

Procedure for determining the specific activities of radionuclides in crustaceans (shrimps) by gamma spectrometry

G- γ -SPEKT-KRUST-01

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1 Scope

The procedure described below is used to examine crustacean meat. Specifically, the specific activity of gamma-ray emitting radionuclides in the flesh of North Sea shrimp (*Crangon crangon*) is recorded in accordance with the Radiation Protection Act (StrlSchG) [1] in the IMIS routine measurement programme [2]. The procedure is particularly suitable for low-level measurements in the context of radioecological investigations, as lower detection limits are achieved as a result of prior incineration.

2 Sampling

North Sea shrimps are mainly caught between spring and late autumn. Sampling is possible during the winter months, provided it is not hindered by ice. Fishing cooperatives or prawn fishermen are usually commissioned to take samples. Shrimp fishermen usually provide precise information about the origin of the shrimp, i. e. the sea coordinates and the date and time of the catch. If shrimp are purchased from a fishing cooperative, the sampler must obtain precise information about their origin. If fish and fishery products, including shrimp, are purchased on the open market, they must be labelled with at least the commercial name (species name) and the catch area in accordance with European Union Regulation 1379/2013 [3].

The shrimps are usually cooked on board, which makes it easier to peel them in the laboratory to produce a ready-to-eat sample. Commercially available products are usually ready to eat, i.e. cooked and peeled. The disadvantage here is that the exact catch coordinates are lost because the shrimps are usually peeled abroad. Additives such as ascorbic acid or sulphites are added to the prawn meat to improve its shelf life.

Ten kilograms of cooked shrimp or three kilograms of cooked and peeled shrimp are required for subsequent analysis. Cooked and peeled shrimp are stored refrigerated or frozen until processing. Cooked but unpeeled shrimp may only be refrigerated, not frozen, to facilitate subsequent peeling.

3 Analysis

3.1 Principle of the procedure

The ready-to-eat shrimp meat is first dried and then ashed at a maximum oven temperature of 420 °C. The homogenised ash is measured using gamma spectrometry.

Note:

The amount of ash is usually sufficient for subsequent Sr-90 and transuranic determination.

The specific activity of iodine isotopes cannot be quantitatively determined using this procedure due to the low boiling point of iodine and its volatile compounds; for this, please refer to the Procedure G- γ -SPEKT-KRUST-02.

3.2 Sample preparation

All tools used for preparing the ashes, e. g. stainless steel and ceramic bowls, as well as measuring cups, must be cleaned with a cleaning solution before each use.

In the laboratory, ensure that the prawns are kept sufficiently cool during peeling to prevent the prawn meat from decomposing. The ready-to-eat prawn meat is transferred to stainless steel bowls lined with ash-free transparent paper and the fresh mass is determined. The shrimp meat is then dried at a temperature of approximately 100 °C to 110 °C until the mass is constant, i. e. for one to two days, and the dry mass is determined. The ratio of fresh mass to dry mass of shrimp meat is approximately 4,0. The dried sample is ashed as described in Procedure G- γ -SPEKT-FISCH-01.

After the ash has cooled, the ash mass is determined, whereby the ratio of wet mass to ash mass for shrimp meat is between 30 and 50. This ratio is required for the subsequent calculation of the specific activity.

The ash is homogenised, transferred to a cylindrical measuring cup made of plastic, e. g. polyvinyl chloride (PVC), with as flat a bottom as possible, and carefully compressed with a stamp. The resulting filling level is documented.

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 geometry and the press density are determined from the ash mass, the filling level and the diameter of the measuring cup; experience shows that this is approximately 0,5 g·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 generally described in the General Chapter γ -SPEKT/GRUNDL of this Procedures' Manual [4] and in the literature [5, 6, 7]. Interferences in the pulse height spectrum are taken into account in accordance with the General Chapter γ -SPEKT/INTERF of this Procedures' Manual [8].

4.2 Background and blanks

As part of quality assurance and to record contamination of the gamma spectrometry measuring device, background effect spectra must be recorded regularly with a measurement duration of at least 1,5 days. A mean value must be calculated for each background effect line from two or three consecutive background effect measurements, and a standard deviation must be calculated from the dispersion of the individual values. The K-40 background effect count rate is of particular importance here, as it is used to verify the plausibility of the measurement results.

Note:

The specific K-40 activity in shrimp meat is usually 50 Bq·kg⁻¹ FM. Therefore, a significant contribution to the pulse height spectrum of a measurement source is due to the Compton spectrum of K-40; this contribution can increase the pulse height spectrum of the measurement source up to three times compared to the background effect spectrum.

For further details on determining the background, reference is given to the General Chapter γ -SPEKT/NULLEF of this Procedures' Manual [9].

4.3 Calibration

For details on calibration, reference is given to Procedure G- γ -SPEKT-FISCH-01.

4.4 Measurement

Gamma spectrometric measurements are usually carried out overnight. However, depending on the purpose of the measurement or the required detection limit (see also Section 6.2), the measurement period can be extended to up to one week. If multi-line nuclides are detected in the ashes, coincidence summations must be taken into account. For more information, reference is given to the General Chapter γ -SPEKT/GRUNDL of this Procedures' Manual [4] and the literature [10, 11, 12].

5 Calculation of the results

The equations for determining the specific activity of a single-line nuclide and its associated standard uncertainty are set out and explained below; for multi-line nuclides, reference is made to the Procedure G-γ-SPEKT-FISCH-01.

5.1 Output quantity

The specific activity a_r of the radionuclide r , based on the fresh mass (FM) and the time of sampling, is calculated according to Equation (1):

$$a_r = \varphi \cdot R_{n,r} = \frac{f_1 \cdot f_3 \cdot f_4}{\varepsilon_A \cdot p_\gamma \cdot m_A \cdot q_F} \cdot (R_{b,r} - R_{T,r} - R_{0,r}) \quad (1)$$

with

$$f_3 = \frac{\lambda_r \cdot t_m}{1 - e^{-\lambda_r \cdot t_m}} \quad (2)$$

$$f_4 = e^{\lambda_r \cdot t_A} \quad (3)$$

In the Equations (1) to (3) are:

- f_1 correction factor for coincidence summation; for an undisturbed line, $f_1 = 1$;
- f_3 correction factor for the decay of the radionuclide r during the measurement;
- f_4 correction factor for the decay of the radionuclide r related to a reference time;
- m_A mass of the ash used for the measurement, in kg;
- p_γ emission intensity of the gamma line of the radionuclide r ;
- q_F ratio between wet mass and ash mass;
- $R_{g,r}$ gross count rate of the gamma line of the radionuclide r , in s^{-1} ;
- $R_{n,r}$ net count rate of the radionuclide r , in s^{-1} ;
- $R_{T,r}$ background count rate underneath the gamma line of the radionuclide r , e. g. as count rate of a trapezoidal background, in s^{-1} ;
- $R_{0,r}$ net count rate at the gamma line of the radionuclide r in a background spectrum, in s^{-1} ;
- t_A time period between sampling and start of the measurement, in s;
- t_m duration of the measurement, in s;
- ε_A detection efficiency in ash, in $Bq^{-1} \cdot s^{-1}$;
- λ_r decay constant of the radionuclide r , in s^{-1} ;
- φ procedural calibration factor, in $Bq \cdot s \cdot kg^{-1}$.

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.

To calculate the variance of the net count rate $u^2(R_{n,r})$ according to Equation (4):

$$u^2(R_{n,r}) = \mu_{0,r} \cdot R_{n,r}^2 + \mu_{1,r} \cdot R_{n,r} + \mu_{2,r} \quad (4)$$

the coefficients μ_k are determined according to Equations (5) to (7):

$$\mu_{0,r} = 0 \quad (5)$$

$$\mu_{1,r} = \frac{1}{t_m} \quad (6)$$

$$\mu_{2,r} = \frac{R_{T,r}}{t_m} \cdot \left(1 + \frac{b}{2 \cdot L}\right) + R_{0,r} \cdot \left(\frac{1}{t_m} + \frac{1}{t_0}\right) + \frac{R_{T,0,r}}{t_0} \cdot \left(1 + \frac{b_0}{2 \cdot L_0}\right) \quad (7)$$

If there is no corresponding line in the background spectrum, the last two terms in Equation (7) are omitted.

Herein are:

- t_0 duration of the background measurement, in s;
- 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 used for background continuum determination to the left and to the right of the peaks, respectively;
- L_0 numbers of channels for the background spectrum used for background continuum determination to the left and to the right of the peaks, respectively;
- $R_{T,0,r}$ 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} .

The relative standard uncertainty of the process-related calibration factor is to be determined according to Equation (8), whereby the uncertainty contributions of the decay corrections are negligible:

$$u_{\text{rel}}(\varphi) = \sqrt{u_{\text{rel}}^2(f_1) + u_{\text{rel}}^2(f_3) + u_{\text{rel}}^2(f_4) + u_{\text{rel}}^2(\varepsilon_A) + u_{\text{rel}}^2(p_Y) + u_{\text{rel}}^2(m_A) + u_{\text{rel}}^2(q_F)} \quad (8)$$

The combined standard uncertainty of the specific activity $u(a_r)$ is calculated using Equation (1) according to Equation (9):

$$u(a_r) = a_r \cdot \sqrt{u_{\text{rel}}^2(\varphi) + \frac{u^2(R_{n,r})}{R_{n,r}^2}} \quad (9)$$

The combined standard uncertainty of the specific activity is usually less than 10 %, with the largest contribution resulting from the net count rate. If the self-attenuation correction is not applied to shrimp meat ash, this leads to an additional systematic error of up to 5 %.

For multi-line nuclides, the uncertainty contribution of the correction factor for coincidence summation must be taken into account, see the General Chapters γ -SPEKT/GRUNDL and γ -SPEKT/SUMESC of this Procedures' Manual [4, 13], which can amount to several percent in the case of significant corrections.

6 Characteristic limits of the procedure

The calculation of the characteristic limits follows the standard series ISO 11929 [14]. For further considerations, it is referred to the General Chapters CHAGR-ISO-01 and CHAGR-ISO-02 of this Procedures' Manual [15, 16].

6.1 Decision threshold

The decision threshold of the specific activity a_r^* is determined according to Equation (10):

$$a_r^* = \varphi \cdot k_{1-\alpha} \cdot \sqrt{\mu_2} = \varphi \cdot k_{1-\alpha} \cdot \sqrt{\frac{1}{t_m} \cdot (R_{T,r} + R_{0,r}) + u^2(R_{T,r}) + u^2(R_{0,r})} \quad (10)$$

with $k_{1-\alpha}$ as quantile of the normal distribution for the probability of the 1st kind error α .

6.2 Detection limit

The detection limit of a determination of the specific activity $a_r^\#$ is calculated using the detection limit a_r^* determined using Equation (10) according to the implicit Equation (11)

$$a_r^\# = a_r^* \cdot \frac{\psi}{\theta} \cdot \left[1 + \sqrt{1 - \frac{\theta}{\psi^2} \cdot \left(1 - \frac{k_{1-\beta}^2}{k_{1-\alpha}^2} \right)} \right] \quad (11)$$

with the auxiliary variables

$$\theta = 1 - k_{1-\beta}^2 \cdot u_{\text{rel}}^2(\varphi) \quad (12)$$

$$\psi = 1 + \frac{k_{1-\beta}^2}{2 \cdot a_r^*} \cdot \frac{\varphi}{t_m} \quad (13)$$

with $k_{1-\beta}$ as quantile of the normal distribution for the probability of the 2nd kind error β .

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.

Since the equations are the same as those used in the Procedure G-γ-SPEKT-FISCH-01, the examples are also transferable. Therefore, only one example for calculating the specific activity of Cs-137 is presented below.

7.1 Manual evaluation

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

The following numerical values are used to calculate the specific Cs-137 activity in 2,9 kg of shrimp meat (FM)

$R_{n,Cs-137}$	=	$2,467 \cdot 10^{-3} \text{ s}^{-1}$;	$R_{T,Cs-137}$	=	$6,316 \cdot 10^{-3} \text{ s}^{-1}$;
$b/(2 \cdot L)$	=	1,125;			
m_A	=	$68,76 \cdot 10^{-3} \text{ kg}$;	$u_{\text{rel}}(m_A)$	=	$4,0 \cdot 10^{-3}$;
q_F	=	42,46;	$u_{\text{rel}}(q_F)$	=	$20,0 \cdot 10^{-3}$;
p_γ	=	0,8499;	$u_{\text{rel}}(p_\gamma)$	=	$2,35 \cdot 10^{-3}$;
ε_A	=	$31,31 \cdot 10^{-3} \text{ Bq}^{-1} \cdot \text{s}^{-1}$;	$u_{\text{rel}}(\varepsilon_A)$	=	$30,08 \cdot 10^{-3}$;
f_1	=	1,000;	$u_{\text{rel}}(f_1)$	=	0.

The standard uncertainty of the following input variables is omissible:

t_A	=	$4,32 \cdot 10^6 \text{ s}$;	λ_{Cs-137}	=	$0,7309 \cdot 10^{-9} \text{ s}^{-1}$;
t_m	=	$576,0 \cdot 10^3 \text{ s}$.			

Equations (2) and (3) are used to calculate the correction factors f_3 and f_4 for the decay during the measurement period and in relation to the reference time point:

$$f_3 = \frac{0,7309 \cdot 10^{-9} \text{ s}^{-1} \cdot 72,0 \cdot 10^3 \text{ s}}{1 - e^{-0,7309 \cdot 10^{-9} \text{ s}^{-1} \cdot 72,0 \cdot 10^3 \text{ s}}} \approx 1,000$$

$$f_4 = e^{0,7309 \cdot 10^{-9} \cdot 4,32 \cdot 10^6} \approx 1,003$$

The value of the procedural calibration factor defined in Equation (1) is

$$\varphi = \frac{1,0 \cdot 1,000 \cdot 1,003}{31,31 \cdot 10^{-3} \cdot 0,8499 \cdot 68,76 \cdot 10^{-3} \cdot 42,46} \text{ Bq} \cdot \text{s} \cdot \text{kg}^{-1} \approx 12,91 \text{ Bq} \cdot \text{s} \cdot \text{kg}^{-1}$$

The specific activity $a_{\text{Cs-137}}$ in shrimp flesh referring to fresh mass (FM) according to Equation (1) calculates to:

$$a_{\text{Cs-137}} = 12,91 \text{ Bq} \cdot \text{s} \cdot \text{kg}^{-1} \cdot 2,467 \cdot 10^{-3} \text{ s}^{-1} \approx 31,85 \cdot 10^{-3} \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)}$$

The relative standard uncertainty of the procedural calibration factor is calculated according to Equation (8):

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

The coefficient μ_2 is determined using the numerical values of the input variables according to Equation (7), omitting the last two terms:

$$\mu_2 = \frac{6,316 \cdot 10^{-3} \text{ s}^{-1}}{576,0 \cdot 10^3 \text{ s}} \cdot (1 + 1,125) \approx 23,30 \cdot 10^{-9} \text{ s}^{-2}$$

The standard uncertainty of the net count rate is calculated according to Equation (9):

$$u(a_{\text{Cs-137}}) = 31,85 \cdot 10^{-3} \text{ Bq} \cdot \text{kg}^{-1} \cdot \sqrt{(36,42 \cdot 10^{-3})^2 + \left(\frac{0,1661 \cdot 10^{-3} \text{ s}^{-1}}{2,467 \cdot 10^{-3} \text{ s}^{-1}}\right)^2} \approx 2,438 \cdot 10^{-3} \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)}$$

This means that the specific Cs-137 activity is:

$$a_{\text{Cs-137}} = (31,9 \pm 2,4) \cdot 10^{-3} \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)}$$

The value of the decision threshold of the specific activity $a_{\text{Cs-137}}^*$ is calculated using the procedural calibration factor φ and the value of 3 for the quantile of the normal distribution for the probability of error of the first type, $k_{1-\alpha}$, with Equation (10):

$$a_{\text{Cs-137}}^* = 12,91 \text{ Bq} \cdot \text{s} \cdot \text{kg}^{-1} \cdot 3,0 \cdot \sqrt{23,30 \cdot 10^{-9} \text{ s}^{-2}} \approx 5,912 \cdot 10^{-3} \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)}$$

The detection limit of the specific activity $a_{\text{Cs-137}}^\#$ is calculated using the Quantile of the normal distribution for the probability of the error of the second type, $k_{1-\beta}$, of 1,645 and the values of the auxiliary variables

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

$$\psi = 1 + \frac{1,645^2}{2 \cdot 5,912 \cdot 10^{-3} \text{ Bq} \cdot \text{kg}^{-1}} \cdot \frac{12,91 \text{ Bq} \cdot \text{s} \cdot \text{kg}^{-1}}{576,0 \cdot 10^3 \text{ s}} \approx 1,005$$

according to Equation (11):

$$a_{\text{Cs-137}}^{\#} = 5,912 \cdot 10^{-3} \text{ Bq} \cdot \text{kg}^{-1} \cdot \frac{1,005}{0,9964} \cdot \left[1 + \sqrt{1 - \frac{0,9964}{1,005^2} \cdot \left(1 - \frac{1,645^2}{3,0^2} \right)} \right] \approx$$

$$\approx 9,284 \cdot 10^{-3} \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)}$$

7.2 Software supported evaluation

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

Final measurement result for a_Cs137 :

Value output quantity:	3.18655E-02	Bq/kg (FM)
extendend (Std.-)uncertainty:	2.43874E-03	Bq/kg (FM)
relative ext.(Std.-)uncertainty:	7.6532	%

Best Bayesian Estimates: ☐ min. Coverage interval

Value output quantity:	3.18655E-02	Bq/kg (FM)
extendend (Std.-)uncertainty:	2.43874E-03	Bq/kg (FM)
lower range limit:	2.70856E-02	Bq/kg (FM)
upper range limit:	3.66453E-02	Bq/kg (FM)

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

Decision thresh. and Detection limit: a_Cs137 :

Decision threshold (DT):	5.9141E-03	Bq/kg (FM)	Iterations: 1
Detection limit (DL):	9.2886E-03	Bq/kg (FM)	Iterations: 1

k_alpha=3.000, k_beta=1.645 Method:ISO 11929:2019, by iteration

Monte Carlo Simulation:

Number of simul. measurements: 100000 ☐ min. Coverage interval
Number of runs: 1

primary estimate:	3.19143E-02	Bq/kg (FM)	reISD%:	0.024
uncertainty primary estimate:	2.43007E-03	Bq/kg (FM)		0.224
Value output quantity:	3.19143E-02	Bq/kg (FM)		0.024
extendend uncertainty:	2.43007E-03	Bq/kg (FM)		0.224
relative extd.(Std.-)uncertainty:	7.6144	%		
lower range limit:	2.72842E-02	Bq/kg (FM)		0.075
upper range limit:	3.67867E-02	Bq/kg (FM)		0.056
Decision threshold (DT):	5.92983E-03	Bq/kg (FM)		0.873
Detection limit (DL):	9.29710E-03	Bq/kg (FM)		0.576

active run: 1 IT: 12 Start MC

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

This procedure requires an ordinary laboratory equipment and the following additional equipment.

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

- incineration oven 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·m⁻² as backing material for the stainless steel containers.

8.2.3 Calibration and activity determination

- gamma-ray emitting 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 spectrometry 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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