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TITLE MOLECULAR SIEVE REGENERATION SYSTEM (MSRS)

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SUMMARY

MOLECULAR SIEVE REGENERATION SYSTEM (MSRS)

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ABSTRACT

A Molecular Sieve Regeneration System (MSRS) was added to the existing Tritium Waste Treatment system (TWT) within the Tritium Systems Test Assembly (TSTA) at Los Alamos National Laboratory. The Department of Energy (DOE) no longer allows "inventory by difference" for radioactive wastes that are to be buried. The MSRS was designed and built to comply with this requirement. Within the TWT, water is generated by the catalytic conversion of hydrogen isotopes and removed by molecular sieve trapping prior to release to the environment. Molecular sieve regeneration is required to remove the trapped water and to rejuvenate the beds. The MSRS permits the collection and direct tritium assay of regenerated tritiated water from molecular sieve beds. This paper describes the MSRS in detail and how it is interfaced with the TWT.

INTRODUCTION

The TWT is a tritium removal system controlled by the TSTA computer, a Master Data Acquisition and Control (MDAC) system. Within the Tritium Waste Treatment system (TWT), hydrogen isotopes including tritium and tritium bearing organic compounds are converted to water and carbon dioxide by the catalytic conversion with oxygen. Water generated in this system is removed from the effluent stream by molecular sieve trapping, therefore, molecular sieve regeneration is required to remove the trapped water from the beds to rejuvenate them. The TWT has four molecular sieve beds, two that contain type 13X sieve and two that contain type 4A sieve. When one of the four molecular sieve beds "breaks through" or becomes saturated it is taken off-line and regenerated. Regeneration of each bed separately is required with the existing TWT configuration. In the past this was accomplished by heating a nitrogen gas stream; passing the hot gas through a selected bed; trapping the regenerated moisture with a moisture collector filled with molecular sieve, and finally recycling the exit gas to the inlet of the TWT. Tritium assay of the moisture collector was determined by difference. The radiation leaving the TWT was integrated and subtracted from the integrated radiation entering the TWT for the time interval the drying bed was on line. Inaccuracies resulting in inventory discrepancies are possible using this method. The Department of Energy (DOE) no longer allows "inventory by

difference" for radioactive wastes that are to be buried. The MSRS is designed and built to comply with this requirement.

Prior to the MSRS molecular sieve regeneration required 190 liters/min of nitrogen gas for up to 16 hours that added to the load on the TWT. Nitrogen is supplied from a large liquid nitrogen dewar located on the roof. Moisture content in the nitrogen is assumed to be low and has not caused problems, however, it is not measured. To reduce this gaseous nitrogen load and to provide a known low dew point regeneration gas for the MSRS a closed-loop design was selected.

SYSTEM DESCRIPTION

The MSRS permits direct tritium assay of molecular sieve bed regenerated water. This is accomplished by two techniques, liquid scintillation counting and calorimetry. The system is controlled by the TSTA computer and provides the following four functions:

Loop Flow - To reduce the gas loading to the TWT, recover the water from the molecular sieve beds (MSB), and to provide a low dew point gas for MSB regeneration.

Hold-Up - To contain and measure the amount of recovered tritiated water prior to assay.

Assay - To transfer recovered tritiated water to sample containers for safe assay operations.

Collection - To safely transfer the collected recovered tritiated water to burial containers.

Loop Flow

With the MSRS molecular sieve bed regeneration is initiated from the Man Machine Interface (MMI). When the MMI is commanded to regenerate a selected bed the following operations are performed by MDAC (1) the liquid holding tank pressure transducer is calibrated; (2) the MSRS valves for loop-flow are configured; (3) the HTO freezer is filled and maintained with liquid nitrogen; (4) the heat exchanger chiller is started; (5) the selected bed heater is activated; (6) one of the nitrogen circulation pumps is turned on, and (7) the MSRS loop is pressurized with nitrogen to 4.76 atm (70 psia).

During loop-flow nitrogen gas is heated by the selected bed 10,000 watt resistance heater. Loop-flow and total flow entering the heater are measured with a 0-4.7 liters/sec (0-10 SCFM) mass flowmeter. The heated gas passes through the MSB in a counter-flow direction to the normal process flow. A two-phase mixture of steam and water at an average temperature of 380 K exits the bed and enters a horizontal condensing U-tube type heat exchanger cooled with a 25 wt. percent ethylene glycol/water mixture from a 30,000 cal/kg/hr (10 ton) chiller. The gas exits the heat

exchanger at 290 K and enters a cyclone separator where the condensate is separated from the gas. Liquid from the cyclone separator falls into a double-walled liquid holding tank while the loop gas enters a liquid nitrogen cooled freezer where the remaining water is removed from the loop gas to a dew point of 193 K (0.68 ppm). From the freezer the loop gas passes through a 5-micron filter to remove any particulates, and returns to the circulation pumps.

Loop-flow and total flow are measured by a flow meter/totalizer. A moisture analyzer provides MDAC with data to determine the progress of the regeneration cycle by sampling the exit bed gas. This instrument permits fast and accurate readings within a range of 123 to 123,000 ppm water. When a pre-determined moisture value is reached MDAC stops loop-flow and displays this condition by a yellow alarm.

Hold Up

Once loop-flow is completed the recovered tritiated water is contained within the liquid holding tank. The hold-up phase of the regeneration process consists of determining the amount of recovered tritiated water. The amount of water in the holding tank is determined by measuring the liquid height with a differential pressure transducer and using the predetermined entered volume measurements of the tank as a function of height.

Assay

Direct assay of the recovered tritiated water from molecular sieve regeneration is the primary function of the MSRS. Two assay techniques are used, calorimetry and scintillation counting. Experience has shown that calorimetry is a more accurate assay method for measuring water containing tritium concentrations $> 500 \text{ Ci/l}$ and scintillation counting is the preferred method at lower concentrations. To eliminate the risk associated with mechanical feed-throughs and still provide a homogeneous mixture for sampling nitrogen-bubble mixing was selected. This technique utilizes the mechanical mixing effect from bubbling nitrogen through the liquid. This is accomplished by opening valve LQGIN and LQGEX and passing the now contaminated nitrogen through the on-line process bed by opening valve MSA1 or MSD1.

Currently, scintillation counting involves direct handling of the sample. To prevent unnecessary handling of tritiated water, calorimetry samples can be taken and analyzed without personnel exposure to the sample. A calorimetry sample vial is attached to valves SCIN and SCEX and evacuated. To remove a sample, water is driven to the moisture collector which in turn fills the calorimetry sample vial with a liter of water. Prior to sample detachment, the moisture collector and sample hand valves are closed and the interconnecting piping is evacuated and purged with nitrogen. The sample vial can now be safely removed from the system, capped, and placed into a Secondary Carrying Container (SCC), transported to the calorimeter, measured, and the results entered into the computer. If the calorimetry results are $< 500 \text{ Ci/l}$ a scintillation

sample vial is attached, evacuated, and a sample taken. Prior to sampling, valve SCIN is closed. Tritiated water in the holding tank is once again mixed and removed to the moisture collector in the same manner used for calorimetry sampling. During the transfer a 10 cm³ sample of water is trapped between the hand valve of the sample vial and valve SCIN. By simply opening the sample vial hand valve a pre-determined amount of water is admitted to the sample vial. The vial is then removed from the system using the same technique as is used for calorimetry sampling, scintillation, and several dilutions of 1 ml to 50 ml, called cocktails, are prepared and counted and the results entered into MDAC.

Collection

At TSTA, the burial containers used to dispose tritiated liquid waste from the TWT are called Moisture Collectors (MC). These 30-gallon capacity DOT-5C containers are filled with Type 13X molecular sieve to absorb the tritiated water, however, they are limited to a 30 percent by weight water loading. Therefore, before any water is transferred from the liquid holding tank to the moisture collector, the on-line MC water capacity value is first determined by MDAC and the appropriate amount of water admitted to the MC. When the moisture collector is full it is removed from the system and a final weight check is performed. Also, MDAC calculates the total amount of tritium in each MC from the tritium assay results.

SAFETY

Construction Standard

The design and construction standards of the TWT, built in 1982, was based on the release and performance data of a similar system at the Tritium Salt Facility (TSF) built in 1974. However, a higher standard of safety was incorporated into the MSRS.

The major safety concern of MSRS is a release of tritiated water. This concern is mitigated by type 316 or 304L stainless steel construction joined by welding or metal gaskets. Prior to hot-startup the complete system will be tested for pressure, helium leak tightness, and functionality. Valves and major components that contact tritiated water are secondarily contained within a hood. A double-walled ASME Section VIII coded and "U" stamped vessel is used for tritiated water storage. If a tritium release within the hood is detected the hood exhaust can be routed to the Emergency Tritium Cleanup system (ETC) for processing. Double-walled construction of the water storage vessel permits continuous tritium monitoring of the primary vessel. Based on TWT construction standards the heat exchanger, HTO freezer, and pumps were not secondarily contained.

High pressure nitrogen (100 psig), regulated to 4.76 atm (70 psia), is used to fill the MSRS with nitrogen. Gas enters the system through valve LQGIN. System pressure is limited by two Back Pressure Regulators (BPR) one set at (75 psia) to

monitor the main loop pressure and a second to protect the Metal Bellows pumps. The BPR for the Metal Bellows pumps is set at 10 psi differential across the pumps. Check valves at the exit of the pumps prevent back-flow through the spare pump. Valve MSBGEX permits LPR loop evacuation, 4 to 8 psia, and valve FLVAC permits house vacuum evacuation of the system, down to 10 torr.

For safety reasons there are no penetrations through the bottom of the liquid holding tank. All transfer tubes within the tank enter from the top. Transferring liquid from the holding tank requires vacuum or pressure.