GROSS ALPHA RADIOACTIVITY IN WATER

1. SCOPE

1.1. This is a method for the measurement of gross alpha radioactivity in water samples.

1.2. This method does not address all aspects of safety, quality control, calibration or instrument set-up. However, enough detail is given for a trained radiochemist to achieve accurate and precise results for the analysis of the analyte(s) from the appropriate matrix, when incorporating the appropriate agency or laboratory safety, quality and laboratory control standards.

2. SUMMARY OF METHOD

2.1. The alpha emitting radionuclides are scavenged from a water sample by batch contact with Eichrom's Actinide Resin. The resin is transferred to a liquid scintillation vial where the alpha radioactivity is counted directly by LSC.

3. SIGNIFICANCE OF USE

3.1. This method is a rapid, reliable method for measurement of total alpha radioactivity in water samples that is more cost-effective and efficient than traditional techniques.

4. INTERFERENCES

4.1. High levels of calcium (above 70 ppm) and magnesium in the sample may interfere with radium absorption on the Actinide Resin. In these cases a smaller aliquot of sample or a larger amount of resin may be indicated.

5. APPARATUS

- Analytical balance, 0.0001 g sensitivity
- Beakers, various sizes
- Filter, 0.45 micron
- Fume hood

- Liquid scintillation counter with alpha/beta discrimination capability
- Liquid scintillation vials
- Magnetic stir bars, suitable for 1L samples
- Petri dishes, plastic, 5-1/2 x 1 cm
- Stir plate
- Vacuum filter apparatus
- Watch glasses

6. REAGENTS

Note: Analytical grade or ACS grade reagents and trace metal grade (or equivalent) acids are recommended. Evaluation of key reagents, such as aluminum nitrate and ammonium hydrogen phosphate, for contribution to method background levels from naturally occurring radioactive materials is recommended.

Actinide resin, 100-150µm particle size resin
Deionized water, All reagents are prepared using deionized water
Hydrochloric acid (37%), concentrated HCl
Ultima Gold™ LLT liquid scintillation cocktail

6.1. *Hydrochloric acid (0.5M)*- Add 42mL of concentrated HCl to 900 mL of water. Dilute to 1L with water.

7. PROCEDURE

7.1. Water Sample Preparation:

7.1.1. If required, filter the sample through a 0.45 micron filter.

7.1.2. Aliquot 100 mL of the sample into an appropriately sized beaker.

7.1.3. If the sample was not acidified to pH 2 in the field, add enough concentrated HCl to adjust the solution to pH 2.

7.2. Concentration of alpha emitters using Eichrom's Actinide Resin:

7.2.1. Add 0.5 gram of Actinide Resin to the acidified sample.

7.2.2. Add a magnetic stir bar and place beaker on a stir plate. Stir the Actinide Resin into the solution for a minimum of 4 hours. (Overnight stirring for 18 hours has been used routinely.)

7.3. Collection of the Actinide Resin

7.3.1. Using a vacuum filtration apparatus, collect the Actinide Resin on a 0.45 micron filter.

7.3.2. Rinse the resin in the filtration apparatus with water.

7.3.3. Transfer the filter paper from the filtration apparatus to a watch glass and dry gently under a heat lamp for approximately 15 minutes.

7.3.4. Carefully transfer the slightly dried resin from the filter paper to a scintillation vial using 1mL of 0.5M HCl.

7.3.5. Add 10mL Ultima Gold LLT liquid scintillation cocktail and mix well on a vortex (or equivalent) mixer. Prepare a blank by adding 0.5 gram Actinide resin, 1mL 0.5M HCl and 10mL Ultima Gold LLT cocktail to a LSC vial.

7.3.6. Measure the total alpha activity in the sample and blank vials on a liquid scintillation counter LSC with alpha beta discrimination capability. Refer to the following comments on set up of the LSC instrument.

7.3.6.1. Alpha window: 90-500 keV

7.3.6.2. Discriminator Setting: The presence of the Actinide Resin in the scintillation vial and the amount of cocktail affect the discriminator setting. It is necessary to follow the instrument manufacturer's guidelines for determining the correct discriminator setting. Once determined, the setting should not change between samples if the tSIE levels do not vary significantly.

7.3.7. Using a Packard Tricarb 2550, 0.5 gram Actinide resin and 10mL cocktail, we found a discriminator setting of 124 to result in the minimum misclassification of alpha and beta when tSIE values are approximately 400.

7.3.8. Typically, we measured blank counts of 0.5 to 1.0 cpm in the window referenced in section 7.3.6.1. The average counting efficiency measured on direct spikes of U-234/238 and Am-241 was 95.5%.

7.3.9. Based on a blank count rate of 0.75 cpm and a 60 minute count time, an MDA of 3 pCi/L is achievable with a 100 mL sample size.

8. CALCULATIONS

Calculate the alpha activity as follows:

$$\text{alpha activity (pCi / L)} = \frac{S - B}{E \times V \times 2.22}$$

where:

S = sample activity, cpm
B = blank activity, cpm
E = counting efficiency
V = sample volume, liters

To convert from pCi/L to Bq/L multiply by 0.037.

9. REFERENCES

1) Horwitz, E.P., et al. “Dipex: A new extraction chromatographic material for the separation and preconcentration of actinides from Aqueous solution” Reactive & Functional Polymers (1997)

2) ASTM Method D7283-13, “Standard Test Method for Alpha and Beta Activity By Liquid Scintillation Counting.”