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**Capability of detection—
Part 2 : Methodology in the linear
calibration case**

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Foreword

This translation has been made based on the original Japanese Industrial Standard established by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee according to the proposal of establishing a Japanese Industrial Standard from the Japanese Standards Association (JSA), with a draft of Industrial Standard based on the provision of Article 12 Clause 1 of the Industrial Standardization Law.

This Standard has been made based on **ISO 11843-2 : 2000** *Capability of detection—Part 2 : Methodology in the linear calibration case* for the purposes of making easy to compare this Standard with International Standard; to prepare Japanese Industrial Standard conforming with International Standard; and to propose a draft of International Standard which is based on Japanese Industrial Standard.

JIS Z 8462 consists of the following two parts with the general title *Capability of detection*.

Part 1 : Terms and definitions

Part 2 : Methodology in the linear calibration case

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In the event of any doubts arising as to the contents,
the original JIS is to be the final authority.

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Capability of detection— Part 2 : Methodology in the linear calibration case

Introduction This part of **JIS Z 8462** has been prepared based on the first edition of **ISO 11843-2 : 2000** *Capability of detection—Part 2 : Methodology in the linear calibration case* published in 2000 without modifying the technical contents.

“Information” underlined with dots are the matters not included in the original International Standard.

An ideal requirement for the capability of detection with respect to a selected state variable would be that the actual state of every observed system can be classified with certainty as either equal to or different from its basic state. However, due to systematic and random distortions, this ideal requirement cannot be satisfied because:

- in reality all reference states, including the basic state, are never known in terms of the state variable. Hence, all states can only be correctly characterized in terms of differences from basic state, i.e. in terms of the net state variable.

In practice, reference states are very often assumed to be known with respect to the state variable. In other words, the value of the state variable for the basic state is set to zero; for instance in analytical chemistry, the unknown concentration or the amount of analyte in the blank material usually is assumed to be zero and values of the net concentration or amount are reported in terms of supposed concentrations or amounts. In chemical trace analysis especially, it is only possible to estimate concentration or amount differences with respect to available blank material. In order to prevent erroneous decisions, it is generally recommended to report differences from the basic state only, i.e. data in terms of the net state variable;

NOTE : In **JIS Q 0030** and in **JIS Z 8461** no distinction is made between the state variable and the net state variable. As a consequence, in these two documents reference states are, without justification, assumed to be known with respect to the state variable.

Information : **JIS Q 0033** rather describes statistical methodology in the calibration curve, while **JIS Q 0030** defines reference materials.

- the calibration and the processes of sampling and preparation add random variation to the measurement results.

In this part of **JIS Z 8462**, the following two requirements were chosen:

- 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.

1 Scope This part of **JIS Z 8462** specifies basic methods to:

- design experiments for the estimation of the critical value of the net state variable, the critical value of the response variable and the minimum detectable value of the net state variable,
- estimate these characteristics from experimental data for the cases in which the calibration function is linear and the standard deviation is either constant or linearly related to the net state variable.

The methods described in this part of **JIS Z 8462** are applicable to various situations such as checking the existence of a certain substance in a material, the emission of energy from samples or plants, or the geometric change in static systems under distortion.

Critical values can be derived from an actual measurement series so as to assess the unknown states of systems included in the series, whereas the minimum detectable value of the net state variable as a characteristic of the measurement method serves for the selection of appropriate measurement processes. In order to characterize a measurement process, a laboratory or the measurement method, the minimum detectable value can be stated if appropriate data are available for each relevant level, i.e. a measurement series, a measurement process, a laboratory or a measurement method. The minimum detectable values may be different for a measurement series, a measurement process, a laboratory or the measurement method.

JIS Z 8462 is applicable to quantities measured on scales that are fundamentally continuous. It is applicable to measurement processes and types of measurement equipment where the functional relationship between the expected value of the response variable and the value of the state variable is described by a calibration function. If the response variable or the state variable is a vectorial quantity the methods of **JIS Z 8462** are applicable separately to the components of the vectors or functions of the components.

Remarks : The International Standard corresponding to this Standard is as follows.

In addition, symbols which denote the degree of correspondence in the contents between the relevant International Standard and **JIS** are IDT (identical), MOD (modified), and NEQ (not equivalent) according to **ISO/IEC Guide 21**.

ISO 11843-2 : 2000 *Capability of detection—Part 2 : Methodology in the linear calibration case* (IDT)

2 Normative references The following standards contain provisions which, through reference in this Standard, constitute provisions of this Standard. If the indication of the year of coming into effect (or the year of publication) is given to these referred standards, only the edition of indicated year constitutes the provision of this Standard but the revision and amendment made thereafter are not applied. The normative references without the indication of the year of coming into effect (or the year of publication) apply limiting only to the most recent edition (including amendments).

JIS Q 0030 : 1997 *Terms and definitions used in connection with reference materials*

Remarks : **ISO Guide 30** : 1992 *Terms and definitions used in connection with reference materials* is identical with the said standard.

JIS Z 8101-1 : 1999 *Statistics—Vocabulary and symbols—Part 1 : Probability and general statistical terms*

Remarks : **ISO 3534-1** : 1993 *Statistics—Vocabulary and symbols—Part 1 : Probability and general statistical terms* is equivalent to the said standard.

JIS Z 8101-2 : 1999 *Statistics—Vocabulary and symbols—Part 2 : Statistical quality control terms*

Remarks : **ISO 3534-2** : 1993 *Statistics—Vocabulary and symbols—Part 2 : Statistical quality control* is equivalent to the said standard.

JIS Z 8101-3 : 1999 *Statistics—Vocabulary and symbols—Part 3 : Design of experiments*

Remarks : **ISO/FDIS 3534-3** : 1999 *Statistics—Vocabulary and symbols—Part 3 : Design of experiments* is identical with the said standard.

JIS Z 8461 : 2001 *Linear calibration using reference materials*

Remarks : **ISO 11095** : 1996 *Linear calibration using reference materials* is identical with the said standard.

JIS Z 8462-1 : 2001 *Capability of detection—Part 1 : Terms and definitions*

Remarks : **ISO 11843-1** : 1997 *Capability of detection—Part 1 : Terms and definitions* is identical with the said standard.

3 Terms and definitions For the purposes of this part of **JIS Z 8462**, the terms and definitions of **JIS Z 8101** (all parts), **JIS Q 0030**, **JIS Z 8461** and **JIS Z 8462-1** apply.

4 Experimental design

4.1 General The procedure for determining values of an unknown actual state includes sampling, preparation and the measurement itself. As every step of this procedure may produce distortion, it is essential to apply the same procedure for characterizing, for use in the preparation and determination of the values of the unknown actual state, for all reference states and for the basic state used for calibration.

For the purpose of determining differences between the values characterizing one or more unknown actual states and the basic state, it is necessary to choose an experimental design suited for comparison. The experimental units of such an experiment are obtained from the actual states to be measured and all reference states used for calibration. An ideal design would keep constant all factors known to influence the outcome and control of unknown factors by providing a randomized order to prepare and perform the measurements.

In reality it may be difficult to proceed in such a way, as the preparations and determination of the values of the states involved are performed consecutively over a period of time. However, in order to detect major biases changing with time, it is strongly recommended to perform one half of the calibration before and one half after

the measurement of the unknown states. However, this is only possible if the size of the measurement series is known in advance and if there is sufficient time to follow this approach. If it is not possible to control all influencing factors, conditional statements containing all unproven assumptions shall be presented.

Many measurement methods require a chemical or physical treatment of the sample prior to the measurement itself. Both of these steps of the measurement procedure add variation to the measurement results. If it is required to repeat measurements the repetition consists in a full repetition of the preparation and the measurement. However, in many situations the measurement procedure is not repeated fully, in particular not all of the preparational steps are repeated for each measurement; see note in 5.2.1.

4.2 Choice of reference states The range of values of the net state variable spanned by the reference states should include

- the value zero of the net state variable, i.e. in analytical chemistry a sample of the blank material, and
- at least one value close to that suggested by a prior information on the minimum detectable value; if this requirement is not fulfilled, the calibration experiment should be repeated with other values of the net state variable, as appropriate.

The reference states should be chosen so that the values of the net state variable (including log-scaled values) are approximately equidistant in the range between the smallest and largest value.

In case in which the reference states are represented by preparations of reference materials their composition should be as close as possible to the composition of the material to be measured.

4.3 Choice of the number of reference states, I and the (numbers of) replications of procedure, J , K and L The choice of reference states, number of preparations and replicate measurements shall be as follows:

- the number of reference states I used in the calibration experiment shall be at least 3; however, $I = 5$ is recommended;
- the number of preparations for each reference state J (including the basic state) should be identical; at least two preparations ($J = 2$) are recommended;
- the number of preparations for the actual state K should be identical to the number J of preparations for each reference state;
- the number of repeated measurements performed per preparation L shall be identical; at least two repeated measurements ($L = 2$) are recommended.

NOTE : The formulae for the critical values and the minimum detectable value in clause 5 are only valid under the assumption that the number of repeated measurements per preparation is identical for all measurements of reference states and actual states.

As the variations and cost due to the preparation usually will be much higher than those due to the measurement, the optimal choice of J , K and L may be derived from an optimization of constraints regarding variation and costs.

5 The critical values y_c and x_c and the minimum detectable value x_d of a measurement series

Information : Symbols y_c , x_c and x_d used here are identical with the symbols y_c , x_c and x_D used in **JIS Z 8462-1**. "The minimum detectable value" which is used hereafter means "the values of minimum detectable net state variable".

5.1 Basic assumptions The following procedures for the computation of the critical values and the minimum detectable value are based on the assumptions of **JIS Z 8461**. The methods of **JIS Z 8461** are used with one generalization; see **5.3**.

Basic assumptions of **JIS Z 8461** are that

- the calibration function is linear,
- measurements of the response variable of all preparations and reference states are assumed to be independent and normally distributed with standard deviation referred to as "residual standard deviation",
- the residual standard deviation is either a constant, i.e. it does not depend on the values of the net state variable [case 1], or it forms a linear function of the values of the net state variable [case 2].

The decision regarding the applicability of this part of **JIS Z 8462** and the choice of one of these two cases should be based on prior knowledge and a visual examination of the data.

5.2 Case 1—Constant standard deviation

5.2.1 Model The following model is based on assumptions of linearity of the calibration function and of constant standard deviation and is given by:

$$Y_{ij} = a + bx_i + \varepsilon_{ij} \dots\dots\dots (1)$$

where

- x_i is the symbol for the net state variable in state i ;
- ε_{ij} are random variables which describe the random component of sampling, preparation and measurement error.

It is assumed that the ε_{ij} are independent and normally distributed with expectation zero and the theoretical residual standard deviation σ : $\varepsilon_{ij} \sim N(0; \sigma^2)$. Therefore, values Y_{ij} of the response variable are random variables with the expectation $E(Y_{ij}) = a + bx_i$ and the variance $V(Y_{ij}) = \sigma^2$, not depending on x_i .

NOTE : In the cases in which J samples are prepared for measurement and each of them is measured L times so that $J \cdot L$ measurements are performed altogether for reference state i , then Y_{ij} refers to the average of the L measurements obtained on the prepared sample.

Information : $\varepsilon_{ij} \sim N(0; \sigma^2)$ indicates that it is followed the normal distribution with ε_{ij} being average value = 0, and standard deviation σ .

5.2.2 Estimation of the calibration function and the residual standard deviation In accordance with **JIS Z 8461**, estimates (see note) for a , b and σ^2 are given by:

$$\hat{b} = \frac{\sum_{i=1}^I \sum_{j=1}^J (x_i - \bar{x})(\bar{y}_{ij} - \bar{y})}{s_{xx}} \dots\dots\dots (2)$$

$$\hat{a} = \bar{y} - \hat{b}\bar{x} \dots\dots\dots (3)$$

$$\hat{\sigma}^2 = \frac{1}{I \times J - 2} \sum_{i=1}^I \sum_{j=1}^J (\bar{y}_{ij} - \hat{a} - \hat{b}x_i)^2 \dots\dots\dots (4)$$

The symbols used here and elsewhere in this part of **JIS Z 8462** are defined in annex A.

NOTE : Estimates are denoted by a symbol ^ to differentiate them from the parameters themselves which are unknown.

Information : $\sum_{j=1}^J$ of equation (2) is erroneously written as $\sum_{j=1}^J$ in the original
International Standard.

5.2.3 Computation of critical values The critical value of the response variable is given by:

$$y_c = \hat{a} + t_{0.95}(\nu) \hat{\sigma} \sqrt{\frac{1}{K} + \frac{1}{I \times J} + \frac{\bar{x}^2}{s_{xx}}} \dots\dots\dots (5)$$

The critical value of the net state variable is given by:

$$x_c = t_{0.95}(\nu) \frac{\hat{\sigma}}{\hat{b}} \sqrt{\frac{1}{K} + \frac{1}{I \times J} + \frac{\bar{x}^2}{s_{xx}}} \dots\dots\dots (6)$$

$t_{0.95}(\nu)$ is the 95 %-quantile of the t -distribution with $\nu = I \cdot J - 2$ degrees of freedom.

The derivation of these formulae is given in annex B.

5.2.4 Computation of the minimum detectable value The minimum detectable value is given by:

$$x_d = \delta \frac{\hat{\sigma}}{\hat{b}} \sqrt{\frac{1}{K} + \frac{1}{I \times J} + \frac{\bar{x}^2}{s_{xx}}} \dots\dots\dots (7)$$

where

$\delta = (\nu; \alpha; \beta)$ is the value of the noncentrality parameter determined in such a way that a random variable following the noncentral t -distribution with $\nu = I \cdot J - 2$ degrees of freedom and the noncentrality parameter δ , $T(\nu; \delta)$, satisfies the equation:

$$P[T(\nu; \delta) \leq t_{1-\alpha}(\nu)] = \beta$$

where $t_{1-\alpha}(\nu)$ is the $(1-\alpha)$ -quantile of the t -distribution with ν degrees of freedom.