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Determination of the detection limit and decision threshold for ionizing radiation measurements —

Part 3: Fundamentals and application to counting measurements by high resolution gamma spectrometry, without the influence of sample treatment

Détermination de la limite de détection et du seuil de décision des mesurages de rayonnements ionisants —

Partie 3: Principes fondamentaux et application aux mesurages de comptage, par spectrométrie gamma haute résolution, sans l'influence du traitement d'échantillon

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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 3.

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.

International Standard ISO 11929-3 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 2, *Radiation protection*, Working Group WG 17 (formerly WG 2), *Radioactivity measurements*.

ISO 11929 consists of the following parts, under the general title *Determination of the detection limit and decision threshold for ionizing radiation measurements*:

— *Part 1: Fundamentals and application to counting measurements without the influence of sample treatment*

— *Part 2: Fundamentals and application to counting measurements with the influence of sample treatment*

— *Part 3: Fundamentals and application to counting measurements by high resolution gamma spectrometry, without the influence of sample treatment*

— *Part 4: Fundamentals and application to measurements by use of linear scale analogue ratemeters, without the influence of sample treatment*

Annex A of this part of ISO 11929 is for information only.

Introduction

This part of ISO 11929 addresses the field of ionizing radiation measurements in which events (in particular pulses) are counted by high resolution gamma spectrometry registrating a pulse-heights distribution (acquisition of a multichannel spectrum), for example on samples. It considers exclusively the random character of radioactive decay and of pulse counting and ignores all other influences (e.g. arising from sample treatment, weighing, enrichment or the instability of the test setup). It assumes that the distance of neighbouring peaks of gamma lines is not smaller than four times the full width half maximum (FWHM) of gamma line and that the background near to gamma line is nearly a straight line. Otherwise ISO 11929-1 or ISO 11929-2 should be used.

It is also assumed that the duration of measurement is smaller in relation to the half-life of the radionuclides involved and that the instrument dead-time losses are negligible. Wherever activities or specific activities are to be determined, it is assumed that the factors for the conversion of pulse rates into activities or specific activities (calibration factor) have been determined to such an accuracy that the influence of their uncertainty of measurement can be ignored.

So far the ISO/TC 85/WG 2 has had meetings in Berlin on 1989-06-06, in Geneva on 1990-01-24, in Berlin on 1990-06-11, in Cadarache on 1991-04-15, in Petten (the Netherlands) on 1992-04-27/29, in Rome on 1993-04-19/20, in Orlando on 1994-10-10/11 and in Albuquerque on 1996-02-16. The draft proposal has been approved for FDIS in Vienna on 1997-07-07/08.

This part of ISO 11929 was prepared in parallel with other International Standards prepared by WG2 (now WG 17): ISO 11932:1996, *Activity measurements of solid materials considered for recycling, re-use or disposal as non-radioactive waste,* and ISO 11929-1, ISO 11929-2 and ISO 11929-4, and is, consequently, complementary to these documents.

The other parts of ISO 11929 deal with counting measurements which take sample treatment into consideration analogue pulse-rate measurements, and specific problems which occur when this part of ISO 11929 is applied (e.g. in the case of integral measurements or continuous monitoring of activity flows).

Determination of the detection limit and decision threshold for ionizing radiation measurements —

Part 3:
Fundamentals and application to counting measurements by high resolution gamma spectrometry, without the influence of sample treatment

1 Scope

This part of ISO 11929 specifies suitable statistical values which allow an assessment of the detection capabilities in high resolution gamma spectrometric ionizing radiation measurements, without the influence of sample treatment [1 to 11]. For this purpose, statistical methods are used to specify the following two statistical values characterizing given probabilities of error.

— The decision threshold, which allows a decision to be made for each measurement with a given probability of error as to whether the registered pulses in a region of interest of the spectrum include a contribution by the sample (e.g. a peak of a gamma line of a nuclide in question).

— The detection limit, which specifies the minimum sample contribution which can be detected with a given probability of error using the measuring procedure in question. This consequently allows a decision to be made as to whether a measuring method defined in this part of ISO 11929 satisfies certain requirements and is consequently suitable for the given purpose of measurement.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this part of ISO 11929. For dated references, subsequent amendments to, or revisions of, such publications do not apply. However, parties to agreements based on this part of ISO 11929 are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

BIPM/IEC/IFCC/ISO/IUPAC/IUPAP/OIML Guide to the Expression of Uncertainty in Measurement. Geneva 1993.

3 Terms and definitions

For the purposes of this part of ISO 11929, the following terms and definitions apply.

3.1
measuring method
use of a high resolution gamma ray detector in combination with a multichannel analyser for measuring specific radionuclides

3.2
sample
whole amount or an aliquot of an inactive material, the content of radioactive nuclides of which has to be determined by ionizing radiation measurement

3.3
decision threshold
critical value of a statistical test for the decision between the null hypothesis $\rho_s = \rho_0$ and the alternative hypothesis $\rho_s > \rho_0$

NOTE It should be the value R_n^* which, when exceeded by the determined value R_n, is taken to indicate that the hypothesis should be rejected. The statistical test should be designed such that the probability of wrongly rejecting the hypothesis (error of the first kind) is equal to a value α which is fixed prior to commencement of the measurement.

3.4
detection limit
smallest expectation value of the counting rate of a net peak area or a region of interest of a spectrum that can be detected on given probabilities and, therefore, the smallest difference $\rho_n = \rho_s - \rho_0$ associated with the statistical test concerned for the decision between the hypothesis $\rho_s = \rho_0$ and the alternative hypothesis $\rho_s > \rho_0$ and having the following characteristic: if in reality $\rho_n \geqslant \rho$, the probability of wrongly not rejecting the hypothesis $\rho_s = \rho_0$ (error of second kind) shall be at most equal to a value β which is fixed prior to commencement of the measurement

NOTE The difference between using the decision threshold and using the detection limit is that measured values are to be compared with the decision threshold while the detection limit is to be compared with the guideline value.

3.5
confidence interval
interval including the true value of ρ_n in at least $(1-\gamma) \times 100$ % of all cases

3.6
region of interest
ROI
group of selected consecutive channels

NOTE The width of a ROI is given by the number of channels concerned.

3.7
gross effect
total of counted pulses in a region of interest, caused by the background effect and the radiation from a particular γ-transition from a nuclide in question in the sample (sample contribution)

3.8
background effect
that component of the total counted pulses, in a region of interest, which is not due to the full energy events of the γ-transition in question and which is mainly due to Compton scattering

3.9
region for determination of background effect
two regions of equal width on both sides of the region of interest (see Figure 1)

3.10
net effect
total of pulses of interest in the region of interest; it is the difference between gross effect and background effect

3.11
guideline value
value constituted by requirements on measuring procedures arising for scientific, legal or other reasons which are specified, for example, as activity, specific activity, dose rate, etc.

NOTE If necessary, a calibration factor can be determined using a radioactive reference standard.

4 Symbols

N_0 Number of background pulses in a ROI

N_s Number of gross effect pulses in a ROI

N_n Number of net pulses in a ROI

t Duration of the measurement (life time)

R_0 Background effect counting rate, $R_0 = N_0/t_0$ in a ROI

ρ_0 Expectation value of R_0

R_s Gross effect counting rate, $R_s = N_s/t_s$ in a ROI

ρ_s Expectation value of R_s

R_n Net effect counting rate, difference between gross and net effect counting rates, $R_n = R_s - R_0$

ρ_n Expectation value of R_n

R_n^* Decision threshold for the net counting rate R_n

ρ_n^* Detection limit for the expectation value of the net counting rate R_n

h Full width half maximum (FWHM) of a peak

b Width of region of interest (in channels)

l_1, l_2 Width of regions for estimating of background (in channels)

N_1, N_2 Total of numbers of background counts in l_1, l_2 respectively

α Error of the first kind; the probability of rejecting the null hypothesis $\rho_s = \rho_0$ for the alternative hypothesis $\rho_s > \rho_0$ when the null hypothesis is true

β Error of the second kind; the probability of accepting the null hypothesis $\rho_s = \rho_0$ against the alternative hypothesis $\rho_s > \rho_0$ when the null hypothesis is false

$1-\gamma$ Confidence level of the confidence interval for ρ_n

$k_{1-\alpha}$, $k_{1-\beta}$, $k_{(1-\gamma/2)}$ } Quantiles of the standard normal distribution (see Table 2)

5 Statistical values and confidence interval

5.1 Principles

It is assumed that the pulses registered in each channel of the multichannel spectrum form Poisson processes. Therefore, the number of pulses N in a region of interest of the spectrum exhibits a Poisson distribution with the expectation $\rho_s \cdot t$. The number of background pulses N_0 likewise exhibits a Poisson distribution with the expectation $\rho_0 \cdot t$. For the calculation of the statistical values, the Poisson distribution will be approximated by a normal distribution. For the estimation of the background in the region of interest, it is assumed that its expectation $\rho_0 \cdot t$ is a linear combination of the expectations of N_1, N_2. This is acceptable because of the proportionality of all expectations of the numbers of counted pulses in the channels to measuring duration t and the principle of superposition of net and background pulses in the spectrum.

5.2 Decision threshold

The decision threshold shall refer to the value R_n^* which, when exceeded by a measured mean net counting rate R_n, is taken to indicate that a sample contribution exists. Otherwise, it shall be assumed in each case that there is no sample contribution.

If this decision rule is observed, a wrong decision occurs with the probability α that there is a sample contribution when in fact only a background effect exists (error of the first kind).

The decision threshold is given by

$$R_n^* = k_{1-\alpha}\sqrt{\mathrm{var}(R_n = 0)} \qquad (1)$$

where $k_{1-\alpha}$ is a factor given in Table 2.

Formulae for calculation of the decision threshold are given in Table 1.

5.3 Detection limit

The detection limit shall refer to the smallest expectation of the net counting rate ρ_n for which a wrong decision occurs with the probability β (if the decision rule as specified in 5.2 is applied) that there is no sample contribution but only a background effect (error of the second kind).

To check whether a measuring procedure is suitable for the purpose of measurement, the detection limit shall be compared with a specified guideline value (e.g. specified requirements on the sensitivity of the measuring procedure for scientific, legal or other reasons).

If in reality $\rho_n \geqslant \rho_n^*$, the probability of wrongly not rejecting the hypothesis $\rho_s = \rho_0$ (error of the second kind) shall be at most equal to a value β which is fixed prior to commencement of the measurement.

So with α and β the detection limit is

$$\rho_n^* = R_n^* + k_{1-\beta}\sqrt{\mathrm{var}(R_n = \rho_n^*)} \qquad (2)$$

$$= k_{1-\alpha}\sqrt{\mathrm{var}(R_n = 0)} + k_{1-\beta}\sqrt{\mathrm{var}(R_n = \rho_n^*)} \qquad (3)$$

and if $\mathrm{var}(R_n = 0) \approx \mathrm{var}(R_n > 0)$

$$\rho_n^* = (k_{1-\alpha} + k_{1-\beta})\sqrt{\mathrm{var}(R_n = 0)} \qquad (4)$$

If $\alpha = \beta$ then the detection limit is

$$\rho_n^* = 2\,R_n^* \qquad (5)$$

where $k_{1-\alpha}$, $k_{1-\beta}$ are factors given in Table 2.

Formulae for calculation of the detection limit are given in Table 1.

5.4 Confidence interval

By way of completion, formulae are given which can be used with a specified confidence level $(1-\gamma)$ to assign a confidence interval to each measured value. The confidence interval for ρ_n includes the true value of ρ_n in at least $(1-\gamma) \times 100$ % of all cases.

6 Application of this part of ISO 11929 (see annex A)

6.1 Specified values

The error probabilities α, β and the confidence level $1-\gamma$ shall be specified. Frequently cited values are $\alpha = \beta = \gamma = 0{,}025$. In special cases other values can be specified, see Table 2.

NOTE If $\alpha = \beta = \gamma/2$ are chosen and if var(R_n) varies just a little with R_n, one obtains for $R_n = R_n^*$ the confidence interval $R_n^* \pm k_{1-\alpha}\sqrt{\mathrm{var}(R_n)}$, that is the interval (0, ρ_n^*). This choice avoids a discontinuity in the expression of results.

6.2 Specifications of regions

If registered events of a single peak of definite localization in the spectrum should be determined and if this peak forms a Gaussian curve of FWHM h, the region of interest should be situated symmetrically over the peak with

$$h \leqslant b \leqslant 2{,}5h \quad (6)$$

and

$$b \geqslant 4 \text{ channels} \quad (7)$$

NOTE 1 Regions of interest of neighbouring peaks should not overlap.

The width of the region for the determination of the background should be (see Figure 1)

$$b \leqslant 2l \leqslant 10b \quad (8)$$

$$2l = l_1 + l_2 \quad (9)$$

NOTE 2 This region should not include tails of other peaks. The FWHM h can be determined or estimated from

— the known resolution of the device;
— a reference standard spectrum measured under identical conditions;
— neighbouring peaks.

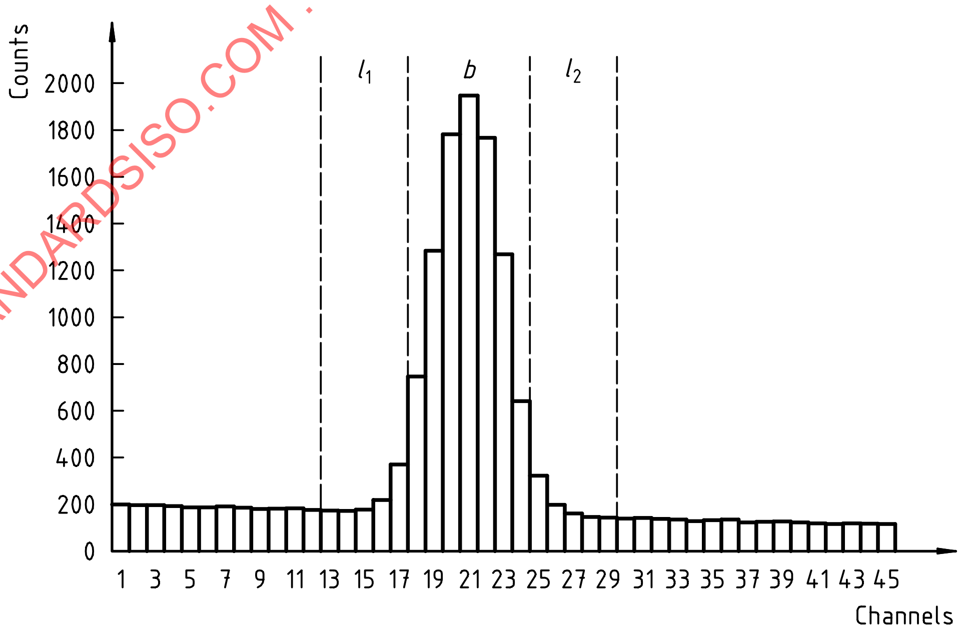

Figure 1 — Specification of regions on a peak

6.3 Determination of background

The number of background counts in the region of interest can be determined from channels of the regions A_1 and A_2 on both sides of the region of interest by the trapezoid rule:

$$N_0 = (N_1 + N_2)\frac{b}{2l} \tag{10}$$

Other procedures which give similar results for the background can also be used.

NOTE This part of ISO 11929 is not applicable to spectra on lines having the same nuclide.

6.4 Assessment of a measuring procedure

The decision as to whether a measuring method (3.1) satisfies certain requirements with respect to the detection limit shall be made by comparing the detection limit which has been determined with the specified guideline value (see 5.3).

The detection limit may be calculated by means of the formulae in Table 1.

If the detection limit thus determined is greater than the guideline value, the measuring procedure is not suitable for the purpose of the measurement.

NOTE Under certain circumstances, a measuring procedure may be suitable for the purpose of measurement, for example, by preselecting a greater duration of measurement or a higher number of pulses, by reducing the background effect, or by increasing sample quantity or chemical enrichment.

6.5 Assessment of measured results

The decision threshold shall be calculated by means of the formulae in Table 1.

A measured result shall be compared with the decision threshold thus obtained (see 5.2).

6.6 Documentation

A report on measurements in accordance with this part of ISO 11929 shall be accompanied by details on the probabilities of error, the decision threshold and the detection limit.

For established sample contributions, in addition to the measured value, confidence intervals determined in accordance with the equations in Table 1 shall also be reported. Activities can be calculated by means of a calibration factor (see 3.8).

Table 1 — Formulae for the calculation of statistical values when trapezoid method is used for the determination of the background

More exact formulae	Simplified formulae
Decision threshold $R_n^* = \frac{k_{1-a}^2}{2t} \cdot \frac{b}{2t} \cdot \left[1+\sqrt{\frac{4R_0 t}{k_{1-\alpha}} \cdot \frac{2l}{b}\left(1+\frac{2l}{b}\right)}\right]$ (11)	$R_n^* = k_{1-\alpha}\sqrt{\frac{R_0}{t}\left(1+\frac{b}{2l}\right)}$ (12) if $b = 2l$ $R_n^* = k_{1-\alpha}\sqrt{\frac{2R_0}{t}}$ (13)
Detection limit $\rho_n^* = \left(k_{1-\alpha}+k_{1-\beta}\right)\sqrt{\frac{\rho_0}{t}\left(1+\frac{b}{2l}\right)} + \frac{1}{4t}\left(k_{1-\alpha}+k_{1-\beta}\right)^2\left(1+\frac{b}{2l}\right)$ (14) Estimate for ρ_0 is R_0	$\rho_n^* = \left(k_{1-\alpha}+k_{1-\beta}\right)\sqrt{\frac{\rho_0}{t}\left(1+\frac{b}{2l}\right)}$ (15) if $b = 2l$ $\rho_n^* = \left(k_{1-\alpha}+k_{1-\beta}\right)\sqrt{\frac{2\rho_0}{t}}$ (16)
Confidence interval $R_n = k_{1-(\gamma/2)}\sqrt{\frac{R_s}{t}+\frac{R_0}{t}\cdot\frac{b}{2l}} \leqslant \rho_n \leqslant R_n + k_{1-(\gamma/2)}\sqrt{\frac{R_s}{t}+\frac{R_0}{t}\cdot\frac{b}{2l}}$ (17) if $b = 2l$ $R_n - k_{1-(\gamma/2)}\sqrt{\frac{R_s}{t}+\frac{R_0}{t}} \leqslant R_n + k_{1-(\gamma/2)}\sqrt{\frac{R_s}{t}+\frac{R_0}{t}}$ (18)	
NOTE These formulae are approximations, which are the more exact, the greater the number $\rho_0 \cdot t$ or $R_0 \cdot t$ respectively. For the values $\alpha = \beta = \gamma/2 = 0{,}025$, the following applies: The actual probability of an error of the first kind lies between 0,045 and 0,055 if, in the case where $b = 2l = 1$; 0,5; 0,25; 0,1, the true value of $\rho_0 \cdot t$ is $\geqslant$ 2; 5; 15; 25. Under these conditions, the actual probability of an error of the second kind lies between 0,04 and 0,055 and the confidence probability is greater than 0,94. The above shows that the range of application of the given formulae for $b = 2l$ is at its broadest, in particular, when small numbers of pulses are to be expected in the ROI. With small values of α, β, γ (e.g. < 0,01), the actual probabilities of error may deviate significantly more from the specified values. Anyone using this part of ISO 11929 should refer to [1] or his own statistical studies.	

Table 2 — Values $k_{1-\alpha}$, $k_{1-\beta}$, $k_{1-(\gamma/2)}$ as a function of the error probabilities α and β and of the confidence level $1-\gamma$ (quantiles of normal distribution)

Error of probability α or β	Confidence level $1-\gamma$	$k_{1-\alpha}$, $k_{1-\beta}$, $k_{1-(\gamma/2)}$
0,1586	0,682	1,000
0,1000	0,800	1,282
0,0500	0,900	1,645
0,0250	0,950	1,960
0,0228	0,955	2,000
0,0100	0,980	2,326
0,0050	0,990	2,576
0,0014	0,997	3,000
0,0010	0,998	3,090