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Chen, Qing Jiang; Aarkrog, A.; Dahlgaard, H.; Nielsen, Sven Poul; Jensen, H.L.; Bruun, J.; Holm Pedersen, A.; Mandrup, K.

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Qing Jiang Chen, Asker Aarkrog, Henning Dahlgaard, Sven P. Nielsen, H.L. Jensen, Jette Bruun, Anna Holm Pedersen, Karen Mandrup

Health Physics Department

Risø National Laboratory, DK-4000 Roskilde, Denmark July 1988



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Abstract. The chemical yield of technetium by an anion exchange (AG 1-X₄) procedure is determined under different conditions from 200 liters sea water. A new method for collecting ⁹⁹Tc from 200-400 liters sea water has been developed by anion exchange. The evaporation rates of RuO₄ and TcO₄⁻ are determined at 100°C in 0.1 N H₂SO₄. A higher decontamination factor 2.5 \times 10⁵ - 2.4 \times 10⁷ for ruthenium is obtained by evaporating the RuO₄ at 100°C in 0.1 N H₂SO₄. The ruthenium is oxidized to RuO₄⁻ with NaClO at pH = 9. Fe(OH)₃ scavenging, AgCl precipitation and 5% TIOA/xylene extraction at controlled valence are used for decontamination of ruthenium, ^{110m}Ag and other nuclides. The chemical yield of technetium-99 in the procedure is about 70%.

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Introduction

Previous work¹ has demonstrated that an anion exchange separation step may be used for the purification of a technetium-99 sample. We have developed a purification procedure² which is a solvent extraction at controlled valence. The technetium-99 is taken from large volume sea water samples collected by $Fe(OH)_2$ precipitate. A large amount of iron is included in the technetium sample. This makes it difficult to purify the sample.

It was therefore necessary to develop a new method for collecting the technetium-99 from large volumes of sea water and for ruthenium decontamination from the technetium sample.

Experimental

1. Determination of the chemical yield of technetium-99 for anion exchange from 200 litres sea water by 3 different pretreatments:

- a) Filter the 200 litres sea water through a filter paper in order to separate the algae etc. Add ^{99m}Tc tracer and 100 ml 30% H_2O_2 to the sea water. Stir with air for 30 min to get TcO_4^- and to allow isotope exchange to be completed. Acidify the 200 litres sea water to pH = 1 with concentrated H_2SO_4 . Stir 30 min again. Prepare three 11 × 2.5 cm (i.d.) anion exchange (AG1-X₄ 100-200 mesh) columns. Load the resin by passing the 200 litres sea water through the 3 columns with a pump (see Figure 1). The speed is 50 ml/min for each column. Wash with 500 ml distilled water, 500 ml 0.1 N HNO₃ and 100 ml 1 N HNO₃ for each column. Strip with 200 ml 10 N HNO₃ for each column. After 25 ml, wait 80 min for ion exchange. It takes 2 hours to finish the stripping. Measure the chemical yield.
- b) Filter the 200 l sea water. Add ^{99m}Tc tracer. Stir with air pump for 1 h to get a uniform mixture. Give same treatment as described under a).
- c) Filter the 200 litres. ^{99m}Tc is reduced to ^{99m}Tc⁺⁴ by adding a solution containing 2 mg/ml K₂S₂O₅ in 0.5 N HCl for 1 h. Add the ^{99m}Tc⁺⁴to the 200 litres sea water. Stir with air for 1 h to get a uniform mixture. Continue as described under a).

The results are shown in Table 1 (see Appendix 1).

2. Determination of ¹⁰³Ru evaporation rate in the 0.1 N H₂SO₄ solution:

Add ¹⁰³Ru tracer, 0.5 ml 5 mg RuCl₃/ml carrier and 2 ml 5% NaClO to 100 ml beaker. Add NaOH to the solution to get pH = 9. Heat 10 min to get RuO₄⁻ and to allow isotope exchange to be completed. Acidify the solution to pH = 1 with H₂SO₄. Heat the solution to evaporate the RuO₄. Measure the counts of ¹⁰³Ru at different times comparing with a standard source. The evaporation rate of the RuO₄ is shown in Table 2 (see Appendix 1).

3. Determination of technetium evaporation rate in 0.1 N H_2SO_4 at 100°C.

Add ^{99m}Tc, ruthenium carrier 5 mg RuCl₃/ml and 2 ml 5% NaClO to a 100 ml beaker. Adjust pH = 9 with NaOH. Heat for 10 min to get a uniform mixture. Acidify the solution to pH = 1 with H₂SO₄. Heat for 30 min in order to evaporate the RuO₄ (carrier). Repeat the operation two times more.

Procedure

Principle

The TcO_4^- is intensely absorbed on an anion exchange resin³. It is therefore difficult to strip the TcO_4^- , but it may be done by a higher affinity group NO_3^- . The high chemical yield of ^{99m}Tc for anion exchange is shown in Table 1. This is the first step for separating the technetium from lower valence cation and a large amount of salt. The second step is oxidation of the technetium and ruthenium to RuO_4^- and TcO_4^- with 5% NaClO at pH = 9. Acidify the solution to pH = 1 with H₂SO₄. Heat the solution at 100°C. The RuO₄ is evaporated. But the TcO_4^- is left in the solution as shown in Tables 2 and 3. This step is taken especially to decontaminate the ruthenium.

The Fe(OH)₃ scavenging and AgCl precipitate are used for cleaning for ^{110m}Ag and other nuclides. This is the third step.

The fourth step is decontaminating for ruthenium and other nuclides by extraction of the TcO_4^- leaving the ruthenium in 1 N H₂SO₄. The valence is controlled with H₂O₂ by 5% TIOA/xylene extraction.

Determination of chemical yield of ⁹⁹Tc and decontamination factor for ¹⁰³Ru of the procedure.

- Acidify 10 litres sea water to pH = 1 with HCl. Add 32 g FeSO₄.7 H₂O, ^{99m}Tc, 60 g K₂S₂O₅. Shake to get a uniform mixture. Let stand for 1 h to get Tc⁺⁴.
- 2. Add NH₄OH to pH = 9. Let stand for 4 h. Discard the sea water. Centrifuge. Dissolve the $Fe(OH)_2$ with 60 ml 10 N H₂SO₄. Add 60 ml 15% NaClO to get TcO_4^- . Add 0.5 ml (5 mg/ml) AgNO₃. Heat for 30 min. Cool and filter the solution. Dilute to 600 ml to get 1 N H₂SO₄.
- 3. Add solution to column 11 cm \times 0.7 cm (AG1-X₄ 50-100 mesh). The speed is about 10 ml/min.
- 4. Wash with 100 ml ion exchange water, 100 ml 0.1 N HNO₃ and 40 ml 1 N HNO₃.
- 5. Strip with 50 ml 7 N HNO₃. After 5 ml, wait for 30 min for ion exchange. It takes 50 min to finish the stripping.
- 6. Evaporate solution to 0.2 ml.
- 7. Add NaOH to pH = 9, 2 ml 5% NaClO, 0.4 ml RuCl₃ (5 mg/ml), and ¹⁰³Ru. Heat for 10 min to allow isotope exchange to be completed. Acidify the solution to pH = 1 with H₂SO₄ to evaporate the RuO₄. Heat for 30 min.
- 8. Repeat step 7 two times.
- 9. Acidify solution to 1 N H₂SO₄. Add 1 ml 30% H₂O₂. Dilute to 70 ml.
- Extract with 35 ml 5% tri-isooctylamine (TIOA)/xylene. Shake for 5 min. Discard the aqueous phase. Wash with 50 ml 1 N H₂SO₄ containing a few drops 30% H₂O₂. Shake for 2 min.
- 11. Back extract with 20 ml 0.5 N NaOH two times. Shake for 2 min.
- 12. Evaporate solution to 20 ml for electrodeposition. The current is 300 mA. The time is about 5 h (or overnight). The results are shown in Table 4 (see Appendix 1).

Flow sheet of the procedure for 200 litres sea water.

- L
- 1) Filter 200 litres sea water through 4 filters (filter paper). This takes 1 h.
- 2) Add ^{99m}Tc and 100 ml 30% H₂O₂. Stir to uniformity to get TcO₄⁻ and allow the isotope exchange to be completed. Let stay for 30 min.
- Acidify the 200 litres sea water to pH = 1 with conc. H₂SO₄. Stir with air pump for 1 h.
- 2.
- 1) Add anion ion exchange AG 1-X₄ resin (100-200 mesh) to the beaker with 1 N H₂SO₄ and stir to uniformity.
- 2) Make a column which is 11 cm long and with a diameter of 2.5 cm in 1 N H₂SO₄.
- 3) Add the sea water to three parallel columns. The speed is about 50 m.l/min for each column. This takes 22 h.
- 4) Wash each column with 500 ml ion exchange water, 500 ml
 0.1 N HNO₃ and 100 ml 1 N HNO₃.
- 5) Strip the Tc with 200 ml 10 N HNO₃ for each column. Eluate with 25 ml for each column and wait for 80 min for the ion exchange. Use about 2 h for stripping.
- 3.

 Add a few drops AgNO₃ carrier. Evaporate the 600 ml stripping solution to 0.5 ml.

 Add 0.5 ml 10 N H₂SO₄. Dilute to 20 ml. Add 1 ml 5% NaClO. Heat for 10 min.

4.

 Dilute to 50 ml. Add 0.5 ml 20 mg/ml AgNO₃ and 0.5 ml 10 mg/ml FeCl₃. Add NaOH to pH = 9. Heat for 5 min. Filter the solution. Wash the precipitate three times with water (pH = 9). Discard the precipitate.

- 5. 1) Add 0.5 ml 5 mg/ml RuCl₃ and 1 ml 5% NaClO. Keep pH =
 9. Heat for 10 min to allow the isotope exchange to be completed and to get RuO₄. (The solution should be bright yellow. If it is not add more 5% NaClO). Add 1 ml 5% NaClO.
 - 2) Acidify the solution to pH = 1 with 10 N H₂SO₄.
 - 3) Heat for 15 min in order to evaporate the RuO₄. The bright yellow disappears and solution is colourless.
- 6.

1) Transfer solution to another beaker. Keep the volume at 70 ml.

- 2) Repeat step 5.
- 7.

1) Transfer solution to another beaker. Keep the volume at 70 ml.

2) Repeat step 5.

8.

- 1) Acidify solution to 1 N H₂SO₄. Add 1 ml 30% H₂O₂ and dilute to 100 ml.
- 2) Extract with 80 ml 5% TIOA (tri-isooctylamine)/xylene. Shake for 5 min.
- 3) Wash with 80 ml 1 N H_2SO_4 containing 0.5 ml 30% H_2O_2 . Shake for 2 min.
- 4) Wash with 80 ml H_2O . Shake 5 min.

9,

Backextract with 40 ml 0.5 N NaOH. Shake for 2 min.
 Backextract again with 20 ml 0.5 N NaOH. Shake for 2 min. Combine the two solutions.

- 10. 1) Evaporate the solution to 20 ml for electrodeposition.
 2) The current is 300 mA. The time is at least 5 h or overnight (about 10-24 h).
 3) Wash the plate with pH = 9 water once.
 - 4) Count the chem. yield.

Flow sheet of the procedure collecting the 99 Tc from Fe(OH)₂ (see Appendix 2).

Flow sheet of analysis of the seaweed (see Appendix 3).

Results and Discussion

Anion exchange

The TcO_4^- is is intensely absorbed on anion exchange AG1-X₄ (100-200 mesh) in 0.1 N -1 N H₂SO₄. Technetium-99 is efficiently collected with 3 columns (11 × 2.5 cm AG 1-X₄) from 200 litres (6 columns for 400 litres and 1 for 50 litres of sea water). Technetium-99 is concentrated from a large amount of salt. The columns are washed with ion exchange water, 0.1 N HNO₃ and 1 N HNO₃ for decontaminating from one, two and three valence cation nuclides. Technetium is successfully stripped with 200 ml 10 N HNO₃ for each column. It takes 2 h for stripping ⁹⁹Tc. The HNO₃ is easily evaporated for next step purification. The chemical yield of ⁹⁹Tc for anion exchange is about 95%. Technetium loss in the column is about 5%. The sea water must be filtered because the organic matter and dirt will destroy the column and thus decrease the chemical yield of technetium.

There are three alternative methods shown in Table 1 for anion exchange of 200 litre sea water:

a) The technetium is oxidized by H_2O_2 in 0.1 N H_2SO_4

b) The technetium is oxidized by oxygen in air (from air pump)

c) The technetium-99m is reduced to $^{99m}Tc^{+4}$ with $K_2S_2O_5$.

Then the ⁹⁹mTc⁺⁴ is added to 200 litre of sea water. Stir with air pump for 1 h. Technetium could be oxidized by oxygen in air (from air pump). By the 3 methods, the same chemical yields of ⁹⁹mTc are obtained. It is shown that the Tc⁺⁴ could not be stable in solution without a reducing agent. The Tc⁺⁴ could easily be oxidized by a gentle oxidizing agent such as H_2O_2 and O_2 . So the technetium valence state in the sea water will be TcO_4^- , because the sea water contains oxygen.

Ruthenium decontamination

Technetium and ruthenium are oxidized to TcO_4^- and RuO_4^- by NaClO at pH = 9. Acidify the solution to pH = 1 with H₂SO₄. In this case, the RuO₄ is evaporated at 100°C in 0.1N H₂SO₄. But the TcO_4^- is left in solution, as shown in Tables 2 and 3. This step is very efficient and convenient for ruthenium decontamination instead of CCl₄ extraction, but the evaporation (and extraction of CCl₄) of the RuO₄ are interferred with by organic agents and iron. If the solution does not change to a bright yellow colour, it is necessary to add more NaClO. The solution may be evaporated to near dryness to remove organic agents, or Fe(OH)₃ may be precipitated in order to remove the iron.

Technetium and ruthenium are separated by extraction of TIOA/xylene at controlled valence with H_2O_2 . If the solution contains concentrated Na₂SO₄, the extraction rate of technetium is decreased. The different valence states of ruthenium are controlled with NaClO and H_2O_2 in different steps in the procedure to get higher ruthenium decontamination. The decontamination factors of ruthenium for each step in the procedure are shown as follows:

Decontamination factors of Ru

Anion exchange	5
Evaporation in 0.1 NH ₂ SO ₄	2 × 10 ⁷
5% TIOA/xylene extraction	250

Step

According to the step-by-step decontamination of the procedure, the decontamination factor for ruthenium should be 2.6×10^{10} . But the large volume of sea water is more complicated, as it contains many interfering elements. Furthermore, the low count of 103 Ru used for the decontamination test may present problems. So the decontamination of the procedure is only 2.5×10^{5} - 2.4×10^{7} for ruthenium, 1.66×10^{5} for 110m Ag. Technetium is lost at each step of the procedure as follows:

Step	Loss rate of 99m Tc %
Left on column	~ 5
Fe(OH) ³ scavenging	~ 10
and AgCl precipitate	
evaporation operation	~ 5
5% TIOA/xylene extraction	~ 5
Electrodeposition	~ 5

The chemical yield of the procedure is about 70%.

Conclusion

- 1. TcO₄ is intensely absorbed on anion exchange AG1-X₄ resin in 0.1 N-1 N H₂SO₄. TcO₄ is stripped with 10 N HNO₃. Use 2 hours for the stripping. The chemical yield of technetium is about 95%.
- 2. Three columns (11 \times 2.5 cm of AG 1-X₄ 100-200 mesh) anion exchange are efficiently used for collecting technetium in a 200-litre sea water sample.
- 3. Ruthenium and technetium are oxidized to RuO_4^- and TcO_4^- with NaClO at pH = 9. Acidify to 0.1 N H₂SO₄. Heat for 10 min at 100°C. 99.64% of RuO₄ is evaporated and the TcO_4^- is left in solution. Repeat the operation 3 times; the decontamination for ruthenium is 2×10^7 . It is more convenient and efficient compared with CCl₄ extraction.
- 4. Fe(OH)₃ scavenging, extraction of 5% TIOA/xylene are used for ruthenium decontamination and other nuclides. AgCl precipitate is used for ^{110m}Ag decontamination.
- 5. The procedure can be applied to different types of environmental samples. The average chemical yield for Tc is about 70%, and the decontamination factors are 2.5×10^{5} - 2.4×10^{7} for ruthenium and 1.66×10^{5} for 110m Ag. The reproducibility of the method is satisfactory (±10%).

References

- 1. S. Foti et al. Anal. Chem. Acta 60, 269 (1972).
- 2. Q.J. Chen et al. Risø-M-2671.
- 3. E. Anders, NAS-NS-3021.

Table 1. Chemical yield of ^{99m}Tc for anion exchange at different conditions of 200 litres sea water and ⁹⁹Tc Bq/m³ (Klint sea water March 1988 in Denmark)

Condition	99mTc valence state	No.	Sea water volume litre	Columns number 11 × 2.5 cm	Stripping volume 10 N HNO3 ml	Time for stripping min	99mTc counts before exchange c.p.m.	99mTc counts after exchange c.p.m.	Chemical yield %	99Tc Bq/m³	(Mean ± 1 SD %) ⁹⁹ Tc Bq/m ³
Filter 0.1 N H ₂ SO ₄ 0.5 ml H ₂ O ₂ per litre Pump air	TcO₄	1 2 3 4	200 200 400 200	3 3 4 3	450 600 800 600	90 120 120 120	28118 40594 25176 8640	25083 37875 20929 7736	89.2 93.3 83.3 89.5	- 0.4427 0.4862 0.5230	
Filter Pump air Filter Pump air	TcO4 Tc+4	1 2 1 2	200 200 200	3 3 3	600 600 600	120 120 120	30798 314755 48502	28946 303757 46348	94.0 96.5 95.6	0.4961 0.4116 0.4861 0.5432	0.4841 ± 0.0450 (±9.3%)
99mTc+4		2	200	5	000	120	40444	40022	93.0	0.3452	

No.	Evaporation time min	Before evaporation counts c.p.s.	After evaporation counts c.p.s.	Loss chemical yield of ¹⁰³ Ru %	Decontamination factor
1	5	276.84	3.63	98.59	76
2	5	267.29	3.26	98.78	82
1	10	373.71	1.33	99.64	281
2	10	266.35	0.99	99.6 3	269

Table 2. Determination of evaporation rate of RuO4 in 0.1 N H2SO4 at 100°C

Table 3. Determination of evaporation rate of 99m Tc in 0.1 N H2SO4 at 100°C

No.	Ruthenium carrier 5 mg/ml RuCl ₃	Evaporation time min	Before evaporation counts c.p.s.	After evaporation counts c.p.s.	Chemical yield %
1	0.1 ml	90	2450	2493	101.8
2	0.1 ml	90	2450	2568	104 8
1	0.4 ml	90	2450	2592	105.8
2	0.4ml	90	2450	2524	103.0
1	0.8 ml	90	2450	2524	103.0
2	0.8ml	90	2450	2494	101.8

Table 4. Chemical yield of technetium and decontamination factor of ¹⁰³Ru of the procedure

No.	Nuclid es	Before the procedure counts c.p.s.	After the procedure counts c.p.s.	^{99m} Tc chemical yield %	¹⁰³ Ru decontamination factor
1	99mTc	78.61	62.63	79.7	· · · · · · · · · · · · · · · · · · ·
2	99mTc	45.58	36.33	79.3	
3	⁹⁹ ™Tc	22.08	14.37	65.1	
1	¹⁰³ Ru	448.73	0.00180		2.5×10^{5}
2	¹⁰³ Ru	400.04	0.00125		3.2×10^{5}
3	¹⁰³ Ru	665.0	0.0000272		2.4×10^{7}

Appendix 2

Determination of ⁹⁹Tc in environmental samples by anion ion exchange (Collecting ⁹⁹Tc by Fe(OH)₂)



- 5. 1) Add 0.5 ml 5 mg/ml RuCl₃ and 1 ml 5% NaClO. Keep pH =
 9. Heat for 10 min to allow the isotope exchange to be completed and to get RuO₄. (The solution should be bright yellow. If it is not add more 5% NaClO). Add 1 ml 5% NaClO.
 - 2) Acidify the solution to pH = 1 with 10 N H₂SO₄.
 - 3) Heat for 15 min in order to evaporate the RuO₄. The bright yellow disappears and solution is colourless.

6.

 Transfer solution to another beaker. Keep the volume at 70 ml.

2) Repeat step 5.

7.

1) Transfer solution to another beaker. Keep the volume at 70 ml.

2) Repeat step 5.

8.

- 1) Acidify solution to 1 N H₂SO₄. Add 1 ml 30% H₂O₂ and dilute to 100 ml.
 - 2) Extract with 80 ml 5% TIOA (tri-isooctylamine)/xylene. Shake for 5 min.
 - 3) Wash with 80 ml ! N H₂SO₄ containing 0.5 ml 30% H₂O₂. Shake for 2 min.
 - 4) Wash with 80 ml H₂O. Shake 5 min.

- 1) Backextract with 40 ml 0.5 N NaOH. Shake for 2 min.
- 2) Backextract again with 20 ml 0.5 N NaOH. Shake for 2 min. Combine the two solutions.

10.

1) Evaporate the solution to 20 ml for electrodeposition.

- 2) The current is 300 mA. The time is at least 5 h or overnight (about 10-24 h).
- 3) Wash the plate with pH = 9 water once.
- 4) Count the chem.yield.

Appendix 3

Determination of ⁹⁹Tc in seaweed by TIOA extraction Flow sheet

- 1) Add to 10 g (10-20 g) dry powdered seaweed, 0.5 ml 5 mg/ml RuCl₃, 0.5 ml 20 mg/ml AgNO₃, ^{99m}Tc and 20 ml concentrated H₂SO₄ (36 N) in a flask (see figure 2). Stir until completely mixed. (Per 1 g seaweed/2 ml conc. H₂SO₄).
 - 2) Let stay for 1 h. The seaweed is carbonated by conc. H_2SO_4 .
 - 3) Add conc. HNO₃ (15 N) drop by drop to the flask. Be careful, the reaction is very strong. Continuously add conc. HNO₃ (per 1 g seaweed/6-8 ml 15 N HNO₃) until the reaction is calm.
 - 4) Heat the solution (in the flask). The reaction is continued until the mixture turns black.
 - 5) Add conc. HNO₃. The black colour disappears. Continuously heat the solution and the black colour returns.
 - 6) Repeat step 5) until the solution becomes greenish yellow.
- 2.
- 1) Transfer the solution to 300 ml beaker. Dilute to 200 ml (the acid concentration is 5-6 N).
 - 2) Add 10 g $K_2S_2O_8$ and heat for 10 min.
 - 3) Cool and filter the solution.
- 3.
- 1) Transfer the solution to 500 ml separator.
- 2) Extract with 80 ml 5% TIOA (tri-isooctylamine)/xylene. Shake for 2 min.
- 3) Wash with 80 ml 1 N H_2SO_4 containing 0.5 ml 30% H_2O_2 . Shake for 2 min.
- 4) Wash with 80 ml $H_2 =$. Shake 5 min.
- 5) Backextract with 40 ml 0.5 N NaOH. Shake for 2 min.
- 6) Backextract again with 20 ml 0.5 N NaOH. Shake for 2 min. Combine the two solutions.
- 4.
- 1) Evaporate the solution to 20 ml for electrodeposition.
- 2) The current is 300 mA. The time is at least 5 h or overnight (10-24 h). The chemical yield is 50-75%.



Figure 2



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Qing Jiang Chen, Asker Aarkrog, Henning Dahlgaard, Even P. Nielsen, H.L. Jensen, Jette Bruun, Anna Holm Pedersen, Karen Mandrup

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