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**Development of a Low Cost Versatile
Method for Measurement
of HTO and HT in Air**

W. E. Sheehan, M. L. Curtis, and D. C. Carter

February 14, 1975

MASTER

Research and Development Report

MOUND LABORATORY

Miamisburg, Ohio

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SUMMARY

A low cost, highly sensitive method for the measurement of HTO and HT in air has been developed. The air is first passed through an ethylene glycol bubbler to remove the HTO, then through a heated palladium catalyst where the HT gas is oxidized to HTO and collected in a second ethylene glycol bubbler. The tritium collected by the bubblers is measured by liquid scintillation counting. Recoveries of 95 to 100% with air flow up to 750 cm³/min are routinely obtained. The sensitivity of the method is basically dependent on the liquid scintillation counter background and the counting techniques employed. Several applications of this method of tritium air sampling are in use at Mound Laboratory and are discussed.

INTRODUCTION

Tritium oxide (HTO) because of its much higher uptake by man presents a much greater health hazard in the atmosphere than elemental or gaseous tritium (HT). The Standards for Radiation Protection reflect this difference in biological hazard. The radiation concentration guide (RCG) for elemental tritium in air for a 168-hr week is $4 \times 10^{-5} \mu\text{Ci}/\text{cm}^3$, whereas the RCG value for tritiated water vapor is $2 \times 10^{-7} \mu\text{Ci}/\text{cm}^3$. To evaluate the impact of a given operation and the effect of stack gas effluents relative to biological hazard to the immediate surroundings, the amount of HTO as well as total tritium in the effluent stream must be known. In response to this need a relatively simple, accurate, and inexpensive method was developed for the simultaneous collection and separation of tritium oxide (HTO) and tritium gas from air on a continuous basis. The technique which employs ethylene glycol water vapor traps and a hot palladium oxidation catalyst was originally developed for stack effluent monitoring and has since been applied to dry box atmosphere analysis, tritium leak rate determinations, and environmental monitoring. This report reviews the development, validation, and current uses of the technique at Mound Laboratory.

BACKGROUND

Ruggedized Field Air Sampler

Several parallel efforts were directed towards the development of differentiating tritium air samplers. One of the earlier efforts was the construction of a ruggedized ultrasensitive field air sampler¹ based upon the Ostlund² technique of combustion and absorption of HT using palladium coated desiccants. This system is designed to handle high air flow rates (up to 75 liter/min) and is capable of detection of near natural background levels of tritium with sampling times of 1 hr.

Real Time Monitor with High Sensitivity

A second effort was the construction of a real-time high-sensitivity tritium monitor that can differentiate between elemental tritium and tritium oxide. This instrument was designed and built under contract to Johnston Laboratories, Inc., Cockeysville, Md., as part of a tritium effluent control development project.³ The instrument employs a flow-through gas proportional counting system with a direct graphical readout in counts per minute. Two modes of operation can be selected, one at a time, to determine either elemental tritium concentrations or total concentration including the oxide. The design criteria were for a sensitivity of $0.01 \mu\text{Ci}/\text{m}^3$ capable of discriminating against an alpha background (radon) of $2 \times 10^{-4} \mu\text{Ci}/\text{m}^3$ and a gamma background of 0.05 mR/hr. Operational experience with this instrument in stack monitoring has proven the

instrument operates up to design criteria with the exception of sensitivity. The detection limit of $0.01 \mu\text{Ci}/\text{m}^3$ was based upon an obtained background of 6 counts/min at Cockeysville, Md. This background was never realized at Mound Laboratory. The lowest background attained at Mound Laboratory prior to exposure to tritium atmosphere was 160 counts/min. This increase in background is believed caused by an increase in the gamma background. With exposure to tritium atmospheres this background has gradually increased to 600 counts/min. This increased background limits the tritium sensitivity to $0.5 \mu\text{Ci}/\text{m}^3$ or 50 times greater than the design criteria following several months of operation.

Low Cost High Sensitivity Air Samplers

A much less costly, versatile method for differentiating between the oxide and gas was developed, originally for measuring grab samples of stack and glovebox atmospheres. The method, with various hardware modifications, has been expanded to include continual measurement of stack and room atmospheres and determination of leak rates from equipment or prototypes. Each method employs a heated palladium oxidizing catalyst (Figure 1). The collected samples are analyzed for tritium by standard liquid scintillation counting techniques.

FIGURE 1 - Palladium catalyst and tube furnace assembly.

EXPERIMENTAL

Oxidation of Tritium Gas with Hot Palladium Catalyst

An early method was developed in which a 5% hydrogen-in-argon carrier gas transported a 2-liter sample (at $400 \text{ cm}^3/\text{min}$) through a dry ice/acetone trap to collect the tritiated water vapor, through a hot ($350\text{-}450^\circ\text{C}$) palladium metal sponge catalyst (4-10 mesh), and finally through a second dry ice/acetone trap to collect the oxidized tritium gas.⁴ The glass

sample flask was flushed 40 min, in two 20-min increments, to effect complete sample recovery. The freeze-out traps were comprised of a standard 25 cm³ plastic scintillation vial half filled with 4-mm glass beads and immersed to the cap in the dry ice/acetone slush. Two-liter tritium gas standards, in air, were analyzed to determine the collection efficiency of the system. (These standards were prepared from a stock tritium standard that had previously been analyzed by gas proportional counting techniques traceable to a certified NBS water standard.) On the basis of nine determinations for a 4×10^5 dis/min standard, an average collection efficiency of 96.3% was attained with standard deviation of 5.4% (Table 1).

Table 1

OXIDATION OF TRITIUM WITH PALLADIUM CATALYST
AT 350°C USING 5% H₂/95% Ar CARRIER GAS^a

Sample Number	HT Added (dis/min)	HTO Recovered (dis/min)	Recovery (%)
1	3.78×10^5	3.48×10^5	92.1
2	3.75×10^5	3.35×10^5	89.3
3	3.78×10^5	3.53×10^5	93.4
4	3.78×10^5	3.58×10^5	94.7
5	3.75×10^5	3.52×10^5	93.9
6	3.78×10^5	3.65×10^5	96.6
7	3.45×10^5	3.40×10^5	98.6
8	2.73×10^5	2.74×10^5	100.4
9	2.74×10^5	2.95×10^5	107.7
Average			96.3
Standard Deviation			5.4

^aAir flow 750 cm³/min.

A further simplification of this grab sample system was made by replacing the 5% hydrogen-in-argon carrier gas with 100% dry air.⁵ The modification allowed the run to be completed in one 40-min increment eliminating the need to change traps after the first 20 min because of dip-tube freeze-up. Further, the expense of a premixed gas was eliminated. The only concern in eliminating the hydrogen carrier was whether complete recovery could be achieved at the lower dew point of the gas stream entering the second trap. On the basis of 11 tritium gas standard determinations, however, an average collection efficiency of 99.0% was attained for a 3×10^5 dis/min standard with a standard deviation of 2.5% (Table 2).

Table 2

OXIDATION OF TRITIUM GAS ON PALLADIUM AT 350°C
WITHOUT USE OF CARRIER GAS^a

Sample Number	HT Added (dis/min)	HTO Recovered (dis/min)	Recovery (%)
1	2.89×10^5	2.84×10^5	98.3
2	2.91×10^5	2.86×10^5	98.3
3	2.82×10^5	2.85×10^5	101.1
4	2.44×10^5	2.35×10^5	96.3
5	2.55×10^5	2.68×10^5	105.1
6	2.64×10^5	2.58×10^5	97.7
7	3.49×10^5	3.46×10^5	99.1
8	1.85×10^5	1.85×10^5	100.0
9	8.21×10^4	8.14×10^4	99.2
10	3.50×10^5	3.38×10^5	96.6
11	2.75×10^5	2.68×10^5	97.5
Average			99.0
Standard Deviation			2.5

^aAir flow 750 cm³/min.

A method based on those already described using freeze-out traps was developed for continuous sampling purposes.³ The dry ice/acetone cold traps were eliminated and a mechanical refrigeration system was incorporated to allow for unattended operation (Figure 2). Two glass freeze-out traps are maintained at a temperature between -50 and -60°C by means of a Neslab Model CC60F cooler and a 4-liter Dewar flask containing Freon TA (duPont) coolant. The regulated gas sample is drawn through the system at 750 cm³/min by means of a small welded bellows vacuum pump compressor, and, because of the large freeze-out traps, 5% hydrogen in argon carrier gas is added at the rate of 50 cm³/min just prior to entering the catalyst tube. The second freeze-out trap which collects the oxidized hydrogen carrier and tritium gas fraction of the sample is the standard cold finger design approximately 1.5 in. (38 mm) in diameter by 12 in. (305 mm) long. The flow has been reversed, however, entering the side and exiting the dip tube, to eliminate clogging during extended sampling periods. The first trap, unlike the second, must collect unpredictable volumes of water depending on the temperature and humidity of the gas sample. To avoid clogging for periods up to 7 days, under most conditions of temperature and humidity, a special high-capacity cold trap was designed which condenses and collects a portion of the incoming moisture (Figure 3). When the gas reaches the lower freeze-out portion of the trap, sufficient water vapor has been removed so that the trap remains

FIGURE 2 - Continuous sampling system.

unclogged for longer periods of time. In order to determine the collection efficiency of the system, two-liter gas standards, 1×10^6 to 1×10^7 dis/min, were flushed into the system at 50 cm³/min, for a total flow of 750 cm³/min, for periods up to 72 hr. On the basis of seven determinations an average collection efficiency of 99.9% was obtained with a standard deviation of 2.9% (Table 3).

Table 3

OXIDATION OF TRITIUM GAS ON PALLADIUM AT 400°C
BY THE CONTINUOUS FREEZE-OUT METHOD

Sample Number	HT Added (dis/min)	HTO Recovered (dis/min)	Recovery (%)
1	1.33×10^7	1.37×10^7	103.0
2	1.33×10^7	1.30×10^7	97.7
3	1.00×10^6	1.04×10^6	104.0
4	9.56×10^7	9.39×10^7	98.2
5	9.56×10^7	9.60×10^7	100.4
6	9.56×10^7	9.16×10^7	95.8
7	9.32×10^6	9.34×10^6	100.2
Average			99.9
Standard Deviation			2.9

FIGURE 3 - High capacity vapor freeze-out trap.

HTO Collection with Ethylene Glycol Bubblers

Ethylene glycol bubblers for trapping HTO were first used in the development of a tritium leak rate method.⁶ Initially a glass-bead cold trap, previously mentioned, was used. The glass-bead cold trap was effective but required a cold temperature bath and had a high background (~180 counts/min) from the ^4K in the glass beads when counted directly. A small volume water bubbler substituted for the glass bead cold trap proved satisfactory if corrections were made for water lost from evaporation. To reduce losses by evaporation ethylene glycol was substituted for the water bubblers. Figure 4 is a sketch of a differential tritium air sampler using ethylene glycol bubblers.

FIGURE 4 - Differential tritium air sampler.

Osborne⁷ summarizes his work with water bubblers by stating that "the most important variables determining the overall collection efficiency for HTO of a bubbler are the intrinsic efficiency and the water lost in the effluent air. Deviation of the former from unity may be disregarded for a practicable range of air flow rates, temperatures, and bubbler types. A linear relationship between collection efficiency and the mass of water lost in the effluent air is precise enough for predicting performance in most monitoring applications." Stated another way, the overall performance of a bubbler is primarily dependent on its retention capabilities since collection is essentially 100% at all times for HTO entering the system.

To evaluate the collection efficiency of the glycol bubbler, five tritium gas standards were passed through a heated palladium catalyst at a flow rate of 750 cm³/min into a 10-cm³ ethylene glycol bubbler. The results given in Table 4 indicate an average overall recovery for the oxidation and collection system of 99.4% with a standard deviation of 2.2%.

Dry air is used for flushing the collection chambers in the tritium leak rate determination, and the retention of tritium in the glycol bubbler is excellent over extended periods of time since essentially no water is being added to the system. In more typical air sampling situations, moisture in the air is added to the system continuously. The pure ethylene glycol is diluted as water is extracted from the air, and tritium losses occur through water lost in the effluent air.

Table 4

COLLECTION OF OXIDIZED HT USING
10-cm³ ETHYLENE GLYCOL BUBBLERS^a

<u>Sample Number</u>	<u>HT Added (dis/min)</u>	<u>HTO Recovered (dis/min)</u>	<u>Recovery (%)</u>
1	3.36 x 10 ⁵	3.35 x 10 ⁵	99.7
2	1.41 x 10 ⁵	1.44 x 10 ⁵	102.1
3	3.21 x 10 ⁵	3.08 x 10 ⁵	96.0
4	8.94 x 10 ⁵	8.96 x 10 ⁵	100.2
5	6.58 x 10 ⁵	6.51 x 10 ⁵	98.9
Average			99.4
Standard Deviation			2.2

^aAir flow 750 cm³/min.

To evaluate the performance of the ethylene glycol bubbler under more typical air sampling conditions, three bubblers in series were connected to a stack air sampling port. Standard 25-cm³ liquid scintillation counting vials were used for bubblers. An air flow rate of 125 cm³/min, stack effluent air* containing 11 g of water per m³, and sampling periods from 8 hr to 168 hr without changing the glycol in the bubblers were used in the evaluation. Three ethylene glycol bubbler volumes were evaluated (3-5 cm³, 3-10 cm³, and 3-15 cm³) for HTO retention with total air sampled ranging from 0.06 to 1.26 m³ of air. Retention of HTO by the 5, 10 and 15 cm³ bubbler train is shown in Tables 5, 6, and 7 respectively.

Table 5

RETENTION OF HTO IN 5-cm³ ETHYLENE GLYCOL BUBBLERS^a

<u>Time Run (hr)</u>	<u>Air Volume (m³)</u>	<u>% Retention #1 Bubbler</u>	<u>% Retention #2 Bubbler</u>	<u>% Retention #3 Bubbler</u>
8	0.06	95.2	4.7	0.1
16	0.12	89.2	10.4	0.4
24	0.18	79.9	18.3	1.8
72	0.54	57.6	29.3	13.1
168	1.26	<40.3	<35.1	<24.6

^aFlow rate 125 cm³/min; stack air 22°C; 50-60% relative humidity.

*Stack effluent air 22°C, relative humidity range 50% to 60%.

Table 6

RETENTION OF HTO IN 10-cm³ ETHYLENE GLYCOL BUBBLERS^a

<u>Time Run (hr)</u>	<u>Air Volume (m³)</u>	<u>% Retention #1 Bubbler</u>	<u>% Retention #2 Bubbler</u>	<u>% Retention #3 Bubbler</u>
8	0.06	97.7	2.2	0.1
16	0.12	93.9	6.0	0.1
24	0.18	92.0	7.8	0.2
72	0.54	68.4	26.2	5.4
168	1.26	<46.9	<34.7	<18.4

^aFlow rate 125 cm³/min; stack air 22°C; 50-60% relative humidity.

Table 7

RETENTION OF HTO IN 15-cm³ ETHYLENE GLYCOL BUBBLERS^a

<u>Time Run (hr)</u>	<u>Air Volume (m³)</u>	<u>% Retention #1 Bubbler</u>	<u>% Retention #2 Bubbler</u>	<u>% Retention #3 Bubbler</u>
72	0.54	81.3	16.9	1.8
96	0.72	77.3	19.6	3.1
168	1.26	61.1	30.0	8.9

^aFlow rate 125 cm³/min; stack air 20°C; 50-60% relative humidity.

Table 8

RETENTION OF TRITIUM OXIDE IN ETHYLENE GLYCOL BUBBLERS^a

<u>Time Run (hr)</u>	<u>Air Volume (m³)</u>	<u>Two 5-cm³ Bubbler (%)</u>	<u>Two 10-cm³ Bubbler (%)</u>	<u>Two 15-cm³ Bubbler (%)</u>
8	0.06	99.9	99.9	---
16	0.12	99.6	99.9	---
24	0.18	98.2	99.8	---
72	0.54	86.9	94.6	98.2
96	0.74	---	---	96.9
168	1.26	<75.6	<81.6	91.9

^aAir ~22°C; 50-60% relative humidity; flow rate 125 cm³/min.

A summary of the collection efficiencies for the first two bubblers in the sampling train for the three bubbler volumes evaluated is shown in Table 8. It is noted that the retention by two 5-cm³ bubblers for 0.18 m³ of air (98.2%) is identical to the retention of two 15-cm³ bubblers for an air volume three times greater (0.54 m³) indicating the linear relationship between air volume and bubbler volume for equivalent HTO bubbler retention under similar sampling conditions.

APPLICATIONS

Several applications of this technique are now in use at Mound Laboratory for tritium in air measurements. Listed in Table 9 are various applications, and the limit of detection which are compared to the RCG values for HTO in air.

Leak Rate Measurements

A method for determining leak rates (on the order of 1×10^{-17} cm³/sec and higher) from containers of tritium waste was developed using the catalyst-glycol bubbler method. The container to be checked is placed in a disposable can and after several days, the accumulated tritium is flushed from the can with compressed air through an ethylene glycol bubbler for collection of HTO. The output of the first bubbler is channeled through a heated palladium catalyst where the HT is oxidized and then to a second ethylene glycol bubbler. This technique replaced the procedures described by one of the authors at the Tritium Symposium where measurements were made by a gas proportional counting technique.⁸ Because of absorption problems this procedure required that the interior of the cans be coated with a cellulose acetate film which was stripped and dissolved in liquid scintillator solution for tritium analysis. This method was time consuming and did not offer the precision or accuracy attainable with the flushing system now in use.

Environmental Monitoring

Mound Laboratory will soon have in operation a network of 21 off-site differentiating tritium air samplers based upon this development work. In this application the lead HTO bubblers have been replaced with a silica gel column which will be returned to the laboratory for desorption on a weekly basis. Large volume (500 cm³) water bubblers now in use will be continued as a means of providing prompt information if needed. Originally Mound Laboratory planned to build differentiating air samplers based on the Ostlund technique of using palladium-coated silica gel for the combustion and collection of HT gas. The pilot unit and the companion desorption equipment was designed and built for Mound Laboratory by Monsanto Research Corporation, Dayton, Ohio. It is estimated that 21 sampling units based upon this particular design would have cost in excess of \$100,000. The 21 units now being fabricated and based upon the methods herein described will cost less than \$20,000. The operational cost of the system being adopted will be considerably less since the desorption work is cut in half over the former technique.

Stack Monitoring

In stack monitoring only the lead HTO and HT bubbler would be changed on a daily basis. If the lead bubbler is changed daily, the water content of the first bubbler is kept at a lower level - with less water and

Table 9

TRITIUM LEVELS ATTAINABLE WITH MOUND DIFFERENTIATING CONTINUOUS AIR SAMPLER

<u>Application</u>	<u>Collection Time</u>	<u>Detection Limit 99% Conf. Level</u>	<u>($\frac{\text{Detection limit}}{\text{RCG}} \times 100$)</u>
			<u>Percent of Applicable HTO Radiological Std.</u>
Emergency Radiological	10 min	$1 \times 10^{-9} \text{ } \mu\text{Ci/cm}^3$	0.02% 40 hr occupational 1.42% general population
Health Physics Monitoring	8 hrs	$2 \times 10^{-10} \text{ } \mu\text{Ci/cm}^3$	0.004% 40 hr occupational 0.01% 168 hr occupational
Stack Monitoring	8 hrs	$2 \times 10^{-10} \text{ } \mu\text{Ci/cm}^3$	0.01% individuals 0.03% general population
Environmental Monitoring	24 hrs	$1 \times 10^{-10} \text{ } \mu\text{Ci/cm}^3$	0.05% individuals 0.15% general population
Leak Rate Measurements	1 week	$1 \times 10^{-9} \text{ } \mu\text{Ci/min}$	N/A
Natural Background (HTO)		$1 \times 10^{-12} \text{ } \mu\text{Ci/cm}^3$	

hence less tritium lost to the second bubbler. Analysis of stack samplings using 10-cm³ bubblers and the lead bubbler changed daily showed losses from the lead bubbler to be 4% and less for a 5-day sampling period of 104 hr. This method of sampling would require changing the second bubbler only twice each week, following the weekend and prior to the weekend, to ensure 95% retention in the lead bubbler and 99% retention overall for sampling conditions at 22°C and 50% relative humidity with a sampling rate of 125 cm³/min.

Room Air Monitoring

The use of this technique for Health Physics surveillance of tritium work areas has great potential. This system, not being real-time monitoring, does not replace the need for Kanne Ionization Chambers and other real-time monitors that provide alarm systems and strip chart recorders for on the spot interpretation, but it does provide historical values of integrated biological dose levels on a daily basis for given work areas. The sensitivity this method provides would permit the evaluation of laboratory tritium levels orders of magnitude below current air concentration guide levels. With a conventional liquid scintillation counter background of 20 counts/min and a 1-min counting time, air concentrations orders of magnitude below the guide limit can be accurately determined.

With greater emphasis and need for reconstruction of radiological working environment, surveillance of this type is of great value. This technique can provide monitoring at levels never before considered feasible at minimal cost with precision and accuracy.

CONCLUSIONS

The use of glycol bubblers in conjunction with the heated palladium catalyst for collection of oxidized HT gas offers several advantages over other commonly used techniques. Methods that use catalyst-coated desiccants require desorption once the sample is collected. Freeze-out systems require carrier gas addition and of course a low-temperature bath. By using the glycol bubblers these additional requirements are eliminated and the sample is collected in a form that can be analyzed with no additional time or sample preparation.

The small volume bubbler does not impose as much limitation on the sensitivity as might appear. The limit of detection for systems that collect water is that volume of water that can be counted. Most liquid scintillation counting techniques can accommodate up to 10 cm³ of water for counting tritium. Unless a tritium enrichment process is applied, only that amount of air containing 10 cm³ of water can be collected for the purpose of increasing the sensitivity.⁹ Additional air increases only the amount of water collected. A 5-cm³ glycol bubbler will retain up to 20% water or 1 cm³ with minimal losses. Thus the bubbler collection system with its convenience offers a sensitivity at most only a factor of 10 greater than the desiccant or freeze-out systems.

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