EMRP Call 2012 – Open Excellence, Industry and SI Broader Scope

Selected Research Topic number: **SRT-s16**
Version: 1.0

Title: Metrology for organic chemical calibrators

Abstract

There are increasing technological and legislative requirements for the provision of SI traceable chemical measurements across a broad range of activities and sectors, and in particular for a growing number of high priority organic measurands. For most quantitative measurements the pinnacle of the traceability chain is an organic pure substance CRM used as a primary calibrator. Current approaches struggle to provide the range and number of primary organic calibrants required. The uncertainty associated with pure substance reference materials can be a significant contributor to the overall uncertainty in trace analysis. This SRT calls for the research needed to develop accurate and accessible methods for the dissemination of a broad range of organic calibrants based on quantitative nuclear magnetic resonance (QNMR) methods, as well as validated procedures for the value assignment of primary calibrants and their use in the calibration of such methods and the extension of current methods to address the growing requirements for the purity assessment of biopolymers.

Conformity with the Work Programme

This Call for JRPs conforms to the EMRP Outline 2008, section on “Grand Challenges” related to Industry & Fundamental Metrology on pages 9 and 13.

Keywords

Primary methods, amount of substance, purity, metrology in chemistry, quantitative NMR, dissemination of traceability

Background to the Metrological Challenges

The Organic Analysis Working Group (OAWG) of CCQM has continually stressed the importance of the purity assessment of all calibrants used for high accuracy measurements to ensure traceability of results. In most priority areas the contribution of the pure primary calibrators is a significant component of the overall measurement uncertainty for matrix materials and is essential in maintaining traceability to the SI. Therefore there is a real need for improving the breadth and capacity of the current EU capability of providing SI traceable primary calibrators.

The purity of an organic substance can be defined as the mass fraction of the substance under investigation in a given sample. However, this cannot be determined directly and traceability of the purity value of an organic substance is complex. Traditionally, organic purity values are determined using analytical techniques such as high performance liquid chromatography (HPLC), gas chromatography (GC) or differential scanning calorimetry (DSC) and subsequent correction for the key purity parameters of water content, inorganic residue and residual solvents. However, HPLC, GC and DSC are secondary methods and are not directly traceable to SI units unless a pure CRM of the measurand is used. A number of direct approaches have been investigated by NMIs. These have included qNMR and semi-preparative HPLC approach based upon the recovery of the pure fraction of the measurand (mass) from an injected sample (mass).

A gap exists between the capacities of NMIs to produce the range of materials, at the quantities required, for an acceptable cost that would enable CRM producers meet the current needs of industry. Investigation of more direct methods for characterising purity and production of a range of primary standards is required.

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Scientific and Technological Objectives

Proposers should address the objectives stated below, which are based on the PRT submissions. Proposers may identify amendments to the objectives or choose to address a subset of them in order to maximise the overall impact, or address budgetary or scientific / technical constraints, but the reasons for this should be clearly stated in the JRP-Protocol.

The JRP shall focus on developing accurate and accessible methods for the dissemination of a broad range of organic calibrants based on quantitative nuclear magnetic resonance (qNMR) methods. It should extend current methods in order to address the growing requirements for the purity assessment of biopolymers and to develop capability for the assessment of chemical purity with low uncertainty.

The specific objectives are:

1. To investigate direct methods and direct approaches (including qNMR), capable of providing traceable mole/mass fraction values of a pure substance including fundamental evaluation of the measurement process, assessment of appropriate calibration methods, provision of suitable calibration standards and a systematic assessment of the uncertainty contributions
2. To include the potential use of direct approaches by secondary suppliers, ensuring traceability of a much broader range of materials
3. To include the analysis of molecules with increased molecular mass (>500 g/mol) and the provision of sequence and purity information of biopolymers

These objectives will require large-scale approaches that are beyond the capabilities of single National Metrology Institutes and Designated Institutes. To enhance the impact of the research work, the involvement of the larger community of metrology R&D resources outside Europe is recommended. A strong industry involvement is expected in order to align the project with their needs and guarantee an efficient knowledge transfer into industry.

Proposers should establish the current state of the art, and explain how their proposed project goes beyond this.

The total eligible cost of any proposal received for this SRT is expected to be around the 2.7 M€ guideline for proposals in this call. The available budget for integral Research Excellence Grants is 42 months of effort.

Potential Impact

Proposals must demonstrate adequate and appropriate participation/links to the “end user” community. This may be through the inclusion of unfunded JRP partners or collaborators, or by including links to industrial/policy advisory committees, standards committees or other bodies. Evidence of support from the “end user” community (e.g. letters of support) is encouraged.

You should detail how your JRP results are going to:

- feed into the development of urgent documentary standards through appropriate standards bodies
- transfer knowledge to the chemistry sector.

You should detail other impacts of your proposed JRP as detailed in the document “Guide 4: Writing a Joint Research Project”

You should also detail how your approach to realising the objectives will further the aim of the EMRP to develop a coherent approach at the European level in the field of metrology and includes the best available contributions from across the metrology community. Specifically the opportunities for:

- improvement of the efficiency of use of available resources to better meet metrological needs and to assure the traceability of national standards
- the metrology capacity of Member States and countries associated with the Seventh Framework Programme whose metrology programmes are at an early stage of development to be increased
- outside researchers & research organisations other than NMIs and DIs to be involved in the work

Time-scale

The project should be of up to 3 years duration.