Evaluation of Tritium Distillation Utilizing Gas Permeable Membrane

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Introduction

Determination of tritium in water, sludge and sanitary sewer discharge was performed by distillation using a gas permeable membrane apparatus. Distillates were counted by liquid scintillation. Results were compared to traditional glass distillation technique. MICRO DIST® tubes were supplied by Zellweger Analytics Inc. for evaluation of tritium separation by gas permeable membrane. MICRO DIST® tubes feature a gas permeable membrane fitted into a disposable plastic tube with detachable sample collection vial.

Equipment

Micro Dist® disposable distillation tube[1]
heating mantel
rheostat
ring stand
clamp
100 and 500 ml boiling flask
scintillation vial
Allihn condenser
Scintillation cocktail
Liquid Scintillation Counter

Procedure[2]

Samples were placed in either a 100 or 500 ml boiling flasks and placed in a heating mantel connected to a variable power supply. The MICRO DIST® tube was attached to the boiling flask and secured in place with a clamp. The mantle power was set to produce a vigorous boil. Gas pressure forced steam through the membrane allowing liquid to condense in the collection vial. After collection of sufficient water, the MICRO DIST® tube was disconnected from the boiling flask and the collection vial was sealed to allow the distillate to cool. The cooled distillate was counted one hour by liquid scintillation.

Analyses were performed on spiked reagent water, sludge samples and sewer discharge samples. Reagent water was spiked with H3, C14, S35 and Cs137 with distillation by membrane and glass condenser to compare the two techniques under controlled conditions. Both distillate and remaining sample were analyzed. Sludge samples containing background activity were evaluated for visible interference i.e. collection of volatile organic material. The sewer discharge contained significant concentrations of H3 and S35 as well as volatile organic material. H3 results were compared for membrane and glass distillation, S35 breakthrough was evaluated and visible organic content was evaluated.

Results

H3 results for all reagent water analyses were within 15% of the expected value. C14, S35 and Cs137 were nondetectable in the distillate with detection limits of 5-10% of the added activity.

Sludge distillation produced similar results by both techniques. The major difficulty in sludge distillation was controlling foam during boiling. In both types of distillation, “boiling over” will ruin the analysis.

H3 results for sewer discharge for the two techniques were within 11% of each other. S35 breakthrough in the gas membrane was approximately 1.8%. S35 breakthrough in glass distillation was not evaluated. However, glass distillation allowed significant visible material to collect in the distillate. Distillate from the membrane was free of solids.

Conclusions

Gas permeable membrane distillation appears to be comparable to glass distillation. The membrane system is simpler allowing time savings during set-up, and clean-up. The membrane system is disposable reducing potential contamination and saving glassware washing time. The membrane system appears to remove some volatile materials which are able to move through the glass condenser. The glass distillation allows collection of a greater amount of distillate, while the membrane system is limited to about 10 ml of distillate. The membrane system has an addition hazard due to the closed system creating pressurized steam produced during distillation.

References

1. MICRO DIST® Simple Effective Distillation, Zellweger Analytics Inc., 1996

2. Water distillation from soil and aqueous matrices using micro-distillation system for tritium distillation, DOE Methods—RP580a, April 1995.