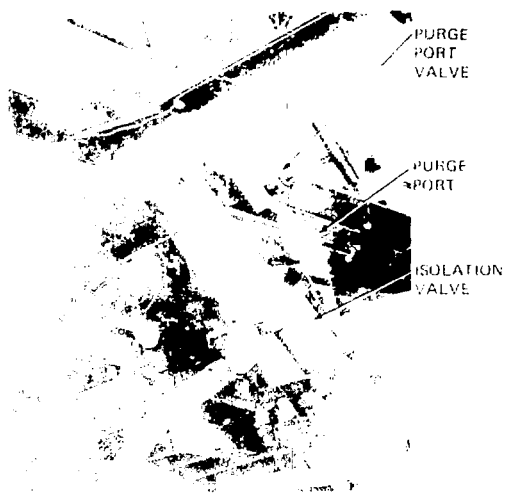


Figure 9. Open full nozzle purge port configuration.

Instrument and Control System Modifications. To enhance both reliability and ease of maintenance, the diagnostics and control systems were changed as follows:

1. The mechanical relay control system was replaced by a microprocessor controlled solid state system to provide increased flexibility and reliability.
2. All heater, motor, and valve control elements except disconnects were removed from the skids and placed outside the hood surrounding the GPS to allow ease of maintenance.
3. The Varian standby heater controls were replaced with silicone controlled rectifier units to achieve better temperature control.
4. Thermocouples were placed in the catalyst bed to provide more accurate temperature control and allow assessment of catalyst performance. Thermocouples were also added at the dryer inlet to provide more accurate determination of regeneration completion.

The GPS control panel in the TRU control room is shown in Figure 10; the wall mounted GPS combustion, air and makeup gas flow instrumentation and control valves are shown in Figure 11. The catalytic reactor standby heater and thermocouple mounting arrangements are presented in Figure 12.

GPS CONTROL PANEL

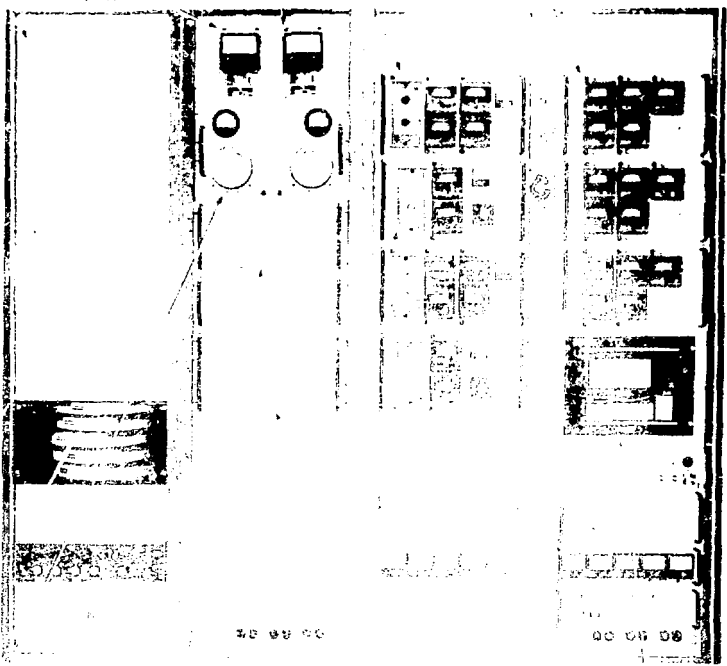


Figure 10 GPS control panel.

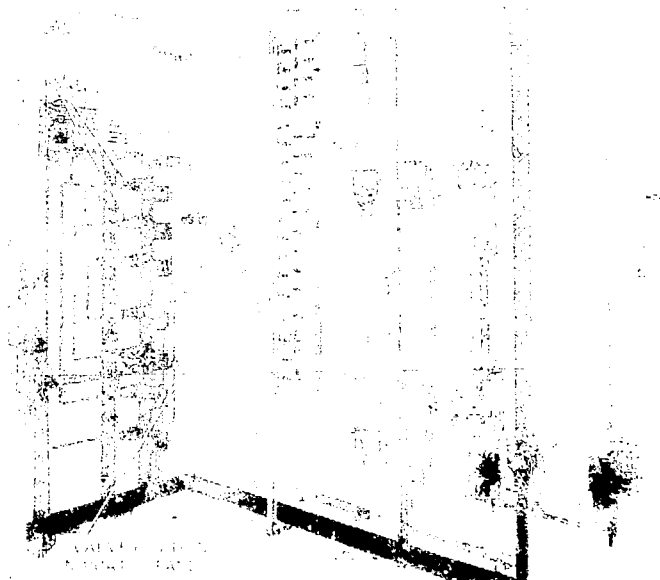


Figure 11 GPS gas flow instrumentation and control valves.

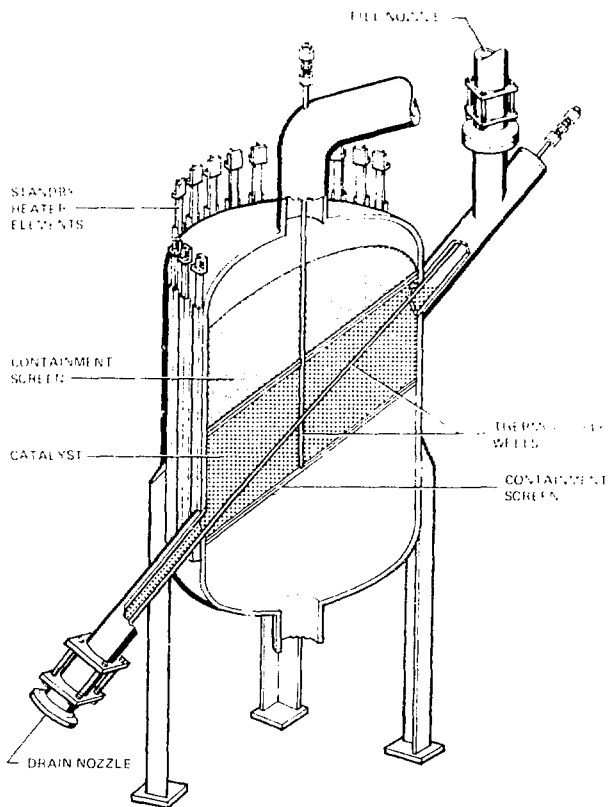


Figure 12 GFS catalytic reactor.

Vacuum Effluent Recovery System

Design and Operation

The VERS, conceptually shown in Figure 13, is used to remove tritium from the exhaust gases of the laboratory vacuum systems before venting to the stack. The VERS removes tritium in the same manner as the GPS and uses the same precious metal catalyst. The system consists primarily of a laboratory vacuum manifold and two holding tanks to collect the contaminated waste gases, a catalytic reactor to oxidize the tritium, two molecular sieve dryers connected in series to collect the tritiated water, the necessary pumps to evacuate the laboratory manifold and transfer the waste gas through the system, and a control and diagnostics section to provide both automatic and manual operation and to assess operational status. That portion of the VERS containing the heater, catalytic reactor, heat exchanger, dryers, and transfer pump is referred to as the "decontamination section."

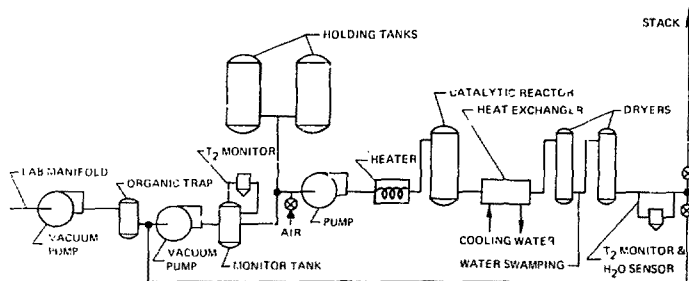


Figure 13 Vacuum effluent recovery system schematic.

The VERS catalytic reactor like that of the GPS is maintained at an elevated temperature (783 K) in standby condition to ensure combustion of tritiated hydrocarbons and readiness to begin processing upon receipt of a start signal. Because holding tank capacity provides adequate time for most maintenance operations, redundant components are limited to the vacuum and gas transfer pumps. Major system performance criteria are summarized in Table II.

The VERS is normally operated in a batch processing mode. The laboratory vacuum manifold is maintained at an average pressure of approximately 3.3 kPa. The effluent exhausted to the manifold is pumped into a 0.3 m³ tank, where its contamination level is monitored. To minimize VERS operating time, the effluent of the 0.3 m³ tank is divided into two levels of tritium concentration which are collected in separate 5.7 m³ holding tanks. The lower concentration is selected such that effluent can be vented directly to the stack while the effluent with the higher tritium concentration is decontaminated before stacking.

Effluent is collected until the holding tank pressure reaches 86 kPa. The decision to stack the holding tank contents is made by control room personnel and

Table II VERS performance criteria.

Processing Capacity	18 m ³ hr ⁻¹
Holding Capacity	9.5 m ³ at 86 kPa
Catalytic Reactor	
Operating Temperature	783 K
Standby Temperature	783 K
Catalyst	Engelhard Minerals and Chemicals No. A-16648
Tritium Concentration Reduction Factor*	1000 per pass for concentration from 20,000 ppm to 1 ppm
Molecular Sieve Dryer	Will use GPS dryers
Molecular Sieve	Type 4A
System Operating Pressure	13 to 86 kPa
System Leak Rate	1 x 10 ⁻¹⁰ m ³ (STP)s ⁻¹ maximum helium at 98 kPa differential

*Ratio of inlet to outlet tritium concentrations.

must be initiated manually. The decision to process the holding tank contents through the decontamination section is automatically made by the VERS control system. The tritium monitor at the exit of the decontamination section generates a signal, depending upon the tritium concentration, either to recirculate the effluent through the VERS or to direct it to the stack.

When a holding tank is to be processed through the decontamination section, the VERS control system alerts the control room by means of an alarm light and begins circulation of the holding tank contents through the decontamination section. When the holding tank pressure is reduced below 13 kPa, the system shuts down. The VERS has the capability for being operated in a continuous stacking and processing mode. However, the decision to use this mode will depend upon operational experience still to be gained.

VERS Modifications

The decontamination section of the VERS is shown in Figure 14 as originally received from the manufacturer.

With the exception of the decontamination section, the VERS was designed and fabricated by Sandia. A major task was to integrate the decontamination section into the rest of the system. Because of space limitations, the manufacturer's skid was disassembled and the major components, with the exception of the catalytic reactor, were mounted on the Sandia skid. A new catalytic reactor was built to provide a larger flow capacity and catalyst entry, and exit nozzles

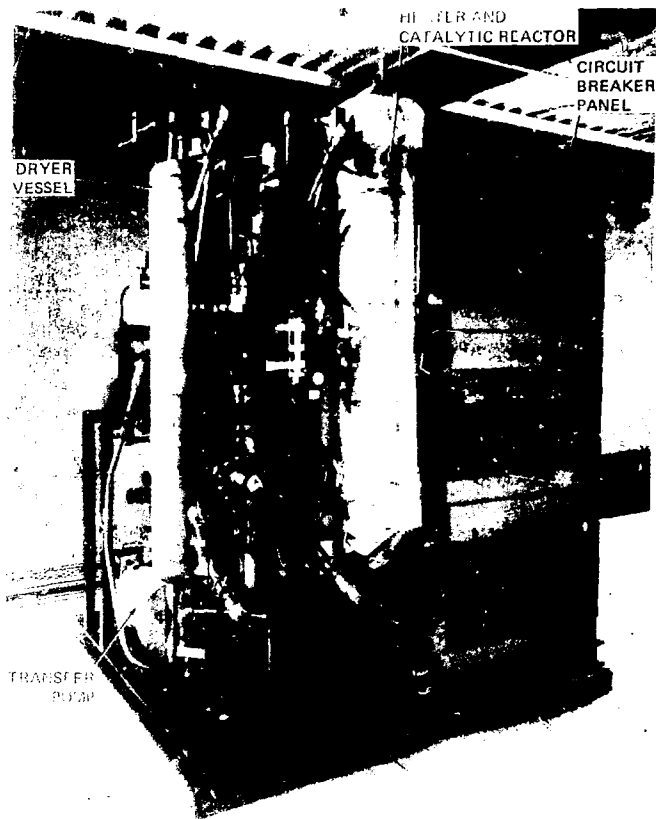


Figure 14 VERS decontamination section as received from manufacturer.

were relocated to facilitate catalyst addition and removal. The VERS exhaust was interconnected to the GPS so that GPS dryers could be used for water collection.

Where possible the control system elements used for acceptance testing of the skid were integrated with the microprocessor controlled solid state system designed by Sandia. As with the GPS, all of the control elements except disconnects were removed from the skid and mounted outside the air hood. The heater control was changed to a silicone controlled rectifier unit, and thermocouples were placed in the catalytic reactor to allow for more accurate temperature control and to provide a means for monitoring catalyst performance. The final VERS skid configuration is shown in Figure 15, and the control panel located in the TRL control room is shown in Figure 16.

Test Program

The GPS and VERS were subjected to acceptance tests at the manufacturer's plant, and preoperational and tritium tests in the TRL. Acceptance testing at the manufacturer's plant verified the ability of the two systems to meet the performance criteria required by the specification (Tables I and II), with the exception of the concentration reduction factors, which required tritium testing for verification. Hydrogen was combusted as part of the acceptance testing to demonstrate that the catalyst was operating and to provide a source of moisture for loading the first dryer. The swamping system was used to load the second and third dryers.

Preoperational testing of the systems in the TRL included essentially a repeat of the manufacturer's acceptance tests. These tests demonstrated that the systems installed in the TRL met the processing capacity, pressure control, and startup operating time, etc., specifications in the actual laboratory operating configuration. The preoperational testing also included a series of catalyst performance tests with hydrogen and methane.⁽²⁾ These tests were run to provide confidence that the catalytic reactors in both systems were operating properly before tritium was introduced and also to determine the appropriate catalyst operating temperature.

The final series of tests was made with tritium and tritiated methane⁽²⁾ to determine that the systems were capable of achieving the design requirement of a single-pass concentration reduction factor (ratio of inlet to exhaust concentration) of 1000 per pass for inlet concentrations of 1 part per million. Sensitivity limitations of gas chromatography required that this confirmation be made with tritium as the test gas. Tests were run with methane and tritiated methane because tritiated hydrocarbons are expected to be present in both systems,⁽³⁾ particularly in the VERS.

GPS and VERS Hydrogen and Methane Tests

The GPS tests were performed first. Approximately ten runs were made to assess the system operation before the parameters were selected for tests measuring temperature effects upon catalyst performance. The VERS test parameters were selected as a result of this experience. The test parameters used are



Figure 15 VERS skid final configuration.

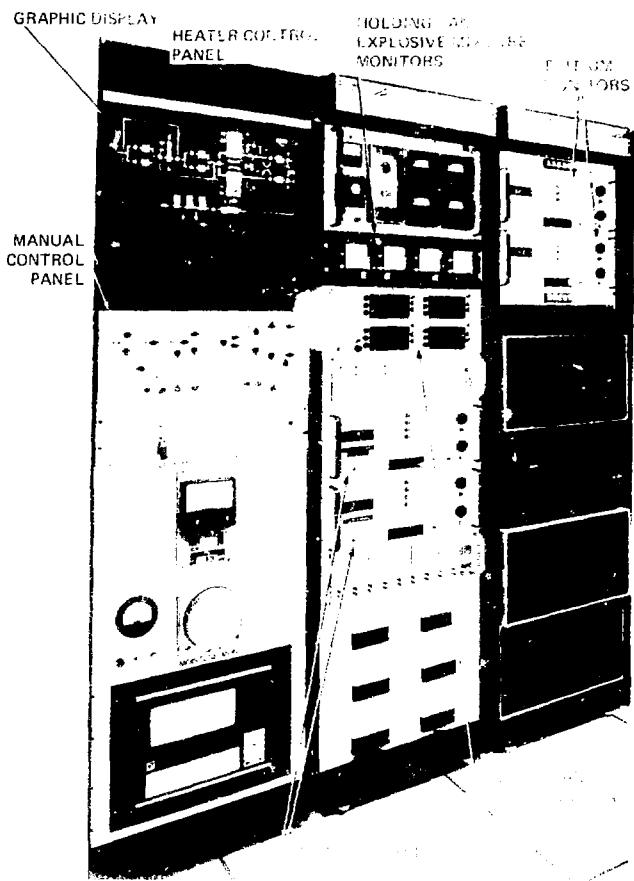


Figure 16 VERS control panel.

summarized in Table III. Inlet concentrations were chosen to provide a reasonable range of detection for the gas chromatograph.

Table III Hydrogen and methane test parameters.

System	Test Gas	Flow Rate (std m ³ hr ⁻¹)	Inlet Concentration (ppm)	Catalyst Temperature (°K)
GPS	Hydrogen	340	120	316-743
GPS	Methane	261	700	316-810
VERS	Hydrogen	16.4	100	302
VERS	Methane	13.3	2000	302-810

The test gases along with nitrogen and combustion air were injected upstream of the catalytic reactor, and inlet and exhaust concentrations were measured with a gas chromatograph upstream and downstream of the catalytic reactor, respectively. Combustion air was injected in excess of stoichiometric requirements. The results of the GPS catalyst temperature tests are displayed in Figure 17 for both hydrogen and methane. Temperatures below 316 K were not achievable because of the heat of compression generated by the circulation blowers

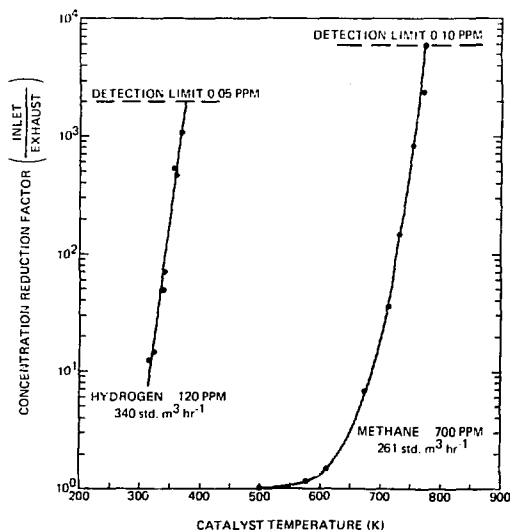


Figure 17 Effect of GPS catalyst temperature on concentration reduction factor.

A glove box cleanup test was also run with methane to determine the rate at which a contaminant could be removed from the glove box atmosphere. Methane was injected into a nitrogen filled glove box until an initial concentration of 500 ppm was achieved. The box atmosphere was then processed by the GPS. The results are presented in Figure 18.

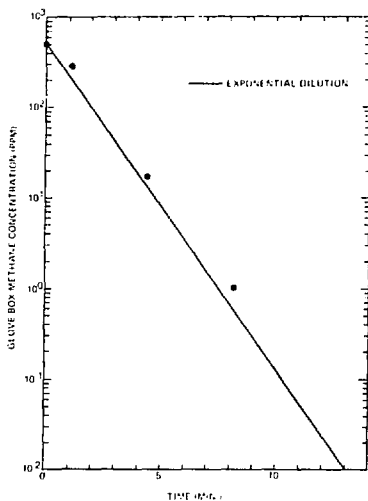


Figure 18 Glove box cleanup test results.

The VERS test results, though similar, cannot be directly compared to those for the GPS since the catalyst residence time for the VERS is approximately 2.5 times that for the GPS. At 320 K, the lowest temperature achievable, the VERS exhaust hydrogen concentration was below the detection limit of the chromatograph, 0.1 ppm. The results of the methane test were essentially the same as for the GPS (Figure 16) and show that to achieve concentration reduction factors greater than 1000, the catalyst must be operated at temperatures in excess of 750 K for methane.

GPS and VERS Tritium Tests

After the preoperational tests and GPS system modifications had been completed, both systems were tested with tritium to verify their ability to achieve concentration reduction factors of 1000 per pass at inlet concentrations of 1 ppm.

Four tritium tests were run on the GPS at a catalyst temperature of 783 K and a flow rate of 340 std m³ hr⁻¹. Seven tests, four with tritium and three with tritiated methane, were run on the VERS at a catalyst temperature of 783 K and a flow rate of 17.0 std m³ hr⁻¹. Hydrogen was added to some test runs to simulate tritium processing at higher concentrations. All of the tests were run without water added ahead of the second dryer. The GPS test gases were injected into a sealed glove box while the VERS test gases were injected into one of the 5.7 m³ holding tanks and then processed through the system. The test results are summarized in Tables IV and V for the GPS and VERS, respectively. Concentration reduction factors were calculated by dividing the maximum inlet tritium concentration by the maximum exhaust tritium concentration.

Table IV GPS tritium test summary.

Test Designation	Sample Composition	Inlet Tritium Concentration Ci m ⁻³	Exhaust Tritium Concentration μCi m ⁻³	Concentration Reduction Factor
GPS T-1	T ₂ in Nitrogen	0.14	2.0	7.0 × 10 ⁴
GPS T-2	T ₂ in Nitrogen	1.3	15.0	8.7 × 10 ⁴
GPS T-3	T ₂ & 2.0% H ₂ in Nitrogen	9.4	1020.0	9.2 × 10 ³
GPS T-4	T ₂ in Nitrogen	115.0	250.0	4.6 × 10 ⁵

Table V VERS tritium test summary.

Test Designation	Sample Composition	Inlet Tritium Concentration Ci m ⁻³	Exhaust Tritium Concentration ^a μCi m ⁻³	Concentration Reduction Factor
VERS T-1	T ₂ in Nitrogen	0.18	≈ 1.0	≈ 1.8 × 10 ⁵
VERS T-2	T ₂ in Nitrogen	0.18	≈ 1.0	≈ 1.8 × 10 ⁵
VERS CH ₃ T-1	CH ₃ T in Nitrogen	0.18	≈ 1.0	≈ 1.8 × 10 ⁵
VERS CH ₃ T-2	CH ₃ T in Nitrogen	1.8	≈ 1.0	≈ 1.8 × 10 ⁶
VERS CH ₃ T-4	CH ₃ T & 0.5% H ₂ in Nitrogen	7.4	≈ 1.0	≈ 7.0 × 10 ⁶
VERS T-5	T ₂ & 0.5% H ₂ in Nitrogen	13.2	≈ 1.0	≈ 1.3 × 10 ⁷
VERS T-6	T ₂ & 2.0% H ₂ in Nitrogen	132.0	≈ 1.0	≈ 1.3 × 10 ⁸

^a1.0 μCi m⁻³ is the least count of the tritium monitor.

An uncertainty of approximately 30 percent should be applied to the GPS concentration reduction factors because of inaccuracies both with tritium measurements and data acquisition methods.

An uncertainty of approximately 50 percent should be applied to the VERS concentration reduction factors because of inaccuracies with tritium measurements at the $1 \mu\text{Ci m}^{-3}$ level of concentration.

Summary

The concept of providing both personnel safety and environmental protection from tritium on a laboratory-wide basis by employing a secondary containment system of sealed glove boxes connected to two central decontamination systems has been implemented by Sandia Laboratories Livermore. Acceptance tests at the manufacturer's plant and preoperational tests in the Tritium Research Laboratory have demonstrated that the systems meet their design specifications and that glove box cleanup rates approaching exponential dilution can be achieved.

The tritium test program to date has demonstrated that both the Gas Purification System and Vacuum Effluent Recovery System perform 10-1000 times better than required by the design specifications, and that tritium removal systems can be designed to achieve concentration reduction factors much in excess of 1000 per pass at inlet concentrations of 1 part per million or less for tritium and for tritiated methane.

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