Title: Metrology for humidity at high temperatures and transient conditions

Abstract

The quality of humidity measurements directly affects final products and efficient use of material and energy in industrial drying processes, being relevant to about 80 % of all product manufacturing. Significant impact in European industry is foreseen by developing in-line water activity (equilibrium relative humidity) measurement methods and humidity calibration methods and procedures for temperatures above 100 °C and under transient conditions. The identified industrial challenges will be solved in partnership with industry.

Keywords

Humidity, water activity, calibration, high temperature, drying, pharmaceutics, food, paper manufacturing

Background to the Metrological Challenges

Humidity is a key parameter in controlling drying processes and ambient conditions in various industrial processes. The quality of humidity measurements directly affects the productivity through energy and material efficiency. For example:

In industrial dryers, humidity measurements are performed from exhaust air/gas at high temperatures. Ideally, higher inlet air temperatures reduce the process time but require a careful and fast in-line temperature-humidity vs. time control for early detection of end-point in order to insure a high starch yield as well to contrast the unwanted cellulose yield. Humidity calibrations are usually not performed at temperatures above 100 °C and the calibration equipment commercially available for industry cannot be operated in this range [www.directindustry.com].

Reliable humidity monitoring of ambient conditions in manufacturing is vital in the pharmaceutical industry. Therefore, extensive on-site calibration activities are continuously carried out in drug production premises. Calibrations of hygrometers are time consuming but essential for quality control. Because of the long stabilisation time needed for each measurement point and potentially significant hysteresis, the overall time needed for a humidity calibration is significantly longer than for other common measurement parameters. Currently, humidity calibrations are performed at discrete measurement points, each in steady-state conditions. On-site calibrations are usually carried out by saturated salt solutions or simple dynamic calibrators based on flow mixing. In industrial laboratories, more advanced calibrators or calibration systems are used. Many research results have been published on these methods but calibration in transient conditions have not been investigated, Test methods applied by manufacturers of humidity instruments include also response time tests but methods or procedures for dynamic calibrations providing SI traceability have not been developed.

An important part of quality assurance in pilot and production phase of drug manufacturing is precise ambient control of sterile microbial processes. Specific microbial transient processes during incubation phases of microbial testing seemingly accumulate high humidity within a small confined environment. Additionally, significant environmental shocks to bacteria from the transient exposure of the sample to different temperatures of agar may occur. Drug manufacturing industry relies on well validated procedures. In specific microbial cases, current state-of-the-art for ambient control at small confined environments seems insufficient, which leads to the unacceptable risk of false microbiological analysis. Modelling has widely been applied to study thermal and mass transfer in various applications but further development is needed to obtain tools meeting the metrological needs of humidity control in transient conditions. The effect of transient conditions on the performance of the humidity instrument during the measurements is not usually taken into account in the uncertainty analysis. No guidance is published on this.

Water activity and water mass fraction are important in the quality control of food production. The relative humidity of air in equilibrium with a food sample is measured as a proxy for bulk moisture content. In order to preserve food nutritional quality and increase the shelf life, industry aims at minimal processing and consequently requires accurate inline detection of water activity. In-line water activity measurements, however, are very challenging because conditions are hardly ever stable and equilibrium cannot be reached between material and air. At the moment, all real time in-line measurements are performed indirectly; only at-line or near-line measurement solutions have been implemented. Some studies have been made on in-line water activity measurements but so far nobody can reliably measure water activity in-line. Water mass fraction is linked to water activity via sorption isotherms which are highly material specific. Information on the metrological quality of these isotherms is insufficient or missing. Appropriate methods and guidance are needed for establishing a traceability link between the two parameters.

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 proposal.

The JRP shall focus on the traceable measurement and characterisation of humidity at high temperatures and/or transient conditions.

The specific objectives are

1. To develop humidity calibration methods and procedures for air temperatures above 100 °C. The uncertainty levels appropriate for relevant applications should be achieved with the minimum additional work load and equipment costs.
2. To develop humidity calibration methods and procedures applicable to transient conditions. The uncertainty of calibration should be the same as when performed under steady state conditions (that is below 2% rh) but the time needed for the calibration should be reduced by at least 50 %.
3. To develop humidity measurement techniques and procedures for the accurate monitoring of temporal and spatial humidity variations in selected applications.
4. To develop water activity measurement (equilibrium relative humidity) techniques for in-line measurement applications (with a measurement uncertainty smaller than 0.02) and to develop methods for establishing the traceability link between water activity and water mass fraction measurements. Tools for analysing error sources in water activity measurements which are an integral part of sorption isotherm measurements should be developed.
5. To validate all of the methods developed in this project in selected industrial applications to facilitate the take up of the technology and measurement infrastructure developed by the project and to support the development of new, innovative products or the efficient use of material and energy, thereby enhancing the competitiveness of EU industry.

Proposers shall give priority to work that meets documented industrial needs and include measures to support transfer into industry by cooperation and by standardisation. An active involvement of industrial stakeholders is expected in order to align the project with their needs – both through project steering boards and participation in the research activities.

Proposers should establish the current state of the art, and explain how their proposed project goes beyond this. In particular, proposers should outline the achievements of the EMRP project SIB64 METefnet and how their proposal will build on those.

EURAMET expects the average EU Contribution for the selected JRPs to be 1.5 M€, and has defined an upper limit of 1.8 M€ for any project.

EURAMET also expects the EU Contribution to the external funded partners to not exceed 30 % of the total EU Contribution to the project. Any deviation from this must be justified.

Any industrial partners that will receive significant benefit from the results of the proposed project are expected to be unfunded partners.

Potential Impact

Proposals must demonstrate adequate and appropriate participation/links to the “end user” community, describing how the project partners will engage with relevant communities during the project to facilitate knowledge transfer and accelerate the uptake of project outputs. Evidence of support from the “end user” community (e.g. letters of support) is also encouraged.

You should detail how your JRP results are going to:

- Address the SRT objectives and deliver solutions to the documented needs,
- Drive innovation in industrial production and facilitate new or significantly improved products through exploiting top-level metrological technology,
- Improve the competitiveness of EU industry,
- Feed into the development of urgent documentary standards through appropriate standards bodies,
- Transfer knowledge to the manufacturing sector.

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

You should also detail how your approach to realising the objectives will further the aim of EMPIR to develop a coherent approach at the European level in the field of metrology and include 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 EURAMET Member States whose metrology programmes are at an early stage of development to be increased
- organisations other than NMIs and DIs to be involved in the work

Time-scale

The project should be of up to 3 years duration.