

Technecium analysis Tc-99 - STUK

Technetium

Measurement	Liquid scintillation counting
Method used for matrices	Environmental Water
Separation Method	Extraction Chromatography
Radionuclide(s)	Tc-99
Quantity of sample used (in kg, l, ...)	0.01 - 0.02 kg
Counting time for the method	300 min
MDA of the technique	1.0 Bq/kg
FWHM (Energy MeV)	-
Method Evaluated	No
Method Accredited	No
Procedure	

Description of the method

Pretreatment and separation methods

The Tc separation method used in this study is based on the Eichroms TEVA extraction chromatography resin, the extractant being an aliphatic quaternary amine. The Tc is strongly retained under neutral conditions while other interfering isotopes are not. The seaweed samples were dried in 105°C and then homogenised. About 10 grams of dry weight seaweed was weighted in a vessel and 40 ml of 8 M HNO₃ was added. Samples were held overnight in icebath and in the next morning Tc-99m tracer was added. Samples were heated 15 minutes in 80°C, 1 h in 100°C and ½ h in 200°C with Tecator combustion apparatus to digest the sample. Then 2 ml of 30% H₂O₂ was added per sample and the samples were heated for ½ h in 90°C. After cooling the samples were filtrated with glassfibre filter (GF/A) to a 250 ml decanter glass. Samples were evaporated to near dryness with hot plate and the residue was dissolved with 25 ml of 0.1 M HNO₃. 3 ml of H₂O₂ was added and samples were heated on a hot plate for ½ h below 90°C. If white precipitate occurred it was filtered off and the sample was passed through a 2 ml Eichrom prefilter column for removing the remaining organic matter. The column was rinsed with 5 ml of 0.1 M HNO₃. In the pertechnetate form (Tc⁷⁺) technetium is highly soluble and stays in solution. When reduced to Tc⁴⁺, technetium can be coprecipitated with iron hydroxide. This can be used to reduce sample volume prior to final technetium separation with TEVA resin. The sea water samples were placed into an 80 liter plastic container and acidified to pH 4 with concentrated HCl and stirred thoroughly. Then 99mTc tracer was added and the sample was stirred for an hour. After that iron carrier was added (50 mg/litre of sample) in the form of FeCl₃ · 6 H₂O and the sample was stirred until all of ferric chloride was dissolved. K₂S₂O₅ was added (about 1 g/ 1 l of sample) to reduce Fe³⁺ to Fe²⁺ and Tc⁷⁺ to Tc⁴⁺ and the sample was stirred until the brown colour of the solution disappeared. 32% NaOH was added (pH 9) to precipitate iron hydroxide (and to coprecipitate Tc). During overnight the precipitate has settled down. The next day the supernatate was discarded and the precipitate was dissolved with concentrated HCl. About 3 ml of 30% H₂O₂ per litre of sample

was added to oxidize Tc again and the samples were heated for ½ h on a hot plate. Then the iron hydroxide was precipitated with NaOH (pH 9) and the samples were centrifuged. Tc remained in the solution and the precipitate was discarded. The solution with the 99Tc was again acidified to pH 4 with concentrated HCl and K₂S₂O₅ and Fe-carrier was added. Ferric hydroxide precipitation together with Tc was done again to reduce the sample volume. The samples were centrifuged and the solution was discarded. Precipitation was dissolved with concentrated HNO₃, pH was adjusted to 1-2, 5 ml of H₂O₂ was added and the samples were heated for ½ h. Then pH was adjusted to 7 with concentrated NH₃ and iron hydroxide was centrifuged off. The solution was evaporated on a hot plate until the sample volume was about 50 to 100 ml. After cooling the solution was passed through a Eichrom prefilter column and the column was rinsed with 5 ml of 0.1 M HNO₃. After prefilter columns both sea water and Fucus samples were passed through Eichrom's TEVA column and the column was rinsed with 5 ml of 0.1 M HNO₃ and 20 ml of 2 M HNO₃. Tc-99 was retained in TEVA resin and the interfering isotopes were removed. The column was cut near the bottom frit with a sharp knife and the resin was rinsed out to 20 ml scintillation vial with four aliquots of distilled water.

Measurement of Tc-99

The chemical yield was measured with gamma spectrometer. Four standard samples were made in the same way as the samples. The TEVA column was cut near the bottom and the resin was rinsed out to the 20 ml scintillation vial with now 3 ml of distilled water. Then 1 ml of 99mTc (same amount as added to the samples) was added to the vial. All standard samples were measured with gamma spectrometer and the average of the results was used to represent the initially added 99mTc amount to the samples. The ratio of measured 99mTc in the sample to the measured average of 99mTc in the standards represents the chemical yield of the analysis. After chemical yield measurement, 15 ml of Ultima Gold AB liquid scintillation cocktail was added to the vial and the samples were left to wait for about a week to ensure that all the 99mTc has completely decayed away. The samples were measured with a low background liquid scintillation counter, Quantulus. The window for 99Tc measurements was chosen to be channels from 100 to 550, where the background was typically about 2.0 to 2.2 cpm. One of the standard samples of the yield measurements was used as a background sample to ensure that the added 99mTc tracer was pure. The samples were counted usually for 600 minutes and the counting efficiency was typically 77 to 81% depending on the sample quenching.

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