Procedure for determining the activity concentration of strontium-90 in seawater by proportional counting

D-Sr-90-MWASS-01

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1 Scope

The procedure outlined in the following is suitable for determining the activity concentration of strontium-90 (Sr-90) by its daughter nuclide yttrium-90 (Y-90) in seawater according to the IMIS-routine and IMIS-intensive programme [1]. This procedure is validated for sample volumes of 20 I to 90 I [2].

The procedure is only applicable if Sr-90 is in radioactive equilibrium with Y-90.

2 Sampling

A detailed description of sampling is given in procedure D-Cs-MWASS-01. The usual sample volume is 70 l, but should be doubled if low activity concentrations of Sr-90 are to be expected.

The on-board sample filling is carried out into drums already containing either 1 ml nitric acid (approx. 6 mol·l⁻¹) per litre seawater or 2 ml of hydrochloric acid (approx. 6 mol·l⁻¹) per litre seawater.

3 Analysis

3.1 Principle of the procedure

From the sample volume, an aliquot of usually 45 l of acidified seawater is used for the determining of the activity concentration of Sr-90.

From this volume, Y-90 is extracted by diisooctyl hydrogen phosphate (DEHP), transferred into a solution of low salt content after various purification steps and finally precipitated as hydroxide. After reprecipitation as oxalate and incinerating of the oxalate residue, Y-90 is measured as yttrium oxide (Y_2O_3).

The Y-90-counting source is measured multiple times consecutively by a low-level proportional counter and the activity concentration of Sr-90 is calculated from the obtained count rates.

The seawater sample may contain radionuclides which distort the determining of the activity concentration of Sr-90. These so-called interfering radionuclides are thorium isotopes as well as trivalent radionuclides. Therefore, appropriate steps should be taken to minimise the influence of these interfering radionuclides on the measurement result.

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Thorium isotopes are separated radiochemically, whereas trivalent radionuclides are taken into account only at the calculation.

Note:

In particular, these interferences have to be taken into account when determining low activity concentrations of Sr-90 in seawater, e. g. in samples of the North Sea and the Atlantic Ocean, respectively. Currently, the contribution of interfering radionuclides are negligible while determining the activity concentration of Sr-90 in samples of the Baltic Sea because of the higher activity concentrations of Sr-90 measured there.

3.2 Sample preparation

From this acidified seawater sample, a volume of 45 l is then decanted into a 50-l-plastic container with volume scale and the volume read off is noted.

3.3 Radiochemical separation

3.3.1 The container together with either the seawater sample prepared after Section 3.2 or the collected eluate after Section 3.3 of the Procedure D-Cs-MWASS-01 is placed underneath a stirrer motor with a propeller stirrer.

3.3.2 While stirring, a pH-value of 1,3 is set with concentrated hydrochloric acid (12 mol·l⁻¹).

3.3.3 Afterwards, a volume of 10 ml of yttrium carrier solution (50 mg Y³⁺) is added to the sample under continuous mixing.

3.3.4 A volume of 250 ml of DEHP solution (0,45 mol·l⁻¹) is added. **This time is noted** as time of the yttrium separation.

3.3.5 The sample is mixed thoroughly for five minutes. Then, the stirrer is removed from the container.

3.3.6 The sample is left for at least 20 minutes, but not more than 30 minutes, for phase separation, whereby the yttrium containing organic phase forms the upper one.

3.3.7 The lower aqueous phase is siphoned to as little volume as possible (from experience between 100 ml to 300 ml) by means of either a hose or a tube and is discarded.

3.3.8 The remaining solution is transferred into a 500 ml-separating funnel.

3.3.9 The plastic container is rinsed successively in each case with approx. 100 ml of distilled water, n-heptane and distilled water again, whereby the rinse solutions are combined with the solution in the separating funnel.

3.3.10 The lower aqueous phase is drained out of the separating funnel and discarded.

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Note:

If required, the aqueous solutions from this step and step 3.3.7 can be combined to be stored as a retention sample. After about 14 days, Sr-90 and Y-90 are in radioactive equilibrium again so that Y-90 can be separated and determined once more.

3.3.11 For cleaning, the organic phase is shaked twice with 100 ml portions each time and once with a 50 ml portion of hydrochloric acid (1 mol·l⁻¹) for one minute, respectively. The hydrochloric phases (lower phases) are drained and discarded in each case after phase separation.

Note:

In case of highly soiled samples, the number of extractions for purification of the organic phase can be increased. However, the total volume of 250 ml should be maintained.

3.3.12 The yttrium remained in the organic phase is re-extracted by shaking twice with 100 ml portions each time and once with a 50 ml portion of hydrochloric acid (9 mol·l⁻¹) for one minute, respectively. After phase separation, the hydrochloric phases (lower phases) are drained and combined in another 500 ml separating funnel already containing a portion of 250 ml of tricaprylylmethylammonium chloride solution.

3.3.13 Afterwards, the solution is shaken for two minutes.

3.3.14 The hydrochloric solution (lower phase) containing the yttrium is drained in a 1-l-beaker after phase separation and diluted to 500 ml with distilled water.

3.3.15 A volume of 350 ml of concentrated ammonia solution (approx. 13,3 mol·l⁻¹) is added slowly to the hydrochloric solution. The solution is then heated on a heating plate with vigorous stirring bringing it closely to the boiling point, whereby yttrium precipitates as yttrium hydroxide.

3.3.16 While cooling down to room temperature, the precipitate of yttrium hydroxide settles down.

3.3.17 The main part of the clear supernatant is carefully removed by vacuum, e.g. by a water jet pump.

Note:

If a radiochemical thorium separation is needed, the steps 3.3.29 to 3.3.35 replace the steps 3.3.18 to 3.3.21.

3.3.18 The yttrium hydroxide is removed by a suction filter (porosity 3) and washed with a little of weak ammoniacal water.

3.3.19 The suction filter together with the precipitate is put on a witt jar already containing a 400-ml-beaker.

3.3.20 The precipitate is completely dissolved from the frit with as little volume of nitric acid (2 mol·l⁻¹) as possible (about 5 ml to 10 ml) and collected in the beaker in the witt jar, where it precipitates again.

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Note:

Usually, the precipitate precipitates again immediately after passing through the frit. Residues of the precipitate are rinsed into the beaker with as little distilled water as possible. If the precipitate does not dissolve, acidified water with a few drops of nitric acid (2 mol·l⁻¹) is used for rinsing. During rinsing, it must be ensured in any case that the volume of the solution in the beaker remains so small that step 3.3.21 can be carried out.

3.3.21 Then, nitric acid (2 mol·l⁻¹) is added dropwise to the suspension until the precipitate has just completely dissolved. A total volume of approx. 150 ml is set in the beaker with distilled water.

3.3.22 The solution is heated on a heating plate bringing it closely to the boiling point and 10 ml of cold saturated oxalic acid solution is added. The yttrium precipitates as yttrium oxalate.

3.3.23 After cooling down to room temperature, a main part of the supernatant is removed by vacuum, e. g. by a water jet pump, and the yttrium oxalate is filtered with a vacuum filtration device of glass by means of a blue ribbon filter.

3.3.24 The precipitate is washed in each case with 5 ml of distilled water and ethanol, respectively.

3.3.25 The filter together with the precipitate is put in a porcelain crucible and is annealed in a muffle furnace at about (900 \pm 100) °C for five minutes

Note:

Yttrium oxalate usually contains different amounts of crystal water. Transferring it into yttrium oxide avoids mistakes in determining the yield gravimetrically.

3.3.26 The obtained yttrium oxide is transferred as quantitatively as possible with 0,1 ml to 0,2 ml of distilled water on a pre-weighed counting tray and a homogenous, flat counting source is produced by rubbing with a metal spatula.

Note:

Usually, 2"-counting trays are used for the preparation of both, calibration and counting sources.

3.3.27 The suspension in the counting tray is dried totally under a surface evaporator.

3.3.28 The counting source is weighed after cooling to determine the chemical yield.

Separation of thorium

3.3.29 The yttrium hydroxide is transferred quantitatively into a 200-ml-centrifuge tube by rinsing three times with approx. 5 ml of weak ammoniacal water in each case.

3.3.30 The sample is centrifuged approx. at the 1910-fold of the gravity acceleration (1910 *g*) for at least three minutes, until the precipitate has completely settled.

Note:

If the centrifuge only shows revolutions per minute, the operating manual of the centrifuge or the centrifuge rotor has to be read.

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3.3.31 The clear supernatant is decanted and discarded. The precipitate is just completely dissolved in approx. 3 ml of nitric acid (5 mol·l⁻¹).

3.3.32 The solution is diluted with distilled water until a pH determination is possible, and then its pH is adjusted to 1,3 to 1,5 with nitric acid (5 mol·l⁻¹).

3.3.33 For co-precipitation, 1 ml of zirconium carrier solution (50 mg Zr⁴⁺) and 1 ml of cold saturated sodium hydrogen phosphate solution are added in excess.

3.3.34 The solution is then centrifuged approx. at the 1910-fold of the gravity acceleration (1910 *g*) for three minutes for a total separation of the solid and liquid phase.

3.3.35 The clear supernatant is then decanted into a 400-ml-beaker and filled up to 150 ml with distilled water.

4 Measuring the activity

4.1 General

The Y-90 activity is measured with a low-level proportional counter.

4.2 Calibration

An yttrium oxide calibration source of known Y-90 activity is used for determining the detection efficiency, produced by using an aqueous solution of Sr-90 of a traceable standard in radioactive equilibria with Y-90.

The production is made by adding a Sr-90 solution of known activity, 10 ml of yttrium carrier solution (50 mg Y^{3+}) and 250 ml of DEHP solution (0,45 mol·l⁻¹) into a 500-ml-separating funnel, followed by a five-minute extraction. The further production follows the radiochemical separation from step 3.3.10 on.

The calibration source is measured in every measuring slot of the proportional counter at least three times for 50 minutes in each case. The detection efficiency is determined specifically for each measuring slot from the mean value of the obtained net count rates.

Note:

Usually, 2"-counting trays are used for the preparation of both, calibration and counting sources.

4.3 Measurement

The counting source obtained from Section 3.3 is measured by eight directly sequential measurements for 360 minutes in each case in a proportional counter.

For reviewing the radiochemical purity of Y-90, a decay curve for Y-90 fitted to the measured values of the net count rates $R_{n,i}$, which is generated by the UncertRadio software, is used.

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If the reduced chi-square value χ^2_R is between 0,5 and 2, it is assumed that counting source is undisturbed, whereby long-lived interfering radionuclides cannot be excluded. If this is the case, the counting source is measured again after 14 days and the hereby determined net count rates are subtracted from the net count rates of the first measurement series.

If χ^2_R deviates significantly from the above values, the measurement result is discarded or the analysis is repeated with a retention sample (see note of step 3.3.10).

4.4 Blank values

Blank value counting sources are prepared and measured according to Section 4.2 on a regular basis, though without addition of a traceable standard. They have to be considered in the calculation of the results if they differ significantly from the background.

Further remarks can be taken from the General Chapter RAD-CHEM/GRUNDL of this Procedures Manual.

5 Calculation of the results

The activity concentration of Sr-90 is calculated using usually eight values to then calculate the mean value at the time of yttrium separation (see step 3.3.4) decay-corrected net count rates $R_{n,i}$ (i = 1, ..., 8) of Y-90.

To reduce the decision threshold and the detection limit, the single measurements can be summed up. For determining the gross count rate of the summed up measurements, see Procedure D-H-3-MWASS-01.

5.1 Equations

5.1.1 Output quantity

The activity concentration c at the time of sampling is calculated according to Equation (1):

$$c = \frac{R_{n,Y-90}^0 \cdot f_A}{\varepsilon_{Y-90} \cdot \eta_Y \cdot V} = \frac{R_{n,Y-90}^0 \cdot e^{\lambda_{Sr-90} \cdot t_A}}{\varepsilon_{Y-90} \cdot \eta_Y \cdot V} = R_{n,Y-90}^0 \cdot \varphi$$
(1)

The Y-90 net count rate $R_{n,Y-90}^{0}$ related to the time of Sr/Y separation is determined by the linear deconvolution method from fitting a decay curve to the measurement points $R_{n,i}$ of the Y-90 net count rate. The decay curve, viz. the sequence of net count rates $R_{n,i}$ as a function of time differences t_i , is described by Equation (2), where $R_{n,Y-90}^{0}$ is the parameter to be fitted:

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$$R_{n,i} = R_{g,i} - R_0 \cong R_{n,Y-90}^0 \cdot e^{-\lambda_{Y-90} \cdot t_i} \cdot \frac{1 - e^{-\lambda_{Y-90} \cdot t_m}}{\lambda_{Y-90} \cdot t_m} = R_{n,Y-90}^0 \cdot f_{Y-90,i}$$
(2)

Analogous to Table 1 and Equations (40) and (41) in [3], Equation (2) can be given in matrix notation:

$$\begin{pmatrix} R_{n,1} \\ R_{n,2} \\ \vdots \\ R_{n,8} \end{pmatrix} \cong \begin{pmatrix} f_{Y-90,1} \\ f_{Y-90,2} \\ \vdots \\ f_{Y-90,8} \end{pmatrix} \cdot R_{n,Y-90}^{0}$$
(3)

In the Equations (1) to (3) are:

c activity concentration of Sr-90, in Bq·m⁻³;

 $f_{\rm A}$ correction factor from the time of sampling to Sr/Y separation;

 $f_{Y-90,i}$ decay correction factor for Y-90 from the time of Sr/Y separation to the start of the *i*th measurement;

$$R_{g,i}$$
 gross count rate of the single measurement *i*, in s⁻¹;

$$R_{n,i}$$
 net count rate of the single measurement *i*, in s⁻¹;

 R_0 background count rate, in s⁻¹;

 $R_{n,Y-90}^{0}$ Y-90 net count rate related to the time of Sr/Y separation, in s⁻¹;

- t_i time period between Sr/Y separation and start of measurement duration of single measurement i, in s;
- V volume of the sample, in m³;

 ε_{Y-90} detection efficiency of Y-90, in Bq⁻¹·s⁻¹;

 $\eta_{\rm Y}$ chemical yield of yttrium;

 $\lambda_{\text{Sr-90}}$ decay constant of Sr-90, in s⁻¹;

 λ_{Y-90} decay constant of Y-90, in s⁻¹;

 φ procedural calibration factor, in Bq·s·m⁻³.

5.1.2 Standard uncertainty of the output quantity

The standard uncertainty of the activity concentration u(c) is calculated according to Equation (4):

$$u(c) = c \cdot \sqrt{u_{\rm rel}^2 (R_{\rm n,Y-90}^0) + u_{\rm rel}^2 (f_{\rm A}) + u_{\rm rel}^2 (\varepsilon_{\rm Y-90}) + u_{\rm rel}^2 (\eta_{\rm Y}) + u_{\rm rel}^2 (V)}$$
(4)

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Herein are:

u(c)	standard uncertainty of the activity concentration of Sr-90, in Bq \cdot m ⁻³ ;
t _m	duration of counting source, in s;
t_0	duration of background measurement, in s;
$R_{n,Y-90}^{0}$	relative standard uncertainty of the Y-90 net count rate related to the time of Sr/Y separation;
$u_{\rm rel}(f_{\rm A})$	relative standard uncertainty of the correction factor for the decay;
$u_{\rm rel}(\varepsilon_{\rm Y-90})$	relative standard uncertainty of the detection efficiency;
$u_{\rm rel}(\eta_{\rm Y})$	relative standard uncertainty of the chemical yield;
$u_{\rm rel}(V)$	relative standard uncertainty of the volume.

In general, the contribution of the relative standard uncertainty of the correction factor for the decay $u_{rel}(f_A)$ can be neglected for the calculation of the combined relative standard uncertainty.The calculation of the standard uncertainty of the Y-90 net count rate u ($R_{n,Y-90}^0$) related to the time of Sr/Y separation is carried out by a software, e.g. UncertRadio.

5.2 Worked example

In the worked examples of the Sections 5.2 and 6.2, the interim results and the result are given with four significant digits. Deviations from the calculated values are possible when using another number of significant digits.

The following values are used in the worked example:

<i>R</i> g,1	=	0,040 s ⁻¹ ;	$u(R_{g,1})$	=	0,0014 s ⁻¹ ;
$R_{\rm g,2}$	=	0,038 s ⁻¹ ;	$u(R_{\rm g,2})$	=	0,0013 s ⁻¹ ;
$R_{g,3}$	=	0,039 s ⁻¹ ;	$u(R_{g,3})$	=	0,0013 s⁻¹;
$R_{\rm g,4}$	=	0,033 s ⁻¹ ;	$u(R_{\rm g,4})$	=	0,0012 s ⁻¹ ;
$R_{\rm g,5}$	=	0,032 s ⁻¹ ;	$u(R_{g,5})$	=	0,0012 s⁻¹;
$R_{\rm g,6}$	=	0,031 s ⁻¹ ;	$u(R_{\rm g,6})$	=	0,0012 s ⁻¹ ;
<i>R</i> g,7	=	0,026 s ⁻¹ ;	$u(R_{g,7})$	=	0,0011 s⁻¹;
$R_{\rm g,8}$	=	0,026 s ⁻¹ ;	$u(R_{g,8})$	=	0,0011 s⁻¹;
R_0	=	0,0017 s ⁻¹ ;	$u(R_0)$	=	0,00014 s ⁻¹ ;
t_1	=	16 200 s;	t_5	=	102 600 s;
t_2	=	37 800 s;	t_6	=	124 200 s;
t_3	=	59 400 s;	t_7	=	145 800 s;

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t_4	=	81 000 s;	t_8	=	167 400 s;
t_0	=	84 000 s;	t _m	=	21 600 s;
t _A	=	1,58 · 10 ⁷ s;	arphi	=	83,456 Bq · s · m ⁻³ ;
$\lambda_{\rm Sr-90}$	=	7,627 · 10 ⁻¹⁰ s ⁻¹ ;	λ_{Y-90}	=	3,0065 · 10⁻ ⁶ s⁻¹;
E _{Y-90}	=	0,35 Bq⁻¹ · s⁻¹;	$u_{\rm rel}(\varepsilon_{\rm Y-90})$	=	0,05;
$\eta_{ m Y}$	=	0,77;	$u_{\rm rel}(\eta_{\rm Y})$	=	0,05;
V	=	0,045 m ³ ;	$u_{\rm rel}(V)$	=	0,02.

The calculations are carried out with the software UncertRadio. The values of the gross count rates, the time intervals t_i and the background count rate are filled into a software dialog. From these, the values and uncertainties of the net count rates are determined. The resulting graphical representation of the decay curve is shown in Figure 1.





The value of the Y-90 net count rate related to the time of Sr/Y separation and the value of the standard uncertainty assigned to it are as follows:

 $R_{n,Y-90}^0 = 0,04230 \text{ s}^{-1}$

$$u(R_{n,Y-90}^0) = 0,6197 \cdot 10^{-3} \text{ s}^{-1}$$

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The activity concentration c is expressed by Equation (1):

$$c = \frac{0.04230 \text{ s}^{-1} \cdot \text{e}^{7.627 \cdot 10^{-10} \cdot 1.58 \cdot 10^{7}}}{0.35 \text{ Bq}^{-1} \cdot \text{s}^{-1} \cdot 0.77 \cdot 0.045 \text{ m}^{3}} = \frac{0.042813 \text{ Bq}}{0.01213 \text{ m}^{3}} = 3.530 \text{ Bq} \cdot \text{m}^{-3}$$

The standard uncertainty u(c) of the activity concentration is calculated according to equation:

$$u(c) = 3,530 \text{ Bq} \cdot \text{m}^{-3} \cdot \sqrt{\left(\frac{0,0006197}{0,04230}\right)^2 + 0,05^2 + 0,05^2 + 0,02^2} = 0,2645 \text{ Bq} \cdot \text{m}^{-3}$$

The activity concentration is thus:

 $c = (3,530 \pm 0,265) \text{ Bq} \cdot \text{m}^{-3}$

5.3 Consideration of the uncertainties

Uncertainty contributions arising from sampling are not taken into account in the framework of this Procedures Manual, as these can depend on many different and often not quantifiable factors.

The standard uncertainty of the analysis takes into consideration the standard uncertainties of the statistical counting, of the calibration, of the chemical yield and of the volume of the sample. The standard uncertainties of the duration of measurement and of the decay constant are neglected.

6 Characteristic limits of the procedure

The calculation of the characteristic limits follows the standard series ISO 11929 [4]. Further considerations concerning the characteristic limits are to be found in the General Chapter CHAGR-ISO-01 of this Procedures Manual.

An Excel spreadsheet (see Section 7.1) is not available. A project file for the software UncertRadio (see Section 0) is available on the website of this Procedures Manual.

6.1 Equations

6.1.1 Decision threshold

The decision threshold c^* is determined using Equation (5):

$$c^* = k_{1-\alpha} \cdot \tilde{u}(0)$$

(5)

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Herein are:

 c^* decision threshold for the activity concentration, in Bq·m⁻³;

 $k_{1-\alpha}$ quantile of the normal distribution for α = 0,0014.

An explicit equation is not presented here due to the complexity of linear deconvolution.

6.1.2 Detection limit

The detection limit $c^{\#}$ is calculated according to the implicit Equation (6):

$$c^{\#} = c^* + k_{1-\beta} \cdot \tilde{u}(c^{\#}) \tag{6}$$

Herein are

 $c^{\#}$ detection limit of the activity concentration, in Bq·m⁻³;

 $k_{1-\beta}$ quantile of the normal distribution for $\beta = 0,05$.

An explicit equation is not presented here due to the complexity of linear deconvolution.

6.1.3 Limits of the coverage interval

The calculation of limits of the coverage interval is not required.

6.2 Worked examples

The characteristic limits of the activity concentration are calculated with the values of Section 5.2 and the following values:

$$k_{1-\alpha} = 3;$$
 $k_{1-\beta} = 1,645;$
 $u_{rel}(\varphi) = 0,0054.$

The values of the decision threshold c^* and the detection limit $c^{\#}$ are calculated with the software:

 $c^* = 0.0566 \text{ Bq} \cdot \text{m}^{-3}$ $c^\# = 0.0922 \text{ Bq} \cdot \text{m}^{-3}$

7 Software supported calculation

7.1 View of the Excel spreadsheet

Due to the complexity of the calculation, no Excel sheet is available.

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7.2 View of the UncertRadio result page

UR UncertRadio: Calculation of uncertainty budget and detect	ion limits - D-Sr-90-MWASS-01_V202	- □ ×
	s 🏢 📰 🗟 💡 🔳	A Belp Save to csv
Procedure Equations Values, Uncert	ainties Uncertainty budg	get Results Text Editor
Final measurement result for c : Value output quantity: 3,5314 extendend (Std)uncertainty: 0.26461 relative ext.(Std)uncertainty: 7,4930 Best Bayesian Estimates: Value output quantity: 3,5314 extendend (Std)uncertainty: 0.26461 lower range limit: 3,0128 upper range limit: 4,050	Bq/m ³ Bq/m ³ Min. Coverage-Intervall Bq/m ³ Bq/m ³ Bq/m ³	Coverage factor k: 1.0 Probability (1-gamma): 0.950 Decision threshold and detection limit for c : Decision threshold (DT): 5.6576E-02 Bq/m ³ Iterations: 1 Detection limit (DL): 9.2199E-02 Bq/m ³ Iterations: 1 k_alpha=3.000, k_beta=1.645 Method: ISO 11929:2019, by iteration PLSQ: standard uncertainty of the fit parameter: from LS analysis: 6.19636E-04 1/s from uncertainty propagation: 5.89439E-04 1/s
Monte Carlo Simulation: Number of simul. measurments 10000 Number of runs: 1 primary estimate: 3,5493 uncertainty primary estimate: 0,26628 Value output quantity: 3,5493 extendend uncertainty: 0,26628 relative extd. (Std)uncertainty: 7,5023 lower range limit: 3,0645 upper range limit: 4,1198 Decision threshold (DT): 5,74294E-02 Detection limit (DL): 9,25585E-02 active run: 1	Values <0 included min. Coverage interval relSD%: Bq/m ³ 0.075 Bq/m ³ 0.707 Bq/m ³ 0.707 Bq/m ³ 0.707 % Bq/m ³ 0.707 % Bq/m ³ 0.232 Bq/m ³ 0.173 Bq/m ³ 0.173 Bq/m ³ 1.78 12 Start MC	reduced Chi-square: 1,5025

The corresponding UncertRadio project file is available on the website of this Procedures Manual.

8 Catalogue of the chemicals und equipment

8.1 Chemicals

The chemicals used should be of analytically pure quality.

	ammonia, NH ₃ :	13,3 mol·l ⁻¹ ;
—	ammoniacal water:	1:1 (v:v) conc. ammonia (13,3 mol·l ⁻¹) and distilled water;
_	diisooctyl hydrogen phosphate (DEHP) or bis-(2-ethylhexyl- phosphoric acid) (HDEHP);	
	DEHP-solution:	0,45 mol·l ⁻¹ :

dissolve 150 ml DEHP in 850 ml n-heptane;

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—	ethanol:	denatured;
	sodium hydrogen phosphate solution:	cold saturated solution from disodium hydrogen phosphate dodecahydrate, Na ₂ HPO ₄ ·12 H ₂ O;
	n-heptane;	
	oxalic acid solution:	cold saturated solution from oxalic acid dihydrate;
	hydrochloric acid, HCl:	1 mol·l ⁻¹ , 9 mol·l ⁻¹ , 12 mol·l ⁻¹ ;
	nitric acid, HNO3:	2 mol·l ⁻¹ , 5 mol·l ⁻¹ , 7 mol·l ⁻¹ , 14,4 mol·l ⁻¹ ;
	toluene;	
—	tricaprylylmethylammonium chloride:	e. g. Aliquat [®] 336;
	tricaprylylmethylammonium chloride solution:	300 ml Aliquat [®] 336 in 700 ml toluene;
	yttrium chloride hexahydrate, YCl ₃ ·6 H ₂ O:	
	yttrium carrier solution:	5 mg Y ³⁺ per ml solution:
		16 ml of nitric acid (approx. 7 mol·l ⁻¹) are added to 34,12 g of yttrium chloride hexahydrate and filled to 2 l with distilled water;
	zirconium(IV)-oxide chloride octa- hydrat, ZrOCl ₂ ·8 H ₂ O;	
_	zirconium solution:	50 mg Zr ⁴⁺ per ml solution: dissolve 17,66 g of zirconium(IV)oxide chloride octahydrate in distilled water and fill to 100 ml.

8.2 Equipment

The following equipment is used for the procedure:

- plastic container, 50 l:
- stirrer motor with a propeller stirrer;
- laboratory centrifuge ;
- suction devices: witt jar with 400-ml-beaker, suction flask, water jet pump;
- suction filter (porosity 3, label 17D, ø 65 mm);
- glass vacuum filtration device (ø 25 mm, volume 30 ml, with glass frit);
- filter paper (ø 24 mm, type blue ribbon, ashless);

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- porcelain crucible (ø 40 mm);
- muffle furnace;
- surface evaporator;
- usual equipment of a radiochemical laboratory;
- counting tray made of stainless steel;
- low-level proportional counter.

References

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