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*Author(s):* Malcolm M. Fowler, Jerry B. Wilhelmy,  
Joseph L. Thompson, and Sixto Maestas

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# Purification of $^3\text{He}$ for use in SNO Neutral Current Detectors

Malcolm M. Fowler, J. B. Wilhelmy, J.L. Thompson, and S. Maestas

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## 1 Introduction

Los Alamos National Laboratory has proposed to use  $^3\text{He}$  filled proportional counters to detect neutrons produced as a result of neutrino induced deuteron breakup. These detectors would be used in the Sudbury Neutrino Observatory (SNO) that is currently being built at the 6800 foot level in the Creighton Mine near Sudbury, Ontario. The sensitive medium in the SNO detector is 1000 tonnes of heavy water contained in an acrylic sphere with a 6 meter radius. The proposed  $^3\text{He}$  counters would be distributed throughout the volume of the heavy water to maximize the probability of detection of neutrons produced by neutrino interaction with the deuterium component of the heavy water.

$^3\text{He}$  as delivered from The Savannah River Project contains a significant amount of tritium. A single measurement of the tritium concentration made in late 1993 gave a value of about 0.53 mCi/STP liter. The  $\beta$ -decay of the tritium contained in the  $^3\text{He}$  used to fill neutron detectors can produce an unacceptably high background through pulse pileup of nearly coincident events.

## 2 Purification Procedure for $^3\text{He}$

The assumption (confirmed by Mound Laboratories) is that most of the tritium in the  $^3\text{He}$  is present as water vapor. There also could be a small amount of free tritiated hydrogen.

The approach to the removal of the tritium is to use a heated getter that would remove any free hydrogen and water vapor followed by a cooled molecular sieve trap to remove any water vapor that was not removed by the getter.

The setup for demonstration of the purification of  $^3\text{He}$  is shown in Fig. 1. Basically, the configuration is a closed loop in which the  $^3\text{He}$  is recirculated with a small metal bellows pump (Model MB-41) through a getter and a cold trap and a sample loop.

The getter we chose is a SAES model PS1GC50R1 designed for purification of rare gasses to be used in gas chromatography. The cold trap is a stainless

steel tube 1.5 cm by 10 cm long filled with 13-X molecular sieve and fitted with 1/8" SS lines. The exit line from the cold trap is wrapped around the outside of the molecular sieve trap to ensure that the circulating gas is cooled to the liquid nitrogen bath temperature before exiting to the sample loop.

### 3 Tritium Measurement Procedure

Measurement of the tritium in the  $^3\text{He}$  is done by converting tritium in the  $^3\text{He}$  to water then incorporating the water in a liquid scintillation cocktail for counting. A sample of  $^3\text{He}$  gas, usually 5 to 7  $\text{cm}^3$  at 800 Torr contained in a 30 cm length of 0.25" stainless steel tube is connected to a small vacuum system. The gas sample is allowed to expand into the system and is pumped out, passing over heated copper oxide where the tritium is converted to water vapor. Next, the  $^3\text{He}$  passes through a cold trap maintained at liquid nitrogen temperature ( $\sim 73\text{K}$  @580 Torr) to trap out the water. After the initial  $^3\text{He}$  is pumped away, additional pure hydrogen is added to the sample tube and is then pumped out through the copper oxide in the same manner the  $^3\text{He}$  had been processed. The hydrogen flush can be repeated until sufficient water is collected in the trap to make quantitative removal possible for preparation of the scintillation cocktail. The assumptions in this step are that tritium is quantitatively removed from the sample container, quantitatively converted to water (if not already in that form), and quantitatively collected in the cold trap.

### 4 Results

In the first purification test, we tried a single pass through the getter and trap without the recirculating pump. We evacuated the recirculating loop while heating the molecular sieve trap and the lines to  $\sim 100^\circ\text{C}$  to degas the system. At the same time the system was helium leak checked to ensure that it was leak free. After the pressure in the loop was reduced to  $\sim 1 \times 10^{-5}$  Torr, the molecular sieve was cooled with liquid nitrogen, the getter was heated to its normal operating temperature, and the loop was valved off from the vacuum pump. Next,  $^3\text{He}$  was expanded into the loop through the getter and molecular sieve. When the loop pressure was at 759 Torr, the sample tube was valved off and removed. Analysis of this single pass purification sample indicated that the tritium content had been reduced by a factor of  $\sim 570$  to  $\sim 0.9 \mu\text{Ci}/\ell$ .

In the second test, we added the recirculating pump between the exit of the molecular sieve trap and the sample tube. The loop was again pumped out and leak checked as in the first test. Next the loop was pressurized to  $\sim 1000$  Torr with  $^3\text{He}$ , then the getter was heated and the trap was cooled. The circulation pump was started and run for about 2 hours before the sample tube was valved off and removed. Analysis of this sample produced the surprising result that the tritium concentration was  $\sim 5.8 \mu\text{Ci}/\ell$ , higher than in the single pass test. We postulated

that during the pressurization of the loop with  $^3\text{He}$ , the Teflon gasket in the metal bellows pump was exposed to the high levels of tritium in the unpurified  $^3\text{He}$ . If some of the tritium diffused into the Teflon and later outgassed during recirculation, then relatively high levels of tritium would be in the gas stream feeding into the sample tube. We tried to test this postulate using helium. We found that after pressurizing the circulation loop with helium to 1000 Torr followed by evacuation, there was still a considerable evolution of helium and the source appeared to be the metal bellows pump.

In the third test, we moved the recirculating pump to a position downstream from the sample tube, then we evacuated the loop for several days while heating all parts of the system. We then repeated the second test, but recirculated the gas for about 1 day rather than for a few hours. Analysis of a sample of this gas gave a concentration of  $\sim 26$  nCi/ $\ell$ . This represents a decontamination factor of about 20000.

Next, we repeated the third test with a longer recirculation time of 4 days and found a decontamination factor of  $\geq 51000$ . The measured concentration in this case was an upper limit of  $\sim 10$  nCi/ $\ell$ .

## 5 Discussion

The effect of the tritium in the  $^3\text{He}$  can be seen in Figures 2. and 3. To generate these figures, we assumed the neutron counters were 3 meters long with an inside diameter of 2 inches. We also assumed that the counters were filled to a pressure of 3 atm. with a mixture that was 80%  $^3\text{He}$  and 20%  $\text{CF}_4$ . The counter would then contain 14.3 STP liters of  $^3\text{He}$ . Next, we assumed that the average energy of the beta particles associated with the decay of the tritium was 6 keV and that we would integrate the signal for 30  $\mu\text{s}$  for each event. Using the tritium concentrations measured earlier, we then calculated the number of tritium decays in the counter during the integration time. Figure 2 shows the average energy deposited in the counter for the different levels of purification discussed earlier. In Fig. 3 is shown the expected distribution of energy deposition for the different tritium concentrations. In the case of the unpurified gas one sees that the tritium energy has a narrow distribution, but is higher than the 760 keV signal expected from the capture of a neutron. As the tritium level is reduced the distribution broadens and becomes assymetrical due to the poisson statistics associated with the few tritium decays during the integration interval. At this level, the contribution of the tritium decay is small compared to the neutron capture signal level. If we can decontaminate the  $^3\text{He}$  to this level then the contribution of false events due to pile up of tritium should be acceptable.

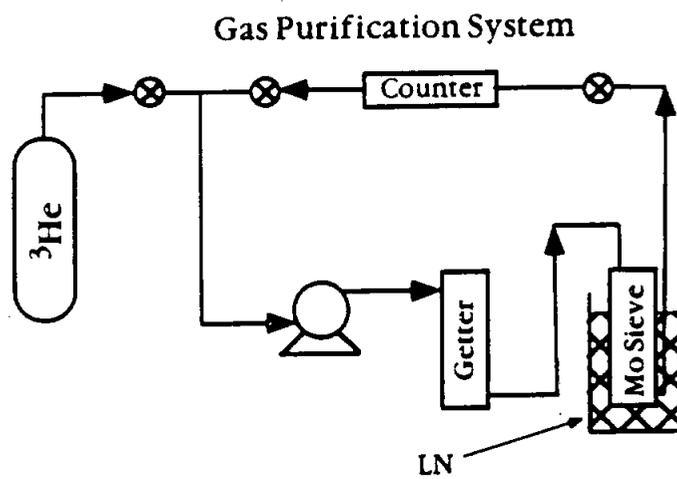


FIGURE 1

# Effects of Tritium in $^3\text{He}$

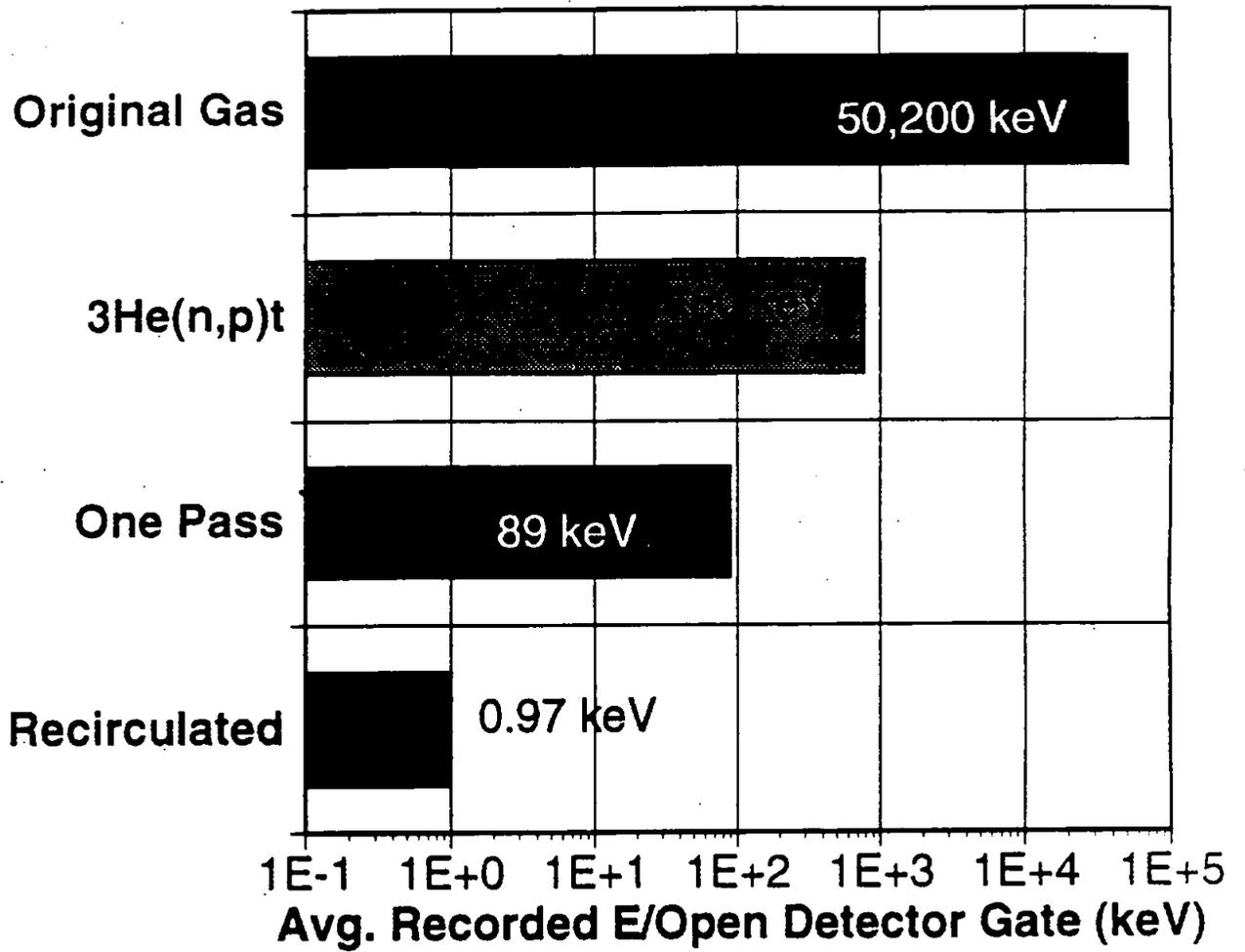


FIGURE 2

# Energy Deposition in Counter

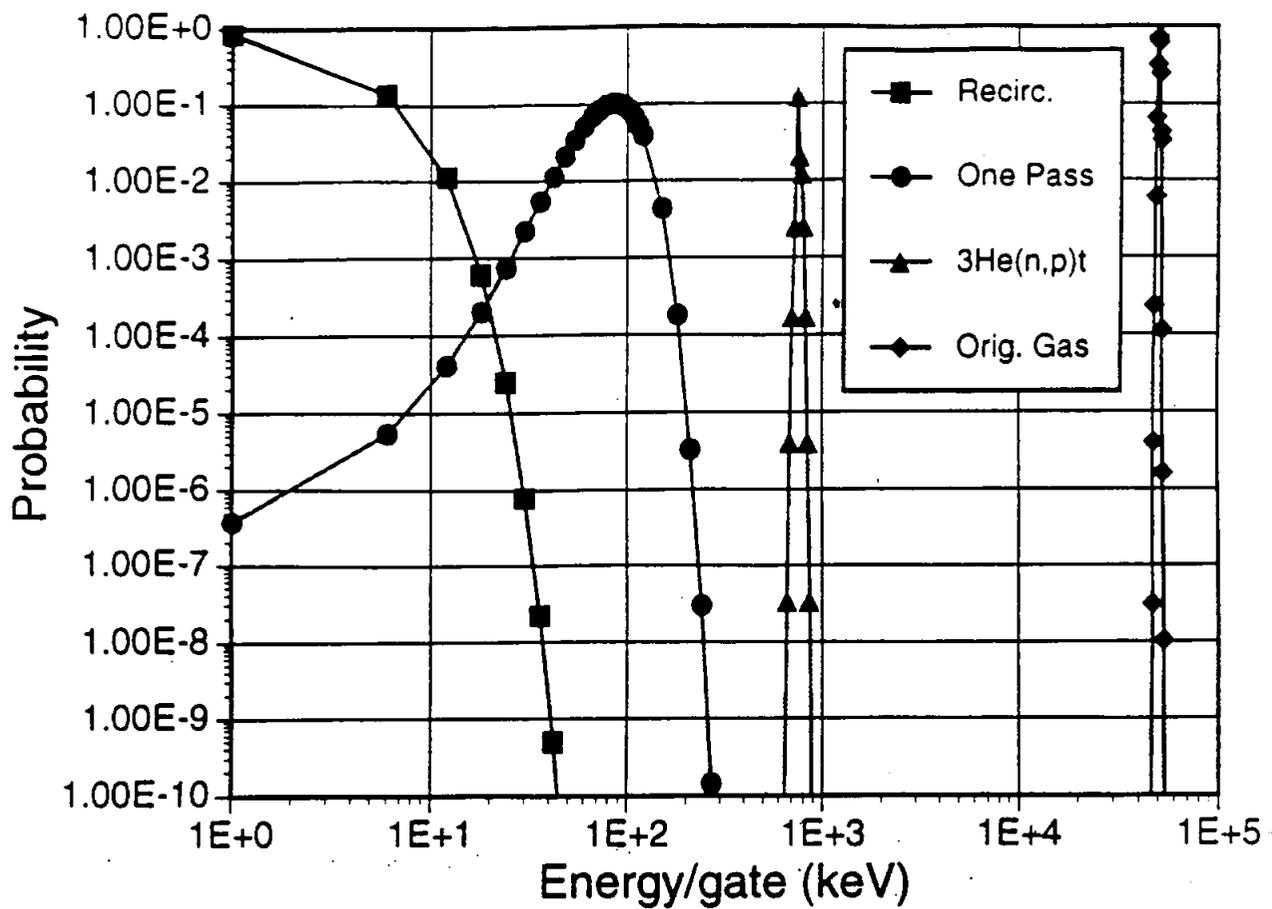


FIGURE 3