

Certified Reference Materials

Certified Reference Materials (CRMs) are controls or standards used to check the quality and traceability of products, to validate analytical measurement methods, or for the calibration of instruments. A certified reference material is a particular form of measurement standard.

Reference materials are particularly important for analytical chemistry and clinical analysis. Since most analytical instrumentation is comparative, it requires a sample of known composition (reference material) for accurate calibration. These reference materials are produced under stringent manufacturing procedures and differ from laboratory reagents in their certification and the traceability of the data provided.

Quality management systems involving laboratory accreditation under national and international accreditation/certification standards such as ISO/IEC 17025 require traceability to Certified Reference Materials (where possible) when using reference materials for calibration.

Requirements for demonstrating quality in the analytical laboratory

Testing and calibration laboratories, including analytical chemical laboratories, are continually requested to provide evidence on the quality of their operations. This is mandatory in cases where legislative limits are involved, e.g. in international trade, food and environmental analysis, clinical chemistry, etc.

The general ISO definition of “**quality**” is given as “*totality of characteristics of an entity that bears on its ability to satisfy stated and implied needs*”.

For the chemical analytical laboratory, the ‘entity’ will in most cases be a measurement result. In a simplified form the quality requirements would then be represented in the form of reliable, comparable (traceable) results, accompanied with stated measurement uncertainty, produced in an agreed time.

The best and easiest way for laboratories to formally demonstrate their quality is to adhere to an appropriate international quality standard and obtain formal accreditation/certification. Various international or national standards have been prepared, e.g. ISO 9000 series of standard, GLP, etc. However, basic quality requirements do not differ significantly. Due to a wide range of activities to which it can be applied and due to the well-established quality assessment structure, the ISO 17025 ‘General requirements for competence of testing and calibration laboratories’ is commonly selected as a standard of choice whenever quality assurance in an analytical laboratory is to be demonstrated.

Quality assurance comprises of all those planned and systematic actions undertaken by the organization necessary to provide adequate confidence that a product or service will satisfy given requirements for quality. In other words, quality assurance describes the overall measures that a laboratory uses to ensure the quality of its operations. One of the main technical components of laboratory quality assurance is the proper use of certified reference materials.

Quality control: Under this term we refer to operational techniques and activities that are used to fulfill requirements for quality. In contrast to quality assurance, which is aimed to assure the quality of laboratory operations, quality control is considered as a set of technical operations aimed to assure the reliability of the results for a specific set of samples (or batches of samples). It describes measures that a specific laboratory takes to assure the quality of its results. Operational quality control techniques in the laboratory may include the use of certified reference materials, proficiency tests, round robin analysis, etc. Although all of them support laboratory quality assurance, it has to be respected that they are

complementary activities. It should be noted that quality assurance and quality control activities will overlap in an operational quality system.

Use of CRMs

Certified reference material: *Reference material accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes traceability to an accurate realization to the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence.*

CRMs are generally prepared in batches for which the property values are determined within the stated uncertainty limits by measurements on sample representative for the whole batch.

All CRMs lie within the definition of 'measurement standards'.

Reference material: *Material or substance one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, assessment of a measurement method, or assigning values to materials.*

In a quality system, CRMs and reference materials are sometimes interchangeable. When a CRM is mentioned alone, it might be assumed that the same is valid also for an RM, and vice versa.

Matrix (or compositional) reference materials: *A "natural" substance more representative of laboratory samples that has been chemically characterized for one or more elements, constituents, etc. with a known uncertainty.*

Matrix reference materials are a specific type of reference materials. Within matrix reference materials different sub-groups exist, i.e. fertilizer RMs, environmental and biological matrix RMs, coal RMs, etc.

Further information to the extent of differences in types of reference materials, and basic guidance on the use of CRMs is given in the ISO Guide 33 'Uses of certified reference materials'.

Producers of CRMs also need to provide all the necessary information in the accompanying certificates or reports. However, before any reference material is selected and applied in the laboratory, it is the user's responsibility to become aware of the material's characteristics, advantages and limitations.

Particularly important are the instructions for the use of the CRM as stated in the certificate. The certified values only apply if the material is strictly used according to these instructions. The user needs to follow closely the recommendations given for storage of the material, eventual drying procedures, and observe the indicated shelf life of the RM.

Besides well established (robust) property values — in analytical chemistry this would normally be a concentration, mass fraction, activity concentration, etc. — the most significant advantage of matrix reference material, when available and correctly selected, is its matrix and measured (analyte) level match in comparison with the test material (sample). In addition, reference materials are normally well characterized for a large number of measurements (analytes) and also in respect to homogeneity of the material. This information is very useful in method development and method validation, providing a basis for estimation of accuracy and precision, as well as for the study of other statistical parameters, such as repeatability, reproducibility, linear range, limit of quantification, robustness and evaluation of eventual interferences. The use of validated — fit for purpose — analytical methods is a prerequisite for any laboratory which would like to claim and formally demonstrate its quality and provide confidence in its measurement results. For almost all of the quality control activities mentioned above, use of CRMs is the most appropriate choice.



LQSi offers Phosphate Fertilizer Certified Reference Material

The Association of Fertilizer and Phosphate Chemists (AFPC) realized that there was a need for phosphate fertilizer certified (matrix) reference materials (CRMs) within the fertilizer industry.

A program was established by AFPC with the goal of generating readily available and low-cost certified reference materials (CRMs) of finished phosphate fertilizers. These CRMs would be used to certify the test parameters typical of commercial transactions within the fertilizer industry.

AFPC recognized that Laboratory Quality Services International (LQSi) was a ready and able partner for the development of these phosphate fertilizer CRMs.

During 2012, AFPC and LQSi began development of a MAP CRM, the first in what is to be a series of phosphate fertilizer CRMs that will cover the range of typically-traded phosphate fertilizers.

LQCRM MAP 11-52 is a Monoammonium Phosphate Fertilizer that has been air dried and milled to pass a 425 µm (No. 40, USA Standard Sieve Series; 35 mesh, Tyler Sieve Series) sieve, homogenized, divided into packets weighing between 71 and 129 g, with an average mass of 103 g, and heat-sealed in Mylar-foil packets (polyester film laminated to aluminum foil, which provides an excellent barrier to oxygen and to moisture, as well as high puncture resistance).

LQCRM MAP 11-52 is a certified reference material that is intended for use in ensuring reliable measurement whenever calibrating instruments and assessing techniques used in the determination of those analytes in monoammonium phosphate fertilizers and materials with a similar matrix for which this **LQCRM** is certified. Certificate available upon request.

Additionally available is a Complex Phosphate Fertilizer **LQCRM MAPSzn 12-41** (Monoammonium Phosphate plus Sulphur & Zinc), and currently under development a Feed Grade Phosphate CRM.

In the future, LQSi will also develop CRMs for non-phosphate fertilizers.

LQSi offers certified reference materials (LQCRMs) that are manufactured and certified according to stringent industry standards as outlined in ISO Guide 34 'General requirements for the competence of reference material producers'.

LQSi is a recognized NIST subcontractor, producing materials subsequently certified by NIST as Standard Reference Materials.

For information on the purchase of these materials, contact:

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**Laboratory Quality Services International (LQSi) is accredited to ISO 17043:2010;
Conformity assessment — General requirements for proficiency testing**