



New OIML Document

Date: 19 March 2015

Reference:  
TC 3/SC 3/p 7/N01/2 CD  
(Clean version)

Supersedes document:  
1 CD

OIML TC 3/SC 3/p 7

Title: OIML D XX:20YY

**General requirements for the programme  
of reference material certification  
in serial production**

Convenership: Russian Federation

Circulated to P- and O-members and liaison  
international bodies and external organizations  
for:

☐

discussion at (date and place of  
meeting):.....

☒

vote (P-members only) and comments  
by O-members and Liaisons

☐

as forwarded for conversion to 1DR by  
BIML

## **Contents**

1 Scope.....	2
2 General concepts.....	3
3 Terminology.....	4
4 Some peculiarities of reference material serial production.....	4
5 General requirements for the programme of reference material certification.....	6
ANNEX A: Bibliography.....	10
ANNEX B: (recommended) Structure and content of the programme of reference material certification.....	11

## **General requirements for the programme of reference material certification in serial production**

### **1 Scope**

This International Document sets out general requirements for the development of a documented procedure, describing the assessment of homogeneity and stability, characterization, assignment of property values and their associated uncertainties, certification of reference materials of the same type, released in series as certified reference materials (CRMs) and used in metrological activities, covered, in conformity with national laws, by state metrological control and supervision, exercised by national services of legal metrology.

*Note:*

In this document the term “reference material” is used as a generic term, to which certified reference material also belongs, - the reference material, characterized by a metrologically valid procedure for one or more specified properties, accompanied by a RM certificate that provides the value of the specified property, its associated uncertainty and a statement of metrological traceability [A.2, A.5].

It is recommended, that the principles, set out in this International Document be implemented by CRM producers in the development and release of CRMs, used in the fields of legal metrological control and supervision (LMCS), in the activities of national services of legal metrology and accreditation bodies, in the development of OIML publications.

This International Document has been developed taking into consideration the general concept, expressed in ISO/REMCO and OIML international publications concerning the development, release and use of CRMs. Account has also been taken of the concepts of OIML publications, establishing the use of certified reference materials in fields, covered by metrological control, exercised by national services of legal metrology [A.1]. This International Document rests upon ISO/REMCO Guides 30-35 (see Annex A) and supplements these Guides as concerns the requirements for documented procedures of CRM producers, by which the determination of characteristics and the certification of released batches or units of the same CRM type, produced in series and intended for use in the field of LMCS, are periodically carried out during a long period of time.

It is recommended, that producers of CRMs, released in series and intended for use in the field of LMCS, follow the requirements of ISO Guides 30-35 (see Annex A) and OIML D 18 (A.1).

When determining characteristics of non-certified reference materials, to be used in the field of LMCS in the absence of corresponding CRMs or, e.g. accepted

for use in pharmacopoeia documents, the recommendations, set out in this document or ISO Guides 30-35 (see Annex A) should be followed as far as possible.

## **2 General concepts**

CRMs are widely used in metrological activities, related to the fields, covered by LMCS, e.g., for the verification, calibration of measurement instruments, validation and control of measurement procedures, etc. General principles of CRM use in the fields, covered by metrological control and supervision, exercised by national services of legal metrology, are set out in OIML D 18 [A.1].

Uninterrupted release of interchangeable CRMs of the given type, used in the field of LMCS from batch to batch or from unit to unit of proper quality during a long period of time can be ensured by the CRM producer, provided, that he has documented procedures (technical documentation, as described in subclause 4.4 [A.1]), clearly and in detail describing the range, sequence and specific work operations on the production and certification of the CRMs.

*Note:*

CRMs, produced in compliance with the same technical documentation, from the same or similar in composition material, having the same property to be certified, intended use, statement of metrological traceability of the certified value, the same or similar property value and accuracy are referred to as CRMs of one and the same type.

The important step in the production of any CRM type is the assessment of homogeneity and stability, characterization, assignment of property values and their associated uncertainties, certification of reference materials. The programme of certification of series produced CRMs (hereinafter referred to as certification programme) is technical documentation, describing the range and the sequence of work on the determination of characteristics of the given CRM type, produced in series. Certification programme relates to the documentation of CRM producer's quality management systems [A.5].

*Note:*

CRM producer's certification programme as a document, which is the part of the technical documentation for CRMs, intended for use in the field of LMCS, is envisaged under subclause 4 of OIML D 18 [A.1]. The requirement to document programmes, procedures instructions and other documents is provided under subclause 4.3.1 of ISO Guide 34 [A.5].

General requirements for the competence of RM producers and requirements for RM certification are established in ISO Guide 34 [A.5] and ISO Guide 35 [A.6] respectively. This International Document specifies the minimum requirements

for the development of the document – certification programme, used in the production of CRMs, intended for use in the field of LMCS. The concepts of this International Document may be extended in national regulatory documents taking into account specific features and practical work experience in various countries.

### **3 Terminology**

The terminology used in this Document is in line with:

- The *International vocabulary of metrology – Basic and general concepts and associated terms*(VIM) [A.7];
- The *International vocabulary of terms in legal metrology*(VIML) [A.8];
- ISO Guides 30-35 (subclauses A.2 –A.6 of Annex A).

### **4 Some peculiarities of reference material serial production**

4.1 Serial production of a specific CRM type envisages periodic release of the CRM in batches or in units. The frequency and the volume of re-occurring CRM batches or units are established by a CRM producer, who when planning production, specifies the relevant information in technical documentation on CRM type. Serial production of CRMs is carried out according to one and the same documented procedures, describing in detail technical and operating requirements subject to the provisions of ISO Guide 34 [A.5].

4.2 In the process of serial production of CRMs, intended for use in the field of LMCS, the producer should follow the provisions of OIML D 18 and ISO Guides 30-35 (see Annex A).

4.3 The determination of CRM characteristics in the framework of serial production, namely, the assessment of homogeneity, stability, the property value and its associated uncertainty, the assessment of commutability (if necessary) and also monitoring of stability should be carried out with each renewal issue of CRM batch or unit of the given type, which is a solid material, including suspensions and sediment in conformity with ISO Guides 34, 35 [A.5, A.6] according to certification programme, developed for the given CRM type. For re-occurring batches or units of CRMs, which are solutions, gases or pure

compounds it is allowed not to carry out homogeneity study in full, as stipulated by ISO Guide 35 [A.6], provided that the necessary work according to [A.6] was carried out for the first CRM batch or unit. In this case, when renewal CRM batches or units are produced applying the same procedures, that were used for the production and certification of the first CRM batch or unit, appropriate verification assessments are required to ensure that estimations of uncertainty due to inhomogeneity, obtained on the first batch or unit remain applicable also for the new CRM batch or unit with account of established acceptance criteria.

In the framework of serial production it is allowed not to carry out stability study in full, as stipulated by ISO Guide 35 [A.6], for renewal release of CRM batch or unit of the given type according to the same procedures, that were used in the production and certification of the first CRM batch or unit. In this case, appropriate verification assessments are sufficient to ensure that estimations of uncertainty due to instability, obtained on the first batch or unit remain applicable also for the new CRM batch or unit with account of established acceptance criteria. Monitoring of stability should be provided for all CRM batches or units of serial production in compliance with the requirements of ISO Guide 34[A.5].

*Note:*

The results of certification of the first and subsequent CRM batches or units of the given type, including the results of the assessment of homogeneity, stability, monitoring of stability, recertification, etc. should be documented by CRM producer in compliance with the requirements of ISO Guide 34 [A.5].

Assumptions, provided under subclause 4.3, hold true, if no amendments were introduced in the documented procedures during the production and certification of re-occurring CRM batches or units, which, eventually could result in property values and estimated uncertainties, different from the planned ones for this CRM type.

4.4 In the long-term serial release of CRMs, the documentation may be amended due to:

- a) the use of new candidate materials (e.g., materials from other producers, other deposits, etc.) for the production of this CRM;
- b) procedures for CRM candidate material processing (subclause 5.8 of ISO Guide 34 [A.5]);
- c) a new CRM container, made of material, different from container material, used for the first CRM batches or units;
- d) the changes in CRM transportation conditions (e.g., temperature), when planning to ship it to other regions of the world; storage and application conditions;

Depending on the type of introduced or proposed amendments in CRM documentation within the works on the determination of characteristics of CRM subsequent batch or unit, the following works, set out in certification programme, should be carried out:

- when amendments are introduced according to a) and b), the assessment of homogeneity and stability of a new CRM batch or unit should be carried out in full in accordance with the requirements of ISO Guide 35 [A.6];
- when amendments are introduced according to c) and d), the assessment of stability of a new CRM batch or unit is carried out in full in accordance with the requirements of ISO Guide 35 [A.6];

Amendments, related to measurement methods and procedures, measurement instruments, CRMs, used in the determination of CRM characteristics, should satisfy the need to release the given CRM type with specified intended use, accuracy and metrological traceability.

4.5 In the framework of CRM serial production it is allowed to recertify the material according to sub-clause 6.7[A.6] when the actual CRM property value deviates from the certified value, provided in the CRM certificate. In this case the work on the assessment of homogeneity and stability, characterization, assignment of certified property values and their associated uncertainties should be repeated in compliance with certification programme.

*Note:*

All CRMs, produced in series, should be accompanied by a certificate, corresponding to ISO Guide 31 [A.3], issued as a result of certification or recertification.

4.6 During the production of CRMs, intended for use in the field of LMCS, the following legal requirements should be observed:

- all measurements, stipulated by procedures, should be made in compliance with ISO/IEC 17025[A.10];
- measurement methods should be validated and comply with ISO/IEC 17025 [A.10] and ISO Guide 34[A.5];
- primary measurement standards, used for measurement (if necessary), should undergo regular comparison with primary measurement standards of other countries, subject to the infrastructure, established by the International Bureau of Weights and Measures and Regional Metrological Organizations. If there are no primary measurement standards in a particular field of measurement in CRM producer's country, national measurement standards, traceable to the implementation of the definition of the unit through a primary measurement standard of another country should be used [A.10];
- to achieve metrological traceability of measurements, measurement standards (see above) and/or certified reference materials with established

metrological traceability should be used, as far as possible;

- measurement instruments should be calibrated and verified and should ensure metrological traceability of measurement results to accepted reference.
- measurements should be made only by a laboratory, competent in the relevant field of measurement.

*Notes:*

1. Additional requirements for measurement methods, measurement instruments and testing equipment are provided in ISO Guides 34 and 35 [A.5, A.6];
2. Requirements, set out in ISO 15189, may be used for tests in the field of medicine.

## **5 General requirements for the programme of reference material certification**

5.1 The CRM producer develops the certification programme to establish the amount, sequence of works, description of specific procedures, carried out when determining characteristics, namely, the assessment of homogeneity, stability, characterization, assignment of certified property values and their uncertainties, the assessment of commutability (if necessary) and also post-certification monitoring of stability of the given CRM type released in series with specified intended use, accuracy and metrological traceability.

*Notes:*

- 1 Certification programme is an integral part of CRM producer's technical documentation for CRM release.
- 2 Certification programme may be issued as a separate document or as a section of CRM technical assignment, section or part of the document (specifications), which establishes the order of CRM serial production.

5.2 Certification programme is intended for use by the personnel of CRM producer, periodically performing specific work on the determination of CRM characteristics in the release of new batches or units of the same CRM type with specified intended use.

*Note:*

The personnel of CRM producer, conducting the work on the determination of characteristics of the given CRM type should be made aware of the certification programme.

5.3 Certification programme is developed prior to the work on the determination of characteristics of re-occurring CRM batches or units based on:

- initial data (e.g., the intended use of the future CRM, planned range of the property values and their associated uncertainties, planned conditions of transportation, storage and safety of the CRM, etc.);
- the data, provided in CRM technical assignment (project);



- the results of certification of the first CRM batch or unit, obtained during the development of the given CRM type.

5.4 During the development of certification programme specific requirements are established for:

- preparation of CRM candidate material for carrying out the studies (if necessary);
- subcontractors, involved in the work (if necessary);
- the procedure for the determination of CRM characteristics;
- the method (-s) of the establishment and demonstration of metrological traceability of CRM property values;
- the documents, issued as a result of the studies;
- the time schedule for carrying out the work on the studies;
- certification;
- the procedure of monitoring of CRM stability;
- recertification.

5.5 The section “The procedure for the determination of CRM characteristics” of certification programme describes:

- the procedure for the assessment of homogeneity;
- the procedure for the assessment of stability;
- the procedure for characterization;
- the procedure for the assessment of CRM commutability (if necessary);
- the procedure for testing with the purpose of the assessment of applicability of estimations of uncertainties due to inhomogeneity and instability, obtained for the first CRM batch or unit, for a new CRM batch or unit (if necessary);
- the algorithms of calculation of the CRM certified value and expanded uncertainty of the certified value.

The section establishes the requirements for:

- the measurement procedures, measurement instruments, CRMs, other equipment, used in the study;
- the measurement standards, ensuring the establishment of metrological traceability or (in their absence) for the accepted references;
- the number of samples, the number of measurement results, needed for the assessment of homogeneity and stability, characterization, monitoring of stability, assessment of CRM commutability (if necessary), testing for the purpose of the assessment of applicability of estimations of uncertainties due to inhomogeneity and instability, frequency of measurements;
- the conditions of carrying out experimental work;
- the algorithms of processing measurement results;
- the algorithms for the calculation of the CRM certified value, its associated uncertainty and its components;

- the form of reporting the obtained results.

*Notes:*

1. Certification programme may include the requirements for measurement conditions, the qualification of operators, in case there are special requirements, not specified in the applied procedures.

2. When describing specific requirements (subclauses 5.3, 5.4), it is recommended to follow provisions set forth in [A.5, A.6].

5.6 Recommendations on the structure and presentation of certification programme are given in Annex B.

5.7 The title page of certification programme should contain the date of its approval. The pages of the document should be enumerated.

5.8 It is recommended to include the sheet of changes in certification programme, which, if required, may be subject to changes and additions over time.

5.9 Certification programme, covering CRMs, intended for the use in the field of LMCS [A.1], is subject to metrological examination, which evaluates:

- the correct use of metrological terms and concepts, designations and symbols of the SI units;

- the completeness of the procedures for the assessment of homogeneity and stability, characterization, assessment of CRM commutability (if necessary), for monitoring of CRM stability, sufficient to carry out the relevant work by the personnel of the CRM producer;

- the conformity of selected measurement procedures, measurement instruments, measurement standards, CRMs with the criteria, specified in [A.5], [A.6], [A.12] and in this document;

- the conformity of requirements for the sub-contractors, the requirements for laboratories, carrying out measurements, with the criteria, specified in [A.5], [A.6] and in this document;

- the validity of choice of the method for establishing metrological traceability of CRM certified values, taking into consideration the provisions, set out in [A.5], [A.6], [A.9], [A.12];

- the conformity of the algorithms of the calculation of the certified value, its associated uncertainty value with the provisions, set out in [A.6] and in national regulatory documents.

*Note:*

Metrological examination of documentation is carried out in conformity with the provisions, adopted in the countries.

Positive results of metrological examination are the basis for the decision, taken by the CRM producer to approve certification programme.

5.10 The CRM producer may introduce amendments in certification programme over time with account of provisions [A.5].

*Note:*

When the amendments, introduced in certification programme, are related to the amendment in the method of RM characterization or/and measurement procedures, used in the assessment of homogeneity and stability, characterization, monitoring of stability, assessment of CRM commutability (if necessary), certification programme is subject to repeated metrological examination to verify the possibility of the release of the given CRM type with specified intended use, accuracy and metrological traceability.

## **Annex A: Bibliography**

- A.1 OIML International Document D 18, *The use of certified reference materials, in fields, covered by metrological control, covered by national services of legal metrology. Basic principles*, 2008
- A.2 ISO Guide 30:2015, *Reference materials – Selected terms and definitions*
- A.3 ISO Guide 31:2000, *Reference materials – content of certificates and labels*
- A.4 ISO Guide 33:2015, *Reference materials- Good practice in using reference materials*
- A.5 ISO Guide 34:2009, *General requirements for the competence of reference material producers*
- A.6 ISO Guide 35:2006, *Reference materials – general and statistical principles for certification*
- A.7 OIML V 2-200, *International Vocabulary of Metrology — Basic and General Concepts and Associated terms(VIM)*, 3<sup>rd</sup> Edition. (Edition 2010 with minor Corrections), 2012
- A.8 OIML V 1, *International vocabulary of terms in legal metrology (VIML)*, 2013
- A.9 *Joint BIPM, OIML, ILAC and ISO Declaration on Metrological Traceability*. OIML Bulletin, Volume LIII, Number 1, January 2012.
- A.10 ISO/IEC 17025:2005, *General requirements for the competence of testing and calibration laboratories*
- A.11 ISO/REMCO Position Paper, *The need for assessment of commutability of reference materials*, 2014
- A.12 OIML D 1:2012, *Considerations for a law on metrology*.

## **AnnexB**

### **(recommended)**

#### **B.1 Structure and content of reference material certification programme**

B.1.1 The name of the document, establishing certification programme, is based on the area of its application.

*Example:*

“The programme of certification of a reference material for composition of diesel fuel of serial production”

B.1.2 Certification programme includes the introductory part and the following sections:

- general concepts (subclause B.1.4);
- preparation of CRM candidate material for carrying out the studies (if necessary) (subclause B.1.5);
- subcontractors, involved in the work (if necessary) (subclause B.1.6);
- procedure for the determination of CRM characteristics (subclause B.1.7);
- the method(-s) of the establishment and demonstration of metrological traceability of CRM property values (subclause B.1.8);
- the documents, issued as a result of the studies (subclause B.1.9);
- the time schedule for carrying out the work on the studies (subclause B.1.10);
- certification (subclause B.1.11);
- the procedure of monitoring of CRM stability (subclause B.1.12);
- recertification (if necessary) (subclause B.1.13).

*Note:*

It is allowed to delete, reduce or supplement these sections taking into consideration CRM specific features and the data, established in the determination of characteristics of the firstCRMbatchor unit.

B.1.3 Introductory part establishes the intended use and scope of certification programme.

Introductory part is set outas follows: “This document establishes certification programme (the name of CRM is indicated), to be used in CRMserial production”.

B.1.4 Section “General concepts” of certification programme includes subsections, specified under subclauses B.1.4.1-.B.1.4.2.

##### **B.1.4.1 Subsection “Information on CRM producer”**

This subsection includes the name and the address of CRM producer.

#### B.1.4.2 Subsection “The names of the property, to be certified, prescribed range of property values”

This subsection includes:

- the name of the property to be certified,
- the range of permissible certified values and their associated uncertainties,
- other information (as appropriate).

Note:

This subsection may provide a reference to other documents of CRM producer, in which this information is given.

#### B.1.5 Section “Preparation of CRM candidate material for carrying out the studies”

This section is prepared as appropriate. It includes the requirements for:

- the procedure for sampling material (as appropriate);
- the procedure for material preparation (as appropriate).

##### B.1.5.1 Subsection “The requirements for sampling material”, prepared as appropriate, includes the requirements for:

- qualification of the operator, conducting the sampling (e.g. the information on qualification of the operator, his education, the information on his skill upgrading and safety training, etc.);
- the period of time within which the sampling of material should be completed;
- the equipment, measurement instruments, used for material sampling (the name of equipment, measurement instruments, identification of documents, according to which they are issued, requirements for calibration and verification of measurement instruments);
- the amount of material, sufficient for all kinds of measurements (e.g. mass and/or volume of material, etc.);
- the procedure for material sampling (description of sampling procedure should be provided);
- the container, into which the borrowed samples of material are placed (the name of container, material, the container is made of, capacity of the container (volume and/or mass);
- the identification and labeling material sample (the type of labelling, the form of labelling);
- storage and transportation conditions (if required) of borrowed samples of material.

B.1.5.2 The subsection “Requirements for material preparation procedure” is included in certification programme when the certified value of CRM is determined by preparation procedure.

Subsection “Requirements for material preparation procedure” includes the information on material preparation procedure, providing the requirements for:

- the initial materials (substances), used for the preparation of material;
- the measurement instruments, used for the preparation of material;
- the qualification of personnel, engaged in the preparation of material;
- the environmental conditions, under which material is prepared;
- the material preparation procedure;
- the identification and labelling of material;
- the containers into which the prepared material is placed;
- the storage conditions for the prepared material.

#### B.1.6 Section “Subcontractors, involved in the work”

This section is included when subcontractors are involved in the work. It includes:

- the requirements for the competence of subcontractors and testing laboratories, taking into consideration recommendations [A.5], [A.6], of this document;
- the specific kind of work, performed by a subcontractor, the terms of interaction.

#### *Note*

It is possible to include other required information on legal entities, involved in the work on laboratory accreditation.

#### B.1.7 Section “The procedure for the determination of CRM characteristics”

This section includes the requirements for the procedure for the determination of CRM characteristics. Recommended content of the procedure is described under subclause B.2.

#### B.1.8 Section “The method(-s) of the establishment and demonstration of metrological traceability of CRM property values”

This section includes the information (e.g. the number, name, etc.) on national or international measurement standards or other accepted references, which ensure the obtaining measurement results with established metrological traceability of property values.

#### *Note:*

It is recommended to provide in this section the unit (-s), to which metrological traceability of property values is ensured and the scheme of the unit transfer in the determination of CRM certified value.

In case, when it is difficult to establish metrological traceability of property values, the information on the proposed procedure (-s), specified under subclauses 5.12 [A.5] is provided.

#### B.1.9 Section “The documents, issued as a result of the studies”

This section provides the requirements for the documents, issued as a result of the assessment of homogeneity and stability, characterization, monitoring of stability, assessment of CRM commutability (if necessary), testing for the purpose of the assessment of applicability of estimations of uncertainties due to inhomogeneity and instability, (including the report, protocols, etc.), taking into consideration the provisions, set out in [A.5], [A.6].

#### B.1.10 Section “The time schedule for carrying out the work on the studies”.

This section indicates the time schedule or frequency for carrying out the work on the studies of assessment of homogeneity and stability, characterization, monitoring of stability, assessment of CRM commutability (if necessary), testing for the purpose of the assessment of applicability of estimations of uncertainties due to inhomogeneity and instability.

#### B.1.11 Section “Certification”

This section describes the work on certification in conformity with clause 11 [A.6]. The CRM certificate, envisaged by [A.5] and [A.6] is issued in accordance with [A.3].

This section establishes the requirements for:

- qualification of personnel (e.g., profession, education, practical experience, position in the company), engaged in the certification.
- the form of certificate for the given CRM type.

#### *Note:*

In certification programme the form of certificate for the given CRM type, released by CRM producer, may be provided as an example.

#### B.1.12 Section “Procedure for monitoring of CRM stability”

The evaluation of monitoring of CRM stability is carried out in accordance with subclause 8.4 [A.6]. The section includes subsections, specified under subclauses B.1.12.1- B.1.12.9.

B.1.12.1 Subsection “Information on the method, used in monitoring of CRM stability” is prepared with account of provisions, specified under subclause 8.4 [A.6] with an indication of methods, used in monitoring CRM stability :

- classical experiment;
- isochronous method.



**B.1.12.2 Subsection “Reference material storage conditions and frequency of measurements during monitoring of CRM stability”**

This subsection includes:

- CRM storage conditions, provided in CRM certificate (environmental conditions, humidity, lighting, etc.);
- the interval of time, at which the study within the framework of monitoring of CRM stability is carried out;
- the frequency of measuring CRM property value in monitoring of CRM stability by the method, selected under subclause B.1.12.

When selecting parameters, it is recommended to follow provisions, set out in [A.6].

**B.1.12.3 Subsection “Measuring instruments, reference materials, chemical reagents, testing equipment etc., used for measurements during monitoring of CRM stability”**

This subsection is prepared in the absence of the relevant information in documented measurement procedures, used during monitoring of CRM stability. The subsection provides the list of measurement instruments, CRMs, supporting devices, materials, chemical reagents used for making measurements.

**B.1.12.4 Subsection “Measurement procedure (-s), used during monitoring of CRM stability”**

This subsection describes measurement procedure (-s), used during monitoring of CRM stability and makes references to the relevant documented procedure (-s).

**B.1.12.5 Subsection “Preparation of CRM for measurements”**

This subsection describes preparatory work, related to CRM preparation for measurements, to be carried out prior to measurements (if necessary).

**B.1.12.6 Subsection “Execution of work”**

This subsection presents the order of work, including:

- the list and sequence of operations;
- the number and frequency of measurements;
- the requirements for reporting intermediate and final measurement results (the number of significant figures, etc.,) etc.

*Note:*

This information is provided for each experiment, envisaged in the framework of stability monitoring.

**B.1.12.7 Subsection “The form of reporting measurement results, obtained during monitoring of CRM stability”**

This subsection includes the form of reporting measurement results, obtained during monitoring of CRM stability.

B.1.12.8 Subsection “The algorithm of processing measurement results with a view to estimate the uncertainty of stability due to instability”.

This subsection indicates:

- the algorithm of calculation of the standard uncertainty due to instability, obtained during monitoring of CRM stability;
- the criterion of CRM stability demonstration according to subclause 8.2.4 [A.6].

B.1.12.9 Subsection “The form of reporting results of monitoring of CRM stability”

This subsection includes the form of report, prepared as a result of monitoring of CRM stability and the form of recommendation on further actions to be taken for the CRM according to [A.6].

B.1.13. Section “Recertification”

This section is prepared with account of provisions, set out under subclause 6.7 [A.6], if necessary. The section includes:

- the reasons for carrying out CRM recertification with account of provisions, set put under subclause 6.7 [A.6] (e.g. the change of the property value, identified as a result of monitoring of stability, etc.);
- the list of work to be carried out during CRM recertification, with an indication of certification programme sections, describing the procedures of the relevant work;
- the procedure of CRM preparation for reassessment of homogeneity, stability, recharacterization (e.g., opening the bottles, mixing the material, etc.) (if necessary).

In case of CRM recertification due to the need to reduce the uncertainty of the property value and clarify the certified value, to establish metrological traceability to a new unit of measurement (when new measurement standards and certified reference materials appear), it is necessary:

- to list the conditions, which will ensure the solution of the problem;
- to prepare and make appropriate amendments to certification programme in accordance with ISO Guide 34 [A.5].

## **B.2 The content of certification programme section “Procedure for the determination of CRM characteristics”**

B.2.1 The section “Procedure for the determination of CRM characteristics” of certification programme includes the following sections:

- the procedure for the assessment of homogeneity (subclause B.2.2);
- the procedure for the assessment of stability (subclause B.2.3);
- the procedure for characterization (subclause B.2.4);
- the procedure for the assessment of CRM commutability (if necessary) (subclause B.2.5);
- the procedure of testing with the purpose of the assessment of applicability of estimations of uncertainties due to inhomogeneity and instability, obtained for the first CRM batch or unit, to a new CRM batch or unit (if necessary) (subclause B.2.6);
- the algorithm of calculation of the certified value and expanded uncertainty of the CRM certified value (subclause B.2.7);

*Notes:*

1 It is allowed to delete or merge these sections or include additional sections taking into consideration CRM specific features.

2 It is allowed to issue the procedure for the assessment of homogeneity, the procedure for the assessment of stability and the procedure for RM characterization as separate documents.

B.2.2 The section “Procedure for the assessment of homogeneity” includes subsections, described under subclauses B.2.2.1-B.2.2.8.

B.2.2.1 Subsection “Properties, by which the assessment of homogeneity is carried out”

This subsection includes:

- the names of the property, by which the assessment of homogeneity is carried out;
- the type of inhomogeneity (between-bottle, within-bottle ), to be studied.

B.2.2.2 Subsection “Measurement instruments, reference materials, chemical reagents, testing equipment, etc., used for carrying out the work on the assessment of homogeneity”.

This subsection is prepared in the absence of appropriate information in documented measurement procedures, used in the assessment of homogeneity.

This subsection provides the list of measurement instruments, CRMs, support devices, materials, chemical reagents, used in measurements.

B.2.2.3 Subsection “Measurement conditions”

Subsection “Measurement conditions” establishes the requirements for the environment, accommodation and other factors, affecting measurements, CRM

candidate material or provides reference to the measurement procedure (-s) in use.

#### B.2.2.4 Subsection “Preparation of CRM candidate material for measurements”

This subsection provides (as appropriate) the description of preparatory work, related to material preparation for measurements, carried out prior to measurements, namely:

- the information on the amount of borrowed samples of material for measurements, on the mass of samples;
- the information on the methods of preparation of material surface for measurements (for solid materials);
- the information on the packaging material, in which the material is stored during the assessment of CRM homogeneity, etc.;

#### B.2.2.5 Subsection “Measurement procedure (-s), used in the assessment of homogeneity”

This subsection describes the measurement procedure (-s), used in the assessment of CRM homogeneity or provides reference to the appropriate documented measurement procedure (-s).

#### B.2.2.6 Subsection “Execution of work”

This subsection describes the procedure of work in the assessment of homogeneity:

- the list and sequence of operations, performed in the assessment of CRM homogeneity;
- the details of the part of CRM, within-bottle homogeneity of which is studied (e.g. disc diameter, cylinder height, etc., for solid material; the mass of the sample to be tested, the mass of the minimum representative sample for disperse material, etc.);
- the number of measurements, the frequency of measurements for the assessment of within-bottle homogeneity;
- the number of measurements, the frequency of measurements for the assessment of between-bottle homogeneity;
- the requirements for reporting intermediate and final measurement results (the number of significant figures, etc.).

#### B.2.2.7 Subsection “Algorithm of the calculation of standard uncertainty due to inhomogeneity”

This subsection contains the algorithm of measurement results processing to calculate the value of the standard uncertainty due to inhomogeneity or the reference to the appropriate document, in which the algorithm is described.

#### B.2.2.8 Subsection “Additional information”

This subsection contains the requirements for the necessity of recording additional information, obtained from the results of the assessment of CRM homogeneity, namely:

- minimum representative sample (for dispersed materials),
- details of the part of solid CRM material, for which the homogeneity study is carried out (for solid materials), etc. for providing the relevant information in the report of CRM certification and in CRM certificate.

B.2.2.9 It is recommended to include in the procedure for the assessment of homogeneity the following subsections:

- “Requirements for qualification of operators” containing the information about the level of skills (e.g. profession, education, experience, etc.) of the personnel, permitted to carry out the work;
- “Requirements for safety of work”, establishing the requirements, that will ensure labor safety, compliance with the norms of workplace sanitation and environmental protection.

*Note:*

If the normative documents, regulating the requirements for safety, workplace sanitation and environmental protection are available, a reference to these documents is made in this subsection.

B.2.3 Section “Procedure for the assessment of stability”

Section “Procedure for the assessment of stability” includes subsections, specified under subclauses B.2.3.1 - B.2.3.12.

B.2.3.1 Subsection “The list of factors, affecting CRM stability”

This subsection provides the list of factors (e.g. temperature, humidity, lighting, etc.), which may affect CRM stability.

B.2.3.2 Subsection “Method of stability study”

This subsection provides the information on the method, used to study CRM short-term and long-term stability in conformity with [A.6]:

- isochronous method,
- method of natural ageing.

B.2.3.3 Subsection “Storage conditions of CRM and frequency of measurements in the assessment of stability”

Subsection includes:

- CRM storage conditions
  - environmental conditions (temperature, humidity, lighting, etc.), under which CRM storage and transportation is planned, within the experiment, corresponding to planned conditions of CRM storage and transportation,

- material of container, in which CRM is stored during stability study;
- the frequency of measuring property values within the implementation of selected method of stability study.

When selecting parameters it is recommended to follow the provisions, set out in [A.6].

B.2.3.4 Subsection “Measurement instruments, reference materials, chemical reagents, testing equipment, etc., used for carrying out the work on the assessment of stability”

This subsection is prepared in the absence of appropriate information in documented measurement procedures, used for carrying out the work on the assessment of stability. It provides the information, specified under subclause B.2.2.2.

B.2.3.5 Subsection “Measurement procedure (-s), used for carrying out the work on the assessment of stability”

This subsection describes the measurement procedure (-s), used for carrying out the work on the assessment of CRM stability or makes reference to the appropriate documented measurement procedure (-s).

B.2.3.6 Subsection “Preparation of CRM candidate material for measurements”

This subsection provides the description of preparatory work, related to material preparation for measurements, carried out prior to measurements.

B.2.3.7 Subsection “Execution of work”

This subsection describes the procedure of work in:

- defining CRM storage conditions,
- defining CRM transportation conditions,
- selecting CRM packaging material,

namely:

- the list and sequence of operations;
- the number of measurements, the frequency of measurements;
- the requirements for reporting intermediate and final measurement

results

(the number of significant figures, etc.).

- others.

*Note:*

This information shall be provided for each experiment in the determination of

- short-term stability,
- long-term stability (when CRM transportation is planned).;

- CRM stability after opening the packaging (when CRM storage and use is allowed after opening the packaging).

B.2.3.8 Subsection “The form of reporting measurement results, obtained in the assessment of stability”

This subsection provides the form of reporting measurement results, obtained in the assessment of CRM stability

B.2.3.9 Subsection “Algorithm of the calculation of standard uncertainty due to instability”

This subsection provides the algorithm of the calculation of:

- standard uncertainty, characterizing CRM short-term and long-term instability;
- CRM life time

or the reference to the appropriate document, in which the algorithm of calculation is set out.

B.2.3.10 Subsection “The form of reporting the results of the assessment of stability”

This subsection provides:

- the form of reporting the standard uncertainty due to short-term and long-term stability, life time of CRM after its production;
- the information on how to achieve CRM integrity:
  - - CRM storage conditions;
  - - CRM transportation conditions;
  - - CRM packaging (including packaging material), CRM shipping container, ensuring CRM stability under storage and transportation conditions during the validity period of CRM certificate and the period of CRM transportation;
  - - possible restriction of CRM life time after opening the packaging, CRM storage conditions after opening the packaging.

B.2.3.11 It is recommended to include in the procedure subsections “Requirements for qualification of operators” and “Requirements for safety of work”, specified under subclause B.2.2.9.

B.2.4 Section “The procedure for characterization” is prepared taking into consideration the recommendations, set out under subclauses B.2.4.1 – B.2.4.9. the

#### B.2.4.1 Subsection “Method of characterization”

This subsection provides the method of characterization, selected in conformity with recommendations, specified in [A.5] and [A.6].

*Note:*

The content of the procedure for the characterization of reference material depends on the selected method of characterization.

B.2.4.2 Subsection “Information on measurement standards, measurement instruments, reference materials, chemical reagents, testing equipment, etc., used in reference material characterization” is prepared in the absence of appropriate information in documented measurement procedures, used in characterization.

This subsection provides the information on measurement standards, measurement instruments, CRMs, testing equipment and establishes the requirements for their characteristics, calibration and certification.

#### B.2.4.3 Subsection “Measurement procedure (-s), used during characterization”

This subsection describes the procedure (-s), used in the determination of CRM property value or makes reference to the document (-s) in which it (they) is (are) established. This subsection provides the information on the units, to which the metrological traceability of measurement results is ensured in the realization of measurement procedure.

B.2.4.4 When the CRM property value is determined using measurement standard, section “Procedure for reference material characterizaion” shall additionally include:

- the information on the measurement standard;
- the information on measurement conditions;
- the information on the number of test results to be obtained when measurements are made using the measurement standard;
- the form of reporting measurement results.

B.2.4.5 When the CRM property value is determined from the results of interlaboratory comparison, section “Procedure for reference material characterization” shall additionally include subsection, specified under subclause B.2.4.5.1.

*Note:*

In case of reference material characterization based on the results of interlaboratory comparison, subsections, specified under subclauses B.2.4.2 – B.2.4.4 are included in section “Procedure for reference material characterization” as appropriate.



#### B.2.4.5.1 Subsection “Information on the programme of interlaboratory comparison”

*Note:*

It is recommended to prepare the programme of interlaboratory comparison as a separate document.

The programme of interlaboratory experiment shall include:

- the information on material (the name of CRM candidate material, the name of the property value to be measured, minimum representative sample, methods of sample storage, safety measures when working with samples, etc.) submitted to laboratories;
- the recommended measurement procedures (methods) to measure property value (as appropriate);
- the time schedule for carrying out the work, including the time for sample distribution and obtaining the protocols of measurement results;
- the requirements for the number of measurement results, obtained in laboratories;
- the requirements procedure for material sampling, performed in laboratories (as appropriate);
- the requirements for measurement conditions to be provided by laboratories when carrying out experimental work;
- the requirements for metrological traceability of measurement results, obtained in each laboratory;
- the form of reporting property values by laboratories.
- other requirements, specified in [A.6] (as appropriate).

B.2.4.6 It is recommended to include in the procedure subsections “Requirements for qualification of operators” and “Requirements for safety of work”, specified under subclauses B.2.2.9.

#### B.2.5 Section “The procedure for the assessment of CRM commutability”

This section is prepared with account of provisions, set out in [A.5], [A.11], if necessary. It includes:

- the information on routine samples, submitted for testing together with CRMs (the name, the information on the material of routine samples, etc.);
- the information on the reference measurement procedure “of a higher order” (the reference is made to the document, describing in detail measurement procedure with an indication of the used measurement standards, and/or measurement instruments, measurement procedure, measurement conditions, measurement result processing, etc.);
- the information on one or more routine measurement procedures “of a lower order” (the reference is made to the document, describing in detail measurement procedure with an indication of the used measurement

instruments, measurement procedure, measurement conditions, measurement result processing, etc.);

- the information on the number of measurement results, to be obtained by measuring property values in routine samples and CRMs using reference measurement procedure “of a higher order” and one or more reference measurement procedures “of a lower order”;

- the form of reporting measurement results, obtained from measurements according to measurement procedures;

- the algorithm of processing results with the purpose of the assessment of CRM commutability with account of provisions, set out in [A.5], [A.11];

- the form of the report, prepared as a result of the assessment of CRM commutability with an indication of measurement procedure, relative to which the CRM is commutative, and the forms of reporting the results of commutability study in CRM certificate.

*Note:*

In the absence of reference measurement procedure “of a higher order”, the information of two or more measurement procedures, subject to harmonization, is provided instead.

B.2.6 Section “The procedure of testing with the purpose of the assessment of applicability of estimations of uncertainties due to inhomogeneity and instability, obtained for the first CRM batch or unit, to a new CRM batch or unit”

This section is prepared, as appropriate, with account of provisions, set out in [A.5] and under clause 3 of this document. The section includes:

- subsection “The procedure of testing with the purpose of the assessment of applicability of estimations of uncertainties due to inhomogeneity, obtained for the first CRM batch or unit, to a new CRM batch or unit”;

- subsection “The procedure of testing for the purpose of the assessment of applicability of estimations of uncertainties due to instability, obtained for the first CRM batch or unit, to a new CRM batch or unit”.

B.2.6.1 The section “The procedure of testing with the purpose of the assessment of applicability of estimations of uncertainties due to inhomogeneity, obtained for the first CRM batch or unit, to a new CRM batch or unit” includes:

- the information of estimations of uncertainties due to inhomogeneity, relative to CRM certified property for the first CRM batch or unit;

- criterion of applicability of estimations of uncertainty due to inhomogeneity, obtained for the first CRM batch or unit, to a new CRM batch or unit;

- the information on the measurement procedure, used for the measurement of property values in material of the subsequent CRM batch or unit (the reference is made to the document, describing in detail measurement procedure with an indication of the used measurement instruments, measurement procedure, measurement conditions, measurement result processing, etc.);
- the information on the number of measurement results, to be obtained by measuring property values in material of the subsequent CRM batch or unit; the requirements for the form of reporting measurement results;
- the algorithm of processing measurement results with the purpose of checking the applicability of estimation of uncertainties due to inhomogeneity, obtained relative to the certified property for the first CRM batch or unit, to a new CRM batch or unit with account of established acceptance criteria;
- the form of the report, prepared as a result of the test with an indication of the form of recommendation on the use of obtained results.

B.2.6.2 The section “The procedure of the testing for the purpose of the assessment of applicability of estimations of uncertainties due to instability, obtained for the first CRM batch or unit, to a new CRM batch or unit” includes subsections, similar to those, specified under subclause 2.6.1, but applied to instability study.

B.2.7 Section “The algorithms of the calculation of the certified value and the expanded uncertainty of CRM certified value”

This section provides specific algorithm of the calculation of the certified value and the expanded uncertainty of the certified value, including the uncertainty due to inhomogeneity, instability and due to characterization taking into account the provisions of [A.5] and [A.6].