Final Publishable JRP Summary for JRP SIB64 METefnet Metrology for Moisture in Materials

Overview

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.

Need for the project

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.

EMRP
European Metrology Research Programme
Programme of EURAMET

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Scientific and technical objectives

The project's objectives were to develop:

1. unambiguous 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

Objectives 1 to 3 developed reference measurement methods and techniques for the basis of SI traceable moisture measurements. Objectives 4, 5 and 7 developed reference materials and calibration techniques that enable dissemination of the SI traceability moisture measurements to a variety of industrial measurement calibration setups. Objectives 6, 8 and 9 provided methods for uncertainty estimations that are essential for moisture measurement and calibration. Objective 10 ensured efficient availability of European metrology services in the field of moisture in materials.

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

Results

Unambiguous methods for moisture in solids in terms of water mass fraction and amount fraction
The ambiguity of an actual measurand is a major obstacle in establishing SI traceability in moisture measurements. To remove this obstacle, water mass fraction and amount fraction were chosen as well-defined and widely used quantities and four unambiguous methods were successfully developed for them. By combining sample weighing, oven drying and water vapour measurement high water specificity was achieved without losing a link to the methods specified by industrial documentary standards. Different approaches in sample drying and water vapour measurements enable measurement systems to be developed for a wide range of materials and sample sizes. The four methods also provide sufficient redundancy for drawing general conclusions needed for the basis of SI traceability in the field of moisture measurements. For the first time, the error due to non-aqueous volatiles, and its uncertainty, can now be properly included in reference moisture measurements for solids.

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
In moisture measurements, a wide range of sample sizes is used to obtain representative results for different materials with grain sizes. Two novel and accurate traceable measurement methods were developed for water vapour detection; one had a cold trap in combination with a capacitive humidity sensor and the other had a dew-point sensor for water vapour detection. Combining these with weighing and oven drying systems, primary standards for water mass fraction were set up. Samples of up to 400 g were analysed using the primary standards. The relative expanded uncertainty (95 % confidence level) of 1.6 % was demonstrated by an inter-laboratory comparison.

Two other primary standards were obtained by carefully validating a commercially evolved water vapour analyser and a coulometric Karl Fischer titrator with sample oven, respectively. The sample mass with these

is a few grams or less. The relative expanded uncertainty (95 % confidence level) of 1.8 % was demonstrated by an inter-laboratory comparison using these two primary standards.

These new measurement primary standards provide results in terms of water mass fraction with validated uncertainties and demonstrated SI traceability. When using these measurement standards, non-aqueous volatiles do not distort the results. These developments are an improvement on existing methods, where uncertainties did not include the effect of non-aqueous volatiles and were up to 4 % of the measured value, depending on method and range – and much worse for trace ranges.

Effective general principles of SI traceability in the field of moisture measurements
The primary standards developed in the second objective were compared to each other and to reference methods at other institutes. These comparisons provided valuable new information about the equivalence of techniques used in laboratories in Europe, and in Asia, Africa and South America. The measurement results obtained by using different materials and different water mass fractions from 50 g/kg to 400 g/kg were in good agreement, indicating a good equivalence of the techniques. On the basis of the results of this project, the project partners made a recommendation on terms, definitions, realisations and principles for SI traceability for moisture measurements and submitted it to the Working Group for Humidity of the International Committee for Weights and Measures. The recommendation is a significant step towards establishing internationally recognised reference measurement services within the CIPM Mutual Recognition Arrangement (CIPM MRA) that is the framework through which National Metrology Institutes (NMIs) demonstrate the international equivalence of their measurement standards and the calibration and measurement certificates they issue.

New certified reference materials with traceability and improved stability
Three new reference materials were developed, certified and studied in 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 certified reference material for water in solid. They provide large sample sizes (up to several hundred grams), they range up to nominally 400 g/kg (40 %), and have good stability. These features extend the use of moisture reference materials to applications where larger sample sizes are needed and wet materials are of interest. The improved stability also extends the shelf life of the reference material and provides better measurement uncertainty.

Novel transfer standards to enable dissemination of SI traceability with optimal accuracy
Two transfer standards were developed for providing a complementary approach for disseminating traceability to a wide range of materials. In the first transfer standard method, moisture sensing with radio-frequency wave and microwave at a low energy level was used to reduce matrix dependent errors. In the second method, the versatility with materials was achieved by the combination of a microwave sensor and reference sample stabilised with a portable humidity generator. With these two transfer standards, performance tests and system validations of moisture measurements in heterogeneous solid samples is more reliable than from current moisture analysers.

Methods for quantifying and reducing the effect of sample transport and handling
The project found the use of humidity and temperature loggers located in wood chip sample bags to be too limited as a method for monitoring moisture changes during transportation. However, weighing sample bags with and without the sample before and after transportation was found to be a powerful method for monitoring the samples during transportation and storage and quantifying any changes and corresponding uncertainties. Based on this a Good Practice Guide for estimating the uncertainty due to sample handling and transportation was prepared and submitted to EURAMET for publishing.

Novel calibration facility with SI traceability for surface moisture meters
Until now, surface moisture meters have been calibrated using material samples with homogenous moisture content. However, in actual measurement applications, there is typically a large moisture gradient close to the surface that significantly affects the meter reading. To solve this problem, a novel calibration system for surface moisture meters was developed. For the first time calibration results can now provide depth characteristic information. Also, a new surface moisture meter based on DRIFT spectroscopy (Diffuse Reflectance Infrared Fourier Transform) was developed. This DRIFT meter improves moisture monitoring in polymer surfaces.

Modelling relevant to moisture metrology to include local moisture variations in the uncertainty
A numerical model and software tool was developed to enable the effects of spatial and temporal moisture differences to be included in the measurement uncertainty analyses. It was experimentally validated and applied to the development of the surface moisture calibration system in objective 7. The software tool proved to be useful for studying the moisture gradients and changes in solids and estimating corresponding uncertainty in moisture measurements. This is relevant to various drying and baking processes.

Practical but metrologically sound methods for estimating uncertainty in selected industrial applications
Using the results and experience gained in the previous objectives, spreadsheet calculation tools were developed for laboratories applying loss-on-drying and coulometric Karl Fischer titration techniques in moisture measurements. Uncertainty components relevant to moisture measurements of forest biomass with new commercial microwave and magnetic resonance moisture analysers were studied experimentally. The results demonstrated the importance of taking into account the effects of material variations and sample handling when calibrating these analysers.

Coherent and developed moisture metrology infrastructure in Europe
Measurement capabilities for a wide range of materials need to be established to provide the moisture metrology services needed by industry, so the project developed a European metrology infrastructure to better serve stakeholders in the moisture metrology field. A network of relevant NMIs was set up in which the institutes 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, (Moisture metrology development; reg. no: 1400) was agreed.

Actual and potential impact

The project achieved all its objectives. The developments of this project enable unambiguous and feasible SI traceability measurements and calibrations of moisture in solid materials. A new measurement infrastructure linking moisture measurements to the SI has now been implemented. This consists of four primary standards (for providing reference measurements) and reference materials, transfer standards and a calibration facility for disseminating traceability to laboratory analysers and surface moisture meters used in industry.

Dissemination

15 articles were submitted and 3 are under preparation for publication in peer-reviewed scientific journals while 12 full papers have been published in conference proceedings. 36 presentations were given in 12 international scientific conferences. In particular, the project was presented by several talks and posters in the International Symposium on Temperature and Thermal Measurements in Industry and Science (TEMPMEKO) 2013 and 2016, CIM 2015 and the International Conference on Electromagnetic Wave Interaction with Water and Moist Substances (ISEMA) 2016. In addition, contributions were provided to 16 scientific seminars and training events. The international metrology community was specifically addressed by presentations and communications in 9 meetings of various working groups of EURAMET, BIPM Consultative Committee for Thermometry CCT and Consultative Committee for Amount of Substance (CCQM).

Project findings were communicated in various face-to-face meetings and seminars with accredited laboratories and other stakeholders. Two international stakeholder workshops and 9 other training events were arranged. A wide range of industry and other fields were represented in the audiences of these events.

Two Good Practice Guides (one on estimating the uncertainty due to sample handling and one on using Karl Fischer titration) are available on the project website. Two sets of training material were developed, and 4 articles were published in professional magazines. Information was provided through the project website, newsletters, partner websites and social media.

Impact on standardisation

To contribute to the development of ISO, CEN and EN standards, the project consortium interacted with 14 working groups of standardisation bodies, covering fertilisers, cereals, thermoplastics, ores and tea. 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.

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.

Actual Impact

Instrument manufacturers and industrial laboratories were involved in the project as collaborators, and they will provide an effective link with end users and companies. The project has highlighted the influence of handling and sampling techniques, and how they can cause greater uncertainty in the moisture measurements than the measurement techniques themselves.

There have been several requests for demonstrations on reference measurements to support the development of new moisture measurement methods and manufacturing processes. Increasing interest in quality control of soil moisture measurements reflects the need for SI traceable reference moisture measurements. The measurement facilities developed at VTT and TUBITAK in this project are being used for feasibility studies to explore candidate approaches for improved traceability for soil moisture measurements in the EMRP project ENV58 MeteoMet2.

The reference materials developed by this project provide a reliable way to carry out comparisons in a wide water mass fraction range, which is essential in demonstrating the equivalence between NMIs on a regular basis. This project created a network of European metrology institutes that are specialised in different subfields of moisture measurements but collaborate closely together to provide cost effective and extensive services for industry. Active collaboration of this network within the global metrology community will ensure effective progress in initiated actions towards international recognition of the general principles of SI traceability for moisture measurements developed in this project.

The new services developed in this project are primarily for accredited and other industrial laboratories that need reliable reference measurements for improving and validating their moisture analysis procedures and obtaining external quality control data. However, the calibrations and other measurement techniques developed in this project will enhance new technologies and products being developed in emerging or regulated markets, e.g. in fuel trade, by providing unambiguous reference measurement results with known uncertainty. The good practice guides and uncertainty budget templates, as well as the training and consultation services of the project partners, will help in realising these developments.

This project created expertise on moisture calibrations and SI traceability in moisture measurements that has already been exploited in industrial R&D projects. It focused on the following material groups: pharmaceuticals, polymer/plastic, foodstuffs, feed, biomass and wood based material. However, many of the outcomes are suitable for extension to wider classes of materials and applications.

Potential impact

The project outputs provide an excellent basis for further development of SI traceable online moisture measurements in industry. Further research is needed to develop measurement, calibration and uncertainty estimation methods that are practical and feasible for implementation in industrial processes.

Using the primary standards and other reference measurement systems and methods developed in this project the metrology institutes of Czech Republic, Denmark, Estonia, Finland, France, Italy, Romania, Slovenia, Turkey and UK are now able to provide reference moisture measurements with demonstrated SI traceability. Services have already been opened and reference measurements have been provided for several customers covering a wide range of materials. Also the metrology institutes of Russia, Korea, Thailand, Egypt and Argentina obtained evidence on the quality of their moisture measurement capabilities in the inter-laboratory comparisons of this project.

The relationships built across metrology and standardisation committees in physical and chemical metrology will continue to provide ongoing consistent and effective metrology infrastructure. A EURAMET project, Moisture metrology development; reg. no: 1400, has been agreed to provide a framework for 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.

List of publications

Full papers published in scientific journals and conference proceedings

1. F. Arpino, G. Cortellessa, M. Dell'Isola, N. Massarotti, A. Mauro, High Order Explicit Solutions For Transient Natural Convection of Incompressible Fluids in Tall Cavities, Numerical Heat Transfer, Part A: Applications: An International Journal of Computation and Methodology, October 2014, Volume 64, Issue 8, pages 839–862, DOI: 10.1080/10407782.2014.892389
2. F. Arpino, G. Cortellessa, A. Mauro, Transient thermal analysis of porous and partially porous cavities, Numerical Heat Transfer, Part A: Applications: An International Journal of Computation and Methodology, March 2015, Volume 67, Issue 6, pages 605-631, DOI: 10.1080/10407782.2014.949133
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
4. M. Ojanen, H. Sairanen, K. Riski, H. Kajastie, M. Heinonen, Moisture measurement setup for wood based materials, NCSLI Measure J. Meas. Sci., 9 (2014), No. 4, pp. 56-60
5. S. Beguš, G. Begeš, J. Drnovšek, D. Hudoklin, A novel NIR laser-based sensor for measuring the surface moisture in polymers, Sensors and Actuators A Physical 221:53-59, November 2014, doi:10.1016/j.sna.2014.10.032
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
7. M. Ojanen-Saloranta, H. Sairanen, J. Salminen, H. Kajastie, M. Heinonen, Moisture measurement setup for wood based materials and biomasses, 17th International Congress of Metrology 08008 (2015), DOI: 10.1051/metrology/20150008008
8. P. Österberg, M. Heinonen, M. Ojanen-Saloranta, A. Mäkynen, The comparison of a microwave based bioenergy moisture measurement instrument against the loss-on-drying method, Proceedings of the XXI IMEKO World Congress, Prague, CZECH REPUBLIC, 2015, Volume 3, p 2170, https://www.imeko.org/publications/wc-2015/IMEKO-WC-2015-TC24-458.pdf
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
12. P. Österberg, M. Heinonen, M. Ojanen-Saloranta, A. Mäkynen, Comparison of an NMR-based Bioenergy Moisture Measurement Instrument against the Loss-on-Drying Method, Proceedings of the 11th International Conference on Electromagnetic Wave Interaction with Water and Moist Substances, Florence, ITALY, 2016, p. 285 – 296, Edifir-Edizione Firenze, May 2016. ISBN 978-88-7970-800-5
13. M. Rudolfová, L. Pitrová Netolická, Z. Ferenčíková, T. Váchová, R. Strnad, Dynamic properties during wood humidification, 17th International Congress of Metrology, 14009 (2015), DOI: 10.1051/metrology/20150014009
14. D Hudoklin, I Muñoz Lopez, G Begeš, J Drnovšek and S Beguš, Industrial implementation of the new nir laser-based sensor for measuring surface moisture in polymers, 17th International Congress of Metrology, 11003 (2015), DOI: 10.1051/metrology/20150011003

15. F. Arpino, G. Cortellessa, N. Massarotti, A. Mauro, Transient thermal analysis of porous cavities, Third International Conference on Computational Methods for Thermal Problems (THERMACOMP2014), ISBN 978-88-7431-727-1

16. F. Arpino, G. Cortellessa, M. Dell'Isola, G. Ficco, A. Carotenuto, N. Massarotti, Transient incompressible flow in a partially porous bouyancy driven tall cavity, ASME-ATI-UIT 2015, ISBN 978-88-98273-17-1

17. M. W. Ben Ayoub, E. Georgin, J. F. Rochas, S. Hubert, P. Achard, P. Sabouroux, L. Neves, New approach for measuring moisture in solids using Radio frequency and Microwave, Proceedings of the 11th International Conference on Electromagnetic Wave Interaction with Water and Moist Substances, 2016, ISBN 978-88-7970-800-5

Full papers under peer-review by scientific journals

18. G. Beltramino, D. Smorgon, V. Fernicola, Design of a portable high-pressure humidity generator based on corrugated-plate heat exchanger, submitted to *Int. J. Thermophys.*

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.*

20. D. Hudoklin, S. Ranogajec, G. Cortellessa, G. Begeš, J. Drnovšek, F. Arpino, V. Fernicola, S. Beguš, Novel approach in characterisation of surface moisture sensors, submitted to *Metrologia*

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.*

24. M. Heinonen, S. Bell, B. Il Choi, G. Cortellessa, V. Fernicola, E. Georgin, D. Hudoklin, G. V. Ionescu, N. Ismail, T. Keawprasert, M. Krasheninina, I. Leito, J. Nielsen, S. Oğuz Aytekin, P. Österberg, J. Skabar, R. Strnad, New methods for establishing SI traceability for moisture measurements in solid materials, submitted to *Int. J. Thermophys.*

25. J. Salminen, H. Sairanen, S. Patel, M.Ojanen-Saloranta, H.Kajastie, Z.Palkova, M.Heinonen, Effects of sample handling and transportation on the moisture content of biomass samples, submitted to *Int. J. Thermophys.*

26. F. Rolle, G. Beltramino, V. Fernicola, M. Sega, A. Verdoja, Metrologically traceable determination of the water content in biopolymers: INRiM activity, submitted to *Int. J. Thermophys.*

27. P. Österberg, M.Heinonen, M. Ojanen-Saloranta, A. Mäkynen, Comparison of the performance of a microwave based and an NMR based biomaterial moisture measurement instruments, submitted to *ACTA IMEKO*

Full papers under preparation for submission to peer-reviewed scientific journals

28. P. Miao, S. Bell, C. McIlroy, C. Spray, Validation of an evolved vapour coulometric technique for water content determination, will be submitted to *Int. J. Thermophys.*

29. S. Oğuz Aytekin, N. Karaböce, M. Eroglu, N. Zorlu, H. Yilmaz, Comparison of methods for moisture content determination in paperboard, will be submitted to *Int. J. Thermophys*

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*

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