Publishable Summary for 14IND11 HIT
Metrology for Humidity at High Temperatures and Transient Conditions

Overview

The aim of this project is to significantly improve the accuracy of industrial humidity measurements at high temperatures up to 180 °C and under transient conditions by developing improved humidity measurement and calibration techniques.

Need

Drying is estimated to cost European industry around 30 000 M€ per year in associated energy costs. Every 0.1 % improvement in drying efficiency due to better process control could save around 30 M€/year. Monitoring humidity under transient conditions and at temperatures above 100 °C is a key factor in controlling drying processes. Thus, by improving the reliability of these humidity measurements annual savings of millions of euros can be achieved in Europe.

The quality and shelf life of *pharmaceutical products* are significantly affected by the humidity of the ambient air during manufacture and storage. Pharmaceutical companies allocate significant resources in order to maintain high quality monitoring of humidity in their production premises and storage facilities. These companies are therefore seeking more efficient calibration methods for humidity sensors and improved methods for spatial humidity monitoring under transient conditions to achieve savings both in costs and materials.

The *food industry* is the second largest manufacturing sector in the EU with a total manufacturing turnover of over 900 billion euros. Drying and baking are key processes in this sector, and water activity – i.e. equilibrium relative humidity – is a key parameter in controlling the quality of food and feed products. In order to determine water activity, material samples are taken from the process and measured using laboratory analysers. A significant quantity of material is wasted because of slow feedback, and the optimisation of energy consumption is limited by the larger safety margins that are required due to uncertainties which are increased by sampling errors and transient conditions.

More than 180 accredited *industrial laboratories* in Europe calibrate hygrometers for their customers but none of them can perform calibrations at temperatures above 100 °C or under transient conditions because appropriate methods and techniques are not available. Furthermore, there is no measurement technology available for monitoring fast transients in humidity in the temperature range above 100 °C. Dynamic humidity measurements are an integral part of the *environmental tests* for various industrial products however no proper methods exist to estimate their uncertainty.

Objectives

The main objectives are:

- **To develop humidity calibration methods and procedures for industrial use for air temperatures above 100 °C and absolute pressures from 0.5 bar to 6 bar.** The uncertainty levels appropriate for relevant applications (typically less than 2 % relative humidity) will be achieved with the minimum additional work load and equipment costs.
- **To develop humidity calibration methods and procedures for industrial use applicable to transient conditions,** including development of a prototype field humidity calibrator. The target is for the uncertainty of the calibration to be the same as when performed under steady state conditions (i.e. better than 2 % relative humidity) but for the time needed for the industrial calibration to be reduced by at least 50 % from up to one day.

- **To develop humidity measurement techniques and procedures for the accurate monitoring of temporal and spatial humidity variations in selected applications**, including development of a new type of hygrometer based on direct Tuneable Diode Laser Absorption Spectroscopy (dTDLAS) for process environments with temperatures up to 180 °C and a new measurement method for detecting the influence of microbiological processes on the transient humidity conditions within small samples. The target relative uncertainty for the water vapour amount fraction at high temperatures is 5 %, and the uncertainty for the relative humidity measurements will be less than 2 %rh.
- **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** including the development of **t**ools for analysing error sources in water activity measurements which are an integral part of sorption isotherm measurements.

Progress beyond the state of the art

Humidity calibration methods for temperatures above 100 °C

Although humidity measurements are made in processes significantly above 100 °C and humidity sensors are specified for these conditions, 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. Extending the temperature range of the calibration systems used by industrial calibration laboratories is possible but research is needed to reach the required uncertainty level of 2 %rh. The project has developed the first primary standard system for humidity at temperatures up to 180 °C and pressures up to 6.5 bar (abs) and two other new calibration/testing facilities that extend the humidity calibration capabilities of the European metrology institutes to temperatures above 100 °C. These facilities are now used in this project to develop a calibration procedure for industrial use that will ensure adequate traceability for humidity measurements at temperatures up to 180 °C. Tests for several capacitive, acoustic and chilled mirror sensors have been completed.

Humidity calibration methods for transient conditions

Currently, calibrations of industrial humidity sensors are performed at discrete measurement points, each under steady-state conditions. On-site calibrations are usually carried out using saturated salt solutions or simple calibrators based on flow mixing, whilst in industrial laboratories, more advanced calibrators or calibration systems such as a combination of climatic chamber and chamber hygrometer are used. To provide a practical approach for carrying out humidity calibrations at non-static conditions, this project has developed a new modular calibration setup for use in commercial temperature/climatic test chambers. New calibration schemes were developed to provide comprehensive but efficient approaches to humidity calibrations at a single temperature and in a wider temperature range. Several capacitive humidity sensors and chilled mirror hygrometers are being tested to investigate the agreement between calibrations at static and non-static conditions. The uncertainty of the calibrations will be smaller than 2 %rh. A new dynamic humidity control method was developed and tested for a field humidity calibrator. The targeted operating range and calibration uncertainty are 10 %rh to 90 %rh and 2 %rh, respectively. Based on the outcomes of the tests and demonstrations in industry recommendations will be produced for humidity calibration procedures applicable to non-static conditions. It is expected that the new non-static calibration approach will reduce the time needed for a RH calibration by 50 %.

Methods for measurement of temporal and spatial humidity variations

The measurement of water vapour content in industrial processes with rapid humidity changes requires fast, robust and well validated measurements methods. Most commercially available hygrometers do not fulfil all of these requirements. For overcoming many of these drawbacks, a new hygrometer was constructed applying direct Tuneable Diode Laser Absorption Spectroscopy (dTDLAS) The response time of the device is well below 1 s, and it provides a good long-term stability with robustness at high temperature, humidity and pressure in industrial applications. After completing a metrological validation the device willenable traceable measurements of humidity transients at temperatures up to 180 °C.

Humidity measurement methods for in-line water activity measurements

In food and feed industry the common way to determine the water activity (a_w) of a product is to sample some of the product and then to analyse it in a laboratory using a commercial water activity analyser. The method is time consuming and feedback in process control is very slow. However, in-line and on-line measurements are problematic due to transient condition and there is no reliable method available for determining water activity in-line in industry. This project developed a novel method for traceable a_w measurements in-line by combining traceable continuous temperature and sample-based sorption isotherm measurements. A software tool was developed for modelling temperature dependence of isotherms and transient water activity in hazelnuts. A measurement system for the determining the absorption-desorption isotherms was developed and tested. The system provides traceability to SI through traceable mass, temperature and humidity measurements.

Results

- **To develop humidity calibration methods and procedures for industrial use for air temperatures above 100 °C and absolute pressures from 0.5 bar to 6 bar.**

  A high-temperature mass fraction water vapour/steam generator and two new calibration/testing facilities have been developed to provide traceable humidity standards at temperatures up to 180 °C and pressures up to 6.5 bar (abs). Several capacitive and acoustic humidity sensors were tested to determine appropriate calibration procedures for industrial use.

- **To develop humidity calibration methods and procedures for industrial use applicable to transient conditions**

  A new calibration scheme with an appropriate calculation method was developed to provide an efficient approach to perform humidity calibration in a range of temperatures. Another scheme based on linear humidity ramps was designed for calibrations at a single temperature. A modular calibration setup has been developed to provide a practical approach for carrying out humidity calibrations at non-static conditions in commercial temperature/climatic test chambers. Several types of capacitive humidity sensors are being tested with different calibration scheme parameters to analyse the effect of non-static conditions on calibration results. The first prototypes of a field humidity calibrator were designed.

- **To develop humidity measurement techniques and procedures for the accurate monitoring of temporal and spatial humidity variations in selected applications**

  A new type of hygrometer based on direct Tuneable Diode Laser Absorption Spectroscopy (dTDLAS) was developed for process environments with temperatures up to 180 °C. The system was introduced and demonstrated at the HIT Workshop in Braunschweig in November 2017.

  To analyse unwanted high humidity occurrences in transient condition within sterile Petri dish samples stacked in a large climatic sterile room, an experimental setup with precision sensors have been developed and tested. The system will be used to perform root cause analysis of the influence of microbiological processes to the transient humidity conditions within the samples.

- **To develop water activity measurement (equilibrium relative humidity) techniques for in-line measurement applications**

  A new in-line water activity measurement method was developed. The method establishes the traceability link between water activity and water mass fraction measurements through sorption isotherm measurements. A sample-based system for sorption isotherm measurement was developed and tested. The development of a software tool for modelling the temperature dependence of sorption isotherms and transient water activity in hazelnuts was completed. Material dependent modifications are needed when applying this software to other materials.

Impact

This project will deliver new and improved methods and techniques for humidity measurement and monitoring at high temperatures and under transient conditions enabling a wide range of industrial enterprises in the EU to enhance their competitiveness through reduced energy consumption and waste production and through more efficient and reliable quality assurance and new products. The outcomes will

be exploited by the industrial partners, manufacturing industry, industrial test and calibration service providers, instrument manufacturers and the wider stakeholder community.

So far, the project has delivered 2 articles in scientific journals, 9 presentations in conferences, 5 training events and 17 presentations and articles in various meetings and trade/professional magazines. The project partners have liaised with standardisation bodies or equivalent and contributed their work in 16 meetings. 11 companies have invited the project partners to demonstrate the project developments.

The project partners communicated actively with metrological communities through participation to three metrology working groups at European level and two other at global level representing both physical and chemical metrology. Presentations were given in the conferences CIM2017, and ICHMT 2017. The project has 6 collaborators. In ResearchGate, HIT project has 26 followers and 315 reads.

Impact on relevant standards

This project will have an impact on the work of several CEN, IEC, ISO, AFNOR, and DIN standardisation groups together with metrology committees The most authoritative guidance documents related to calibration and traceability are prepared and published by the Joint Committee for Guide in Metrology (JCGM) (which includes BIPM, IEC, IFCC, ILAC, ISO, IUPAC, IUPAP and OIML) and regional metrology organisations, such as EURAMET e.V. in Europe. The quality assurance of measurements is often evaluated in certification (ISO9001:2000; GMP etc.) and accreditation (ISO17025:2005) in Europe using the requirements set in the JCGM and EURAMET guidance documents. Until now there is no European standard or guideline on humidity calibrations available for industry. This project will develop a draft version of a EURAMET Guide cg NN “Calibration of humidity measuring instruments” based on the advances in the project in consultation with EURAMET TC-T WG Best Practice.

Until now, the project consortium has contributed to two standardisation groups of DIN and ISO and five metrology working groups of EURAMET and CIPM.

Impact on industrial and other user communities

A wide range of industrial enterprises in EU will benefit from the outcomes of this project through improved humidity control in drying, baking, storage and testing and through more efficient calibration methods. As a result, the enterprises will be able to improve their productivity through reduced energy consumption and waste production and through more efficient testing and calibration.

The project will have significant direct impact on the application of humidity measurements in industrial process and quality control through new humidity calibration methods and procedures, the new type of transfer standard hygrometer and a field humidity calibrator, new measurement and uncertainty estimation methods in applications with significant temporal and spatial humidity variations and the novel in-line water activity measurement techniques.

The new type of field humidity calibrator developed in the project will significantly reduce the time needed for humidity calibrations on-site by up to 50 %, thus reducing costs and downtime for instruments. Extending calibrations to dynamic humidity measurements will reduce the calibration time e.g. in pharmaceutical companies and in humidity sensor manufacturing and will improve humidity control e.g. in environmental testing.

All the outcomes of this project will directly be exploited by industry.

Active communication with industrial partners, collaborators and other stakeholders has been maintained to ensure efficient two-way exchange of information. This includes face-to-face meetings with 13 companies, contribution to 5training events, presentations in 5 conferences and 10 workshops/seminars, 4 articles in professional magazines, a LinkedIn group, project website (www.empir-hit.eu) and e-Newsletter. The project partners have extended their measurement, consultation and training services for industry in the fields of high temperature humidity measurements and water activity measurements.

Impact on the metrological and scientific communities

As a result of this project for the first time there will be two NMIs in the world providing traceability for humidity measurements at dew-point temperatures up to 150 °C, air temperatures up to 180 °C and air pressures up to 6 bar. This will ensure the availability of NMI level traceability services (i.e. calibration of reference instruments and interlaboratory comparisons) which are vital in enabling industrial calibration laboratories to directly exploit the project outcomes. By applying the calibration methods developed in this project other NMIs/DIs will also be able to efficiently extend their capabilities according to national needs to cover humidity calibrations at temperatures above 100 °C and dynamic humidity measurements. The outcomes of the work on relative humidity calibrations above 100 °C will underpin the task of CCT WG Humidity related to internationally harmonised humidity terms and definitions. The project will provide direct input to EURAMET in the form of recommendations on humidity calibration procedures for high temperatures and under transient conditions and the draft EURAMET guide on the Calibration of humidity measuring instruments.

The advances in estimating uncertainty in water activity measurements and the determination of sorption isotherms will enable food scientists to improve the characterisations of raw and processed food materials.

List of Publications

1. N. Massarotti et al., New benchmark solutions for transient natural convection in partially porous annuli, International Journal of Numerical Methods for Heat & Fluid Flow, Vol. 26 No. 3/4, 2016 pp. 1-40

Project start date and duration:	01 September 2015, 36 months	
Coordinator: Dr Martti Heinonen, VTT, Tel: +358 400 686 553 E-mail: martti.heinonen@vtt.fi Project website address: http://www.empir-hit.eu/		
Internal Funded Partners:	External Funded Partners:	Unfunded Partners:
Partner 1 VTT, Finland Partner 2 CETIAT, France Partner 3 FORCE, Denmark Partner 4 DTI, Denmark Partner 5 INRIM, Italy Partner 6 PTB, Germany Partner 7 UL, Slovenia Partner 8 VSL, Netherlands	Partner 9 CNR, Italy Partner 10 GBV, Italy Partner 11 IH, Netherlands Partner 12 Michell, Netherlands Partner 13 TU-DA, Germany Partner 14 UNICAS, Italy	Partner 15 Vaisala, Finland