Procedure for determining the activity concentration of tritium in seawater by liquid scintillation counting

D-H-3-MWASS-01

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Procedures Manual for monitoring of radioactive substances in the environment and of external radiation
(Messanleitungen für die „Überwachung radioaktiver Stoffe in der Umwelt und externer Strahlung")

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1 Scope

The procedure outlined in the following is suitable for determining the activity concentration of tritium (H-3) in seawater according to both the IMIS-routine and intensive measurement programme in accordance with the Radiation Protection Act (StrlSchG) [1].

The procedure is applicable for activity concentrations of H-3 of more than 100 Bq·m^{-3} in seawater after electrolytic enrichment in the routine operation mode. In the intensive operation mode, the procedure is applied without electrolytic enrichment [2, 3, 4, 5].

2 Sampling

A detailed description of sampling is given in Procedure D-Cs-MWASS-01.

A 1-litre glass bottle and its screw cap with seal are pre-rinsed three times with seawater. One litre of seawater is filled into the rinsed glass bottle without prior filtering or acidification to overflowing and the glass bottle is sealed airtight with the screw cap. In this way, the contact with the ambient air is limited so that the seawater sample can be stored.

3 Analysis

3.1 Principle of the procedure

The procedure is designed for sample volumes of 0,5 l.

To separate off the salt matrix, the seawater sample is distilled under vacuum without further pre-treatment. For expected activity concentrations above 2 Bq·l^{-1}, an aliquot is taken from the resulting distillate and is measured directly using a liquid scintillation spectrometer (LSC) after adding a scintillation cocktail.

For expected lower H-3 activity concentrations, the tritium contained in the distillate must be enriched electrolytically. For this purpose, the distillate is decomposed in an electrolysis cell to oxygen and hydrogen, wherein the heavy hydrogen isotopes deuterium and tritium accumulate at the anode. The resulting solution is again distilled under vacuum to remove impurities which are still present. An aliquot is then taken out of the distillate and, after adding a scintillation cocktail, is measured in a liquid scintillation spectrometer.

3.2   Sample preparation

A little more than 0,5 litre of the seawater sample is distilled under vacuum at a pressure of about $3 \cdot 10^3$ Pa and, if necessary, is subjected to electrolytic enrichment afterwards.

3.2.1   Distillation

The distillation apparatus used in the federal coordinating office, with which up to four samples can be distilled simultaneously, is shown in Figures 1 and 2.
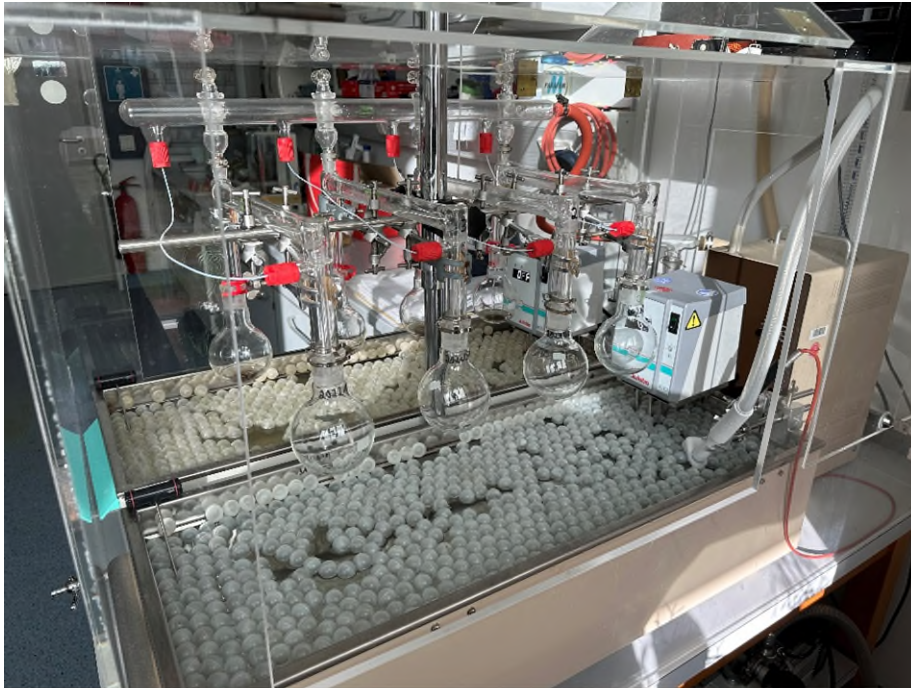

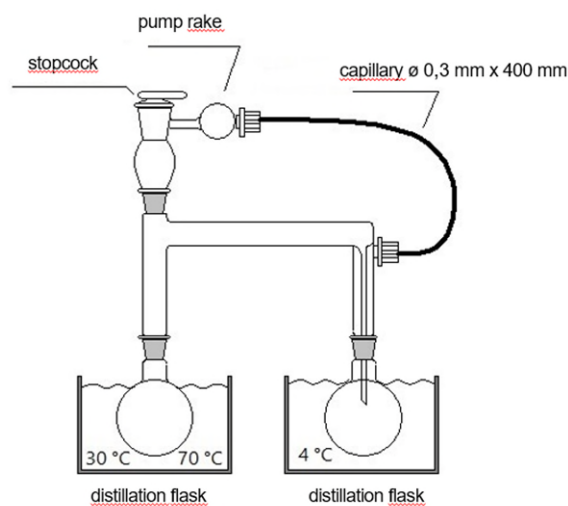**Fig. 1:**   Distillation apparatus with four distillation units

Fig. 2:   Sketch of the distillation apparatus

3.2.1.1 About 500 ml of the seawater sample are transferred into a tared 500 ml round-bottom flask (distillation flask) and the weight of both together is determined. The round-bottom flask is attached to a vacuum distillation apparatus.

Note:

Using commercially available 500 ml round-bottom flasks, parts of the seawater sample can overflow into the distillation bridge, thus increasing the conductivity of the distillate. Therefore, a special design with a wider neck, see Figure 3, is recommended.

Fig. 3: Special design of a round-bottom flask (right) compared with a commercially available round bottom flask (left)

3.2.1.2 A 500 ml round-bottom flask (distillate flask), also tared, is used to collect the distillate.

3.2.1.3 The distillation flask is heated to about 30 °C by means of a heat bath and the distillate flask is cooled to 4 °C by means of a cold bath.

Note:

Cold baths of this size are generally not commercially available. The cold bath used in the federal coordinating office was constructed from an ordinary water bath with an electric heating and cooling element. To prevent freezing of the water in the cold bath in case of failure of the heating element, ethylene glycol is added to the water.

3.2.1.4 The sample is then distilled to dryness at a pressure of about $3 \cdot 10^3$ Pa. Depending on the volume used, the distillation process takes between 15 hours and 20 hours.

Note:

In practice, other bath temperatures and distillation times may be required. If dryness is not reached after 24 hours, the distillation is continued, wherein the temperature of the heat bath can be increased to up to 50 °C.

3.2.1.5 The mass of the distillate flask and the distillate is determined by weighing the cooled and dried round-bottom flasks.

3.2.1.6 For expected activity concentrations above 2 Bq·l⁻¹, the distillate is directly prepared for measurement in accordance with Section 3.4. Otherwise, an electrolytic enrichment according to Section 3.2.2 is carried out.

3.2.2   Electrolytical enrichment

The enrichment apparatus used in the federal coordinating office is shown in Figure 4 and consists of the following components:

— up to nine 500 ml electrolysis cells, each consisting of two parts (see Figure 5):

- an inner part, the cathode, with a device for discharging the resulting explosive gas and the connection to the power control unit. As cathode material a structural steel with phosphated surface is usually used.

- an outer part, the anode, into which the electrolyte is filled. The anode material is usually made of stainless steel.

— a freezer;

— a control unit with alarm and monitoring function;

— an exhaust gas discharge.

The extent of enrichment depends on the type, concentration and temperature of the electrolyte, on the electrode material and its surface condition as well as on the current density at the cathode surface.

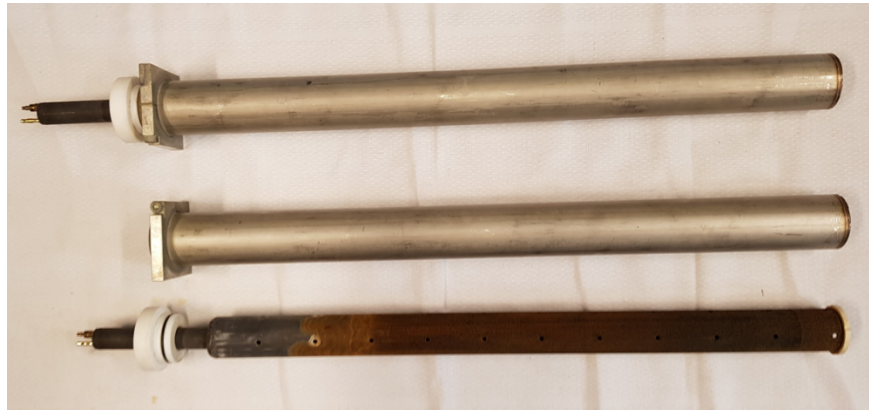**Fig. 4:**   Enrichment apparatus with nine electrolysis cells

Fig. 5: Electrolysis cell (top) consisting of anode/outer part (center) and cathode/inner part (bottom)

3.2.2.1 The tare of the anode and cathode are determined.

3.2.2.2 About 1,3 g of sodium peroxide are weighed into the anode of the electrolysis cell. **The exact amount must be noted.**

3.2.2.3 Then, about 500 g of the distillate are weighed into the anode. **The exact amount must be noted.**

3.2.2.4 The sodium peroxide is dissolved in the distillate by swirling the anode.

3.2.2.5 Thereafter, the anode and the cathode of the electrolysis cell are brought together and the total mass of the electrolysis cell is determined. The electrolysis cell is placed in a freezer and the freezer is switched on.

3.2.2.6 An exhaust gas hose (blue hoses in Figure 4) is attached to the cathode to dissipate the resulting oxyhydrogen, which is connected to the exhaust of the enrichment equipment.

3.2.2.7 Anode and cathode are connected to the power supply.

3.2.2.8 When a temperature of 5 °C is reached in the freezer, the electrolysis is started, the current being increased to 8 A within two to three minutes.

3.2.2.9 If a current of 8 A is reached, the individual voltages of the electrolysis cells are checked after about 15 minutes. The values of the cell voltages are generally 2,4 V. If increased individual voltages occur, the temperature of the affected cell must additionally be checked with an infrared thermometer. If the individual voltage of a cell exceeds 3,5 V, the cell must be taken out of operation.

3.2.2.10 After about 30 minutes, the current is increased to 10 A. At this current, about 80 ml of water are decomposed per day. The corresponding voltage is about 2,6 V and decreases during the enrichment to approx. 2,2 V.

3.2.2.11 The individual voltages of the electrolysis cells are checked at least once a working day.

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Note:

If the voltages of individual cells fluctuate in the Steps 3.2.2.9 and 3.2.2.11 for longer than 2 minutes and more than 0,5 V of the normal value, the cell has to be taken out of operation due to possible corrosion.

3.2.2.12   The electrolytic enrichment is terminated after about 6 days when the volume of the weighed distillate has decomposed to a residual volume of about 20 ml.

3.2.2.13   The mass of the residual volume is determined by weighing.

3.2.2.14   The electrolyte is transferred to a tared 250 ml round-bottom flask for cleaning any remaining impurities, noting the weighing into the round-bottom flask. The distillate flask is also a 250 ml round-bottom flask.

3.2.2.15   Then, a new distillation is carried out in accordance with Section 3.2.1 heating the heat bath to approx. 70 °C instead of approx. 30 °C.

Note:

Due to the higher temperature and lower volume, this distillation takes between two hours and four hours.

3.2.2.16   The resulting distillate is used for preparing the counting source according to Section 3.4.

3.3   Radiochemical separation

A radiochemical separation is not required.

3.4   Counting source preparation

An aliquot of 8 ml is taken out of the distillate obtained according to Section 3.2.1 or 3.2.2 and is pipetted into a tared 20 ml scintillation vial. The mass of the aliquot is determined by weighing. Then, 12 ml scintillation cocktail are added to the scintillation vial. The solutions are thoroughly mixed by vigorous shaking of the vial.

4   Measuring the activity

4.1   General

The counting sources and the background source are pre-cooled in the low-level liquid scintillation spectrometer for several hours in the dark for minimising luminescence effects. Afterwards, the activity of tritium is measured. For each measurement series, tritium-poor water, e. g. Atlantic water or deep well water, is used for determining the background count rate.

Note:

Pre-cooling for 12 hours is recommended.

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4.2   Calibration

4.2.1   Low-level liquid scintillation spectrometer

The detection efficiency of the liquid scintillation spectrometer is determined with a calibration source whose activity is traceable to a national primary standard.

If the calibration source is produced by oneself, this is done in accordance with Section 3.4. In this case, an activity of approx. 10 Bq of tritium in the form of tritiated water (HTO) is usually presented and filled up to a total volume of 8 ml with tritium-poor water.

4.2.2   Determination of the yield of the electrolysis cells

For each electrolysis cell, the tritium yield of the electrolytic enrichment $\eta_{A,i}$ must be determined and checked regularly.

For this purpose, a calibration solution consisting of tritium-poor, distilled water and a traceable activity standard is prepared, which contains an activity concentration of about 10 Bq·l^{-1}. The electrolytic enrichment is carried out according to Section 3.2.2 with a volume of 500 ml of calibration solution per electrolytic cell.

The tritium yield $\eta_{A,i}$ for each electrolytic cell i is calculated after measurement in the liquid scintillation spectrometer according to Equation (1):

$$\eta_{A,i} = \frac{a_{n,i} \cdot m_{n,i}}{a_{v,i} \cdot m_{v,i}} \tag{1}$$

Herein are:

$\eta_{A,i}$ electrolysis cell-specific tritium yield of the electrolytic enrichment;

$a_{n,i}$ specific H-3 activity after electrolysis, in Bq·g^{-1};

$a_{v,i}$ specific H-3 activity before electrolysis, in Bq·g^{-1};

$m_{n,i}$ mass of the solution remaining in the electrolysis cell i after electrolysis, in g;

$m_{v,i}$ mass of solution filled into the electrolysis cell i before electrolysis, in g.

4.3   Measurement

The counting sources obtained according to Section 3.4 are measured by five directly sequential measurements for 200 minutes in each case in a liquid scintillation spectrometer. The count rates obtained are then summed up to determine the activity of the tritium (see Section 5.1).

The measurement of the counting sources for the determination of the background, the detection efficiency and the tritium yield of the electrolytic enrichment takes place in an analogous manner.

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5 Calculation of the results

5.1 Equations

5.1.1 Output quantity

If electrolytic enrichment was carried out, the H-3 activity concentration c at the time of sampling is calculated according to Equation (2).

$$c = \frac{m_{\mathrm{n}} \cdot \rho \cdot f_{\mathrm{A}}}{\varepsilon \cdot \eta_{\mathrm{A},i} \cdot m_{\mathrm{v}} \cdot m_{\mathrm{M}}} \cdot R_{\mathrm{n}} = \frac{m_{\mathrm{n}} \cdot \rho \cdot \mathrm{e}^{\lambda \cdot t_{\mathrm{A}}}}{\varepsilon \cdot \eta_{\mathrm{A},i} \cdot m_{\mathrm{v}} \cdot m_{\mathrm{M}}} \cdot R_{\mathrm{n}} = \varphi \cdot R_{\mathrm{n}} \tag{2}$$

If the enrichment step is omitted, Equation (3) is used to calculate the activity concentration.

$$c = \frac{\rho \cdot f_{\mathrm{A}}}{\varepsilon \cdot m_{\mathrm{M}}} \cdot R_{\mathrm{n}} = \frac{\rho \cdot \mathrm{e}^{\lambda \cdot t_{\mathrm{A}}}}{\varepsilon \cdot m_{\mathrm{M}}} \cdot R_{\mathrm{n}} = \varphi \cdot R_{\mathrm{n}} \tag{3}$$

In the Equations (2) and (3) are:

c activity concentration, in Bq·m^{-3};

f_{A} correction factor for the decay;

m_{M} mass of distillate in the scintillation vial, in g;

m_{n} mass of solution remaining in the electrolysis cell after electrolysis, in g;

m_{v} mass of the solution filled into the electrolysis cell before electrolysis, in g;

R_{n} net count rate of the counting source, in s^{-1}:

        with $R_{\mathrm{n}} = R_{\mathrm{g}} - R_0$

t_{A} time period between sampling and beginning of measurement, in s;

ε detection efficiency, in Bq^{-1}·s^{-1};

λ decay constant of tritium, in s^{-1};

ρ density of the seawater, in kg·m^{-3};

φ procedural calibration factor, in Bq·s·m^{-3}.

For determining the total gross count rate R_{g} from the individual gross count rates $R_{\mathrm{g},j}$, Equation (4) is used:

$$R_{\mathrm{g}} = \frac{\sum_1^j R_{\mathrm{g},j} \cdot t_{\mathrm{m},j}}{\sum_1^j t_{\mathrm{m},j}} = \frac{\sum_1^j R_{\mathrm{g},j} \cdot t_{\mathrm{m},j}}{t_{\mathrm{tot}}} \tag{4}$$

Herein are:

$R_{\mathrm{g},j}$ gross count rate of the single measurement j of the counting source, in s^{-1};

$t_{\mathrm{m},j}$ duration of the single measurement j, in s;

t_{tot} sum of the durations of single measurements in s.

5.1.2   Standard uncertainty of the output quantity

The standard uncertainty of the activity concentration $u(c)$ is calculated according to Equation (5). If no electrolysis is carried out, the relative standard uncertainties for the tritium yield and for the masses before and after the electrolysis are eliminated:

$$u(c) = c \cdot \sqrt{\frac{1}{\left(R_{\mathrm{g}} - R_0\right)^2} \cdot \left(\frac{R_{\mathrm{g}}}{t_{\mathrm{tot}}} + \frac{R_0}{t_0}\right) + u_{\mathrm{rel}}^2(\varphi)} \tag{5}$$

with

$$u_{\mathrm{rel}}^2(\varphi) = u_{\mathrm{rel}}^2(f_{\mathrm{A}}) + u_{\mathrm{rel}}^2(m_{\mathrm{n}}) + u_{\mathrm{rel}}^2(m_{\mathrm{v}}) + u_{\mathrm{rel}}^2(m_{\mathrm{M}}) + u_{\mathrm{rel}}^2(\varepsilon) + u_{\mathrm{rel}}^2\left(\eta_{\mathrm{A},i}\right) + u_{\mathrm{rel}}^2(\rho)$$

Herein are:

$u(c)$ standard uncertainty of the activity concentration, in Bq·m⁻³;

t_0 sum of the duration of the single background measurements $t_{0,j}$, in s;

$u_{\mathrm{rel}}(f_{\mathrm{A}})$ relative standard uncertainty of the correction factor for the decay;

$u_{\mathrm{rel}}(m_{\mathrm{M}})$ relative standard uncertainty of the mass of distillate in the scintillation vial;

$u_{\mathrm{rel}}(m_{\mathrm{n}})$ relative standard uncertainty of the mass of solution remaining in the electrolysis cell after electrolysis;

$u_{\mathrm{rel}}(m_{\mathrm{v}})$ relative standard uncertainty of the mass of the solution filled into the electrolysis cell before electrolysis;

$u_{\mathrm{rel}}(\varepsilon)$ relative standard uncertainty of the detection efficiency;

$u_{\mathrm{rel}}(\eta_{\mathrm{A},i})$ relative standard uncertainty of the tritium yield of the electrolysis cell i;

$u_{\mathrm{rel}}(\rho)$ relative standard uncertainty of the density of seawater;

$u_{\mathrm{rel}}(\varphi)$ relative standard uncertainty of the procedural calibration factor.

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 worked example shows the calculation of the activity concentration of H-3 after electrolytic enrichment, based on the following values. The relative standard uncertainty of the decay correction factor $u_{\mathrm{rel}}(f_{\mathrm{A}})$ is neglected.

$R_{g,1}$	= 0,160 s⁻¹;	$t_{m,1}$	= 200 min	= 12 000 s;

$R_{g,1}$ = 0,160 s^{-1}; $t_{m,1}$ = 200 min = 12 000 s;
$R_{g,2}$ = 0,140 s^{-1}; $t_{m,2}$ = 12 000 s;
$R_{g,3}$ = 0,180 s^{-1}; $t_{m,3}$ = 12 000 s;
$R_{g,4}$ = 0,150 s^{-1}; $t_{m,4}$ = 12 000 s;
$R_{g,5}$ = 0,170 s^{-1}; $t_{m,5}$ = 12 000 s;
R_0 = 0,020 s^{-1}; t_0 = 60 000 s;
t_A = 4,50·10^7 s; t_{tot} = 60 000 s;
m_v = 0,5 kg; $u_{rel}(m_v)$ = 0,01;
m_n = 0,021 kg; $u_{rel}(m_n)$ = 0,01;
m_M = 0,008 kg; $u_{rel}(m_M)$ = 0,01;
ε = 0,33 Bq^{-1}·s^{-1}; $u_{rel}(\varepsilon)$ = 0,05;
$\eta_{A,i}$ = 0,85; $u_{rel}(\eta_{A,i})$ = 0,02;
ρ = 1·10^3 kg·m^{-3}; $u_{rel}(\rho)$ = 0,03;
λ = 1,78·10^{-9} s^{-1}.

First, the total gross count rate R_g is determined according to Equation (4):

$$R_g = \frac{(0{,}160\ \text{s}^{-1} + 0{,}140\ \text{s}^{-1} + 0{,}180\ \text{s}^{-1} + 0{,}150\ \text{s}^{-1} + 0{,}170\ \text{s}^{-1}) \cdot 12\ 000\ \text{s}}{60\ 000\ \text{s}} =$$

$$= 0{,}1600\ \text{s}^{-1}$$

Then, the activity concentration c is calculated according to equation (2):

$$c = \frac{0{,}021\ \text{kg} \cdot 1 \cdot 10^3\ \text{kg} \cdot \text{m}^{-3} \cdot e^{1{,}78 \cdot 10^{-9}\ \text{s}^{-1} \cdot 4{,}50 \cdot 10^7\ \text{s}}}{0{,}33\ \text{Bq}^{-1} \cdot \text{s}^{-1} \cdot 0{,}85 \cdot 0{,}5\ \text{kg} \cdot 0{,}008\ \text{kg}} \cdot 0{,}140\ \text{s}^{-1} =$$

$$= 20\ 277\ \text{Bq} \cdot \text{s} \cdot \text{m}^{-3} \cdot 0{,}140\ \text{s}^{-1} = 2839\ \text{Bq} \cdot \text{m}^{-3}$$

with

$$R_n = 0{,}160\ \text{s}^{-1} - 0{,}020\ \text{s}^{-1} = 0{,}140\ \text{s}^{-1}$$

The standard uncertainty of the activity concentration $u(c)$ is calculated according to Equation (5):

$$u(c) = 2839\ \text{Bq} \cdot \text{m}^{-3} \cdot \sqrt{\frac{1}{(0{,}140\ \text{s}^{-1})^2} \cdot \frac{(0{,}160\ \text{s}^{-1} + 0{,}020\ \text{s}^{-1})}{60\ 000\ \text{s}} + 0{,}004100} =$$

$$= 185{,}1\ \text{Bq} \cdot \text{m}^{-3}$$

with

$$u_{\text{rel}}^2(\varphi) = 0{,}01^2 + 0{,}01^2 + 0{,}01^2 + 0{,}05^2 + 0{,}02^2 + 0{,}03^2 = 0{,}004100$$

The activity concentration c is thus:

$$c = (2839 \pm 185{,}1) \, \text{Bq} \cdot \text{m}^{-3}$$

5.3 Consideration of the uncertainties

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

The standard uncertainty of the analysis includes the standard uncertainties of the counting statistics, the calibration of the liquid scintillation spectrometer and the tritium yield of the electrolysis cell and the volume of the sample in the form of the product of density and mass. The standard uncertainties of the measurement duration and the decay constant are neglected.

6 Characteristic limits of the procedure

The calculation of the characteristic limits follows the standard series ISO 11929 [6]. 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) as well as a project file for the software Uncert-Radio (see Section 7.2) are available on the website of this Procedures Manual.

6.1 Equations

6.1.1 Decision threshold

The decision threshold c^* is determined using Equation (6):

$$c^* = k_{1-\alpha} \cdot \varphi \cdot \sqrt{R_0 \cdot \left(\frac{1}{t_{\text{tot}}} + \frac{1}{t_0}\right)} \tag{6}$$

Herein are:

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

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

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6.1.2 Detection limit

The detection limit $c^\#$ is calculated according to the implicit Equation (7):

$$c^\# = c^* \cdot k_{1-\beta} \cdot \sqrt{c^{\#2} \cdot u_{\text{rel}}^2(\varphi) + \varphi^2 \cdot \left(\frac{c^\#}{t_{\text{tot}} \cdot \varphi} + \frac{R_0}{t_{\text{tot}}} + \frac{R_0}{t_0} \right)} \tag{7}$$

In Equation (7) are:

$c^\#$ detection limit for the activity concentration, 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 (8):

$$c^\# = \frac{c^* \cdot \Psi}{\theta} \cdot \left[1 + \sqrt{1 - \frac{\theta}{\Psi^2} \cdot \left(1 - \frac{k_{1-\beta}^2}{k_{1-\alpha}^2} \right)} \right] \tag{8}$$

with

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

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

6.1.3 Limits of the coverage interval

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

6.2 Worked examples

For an electrolytically enriched 0,5 litre sample with the values

$k_{1-\alpha}$	=	3;	$k_{1-\beta}$	=	1,645;
φ	=	20 277 Bq·s·m^{-3};	$u_{\text{rel}}(\varphi)$	=	0,0041;

the following decision threshold c^* is obtained according to Equation (6):

$$c^* = 3 \cdot 20\,277 \,\text{Bq} \cdot \text{s} \cdot \text{m}^{-3} \cdot \sqrt{0{,}020 \,\text{s}^{-1} \cdot \left(\frac{1}{60\,000 \,\text{s}} + \frac{1}{60\,000 \,\text{s}} \right)} = 49{,}67 \,\text{Bq} \cdot \text{m}^{-3}$$

For the detection limit $c^\#$ the following value is determined according to Equation (8):

$$c^\# = \frac{49{,}67 \,\text{Bq} \cdot \text{m}^{-3} \cdot 1{,}009}{0{,}9889} \cdot \left[1 + \sqrt{1 - \frac{0{,}9889}{1{,}009^2} \cdot \left(1 - \frac{1{,}645^2}{3^2} \right)} \right] = 79{,}38 \,\text{Bq} \cdot \text{m}^{-3}$$

with

$$\theta = 1 - 1{,}645^2 \cdot 0{,}0041 = 0{,}9889$$

$$\Psi = 1 + \frac{1{,}645^2}{2 \cdot 49{,}67\ \text{Bq} \cdot \text{m}^{-3}} \cdot 20\ 277\ \text{Bq} \cdot \text{s} \cdot \text{m}^{-3} \cdot \frac{1}{60\ 000\ \text{s}} = 1{,}009$$

7 Software supported calculation

7.1 View of the Excel spreadsheet

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SAMPLE IDENTIFICATION:　　　　　Seawater

#Number of parameters p	13
k_alpha: quantile for (1-α)	3
k_beta: quantile for (1-β)	1,645
gamma: probability for conf.limits	0,05

Create variables for parameters

User-Input:	Input of values
	Definition Excel variables
	Input of Excel formulae
Excel-VBA:	#Keywords
	Values from Vbasic

Data input: | | variable names: | | | **Uncertainty budget:** | | |

	#Values of parameters p	Unit:		Input values	StdDev:	partial derivatives	uncertainty budget:	budget: in %
p1	#Summed number of gross counts Ng		Ng	9600,00	97,98	0,3380	33,1147	3,20
p2	Number of single measurements		m	5,00	0,00	0,0000	0,0000	0,00
p3	Half-live of H-3	1/s	lam_H3	1,7800E-09	0,0000E+00	1,2784E+11	0,0000	0,00
p4	Detection efficiency	1/(Bq s)	eps	3,3000E-01	1,6500E-02	-8603,0070	141,9496	58,78
p5	Chemical yield of the electrolytical cell i		etaAi	8,5000E-01	1,7000E-02	-3339,9910	56,7798	9,40
p6	Density of seawater	kg/m³	rho	1000,00	30,00	2,8390	85,1699	21,16
p7	Time elapsed sampling --> startmeas	s	tA	4,5000E+07	0,0000E+00	0,0000	0,0000	0,00
p8	Total duration of measurements	s	tm	60000,00	0,00	-0,0541	0,0000	0,00
p9	Duration of background measurement	s	_t0	60000,00	0,00	0,0000	0,0000	0,00
p10	Background count rate	1/s	R0	2,0000E-02	5,7735E-04	-20278,5369	11,7078	0,40
p11	Rest mass of solution after electrolysis	kg	mn	2,1000E-02	2,1000E-04	135190,2459	28,3900	2,35
p12	Total mass of solution before electrolysis	kg	mv	5,0000E-01	5,0000E-03	-5677,9846	28,3899	2,35
p13	Mass of solution in the counting vial	kg	mM	8,0000E-03	8,0000E-05	-354874,0405	28,3899	2,35

(List can be continued here)

Model section		c = Factor * Rn	
Auxiliary equations h		(Formulae)	
#Gross count rate Rg	1/s	Rg	1,6000E-01
h1	Decay correction during measurement		_f3	9,9995E-01
h2	Decay correction sampling		_f4	1,0834E+00

(List can be continued here)

#Net count rate Rn	1/s	Rn	1,4000E-01	
#Calibration factor, proc.dep.	Bq*s/m³	Factor	2,0279E+04	
#Value output quantity	Bq/m³	Erg	2,8390E+03	79,4116014 <-- output value modifiable by VBA
#Combined standard uncertainty	Bq/m³	uErg	1,8515E+02	

Calculate!

| #Decision threshold | Bq/m³ | | 49,6720681 |
| #Detection limit | Bq/m³ | | 79,4115851 |

further derived values			
Auxiliary quantity Omega		Omega	1,0000E+00
Best estimate	Bq/m³	BestEst	2,8390E+03
Uncertainty best estimate	Bq/m³		1,8515E+02
Lower confidence limit	Bq/m³		2,4761E+03
Upper confidence limit	Bq/m³		3,2019E+03

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

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7.2   View of the UncertRadio result page

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.

—   sodium peroxide, Na_2O_2;

—   ethylene glycol (optionally);

—   tritium-poor, distilled water;

—   scintillation cocktail: e.g. Quicksafe 450.

8.2   Equipment

The following equipment is used for the procedure:

— usual equipment of a radiochemical laboratory;

— 1 l glass bottle with screw cap and plastic seal;

— round-bottom flask (250 ml and 500 ml);

— vacuum distillation apparatus incl. pressure display;

— temperature-controlled water baths;

— apparatus for electrolytic enrichment, incl. accessories;

— piston pipette (1 ml to 10 ml or 1 ml to 12 ml, variable);

— 20-ml-sld-scintillation vials (**s**uper **l**ow **d**iffusion);

— low-level liquid scintillation spectrometer.

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