DRAFT INTERNATIONAL STANDARD ISO/DIS 11843-6

ISO/TC **69**/SC **6** Secretariat: **JISC**

Voting begins on
2010-11-22

Voting terminates on
2011-04-22

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Capability of detection —

Part 6:
Methodology for the determination of the critical value and the minimum detectable values in Poisson distribution measurements

Capacité de détection —

Partie 6: Méthodologie de détermination de la valeur critique et des valeurs minimales détectables dans les mesures de distribution selon la loi de Poisson

ICS 03.120.30; 17.020

In accordance with the provisions of Council Resolution 15/1993 this document is circulated in the English language only.

Conformément aux dispositions de la Résolution du Conseil 15/1993, ce document est distribué en version anglaise seulement.

To expedite distribution, this document is circulated as received from the committee secretariat. ISO Central Secretariat work of editing and text composition will be undertaken at publication stage.

Pour accélérer la distribution, le présent document est distribué tel qu'il est parvenu du secrétariat du comité. Le travail de rédaction et de composition de texte sera effectué au Secrétariat central de l'ISO au stade de publication.

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

iTeh STANDARD PREVIEW
(standards.iteh.ai)
Full standard:
https://standards.iteh.ai/catalog/standards/sist/bb9cdfb6-
f505-44e8-8d28-da8cad5c524e/iso-11843-6-2013

Copyright notice

This ISO document is a Draft International Standard and is copyright-protected by ISO. Except as permitted under the applicable laws of the user’s country, neither this ISO draft nor any extract from it may be reproduced, stored in a retrieval system or transmitted in any form or by any means, electronic, photocopying, recording or otherwise, without prior written permission being secured.

Requests for permission to reproduce should be addressed to either ISO at the address below or ISO’s member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Reproduction may be subject to royalty payments or a licensing agreement.

Violators may be prosecuted.

© ISO 2010 – All rights reserved

Contents

Page

Figures

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 11843-6 was prepared by Technical Committee ISO/TC 69, *Application of statistical methods*, Subcommittee SC 6, *Measurement methods and results*.

ISO 11843 consists of the following parts, under the general title *Capability of detection*:

- *Part 1: Terms and definitions*
- *Part 2: Methodology in the linear calibration case*
- *Part 3: Methodology for determination of the critical value for the response variable when no calibration data are used*
- *Part 4: Methodology for comparing the minimum detectable value with given value*
- *Part 5: Methodology in the linear and non-linear calibration cases*
- *Part 6: Methodology for the determination of the critical value and the minimum detectable values in Poisson distribution measurements*

Introduction

Many types of instruments use the pulse-counting method for detecting signals. X-ray, electron and ion-spectroscopy detectors, such as X-ray diffractometer (XRD), X-ray fluorescence spectrometer (XRF), X-ray photoelectron spectrometer (XPS), Auger electron spectrometer (AES), and secondary ion mass spectrometer (SIMS), are of this type. These signals consist of a series of pulses produced at random and irregular intervals that can be understood statistically using a Poisson distribution. Since the variation in a signal follows a Poisson distribution, a methodology for determining the minimum detectable value can be deduced from statistical principles.

Determining the minimum detectable value of signals is sometimes important in practical work. It provides a criterion for deciding when "the signal is certainly not detected", or when "the signal is significantly different from the background noise level" [1-8]. For example, it is valuable when measuring the presence of hazardous substances or surface contamination of a semi-conductor materials. It also allows the condition of an analyzer to be quantified when assessing the limiting performance of an instrument.

The methods used to set the minimum detectable value have for some time been in widespread use in the field of chemical analysis, although not where pulse-counting measurements are involved. The need to establish a methodology to determine the minimum detectable value in that area is recognized [9].

This part of ISO 11843 considers how to approximate the Poisson distribution to the Normal distribution, ensuring consistency with the IUPAC approach and, particularly, with the series of ISO 11843 [10] standards. Two types of approximation, the Simple approximation and Square Root approximation, are used for this purpose. Definitions are given for the variance, for the critical value of the response variable, for the capability of detection criteria and for the minimum detectability level [17].

The methods considered in this standard should be applied only when the mean value of Poisson counts is not too small.

In this part of ISO 11843

- the probability is α of detecting (erroneously) that a system is not in the basic state when it is in the basic state;
- the probability is β of (erroneously) not detecting that a system, for which the value of the net state variable is equal to the minimum detectable value(x_d) is not in the basic state.

This standard is fully compliant with ISO 11843, part 1, 3 and 4.

Capability of detection —

Part 6:
Methodology for the determination of the critical value and the minimum detectable values in Poisson distribution measurements

1 Scope

This part of ISO 11843 specifies statistical values for assessing the capability of detection in Poisson distribution measurements and whether a specific signal is significant or not. It is applicable when variations in both the background noise and the signal are describable by the Poisson distribution. Two types of approximation, the Simple approximation and the Square Root approximation, are used to approximate the Poisson distribution to the Normal distribution, and are used to determine the variance, the critical value of the response variable, and the minimum detectable value. This part of ISO 11843 should be applied to a sufficient number of counts of the response variable.

2 Normative references

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

ISO 11843-1:1997, *Capability of detection - Part 1: Terms and definitions*
ISO11843-3: 2003, *Capability of detection - Part 3: Methodology for the determination of the critical value for the response variable when no calibration data are used*
ISO11843-4: 2003, *Capability of detection - Part 4: Methodology for comparing the minimum detectable value with a given value*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 3534 (all parts), ISO 5479, ISO 5725-2, ISO 11095, ISO 11843-1 and ISO Guide 30 apply.

4 Symbols

The meanings of the symbols used here are given in Annex A.

5 Measurement system and data handling

The conditions under which Poisson distribution measurement is made are usually specified by the experimental set-up. The number of pulses that are detected increases with both the time and with the width of the region over which the spectrum is being observed. These two parameters should therefore be noted and not changed during the course of the measurement.

The following conditions are essential for determining the minimum detectable value:

(1) Both the signal and the background noise should have Poisson distributions.

(2) The spectra under consideration should not have received any data treatment, such as smoothing.

(3) Time interval: A single measurement over a longer period of time is preferable to several shorter measurements. A measurement taken for over one second is better than 10 measurements over 100 milliseconds each. The approximation of the Poisson distribution by the Normal distribution is more reliable with higher mean values.

(4) The number of measurements: Since only mean values are used in the approximations presented here, repeated measurements are needed to determine them. Moreover, the detectability of the minimum value increases with the number of measurements.

(5) Number of channels used by the detector: There should be no overlap of neighboring peaks. The number of channels used to measure the background noise and sample spectra should be identical (Annex D, Figure 4).

(6) Peak width: The full width at half maximum (FWHM) is the recommended coverage for monitoring a single peak, and is preferable to measurements based on the top and/or bottom of the peak with noise. The appropriate FWHM should be determined beforehand by measurements on a standard sample. An identical value of the FWHM width should be adopted for both background noise and sample measurements.

NOTE Additional conditions for the interpretation of the data are: The instrument works correctly. The detector works in the linear counting range. Both the ordinate and the abscissa axes are calibrated. There is no signal that cannot be identified, except statistical fluctuation. Degradation of the specimen during measurement is negligibly small. At least one signal or peak that belongs to an element can be observed.

6 Computation by the simple approximation (Annex B.1)

6.1 General

The critical value, y_c ,is defined as the value of the response variable, y ,if the system is considered to move from a dormant or ‘basic’ state to an ‘active’ state. Its value is chosen so that, when it is in the ‘basic’ state, the system is considered to be ‘active’ with only a small probability, α. In other words, y_c is the lowest significant value of the measurement or signal that can be discriminated from the background noise [5,6,10].

The decision about whether the measured signal is ‘significant’ or ‘not significant’ is made by comparing the arithmetic mean of the actual measured values, $\bar{y}_a$,with the critical value, y_c. The probability that $\bar{y}_a$ exceeds the critical value, y_c, for the distribution in the basic state should be less than or equal to an appropriate pre-selected probability, α.

The critical value, y_c, of the response variable generally satisfies

$$P(\bar{y}_a > y_c | x = 0) \leq \alpha \,, \tag{1}$$

where

x	a value of net state variable;
y	a value of response variable;
$\bar{y}_a$	arithmetic mean of measured responses of an actual states;
α	the probability that an error of the first kind has occurred : the probability of rejecting the null hypothesis $\eta_g = \eta_b$ when the null hypothesis is true;
η_b	the expected value under the actual performance conditions for the responses of the basic state;

η_g	the expected value under the actual performance conditions for the responses of a sample with the net state variable equal to x_g .

6.2 Computation of the critical value of the response variable by the simple approximation

Eq. (1) gives the probability that $\bar{y}_{\mathrm{a}} > y_{\mathrm{c}}$ under the condition that $x = 0$. If the response variable follows a Poisson distribution with a large mean value, λ ,and the standard deviation, σ_0 ,equals to $\sqrt{\lambda}$, and then the critical value of the response variable is given by the following simplified version of Eq. (1):

$$y_{\mathrm{c}} = \bar{y}_{\mathrm{b}} \pm z_{1-\alpha}\sigma_0\sqrt{\frac{1}{J}+\frac{1}{K}} \ . \tag{2}$$

Also, if σ_0 is replaced by $\sqrt{\bar{y}_b}$, and then

$$y_{\mathrm{c}} = \bar{y}_{\mathrm{b}} \pm z_{1\text{-}\alpha}\sqrt{\bar{y}_{\mathrm{b}}}\sqrt{\frac{1}{J}+\frac{1}{K}} \quad , \tag{3}$$

where

$z_{1-\alpha}$	$(1-\alpha)$-quantile of the standard normal distribution;
σ_0	actual standard deviation at zero level of state variable;
$\bar{y}_{\mathrm{b}}$	observed mean response of the basic state;
J	number of replication of measurements on the reference material representing the value of the net state variable (blank sample) in an application of the method;
K	number of replication of measurements on the actual state (test sample) n an application of the method.

Note that the + sign is used when the response variable increases with increasing level of the net state variable and the - sign is used when the response variable decreases with increasing level of the net state variable. The + sign is preferable in Poisson distribution measurements as the response variable increases with increasing level of the net state variable. In general, when the value of the net state variable in the basic state (the baseline for the response variable) is known without a significant error, then $\sigma_0 = \sigma_{\mathrm{b}}$. The latter is estimated through $\bar{y}_{\mathrm{b}}$, the standard deviation of the repeated measurements of the response variable in the basic state. As mentioned in the *simple approximation* section, $\sqrt{\bar{y}_{\mathrm{b}}}$ gives an estimate of σ_{b}. The concept of the critical value is illustrated in Figure 1.

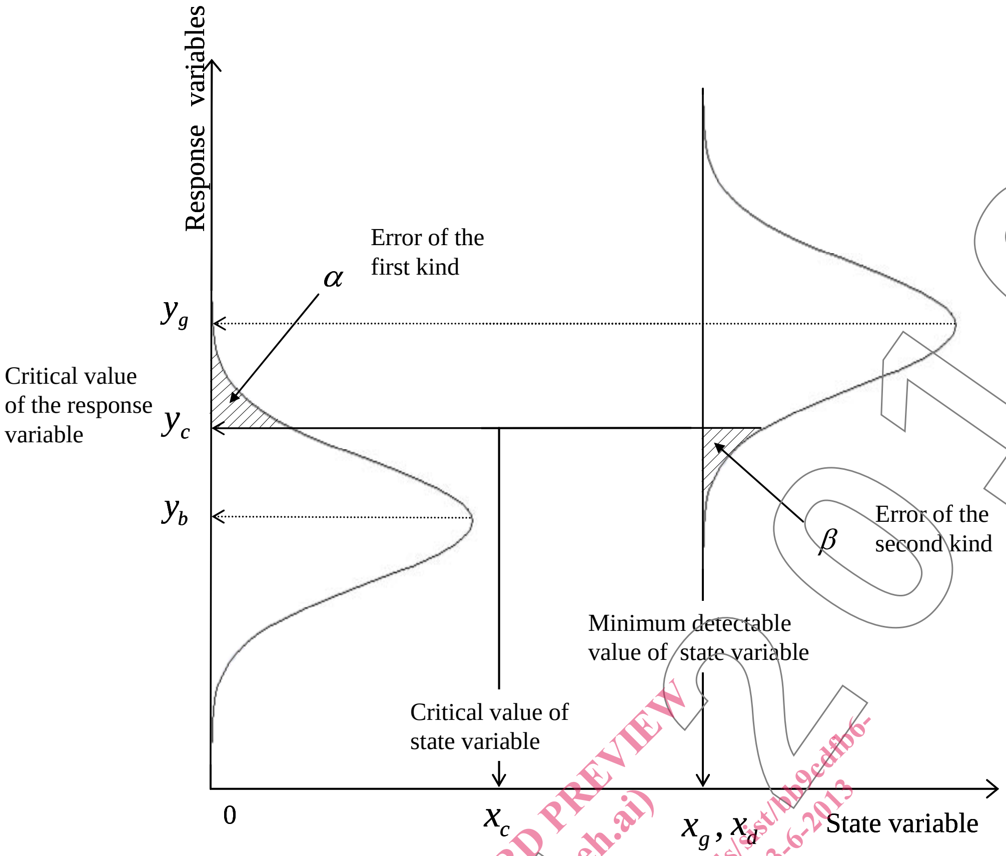

Figure 1 Relation between the critical value of the response variable and the minimum detectable value of the state variable [10].

6.3 Sufficient capability of the detection criterion by the simple approximation

The sufficient capability of detection criterion enables decisions to be made about the detection of a signal by comparing the critical value probability with a specified value, $1-\beta$. If the criterion is satisfied, it may be concluded that the minimum detectable value, x_d ,is less than or equal to the value of the net state variable, x_g .The minimum detectable value then defines the smallest expectation of the response variable, η_g ,for which an incorrect decision occurs with a probability, β, that there is no signal, but only background noise. This is termed an ‘error of the second kind’ [10].

If the standard deviation of the responses for a given value x_g is σ_g, the criterion for the probability to be greater than or equal to $1-\beta$ is defined by inequality (4), and it is actually given by inequalities (5) and (6):

$$\eta_g \ge y_c + z_{1-\beta}\sqrt{\frac{1}{J}\sigma_b^2+\frac{1}{K}\sigma_g^2}, \qquad (4)$$

If y_c is replaced by $\eta_b + z_{1-\alpha}\sigma_b\sqrt{\frac{1}{J}+\frac{1}{K}}$, defined in Eq. (2), then

$$\eta_g - \eta_b \ge z_{1-\alpha}\sigma_b\sqrt{\frac{1}{J}+\frac{1}{K}} + z_{1-\beta}\sqrt{\frac{1}{J}\sigma_b^2+\frac{1}{K}\sigma_g^2}, \qquad (5)$$