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

Overview

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

The project succeeded in developing humidity calibration methods and procedures for industrial use for air temperatures above 100 °C and absolute pressures up to 6 bar with an uncertainty of less than 2 %. Calibration methods for non-static humidity conditions were developed – also with an uncertainty of less than 2 %. Humidity measurement techniques and procedures for the accurate monitoring of temporal and spatial humidity variations were successfully developed and demonstrated in the temperature range up to 100 °C. Above 100 °C, it was not possible to achieve the desired uncertainty of 5 %.

SI traceable in-line water activity measurement (equilibrium relative humidity) techniques, as well as methods for establishing the traceability link between water activity and water mass fraction measurements, were developed. In-line water activity measurements provide a direct feedback to the drying, baking or roasting process. The achieved uncertainty in the water activity measurements was 2 %. The methods developed in this project were demonstrated in relevant industrial applications.

Need

Prior to the start of the project, drying was 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 could 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 were 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, at the end of the project, only one of them was able to perform calibrations at temperatures above 100 °C. In addition, calibration methods for transient humidity conditions were not available. Furthermore, there was 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 existed to estimate their uncertainty.

Objectives

The overall objective of this research was to improve the efficiency of material and energy usage and to improve quality control in industry by developing improved measurement and calibration techniques for humidity at high temperatures and under transient conditions. The main objectives were:

- **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.
- **To validate all of the methods developed in this project through demonstration in selected industrial applications** in order to facilitate the take up of the technology and measurement infrastructure developed by the project and to recommend what further actions are necessary to ensure the uptake.

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 was needed to reach the required uncertainty level of 2 %rh. The project has developed a primary standard system for humidity at temperatures up to 180 °C and pressures up to 6 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. An informal inter-laboratory comparison has demonstrated good agreement within these three institutes. These facilities were used in this project to develop calibration procedures 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 carried out.

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 a climatic chamber and a chamber hygrometer are used. To provide a practical approach for carrying out humidity calibrations at non-static conditions, this project 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 from different manufacturers were tested to investigate the agreement between calibrations at static and non-static conditions. The new non-static calibration approach reduced the time needed for a typical RH calibration by 40 % or more, with multiple measurement points. The uncertainty of the calibrations was around 1 %rh. The non-static calibration schemes were validated against the static ones, and the results were in agreement. A new dynamic humidity control method was developed and tested for a field humidity calibrator. The targeted operating range and calibration uncertainty were 10 %rh to 90 %rh and 2 %rh, respectively. Based on the outcomes of the tests and demonstrations in industry, recommendations for humidity calibration procedures, which were applicable to non-static conditions, were produced.

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 measurement 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 was well below 1 s, and it provided a good long-term stability with robustness at high temperature, humidity and pressure in industrial applications. The method was validated as a comparison with two different humidity generators in the temperature ranges 40 ^{0} C to 100 ^{0} C and 100 ^{0} C to 180 ^{0} C (in 0.6 MPa up to 140 ^{0} C and 140 ^{0} C to 180 ^{0} C in atmospheric pressure). In the temperature range below 100 ^{0} C, the method met its target uncertainty, less than 5 %. In the temperature range above 100 ^{0} C and for larger pressures, the observed deviation was larger. This was due to fitting issues with respect to the spectral reference data.

Humidity measurement methods for in-line water activity measurements

In the 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 determining absorption-desorption isotherms was developed and tested. The system provided traceability to the SI through traceable mass, temperature and humidity measurements.

Method validation and industrial demonstrations

The methods and techniques developed in the project were to be validated by demonstration in selected industrial applications. An acoustic humidity sensor calibrated and characterised at high humidity, temperature and pressure was demonstrated in a dairy manufacturing process in the Netherlands. Calibration methods developed in this project were demonstrated in sensor calibration for a biscuit manufacturing company. Real-time humidity and temperature measurements combined with water activity measurements were demonstrated in whole hazelnut drying process in tunnel drying and in a roasting oven. With the dTDLAS instrument, humidity transients in high temperatures could be measured traceably. Water activity and sorption isotherm measurement and modelling methods were validated in a drying process in a pet-feed factory. A transportable humidity field calibrator developed in the project was demonstrated in a pharmaceutical product plant and with a sensor manufacturer in Finland, as well as in a humidity measurement training course in Denmark. Based on the feedback and suggestions, the commercialisation of the calibrator was considered. In addition, a method to measure local gradients of 1 %rh/cm was developed. The method was demonstrated with an experimental setup of a stack of 6 Petri dishes. The goal was to measure unwanted high humidity occurrences in transient conditions within sterile Petri dish samples stacked in a large climatic sterile room. The system was used to perform root cause analysis of the influence of microbiological processes to the transient humidity conditions within the samples.

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 were developed to provide traceable humidity standards at temperatures up to 180 °C and pressures up to 6 bar (abs). Several capacitive and acoustic humidity sensors were tested to determine appropriate calibration procedures for industrial use. With the developed methods, the uncertainty in relative humidity calibrations was 1 % and 0.13 °C in dew-point temperature. The procedures developed were used as input to a draft guideline “A practical approach for estimating the uncertainty of humidity measurements at high temperatures (up to 180 °C) and under transient conditions in industry”. This objective was fully achieved.

- **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 at several temperatures, in the temperature range from 100 °C to 180 °C. Another scheme based on linear humidity ramps was designed for calibrations at a single temperature. These calibration schemes save 40 % of the calibration time compared with a typical calibration scheme. The schemes were validated with a static method, and the agreement was good. A modular calibration setup was 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 were tested with different calibration scheme parameters to analyse the effect of non-static conditions on calibration results. Three prototypes of a field humidity calibrator were designed and tested. The procedures developed were used as input to a draft guideline “A practical approach for estimating the uncertainty of humidity measurements at high temperatures (up to 180 °C) and under transient conditions in industry”. The typical calibration time saving obtained was 40 % instead of the original aim of at least 50 %, but otherwise this objective was achieved.

- **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. Validation measurements were successfully performed at ambient pressure and temperatures between 40°C to 100°C: The dTDLAS hygrometer showed relative deviations of less than 5 % compared to the reference value. Based on this validation, the dTDLAS hygrometer was subsequently applied at an industrial site within the project. However, the first validation trial in the temperature range 100 ^{0} C to 180 ^{0} C showed a larger deviation than the targeted 5 % at higher pressures. The reason was a mismatch of the line data and the fitting approach that was used. Line data parameters are typically reported for gas mixtures of the measured gas in air. During the validation experiments, the mole fraction and the pressure were increased to a level where those parameters were no longer valid.

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

  Studies were carried out to characterise the behaviour and to estimate the measurement uncertainties of impedance humidity sensors, the effects of direct thermal irradiation, the response times, the heating/purging feature and the response to cyclic and stepwise temperature and humidity changes. Based on these results, a draft guideline “A practical approach for estimating the uncertainty of humidity measurements at high temperatures (up to 180 °C) and under transient conditions in industry” was prepared. It will be published as a EURAMET guide. The target uncertainty of 5 % was not met in the temperature range above 100 ^{0} C, but otherwise this objective was achieved.

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

  A new SI traceable in-line water activity measurement method was developed and validated. 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, tested and launched as a service. The uncertainty of the method was around 2 %.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 will be needed when applying this software to other materials. This objective was fully achieved.

- **To validate all of the methods developed in this project through demonstration in selected industrial applications** in order to facilitate the take up of the technology and measurement infrastructure developed by the project and to recommend what further actions are necessary to ensure the uptake.

  An acoustic humidity sensor calibrated and characterised up to 120 °C, at pressures up to 500 kPa and at dew-point temperatures up to 117 °C, was successfully demonstrated in a dairy manufacturing process in the Netherlands. The water activity measurement was demonstrated in pet-food manufacture and in whole hazelnut drying process monitoring. The measurement of water activity during drying and roasting improves the product quality and thus their shelf life. The field calibrator was demonstrated at a pharmaceutical plant to calibrate their humidity sensors. The importance of sensor calibration at high temperatures using the new methods developed in the project was demonstrated in biscuit production. This objective was fully achieved.

Impact

This project delivered 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 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 are available to be exploited by the industrial partners, manufacturing industry, industrial test and calibration service providers, instrument manufacturers and the wider stakeholder community. To assist the industrial and scientific communities, new SI traceable measurement and calibration services in high dew-point and air temperatures as well as in sorption isotherm and water activity determination have been launched.

The project has already delivered 3 articles published in scientific journals, 11 presentations in conferences, 13 training events and workshops, and 33 presentations and articles in various meetings and trade/professional magazines. 7 abstracts have been submitted to TEMPMEKO 2019 conference, and 6 of them will be published as peer-reviewed scientific articles after the conference. One article for a peer-reviewed scientific journal has been submitted for review, and another is being drafted phase. The project partners have liaised with standardisation bodies or the equivalent and demonstrated their work at 16 meetings. Fourteen companies have invited the partners to demonstrate the project’s developments.

The partners communicated actively with metrological communities through participation in three metrology working groups at European level and two others at global level representing both physical and chemical metrology. Presentations were given in the conferences CIM 2015 and 2017, TEMPMEKO 2016, THERMACOMP 2016, ICHMT 2017, ICWPS 2018 and submitted to TEMPMEKO 2019. The project has 6 collaborators. In ResearchGate, this project has 27 followers and 375 reads.

Impact on industrial and other user communities

A wide range of industrial enterprises in the 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 had 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 significantly reduced the time needed for humidity calibrations on-site by 40 % or more, 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. The field calibrator has been demonstrated to industrial partners. The feedback obtained will be exploited in its future commercialisation attempts.

New services for traceably determining sorption isotherms and water activity at temperatures up to 70 °C have been launched. This supports the food and feed industry in their product quality assurance in a cost-effective manner.

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 14 companies, contributions to 11 training events, 9 conference presentations in 7 conferences and 17 workshops/seminars, 4 articles in professional magazines, a LinkedIn group, project website (www.empir-hit.eu) and e-Newsletter. The 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 metrology and scientific communities

The high-temperature mass fraction water vapour/steam generator and two new calibration/testing facilities developed in this project have extended the reference humidity measurement capabilities of European NMIs to dew-point temperatures up to 150 °C, air temperatures up to 180 °C and air pressures up to 6 bar (abs). One of the facilities has already launched the service, while two others are progressing towards it. This enhances the wider availability of NMI level traceability services (i.e. calibration of reference instruments and inter-laboratory comparisons) which are vital in enabling industrial calibration laboratories.

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, as well as dynamic humidity measurements. The outcomes of the work on relative humidity calibrations above 100 °C underpins the task of CCT WG Humidity related to internationally harmonised humidity terms and definitions. The project provides 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.

As a result of this project, the customer offering at the NMI level has been extended to water activity. New services for SI traceable determination of sorption isotherms and water activity at temperatures up to 70 °C have been launched. This development, combined with the numerical tools in estimating uncertainty in water activity measurements and the determination of sorption isotherms, enables food scientists to improve the characterisations of raw and processed food materials.

Impact on relevant standards

The partners contributed actively to 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 (ISO17025:2017) in Europe using the requirements set in the JCGM and EURAMET guidance documents. Until now there has been no European standard or guideline on humidity calibrations available for industry. This project developed 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.

The consortium has been contributing to two standardisation groups of DIN and ISO and to five metrology working groups of EURAMET and CIPM. Experiments have been carried out and results analysed for formulating recommendations to EURAMET and CIPM and the draft EURAMET guide. The recommendations are about applying the new calibration methods developed in this project.

Longer-term economic, social and environmental impacts

Due to improved humidity calibration methods for temperatures above 100 °C and for non-static conditions, the calibrations can be carried out in conditions that correspond to the actual use of the humidity sensors. More accurate measurements improve product quality in food, pharmaceutical and paper manufacturing, which results in reduced amounts of waste. On the other hand, energy savings can be expected, as the products will no longer be over-dried. Faster and more automatic calibration schemes reduce the downtime of the devices under calibration, but they also make the calibration work more economically sustainable for the accredited laboratories. In-line measurements of water activity applied in the food and feed industry will improve consumer safety by improved product safety and prolonged shelf-life of the food or feed products - in a cost-effective manner.

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. 1187-1285. https://doi.org/10.1108/HFF-11-2015-0464
2. F. Arpino, G. Cortellessa, N. Massarotti, M, Scungio, Two-phase explicit CBS procedure for compressible viscous flow transport in porous materials, Journal of Numerical Methods for Heat & Fluid Flow, Vol. 28 Issue: 2, pp.336-360. https://doi.org/10.1108/HFF-02-2017-0080
3. R. Bosma, R.J. Pouw, W. van Schaik and A. Peruzzi, Climatic chamber for dew-point temperatures up to 150 ^{0} C, Metrologia 55 (2018) 597-608. https://doi.org/10.1088/1681-7575/aacecc

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