

The APLMF Guide to

The selection and use of certified reference materials for legal metrological control in food safety & agricultural products

Foreword

The Asia-Pacific Legal Metrology Forum (APLMF) is a regional, inter-governmental organization whose primary aim is to harmonize the regulations and metrological controls applied by the national metrological services, or related organizations, of its Member States.

APLMF Draft Recommendations and Guides are developed by Working Groups concerned formed by representatives from Member Economies. Certain international and regional institutes also participate on a consultation basis if necessary.

This document was developed in accordance with the general concepts and principles set in related OIML and ISO/REMCO publications concerning the selection and use of CRMs in metrological activities. Account has also been taken of concepts set out in other OIML publications establishing general metrological requirements and rules.

This document is also based on ISO/REMCO Guides 30 to 35 with a view to facilitating a wider use of CRMs for legal metrological control in the field of food safety and agricultural products. In particular, the principle on traceability that has been widely recognized by the international metrology community is also emphasized in this guide.

The aim of this guide is to provide information on the selection and use of CRMs for legal metrology control with respect to agricultural products and food safety. This guide was developed by the Working Group on Metrology Control (MCWG) under APLMF.

The APLMF Publications may be downloaded from the APLMF website in the form of PDF files. Additional information on APLMF Publications may be obtained from the Organization's headquarters.

1. Introduction - Scope

Certified reference materials (CRMs) (defined in 3.2) make possible the establishment of metrological traceability (defined in 3.3) for measurements, which is indeed essential to ensure the reliability of the measurement results. This is very important when we come to consider measurements in support of international trade and national legal metrological control (defined in 3.4). For instance, CRMs are widely used for the calibration of measuring instrument, evaluation of measuring procedures and quality assurance purposes. All kinds of CRMs are playing an increasingly important role in national and international measurement activities, in proficiency testing, in the accreditation of laboratories, and in legal metrological supervision as well.

In recent years, food safety has become a very important issue around the world as the result of globalization of trade on food products. This issue raises a pressing need for reliable measurements to facilitate food registration; market surveillance; certification of food products for import, export and internal consumption. Typical examples include testing of agricultural products for the presence of pesticide residues, mycotoxins and other harmful contaminants. In fact, the number of inspection and laboratory testing related to these activities keep on increasing rapidly in many economies. Moreover, many economies have imposed legal and regulatory control on food products produced locally or imported from other economies. In this connection, CRMs are needed in management of measuring instrument and regulatory control of measurement results where metrological traceability is imperative to avoid unnecessary disputes or loss. However, as we could observe, the testing community and the authoritative bodies are faced with problems in the selection and use of CRMs especially in the field of agricultural products and food safety.

This document is intended to serve as a guide to selection and use of CRMs for the stated purposes. However, as a general guide, this document would only provide basic information on the topics. For more detailed information or procedures, users are encouraged to refer to other technical guides or standards issued by other international organizations such as OIML, CIPM and ISO. The content of this guide shall make reference to these relevant documents on the selection and use of certified reference materials (CRMs) wherever appropriate.

2. General concepts

2.1 CRM

CRMs are normally developed and issued by national metrology institutes or other authoritative bodies with proven competency. They are widely used in metrological activities such as verification, calibration of measuring system and quality control of measurement procedures. When used for such purposes, CRMs also become the object of metrological control and supervision. In this aspect, detailed procedures have been set out in OIML publication D18.

However, when selecting a CRM, it is imperative to take into consideration the intended metrological use, the metrological characteristics and the certification method of the CRM as indicated in its certificate. In particular, in some economies, there might be legal requirements on the imported CRMs. Besides, technical and administrative requirements have to be considered also. In this regard, recommendations of ISO Guides 30 and 33 should be taken into account respectively.

In cases where appropriate CRMs are not available to meet identified needs, the following alternatives might be helpful.

- Laboratory or regulatory body concerned may attempt to develop its own in-house RM. Considering the lengthy and costly operation that might be involved, it is highly recommended that CRM producers capable of developing such a new CRM should be contacted to explore the possibilities. Or groups of users with the same need be contacted and try to set up a joint project with the assistance of the national laboratory responsible for CRMs.
- Laboratory or regulatory body may seek the assistance from their national laboratory or other metrological institutes abroad to assign a reference value for their in-house RMs in a way the requirements on traceability and uncertainty could be fulfilled.
- Instead of RMs, laboratory or regulatory body may apply recognized reference methods in the measurement. Of course, it is presumed that the procedures were strictly followed and the required experimental conditions were used as stipulated in the reference methods. One of the sources of reference methods for food analysis is the Official Methods of Analysis of AOAC INTERNATIONAL which covers a wide range of analysis including microbiology, composition, nutrition,

contaminants in foods. An important feature of the Official Methods of Analysis is the international source of many of the methods, with many countries and international organizations contributing their expertise to method standardization.

The majority of measurements made in the field of agricultural products and food safety are within the framework of the International System of units (SI), i.e. measurement results directly expressed in SI units or derived SI units. However, there are measurements, which involve conventional scales that could not be expressed in SI units, for e.g. pH value. For this, national standard specifications shall be applied to the certification of related reference materials that could then be used as CRMs for that kind of measurement.

2.2 Assurance of metrological control

The essential goal of legal metrology is to ensure fairness in the marketplace and to contribute to the health and safety of the general public. This would not be reached unless the legal metrology service give an assurance of entire measurement process which includes the instrument, operator, environment, procedure and special characteristics of the item being measured, performs properly. In particular, CRMs are needed as a kind of metrological tool to monitor the performance in terms of minimum required accuracy, target compliance and desired level of confidence. However, to ensure an effective metrological control, other factors like cost, flexibility, training, verification procedures, reliability of instruments have to be considered.

3. Terminology

The terminology used in this guide is in line with:

- the *International vocabulary of basic and general terms in metrology* (VIM);
- the *International vocabulary of terms in legal metrology* (VIML); and
- relevant ISO & OIML publications.

3.1 Reference material (RM)

Material, sufficiently homogeneous and stable with reference to specified properties, which has been established to be fit for its intended use in measurement or in examination of nominal properties.

3.2 Certified reference material (CRM)

Reference material, accompanied by documentation issued by an authoritative body and providing one or more specified property values with associated uncertainties and traceabilities, using valid procedures.

3.3 Metrological traceability

Property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to its measurement uncertainty.

3.4 Legal metrological control

The whole of legal metrology activities which contribute to metrological assurance. Legal metrological control includes:

- 3.4.1 legal control of measuring instruments;
- 3.4.2 metrological supervision;
- 3.4.3 metrological expertise.

Note: Detailed descriptions of the above elements are provided in OIML publication D9.

5. Selection of CRMs

5.1 Search for CRMs

Information about reference materials is available from a number of sources. The COMAR Database, contains information on more than 11,000 RMs/CRMs which can be accessed directly through the internet (www.comar.bam.de). Users can also obtain the information directly from the RM producers such as national metrology institutes or related international organizations.

The BIPM have developed two databases, one is categorized under Appendix C of the CIPM-MRA and the second under the remit of the Joint Committee for Traceability in Laboratory Medicine (JCTLM). Both data bases (www.bipm.org) provide useful information on traceability of the assigned property values.

The following are examples of matrix CRMs for food and agriculture that are currently available:

Matrix	Analytical parameters
Fruit, vegetable, oyster tissue, fish	Element analysis
Vegetable, cod liver oil, milk powder	Pesticide residue
Egg powder, corn starch, milk powder, fat oil	Fatty acids
Fish tissue, vegetables, milk powder	Nutritional constituent

Fish tissue, mussel tissue	Mercury, methyl-mercury, organo-arsenic, butyltins
Maize, peanut meal, peanut butter, milk powder	Mycotoxins such as aflatoxins, ochratoxins, zearalenone
Milk powder	Dioxins
Milk powder	Salmonella typhimurium, Escherichia coli
Soybeans, maize and other crops	Genetic modified organisms (GMO)

To calibrate the instruments used for food analysis, some calibration solutions (CRMs in solution form) may be used.

5.2 Aspects should be considered in selecting CRMs

In selecting the CRMs, the users would need to consider the following aspects. However, depending on the situation and usage, some of them may be accorded priority over others.

- **Fit for purposes.** The CRM should have properties at the appropriate level, have a matrix in a physical state and form that is as close as possible to those of the material subjected to the measurement process.
- **Characteristics.** The CRM should be effectively certified with traceability established for the certified value. Also, the uncertainty of the certified value should be compatible with the precision and trueness requirements.
- **Legal requirements.** The CRM should be able to fulfil the legal requirements on the precision and trueness for some special analyses. Besides, in some countries, there may be special requirements on the packaging and label especially for CRMs to be imported from abroad.
- **Regulation on transportation.** Countries normally would have restrictions on importing of special kind of goods, for e.g. materials of plant or biological in nature. So, CRMs fall within the restricted categories may need to obtain permit for the import.
- **Safety.** The CRMs manufacturers have to ensure the safety and stability of their materials especially for materials of biological in nature, for e.g. the materials

were properly irradiated before packaging.

- **CRMs producer.** Most of the CRMs currently available in the market are provided by national metrology institutes. Their competency in particular testing areas are demonstrated by their calibration and measurement capability (CMC) claims posted on the Appendix C of the Key Comparison Database (KCDB) set up by BIPM (<http://kcdb.bipm.org>). CRMs user should refer to the CMC information while making decision on which institute's products are preferred.

6. Use of CRMs

6.1 Typical procedures

For legal metrological control, there are a number of procedures that involve the use of CRMs. These procedures are summarized in Figure 1 for easy reference.

6.2. Real examples

6.2.1. Residues and contaminants in honey

In Mexico, there are laws for the control of residues and contaminants in food and feed. For honey, in particular, maximum residue limits (MRLs) for some specific substances are stipulated in laws, which are taken as legal limits under the National Residue Control Plan. Health authorities are charged with duties to conduct surveillance on residue levels in honey for local consumption, and issue health certificate for honey to be exported upon satisfactory testing results were obtained.

However, the determination of residues in honey needs validated analytical methods capable of meeting international requirements. Metrological tools like CRMs are normally required to calibrate measuring instruments and methods of measurement. To this end, testing laboratories under the health authorities employ CRMs on pesticides and antibiotics in honey matrix produced jointly by CENAM, the national metrology institute of Mexico, and national reference laboratories. These CRMs are used to validate analytical methods and serve as calibrants for the control of the measuring system.

6.2.2. Cadmium in rice

There is a world-wide safety concern on heavy metal contamination, such as cadmium, in foods. As rice is the staple diet in many countries, Codex/WHO set a maximum level of cadmium in rice as 0.2 mg/kg. To facilitate international trade on rice, exporters are required to supply analytical data proving that the Codex limit was not

exceeded. Also, the analytical results have to be metrologically traceable to a stated reference in order to be accepted. These requirements warrant the use of CRMs for method validation, quality control of measuring procedures and establishing the metrological traceability for the analytical results.

In China, a series of CRMs on cadmium in rice flour is available from the National Institute of Metrology (NIM). Testing laboratories can use them to validate their analytical methods and effect the control of their measuring system. To demonstrate the metrological traceability of the certified values of the CRMs, NIM had established the degree of equivalence for the analytical methodology they used with other national metrology institutes through the participation of inter-comparison programmes organized by CCQM.

In Japan, the food sanitary law stated that rice is not allowed to be on sale if the cadmium level exceeded 0.4 mg/kg. The CRMs available in Japan are mainly used as calibrants for nuclear absorbance method. For the effective metrological control of the quality of rice, the government stipulated that production of these CRMs has to follow the relevant international standards such as ISO Guide 34 and ISO/IEC 17025.

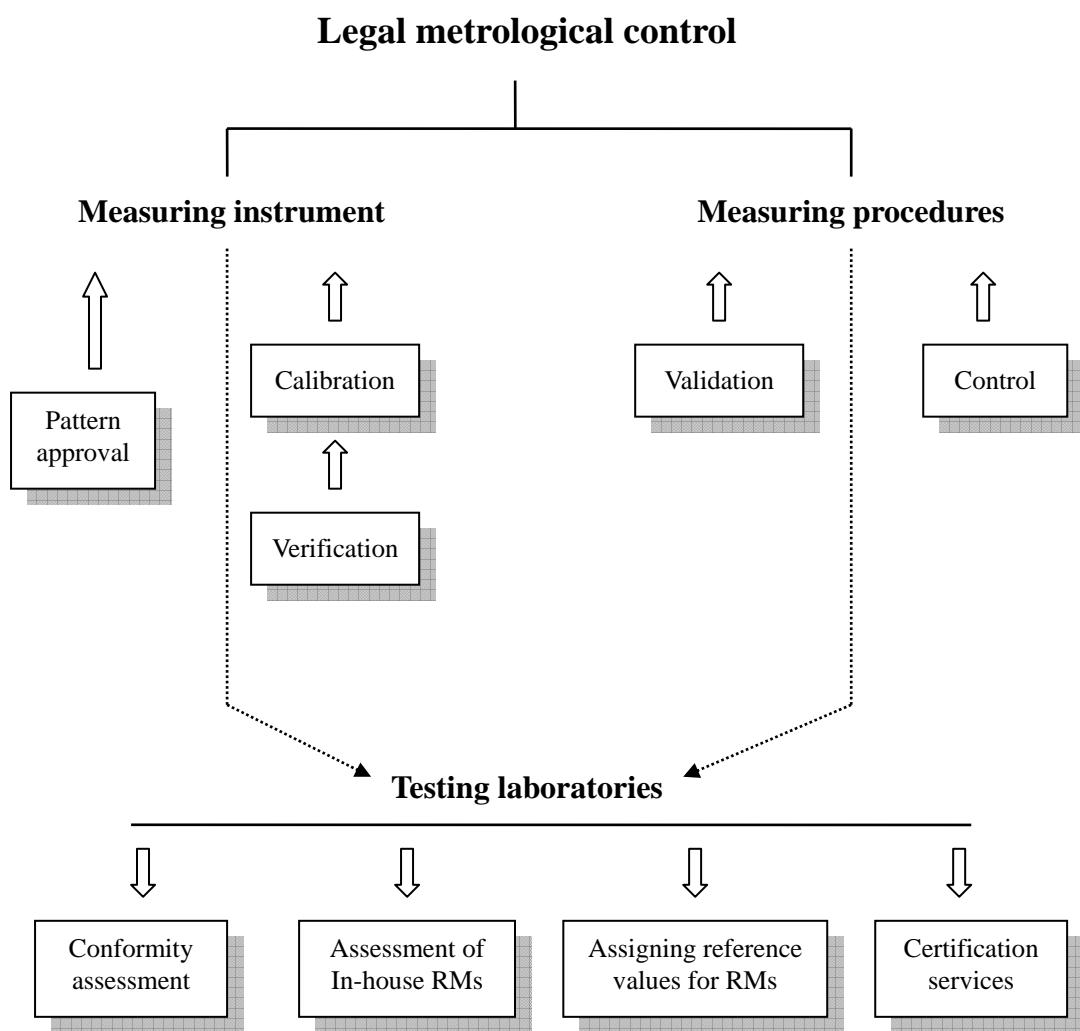


Figure 1 : Procedures under the legal metrology control that require the use of CRMs

Annex A

Bibliography

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- A.10 ISO Guide 33:2000, *Uses of certified reference materials*
- A.11 ISO Guide 34:2000, *General requirements for the competence of reference material producers*
- A.12 ISO Guide 35:2006, *Certification of reference materials – general and statistical principles*