# Procedure for determining the specific activity of strontium-90 in food by proportional counting (Dicyclohexyl-18-crown-6 method)

E-Sr-90-LEBM-03

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# Procedure for determining the specific activity of strontium-90 in food by proportional counting (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.

# 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  $E-\gamma$ -SPEKT-LEBM-01.

This procedure requires the dry ashing of fresh samples at 400 °C. In most cases, the quality of the ash obtained in this way is not sufficient for the Sr-90 analysis. For routine samples, it is often advantageous to reduce the required large quantities of samples by drying and to store the dried sample until later ash. Drying can be performed in a circulating drying oven at 105 °C (e. g. for leafy vegetable samples) or in a freeze-drying unit (e. g. mixed diet, meat, eggs). Since the quality of the ash is decisive for the extraction of the strontium and the chemical yield, modifications concerning the ashing process differing from the procedure above are listed below.

 All air-dried samples are generally ground to a particle size smaller than 1 mm before ashing.

#### Note:

Cutting mills with sample suction (cyclone suction) and centrifugal mills have proven to be the best choice for grinding. For sample types that clog the sieve of the cutting mill, only a particle size of about

2 mm can be ground. The resulting sample is then ground with the centrifugal mill to a particle size smaller than 1 mm.

- Freeze-dried samples in particular samples with high fat content, hard-to-grind, such as mixed diet – are homogenized to a fine-grained bulk material; depending on the fat content, they may easily clump together.
  - Note:

For the homogenization of fat-containing, freeze-dried samples, so-called emulsifying mixers, which are used in professional kitchens have proven to be effective. The freeze-dried sample is placed in the bowl (capacity 3,5 liters) of the device at approx. 20 °C and homogenized for a maximum of one minute at an increasing number of revolutions in the range of 300 revolutions per minute to 3000 revolutions per minute.

— In this process, the ashing is carried out with a temperature program up to 600 °C.

In addition to the temperature, a complete ashing of the sample depends on other factors such as sample type, sample preparation, total mass of the samples in the furnace, furnace construction and air supply conditions. Therefore, the prepared sample material is distributed up to a maximum filling height of 10 mm in a quartz or fused silica bowl.

In the Federal Coordinating Office, an ashing furnace (see Section 8.2) has proved to be effective for sample masses up to 1,6 kg of dry mass, using the following temperature sequence:

- heat linearly within 2 hours to a temperature of 250 °C with the air inlet valve of the furnace closed;
- maintain the temperature of 250 °C for 2 hours;
- heat linearly to a temperature of 400 °C within 3 hours;
- maintain the temperature of 400 °C for 2 hours (most of the organic material is then removed and the air inlet valve can be opened);
- heat linearly to a temperature of 600 °C within 4 hours;
- maintain the temperature of 600 °C for 10 hours.

If small brown or black particles are visible in the ash, the ashing must be repeated to avoid problems during radiochemical separation. Ashes of samples distributed over several bowls are combined and homogenized.

The ashes are removed from the furnace at about 100 °C, processed immediately or stored in a desiccator, as the ashes of some food samples are sometimes highly hygroscopic. On the one hand, this can lead the ash to combine with the surface of the ash bowl; on the other hand, the ash yield can be falsified by the absorption of water from the air. If ash samples have been stored in plastic bags or bottles over a long period of time, a moisture determination must be performed before further use of the ash.

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## 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.

#### 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 carried out according to procedure F-Sr-90-MILCH-04.

# 5 Calculation of the results

## 5.1 Equations

## 5.1.1 Quantity

The specific activity of Sr-90 is calculated according to Equation (1):

$$a = f_2 \cdot \frac{\varphi_A}{\eta_{\text{Sr}} \cdot m_{\text{FM}}} \cdot R_n = e^{\lambda_{\text{Sr}-90} \cdot t_A} \cdot \frac{\varphi_A}{\eta_{\text{Sr}} \cdot m_{\text{FM}}} \cdot (R_g - R_0)$$
(1)

Herein are:

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

 $\varphi_A$  activity-related calibration factor, in Bq·s;

 $t_{\rm A}$  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;

 $\lambda_{\rm Sr-90}$  decay constant of Sr-90, in s<sup>-1</sup>;

$$\lambda_{\text{Sr-90}} = \frac{\ln 2}{t_{\text{Sr-90}}}$$
  
with:  $t_{\text{Sr-90}}$  half-life of Sr-90, in s;

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- $\eta_{\rm Sr}$  chemical yield for strontium;
- $R_{\rm g}$  gross count rate, in s<sup>-1</sup>;
- $R_0$  background count rate, in s<sup>-1</sup>;

 $R_n$  net count rate of the (Sr-90 + Y-90) counting source, in s<sup>-1</sup>;

 $m_{\rm FM}$  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;
- *q*<sub>2</sub> ratio of fresh mass to dry mass.

## 5.1.2 Standard uncertainty of the quantity

The relative standard uncertainty of the specific activity  $u(a) \cdot a^{-1}$  is according to Equation (2):

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

Herein are:

u(a) standard uncertainty of the specific activity a at the time of sampling, based on fresh mass, in Bq·kg<sup>-1</sup>;

 $u_{\rm rel}(\varphi_{\rm A})$  relative standard uncertainty of the activity-related calibration factor;

- $u_{rel}(f_2)$  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;
- *t*<sub>m</sub> duration of measurement, in s;
- t<sub>0</sub> 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  $u_{rel}(f_2)$  and the mass of the sample used  $u_{rel}(m_{FM})$  can be neglected.

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## 5.2 Worked example

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 based on the fresh mass of a kale sample is presented below.

R <sub>g</sub>	=	0,05755 s⁻¹;	$t_{ m m}$	=	0,17·10 <sup>6</sup> s;
R <sub>0</sub>	=	0,00583 s <sup>-1</sup> ;	t <sub>0</sub>	=	0,25·10 <sup>6</sup> s;
m <sub>a</sub>	=	0,010 kg;	$q_1$	=	10,0;
$m_{ m FM}$	=	0,588 kg;	<i>q</i> <sub>2</sub>	=	5,88;
$arphi_{ m A}$	=	1,615 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.			

According to Equation (1), the specific activity of Sr-90 is:

$$a = \frac{1,00 \cdot 1,615 \cdot (0,05755 - 0,00583)}{0,750 \cdot 0,588} \quad \text{Bq} \cdot \text{kg}^{-1} \text{ (FM)} = 0,189 \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)}$$

With the above values, according to Equation (2), the relative standard uncertainty of the specific activity of Sr-90 is:

$$\frac{u(a)}{a} = \sqrt{\frac{1}{(0,05755 - 0,00583)^2} \cdot \left(\frac{0,05755}{0,17 \cdot 10^6} + \frac{0,00583}{0,25 \cdot 10^6}\right) + 0,04^2 + 0,05^2} = \sqrt{4,235 \cdot 10^{-3}} = 0,0651$$

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

$$a = (0,189 \pm 0,012) \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)}$$

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

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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<sup>-1</sup> with a standard deviation of 6,7 Bq·kg<sup>-1</sup>; 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<sup>-1</sup> and standard deviation of 19 Bq·kg<sup>-1</sup> [4].

# 6 Characteristic limits of the procedure

The calculation of the characteristic limits follows the standard ISO 11929 series [5].

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.

## 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 (3):

$$a^* = k_{1-\alpha} \cdot f_2 \cdot \frac{\varphi_A}{\eta_{\rm Sr} \cdot m_{\rm FM}} \cdot \sqrt{R_0 \cdot \left(\frac{1}{t_{\rm m}} + \frac{1}{t_0}\right)}$$
(3)

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

## 6.1.2 Detection limit

Thus, the detection limit  $a^{\#}$  can be calculated according to the implicit Equation (4):

$$a^{\#} = a^{*} + k_{1-\beta} \cdot \sqrt{a^{\#^{2}} \cdot u_{\text{rel}}^{2}(\varphi) + \varphi^{2} \cdot \left(\frac{a^{\#}}{t_{\text{m}} \cdot \varphi} + \frac{R_{0}}{t_{\text{m}}} + \frac{R_{0}}{t_{0}}\right)}$$
(4)

with

$$\varphi = f_2 \cdot \frac{\varphi_A}{\eta_{\text{Sr}} \cdot m_{\text{FM}}}$$
$$u_{\text{rel}}(\varphi) = \sqrt{u_{\text{rel}}^2(f_2) + u_{\text{rel}}^2(\varphi_A) + u_{\text{rel}}^2(\eta_{\text{Sr}}) + u_{\text{rel}}^2(m_{\text{FM}})}$$

After solving Equation (4), the detection limit is calculated according to Equation (5)

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$$a^{\#} = \frac{a^* \cdot \psi}{\theta} \cdot \left[ 1 + \sqrt{1 - \frac{\theta}{\psi^2} \cdot \left(1 - \frac{k_{1-\beta}^2}{k_{1-\alpha}^2}\right)} \right]$$
(5)

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 (4) and (5) are:

- $k_{1-\beta}$  quantile of the standard normal distribution for the probability of the type II error  $\beta$ ;
- $\varphi$  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 and the values for the quantiles  $k_{1-\alpha} = 3$  and  $k_{1-\beta} = 1,645$ , the decision threshold  $a^*$ , according to Equation (3) is:

$$a^* = 3 \cdot 1,000 \cdot \frac{1,615}{0,750 \cdot 0,588} \cdot \sqrt{0,00583 \cdot \left(\frac{1}{0,17 \cdot 10^6} + \frac{1}{0,25 \cdot 10^6}\right)} \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)} = 10,99 \cdot 0,00024 \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)} = 0,00264 \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)}$$

According to Equation (5), the detection limit  $a^{\#}$  is:

$$a^{\#} = \frac{0,00264 \cdot 1,0110}{0,9889} \cdot \left[ 1 + \sqrt{1 - \frac{0,9889}{1,0221}} \cdot \left(1 - \frac{2,706}{9}\right) \right] \text{Bq} \cdot \text{kg}^{-1} \text{ (FM)} = 0,00270 \cdot 1,5687 \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)} = 0,00424 \text{ Bq} \cdot \text{kg}^{-1} \text{ (FM)}$$

with

$$\theta = 1 - 2,706 \cdot (0,0016 + 0,0025) = 1 - 2,706 \cdot 0,0041 = 0,9889$$
$$\psi = 1 + \frac{2,706}{2 \cdot 0,00264} \cdot 1,000 \cdot \frac{1,615}{0,750 \cdot 0,588} \cdot \frac{1}{0,17 \cdot 10^6} =$$
$$= 1 + 512,5 \cdot 3,66 \cdot 5,882 \cdot 10^{-6} = 1,0110$$

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## 7 Software supported calculation

#### 7.1 View of the Excel spreadsheet

Procedure for determining the specific activity of strontium-90 in food with the proportional counter (dicyclohexyl-18-crown-6 method)

kale

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SAMPLE IDENTIFICATION:

#Number of parameters p	8		User-Input:	Input of values
k_alpha	3	Create Eventurrichted		Definition Excel variables
k_beta	1,645	Create Excel variables!		Input of Excel formulae
gamma	0,05		Excel-VBA:	#Keywords
	<u> </u>			Values from Vbasic

	Data input:		variable names	:		Uncertainty b	udget:	
	#Values of parameters p	Unit	Excel variable	Input values	StdDev	partial	uncertainty	budget
						derivatives	budget:	in %
p 1	#Number of gross counts Ng		Ng	9783,5	98,9115767	2,1542E-05	0,00213075	2,975423442
p 2	background count rate	1/s	RO	5,83000E-03	0,00015271	-3,66213152	0,00055924	0,204965563
р З	duration of measurement	S	tm	1,70000E+05		-1,2397E-06	0	0
p 4	duration of background measurement	S	t0	2,50000E+05		0	0	0
р 5	fresh mass	kg	mFM	0,58800000	0,002500	-0,32211778	0,00080529	0,425004866
р б	activity-related calibration factor	Bq*s	phia	1,61500000	0,064600	0,11727891	0,00757622	37,61745314
р7	chemical yield		eta	0,75000000	0,037500	-0,25254034	0,00947026	58,77715298
p 8	correction factor for decay		_f2	1,0000000		0,18940544	0	0
	(List can be continued here)				1			
	Model section		c = phix * Rn					
	Auxiliary equations h		_	(Formulae)				
h 1	#Gross count rate Rg	1/s	Rg	0,05755				
	(List can be continued here)							
	#Net count rate Rn	1/s	Rn	5,17200E-02				
	#Calibration factor, proc.dep.	Bq*s/kg	phix	3,662131519				
	#Value output quantity	Bq/kg	Result	0,189405442	0,0042298	< output valu	ie modifiable b	y VBA
	#Combined standard uncertainty	Bq/kg	uResult	0,012352582				
					-			
	#Decision threshold	Bq/kg		0,002637058				
	#Detection limit	Bq/kg		0,004229797				
	further derived values							1
	Auxiliary quantity Omega	D (1	Omega	1		Calc	ulate!	
	Best estimate	Bq/kg	BestEst	0,189405442		Calco	alate:	
	Uncertainty best estimate Lower confidence limit	Bq/kg		0,012352582				-
		Bq/kg		0,165194826				
	Upper confidence limit	Bq/kg		0,213616058				

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

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File Edit Options <u>H</u> elp					
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Procedure Equations Values, Un	certainties Uncertain	nty budget Results Text Editor			
Final measurement result for aSr90 :		1			
Coverage factor k: 1.0					
Value output quantity: 0.1894	Bq/kg	Decision threshold and detection limit for aSr90 : Decision threshold (DT): 2.6371E-03 Bg/kg Iterations: 1			
extendend (Std)uncertainty: 1.2328E-02	Bq/kg				
relative ext.(Std)uncertainty: 6.509	%				
Best Bayesian Estimates:	min. Coverage-Intervall				
Value output quantity: 0.1894	Bq/kg				
extendend (Std)uncertainty: 1.2328E-02	Bq/kg	k_alpha=3.000, k_beta=1.645 Method: ISO 11929:2019, b			
lower confidence limit: 0.1652	Bq/kg	iteration			
upper confidence limit: 0.2136	Bq/kg				
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	min. Coverage interval				
	relSD%:	from uncertainty propagation:			
Value output quantity: 0.1898	Bq/kg 0.021	reduced Chi-square:			
extendend uncertainty: 1.2423E-02	Bq/kg 0.224	•			
relative extd.(Std)uncertainty: 6.544	%				
lower confidence limit: 0.1668	Bq/kg 0.063				
upper confidence limit: 0.2154	Bq/kg 0.049 Bg/kg 0.873				
Decision threshold (DT): 2.6651E-03 Detection limit (DL): 4.2316E-03	Bq/kg 0.873 Bq/kg 0.571				
active run: 1 IT	: 9 Start MC				

## 7.2 View of the UncertRadio result page

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 <sup>-1</sup> ;
—	sodium acetate – acetic acid solution:	0,05 mol·l <sup>-1</sup> sodium acetate in 0,05 mol·l <sup>-1</sup> acetic acid;
	sodium dichromate solution, Na <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> :	1,31 mol·l <sup>-1</sup> ;
	sodium hydroxide solution, NaOH:	3 mol·l <sup>-1</sup> , 10 mol·l <sup>-1</sup> ;
—	nitric acid, HNO <sub>3</sub> :	6 mol·l⁻¹.

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#### **Carrier solutions**

	barium carrier solution:	2 mg Ba <sup>2+</sup> per ml solution:			
		dissolve 0,356 g barium chloride dihydrate (BaCl <sub>2</sub> $\cdot$ 2 H <sub>2</sub> O) in deionized water, add 1 ml hydrochloric acid (3 mol·l <sup>-1</sup> ), then fill to 100 ml with deionized water.			
	strontium carrier solution:	20 mg Sr <sup>2+</sup> per ml solution:			
		dissolve 6,086 g strontium chloride hexahydrate (SrCl <sub>2</sub> $\cdot$ 6 H <sub>2</sub> O) in deionized water, add 1 ml hydrochloric acid (3 mol·l <sup>-1</sup> ), then fill to 100 ml with deionized water;			
—	yttrium carrier solution:	20 mg Y <sup>3+</sup> per ml solution:			
		dissolve 6,83 g yttrium chloride hexahydrate (YCl <sub>3</sub> $\cdot$ 6 H <sub>2</sub> O) in deionized water, add 1 ml hydrochloric acid (3 mol·l <sup>-1</sup> ), 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;
- kitchen emulsifying mixer with stainless steel bowl with a capacity of 3,5 litres and with an adjustable speed in the range of 300 revolutions per minute to 3000 revolutions per minute (e. g. Blixer 5 V.V. from Robot-Coup);
- 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 catalytic exhaust gas cleaning, e. g. chamber furnace type N150 from Nabertherm, modified with air inlet valves and distribution slots for uniform ashing of the samples in the chamber;
- optional: microwave oven with 250 ml pressure vessels, e. g. from MLS;
- fine quantitative paper filters with a pore diameter of less than 2  $\mu$ m, or nitrocellulose filters with a pore diameter of 0,45  $\mu$ m;
- low level beta proportional counter with anticoincidence unit (background count rate less than 0,008 s<sup>-1</sup>);
- laboratory centrifuge.

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[last accessed on 26 May 2020].

[5] 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.

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