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(ISO Guide 33 : 2000)

## **Uses of certified reference materials**

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Q 0033 : 2002 (ISO Guide 33 : 2000)

## Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law. Consequently **JIS Q 0033:1997** is replaced with **JIS Q 0033:2002**.

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## Uses of certified reference materials

**Introduction** This Japanese Industrial Standard has been prepared based on the second edition of **ISO Guide 33** *Uses of certified reference materials* published in 2000 without modifying the technical contents.

Portions underlined with dots in this Standard are the matters not stated in the original Guide.

Today's world of modern technology requires a large number of certified reference materials (CRMs) in widely diverse fields, and the demand for such materials is expected to increase. The preparation of a CRM is a time-consuming, meticulous and expensive endeavour and consequently it has not always been, and will continue not to be, possible to satisfy the demand for all types and quantities of CRMs. For this reason, CRMs should be used properly, i.e. effectively, efficiently and economically.

Certified reference materials should be used on a regular basis to ensure reliable measurements. However, in doing so, the magnitude of the supply of that CRM, its relative cost, its availability (accessibility) and the measurement technique, be it destructive or non-destructive, should be considered. Also important to the user is the fact that the misuse of a CRM may not provide the intended information.

Misuse of CRMs differs from incorrect use. The user of a CRM is expected to be familiar with all information pertinent to the use of the CRM as specified in its certificate. He should comply with such factors as the period of validity of the CRM, the prescribed conditions for storage of the CRM, instructions for the use of the CRM, and specifications for validity of the certified properties of the CRM. A CRM should not be used for a purpose other than that for which it was intended. Nevertheless, from time to time, when a user must resort to applying a CRM in an incorrect manner because of the unavailability of a suitable CRM, he should be fully cognizant of the potential pitfalls and therefore assess his measurement output accordingly.

There are many measurement processes in which CRMs are in general use but are replaceable by a host of working standards, such as homogeneous materials, previously analysed materials, pure compounds, solutions of pure elements, etc. Some examples are where only a "rough" estimate of the trueness or precision of a method is sought, where "blind" unknown check samples are used routinely in quality control programmes, and where only the variation in trueness or precision of a method with some parameter such as time, analyst, instrument, etc., is being evaluated. The first example illustrates the use of a CRM in which the well-defined certified value and uncertainty of the CRM is under-utilized. The others illustrate the case in which a series of "one-time" trueness and precision assessments are compared with one another. There is no need to base that comparison on a well-defined certified value and uncertainty of a CRM. The advantages in using CRMs are that the user has the means to assess the trueness and precision of his measurement method and establishes metrological traceability for his results.

Whether the use of CRMs in these procedures is in fact "misuse" depends largely on the availability and relative cost of the CRMs. Where CRMs are in short supply or very expensive, their use would indeed be misuse. However, for CRMs in ample supply or where similar CRMs are available from one or more sources, it is strongly recommended that CRMs always be used instead of in-house standards because of the resultant enhanced confidence in the measurement output.

It is important that users remain aware that the preparation of in-house standards for use instead of CRMs has an associated cost based on factors such as material cost, facility usage charges, personnel labour rates, etc., in which the material cost is in general the lowest. For some CRMs such as the complex compositional materials certified for chemical composition, the cost of preparing in-house standards to match the composition of real samples can exceed that of available CRMs. In these cases, the use of CRMs is recommended.

The user should be aware of the potential misuse of CRMs as "blind" unknown check samples in quality control programmes. Where there are only a few CRMs in an area of expertise, they are easily recognized and they may therefore not satisfy the intended purpose. Moreover, the same CRM should never be used for both calibration purposes and as "blind" unknown check sample in a measurement process.

The misuse of CRMs can also occur when the user does not fully take into account the uncertainty in the certified property. The combined standard uncertainty of a certified property of a CRM can have contributions from the inhomogeneity of the material, the within-laboratory uncertainty and, where applicable, the between-laboratory uncertainty. The level of homogeneity defined for a CRM by the producer is dependent on both the statistical design used to evaluate it and the repeatability of the method of measurement. For certain CRMs, the level of homogeneity is valid for a test portion defined by mass, physical dimension, time of measurement, etc. The user should be aware that the use of a test portion that does not meet or exceed that specification could severely increase the contribution of the inhomogeneity of the CRM to the uncertainty of the certified property, to the point where the statistical parameters of certification are no longer valid.

The variation in the repeatability of different methods has another implication for the user. Since the degree of inhomogeneity of a CRM is dependent on the repeatability of the method of measurement, it is possible that a user, in applying a method capable of better repeatability, could detect inhomogeneity in that CRM. In such cases, the observed inhomogeneity is already accounted for in the statistical parameters for the certified property and therefore the statistical tests presented in this Standard remain valid, but the scientific basis for using that particular CRM to give a true assessment of the user's method should again be questioned.

It is well known that different methods of measurement of a property are not capable of equal repeatability. Accordingly there could arise instances where the user may wish to assess a method that has greater repeatability than that or those used in the certification of the CRM. In such cases, the statistical tests presented in this Standard remain valid but the scientific basis for using that particular CRM to give a true assessment of the precision (and possibly the trueness) normally expected from the user's method should be questioned. It is recommended that the user resorts to a CRM of lesser uncertainty, if available.

For CRMs certified by a primary method, the user should not assume that his method is capable of matching the precision and trueness reported for the CRM. It is unreasonable therefore to apply the statistical procedures in this Standard for assessing the trueness and precision of a method by application to a CRM using the certification parameters for a property reported in the certificate. The user, as a consequence, should either experimentally establish or make estimates based on available information for those parameters that are more appropriate. Similarly, where a user applies a method to a CRM that has been certified by a single different method, the user should not assume that the certification parameters for the certified property are applicable to his method except in cases where the trueness and precision capable by both methods are known to be comparable.

One of the important considerations in selecting a CRM for use, either in assessing the trueness and precision of a method or in the calibration of instruments in a method is the level of uncertainty required by the end-use of the method. Obviously the user should not apply a CRM of greater uncertainty than permitted by the end-use.

The selection of CRMs should take into account not only the level of uncertainty required for the intended purpose but also their availability, cost, and chemical and physical suitability for the intended purpose. For example, the unavailability or high cost of one CRM could force a user to resort to using another CRM of greater uncertainty than the preferred one. Also, in chemical analysis, a CRM of greater, but still acceptable, uncertainty in the certified property may be preferred over another CRM because of better matching with the composition of real samples. This could result in minimizing "matrix" or chemical effects in the measurement process which are capable of causing errors far greater than the difference between the uncertainties of the CRMs.

In conclusion, CRMs are meant to fulfil many purposes. Accordingly, a CRM used properly for one purpose in one laboratory may be misused for another purpose in another laboratory. It is recommended that the user consider the suitability of a CRM for his intended purpose on a case-by-case basis.

**1 Scope** This Standard discusses the uses of certified reference materials (CRMs) and their correct applications. Clause 2 of this Standard presents definitions (with indication of their sources) of terms used and clause 4 sets out the statistical considerations on which this Standard is based. Clause 5 discusses the role of CRMs in measurement science and in realization of conventional measurement scales. Clause 6 presents recommendations for developing criteria for the assessment of the precision and trueness of a measurement procedure by the use of CRMs. It pertains only to CRMs characterized to be homogeneous as described in **JIS Q 0035** [4].

**NOTE 1** The use of CRMs is essential for assessment of trueness and optional for assessment of precision.

**2** 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 Guide 33: 2000 *Uses of certified reference materials* (IDT)

This Standard does not describe the use of certified reference materials as calibrants. This subject is treated in **JIS Q 0032** [3].

**2 Terms and definitions** For the purposes of this Standard, the following terms and definitions apply.

**2.1 measurement process** all the information, equipment and operations relevant to a given measurement

**NOTE:** This concept embraces all aspects relating to the performance and quality of the measurement; it includes, for example, the principle, method, procedure, values of the influence quantities and the measurement standards.

[see VIM, *International vocabulary of basic and general terms in metrology*, issued by BIPM, **IEC**, **IFCC**, **ISO**, **IUPAC**, **IUPAP** and **OIML**]

Information: This term existed in VIM1984 referred in the original Guide, but it is deleted in 1993 edition.

**2.2 influence quantity** quantity that is not the measurand but that affects the result of the measurement

**EXAMPLE** Ambient temperature; frequency of an alternating measured voltage.

[see VIM, *International vocabulary of basic and general terms in metrology*, issued by BIPM, **IEC**, **IFCC**, **ISO**, **IUPAC**, **IUPAP** and **OIML**]

Information: Other examples

- 1 Temperature of micrometers used for measurement of length.
- 2 Bilirubin concentration in hemoglobin concentration determination of the sample of human blood plasma.

**2.3 reference material, RM** a material or substance one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials [see **JIS Q 0030:1997**]

Information: A reference material may be in the form of a pure or mixed gas, liquid or solid. Examples are water for the calibration of viscometers, sapphire as a heat-capacity calibrant in calorimetry, and solutions used for calibration in chemical analysis.

**2.4 certified reference material, CRM** a reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes traceability to an accurate realization of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence [see **JIS Q 0030:1997**]

Information 1 The definition of a reference material certificate is given in **4.2**.

- 2 CRMs are generally prepared in batches for which the property values are determined within stated uncertainty limits by measurements on samples representative of the whole batch.
- 3 The certified properties of reference materials are sometimes conveniently and reliably realized when the material is incorporated into a specially fabricated device, e.g. a substance of known triple-point into a triple-point cell; a glass of known optical density into a transmission filter; spheres of uniform particle size mounted on a microscope slide. Such devices may also be considered as CRMs.

4. All CRMs lie within the definition of measurement standards or etalons given in the International vocabulary of basic and general terms in metrology (VIM).
5. Some RMs and CRMs have properties which, because they cannot be correlated with an established chemical structure or for other reasons, cannot be determined by exactly defined physical and chemical measurement methods. Such materials include certain biological materials such as vaccines to which an International unit has been assigned by the World Health Organization.

**2.5 precision** the closeness of agreement between independent test results obtained under prescribed conditions [see **JIS Z 8101-1:1999**]

**2.6 repeatability** precision under repeatability conditions [see **JIS Z 8101-1:1999**]

**2.7 repeatability conditions** conditions where independent test results are obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within short intervals of time [see **JIS Z 8101-1:1999**]

**2.8 repeatability standard deviation** the standard deviation of test results obtained under repeatability conditions

NOTE: It is a measure of the dispersion of the distribution of test results under repeatability conditions [see **JIS Z 8101-1:1999**].

**2.9 repeatability limit,  $r$**  the value less than or equal to which the absolute difference between two single test results obtained under repeatability conditions is expected to be with a probability of 95 % [see **JIS Z 8101-1:1999**]

**2.10 reproducibility** precision under reproducibility conditions [see **JIS Z 8101-1:1999**]

**2.11 reproducibility conditions** conditions where test results are obtained with the same method on identical material in different laboratories by different operators using different equipment [see **JIS Z 8101-1:1999**]

**2.12 reproducibility limit,  $R$**  the value less than or equal to which the absolute difference between two single test results obtained under reproducibility conditions is expected to be with a probability of 95 % [see **JIS Z 8101-1:1999**]

**2.13 bias** the difference between the expectation of the test results and an accepted reference value

NOTE: Bias is a systematic error as contrasted to random error. There may be one or more systematic error components contributing to the bias. A larger systematic difference from the accepted reference value is reflected by a larger bias value [see **JIS Z 8101-1:1999**].