

VTT TECHNICAL RESEARCH CENTRE OF FINLAND LTD

Centre for Metrology MIKES



**Metrology for Moisture
in Materials**

Measurement of Moisture in Materials: Challenges and on-going European research

Triple M Conference, DTI, Taastrup 20.10.15
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Contents

- Challenges
 - Fundamentals
 - Industrial applications
- On-going European research
- Future

Challenges

- Moisture control is vital for product quality and shelf life of pharmaceuticals, foodstuffs and biomaterials



- Better moisture control allows reduced over-drying:
 - 0.1 % reduction in over-drying could save about 5×10^5 GWh/year in Europe



- Improved moisture control enables higher process speed, e.g. in polymer and wood based material production



Challenges: Fundamentals

- In most moisture measurement applications, Loss on Drying (LoD) is recognized as the ultimate reference for moisture content in solids
- Moisture content values are distorted by **other volatiles** and **varying binding** of water
 - ⇒ inconsistent results
 - ⇒ over 1300 documentary standards
- In measurements of moisture in solids:
 - uncertainties are unknown
 - means to establish traceability links are insufficient/missing



Challenges: Industrial applications

- Laboratory measurements
 - Reference:
 - instrument/procedure validation
 - traceability
 - material specificity
 - Sampling & sample handling
 - Uncertainty
- On-line measurements
 - Calibration
 - Material specific
 - Sampling method (if used)
 - Uncertainty



On-going European research



Metrology for Moisture
in Materials

- METefnet – Metrology for Moisture in Materials 2013 – 2016
 - Focus in fundamental challenges
 - Extension to industrial analysers (research excellence grant)



- HIT – Metrology for Humidity at High Temperatures and Transient Conditions 2015 – 2018
 - Includes development of in-line water activity measurement for industry



Metrology for Moisture in Materials

EMRP
European Metrology Research Programme
■ Programme of EURAMET



The EMRP is jointly funded by the EMRP participating countries within EURAMET and the European Union

TOWARDS EFFECTIVE SI TRACEABILITY FOR MEASUREMENTS OF MOISTURE IN SOLIDS

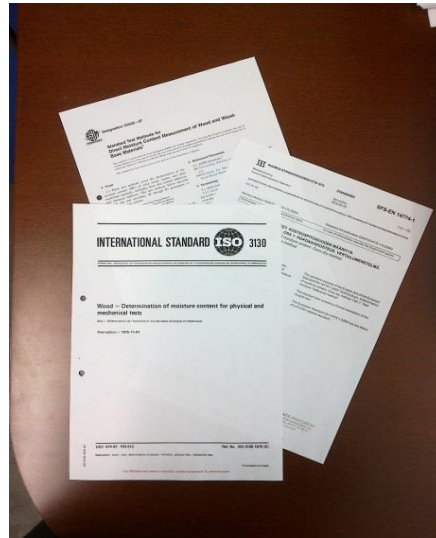
Introduction to the EMRP SIB64 METefnet -project

Vision

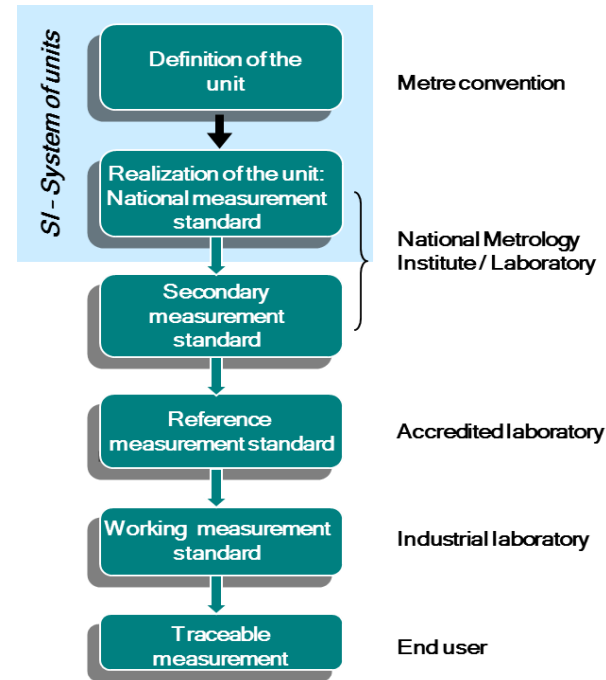
- Quality control of moisture measurements in solids through SI traceability in terms of mass fraction and amount fraction



antiquity



present



future



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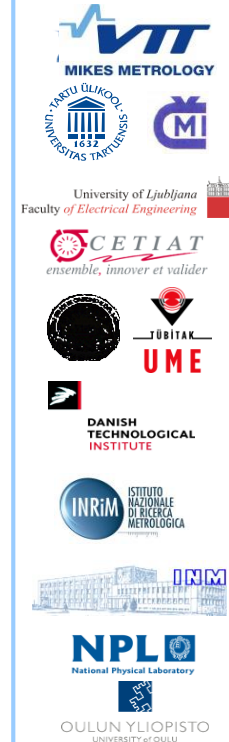
OULUN YLIOPISTO
UNIVERSITY OF OULU



Scope of METefnet

- Principles of SI traceability
- Primary realisations for water mass fraction and amount fraction
- Dissemination from primary realisations
 - Incl. methods for intercomparisons
 - Also, method for calibrating surface moisture meters is included
- Methods for estimating uncertainty
- Materials:

Pharmaceuticals	Polymer/plastic	Foodstuff
Feed	Biomass	Wood based



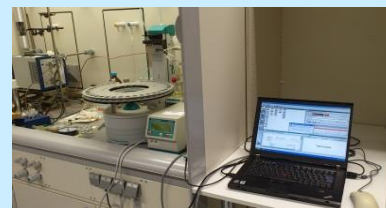
WP1: Realisation of moisture units

New principles and methods for SI unit realisations:

- measurands = true SI quantities
- Loss-on-drying + water detection and improved Karl Fischer
- uncertainties better than 0.5 % to 4 % (incl. **all** factors)
- links to classical measurands

Status:

- LoD + cold trap completed at VTT MIKES
- LoD + chilled mirror completed at DTI
- LoD + electrolytic: validation of a commercial analyser completed at NPL
- cKF with oven have been set up and studied at UT and BRML
- Preliminary comparative measurements with 1) wood pellets and 2) alpha-Lactose and calciumoxalate monohydrate powder
- Final comparisons started in October 2015



Robust basis
for SI
traceability
for moisture
in solids

materials |



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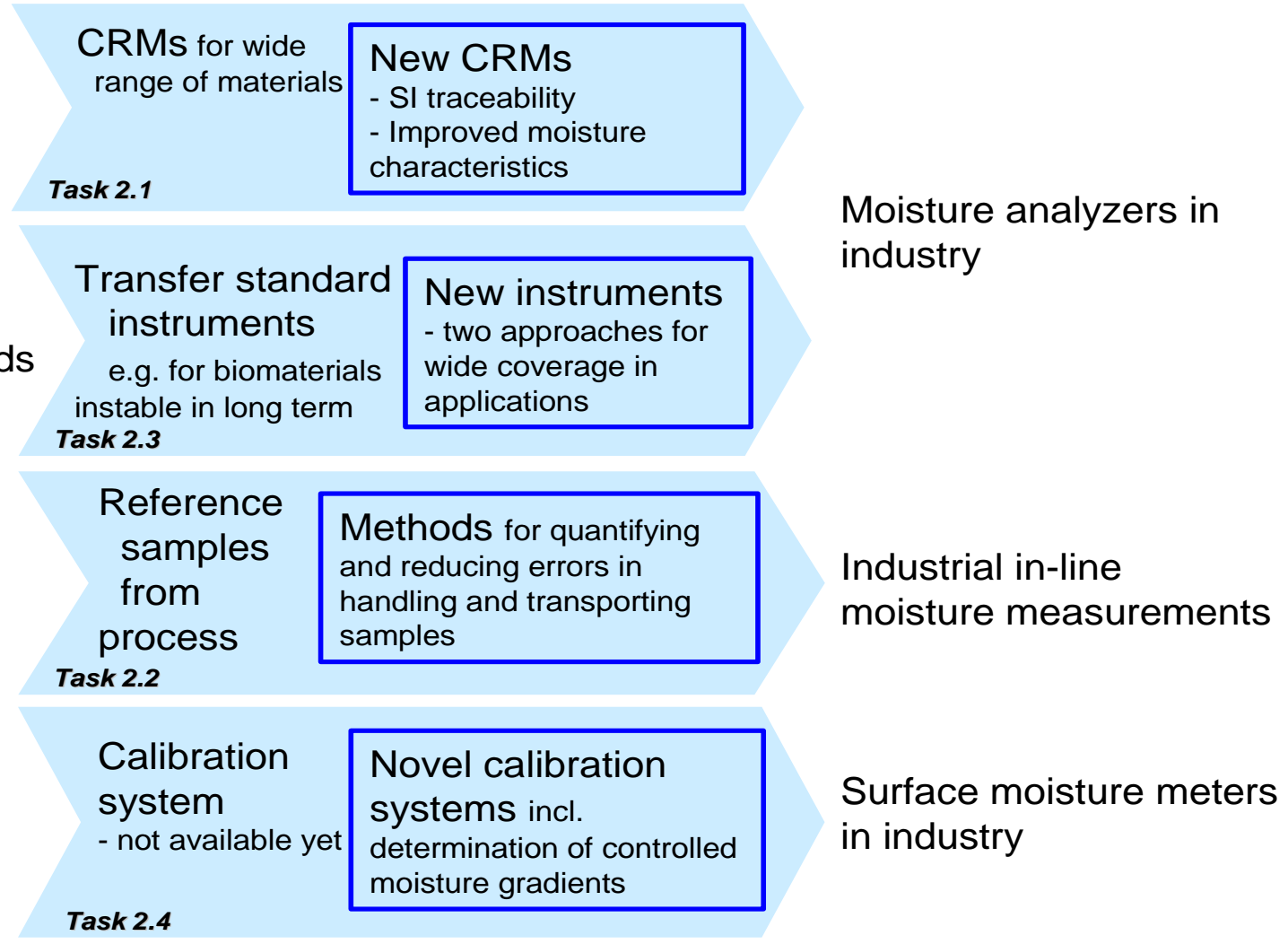


NPL
National Physical Laboratory



WP2: Traceability and dissemination

Dissemination method Development in this JRP



WP2: Traceability and dissemination

Dissemination method

Development in this JRP

CRMs for wide range of materials

New CRMs
- SI traceability
- Improved moisture characteristics

Status:

- Two candidates for CRMs identified and under tests for certification at NPL: calciumoxalate monohydrate (12 %) and di-sodium tetraborate decahydrate (47 %)
- RF/MW instrument development is on-going at CETIAT
- MW resonator + portable humidity generator prototype designed , constructed and tested at INRIM
- UL has designed and evaluated NIR sensor for measuring surface moisture in polymer elements

system
- not available yet

systems incl.
determination of controlled moisture gradients

Task 2.4

analyzers in

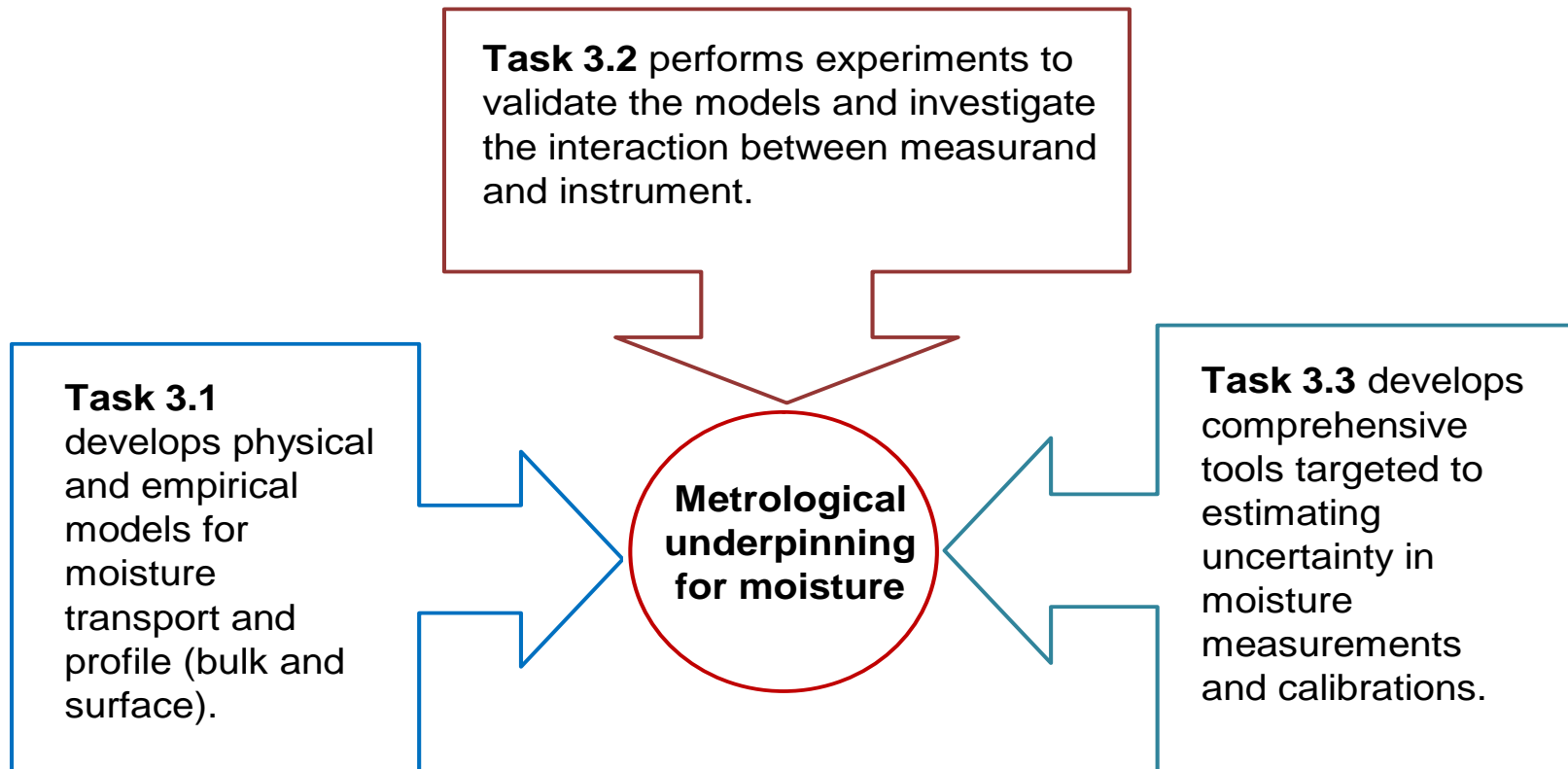
on-line

measurements

moisture meters in industry



WP3: Metrological underpinning for moisture



WP3: Metrological underpinning for moisture

Task 3.2 performs experiments to validate the models and investigate the interaction between measurand and instrument.

Status:

- UNICLAM has been successfully developing numerical modelling for estimating the effect of moisture transportation on uncertainty in moisture measurements.

Task 3.1 develops and implements a moisture transport model for moisture profile (bulk and surface).

Task 3.3 develops extensive metrological competence to support metrology in measurements and calibrations.



REG2(UOULU): Research in moisture measurement instruments

Status:

- Measurement have been made with
 - commercial microwave analyser
 - commercial NMR analyser
 - LoD according to EN 14774-1
- First results were published in IMEKO World Congress 2015



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Partners:

Short Name	Organisation legal full name	Country
MIKES	Mittatekniikan Keskus	Finland
BRML	Biroul Roman de Metrologie Legala	Romania
CETIAT	Centre Technique des Industries Aéronautiques et Thermiques	France
CMI	Cesky Metrologický Institut Brno	Czech Rep.
DTI	Teknologisk Institut	Denmark
INRIM	Istituto Nazionale di Ricerca Metrologica	Italy
NPL	NPL Management Limited	UK
TUBITAK	Türkiye Bilimsel ve Teknolojik Arastırma Kurumu	Turkey
UL	Univerza v Ljubljani	Slovenia
UT	Tartu Ülikool	Estonia
UNICLAM	Università degli Studi di Cassino e del Lazio Meridionale	Italy REG1
UOULU	University of Oulu	Finland REG2

Collaborators:

	Raute Oyj Mecano Business Unit, Finland
	Valmet Automation Inc., Finland
	Intertek Pharmaceutical Services Manchester, ITS Testing Services, Ltd, United Kingdom
	UCL School of Pharmacy, United Kingdom
	Henkel Slovenija d.o.o., Slovenia
	Seltek Ltd, Turkey
	The Ural Research Institute for Metrology, Russian Federation
	Korea Research Institute of Standards and Science, Republic of Korea
	NIS - National Institute for Standards, Egypt
	Universidad Politécnica de Cartagena, Spain



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Further information:

- www.metef.net
 - Incl. presentations of the workshop at CETIAT 18.6.2015
- Events:
 - Workshop summarising outcomes of METefnet in Copenhagen 11 – 12 May 2016
 - TEMPMEKO 2016 Conference in Zakopane, Poland 26 June to 1 July 2016



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Future

- After completing METefnet, European metrology institutes will provide traceability and expert services for customers in moisture measurements.
- We are looking for possibilities to research and development addressing the challenges in industrial applications.
 - All expressions of interest and proposals are welcome!!



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