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Technetium-99 in Water (TEVA Disc Method)

1. Scope

1.1 This procedure describes a method to separate and measure technetium-99 in water.

2. Summary of Method

2.1 Technetium-99 is separated from water samples using Eichrom's TEVA Disc prior to liquid scintillation counting. After concentration of the pertechnetate ion (TcO_4^-) on the disc, Tc-99 is measured by liquid scintillation counting by adding the disc directly to the scintillation cocktail. The sample can be analyzed with and without a Tc-99 spike to determine chemical recovery. Alternately, either of the short-lived gamma emitters, Tc-99m or Tc-95m, can be used as a tracer. The detection limit for this method is 1 pCi/L (37 mBq/L).

3. Significance of Use

3.1 This method is a rapid, reliable method for measurement of Tc-99 in environmental samples that combines the selectivity of Eichrom's TEVA Resin with the fast flow rates achievable with a disc format.

4. Interferences

4.1 All radionuclides that undergo beta emission or quench the liquid scintillation counting are effectively removed (including C-14, P-32, S-35, Cs-137, and Sr-90) using the Eichrom TEVA disc. Tritium may sometimes follow the technetium due to the absorption of tritium-labeled compounds by the disc. Possible interference by tritium is eliminated by setting the Tc-99 counting window above the maximum energy for tritium beta particles.

4.2 Organic matter present in the sample can interfere by quenching during liquid scintillation counting.

4.3 Six (6) liters of solution may be passed through an Eichrom TEVA Disc without breakthrough of Tc-99. Larger volumes have not been tested.

5. Apparatus

5.1 *Filter apparatus:* suitable to hold 47mm TEVA Disc: Nalgene Reusable Filter Holder recommended (nalgene part # 300-4000 or 300-4050 or 300-4100)
5.2 *Forceps*
5.3 *Glass beakers*
5.4 *Liquid scintillation counter*
5.5 *Liquid scintillation vials, glass*
5.6 *Watch glass*

6. Reagents

6.1 Unless otherwise indicated, all references to water should be understood to mean deionized distilled water.
6.2 *Hydrogen peroxide (30 wt %)*
6.3 *Liquid scintillation cocktail* - Ultima Gold®-LLT (Packard Instrument Company)
6.4 *Nitric acid solution (0.01M)*- Add 640 µL concentrated HNO_3 (sp gr 1.42) to 900 mL water and dilute to 1L with water.

6.5 *TEVA Disc* - 47 mm, Part number: TE-D50-FX (50 discs) or TE-D200-FX (200 discs)

7. Procedure

7.1 *Water Sample Preparation:*

7.1.1 Measure the sample volume using a graduated cylinder (or equivalent) and transfer the volume to an appropriate size beaker.

7.1.2 Adjust the pH of the sample to pH 2, if required.

7.1.3 Analyze the sample with and without adding Tc-99 spike to determine chemical recovery.

> Note: An alternative is to use Tc-99m or Tc-95m as a tracer, measuring the gamma activity of Tc-99m or Tc-95m. If Tc-99m is used, allow the Tc-99m to decay for approximately 1 week after measuring the gamma activity and before measuring Tc-99 using liquid scintillation counting.

7.1.4 Add 10 mL of 30 wt % H_2O_2 (per liter of sample) to the beaker, stir and cover with a watch glass. Remove cover once the sample begins to boil.

7.1.5 Heat the sample to about 90°C for 1 hour to oxidize Tc(IV) toTc (VII), forming TcO_4^-, oxidize some of the organics present, and destroy excess H_2O_2.

7.1.6 If bubbling due to decomposition of the hydrogen peroxide has not stopped as the sample cools, continue heating until bubbling has stopped. Stir occasionally with glass stirring rod.

7.1.7 Allow the beaker to cool to room temperature.

7.1.8 If the sample contains insoluble matter, filter the sample to remove solids prior to using the TEVA Disc.

7.2. *Eichrom TEVA Disc separation*

7.2.1 Using forceps, carefully place a TEVA Disc on the filtering apparatus.

7.2.2 Place the funnel over the disc and transfer the water sample from step 7.1.7 into the filtering funnel.

7.2.3 Allow the sample solution to filter through the disc by gravity or vacuum. (No vacuum is necessary. Depending on which filtering apparatus is used, it is possible to achieve a flow rate as high as 33 mL/min with gravity. An average of 98% Tc-99 recovery achieved using 200 mL/min with vacuum on 1 L simulated ground water sample type)

7.2.4 After filtering the sample, rinse the original beaker or container with the minimal volume of water required (depending on beaker size) and transfer this rinse into the filtering funnel.

7.2.5 Allow the rinse solution to drain through the disc completely.

7.2.6 Rinse disc with 50 mL 0.01M nitric acid solution.

7.2.7 Squirt 5 mL of water using a squeeze bottle to rinse the sides of the funnel. This will remove any residual material of the disc which may have suspended during filteration.

7.2.8 Connect the filtering apparatus to a vacuum pump (if gravity flow used to this point) and apply vacuum for a few seconds to remove any residual liquid from the disc. Alternately, if the filtration apparatus has a Luer fitting, attach a 50 cc syringe and draw residual liquid from the disc.

7.3 *Counting preparation:*

7.3.1 With forceps, remove the TEVA Disc from the filtering apparatus and transfer it into a liquid scintillation vial.

Note: The disc can be rolled gently while it is still on the filter apparatus so that it can be easily inserted into the LSC vial.

7.3.2 Add 15 mL of Ultima Gold LLT into the LSC vial containing the disc. Cap the vial and vortex until the disc disintegrates and forms a colorless translucent gel.

7.3.3 Set up the scintillation counting window to measure from 26 Kev to 350 Kev or according to lab protocol.

8. Calculations

8.1 *Calculate chemical yield*

8.1.1 *Measurement of spiked and unpsiked samples*

$$Y = \frac{(C_S - C_U)}{E \times A_S}$$

where:

Y = chemical yield
C_S = count rate spiked sample, cpm
C_U = count rate unspiked sample, cpm
E_S = counting efficiency for Tc-99
A_S = Tc-99 tracer activity, dpm, corrected for decay from reference date

8.1.2 *Tc-99m (or Tc-95m) Tracer Method*

$$Y = \frac{(Cs - Bs)}{Es * As}$$

where:

C_S = measured Tc-99m (or Tc-95m) tracer, gamma cpm
B_S = background, gamma cpm
E_S = gamma counting efficiency for Tc-99m (or Tc-95m)
A_S = Tc-99m (or Tc-95m) tracer activity, dpm, corrected for decay from reference date

8.2 *Calculate the Tc-99 activity as follows:*

$$A = \frac{(S - B)}{E \times V \times Y \times 2.22}$$

where:

A = Tc-99 activity in the sample (pCi/L)
S = sample counts/time in minutes, cpm

B = blank counts/time in minutes, cpm
E = counting efficiency for Tc-99 determined from a direct spike of Tc-99 in LSC vial containing a TEVA Disc dissolved in 15 mL Ultima Gold LLT.
V = sample volume, L
Y = chemical yield (determined in section 8.1)

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(3) Mann, Darrin. Interview: K-25 Plant, Oak Ridge Site.

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(5) Wyse, E.J. and Fadeff, S.K., "Alternative Techniques for the Determination of Technetium-99 in Groundwaters: ICP/MS and Extraction Resin." Submitted for publication.