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Status and Strategy for Moisture Metrology in European Metrology Institutes

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Abstract Measurement of moisture in materials presents many challenges, due to diverse measuring principles, sample interactions with atmosphere, and variation in what is measured (either water content alone or moisture including other liquids). Calibrations are variously referenced to published standard methods, primary calibration facilities, or certified reference materials, but each of these addresses limited substances and ranges of measurement. Overall, metrology infrastructure is not as fully developed or coherent for this field as it is for many other areas of measurement. In order to understand the metrology needs and to support developments, several European national metrology institutes (NMIs) have undertaken some collaborative activities. These have included a “cooperation in research” project for sharing of information, a survey of moisture capabilities at NMIs, the formulation of a strategy for moisture metrology at the NMI level, and a funded research project to develop improved metrology for the moisture field. This paper summarizes the information gathered, giving an overview of the status of moisture metrology at NMIs, and it reports a proposed strategy to improve the current situation.

Keywords Certified reference materials · Measurement traceability · Moisture content · Strategy · Water content

1 Moisture Metrology—Background

Measurements of moisture content in materials are of wide-ranging importance in areas such as food, pharmaceuticals, fuels, and construction materials. Moisture adversely affects many materials and products in their dimensional stability, mechanical strength, microbial activity, chemical stability, handling characteristics, thermal properties, and more. These moisture-dependent phenomena have consequences in many areas of industry. Examples are the thermal efficiency of buildings, product quality in foods, pharmaceuticals, agriculture, solid and liquid fuels, concrete, timber, chemicals, and materials processing. Moisture is critical to the performance of many materials with specialized functions, particularly coatings and adhesives, and hence, reliable moisture measurement can also be the key to innovation in a wide variety of areas. The EURAMET (European Association of National Metrology Institutes) roadmap for humidity and moisture [1,2] outlines the metrology needs and high-level drivers such as energy efficiency and climate research.

Moisture measurement is a challenging field, and the measurements needed can vary considerably in scope. In simple cases, the quantity of interest is average or bulk moisture content, while in other cases, it is surface or near-surface moisture. Some users would like to measure the moisture profile in a material and real-time changes in moisture profile, which are far more difficult to measure. Most significantly, some moisture

measurement methods measure water specifically, while others measure all moisture, including other liquids or volatiles. The term “moisture” is widely used with either meaning [3]. In what follows, the term “moisture” is used where it is useful to combine both meanings, but where specifically water is concerned, this is identified. Measurement of water vapor in air is generally not discussed here (although the term “moisture” colloquially covers this). Measurement of water in liquids, such as oils, is not discussed, although some of the issues are similar to those for moisture in solid materials.

Methods of measurement of moisture in solids are diverse. Techniques for these measurements can be classed as “absolute” (direct) or “inferential” (indirect) techniques. The so-called absolute techniques of moisture measurement include Karl Fischer titration [4] and mass loss on drying (thermogravimetric analysis) [5], and these are treated as reference methods. Indirect techniques include electrical conductance, electrical capacitance, near-infrared reflection (NIR), radio frequency and microwave absorption, neutron moderation, and nuclear magnetic resonance (NMR) [6], and these generally require calibration [7].

Despite widespread interest in measuring material moisture content, there is limited measurement traceability infrastructure currently in place. Standards and traceability are slightly more established for “chemical” methods of measurement than for “physical” methods. Measurement traceability of weighings for loss-on-drying measurements is easily implemented, but this can neither account for any incomplete removal of moisture nor distinguish water from other volatiles. Certified reference materials (CRMs) offer a route of measurement traceability that can address these problems, but the limited CRMs available may not be applicable for the material or for the method being used [8].

In view of the measurement challenges and of the limitations of existing traceability, there is scope for improving calibration approaches, to benefit users of measurements. In order to gain a clear view of NMI capabilities and to identify a course of action, a group of NMIs in Europe set up a EURAMET Project for cooperation in the field of measurement of moisture in materials. Within this cooperation, a survey of capabilities was carried out, followed later by the development of a strategy for improving metrology for moisture in materials within Europe.

This paper summarizes the information gathered, giving an overview of the current status of moisture metrology at NMIs, and it reports a proposed strategy to improve the current situation. In what follows, Section 2 gives details of the EURAMET Project 1065 cooperation. Section 3 reports the survey of capabilities for moisture metrology among the Project 1065 participants. Section 4 outlines an international infrastructure for moisture metrology—where measurement of moisture in materials is addressed by a small number of NMIs worldwide, in metrology working groups, in legal metrology, and in published standards. Section 5 reports the development of a strategy for moisture metrology, and Section 6 outlines the resulting funded joint research project [9].

2 EURAMET Project 1065

In 2008, at a time when several NMIs across Europe were beginning to set up new moisture facilities, a EURAMET *Cooperation in research* project was initiated, on the subject “Survey & strategic planning in the field of measurement of moisture in materials” (EURAMET Project 1065) [10]. The project was categorized in the field of thermometry (which includes humidity) because most of the NMIs concerned

were developing interests in moisture in materials as an extension of existing humidity capability, although moisture measurement is often also classed as chemical metrology.

The EURAMET Project 1065 had the agreed objectives of

- Reviewing existing moisture measurement research and standards at participating institutes and other NMIs
- Reviewing moisture measurement needs generally across Europe
- Developing a view of the metrology infrastructure required in Europe for moisture measurements
- Identifying opportunities for information exchange, cooperative working, or secondments between institutes
- Considering how participant expertise might be combined to formulate a proposal on moisture measurement relevant to the theme of energy, or other appropriate subject area, for submission to the European Metrology Research Programme (EMRP).

The Project has continued from 2008 to the present (2015), and membership to date includes Delta (Denmark), DTI (Denmark), INM (Romania), INRiM (Italy), LNE-CETIAT (France), MIKES (Finland), NPL (UK), PTB (Germany), Tubitak-UME (Turkey), VSL (the Netherlands), plus NIS (Egypt) for part of the lifetime of the project.

3 Survey of P1065 Participants

3.1 Survey Questionnaire

A survey was carried out from 2008 to 2010 (and slightly updated after that) to gain an overview of the status of moisture metrology at participating NMIs. The survey covered subjects including moisture facilities and services established, links with stakeholders, organizational structure and staffing, and future work envisaged. The survey questions are listed in Table 1.

The results of the survey give a “snapshot” of the interests and activities of the P1065 participants, who represented most of the EURAMET NMIs actively developing capability in the area of moisture in materials alongside humidity metrology. In addition, it is recognized that a number of NMIs, and other designated institutes (DIs) within Europe, include moisture measurement within the discipline of chemistry, and that numerous NMIs are also active in this field outside EURAMET, as are other bodies such as the International Organization of Legal Metrology (OIML). The survey was not designed to capture these, but they all form parts of the metrology framework for the field. It should also be noted that NMI capabilities are changing relatively rapidly in this area due to the growing recognition of the metrology need, as well as the increasing capacity to address this work in NMI humidity laboratories.

3.2 Survey Responses

Selected results of the survey are as follows.

3.2.1 Level of Establishment of Work in the Moisture Field

There was an even spread between those NMIs in each category of “established,” “developing,” or “in planning,” with staffing levels ranging between “minimal” and two to three persons. Most respondents said their NMI had (or was developing) active links with measurement users in industries, university researchers, moisture instrument companies, or a mixture of all three.

Table 1 Survey questions for participants of EURAMET Project 1065

1.	What is your institute’s general activity in measurement of moisture in materials?
2.	Is the work in planning, under development, established?
3.	What equipment/facilities for moisture measurement are available in your laboratory?
4.	Do you have any activity on reference materials for moisture measurement (developed, characterized, used)?
5.	Do you have moisture modeling capability/expertise?
6.	What amount of staff effort is dedicated to moisture measurement?
7.	Do you offer any moisture measurement services to customers?
8.	Have you participated in any measurement intercomparisons?
9.	Is this area of work in your organization grouped with thermal measurements, chemical measurements, or other?
10.	Does any other institute in your country also have responsibility for moisture metrology and standards?
11.	What you see as the industrial drivers in your country, for moisture measurement in materials?
12.	Do you have active contact with industrial users of these measurements?
13.	Do you have links with any university working in this area?
14.	Do you have contact with instrument companies in this field?
15.	Do you participate in national or international committees in this field?
16.	What is your expectation for NMI work in this field in future? Please give your ideas on: <ul style="list-style-type: none">• Areas for research/collaboration• Mechanism for funding• Long-term vision—what do you see as desirable (and realistic) future developments in moisture metrology
17.	Do you have any interest in secondment of a scientist in this work: <ul style="list-style-type: none">• Inward—visiting scientist to your institute?• Outward to another institute?
18.	Please feel free to add anything else not covered by the questions above

3.2.2 Equipment or Facilities for Moisture Measurement Available in Laboratories

At the time of surveying, there were a variety of NMI moisture facilities available as shown in the chart in Fig. 1. Those in use included classic loss-on-drying method (oven and balance), Karl Fischer titration, integrated thermogravimetric analyzers, and some less-common methods, using evolved vapor, microwave, infrared, and electrical conductivity techniques. Water activity measurement was also listed, although not a direct measurement of water content but of air humidity in equilibrium with a given (moist) material. Overall, the methods in use among respondents represent most of the conventional laboratory approaches to moisture measurement.

3.2.3 Moisture Modeling Capability

A small number of respondents had established limited capability for relevant

modeling—for drying of concrete (DTI), porous media (CETIAT), and 1-D diffusion in laminate and composite materials (NPL).

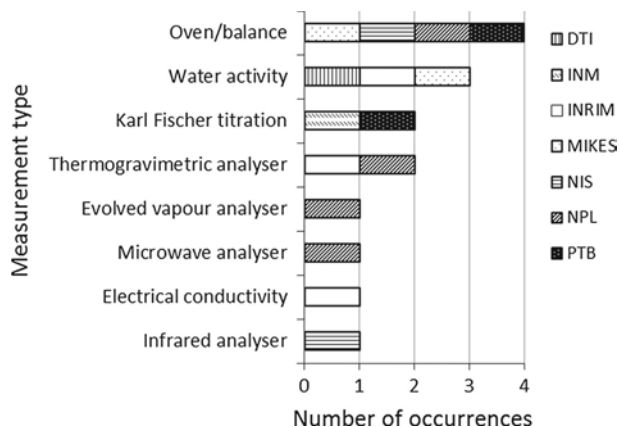


Fig. 1 Chart showing methods of moisture measurement in use at participant NMIs at the time of survey, illustrating the variety of facilities, and showing which are most common

3.2.4 Activity on Reference Materials for Moisture Measurement Development, Characterization, and Usage

Apart from using CRMs for instrument calibration, there was interest in research of moisture reference materials but little activity at the time of the survey. Some limited work had been done on water in oil (INM) and wood samples (INRiM) as potential references.

3.2.5 Moisture Measurement Services to Customers

Three respondents (DTI, INM, and NIS) were actively providing some form of moisture measurement services to customers, and two were just beginning (MIKES, NPL).

3.2.6 Interlaboratory Measurement Comparisons

From the survey, only two formal NMI interlaboratory comparisons were known to have taken place, both COOMET pilot comparisons in grain moisture. These were

- COOMET Project 379/RU/06—“Pilot comparisons in the field of humidity mass fraction measurements in cereal grain and cereal products.” Participants: BelGIM (Belarussian State Institute for Metrology), UMTS (Ukrmetrteststandard, Ukraine), SP (Technical Research Institute of Sweden), PTB (Germany), and UNIIM (Ural Scientific and Research Institute for Metrology, Russia)
- COOMET Project 436/RU/08—“Pilot comparisons in the field of moisture mass fraction measurements in barley cereal grain.” Participants: UNIIM (Russia), BelGIM (Belarus), UMTS (Ukraine), SP (Sweden), PTB (Germany), and KazInMetr

(Kazakhstan Institute of Metrology).

Table 2 Some reported industrial sectors where measurement of moisture in materials is relevant, according to initial survey responses of NMIs

Industrial sectors ?	DTI	INM	INRiM	MIKES	NIS	NPL	PTB
Oil, gas		x					
Solid biofuel				x			
Building and construction	x		x	x			
Wood, wood products		x		x	x		
Paper		x		x			
Food		x			x		
Agriculture (grain)		x			x		x
Soil					x		
Pharmaceuticals			x		x	x	
Plastics						x	
Powders						x	
Moisture instrument manufacturers				x			

3.2.7 Grouping of Moisture Work Organizationally with Temperature and Humidity Metrology, Chemical Metrology, or Other

For those NMIs responding to the survey, moisture metrology was aligned with thermal (or humidity) metrology in four cases and with chemistry in two cases. This balance may be a reflection of the primary contacts for the Project and questionnaire being through a temperature field technical committee. In certain cases, designated institutes were reported to have significant responsibility for the moisture measurement field, such as LCG (UK), and BAM (Bundesanstalt für Materialforschung und -prüfung) and the Max Rubner-Institut (Germany).

3.2.8 Reported Industry Drivers for Moisture Measurement in Materials

Some perceived main industry drivers were reported, although far from an exhaustive list. Table 2 summarizes the responses. Even without being comprehensive, the responses give an interesting flavor of industrial priorities in different respondent countries.

3.2.9 Participation in National or International Committees in the Moisture Field

About half the respondents were active on moisture-related standardization committees at either the national level or internationally, particularly through OIML, on technical committee OIML TC17 SC1 Humidity (covering grain and wood moisture meters).

3.2.10 Future Areas of NMI Moisture Work Envisaged by Respondents

Respondents individually suggested future streams of moisture work in two broad categories: developments in techniques and applications, and developments in NMI roles and infrastructure for the moisture field.

Envisaged moisture measurement techniques and application areas for future development included the following suggestions (in no particular order):

- Biofuels
- On-line real-time moisture measurements by physical methods; radio frequency/microwave/ terahertz/magnetic resonance imaging/nuclear magnetic resonance—variously for research or process use
- Wireless sensor technology for monitoring of moisture on building sites and production of building materials.
- Electrical impedance spectroscopy (EIS) and microwave techniques for wood moisture measurements, and others
- Porous media (especially modeling of these)
- Investigating the relationship between the equilibrium relative humidity (water activity) and the moisture content of materials.

Envisaged directions of development of NMI provision identified by one or more respondents included

- Developing national (primary) standards in some form
- Effective, disseminated traceability for laboratory moisture measurements
- Developing reference or calibration methods for traceable on-line moisture measurements, especially *in situ* reference materials
- Investigating the relationship between the standardized gravimetric reference methods and actual moisture content
- Investigation of influences affecting the reliability of moisture measurements
- Provision of interlaboratory comparisons
- Support of good measurement practice in the moisture field
- Coherent interaction between chemical and physical approaches to moisture metrology and, where relevant, with the legal metrology field

Existing measurement interests (Table 2) do not necessarily imply a need for future research. For example, for grain moisture, measurement methods for legal metrology are well established, and in this context, any benefits of improving techniques might not outweigh the disruption.

It should be emphasized that the survey responses represented only participants in EURAMET Project 1065, mostly working in temperature and humidity metrology areas of NMIs and representing a snapshot of information gathered mainly between 2008 and 2010, with only minor updating after that. Several of the NMIs involved have continued to incrementally develop their moisture measurement capabilities and interests since the survey.

4 Moisture Metrology Infrastructure

In considering the status of moisture metrology, it is useful to understand the roles of certain organizations worldwide. These include NMIs generally (not only those surveyed within the EURAMET Project), organizations overseeing scientific and legal metrology, and standardization bodies, as outlined below.

4.1 National Measurement Institutes

In addition to those surveyed in EURAMET Project 1065, a number of other NMIs have activity and expertise in moisture metrology. These include BAM (Germany), BelGIM (Belarus), KazInMetr (Kazakhstan), KRISS (Korea Research Institute of Standards and Science), LGC (UK), NIST (National Institute of Standards and Technology, USA), NMIJ (National Metrology Institute of Japan), SP (Sweden), UMTS (Ukraine), UME (Turkey), UNIIM (Russia), University of Tartu (Estonia), and others.

4.2 International Metrology Working Groups

Moisture metrology is addressed in working groups within the consultative committee structure of the CIPM (International Committee for Weights and Measures). Since 2006, the humidity working group of the Consultative Committee for Thermometry (CCT) has been tasked with responsibility for the physical metrology of moisture in materials, particularly where this overlaps with humidity metrology. This includes liaising with relevant chemistry experts and working groups in CCQM (Consultative Committee for Amount of Substance—Metrology in Chemistry). Moisture content, being an aspect of chemical composition, falls naturally within the remit of CCQM and is addressed within its Inorganic Analysis Working Group. In 2010, CCQM formed an ad hoc Working Group on “Moisture in Grain” to hold discussions for this area of metrology, to address the growing interest in proposing a key comparison that would support BIPM (International Bureau of Weights and Measures) Calibration and Measurement Capabilities for NMIs in this area of work.

4.3 International Organization of Legal Metrology

The OIML legal metrology infrastructure relates to moisture measurements for trade purposes. In the moisture field, there are two significant moisture-related International Recommendation documents OIML R 92, “Wood-moisture meters—Verification methods and equipment: general provisions” (First published 1989) and OIML R 59 “Moisture Meters for Cereal Grain and Oilseeds” both under the responsibility of TC 17/SC 1 (Humidity). These recommendations cover metrological traceability of measuring instruments, but also concern matters of instrument design and pattern approval, in the context of legal metrology.

4.4 Published ISO and Other Standards

There are hundreds of documentary standards specifying moisture measurement methods, published as ISO, EN, or ASTM standards and by other national standardization bodies. These cover moisture measurement approaches for particular applications, but also numerous reference methods, mostly using mass loss on drying. While each reference method usually specifies elements of best practice, and measurement traceability of some kind, the protocols for sampling, drying, and determining the end point vary

in detail for every different material or field of application. The large number of these different protocols illustrates how very non-standard these reference loss-on-drying methods are.

5 Moisture Strategy

5.1 Moisture Workshop

In 2012, DTI hosted a two-day workshop on moisture in materials. Day 1 was an open session with presentations from industries and NMIs. A number of topics were covered, including industrial measurement problems and priorities in sectors of food, biofuels, and drying technology; moisture work at some NMIs; and a review of the challenges in moisture measurement. The industrial presenters gave examples of the benefits and savings achievable if better moisture measurements were available. A clear perspective emerged of their drivers, which were typically cost savings associated with maximizing utilization and productivity—by improving handling and by minimizing wastage, downtime, or re-work. Quality improvements and carbon reduction were also motivations—less so in some industries.

Day 2 was an NMI strategy workshop for participants of EURAMET Project 1065. The workshop began with an ideas session on the subject of metrology development for moisture in materials, both generally and with a particular view to how a proposal could be made to EMRP 2013 calls for funding proposals in areas of “Industry” or “SI Broader Scope.” The workshop generated a SWOT analysis (strengths, weaknesses, opportunities, and threats) for the potential objective of “a funded metrology infrastructure for moisture in materials, serving industries and users providing traceable and best-practice measurements of moisture in materials.” A summary of the SWOT analysis is shown in Table 3, indicating, for this field, what main opportunities were identified constituting areas or stimuli for potential action. The issues were ranked according to the importance, and this is indicated in the table. From the SWOT analysis, a strategy statement was agreed.

5.2 Moisture Strategy

Based on strongly ranked items in the SWOT analysis, a set of strategic objectives was formed by the EURAMET Project 1065 participants, and then presented to a meeting of the EURAMET Humidity Subfield for comment and approval. The agreed objectives are as follows:

1. The goal of the strategy is the development of a funded metrology infrastructure for moisture in materials, serving industries and measurement users, and enabling traceable and best-practice measurements of moisture in materials.
2. In order to address identified weaknesses in current moisture metrology, work should be proposed that can
 - address moisture measurement traceability problems
 - clarify and disseminate relevant understanding of definitions of water content versus moisture content

- address method dependence of moisture measurements and calibrations
 - involve and collaborate with existing contact instrument companies, who have known interests in this.
3. Any proposed program of work to improve moisture metrology should respond to the challenges posed by
 - existing regulations and procedure-driven standards for moisture calibration
 - difficult measurement problems
 - needs for new solutions to fit existing process control systems
 - measurement users (industries) being change-averse
 - natural reluctance of companies to expose weaknesses in existing techniques
 - the prospect that, with realistic assessment, uncertainties will increase
 - dependence on cooperation of instrument companies for new developments to be adopted.
 4. Progression toward an improved metrology infrastructure for moisture should make use of identified NMI strengths in this context, such as
 - expertise in metrology of humidity and other related fields (e.g., mass and temperature)
 - good calibration capabilities and expertise
 - general metrology infrastructure in place
 - good industry-NMI cooperation (in some cases)
 - past experience of forming funded collaborative projects in EMRP.
 5. In order to succeed in such development of moisture metrology, NMIs will need to improve in areas such as their
 - limited experience and critical mass in the field of moisture metrology generally
 - ability to address industry with relevance “at their level.”

Table 3 Main findings of SWOT analysis from EURAMET Project 1065 Strategy Workshop

Strengths	Weaknesses
++ Good calibration capabilities	-- Limited experience of NMIs in this field generally
++ Metrology infrastructure	-- NMIs can be too high-level in talking to industry
++ Expertise in humidity and other related fields (mass, temperature)	-- NMIs do not yet cater for all sample types or sizes
++ Good industry-NMI cooperation	- Risk of being inward looking
++ Past EMRP experience	- Limited knowledge of industrial control systems
+ NMIs are familiar with diverse approaches	
+ Uncertainty analysis	
+ Can choose to develop instruments, methods or references	
+ Measurement expertise	
+ Modeling expertise	
+ Some good industrial and university contacts	
+ Cost-benefit arguments can be quantified	
Opportunities	Threats
++ Industries recognize there is a problem	-- Hard-to-solve difficult practical problems
++ Definitions moisture-water etc. need to be clarified	-- Industries are change-averse
++ Existing instrument company interests/contacts	-- Companies commercially do not want to expose weaknesses
++ Method-dependence of moisture measurement	-- Declared uncertainties will increase
+ Energy conservation and savings	-- Existing standards-regulations
+ productivity and quality are industrial priorities	-- New solutions must fit existing control systems (industry)
+ Diverse approaches available	-- Instrument companies may not cooperate
+ Moisture and temperature distributions needed	- NMI-industry communication can be poor
+ Novel techniques steam/oil drying	- The “medicine tastes bad”!—solutions may not be palatable to users
+ NMIs outside Europe interested	
+ Key industries may vary nationally	
+ Drying	
+ Energy production	
+ Innovation	
+ Cultural heritage, academia, other stakeholders	
+ Liquid concentration measurement and calibration (including moisture/water content (dry basis) above 100 %	
+ Standards for moisture measurement not available in all areas	
+ CRMs not widely applicable in current form	
+ Destructive testing can be problematic	

Markings from ++ to -- were used to rate the degree of positive or negative impact of issues on the proposition of “a funded metrology infrastructure for moisture in materials, serving industries/users, and enabling traceable and best-practice measurements of moisture in materials.” The strengths and weaknesses are those of NMIs to fulfill the proposition

5.3 Workshop and Strategy Conclusions

The industrial presentations gave some clear ideas of user needs, and the strategy process was very much guided by the industrial perspectives. This led the workshop to consider possible courses of action such as a proposal in the EMRP Industry Call, or a proposal for an EU project in any one of a number of industry sectors where moisture is important. However, in spite of a strong emphasis on discussing industrial drivers for the work, it became clear that the best course of action would be to propose some enabling work in the EMRP “SI Broader Scope” Call, which could uniquely support improvements to metrology infrastructure in the field of moisture in materials. If successful, this metrology infrastructure would underpin other more industry-focused future developments in the field of moisture measurement. As a result of these conclusions, it was decided to propose the Joint Research Project known as *METefnet—Metrology for Moisture in Materials*, which is outlined in the following section.

6 EMRP Joint Research Project METefnet—Metrology for Moisture in Materials

In 2013, the EMRP Joint Research Project *METefnet—Metrology for Moisture in Materials* was set up to develop unambiguous principles, methods, and equipment for establishing and disseminating traceability to the International System of Units (SI) for measurements of moisture in solids by

- developing unambiguous realization methods for water mass fraction and new primary standards based on these methods
- improving the coulometric Karl-Fischer titration-based realization for water amount fraction
- creating effective general principles of SI traceability in the field of moisture measurements
- developing new/adapted CRMs and novel transfer standard instruments to enable dissemination of SI traceability with optimal accuracy
- developing methods for quantifying and reducing the effect of moisture change during transporting and handling samples
- developing a novel calibration facility with SI traceability for surface moisture meters
- developing modeling to include local moisture variations in the uncertainty estimations, and developing uncertainty analysis tools for selected industrial applications and
- establishing a coherent and developed moisture metrology infrastructure in Europe.

The 3-year project brings together the main European national metrology research institutions active in the field of moisture measurements in collaboration with metrology institutes worldwide, accredited testing and calibration laboratories, suppliers of moisture instrumentation and drying equipment, commercial research organizations, university research departments, and companies manufacturing moisture-critical products. More information is given on the project website at www.metef.net [11].

7 Conclusion

The survey of participants in the EURAMET Project 1065 provided an overview of the level of development of moisture metrology at participating NMIs, especially those with moisture work being developed alongside humidity work. Seen together with provision at other NMIs and elsewhere, this gave a picture of the status of the field of metrology for moisture in materials at the time of the survey.

In view of existing metrology provision, and informed by knowledge of some of the industrial needs for support for moisture measurements, a concise strategy document was agreed for the further development of a metrology infrastructure for moisture in materials, serving industries and measurement users, and enabling traceable and best-practice measurements of moisture in materials. This strategy is being pursued through the EMRP Joint Research Project METefnet—Metrology for Moisture in Materials.

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