

JAPANESE INDUSTRIAL STANDARD

JIS Z 8402—3 : 1999

Accuracy (trueness and precision) of measurement methods and results—Part 3:

Intermediate measures of the precision of a standard measurement method

September 2003

ERRATA

Page 18

Annex B (normative)

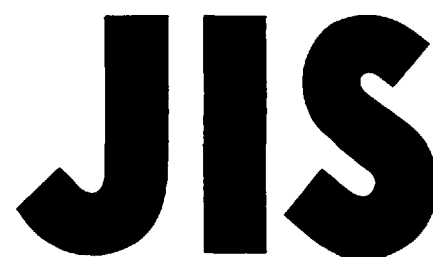
B.1

Error: $SS0 = \dots - 4 \left(\overline{y} \right)^2$

Correct: $SS0 = \dots - 4 \rho \left(\overline{y} \right)^2$

Remarks: This erratum is for correcting the first edition of this Standard.

Japanese Standards Association



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INDUSTRIAL
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(ISO 5725-3 : 1994)

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of measurement methods and
results—**

**Part 3 : Intermediate measures
of the precision of a standard
measurement method**

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Foreword

This translation has been made based on the original Japanese Industrial Standard established by the Minister of International Trade and Industry through deliberations at the Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law. By this establishment, **JIS Z 8402 : 1991** was withdrawn and replaced with this Standard. This Standard has been prepared based on the first edition of **ISO 5725-3** issued in 1994.

JIS Z 8402 : 1999 consists of the following 6 parts under the title "*Accuracy (trueness and precision) of measurement methods and results*".

- Part 1 : *General principles and definitions*
- Part 2 : *Basic method for the determination of repeatability and reproducibility of a standard measurement method*
- Part 3 : *Intermediate measures of the precision of a standard measurement method*
- Part 4 : *Basic methods for the determination of the trueness of a standard measurement method*
- Part 5 : *Alternative methods for the determination of the precision of a standard measurement method*
- Part 6 : *Use in practice of accuracy values*

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the original JIS is to be the final authority.

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**Accuracy (trueness and precision)
of measurement methods and results—
Part 3 : Intermediate measures
of the precision of a standard
measurement method**

Introduction This Japanese Industrial Standard has been prepared based on ISO 5725-3 *Accuracy (trueness and precision) of measurement methods and results—Part 3 : Intermediate measures of the precision of a standard measurement method* issued in 1994 as the first edition without changing the technical contents.

The “Informative references” underlined with dots in this Standard show the matters not specified in the original International Standard.

0.1 JIS Z 8402 uses two terms “trueness” and “precision” to describe the accuracy of a measurement method. “Trueness” refers to the closeness of agreement between the average value of a large number of test results and the true or accepted reference value. “Precision” refers to the closeness of agreement between test results.

0.2 General consideration of these quantities is given in Part 1 of **JIS Z 8402** and so is not repeated here. It is stressed that Part 1 of this Standard should be read in conjunction with all other parts of **JIS Z 8402** because the underlying definitions and general principles are given there.

0.3 Many different factors (apart from variations between supposedly identical specimens) may contribute to the variability of results from a measurement method, including:

- a) the operator;
- b) the equipment used;
- c) the calibration of the equipment;
- d) the environment (temperature, humidity, air pollution, etc.);
- e) the batch of a reagent;
- e) the time elapsed between measurements.

The variability between measurements performed by different operators and/or with different equipment will usually be greater than the variability between measurements carried out within a short interval of time by a single operator using the same equipment.

0.4 Two conditions of precision, termed repeatability and reproducibility conditions, have been found necessary and, for many practical cases, useful for describing the variability of a measurement method. Under repeatability conditions, factors a) to f) in 0.3 are considered constants and do not contribute to the variability, while under reproducibility conditions they vary and do contribute to the variability of the test results. Thus repeatability and reproducibility conditions are the two extremes of precision, the first describing the minimum and the second the maximum variability.

in results. Intermediate conditions between these two extreme conditions of precision are also conceivable, when one or more of factors a) to f) are allowed to vary, and are used in certain specified circumstances.

Precision is normally expressed in terms of standard deviations.

0.5 This part of **JIS Z 8402** focuses on intermediate precision measures of a measurement method. Such measures are called intermediate as their magnitude lies between the two extreme measures of the precision of a measurement method: repeatability and reproducibility standard deviations.

To illustrate the need for such intermediate precision measures, consider the operation of a present-day laboratory connected with a production plan involving, for example, a three-shift working system where measurements are made by different operators on different equipment. Operators and equipment are then some of the factors that contribute to the variability in the test results. These factors need to be taken into account when assessing the precision of the measurement method.

0.6 The intermediate precision measures defined in this part of **JIS Z 8402** are primarily useful when their estimation is part of a procedure that aims at developing, standardizing, or controlling a measurement method within a laboratory. These measures can also be estimated in a specially designed interlaboratory study, but their interpretation and application then requires caution for reasons explained in 1.3 and 9.1.

0.7 The four factors most likely to influence the precision of a measurement method are the following.

- a) **Time:** whether the time interval between successive measurements is short or long.
- b) **Calibration:** whether the same equipment is or is not recalibrated between successive groups of measurements.
- c) **Operator:** whether the same or different operators carry out the successive measurements.
- d) **Equipment:** whether the same or different equipment (or the same or different batches of reagents) is used in the measurements.

0.8 It is, therefore, advantageous to introduce the following M -factor-different intermediate precision conditions ($M = 1, 2, 3$ or 4) to take account of changes in measurement conditions (time, calibration, operator and equipment) within a laboratory.

- a) $M = 1$: only one of the four factors is different;
- b) $M = 2$: two of the four factors are different;
- c) $M = 3$: three of the four factors are different;
- d) $M = 4$: all four factors are different.

Different intermediate precision conditions lead to different intermediate precision standard deviations denoted by $s_{I(\)}$, where the specific conditions are listed within the parentheses. For example, $s_{I(TO)}$ is the intermediate precision standard deviation with different times (T) and operators (O).

0.9 For measurements under intermediate precision conditions, one or more of the factors listed in **0.7** is or are different. Under repeatability conditions, those factors are assumed to be constant.

The standard deviation of test results obtained under repeatability conditions is generally less than that obtained under the conditions for intermediate precision conditions. Generally in chemical analysis, the standard deviation under intermediate precision conditions may be two or three times as large as that under repeatability conditions. It should not, of course, exceed the reproducibility standard deviation.

As an example, in the determination of copper in copper ore, a collaborative experiment among 35 laboratories revealed that the standard deviation under one-factor-different intermediate precision conditions (operator and equipment the same but time different) was 1.5 times larger than that under repeatability conditions, both for the electrolytic gravimetry and $\text{Na}_2\text{S}_2\text{O}_3$ titration methods.

1 Scope

1.1 This part of **JIS Z 8402** specifies four intermediate precision measures due to changes in observation conditions (time, calibration, operator and equipment) within a laboratory. These intermediate measures can be established by an experiment within a specific laboratory or by an interlaboratory experiment.

Furthermore, this part of **JIS Z 8402**,

- a) discusses the implications of the definitions of intermediate precision measures;
- b) presents guidance on the interpretation and application of the estimates of intermediate precision measures in practical situations;
- c) does not provide any measure of the errors in estimating intermediate precision measures;
- d) does not concern itself with determining the trueness of the measurement method itself, but does discuss the connections between trueness and measurement conditions.

1.2 This part of **JIS Z 8402** is concerned exclusively with measurement methods which yield measurements on a continuous scale and give a single value as the test result, although the single value may be the outcome of a calculation from a set of observations.

1.3 The essence of the determination of these intermediate precision measures is that they measure the ability of the measurement method to repeat test results under the defined conditions.

1.4 The statistical methods developed in this part of **JIS Z 8402** rely on the premise that one can pool information from "similar" measurement conditions to obtain more accurate information on the intermediate precision measures. This premise is a powerful one as long as what is claimed as "similar" is indeed "similar". But it is very difficult for this premise to hold when intermediate precision measures are estimated from an interlaboratory study. For example, controlling the effect of "time" or of "operator" across laboratories in such a way that they are "similar", so that pooling information from different laboratories makes sense, is very difficult. Thus,

using results from interlaboratory studies on intermediate precision measures requires caution. Within-laboratory studies also rely on this premise, but in such studies it is more likely to be realistic, because the control and knowledge of the actual effect of a factor is then more within reach of the analyst.

1.5 There exist other techniques besides the ones described in this part of **JIS Z 8402** to estimate and to verify intermediate precision measures within a laboratory, for example, control charts (see **JIS Z 8402-6**). This part of **JIS Z 8402** does not claim to describe the only approach to the estimation of intermediate precision measures within a specific laboratory.

Note 1 This part of **JIS Z 8402** refers to designs of experiments such as nested designs. Some basic information is given in Annexes B and C. Other references in this area are given in Annex E.

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 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 apply limiting only to the most recent edition (including the amendment).

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

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

JIS Z 8402-1 : 1999 *Accuracy (trueness and precision) of measurement methods and results—Part 1 : General principles and definitions*

JIS Z 8402-2 : 1999 *Accuracy (trueness and precision) of measurement methods and results—Part 2 : Basic method for the determination of repeatability and reproducibility of a standard measurement method*

JIS Q 0033 : 1997 *Uses of certified reference materials*

JIS Q 0035 : 1997 *Certification of reference materials—General and statistical principles*

3 Definitions

For the purposes of this part of **JIS Z 8402**, the definitions given in **JIS Z 8101** and **JIS Z 8402-1** apply.

The symbols used in **JIS Z 8402** are given in Annex A.

4 General requirement

In order that the measurements are made in the same way, the measurement method shall have been standardized. All measurements forming part of an experiment within a specific laboratory or of an interlaboratory experiment shall be carried out according to that standard.

5 Important factors

5.1 Four factors (time, calibration, operator and equipment) in the measurement conditions within a laboratory are considered to make the main contributions to the variability of measurements (see Table 1).

Table 1 Four important factors and their states

Factor	Measurement conditions within a laboratory	
	State 1 (same)	State 2 (different)
Time	Measurements made at the same time	Measurements made at different times
Calibration	No calibration between measurements	Calibration carried out between measurements
Operator	Same operator	Different operators
Equipment	Same equipment without recalibration	Different equipment

5.2 “Measurements made at the same time” include those conducted in as short a time as feasible in order to minimize changes in conditions, such as environmental conditions, which cannot always be guaranteed constant. “Measurements made at different times”, that is those carried out at long intervals of time, may include effects due to changes in environmental conditions.

5.3 “Calibration” does not refer here to any calibration required as an integral part of obtaining a test result by the measurement method. It refers to the calibration process that takes place at regular intervals between groups of measurements within a laboratory.

5.4 In some operations, the “operator” may be, in fact, a team of operators, each of whom performs some specific part of the procedure. In such a case, the team should be regarded as the operator, and any change in membership or in the allotment of duties within the team should be regarded as providing a different “operator”.

5.5 “Equipment” is often, in fact, sets of equipment, and any change in any significant component should be regarded as providing different equipment. As to what constitutes a significant component, common sense must prevail. A change of thermometer would be considered a significant component, but using a slightly different vessel to contain a water bath would be considered trivial. A change of a batch of a reagent should be considered a significant component. It can lead to different “equipment” or to a recalibration if such a change is followed by calibration.

5.6 Under repeatability conditions, all four factors are at state 1 of Table 1. For intermediate precision conditions, one or more factors are at state 2 of Table 1, and are specified as “precision conditions with M factor(s) different”, where M is the number of factors at state 2. Under reproducibility conditions, results are obtained by different laboratories, so that not only are all four factors at state 2 but also there are additional effects due to the differences between laboratories in management and maintenance of the laboratories, general training levels of operators, and in stability and checking of test results, etc.

5.7 Under intermediate precision conditions with M factor(s) different, it is necessary to specify which factors are at state 2 of Table 1 by means of suffixes, for example:

- time-different intermediate precision standard deviation, $s_{I(T)}$;
- calibration-different intermediate precision standard deviation, $s_{I(C)}$;
- operator-different intermediate precision standard deviation, $s_{I(O)}$;
- [time + operator]-different intermediate precision standard deviation, $s_{I(TO)}$;
- [time + operator + equipment]-different intermediate precision standard deviation, $s_{I(TOE)}$;
- and many others in a similar fashion.

6 Statistical model

6.1 Basic model

For estimating the accuracy (trueness and precision) of a measurement method, it is useful to assume that every test result, y , is the sum of three components:

$$y = m + B + e \dots\dots\dots (1)$$

where, for the particular material tested,

- m is the general mean (expectation);
- B is the laboratory component of bias under repeatability conditions;
- e is the random error occurring in every measurement under repeatability conditions.

A discussion of each of these components, and of extensions of this basic model, follows.

6.2 General mean, m

6.2.1 The general mean, m , is the overall mean of the test results. The value of m obtained in a collaborative study (see JIS Z 8402-2) depends solely on the "true value" and the measurement method, and does not depend on the laboratory, equipment, operator or time by or at which any test result has been obtained. The general mean of the particular material measured is called the "level of the test"; for example, specimens of different purities of a chemical or different materials (e.g. different types of steel) will correspond to different levels.

In many situations, the concept of a true value μ holds good, such as the true concentration of a solution which is being titrated. The level m is not usually equal to the true value μ ; the difference ($m - \mu$) is called the "bias of the measurement method".

In some situations, the level of the test is exclusively defined by the measurement method, and the concept of an independent true value does not apply; for example, the Vicker's hardness of steel and the Micum indices of coke belong to this category. However, in general, the bias is denoted by δ ($\delta = 0$ where no true value exists), then the general mean m is