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JRP-Coordinator	
Name, title, organisation	Martti Heinonen, Dr., VTT MIKES
Tel:	+358 40 0686 553
Email:	martti.heinonen@vtt.fi
JRP website address	www.metef.net
Other JRP-Partners	
Short name, country	BRML, Romania CETIAT, France CMI, Czech Republic DTI, Denmark INRIM, Italy NPL, United Kingdom TUBITAK, Turkey UL, Slovenia UT, Estonia

REG1-Researcher (associated Home Organisation)	Gino Cortellessa (UNICLAM, Italy)	Start date: 1st June 2013 Duration: 36 months
REG2-Researcher (associated Home Organisation)	Petri Österberg (UOULU, Finland)	Start date: 1st January 2014 Duration: 24 months

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1 Executive Summary

Introduction

Maintaining correct moisture levels during the production of pharmaceuticals, foods and paper is vital for ensuring quality but there is a lack of reliable measurements to determine 'dryness'. Water content may change due to evaporation during processing, transportation or storage. In order to determine the water content of samples, they can be weighed before and after, and the amount of moisture lost calculated. However, the sample may still contain a certain amount of moisture after drying and other volatiles may also have evaporated, and so measurement uncertainty often remains unknown. By developing reference materials and comparing methods, this project established moisture measurements with SI traceability and known measurement uncertainties for the first time. The results will allow improvement of online measurement techniques for moisture and removal of variation in results between different techniques.

The Problem

Active ingredients in pharmaceuticals, carbon-fibre composites, polymers, food powders, paper and biomass are affected by the amount moisture present during processing. Errors and inconsistencies in measurement and control of moisture in industrial processes can lead to decreased process throughput, increased wastage and reduced durability of biomaterials. Increasing drying times to remove moisture can increase energy consumption.

The quality of moisture measurements in solids is assured by reference moisture determinations according to standardised procedures. Usually the amount of moisture is determined as the mass loss measured by weighing a sample before and after drying. However, drying can extract other volatiles as well as the water, and even after drying the sample may contain residual water. Moisture measurements can also be affected by the transport and handling of a sample, meaning that the measurement uncertainty is in many cases unknown.

Moisture measurements are important in many different industrial sectors, many of who have developed their own techniques and measurement ranges. More than 1300 national or international documentary standards are in active use due to the range of available measurement methods, reference methods and even the current definitions for moisture are material or industry specific. This diversity has hampered the development of common reference standards and procedures.

Making reliable moisture measurements in solids is very challenging as it depends on the method chosen, the reference materials used and a knowledge of actual uncertainty determination. Measurements are often not comparable, leading to reduced process speed/throughput and increased wastage. In the process industry, online measurements are often performed using microwave or near-infrared (NIR) techniques to measure and control the moisture content of the products, but the final quality control is made by weighing and calculating the mass loss.

There is a fundamental need to move from method-based standardisation of procedures, towards outcome-based verification of measurement results. The quality of moisture control measurements would be improved with robust calibration traceable to the SI and the use of certified reference materials. This would also enable the development of real time process control based on on-line moisture monitoring.

The Solution

To solve this problem, we developed new methods for unambiguous realisation for moisture in solids, establishing traceability link to these primary realisations and estimating measurement uncertainty. New primary realisations and calibration systems were set up and new certified reference materials developed. Guidelines were prepared for uncertainty estimations. Measurement, calibration and consultancy services for customers were developed by the partner institutes.

Impact

The outcomes of this project opened up a new feasible way to control the quality of moisture measurements in industrial laboratories through appropriate traceability chains. This will enable the development of effective calibration methods for on-line moisture sensors in industry.

The network of national metrology institutes established in this project serves now customers throughout Europe providing:

- reference water mass fraction measurements for different kinds of solid material samples;
- calibration of surface moisture meters;
- certified reference materials for moisture measurements;
- consultancy services on moisture measurements.

The reference measurements have already been provided for several industrial customers to verify and improve their measurements and develop new services. Five metrology institutes from Asia, Africa and South America have compared their measurement capabilities with the facilities developed in this project. Supported by the project outcomes, several laboratories are considering applying for accreditation for moisture measurement. The international metrology community has started preparing guidance for SI traceable moisture measurements.

2 Project context, rationale and objectives

2.1 Context and rationale

Improving energy efficiency has been identified as one of the most effective ways to reduce greenhouse emissions and other pollutants and thus to mitigate climate change. A reduction of 0.1 % in the energy consumed by industrial drying would prevent around 10^5 tonnes of CO_2 emissions per year across Europe. Moisture is a key parameter in controlling drying processes. In various combustion processes, improved moisture control reduces particle emissions and improves the efficiency. Thus, improvements in moisture measurements have potential for large environmental impact.

Pharmaceuticals, carbon-fibre composites, polymers, foodstuffs, paper, biomass and many other solid materials are highly affected by moisture when processed into various products. Errors and inconsistencies in moisture measurement and control in industrial processes lead to decreased process speed/throughput and increased wastage, shortened durability of biomaterials, increased energy consumption in drying, and increased fine particle emissions in biomass combustion. Some 70 % of all producer industries use drying for removal of water or another solvent by evaporation of their products. Yet direct measurements of “dryness” are under-used, due to insufficient reliability and lack of traceability in moisture measurements. In chemical metrology, moisture is critical wherever substance purity is concerned; especially in certified reference materials. In the process industry, in-line measurements are often performed by means of microwave or NIR techniques to measure and control the moisture content of the products, but the final quality control is made by mass loss on drying or titration methods.

Reliable moisture measurements in solids are very challenging. Applied methods are material specific and the results obtained are distorted by ambiguity of the actual measurands defined by applied procedures. More than 1300 national or international documentary standards are in active use because available measurement methods, reference methods and even the current definitions for moisture as a measurand are material specific. Due to method dependency, different reference methods and lack of knowledge of the actual uncertainty, the measurements are not comparable, often leading to heavily decreased process speed/throughput and increased wastage.

This project was set up to enable moving away from method-based standardisation of procedures, towards outcome-based verification of measurement results through meaningful calibrations with traceability to the SI. With improved comparability and quality control of moisture measurements, industry will be able to significantly improve moisture related process control. The feasible SI traceability will enable a leap forward in developing real time process control based on on-line moisture measurements.

2.2 Objectives

This project aimed at removing ambiguities and inconsistencies in moisture measurement and calibration techniques to enable complete SI traceability chains in industry. To achieve this novel, relevant and effective methods of realising and disseminating SI units of moisture were developed with provision of metrology infrastructure for moisture measurements. The objectives of the project were

1. unambiguous realisation methods for moisture in solids in terms of water mass fraction and amount fraction

2. new primary standards for water mass fraction featuring uncertainties better than the existing state-of-the-art for samples of between 25 mg and 400 g
3. effective general principles of SI traceability in the field of moisture measurements
4. new certified reference materials with traceability and improved stability
5. novel transfer standards to enable dissemination of SI traceability with optimal accuracy
6. methods for quantifying and reducing the effect of sample transport and handling
7. a novel calibration facility with SI traceability for surface moisture meters
8. modelling relevant to moisture metrology to include local moisture variations in the uncertainty
9. practical but metrologically sound methods for estimating uncertainty in selected industrial applications
10. a coherent and developed moisture metrology infrastructure in Europe

The work was focused on the following material groups: pharmaceuticals, polymer/plastic, foodstuffs, feed, biomass, wood based material. However, many of the outcomes are suitable for extension to wider classes of materials and applications.

3 Research results

3.1 Effective methods for realising SI of moisture in solid materials

3.1.1 Unambiguous realisation methods for moisture in solids in terms of water mass fraction and amount fraction

In most moisture measurement applications, measurement results are expressed in terms of moisture content. This measurand, however, is ambiguous because it is defined through the Loss-on-Drying method (LoD) that is not water specific. When drying a solid material sample, the measured mass loss does not depend only on the water content but also on the content of other volatile components in the sample. In addition, the measurand is also affected by the degree of binding of water. Because of this ambiguity, there are many standardised variations of the LoD method in use for different materials and applications and the actual measurand may differ from the water mass fraction that is the relevant SI quantity. In some application fields, the coulometric Karl Fischer titration (cKF) is used as a reference method for moisture measurements in terms of water mass fraction or water amount fraction. When applying this method to solid materials, water is often extracted from a sample by heating in a sample oven and the measurement results are affected by variations in water binding.

To create a solid basis for SI traceability in moisture measurements, unambiguous realisation methods were developed in this project for water mass fraction and water amount fraction in solid samples. Our approach is to dry an initially weighed sample by heating in a measurement cell. Gas (air or nitrogen) flows through the cell to a water vapour detection unit. The measurement result is obtained by dividing the total mass of water vapour detected by the initial sample mass. By weighing the sample again after drying and controlling the inlet air humidity, the corresponding moisture content value according to a relevant standardised LoD procedure can be determined. Thus, the link to the standardised reference measurements can be established.

Aligned with this approach, four realisation methods were developed applying different principles of water vapour detection:

- Weighing the water condensed out of gas in a cold trap
- Measuring the dew-point temperature and flow rate of the gas
- Measuring the amount of water vapour in the gas with an electrolytic cell
- Measuring the amount of water vapour in the gas with a coulometric Karl Fischer titrator.

For the first two methods, prototype primary standards were designed, constructed and thoroughly tested. Novel solutions were applied to fully comply with the requirements of realisation and linkage to standardised LoD methods. The realisation methods based on an electrolytic cell and cKF were developed by a thorough characterisation of commercially available equipment. Novel methods for determining the cell factor of an electrolytic cell and the equivalent water mass fraction in a case of incomplete water release were developed to improve the applicability of electrolytic and cKF methods in realisation measurements.

Tests with wood materials demonstrated the effect of non-aqueous volatiles and the capability of our approach to take this effect into account. Also, the capability to study the effect of water binding was demonstrated in studies with varying the oven temperature.

The four different realisation methods provide sufficient redundancy for drawing general conclusions needed for the basis of SI traceability in the field of moisture measurements.

3.1.2 New primary standards for water mass fraction featuring uncertainties better than the existing state-of-the-art for samples of between 25 mg and 400 g

Figure 1 illustrates the principles of operation of the new primary standards developed in this project applying a cold trap and a combination of capacitive humidity sensor and dew-point sensor for water vapour detection.

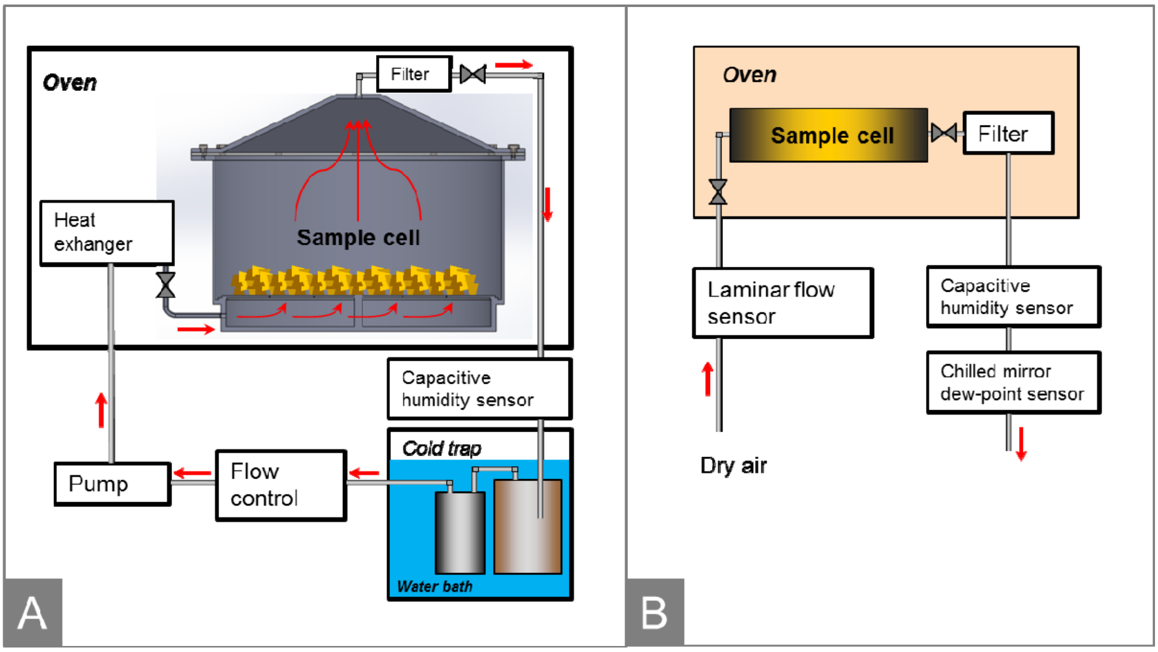

Figure 1. Schematic drawings of the primary standards for water mass fraction developed at VTT MIKES (A) and DTI (B). The balances used for weighing the sample cells are not shown in the drawings.

In the setup developed by VTT MIKES (Fig, 1 A), the water vapour is collected to a cold trap and measured by weighing. A contribution of possibly trapped non-aqueous volatiles is determined by chemical analysis from the trapped water. The system was designed for larger samples (max. 400 g / 9 dm^3). The traceability to SI is obtained directly through traceable mass measurements. The relative expanded uncertainty at the 95 % confidence level is 2 %. A smaller sample cell and a desiccant dryer were developed for samples smaller than 50 g.

In the primary standard of DTI (Fig. 1 B), the total mass of evaporated water is determined by integrating the amount of water vapour in exhaust air over the heating time period. The mass of water vapour is calculated from the measured dew-point temperature, air temperature and air flow rate. The combination of capacitive and chilled mirror sensors enables working in the presence of contaminants that may have evaporated from the sample. The traceability to SI is obtained through traceable measurements of sample mass, air flow, air temperature and dew-point temperature. The relative expanded uncertainty (95 % confidence level) of 1.6 % was demonstrated by an inter-laboratory comparison.

At NPL and UT, the primary standards were obtained by carefully validating a commercial evolved water vapour analyser and O-cKF system, respectively. The sample mass with these systems is a few grams or less. The total amount of water moles evaporated from the sample is determined by electrolysis. This provides the possibility to determine the moisture in terms of water amount fraction in rare cases where total amount of moles of dry sample can be determined. Most often, however, the mass fraction is the primary measurand and determined by weighing the initial mass of a sample. In EWVA, the amount of water moles is detected by electrolytic current in highly hygroscopic P_2O_5. In O-cKF, the amount of water moles is calculated from the electric current used for I_2 production in the titrator cell. To take into account the non-ideal efficiency and drift of the electrolytic cell when operating with EWVA as a primary standard, the cell is calibrated against a dew-point temperature standard using a stable humid gas source with well controlled flow rate (see Figure 2). Thus, the traceability to SI is obtained with this EWVA through traceable measurements of sample mass, flow rate, temperature and dew-point temperature. With the O-cKF, the traceability is obtained through gravimetrically prepared methanol-water mixture. The relative expanded uncertainty (95 % confidence level) of 1.8 % was demonstrated by an inter-laboratory comparison. The parameters affecting the uncertainty with O-cKF were jointly studied by UT and BRLM.

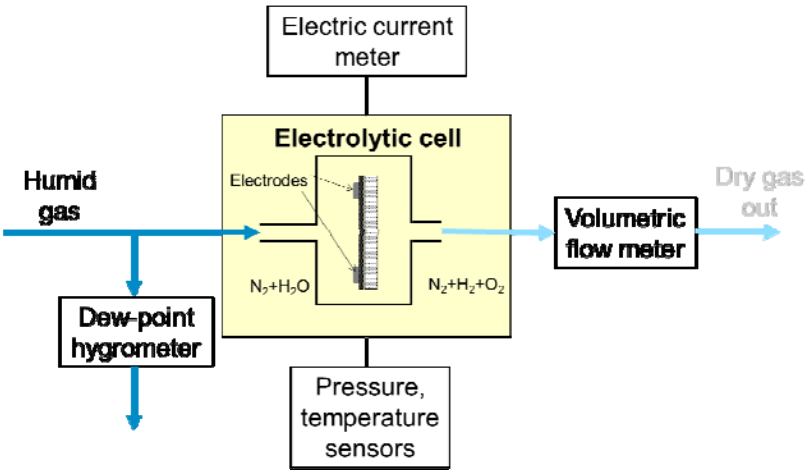

Figure 2. Novel approach for demonstrating SI traceability of an evolved water vapour analyser through humidity standards developed at NPL.

3.1.3 Effective general principles of SI traceability in the field of moisture measurements

The general validity of the developed realisation methods were probed by comparing the developed primary standards to each other and to reference methods at other institutes. These comparisons provided valuable new information about the equivalence between techniques applied by laboratories not only in Europe but also in Asia, Africa and South America.

Three inter-laboratory comparisons were carried out with polymer, wood pellet and powder samples ranging water mass fractions from 2 g/kg to 400 g/kg, i.e. 0.2 % to 40 %. In addition to the project partners BRML, DTI, INRIM, NPL and VTT MIKES and UT, the comparisons were joined by Instituto Nacional de Tecnologia Industrial in Argentina, National Institute of Metrology in Thailand, National Institute for Standards in Egypt and Ural Research Institute for Metrology in Russia. As it can be seen in Figures 3 to 7, the measurement results obtained with different materials and different water mass fractions are in a good agreement, indicating a good equivalence of the techniques. The numbers after the instrument types on the x-axes indicate that each result was derived with different measurement setup.

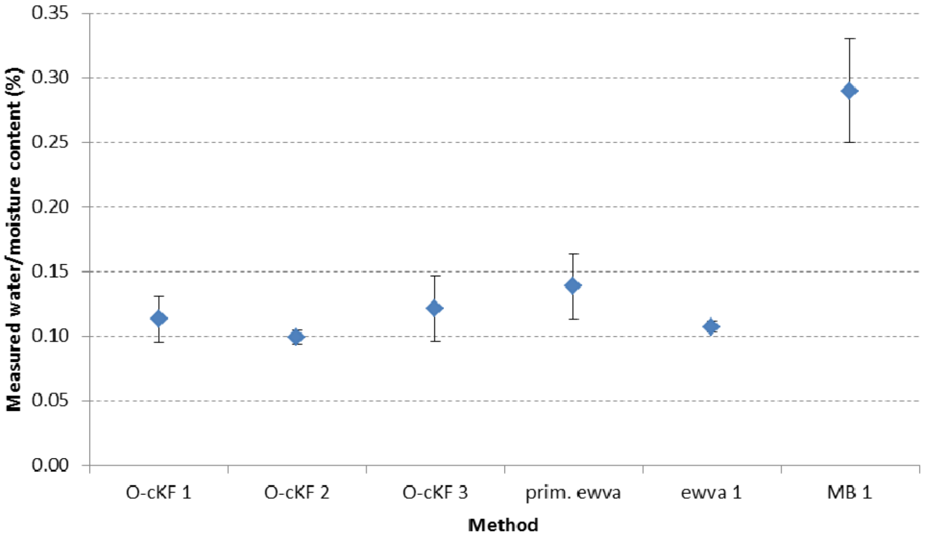

Figure 3. Results obtained with different methods in the inter-laboratory comparison with polycaprolactone polymer. Each result was derived with different measurement equipment. O-cKF = coulometric Karl Fischer titrator with oven, ewva = evolved water vapour analyser, MB = moisture balance. The primary standard is identified with 'prim'. Error bars show estimated uncertainties at about 95 % confidence level.

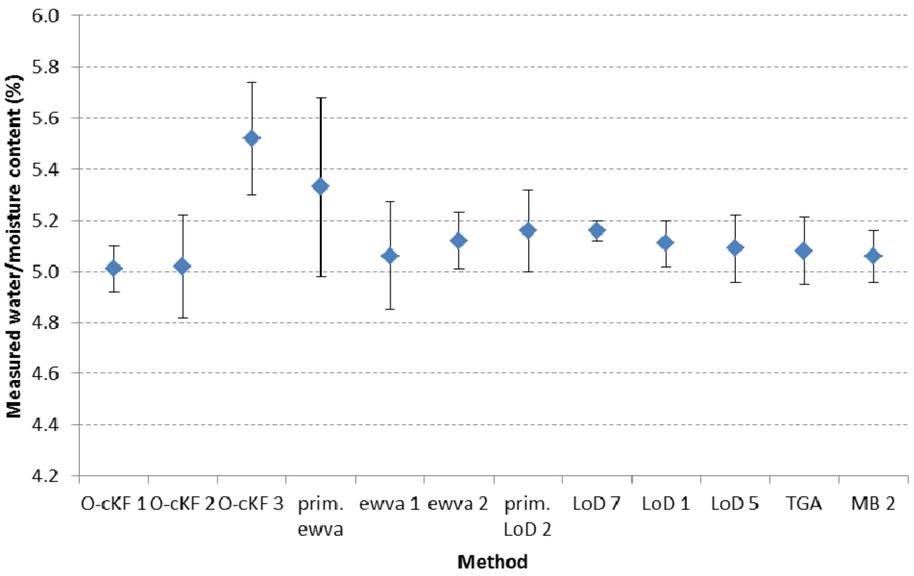

Figure 4. Results obtained with different methods in the inter-laboratory comparison with α-D-lactose monohydrate. Each result was derived with different measurement equipment. O-cKF = coulometric Karl Fischer titrator with oven, ewva = evolved water vapour analyser, LoD = oven drying system, MB = moisture balance. The primary standards are identified with 'prim'. Error bars show estimated uncertainties at about 95 % confidence level.

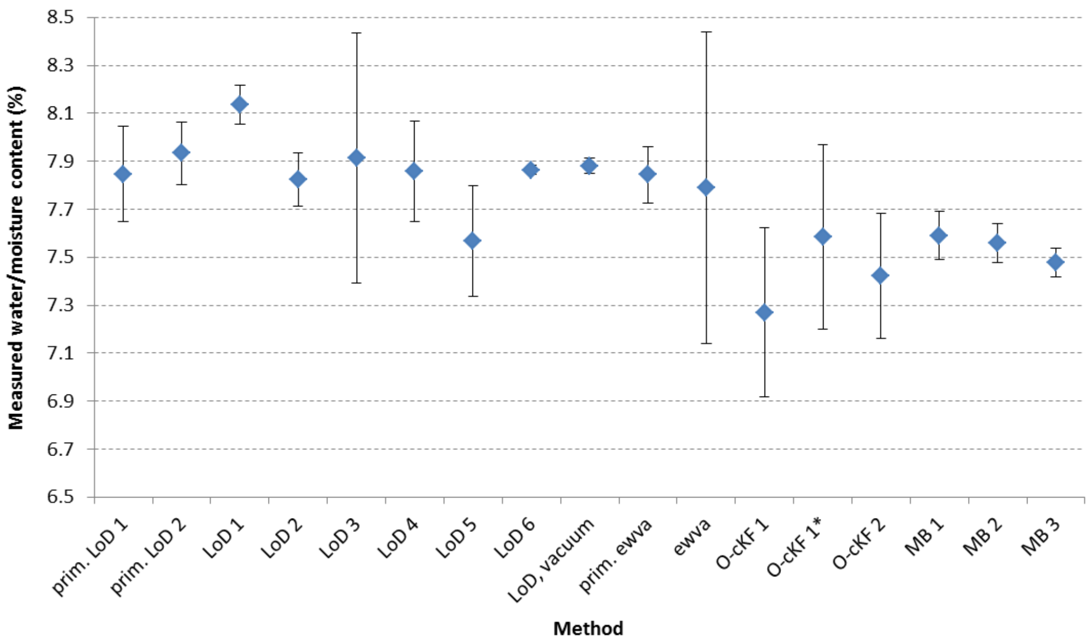

Figure 5. Results obtained with different methods and instruments in the inter-laboratory comparison with wood pellets. O-cKF = coulometric Karl Fischer titrator with oven, ewva = evolved water vapour analyser, LoD = oven drying system, MB = moisture balance. The primary standards are identified with 'prim'. Error bars show estimated uncertainties at about 95 % confidence level.

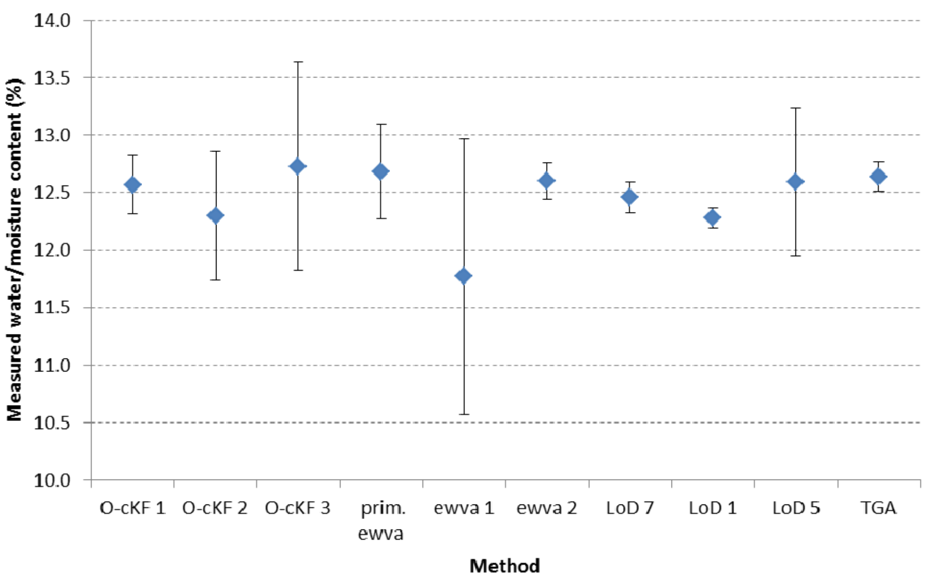

Figure 6. Results obtained with different methods in the inter-laboratory comparison with calcium oxalate monohydrate. Each result was derived with different measurement equipment. O-cKF = coulometric Karl Fischer titrator with oven, ewva = evolved water vapour analyser, LoD = oven drying system, TGA = thermogravimetric analyser, MB = moisture balance. The primary standards developed in this project are identified with 'prim'. Error bars show estimated uncertainties at about 95 % confidence level.

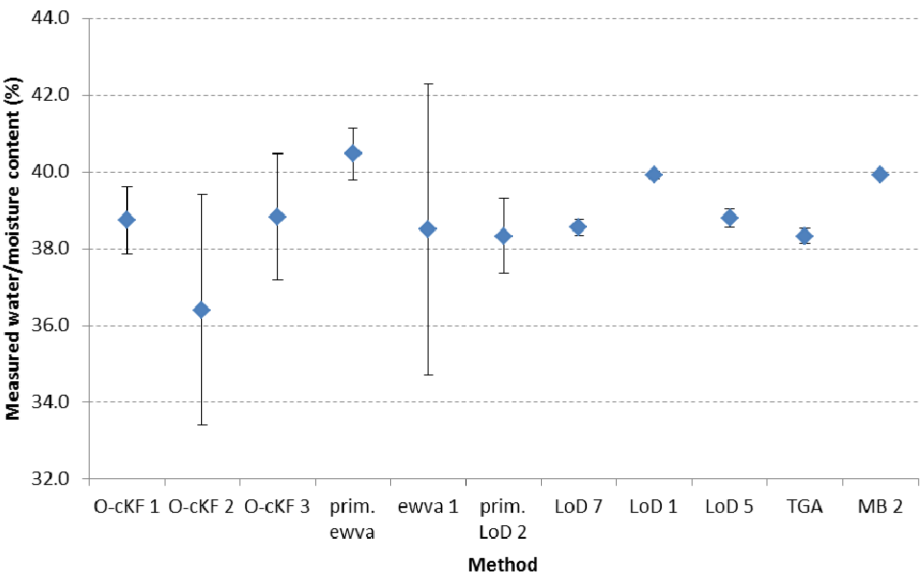

Figure 7. Results obtained with different methods in the inter-laboratory comparison with sodium succinate hexahydrate. Each result was derived with different measurement equipment. O-cKF = coulometric Karl Fischer titrator with oven, ewva = evolved water vapour analyser, LoD = oven drying system, TGA = thermogravimetric analyser, MB = moisture balance. The primary standards developed in this project are identified with 'prim'. Error bars show estimated uncertainties at about 95 % confidence level.

The comparison measurements with wood pellets revealed that O-cKF tends to show smaller water mass fraction indicating incomplete water release due to a short heating time. The results also show that the method developed at UT for determining the equivalent water mass fraction by measuring at several oven temperatures and modelling water release and decomposition in the sample produces results that agree within the estimated uncertainties (see result O-cKF 1* in Figure 5).

On the basis of the developments and comparison results of this project, effective general principles of SI traceability in the field of moisture measurements were outlined in cooperation between the partners and proposed to EURAMET and CIPM. It is recommended using a measurand of three components: analyte, matrix and quantity. Also, sample drying conditions should be reported with measurement results. In SI traceable measurements it is recommended using water as the analyte and mass fraction or amount of substance concentration as the quantity. The best way to avoid confusions when expressing results with uncertainties is to express the results as a ratio of two units of the same kind (e.g. g/kg, mmol/mol etc.).

3.2 Effective methods for disseminating SI traceability in moisture measurements

The matrix dependency of moisture measurement methods and appropriate uncertainty estimation are most challenging in disseminating traceability in the field of moisture measurements. To provide improved tools for this we developed new certified reference materials (CRM), transfer standards, calibration method for surface moisture meters and methods for estimating the measurement uncertainty.

3.2.1 New certified reference materials with traceability and improved stability

Until now only a few CRMs have been available for water mass fraction measurements and they are very limited in type and applicability. In particular, the available range of water mass fraction is highly limited (often focused towards the dry end).

In this project, several candidate materials available in larger sample sizes and with low price were studied with a focus on the stability, homogeneity, commutability (between measurement methods), stability after temperature change and lifespan. Based on these studies three of the materials (α-D-lactose monohydrate, calcium oxalate monohydrate and sodium succinate hexahydrate) were selected for certification and studied in one of the inter-laboratory comparisons. The results show that these materials offer reference values of water mass fraction with features rarely – if ever – available before in a water CRM: they provide large sample sizes (up to several hundred grams), they range up to nominally 400 g/kg (40 %), and two of the three have very good stability. Certificates have now been produced for them by NPL.

3.2.2 Novel transfer standards to enable dissemination of SI traceability with optimal accuracy

In applications where appropriate reference materials are not available and materials are highly sensitive to changes in ambient conditions, transfer standards provide a complementary approach for disseminating traceability. The applicability of commercially available instruments is, however, highly limited by their sensitivity to material matrix. To reduce material sensitivity in moisture measurements, CETIAT developed a new instrument that measures complex permittivity of a moist material by using radio-frequency wave and microwave at a low energy level. The system comprises a VirtualNetwork Analyser and two non-resonant and broadband measurement cells: the capacitive one (Figure 8 A) covers the frequency range 1 MHz to 400 MHz while the coaxial cell (Figure 8 B) operates in the range 200 MHz to 5 GHz. Tests carried out for the instrument showed its potential to reliable moisture measurements with a wide range of materials.

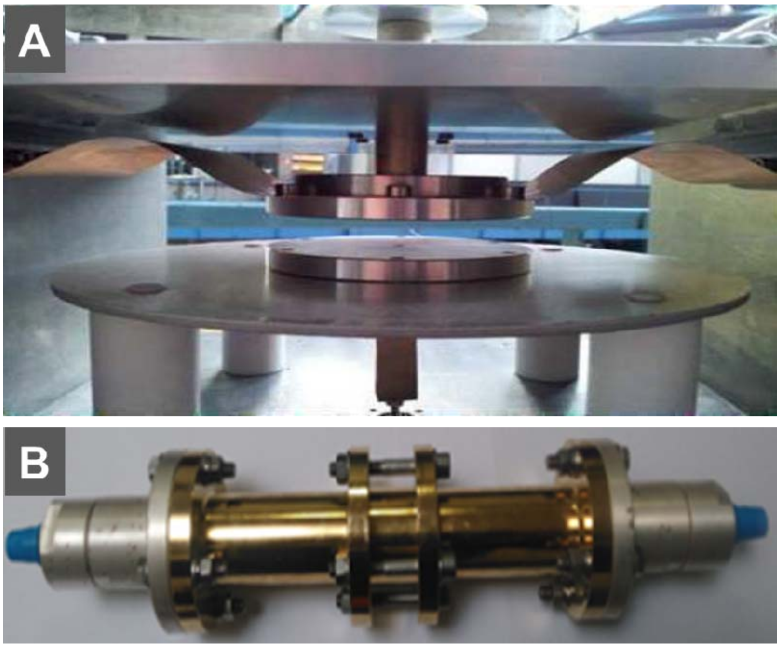

Figure 8. Photographs of the capacitive (A) and coaxial (B) measurement cells developed by CETIAT.

To enable usage of a wide range of materials in validations and comparisons demanding moisture measurement applications, a portable system was developed at INRIM in which constant ambient conditions for a reference material are maintained during transport (see Figure 9). A microwave sensor was developed for carrying out the comparative measurements on site.

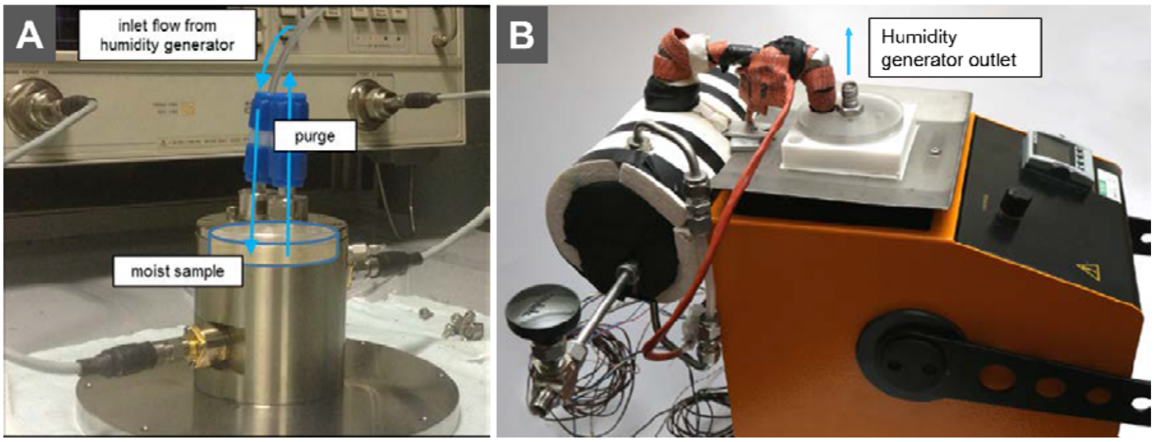

Figure 9. Photographs showing details of the transfer standard system developed at INRIM: a microwave sensor unit (A) and a portable humidity generator unit (B).

In the development of this equipment, INRIM and CETIAT worked closely together, benefitting from their previous expertise in microwave measurements. Validation tests carried out for the system show that this system provides better reliability over wide ranges of water content and materials than was achievable before in validations and comparisons of reference moisture equipment.

3.2.3 Methods for quantifying and reducing the effect of sample transport and handling

Sample handling and transport are an essential part of reference moisture measurements. They significantly affect the quality of the measurement result. If the equilibrium relative humidity of a solid sample is far from the relative humidity of ambient air, even a short exposure time of the sample may significantly reduce or increase the amount of water that is measured with the reference system. Changes in bulk moisture of sample may occur during transportation due to water permeation through sample bags/containers. Large variations in temperature may increase this effect and also water transport within a sample causing inhomogeneity of the sample. BRML, CMI, INRIM, MIKES and TUBITAK studied these effects in paper and wood samples.

It was demonstrated that monitoring wood chip samples during transportation with RH/T loggers located in the sample bags provided useful information about changes in the water mass fraction of the samples. However, because of limited accuracy in the low moisture range, the limitation to the equilibrium relative humidity below 100 % and specificity to matrix, the method is considered too limited in range and accuracy for common use in industry.

Weighing sample bags with and without the sample before and after transportation was found a powerful method for monitoring the samples during transportation and storage and quantifying any changes and corresponding uncertainties. For estimating the uncertainty due to sample handling, the mass of a parallel sample in an open container is monitored with respect to exposure time.

A good practice guide for estimating the uncertainty due to sample handling and transportation was prepared and submitted to EURAMET for publication.

3.2.4 Novel calibration facility with SI traceability for surface moisture meters

Besides bulk moisture, surface moisture is also measured in industry for controlling quality e.g. in gluing process. These measurements are carried out for determining local moisture content at declared depth in materials that have typically large moisture gradient close to the surface. Until now there has not been a calibration method available or even appropriate way to express a calibration result relevant for such conditions. Calibration with homogenous material samples does not provide the information about the depth characteristics which is vital for the users.

In this project, a novel calibration system for surface moisture meters was developed in cooperation between UL, INRIM and UNICLAM. Also, a new surface moisture meter based on DRIFT spectroscopy (Diffuse

Reflectance Infrared Fourier Transform) was developed at UL for monitoring moisture in polymer surfaces. In the calibration system (Figure 10), a known linear moisture gradient is generated a sample specimen by controlling the humidity accurately at both sides of the specimen. The calibration result is expressed in terms of equivalent depth. At this depth, the actual local moisture in the specimen is equal to the reading of the device under calibration.

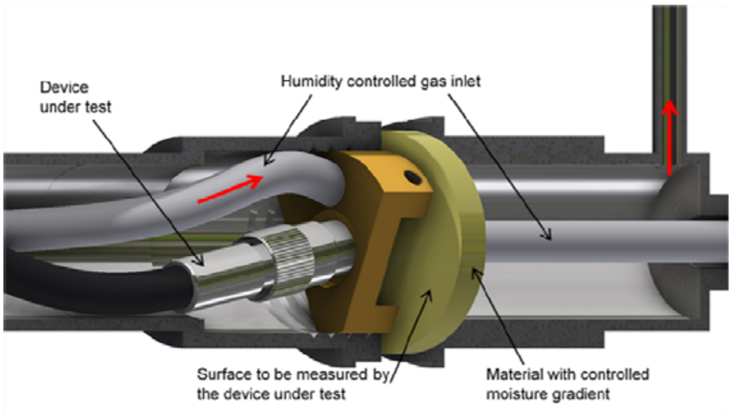

Figure 10. Schematic drawing of the calibration system developed at UL for surface moisture meters.

3.2.5 Modelling relevant to moisture metrology to include local moisture variations in the uncertainty

Spatial and temporal moisture differences cause errors in moisture measurements and should be addressed in measurement uncertainty analyses. On the other hand, accurate information about a moisture gradient is essential when performing calibrations for surface moisture meters. Usually, experimental methods are not feasible in moisture measurement and calibration applications but simulations using appropriate modelling are needed.

In this project, a general mathematical model for heat and mass transfer in wet porous domains was selected. The Eulerian approach was used to derive a set of conservation equations for the analysis of single-phase and multiphase systems. A fully numerical tool was developed by UNICLAM in order to estimate temperature, velocity and moisture profiles in materials. The different parts of the mathematical and numerical models were separately verified by selecting representative numerical and analytical benchmarks available in the scientific literature. The numerical model was applied to the determination of the moisture profiles and distributions in selected materials. The tool was validated considering real experiments realised in collaboration with DTI, INRIM and UL. An example of these is given in Figure 11. The tool had a significant role in the development of the surface moisture calibration system.

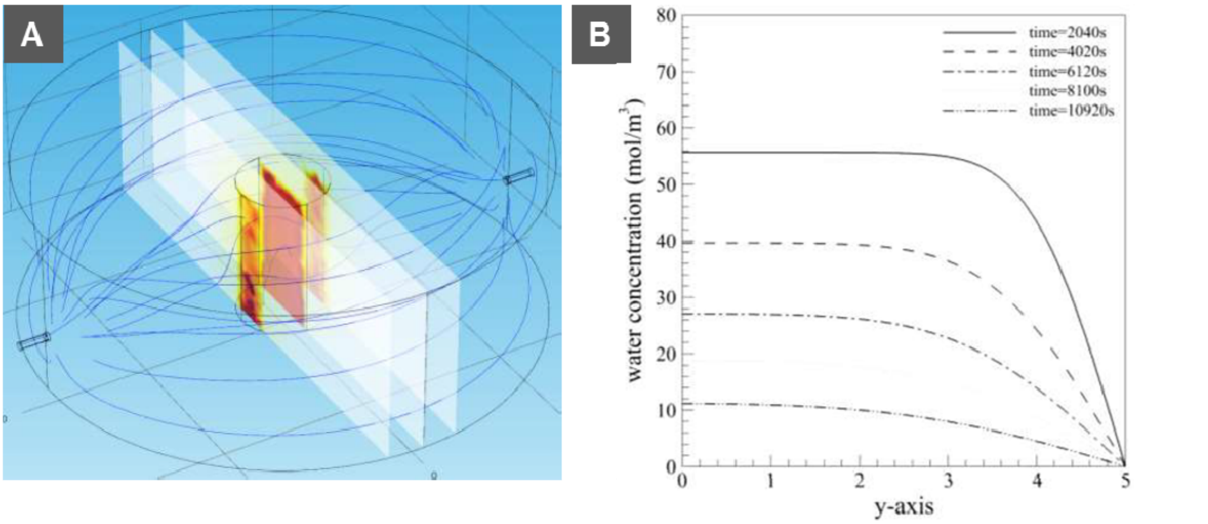

Figure 11. An example of cases applied the developed numerical simulation tool. A) Water concentrations within the porous sample evaluated in correspondence of three different sections and a computational time of 1000s. B) Water concentration of the sample as a function of the y-axis obtained in correspondence of different computational times.

3.2.6 Practical but metrologically sound methods for estimating uncertainty in selected industrial applications

Uncertainty analysis had a major role in the developments reported above as appropriate uncertainty information is a key feature of SI traceability. Using the results and gained experience spreadsheet calculation tools were developed for laboratories applying LoD and cKF in moisture measurements.

In industrial laboratories new rapid moisture measurement methods have been challenging the conventional oven drying method in determining moisture in solid biofuels. Uncertainty components relevant to measurements with commercial microwave and magnetic resonance analysers were studied by UOULU in cooperation with VTT MIKES. Figure 12 shows the repeatability characteristics of the commercial instruments tested. With the tested individual instruments (one of each kind) the accuracy and precision of LoD method was not achieved but it was concluded that results good enough for biomaterial invoicing can be achieved.

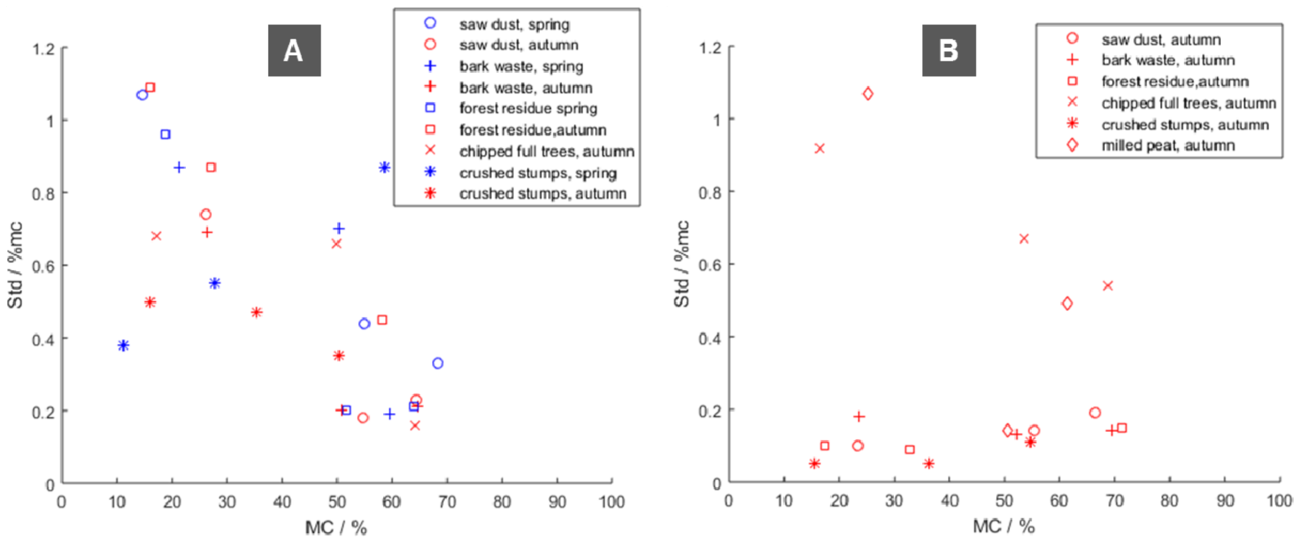

Figure 12. Repeatability test results of A) a magnetic resonance instrument and B) a microwave moisture analyser.

3.3 Coherent and developed moisture metrology infrastructure in Europe

Metrology of moisture in materials is multidisciplinary. National metrology institutes (NMIs) with moisture capability often address this within thermometry/humidity laboratories (because of the interaction with air humidity), or in chemistry, or partly in other areas where relevant metrological techniques are researched or used (RF/microwave/submillimetre electromagnetic radiation, mass, electrical, radiometry etc.). At the international metrology level, moisture is in the remit of a variety of CIPM Consultative Committees and technical committees of regional metrology organisations. The treatment of the subject is fragmented because it spans several metrology fields and it is difficult to ensure a cross-disciplinary view. Moisture traceability services for solids are increasing at the NMI level but none of them have been published so far in the BIPM CMC database.

In this project a coherent moisture metrology infrastructure was set up in Europe in which NMIs specialise in different subfields of moisture measurements but collaborate closely with each other. To support this development after completion of this project, a EURAMET project was agreed and registered. We actively cooperated with other regional metrology organisations and participated in the work of CIPM CCT and CIPM CCQM for enhancing the coherence at the global level as well.

3.4 Summary

As a summary, the key results of this project are:

- New measurement capabilities at NMIs/DIs
    - As the first in the world, a group of NMIs/DIs (DTI, NPL, UT and VTT MIKES) now has the capability to provide moisture measurements for solid samples using primary standards with demonstrated traceability to SI in a wide range of materials and water mass fraction up to 400 g/kg.
    - The capability of calibrating surface moisture meters in terms of moisture equivalent depth at UL is first in the world.
    - TUBITAK and INRIM have extended their measurement capabilities to moisture in solids.
- Reference materials
    - NPL has certified new reference materials offering reference values of water mass fraction with features rarely if ever available before in a water CRM: they provide large sample sizes (up to several hundred grams) and they range up to nominally 400 g/kg. These CRMs will be available for NMIs/DIs, the calibration sector and wider industry.
    - CMI will provide reference wood samples for quality control of wood moisture meters used in industry.
- New methods and procedures:
    - For the first time in the world, effective principles of SI traceability for moisture in solids were introduced in this project and methods for unambiguous realisation of water mass fraction in solid samples were developed. These methods will primarily be exploited by NMIs/DIs.
    - Two good practice guides were developed: one for usage and estimating uncertainty with coulometric Karl Fischer titration and another one for estimating uncertainty due to sample handling and transportation. These guides are for use in calibration sector.
    - To support uncertainty estimations in industry and NMIs/DIs, spreadsheet uncertainty budget templates were developed for LoD and cKF and a method and software tool were developed for simulating moisture gradients in solids.
- A European moisture metrology infrastructure was set up in this project with specialisation of NMIs/DIs in different subfields of moisture measurements.

4 Actual and potential impact

4.1 Impacts addressing specific stakeholder needs

This project is creating direct impact for the following stakeholder groups:

- -the metrology community in Europe and wider
- -accredited calibration and testing laboratories providing services to industry
- -industrial laboratories assuring the quality of moisture measurements in their companies
- -manufacturers of moisture measuring instruments
- -research groups carrying out research and development on materials and processes
- -standardisation bodies
- -accreditation bodies
- -users of moisture meters and analysers

Metrology community in Europe and wider

Using the primary standards developed in this project, four participating NMIs/DIs are now able to provide reference moisture measurements with demonstrated SI traceability. Four other European and five non-European NMIs/DIs obtained evidence on the quality of their moisture measurement capabilities in the comparisons carried out in this project. By applying the methods developed for unit realisations and uncertainty estimations, other NMIs/DIs are able to set up primary standards for moisture in solids. The developed CRMs provide a reliable way to carry out inter-laboratory comparisons in a wide water mass fraction range, which is essential in demonstrating the equivalence between NMIs/DIs. The European metrology infrastructure established through close collaboration between the project partners can now provide measurement and consultancy services for a wide range of moisture measurement applications.

The general principles of SI traceability for moisture measurements developed in this project and submitted to working groups of CIPM and EURAMET will support the implementation of key comparisons and CMC declarations.

Accredited and other industrial laboratories

With the methods and facilities developed in this project, the partner NMIs/DIs are now providing services for accredited and industrial laboratories to fulfil their needs to validate their moisture analysis procedures and external quality control data, and to improve measurement procedures. Demonstrated SI traceability in water mass fraction measurements will also open possibilities for the industrial laboratories to develop new measurement services. These advances were made possible by the developed reference measurement systems, transfer standards, reference materials and gained expertise. The developed good practice guides and uncertainty budget templates will help the improvement of existing laboratory services and the setting up of new ones.

It is foreseen that for practical reasons, reference measurements in terms of moisture content (without water specificity) will maintain their importance in industry for some time. By performing comparisons against a primary standard, industrial laboratories are better able to understand and study the effect of variations in their samples with respect to the amount of non-aqueous volatiles and binding of water.

Instrument manufacturers

The comparative research on different types of commercial moisture analysers and LoD was carried out in collaboration with instrument manufacturers. The work provided valuable information about the characteristics of these instruments. Other instrument manufacturers were involved as collaborators, obtaining reference measurements and exchange of information. The use of SI traceable water mass fraction reference measurements is useful in particular in the development of fully water selective techniques.

Calibrations and other experimental studies can be carried out with the facilities developed in this project, which will enhance new technologies and products for emerging markets under regulations, e.g. in the fuel trade.

Research on materials and processes

The project has potential impact for R&D on materials and industrial processes as demonstrated by the number of requests received and demonstrations provided on reference measurements to support the development of new moisture measurement methods and manufacturing processes for various applications. Growing interest in the quality control of soil moisture measurements is increasing the need for SI traceable reference moisture measurements. The measurement facilities developed at VTT MIKES and TUBITAK in this project will be used for feasibility studies related to soil moisture measurements in the EMRP MeteoMet2 project (ENV58). The development of the calibration method for surface moisture meters at UL induced the development of a new surface moisture meter that is applicable to various moisture measurement applications.

Standardisation and accreditation bodies

Highly skilled experts willing to contribute to standard development and technical assessments are vital for standardisation and accreditation bodies, respectively. Within this project, input was provided to eight standards under preparation/revision by standardisation committees of ISO, CEN and OIML. Because of this project, the European NMIs/DIs are now able to provide experts of SI traceability in moisture measurements for accreditation bodies.

Users of moisture meters and analysers

The European moisture metrology infrastructure set up in this project makes the measurement, calibration and consultancy services as well as the reference materials developed in the project available for any user of moisture meters and analysers.

During the project, training on carrying out reliable moisture measurements was provided to various stakeholders and good practice guides were developed. Training material for future events has been developed and made publically available through the project website.

This project provides an excellent basis for further development of traceability to online moisture measurements in industry. Actions have been taken to set up a follow-on project addressing this need.

4.2 Early impacts

The interaction and work with the EN TC346 WGs resulted in clear inputs to the preparation of the draft norm prEN 16682. The recent public enquiry among national standardisation bodies resulted in over 90 % approval rate for this standard.

Using the developed primary standards, reference measurements have been provided to several companies seeking improvements and validation of their processes. These include measurements of paperboard, plywood, polymer, active pharmaceutical ingredients and carbon samples. The new surface moisture meter developed in this project was demonstrated for an industrial company for use as a quality control tool in a gluing process.

Three NMIs are planning to include new measurement services developed in this project in their scopes of accreditation. One partner is applying for extending its DI scope to moisture in materials. With the project developments, the partners have already started providing new measurement, training and consultancy services and reference materials for customers.

Several invitations to present the findings of the project have been received from companies and non-European NMIs/DIs.

A new EURAMET project (P1400) has been set up to pursue further progress towards metrology infrastructure for the moisture field, such as intercomparisons, potential BIPM CMCs, ILAC accreditations, and dissemination of measurement traceability. Cross-disciplinary cooperation is enhanced by bringing together expertise in physical and chemical metrology relevant to the moisture field.

The outcomes of the project were presented for the CCT WG-Hu in its meeting in July 2016. The working group initiated plans for actions enabling publication of BIPM CMCs on the basis of the outcomes.

4.3 Summary of dissemination activities

For sharing the research findings of this project with wider scientific community, 15 articles have been submitted and three are under preparation for publication in peer-reviewed scientific journals while 12 full papers have been published in conference proceedings. In total 36 presentations were given in 12 international scientific conferences. In particular, the project was presented by several talks and posters at TEMPMEKO 2013, CIM 2015, ISEMA 2016 and TEMPMEKO 2016. In addition, contributions were provided to 16 scientific seminars and training events arranged in Estonia, Italy, Turkey and UK. The international metrology community was specifically addressed by presentations and communications in 9 meetings of various working groups of EURAMET, CCT and CCQM.

The project findings were communicated to accredited laboratories and other stakeholders in industry at various face-to-face meetings and seminars. Two international stakeholder workshops (Lyon, June 2015 and Copenhagen, May 2016) and 9 other training events were arranged in Denmark, Finland, France, Turkey and UK. A wide range of industry and other fields were represented in the audiences of these events. Two good practice guides and two sets of training material were developed, and four articles were published in professional magazines. To contribute to the development of ISO, CEN and EN standards, the project consortium interacted with 14 working groups of standardisation bodies. Many of the standards relevant for these groups are dealing with moisture as a significant material parameter in different materials ranging from tea to thermoplastic materials.

For the general public audience, information was provided through the project website, newsletters, partner websites and social media. Updates were published in a LinkedIn group established for the project, on Twitter and Facebook.

4.4 Future impacts

The developed capabilities will continue to be implemented after the end of the project. Several project partners have explicitly obtained funding to maintain the developed facilities, and to promote the new services. The relationships built across metrology and standard committees in physical and chemical metrology will continue to provide ongoing consistent and effective metrology infrastructure. A EURAMET project has been agreed to provide a framework for the strategic coordination of these activities.

In the application areas, biomass fuel is a key area where improved moisture measurements will allow future development of efficient processes for this renewable energy source. Improved measurements in other industries such as milk powder production are beginning to be underpinned by meaningful metrological traceability, and this will provide a reduction in process energy, waste and re-work. Where measurements are being taken up for research on materials and processes (such as novel polymers) this supports innovation with diverse long-term future benefits.

5 Website address and contact details

The project website is: www.metef.net

The contact persons are:

- Dr Martti Heinonen, VTT MIKES, martti.heinonen@vtt.fi
- Dr Stephanie Bell, NPL, stephanie.bell@npl.co.uk

6 List of publications

Full papers published in scientific journals and conference proceedings

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3. S Bell, N Boese, R. Bosma, M Buzoianu, P Carroll, V Fernicola, E Georgin, M Heinonen, A Kentved, C Melvad, J Nielsen, Status and strategy for moisture metrology in European metrology institutes, Int J Thermophys, August 2015, Volume 36, Issue 8, pp 2185–2198, DOI 10.1007/s10765-015-1859-6

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6. S. Bell , R. Aro, F. Arpino, S. Aytekin, G. Cortellessa, M. Dell'Isola, Z. Ferenčíková, V. Fernicola, R. Gavioso, E. Georgin, M. Heinonen, D. Hudoklin, L. Jalukse, N. Karaböce, I. Leito, A. Mäkynen, P. Miao, J. Nielsen, I. Nicolescu, M. Rudolfová, M. Ojanen-Saloranta, P. Österberg, P. Østergaard, M. Rujan, M. Sega, R. Strnad, T. Vachova, METefnet: developments in metrology for moisture in materials, 17th International Congress of Metrology, 15003 (2015), doi:10.1051/metrology/20150015003

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9. E. Georgin, J. F. Rochas, S. Hubert, P. Achard, M. W. Ben Ayoub, P. Sabouroux, First steps in development of a new transfer standard, for moisture measurement, based on radio-frequency wave and micro-wave, 17th International Congress of Metrology, 15008 (2015), DOI: 10.1051/metrology/201515008

10. M. Sega, G. Beltramino, V. Fernicola, F. Rolle, A. Verdoja, Metrological traceability for moisture content analysis in wood pellets, 17th International Congress of Metrology, 08002 (2015), DOI: 10.1051/metrology/20150008002

11. F. Rolle, G. Beltramino, V. Fernicola, M. Sega, A. Verdoja, Moisture determination for food quality assessment, 17th International Congress of Metrology, 15006 (2015), DOI: 10.1051/metrology/20150015006

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19. E. Georgin, M. W. Ben Ayoub, J. F. Rochas, S. Hubert, P. Achard, L. Neves, P. Sabouroux, First steps in development of a new transfer standard, for moisture measurement, based on radio-frequency wave and micro-wave, submitted to *Meas. Sci. Technol.*

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21. Z. Pálková, M. Rudolfová, E. Georgin, M. W. Ben Ayoub, V. Fernicola, G. Beltramino, N. Ismail, B. Il Choi, M. Heinonen, Effect of handling, packing and transportation on the moisture of timber wood, submitted to *Int. J. Thermophys.*

22. P. Østergaard, J. Nilsen, SI-traceable water content in solids, bulks and powders, submitted to *Int. J. Thermophys.*

23. M. Ojanen-Saloranta, H. Sairanen, P. Österberg, J. Salminen, H. Kajastie, T. Rajamäki, M. Heinonen, Reference measurement system for moisture in wood-based materials and biomasses, submitted to *Meas. Sci. Technol.*

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30. M. Heinonen, R. Aro, S. Bell, R. Blasco, B. Il Choi, D. abd El – Galil, V. Fernicola, N. Ismail, L. Jalukse, N. Karböce, T. Keawprasert, M. Krasheninina, I. Leito, M. Medvedevskikh, P. Miao, J. Nielsen, S. Oğuz Aytekin, M. Ojanen-Saloranta, P. Østergaard, S. Pepe, F. Rolle, J. Salminen, M. Sega, J. Skabar, An intercomparison of water content measurements of wood pellets, will be submitted to *Metrologia*