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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Procedures' Manual for monitoring of radioactive substances in the environment and of external radiation
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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.

Note:
In the context of an IMIS proficiency test [4], the mean value of the specific activity of Sr-90 in a spinach powder sample using this procedure was (102 ± 6,7) $Bq \cdot kg^{-1}$; this is in good agreement with the total mean value of the proficiency test of (103 ± 19) $Bq \cdot kg^{-1}$.

2 Sampling

The sampling is carried out according to procedure E-γ-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

For sample preparation, reference is made to procedure E-γ-SPEKT-LEBM-01.

This procedure requires 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 carried out 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 of less 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 of less 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.

— 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 two hours to a temperature of 250 °C with the air inlet valve of the furnace closed;

— maintain the temperature of 250 °C for two hours;

— heat linearly to a temperature of 400 °C within three hours;

— maintain the temperature of 400 °C for two 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 four hours;

— maintain the temperature of 600 °C for ten 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.

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 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

The calibration, yield determination and the measurement of the background and of the Sr-90 counting source are described in procedure F-Sr-90-MILCH-04.

5 Calculation of the results

5.1 Output quantity

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

$$a = \varphi \cdot R_{\mathrm{n}} = f_2 \cdot \frac{\varphi_{\mathrm{A}}}{\eta_{\mathrm{Sr}} \cdot m} \cdot \left(R_{\mathrm{g}} - R_0\right) \tag{1}$$

Herein are:

a specific activity of radionuclide Sr-90, in $\mathrm{Bq \cdot kg^{-1}}$, related to fresh mass (FM);

f_2 correction factor for the decay of the activity of Sr-90 for the time span between sampling and start of measurement. The correction is only required if the time period t_{A} is greater than 0,5 years;

$f_2 = \mathrm{e}^{\lambda_{\mathrm{Sr-90}} \cdot t_{\mathrm{A}}}$

m mass of the sample, in kg,

related to dry mass (DM): $m = m_{\mathrm{DM}} = m_{\mathrm{a}} \cdot q_1$

related to fresh mass (FM): $m = m_{\mathrm{FM}} = m_{\mathrm{a}} \cdot q_1 \cdot q_2$

m_{a} aliquot of the mass of sample ash, in kg;

q_1 ratio of dry mass to ash mass;

q_2 ratio of fresh mass to dry mass;

R_{g} gross count rate of (Sr-90 + Y-90) counting source, in s^{-1};

R_{n} net count rate of (Sr-90 + Y-90) counting source, in s^{-1};

R_0 background count rate, in s^{-1};

t_{A} time span between sampling and the start of the measurement, in s;

$t_{\mathrm{Sr\text{-}90}}$ half-life of radionuclide Sr-90, in s;

$\lambda_{\mathrm{Sr\text{-}90}}$ decay constant of the radionuclide Sr-90, in s^{-1};

$$\lambda_{\mathrm{Sr-90}} = \frac{\ln 2}{t_{\mathrm{Sr\text{-}90}}}$$

η_{Sr} chemical yield for strontium;

φ procedural calibration factor, in $\mathrm{Bq \cdot s \cdot kg^{-1}}$;

φ_A activity-related calibration factor, in Bq·s.

$$\varphi_A = \frac{A_{\mathrm{Std}}}{R_{\mathrm{n,Std}}}$$

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.

The standard uncertainty includes not only the counting statistics but also the standard uncertainties of mass, chemical separation, yield determination and calibration. Standard uncertainties of time indications, on the other hand, are neglected.

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

$$\frac{u(a)}{a} = \sqrt{\frac{1}{\left(R_{\mathrm{g}} - R_0\right)^2} \cdot \left(\frac{R_{\mathrm{g}}}{t_{\mathrm{m}}} + \frac{R_0}{t_0}\right) + u_{\mathrm{rel}}^2(\varphi)} \tag{2}$$

with

$$u_{\mathrm{rel}}^2(\varphi) = u_{\mathrm{rel}}^2(f_2) + u_{\mathrm{rel}}^2(\varphi_{\mathrm{A}}) + u_{\mathrm{rel}}^2(\eta_{\mathrm{Sr}}) + u_{\mathrm{rel}}^2(m) \tag{3}$$

Herein are:

t_m duration of measurement, in s;

t_0 duration of background measurement, in s;

$u(a)$ standard uncertainty of the specific activity of Sr-90 at the time of sampling, in Bq·kg^{-1}, related to fresh mass (FM);

$u_\mathrm{rel}(f_2)$ relative standard uncertainty of the decay correction factor for Sr-90 for the time period between sampling and the start of the measurement;

$u_\mathrm{rel}(m)$ relative standard uncertainty of the sample mass;

related to dry mass: $u_\mathrm{rel}^2(m) = u_\mathrm{rel}^2(m_\mathrm{DM}) = u_\mathrm{rel}^2(m_\mathrm{a}) + u_\mathrm{rel}^2(q_1)$

related to fresh mass: $u_\mathrm{rel}^2(m) = u_\mathrm{rel}^2(m_\mathrm{FM}) = u_\mathrm{rel}^2(m_\mathrm{a}) + u_\mathrm{rel}^2(q_1) + u_\mathrm{rel}^2(q_2)$

$u_\mathrm{rel}(m_\mathrm{a})$ relative standard uncertainty of the mass of sample ash used;

$u_\mathrm{rel}(q_1)$ relative standard uncertainty of the dry mass to ash mass ratio;

$u_\mathrm{rel}(q_2)$ relative standard uncertainty of the fresh mass to dry mass ratio;

$u_\mathrm{rel}(\eta_\mathrm{Sr})$ relative standard uncertainty of chemical yield for strontium;

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

$u_\mathrm{rel}(\varphi_A)$ relative standard uncertainty of the activity-related calibration factor.

When calculating the relative standard uncertainty of the specific activity, the contributions of the relative standard uncertainties of the correction factor $u_\mathrm{rel}(f_2)$ and the mass of the sample ash used $u_\mathrm{rel}(m_\mathrm{a})$ as well as the conversion factors $u_\mathrm{rel}(q_1)$ and $u_\mathrm{rel}(q_2)$ are generally neglected.

6 Characteristic limits of the procedure

The calculation of the characteristic limits follows the standard series ISO 11929 [5]. For further considerations, it is referred to the General Chapters CHAGR-ISO-01 and CHAGR-ISO-02 of this Procedures' Manual [6, 7].

6.1 Decision threshold

In order to calculate the characteristic limits of the procedure, the decision threshold a^* is first determined by the Equation (4):

$$a^* = k_{1-\alpha} \cdot \varphi \cdot \sqrt{R_0 \cdot \left(\frac{1}{t_\mathrm{m}} + \frac{1}{t_0}\right)} \tag{4}$$

This means:

a^* decision threshold of the specific Sr-90 activity, in Bq·kg^{-1} (FM);

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

6.2 Detection limit

The detection limit $a^{\#}$ is calculated according to the implicit Equation (5):

$$a^{\#} = a^{*} + k_{1-\beta} \cdot \sqrt{a^{\#2} \cdot u_{\mathrm{rel}}^{2}(\varphi) + \varphi^{2} \cdot \left(\frac{a^{\#}}{t_{\mathrm{m}} \cdot \varphi} + \frac{R_0}{t_{\mathrm{m}}} + \frac{R_0}{t_0} \right)} \tag{5}$$

After resolution and simplification of the Equation (5), Equation (6) is obtained for the calculation of the detection limit:

$$a^{\#} = \frac{a^{*} \cdot \psi}{\theta} \cdot \left[1 + \sqrt{1 - \frac{\theta}{\psi^{2}} \cdot \left(1 - \frac{k_{1-\beta}^{2}}{k_{1-\alpha}^{2}} \right)} \right] \tag{6}$$

with the auxiliary quantities

$$\theta = 1 - k_{1-\beta}^{2} \cdot u_{\mathrm{rel}}^{2}(\varphi) \tag{7}$$

$$\psi = 1 + \frac{k_{1-\beta}^{2}}{2 \cdot a^{*}} \cdot \frac{\varphi}{t_{\mathrm{m}}} \tag{8}$$

This means:

$a^{\#}$ detection limit of the specific Sr-90 activity, in Bq·kg^{-1} (FM);

$k_{1\text{-}\beta}$ quantile of the normal distribution for the probability of the type I 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.

In the following example, the specific Sr-90 activity related to fresh mass in a kale sample is calculated from the values obtained below.

R_{g}	=	57,55·10^{-3} s^{-1};	t_{m}	=	0,17·10^{6} s;
R_0	=	5,83·10^{-3} s^{-1};	t_0	=	0,25·10^{6} s;
t_{A}	=	3,456·10^{6} s;	$t_{\mathrm{Sr\text{-}90}}$	=	0,9088·10^{9} s;
m_{a}	=	0,010 kg;	$u_{\mathrm{rel}}(m_{\mathrm{a}})$	=	0,0;
q_1	=	10,0;	$u_{\mathrm{rel}}(q_1)$	=	0,0;

q_2	=	5,88;	$u_{\mathrm{rel}}(q_2)$	=	0,0;
η_{Sr}	=	0,750;	$u_{\mathrm{rel}}(\eta_{\mathrm{Sr}})$	=	0,05;
φ_A	=	1,615 Bq·s;	$u_{\mathrm{rel}}(\varphi_A)$	=	0,04.

The relative standard uncertainties for the mass m_{a} and for the time indications can be neglected.

7.1 Manual evaluation

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

The net counting rate R_{n} and the procedural calibration factor φ are calculated by Equation (1):

$$R_{\mathrm{n}} = 57{,}55 \cdot 10^{-3}\ \mathrm{s}^{-1} - 5{,}83 \cdot 10^{-3}\ \mathrm{s}^{-1} \approx 51{,}72 \cdot 10^{-3}\ \mathrm{s}^{-1}$$

$$\varphi = \frac{1{,}615\ \mathrm{Bq} \cdot \mathrm{s}}{0{,}750 \cdot 0{,}010\ \mathrm{kg} \cdot 10{,}0 \cdot 5{,}88} \cdot \mathrm{e}^{\frac{\ln 2 \cdot 3{,}456 \cdot 10^6\ \mathrm{s}}{0{,}9088 \cdot 10^9\ \mathrm{s}}} \approx 3{,}662\ \mathrm{Bq} \cdot \mathrm{s} \cdot \mathrm{kg}^{-1} \cdot 1{,}00 \approx$$

$$\approx 3{,}662\ \mathrm{Bq} \cdot \mathrm{s} \cdot \mathrm{kg}^{-1}$$

The specific activity of the radionuclide Sr-90 by Equation (1):

$$a = 3{,}662\ \mathrm{Bq} \cdot \mathrm{s} \cdot \mathrm{kg}^{-1} \cdot 51{,}72 \cdot 10^{-3}\ \mathrm{s}^{-1}\ \mathrm{s}^{-1} \approx 0{,}1894\ \mathrm{Bq} \cdot \mathrm{kg}^{-1}\ (\mathrm{FM})$$

The variance of the procedural calibration factor $u_{\mathrm{rel}}(\varphi)$ is determined by Equation (3):

$$u_{\mathrm{rel}}^2(\varphi) = 0^2 + 0{,}04^2 + 0{,}05^2 + 0^2 + 0^2 + 0^2 = 4{,}1 \cdot 10^{-3}$$

The value of the relative standard uncertainty of the specific activity $u_{\mathrm{rel}}(a)$ is obtained by Equation (2):

$$\frac{u(a)}{a} = \sqrt{\frac{1}{(51{,}72 \cdot 10^{-3}\ \mathrm{s}^{-1})^2} \cdot \left(\frac{57{,}55 \cdot 10^{-3}\ \mathrm{s}^{-1}}{0{,}17 \cdot 10^6\ \mathrm{s}} + \frac{5{,}83 \cdot 10^{-3}\ \mathrm{s}^{-1}}{0{,}25 \cdot 10^6\ \mathrm{s}}\right) + 4{,}1 \cdot 10^{-3}} \approx$$

$$\approx \sqrt{0{,}1353 \cdot 10^{-3} + 4{,}1 \cdot 10^{-3}} \approx 0{,}0651$$

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

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

With the above values as well as the value 3 for the quantile of the standard normal distribution $k_{1-\alpha}$ and the value 1,645 for the quantile of the standard normal distribution $k_{1-\beta,}$ the detection limit a^* is calculated by Equation (4):

$$a^* = 3 \cdot 3{,}672\ \mathrm{Bq \cdot s \cdot kg^{-1}} \cdot \sqrt{5{,}83 \cdot 10^{-3}\ \mathrm{s^{-1}} \cdot \left(\frac{1}{0{,}17 \cdot 10^6\ \mathrm{s}} + \frac{1}{0{,}25 \cdot 10^6\ \mathrm{s}}\right)} \approx$$

$$\approx 11{,}02\ \mathrm{Bq \cdot s \cdot kg^{-1}} \cdot \sqrt{57{,}61 \cdot 10^{-9}\ \mathrm{s^{-2}}} \approx 2{,}644 \cdot 10^{-3}\ \ \mathrm{Bq \cdot kg^{-1}\ (FM)}$$

For the calculation of the value for the detection limit $a^{\#}$, the auxiliary quantities θ and ψ are first determined by the Equations (7) and (8):

$$\theta = 1 - 1{,}645^2 \cdot 4{,}1 \cdot 10^{-3} \approx 0{,}9889$$

$$\psi = 1 + \frac{1{,}645^2}{2 \cdot 2{,}644 \cdot 10^{-3}\ \ \mathrm{Bq \cdot kg^{-1}}} \cdot \frac{3{,}672\ \mathrm{Bq \cdot s \cdot kg^{-1}}}{0{,}17 \cdot 10^6\ \mathrm{s}} \approx 1{,}011$$

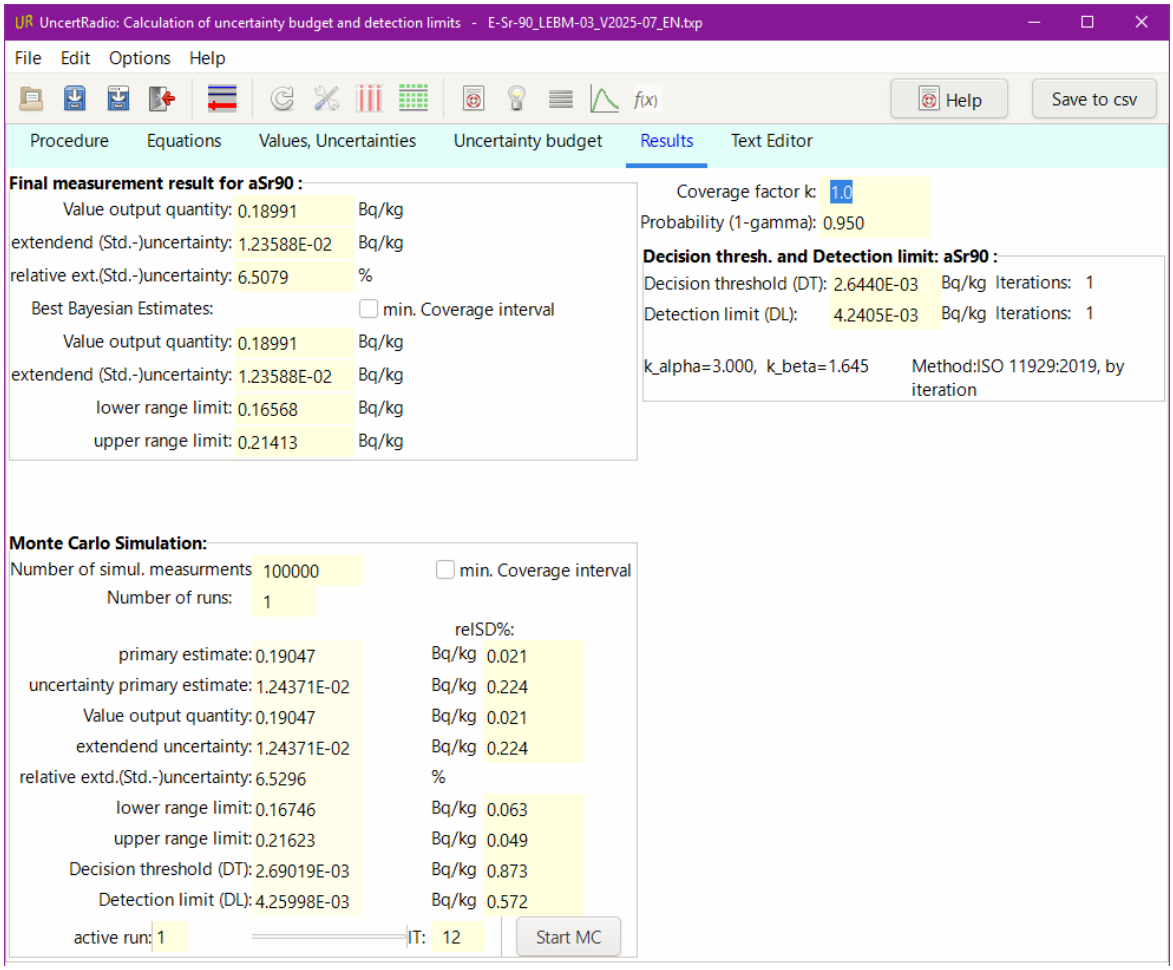
Using Equation (6), the value of detection limit $a^{\#}$ is:

$$a^{\#} = \frac{2{,}644 \cdot 10^{-3}\ \ \mathrm{Bq \cdot kg^{-1}\ (FM)}\ \cdot 1{,}011}{0{,}9889} \cdot \left[1 + \sqrt{1 - \frac{0{,}9889}{1{,}011^2} \cdot \left(1 - \frac{1{,}645^2}{3^2}\right)}\right] \approx$$

$$\approx 2{,}703 \cdot 10^{-3}\ \mathrm{Bq \cdot kg^{-1}\ (FM)} \cdot 1{,}569 \approx 4{,}241 \cdot 10^{-3}\ \ \mathrm{Bq \cdot kg^{-1}\ (FM)}$$

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.



8 Catalogue of the chemicals und equipment

8.1 Chemicals

The chemicals used shall 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, $Na_2Cr_2O_7$: 1,31 mol·l^{-1};
- sodium hydroxide solution, NaOH: 3 mol·l^{-1}, 10 mol·l^{-1};
- nitric acid, HNO_3: 6 mol·l^{-1}.

Carrier solutions

— barium carrier solution: 2 mg Ba^{2+} per ml solution:

dissolve 0,356 g barium chloride dihydrate ($BaCl_2 \cdot 2\ H_2O$) in deionized water, add 1 ml hydrochloric acid (3 $mol \cdot l^{-1}$), 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 ($SrCl_2 \cdot 6\ H_2O$) in deionized water, add 1 ml hydrochloric acid (3 $mol \cdot l^{-1}$), 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 ($YCl_3 \cdot 6\ H_2O$) in deionized water, add 1 ml hydrochloric acid (3 $mol \cdot l^{-1}$), then fill to 100 ml with deionized water.

8.2 Equipment

This procedure requires an ordinary laboratory equipment.

Additionally, further equipment is necessary:

— 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 of 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 µm, or nitrocellulose filters with a pore diameter of 0,45 µm;

— low level beta proportional counter with anticoincidence unit (background count rate less than 0,008 s^{-1});

— laboratory centrifuge.

References

[1] *Act on Protection against the Harmful Effects of Ionising Radiation (Radiation Protection Act - StrlSchG)*
Available at: https://www.bundesumweltministerium.de/GE241-1.

[2] *Richtlinie zur Emissions- und Immissionsüberwachung kerntechnischer Anlagen (REI)*. Gemeinsames Ministerialblatt Nr. 6-9, p. 102.

[3] *Allgemeine Verwaltungsvorschrift zum integrierten Mess- und Informationssystem zur Überwachung der Radioaktivität in der Umwelt (IMIS) nach dem Strahlenschutzvorsorgegesetz (AVV-IMIS)*. Bundesanzeiger Nr. 244a, p. 4-80.

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[5] Standard series ISO 11929:2019-02, *Determination of the characteristic limits (decision threshold, detection limit and limits of the coverage interval) for measurements of ionizing radiation – Fundamentals and application (Parts 1 to 3)*.

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