# Procedure for determining thorium isotopes in wastewater by alpha spectrometry

 $H-\alpha$ -SPEKT-AWASS-05

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# 1 Scope

The procedure for determining the thorium isotope Th-232, its decay product Th-228, as well as Th-230 from the uranium-radium decay chain described here, is suitable for use with wastewaters from nuclear facilities, wastewaters from users of isotopes, and drains of water purification plants, pile seepage waters, and waters emanating from mines. The principles of the procedure may also be adapted – if needed after enrichment by means of evaporating larger volumes – to other waters, including, e. g., groundwater, drinking water and surface waters.

High levels of salinity, which may be present in seepage waters from piles and mines, for example, will hamper the processing of samples. Low chemical yields might have to be expected in these cases.

# 2 Sampling

Depending on the expected thorium activity concentrations, an adequately large volume of water is sampled. The water sample has to be representative of the sampled location (see also prodedure H- $\gamma$ -SPEKT-AWASS-01 for collecting water samples). A volume between 1 l and 10 l will usually be sufficient. The samples are acidified with a minimum of 1 ml of nitric acid (14 mol·l<sup>-1</sup>) per litre of water each and may then be stored in polyethylene bottle until they can be processed further.

# 3 Analysis

### **3.1 Principle of the procedure**

The principle of the chemical separation is explained in Figure 1. The sample is acidified and concentrated. The thorium isotopes are pre-concentrated by co-precipitating them with yttrium fluoride. The precipitated fluorides are then dissolved in nitric acid following their conversion into sulphates. The thorium isotopes are extracted from the solution with tri-octyl methyl ammonium nitrate (TOA)/xylene and re-extracted with diluted hydrochloric acid. This step is then followed up with a fine-purification process that separates the iron and uranium by means of an anion exchange from the hydrochlorated solution. If plutonium isotopes are to be expected, these have to be removed from a solution containing nitric acid by an anion exchange process. Liberated of interfering ions and nuclides thus, the thorium isotopes are extracted from the solution by electrodeposition. The activities of the isotopes Th-228, Th-230 and Th-232 are determined by alpha spectrometry. The chemical yield may be determined by adding a thorium isotope as a tracer. Being alpha emitters, Th-229 or Th-227 may be considered here. It needs to be taken into account, however, that the decay of Th-228 produces short-lived daughter nuclides whose alpha energies complicate determining Th-227; for its part, Th-229 has alpha energies that lie close to those of the Th-230, so that determining the latter may sometimes not be possible. In the case of elevated Th-230 activity concentrations, chemical yields of more than 100 % may then be feigned.

#### Sample Preparation

Acidification of the sample with HNO<sub>3</sub>, +MgCl<sub>2</sub>, +YCl<sub>3</sub> mixing with Th-234 tracer

Dry-ashing at 450 °C						
Smokina	off with +	· H <sub>2</sub> SO <sub>4</sub> /	HNO <sub>3</sub> ,	dilutina,	filterina	off

### Scavenger-precipitation of Th

with YF<sub>3</sub>, dissolving in  $H_2SO_4$ 

Extraction with

tri-octyl methyl ammonium nitrate (TOA) in xylene from HNO<sub>3</sub> solution (3 mol·l<sup>-1</sup>)

**Re-extraction** 

into an aqueous phase with warm HCl (0,5 mol·l<sup>-1</sup>)

#### Anion exchange

Removing of uranium as a hexa-chlorine complex from HCl solution (9 mol·l<sup>-1</sup>)

If necessary, removing the Pu as nitrate complex from  $HNO_3$  solution (7,2 mol·l<sup>-1</sup>)

#### Separation of short-lived alpha decay products of Th-228 by another TOA-extraction

### Cathodic electrochemical separation

on stainless steel discs

#### alpha spectrometric measurement

by means of a surface barrier detector

determining the chemical yield (e. g., by gamma spectrometry of Th-234)

Fig. 1: Principle of determining thorium isotopes in wastewater

Determining the yield therefore makes use of Th-234 as a tracer and is effected by means of gamma spectrometry. The lines at 63 keV and 1001 keV (Pa-234m) are well suited for analysing the gamma spectra.

Th-234 may be extracted from U-238 (natural or depleted uranium) by anion exchange (see section 7.1.3). If Th-234 is to be used as a tracer, a sufficiently high activity needs to be ensured (e. g., some kBq), in order to keep the Th-234 already contained in the sample from interfering.

# 3.2 Sample preparation

The water sample is filled into an adequately large beaker and spiked with Th-234 (of several kBq) as a tracer for determining the chemical yield, about 1 g of magnesium chloride, as well as 500 mg of yttrium chloride, and concentrated on a sand bath to a volume of 100 ml. For preparing the Th-234 tracer, see section 7.1.3.

#### Note

If the alpha emitter Th-229 is used instead of the isotope Th-234 as a tracer, the activity added should amount to about 0,5 Bq; see section 5 for details.

The solution is transferred quantitatively to a 250 ml-beaker and evaporated to dryness. The beaker containing the dried sample is heated in a muffle furnace to 450 °C for about 24 hours. Once it has cooled to room temperature, the residue is dissolved with 5 ml of sulphuric acid (18 mol·l<sup>-1</sup>) and 10 ml of nitric acid (14 mol·l<sup>-1</sup>) and fumed off to dryness on a sand bath.

### **3.3** Radiochemical separation

**3.3.1** 50 ml of hydrochloric acid  $(1 \text{ mol} \cdot l^{-1})$  is added to the residue and stirred at elevated temperatures for about 10 minutes. If necessary, it is then extracted via a membrane filter and washed with hydrochloric acid  $(1 \text{ mol} \cdot l^{-1})$ . The filtrate and the wash solution are combined and the precipitate is discarded. 5 g of ammonium fluoride is added to the clear solution and stirred at elevated temperatures for about an hour.

**3.3.2** The yttrium/thorium fluoride precipitate is centrifuged off and the supernatant solution discarded. It is then converted into sulphate by adding 5 ml of sulphuric acid (18 mol·l<sup>-1</sup>) and fuming it off to dryness on a sand bath twice.

**3.3.3** The residue is dissolved in ca. 100 ml of nitric acid (3 mol·l<sup>-1</sup>) under stirring at elevated temperatures. Insoluble constituents are filtered off using a membrane filter. The precipitate is discarded. The filtrate is left to cool and then shaken out twice with 50 ml of conditioned TOA-extraction solution (see section 7.1.2) for 5 minutes each. The two organic phases are combined and united once with nitric acid (3 mol·l<sup>-1</sup>). The nitrates of thorium, uranium and plutonium isotopes are now contained in the organic phase whereas all other ions are contained in the aqueous phase, which can be discarded.

**3.3.4** The organic phase now serves to re-extract the thorium isotopes in five steps, using 50 ml of hydrochloric acid  $(0,5 \text{ mol}\cdot\text{l}^{-1})$  with a temperature of about 60 °C each. The combined hydrochloric acid extracts are infused with 10 ml of nitric acid (14 mol·l<sup>-1</sup>) and 5 ml of sulphuric acid (18 mol·l<sup>-1</sup>) in order to remove all organic remnants. The solution is then evaporated to dryness. If the residue fails to be colourless, this step needs to be repeated.

**3.3.5** The residue is dissolved in ca. 50 ml of hydrochloric acid (9 mol·l<sup>-1</sup>) in a warm environment for an hour, left to cool to room temperature, and then applied to a pre-conditioned anion exchange column (see section 7.1.1) in order to remove the uranium isotopes contained. Iron and uranium will form chlorine complexes and attach themselves to the exchanger while the thorium isotopes are contained in the run-off that is collected. 50 ml of hydrochloric acid (9 mol·l<sup>-1</sup>) are used for washing. The wash solution is combined with the run-off.

**3.3.6** The solution is evaporated to dryness and wet-ashed twice with a little nitric acid (14 mol·l<sup>-1</sup>) and sulphuric acid (18 mol·l<sup>-1</sup>) in order to destroy organic remnants. It is then processed further according to steps 3.3.7 or 3.3.8.

#### Note

If the sample has a very high uranium concentration relative to thorium, it will be expedient to repeat the anion exchange process according to step 3.3.5.

**3.3.7** If the presence of Pu-isotopes must be expected, these are to be separated from a nitric acid (7 mol·l<sup>-1</sup>) solution by anion exchange, as described in H- $\alpha$ -SPEKT-AWASS-03, step 3.3.5. The run-off containing the Th-isotopes is collected with the nitric acid wash solution and evaporated to dryness.

**3.3.8** If elevated contents of interfering impurities are to be expected, another purification step is recommended. To this end, the residue is dissolved in about 50 ml of warm nitric acid  $(3 \text{ mol} \cdot l^{-1})$  and extracted with 20 ml of TOA in a 100 ml-separation funnel, as described in step 3.3.3, and re-extracted five times from the combined organic phases with 20 ml of hydrochloric acid (0,5 mol·l<sup>-1</sup>) each. The combined hydrochloric acid solutions are then evaporated to dryness and wet-ashed with sulphuric acid (18 mol·l<sup>-1</sup>) and nitric acid (14 mol·l<sup>-1</sup>).

**3.3.9** For the purpose of electrodeposition of the thorium isotopes, the residue is dissolved in sulphuric acid (1,5 mol·l<sup>-1</sup>) in the heat and transferred quantitatively with sulphuric acid (0,5 mol·l<sup>-1</sup>) to the electrolytic cell (Figure 2 in H- $\alpha$ -SPEKT-AWASS-01). Using sulphuric acid (1,5 mol·l<sup>-1</sup>), it is adjusted to a pH of 2,4 to 2,5 (verified with Acilit<sup>®</sup> paper).

**3.3.10** Subsequently, the thorium is electrochemically deposited on the cathode at 300 mA for four hours. One minute before the electrolytic process is completed, 1 ml of ammonia solution  $(13 \text{ mol} \cdot l^{-1})$  is added. The cell is dismounted; the steel disc is rinsed with distilled water and then dried by rinsing it with acetone; the cell, seal and bottom plate are rinsed with distilled water, and the upper part of the cell and a fresh steel disc (for the next analysis) are stored in RBS solution.

#### Note

In order to prevent a renewed increase in the alpha-activity of short-lived decay products, the electrodeposition process should be started immediately after the fine-tuned purification step (step 3.3.8), and the alpha spectrometric measurement should commence right after the electrodeposition process in order not to unnecessarily complicate the analysis of the alpha spectrum.

# 4 Measuring the activity

The alpha spectrum of the electrochemically separated thorium is measured with the aid of a surface barrier detector operating in a vacuum (ca.  $10^3$  Pa residual pressure) that is linked to a multi-channel analyser and a corresponding data output unit. Further details on the geometry of the alpha measuring station and calibration are provided in chapter IV.2 of this procedures manual as well as in the procedures H- $\alpha$ -SPEKT-AWASS-01 and H- $\alpha$ -SPEKT-AWASS-03. The alpha energies and emission probabilities of the thorium isotopes encountered here are compiled in Table 1. Because the resolution of semiconductor detectors is normally insufficient to detect twin and multiple lines separately for analysis, the weighted average energies of these lines and the sum of their emission probabilities in the analysed energy ranges are provided.

Nuclide	Alpha energy (MeV) weighted average of twin and multiple lines, respectively	Emission probability $p_{\alpha,r}$ (%)
Th-227	5,93 (multiple line)	95
Th-228	5,40 (twin line)	99
Th-229	4,85 (multiple line)	86
Th-230	4,67 (twin line)	100
Th-232	4,17 (twin line)	100

Tab. 1: Alpha energies and emission probabilities of thorium isotopes

#### Note

Imperfect electrolytic deposition of the thorium on the steel disc or impurities in the counting sources, in particular incomplete separation of other alpha emitters, such as uranium, may cause a substantial broadening of the lines during the alpha spectrometric measurement. In such case, it is expedient to separate the thorium from the steel disc with hydrochloric acid (8 mol·l<sup>-1</sup>) (which only takes a few seconds), to evaporate the solution to dryness, and to repeat the preparation process from step 3.3.8.

Following the alpha spectrometric measurement, the disc is measured by gamma spectrometry to determine the yield (cf. section 3.2). In a separate step, Th-234 is removed from the tracer solution that was added to the sample at the start of its chemical processing, and is measured by gamma spectrometry without chemical separation under the same geometry (e. g., after evaporating it in a beaker of adequate size).

# 5 Calculation of the results

The activity concentrations,  $c_r$ , of the individual thorium isotopes are calculated according to equation (1):

$$c_{\rm r} = \frac{\varphi_{\rm A}}{V \cdot \eta \cdot p_{\alpha,\rm r}} \cdot \left(R_{\rm g} - R_{\rm 0}\right) \cdot f_{\rm 1} \tag{1}$$

where

 $c_r$  activity concentration of the nuclide r, in Bq·l<sup>-1</sup>;

 $R_0$  background count rate in the region of the line at  $E_{\alpha}$ , in s<sup>-1</sup>;

 $R_{g}$  gross count rate in the region of the line at  $E_{\alpha}$ , in s<sup>-1</sup>;

*V* sample volume, in I;

 $\varphi_A$  calibration factor, in Bq·s;

 $p_{\alpha r}$  emission probability of alpha radiation of the nuclide r;

 $\eta$  chemical yield of the procedure.

The factor  $f_1$  takes into consideration the decay correction, which will be of relevance only for Th-228, however. Equation (2) applies:

$$f_{1} = \exp\left(\ln 2 \cdot \frac{t_{A1}}{t_{r1}}\right)$$
(2)

where:

*t*<sub>A1</sub> period of time between sampling and measuring, in s;

 $t_{r1}$  half-life of Th-228, in s.

To determine the chemical yield,  $\eta,$  either Th-229 or Th-234 may be used as tracer:

 If Th-234 is used as a tracer, equation (3) applies if sample and standard are measured in an approximately equal geometry, if possible on the same day, with the same gamma spectrometric workstation:

$$\eta = \frac{R_{P, Th-234} - R_{r, \gamma}}{R_{S, Th-234} - R_{0, \gamma}}$$
(3)

where:

 $R_{P,Th-234}$  gross count rate of the counting source, in s<sup>-1</sup>;

 $R_{S,Th-234}$  gross count rate of the standard source, in s<sup>-1</sup>;

 $R_{0,\gamma}$  background count rate, in s<sup>-1</sup>.

Possible Th-230 impurities in the Th-234 tracer may need to be taken into consideration.

- If Th-229 is used as a tracer, equation (4) applies:

$$\eta = \frac{\varphi_{A} \cdot R_{\text{Th}-229}}{A_{\text{Th}-229} \cdot p_{\alpha,r}}$$
(4)

where:

 $R_{Th-229}$ net count rate in the region of the Th-229 line, in s<sup>-1</sup>; $A_{Th-229}$ activity of Th-229, in Bq; $p_{ar}$ emission probability of Th-229.

The alpha energy of Th-230 differs so little to the corresponding alpha energy of Th-229 that these two nuclides cannot usually be distinguished. Determining Th-230 may therefore not be possible if Th-229 has been used as a tracer. Higher Th-230 activities in the sample may seemingly show yields in excess of 100 %.

# 5.1 Worked example

Using Th-234 as a tracer for determining the yield of the nuclide Th-228 will produce the following result if the following numerical values are inserted:

V	= 2 l;				
$arphi_{A}$	= 5,46 Bq∙s;				
t <sub>r1</sub>	= 6,038·10 <sup>7</sup> s (699 d) for Th-228;				
t <sub>A1</sub>	= 7,080·10 <sup>6</sup> s	(82 d);			
<b>R</b> <sub>0,γ</sub>	= 1,32·10 <sup>-3</sup> s <sup>-1</sup>				
<b>R</b> <sub>P,Th-234</sub>	= 0,243 s <sup>-1</sup> ;				
$R_{ m S,Th-234}$	= 0,342 s <sup>-1</sup> ;				
R <sub>0</sub>	= 3,8·10 <sup>-5</sup> s <sup>-1</sup>	in the region of the Th-228 line;			
Rg	= 3,9·10 <sup>-3</sup> s <sup>-1</sup>	in the region of the Th-228 line;			
$p_{lpha}$	= 0,99	for Th-228.			

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according to equation (3) for the chemical yield,  $\eta$ :

$$\eta = 0,709 (70,9 \%)$$

according to equation (2) for the decay correction of Th-228:

$$f_1 = 1,085$$

according to equation (1) for the activity concentration, c, of Th-228 at the point of time of sampling:

$$c_{\text{Th-228}} = 1,63 \cdot 10^{-2} \text{ Bq} \cdot \text{I}^{-1}.$$

Besides the lines of the thorium isotopes, additional lines stemming from the shortlived decay products are illustrated in the alpha spectrum of a thorium sample (Figure 2).



Fig. 2: Alpha spectrum of a thorium sample

#### 5.2 Consideration of uncertainties

To estimate the total uncertainty of the activity concentrations,  $c_r$ , statistical counting uncertainty, the uncertainty in determining the detection efficiency (calibration uncertainties), and the uncertainty incurred from determining the chemical yield needs to be taken into account, whereas the uncertainty associated with determining the sample volume may be neglected. The relative uncertainty in the detection efficiency is around 5 %, while the relative uncertainty in the chemical yield fluctuates between 15 % and 20 %, depending on the yield.

Systematic uncertainties, e. g., overlapping lines as a result of other alpha emitters (such as uranium isotopes) having been incompletely separated may be substantially larger. If Th-227 is used as a tracer for determining the yield, the decay pro-

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ducts of Th-228 will cause systematic errors. If Th-229 is used, Th-230 that is present in the sample may render determining the yield impossible. The presence of Po-210 may complicate determining the Th-228 content.

Applying equation (2.7) given in chapter IV.5 of this procedures manual for the standard deviation,  $s(R_n)$ , of the net count rate, the relative uncertainty of the concentration can be calculated according to the following equation:

$$\frac{s(c)}{c} = \sqrt{\frac{\frac{R_g}{t_m} + \frac{R_0}{t_0}}{\left(R_g - R_0\right)^2} + \left(\frac{s(\varphi)}{\varphi}\right)^2 + \left(\frac{s(\eta)}{\eta}\right)^2}$$
(5)

Using the values for  $R_g$  and  $R_0$  given in section 5.1, a measuring period of the background effect of  $t_0 = 86400$  s and a measuring period for the sample,  $t_m = 86400$  s, as well as

$$\frac{s(\varphi)}{\varphi} = 0,05$$
 and  $\frac{s(\eta)}{\eta} = 0,15$ 

the relative uncertainty of the Th-228 concentration yields:

$$\frac{s(c)}{c} = \sqrt{0,003 + 0,05^{2} + 0,15^{2}} = 0,16$$
(6)

The result of measuring the activity therefore reads:

$$C_{\text{Th-228}} = (0,016 \pm 0,003) \text{ Bq} \cdot \text{I}^{-1}$$

# 6 Characteristic limits of the procedure

To estimate the characteristic limits, reference is made to chapter IV.5, sections 2.1, 2.3 and 4.8 of this procedures manual. The approach for determining the detection limit is based here on regarding the analysis of an alpha line with a base width, b, as an integral measurement that is taken with a single-channel analyser.

The magnitude of achievable detection limits is estimated in the following example: Applying long-term measuring, the following mean background count rates were determined in the places of the lines of Th-228, Th-230 and Th-232 under the given measuring configuration:

Isotope	$E_{\alpha}$ (MeV)	Base width	$R_0$ (s <sup>-1</sup> )	$R_0$ (s <sup>-1</sup> )
		(channels)	within range <i>b</i>	per channel
Th-227	5,93	42	4,4·10 <sup>-5</sup>	1,0·10 <sup>-6</sup>
Th-228	5,40	25	3,8·10⁻⁵	1,5·10 <sup>-6</sup>
Th-229	4,85	30	1,1·10 <sup>-5</sup>	3,6·10 <sup>-7</sup>
Th-230	4,67	20	1,4·10 <sup>-5</sup>	7,1·10 <sup>-7</sup>
Th-232	4,17	20	1,8·10 <sup>-5</sup>	8,8·10 <sup>-7</sup>

According to equation (2.4) in section 2.1.2 of chapter IV.5 and applying measuring periods of  $t_m$  (sample) and  $t_0$  (background effect) of 86400 s (24 h), a value for k of 4,645 ( $k_{1-\alpha} = 3$ ,  $k_{1-\beta} = 1,645$ ), und a calibration factor of  $\varphi_A = 5,46$  Bq·s, the detection limits for the activity for Th-228 amount to 1,5 mBq, and 1,2 mBq for Th-230 and Th-232.

The detection limits,  $g_r$ , for activity concentrations are obtained according to equation (7):

$$g = \frac{G}{V \cdot \eta \cdot p_{\alpha,r}}$$
(7)

Using an original sample volume of  $V = 2 \,\text{I}$  and obtaining a chemical yield,  $\eta$ , of 71 %, the detection limits for activity concentrations,  $g_r$ , for Th-228 as well as Th-230 and Th-232 amount to 0,94 mBq·l<sup>-1</sup> and 0,75 mBq·l<sup>-1</sup>, respectively, that is about 1 mBq·l<sup>-1</sup>.

# 7 Catalogue of chemicals and equipment

### 7.1 Chemicals

All chemicals used should be of the purity grade "pro analysi":

- Ammonia solution, NH<sub>3</sub>: 13 mol·l<sup>-1</sup>;
- Ammonium fluoride, NH<sub>4</sub>F;
- Anion exchanger: strongly alkaline, 100 mesh to 200 mesh, Cl<sup>-</sup>-form;
- Hydroxyl ammonium sulphate, (NH<sub>3</sub>OH)<sub>2</sub>SO<sub>4</sub>;
- Magnesium chloride, MgCl<sub>2</sub>;
- RBS-50 solution: ca. 10 %;
- Nitric acid, HNO<sub>3</sub>: 0,5 mol·l<sup>-1</sup>, 3 mol·l<sup>-1</sup> and 14 mol·l<sup>-1</sup>;
- Hydrochloric acid, HCI: 0,1 mol·l<sup>-1</sup>, 1 mol·l<sup>-1</sup>, 3 mol·l<sup>-1</sup> and 9 mol·l<sup>-1</sup>;
- Sulphuric acid, H<sub>2</sub>SO<sub>4</sub>: 0,5 mol·l<sup>-1</sup> and 18 mol·l<sup>-1</sup>;
- Th-234 tracer solution: about 0,2 MBq·l<sup>-1</sup>, see section 7.1.3 if necessary, Th-227 or Th-229 tracer solution, about 300 Bq·l<sup>-1</sup>;
- Trioctylmethylammonium chloride, [(C<sub>8</sub>H<sub>18</sub>)<sub>3</sub> · CNH<sub>3</sub>]Cl (e. g., from Serva, Heidelberg);
- Xylene, C<sub>6</sub>H<sub>4</sub>(CH<sub>3</sub>)<sub>2</sub> (mixed isomers);
- Yttrium chloride,  $YCl_3 \cdot 6H_2O$  (thorium-free).

### 7.1.1 Conditioning of the anion exchanger

For conditioning the anion exchanger, reference is made to the procedures H- $\alpha$ -SPEKT-AWASS-01 and H- $\alpha$ -SPEKT-AWASS-02.

### 7.1.2 Conditioning of the extraction solution

10 % tri-octyl methyl ammonium chloride are dissolved in 90 % xylene. The solution is shaken out twice with nitric acid (3 mol·l<sup>-1</sup>). This produces the tri-octyl methyl ammonium nitrate required for the separation process.

### 7.1.3 Preparation of the Th-234 tracer

About 10 g<sup>1</sup> of uranyl chloride, UO<sub>2</sub>Cl<sub>2</sub>·6H<sub>2</sub>O (matured at least for one month after the last separation; the equilibrium with the daughter nuclide Th-234 is reached after about 240 days) are dissolved in about 100 ml of hydrochloric acid (9 mol·l<sup>-1</sup>). The solution is applied to a column (diameter = 30 mm, length = 200 mm) that has been filled with about 150 g of anion exchanger resin and conditioned with about 200 ml of hydrochloric acid (9 mol·l<sup>-1</sup>). The acid used for conditioning is discarded. The run-off of the applied uranium solution is collected. The anion exchanger is purified twice with 190 ml of hydrochloric acid (9 mol·l<sup>-1</sup>) each. The wash solutions are combined with the run-off and concentrated to dryness<sup>2</sup>.

The residue is wet-ashed with 2 ml of sulphuric acid (18 mol·l<sup>-1</sup>) and 5 ml of nitric acid (14 mol·l<sup>-1</sup>). The residue is dissolved in 20 ml of hydrochloric acid (9 mol·l<sup>-1</sup>) in a warm environment and applied to a conditioned anion exchanger column (section 7.1.1). The run-off is collected and the column is washed with 100 ml of hydrochloric acid (9 mol·l<sup>-1</sup>). The wash solution and the run-off are combined and filled up to the mark with distilled water in a 250 ml-volumetric flask.

Once the alpha spectrometric measurement has been completed, the steel discs of the spiked samples are measured by gamma spectrometry in the same geometry as the standard, either right before or after measuring the standard.

# 7.2 Equipment

- Stainless steel discs (V2A steel, austenitic,  $\emptyset = 25$  mm, thickness 0,3 mm);
- Electrolytic cell: see Fig. 2 in H-a-SPEKT-AWASS-01;
- Direct-current generator, if possible with current stabilizer;
- Ion-exchanger columns: length 20 cm,  $\emptyset = 1,5$  cm and 3 cm, frit G0;
- Measuring station for alpha spectrometry, consisting of: surface barrier detector (e. g., with 400 mm<sup>2</sup> active surface, 100 µm depth of the depletion zone, 25 keV resolution), vacuum measuring chamber, vacuum pump, power supply, amplifier, analogue-digital converter (ADC), multi-channel pulse peak analyser, data recording device/PC;
- Measuring station for gamma spectrometry;
- Muffle furnace with a large sample chamber;
- Basic equipment of a radiochemical laboratory.

#### 7.2.1 Preparing the electrolytic cell

The electrolytic cell and stainless steel disc are stored in ca. 10 % RBS-50 solution, warmed for about 10 minutes prior to the startof the electrolysis, and then thoroughly rinsed with distilled water. The stainless steel disc is rinsed with distilled water after having been treated with RBS-50.

<sup>&</sup>lt;sup>1</sup>) 10 g UO<sub>2</sub>Cl<sub>2</sub> · 6H<sub>2</sub>O (449,03 g·mol·l<sup>-1</sup>) correspond to 65452 Bq Th-234 (in equilibrium). Almost all commercially available – even «pure» – uranium products contain polluting traces of Th-230. Repeating the anion exchange process will render the uranium increasingly pure. If uranyl chloride of unknown origin is used, pollutions by, e. g., Th-230 have to be tested for by means of alpha spectrometry.

<sup>&</sup>lt;sup>2</sup>) The uranium from the ion exchanger is eluted with at least 200 ml of hydrochloric acid (1,2 mol·l<sup>-1</sup>). The elute is evaporated and may then be reused once more for manufacturing Th-234 tracer following an adequate period of time for the ingrowth of Th-234 ( $t_{\text{Th-234}} = 24,1$  days). The anion exchanger may be reused twice for the same purpose.

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