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NEW PRIMARY STANDARDS FOR ESTABLISHING SI TRACEABILITY FOR MOISTURE MEASUREMENTS IN SOLID MATERIALS

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Abstract

A European research project METefnet addresses a fundamental obstacle to improving energy intensive drying process control: due to ambiguous reference analysis methods and insufficient methods for estimating uncertainty in moisture measurements, the achievable accuracy in the past was limited and measurement uncertainties were largely unknown. This paper reports the developments in METefnet that provide a sound basis for the SI traceability: four new primary standards for realising the water mass fraction were set up, analysed and compared to each other. The operation of these standards is based on combining sample weighing with different water vapour detection techniques; cold trap, chilled mirror, electrolytic and coulometric Karl Fischer titration. The results show that an equivalence of 0.2 % has been achieved between the water mass fraction realisations and that the developed methods are applicable to wide range of materials.

Keywords: Moisture; traceability; Loss-on-drying, Karl Fischer, oven drying

1 Introduction

Moisture content of solid materials is important in a vast range of applications. The presence of water influences in pharmaceuticals, carbon-fibre composites, polymers, food powders, biomass, paper, and many others – during processing, storage and use. In drying and combustion processes water has major effect on energy consumption/generation due to its large latent heat of evaporation. Thus, significant advancements in energy efficiency can be achieved by improving moisture control. In various production processes, water influences active ingredients, mechanical quality, product stability and shelf life. Errors and inconsistencies in moisture measurement and control lead to increased wastage and decreased process speed and throughput.

Reliable measurement and quantifying the reliability in moisture measurements pose various challenges. The most fundamental one is the measurand itself: applying a measurement method you may measure water content alone while with another one you actually measure water and other liquid or volatile contents. For example, results obtained with the widely recognised loss-on-drying methods take into account water and other volatiles while only water is determined with the coulometric Karl Fischer titration method. Also, disseminating traceability and estimating uncertainty are challenging in moisture measurements. Overall there is a need in the moisture field to reduce dependence solely on method-based standardisation of procedures, moving instead towards outcome-based verification of measurement results.

A 3-year European research project METefnet was started in 2013 to develop well-defined principles, methods and equipment for establishing and disseminating SI traceability to measurements of moisture in solids. The project addresses a wide range of materials relevant to industrial production of pharmaceuticals, polymers, foodstuff, animal feed, biomass, wood products, and others [1].

This paper focuses in the development of SI unit realisations for moisture measurements carried out in the METefnet project. As the target was new primary standards, an emphasis was put to the determination of mass of water in a sample and its uncertainty without reference to other moisture standards. The paper reports two new primary measurement setups, upgrade of a commercial evolved water vapour analyser to a primary standard and a new method for obtaining more comprehensive uncertainty estimation and finding optimal operation parameters with an oven equipped coulometric Karl Fischer titrator. Finally, these

methods are compared to each other and to conventional moisture standards and the potentiality of the SI traceability based on calibrations and reference materials in quality assurance of moisture measurements is discussed.

2 Background

When determining gravimetrically the moisture content (w_{mc}) applying loss-on-drying methods (LoD) the result is obtained as a percentage of its wet mass (wet basis) or dry mass (dry basis):

$$w_{mc,wet} = \frac{m_w^*}{m_m} \times 100 \% = \frac{m_w^*}{m_w^* + m_d} \times 100 \% = \frac{m_m - m_d}{m_w^* + m_d} \times 100 \% \quad (1)$$

$$w_{mc,dry} = \frac{m_w^*}{m_d} \times 100 \% = \frac{m_m - m_d}{m_d} \times 100 \% \quad (2)$$

where: m_w^* = mass of water and other volatiles determined as the mass loss during drying of a material sample, m_m = mass of the material sample before drying and m_d = mass of the material sample after drying. This method is most widely recognized for moisture determination for its simplicity and fundamentality. In practical realisations of reference measurements, a sample is dried by heating in an oven (often called as the oven-drying method) but also vacuum drying is applied. However, the duration and the conditions in each part of the process (weighings, handling the sample, heating in an oven, pressure, cooling down) affect significantly on the results. Because of differences in water evaporation when heating, water adsorption when cooling, chemical decomposition etc. with different materials, a large number of standardised methods with different heating temperatures and durations for different materials have been set up in industry to obtain acceptable level of reproducibility. A review of LoD methods and relevant standards is presented in [2]. Although the repeatability and reproducibility are included in the uncertainty analysis of the moisture content measurements, bias effects due to non-aqueous volatiles are usually not known and taken into account in the analysis of measurement results. To remove this shortcoming and to reduce the material dependency new primary standards were developed in the METefnet project.

Coulometric Karl Fischer titration (cKF) provides another fundamental approach to moisture determination. When measuring moisture in solids, the titration setup needs often to be equipped with a sample oven. In this case, the method is analogous to LoD but instead of measuring mass loss we measure the actual amount of water evaporated from the sample (n_w) and determine finally the water mass fraction (w_w):

$$w_w = \frac{n_w M_w}{m_m} = \frac{m_w}{m_m} \quad (3)$$

Here m_w and M_w are the mass of evaporated water and the molar mass of water, respectively. As a dimensionless quantity, water mass fraction is often given in % (that is equivalent with g/100 g) or g/kg. The uncertainty sources of the cKF method are rather well known but quantitative information on the actual uncertainty contributions is very limited in the literature [3]. In particular, effects related to the sample oven temperature and extraction time needed further consideration. In this project, a procedure was developed for finding the optimal values of these parameters.

3 New primary standards for water mass fraction

3.1 New oven-drying primary standard setups

To investigate and demonstrate the feasibility of water mass fraction as a well-defined measurand in moisture measurements two new primary standards were developed and validated at the Centre for Metrology MIKES of VTT Technical Research Centre of Finland Ltd (VTT MIKES) and the Danish Technological Institute (DTI). In these systems, the actual water mass fraction is determined by combining the oven-drying method realised in well controlled environment with hygrometer-based and water-trap-based water vapour detection, respectively. The contributions of non-aqueous volatiles and residual water after drying to the measurement result are minimised and included in the uncertainty

The VTT MIKES standard was designed for relatively large samples. The maximum volume and mass of a sample are 9 dm³ and 400 g, respectively. Figure 1a shows the principle of operation of this system: A sample cell, a monitoring capacitive humidity sensor, a cold trap, a flow controller and an air pump form a closed air circulation loop. The water vaporised by heating in the sample cell is extracted from the

circulating air by condensation in the cold trap. Throughout the measurement process, the dew-point temperature of the cold trap outlet air is constant, which ensures stable and well-controlled air supply to the sample cell. Platinum resistance thermometers in the cell and close to it are used for monitoring thermal conditions of the sample during heating, and the capacitive humidity sensor is used to monitor the drying process. As illustrated in Figure 1a, air flushes the sample when passing through the sample cell enhancing homogenous drying of the sample. The mass loss of the sample is determined by weighing the sample cell with the sample before and after drying without opening the cell. Similarly, the mass gain of the cold trap is determined by weighing the trap before and after the drying the sample. A single balance is used for all the weighings. The balance is calibrated regularly using standard weights traceable to the Finnish National Standard of mass. The water mass fraction in the sample is determined by dividing the mass gain of cold trap by the original mass of the sample. If there are non-aqueous volatiles in the sample, the determined mass loss of the sample cell is slightly larger than the mass gain of the water trap. In practice, however, full water selectivity is not achieved with the cold trap method. To take this into account in the water mass fraction measurement, chemical analysis is applied to a water sample taken from the cold trap. Combining the uncertainties of weighings, the measurement process and the determination of non-aqueous volatiles, the relative expanded uncertainty of the measured water mass fraction at the 95 % confidence level is 2 % to 3 % depending on the sample properties (chemical matrix, sample size and water mass fraction level). The SI traceability is realised to mass through the calibrated balance. Further technical details of the system are given in [4].

In the DTI standard, the amount water evaporated from the sample during drying is continuously monitored with a pair of hygrometers and a pressure gauge. The combination of these two independent humidity detection systems allows discrimination of water from other volatiles possibly evaporated from the sample during heating. As illustrated in Figure 1b, dry air is supplied to a measurement cell where a sample is dried by heating. The flow rate of the dry air is measured with a laminar flow meter. The amount of water in the air exiting the sample cell is calculated from the measured air pressure, dew-point temperature and flow rate. The total amount of evaporated water is determined by integrating over the whole drying period. This value is then finally divided by the original mass of the sample to obtain the water mass fraction. The traceability to SI is realised through calibrations of the pressure gauge, the hygrometers, the flow meter and the balance

used for weighing the original mass of the sample. In this system, the relative expanded uncertainty (95 % confidence level) can be as low as 1.5 %. The DTI standard (Figures 1b) is described in detail in [5]. In both the VTT and the DTI primary standards the measurement chambers are much larger than what is common in commercially moisture analysers, allowing representative measurement on most industrial products without having to pretreat the sample with a risk of contaminating it.

Figure 1. Schematic drawings of the primary standards for water mass fraction developed at a) VTT MIKES and b) DTI. Balances used for weighing the sample cells and the cold trap are not shown here.

3.2 Primary standards based on an evolved water vapour analyser and a coulometric Karl Fischer titrator

The primary realisation at the National Physical Laboratory (NPL) is based on a commercial evolved water vapour analyser in which a sample of up to 2 g is flushed with dry gas during heating and the evaporated water is determined using a phosphorus pentoxide humidity sensor. The sensor passes an electrolytic current according to the amount of water in the carrier gas, in moles per unit time, and the current is integrated over the time of release of water, to find the total amount. To obtain water mass fraction in the material sample, the initial mass of the sample is measured using a calibrated balance. Traceability of the water content was established through a novel approach: by substituting the sample carrier gas with a defined flow of reference gas of known water content, traceable to primary humidity standards. The rate of delivery of water vapour to

the sensor, in moles per unit time, was evaluated from the dew point of the reference gas, converted to amount fraction, together with the measured volumetric flow rate, pressure and temperature of the reference gas. In addition the measurement of current was traceably calibrated. (see Figure 2). Thus, the traceability to SI is obtained through traceable measurements of sample mass, flow rate, temperature and dew-point temperature. More detailed information about the NPL standard is given in [6].

Figure 2 A new approach for providing the SI traceability of an evolved water vapour analyser through humidity standards developed at NPL.

In a coulometric Karl Fischer titrator the amount of water moles is determined from the electric current used for I_2 production in a titrator cell, as I_2 is consumed at a stoichiometric ratio of 1:1 per each molecule of water. University of Tartu (UT) carried out a comprehensive uncertainty analysis for a commercial cKF titrator equipped with an oven sample processor (Figure 3). In this system, a sample of up to a few grams is heated in a sealed glass container. Dry nitrogen flushes the sample and transports the evaporated water to the titrator cell. The water mass fraction of the sample is determined from the net electric charge applied to the titrator for iodine production and the initial mass of the sample according to Eq. (3). The mass of water is integrated over the heating time. The analysis showed that the most important parameters are oven temperature, the current between indicator electrodes and titration speed. In particular, too low oven temperature causes incomplete release of the water within the sample while too high temperature may lead to sample decomposition. For finding the optimal values of the oven temperature and the extraction time, a procedure based on series of measurements with different temperature change rates was developed by UT. [7]. With the UT system, the traceability is obtained through a gravimetric standard solution, several of

which were analysed to ensure the reliability of the results. It is prepared by mixing high purity methanol and water. Methanol is used as a solvent in the Karl Fischer reagent and does not interfere with the reaction. Knowing the mass of both the added water and the methanol (along with its water mass fraction), the water content of the solution and its associated uncertainty were calculated. The relative expanded uncertainty (95 % confidence level) of 1.8 % was demonstrated by an inter-laboratory comparison.

Figure 3 Schematic representation of the measurement system used at UT.

4. Equivalence of different water mass fraction measurement techniques

In order to serve as an ultimate source of traceability for water mass fraction defined by Eq. (3), results obtained with a primary standard should be equivalent to result obtained with any other primary standard of the same quantity. As four different techniques and traceability routes to realisations of SI base units have been applied in the primary standards introduced in this paper, it is essential to investigate their equivalence to each other. For this purpose, inter-laboratory comparisons with five different types of solid materials were carried out. They were selected to cover a wide range of moisture levels (from 0.2 % to 40 %) and several relevant industrial applications (plastic manufacturing, biomaterials and biomass energy production, pharmaceuticals and food powder manufacturing) and to provide a good stability. To obtain further information about the equivalence with conventional reference moisture measurement techniques, the comparisons were extended to include also conventional LoD and cKF methods used at several other national metrology institutes , namely Istituto Nazionale di Ricerca Metrologica (INRIM) in Italy, Instituto

Nacional de Tecnologia Industrial (INTI) in Argentina, National Institute of Metrology (BRML) in Romania, National Institute of Metrology (NIMT) in Thailand, TÜBİTAK Ulusal Metroloji Enstitüsü (TUBITAK) in Turkey, National Institute for Standards (NIS) in Egypt and Ural Research Institute for Metrology (UNIIM) in Russia. The materials used in the comparisons were: polycaprolactone polymer (about 0.2 %), wood pellets (about 8 %), α -D-lactose monohydrate (about 5 %), calcium oxalate monohydrate (about 12 %) and sodium succinate dibasic hexahydrate (about 40 %). The polymer samples with a granule size of 3.5 mm (average diameter) were delivered to the participants in 40 ml glass vials (approximately 25 g of polymer per vial) with PTFE lined screw caps and wrapped with parafilm to limit diffusion of water to and from the sample during transportation. The wood pellets were cylindrical with a length of 10 – 20 mm and a diameter of approximately 10 mm. Pellet samples of about 200 g were delivered to the participants in plastic bags with diffusion barriers. The α -D-lactose monohydrate and calcium oxalate monohydrate were purchased from Acros Organics in 2 kg quantities packaged in plastic bottles and in 500 g quantities packaged in glass bottles respectively. Sodium succinate dibasic hexahydrate was purchased from Sigma-Aldrich packed in 500 g quantities packages in plastic bottles. The samples were from the same batch to avoid potential batch to batch variation in water content, and they were distributed in the original supplier's packaging without breaking the seals.

Figures 4 to 8 summarise the results of the inter-laboratory comparisons. As the focus of the analysis in this paper is in the equivalence between different techniques, the results are arranged in the figures according to the applied techniques. Identifications of the techniques are:

O-cKF = coulometric Karl Fischer titrator with a sample oven

ewva = evolved water vapour analyser

LoD = oven drying system

TGA = thermogravimetric analyser

MB = moisture balance (i.e. a commercial moisture analyser comprising of a balance and a sample heater integrated in a single unit).

The primary standards presented in the previous sections are labelled with 'prim.' on horizontal axes. UNIIM applied also vacuum drying technique, which is identified with 'vacuum' in Fig. 5. Identifications of

the laboratories are given in parentheses. Although multiple results are shown for most of the measurement techniques, each result within single comparison was obtained with different measurement equipment.

Figure 4 Results obtained with polycaprolactone polymer samples applying different measurement techniques. Each result was derived with different measurement equipment. Error bars show estimated uncertainties at about 95 % confidence level.

Figure 5 Results obtained with wood pellets applying different measurement techniques. Each result was derived with different measurement equipment. Error bars show estimated uncertainties at about 95 % confidence level.

Figure 6 Results obtained with α -D-lactose monohydrate applying different measurement techniques. Each result was derived with different measurement equipment. Error bars show estimated uncertainties at about 95 % confidence level.

Figure 7 Results obtained with calcium oxalate monohydrate applying different measurement techniques. Each result was derived with different measurement equipment. Error bars show estimated uncertainties at about 95 % confidence level.

Figure 8 Results obtained with sodium succinate hexahydrate applying different measurement techniques. Error bars show estimated uncertainties at about 95 % confidence level.

Figures 4 to 7 show that the results obtained with the primary standards agree within the estimated uncertainties indicating a good equivalence between the techniques and the practical realisations. The agreement was poorer with the sodium succinate dibasic hexahydrate (Figure 8), which is probably related to residual water but needs to be studied further. When analysing all results, we can identify several conventional measurement systems providing results that are not consistent with others. In these cases, the uncertainty estimations did not probably take sufficiently into account error sources in the applied procedures. However, the overall equivalence between the different measurement techniques is good. LoD measurements were not successful with the polymer samples due to very low moisture level and possibly non-aqueous volatiles but cKF and ewva showed a good agreement. The agreement between water specific and non-water specific methods indicate low concentrations of non-aqueous volatiles in the pellets and powders used as the sample materials. With wood pellets, conventional O-cKF method tends to show lower water mass fraction due to short heating time. However, applying the procedure based on series of measurements with different temperature change rates (see Section 3.2) also this technique provides equivalent results.

In the comparisons, each laboratory applied its own procedures for handling samples, performing measurements and analysing the measurement uncertainty. The uncertainty analyses were carried out according to GUM [8]. The stability and homogeneity of the samples were found good. A comparison of the uncertainty estimations for different measurement systems showed that they varied significantly from each other due to very different kinds of techniques and procedures.

5 Discussion

In the development of the new primary standards presented in this paper, it was essential to identify the well-defined quantity and its unit. There is a need to avoid ambiguity due to non-aqueous volatiles; and to avoid confusion between mass-based and volume-based quantities, and between expression as fraction (of total wet material) and ratio (relative to dry matrix material). In view of these points, , we have used water mass fraction as the primary measurand: it is well defined in terms of an SI base quantity (mass), and mass fraction is included in the SI brochure published by BIPM [9]. To enable easy comparison with conventional moisture measurement methods, the results in this paper are reported percentages. However, to avoid confusion between quantity values and relative uncertainty values, we recommend distinguishing clearly between “percent of value” and “percentage points”. Greater clarity can be gained by expressing water mass fraction results as a quotient of mass units, e.g. g/kg, g/100 g etc. A full definition of the measurand also identifies both the analyte (with might be water, or alternatively moisture including all volatiles) and the matrix or background material.

This paper reports new measurement facilities that are considered to be primary standards because they measure or realise a well-defined moisture quantity with well characterised uncertainty. Both their chemical and physical processes are well understood, resulting in low uncertainty estimates. To enhance dissemination the traceability from these primary standards, the METefnet project also developed transfer standards [10-12], certified reference materials [13], simulations [14] and uncertainty estimation methods [15-17] for disseminating SI traceability in the field of moisture measurements. In addition, a new

calibration method was developed for disseminating traceability to surface moisture meters. However, further research is needed to enable the dissemination of traceability from primary standards down to online measurements in industry.

6 Conclusion

This paper reports the development of the primary standards for establishing SI traceability in the measurements of moisture in solid materials. Two new primary standard systems were constructed, an evolved water vapour analyser was upgraded to a primary standard instrument and a new measurement procedure and a comprehensive uncertainty analysis were developed for a coulometric Karl Fischer titrator with sample oven. The inter-laboratory comparison results show that the uncertainty estimations carried out for the primary standards are realistic and that a relative uncertainty of 2 % with the coverage probability of 95 % was achieved in terms of water mass fraction. The comparisons were carried out using polymer, wood and powder samples with moisture levels from 0.2 % to 40 % (2 g/kg to 400 g/kg) but the measurement setups can be applied to measurements of water mass fraction in a wide range of material samples. The extensions of the comparisons to conventional loss-on-drying and coulometric Karl Fischer titrator systems in different laboratories showed no systematic technique-dependent inconsistencies but uncertainty estimations were in some cases found too optimistic.

With the methods and metrology infrastructure developed in this METefnet project, SI traceability with known measurement uncertainties can be established for the first time in various moisture measurement applications. The work reported here has provided a solid basis for establishing SI traceability in moisture measurements with the water mass fraction as the primary measurand. The obtained results form a sound basis for future improvements of in-line measurement techniques and for minimising the method dependency in determining the water content of materials. Further work is needed to provide more efficient methods for industrial measurements. Wide international cooperation is needed to obtain generally accepted principles of SI traceability in moisture measurements and internationally recognised measurement capabilities at national metrology institutes for moisture in various materials.

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