

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

D-H-3-MWASS-01

Authors:

I. Bendler

K. Becker

J. Herrmann

S. Schmied

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

(Leitstelle für Meerwasser, Meeresschwebstoff und -sediment)

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

1 Scope

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

The procedure is applicable for activity concentrations of H-3 of more than $100 \text{ Bq}\cdot\text{m}^{-3}$ in sea water after electrolytic enrichment in the routine operation mode. In the intensive operation mode, the procedure is applied without electrolytic enrichment.

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 twice with sea water. 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 sea water sample is distilled under vacuum without further pretreatment. For expected activity concentrations above $2 \text{ Bq}\cdot\text{l}^{-1}$, an aliquot is taken from the resulting distillate and is measured directly using a liquid scintillation spectrometer 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 may still be 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 maximum of 0,5 litre of the sea water sample is distilled under vacuum at a pressure of about $4 \cdot 10^3$ 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 five samples can be distilled simultaneously, is shown in figures 1 and 2.



Fig. 1: Distillation apparatus with five distillation units

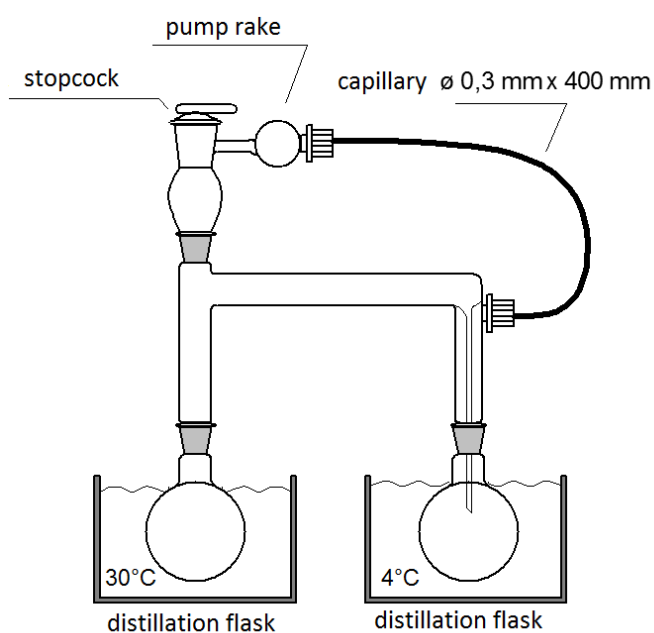


Fig. 2: Sketch of the distillation apparatus

3.2.1.1 About 500 ml of the sea water 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.

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.

3.2.1.4 The sample is then distilled to dryness at a pressure of about $4 \cdot 10^3$ 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 40 °C.

3.2.1.5 By weighing the cooled and dried round-bottom flasks, the weight of the distillate flask and the distillate is determined.

3.2.1.6 For expected activity concentrations above $2 \text{ Bq} \cdot \text{l}^{-1}$, 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 Electrolytic enrichment

The enrichment apparatus used in the federal coordinating office is shown in figure 3 and consists of the following components:

- up to nine 500 ml electrolysis cells, each consisting of two parts (see figure 4):
 - 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. 3: Enrichment apparatus with nine electrolysis cells



Fig. 4: Electrolysis cell (top) consisting of anode/outer part (center) and cathode/inner part (bottom)

3.2.2.1 About 1,3 g of sodium peroxide are weighed into the anode of the electrolysis cell and the total mass, consisting of the masses of the anode, the cathode and the sodium peroxide, is determined.

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

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

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

3.2.2.5 An exhaust gas hose (blue hoses in figure 3) is attached to the cathode to dissipate the resulting oxyhydrogen, which is connected to the exhaust of the enrichment equipment.

3.2.2.6 Anode and cathode are connected to the power supply.

3.2.2.7 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.

Note:

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

3.2.2.8 After about 30 minutes, the current is increased to 10 A. At this current, an amount of water of about 80 ml per day is decomposed.

Note:

The single voltages of the electrolysis cells are checked at least once a working day. In the following, the note in step 3.2.2.7 is taken into account.

3.2.2.9 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.

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

3.2.2.11 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.

3.2.2.12 Then, a new distillation is carried out in accordance with section 3.2.1. From the resulting distillate a counting source is prepared 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 activity of the tritium is measured after sufficient precooling of the scintillation vial in a low-level liquid scintillation spectrometer. For each measurement series, tritium-poor water, e. g. Atlantic water or deep well water, is used for determining the background count rate.

Note:

The scintillation vials should be precooled for at least 2 hours to minimize luminescent effects.

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 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):

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

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;

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

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

$m_{v,i}$ 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.

5 Calculation of the results

5.1 Equations

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

$$c = \frac{m_n \cdot \rho \cdot f_A}{\varepsilon \cdot \eta_{A,i} \cdot m_v \cdot m_M} \cdot R_n = \frac{m_n \cdot \rho \cdot e^{\lambda \cdot t_A}}{\varepsilon \cdot \eta_{A,i} \cdot m_v \cdot m_M} \cdot R_n = \varphi \cdot R_n \quad (2)$$

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

$$c = \frac{\rho \cdot f_A}{\varepsilon \cdot m_M} \cdot R_n = \frac{\rho \cdot e^{\lambda \cdot t_A}}{\varepsilon \cdot m_M} \cdot R_n = \varphi \cdot R_n \quad (3)$$

In equations (2) and (3) are:

c activity concentration in $\text{Bq} \cdot \text{m}^{-3}$;

f_A correction factor for the decay;

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

t_A time period between sampling and beginning of measurement in s;

R_n net count rate of the counting source in s^{-1} :

$$\text{with } R_n = R_g - R_0$$

ε detection efficiency in $\text{Bq}^{-1} \cdot \text{s}^{-1}$;

ρ density of the sea water in $\text{kg} \cdot \text{m}^{-3}$;

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;

m_M mass of distillate in the scintillation vial, in g;

φ procedural calibration factor in $\text{Bq} \cdot \text{s} \cdot \text{m}^{-3}$.

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

$$R_g = \frac{\sum_1^j R_{g,j} \cdot t_{m,j}}{\sum_1^j t_{m,j}} = \frac{\sum_1^j R_{g,j} \cdot t_{m,j}}{t_{\text{tot}}} \quad (4)$$

Herein are:

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

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

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

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:

$$u(c) = c \cdot \sqrt{\frac{1}{(R_g - R_0)^2} \cdot \left(\frac{R_g}{t_{\text{tot}}} + \frac{R_0}{t_0} \right) + u_{\text{rel}}^2(\varphi)} \quad (5)$$

with

$$u_{\text{rel}}^2(\varphi) = u_{\text{rel}}^2(f_A) + u_{\text{rel}}^2(m_n) + u_{\text{rel}}^2(m_v) + u_{\text{rel}}^2(m_M) + u_{\text{rel}}^2(\varepsilon) + u_{\text{rel}}^2(\eta_{A,i}) + u_{\text{rel}}^2(\rho)$$

Herein are:

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

$u_{\text{rel}}(f_A)$ relative standard uncertainty of the correction factor for the decay;

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

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

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

$u_{\text{rel}}(m_M)$ relative standard uncertainty of the mass of distillate in the scintillation vial;

$u_{\text{rel}}(\rho)$ relative standard uncertainty of the density of sea water;

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

5.2 Worked example

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 $u_{\text{rel}}(f_A)$ is neglected.

$R_{g,1}$	= 0,160 s ⁻¹ ;	$t_{m,1}$	= 200 min = 12000 s;
$R_{g,2}$	= 0,140 s ⁻¹ ;	$t_{m,2}$	= 12000 s;
$R_{g,3}$	= 0,180 s ⁻¹ ;	$t_{m,3}$	= 12000 s;
$R_{g,4}$	= 0,150 s ⁻¹ ;	$t_{m,4}$	= 12000 s;
$R_{g,5}$	= 0,170 s ⁻¹ ;	$t_{m,5}$	= 12000 s;
R_0	= 0,020 s ⁻¹ ;	t_0	= 60000 s;
t_A	= 4,50 · 10 ⁷ s;	t_{tot}	= 60000 s;
m_v	= 0,5 kg;	$u_{\text{rel}}(m_v)$	= 0,01;
m_n	= 0,021 kg;	$u_{\text{rel}}(m_n)$	= 0,01;
m_M	= 0,008 kg;	$u_{\text{rel}}(m_M)$	= 0,01;
ε	= 0,33 Bq ⁻¹ · s ⁻¹ ;	$u_{\text{rel}}(\varepsilon)$	= 0,05;
$\eta_{A,i}$	= 0,85;	$u_{\text{rel}}(\eta_{A,i})$	= 0,02;
ρ	= 1 · 10 ³ kg · m ⁻³ ;	$u_{\text{rel}}(\rho)$	= 0,03;
λ	= 1,78 · 10 ⁻⁹ s ⁻¹ ;	φ	= 20277 Bq · s · m ⁻³ .

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

$$R_g = \frac{(0,160 \text{ s}^{-1} + 0,140 \text{ s}^{-1} + 0,180 \text{ s}^{-1} + 0,150 \text{ s}^{-1} + 0,170 \text{ s}^{-1}) \cdot 12000 \text{ s}}{60000 \text{ s}} = 0,160 \text{ s}^{-1}$$

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

$$c = \frac{0,021 \text{ kg} \cdot 1 \cdot 10^3 \text{ kg} \cdot \text{m}^{-3} \cdot e^{1 \cdot 10^3 \text{ s}^{-1} \cdot 4,50 \cdot 10^7 \text{ s}}}{0,33 \text{ Bq}^{-1} \cdot \text{s}^{-1} \cdot 0,85 \cdot 0,5 \text{ kg} \cdot 0,008 \text{ kg}} \cdot 0,140 \text{ s}^{-1} = 20277 \text{ Bq} \cdot \text{s} \cdot \text{m}^{-3} \cdot 0,140 \text{ s}^{-1} = 2839 \text{ Bq} \cdot \text{m}^{-3}$$

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):

$$u(c) = 2839 \text{ Bq} \cdot \text{m}^{-3} \cdot \sqrt{\frac{1}{(0,140 \text{ s}^{-1})^2} \cdot \frac{0,160 \text{ s}^{-1} + 0,020 \text{ s}^{-1}}{60000 \text{ s}} + 0,0041} = 189 \text{ Bq} \cdot \text{m}^{-3}$$

with

$$u_{\text{rel}}^2(\varphi) = 0,01^2 + 0,01^2 + 0,01^2 + 0,05^2 + 0,02^2 + 0,03^2 = 0,0041$$

The activity concentration c is thus:

$$c = (2839 \pm 189) \text{ Bq} \cdot \text{m}^{-3}$$

5.3 Consideration of uncertainties

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 DIN ISO 11929.

An excel spreadsheet (see section 7.3.1) as well as 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 procedure manuals.

6.1 Equations

6.1.1 Decision threshold

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

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

Herein are:

c^* decision threshold in $\text{Bq} \cdot \text{m}^{-3}$;

$k_{1-\alpha}$ 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 (7):

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

In equation (7) are:

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

$k_{1-\beta}$ 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):

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

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 Confidence intervals

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

6.2 Worked example

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

$$\begin{aligned} k_{1-\alpha} &= 3; & k_{1-\beta} &= 1,645; \\ \varphi &= 20277 \text{ Bq}\cdot\text{s}\cdot\text{m}^{-3}; & u_{\text{rel}}^2(\varphi) &= 0,0041. \end{aligned}$$

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

$$c^* = 3 \cdot 20277 \text{ Bq}\cdot\text{s}\cdot\text{m}^{-3} \cdot \sqrt{0,020 \text{ s}^{-1} \cdot \left(\frac{1}{60000 \text{ s}} + \frac{1}{60000 \text{ s}} \right)} = 50 \text{ Bq}\cdot\text{m}^{-3}$$

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

$$c^\# = \frac{50 \text{ Bq}\cdot\text{m}^{-3} \cdot 1,009}{0,99} \cdot \left\{ 1 + \sqrt{1 - \frac{0,99}{1,009^2} \cdot \left(1 - \frac{1,645^2}{3^2} \right)} \right\} = 80 \text{ Bq}\cdot\text{m}^{-3}$$

with

$$\theta = 1 - 1,645^2 \cdot 0,0041 = 0,99$$

$$\Psi = 1 + \frac{1,645^2}{2 \cdot 50 \text{ Bq}\cdot\text{m}^{-3}} \cdot 20277 \text{ Bq}\cdot\text{s}\cdot\text{m}^{-3} \cdot \frac{1}{60000 \text{ s}} = 1,009$$

7 Catalogue of chemicals and equipment

7.1 Chemicals

The chemicals used should be of analytically pure quality.

- sodium peroxide, Na_2O_2 ;
- tritium-poor, distilled water;
- scintillation cocktail: e.g. Quicksafe 400.

7.2 Equipment

- 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, variable);
- 20 ml-sld-scintillation vials (**super low diffusion**);
- low-level liquid scintillation spectrometer.

7.3 Software supported calculation

7.3.1 Example of an Excel spreadsheet

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

D-H-3-MWASS-01

Version

November 2018

Procedures manual for monitoring of radioactive substances in the environment and of external radiation (ISSN 1865-8725)

SAMPLE IDENTIFICATION: Sea water

#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
p4	Detection efficiency	1/(Bq s)	eps	3,3000E-01	1,6500E-02	-8603,0070	141,9496
p5	Chemical yield of the electrolytical cell i		etaAi	8,5000E-01	1,7000E-02	-3339,9910	56,7798
p6	Density of sea water	kg/m ³	rho	1000,00	30,00	2,8390	85,1699
p7	Time elapsed sampling --> startmeas	s	tA	4,5000E+07	0,0000E+00	0,0000	0,0000
p8	Total duration of measurements	s	tm	60000,00	0,00	-0,0541	0,0000
p9	Duration of background measurement	s	_t0	60000,00	0,00	0,0000	0,0000
p10	Background count rate	1/s	R0	2,0000E-02	5,7735E-04	-20278,5369	11,7078
p11	Rest mass of solution after electrolysis	kg	mn	2,1000E-02	2,1000E-04	135190,2459	28,3900
p12	Total mass of solution before electrolysis	kg	mv	5,0000E-01	5,0000E-03	-5677,9846	28,3899
p13	Mass of solution in the counting vial	kg	mM	8,0000E-03	8,0000E-05	-354874,0405	28,3899
(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/m ³	Factor	2,0279E+04			
	Value output quantity	Bq/m ³	Erg	2,8390E+03	79,4116014	<-- output value modifiable by VBA	
	#Combined standard uncertainty	Bq/m ³	uErg	1,8515E+02			
	#Decision threshold	Bq/m ³		49,6720681			
	#Detection limit	Bq/m ³		79,4115851			
Calculate!							
further derived values							
	Auxiliary quantity Omega		Omega	1,0000E+00			
	Best estimate	Bq/m ³	BestEst	2,8390E+03			
	Uncertainty best estimate	Bq/m ³		1,8515E+02			
	Lower confidence limit	Bq/m ³		2,4761E+03			
	Upper confidence limit	Bq/m ³		3,2019E+03			

The corresponding excel spreadsheet can be found on the website of this procedures manual.

7.3.2 Example of an UncertRadio project

The screenshot displays the 'Results' tab of the UncertRadio software. The window title is 'UncertRadio: Calculation of uncertainty budget and detection limits - D-H-3-MWASS-01_V2018-11_EN.bxp'. The interface includes a menu bar (File, Edit, Options, Help), a toolbar, and a status bar at the bottom showing the project path and 'Ready!'.

Final measurement result for c :

Coverage factor k:	1.0
Value output quantity:	2838.844 Bq/m ³
extendend (Std.-)uncertainty:	188.4508 Bq/m ³
relative ext.(Std.-)uncertainty:	6.638294 %
Best Bayesian Estimates:	<input type="checkbox"/> min. Coverage-Intervall
Value output quantity:	2838.844 Bq/m ³
extendend (Std.-)uncertainty:	188.4508 Bq/m ³
lower confidence limit:	2469.487 Bq/m ³
upper confidence limit:	3208.20 Bq/m ³
Probability (1-gamma):	0.950

Decision threshold and detection limit for c :

Decision threshold (DT):	49.66942 Bq/m ³	Iterations:	1
Detection limit (DL):	79.45074 Bq/m ³	Iterations:	5

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

LinFit: Standard uncertainty of fit parameter ai:

from LS analysis: []

from uncertainty propagation: []

reduced Chi-square: []

Monte Carlo Simulation:

Number of simul. measurments: 100000 Values <0 included
 Number of runs: 1 min. Coverage interval

Value output quantity:	2848.237 Bq/m ³	relSD%:	0.021
extendend uncertainty:	190.0572 Bq/m ³		0.224
relative extd.(Std.-)uncertainty:	6.672802 %		
lower confidence limit:	2499.690 Bq/m ³		0.064
upper confidence limit:	3242.868 Bq/m ³		0.050
Decision threshold (DT):	50.75171 Bq/m ³		0.873
Detection limit (DL):	80.26966 Bq/m ³		0.573

active run: 1 IT: 10 Start MC

Project: -MWASS-01\Berechnungsdateien\D-H-3-MWASS-01_V2018-11_EN.bxp Ready!

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

References

- (1) Mahrla, P.: *Die Meßgenauigkeit des Heidelberger Low-Level-Tritiummeßverfahrens für ozeanische Proben*. Diplomarbeit 1978, Institut für Umweltphysik der Universität Heidelberg.
- (2) Zutshi, P. K., Sas-Hubicki, J.: *A new cathode treatment for the reproducible electrolytic enrichment of tritium*. Int. J. Appl. Radiat. Isot., 1966, Vol. 17, S. 670 – 671.
- (3) Östlund, H. G., Werner, E.: *The electrolytic enrichment of tritium and deuterium for natural tritium measurements*. Tritium in the Physical and Biological Sciences, Vol. 1, S. 95 – 104.
- (4) Weiss, W., Roether, W., Bader, G.: *Determination of blanks in low-level tritium measurements*. Int. J. Appl. Radiat. Isot., 1976, Vol. 27, S. 217 – 225.