

# **Procedure for determining radionuclides in precipitation by gamma spectrometry**

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Authors:

W. Kiesewetter  
H. Diedrich  
W. Dyck  
T. Steinkopff  
H. Ulbricht

Federal coordinating office for air and precipitation  
(Leitstelle für Luft und Niederschlag)

# Procedure for determining radionuclides in precipitation by gamma spectrometry

## 1 Scope

The procedure described in the following serves to identify gamma-emitting radionuclides contained in precipitation (total deposition). It conforms to the requirements for measuring programmes of the AVV-IMIS (1) and the guidelines for the monitoring of emissions and immissions of nuclear installations (REI) (2).

## 2 Sampling

The total deposition (dry and wet deposition) is determined by using open collectors that collect both rain and dry deposition. Preconditions are a sufficiently large collection area and a culvert to a collection vessel that must be sized to suit both the collection interval and the maximum expected amount of rain.

The particular problems associated with sampling the constituents of rain are discussed in sections 3 and 4. Two different sampling methods that are employed in practice are described in the following.

### **Sampling installations at the measuring points for radioactivity of the DWD**

Tubs made from enamelled stainless steel (Figure 1) are set up at the measuring points for radioactivity of the Deutscher Wetterdienst (DWD). Their collection surface covers ca. 0,64 m<sup>2</sup> and they are mounted ca. 1 m above the ground. Each tub is flush-mounted in the top of a cupboard made from zinc-plated sheet metal and painted with weather-resistant paint. The cupboard can be locked, is insulated against heat, and contains a plastic collection container (20 litres) for the drained deposition. The interior of the cupboard can be heated by means of two thermostat-controlled infrared heaters of 250 Watt each. This heating is meant to melt ice and snow accumulating on the collection surfaces and at the same time prevent the freezing of precipitation in the collection vessel. If the collected snow does not melt completely in the tub, it is recovered manually, melted in the laboratory, and then added to the liquid precipitation collected.

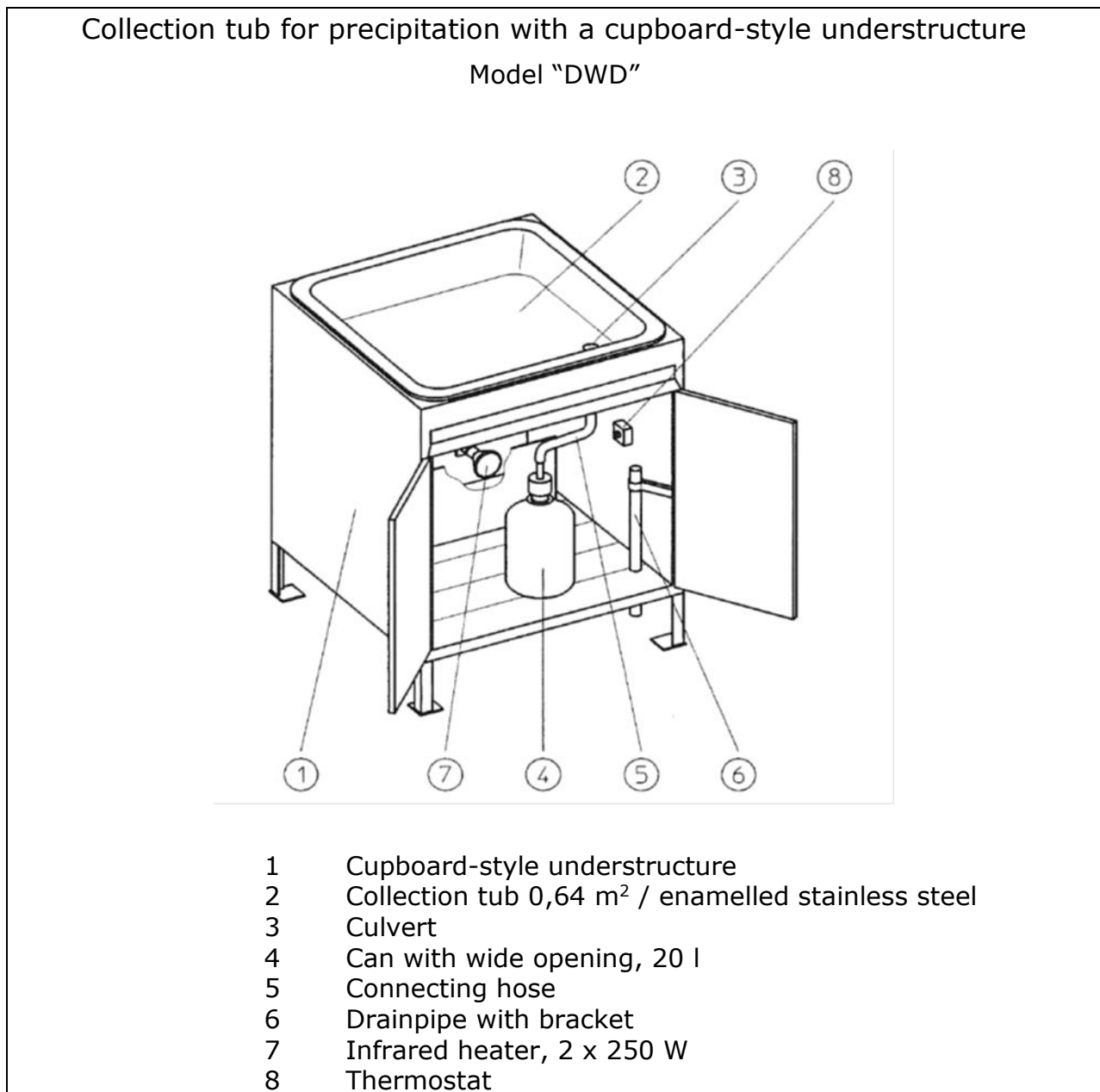
The collection of snow is subject to varying degrees of collection efficiency, depending on wind speeds.

### **Alternative sampling installation for precipitation**

An alternative to the collector configuration of the DWD is the funnel-type configuration illustrated in Figure 2. It is described in the recommendations for the monitoring of radioactivity in the environment (5) by the Arbeitskreis Umweltüberwachung (AKU) and employed, for example, at monitoring points for radioactivity in Switzerland.

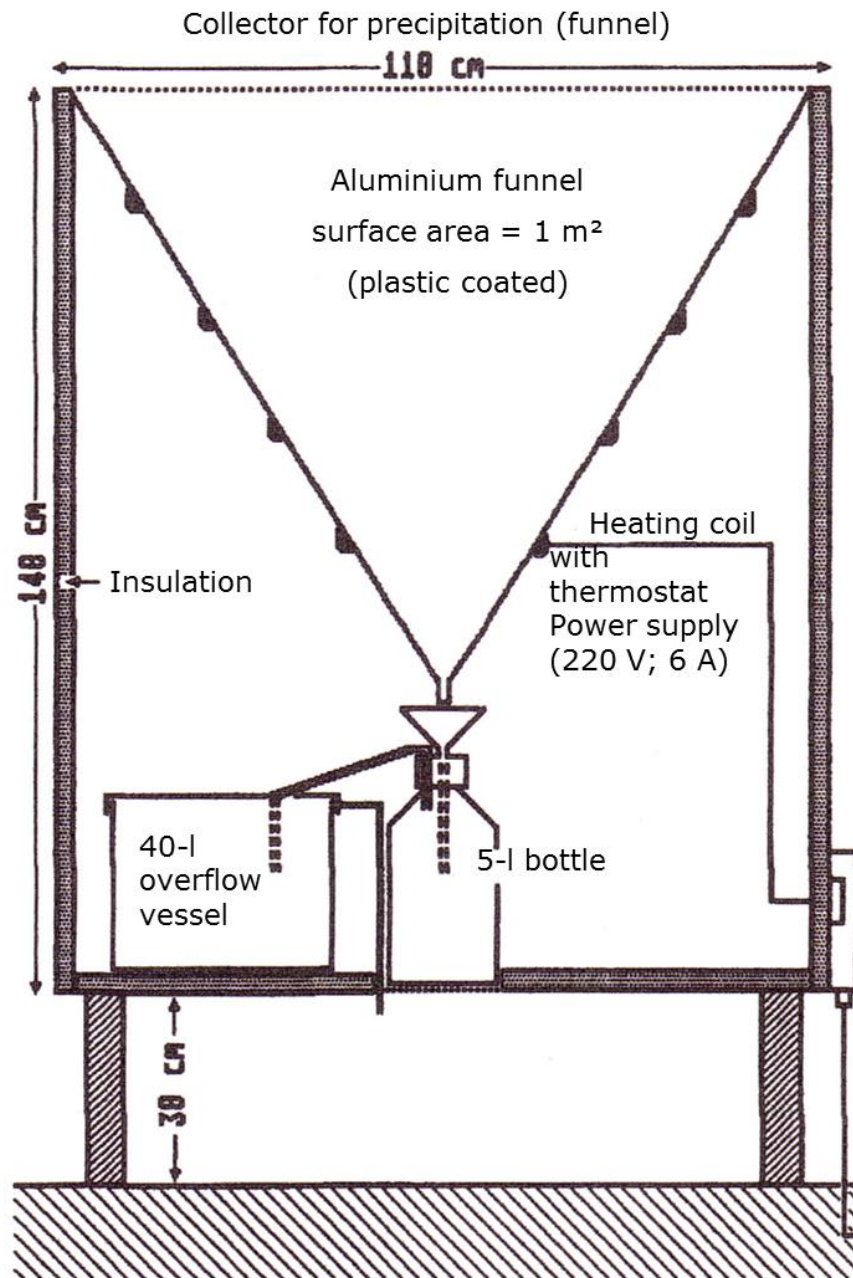
The funnel has a collection surface of 1 m<sup>2</sup> so that, in an ideal scenario, 1 mm of precipitation will correspond to one litre. It is made of welded sheet aluminium (thickness 2 mm, with a fine brushed finish on the inside) and coated with a weather-resistant plastic coating (e. g., RILSAN PA 11, grey) that minimises the adhesion of particles to the funnel surface. The outside wall of the precipitation-

collection casing (2 mm sheet iron) is hot-dip galvanized and painted in a light colour to prevent overheating of the funnel in summer. The inside of the casing is insulated with foam rubber.



**Fig. 1:** Sampling installation as used at the measuring points for radioactivity of the DWD

By gluing a thermostat-controlled heating cable (ca. 1 kW) to the outside of the funnel in winter, the collecting surface of the funnel is kept a few degrees above freezing point in order to also quantitatively collect precipitation as snow and prevent water collected on the collection surface from freezing. A proven alternative is to install an electrical heater (1 kW - 2 kW heating output) with a fan and a thermostat in the interior of the precipitation collector casing.



**Fig. 2:** Sampling installation for precipitation according to (5)

Collected liquid precipitation drains into a 5 litre plastic bottle that is also used for transporting the sample to a laboratory. In order to minimize the evaporation of water, the mouth of the funnel should have a small diameter (28 mm). A graduated overflow-collecting vessel with a lid and a capacity of at least 40 litres is placed next to the plastic collecting bottle to take excess precipitation water. Subsequent analyses are limited to the contents of the 5 litre-plastic can. The total amount of precipitation is quantified from the contents of both vessels, however.

The bottom of the precipitation collector casing is fitted with a grille or a piece of perforated sheet metal so that no water may collect in the casing. The precipitation collector is mounted on 30 cm stilts that are anchored in concrete foundations. Its operation requires a power supply of 220 V and 6 A.

## 3 Analysis

### 3.1 Principle of the procedure

If the concentration of radionuclides is of the order of  $1 \text{ Bq}\cdot\text{l}^{-1}$  or higher, a defined volume is measured directly in a plastic bottle. No preparatory enrichment of the radionuclides is required, and the required measuring period is of the order of magnitude of several hours. Suspended particles need to be distributed homogeneously during the measuring process. This can be achieved by stirring the sample, for example.

If the activity concentration is of the order of a few  $\text{mBq}\cdot\text{l}^{-1}$ , the direct measurement without preceding enrichment will not be sufficiently sensitive. In this case, a sufficiently large amount of precipitation is carefully evaporated. The remaining concentrate or residue is measured thereafter. Here, the volatility of certain radionuclides, such as I-131, needs to be taken into consideration.

### 3.2 Sample preparation

The collected precipitation is removed from the collection bottle and its volume measured. The amount of precipitation (mm, or  $\text{l}\cdot\text{m}^{-2}$ ) at the point of collection is recorded by means of standardized meteorological instruments (e. g., Hellmann collector).

In the case of open collectors, the proportion of dry depositions to the total deposition depends on the collecting period, the location, and the meteorological situation. The result of the collection of precipitation, with respect to fixed collection intervals, will be present either as both wet and dry deposition or only as a dry deposition:

#### a) Wet and dry deposition

It can be supposed that the sedimenting, insoluble particles accumulate at the bottom of the collecting bottle whereas soluble and finely dispersing constituents are distributed throughout the aqueous precipitation. In order to record the dry deposition during extended collection intervals, the collection surface is flushed with 0,5 litre to 1 litre of deionised water at shorter intervals, e. g., daily in the case of a monthly sampling period. The water used for flushing is thus added to the sample and must be subtracted when the total volume is quantified later.

The following approaches are available:

- Processing the entire precipitation sample  
The collection interval, the collection surface, and the collecting vessel need to be designed in order for the required detection limit to be attained. The activity per unit ground area is calculated from the activity concentration that is attributed to the cumulative sample and the precipitation height.
- Taking a partial sample  
Prior to taking a partial sample, the entire sample is stirred thoroughly in order to achieve a homogeneous distribution of its constituents. Without permitting a renewed deposition of the fine and coarse particles, an aliquot of this suspension is smoothly poured into a sufficiently large container and measured in a measuring cylinder prior to further processing or shipping as a mixed sample. The activity per unit ground area is calculated from the activity concentration that is attributed to the cumulative sample and the precipitation height.

## **b) Dry deposition**

To record the dry deposition ( $< 0,05$  mm precipitation during the collection period), the collection tub is flushed three times with 150 ml to 200 ml of deionised water each after the predetermined sampling interval. Using a rubber wiper, coarse particles are washed into the drain hole with the first flushing, and smaller particles that may still be adhering will then be collected in the same manner with the second flushing. Remnant deposits in the corners of the tub are flushed out with the final flushing. The activity of the dry deposition contained in the flushing water is measured right away or following an evaporation process. The activity per unit ground area is calculated from the activity recorded and the collection surface.

For enrichment of the radioactive substances, the entire amount of precipitation collected during the collection interval is employed. The monthly amount of precipitation to be processed, collected with the sampling installation of the DWD, averages about 70 litres. Even if the collection interval is long (e. g., a month), samples should preferably be recovered daily. The volume of the precipitation sample needs to be recorded before it is transferred to an evaporation vessel (stainless steel pot or ceramic bowl with 5 l in net volume). By carefully evaporating it (no visible boiling) while continuously adding more of the precipitation sample, a thick but pourable evaporation residue is produced. The latter is deposited in a petri dish and further desiccated under a heating lamp until it forms a homogeneous deposit on the bottom of the dish.

## **3.3 Radiochemical separation**

No radiochemical separation is required for the procedure described here.

# **4 Measuring the activity**

## **4.1 General**

An ultrapure germanium detector with a relative detection efficiency between 20 % and 60 % should be employed. Here, the detection efficiency refers to a  $3 \times 3''$  NaI(Tl) crystal and a half-width of the 1332 keV gamma line of the radionuclide Co-60 of  $< 2,0$  keV. The shielding of the detector should be made of minimum-radiation lead at least 10 cm thick. A sheet of copper needs to be installed between the lead shield and the detector in order to suppress the X-ray radiation of lead at 76 keV and 85 keV, and Perspex.

Further details on gamma spectrometry are outlined in chapter IV.1.1 of this procedures manual.

For a direct measurement, a defined volume, but a maximum of 1 litre of the aqueous sample or the flush water is taken from the precipitation collection vessel and transferred to a 1 litre-plastic bottle (Kautex) with a wide opening. If suspended particles are present, the sample needs to be stirred during the measuring process.

## **4.2 Calibration**

Calibration is effected with a corresponding volume or area-activity calibration source that contains several radionuclides of known activity. The area-activity standard source for the calibration is placed on a spacer 1 mm high in a plastic petri dish, placed on the detector, and then measured.

## 5 Calculation of the results

The calculation of the activity concentration,  $c_r$ , at the time the sample was collected, according to sections 7.2 and 7.4 of chapter IV.1.1 of this procedures manual, corresponds to:

$$c_r = \frac{N_n}{\varepsilon_r \cdot p_\gamma \cdot t_m \cdot V} \cdot f_1 \cdot f_2 \cdot f_3 \quad (1)$$

with

$$f_2 = e^{\lambda_r \cdot t_A} \quad (2)$$

and

$$f_3 = \frac{\lambda_r \cdot t_m}{1 - e^{-\lambda_r \cdot t_m}} \quad (3)$$

where

$c_r$  activity concentration of the radionuclide,  $r$ , in  $\text{Bq} \cdot \text{l}^{-1}$ ;

$N_n$  net count number;

$\varepsilon$  detection efficiency of the radionuclide,  $r$ , in  $\text{Bq}^{-1} \cdot \text{s}^{-1}$ ;

$p_\gamma$  emission probability per nuclear transformation;

$t_m$  duration of sample measurement, in s;

$V$  aliquot of the sample volume, in l;

$t_A$  period from the middle of the sampling period to the commencement of measuring, in s;

$\lambda_r$  decay constant of the radionuclide,  $r$ , in  $\text{s}^{-1}$ ;

$f_1$  summation correction factor;

$f_2$  correction factor for the decay of radionuclides within the period from the middle of the sampling period to the commencement of measuring;

$f_3$  correction factor for the decay of the radionuclides during the measuring process.

The summation correction factor,  $f_1$ , is determined according to chapter IV.1, section 5, of this procedures manual.

If the period  $t_A$  or  $t_m$ , as may be the case, is much smaller than the half-life of the radionuclide to be measured,  $f_2 = 1$  or  $f_3 = 1$  will apply, respectively.

The activity per area,  $a_{F,r}$ , of the radionuclide,  $r$ , is given by

$$a_{F,r} = c_r \cdot V_F$$

where

$V_F$  amount of precipitation per area in  $\text{l} \cdot \text{m}^{-2}$ .

If only a dry deposition is available, the ratio of the volume of flush water (l) to the collecting surface ( $\text{m}^2$ ) of the sampling installation needs to be inserted in equations (4) and (8) for  $V_F$ .

According to chapter IV.5, section 2.2.3, of this procedures manual, the standard deviation,  $s(R_n)$  of the net count rate,  $R_n$ , is calculated as follows:

$$s(R_n) = \sqrt{\frac{R_n}{t_m} + \frac{b \cdot \bar{R}_0(E_\gamma)}{t_m} \cdot \left(1 + \frac{b}{2 \cdot L}\right)} \quad (5)$$

where

- $s(R_n)$  standard deviation of the net count rate, in  $s^{-1}$ ;
- $R_n$  net count rate, in  $s^{-1}$ ;
- $b$  base width of the gamma line, in number of channels;
- $\bar{R}_0(E_\gamma)$  mean background count rate per channel, in  $s^{-1}$ ;
- $E_\gamma$  energy of the gamma line;
- $L$  number of channels for determining the background.

For the standard deviation of the activity concentration,  $s(c_r)$ , the following equation applies:

$$s(c_r) = s(R_n) \cdot \frac{c_r}{R_n} = \frac{1}{\varepsilon_r \cdot p_\gamma \cdot V} \cdot f_1 \cdot f_2 \cdot f_3 \cdot s(R_n) \quad (6)$$

This results in a standard deviation of activity per area of:

$$s(a_{F,r}) = s(R_n) \cdot \frac{a_{F,r}}{R_n} = \frac{V_F}{\varepsilon_r \cdot p_\gamma \cdot V} \cdot f_1 \cdot f_2 \cdot f_3 \cdot s(R_n) \quad (7)$$

## 5.1 Worked examples

For demonstration purposes, worked examples are given where activity concentrations of Cs-137 and I-131 are derived from which the respective activity per area (deposition) is calculated.

### a) Cs-137 contained in precipitation

For calculating the activity concentration of Cs-137 contained in a monthly sample, the following numerical values are inserted:

$N_g$ = 3005;	$N_0$ = 408;
$N_n$ = 2597;	$R_n$ = $3,61 \cdot 10^{-2} s^{-1}$ ;
$\varepsilon_{Cs-137}$ = $0,03734 Bq^{-1} \cdot s^{-1}$ ;	$p_\gamma$ = 0,851;
$b$ = 10;	$L$ = 5;
$R_0(E_\gamma)$ = $5,67 \cdot 10^{-4} s^{-1}$ ;	$E_\gamma$ = 662 keV;
$t_m$ = 72000 s;	$V$ = 66,97 l;
$t_A$ = $1,88 \cdot 10^6 s$ ;	$\lambda_{Cs-137}$ = $7,26 \cdot 10^{-10} s^{-1}$ ;
$f_1$ = 1,00;	$V_F$ = $72 l \cdot m^{-2}$ .

with

$$f_2 = e^{7,26 \cdot 10^{-10} \cdot 1,88 \cdot 10^6} = 1,00$$



and

$$f_3 = \frac{7,26 \cdot 10^{-10} \cdot 72000}{1 - e^{-7,26 \cdot 10^{-10} \cdot 72000}} = 1,00$$

These numerical values yield a Cs-137 activity concentration of:

$$c_{\text{Cs-137}} = \frac{2597}{0,03734 \cdot 0,851 \cdot 72000 \cdot 66,97} \cdot 1,00 \cdot 1,00 \cdot 1,00 \text{ Bq} \cdot \text{l}^{-1} = 1,69 \cdot 10^{-2} \text{ Bq} \cdot \text{l}^{-1}$$

and the activity of Cs-137 per unit area of:

$$a_F(\text{Cs} - 137) = 1,69 \cdot 10^{-2} \cdot 72 \text{ Bq} \cdot \text{m}^{-2} = 1,22 \text{ Bq} \cdot \text{m}^{-2}$$

The standard deviation of the net count rate is calculated as:

$$s(R_n) = \sqrt{\frac{3,61 \cdot 10^{-2}}{72000} + \frac{10 \cdot 5,67 \cdot 10^{-4}}{72000}} \cdot 2 \text{ s}^{-1} = 8,12 \cdot 10^{-4} \text{ s}^{-1}$$

which produces a standard deviation for the Cs-137 activity concentration of:

$$s(c_{\text{Cs-137}}) = 8,12 \cdot 10^{-4} \cdot \frac{1,69 \cdot 10^{-2}}{3,61 \cdot 10^{-2}} \text{ Bq} \cdot \text{l}^{-1} = 1,40 \cdot 10^{-6} \text{ Bq} \cdot \text{l}^{-1}$$

The result of this example calculation for measuring the Cs-137 activity concentration in precipitation is therefore:

$$c_{\text{Cs-137}} = (1,69 \pm 0,04) \cdot 10^{-2} \text{ Bq} \cdot \text{l}^{-1}$$

The standard deviation for the activity per area results in:

$$s(a_F(\text{Cs} - 137)) = 8,12 \cdot 10^{-4} \cdot \frac{1,217}{3,61 \cdot 10^{-2}} \text{ Bq} \cdot \text{m}^{-2} = 2,74 \cdot 10^{-2} \text{ Bq} \cdot \text{m}^{-2}$$

which produces a measured result of Cs-137 activity per area of:

$$a_F(\text{Cs} - 137) = (1,22 \pm 0,03) \text{ Bq} \cdot \text{m}^{-2}$$

## b) I-131 contained in precipitation (dry deposition)

The following numerical values are inserted:

$N_g$	= 1830;	$N_0$	= 1198;
$N_n$	= 632;	$R_n$	= $4,39 \cdot 10^{-2} \text{ s}^{-1}$ ;
$\epsilon_{\text{I-131}}$	= $0,044 \text{ Bq}^{-1} \cdot \text{s}^{-1}$ ;	$p_\gamma$	= 0,816;
$b$	= 10;	$L$	= 5;
$R_0(E_\gamma)$	= $8,32 \cdot 10^{-3} \text{ s}^{-1}$ ;	$E_\gamma$	= 364 keV;
$t_m$	= 14400 s;	$V$	= 1 l;
$t_A$	= 43200 s;	$\lambda_{\text{I-131}}$	= $1,00 \cdot 10^{-6} \text{ s}^{-1}$ ;
$f_1$	= 1,00;	$V_F$	= 1,0.

with

$$f_2 = e^{1,00 \cdot 10^{-6} \cdot 43200} = 1,04$$

and

$$f_3 = \frac{1,00 \cdot 10^{-6} \cdot 14400}{1 - e^{-1,00 \cdot 10^{-6} \cdot 14400}} = 1,007$$

The activity concentration of I-131 yields:

$$c_{\text{I-131}} = \frac{632}{0,044 \cdot 0,816 \cdot 14400 \cdot 1} \cdot 1,0 \cdot 1,04 \cdot 1,007 \text{ Bq} \cdot \text{l}^{-1} = 1,28 \text{ Bq} \cdot \text{l}^{-1}$$

Given a collecting surface of 0,64 m<sup>2</sup> and a flush water volume of 1 litre, the activity per area reads:

$$a_F(\text{I} - 131) = \frac{1,28 \cdot 1}{0,64} \text{ Bq} \cdot \text{m}^{-2} = 2,0 \text{ Bq} \cdot \text{m}^{-2}$$

The standard deviation of the net count rate amounts to:

$$s(R_n) = \sqrt{\frac{4,39 \cdot 10^{-2}}{14400} + \frac{10 \cdot 8,32 \cdot 10^{-3}}{14400}} \cdot 2 \text{ s}^{-1} = 3,82 \cdot 10^{-3} \text{ s}^{-1}$$

which produces a standard deviation for the I-131 activity per area of:

$$s(a_F(\text{I} - 131)) = 3,82 \cdot 10^{-3} \cdot \frac{2,0}{4,39 \cdot 10^{-2}} \text{ Bq} \cdot \text{m}^{-2} = 0,174 \text{ Bq} \cdot \text{m}^{-2}$$

The result of this example calculation for measuring the I-131 activity concentration contained in a dry deposition therefore reads:

$$a_F(\text{I} - 131) = (2,0 \pm 0,17) \text{ Bq} \cdot \text{m}^{-2}$$

## 5.2 Consideration of uncertainties

The variation of individual measurements is owed to the statistical uncertainty in the counting of pulses in this measuring technique. A larger proportion of the total uncertainty arises from uncertainties associated with collecting samples.

Errors stemming from the use of a non-representative sample have to be estimated for each individual configuration, using sections 2 and 3.2 as a guideline.

## 6 Characteristic limits of the procedure

The detection limits for activity concentrations,  $g$ , and those for the activity per area,  $g_F$ , are calculated using equations (8) and (9), respectively:

$$g = \frac{1}{\varepsilon_r \cdot p_\gamma \cdot V} \cdot f_1 \cdot f_2 \cdot f_3 \cdot (k_{1-\alpha} + k_{1-\beta}) \cdot \sqrt{\frac{2 \cdot b \cdot \bar{R}_0(E_\gamma)}{t_m}} \quad (8)$$

$$g_F = g \cdot V_F = \frac{V_F}{\varepsilon_r \cdot p_\gamma \cdot V} \cdot f_1 \cdot f_2 \cdot f_3 \cdot (k_{1-\alpha} + k_{1-\beta}) \cdot \sqrt{\frac{2 \cdot b \cdot \bar{R}_0(E_\gamma)}{t_m}} \quad (9)$$

Applying the quantiles of the standard distribution,  $k_{1-\alpha} = 3,0$  and  $k_{1-\beta} = 1,645$ , and the numerical values used in section 5.1, the following equations arise:

### a) for Cs-137:

$$g = \frac{1}{0,03734 \cdot 0,851 \cdot 66,97} \cdot (3 + 1,645) \cdot \sqrt{\frac{2 \cdot 10 \cdot 5,67 \cdot 10^{-3}}{72000}} \text{ Bq} \cdot \text{l}^{-1} = 8,66 \cdot 10^{-4} \text{ Bq} \cdot \text{l}^{-1}$$

This produces a detection limit for the Cs-137 activity concentration of  $8,66 \cdot 10^{-4} \text{ Bq} \cdot \text{l}^{-1}$ , and:

$$g_F = 8,66 \cdot 10^{-4} \cdot 72 \text{ Bq} \cdot \text{m}^{-2} = 6,23 \cdot 10^{-2} \text{ Bq} \cdot \text{m}^{-2}$$

for the activity per area, the detection limit therefore reads  $6,23 \cdot 10^{-2} \text{ Bq} \cdot \text{m}^{-2}$ , depending on the amount of precipitation.

### b) for I-131:

$$g_F = \frac{1}{0,044 \cdot 0,816 \cdot 1 \cdot 0,64} \cdot (3 + 1,645) \cdot \sqrt{\frac{2 \cdot 8,32 \cdot 10^{-2}}{14400}} \text{ Bq} \cdot \text{m}^{-2} = 6,87 \cdot 10^{-1} \text{ Bq} \cdot \text{m}^{-2}$$

The detection limit for the area-related I-131 activity amounts to  $0,69 \text{ Bq} \cdot \text{m}^{-2}$ .

More examples for achievable detection limits using the procedure applied by the DWD, supposing an evaporated amount of precipitation of 51 litres, a factual volume of precipitation of  $65 \text{ l} \cdot \text{m}^{-2}$ , a relative detection efficiency by the high-purity germanium detector of 40 % at the 1332 keV gamma line of Co-60, and a measuring period of 24 h, are given in the following table:

Radionuclide	$g$ in $\text{Bq} \cdot \text{l}^{-1}$	$g_F$ in $\text{Bq} \cdot \text{m}^{-2}$
Zr-95	$6,34 \cdot 10^{-4}$	$4,12 \cdot 10^{-2}$
Nb-95	$3,91 \cdot 10^{-4}$	$2,54 \cdot 10^{-2}$
Cs-134	$3,58 \cdot 10^{-4}$	$2,33 \cdot 10^{-2}$
Ce-141	$7,09 \cdot 10^{-4}$	$4,61 \cdot 10^{-2}$
Ce-144	$2,07 \cdot 10^{-3}$	$1,35 \cdot 10^{-1}$

## 7 Catalogue of chemicals and equipment

### 7.1 Chemicals

- Deionised water.

### 7.2 Equipment

- Collecting tub for precipitation with heat-insulated sub-construction and radiant heater;
- Plastic collecting container for precipitation;
- Stainless steel pot or ceramic bowl with a capacity of 5 litre;
- Small ceramic bowls (e. g., 100 ml, 50 ml);
- Hotplate;
- Sand bath;
- Infrared heat;
- Rubber wiper;
- Glass petri dishes for evaporated samples (d = 50 mm) with a thin bottom;
- Plastic cans with a wide opening: 100 ml, 250 ml, 500 ml and 1000 ml;
- Calibration source that contains several radionuclides of known activity;
- Gamma spectrometry measuring system;
- Precipitation measuring instrument;
- Measuring cylinder.

### References

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