

DECONTAMINATION OF TRITIATED WATER SAMPLES PRIOR TO TRITIUM ASSAY

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The liquid scintillation assay of tritiated water samples in radiation protection control is often inaccurate and unreliable due to the presence of other radionuclides. A simple and efficient separation procedure based on distillation and recovery of the tritiated water was published not long ago (1). In the work presented here various aspects of the above method were investigated, with the view of improving the purification and decontamination of the sample from interfering nuclides. The technique is especially useful in the tritium assay of liquid waste effluents from hospitals, biological and veterinary institutes, as well as in the production facilities for labelled compounds.

METHOD

Measured aliquots of tritiated water samples, containing other additional radionuclides, are introduced into one of the distillation apparatuses shown in the illustrations. The larger apparatus (fig.1) consists of a test tube for 25 ml samples and is provided with a small aluminum condenser. The sample is heated to boiling in a glycerol bath and the distillate recovered into a 20 ml plastic vial. About 300 mg of a hold-back carrier are added prior to the distillation; it consists of a dry mixture of AgNO_3 (40%), NaI (20%), CuS (10%), anhydrous Na_2CO_3 (10%) and anhydrous $\text{Sr}(\text{NO}_3)_2$. CuS cannot be exchanged with sodium thiosulfate, which forms a soluble complex with AgI , but NaHSO_3 can be used. Salts containing crystallization water must be absent.

A device for smaller samples is useful when only limited amounts of tritiated water are available, or if volatile compounds of radioiodine are present (fig. 2). The glass-tripod with a 4 ml cup on top is placed into a 100 ml beaker and covered with a watch-glass (2); the water sample at the bottom of the beaker is slowly evaporated on a sand bath at 70°C and in presence of the hold-back carrier, until the cup is filled with a sufficient amount of distillate.

In both apparatuses most of the interfering nuclides are completely removed by the hold-back carrier, except for some traces of radiocesium and radoruthenium, which sometimes appear in the distillate. They are removed by shaking the sample with (< 100 mg of) $\text{K}_2\text{Co}[\text{Fe}(\text{CN})_6]$ before or after the distillation, and discarding the solid residue (3).

The sample should be distilled to dryness whenever time and the presence of foreign nuclides permit it. The separation lasts up to one hour with the larger apparatus and about 4 hours with the smaller one. Twelve samples can be easily separated in one run. The

purified sample is now mixed with the liquid scintillator and assayed.

RESULTS AND DISCUSSION

The method was tried out on aqueous solutions of sixteen of the most common nuclides (in ordinary water, no H-3 !), in order to determine their "decontamination factors". The following tracers (mostly as chlorides or nitrates) were used: Am-241, C-14 (as carbonate or uridine), Cl-36, Co-60, Cs-134, Fe-55, gold-198, Hg-203, I-125, Mn-54, Na-22, Ru-103, Sr-85, Tc-99, Th-230 and Zn-65; their activities ranged from 10^{-3} to 1.1 μCi . They were subjected to the distillation procedure either individually or in prepared mixtures. As expected, most of the radionuclides were absent from the distillate except for some traces of radioiodine, Hg-203 and Ru-103; decontamination factors of these nuclides are presented in table 1.

TABLE 1. Decontamination factors for I-125, Hg-203 and Ru-103;
R = cpm(distillate)/cpm(sample)

| Nuclide | sample (ml) | distillate (ml) | hold-back carrier | R |
|---------------------|----------------|--------------------|--|--------------------|
| I-125 | | | | |
| 0.3 μCi | 10 | 5 | Hamiltons (1) | 4×10^{-5} |
| " | 10 | 4 | " without CuS, | 10^{-3} |
| " | 10 | 4 | but with $\text{Na}_2\text{S}_2\text{O}_3$ | |
| | | | Hamiltons, no CuS, | 7×10^{-5} |
| | | | but with NaHSO_3 | |
| I-125 | | | | |
| 0.05 μCi | 25 | first 8 | Hamiltons | 3×10^{-4} |
| " | 25 | second 8 | " | 4×10^{-4} |
| " | 25 | last 9 | " | 0.034 |
| Hg-203 | | | | |
| 0.02 μCi | 18 | first 15 | " | 2×10^{-5} |
| " | 18 | last 3 | " | 10^{-4} |
| Ru-103 | | | | |
| 0.02 μCi | 20 | first 7 | " | 0.027 |
| " | 20 | second 7 | " | 7×10^{-3} |
| " | 20 | last 6 | " | 0.026 |

All the activities were measured by liquid scintillation counting using the Instagel scintillator. A Packard Tricarb Model 3390 spectrometer was used, at optimum gain settings and window openings.

OPTIMAL FIGURES OF MERIT FOR TRITIUM COUNTING

The "figure of merit" (FM) in tritium assay is obtained by multiplying the sample volume with the counting efficiency ϵ . The highest FM with polyethylene vials (20 ml) were with 7 ml of sample and 9 ml of Instagel ($\epsilon = .18$); with the small (7 ml) vials FM was highest for a 1:1 ratio in a 5 ml total volume ($\epsilon = .16$).

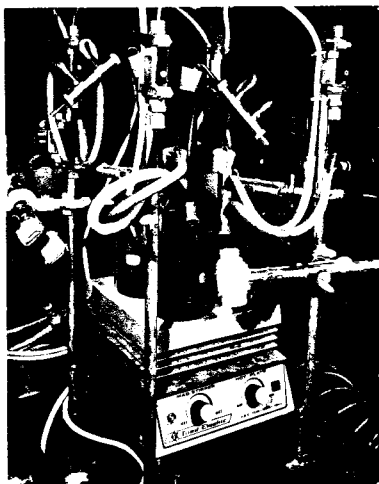


Figure 1.



Figure 2.

REFERENCES

1. Hamilton, R.A. (1974) : Determination of Tritium in Waste Processing Effluents by Distillation and Liquid Scintillation Emulsion Counting. - USAFC report no. ARH-SA-188.
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3. Boni, I.L. (1966) : Analyt. Chem., 38, 89.