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

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

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

Partie 2: Principes fondamentaux et application aux mesures par comptage, avec 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 11929-2 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) on samples are counted after treating them (e.g. aliquotation, solution, enrichment, separation). It considers, besides the random character of radioactive decay and of pulse counting, all other influences arising from sample treatment, (e.g. weighing, enrichment, calibration or the instability of the test setup).

In general, it can be assumed that the results of such measurements follow a mixture of Poisson distributions. Special kinds of mixtures of Poisson distributions are negative binominal distributions [1, 3, 8].

It is assumed part of ISO 11929 that the results from counting of samples and blanks follow a negative binominal distribution. If the influence of sample treatment and instability of the device is small in comparison to statistical counting errors, the results may be Poisson distributed. In this case, ISO 11929-1 can be applied for specification of the statistical values as long as the dispersion test [2] indicates that the Poisson distribution cannot be rejected.

It is also assumed that the duration of measurement is small compared to the half-life of the radionuclides concerned and that the instrument dead time losses are negligible.

This part of ISO 11929 was prepared in parallel with other International Standards prepared by WG 2 (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 11929-3 and ISO 11929-4 and is, consequently, complementary to these documents.

The other parts of ISO 11929 deal with counting measurements which do not take sample treatment into consideration, analogue pulse rate-measurement, and specific problems which occur when this part of ISO 11929 is applied (e.g. in the case of spectrometric measurements or continuous monitoring of radioactivity effluents).

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

Part 2:
Fundamentals and application to counting measurements with 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 ionizing radiation measurements with the influence of sample treatment. 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 include a contribution by the sample.

— 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
combination of a specific sample treatment procedure and the use of a measuring instrument for counting measurements under given conditions

3.2
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 null 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

3.3
detection limit
smallest expectation value of the net counting rate 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.

3.4
confidence interval
interval including the true value of ρ_n in at least $(1 - \gamma) \times 100$ % of all cases

3.5
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 after treatment

3.6
blank
aliquot of an inactive material with properties similar to the sample, which will be treated and measured in the same way as the sample

3.7
background effect
measured counting rate from the blank and radiation from external sources and radionuclides in the dectector and shielding

3.8
gross effect
measured counting rate from the sample (sample contribution) and the background radiation

3.9
net effect
⟨sample contribution⟩ gross effect minus the background effect

3.10
guideline value
value constituted by requirements on measuring procedures arising for scientific, legal or other reasons which are specified, for example, as activity, activity concentration, dose rate, etc.

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

4 Symbols

n_0 Number of measured blanks

n_m Number of measured spiked blanks

n_s Number of measured samples

n_u Number of external background measurings using a LSC cocktail with the same amount of demineralized water added instead of strontium solution in the device

$N_{0,i}$ Number of pulses counted with the i-th blank

$N_{m,i}$ Number of pulses counted with the i-th spiked blank

$N_{s,i}$ Number of pulses counted with the i-th sample

$N_{u,i}$ Number of pulses counted during the i-th run of external background measurement using a LSC cocktail with the same amount of demineralized water added instead of strontium solution

t_0 Duration of the measurement with a blank

t_s Duration of the measurement with a sample

t_m Duration of the measurement with a spiked blank

t_u Duration of the measurement with neither a blank nor a sample in the device

$R_{0,i}$ Background effect: counting rate with a blank, quotient of the pulses N_0 counted during the preselected duration of measurement t_0 and the duration of measurement t_0: $R_{0,i} = N_{0,i}/t_0$

$R_{s,i}$ Gross effect counting rate, quotient of the number of pulses counted during the preselected duration of measurement t_s and the duration of measurement t_s: $R_{s,i} = N_{s,i}/t_s$

$R_{m,i}$ Counting rate with a spiked blank, quotient of the number of pulses counted during the preselected duration of measurement t_m and the duration of measurement t_m: $R_{m,i} = N_{m,i}/t_m$

$R_{u,i}$ External background radiation counting rate using a LSC cocktail with the same amount of demineralized water added instead of strontium solution, quotient of the number of pulses counted during the preselected duration of measurement t_u and the duration of measurement t_u: $R_{u,i} = N_{u,i}/t_u$

$\overline{R_0}$ Mean of all $R_{0,i}$

$\overline{R_s}$ Mean of all $R_{s,i}$

$\overline{R_m}$ Mean of all $R_{m,i}$

$\overline{R_u}$ Mean of all $R_{u,i}$

$\overline{R_n}$ $\overline{R_n} = \overline{R_s} - \overline{R_0}$

ρ_0 Expectation value of all $R_{0,i}$

ρ_s Expectation value of all $R_{s,i}$

ρ_m Expectation value of all $R_{m,i}$

ρ_u Expectation value of all $R_{u,i}$

ρ_n Expectation value of all $R_{n,i}$

R_n^* Decision threshold for the net counting rate R_n (see Tables 1 and 2)

ρ_n^* Detection limit for the expectation value of the net counting rate R_n (see Tables 1 and 2)

σ_0^2 Variance of all $R_{0,i}$

σ_s^2 Variance of all $R_{s,i}$

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 (see Tables 1 and 2)

θ Relative standard deviation of the error due to the influence of the sample treatment and of the device instability

$k_{1-\alpha}$, $k_{1-\beta}$, $k_{(1-\gamma/2)}$ Quantiles of the standard normal distribution (see Table 3)

$t_{1-\alpha,f}$ Quantiles of the t-distribution for degree of freedom f (see Table 4)

5 Statistical values and confidence interval

5.1 Principles

5.1.1 General aspects

The definition of the statistical values for decision threshold, detection limit and confidence interval are based on the variances of the measured results. They are a combination of the variations caused by counting statistics, measurement equipment instability and sample treatment. Measurement equipment instability normally can be neglected because usually it is small in comparison to the other influences. The contribution of counting statistics can be calculated by the Poisson formula. The parameter describing the contribution of the sample treatment can be determined by special experiments with spiked blanks, by experimental experience or with reduced quality by the variances of the given measured results.

5.1.2 Model

If device instabilities are neglected, the following model can be applied. The number of pulses, N_0, counted with a blank is the sum of background radiation (external sources and detector noise) and matrix radiation.

The number of pulses, N_s, counted with a sample is the sum of background, matrix and sample radiation (net counting):

$$N_s = N_0 + N_n \quad (1)$$

It is assumed that, for constant radioactive emission, the numbers of pulses counted are Poisson distributed. In this case, the expectations of the counting rates R_0 and R_s are ρ_0 and $\rho_s = \rho_0 + \rho_n$, respectively.

The variances of R_0 and R_s are ρ_0/t_0 and ρ_s/t_s, respectively.

In a more general case than the previous one, it can be assumed that the sampling error (and errors caused by sample treatment) related to the sum $(\rho_0 + \rho_n)$ is a random variable and its relative standard deviation is θ. The expectations of R_0 and R_s are not modified but the variances of them become

$$\mathrm{var}(R_0) = \rho_0/t_0 + (\rho_0 - \rho_u)^2\theta^2 \text{ and } \mathrm{var}\left(\overline{R_0}\right) = 1/n_0[\rho_0/t_0 + (\rho_0 - \rho_u)^2\theta^2] \quad (2)$$

$$\mathrm{var}(R_s) = \rho_s/t_s + (\rho_s - \rho_u)^2\theta^2 \text{ and } \mathrm{var}\left(\overline{R_s}\right) = 1/n_s[\rho_s/t_s + (\rho_s - \rho_u)^2\theta^2] \quad (3)$$

The net counting rate $R_n = (R_s - R_0)$ has the expectation ρ_n and the variance

$$\mathrm{var}(R_n) = \mathrm{var}(R_0) + \mathrm{var}(R_s) \tag{4}$$

In this case, ρ_0 and ρ_s are unknown parameters; the measured values R_0, R_s can be taken as estimates and θ can be obtained separate measurements.

5.1.3 Determination of parameter θ

The parameter $\theta^2 \geqslant 0$ is assumed to be well known, if it is determined using a line of at least $n_m > 20$ spiked blanks. The activity added to the blank for spiking should be high enough to make the variance of counting statistics small in comparison to the expected variance of sample treatment. θ can be calculated using this formula:

$$\theta^2 = \frac{1}{\left(\overline{R_m} - \rho_u\right)^2} \left[\frac{\sum_{i=1}^{n_m} \left(R_{m,1} - \overline{R_m}\right)^2}{n_m - 5/8} - \frac{\overline{R_m}}{t_m} \right] \tag{5}$$

If the result is negative, take $\theta^2 = 0$.

ρ_u should be determined with very high precision.

It is assumed, that θ is the same for a spiked blank and a sample.

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).

If θ is known the decision threshold is given by

$$R_n^* = k_{1-\alpha} \sqrt{\mathrm{var}\left(R_n = 0\right)} \tag{6}$$

where $k_{1-\alpha}$ is a factor given in Table 3.

Formulae for calculation of the decision threshold are given in Table 1 (if θ is known) and in Table 2 (if θ is unknown), respectively.

5.3 Detection limit

The detection limit shall refer to the smallest expectation of the net counting rate ρ 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).

So, if θ is known and with α and β, the detection limit is

$$\rho_n^* = R_n^* + k_{1-\beta}\sqrt{\mathrm{var}(R_n = \rho_n^*)} \tag{7}$$

$$= k_{1-\alpha}\sqrt{\mathrm{var}(R_n = 0)} + k_{1-\beta}\sqrt{\mathrm{var}(R_n = \rho_n^*)} \tag{8}$$

and if var(R_n = 0) ≈ var(R_n > 0)

$$\rho_n^* = \left(k_{1-\alpha} + k_{1-\beta}\right)\sqrt{\mathrm{var}(R_n = 0)} \tag{9}$$

If $\alpha = \beta$ as recommended in ISO 11929-1, the detection limit is

$$\rho_n^* = 2\,R_n^* \tag{10}$$

where $k_{1-\alpha}$, $k_{1-\beta}$ are factors given in Table 3.

Formulae for calculation of the detection limit under different conditions are given in Table 1 (if θ is known) and in Table 2 (if θ is unknown), respectively.

5.4 Confidence interval

Formulae for calculation of the confidence interval are given in Table 1 (if θ is known) and in Table 2 (if θ is unknown).

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 in advance. Frequently cited values are $\alpha = \beta = \gamma = 0{,}025$. The number of blanks and samples shall be chosen as great as is reasonably achievable. The measuring times shall be chosen such that the detection limit is below the guideline value.

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 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).

This may be carried out either in advance, for the assessment of an intended measuring method on the basis of an empirically determined value for the background effect or a separate measurement, or else in retrospect, for the assessment of a measurement already carried out on the basis of the blank results then available.

The detection limit may be calculated by means of the formulae in 5.3 or in Table 1 (if θ is known) or in Table 2 (if θ is unknown), respectively.

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.

6.3 Assessment of measured results

The decision threshold may be calculated by means of the formulae in Table 1 (if θ is known) and in Table 2 (if θ is unknown), respectively.

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

6.4 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 (if θ is known) and in Table 2 (if θ is unknown), and the confidence level shall also be reported.

Table 1 — Formulae for the calculation of statistical values if θ is known

$$q = \frac{n_s t_s}{n_0 t_0} \quad (11)$$

$$c_1 = \frac{1}{n_0 t_0} + \frac{1}{n_s t_s} \quad (12)$$

$$c_2 = \frac{1}{n_0} + \frac{1}{n_s} \quad (13)$$

$$c_3 = \rho_0\left(\frac{1}{n_0 t_0} + \frac{1}{n_s t_s}\right) + \theta^2(\rho_0 - \rho_u)^2\left(\frac{1}{n_0} + \frac{1}{n_s}\right) \quad (14)$$

$$c_4 = \frac{1}{2}\left(k_{1-\alpha} + k_{1-\beta}\right) \quad (15)$$

$$c_5 = \frac{q}{q+1} \quad (16)$$

Decision threshold

$$R_n^* = \frac{k_{1-\alpha}^2\, c_5\left[c_1 + 2c_2\theta^2\left|\overline{R_0} - \rho_u\right|\right]}{2\left[1 - k_{1-\alpha}^2\theta^2 c_2 c_5^2\right]} \times \left[1 + \sqrt{1 + \frac{4\left[1 - k_{1-\alpha}^2\theta^2 c_2 c_5^2\right]\left[c_1\overline{R_0} + c_2\theta^2\left(\overline{R_0} - \rho_u\right)^2\right]}{k_{1-\alpha}^2\, c_5^2\left[c_1 + 2c_2\theta^2\left|\overline{R_0} - \rho_u\right|\right]^2}}\right] \quad (17)$$

If $n_s \cdot t_s \cdot \rho_0$ is big enough and θ is small, the following simplified formula may be applied

$$R_n^* = k_{1-\alpha}\sqrt{\overline{R_0}\left(\frac{1}{n_0 t_0} + \frac{1}{n_s t_s}\right) + \theta^2\left(\overline{R_0} - \rho_u\right)^2\left(\frac{1}{n_0} + \frac{1}{n_s}\right)} \quad (18)$$