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METROLOGY

Standardization of test methods

Draft Guidelines on Methodology for Measuring Procedures*

Note submitted by the Rapporteur for metrology

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FOREWORD

1. The UN/ECE Working Party on Technical Harmonization and Standardization Policies is unique as an international standards-related forum with governmental representation. Its efforts serve to reduce, if not to eliminate, non-tariff standards-related trade barriers.

2. The eighth session of the Working Party supported the proposal to prepare the draft of guidelines on methodology for measuring procedures. Before these guidelines are prepared interested international (International Organization for Standardization (ISO)), International Electrotechnical Commission (IEC)), International Organization of Legal Metrology (OIML)) and regional organizations (European Committee for Standardization (CEN)), European Committee for Electrotechnical Standardization (CENELEC)) should be consulted on its scope and content.

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Introduction

3. Successful solving of metrological problems by testing requires a corresponding methodology that would ensure the necessary confidence in test results and evaluation of the quality characteristics of a product. Experience based on the concept “traceability of the measurements” leads to the possible feasibility of introducing the concept of “traceability of the tests” (experimental test operations). In this respect, it is extremely important to ensure conformity of the methods, i.e. practical measuring procedures, test and control methods.

4. Acoustical, magnetic, radiation, optical, radio waves and thermal methods are widely used in testing. That is why confidence in results is linked directly to accuracy of application of measuring procedures and to links of measuring instruments to corresponding primary and secondary standards of units of measurement.

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Guide for measurement procedures

1. Scope

1.1. This guide covers all the procedures of measurements (PM) that are being developed or revised, including the quantitative chemical analysis technique (QCAT), and provides the general principles and requirements for their development, certification, standardization and metrological assurance.

1.2. The guide does not cover PM, for which the measurement uncertainty characteristics are determined during or after their application.

2. Normative references

2.1 Guide to the Expression of Uncertainty in Measurement. ISO, Geneva, Switzerland, 1993.

2.2 Quantifying Uncertainty in Analytical Measurement. EUROCHEM, version 6.

3. Definitions and abbreviations

3.1 Procedures of measurements (PM)

– A set of operations and rules the fulfillment of which ensures the obtainment of measurement results with a known uncertainty.

3.2 PM certification

– A procedure for the determination and verification of PM conformity with the relevant metrological requirements.

3.3 Metrological examination of PM

- An analysis and assessment of techniques and means of measurement selection, operations and rules of measurement execution and measurement results processing with the aim of establishing the conformity of PM with relevant metrological requirements.

3.4 Assigned characteristic of measurement uncertainty

– Measurement uncertainty characteristic of any result of a set of measurements obtained in conformity with the rules of this procedure.

4. General considerations

4.1 PM are developed and used with the aim of ensuring that the measuring execution uncertainty does not exceed a required or assigned characteristic.

4.2 Depending on the complexity and application scope, PM should be stated in:

- a separate document (standard, instructions, recommendation, etc.);
- a section or part of the document (standard section, specifications, design or engineering document, etc.).

5. PM development

5.1 PM development is carried out on the basis of reference data, which include:

- purpose of PM, measurement uncertainty requirements, measurement conditions and other requirements for PM.

The reference data should be stated in performance specifications, specifications, scientific research reports and other documents.

5.1.1 The purpose of PM should comprise:

- scope (object of measurement, including the product and controlled parameter names, as well as the field of application – for a separate enterprise, for a branch of industry, for a net of departmental and interdepartmental laboratories, etc.);

- name (expanded definition, if necessary) of a measured quantity;

- characteristics of the measured quantity (region and frequency spectrum, values of non-informational parameters, etc.);

- characteristics of a measurement object if they can influence the measurement uncertainty (output resistance, stiffness at a probe contact place, sample composition, etc.).

5.1.2 Measurement uncertainty requirements should be expressed in accordance with the guide 1/. In the PM regulating document, the measurement uncertainties may be given by reference to the document in which these requirements are specified.

The measurement uncertainty requirements should be established taking into account all the components of uncertainty (procedural, instrumental, human, sampling and sample preparation). Typical measurement uncertainty components are given in Appendix A.

If the measurement uncertainty requirements are not given explicitly, initial requirements should include indications allowing a rational choice of measurement techniques and means, and these should be used when certifying PM (controlled parameter allowance, credibility indices of measurement inspection, etc.).

1/ See page 5, paragraph 2.1.

5.1.3 Measurement requirements should be given as nominal values and/or range limits of the possible values of influencing quantities. Limit speed changes or other characteristics of influencing values, as well as restrictions on the measurement duration, number of parallel determinations, etc., should be given, if necessary.

When, by determining the initial requirements, it is known *a priori* that the measurements will be carried out by use of means disposed of at different locations, the measurement conditions should be indicated for all the places of disposition of the measurement means constituting the measuring system.

5.2. The development of PM will normally comprise:

– choice of methods and means of measurement (including certified reference materials, certified mixtures), auxiliary and other technical facilities;

– determination of the sequence and contents of operations when preparing and executing measurements, processing of intermediate results and calculation of final measurement results;

– determination of assigned measurement uncertainty characteristics;

– development of normative documents and procedures for the inspection of the accuracy of the obtained measurement results;

– development of a document (section, part of a document) for PM;

– metrological design review of a PM draft document;

– PM certification;

– PM standardization.

Note. PM certification and standardization may be carried out as independent projects.

5.2.1 Methods and means of measurement should be selected in compliance with currently valid documents for the choice of methods and means for this type of measurement, and if these documents are unavailable they should be chosen in accordance with general considerations.

5.2.2 Methods of expression for assigned measurement uncertainty characteristics should comply with the method given in the reference data. If the measurement uncertainty requirements are not given, the assigned measurement uncertainty characteristics may be expressed in accordance with the guide 1/.

When developing QCAT, the methods of assigned measurement uncertainty characteristics may be expressed in accordance with the recommendation 2/.

1/ See page 5, paragraph 2.1.
2/ See page 5, paragraph 2.2.

5.2.3 Generally, in PM regulating documents (sections, parts of documents) should be pointed out:

- PM purpose;
- measurement conditions;
- requirements for measurement uncertainty and/or assigned measurement uncertainty characteristics;
- method(s) of measurements;
- requirements for measuring means (including those for certified reference materials, certified mixtures), auxiliary devices, materials, solutions; otherwise, types of measuring means, their characteristics and document designations where the requirements for the measuring means are given should be pointed out;
- preparatory operations for measurements execution;
- operations during measurements execution;
- processing and measurement results calculation operations;
- normative documents, procedure and periodicity of measurement uncertainty inspections of executed measurements;
- requirements for measurement data drawing up;
- requirements for operators' qualifications;
- requirements for ensuring occupational safety;
- requirements for ensuring environmental safety;
- other requirements and operations (if required).

PM documents should comprise those requirements and operations from the list above which ensure the fulfillment of requirements for the measurement uncertainty or assigned measurement uncertainty characteristics.

Notes:

1. Serial numbers (inventory, etc.), identification numbers of individual measuring means and other technical facilities should be additionally pointed out in PM documents, in which the use of specific items of measuring means and other technical facilities is provided for.
2. Instead of the PM information listed above, the document (sections of document) may have references to other documents where this information is noted.
3. When developing procedures for the inspection of uncertainty of quantitative chemical analysis results the recommendation 1/ should be used.
4. The recommendations for the fabrication and wording of individual PM documents are given in Appendix B.

6. PM certification

6.1 The main object of a PM certification is to demonstrate that when measures are made in conformance with the PM in question, the range of possible measurement results (i.e. the measurement uncertainty) is not greater than that indicated in the PM regulating document.

1/ See page 5, paragraph 2.2.

6.2 The certification of a PM should be carried out by metrological services and other organizational bodies that can assure the traceability of measurements of enterprises (bodies) developing or using the PM.

The metrological service (organizational body) of an enterprise (body) certifying a PM should be accredited for the certification of PM.

6.3 The certification of PM should be carried out on the basis of a metrological examination of the PM development documents and a document (section, part of a document) regulating PM, and/or theoretical and/or experimental study of PM.

6.4 The following documents should be presented at the certification:

- initial requirements for the development of the PM;
- PM regulating document (draft document);
- program and results of experimental and analytical assessment of PM uncertainty characteristics if this has been carried out.

6.5 In the case of positive certification results, a certificate of PM conformity, an exemplary form of which is shown in Appendix B, should be registered officially.

7. Metrological supervision of certified PM

7.1. When executing metrological supervision, metrological services of corresponding juridical persons should audit:

- availability of the PM regulating document with the mark or certificate of conformity;
- conformity of used measuring means and other technical facilities, measurement conditions, order of preparation and execution of measurement.

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APPENDIX A
(reference)

Typical measurement uncertainty components

A.1 Procedural components of measurement uncertainties

A.1.1. Non-adequacy of a controlled object to a model whose parameters are used as quantities to be measured.
A.1.2. Deviations from conventional values of arguments of the function relating the measured value with the value at “the input” of measuring means (a primary measuring transducer).
A.1.3. Deviations from conventional values for the difference between the measuring value of a measured quantity at the measuring means input and its sampling point value.
A.1.4. Uncertainty due to quantum effects.
A.1.5. Difference of a calculation algorithm from a precise function relating measurement results to the measured quantity.
A.1.6. Sampling and sample preparation uncertainties.
A.1.7. Uncertainties due to hindering influence of sample factors (hindering sample components, dispersity, porosity, etc.).

A.2 Instrumental components of measurement uncertainties

A.2.1. Basic uncertainties and additional static uncertainties of measuring means due to slow changing influencing external factors.
A.2.2. Uncertainties due to the limited resolution of measuring means.
A.2.3. Dynamic uncertainties of measuring means (uncertainties due to the inertial properties of measuring means).
A.2.4. Uncertainties due to the interaction of measuring means with the object of measurement and measuring means attached to its input or output.
A.2.5. Uncertainties due to measurement data transmission.

A.3 Operator’s uncertainties (human uncertainties)

A.3.1. Uncertainties of the reading of a measured value from scales and charts.
A.3.2. Uncertainties of chart processing without using instruments (by elimination, summation of measured values, etc.).
A.3.3. Uncertainties due to the operator’s influence on the object and measuring means (distortions of a temperature field, mechanical actions, etc.).

A.4 Analysis of measurement uncertainty components should be carried out in accordance with the guide 1/.

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1/ See page 5, paragraph 2.1.

APPENDIX B
(recommendatory)

Creation and wording of individual PM documents

B.1 The title of the PM document should reflect the essence of the procedure. It is permissible to involve in the title a specific character of a measurement quantity. For instance: "STATE SYSTEM OF UNIFORM MEASUREMENTS: MASS OF RAILWAY TRANSPORTED CARGOS: Procedure of measurements by a large wagon balance".

If there is a large volume of measured quantities, their general description should be used, e.g.: "Parameters of electromagnetic fields in flares of high directivity antennas".

B.2 Documents for PM should consist of an introduction and the following sections:

- requirements for measurement uncertainty or assigned measurement uncertainty characteristics;
- measuring means, auxiliary devices, materials, solutions;
- method(s) of measurement(s);
- safety and environmental protection requirements;
- staff qualification requirements;
- measurement conditions;
- preparation for measurement execution;
- execution of measurements;
- processing (calculation) of measurement results;
- inspection of the accuracy of measurement results;
- expression of measurement results.

It is permitted to eliminate or combine the above-mentioned sections as well as change their titles and include additional sections, taking into account the specific character of measurements.

B.3 An introduction establishes a purpose and a scope of the PM document

B.3.1 The introduction should be stated as follows: "This document (specific type of PM document should be pointed out) provides the procedures of measurements (for the name of a measured quantity with the indication of its specific character and specific character of measurement should be indicated further, if required)".

An introduction for QCAT might be written, for example, as follows: "This standard provides an extraction – photometric technique for the quantitative chemical analyses of samples of rare metals oxides for determination in them of cobalt content at the cobalt mass concentration from 2×10-6 to 2×10-4 %".

B.3.2. When, in the introduction, a reference is made to the specific product, the designation of a normative document covering this product should be given, e.g.: "This document (specific type of PM document should be indicated) provides

procedures of measurements for the determination of magnetically soft alloy characteristics in compliance with the GOST 10160 at any point of the hysteresis loop. Magnetically soft alloy characteristics comprise:

- induction coercive force;
- coefficient of the rectangularity of the hysteresis loop;
- magnetization coercive force;
- temperature coefficients of the above-mentioned parameters.

B.4 The section "Measurement uncertainty requirements" or "Characteristics of the measurement uncertainty" will contain the numeric values of required and assigned measurement uncertainty characteristics, or a reference to the document where they are specified

B.4.1. The first clause of this section should be stated as follows: "The limits of the permissible fractional measurement uncertainty in accordance with this procedure should be ±1.5%" or "The measurement uncertainty should correspond to the requirements given in …" (reference to a normative document). In indicating assigned measurement uncertainty characteristics, instead of "Limits of the permissible uncertainty …". The wording should be: "Uncertainty limits …", and instead of "The measurement uncertainty should correspond to the requirements given in …". The wording should be: "The measurement uncertainty corresponds to the characteristics given in …".

If it is presumed that there is a substantial random component in the measurement uncertainty, the term "limits" should be changed to "boundaries" followed by a probability value (e.g. P=0.95).

The requirements for measurement uncertainty and assigned measurement uncertainty characteristics may be expressed by other methods given in the guide 1/ as well.

B.4.2. The measurement uncertainty requirements (assigned characteristics) of the same quantity may differ for various values of the quantity, various products, various measurement conditions and use of the measurement results. In this case, as well as in the case of several measured quantities, the measurement uncertainty requirements (assigned characteristics) should be given in the form of tables, graphs or equations.

B.5 The section "Measuring means, auxiliary devices, materials, solutions" comprises the list of measuring means and other technical facilities used for the execution of measurements

Designations of national standards (or standards of other categories) or specifications, designation of types of measurement means (modules), their metrological characteristics (accuracy class, permissible uncertainty limits, measurement limits, etc.) should be pointed out in the list of these means along

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1/ See page 5, paragraph 2.1.

with their names. If the number of metrological characteristics is large, they may be given in an appendix.

B.5.1. Drawings, specifications and descriptions of measuring means and other technical devices of one-off production may be given in an appendix.

B.5.2. The first item of this clause should be stated as follows: “In the execution of measurements the following measuring means and other technical facilities: (a list follows) should be used ” or “In the execution of measurements, the measuring means and other technical facilities, given in Table 1, should be used”.

Table 1

Ordinal number and names of measuring means, technical facilities	Designation of standard, specifications, type of the measuring means or its metrological characteristics, or reference to a drawing or appendix	Name of measured quantity

B.6 The section “Method of measurements” comprises the descriptions of measured quantity comparison methods with a unit, in accordance with the basic concept of the method

If a quantity is measured by several methods or a document provides PM for two or more quantities, the description of each method should be given in a separate subsection.

B.6.1. The first clause (of the subsection) should be stated as follows: “The measurements of (the name of the quantity follows) should be carried out by (the description of the physical principle of the method)”.

B.7 The section “Safety and environmental protection requirements” comprises the requirements, the implementation of which ensure occupational safety, conformance with industrial sanitary regulations and environmental protection at the time and place of measurement execution

B.7.1. In the case of available normative documents regulating the requirements of safety, industrial sanitary regulations and environmental protection, in this section reference should be made to these documents.

B.7.2. The first clause of the subsection should be stated as follows: “In the execution of measurements of (the name of the measured quantity follows) the following requirements should be observed: (the list of safety, industrial sanitary regulations and environmental protection requirements follows)”.

B.8 The section "Operators' qualification requirements" comprises information about the qualification level (occupation, education, skills, etc.) of persons which are allowed to execute measurements. The section should be included in the PM document when sophisticated non-automated methods of measurements and measurement results processing are used

B.8.1. The first clause of the section should be stated as follows: "The person(s) allowed to execute the measurements and/or process the results of measurements have the information about qualification level follows.

B.9 The section "Measurement conditions" comprises the list of influencing quantities, their nominal values and/or limits of the range of possible values, as well as other characteristics of influencing quantities and requirements for the object of measurements. The influencing quantities include the medium (sample) parameters, voltage, frequency of the supplying current, internal impedances of measurement objects and other characteristics

The list of influencing quantities may be given in a table form.

B.9.1. The first clause of the subsection should be stated as follows: "When executing the measurements, the following conditions (a list follows) should be observed" or "When executing the measurements the conditions given in Table 2 should be observed".

Table 2

Name of the measured quantity	Name of the influencing quantity	Limit deviations

B.10 The section "Preparation for measurement execution" comprises the descriptions of preparatory work which is to be carried out immediately before the execution of measurements. This work includes the preliminary determination of the value of influencing quantities, circuits assembly (equivalent circuits should be given in this section or in an appendix for this purposes), preparation and performance check of measuring means and other facilities (zero adjustment, warming up, testing, etc.), and sample preparation for measurements

B.10.1. If determination of the calibration curve is provided for in the process of quantitative chemical analysis, the method of its determination and inspection should be described, as well as the sequence for using calibration samples and the preparation of mixed samples should be explained.

B.10.2. If the sequence of preparatory work is specified in the documents for measuring means and other facilities, this section should comprise references to these documents.

B.10.3. The first clause of this section should be stated as follows: “When preparing for the measurement execution the following work should be carried out: (a list and descriptions of the preparatory work follows)”.

B.11 The clause “Measurement execution” comprises a list, volume, sequence of operations, periodicity and number of measurements, operation descriptions, requirements for the presentation of intermediate and final results (number of significant digits, etc.)

For QCAT the requirement for the mass and number of sample charges and, if necessary, instructions for the “check trial” execution and descriptions of operations to eliminate the hindering sample components.

B.11.1. If the sequence of operation execution is specified in the documents for the used measuring means and other facilities, then reference should be made to these documents in this section.

B.11.2. If a quantity is measured by several methods or a document provides PM for two or more quantities, the description of each method should be given in a separate subsection.

B.11.3. In the section (subsection) the requirements necessary for registering intermediate measurement results and values of influencing quantities should be pointed out. The forms of intermediate measurement results and values of influencing quantities should be indicated, if necessary.

B.11.4. The first clause of this section should be stated as follows: “For the execution of measurements (of the name of the measured quantity follows) the following operations are carried out: (the description of operations follows)”.

B.12 The section “Processing (calculation) of measurement results” comprises the descriptions of methods of processing and obtaining final measurement results. If the methods of processing of measurement results are specified in other documents, references should be given in this section to these documents

B.12.1. If a quantity is measured by several methods or a document provides PM for two or more quantities, the description of each method should be given in a separate subsection.

B.12.2. If necessary, the data required for obtaining the final results of measurements (constants, tables, graphs of equations, etc.) should be given in this section. If there are large volumes of data they should be given in appendix.

B.12.3. The necessary requirements for registering preliminary measurement results processing and, if necessary, the form of this registration (magnetic tape, printout, etc.) should be pointed out.

B.12.4. The first clause of the section should be stated as follows: “The processing of measurement results of (the name of measured quantity follows) should be carried out by the method of (the description of the method follows)”.

B.13 The section “Inspection of the accuracy of measurement results” comprises information about normative documents, methods, means and schedule of performing of a primary (operative) and periodic (statistical) inspection of the uncertainty of measurement results obtained in accordance with this PM

B.14 The section “Expression of measurement results” comprises the requirements for the form of presentation of the obtained measurement results. The type of medium for measurement data (document, magnetic tap, recorder tape, etc.) should be pointed out in this section. If necessary, information about used measuring means and other technical facilities, the date and time of measurement result should be given together with the results

B.14.1. The document or record should be signed by the person who conducted the measurements and, if necessary, by the Head of the organization (enterprise), whose signature should be accompanied by the organization’s (enterprise’s) stamp.

B.14.2. The first clause of this section should be stated as followed: “ The measurement results should be presented in a protocol, the form of which is given in Appendix (number of Appendix)” or “Measurement results should be recorded in the log journal in accordance with the following form (a table, graph or other form of presentation follows) or “Measurement results should be stored (instructions on data storage on computer media follow)”.

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APPENDIX C
(recommendatory)

The form of the certificate of conformity for procedures of measurements

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name and legal address of organization (enterprise) that conducted the certification of PM

C E R T I F I C A T E
of conformity for the procedure of measurements

Procedure of measurements _____
name of measured quantity,

_____ ,
developed by
if necessary, the object and method of measurements should be pointed out

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name of organization (enterprise) that developed the procedure of measurements

and regulated by _____
designation and title of the document

The certification was carried out according to the results of _____

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type of works: metrological examination of the documents on PM development, theoretical or experimental study of PM, other types of work

As a result of the certification of the procedure of measurements, it is established that this procedure of measurements is in conformity with the relevant metrological requirements and has the following main metrological characteristics:

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measurement range, uncertainty characteristics (if necessary, characteristics of its components, as well as normative documents on the inspection of measurement results accuracy, etc.)

Head of organization (enterprise) _____ _____
(personal signature) (name in capitals)

data stamp