

**Procedure for determining the specific activity
of strontium-90 in animal feed and
vegetation samples
by liquid scintillation spectrometry
(Dicyclohexyl-18-crown-6 method)**

F-Sr-90-FUMI-04

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Procedure for determining the specific activity of strontium-90 in animal feed and vegetation samples by liquid scintillation spectrometry (Dicycloheyl-18-crown-6 method)

1 Scope

The procedure is suitable for monitoring the specific activity of strontium-90 (Sr-90) in food according to the German Radiation Protection Act (StrlSchG) [1] and the Guideline concerning Emission and Immission Monitoring of Nuclear Facilities (REI) [2]. With this procedure, the detection limits required in the General Administrative Regulation on the Integrated Measurement and Information System for the Monitoring of Environmental Radioactivity (AVV-IMIS) [3] can be achieved.

Note:

The mass reference of the specific activity of Sr-90 according to AVV-IMIS is dry mass for animal feed and feed raw materials, and fresh mass for pasture and meadow vegetation. In a few cases, it is necessary to refer the specific activity to dry mass. These different mass references are taken into account when calculating the results in Section 5.

2 Sampling

The sampling is carried out according to procedure F- γ -SPEKT-FUMI-01.

3 Analysis

3.1 Principle of the procedure

The principle of this procedure is described in procedure F-Sr-90-BODEN-02.

3.2 Sample preparation

The sample preparation is carried out according to procedure F-Sr-90-FUMI-03.

3.3 Radiochemical separation

The radiochemical separation is carried out according to procedure F-Sr-90-BODEN-02. The information provided at the beginning of Section 3.3 of the above-mentioned procedure serves as guidance.

Most ashes of animal feed and vegetation samples are almost completely dissolved after only two minutes when strontium is extracted conventionally with boiling diluted nitric

acid. If a residue remains after conventional extraction, a subsequent microwave-assisted extraction at 10 bar may be necessary.

Up to 10 g of ash are used for extraction.

Note:

In the liquid-liquid extraction described in step 3.3.2.2 of procedure F-Sr-90-BODEN-02, the phases occasionally separate only slowly. In such cases, the addition of a maximum of 5 ml of methanol leads to a rapid phase separation.

4 Measuring the activity

Information about the calibration and the measurement with the liquid scintillation spectrometer is given in the corresponding section of procedure F-Sr-90-MILCH-05.

5 Calculation of the results

The calculation of the analytical results is based on procedure F-Sr-90-FUMI-03. However, the count rates and the activity-related calibration factor φ_A must be determined

- in the same energy range and
- under the same quench conditions, i. e. the same quench factor for the measurement.

If Sr-85-tracer has been added to the sample to determine the chemical yield, possible contributions of the tracer to the count rate must be taken into account. In this case, the expected Sr-85 contribution in the energy range used to determine the activity of (Sr-90+Y-90) at the time of the measurement must be calculated and subtracted from the gross count rate R_g . Therefore, calculation examples are given for both methods of calculating the chemical yield.

5.1 Equations

5.1.1 Quantity

5.1.1.1 Counting source with addition of Sr-85

When Sr-85 is added to the sample, the expected Sr-85 contribution is determined in the energy range used to determine the specific activity of (Sr-90+Y-90) at the time of the measurement according to Equation (1):

$$R_{\text{Sr-85}} = \frac{A_{\text{Sr-85}}}{\varphi_{\text{A,Sr-85}}} \cdot \eta_{\text{Sr}} \cdot f_b = \frac{A_{\text{Sr-85}}}{\varphi_{\text{A,Sr-85}}} \cdot \eta_{\text{Sr}} \cdot e^{-\lambda_{\text{Sr-85}} \cdot t_b} \quad (1)$$

Herein are:

$R_{\text{Sr-85}}$ count rate of Sr-85, in s^{-1} ;

- $A_{\text{Sr-85}}$ activity of Sr-85 added to the sample, in Bq;
- $\varphi_{\text{A,Sr-85}}$ activity-related calibration factor of Sr-85, in Bq·s;
- f_{b} correction factor for the decay of the activity of Sr-85 for the time period between addition of the tracer to the sample and start of the measurement;
- $\lambda_{\text{Sr-85}}$ decay constant of Sr-85, in s^{-1} ;
- t_{b} time period between addition of the tracer to the sample and start of the measurement, in s;
- η_{Sr} chemical yield for strontium.

The specific activity of Sr-90 is calculated according to Equation (2). According to the legally required mass reference for the specific activity in the sample type, m in this equation stands for either the dry mass (m_{TM}) or the fresh mass (m_{FM}).

$$a = f_2 \cdot \frac{\varphi_{\text{A}}}{\eta_{\text{Sr}} \cdot m} \cdot R_{\text{n}} = e^{\lambda_{\text{Sr-90}} \cdot t_{\text{A}}} \cdot \frac{\varphi_{\text{A}}}{\eta_{\text{Sr}} \cdot m} \cdot [R_{\text{g}} - (R_0 + R_{\text{Sr-85}})] \quad (2)$$

Herein are:

- a specific activity of Sr-90, based on fresh mass, in $\text{Bq} \cdot \text{kg}^{-1}$;
- φ_{A} activity-related calibration factor, in Bq·s;
- t_{A} time period between sampling and start of the measurement, in s;
- f_2 correction factor for the decay of the activity of Sr-90 for the time period between sampling and start of the measurement. Correction is only required if the time period t_{A} is greater than 0,5 years;
- $\lambda_{\text{Sr-90}}$ decay constant of Sr-90, in s^{-1} ;
- R_{g} gross count rate, in s^{-1} ;
- R_0 background count rate, in s^{-1} ;
- R_{n} net count rate of the (Sr-90 + Y-90) counting source, in s^{-1} ;
- m mass of the sample used, in kg,
 for dry mass: $m_{\text{TM}} = m_{\text{a}} \cdot q_1$
 for fresh mass: $m_{\text{FM}} = m_{\text{a}} \cdot q_1 \cdot q_2$
 with: m_{a} mass of ash used, in kg;
 q_1 ratio dry mass to ash mass;
 q_2 ratio of fresh mass to dry mass.

Note:

In addition to the calculation, the sum ($R_0 + R_{\text{Sr-85}}$) of the contributions of the background and the Sr-85 can be determined empirically. For this purpose, the activity of Sr-85 in the counting source is determined by gamma spectrometry. The same activity of Sr-85 is added to the counting vial used for the background determination of the scintillation measurements. It is assumed that the same measurement conditions are maintained.

5.1.1.2 Counting source without addition of Sr-85

If no Sr-85 is added to the sample, but a control counting source is used to determine the chemical yield, the specific activity of Sr-90 is calculated according to Equation (3):

$$a = f_2 \cdot \frac{\varphi_A}{\eta_{\text{Sr}} \cdot m} \cdot R_n = e^{\lambda_{\text{Sr-90}} \cdot t_A} \cdot \frac{\varphi_A}{\eta_{\text{Sr}} \cdot m} \cdot (R_g - R_0) \quad (3)$$

5.1.2 Standard uncertainty of the quantity

5.1.2.1 Counting source with addition of Sr-85

The combined relative standard uncertainty of the specific activity $u(a) \cdot a^{-1}$ is calculated according to Equation (4):

$$\frac{u(a)}{a} = \left\{ \frac{1}{[R_g - (R_0 + R_{\text{Sr-85}})]^2} \cdot \left[\frac{R_g}{t_m} + \frac{R_0}{t_0} + u^2(R_{\text{Sr-85}}) \right] + \right. \\ \left. \left. + [u_{\text{rel}}^2(f_2) + u_{\text{rel}}^2(\varphi_A) + u_{\text{rel}}^2(\eta_{\text{Sr}}) + u_{\text{rel}}^2(m)] \right\}^{\frac{1}{2}} \quad (4)$$

with

$$u^2(R_{\text{Sr-85}}) = R_{\text{Sr-85}}^2 \cdot [u_{\text{rel}}^2(A_{\text{Sr-85}}) + u_{\text{rel}}^2(\varphi_{A, \text{Sr-85}}) + u_{\text{rel}}^2(\eta_{\text{Sr}}) + u_{\text{rel}}^2(f_b)] \quad (5)$$

Herein are:

- $u(a)$ standard uncertainty of the specific activity a at the time of sampling, based on fresh mass, in $\text{Bq} \cdot \text{kg}^{-1}$;
- $u(R_{\text{Sr-85}})$ standard uncertainty of the count rate of Sr-85, in s^{-1} ;
- $u_{\text{rel}}(A_{\text{Sr-85}})$ relative standard uncertainty of the added activity of Sr-85;
- $u_{\text{rel}}(\varphi_{A, \text{Sr-85}})$ relative standard uncertainty of the activity-related Sr-85 calibration factor;
- $u_{\text{rel}}(\varphi_A)$ relative standard uncertainty of the activity-related calibration factor;
- $u_{\text{rel}}(f_b)$ relative standard uncertainty of the correction factor for the decay of the activity of Sr-85 for the time period between addition of the tracer and start of the measurement;
- $u_{\text{rel}}(f_2)$ relative standard uncertainty of the correction factor for the decay of the activity of Sr-90 for the period between sampling and the beginning of the measurement;
- $u_{\text{rel}}(\eta_{\text{Sr}})$ relative standard uncertainty of the chemical yield for strontium;
- $u_{\text{rel}}(m)$ relative standard uncertainty of the mass of the sample used based on dry mass, $u_{\text{rel}}(m_{\text{TM}})$, or fresh mass $u_{\text{rel}}(m_{\text{FM}})$;

t_m	duration of measurement, in s;
t_0	duration of background measurement, in s.

For the calculation of the combined relative standard uncertainty of the specific activity, the contributions of the relative standard uncertainty of the correction factor $u_{\text{rel}}(f_2)$ and of the mass of the sample used $u_{\text{rel}}(m)$ can be neglected. For an energy range from 350 keV to 1000 keV used for the measurement of the specific activity of (Sr-90+Y-90) and an activity of Sr-85 of only a few Bq, the standard uncertainty $u(R_{\text{Sr-85}})$ is negligible in comparison to the standard uncertainty of the background $u(R_0)$ (see Section 5.2.1).

5.1.2.2 Counting source without addition of Sr-85

The combined relative standard uncertainty of the specific activity $u(a) \cdot a^{-1}$ is calculated according to Equation (6):

$$\frac{u(a)}{a} = \sqrt{\frac{1}{(R_g - R_0)^2} \cdot \left(\frac{R_g}{t_m} + \frac{R_0}{t_0} \right) + [u_{\text{rel}}^2(f_2) + u_{\text{rel}}^2(\varphi_A) + u_{\text{rel}}^2(\eta_{\text{Sr}}) + u_{\text{rel}}^2(m)]} \quad (6)$$

For the calculation of the combined relative standard uncertainty of the specific activity, the contributions of the relative standard uncertainty of the correction factor $u_{\text{rel}}(f_2)$ and the mass of the sample used $u_{\text{rel}}(m)$ may generally be neglected.

5.2 Worked example

5.2.1 Counting source with addition of Sr-85

In the worked examples of the Sections 5.2 and 6.2, the interim results and the result are given with four significant digits. Deviations from the calculated values are possible when using another number of significant digits.

An example for the calculation of the results based on the dry mass of the sample is given in procedure F-Sr-90-Boden-02. Therefore, an example based on the fresh mass of a pasture vegetation sample is presented here.

In this calculation example, 5,0 Bq Sr-85 were added to the sample 20 days prior to the start of the measurement. To calculate the specific activity of Sr-90, the energy range between 350 keV and 1000 keV in the pulse height spectrum was used.

Furthermore, the following values were obtained:

$\varphi_{\text{A,Sr-85}}$	=	4167 Bq·s;	$u(R_{\text{Sr-85}})$	=	0;
η_{Sr}	=	0,750;	$u_{\text{rel}}(\eta_{\text{Sr}})$	=	0,05;
f_b	=	0,808;	m_a	=	0,010 kg;

R_g	=	0,02813 s ⁻¹ ;	t_m	=	60·10 ³ s;
R_0	=	0,0160 s ⁻¹ ;	t_0	=	60·10 ³ s;
q_1	=	11,11;	q_2	=	5,0;
m_{FM}	=	0,555 kg;	$u_{rel}(m_{FM})$	=	0;
φ_A	=	2,632 Bq·s;	$u_{rel}(\varphi_A)$	=	0,04;
f_2	=	1,000;	$u_{rel}(f_2)$	=	0.

According to Equation (1), the count rate of Sr-85 is:

$$R_{Sr-85} = \frac{5,0}{4167} \cdot 0,750 \cdot 0,808 \text{ s}^{-1} = 0,7271 \cdot 10^{-3} \text{ s}^{-1}$$

According to Equation (2), the specific activity of Sr-90 is:

$$a = \frac{1,00 \cdot 2,632 \cdot [0,02813 - (0,0160 + 0,7271 \cdot 10^{-3})]}{0,750 \cdot 0,555} \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)} =$$

$$= 72,10 \cdot 10^{-3} \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)}$$

With the values above and with the corresponding additions to Equation (4) to take into account the contributions of Sr-85, the relative standard uncertainty of the specific activity of Sr-90 is:

$$\frac{u(a)}{a} = \sqrt{\frac{1}{[0,02813 - (0,0160 + 0,7271 \cdot 10^{-3})]^2} \cdot \left(\frac{0,02813}{60 \cdot 10^3} + \frac{0,0160}{60 \cdot 10^3}\right) + 0,04^2 + 0,05^2} =$$

$$= 0,09878$$

The specific activity of Sr-90 in the pasture vegetation sample at the time of sampling is:

$$a = (72,10 \pm 7,12) \cdot 10^{-3} \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)}$$

The evaluation can be carried out software supported as shown in Section 7.

5.2.2 Counting source without addition of Sr-85

In this worked example, the energy range between 20 keV and 1000 keV in the pulse height spectrum was used to calculate the specific activity of Sr-90. The values are:

R_g	=	0,1303 s ⁻¹ ;	t_m	=	60·10 ³ s;
R_0	=	0,0790 s ⁻¹ ;	t_0	=	60·10 ³ s;
m_a	=	0,010 kg;	m_{FM}	=	0,555 kg;

$$\begin{array}{llll}
 q_1 & = & 11,11; & q_2 & = & 5,00; \\
 \varphi_A & = & 0,585 \text{ Bq}\cdot\text{s}; & u_{\text{rel}}(\varphi_A) & = & 0,04; \\
 \eta_{\text{Sr}} & = & 0,750; & u_{\text{rel}}(\eta_{\text{Sr}}) & = & 0,05; \\
 f_2 & = & 1,000. & & &
 \end{array}$$

According to Equation (3), the specific activity of Sr-90 is

$$a = \frac{1,00 \cdot 0,585 \cdot (0,1303 - 0,0790)}{0,750 \cdot 0,555} \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)} = 72,10 \cdot 10^{-3} \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)}$$

With the above values, according to Equation (6), the relative standard uncertainty of the specific activity of Sr-90 is:

$$\frac{u(a)}{a} = \sqrt{\frac{1}{(0,1303 - 0,0790)^2} \cdot \left(\frac{0,1303}{60 \cdot 10^3} + \frac{0,0790}{60 \cdot 10^3} \right) + 0,04^2 + 0,05^2} = 0,0737$$

The specific activity of Sr-90 in the pasture vegetation sample at the time of sampling is:

$$a = (72,10 \pm 5,31) \cdot 10^{-3} \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)}$$

The evaluation can be carried out software supported as shown in Section 7.

5.3 Consideration of the uncertainties

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 combined standard uncertainty includes the standard statistical uncertainty and the standard uncertainties of the chemical separation, the yield determination and the calibration.

6 Characteristic limits of the procedure

The calculation of the characteristic limits follows the standard ISO 11929 series [4].

An Excel spreadsheet (see Section 7.1) as well as a project file for the software Uncert-Radio (see Section 7.2) are available on the website of this Procedures Manual.

Further considerations concerning the characteristic limits are to be found in the General Chapters ERK/NACHWEISGR-ISO-01 and ERK/NACHWEISGR-ISO-02 of this Procedures Manual.

The worked example considers the case that no detectable contribution of Sr-85 is to be expected in the test sample, i. e. no addition of Sr-85 to the sample, or the added tracer has already disintegrated sufficiently.

6.1 Equations

6.1.1 Decision threshold

For the calculation of the characteristic limits of the procedure, in a first step the decision threshold a^* is determined according to Equation (7):

$$a^* = k_{1-\alpha} \cdot f_2 \cdot \frac{\varphi_A}{\eta_{\text{Sr}} \cdot m} \cdot \sqrt{R_0 \cdot \left(\frac{1}{t_m} + \frac{1}{t_0} \right)} \quad (7)$$

$k_{1-\alpha}$ is the quantile of the normal distribution for the probability of the type I error α .

6.1.2 Detection limit

Thus, the detection limit $a^\#$ can be calculated according to the implicit Equation (8):

$$a^\# = a^* + k_{1-\beta} \cdot \sqrt{a^{\#2} \cdot u_{\text{rel}}^2(\varphi) + \varphi^2 \cdot \left(\frac{a^\#}{t_m \cdot \varphi} + \frac{R_0}{t_m} + \frac{R_0}{t_0} \right)} \quad (8)$$

with

$$\varphi = f_2 \cdot \frac{\varphi_A}{\eta_{\text{Sr}} \cdot m}$$

$$u_{\text{rel}}(\varphi) = \sqrt{u_{\text{rel}}^2(f_2) + u_{\text{rel}}^2(\varphi_A) + u_{\text{rel}}^2(\eta_{\text{Sr}}) + u_{\text{rel}}^2(m)}$$

After solving Equation (8), the detection limit is calculated according to Equation (9)

$$a^\# = \frac{a^* \cdot \psi}{\theta} \cdot \left[1 + \sqrt{1 - \frac{\theta}{\psi^2} \cdot \left(1 - \frac{k_{1-\beta}^2}{k_{1-\alpha}^2} \right)} \right] \quad (9)$$

with the auxiliary quantities:

$$\theta = 1 - k_{1-\beta}^2 \cdot [u_{\text{rel}}^2(f_2) + u_{\text{rel}}^2(\varphi_A) + u_{\text{rel}}^2(\eta_{\text{Sr}}) + u_{\text{rel}}^2(m)]$$

$$\psi = 1 + \frac{k_{1-\beta}^2}{2 \cdot a^*} \cdot f_2 \cdot \frac{\varphi_A}{\eta_{\text{Sr}} \cdot m} \cdot \frac{1}{t_m}$$

In the Equations (8) and (9) are:

φ procedural calibration factor;

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

$k_{1-\beta}$ quantile of the standard normal distribution for the probability of the type II error β .

6.1.3 Limits of the coverage interval

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

6.2 Worked examples

With the values from Section 5.2.2 and the values of the quantiles $k_{1-\alpha} = 3$ and $k_{1-\beta} = 1,645$, the decision threshold a^* , according to Equation (7) is:

$$\begin{aligned} a^* &= 3 \cdot 1,000 \cdot \frac{0,585}{0,750 \cdot 0,555} \cdot \sqrt{0,0790 \cdot \left(\frac{1}{60 \cdot 10^3} + \frac{1}{60 \cdot 10^3} \right)} \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)} = \\ &= 4,216 \cdot 0,00162 \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)} = 6,84 \cdot 10^{-3} \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)} \end{aligned}$$

According to Equation (9), the detection limit $a^\#$ is:

$$\begin{aligned} a^\# &= \frac{6,84 \cdot 10^{-3} \cdot 1,0046}{0,9889} \cdot \left[1 + \sqrt{1 - \frac{0,9889}{1,0092} \cdot \left(1 - \frac{2,706}{9} \right)} \right] \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)} = \\ &= 6,949 \cdot 10^{-3} \cdot 1,5610 \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)} = 10,9 \cdot 10^{-3} \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)} \end{aligned}$$

with

$$\theta = 1 - 2,706 \cdot (0,0016 + 0,0025) = 1 - 2,706 \cdot 0,0041 = 0,9889$$

$$\begin{aligned} \psi &= 1 + \frac{2,706}{2 \cdot 6,84 \cdot 10^{-3}} \cdot 1,000 \cdot \frac{0,585}{0,750 \cdot 0,555} \cdot \frac{1}{60 \cdot 10^3} = \\ &= 1 + 197,8 \cdot 1,405 \cdot 16,66 \cdot 10^{-6} = 1,0046 \end{aligned}$$

The detection limit of the specific activity of $50 \cdot 10^{-3} \text{ Bq} \cdot \text{kg}^{-1}$ (FM) required in the routine measurement program according to AVV-IMIS is reached at a measurement duration of approx. two hours, if a Sr-85 control counting source is used [3].

If Sr-85 is added directly to the test sample as yield tracer, the count rate is increased. To keep this contribution low, care must be taken to select a smaller energy range for the measurement of the activity of (Sr-90+Y-90) in the upper region of the pulse height spectrum. This reduces the detection efficiency and the detection limit increases for the same measurement duration. In this case, the detection limit of the activity concentration required in the routine measurement program according to AVV-IMIS is reached after a measurement time of approx. three hours to four hours.

7 Software supported calculation

7.1 View of the Excel spreadsheet

7.1.1 Counting source with added Sr-85

Procedure for determining the specific activity of strontium-90 in animal feed and vegetation samples by liquid scintillation spectrometry (dicyclohexyl-18-crown-6 method)

F-Sr-90-FUMI-04

Version July 2015 / verified June 2020

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

SAMPLE IDENTIFICATION: kale

#Number of parameters p	11
k_alpha	3
k_beta	1,645
gamma	0,05

Create Excel variables!

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

Data input:		variable names:		Uncertainty budget:			
#Values of parameters p	Unit	Excel variable	Input values	StdDev	partial derivatives	uncertainty budget:	budget in %
p 1	#Number of gross counts Ng	Ng	1687,8	41,0828431	0,00010539	0,00432953	35,7609808
p 2	background count rate	_RO	1,60000E-02	0,0005164	-6,32312312	0,00326525	20,34040856
p 3	duration of measurement	tm	6,00000E+04		-2,9645E-06	0	0
p 4	duration of background measurement	_t0	6,00000E+04		0	0	0
p 5	added Sr-85 activity	_ASr85	5,00000E+00	0,1	-0,00091956	9,1956E-05	0,016132036
p 6	calibration factor for Sr-85	_phiSr85	4,16700E+03	166,7	1,1034E-06	0,00018393	0,064543502
p 7	fresh mass	mFM	0,55500000	0,000015	-0,1299128	1,9487E-06	7,24459E-06
p 8	calibration factor for Sr-90	phia	2,63200000	0,10492	0,02739425	0,00287421	15,76025278
p 9	chemical yield	eta	0,75000000	0,0375	-0,10226588	0,00383497	28,05767508
p 10	decay factor for Sr-85	fb	0,80800000		-0,00569036	0	0
p 11	decay factor for Sr-90	_f2	1,00000000		0,07210168	0	0
(list prolongable here)							
Model section		c = phix * Rn					
Auxiliary equations h				(Formulae)			
h 1	#Gross count rate Rg	1/s	Rg	0,02813			
h 2	Sr-85 count rate	1/s	_RSr85	7,27142E-04			
(list prolongable here)							
	#Net count rate Rn	1/s	Rn	1,14029E-02			
	#Calibration factor, proc.dep.	Bq*s/kg	phix	6,323123123			
	#Value output quantity	Bq/kg	Result	0,072101676	0,02256954	<-- output value modifiable by VBA	
	#Combined standard uncertainty	Bq/kg	uResult	0,00723996			
	#Decision threshold	Bq/kg		0,014040305			
	#Detection limit	Bq/kg		0,022569532			
further derived values							
	Auxiliary quantity Omega		Omega	1			
	Best estimate	Bq/kg	BestEst	0,072101676			
	Uncertainty best estimate	Bq/kg		0,00723996			
	Lower confidence limit	Bq/kg		0,057911615			
	Upper confidence limit	Bq/kg		0,086291737			

Calculate!

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

7.1.2 Counting source without added Sr-85

Procedure for determining the specific activity of strontium-90 in animal feed and vegetation samples by liquid scintillation spectrometry (dicyclohexyl-18-crown-6 method)

F-Sr-90-FUMI-04

Version July 2015 / verified June 2020

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

SAMPLE IDENTIFICATION: meadow vegetation

#Number of parameters p	8
k_alpha	3
k_beta	1,645
gamma	0,05

Create Excel variables!

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	Excel variable	Input values	StdDev	partial derivatives	uncertainty budget:	budget in %
p 1	#Number of gross counts Ng		Ng	7818	88,4194549	2,3423E-05	0,00207109	15,209613
p 2	background count rate	1/s	_R0	7,90000E-02	0,00114746	-1,40540541	0,00161265	9,221484
p 3	duration of measurement	s	tm	6,00000E+04		-3,0521E-06	0	0,000000
p 4	duration of background measurement	s	_t0	6,00000E+04		0	0	0,000000
p 5	fresh mass	kg	mFM	0,55500000	0,000015	-0,12990491	1,9486E-06	0,000013
p 6	calibration factor	Bq*s	phia	0,58500000	0,0234	0,12324324	0,00288389	29,490334
p 7	chemical yield		eta	0,75000000	0,0375	-0,09612963	0,00360486	46,078555
p 8	decay factor for Sr-90		_f2	1,00000000		0,0720973	0	0,000000
(list prolongable here)								
Model section		c = phix * Rn						
Auxiliary equations h		(Formulae)						
h 1	#Gross count rate Rg	1/s	Rg	1,30300E-01				
(list prolongable here)								
	#Net count rate Rn	1/s	Rn	5,13000E-02				
	#Calibration factor, proc.dep.	Bq*s/kg	phix	1,405405405				
	#Value output quantity	Bq/kg	Result	0,072097297	0,01085047	<-- output value modifiable by VBA		
	#Combined standard uncertainty	Bq/kg	uResult	0,005310545				
	#Decision threshold	Bq/kg		0,006841885				
	#Detection limit	Bq/kg		0,010850465				
further derived values								
	Auxiliary quantity Omega		Omega	1,00000000				
	Best estimate	Bq/kg	BestEst	0,072097297				
	Uncertainty best estimate	Bq/kg		0,005310545				
	Lower confidence limit	Bq/kg		0,06168882				
	Upper confidence limit	Bq/kg		0,082505775				

Calculate!

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

7.2 View of the UncertRadio result page

7.2.1 Counting source with added Sr-85

UJR UncertRadio: Calculation of uncertainty budget and detection limits - F-Sr-90-FUMI-04_var1_V2015-07_gp2020-06_EN.TXP

File Edit Options Help

Procedure Equations Values, Uncertainties Uncertainty budget **Results** Text Editor

Final measurement result for aSr90 :

Coverage factor k: 1.0

Value output quantity: 7.2102E-02 Bq/kg

extendend (Std.-)uncertainty: 7.2439E-03 Bq/kg

relative ext.(Std.-)uncertainty: 10.05 %

Best Bayesian Estimates: min. Coverage-Intervall

Value output quantity: 7.2102E-02 Bq/kg

extendend (Std.-)uncertainty: 7.2439E-03 Bq/kg

lower confidence limit: 5.7904E-02 Bq/kg

upper confidence limit: 8.6299E-02 Bq/kg

Probability (1-gamma): 0.950

Decision threshold and detection limit for aSr90 :

Decision threshold (DT): 1.4040E-02 Bq/kg Iterations: 1

Detection limit (DL): 2.2570E-02 Bq/kg Iterations: 5

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

Monte Carlo Simulation:

Number of simul. measurements: 100000 Values <0 included

Number of runs: 1 min. Coverage interval

relSD%:

Value output quantity: 7.2276E-02 Bq/kg 0.032

extendend uncertainty: 7.3045E-03 Bq/kg 0.224

relative extd.(Std.-)uncertainty: 10.11 %

lower confidence limit: 5.8680E-02 Bq/kg 0.105

upper confidence limit: 8.7276E-02 Bq/kg 0.071

Decision threshold (DT): 1.4315E-02 Bq/kg 0.873

Detection limit (DL): 2.2662E-02 Bq/kg 0.573

active run: 1 IT: 10 Start MC

LinFit: Standard uncertainty of fit parameter ai:

from LS analysis:

from uncertainty propagation:

reduced Chi-square:

Project: Bearbeitung\F-Sr-90-FUMI-04_var1_V2015-07_gp2020-06_EN.T... Ready!

The corresponding UncertRadio project file is available on the website of this Procedures Manual.

7.2.2 Counting source without added Sr-85

UJR UncertRadio: Calculation of uncertainty budget and detection limits - F-Sr-90-FUMI-04_var2_V2015-07_gp2020-06_EN.TXP

File Edit Options Help

Procedure Equations Values, Uncertainties Uncertainty budget **Results** Text Editor

Final measurement result for aSr90 :

Coverage factor k: 1.0

Value output quantity: 6.8051E-02 Bq/kg

extendend (Std.-)uncertainty: 5.0125E-03 Bq/kg

relative ext.(Std.-)uncertainty: 7.366 %

Best Bayesian Estimates: min. Coverage-Intervall

Value output quantity: 6.8051E-02 Bq/kg

extendend (Std.-)uncertainty: 5.0125E-03 Bq/kg

lower confidence limit: 5.8227E-02 Bq/kg

upper confidence limit: 7.7875E-02 Bq/kg

Probability (1-gamma): 0.950

Decision threshold and detection limit for aSr90 :

Decision threshold (DT): 6.4579E-03 Bq/kg Iterations: 1

Detection limit (DL): 1.0242E-02 Bq/kg Iterations: 5

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

Monte Carlo Simulation:

Number of simul. measurements: 100000 Values <0 included

Number of runs: 1 min. Coverage interval

relSD%:

Value output quantity: 6.8203E-02 Bq/kg 0.023

extendend uncertainty: 5.0463E-03 Bq/kg 0.224

relative extd.(Std.-)uncertainty: 7.399 %

lower confidence limit: 5.8892E-02 Bq/kg 0.072

upper confidence limit: 7.8713E-02 Bq/kg 0.054

Decision threshold (DT): 6.5554E-03 Bq/kg 0.873

Detection limit (DL): 1.0288E-02 Bq/kg 0.576

active run: 1 IT: 8 Start MC

LinFit: Standard uncertainty of fit parameter ai:

from LS analysis:

from uncertainty propagation:

reduced Chi-square:

Project: Bearbeitung\F-Sr-90-FUMI-04_var2_V2015-07_gp2020-06_EN.T... Ready!

The corresponding UncertRadio project file is available on the website of this Procedures Manual.

8 Catalogue of the chemicals und equipment

8.1 Chemicals

The chemicals used should be of analytically pure quality.

- ammonium carbamate;
- dicyclohexyl-18-crown-6 in chloroform: 0,05 mol·l⁻¹;
- sodium acetate – acetic acid solution: 0,05 mol·l⁻¹ sodium acetate in 0,05 mol·l⁻¹ acetic acid;
- sodium dichromate solution, Na₂Cr₂O₇: 1,31 mol·l⁻¹;
- sodium hydroxide solution, NaOH: 3 mol·l⁻¹, 10 mol·l⁻¹;
- nitric acid, HNO₃: 6 mol·l⁻¹;
- scintillation cocktail: e. g. InstantScintGelPlus, UltimaGold LLT;
- toluenesulfonic acid in aqueous solution: 25 g toluenesulfonic acid in 75 ml deionized water (in total 100 ml).

Carrier solutions

- barium carrier solution: 2 mg Ba²⁺ per ml solution:
dissolve 0,356 g barium chloride dihydrate (BaCl₂ · 2 H₂O) in deionized water, add 1 ml hydrochloric acid (3 mol·l⁻¹), then fill to 100 ml with deionized water.
- strontium carrier solution: 20 mg Sr²⁺ per ml solution:
dissolve 6,086 g strontium chloride hexahydrate (SrCl₂ · 6 H₂O) in deionized water, add 1 ml hydrochloric acid (3 mol·l⁻¹), then fill to 100 ml with deionized water;
- yttrium carrier solution: 20 mg Y³⁺ per ml solution:
dissolve 6,83 g yttrium chloride hexahydrate (YCl₃ · 6 H₂O) in deionized water, add 1 ml hydrochloric acid (3 mol·l⁻¹), then fill to 100 ml with deionized water.

8.2 Equipment

The following equipment is used for the procedure:

- cutting mill with sieves for particle size less than 2 mm or 1 mm and sample suction as well as centrifugal mill with a sieve for particle size less than 1 mm;
- ash trays, e. g. made of quartz or fused silica, 55 mm high, 145 mm wide, 205 mm long and a wall thickness of 4 mm to 5 mm;

- chamber furnace with air inlet slots and catalytic exhaust gas cleaning, e. g. chamber furnace type N150 from Nabertherm;
- optional: microwave oven with 250 ml pressure vessels, e. g. from MLS;
- fine quantitative paper filters with a pore diameter of less than 2 µm, e. g. blue band filter grade 589/3, or nitrocellulose filters with a pore diameter of 0,45 µm;
- liquid scintillation measurement vials made of low potassium glass;
- liquid scintillation spectrometer, if possible low-level version with multi-channel analyzer;
- laboratory centrifuge.

References

- [1] *Act on Protection against the Harmful Effects of Ionising Radiation* (Radiation Protection Act, StrlSchG). Available at: <https://www.bmu.de/GE241-1> [last accessed on 12 November 2020].
- [2] *Guideline concerning Emission and Immission Monitoring of Nuclear Installations* (REI). Available at: <https://www.base.bund.de/SharedDocs/Downloads/BASE/EN/hns/a1-english/A1-12-05-REI.html> [last accessed on 12 November 2020].
- [3] *General Administrative Regulation on the Integrated Measurement and Information System for the Monitoring of Environmental Radioactivity* (AVV-IMIS). BAnz. 2006, Nr. 244a. Only available in German.
- [4] ISO 11929-1:2019 *Determination of the characteristic limits (decision threshold, detection limit and limits of the coverage interval) for measurements of ionizing radiation — Fundamentals and application — Part 1: Elementary applications*.