Procedure for determining the specific activities of radionuclides in fish and fishery products by gamma spectrometry

G-γ-SPEKT-FISCH-02

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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 described in the following is suitable for gamma-ray-spectrometric analysis of fish samples in case of higher releases of radioactive substances to get a rapid overview on the extent of contamination according to the IMIS-intensive measurement programme [1]. It offers the main advantage to determine the specific activities of iodine isotopes, which is not possible with the Procedure G-γ-SPEKT-FISCH-01 due to the volatility of iodine.

The procedure described in the following may also be used for gamma spectrometric analysis of samples of fish that have to be monitored in the IMIS-routine measurement programme [1] according to the German Precautionary Radiation Protection Act (StrlSchG) [2] and the Guideline concerning Emission and Immission Monitoring of Nuclear Installations [3] in normal operation.

2 Sampling

The fundamentals of sampling are described in the Procedure G-γ-SPEKT-FISCH-01. The sample mass has to be dimensioned large enough that 1,5 kg fresh mass is available for the analysis, which is filled into a 1 litre Marinelli beaker (reentrant beaker).

Special care must be taken if fish is sampled in higher contaminated regions [4]. The freshly collected fish are killed quickly at the point of sampling and packed into plastic bags, which are stored on ice inside a suitable container. Freezing of the sample should be avoided as they should be measured as direct after sampling as possible. If freezing of the sample cannot be avoided, special care must be taken to include tissue fluids that may be liberated during the defrosting process in the sample. Furthermore, it must be distributed homogenously in the sample during the measurement process.

In case of import goods, crustaceans and shellfish and also tinned fish (fish commodities) are sampled besides fish. Samples are obtained from importers or super markets operating in the respective Federal State. Only the consumable parts considered as generally consumed by the public are analysed.

For correct labeling of samples, it is referred to Procedure G-γ-SPEKT-FISCH-01 and the literature [5] .

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3 Analysis

3.1 Principle of the procedure

Fresh fish is filleted first. Fillets and tinned fish are chopped and homogenized. The specific activity is of gamma-ray emitting radionuclides including iodine is determined inside a 1 litre Marinelli-beaker by gamma spectrometry.

3.2 Sample preparation

All items used for sample preparation, e. g. cutting boards, knives and Marinelli-beakers, must be cleaned using cleaning solution prior each use.

Fish are cleaned like in common household. Afterwards, they are filleted and bones and skin are removed as much as possible. The fish flesh obtained is finely cubed, a preserving agent, e. g. sodium azide is added and both are thoroughly mixed (see below). The use of a mixer or meat grinder must be avoided in cases of higher contamination.

In case of tinned fishery products, it is recommended that the included liquids (e. g. sauces) are processed according to their general consumption by the public. When they are not consumed, they may be discarded; otherwise they must be uniformly distributed inside the sample. The filling is not cleaned, but the texture may require mincing. The addition of preserving agents takes place before homogenization of the sample.

Generally, the preparation is only stable for a short time. The addition of preserving agents like sodium azide decelerates microbial processes inside the preparation by which gases are released that lead to a swelling of the preparation. During measurement, it is strictly advised that the cap is only loosely placed on top of the beaker. Additionally, the measurement chamber should be coated with foil or the beaker should be placed inside a large plastic bag.

At longer idle times before analysis, the preparation must be stored in the fridge.

3.3 Radiochemical separation

A radiochemical separation is not required.

4 Measuring the activity

4.1 General

The fundamentals of gamma spectrometry are discussed in the General Chapter γ-SPEKT/GRUNDL of this Procedures' Manual [6] and in the literature [7, 8, 9].

4.2 Background

The fundamentals of measuring the background effects are described in the Procedure G-γ-SPEKT-FISCH-01 and in the General Chapter γ-SPEKT/NULLEF of this Procedures' Manual [10].

In cases of specific activities two orders larger compared to the routine measuring programme, the background effect must be determined with smaller measurement duration but higher frequency, e. g. each working day.

For fish samples, reliably determining the K-40 background count rate of the background effect is important in order to be able to use the K-40 activity determined in fish for verifying the correctness of the subsequent sample spectrum analysis.

4.3 Calibration

The calibration is carried out with a suitable calibration source, e. g. an aqueous solution of a traceable standard or a resin containing traceable activities of the radionuclides of interest.

Because of the small difference in densities between water and fish flesh, a density correction for fish flesh may be omitted. This is due to the fact the density of the aqueous calibration solution is close to that of water (1 $g \cdot cm^{-3}$) and fish flesh contains from water by approx. 80 %.

For further information concerning calibration and self-attenuation, reference is made to General Chapter γ-SPEKT/GRUNDL of this Procedures' Manual [6].

4.4 Measurement

The measurement is described in Section 4.4 of the Procedure G-γ-SPEKT-FISCH-01 in detail. Instead of the cylindrical beaker described there, a 1-liter-Marinelli-beaker is used here.

5 Calculation of the results

5.1 Output quantity

When determining specific activities of radionuclides in fish flesh, the gamma lines of different radionuclides usually do not interfere. Therefore, specific activities of radionuclides are determined from a single gamma line. In case of multiple-line radionuclides, a weighted mean can be used. For details, reference is made to the General Chapters γ-SPEKT/GRUNDL and γ-SPEKT/INTERF of this Procedures' Manual [6, 11] .

If a net count rate $R_{\mathrm{n,r}}$ associated with the gamma-ray energy of the radionuclide r has been detected, the resulting specific activity a_{r}, relative to wet mass (FM) and the date and time of sampling, is calculated according to Equation (1):

$$a_{\mathrm{r}} = \varphi \cdot R_{\mathrm{n,r}} = \frac{f_1 \cdot f_2 \cdot f_3 \cdot f_4}{\varepsilon_{\mathrm{W}} \cdot p_{\gamma,\mathrm{r}} \cdot m_{\mathrm{F}}} \cdot R_{\mathrm{n,r}} \quad (1)$$

with

$$f_3 = \frac{\lambda_{\mathrm{r}} \cdot t_{\mathrm{m}}}{1 - \mathrm{e}^{-\lambda_{\mathrm{r}} \cdot t_{\mathrm{m}}}} \quad (2)$$

$$f_4 = \mathrm{e}^{\lambda_{\mathrm{r}} \cdot t_{\mathrm{A}}} \quad (3)$$

Herein are:

f_1 correction factor for coincidence; for an undisturbed gamma line, $f_1 = 1$;

f_2 correction factor for self-attenuation for fish flesh relative to water, $f_2 = 1$;

f_3 correction factor for the decay of the activity of the radionuclide r during the measurement;

f_4 correction factor for the decay of the activity of the radionuclide r related to a reference time;

m_{F} mass of fish flesh used for the measurement, in kg;

$p_{\gamma,\mathrm{r}}$ emission intensity of the considered gamma line of the radionuclide r;

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

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

ε_{W} detection efficiency for water, depending on energy and fill level, in $\mathrm{Bq^{-1} \cdot s^{-1}}$;

λ_{r} decay constant of the radionuclide r, in $\mathrm{s^{-1}}$;

φ procedural calibration factor, in $\mathrm{Bq \cdot s \cdot kg^{-1}}$.

The net count rate of the gamma-line of the radionuclide r is calculated according to Equation (4):

$$R_{\mathrm{n,r}} = R_{\mathrm{g,r}} - R_{\mathrm{T,r}} - R_{0,\mathrm{r}} \quad (4)$$

Herein are:

$R_{\mathrm{g,r}}$ gross count rate of the considered gamma line of the radionuclide r, in $\mathrm{s^{-1}}$;

$R_{\mathrm{T,r}}$ background count rate underneath the considered gamma line of the radionuclide r, e. g. as count rate of a trapezoidal background, in $\mathrm{s^{-1}}$;

$R_{0,\mathrm{r}}$ net count rate at the considered gamma line of the radionuclide r in a background spectrum, in $\mathrm{s^{-1}}$.

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 generalized expression to calculate the uncertainty of the net count rate is given in Equation (5),

$$u^2(R_{\mathrm{n,r}}) = \mu_0 \cdot R_{\mathrm{n,r}}^2 + \mu_1 \cdot R_{\mathrm{n,r}} + \mu_2 \tag{5}$$

with the coefficients μ_k:

$$\mu_0 = 0 \tag{6}$$

$$\mu_1 = \frac{1}{t_{\mathrm{m}}} \tag{7}$$

$$\mu_2 = \frac{R_{\mathrm{T,r}} + R_{0,\mathrm{r}}}{t_{\mathrm{m}}} + u^2(R_{\mathrm{T,r}}) + u^2(R_{0,\mathrm{r}}) \tag{8}$$

Using the trapezoidal method (assuming a linear background continuum) the coefficient μ_2 is given by Equation (9):

$$\mu_2 = \frac{R_{\mathrm{T,r}}}{t_{\mathrm{m}}} \cdot \left(1 + \frac{b}{2 \cdot L}\right) + R_{0,\mathrm{r}} \cdot \left(\frac{1}{t_{\mathrm{m}}} + \frac{1}{t_0}\right) + \frac{R_{\mathrm{T},0,\mathrm{r}}}{t_0} \cdot \left(1 + \frac{b_0}{2 \cdot L_0}\right) \tag{9}$$

Herein are:

b line widths of sample spectra at the respective peak baselines, in channels;

b_0 line widths of background spectra at the respective peak baselines, in channels;

L numbers of channels for the sample spectrum, respectively, over which the background continuum to the left and to the right of the peaks are estimated;

L_0 numbers of channels for the background spectrum, respectively, over which the background continuum to the left and to the right of the peaks are estimated;

$R_{\mathrm{T},0,\mathrm{r}}$ background continuum count rate at the line of the radionuclide r within the background spectrum, e. g. as a trapezoidal background count rate, in s^{-1};

t_0 duration of the background measurement, in s.

Equation (9) is a sufficient approximation for the background estimated by an empirical background step function.

The μ_k coefficients only need to be inserted into Equation (5) to receive the standard uncertainty of the net count rate $u(R_{\mathrm{n,r}})$ given by Equation (10):

$$u^2(R_{\mathrm{n,r}}) = \frac{R_{\mathrm{n,r}}}{t_{\mathrm{m}}} + \mu_2 = \\ = \frac{R_{\mathrm{n,r}}}{t_{\mathrm{m}}} + \frac{R_{\mathrm{T,r}}}{t_{\mathrm{m}}} \cdot \left(1 + \frac{b}{2 \cdot L}\right) + R_{0,\mathrm{r}} \cdot \left(\frac{1}{t_{\mathrm{m}}} + \frac{1}{t_0}\right) + \frac{R_{\mathrm{T,0,r}}}{t_0} \cdot \left(1 + \frac{b_0}{2 \cdot L_0}\right) \tag{10}$$

When no count rate occurs at corresponding gamma ray energy of background spectrum the Equations (9) and (10) are reduced by the last two terms.

The relative standard uncertainty of the procedural calibration factor $u_{\mathrm{rel}}(\varphi)$ is determined according to Equation (11), where the uncertainty of the decay correction may be neglected:

$$u_{\mathrm{rel}}(\varphi) = \left[u_{\mathrm{rel}}^2(f_1) + u_{\mathrm{rel}}^2(f_2) + u_{\mathrm{rel}}^2(f_3) + u_{\mathrm{rel}}^2(f_4) + u_{\mathrm{rel}}^2(\varepsilon_{\mathrm{W}}) + \right. \\ \left. + u_{\mathrm{rel}}^2(p_\gamma) + u_{\mathrm{rel}}^2(m_{\mathrm{F}})\right]^{1/2} \tag{11}$$

The combined standard uncertainty of the specific activity $u(a_{\mathrm{r}})$ using Equation (1) is calculated as:

$$u(a_{\mathrm{r}}) = a_{\mathrm{r}} \cdot \sqrt{u_{\mathrm{rel}}^2(\varphi) + \frac{u^2(R_{\mathrm{n,r}})}{R_{\mathrm{n,r}}^2}} \tag{12}$$

The combined standard uncertainties of the specific activity amount to be 20 % or less except that the activities are so small that the relative uncertainties of the net counting rate already clearly exceed 20 %. The standard uncertainty of the self-attenuation correction factor does not contribute anything to this.

For multiple-line radionuclides such as Cs-134, the uncertainty contribution of the coincidence summing correction factors has to be taken into account, which may lead to additional few percent in case of significant corrections. It is referred to General Chapters γ-SPEKT/GRUNDL and γ-SPEKT/SUMESC of this Procedures' Manual [6, 12].

6 Characteristic limits of the procedure

The calculation of the characteristic limits follows the standard series ISO 11929 [13]. For further considerations, it is referred to the General Chapters CHAGR-ISO-01 and CHAGR-ISO-02 of this Procedures' Manual [14, 15].

6.1 Decision threshold

The decision threshold of the specific activity a_{r}^{*} of the radionuclide r is calculated according to Equation (13):

$$a_{\mathrm{r}}^{*} = \varphi \cdot k_{1-\alpha} \cdot \sqrt{\mu_2} = \varphi \cdot k_{1-\alpha} \cdot \sqrt{\frac{1}{t_{\mathrm{m}}} \cdot \left(R_{\mathrm{T,r}} + R_{0,\mathrm{r}}\right) + u^2\left(R_{\mathrm{T,r}}\right) + u^2\left(R_{0,\mathrm{r}}\right)} \tag{13}$$

Herein is:

$k_{1-\alpha}$ quantile of the normal distribution associated with the type I error.

6.2 Detection limit

The detection limit $a_{\mathrm{r}}^{\#}$ of single-line radionuclides is calculated with Equation (13) using the decision threshold of the radionuclide's specific activity a_{r}^{*} obtained by Equation (14):

$$a_{\mathrm{r}}^{\#} = a_{\mathrm{r}}^{*} \cdot \frac{\psi}{\theta} \cdot \left[1 + \sqrt{1 - \frac{\theta}{\psi^2}\left(1 - \frac{k_{1-\beta}^2}{k_{1-\alpha}^2}\right)}\right] \tag{14}$$

Herein is

$k_{1-\beta}$ quantile of the normal distribution associated with the type II error.

with the auxiliary quantities:

$$\theta = 1 - k_{1-\beta}^2 \cdot u_{\mathrm{rel}}^2(\varphi) \tag{15}$$

$$\psi = 1 + \frac{k_{1-\beta}^2}{2 \cdot a_{\mathrm{r}}^{*}} \cdot \frac{\varphi}{t_{\mathrm{m}}} \tag{16}$$

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 the software UncertRadio (see Section 7.2). A project file for the software UncertRadio is available on the website of this Procedures' Manual.

7.1 Manual evaluation

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

The following example is used to calculate the specific I-131 activity in 1 kg fresh fish flesh (FM) using only the intensity of the gamma-ray energy at 364,5 keV. The following values are used:

$R_{\mathrm{n,I\text{-}131}}$	=	0,5115 s^{-1};	$R_{\mathrm{T,I\text{-}131}}$	=	1,96·10^{-3} s^{-1};
m_{F}	=	1,00 kg;	$u_{\mathrm{rel}}(m_{\mathrm{F}})$	=	4,0·10^{-3};
$p_{\gamma,\mathrm{I\text{-}131}}$	=	0,812;	$u_{\mathrm{rel}}(p_{\gamma,\mathrm{I\text{-}131}})$	=	9,85·10^{-3};
ε_{w}	=	17,75·10^{-3} $Bq^{-1}\cdot s^{-1}$;	$u_{\mathrm{rel}}(\varepsilon_{\mathrm{w}})$	=	0,029;
f_1	=	1,000;	$u_{\mathrm{rel}}(f_1)$	=	0;
f_2	=	1,000;	$u_{\mathrm{rel}}(f_2)$	=	0;
$b/(2\cdot L)$	=	0,523.			

The standard uncertainties of the following input quantities are negligible:

t_{A}	=	57,6·10^3 s;	$\lambda_{\mathrm{I\text{-}131}}$	=	9,999·10^{-7} s^{-1};
t_{m}	=	1,8·10^3 s.			

According to the Equations (2) and (3), to the correction factors f_3 and f_4 for the decay during the duration of the measurement and the decay related to sampling, respectively, are calculated:

$$f_3 = \frac{9{,}999 \cdot 10^{-7}\ \mathrm{s^{-1}} \cdot 1800\ \mathrm{s}}{1 - \mathrm{e}^{-9{,}999\cdot 10^{-7}\mathrm{s^{-1}} \cdot 1800\ \mathrm{s}}} \approx 1{,}0009$$

$$f_4 = \mathrm{e}^{9{,}999\cdot 10^{-7}\mathrm{s^{-1}} \cdot 57600\ \mathrm{s}} \approx 1{,}059$$

The procedural calibration factor is defined in Equation (1):

$$\varphi \approx \frac{1{,}000 \cdot 1{,}000 \cdot 1{,}0009 \cdot 1{,}059}{17{,}75 \cdot 10^{-3} \cdot 0{,}812 \cdot 1{,}00}\ \mathrm{Bq \cdot s \cdot kg^{-1}} \approx\ 73{,}54\ \mathrm{Bq \cdot s \cdot kg^{-1}}$$

The specific activity $a_{\mathrm{I\text{-}131}}$ in the fresh fish flesh (FM) results from Equation (1):

$$a_{\mathrm{I-131}} \approx 73{,}54\ \mathrm{Bq \cdot s \cdot kg^{-1}} \cdot 0{,}5115\ \mathrm{s^{-1}} \approx 37{,}62\ \mathrm{Bq \cdot kg^{-1}}\ (\mathrm{FM})$$

The relative standard uncertainty of the procedural calibration factor is calculated by Equation (11):

$$u_{\mathrm{rel}}\,(\varphi) = \sqrt{0^2 + 0^2 + 0^2 + 0^2 + 0{,}004^2 + 0{,}029^2 + (9{,}85 \cdot 10^{-3})^2} \approx 30{,}89 \cdot 10^{-3}$$

The standard uncertainty of the net count rate is obtained by Equation (10) with neglecting the last two terms:

$$u\big(R_{\mathrm{n,I-131}}\big) \approx \sqrt{\frac{0{,}5115\ \mathrm{s^{-1}}}{1800\ \mathrm{s}} + \frac{1{,}96 \cdot 10^{-3}\ \mathrm{s^{-1}}}{1800\ \mathrm{s}} \cdot (1 + 0{,}523)} \approx\ 16{,}91 \cdot 10^{-3}\ \mathrm{s^{-1}}$$

The combined standard uncertainty of the specific activity $u(a_{\text{I-131}})$ is calculated with Equation (12):

$$u(a_{\mathrm{I-131}}) \approx 37{,}62\ \cdot \sqrt{(30{,}89 \cdot 10^{-3})^2 + \left(\frac{16{,}91 \cdot 10^{-3}}{0{,}5115}\right)^2}\ \mathrm{Bq \cdot kg^{-1}} \approx$$

$$\approx 37{,}62\ \cdot 45{,}25 \cdot 10^{-3}\mathrm{Bq \cdot kg^{-1}} = 1{,}702\ \mathrm{Bq \cdot kg^{-1}}\ (\mathrm{FM})$$

Finally, the specific I-131 activity is obtained as:

$$a_{\mathrm{I-131}}\ =\ (37{,}6\ \pm\ 1{,}7)\ \mathrm{Bq \cdot kg^{-1}}\ (\mathrm{FM})$$

Using the values of the input quantities, the coefficient μ_2 is derived by Equation (9):

$$\mu_2 = \frac{1{,}96 \cdot 10^{-3}}{1800} \cdot (1 + 0{,}523)\ \mathrm{s^{-2}} \approx 1{,}658 \cdot 10^{-6}\ \mathrm{s^{-2}}$$

Using this value, the decision threshold of the specific activity $a^{*}_{\text{I-131}}$ is obtained from the value of the procedural calibration factor φ and the value of the normal distribution quantile $k_{1\text{-}\alpha}$ = 3:

$$a^{*}_{\mathrm{I-131}} \approx\ 73{,}54 \cdot 3{,}0 \cdot \sqrt{1{,}658 \cdot 10^{-6}}\ \mathrm{Bq \cdot kg^{-1}} \approx 0{,}2841\ \mathrm{Bq \cdot kg^{-1}}\ (\mathrm{FM})$$

Using a value of 1,645 for the normal distribution quantile $k_{1\text{-}\beta}$, the values of the auxiliary quantities are calculated by the Equations (15) and (16):

$$\theta \approx 1 - 1{,}645^2\ \cdot (30{,}89 \cdot 10^{-3})^2 \approx 0{,}9974$$

$$\psi \approx 1 + \frac{1{,}645^2}{2\ \cdot 0{,}2841\ \mathrm{Bq \cdot kg^{-1}}} \cdot \frac{73{,}54\ \mathrm{Bq \cdot s \cdot kg^{-1}}}{1800\ \mathrm{s}} \approx 1{,}1946$$

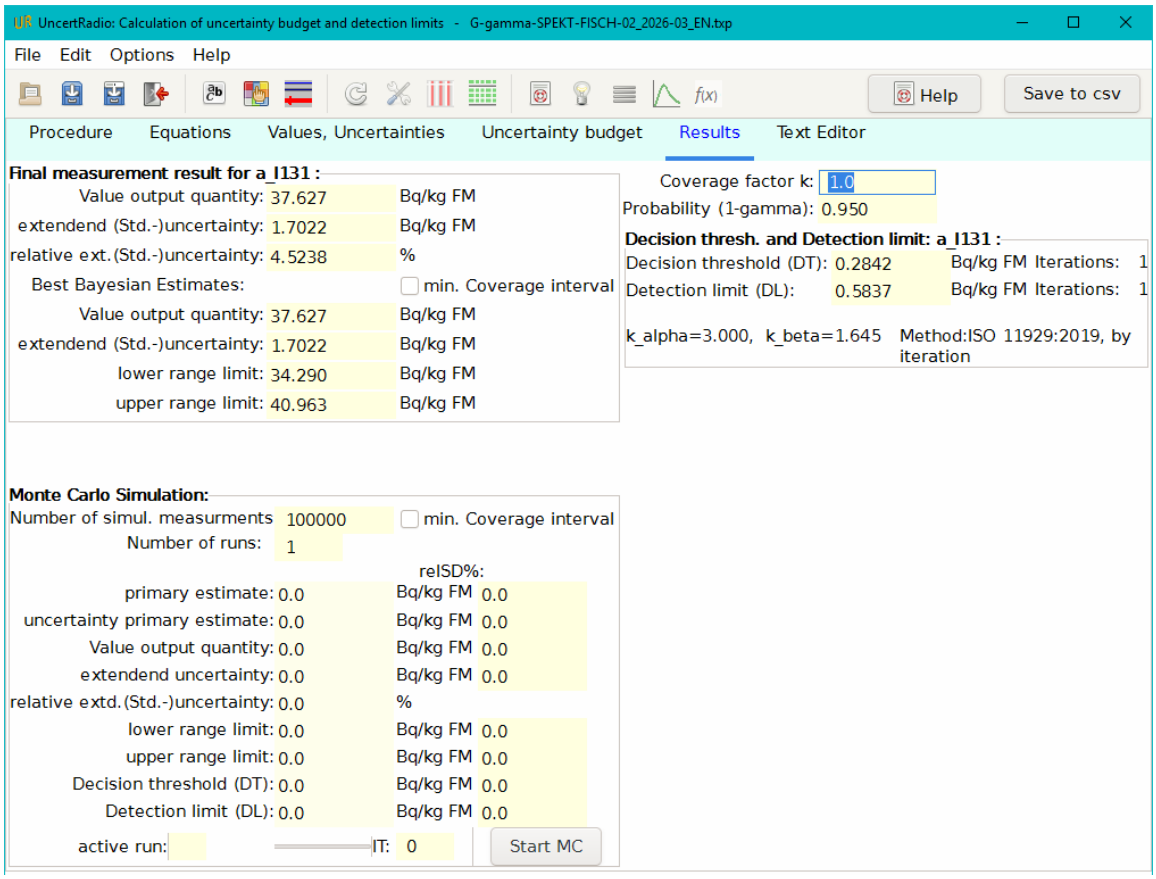
Then, the detection limit of the specific activity $a^{\#}_{\text{I-131}}$ is determined by Equation (14):

$$a^{\#}_{I-131} \approx 0{,}2841 \cdot \frac{1{,}1946}{0{,}9974} \cdot \left[1 + \sqrt{1 - \frac{0{,}9974}{1{,}1946^2} \cdot \left(1 - \frac{1{,}645^2}{3{,}0^2}\right)}\right] \text{Bq} \cdot \text{kg}^{-1} \approx$$

$$\approx 0{,}5836 \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)}$$

7.2 Software supported evaluation

In the following, the result page of the UncertRadio project file corresponding to this procedure is shown. The file itself 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.

— cleaning agent: e. g. RBS-50-Super-liquid concentrate® 2 %;

— sodium azide, NaN_3: Preserving agent

required for longer sample storage or in case of long (overnight) duration of measurement; generally, 3,5 ml NaN_3 solution (5 %) per kg wet mass.

8.2 Equipment

Besides the ordinary equipment of a radiochemical laboratory, the following is necessary:

8.2.1 Sampling

— ice container / cooling boxes;

— plastic bags;

— deep freezer (ca. -18 °C), if the samples have to be stored.

8.2.2 Sample preparation

— filleting board made of plastic;

— sharpened filleting knifes;

— cut-resistant gloves.

8.2.3 Calibration and measurement

— Marinelli-beaker (reentrant beaker, 1 l);

— plastic foil or plastic bags, as splash guard for detector and interior room of the lead shielding.

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