Procedure of determining the activity concentration of strontium-90 in sea water by proportional counting

D-Sr-90-MWASS-01

Authors:

J. Herrmann
A. Gottschalk
C. Wehner
S. Schmied

Federal coordinating office for sea water, suspended particulate matter and sediments

(Leitstelle für Meerwasser, Meeresschwebstoff und -sediment)

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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 sea water according to the IMIS-routine programme (1). This procedure is validated for sample volumes of 20 l to 90 l.

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 $mol \cdot l^{-1}$) per litre sea water or 2 ml of hydrochloric acid (approx. 6 $mol \cdot l^{-1}$) per litre sea water.

3 Analysis

3.1 Principle of the procedure

From the sample volume, an aliquot of usually 45 l of acidified sea water 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 sea water 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. Thorium isotopes are separated radiochemically, whereas trivalent radionuclides are taken into account only at the calculation.

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Note:
In particular, these interferences have to be taken into account when determining low activity concentrations of Sr-90 in sea water, 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 sea water 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 sea water sample prepared after step 3.2 or the collected eluate after step 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 $\mathrm{mol \cdot l^{-1}}$).

3.3.3 Afterwards, a volume of 10 ml of yttrium carrier solution (50 mg Y^{3+}) is added to the sample under continuous mixing.

3.3.4 A volume of 250 ml of DEHP solution (0,45 $\mathrm{mol \cdot l^{-1}}$) is added and the time of this addition is noted.

3.3.5 Then, the sample is mixed thoroughly for five minutes.

3.3.6 The stirrer is removed from the container. **This time is noted as time of the yttrium separation**.

3.3.7 The sample is left for at least 20 minutes for phase separation, whereby the yttrium containing organic phase forms the upper one.

3.3.8 The lower aqueous phase is siphoned except for approx. 100 ml by means of either a hose or a tube and is discarded.

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

3.3.10 The plastic container is rinsed successively in each case with approx. 20 ml of distilled water, n-heptane and distilled water again, whereby the rinse solutions are combined with the solution in the separating funnel.

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

Note:
If required, the aqueous solutions from this step and step 3.3.8 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.

3.3.12 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 $\mathrm{mol \cdot l^{-1}}$) for one minute, respectively. The hydrochloric phases (lower phases) are drained and discarded in each case after phase separation.

3.3.13 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 pha¬ses (lower phases) are drained and combined in another 500 ml-separating funnel already containing a portion of 250 ml of trioctylmethylammonium chloride solution.

3.3.14 Afterwards, the solution is shaked for two minutes.

3.3.15 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.

3.3.16 A volume of 350 ml of concentrated ammonia solution (approx. 13,3 mol·l^{-1}) is then added slowly while stirring the hydrochloric solution whereby yttrium precipitates as yttrium hydroxide.

3.3.17 The solution is then heated with boiling capillaries on a heating plate bringing closely to boiling point. The appearing gas bubbles are removed by stirring occasionally.

3.3.18 While cooling down to room temperature, the precipitate of yttrium hydro¬xide settles down.

3.3.19 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, steps 3.3.30 to 3.3.36 replace steps 3.3.20 to 3.3.22.

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

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

3.3.22 The precipitate is dissolved with 5 ml to 10 ml nitric acid (2 mol·l^{-1}) and is then collected in the beaker inside the witt jar. The suction filter is then washed with distilled water until a total volume of approx. 150 ml is collected in the beaker.

3.3.23 The solution is heated briefly on a heating plate and 10 ml of a cold saturated solution of oxalic acid are added. The yttrium precipitates as yttrium oxalate.

3.3.24 After cooling down, 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.

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

3.3.26 The filter together with the precipitate is put in a porcelain crucible and is annealed in a muffle furnace at 900 °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.

3.3.27 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.

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

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

Separation of thorium

3.3.30 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.

3.3.31 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.

3.3.32 The clear supernatant is decanted and discarded. The precipitate is dissolved in approx. 3 ml of nitric acid (5 $\text{mol} \cdot \text{l}^{-1}$).

3.3.33 The pH-value of the solution is adjusted to 1,3 to 1,5 by distilled water.

3.3.34 For co-precipitation, 1 ml of zirconium carrier solution (50 mg Zr^{4+}) and 1 ml of cold saturated sodium hydrogen phosphate solution are added in excess.

3.3.35 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.

3.3.36 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 $\text{mol} \cdot \text{l}^{-1}$) into a 500-ml-separating funnel, followed by a five-minute extraction. The further production follows the radiochemical separation from step 3.3.11 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 step 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 function is determined by a semi logarithmic scale of the non-decay-corrected net count rates against the times elapsed from the beginning of the first measurement. A maximum of two outliers of the non-decay-corrected net count rates are acceptable; the corresponding values are eliminated during determination of the decay function.

If the coefficient of determination ranges between 0,7 and 1, an undisturbed counting source is assumed, whereby long-lived interfering radionuclides cannot be excluded. In this 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. At a coefficient of determination of less than 0,7, a measurement value close to the decision threshold is present as a rule, which cannot necessarily be interpreted as a Sr-90 activity concentration. In this case, the measurement has to be discarded or, if necessary, has to be reproduced with a retention sample (see step 3.3.11).

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 these procedures manuals.

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 3.3.6) decay-corrected net count rates $R_{\mathrm{n},i}$ ($i = 1,\ldots, 8$) 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

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

$$c = \frac{R_\mathrm{n} \cdot f_\mathrm{A}}{\varepsilon \cdot \eta \cdot V} = \frac{R_\mathrm{n} \cdot \mathrm{e}^{\lambda \cdot t_\mathrm{A}}}{\varepsilon \cdot \eta \cdot V} = R_\mathrm{n} \cdot \varphi \tag{1}$$

with

$$R_\mathrm{n} = \overline{R}_\mathrm{g} - R_0$$

Herein are:

c	activity concentration of Sr-90 in Bq·m^{-3};
f_A	correction factor for the decay;
λ	decay constant of Sr-90 in s^{-1};
t_A	time period between sampling and beginning oft he yttrium separation in s;
R_n	net count rate in s^{-1};
$\overline{R}_\mathrm{g}$	mean, at the time of yttrium separation decay-corrected gross count rate of m measurements of the counting source in s^{-1},

with $$\overline{R}_\mathrm{g} = \frac{1}{m} \cdot \sum_{i=1}^{m} R_{\mathrm{g},i} \cdot \mathrm{e}^{\lambda_{\mathrm{Y-90}} \cdot t_{\mathrm{B},i}}$$

$R_{\mathrm{g},i}$	gross count rate of the single measurement i in s^{-1};
R_0	background count rate in s^{-1};
$\lambda_{\mathrm{Y-90}}$	decay constant of Y-90 in s^{-1};
$t_{\mathrm{B},i}$	time period between yttrium separation and end of the measurement period of the single measurement i in s;
φ	procedural calibration factor in Bq·s·m^{-3};
ε	detection efficiency in Bq^{-1}·s^{-1};
η	chemical yield;
V	volume of the sample in m^3.

The standard uncertainty of the activity concentration $u(c)$ is calculated according to equation (2):

$$u(c) = c \cdot \sqrt{\frac{1}{m} \cdot \frac{\overline{R}_\mathrm{g}}{t_\mathrm{m}} + \frac{R_0}{t_0} + u_\mathrm{rel}^2(f_\mathrm{A}) + u_\mathrm{rel}^2(\varepsilon) + u_\mathrm{rel}^2(\eta) + u_\mathrm{rel}^2(V)} \tag{2}$$

Herein are:

$u(c)$	standard uncertainty of the activity concentration in $\mathrm{Bq \cdot m^{-3}}$;
t_m	duration of measurement in s;
t_0	duration of the background measurement in s;
$u_\mathrm{rel}(f_\mathrm{A})$	relative standard uncertainty of the correction factor for the decay;
$u_\mathrm{rel}(\varepsilon)$	relative standard uncertainty of the detection efficiency;
$u_\mathrm{rel}(\eta)$	relative standard uncertainty of the chemical yield;
$u_\mathrm{rel}(V)$	relative standard uncertainty of the volume.

In general, the contribution of the relative standard uncertainty of the correction factor for the decay $u_\mathrm{rel}(f_\mathrm{A})$ can be neglected for the calculation of the combined relative standard uncertainty.

5.2 Worked example

The following values are used in the worked example:

$R_{\mathrm{g},1} = 0{,}040\ \mathrm{s^{-1}}$; $u(R_{\mathrm{g},1}) = 0{,}0014\ \mathrm{s^{-1}}$;
$R_{\mathrm{g},2} = 0{,}038\ \mathrm{s^{-1}}$; $u(R_{\mathrm{g},2}) = 0{,}0013\ \mathrm{s^{-1}}$;
$R_{\mathrm{g},3} = 0{,}039\ \mathrm{s^{-1}}$; $u(R_{\mathrm{g},3}) = 0{,}0013\ \mathrm{s^{-1}}$;
$R_{\mathrm{g},4} = 0{,}033\ \mathrm{s^{-1}}$; $u(R_{\mathrm{g},4}) = 0{,}0012\ \mathrm{s^{-1}}$;
$R_{\mathrm{g},5} = 0{,}032\ \mathrm{s^{-1}}$; $u(R_{\mathrm{g},5}) = 0{,}0012\ \mathrm{s^{-1}}$;
$R_{\mathrm{g},6} = 0{,}031\ \mathrm{s^{-1}}$; $u(R_{\mathrm{g},6}) = 0{,}0012\ \mathrm{s^{-1}}$;
$R_{\mathrm{g},7} = 0{,}026\ \mathrm{s^{-1}}$; $u(R_{\mathrm{g},7}) = 0{,}0011\ \mathrm{s^{-1}}$;
$R_{\mathrm{g},8} = 0{,}026\ \mathrm{s^{-1}}$; $u(R_{\mathrm{g},8}) = 0{,}0011\ \mathrm{s^{-1}}$;
$t_{\mathrm{B},1} = 17520\ \mathrm{s}$; $t_{\mathrm{B},2} = 39120\ \mathrm{s}$;
$t_{\mathrm{B},3} = 60720\ \mathrm{s}$; $t_{\mathrm{B},4} = 82320\ \mathrm{s}$;
$t_{\mathrm{B},5} = 103920\ \mathrm{s}$; $t_{\mathrm{B},6} = 125520\ \mathrm{s}$;
$t_{\mathrm{B},7} = 147120\ \mathrm{s}$; $t_{\mathrm{B},8} = 168720\ \mathrm{s}$;
$R_0 = 0{,}0017\ \mathrm{s^{-1}}$; $u(R_0) = 0{,}00014\ \mathrm{s^{-1}}$;
$\lambda = 7{,}62 \cdot 10^{-10}\ \mathrm{s^{-1}}$; $\lambda_\text{Y-90} = 3{,}006 \cdot 10^{-6}\ \mathrm{s^{-1}}$;
$t_\mathrm{A} = 6\ \text{Monate} = 1{,}58 \cdot 10^{7}\ \mathrm{s}$; $t_\mathrm{m} = 360\ \mathrm{min} = 21600\ \mathrm{s}$;
$t_0 = 1400\ \mathrm{min} = 84000\ \mathrm{s}$; $\varphi = 83{,}456\ \mathrm{Bq \cdot s \cdot m^{-3}}$;
$\varepsilon = 0{,}35\ \mathrm{Bq^{-1} \cdot s^{-1}}$; $u_\mathrm{rel}(\varepsilon) = 0{,}05$;
$\eta = 0{,}77$; $u_\mathrm{rel}(\eta) = 0{,}05$;
$V = 0{,}045\ \mathrm{m^3}$; $u_\mathrm{rel}(V) = 0{,}02$.

The activity concentration *c* is calculated according to equation (1):

$$\overline{R}_\mathrm{b} = \frac{1}{8} \cdot \big(0{,}04\ \mathrm{s}^{-1} \cdot \mathrm{e}^{3{,}006 \cdot 10^{-6} \cdot 17520} + 0{,}038\ \mathrm{s}^{-1} \cdot \mathrm{e}^{3{,}006 \cdot 10^{-6} \cdot 39120} +$$

$$+ 0{,}039\ \mathrm{s}^{-1} \cdot \mathrm{e}^{3{,}006 \cdot 10^{-6} \cdot 60720} + 0{,}033\ \mathrm{s}^{-1} \cdot \mathrm{e}^{3{,}006 \cdot 10^{-6} \cdot 82320} +$$

$$+ 0{,}032\ \mathrm{s}^{-1} \cdot \mathrm{e}^{3{,}006 \cdot 10^{-6} \cdot 103920} + 0{,}031\ \mathrm{s}^{-1} \cdot \mathrm{e}^{3{,}006 \cdot 10^{-6} \cdot 125520} +$$

$$+ 0{,}026\ \mathrm{s}^{-1} \cdot \mathrm{e}^{3{,}006 \cdot 10^{-6} \cdot 147120} + 0{,}026\ \mathrm{s}^{-1} \cdot \mathrm{e}^{3{,}006 \cdot 10^{-6} \cdot 168720}\big) = 0{,}0433\ \mathrm{s}^{-1}$$

$$R_\mathrm{n} = 0{,}0433\ \mathrm{s}^{-1} - 0{,}0017\ \mathrm{s}^{-1} = 0{,}0416\ \mathrm{s}^{-1}$$

$$c = \frac{0{,}0416\ \mathrm{s}^{-1} \cdot \mathrm{e}^{7{,}62 \cdot 10^{-10} \cdot 1{,}58 \cdot 10^{-7}}}{0{,}035\ \mathrm{Bq}^{-1} \cdot \mathrm{s}^{-1} \cdot 0{,}77 \cdot 0{,}045\ \mathrm{m}^3} = \frac{0{,}0421\ \mathrm{Bq}}{0{,}012\ \mathrm{m}^3} = 3{,}47\ \mathrm{Bq} \cdot \mathrm{m}^{-3}$$

The standard uncertainty *u*(*c*) of the activity concentration *c* is calculated according to equation (2):

$$u(c) = 3{,}47\ \mathrm{Bq} \cdot \mathrm{m}^{-3} \cdot \sqrt{\frac{1}{8} \cdot \frac{0{,}0433}{21600} + \frac{0{,}017}{84000} + 0{,}05^2 + 0{,}05^2 + 0{,}02^2} = 0{,}26\ \mathrm{Bq} \cdot \mathrm{m}^{-3}$$

Thus, the activity concentration is:

$$c = (3{,}47 \pm 0{,}26)\ \mathrm{Bq} \cdot \mathrm{m}^{-3}$$

5.3 Consideration of uncertainties

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 ISO 11929.

Currently, a project file for the software UncertRadio (see section 7.3.2) are available on the website of this procedures manual.

Further considerations concerning the characteristic limits are to be found in the general chapters ERK/NACHWEISGR-ISO-01 and ERK/NACHWEISGR-ISO-02 of these procedures manuals.

6.1 Equations

6.1.1 Decision threshold

Before calculating the detection limit $c^{\#}$ the decision threshold c^{*} is determined using equation (3):

$$c^{*} = k_{1-\alpha} \cdot \varphi \cdot \sqrt{R_0 \cdot \left(\frac{1}{m} \cdot \frac{1}{t_\mathrm{m}} + \frac{1}{t_0}\right)} \tag{3}$$

Herein are:

c^{*} decision threshold in Bq·m^{-3};

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

6.1.2 Detection limit

The detection limit $c^{\#}$ is calculated according to the implicit equation (4):

$$c^{\#} = c^{*} + k_{1-\beta} \cdot \sqrt{{c^{\#}}^2 \cdot u_\mathrm{rel}^2(\varphi) + \varphi^2 \cdot \left(\frac{c^{\#}}{t_\mathrm{m} \cdot \varphi} + \frac{1}{m} \cdot \frac{R_0}{t_\mathrm{m}} + \frac{R_0}{t_0}\right)} \tag{4}$$

The relative measurement uncertainty of the procedural calibration factor $u_\mathrm{rel}(\varphi)$ is calculated according to equation (5):

$$u_\mathrm{rel}(\varphi) = \sqrt{u_\mathrm{rel}^2(f_\mathrm{A}) + u_\mathrm{rel}^2(\varepsilon) + u_\mathrm{rel}^2(\eta) + u_\mathrm{rel}^2(V)} \tag{5}$$

In equations (4) and (5) are:

$c^{\#}$ detection limit in Bq·m^{-3};

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

After implementation of the auxiliary quantities Ψ and θ the detection limit $c^{\#}$ is calculated using equation (6):

$$c^{\#} = \frac{c^{*} \cdot \Psi}{\theta} \cdot \left\{1 + \sqrt{1 - \frac{\theta}{\Psi^2} \cdot \left(1 - \frac{k_{1-\beta}^2}{k_{1-\alpha}^2}\right)}\right\} \tag{6}$$

with

$$\theta = 1 - k_{1-\beta}^2 \cdot u_\mathrm{rel}^2(\varphi)$$

$$\Psi = 1 + \frac{k_{1-\beta}^2}{2 \cdot c^{*}} \cdot \varphi \cdot \frac{1}{t_\mathrm{m}}$$

6.1.3 Confidence intervals

The calculation of the upper and lower confidence intervals is not required in this case.

6.2 Worked example

Following values are taken for a sea water sample of 45 l:

R_0	=	0,0017 s^{-1};	t_m	=	360 min = 21600 s;
t_0	=	1400 min = 84000 s;			
$k_{1-\alpha}$	=	3;	$k_{1-\beta}$	=	1,645;
φ	=	83,456 Bq·s·m^{-3};	$u_{\mathrm{rel}}^2(\varphi)$	=	0,0054.

The value of the decision threshold c^* is

$$c^* = 3 \cdot 83{,}456 \cdot \sqrt{0{,}0017 \cdot \left(\frac{1}{8} \cdot \frac{1}{21600} + \frac{1}{84000}\right)}\ \mathrm{Bq \cdot m^{-3}} = 0{,}0434\ \mathrm{Bq \cdot m^{-3}}$$

and the value of the detection limit $c^\#$ is

$$c^\# = \frac{0{,}0434 \cdot 1{,}120}{0{,}985} \cdot \left\{1 + \sqrt{1 - \frac{0{,}982}{1{,}120^2} \cdot \left(1 - \frac{1{,}645^2}{3^2}\right)}\right\}\ \mathrm{Bq \cdot m^{-3}} = 0{,}0825\ \mathrm{Bq \cdot m^{-3}}$$

The values of the auxiliary quantities are

$$\theta = 1 - 1{,}645^2 \cdot 0{,}0054 = 0{,}985$$

$$\Psi = 1 + \frac{1{,}645^2}{2 \cdot 0{,}0434\ \mathrm{Bq \cdot m^{-3}}} \cdot 83{,}456\ \mathrm{Bq \cdot s \cdot m^{-3}} \cdot \frac{1}{21600\ \mathrm{s}} = 1{,}120$$

7 Catalogue of chemicals and equipment

7.1 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-Heptan;

— ethanol:	denatured;
— sodium hydrogen phosphate solution:	cold saturated solution from disodium hydrogen phosphate dodekahydrate, $Na_2HPO_4 \cdot 12\ H_2O$;
— n-heptane;	
— oxalic acid solution:	cold saturated solution from oxalic acid dihydrate;
— hydrochloric acid, HCl:	1 $\mathrm{mol \cdot l^{-1}}$, 9 $\mathrm{mol \cdot l^{-1}}$, 12 $\mathrm{mol \cdot l^{-1}}$;
— nitric acid, HNO_3:	2 $\mathrm{mol \cdot l^{-1}}$, 5 $\mathrm{mol \cdot l^{-1}}$, 7 $\mathrm{mol \cdot l^{-1}}$, 14,4 $\mathrm{mol \cdot l^{-1}}$;
— toluene;	
— trioctylmethylammonium chloride:	e. g. Adogen® 464;
— trioctylmethylammonium chloride solution:	300 ml Adogen® 464 in 700 ml toluene;
— yttrium chloride hexahydrate, $YCl_3 \cdot 6\ H_2O$:	
— yttrium carrier solution:	5 mg Y^{3+} per ml solution: 16 ml of nitric acid (approx. 7 $\mathrm{mol \cdot l^{-1}}$) are added to 34,12 g of yttrium chloride hexahydrate and filled to 2 l with distilled water;
— zirconium(IV)-oxide chloride octahydrat, $ZrOCl_2 \cdot 8\ H_2O$;	
— 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.

7.2 Equipment

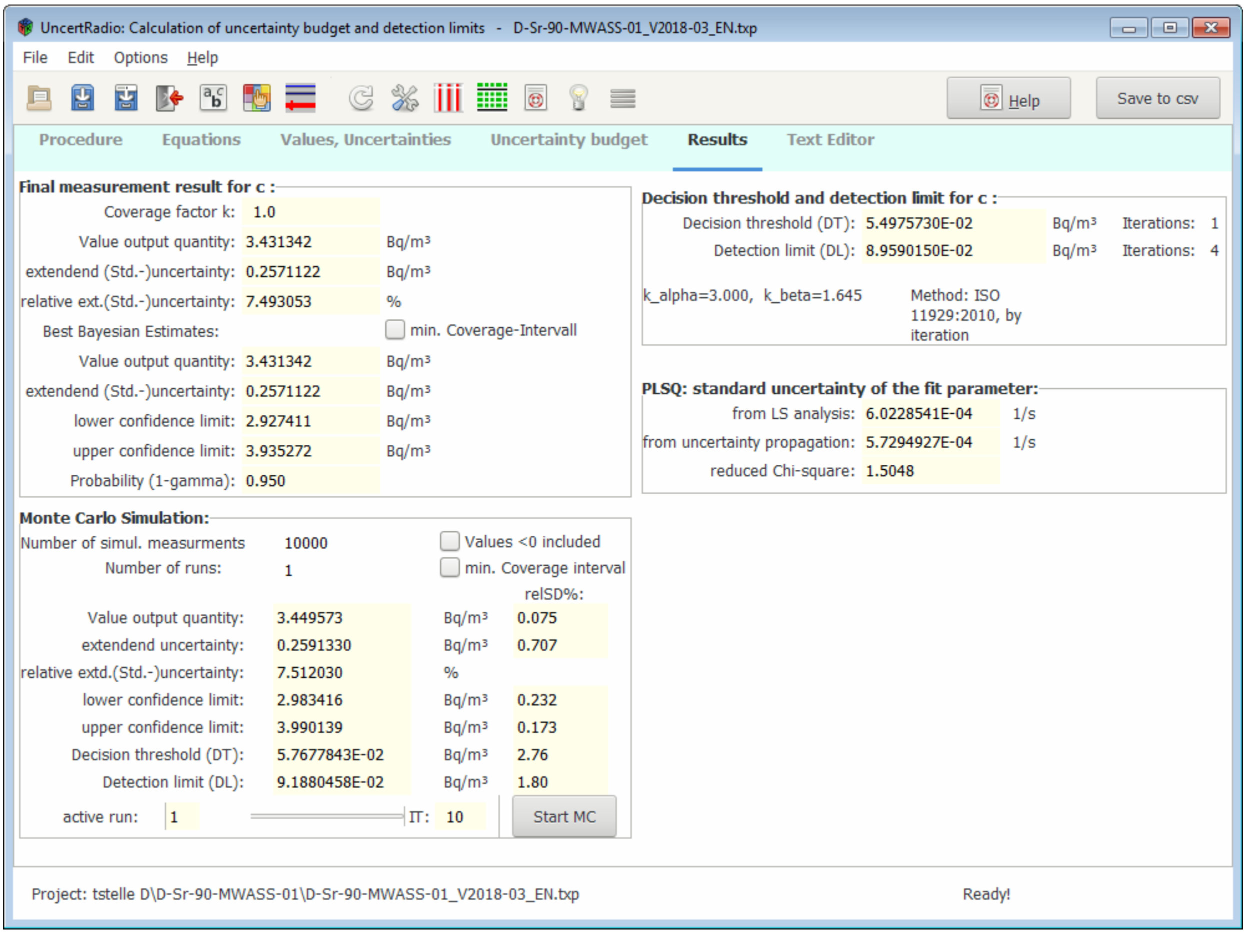
- plastic container, 50 l:
- stirrer motor with a propeller stirrer;
- laboratory centrifuge ;
- suction devices: witt jar with 400-ml-beaker, suction flask, water jet pump;
- suction filter (porosity 3, label 17D, Ø 65 mm);
- glass vacuum filtration device (Ø 25 mm, volume 30 ml, with glass frit);
-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

7.3 Software supported calculation

7.3.1 Example of an Excel spreadsheet

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

7.3.2 Example of an UncertRadio project



Final measurement result for c :

Coverage factor k: 1.0
Value output quantity: 3.431342 Bq/m^3
extendend (Std.-)uncertainty: 0.2571122 Bq/m^3
relative ext.(Std.-)uncertainty: 7.493053 %
Best Bayesian Estimates: ☐ min. Coverage-Intervall
Value output quantity: 3.431342 Bq/m^3
extendend (Std.-)uncertainty: 0.2571122 Bq/m^3
lower confidence limit: 2.927411 Bq/m^3
upper confidence limit: 3.935272 Bq/m^3
Probability (1-gamma): 0.950

Monte Carlo Simulation:

Number of simul. measurments 10000 ☐ Values <0 included
Number of runs: 1 ☐ min. Coverage interval

			relSD%:
Value output quantity:	3.449573	Bq/m^3	0.075
extendend uncertainty:	0.2591330	Bq/m^3	0.707
relative extd.(Std.-)uncertainty:	7.512030	%	
lower confidence limit:	2.983416	Bq/m^3	0.232
upper confidence limit:	3.990139	Bq/m^3	0.173
Decision threshold (DT):	5.7677843E-02	Bq/m^3	2.76
Detection limit (DL):	9.1880458E-02	Bq/m^3	1.80

active run: 1 IT: 10

Decision threshold and detection limit for c :

Decision threshold (DT): 5.4975730E-02 Bq/m^3 Iterations: 1
Detection limit (DL): 8.9590150E-02 Bq/m^3 Iterations: 4

k_alpha=3.000, k_beta=1.645 Method: ISO 11929:2010, by iteration

PLSQ: standard uncertainty of the fit parameter:

from LS analysis: 6.0228541E-04 1/s
from uncertainty propagation: 5.7294927E-04 1/s
reduced Chi-square: 1.5048

Project: tstelle D\D-Sr-90-MWASS-01\D-Sr-90-MWASS-01_V2018-03_EN.txp Ready!

The corresponding UncertRadio project file can be found on the website of this procedures manual.

References

(1) Borcherding, J.; Nies, H.: *An Improved Method for the Determination of ^{90}Sr in Large Samples of Seawater*. J. Radioanal. Nucl. Chem., 1986, Vol. 98, Nr. 1, S. 127 – 131.