Procedure for determining the specific activity of strontium-90 in food by liquid scintillation spectrometry (Dicyclohexyl-18-crown-6 method)

E-Sr-90-LEBM-04

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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.

2 Sampling

The sampling is carried out according to procedure $E-\gamma$ -SPEKT-LEBM-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 E-Sr-90-LEBM-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 food 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.

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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

Calibration and measurement are described in procedure F-Sr-90-MILCH-05.

5 Calculation of the results

The calculation of the analytical results is based on procedure E-Sr-90-LEBM-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):

$$R_{\rm Sr-85} = \frac{A_{\rm Sr-85}}{\varphi_{\rm A,Sr-85}} \cdot \eta_{\rm Sr} \cdot f_{\rm b} = \frac{A_{\rm Sr-85}}{\varphi_{\rm A,Sr-85}} \cdot \eta_{\rm Sr} \cdot e^{-\lambda_{\rm Sr-85} \cdot t_{\rm b}}$$
(1)

Herein are:

 $\begin{array}{ll} R_{\rm Sr-85} & {\rm count\ rate\ of\ Sr-85,\ in\ s^{-1};} \\ A_{\rm Sr-85} & {\rm activity\ of\ Sr-85\ added\ to\ the\ sample,\ in\ Bq;} \\ \varphi_{\rm A,Sr-85} & {\rm activity\ related\ calibration\ factor\ of\ Sr-85,\ in\ Bq\cdot s;} \\ f_{\rm b} & {\rm correction\ factor\ for\ the\ decay\ of\ the\ activity\ of\ Sr-85\ for\ the\ time\ period}} \end{array}$

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between addition of the tracer to the sample and start of the measurement;

 $\lambda_{\rm Sr-85}$ 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;

 $\eta_{\rm Sr}$ chemical yield for strontium.

The specific activity of Sr-90 is calculated according to Equation (2):

$$a = f_2 \cdot \frac{\varphi_A}{\eta_{\text{Sr}} \cdot m_{\text{FM}}} \cdot R_n = e^{\lambda_{\text{Sr}-90} \cdot t_A} \cdot \frac{\varphi_A}{\eta_{\text{Sr}} \cdot m_{\text{FM}}} \cdot \left[R_g - (R_0 + R_{\text{Sr}-85}) \right]$$
(2)

Herein are:

a specific activity of Sr-90, based on fresh mass, in $Bq kg^{-1}$;

 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;

 λ_{Sr-90} decay constant of Sr-90, in s⁻¹;

- $R_{\rm g}$ 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;
- $m_{\rm FM}$ fresh mass of the sample used, in kg,

 $m_{\rm FM} = m_{\rm a} \cdot q_1 \cdot q_2$

with: m_a mass of ash used, in kg;

- q_1 ratio dry mass to ash mass;
 - q₂ ratio of fresh mass to dry mass.

Note:

In addition to the calculation, the sum of the contributions of the background and the Sr-85 ($R_0 + R_{Sr-85}$) 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):

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

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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):

$$\frac{u(a)}{a} = \left\{ \frac{1}{\left[R_{\rm g} - (R_0 + R_{\rm Sr-85})\right]^2} \cdot \left[\frac{R_{\rm g}}{t_{\rm m}} + \frac{R_0}{t_0} + u^2(R_{\rm Sr-85})\right] + - \left[u_{\rm rel}^2(f_2) + u_{\rm rel}^2(\varphi_{\rm A}) + u_{\rm rel}^2(\eta_{\rm Sr}) + u_{\rm rel}^2(m_{\rm FM})\right] \right\}^{\frac{1}{2}}$$

$$(4)$$

with

$$u^{2}(R_{\rm Sr-85}) = R_{\rm Sr-85}^{2} \cdot \left[u_{\rm rel}^{2}(A_{\rm Sr-85}) + u_{\rm rel}^{2}(\varphi_{\rm A, Sr-85}) + u_{\rm rel}^{2}(\eta_{\rm Sr}) + u_{\rm rel}^{2}(f_{\rm b}) \right]$$
(5)

Herein are:

u(a)	standard uncertainty of the specific activity a at the time of sampling, based on fresh mass, in Bq·kg ⁻¹ ;
$u_{\rm rel}(\varphi_{\rm A})$	relative standard uncertainty of the activity-related calibration factor;
$u_{\rm rel}(f_2)$	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;
$u_{\rm rel}(\eta_{\rm Sr})$	relative standard uncertainty of the chemical yield for strontium;
$u_{\rm rel}(m_{\rm FM})$	relative standard uncertainty of the mass of the sample used based on fresh mass;
$u(R_{\rm Sr-85})$	standard uncertainty of the count rate of Sr-85, in s ⁻¹ ;
$u_{\rm rel}(A_{\rm Sr-85})$	relative standard uncertainty of the added activity of Sr-85;
$u_{\rm rel}(\varphi_{\rm A,Sr-85})$	relative standard uncertainty of the activity-related calibration factor for Sr-85;
$u_{\rm rel}(f_{\rm b})$	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;
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 $u_{\rm rel}(f_2)$ and of the mass of the sample used $u_{\rm rel}(m_{\rm FM})$ can be neglected. For an energy range from

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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 $u(R_{Sr-85})$ 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):

$$\frac{u(a)}{a} = \sqrt{\frac{1}{\left(R_{\rm g} - R_0\right)^2} \cdot \left(\frac{R_{\rm g}}{t_{\rm m}} + \frac{R_0}{t_0}\right) + \left[u_{\rm rel}^2(f_2) + u_{\rm rel}^2(\varphi_{\rm A}) + u_{\rm rel}^2(\eta_{\rm Sr}) + u_{\rm rel}^2(m_{\rm FM})\right]} \tag{6}$$

For the calculation of the combined relative standard uncertainty of the specific activity, the contributions of the relative standard uncertainty of the correction factor $u_{rel}(f_2)$ and the mass of the sample used $u_{rel}(m_{FM})$ 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.

In the following example "fresh mass of a kale sample", 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:

$\varphi_{\mathrm{A,Sr-85}}$	=	4167 Bq·s;	$u(R_{\rm Sr-85})$	=	0;
$\eta_{ m Sr}$	=	0,750;	$u_{\rm rel}(\eta_{\rm Sr})$	=	0,05;
f _b	=	0,808;	m _a	=	0,010 kg;
R _g	=	0,04167 s⁻¹;	t _m	=	60·10 ³ s;
R_0	=	0,0160 s ⁻¹ ;	t_0	=	60·10³ s;
q_1	=	10,0;	q_2	=	5,88;
$m_{ m FM}$	=	0,588 kg;	$u_{\rm rel}(m_{\rm FM})$	=	0;
$arphi_{ m A}$	=	2,632 Bq·s;	$u_{ m rel}(\varphi_{ m A})$	=	0,04;
f_2	=	1,000;	$u_{\rm rel}(f_2)$	=	0.

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

$$R_{\rm Sr-85} = \frac{5.0}{4167} \cdot 0.750 \cdot 0.808 \, \rm s^{-1} = 0.000727 \, \rm s^{-1}$$

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

$$a = \frac{1,00 \cdot 2,632 \cdot [0,04167 - (0,0160 + 0,000727)]}{0,750 \cdot 0,588} \quad \text{Bq} \cdot \text{kg}^{-1} \text{ (FM)} =$$
$$= 0,149 \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)}$$

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:

$$\frac{u(a)}{a} = \sqrt{\frac{1}{[0,04167 - (0,0160 + 0,000727)]^2} \cdot \left(\frac{0,04167}{60 \cdot 10^3} + \frac{0,0160}{60 \cdot 10^3}\right) + 0,04^2 + 0,05^2} = \sqrt{0,005646} = 0,0751$$

For this example, the specific Sr-90 activity in the kale sample at the time of sampling is:

$$a = (0,149 \pm 0,011) \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)}$$

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	=	0,1912 s⁻¹;	$t_{ m m}$	=	60·10³ s;
R_0	=	0,0790 s ⁻¹ ;	t_0	=	60·10³ s;
m _a	=	0,010 kg;	$m_{ m FM}$	=	0,588 kg;
q_1	=	10,0;	q_2	=	5,88;
φ_{A}	=	2,632 Bq·s;	$u_{ m rel}(\varphi_{ m A})$	=	0,04;
$\eta_{ m Sr}$	=	0,750;	$u_{ m rel}(\eta_{ m Sr})$	=	0,05;
f_2	=	1,000.			

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

$$a = \frac{1,00 \cdot 0,585 \cdot (0,1912 - 0,0790)}{0,750 \cdot 0,588} \quad \text{Bq} \cdot \text{kg}^{-1} \text{ (FM)} = 0,149 \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)}$$

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

$$\frac{u(a)}{a} = \sqrt{\frac{1}{(0,1912 - 0,0790)^2} \cdot \left(\frac{0,1912}{60 \cdot 10^3} + \frac{0,0790}{60 \cdot 10^3}\right) + 0,04^2 + 0,05^2} = \sqrt{4,46 \cdot 10^{-3}} = 0,0668$$

The specific activity of Sr-90 in the kale sample at the time of sampling is for this example:

$$a = (0,149 \pm 0,010) \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)}$$

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 uncertainties and the systematic standard uncertainties, e. g. those of yield determination. The mean value of the specific activity of triple determinations of a total of nine aliquots (i. e. a total of 27 determinations) of a spinach powder sample in the context of an IMIS round robin test using this procedure was 102 Bq·kg⁻¹ with a standard deviation of 6,7 Bq·kg⁻¹; this is in good agreement with the mean value of the round robin test (mean value of all participants after outlier correction) of 103 Bq·kg⁻¹ and standard deviation of 19 Bq·kg⁻¹ [4].

In the round robin test of the Joint Research Centre of the European Commission with wildberries as sample material, a mean value for the specific activity of Sr-90 of 159 Bq·kg⁻¹ with an extended uncertainty (covering factor k = 2) of 12 Bq·kg⁻¹ was determined in three analyses using this procedure. This value is in good agreement with the reference value of 153 Bq·kg⁻¹ with an extended uncertainty (k = 2) of 8 Bq·kg⁻¹ [5].

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6 Characteristic limits of the procedure

The calculation of the characteristic limits follows the standard ISO 11929 series [6].

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):

$$a^* = k_{1-\alpha} \cdot f_2 \cdot \frac{\varphi_A}{\eta_{\rm Sr} \cdot m_{\rm FM}} \cdot \sqrt{R_0 \cdot \left(\frac{1}{t_{\rm m}} + \frac{1}{t_0}\right)}$$
(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):

$$a^{\#} = a^{*} + k_{1-\beta} \cdot \sqrt{a^{\#^{2}} \cdot u_{\text{rel}}^{2}(\varphi) + \varphi^{2} \cdot \left(\frac{a^{\#}}{t_{\text{m}} \cdot \varphi} + \frac{R_{0}}{t_{\text{m}}} + \frac{R_{0}}{t_{0}}\right)}$$
(8)

with

$$\varphi = f_2 \cdot \frac{\varphi_{\rm A}}{\eta_{\rm Sr} \cdot m_{\rm FM}}$$

$$u_{\rm rel}(\varphi) = \sqrt{u_{\rm rel}^2(f_2) + u_{\rm rel}^2(\varphi_{\rm A}) + u_{\rm rel}^2(\eta_{\rm Sr}) + u_{\rm rel}^2(m_{\rm FM})}$$

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

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

with the auxiliary quantities:

$$\theta = 1 - k_{1-\beta}^2 \cdot \left[u_{\text{rel}}^2(f_2) + u_{\text{rel}}^2(\varphi_A) + u_{\text{rel}}^2(\eta_{\text{Sr}}) + u_{\text{rel}}^2(m_{\text{FM}}) \right]$$
$$\psi = 1 + \frac{k_{1-\beta}^2}{2 \cdot a^*} \cdot f_2 \cdot \frac{\varphi_A}{\eta_{\text{Sr}} \cdot m_{\text{FM}}} \cdot \frac{1}{t_{\text{m}}}$$

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

- $k_{1-\beta}$ quantile of the standard normal distribution for the probability of the type II error β ;
- φ procedural calibration factor

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

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:

$$a^* = 3 \cdot 1,000 \cdot \frac{0,585}{0,750 \cdot 0,588} \cdot \sqrt{0,0790 \cdot \left(\frac{1}{60 \cdot 10^3} + \frac{1}{60 \cdot 10^3}\right)} \text{ Bq} \cdot \text{kg}^{-1} (\text{FM}) = 3,980 \cdot 0,00162 \text{ Bq} \cdot \text{kg}^{-1} (\text{FM}) = 6,45 \cdot 10^{-3} \text{ Bq} \cdot \text{kg}^{-1} (\text{FM})$$

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

$$a^{\#} = \frac{6,45 \cdot 10^{-3} \cdot 1,0046}{0,9889} \cdot \left[1 + \sqrt{1 - \frac{0,9889}{1,0092}} \cdot \left(1 - \frac{2,706}{9}\right) \right] \text{Bq} \cdot \text{kg}^{-1} \text{ (FM)} = 6,552 \cdot 10^{-3} \cdot 1,5610 \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)} = 10,2 \cdot 10^{-3} \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)}$$

with

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

$$\psi = 1 + \frac{2,706}{2 \cdot 6,45 \cdot 10^{-3}} \cdot 1,000 \cdot \frac{0,585}{0,750 \cdot 0,588} \cdot \frac{1}{60 \cdot 10^{3}} =$$
$$= 1 + 209,8 \cdot 1,327 \cdot 16,66 \cdot 10^{-6} = 1,0046$$

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The detection limit of the specific activity of $40 \cdot 10^{-3}$ Bq·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.

Software supported calculation 7

View of the Excel spreadsheet 7.1

7.1.1 **Counting source with added Sr-85**

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SAMPLE IDENTIFICATION:

	#Number of parameters p		11			User-Input:	Input of value	25
	k_alpha		3				Definition Exc	cel variables
	k_beta		1,645 Create	Excel variable	les!		Input of Exce	l formulae
	gamma		0,05			Excel-VBA:	#Keywords	
							Values from V	/basic
	Data input:		variable names:			Uncertainty b	oudget:	
	#Values of parameters p	Unit	Excel variable	Input values	StdDev	partial	uncertainty	budget
						derivatives	budget:	in %
p 1	#Number of gross counts Ng		Ng	2500,2	50,002	9,9471E-05	0,00497374	19,30114816
p 2	background count rate	1/s	_R0	1,60000E-02	0,0005164	-5,96825397	0,00308199	7,411048006
р З	duration of measurement	S	tm	6,00000E+04		-4,1449E-06	0	0
p 4	duration of background measurement	S	_t0	6,00000E+04	• •	0	0	0
p 5	added Sr-85 activity	Bq	_ASr85	5,00000E+00	0,1	-0,00086795	8,6795E-05	0,005877723
р6	calibration factor for Sr-85	Bq*s	_phiSr85	4,16700E+03	166,7	1,0415E-06	0,00017361	0,023516489
p 7	fresh mass	kg	mFM	0,58800000	0,000015	-0,25317205	3,7976E-06	1,1252E-05
р8	calibration factor for Sr-90	Bq*s	phia	2,63200000	0,10492	0,05655977	0,00593425	27,47565934
р9	chemical yield		eta	0,75000000	0,0375	-0,20427323	0,00766025	45,78273904
p 10	decay factor for Sr-85		fb	0,8080000)	-0,005371	0	0
p 11	decay factor for Sr-90		_f2	1,0000000		0,14886531	0	0
	(list prolongable here)							
	Model section		c = phix * Rn					
	Auxiliary equations h			(Formulae)				
h 1	#Gross count rate Rg	1/s	Rg	0,04167	,			
h 2	Sr-85 count rate	1/s	_RSr85	7,27142E-04				
	(list prolongable here)							
	#Net count rate Rn	1/s	Rn	2,49429E-02				
	#Calibration factor, proc.dep.	Bq*s/kg	g phix	5,968253968	8	_		
	#Value output quantity	Bq/kg	Result	0,148865312	0,02130288	< output val	ue modifiable b	y VBA
	#Combined standard uncertainty	Bq/kg	uResult	0,011321184	ŀ			
	#Decision threshold	Bq/kg		0,013252329)			
	#Detection limit	Bq/kg		0,021302875	i	Calc	امتدار	
	further derived values					Carc	ulate:	
	Auxiliary quantity Omega		Omega	1				
	Best estimate	Bq/kg	BestEst	0,148865312				
	Uncertainty best estimate	Bq/kg		0,011321184				
	Lower confidence limit	Bq/kg		0,126676199				
	Upper confidence limit	Bq/kg		0,171054425				

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

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7.1.2 **Counting source without added Sr-85**

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SAMPLE IDENTIFICATION:

	#Number of parameters p	8	3		1	User-Input:	Input of value	es
	k_alpha	3	3 Creat	- Evenl variabl	ocl		Definition Exc	cel variables
	k_beta	1,645			es:		Input of Exce	l formulae
	gamma	0,05	5			Excel-VBA:	#Keywords	
							Values from \	/basic
	Data input:		variable names:		0.15	Uncertainty b	oudget:	
	#Values of parameters p	Unit	Excel variable	Input values	StdDev	partial	uncertainty	budget
						derivatives	budget:	in %
p1	#Number of gross counts Ng		Ng	114/2	107,107423	2,2109E-05	0,00236802	5,678559067
p 2	background count rate	1/s	_R0	7,90000E-02	0,00114746	-1,32653061	0,00152214	2,34626656
p 3	duration of measurement	S	tm	6,0000E+04		-4,22/2E-06	0	0
p 4	duration of background measurement	S	_t0	6,0000E+04		0	0	0
p 5	fresh mass	kg	mFM	0,58800000	0,000015	-0,25312345	3,7969E-06	1,45987E-05
p 6	calibration factor	Bq*s	phia	0,58500000	0,0234	0,25442177	0,00595347	35,89278905
p 7	chemical yield		eta	0,75000000	0,0375	-0,19844878	0,00744183	56,08237073
p 8	decay factor for Sr-90		_f2	1,0000000		0,14883673	0	0
	(list prolongable here)				T			
	Model section		c = phix * Rn					
	Auxiliary equations h			(Formulae)				
h 1	#Gross count rate Rg	1/s	Rg	1,91200E-01				
	(list prolongable here)							
	#Net count rate Rn	1/s	Rn	1,12200E-01				
	#Calibration factor, proc.dep.	Bq*s/kg	phix	1,326530612				
	#Value output quantity	Bq/kg	Result	0,148836735	0,01024151	< output val	ue modifiable b	y VBA
	#Combined standard uncertainty	Bq/kg	uResult	0,009937257	•			
	#Decision threshold	Ba/kg		0 006457902				
	#Detection limit	Bq/kg		0.010241511				
		04/16		0,010241311				
	further derived values							
	Auxiliary quantity Omega		Omega	1,0000000				
	Best estimate	Bq/kg	BestEst	0,148836735		Calc	ulate!	
	Uncertainty best estimate	Bq/kg		0,009937257				
	Lower confidence limit	Bq/kg		0,129360069				
	Upper confidence limit	Bq/kg		0,168313401				

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

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7.2 View of the UncertRadio result page

7.2.1 Counting source with added Sr-85

File Edit Options Help		
🕒 😫 🖬 📴 🏗 🌆 🧮 🖸 💥 🗍	II 🖩 🛛 🖇 🔳 📐	Help Save to csv
Procedure Equations Values, Un	certainties Uncertain	nty budget Results Text Editor
Final measurement result for a Sr90 : Coverage factor k: 1.0 Value output quantity: 0.1489 extendend (Std)uncertainty: 1.1332E-02 relative ext.(Std)uncertainty: 7.612 Best Bayesian Estimates: Value output quantity: Value output quantity: 0.1489 extendend (Std)uncertainty: 1.1332E-02 lower confidence limit: 0.1267	Bq/kg Bq/kg [%] ☐ min. Coverage-Intervall Bq/kg Bq/kg Bq/kg	Decision threshold and detection limit for aSr90 : Decision threshold (DT): 1.3252E-02 Bq/kg Iterations: Detection limit (DL): 2.1304E-02 Bq/kg Iterations: k_alpha=3.000, k_beta=1.645 Method: ISO 11929:2019, b iteration
upper confidence limit: 0.1711 Probability (1-gamma): 0.950 Monte Carlo Simulation:	Bq/kg	
Number of simul. measurments 100000 Number of runs: 1 Value output quantity: 0.1492	Values <0 included min. Coverage interval relSD%:	LinFit: Standard uncertainty of fit parameter ai: from LS analysis: from uncertainty propagation:
extendend uncertainty: 1.1399E-02 relative extd.(Std)uncertainty: 7.639	Bq/kg 0.224	reduced Chi-square:
lower confidence limit: 0.1282 upper confidence limit: 0.1729 Decision threshold (DT): 1.3585E-02	Bq/kg 0.075 Bq/kg 0.056 Bq/kg 0.873	
Detection limit (DL): 2.1493E-02 active run: 1	Bq/kg 0.573 8 Start MC	

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

7.2.2 Counting source without added Sr-85

\bigcup^{R} UncertRadio: Calculation of uncertainty budget an	d detection limits - E-Sr-90-L	EBM-04_var2_V2015-07_gp2020-06_EN.TXP - 🗆 🗙
File Edit Options <u>H</u> elp		
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Procedure Equations Values, Un	certainties Uncertair	ty budget Results Text Editor
Final measurement result for aSr90 :		
Value output quantity: 0.1488	Ba/ka	
extendend (Std -)uncertainty: 9.9373E-03	Bq/kg	
relative ext (Std -)uncertainty, 5.53732-03	64/Kg	Decision threshold and detection limit for aSr90 :
Post Payerian Estimated	min Coverage-Intervall	Decision threshold (DT): 6.4579E-03 Bq/kg Iterations: 1
Value eutrut guertitu 01488	Dallia	Detection limit (DL): 1.0242E-02 Bq/kg Iterations: 5
extendend (Std.)uncertainty, 0.02725-02	Bq/kg	k alpha=3.000 k beta=1.645 Method: ISO 11929:2019 by
lever confidence limits 01204	bq/kg	iteration
lower confidence limit: 0.1294	Bq/kg	
Drahability (1. samma): 0.050	БЧ/КУ	
Probability (1-gamma): 0.950		
Monte Carlo Simulation:		
Number of simul. measurments 100000	Values <0 Included	LinFit: Standard uncertainty of fit parameter ai:
Number of runs: 1		from LS analysis:
Value output quantity: 0.1492	Ba/ka 0.021	from uncertainty propagation:
extendend uncertainty: 1.0020E-02	Bq/kg 0.224	reduced Chi-square:
relative extd.(Std)uncertainty: 6.715	%	
lower confidence limit: 0.1307	Bq/kg 0.065	
upper confidence limit: 0.170	Bq/kg 0.050	
Decision threshold (DT): 6.6437E-03	Bq/kg 0.873	
Detection limit (DL): 1.0382E-02	Bq/kg 0.578	
active run: 1	Start MC	
Project: Rearboitung/E-Sr-90-I ERM-04 var	2 V201E-07 cp2020-06 E	N T Posdvil
	_v2013-07_gp2020-00_c	Neauy:

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

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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, Na ₂ Cr ₂ O ₇ :	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 Ba ²⁺ per ml solution: dissolve 0,356 g barium chloride dihydrate (BaCl ₂ \cdot 2 H ₂ O) 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 Sr ²⁺ per ml solution: dissolve 6,086 g strontium chloride hexahydrate (SrCl ₂ · 6 H ₂ O) 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 Y^{3+} per ml solution: dissolve 6,83 g yttrium chloride hexahydrate (YCl ₃ · 6 H ₂ O) 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;

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- 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.

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