Procedure for determining the activity concentration of radon-222 in drinking water

H-Rn-222-TWASS-01

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1 Scope

The isotope radon-222 (Rn-222) is a natural, alpha-emitting radioactive noble gas with a half-life of about 3,8 days, and is part of the decay series of uranium-238 (U-238).

The procedure described here is suitable for clear and colourless water samples of any origin, in particular drinking water, ground water, spring water and surface water in accordance with Section 162 of the Radiation Protection Act [1]. Applying this procedure under normal boundary conditions ensures a detection limit of 10 $Bq \cdot l^{-1}$ [2] in particular in the case of a short delay between sampling and measurement. A radiochemical sample preparation is not required.

2 Sampling

In order to determine the activity concentration of Rn-222 following the procedure described here, a sample volume of about 10 ml to 500 ml is required.

The sampling procedure depends on the local conditions and the specifications of the testing or measuring point. Two options are available:

- Sampling from a laminar flow of water is carried out using a disposable syringe with a 5 cm silicone hose attached, as illustrated in Figure 1a. Counting sources can be prepared on site immediately after sampling as described in Section 3.2.
- If the water sample cannot be prepared immediately for measurement, a volume (usually 500 ml) is filled to the brim (without air volume) into a suitable gas-tight sample container made of glass or polyethylene terephthalate (PET). From this sample bottle, the sub-sample to be measured is later taken with a suitable pipette or disposable syringe.

Note:
Test results on the suitability of various container materials and closures can be found in Annex A.

For further information on sampling as well as the transport and storage of samples, it is referred to the preface H-VORBEMERK-TWASS-02.

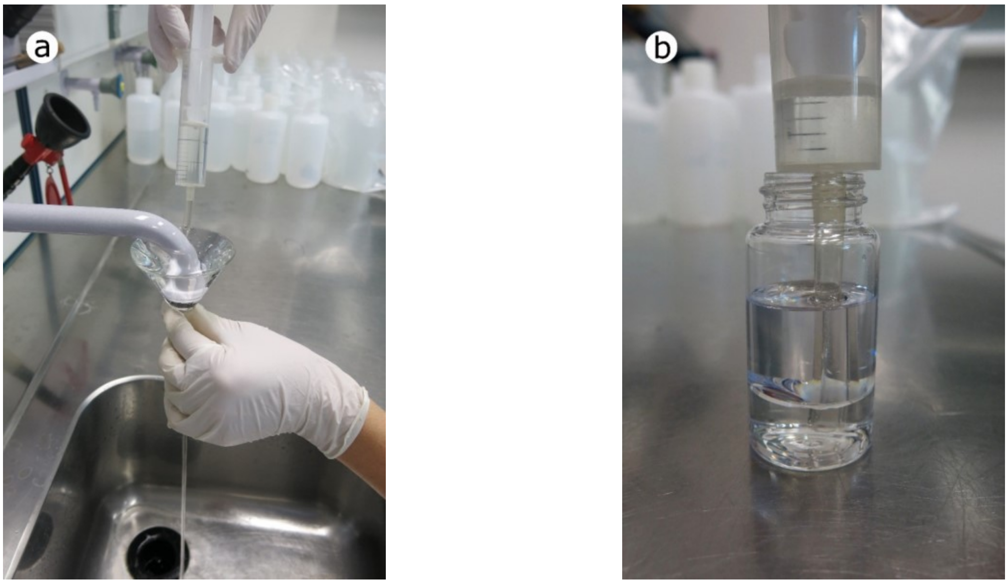


Fig. 1: a) Sampling from a laminar flow of water with a disposable syringe with a 5 cm silicone hose attached;
b) Careful layering of the water sample underneath the scintillation cocktail in a 20 ml glass vial.

3 Analysis

3.1 Principle of the procedure

For the purpose of determining the activity concentration of Rn-222, an aliquot of the water sample is mixed with a commercially available scintillation cocktail without pre-treatment. After equilibration of Rn-222 and its short-lived progenies, the counting source is measured in a liquid scintillation counter (LSC).

3.2 Sample preparation

To prepare a counting source, as shown in Figure 1b, 10 ml of the sampled water is transferred into a counting vial made of glass or polytetrafluoroethylene-coated high-density polyethylene (PTFE-HDPE), prefilled with 10 ml of a water-immiscible scintillation cocktail, suitable for the extraction of radon. After reaching the radioactive equilibrium of Rn-222 and its progenies, the counting source is measured in a liquid scintillation counter.

Note:
Counting vials made exclusively of polyethylene (PE) are not suitable for this measurement, because Rn-222 can diffuse through this material.

3.3 Radiochemical separation

A radiochemical separation is not required.

4 Measuring the activity

4.1 General

Rn-222 is part of the decay series of naturally occurring U-238 (Figure 2). The activity of Rn-222 is determined by LSC measurement. For this purpose, a measuring window covering the energy range of all contributions arising from Rn-222 and its short-lived alpha and beta emitting progenies to the LSC spectrum has to be defined. The parameters of the measuring window are determined by recording the spectrum of a calibration source.

Note:
Optimising the measurement window is possible by means of the '*Figure of Merit*', the quotient of the square of the detection efficiency ε and the background count rate R_0 in the measurement window [3].

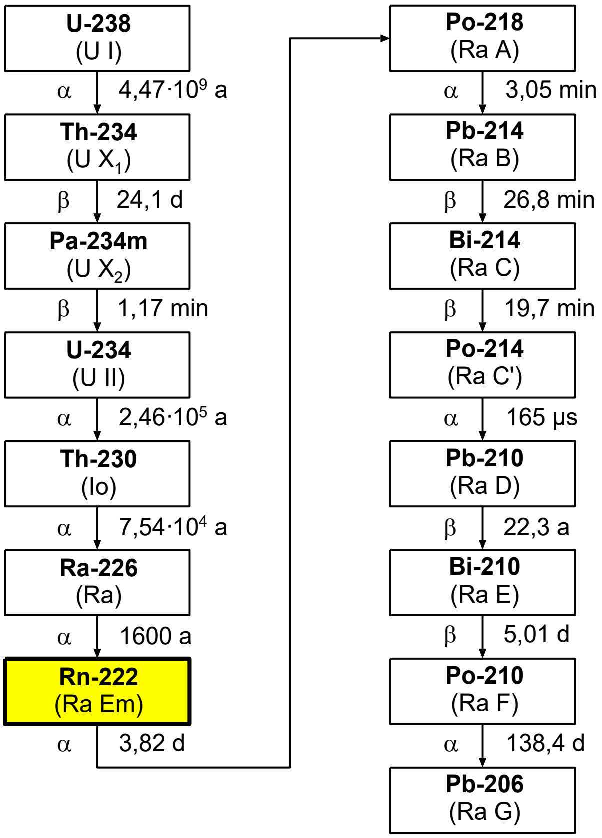


Fig. 2: Uranium-238 decay series; decay processes with emission intensities less than 0,1 % are omitted. The historical names of the individual isotopes are given in brackets.

4.2 Calibration

To prepare a calibration source, 10 ml of a Ra-226 standard solution are overlayered with the same scintillation cocktail used in Section 3.2 in a 20 ml glass counting vial. The counting vial has to be firmly closed and kept in the refrigerator upside down for at least 30 days in order to reach radioactive equilibration between Ra-226 and Rn-222.

Note:

The counting vials are stored upside down in order to avoid losses of radon and water vapour through the closure of the counting vial. The calibration sources stored in this manner can be used for up to three years.

Prior to each measurement, the calibration source is vigorously shaken and stored dark and cool, e. g. in the liquid scintillation counter, for three hours to avoid interfering chemical luminescence effects and to reach the radioactive equilibrium of Rn-222 and its short-lived progenies. Afterwards, the calibration source is directly measured for a duration between 600 s and 1800 s.

Note:

When evaluating the measurement of the calibration source, no decay correction is to be applied. The activities of Rn-222, which decays in the organic phase after shaking, and Rn-222, which is reproduced from Ra-226 in the aqueous phase, are identical. Since the reproduced Rn-222 diffuses very quickly and almost quantitatively into the organic phase even without shaking, the period between shaking and the beginning of the measurement as well as the measurement duration must be disregarded when measuring the calibration sources.

An example of a calibration spectrum is shown in Figure 3.

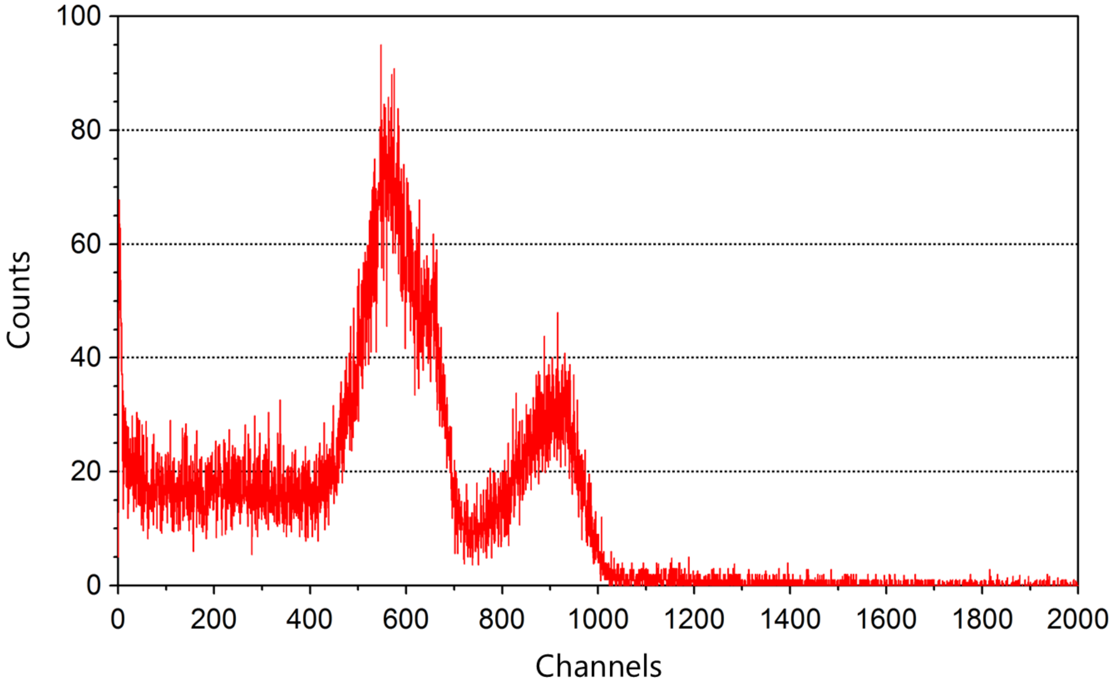


Fig. 3: Example of a calibration spectrum; for preparing the calibration source, the scintillation cocktail MaxiLight$^{\mathrm{TM}}$ was used.

The calibration factor φ_A is calculated according to Equation (1):

$$\varphi_A = \frac{c_\mathrm{s} \cdot V_\mathrm{s}}{R_\mathrm{s} - R_0} \tag{1}$$

Herein are:

c_s	activity concentration of the Ra-226 standard solution, in Bq·l^{-1};
V_s	volume of the standard solution used, in l;
R_s	gross count rate of the standard solution, in s^{-1};
R_0	Background count rate, in s^{-1}.

In order to estimate the uncertainty of the calibration factor, it is recommended to prepare and measure at least three calibration sources of different Ra-226 activities.

Note:
Since the measurement window also covers the short-lived progenies, the calibration factor φ based on Rn-222 is less than one, which corresponds to a detection efficiency ε higher than one.

4.3 Measurement

The counting source is prepared according to Section 3.2. Prior to the measurement, the counting source is vigorously shaken and stored dark and cool, e. g. in the liquid scintillation counter, for three hours to avoid interfering chemical luminescence effects and to reach the radioactive equilibrium of Rn-222 and its short-lived progenies. Afterwards, the counting source is directly measured for a duration between 600 s and 1800 s. Because of the half-life of Rn-222 of approximately 3,8 days, the measurement should be started within one day.

Note:
If a longer storage of the counting sources cannot be avoided, they should always be stored upside down, dark and cool.

5 Calculation of the results

5.1 Output quantity

The Rn-222 activity concentration c at the time of sampling shall be calculated according to Equation (2):

$$c = \varphi \cdot \left(R_\mathrm{g} - R_0\right) = \varphi \cdot R_\mathrm{n} \qquad (2)$$

with

$$\varphi = \frac{\varphi_A}{V} \cdot \mathrm{e}^{\frac{-\ln 2}{t_{\mathrm{Rn-222}}} \cdot t_A} = \frac{\varphi_A}{V} \cdot f_1 \qquad (3)$$

Herein are:

R_g	gross count rate of the counting source, in s^{-1};
R_n	net count rate of the counting source, in s^{-1};

f_1	decay correction factor for Rn-222;
t_A	time period between sampling and half of the measurement duration, in s;
$t_\text{Rn-222}$	half-life of Rn-222, in s;
V	sample volume, in l;
φ	procedural calibration factor, in Bq·s·l^{-1};
φ_A	activity-related calibration factor, in Bq·s.

5.2 Standard uncertainty of the output quantity

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 combined relative standard uncertainty of the Rn-222 activity concentration is calculated according to Equation (4):

$$u_\mathrm{rel}(c) = \frac{u(c)}{c} = \sqrt{u_\mathrm{rel}^2(R_\mathrm{n}) + u_\mathrm{rel}^2(\varphi)} \tag{4}$$

The relative statistical uncertainty contribution of the net count rate R_n or the type A uncertainty is given by the following Equation (5):

$$u_\mathrm{rel}(R_n) = \frac{u(R_\mathrm{n})}{R_\mathrm{n}} = \frac{1}{R_\mathrm{n}} \cdot \sqrt{\frac{R_\mathrm{g}}{t_\mathrm{m}} + \frac{R_0}{t_0}} \tag{5}$$

The relative standard uncertainty of the procedural calibration factor $u_\mathrm{rel}(\varphi)$ or type B uncertainty is determined by Equation (6).

$$u_\mathrm{rel}(\varphi) = \frac{u(\varphi)}{\varphi} = \sqrt{u_\mathrm{rel}^2(\varphi_A) + u_\mathrm{rel}^2(V)} \tag{6}$$

In the Equations (4) to (6) are:

$u(x)$	relative standard uncertainty of the measurand under consideration x;
$u_\mathrm{rel}(x)$	standard uncertainty of the measurand under consideration x;
t_m	duration of measurement of the counting source, in s;
t_0	duration of measurement of the background measurement, in s.

The relative standard uncertainty of the activity-related calibration factor $u_\mathrm{rel}(\varphi_A)$ is 15 % and takes into account uncertainties regarding the activity of the calibration standard, the representativeness of the sample and the extraction of radon into the scintillation cocktail. The relative standard uncertainty of the sample volume $u_{\mathrm{rel}}(V)$ corresponds to the uncertainty when taking the sample volume with a syringe or pipette and is about 3 %. The relative standard uncertainty of the decay correction factor f_1 is neglected.

6 Characteristic limits of the procedure

The calculation of the characteristic limits follows the standard series ISO 11929 [4]. For further considerations, it is referred to the General Chapters CHAGR-ISO-01 and CHAGR-ISO-02 of this Procedures' Manual [5, 6].

6.1 Decision threshold

The decision threshold of Rn-222 activity concentration c^* is calculated according to Equation (7)

$$c^* = k_{1-\alpha} \cdot \varphi \cdot \sqrt{R_0 \cdot \left(\frac{1}{t_{\mathrm{m}}} + \frac{1}{t_0}\right)} \tag{7}$$

where $k_{1\text{-}\alpha}$ is the quantile of the standard normal distribution for a type I error.

6.2 Detection limit

The limit of detection of Rn-222 activity concentration $c^{\#}$ is determined by Equation (8)

$$c^{\#} = c^* + k_{1-\beta} \cdot \sqrt{[c^{\#} \cdot u_{\mathrm{rel}}(\varphi)]^2 + \varphi^2 \cdot \left[\frac{c^{\#}}{t_{\mathrm{m}} \cdot \varphi} + R_0 \cdot \left(\frac{1}{t_{\mathrm{m}}} + \frac{1}{t_0}\right)\right]} \tag{8}$$

The iteratively solvable Equation (8) is converted into the explicit Equation (9)

$$c^{\#} = \frac{c^* \cdot \psi}{\theta} \cdot \left[1 + \sqrt{1 - \frac{\theta}{\psi^2} \cdot \left(1 - \frac{k_{1-\beta}^2}{k_{1-\alpha}^2}\right)}\right] \tag{9}$$

with the auxiliary quantities determined by the Equations (10) and (11)

$$\theta = 1 - k_{1-\beta}^2 \cdot u_{\mathrm{rel}}^2(\varphi) \tag{10}$$

$$\psi = 1 + \frac{k_{1-\beta}^2}{2 \cdot c^*} \cdot \frac{\varphi}{t_{\mathrm{m}}} \tag{11}$$

where $k_{1\text{-}\beta}$ is the quantile of the standard normal distribution for a type II error.

6.3 Limits of the coverage interval

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

7 Worked examples

The evaluation can be carried out either manually (see Section 7.1) or software supported by Excel® or by the software UncertRadio (see Section 7.2). An Excel® spreadsheet as well as a project file for the software UncertRadio are available on the website of this Procedures' Manual.

The following numerical values are used for the calculation example:

R_g = 0,40 s^{-1};
t_m = 1800 s;
R_0 = 0,30 s^{-1};
t_0 = 6000 s;
t_A = $108{,}0 \cdot 10^3$ s;
$t_\mathrm{Rn\text{-}222}$ = $330{,}32 \cdot 10^3$ s;
V = 0,010 l;
$u_\mathrm{rel}(V)$ = 0,03;
φ_A = 0,30 Bq·s;
$u_\mathrm{rel}(\varphi_A)$ = 0,15.

7.1 Manual evaluation

In the manual evaluation, the interim results and the result are given rounded with four significant digits.

The procedural calibration factor shall be calculated with Equation (3):

$$\varphi = \frac{0{,}30\ \mathrm{Bq} \cdot \mathrm{s}}{0{,}010\ \mathrm{l}} \cdot \mathrm{e}^{\frac{-\ln 2}{330{,}32 \cdot 10^3\ \mathrm{s}} \cdot 108{,}0 \cdot 10^3\ \mathrm{s}} \approx 37{,}63\ \mathrm{Bq} \cdot \mathrm{s} \cdot \mathrm{l}^{-1}$$

Thus, the activity concentration of Rn-222 is obtained with Equation (2):

$$c \approx 37{,}63 \mathrm{Bq} \cdot \mathrm{s} \cdot \mathrm{l}^{-1} \cdot (0{,}40\ \mathrm{s}^{-1} - 0{,}3\ \mathrm{s}^{-1}) \approx 3{,}763\ \mathrm{Bq} \cdot \mathrm{l}^{-1}$$

For the calculation of the standard uncertainty, the values of the relative standard uncertainties of the net count rate and the procedural calibration factor are first calculated with the Equations (5) and (6):

$$u_\mathrm{rel}(R_\mathrm{n}) = \frac{1}{0{,}10\ \mathrm{s}^{-1}} \cdot \sqrt{\frac{0{,}40\ \mathrm{s}^{-1}}{1800\ \mathrm{s}} + \frac{0{,}30\ \mathrm{s}^{-1}}{6000\ \mathrm{s}}} = 0{,}1650$$

$$u_\mathrm{rel}(\varphi) = \sqrt{0{,}15^2 + 0{,}03^2} = 0{,}1530$$

With these values and after conversion of Equation (4), the standard uncertainty of the activity concentration is determined as follows:

$$u(c) \approx 3{,}763\ \mathrm{Bq} \cdot \mathrm{l}^{-1} \cdot \sqrt{0{,}165^2 + 0{,}153^2} \approx 0{,}8467\ \mathrm{Bq} \cdot \mathrm{l}^{-1}$$

The Rn-222 activity concentration in the sample is therefore

$$c = (3{,}763 \pm 0{,}847)\ \mathrm{Bq \cdot l^{-1}}$$

With the value $k_{1\text{-}\alpha}$ of 3, the following detection limit is obtained after Equation (7):

$$c^{*} \approx 3 \cdot 37{,}63\ \mathrm{Bq \cdot s \cdot l^{-1}} \cdot \sqrt{0{,}30\ \mathrm{s^{-1}} \cdot \left(\frac{1}{1800\ \mathrm{s}} + \frac{1}{6000\ \mathrm{s}}\right)} \approx 1{,}662\ \mathrm{Bq \cdot l^{-1}}$$

The values of the auxiliary quantities θ and ψ are determined with the Equations (10) and (11) using a value of 1,645 for $k_{1\text{-}\beta}$.

$$\theta \approx 1 - 1{,}645^2 \cdot 0{,}153^2 \approx 0{,}9367$$

$$\psi \approx 1 + \frac{1{,}645^2}{2 \cdot 1{,}662\ \mathrm{Bq \cdot l^{-1}}} \cdot \frac{37{,}63\ \mathrm{Bq \cdot s \cdot l^{-1}}}{1800\ \mathrm{s}} \approx 1{,}017$$

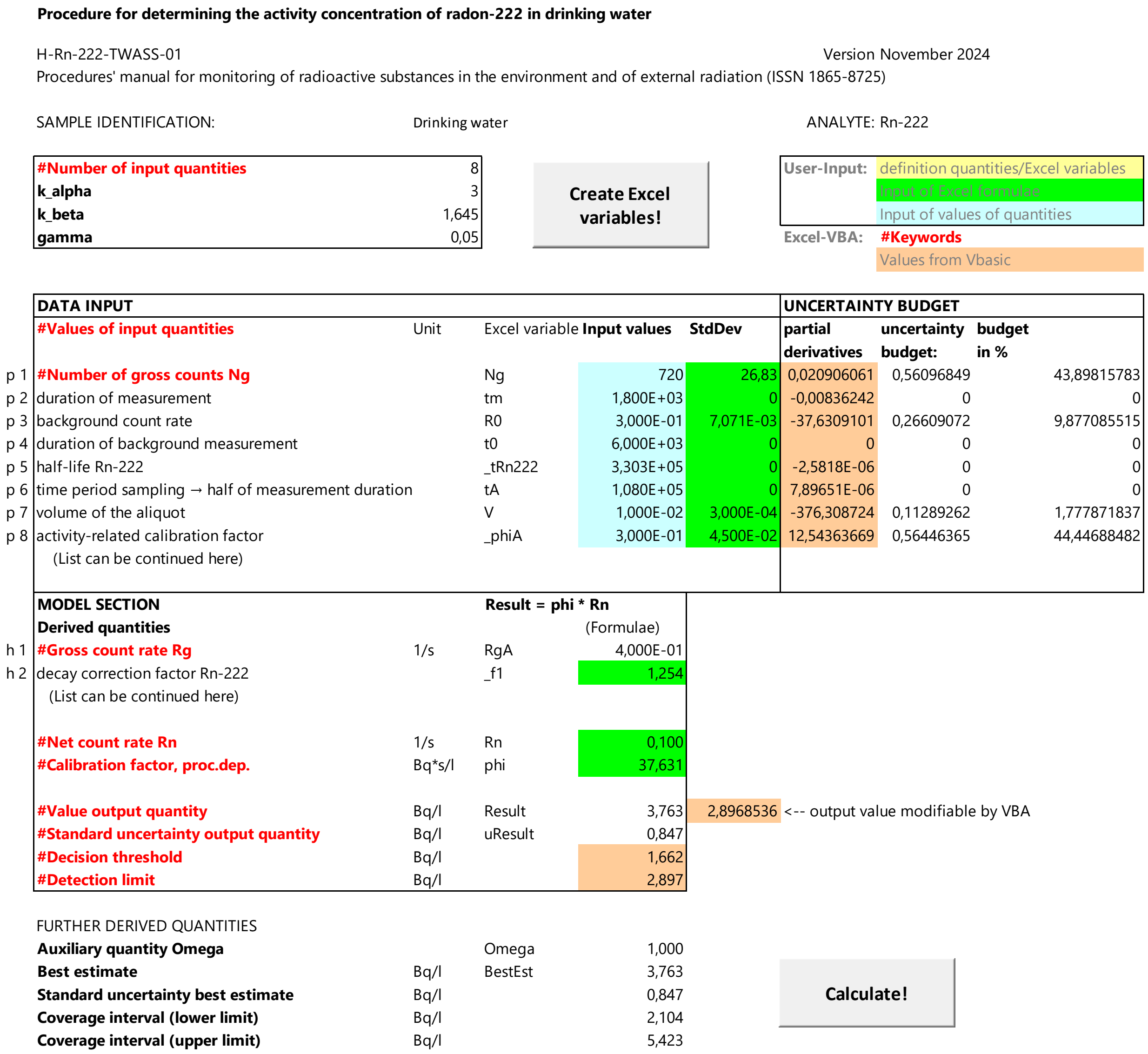
The detection limit is calculated with Equation (9):

$$c^{\#} \approx \frac{1{,}662\ \mathrm{Bq \cdot l^{-1}} \cdot 1{,}017}{0{,}9367} \cdot \left[1 + \sqrt{1 - \frac{0{,}9367}{1{,}017^2} \cdot \left(1 - \frac{1{,}645^2}{3^2}\right)}\right] \approx 2{,}897\ \mathrm{Bq \cdot l^{-1}}$$

Note:
With a sample volume of 0,010 l, detection limits of approximately 3 $\mathrm{Bq \cdot l^{-1}}$ are realistic. By extending the measurement duration, the detection limit can be further reduced.

7.2 Software supported evaluation

7.2.1 View of the Excel® spreadsheet



Procedure for determining the activity concentration of radon-222 in drinking water

H-Rn-222-TWASS-01 Version November 2024
Procedures' manual for monitoring of radioactive substances in the environment and of external radiation (ISSN 1865-8725)

SAMPLE IDENTIFICATION: Drinking water ANALYTE: Rn-222

#Number of input quantities	8
k_alpha	3
k_beta	1,645
gamma	0,05

User-Input:	definition quantities/Excel variables
	Input of Excel formulae
	Input of values of quantities
Excel-VBA:	**#Keywords**
	Values from Vbasic

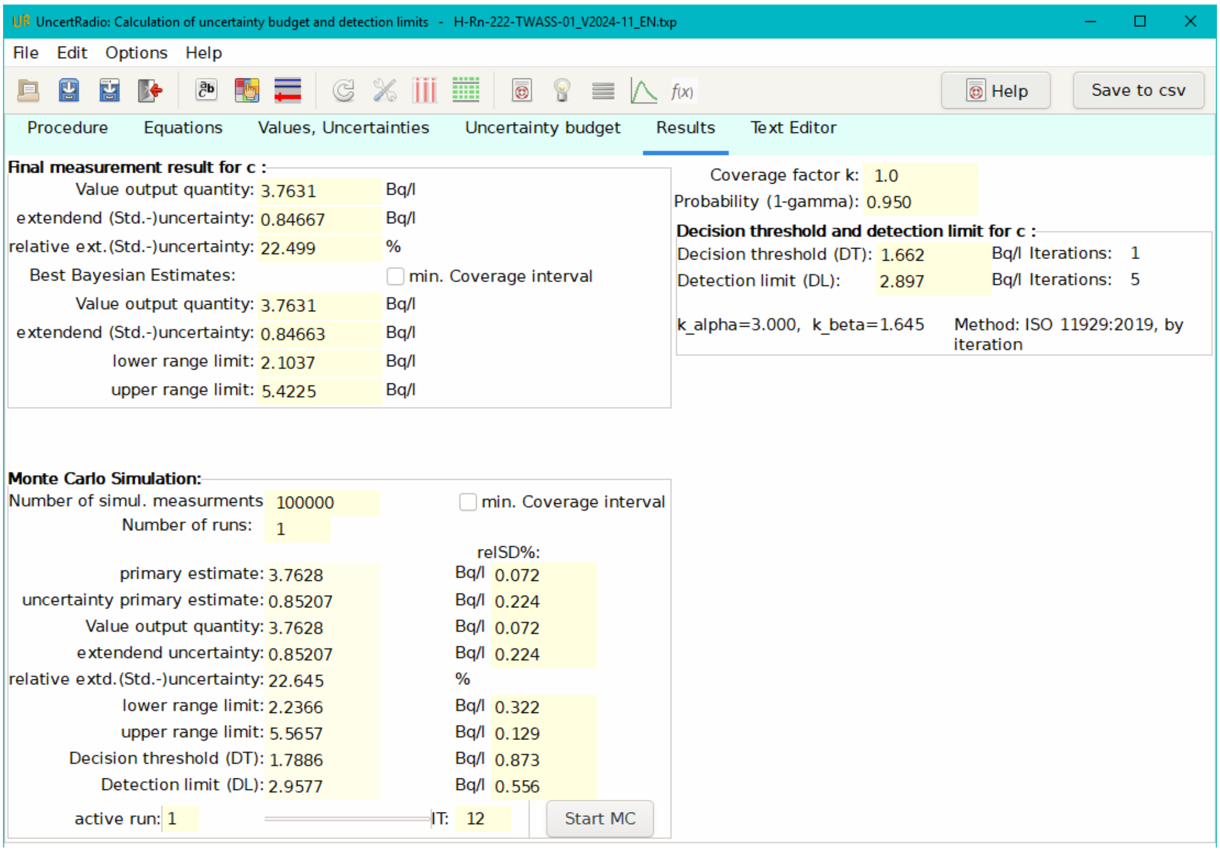
	DATA INPUT	Unit	Excel variable	Input values	StdDev	UNCERTAINTY BUDGET partial derivatives	uncertainty budget:	budget in %
	#Values of input quantities							
p 1	**#Number of gross counts Ng**		Ng	720	26,83	0,020906061	0,56096849	43,89815783
p 2	duration of measurement		tm	1,800E+03	0	-0,00836242	0	0
p 3	background count rate		R0	3,000E-01	7,071E-03	-37,6309101	0,26609072	9,877085515
p 4	duration of background measurement		t0	6,000E+03	0	0	0	0
p 5	half-life Rn-222		_tRn222	3,303E+05	0	-2,5818E-06	0	0
p 6	time period sampling → half of measurement duration		tA	1,080E+05	0	7,89651E-06	0	0
p 7	volume of the aliquot		V	1,000E-02	3,000E-04	-376,308724	0,11289262	1,777871837
p 8	activity-related calibration factor		_phiA	3,000E-01	4,500E-02	12,54363669	0,56446365	44,44688482
	(List can be continued here)							

	MODEL SECTION		Result = phi * Rn		
	Derived quantities			(Formulae)	
h 1	**#Gross count rate Rg**	1/s	RgA	4,000E-01	
h 2	decay correction factor Rn-222		_f1	1,254	
	(List can be continued here)				
	#Net count rate Rn	1/s	Rn	0,100	
	#Calibration factor, proc.dep.	Bq*s/l	phi	37,631	
	#Value output quantity	Bq/l	Result	3,763	2,8968536 <-- output value modifiable by VBA
	#Standard uncertainty output quantity	Bq/l	uResult	0,847	
	#Decision threshold	Bq/l		1,662	
	#Detection limit	Bq/l		2,897	

FURTHER DERIVED QUANTITIES			
Auxiliary quantity Omega		Omega	1,000
Best estimate	Bq/l	BestEst	3,763
Standard uncertainty best estimate	Bq/l		0,847
Coverage interval (lower limit)	Bq/l		2,104
Coverage interval (upper limit)	Bq/l		5,423

The corresponding Excel® spreadsheet is available on the website of this Procedures Manual.

7.2.2 View of the UncertRadio result page



Final measurement result for c :
Value output quantity: 3.7631 Bq/l
extendend (Std.-)uncertainty: 0.84667 Bq/l
relative ext.(Std.-)uncertainty: 22.499 %
Best Bayesian Estimates: ☐ min. Coverage interval
Value output quantity: 3.7631 Bq/l
extendend (Std.-)uncertainty: 0.84663 Bq/l
lower range limit: 2.1037 Bq/l
upper range limit: 5.4225 Bq/l

Coverage factor k: 1.0
Probability (1-gamma): 0.950

Decision threshold and detection limit for c :
Decision threshold (DT): 1.662 Bq/l Iterations: 1
Detection limit (DL): 2.897 Bq/l Iterations: 5
k_alpha=3.000, k_beta=1.645 Method: ISO 11929:2019, by iteration

Monte Carlo Simulation:
Number of simul. measurments 100000 ☐ min. Coverage interval
Number of runs: 1

			relSD%:
primary estimate:	3.7628	Bq/l	0.072
uncertainty primary estimate:	0.85207	Bq/l	0.224
Value output quantity:	3.7628	Bq/l	0.072
extendend uncertainty:	0.85207	Bq/l	0.224
relative extd.(Std.-)uncertainty:	22.645	%	
lower range limit:	2.2366	Bq/l	0.322
upper range limit:	5.5657	Bq/l	0.129
Decision threshold (DT):	1.7886	Bq/l	0.873
Detection limit (DL):	2.9577	Bq/l	0.556

active run: 1 IT: 12

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 shall be of analytically pure quality.

- Ra-226 standard solution;
- Scintillation cocktail: e. g. Ultima Gold F^{TM}, MaxiLight^{TM}, Opti-Fluor^{TM}, Opti-Fluor O^{TM};

  Note:
  In the literature, a distribution coefficient of 48 is given for the scintillation cocktail Opti-Fluor^{TM} [7].
- Water, de-emanated: from deionised water:

  discharge of nitrogen or radon-free air through deionized water; the duration depends, inter alia, on the ratio between the volume of air passed through and the volume of water.

8.2 Equipment

The following equipment is used for the procedure:

- of a radiochemical laboratory;
- disposable syringes, nominal volume 20 ml;
- silicone hose;
- counting vials made of K-40-poor glass or high-density polytetrafluoroethylene-coated polyethylene (PTFE-HDPE), nominal volume 20 ml;
- liquid scintillation counter (LSC).

Annex A
Leak tightness of sampling containers

A.1 Introduction

To identify suitable container for Rn-222 sampling and storage, leak tests were carried out with PET-veral bottles and glass-syrup bottles covered with aluminium-coated screw caps or conical PE screw caps.

A.2 Method and results

For this purpose, water samples with a Rn-222 activity concentration of (673 ± 75) $Bq \cdot l^{-1}$ were stored in the respective sampling containers for up to 13 days at room temperature. During this storage period, five triple determinations were carried out. The results are shown in Figure A1 as averages with the simple standard measurement uncertainty

In addition, a postal delivery scenario was simulated at the selected sampler, in which these were shaken over three working days, i. e. a total of about 13 hours, on a shaking machine. The subsequent sampling and measurement were carried out as in the Sections 2, 3.2 and 4.3 described.

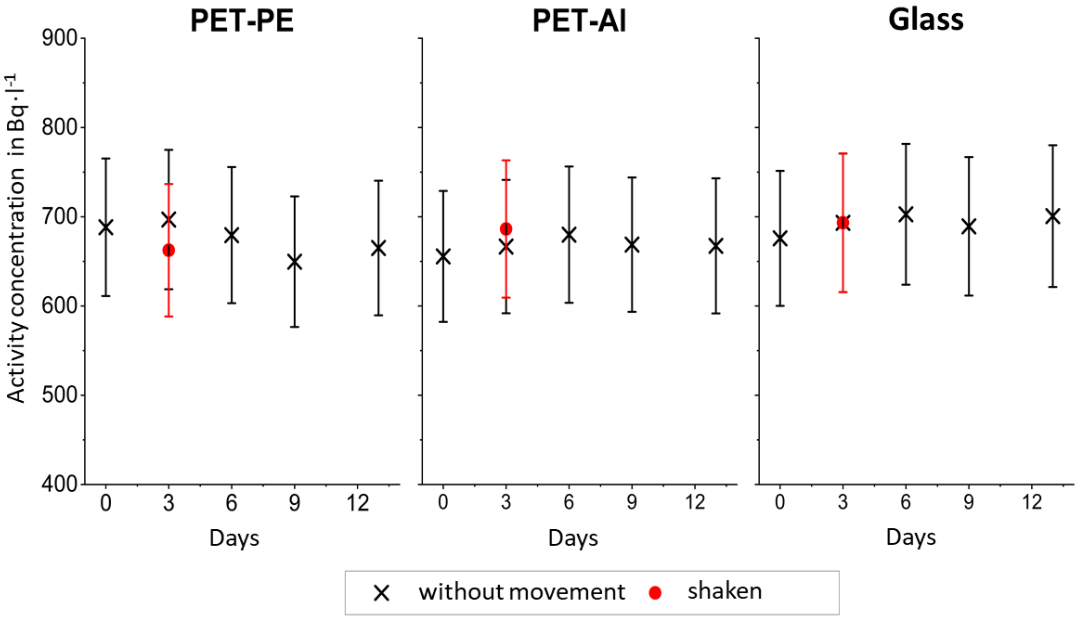


Fig. A1: Measurement results of the Rn-222 activity concentration depending on the state of the sample, the sampling container and the screw cap

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