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Measurement of radioactivity in the environment — Soil —

Part 6:
Measurement of gross alpha and gross beta activities

Mesurage de la radioactivité dans l'environnement — Sol —

Partie 6: Mesurage des activités alpha globale et bêta globale

Reference number
ISO 18589-6:2009(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 18589-6 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 2, *Radiation protection*.

ISO 18589 consists of the following parts, under the general title *Measurement of radioactivity in the environment — Soil*:

- *Part 1: General guidelines and definitions*
- *Part 2: Guidance for the selection of the sampling strategy, sampling and pre-treatment of samples*
- *Part 3: Measurement of gamma-emitting radionuclides*
- *Part 4: Measurement of plutonium isotopes (plutonium 238 and plutonium 239 + 240) by alpha spectrometry*
- *Part 5: Measurement of strontium 90*
- *Part 6: Measurement of gross alpha and gross beta activities*

Introduction

ISO 18589 is published in several parts for use jointly or separately according to needs. Parts 1 to 6 concerning the measurements of radioactivity in the soil have been prepared simultaneously. These parts are complementary and are addressed to those responsible for determining the radioactivity present in soil. The first two parts are general in nature. Parts 3 to 5 deal with nuclide-specific measurements and Part 6 with non-specific measurements of gross alpha or gross beta activities.

Additional parts can be added to ISO 18589 in the future if the standardization of the measurement of other radionuclides becomes necessary.

INTERNATIONAL STANDARD **ISO 18589-6:2009(E)**

Measurement of radioactivity in the environment — Soil —

Part 6:
Measurement of gross alpha and gross beta activities

1 Scope

This part of ISO 18589 provides a method that allows an estimation of gross radioactivity of alpha- and beta-emitters present in soil samples. It applies, essentially, to systematic inspections based on comparative measurements or to preliminary site studies to guide the testing staff both in the choice of soil samples for measurement as a priority and in the specific analysis methods for implementation.

The gross α or β radioactivity is generally different from the sum of the effective radioactivities of the radionuclides present since, by convention, the same alpha counting efficiency is assigned for all the alpha emissions and the same beta counting efficiency is assigned for all the beta emissions

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 31-9, *Quantities and units — Part 9: Atomic and nuclear physics*

ISO 11074, *Soil quality — Vocabulary*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

ISO 18589-1, *Measurement of radioactivity in the environment — Soil — Part 1: General guidelines and definitions*

ISO 18589-2, *Measurement of radioactivity in the environment — Soil — Part 2: Guidance for the selection of the sampling strategy, sampling and pre-treatment of samples*

3 Terms, definitions and symbols

For the purposes of this document, the terms, definitions and symbols given in ISO 18589-1, ISO 11074 and ISO 31-9 and the following symbols apply.

m	Mass of the test portion, expressed in kilograms
a	Activity per unit of mass, expressed in becquerel per kilogram
A_α, A_β	Activity of the standard in the α and β calibration sources, expressed in becquerel
t_g	Sample counting time, expressed in seconds

t_0	Background counting time, expressed in seconds
$t_{s\alpha}$, $t_{s\beta}$	Calibration α and β sources counting time, expressed in seconds
$r_{g\alpha}$, $r_{g\beta}$	Gross count rate per second, from the α and β windows, respectively
$r_{0\alpha}$, $r_{0\beta}$	Background count rate per second, from the α and β windows, respectively
$r_{s\alpha}$, $r_{s\beta}$	Calibration count rate per second, from the α and β windows, respectively
ε_α, ε_β	Counting efficiency for alpha and beta, respectively
χ	Alpha-beta cross-talk, percentage of α count going into β window from the calibration source
$u(a)$	Standard uncertainty associated with the measurement result
U	Expanded uncertainty, calculated by $U = k \cdot u(a)$ with $k = 1, 2, \ldots$, expressed in becquerel per kilogram
a^*	Decision threshold, expressed in becquerel per kilogram
$a^\#$	Detection limit, expressed in becquerel per kilogram
$a^\triangleleft$, $a^\triangleright$	Lower and upper limits of the confidence interval, expressed in becquerel per kilogram

4 Principle

Gross alpha and beta radioactivity are determined by using gas-flow proportional counting or solid scintillation counting on a thin layer of fine soil on a planchette. [1],[2],[3]

Gross alpha and beta determinations are not absolute determinations of the radioactivity of the sample but relative determinations referred to a specific alpha- or beta-emitter that constitutes the standard calibration source. These types of determinations are also known as the alpha index and beta index.

5 Chemical reagents and equipment

5.1 Degreasing solvent.

5.2 Fixer, for example cellulose nitrate (Parlodion®1)), up to 10 g/l in acetone.

5.3 Planchette, stainless steel, with a diameter adapted to the measuring apparatus.

5.4 Analytical balance, accurate to 0,1 mg.

5.5 Gas-flow proportional counter or **solid-state scintillation counter** (such as ZnS), designed to discriminate between the alpha and beta radioactivity.

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1) Parlodion® is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 18589 and does not constitute an endorsement by ISO of this product.

6 Procedure

6.1 Preparation of sources

The preparation of sources involves the following stages.

a) Clean the planchette (5.3) using a degreasing solvent (5.1).

b) Evenly deposit a known mass of the test sample, m, prepared in accordance with ISO 18589-2 in order to obtain the thinnest possible layer with a surface deposit below 20 mg/cm^2.

c) The mass of the sample shall fall between the maximum and the minimum values of the calibration curve.

d) Place the planchette on a horizontal surface.

e) It is recommended to cover the deposit using the fixer (for example 10 ml of Parlodion® dissolved in a 130 mm test dish) and leave it to evaporate at ambient temperature (take the same quantity of fixer as used to prepare the standard sources). The purpose of this operation is to permanently fix a thin film on the surface of the sample on the planchette.

The source is ready for the determination of the gross alpha and gross beta radioactivity indices using previously calibrated measuring equipment.

The planchette should be suitably stored before measuring, to avoid any risk of contamination.

IMPORTANT —

- **The self-absorption phenomena that depend on the matrix constituent as well as the thickness of the deposit tend to underestimate the gross radioactivity, especially the alpha radioactivity.**
- **It is difficult to prepare thin sources with a uniform thickness, which shall be constant from one preparation to another.**

6.2 Calibration

6.2.1 Principle

The counting efficiency and associated uncertainty are determined using soil reference calibration sources [4],[5],[6] prepared by

a) metrological bodies using the reference soil supplied;

b) the laboratory using soil of the same nature as the soil being analysed, whose homogeneity has been thoroughly evaluated. These soils become reference soils after spiking, separately, with calibrated alpha and beta solutions with sufficient activity to allow the activity of the natural or artificial radionuclides present in the soil to represent less than 2 % of the total activity in the reference soils.

Among the standard solutions available for artificial alpha-emitting radionuclides, ^{241}Am and ^{239}Pu are commonly selected. When ^{239}Pu is used, it is necessary to take into account the presence of the ^{241}Pu impurity. Among the standard solutions available for artificial beta-emitting radionuclides, ^{90}Sr+^{90}Y is commonly selected. Finally, among the natural standard solutions, $\mathrm{U}_{\mathrm{nat}}$ and ^{40}K are commonly selected.

An example of the method of the preparation of sources calibrated with plutonium to measure the gross α-radioactivity is given in Annex A. A similar procedure may be implemented by spiking.

The calibration sources shall be stored in a desiccator due to the hydrophilic nature of the soil.

It is advisable to choose the alpha and beta windows so that the beta-alpha cross-talk is close to zero. Then it is sufficient to consider only the alpha-beta cross-talk correction factor. If this cannot be done, it is necessary to consider both the alpha-beta and the beta-alpha cross-talk correction factors.

6.2.2 Procedure

6.2.2.1 Beta calibration

The procedure for calibrating the counters is as follows.

a) Deposit a thin layer of the reference soil following the procedure given in 6.1.

b) Select the width of the alpha and beta windows.

c) Select $t_{\mathrm{s}\beta}$ to collect at least 10^4 counts in the beta window.

d) Determine the count rate of the calibration source in its window.

e) Verify that the count rate in the alpha window is zero or compatible with the equipment background count rate.

f) Calculate the counting efficiency of the counter, dividing the recorded count rate by the activity of the calibration source as given by Equation (1):

$$\varepsilon_\beta = \left(r_{\mathrm{s}\beta} - r_{0\beta}\right) / A_\beta \tag{1}$$

6.2.2.2 Alpha calibration

The procedure for calibrating the counters is as follows.

a) Deposit a thin layer of the reference soil following the procedure given in 6.1.

b) Select the width of the alpha and beta windows.

c) Select the $t_{\mathrm{s}\alpha}$ to collect at least 10^4 counts in the alpha window.

d) Determine the count rate of the calibration source in its window.

e) Calculate the alpha-beta cross-talk correction factor, χ, as given by Equation (2):

$$\chi = r_{\mathrm{s}\alpha\to\beta} / r_{\mathrm{s}\alpha} \tag{2}$$

where $r_{\mathrm{s}\alpha\to\beta}$ is the count rate in the beta window when the alpha calibration source is measured.

f) Calculate the counting efficiency of the counter by dividing the recorded count rate by the activity of the calibration source as given by Equation (3):

$$\varepsilon_\alpha = \left(r_{\mathrm{s}\alpha} - r_{0\alpha}\right) / A_\alpha \tag{3}$$

6.3 Calibration curves

In general, it is difficult to produce a sample source identical to the calibration source. To avoid self-absorption differences between them, it is convenient to construct a calibration curve of sample mass versus counting efficiency, $\varepsilon(m)$. The self-absorption effect is more important in alpha counting.

6.4 Background determination

Measure the background using a planchette prepared in a manner similar to that for the sample source. Let the measured count rates be $r_{0\alpha}$ and $r_{0\beta}$.

6.5 Measurement

The alpha and beta activities per unit mass are estimated by counting the sample source for an appropriate time.

The same equipment conditions should be used for the sample, the background and the calibration source measurements.

The counting time depends on the sample and background count rates and the required detection limit and decision threshold.

$r_{g\alpha}$ and $r_{g\beta}$ are corrected for the background contribution and $r_{g\beta}$ for the alpha-beta cross-talk contribution.

7 Expression of results

7.1 Activities per unit mass

7.1.1 Calculation of alpha activity per unit of mass

The gross alpha activity per unit of mass, a_α, is calculated as given by Equation (4):

$$a_\alpha = \frac{r_{g\alpha} - r_{0\alpha}}{m \cdot \varepsilon_\alpha(m)} = \left(r_{g\alpha} - r_{0\alpha}\right) \cdot w \tag{4}$$

where $w = \dfrac{1}{m \cdot \varepsilon_\alpha(m)}$.

7.1.2 Calculation of beta activity per unit of mass

The gross beta activity per unit of mass, a_β, is calculated as given by Equation (5):

$$a_\beta = \frac{r_{g\beta} - r_{0\beta} - \chi\left(r_{g\alpha} - r_{0\alpha}\right)}{m \cdot \varepsilon_\beta(m)} = \left[r_{g\beta} - r_{0\beta} - \chi\left(r_{g\alpha} - r_{0\alpha}\right)\right] \cdot w \tag{5}$$

where $w = \dfrac{1}{m \cdot \varepsilon_\alpha(m)}$.

If the alpha-beta cross-talk can be neglected, $\chi = 0$, and Equation (5) becomes similar to Equation (4).

7.2 Standard uncertainty

7.2.1 Standard uncertainty of the alpha activity per unit of mass

According to ISO/IEC Guide 98, the standard uncertainty of a_α is calculated as given by Equation (6):

$$u(a_\alpha) = \sqrt{w^2 \cdot \left[u^2\left(r_{g\alpha}\right) + u^2\left(r_{0\alpha}\right)\right] + {a_\alpha}^2 \cdot u_{\text{rel}}^2(w)} = \sqrt{w^2 \cdot \left(r_{g\alpha}/t_g + r_{0\alpha}/t_0\right) + a_\alpha^2 \cdot u_{\text{rel}}^2(w)} \tag{6}$$

where the uncertainty of the counting time is neglected.

The relative standard uncertainty of w is calculated as given by Equation (7):

$$u_{\mathrm{rel}}^2(w) = u_{\mathrm{rel}}^2\left[\varepsilon_\alpha(m)\right] + u_{\mathrm{rel}}^2(m) \tag{7}$$

and the relative standard uncertainty of ε_α is calculated as given by Equation (8):

$$\begin{aligned} u_{\mathrm{rel}}^2\left[\varepsilon_\alpha(m)\right] &= u_{\mathrm{rel}}^2\left(r_{\mathrm{s}\alpha} - r_{0\alpha}\right) + u_{\mathrm{rel}}^2\left(A_\alpha\right) \\ &= \left(r_{\mathrm{s}\alpha}/t_{\mathrm{s}\alpha} + r_{0\alpha}/t_0\right)\Big/\left(r_{\mathrm{s}\alpha} - r_{0\alpha}\right)^2 + u_{\mathrm{rel}}^2\left(A_\alpha\right) \end{aligned} \tag{8}$$

$u_{\mathrm{rel}}^2\left(A_\alpha\right)$ includes all the uncertainties related to the calibration source, that is, in the standard solution and the preparation of the calibration source.

The calculation of the characteristic limits (see ISO 11929 [8]) requires the calculation of $\tilde{u}\left(\tilde{a}_\alpha\right)$, i.e., the standard uncertainty of a_α as a function of its true value, calculated [7] as given by Equation (9):

$$\tilde{u}\left(\tilde{a}_\alpha\right) = \sqrt{w^2 \cdot \left[\left(\tilde{a}_\alpha/w + r_{0\alpha}\right)/t_{\mathrm{g}} + r_{0\alpha}/t_0\right] + \tilde{a}_\alpha^2 \cdot u_{\mathrm{rel}}^2(w)} \tag{9}$$

7.2.2 Standard uncertainty of the beta activity per unit of mass

According to ISO/IEC Guide 98, the standard uncertainty of a_β is calculated as given by Equations (10) and (11):

$$u\left(a_\beta\right) = \sqrt{w^2\left[\left(r_{\mathrm{g}\beta}/t_{\mathrm{g}} + r_{0\beta}/t_0\right) + T(\chi)\right] + a_\beta^2 \cdot u_{\mathrm{rel}}^2(w)} \tag{10}$$

where

$$T(\chi) = \left(r_{\mathrm{g}\alpha} - r_{0\alpha}\right)^2 u^2(\chi) + \chi^2\left(r_{\mathrm{g}\alpha}/t_{\mathrm{g}} + r_{0\alpha}/t_0\right) \tag{11}$$

where the uncertainty of the counting time is neglected.

The relative standard uncertainty of w is calculated as given by Equation (12):

$$u_{\mathrm{rel}}^2(w) = u_{\mathrm{rel}}^2\left[\varepsilon_\beta(m)\right] + u_{\mathrm{rel}}^2(m) \tag{12}$$

The relative standard uncertainty of ε_β is calculated as given by Equation (13):

$$u_{\mathrm{rel}}^2\left[\varepsilon_\beta(m)\right] = u_{\mathrm{rel}}^2\left(r_{\mathrm{s}\beta} - r_{0\beta}\right) + u_{\mathrm{rel}}^2\left(A_\beta\right) = \left(r_{\mathrm{s}\beta}/t_{\mathrm{s}\beta} + r_{0\beta}/t_0\right)\Big/\left(r_{\mathrm{s}\beta} - r_{0\beta}\right)^2 + u_{\mathrm{rel}}^2\left(A_\beta\right) \tag{13}$$

The relative standard uncertainty of χ is calculated as given by Equation (14):

$$u(\chi) = \sqrt{\chi(\chi + 1)/\left(r_{\mathrm{s}\alpha} \cdot t_{\mathrm{s}\alpha}\right)} \tag{14}$$

For the calculation of the characteristic limits (see ISO 11929), one needs $\tilde{u}\left(\tilde{a}_\beta\right)$, i.e., the standard uncertainty of a_β as a function of its true value, calculated [7] as given by Equation (15):

$$\tilde{u}\left(\tilde{a}_\beta\right) = \sqrt{w^2\left\{\left[\tilde{a}_\beta/w + \chi\left(r_{\mathrm{g}\alpha} - r_{0\alpha}\right) + r_{0\beta}\right]\Big/t_{\mathrm{g}} + r_{0\beta}/t_0 + T(\chi)\right\} + \tilde{a}_\beta^2 \cdot u_{\mathrm{rel}}^2(w)} \tag{15}$$

If the alpha-beta cross-talk can be neglected, $\chi = 0$, and Equations (10) and (15) become similar to Equations (6) and (9).

7.3 Decision threshold

7.3.1 Decision threshold of the alpha activity per unit of mass

The decision threshold, a_α^* (see ISO 11929), is obtained from Equation (9) for $\tilde{a}_\alpha = 0$, which yields Equation (16):

$$a_\alpha^* = k_{1-\alpha} \cdot \tilde{u}(0) = k_{1-\alpha} \cdot w \cdot \sqrt{r_{0\alpha}/t_\mathrm{g} + r_{0\alpha}/t_0} \tag{16}$$

$\alpha = 0{,}05$ with $k_{1-\alpha} = 1{,}65$ is often chosen by default.

7.3.2 Decision threshold of the beta activity per unit of mass

The decision threshold, a_β^* (see ISO 11929), is obtained from Equation (15) for $\tilde{a}_\beta = 0$, which yields Equation (17):

$$a_\beta^* = k_{1-\alpha} \cdot \tilde{u}(0) = k_{1-\alpha} \cdot w \cdot \sqrt{\left[\chi\left(r_{\mathrm{g}\alpha} - r_{0\alpha}\right) + r_{0\beta}\right]\Big/t_\mathrm{g} + r_{0\beta}/t_0 + T(\chi)} \tag{17}$$

$\alpha = 0{,}05$ with $k_{1-\alpha} = 1{,}65$ is often chosen by default.

If the alpha-beta cross-talk can be neglected, $\chi = 0$, and Equation (17) becomes similar to Equation (16).

7.4 Detection limit

7.4.1 Detection limit of the alpha activity per unit of mass

The detection limit, $a_\alpha^\#$ (see ISO 11929), is calculated as given by Equation (18):

$$\begin{aligned} a_\alpha^\# &= a_\alpha^* + k_{1-\beta} \cdot \tilde{u}\left(a_\alpha^\#\right) \\ &= a_\alpha^* + k_{1-\beta} \cdot \sqrt{w^2\left[\left(a_\alpha^\#/w + r_{0\alpha}\right)\Big/t_\mathrm{g} + r_{0\alpha}/t_0\right] \cdot + \left(a_\alpha^\#\right)^2 \cdot u_\mathrm{rel}^2(w)} \end{aligned} \tag{18}$$

$\beta = 0{,}05$ with $k_{1-\beta} = 1{,}65$ is often chosen by default.

The detection limit can be calculated by solving Equation (18) for $a_\alpha^\#$ or, more simply, by iteration with a starting approximation of $a_\alpha^\# = 2 \cdot a_\alpha^*$.

By taking $\alpha = \beta$, then $k_{1-\alpha} = k_{1-\beta} = k$, and the solution of Equation (18) is given by Equation (19):

$$a_\alpha^\# = \frac{2 \cdot a_\alpha^* + \left(k^2 \cdot w\right)\Big/t_\mathrm{g}}{1 - k^2 \cdot u_\mathrm{rel}^2(w)} \tag{19}$$

7.4.2 Detection limit of the beta activity per unit of mass

The detection limit, $a_\beta^\#$ (see ISO 11929), is calculated as given by Equation (20):

$$\begin{aligned} a_\beta^\# &= a_\beta^* + k_{1-\beta} \cdot \tilde{u}\left(a_\beta^\#\right) \\ &= a_\beta^* + k_{1-\beta} \cdot \sqrt{w^2\left\{\left[a_\beta^\# / w + \chi\left(r_{\mathrm{g}\alpha} - r_{0\alpha}\right) + r_{0\beta}\right]\Big/t_\mathrm{g} + r_{0\beta} / t_0 + T(\chi)\right\} + \left(a_\beta^\#\right)^2 \cdot u_\mathrm{rel}^2(w)} \end{aligned} \tag{20}$$

$\beta = 0{,}05$ with $k_{1-\beta} = 1{,}65$ is often chosen by default.

The detection limit can be calculated by solving Equation (20) for $a_\beta^\#$ or, more simply, by iteration with a starting approximation of $a_\beta^\# = 2 \cdot a_\beta^*$.

If the alpha-beta cross-talk can be neglected, $\chi = 0$, and Equation (20) becomes similar to Equation (18).

By taking $\alpha = \beta$, then $k_{1-\alpha} = k_{1-\beta} = k$, and the solution of Equation (20) is given by Equation (21):

$$a_\beta^\# = \frac{2 \cdot a_\beta^* + \left(k^2 \cdot w\right) / t_\mathrm{g}}{1 - k^2 \cdot u_\mathrm{rel}^2\left(w\right)} \tag{21}$$

7.5 Confidence limits

The lower confidence limit, $a^\triangleleft$, and the upper confidence limit, $a^\triangleright$, are calculated using Equations (22) and (23):

$$a^\triangleleft = a - k_p \cdot u\left(a\right); \quad p = \omega \cdot \left(1 - \gamma/2\right) \tag{22}$$

$$a^\triangleright = a + k_q \cdot u\left(a\right); \quad q = 1 - \omega \cdot \gamma/2 \tag{23}$$

where $\omega = \Phi\left[y/u\left(y\right)\right]$

where Φ is the distribution function of the standardized normal distribution.

ω may be set equal to 1 if $a \geqslant 4 \cdot u\left(a\right)$, yielding Equation (24):

$$a^{\triangleleft,\triangleright} = a \pm k_{1-\gamma/2} \cdot u(a) \tag{24}$$

$\gamma = 0{,}05$ with $k_{1-\gamma/2} = 1{,}96$ is often chosen by default.

8 Test report

The test report shall be in accordance with the ISO/IEC 17025 requirements and shall contain the following information:

a) reference to this part of ISO 18589;

b) identification of the sample;

c) units in which the results are expressed;

d) test result, $a \pm u\left(a\right)$ or $a \pm U$, with the associated k value.

Complementary information can be provided, such as the following:

- probabilities α, β and $(1 - \gamma)$;
- the decision threshold and the detection limit; depending on the customer request, there are different ways to present the result:
  - when the activity per unit of mass, a, is compared with the decision threshold (see ISO 11929), the result of the measurement should be expressed as $\leqslant a^*$ when the result is below the decision threshold;

  - when the activity per unit of mass, a, is compared with the detection limit, the result of the measurement can be expressed as $\leqslant a^{\#}$ when the result is below the detection limit; if the detection limit exceeds the guideline value, it shall be documented that the method is not suitable for the measurement purpose;

- any relevant information likely to affect the results.

Annex A
(informative)

Preparation of calibration sources with plutonium 239

A.1 Principle

A known quantity of ^{239}Pu is added to a soil sample in an aqueous medium. Any loss in activity during the operation shall be taken into account.

A.2 Preparation of the spiked sample

The preparation of the spiked sample consists of the following stages.

a) Prepare the soil, following the procedure described in ISO 18589-2.

b) Transfer an aliquot of this soil (approximately 5 g) into a beaker, B1, whose weight is known.

c) Dry in an oven at (105 ± 10) °C for 1 h, leave to cool in a dessicator and determine the mass of the soil present by calculating the difference in mass, m_1.

d) Add 10 ml of water and stir vigorously for 1 h until a uniform solution is obtained.

e) Add by weighing a known quantity of standard ^{239}Pu, A_{th}, and stir for approximately 1 h.

f) Heat the solution whilst stirring until the sample has solidified.

g) Desiccate in the oven at (105 ± 10) °C to a constant mass.

h) Leave it to cool, then pass the spiked sample through a 200 µm sieve using a spatula. This is the reference soil.

A.3 Determination of the activity of the spiked sample

To determine the activity of the reference soil, the following operations shall be carried out.

a) Determine the mass of the reference soil, m_2.

b) Determine the activity of the reference soil, A_{exp}, following the procedure in ISO 18589-4.

A.4 Determination of the residual activity

The residual activity corresponds to the loss of activity during the preparation of the spiked sample. To determine this activity, the following operations can be carried out.

a) Place the sieve and agitator used in beaker B1.

b) Rinse with a nitric acid solution, $c(\text{HNO}_3) = 4$ mol/l, and retain the rinsing solution.

c) Determine the residual activity, A_{res}, of ^{239}Pu by measuring the α-activity of the rinsing solution.

A.5 Preparation and measurement of the calibration source

The procedure is carried out as follows.

a) Deposit a thin layer of a known quantity, m_3, of reference soil on a clean stainless steel test dish following the procedure defined in 6.1. This is the calibration source.

b) Add the fixer to the planchette.

c) Determine the α count rate of the calibration source using the same equipment conditions as used for all the measurements.

To prepare these calibration sources, the same quantities of spiked soil and fixer shall be used as during the preparation of the sources to be measured (see 6.1).

A.6 Determination of the efficiency

If, with the exception of the uncertainties, Equation (A.1) is verified, the counting efficiency can be determined using Equation (A.2):

$$A_{\text{exp}} = A_{\text{th}} - A_{\text{res}} \tag{A.1}$$

$$\varepsilon_{\alpha} = \frac{\left(r_{\text{s}\alpha} - r_{0\alpha}\right)}{A_{\alpha}} \tag{A.2}$$

where

$$A_{\alpha} = A_{\text{exp}} \cdot m_3 / m_2$$

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