

TECHNETIUM-99 IN URINE

(TEVA DISC METHOD)

1. SCOPE

- 1.1. This procedure describes a method to separate and measure technetium-99 in urine.
- 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 sample is digested with nitric acid and evaporated to dryness. The residue is ashed in the muffle furnace. The urine ash is redissolved in weak nitric solution. The pertechnetate ion (TcO_4^-) is concentrated on a TEVA disc. ^{99}Tc is measured by liquid scintillation counting by adding the disc directly to the scintillation cocktail. The sample can be analyzed with and without a ^{99}Tc spike to determine chemical recovery. The short-lived gamma emitter, $^{99\text{m}}\text{Tc}$, can also be used as a tracer.

3. SIGNIFICANCE OF USE

- 3.1. This is a rapid, reliable method for measurement of ^{99}Tc 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. Beta emitting radionuclides (including ^{14}C , ^{32}P , ^{35}S , and ^{90}Sr) and components that quench the liquid scintillation counting are effectively removed using Eichrom TEVA Resin. Tritium may follow the technetium due to the absorption of tritium-labeled compounds by the resin. Possible interference by tritium can be minimized by setting the ^{99}Tc liquid scintillation 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.

5. APPARATUS

- Beakers, glass
- Filter apparatus: suitable to hold 47mm TEVA Disc: Nalgene reusable filter holder recommended (Nalgene Part: 300-4000 or 300-4050 or 300-4100)
- Forceps
- Hot plate
- Liquid scintillation counter
- Liquid scintillation vials, glass
- Muffle furnace
- Watch glass

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.

<i>Deionized water</i> , all reagents are prepared using deionized water
<i>Hydrogen peroxide (30%)</i> , concentrated H ₂ O ₂
<i>Liquid Scintillation Cocktail</i>
<i>Nitric acid (70%)</i> , concentrated HNO ₃
<i>TEVA® Disc</i> , 47 mm, Part TE-D50-F (50 discs) or TE-D200-F (200 discs)

6.1. *Nitric acid solution (0.01M)*- Add 640 µL concentrated HNO₃ (sp gr 1.42) to 900 mL water and dilute to 1L with water.

7. PROCEDURE

7.1. Urine Sample Preparation:

- 7.1.1. Measure 300mL of sample using a graduated cylinder (or equivalent) and transfer the volume to an appropriate size beaker. A larger sample may be used as needed.
- 7.1.2. Add 30mL of concentrated nitric acid to each sample.

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

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

- 7.1.4. Evaporate sample to dryness at 90°C. (Caution: Sample may ignite as it nears dryness, beaker should be removed from the hot plate, if this occurs).
- 7.1.5. Cool the beaker and add 10mL of concentrated nitric acid, by rinsing the walls of the beaker.
- 7.1.6. Evaporate to dryness at 90°C. Cool the beaker.
- 7.1.7. Place the beaker in a muffle furnace and muffle the sample at 500°C for 30 minutes, until a white residue is achieved.
- 7.1.8. If the sample residue is not completely white then repeat steps 7.1.5 through 7.1.7.
- 7.1.9. Cool the beaker. Add 5mL of concentrated nitric acid and evaporate to dryness.
- 7.1.10. Add approximately 500mL of water and 0.5mL of nitric acid to the residue and stir to dissolve the residue.

Note: Additional water and nitric acid could be added to dissolve the residue.

- 7.1.11. Add 5mL of H_2O_2 to the solution and place the beaker on a hot plate.
- 7.1.12. Heat the sample solution to about 90°C for 1 hour to oxidize Tc(IV) to Tc(VII), forming TcO_4^- .
- 7.1.13. 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.
- 7.1.14. Allow the beaker to cool to room temperature.
- 7.1.15. 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 33mL/min with gravity. Vacuum may be used at up to 100mL/min.
 - 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 50mL 0.01M nitric acid solution.
 - 7.2.7. Add 5mL 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 filtration.
 - 7.2.8. Connect the filtering apparatus to a vacuum pump (if gravity flow was used to this point) and apply vacuum for a few seconds to remove any residual liquid from the disc. Alternatively, if the filtration apparatus has a Luer fitting, attach a 50cc 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 15mL 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

Calculate chemical yield

Measurement of spiked and unspiked 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

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

$$Y = \frac{(C_S - B_S)}{E_S \times A_S}$$

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

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)

9. REFERENCE

- 1) DOE Methods Compendium, RP550. "Technetium-99 Analysis Using Extraction Chromatography,"
- 2) Banavali, A.D., "The Determination of Technetium-99 in Low-Level Radioactive Waste, *Radioactivity & Radiochemistry*," 6(3), 26-35 (1995).
- 3) Sullivan, T., et al., "Determination of Technetium-99 in Borehole Waters Using an Extraction Chromatographic Resin," 37th Annual Conference on Bioassay, Analytical and Environmental Radiochemistry, Ottawa, Canada. 1991.
- 4) Mas, J.L. "Method for the detection of Tc in seaweed samples coupling the use of Re as a chemical tracer and isotope dilution inductively coupled plasma mass spectrometry," *Analytica Chimica Acta*, 509, 83-88 (2004).