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IONIZATION CHAMBER MEASUREMENTS ON LIQUID SAMPLES FROM P-10

ABSTRACT

A method of measuring low level tritium in liquid samples has been investigated. The water containing the tritium oxide is vaporized in a heated ion chamber, and a vibrating reed electrometer is used as the current measuring instrument. With a one liter ion chamber operated at 80°C, the sensitivity is $1.3 \pm 0.3 \frac{mv}{min}$ per microcurie/liter.

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INTRODUCTION

Several methods of measuring tritium oxide have been used previously. At Hanford, the Health Instrument Methods Group has devised a method whereby a body fluid is converted to methane via aluminum carbide, and counted in a proportional counter[1].

N. Pace, L. Kline, H. K. Schachman, and M. Harfenist[2] have counted tritium oxide vapor in a Geiger counter filled with 2 mm Hg of water vapor, 2.5 cm Hg alcohol vapor, and 2.0 cm Hg Argon vapor. Allen and Ruben[3] have also counted water vapor containing tritium. They used an 18% ethanol-argon counting gas. Taschek and Gittings[4] have detected tritium beta particles as well as the soft X-rays produced by the beta particles, with finely divided napthalene and a 1P21 phototube.

Analyses of samples containing tritium oxide using ion chamber techniques have been accomplished by F. C. Henriques and C. Margnetti.[5] Their liquid samples were converted to hydrogen before inserting in the chamber. An ion chamber method is also used by the Health Instrument Methods Group here at Hanford.[6] The liquid tritium containing sample is converted to acetylene by treatment with calcium carbide, and the acetylene is measured in a one liter ion chamber with a vibrating reed electrometer. All of the above methods involve considerable apparatus, both chemical and physical, and it was thought that a simpler technique might be devised.

Feasibility of Vapor Method

For a concentration of one microcurie per liter in body fluids, it can be shown that, for an ion chamber containing such liquid as steam at atmospheric pressure and 100°C, 5.22 ion pairs/cc sec will be produced. For 80°C vapor, 2.61 ion pairs/cc sec will be produced. These results depend upon the use of 5.7 Kev as the average beta energy and 32.5 ev. per ion pair produced, as well as upon the assumption that HTO, T_2O, and H_2O evaporate at rates determined only by their concentration in the solution to be tested. This is probably a fairly good assumption, since most of the mass of the molecule is made up of the oxygen atom.

Background radiation, including cosmic rays, local radiation, and alpha wall contamination, varies from 6 to 18 ion prs/cc sec.[7] If the largest alpha pulses can be subtracted by means of a chart recorder, the background without alpha will be 6 to 8 ion pairs/cc/sec.[8] Therefore, it should be possible to detect concentrations of one microcurie/liter or greater in a liquid sample. Actually, it will be seen that instrumentation troubles will raise this figure considerably.

Method

The ion chamber used was a one liter flask coated on the inside with aquadag. A ground glass joint joined the flask with a guard ringed electrode system, which in turn fastened directly to the head of a vibrating reed electrometer. The vibrating reed electrometer was an M-3 Applied Physics Corp. model, and was followed by a Brown Electronik self balancing potentiometer type chart recorder.

Currents were measured by the rate of drift method. The input capacitance of the electrometer was measured utilizing a cobalt-60 source in fixed geometry, and a known input resistor.

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With the resistor across the input, the current was measured as E/R. With the resistor removed, the current was again measured as $dq/dt = C\frac{dE}{dt} = C\frac{\Delta E}{\Delta t}$. Equating the two currents, one obtains

$$C = \frac{E}{\Delta E} \quad \frac{\Delta t}{R}$$

The E and Δ E were measured using the meter on the vibrating reed. The input capacitance of the electrometer used was 12.25 ± .20 micromicrofarads. The active volume was estimated at 980 cc by filling the active volume of the chamber with water, and measuring the water volume. It was 980 cc. ± 30 cc.

In taking measurements, first a background was run with air in the chamber. Next a measured amount of distilled water was added, the flask brought up to temperature, and the drift rate measured again. The chamber was dried thoroughly by evacuation. A fluid sample containing the tritium oxide was then inserted, and the temperature again stabilized before taking measurements. During most of the work, considerably more than enough sample to saturate the chamber was used, since the walls and electrodes seemed to hold the water, either by absorption or condensation.

Experimental Results

The experimentally determined sensitivity of this method is 1.3 ± 0.3 mv/min per microcurie/liter, as compared to the calculated value of 1.5 mv/min per microcurie/liter. Most of the lack of reproducibility is due to insulator troubles during water background and P-10 measurements. Covering the insulator with ceresin wax did not seem to help.

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The trouble was partly overcome by the use of a heat lamp on the neck of the flask, in addition to the normal water bath or oven used as a heat supply. This kept the insulator hotter than the vapor, and tended to evaporate the moisture film. Moisture condensing on the central electrode and guard ring was also the source of some difficulty.

Approximately forty-five minutes were required for the vapor to reach equilibrium at 80°C. No information was obtained at higher temperatures, since the ion chamber incorporated wax seals that were beginning to melt.

Background drift rates corresponded to 5.5 to 7.5 ion pairs/cc sec, both for a water vapor chamber, and the air filled chamber. A major portion of the drift due to alpha particles had been subtracted to obtain the above result. The value of background radiation in 3746-D was 6.90 ± .63 ion pairs per cc sec at standard conditions. This corresponds well to Strong's value of 6 to 8 ion pairs/cc sec.

Conclusions:

The sensitivity of this method is high enough for it to be used in measuring tolerance amounts of tritium. It suffers rather severely from instrumental troubles. Some of them could, I believe, be eliminated by proper design of the ion chamber. This was not attempted, since the proportional counting method[9] used by the Methods' Group seems to be superior, both in stability and in signal to background ratio. Also, there is little gain in time, since the time necessary to reach vapor equilibrium in this method is about the same as the chemical process and flushing time for the counting method. Therefore this method was not pursued to the point of having special heated ion chambers made up.

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I. T. Myers
Physics Group
H. I. Development Division

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