# Procedure for determining the specific activities of radionuclides in fish by gamma spectrometry

G-γ-SPEKT-FISCH-01

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Procedures' Manual for monitoring of radioactive substances in the environment and of external radiation
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# Procedure for determining the specific activities of radionuclides in fish by gamma spectrometry

## 1 Scope

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

The procedure is suitable for low-level measuring in the framework of radio-ecological studies due to its low limit of determination after dry-ashing of the sample.

## 2 Sampling

The sampling of fish – especially marine fish – relies to a wide extent on the co-operation of professionals in the respective industries (professional fishermen) and the product range available at fish markets.

It is in the responsibility of the person collecting the sample to ensure that a sample taken can be traced back to the sampled water body. The sampling position needs to be recorded as exact as possible. The name of the sampled fish species is also an essential part of the sample label. In case of rivers and streams, the river kilometre has to be additionally reported, while for saltwater fish the FAO fishing areas or coordinates of the catch must be reported by the fishermen. According to an EU regulation, marketed fish must be labelled with its registered trade name and the fishing area [4].

The required sample size depends on the type of study to be carried out. For studies according to the Radiation Protection Act and the Guideline for the Monitoring of Emissions and Immissions of Nuclear Installations, at least 1,5 kg of fish flesh is required. This equals a minimum of 3 kg non-filleted original sample. If the samples are to be used for additional analyses like strontium or isotopes of plutonium and americium, the sample size should be enlarged to 5 kg of fish flesh. Some species, e. g. sprat, must be examined as a whole fish because of their small size. The determining factor for sample preparation is the typical consumption of this species. In case of individual fishes, the person collecting the sample must ensure that only marketable fish is sampled.

The freshly collected fish are killed immediately at the point of sampling, sorted by species, packed in plastic bags, and transported to the laboratory in ice-filled containers.

The fish samples are stored deep-frozen at ca. -20 °C until they can be processed further in the laboratory.

# 3 Analysis

## 3.1 Principle of the procedure

The whole fish, or fish flesh obtained after filleting, is dried at 100 °C to 110 °C and then ashed at a maximum temperature of 420 °C. The specific activity of radionuclides in the homogenised ash are analysed using a gamma spectrometric measurement system.

The specific activities of iodine isotopes cannot be determined quantitatively because of the low boiling point of iodine (184 °C). The quantitative determination of iodine isotopes in fish requires the use of the Procedure G-γ-SPEKT-FISCH-02.

## 3.2 Sample preparation

All items used for preparation of the ashes, e. g. stainless steel bowls, ceramic bowls, measuring beakers, etc. must be cleaned using cleaning solution prior each use.

During filleting of fresh or defrosted fish, attention needs to be paid to deboning them as completely as possible. The obtained fish flesh is roughly chopped up. Chopped fish flesh or whole fish samples are placed in suitably large stainless steel or ceramic bowls that are laid out with ash-free transparent paper. Frozen fillets are defrosted directly in prepared stainless steel bowls in order to include tissue fluids liberated during the defrosting process in the subsequent drying and ashing steps. In case of frozen whole fish, the cell liquids from defrosting of roundfish, e. g. herring, sprat or cod, and flatfish, e. g. dab, flounder or plaice, are treated differently. As the latter release large amounts of mucus during frosting and defrosting which may disturb a later radiochemical analysis, the liquid is discarded. The total fresh mass used is to be recorded.

Afterwards, fish samples are dried until constant mass is reached, i. e. about one day or two days at a temperature of about 110 °C. Thereafter, the dry mass (TM) is determined. The ratio between fresh mass and dry mass in fish meat is about 5.

The time to reach the maximum ashing temperature depends on the lipid content of the sample; for normal fish it takes 45 hours. If fish with high lipid content, e. g. eel, mackerel, herring or sprat are to be ashed, this time needs to be extended in order to avoid the ignition of the sample.

In order to achieve a complete ashing of the sample, the final temperature is held for another 96 hours to 154 hours. The final temperature of 420 °C is selected to avoid the

volatilisation of caesium. Experiments to this effect, using the temperature program listed above, revealed ashing-induced losses of about 1 %.

The following temperature program is commonly used for fish samples with average lipid content:

Step 1: Linear increase of the temperature to 230 °C within 3 hours;

Step 2: Linear increase to the final temperature of 420 °C within 42 hours;

Step 3: Hold the final temperature for at least 96 hours, optimal 154 hours.

**Note:**
The danger of ignition during the ashing process rises, when the sample consists of material high in lipid content, e. g. cod liver. Therefore, the bowls must contain only a thin layer of these samples. The final temperature of 380 °C must be selected in these instances.

Once ashing has been completed and the sample has cooled down, the mass of the ash is recorded. A ratio of fresh mass to ash mass of about 80 (range 65 to 95) has usually to be expected in fish flesh; in whole fish, it may be lower by a factor of 2. Carbon residues still contained in the ash will not affect the results of the gamma spectrometric analysis. The ash is homogenised using a mortar, filled into a suitably large measuring beaker, and then carefully manually compressed with a stamp. The obtained filling level is recorded. The measurement geometry is calculated from the mass and filling level of the sample and the diameter of the measuring beaker. The compressed density is commonly around 0,5 $g \cdot cm^{-3}$.

### 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 [5] and in the literature [6, 7, 8].

### 4.2 Background

Background spectra need to be recorded at regular intervals to check the gamma spectrometric measurement system for contaminations. Measuring periods for this purpose should be at least 1,5 days to 3 days. It is recommended to obtain a mean value and standard deviation of the net count rate of each individual background gamma-ray

energy from two or three background spectra. The K-40 background count rate is of particular importance here, as it is used to plausibly check the measurement results.

**Note:**
Typically, the specific activity of K-40 in fish flesh is about 110 $Bq \cdot kg^{-1}$ FM, for whole fish it is about 80 $Bq \cdot kg^{-1}$ FM. Therefore, a significant contribution in the pulse height spectrum of a measuring source can be traced back to the compton spectrum of the K-40; this contribution can increase the pulse height spectrum of the measuring source by up to three times compared to the background spectrum

For further considerations, reference is made to the General Chapter γ-SPEKT/NULLEF of this Procedures' Manual [9].

## 4.3 Calibration

Usually, a calibration source similar in composition and geometry to the measuring source is used for calibration. Suitable calibration sources for fish ash are not available; therefore, the calibration for ash has to be carried out using other calibration media. The detection efficiency in the specific calibration medium $\varepsilon_{\mathrm{Kal}}(E)$ takes into account the coincidence correction $f_1$, the correction of the self-attenuation for the calibration medium $f_2$, the correction factor for the radioactive decay during the measurement, $f_3$, and the correction factor for the radioactive decay based on the reference time of the activity normal, $f_4$. In addition, an energy-dependent correction factor for self-attenuation of the ash $f_5(E)$ is required.

The detection efficiency in ash $\varepsilon_{\mathrm{A}}(E)$ is calculated according to Equation (1):

$$\varepsilon_{\mathrm{A}}(E) = \frac{\varepsilon_{\mathrm{Kal}}(E)}{f_5(E)} \tag{1}$$

The relative standard uncertainty is determined by Equation (2)

$$u_{\mathrm{rel}}(\varepsilon_{\mathrm{A}}) = \sqrt{u_{\mathrm{rel}}^2(\varepsilon_{\mathrm{Kal}}) + u_{\mathrm{rel}}^2(f_5)} \tag{2}$$

For further considerations on the calibration of a gamma spectrometry measurement system, required correction factors and filling level dependent detection efficiencies, reference is made to the General Chapter γ-SPEKT/GRUNDL of this Procedures' Manual [5] and the literature [10, 11, 12].

## 4.4 Measurement

For the measurement, a commercially available gamma spectrometry measurement system as well as cylindrical measuring beakers made of plastic, e. g. polyvinyl chloride (PVC), with plane bottom are used for analysis.

**Note:**
Compared to other plastic materials, PVC has got the advantage that the detection efficiency for X-ray energies is reduced due to the higher absorption of smaller energies. This reduces coincidence effects.

The measurement is normally carried out overnight. The measuring period may be enlarged, depending on the scope of the analysis or the required detection limit (see Section 6.2).

# 5 Calculation of the results

## 5.1 Output quantity

When determining specific activities of radionuclides in ash from fish flesh, interferences due to gamma lines of different radionuclides are scarcely occurring. Therefore, specific activities of radionuclides may be calculated either from a single gamma line (see Chapter 5.1.1) or, in the case of multiple-line radionuclides, by using a weighted mean (see Chapter 5.1.2).

If interferences occur, reference is made to the General Chapter γ-SPEKT/INTERF of this Procedures' Manual [13].

### 5.1.1 Specific activity from a single gamma line

If a net count rate $R_{\mathrm{n,r}}$ at the gamma-ray energy of the radionuclide r has been detected, the resulting specific activity $a_{\mathrm{r}}$, relative to fresh mass (FM) and the date and time of sampling, is calculated according to Equation (3):

$$a_{\mathrm{r}} = \varphi \cdot R_{\mathrm{n,r}} = \frac{f_1 \cdot f_3 \cdot f_4}{\varepsilon_{\mathrm{A}} \cdot p_{\gamma} \cdot m_{\mathrm{A}} \cdot q_{\mathrm{F}}} \cdot \left(R_{\mathrm{g,r}} - R_{\mathrm{T,r}} - R_{0,\mathrm{r}}\right) \tag{3}$$

with

$$f_3 = \frac{\lambda_{\mathrm{r}} \cdot t_{\mathrm{m}}}{1 - \mathrm{e}^{-\lambda_{\mathrm{r}} \cdot t_{\mathrm{m}}}} \tag{4}$$

$$f_4 = \mathrm{e}^{\lambda_{\mathrm{r}} \cdot t_{\mathrm{A}}} \tag{5}$$

In the Equations (3) to (5) are:

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

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

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

$m_\mathrm{A}$ mass of the ash used for the measurement, in kg;

$p_\gamma$ emission intensity of the considered gamma line of the radionuclide r;

$q_\mathrm{F}$ ratio of fresh mass to ash mass;

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

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

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

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

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

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

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

### 5.1.2 Specific activity from multiple gamma lines

The specific activity $a_\mathrm{r}$ relative to fresh mass (FM) and the time of sampling of a number of $j$ intensities ($j$ greater than 1) at gamma-ray energies of radionuclide r sufficiently detected are calculated according to Equation (6):

$$a_\mathrm{r} = \varphi_m \cdot A_\mathrm{r} = \frac{f_3 \cdot f_4}{m_\mathrm{A} \cdot q_\mathrm{F}} \cdot A_\mathrm{r} \tag{6}$$

Herein, $A_\mathrm{r}$ represents the activity at the time of measurement, which is calculated as a weighted mean from the intensity of a number of single gamma-ray energies of nuclides,

$$A_\mathrm{r} = u^2(A_\mathrm{r}) \cdot \sum_j \frac{A_{\mathrm{r},j}}{u^2(A_{\mathrm{r},j})} \tag{7}$$

wherein the variance of the uncertainty $u^2(A_\mathrm{r})$ is given by Equation (8):

$$u^2(A_\mathrm{r}) = \frac{1}{\sum_j \frac{1}{u^2(A_{\mathrm{r},j})}} \tag{8}$$

The index $j$ in these equations identifies single gamma-ray energies. The activities $A_{\mathrm{r},j}$ of the individual gamma-ray energies are calculated according to Equation (3):

$$A_{\mathrm{r},j} = R_{\mathrm{n,r},j} \cdot \frac{f_{1,\mathrm{r},j}}{\varepsilon_{A,\mathrm{r},j} \cdot p_{\gamma,\mathrm{r},j}} = R_{\mathrm{n,r},j} \cdot \varphi_{\mathrm{r},j} \quad (9)$$

Herein are:

$f_{1,\mathrm{r},j}$ coincidence summing correction factor for the gamma-ray energy $j$;

$p_{\gamma,\mathrm{r},j}$ emission intensity of the gamma-ray energy $j$;

$R_{\mathrm{n,r},j}$ net count rate of the gamma-ray energy $j$ of the radionuclide r, in $s^{-1}$;

$\varepsilon_{\mathrm{A,r},j}$ detection efficiency of the gamma-ray energy $j$ in ash, in $Bq^{-1} \cdot s^{-1}$ (see Section 4.3);

$\varphi_m$ line-independent, mass-related calibration factor, in $kg^{-1}$;

$\varphi_{\mathrm{r},j}$ line specific 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.

### 5.2.1 Standard uncertainty of the specific activity from a single gamma line

The generalized expression to calculate the uncertainty of the net count rate is given in Equation (3), with the coefficients $\mu_k$ are to be determined according to Equation (8):

$$u^2(R_{\mathrm{n,r}}) = \mu_0 \cdot R_{\mathrm{n,r}}^2 + \mu_1 \cdot R_{\mathrm{n,r}} + \mu_{2,\mathrm{r}} \quad (10)$$

with the coefficients:

$$\mu_{0,\mathrm{r}} = 0 \quad (11)$$

$$\mu_{1,\mathrm{r}} = \frac{1}{t_{\mathrm{m}}} \quad (12)$$

$$\mu_{2,\mathrm{r}} = \frac{R_{\mathrm{T,r}} + R_{0,\mathrm{r}}}{t_{\mathrm{m}}} + u^2(R_{\mathrm{T,r}}) + u^2(R_{0,\mathrm{r}}) \quad (13)$$

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;

The relative standard uncertainty of the procedural calibration factor is determined according to Equation (3), where the uncertainty of the decay correction may be neglected:

$$u_{\mathrm{rel}}(\varphi) = \sqrt{u_{\mathrm{rel}}^2(f_1) + u_{\mathrm{rel}}^2(f_3) + u_{\mathrm{rel}}^2(f_4) + u_{\mathrm{rel}}^2(\varepsilon_{\mathrm{A}}) + u_{\mathrm{rel}}^2(p_\gamma) + u_{\mathrm{rel}}^2(m_{\mathrm{A}}) + u_{\mathrm{rel}}^2(q_{\mathrm{F}})} \quad (14)$$

The combined standard uncertainty of the specific activity $u(a_{\mathrm{r}})$ using Equation (3) is calculated as:

$$u(a_{\mathrm{r}}) = a_{\mathrm{r}} \cdot \sqrt{u_{\mathrm{rel}}^2(\varphi) + \frac{u^2(R_{\mathrm{n,r}})}{R_{\mathrm{n,r}}^2}} \quad (15)$$

The combined standard uncertainty of the specific activity of Cs-137 amounts to be less than 10 % in the IMIS routine measurement program [2], where the largest contribution originates from the net count rate.

### 5.2.2 Standard uncertainty of the specific activity from multiple gamma lines

The variances of $A_{\mathrm{r},j}$ are calculated according to Equation (16):

$$u^2(A_{\mathrm{r},j}) = \varphi_{\mathrm{r},j}^2 \cdot u^2(R_{\mathrm{n,r},j}) + R_{\mathrm{n,r},j}^2 \cdot u^2(\varphi_{\mathrm{r},j}) \quad (16)$$

When the net count rates are calculated using the trapezoidal background procedure, their standard uncertainties are calculated according to Equation (10). If peak fitting is used instead, that factor $(1+b/2L)$, representing the trapezoidal background procedure, changes into a factor $f_{\mathrm{B}}$, which depends on the method used for peak fitting:

$$\mu_{2,\mathrm{r},j} = \frac{R_{\mathrm{T,r},j}}{t_{\mathrm{m}}} \cdot f_{\mathrm{B}} + R_{0,\mathrm{r}} \cdot \left(\frac{1}{t_{\mathrm{m}}} + \frac{1}{t_0}\right) + \frac{R_{\mathrm{T},0,\mathrm{r}}}{t_0} \cdot \left(1 + \frac{b_0}{2 \cdot L_0}\right) \quad (17)$$

The factor $f_{\mathrm{B}}$ generally depends on the averaged background continuum per channel underneath the gamma-ray energy and on a ratio of count rates. The factor $f_{\mathrm{B}}$ may be approximated by a fixed value; see the calculation example in Section 7.1.2.

**Note:**
The value of the factor $f_{\mathrm{B}}$ is usually a little bit above one.

The standard uncertainty of the specific activity is calculated as follows:

$$u(a_\mathrm{r}) = a_\mathrm{r} \cdot \sqrt{u_\mathrm{rel}^2(\varphi_m) + u_\mathrm{rel}^2(A_\mathrm{r})} \tag{18}$$

with

$$u_\mathrm{rel}(\varphi_m) = \sqrt{u_\mathrm{rel}^2\,(m_\mathrm{A}) + u_\mathrm{rel}^2(q_\mathrm{F})} \tag{19}$$

For multi-line radionuclides such as Cs-134, the uncertainty contribution of the coincidence summing correction factor has to be taken into account, which may lead to additional few per cents in case of significant corrections. Further information to the coincidence summing correction factor is given in the General chapters γ–SPEKT/GRUNDL and γ-SPEKT/SUMESC of this Procedures' Manual [5, 14].

# 6 Characteristic limits of the procedure

The calculation of the characteristic limits follows the standard series ISO 11929 [15]. For further considerations, it is referred to the General Chapters CHAGR-ISO-01 and CHAGR-ISO-02 of this Procedures' Manual [16, 17].

## 6.1 Decision threshold

### 6.1.1.1 Decision threshold of the specific activity of single-line radionuclides

The decision threshold of the single-line radionuclide's specific activity $a_\mathrm{r}^*$, e. g. Cs-137, is determined using Equation (20):

$$a_\mathrm{r}^* = \varphi \cdot k_{1-\alpha} \cdot \sqrt{\mu_2} = \varphi \cdot k_{1-\alpha} \cdot \sqrt{\frac{1}{t_\mathrm{m}} \cdot \left(R_\mathrm{T,r} + R_\mathrm{0,r}\right) + u^2\left(R_\mathrm{T,r}\right) + u^2(R_\mathrm{0,r})} \tag{20}$$

Herein is:

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

### 6.1.1.2 Decision threshold of the specific activity of multi-line radionuclides

The decision threshold $a_\mathrm{r}^*$ for multi-line radionuclides, e. g. Cs-134, may be calculated directly by Equation (21):

$$a_\mathrm{r}^* = \varphi_m \cdot k_{1-\alpha} \cdot u(A_\mathrm{r} = 0) = \varphi_m \cdot k_{1-\alpha} \cdot \sqrt{\frac{1}{\sum_j \frac{t_\mathrm{m}}{\varphi_{\mathrm{r},j}^2 \cdot R_{\mathrm{T,r},j} \cdot f_\mathrm{B}}}} \tag{21}$$

## 6.2 Detection limit

### 6.2.1.1 Detection limit of the specific activity of single-line radionuclides

The detection limit $a_r^{\#}$ of single-line radionuclides is calculated with Equation (22) using the decision threshold of the radionuclide's specific activity $a_r^*$ obtained by Equation (20):

$$a_r^{\#} = a_r^* \cdot \frac{\psi}{\theta} \cdot \left[1 + \sqrt{1 - \frac{\theta}{\psi^2}\left(1 - \frac{k_{1-\beta}^2}{k_{1-\alpha}^2}\right)}\right] \tag{22}$$

with the auxiliary quantities:

$$\theta = 1 - k_{1-\beta}^2 \cdot u_{rel}^2(\varphi) \tag{23}$$

$$\psi = 1 + \frac{k_{1-\beta}^2}{2 \cdot a_r^*} \cdot \frac{\varphi}{t_m} \tag{24}$$

Herein is:

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

### 6.2.1.2 Detection limit of the specific activity of multi-line radionuclides

The detection limit of the specific activity of multi-line radionuclides $a_r^{\#}$, e. g. Cs-134, is estimated according to Equation (25).

$$a_r^{\#} \approx a_r^* + k_{1-\beta} \cdot u(a_r^{\#\prime}) \tag{25}$$

with the standard uncertainty of an iterated specific activity $u(a_r^{\#'})$:

$$u(a_r^{\#\prime}) = \sqrt{\left(\frac{a_r^*}{k_{1-\alpha}}\right) + \left[u^2(a_r) - \left(\frac{a_r^*}{k_{1-\alpha}}\right)^2\right] \cdot \frac{a_r^{\#\prime}}{a_r}} \tag{26}$$

## 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). Project files for the software UncertRadio are available on the website of this Procedures' Manual.

In case of single-line-radionuclides, the output quantity, the associated standard uncertainty and the characteristic limits can be calculated explicitly. For multi-line emitters, the output quantity and the associated standard uncertainty can still be calculated directly, while the characteristic limits can only be estimated. Therefore, a software supported calculation should be preferred.

## 7.1 Manual evaluation

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

### 7.1.1 Determining the specific Cs-137 activity

The following values are used for calculating the specific Cs-137 activity in 1,1 kg fish flesh (FM):

| | | | | | |
|---|---|---|---|---|---|
| $R_{\mathrm{n,Cs\text{-}137}}$ | = | $6{,}32\cdot10^{-3}$ s$^{-1}$; | $R_{\mathrm{T,Cs\text{-}137}}$ | = | $2{,}71\cdot10^{-3}$ s$^{-1}$; |
| $b/(2\cdot L)$ | = | 0,523; | | | |
| $m_{\mathrm{A}}$ | = | $13{,}17\cdot10^{-3}$ kg; | $u_{\mathrm{rel}}(m_{\mathrm{A}})$ | = | $4{,}0\cdot10^{-3}$; |
| $q_{\mathrm{F}}$ | = | 83,54; | $u_{\mathrm{rel}}(q_{\mathrm{F}})$ | = | $20{,}0\cdot10^{-3}$; |
| $p_{\gamma}$ | = | 0,8499; | $u_{\mathrm{rel}}(p_{\gamma})$ | = | $2{,}35\cdot10^{-3}$; |
| $\varepsilon_{\mathrm{W}}$ | = | $30{,}56\cdot10^{-3}$ Bq$^{-1}\cdot$s$^{-1}$; | $u_{\mathrm{rel}}(\varepsilon_{\mathrm{W}})$ | = | $29{,}0\cdot10^{-3}$; |
| $f_1$ | = | 1,000; | $u_{\mathrm{rel}}(f_1)$ | = | 0; |
| $f_5$ | = | 0,976; | $u_{\mathrm{rel}}(f_5)$ | = | $8{,}0\cdot10^{-3}$. |

The standard uncertainties of the following input quantities are negligible:

| | | | | | |
|---|---|---|---|---|---|
| $t_{\mathrm{A}}$ | = | $19{,}79\cdot10^{6}$ s; | $\lambda_{\mathrm{Cs\text{-}137}}$ | = | $0{,}7309\cdot10^{-9}$ s$^{-1}$; |
| $t_{\mathrm{m}}$ | = | $72{,}0\cdot10^{3}$ s. | | | |

According to the Equations (4) and (5), the correction factors $f_3$ and $f_4$ are

$$f_3 = \frac{0{,}7309\cdot10^{-9}\ \mathrm{s}^{-1}\cdot 72{,}0\cdot10^{3}\ \mathrm{s}}{1-\mathrm{e}^{-0{,}7309\cdot10^{-9}\ \mathrm{s}^{-1}\cdot 72{,}0\cdot10^{3}\ \mathrm{s}}} \approx 1{,}000$$

$$f_4 = \mathrm{e}^{0{,}7309\cdot10^{-9}\cdot 19{,}79\cdot10^{6}} \approx 1{,}015$$

The detection efficiency in ash $\varepsilon_{\mathrm{A}}$ and the relative standard uncertainty $u_{\mathrm{rel}}(\varepsilon_{\mathrm{A}})$ are calculated according to the Equations (1) and (2):

$$\varepsilon_{\mathrm{A}} = \frac{30{,}56\cdot10^{-3}}{0{,}976}\ \mathrm{Bq}^{-1}\cdot\mathrm{s}^{-1} \approx 31{,}31\cdot10^{-3}\ \mathrm{Bq}^{-1}\cdot\mathrm{s}^{-1}$$

$$u_{\mathrm{rel}}(\varepsilon_{\mathrm{A}}) = \sqrt{(8{,}0 \cdot 10^{-3})^2 + (29{,}0 \cdot 10^{-3})^2} = 30{,}08 \cdot 10^{-3}$$

The procedural calibration factor defined in Equation (3) is given by:

$$\varphi = \frac{1{,}0 \cdot 1{,}000 \cdot 1{,}015}{31{,}31 \cdot 10^{-3} \cdot 0{,}8499 \cdot 13{,}17 \cdot 10^{-3} \cdot 83{,}54}\ \mathrm{Bq \cdot s \cdot kg^{-1}} \approx\ 34{,}67\ \mathrm{Bq \cdot s \cdot kg^{-1}}$$

The specific activity $a_{\mathrm{Cs\text{-}137}}$ in the fish flesh relative to the fresh mass (FM) results from Equation (3):

$$a_{\mathrm{Cs-137}} = 34{,}67\ \mathrm{Bq \cdot s \cdot kg^{-1}} \cdot 6{,}32 \cdot 10^{-3}\ \mathrm{s^{-1}} \approx 219{,}1 \cdot 10^{-3}\ \mathrm{Bq \cdot kg^{-1}}\ (\mathrm{FM})$$

The relative standard uncertainty of the procedural calibration factor is calculated by Equation (14):

$$u_{\mathrm{rel}}\,(\varphi) = \sqrt{(30{,}08 \cdot 10^{-3})^2 + (2{,}35 \cdot 10^{-3})^2 + (4{,}0 \cdot 10^{-3})^2 + (20{,}0 \cdot 10^{-3})^2} \approx$$
$$\approx 36{,}42 \cdot 10^{-3}$$

With the values of the input quantities, the coefficient $\mu_2$ is obtained by Equation (13) with neglecting the last two terms:

$$\mu_2 = \frac{2{,}71 \cdot 10^{-3}\ \mathrm{s^{-1}}}{72{,}0 \cdot 10^{3}\ \mathrm{s}} \cdot (1 + 0{,}523) \approx 57{,}32 \cdot 10^{-9}\ \mathrm{s^{-2}}$$

The standard uncertainty of the net count rate is calculated according to Equation (10):

$$u\big(R_{\mathrm{n,Cs-137}}\big) = \sqrt{\frac{6{,}32 \cdot 10^{-3}\ \mathrm{s^{-1}}}{72{,}0 \cdot 10^{3}\ \mathrm{s}} + 57{,}32 \cdot 10^{-9}\ \mathrm{s^{-2}}} \approx\ 0{,}3809 \cdot 10^{-3}\ \mathrm{s^{-1}}$$

The relative standard uncertainty of the specific activity $u(a_{\mathrm{Cs\text{-}137}})$ is calculated according to Equation (15):

$$u(a_{\mathrm{Cs-137}}) = 219{,}1 \cdot 10^{-3}\ \mathrm{Bq \cdot kg^{-1}} \cdot \sqrt{(36{,}42 \cdot 10^{-3})^2 + \left(\frac{0{,}3809 \cdot 10^{-3}\ \mathrm{s^{-1}}}{6{,}32 \cdot 10^{-3}\ \mathrm{s^{-1}}}\right)^2} \approx$$
$$\approx 15{,}43 \cdot 10^{-3}\ \mathrm{Bq \cdot kg^{-1}}\ (\mathrm{FM})$$

Finally, the specific Cs-137 activity is obtained as:

$$a_{\mathrm{Cs-137}}\ =\ (219{,}1\ \pm\ 15{,}4) \cdot 10^{-3}\ \mathrm{Bq \cdot kg^{-1}}\ (\mathrm{FM})$$

Using this value, the decision threshold of the specific activity $a^{*}_{\mathrm{Cs\text{-}137}}$ is obtained from the procedural calibration factor $\varphi$ and the normal distribution quantile $k_{1\text{-}\alpha}$ = 3:

$$a^{*}_{\mathrm{Cs-137}} = 34{,}67\ \mathrm{Bq \cdot s \cdot kg^{-1}} \cdot 3{,}0 \cdot \sqrt{57{,}32 \cdot 10^{-9}\ \mathrm{s^{-2}}} \approx 24{,}90 \cdot 10^{-3}\ \mathrm{Bq \cdot kg^{-1}}\ (\mathrm{FM})$$

According to Equation (22), the detection limit of the specific activity $a^{\#}_{\mathrm{Cs\text{-}137}}$ is

$$a^{\#}_{\mathrm{Cs-137}} = 24{,}90 \cdot 10^{-3}\ \mathrm{Bq \cdot kg^{-1}} \cdot \frac{1{,}026}{0{,}9964} \cdot \left[1 + \sqrt{1 - \frac{0{,}9964}{1{,}026^2} \cdot \left(1 - \frac{1{,}645^2}{3{,}0^2}\right)}\right] \approx$$

$$\approx 40{,}56 \cdot 10^{-3}\ \mathrm{Bq \cdot kg^{-1}}\ (\mathrm{FM})$$

with values of the auxiliary quantities and the normal distribution quantile $k_{1\text{-}\beta}$ = 1,645:

$$\theta = 1 - 1{,}645^2 \cdot (36{,}42 \cdot 10^{-3})^2 \approx 0{,}9964$$

$$\psi = 1 + \frac{1{,}645^2}{2 \cdot 24{,}90 \cdot 10^{-3}\ \mathrm{Bq \cdot kg^{-1}}} \cdot \frac{34{,}67\ \mathrm{Bq \cdot s \cdot kg^{-1}}}{72{,}0 \cdot 10^{3}\ \mathrm{s}} \approx 1{,}026$$

### 7.1.2 Determining the specific Cs-134 activity

For calculating the specific Cs-134 activity, the two main gamma energies at 604,7 keV and 795,9 keV were evaluated in a sample 3,7 kg (FM) of flesh from North Atlantic cod off Greenland, whose ash was measured over a period of about 15 days on a Germanium detector with relative efficiency of 35 %. The numerical values used for the calculations are the following:

| | | | | | |
|---|---|---|---|---|---|
| $m_{\mathrm{A}}$ | = | 51,21·10$^{-3}$ kg; | $u_{\mathrm{rel}}(m_{\mathrm{A}})$ | = | 4,0·10$^{-3}$; |
| $q_{\mathrm{F}}$ | = | 72,4; | $u_{\mathrm{rel}}(q_{\mathrm{F}})$ | = | 20,0·10$^{-3}$; |

The values for calculating the activities of the two Cs-134 gamma lines are:

***1. Gamma line at the energy of 604,7 keV***

| | | | | | |
|---|---|---|---|---|---|
| $R_{\mathrm{n,Cs\text{-}134,1}}$ | = | 0,4796·10$^{-3}$ s$^{-1}$; | $R_{\mathrm{T,Cs\text{-}134,1}}$ | = | 4,973·10$^{-3}$ s$^{-1}$; |
| $p_{\gamma,\mathrm{Cs\text{-}134,1}}$ | = | 0,9763; | $u_{\mathrm{rel}}(p_{\gamma,\mathrm{Cs\text{-}134,1}})$ | = | 0,8194·10$^{-3}$; |
| $\varepsilon_{\mathrm{w,Cs\text{-}134,1}}$ | = | 20,71·10$^{-3}$ Bq$^{-1}$·s$^{-1}$; | $u_{\mathrm{rel}}(\varepsilon_{\mathrm{w,Cs\text{-}134,1}})$ | = | 19,5·10$^{-3}$; |
| $f_{1,\mathrm{Cs\text{-}134,1}}$ | = | 1,133; | $u_{\mathrm{rel}}(f_{1,\mathrm{Cs\text{-}134,1}})$ | = | 12,09·10$^{-3}$; |
| $f_{5,\mathrm{Cs\text{-}134,1}}$ | = | 0,9243; | $u_{\mathrm{rel}}(f_{5,\mathrm{Cs\text{-}134,1}})$ | = | 10,0·10$^{-3}$. |

***2. Gamma line at the energy of 795,9 keV***

| | | | | | |
|---|---|---|---|---|---|
| $R_{\mathrm{n,Cs\text{-}134,2}}$ | = | 0,3835·10$^{-3}$ s$^{-1}$; | $R_{\mathrm{T,Cs\text{-}134,2}}$ | = | 4,961·10$^{-3}$ s$^{-1}$; |
| $p_{\gamma,\mathrm{Cs\text{-}134,2}}$ | = | 0,854; | $u_{\mathrm{rel}}(p_{\gamma,\mathrm{Cs\text{-}134,2}})$ | = | 1,053·10$^{-3}$; |
| $\varepsilon_{\mathrm{w,Cs\text{-}134,2}}$ | = | 16,38·10$^{-3}$ Bq$^{-1}$·s$^{-1}$; | $u_{\mathrm{rel}}(\varepsilon_{\mathrm{w,Cs\text{-}134,2}})$ | = | 16,32·10$^{-3}$; |

| | | | | | |
|---|---|---|---|---|---|
| $f_{1,\text{Cs-134,2}}$ | = | 1,137; | $u_{\text{rel}}(f_{1,\text{Cs-134,2}})$ | = | $14{,}15 \cdot 10^{-3}$; |
| $f_{5,\text{Cs-134,2}}$ | = | 0,932; | $u_{\text{rel}}(f_{5,\text{Cs-134,2}})$ | = | $10{,}0 \cdot 10^{-3}$. |

The standard uncertainties of the following input quantities are negligible:

| | | | | | |
|---|---|---|---|---|---|
| $t_{\text{A}}$ | = | $13{,}65 \cdot 10^{6}$ s; | $\lambda_{\text{Cs-134}}$ | = | $10{,}64 \cdot 10^{-9}$ s$^{-1}$; |
| $t_{\text{m}}$ | = | $1{,}314 \cdot 10^{6}$ s; | $f_{\text{B}}$ | = | 1,08. |

According to the Equations (4) and (5), the correction factors $f_3$ and $f_4$ are

$$f_3 = \frac{10{,}64 \cdot 10^{-9}\ \text{s}^{-1} \cdot 1{,}314 \cdot 10^{6}\ \text{s}}{1 - \text{e}^{-10{,}64 \cdot 10^{-9}\ \text{s}^{-1} \cdot 1{,}314 \cdot 10^{6}\ \text{s}}} \approx 1{,}007$$

$$f_4 = \text{e}^{10{,}64 \cdot 10^{-9} \cdot 13{,}65 \cdot 10^{6}} \approx 1{,}156$$

The factor $\varphi_{\text{m}}$ and its relative standard uncertainty $u_{\text{rel}}(\varphi_{\text{m}})$ are obtained according the Equations (6) and (19).

$$\varphi_m = \frac{1{,}007 \cdot 1{,}156}{51{,}21 \cdot 10^{-3}\ \text{kg} \cdot 72{,}4} \approx 314{,}0 \cdot 10^{-3}\ \text{kg}^{-1}$$

$$u_{\text{rel}}(\varphi_m) = \sqrt{(4{,}0 \cdot 10^{-3})^2 + (20{,}0 \cdot 10^{-3})^2} \approx 20{,}40 \cdot 10^{-3}$$

With the line specific calibration factors $\varphi_{\text{r},j}$, the activities in each of both Cs-134-gamma lines are calculated according Equation (9):

$$A_{\text{Cs-134,1}} = 0{,}4796 \cdot 10^{-3}\ \text{s}^{-1} \cdot \frac{1{,}133 \cdot 0{,}9243}{20{,}71 \cdot 10^{-3}\ \text{Bq}^{-1} \cdot \text{s}^{-1} \cdot 0{,}9763} \approx$$

$$\approx 0{,}4796 \cdot 10^{-3} \cdot 51{,}79\ \text{Bq} \approx 24{,}84 \cdot 10^{-3}\ \text{Bq}$$

$$A_{\text{Cs-134,2}} = 0{,}3835 \cdot 10^{-3}\ \text{s}^{-1} \cdot \frac{1{,}137 \cdot 0{,}932}{16{,}38 \cdot 10^{-3}\ \text{Bq}^{-1} \cdot \text{s}^{-1} \cdot 0{,}854} \approx$$

$$\approx 0{,}3835 \cdot 10^{-3} \cdot 75{,}75\ \text{Bq} \approx 29{,}05 \cdot 10^{-3}\ \text{Bq}$$

The standard uncertainties of both net count rates are calculated by Equations (10) and (17) neglecting the last two terms in Equation (17):

$$u\left(R_{\text{n,Cs-134,1}}\right) = \sqrt{\frac{0{,}4796 \cdot 10^{-3}\ \text{s}^{-1}}{1{,}314 \cdot 10^{6}\ \text{s}} + \frac{4{,}973 \cdot 10^{-3}\ \text{s}^{-1}}{1{,}314 \cdot 10^{6}\ \text{s}} \cdot 1{,}08} \approx 66{,}73 \cdot 10^{-6}\ \text{s}^{-1}$$

$$u\left(R_{\text{n,Cs-134,2}}\right) = \sqrt{\frac{0{,}3835 \cdot 10^{-3}\ \text{s}^{-1}}{1{,}314 \cdot 10^{6}\ \text{s}} + \frac{4{,}961 \cdot 10^{-3}\ \text{s}^{-1}}{1{,}314 \cdot 10^{6}\ \text{s}} \cdot 1{,}08} \approx 66{,}10 \cdot 10^{-6}\ \text{s}^{-1}$$

The relative standard uncertainties of both line specific calibration factors $u_{\mathrm{rel}}(\varphi_{\mathrm{r},j})$ result in:

$$u_{\mathrm{rel}}\left(\varphi_{\mathrm{Cs-134,1}}\right) = \sqrt{(12{,}09^2 + 10{,}0^2 + 19{,}50^2 + 0{,}8194^2) \cdot (10^{-3})^2} \approx 25{,}04 \cdot 10^{-3}$$

$$u_{\mathrm{rel}}\left(\varphi_{\mathrm{Cs-134,2}}\right) = \sqrt{(14{,}15^2 + 10{,}0^2 + 16{,}32^2 + 1{,}053^2) \cdot (10^{-3})^2} \approx 23{,}83 \cdot 10^{-3}$$

With these values, the variances of the activities of the two lines are calculated according to Equation (16):

$$u^2\left(A_{\mathrm{Cs-134,1}}\right) = [51{,}79^2 \cdot (66{,}73 \cdot 10^{-6})^2 + (0{,}4796 \cdot 10^{-3})^2 \cdot (25{,}04 \cdot 10^{-3})^2]\ \mathrm{Bq}^2 \approx$$
$$\approx 11{,}94 \cdot 10^{-6}\ \mathrm{Bq}^2$$

$$u^2\left(A_{\mathrm{Cs-134,2}}\right) = [75{,}75^2 \cdot (66{,}10 \cdot 10^{-6})^2 + (0{,}3835 \cdot 10^{-3})^2 \cdot (23{,}83 \cdot 10^{-3})^2]\ \mathrm{Bq}^2 \approx$$
$$\approx 25{,}07 \cdot 10^{-6}\ \mathrm{Bq}^2$$

Now, the variance of the Cs-134 activity is calculated according to Equation (8) followed by the calculation of the standard uncertainty:

$$u^2(A_{\mathrm{Cs-134}}) = \frac{1}{\frac{1}{11{,}94 \cdot 10^{-6}\ \mathrm{Bq}^2} + \frac{1}{25{,}07 \cdot 10^{-6}\ \mathrm{Bq}^2}} \approx 8{,}088 \cdot 10^{-6}\ \mathrm{Bq}^2$$

$$u(A_{\mathrm{Cs-134}}) = 2{,}844 \cdot 10^{-3}\ \mathrm{Bq}$$

Afterwards, the activity of Cs-134 $A_{\mathrm{Cs\text{-}134}}$ is obtained using Equation (7):

$$A_{\mathrm{Cs-134}} = 8{,}088 \cdot 10^{-6}\ \mathrm{Bq}^2 \cdot \left(\frac{24{,}84 \cdot 10^{-3}\ \mathrm{Bq}}{11{,}94 \cdot 10^{-6}\ \mathrm{Bq}^2} + \frac{29{,}05 \cdot 10^{-3}\ \mathrm{Bq}}{25{,}07 \cdot 10^{-6}\ \mathrm{Bq}^2}\right) \approx$$
$$\approx 8{,}088 \cdot 10^{-6} \cdot\ 3{,}239 \cdot 10^{3}\ \mathrm{Bq} \approx 26{,}20 \cdot 10^{-3}\ \mathrm{Bq}$$

Finally, the value and the associated standard uncertainty of the specific Cs-134 activity are determined according to Equations (6) and (18):

$$a_{\mathrm{Cs-134}} = 314{,}0 \cdot 10^{-3}\ \mathrm{kg}^{-1} \cdot 26{,}20 \cdot 10^{-3}\ \mathrm{Bq} \approx 8{,}227 \cdot 10^{-3}\ \mathrm{Bq} \cdot \mathrm{kg}^{-1}\ (\mathrm{FM})$$

$$u(a_{\mathrm{Cs-134}}) = 8{,}227 \cdot 10^{-3}\ \mathrm{Bq} \cdot \mathrm{kg}^{-1} \cdot \sqrt{(20{,}40 \cdot 10^{-3})^2 + \left(\frac{2{,}844 \cdot 10^{-3}\ \mathrm{Bq}}{26{,}20 \cdot 10^{-3}\ \mathrm{Bq}}\right)^2} \approx$$
$$\approx 0{,}9087 \cdot 10^{-3}\ \mathrm{Bq} \cdot \mathrm{kg}^{-1}\ (\mathrm{FM})$$

First, the following auxiliary quantity from Equation (21) is calculated using the values of the quantities mentioned above and the normal distribution quantile $k_{1-\alpha}$ = 3:

$$\frac{1{,}314 \cdot 10^6}{51{,}79^2 \cdot 4{,}973 \cdot 10^{-3} \cdot 1{,}08}\ \mathrm{Bq}^{-2} + \frac{1{,}314 \cdot 10^6}{75{,}75^2 \cdot 4{,}961 \cdot 10^{-3} \cdot 1{,}08}\ \mathrm{Bq}^{-2} \approx$$

$$\approx 91{,}21 \cdot 10^3\ \mathrm{Bq}^{-2} + 42{,}74 \cdot 10^3\ \mathrm{Bq}^{-2} \approx 134{,}0 \cdot 10^3\ \mathrm{Bq}^{-2}$$

Therewith, the decision threshold of the specific Cs-134 activity amounts to:

$$a^*_{\mathrm{Cs-134}} = 314{,}0 \cdot 10^{-3} \cdot 3{,}0 \cdot \sqrt{\frac{1}{134{,}0 \cdot 10^3}}\ \mathrm{Bq \cdot kg^{-1}} \approx 2{,}573 \cdot 10^{-3}\ \mathrm{Bq \cdot\ kg^{-1}}\ (\mathrm{FM})$$

This decision threshold value agrees quite well with that value obtained from using UncertRadio (see Section 7.2.2). It is smaller compared to the two single-line decision threshold values, which are 3,12 mBq·kg$^{-1}$ and 4,55 mBq·kg$^{-1}$, respectively.

The detection limit of the specific Cs-134 activity is estimated with a first step of iteration according to Equations (25) and (26) as follows:

$$\left(a_{\mathrm{r}}^{\#\prime}\right) = \left\{\left(\frac{2{,}573 \cdot 10^{-3}\ \mathrm{Bq \cdot\ kg^{-1}}}{3{,}0}\right)^2 + \frac{3{,}0 + 1{,}645}{3{,}0} \cdot \frac{2{,}573 \cdot 10^{-3}\ \mathrm{Bq \cdot\ kg^{-1}}}{8{,}227 \cdot 10^{-3}\ \mathrm{Bq \cdot kg^{-1}}} \cdot \right.$$

$$\left. \cdot \left[(0{,}9087 \cdot 10^{-3}\ \mathrm{Bq \cdot kg^{-1}})^2 - \left(\frac{2{,}573 \cdot 10^{-3}\ \mathrm{Bq \cdot\ kg^{-1}}}{3{,}0}\right)^2\right]\right\}^{1/2} \approx$$

$$\approx \{0{,}7356 \cdot 10^{-6}\ \mathrm{Bq^2 \cdot kg^{-2}} + 0{,}4842 \cdot 0{,}092 \cdot 10^{-6}\ \mathrm{Bq^2 \cdot kg^{-2}}\}^{1/2} \approx$$

$$\approx 0{,}8838 \cdot 10^{-3}\ \mathrm{Bq \cdot kg^{-1}}\ (\mathrm{FM})$$

$$a^{\#}_{\mathrm{Cs-134}} \approx\ 2{,}573 \cdot 10^{-3} + 1{,}645 \cdot 0{,}8838 \cdot 10^{-3}\ \mathrm{Bq \cdot kg^{-1}} \approx$$

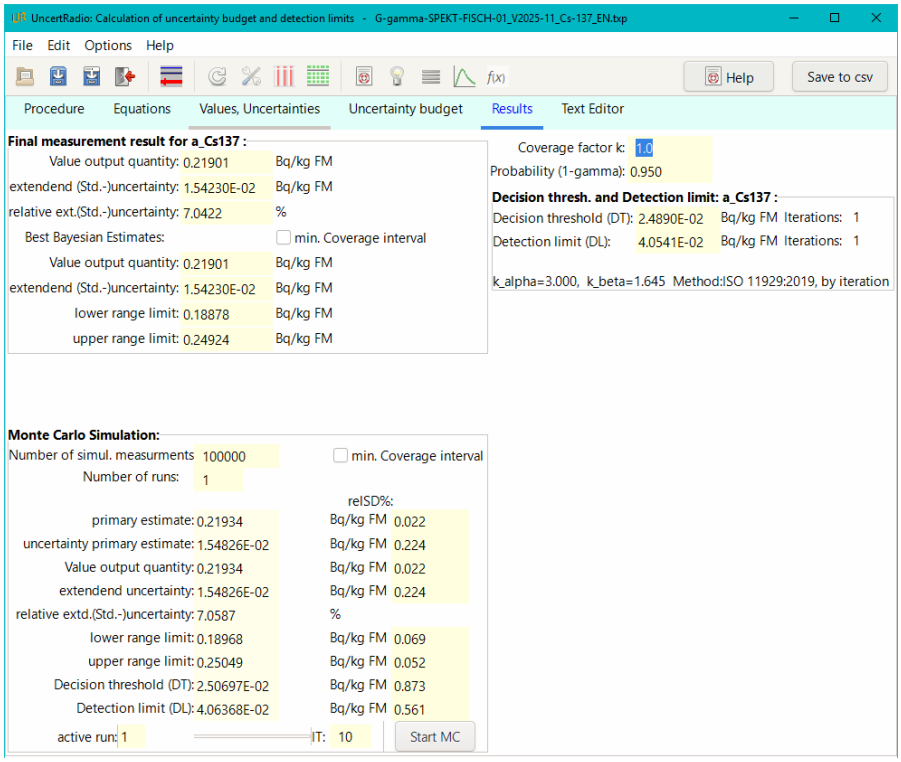
$$\approx 4{,}027 \cdot 10^{-3}\ \mathrm{Bq \cdot kg^{-1}}\ (\mathrm{FM})$$

This iteratively estimated detection limit is in good agreement with the result of the software UncertRadio, 4,031·10$^{-3}$ Bq·kg$^{-1}$ (see Section 7.2.2).

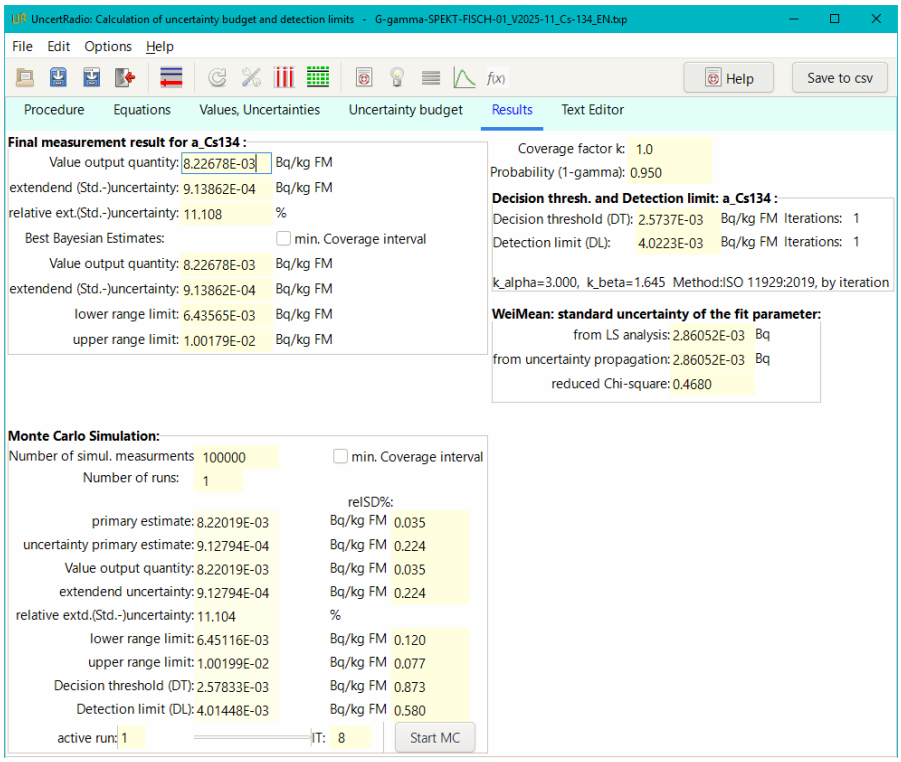
## 7.2 Software supported evaluation

In the following, the result pages of the UncertRadio project file corresponding to this procedure are shown. The files are available on the website of this Procedures' Manual.

### 7.2.1 View of the UncertRadio result page concerning Cs-137




### 7.2.2 View of the UncertRadio result page concerning Cs-134



# 8 Catalogue of the chemicals und equipment

## 8.1 Chemicals

The chemicals used shall be of analytically pure quality.

— Cleaning agent: strong alkaline cleaning agent, pH value above 13, diluted to 2 %.

## 8.2 Equipment

Besides the ordinary equipment of a radiochemical laboratory, the following equipment is used for the procedure.

### 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.
— incineration ovens with catalytical treatment of exhaust gases; organic exhaust gases shall largely be burned to carbon dioxide und water;
— stainless steel bowls (V4A) or ceramic bowls adapted in size to the internal volume of the oven,
— transparent paper with an areal density of about 90 $g \cdot m^{-2}$ as backing material for the stainless steel containers.

### 8.2.3 Calibration and activity determination

— gamma mitting radionuclides, preferably single-line emitters; if applicable, within a single solution covering the whole energy range to be calibrated;
— cylindrical plastic containers, e. g. made from PVC, with an inner diameter of about 7 cm and a capacity of ca. 220 ml, preferably with plane bottom;
— Gamma spectrometric measurement system, e. g. with a high-purity germanium-semiconductor detector with a relative efficiency preferably between 20 % and 60 %

relative to a 3'' x 3'' NaI(Tl)-crystal and a full-width-at-half-maximum less than 2,0 keV for the 1,33 MeV gamma-line of Co-60.

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