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URINE ANALYSIS FOR TRITIUM OXIDE.

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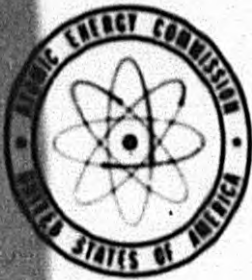
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The following procedure is based on the production of acetylene from the active water, with subsequent measurement of the ionization caused by the tritium beta particle. This eliminates the troublesome absorption on the chamber walls when hydrogen is used, and provides a rapid method for the preparation of the sample.

MATERIALS

A. Gas Generator

The gas generator consists of a 25 ml. boiling flask with a two hole rubber stopper. One hole contains a thistle tube and the other a tube leading to a four liter water-displacement collection bottle. About 2-3 gms. of calcium carbide are placed in the generator before final assembly.

B. Ion Chamber and Electrometer

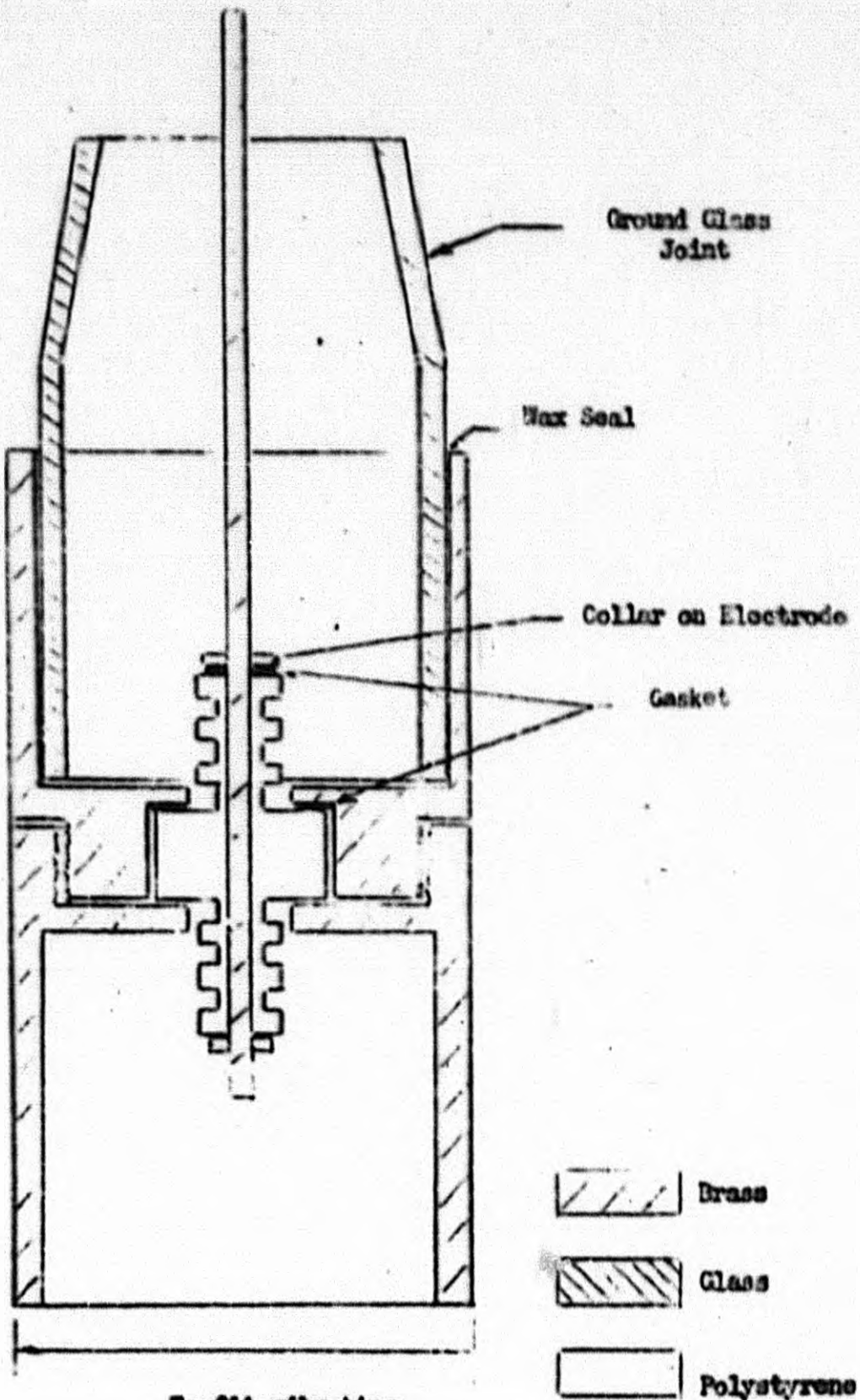
The chamber consists of a one or two liter glass boiling flask with a ground glass joint, a two way stopcock in the neck, and the interior coated with aquadag. A potential of -180 volts from B batteries is applied to the conducting coating by means of a wire sealed through the glass. A condenser of 125 uf is placed across the battery to minimize "noise".

A rough sketch of the collecting electrode and insulator assembly is given in figure 1. The bottom portion of this assembly fits directly into the vibrating reed head and the end of the electrode seats against a spring loaded contact. The nut supporting the electrode should not be made too tight or strains in the polystyrene may result.

PROCEDURE

1. Obtain 10-20 ml. sample of urine. This volume permits re-runs if necessary.
2. Pour about 4-5 ml. of sample into the thistle tube of gas generator.

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To fit vibrating
Beard Head
Rod

FIGURE 1

Insulator System for Glass Chamber

3. Generate the acetylene by allowing the urine to drop slowly on the calcium carbide. Allow all of the air to flush from the lines before starting the collection in the gas bottle.
4. Upon completion of the generation, the gas may be stored for several minutes or several days in the collection vessel.
5. Evacuate the chamber and fasten to the collection vessel through a tube containing "drierite".
6. Adjust pressure in the chamber to 760 mm. of mercury by means of a water column.
7. Close stopcock to chamber and read the ionization current on the vibrating reed electrometer by the rate of drift method.

In practice, it has been found necessary to obtain background measurements before and after each sample because of occasional high readings believed to be due to insulator strains. Successive readings are made on each sample until the drift rate appears to be reasonably constant in order to eliminate measurement of charges induced by the gas flowing into the chamber. Measurements are made on each sample for a total time of 10-20 minutes to average random fluctuations in current. This is accomplished by recording the drift rate on an Esterline-Angus meter and measuring the time of drift between two fixed points.

The gas for background measurements has been acetylene generated from the urine of personnel working in the laboratory with presumably no exposure to tritium.

RESULTS

A series of 16 background measurements on a one liter chamber during the routine analysis of samples gave an average of 5.2×10^{-15} amperes with a standard deviation of 0.45×10^{-15} amperes. Air appears to give about 1/3 to 1/5 of this value. The significance of the current from any particular sample is tested by "t" testing the one value against the average background:

$$t = \frac{x - \bar{x}}{\sigma}$$

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Where x is the individual reading, \bar{x} is the average background reading, and σ is the standard deviation of the average background. The significance for any value of " t " is obtained from standard tables.

In general, backgrounds measured before and after the sample should be approximately the same or the results would be open to question. When any value is found positive, at least one re-run is made to insure against the possibility of stray charges or insulator strain.

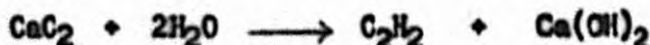
INTERPRETATION OF RESULTS:

The resulting ion current above background is interpreted to microcuries by assuming: (1) 5.69 kev average energy of the beta particle; (2) no loss in the chamber walls due to particles striking the wall; and (3) 32 ev are required to produce one ion pair in acetylene. Assumption (2) will cause the results to be low by 5-10%. Until recently, variations in the performance of the equipment caused uncertainties of a greater magnitude than this and no correction factor was obtained. Assumption (3) is open to question. The only test, to date, has been to measure the current from a plutonium source both in air and in acetylene. Both values were lower than theoretical, but were practically identical.

Using these assumptions, the relation between microcuries and current is:

$$\begin{aligned} \text{Microcuries} &= \frac{\text{amperes} \times 6.281 \times 10^{18} \times 32}{5.69 \times 10^3 \times 3.7 \times 10^4} \\ &= (9.55 \times 10^{11}) \text{ (amperes)} \end{aligned}$$

The reaction between calcium carbide and water is:



Thus one mole of acetylene is produced from two moles of water or 1.61 gas. of water are required to produce one liter of acetylene. The hydrogen from only one mole of water goes to produce acetylene, however. This means that the one

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liter of acetylene in the chamber represents the hydrogen from only 0.805 gm. of water.

To convert the result to microcuries per liter of sample:

$$\begin{aligned} \text{uc/l} &= \frac{116}{0.805 \times 10^{-3}} = 1240 \text{ (uc)} \\ &= (1240) (9.55 \times 10^{11}) \text{ (amperes)} \\ &= (1.26 \times 10^{15}) \text{ (amperes)} \end{aligned}$$

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