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SAMPLING SYSTEM FOR TRITIUM AND CARBON-14  
IN ENVIRONMENTAL AIR

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ABSTRACT

An inexpensive sampler is described for general environmental sampling of tritiated water vapor and carbon-14 as  $\text{CO}_2$  in the air. The components cost less than \$20.00. For sampling tritium, use is made of the rapid exchange of water in a small silica gel column. A quick and easy method for extracting the sampled water from the column is also described. The sampler can detect less than 1% of the MPC for uncontrolled areas; its sensitivity is therefore adequate for most purposes.

## DESCRIPTION OF SAMPLER

1. Air Mover A small diaphragm pump, sold as an aquarium aerator, is used to circulate the air. This pump has a capacity far greater than needed, so a small filament transformer is used to reduce the voltage at the pump from 117 volts to 18 volts, thus ensuring quiet operation and less heat. All components are mounted in a small electronic cabinet.
2. Flow Regulation and Measurement The flow rate is controlled by a small needle valve and measured by a small plastic rotameter with a styrofoam float. The desired flow rate of 10 cc/minute corresponds to about 1/3 of full scale.
3. Tritium Sampler A column containing about 12 grams of 6-12 mesh silica gel is used for extracting the tritiated water vapor from the air. The silica gel is not previously dehydrated, but is allowed to equilibrate with ambient conditions before use. We have discovered that this in no way impairs its effectiveness in removing HTO vapor. <sup>(1)</sup>
4. Carbon-14 (CO<sub>2</sub>) Sampler A conventional technique of bubbling the air through a caustic solution is used. A wide mouth, 2 ounce bottle with a 2-hole rubber stopper serves as a convenient container and bubbler.

## OPERATING THE SYSTEM

The routine sampling period is one week at a flow rate of 10 cc/minute, giving a total sample volume of 1/10 cubic meter. At the end

of one week, the silica gel column is about half spent and about 30 cc of  $\text{CO}_2$  have been deposited in the bubbler solution. The sampling flow rate remains quite steady and no attention is required other than the weekly sample changes. Each week a fresh bottle of bubbler solution (0.2 molar NaOH) is put in place and the silica gel column is replaced.

A very simple technique for counting the tritium activity has been devised. We pour the exposed silica gel into 25 cc of water in a 100 cc beaker. It is stirred at this time and again  $1/2$  hour later. Thirty minutes after the last stirring, a 3 ml sample is withdrawn from the water and placed in a vial containing 17 ml of dioxane type liquid scintillation cocktail. Because the silica gel already contains 3 or 4 ml of water when poured into the beaker, the total volume of water present is close to 30 ml, and the 3 ml portion taken represents about a 10% aliquot. There is some error because of variations in humidity, but the error is only a few percent and of no consequence in ordinary environmental monitoring.

The sensitivity obtainable, using a 20 minute counting time, is about 1 nanocurie per cubic meter or 1% of the non-occupational MPC, and therefore this simple technique is adequate for most purposes.

The carbon-14 activity is also counted in a liquid scintillation counter. An excess of 12N sulphuric acid is added to the solution in the sample bottle, converting the collected carbonate into  $\text{CO}_2$ . The  $\text{CO}_2$  released is led directly into a counting vial and bubbled through

20 ml of liquid scintillation cocktail containing ethanolamine. Air is then bubbled through the acid bubbler solution to ensure that all  $\text{CO}_2$  is transferred into the counting vial. Limit of sensitivity is about 20 pCi/m<sup>3</sup>.

### COLLECTION EFFICIENCIES

1. A column was loaded with five 5-gram portions of silica gel, separated by fiberglass discs, and operated as a stack sampler for 1 week at 10 cc/minute. A low, steady concentration of HTO was present in the stack during this period.

Table 1. Distribution of HTO in the column.

<u>Portion</u>	<u>c/m Tritium</u>
1	1370
2	41
3	3
4	2
5	0

It is apparent that more than 99% of the tritium collected was in the first two 5-gram sections.

A further check was made by running two parallel samplers for 6 weeks. One was of the silica gel type described here; the other was a column of indicating drierite whose collected water was separated

from it by distillation with xylene.

Table 2. Comparison of the two Samplers.

Week	Concentration, nCi/m <sup>3</sup>	
	Silica Gel	Drierite
1	150	73
2	64	55
3	220	160
4	73	58
5	5.0	2.8
6	6.0	3.3

Apparently the silica gel method is more efficient than the drierite method. These findings are further substantiated by other work we have done with silica gel columns, work which formed the basis for a tritium control method described in Reference 1. The evidence is ample to prove that the collection efficiency is close to 100%.

2. CO<sub>2</sub> Bubbler Because bubbling air through an alkaline solution is a standard method for removing CO<sub>2</sub>, less extensive tests were made of this sampling technique. Two bubblers were operated in series for one week. Addition of barium ion to the first one produced a large quantity of BaCO<sub>3</sub> precipitate, but barium ion produced no visible precipitate when added to the second. There seems to be no doubt that a 0.2 molar NaOH solution is 100% effective in trapping CO<sub>2</sub>.



Retrieving  $\text{CO}_2$  from the bubbler solution is the difficult part of the process. When the solution is acidified, the gas that is formed tends to remain dissolved and only slowly evolves. About an hour of bubbling air through the solution is required to release most of the  $\text{CO}_2$ , as indicated by barium tests. The process is aided by the substantial amount of heat from neutralization and dilution of the acid.

Acknowledgment—Work done under the auspices of the U. S. Atomic Energy Commission.

## REFERENCE

1. R. G. Aune, H. P. Cantelow, R. L. Boltin, Complete Control of Tritium Water Vapor by the Use of Silica Gel, Lawrence Berkeley Laboratory Report LBL-2 (1971).



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Fig. 1. Environmental Tritium and  $^{14}\text{C}$  Sampler.

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