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TITLE: An Identification Checker for Measurement of High-Level Tritium Gas*

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AN IONIZATION CHAMBER FOR MEASUREMENTS OF [illegible]

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ABSTRACT

The construction and calibration of a steady-ionization-chamber apparatus for measurement of high level tritium gas is described. The apparatus uses an easily constructed but rugged chamber for containing the unknown gas and an inexpensive digital multimeter for measuring the ion current. The equipment after calibration is suitable for measuring 0.01 to 100% tritium gas in hydrogen-helium mixes with an accuracy of a few percent. At both the high and low limits of measurements, deviations from the predicted theoretical current are observed. These are briefly discussed.

INTRODUCTION

In our tritium facility at Los Alamos Scientific Laboratory we often need to assay low concentrations of tritium (1% and below) in helium gas mixes. In the past these analyses have been made using mass-spectrometric techniques but such measurements are time consuming and, because of the low concentrations, often inaccurate. We felt a measuring system based on an ionization chamber would be simpler to use and more accurate. Although such chambers are routinely used for low level measurements in the gas phase (ppm and below),[1,2] their use at higher concentrations have been limited. Anthony[3] has described such a chamber but its design is somewhat more complicated than the one described here.

This ionization chamber technique has the advantage of providing rapid results and does not suffer the same interpretation problems as low resolution mass spectrometric techniques. A further advantage is that such a chamber could be used to continuously monitor the total tritium content of a flowing stream. A major disadvantage is that it does not differentiate between different tritium species.

EXPERIMENTAL DETAILS

Because of the large ionization currents found for high level gases, the equipment is simple and inexpensive. The materials used in the construction of the ionization chamber were chosen

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more for ruggedness and impermeability to tritium rather than for low background current as is usual in conventional chamber design. The chamber (Fig. 1) is constructed from a drilled brass rod and has an effective volume of 17 cc. A stainless steel rod is used as the collector and it is supported and insulated from the chamber with a glass and Kovar insulator. The assembly is soft soldered. The chamber is biased with a 65 V battery and the ionization current is measured with 3-1/2 digit, digital multimeter having current ranges down to 19.99 nA and a stated accuracy of ± 0.5%. In our arrangement the collector is above ground potential whereas the chamber is grounded, simplifying the apparatus. This is contrary to usual design and is possible because both inputs of the multimeter used can float above ground.

During calibrations background currents varied between 5 and 100 nA and appeared to arise primarily from strain currents and leakage rather than from tritium contamination. The noise level generally was in the 5 nA range, thus currents could be measured down to about 10 nA, corresponding to about 0.01% tritium, with good accuracy.

Calibration gases were made up from mixes containing predominantly tritium and deuterium

Fig. 1. The construction of the ionization chamber and the experimental setup.

with traces of protium and helium (as measured with a mass spectrometer). A series of more dilute gas mixes was prepared from each starting mix by several successive dilutions with ^{4}He. Pressures were measured with a calibrated Wallace and Tiernan gauge accurate to ± 1% and each mixture after preparation was circulated through a loop in the system for several minutes with a transfer pump to assure a homogeneous mix. The prepared gases covered the region of 0.01-88% tritium. The ionization currents for each gas mix at pressures between 50 and 600 torr were then measured to obtain calibration data.

The variation of current with voltage at constant pressure was measured for several gas mixes at 600 torr. For mixtures containing less than 1% tritium, constant ionization currents were obtained for voltages above 45 V. The flat curves obtained indicated saturation currents were achieved. For higher concentrations (above 5% tritium) plateaus were not reached even at 90 V, the highest voltage used to bias the chamber. For consistency, the calibration experiments were performed with a bias voltage of 45 V, leading to a lowered ionization current at high concentrations.

DISCUSSION

For an infinitely large chamber with no wall effects and in the absence of recombination, the saturation current due to ionizations arising from tritium decay is given by $I_s = (33.29/W)\mu A/Ci$ where W is the energy in eV required to produce one ion pair in the gas within the chamber.[1,2] Experimentally determined[4] values of W yield 1_s = 0.909 for hydrogen isotopes and 0.795 for helium isotopes. For the gas mixes used here we can then write $1_s = 0.909\ f + 0.795\ (1-f)$ where f is the partial fraction of hydrogen isotopes in the calibration gas. I_s represents the ideal current and is a good approximation for large chambers and small concentrations of tritium. Two effects, collisions of the beta particles with the wall before they can deposit their entire energy to the gas and recombination of carrier ions, both work to decrease the efficiency of the chamber giving lower currents.

Results for two calibration runs are given in Fig. 2. The solid line in the figure represents the ideal current based on the above equations. The dropoff at high concentrations as discussed above, is due to recombination of carrier ions and resultant lack of saturation. Two attempts using published techniques[5,6] to calculate the losses due to recombination were tried. Both calculations overestimated the effect, but they did point out the importance of this effect at high concentrations of tritium.

Soudain[5] has estimated the effects of wall collisions using geometrical considerations based on the known average pathlength of the beta particle. Similar calculations for our chamber indicated a loss of about ½% in efficiency for pressures of 600 torr. Mueller[7] has done a more detailed calculation based on linear absorption

Fig. 2. Experimental currents for two runs compared to the theoretical saturation current I_s (solid line).

coefficients and which allowed for partial reflections of the beta particles from the walls. This indicated the loss should be somewhat higher, approaching perhaps 50%. Both results indicated the effects should be approximately constant for varying amounts of tritium; thus, wall collisions should result in a general lowering of the curve by a constant amount.

As can be seen from the figure, the experimental currents go above theoretical by a factor approaching ten at low tritium concentrations. This is thought to be an artifice of the experimental setup arising from tritium gas desorbing from the walls of the tanks used to prepare the mixes and from the oil in the transfer pump used to stir the gases. This desorption would increase the concentration and ionizaton current and would become of greater importance at low concentrations as is apparent in the results. A similar desorption has been seen in other systems used to process high level tritium gas.[8] The contamination is definitely not in the chamber as measurements of pure helium following a calibration run showed no increase in background. The apparent "memory" effect could be checked by running a calibration gas of known composition in the range 0.001-0.01%, an experiment which would be difficult with our system. The second run shown in the figure was made under conditions which tended to decrease the desorption contamination. This gave better agreement with theoretical, supporting this hypothesis.

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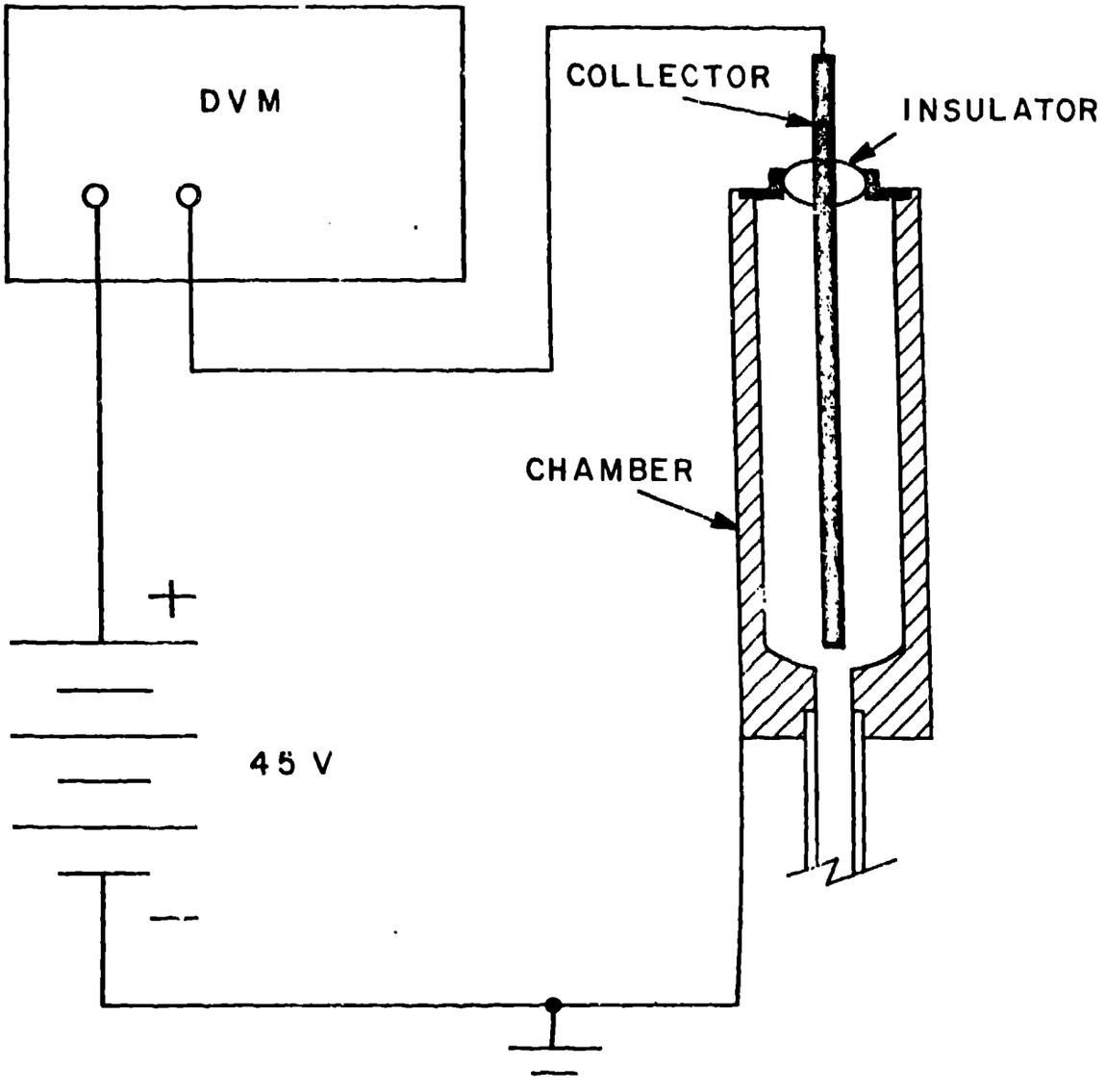
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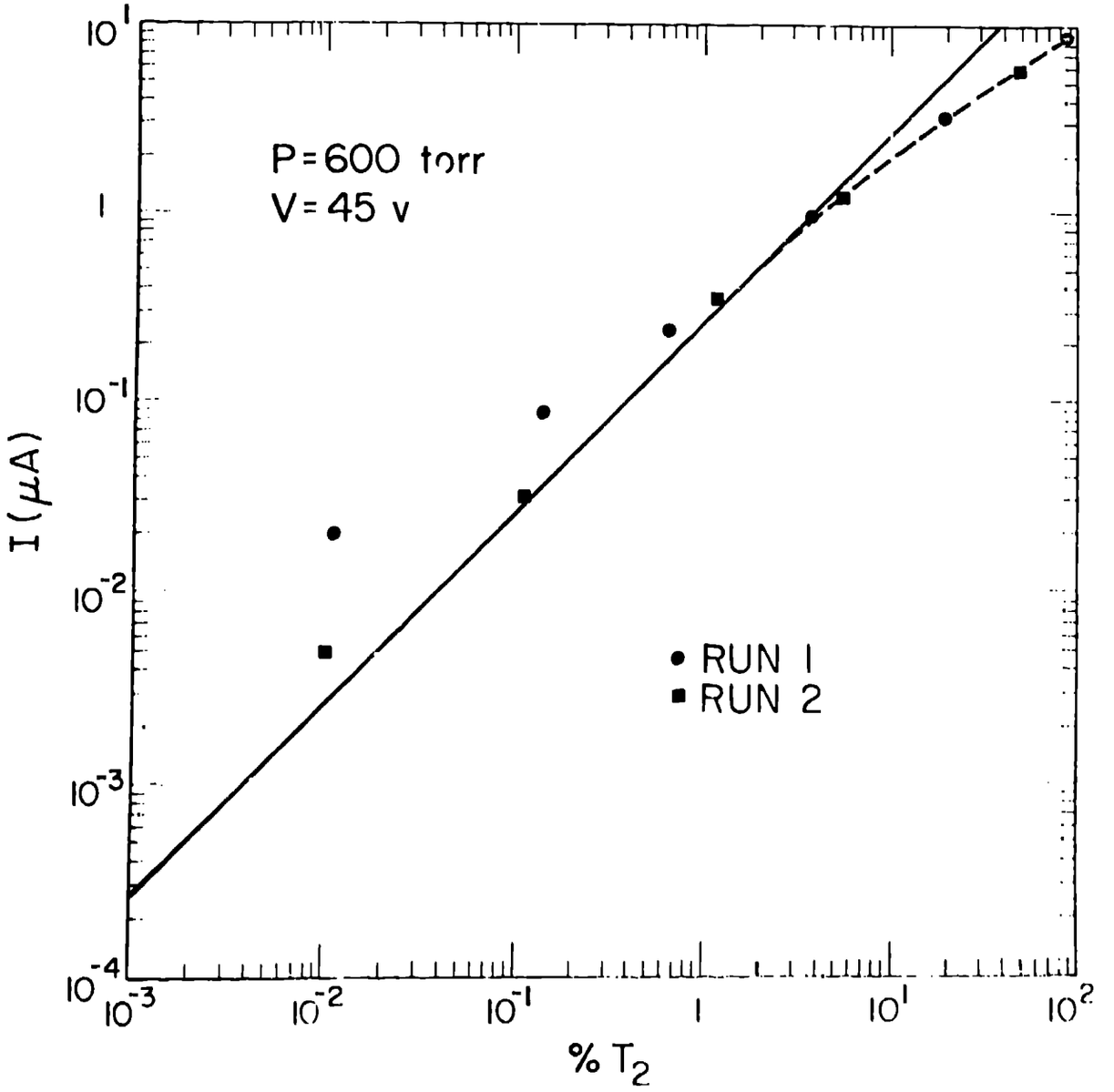
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RUN 2
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% T2
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1
10^-1
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10^-2
10^-1
1
10^1
10^2