



BSI Standards Publication

Conservation of cultural heritage — Methods of measurement of moisture content, or water content, in materials constituting immovable cultural heritage

National foreword

This British Standard is the UK implementation of EN 16682:2017.

The UK participation in its preparation was entrusted to Technical Committee B/560, Conservation of tangible cultural heritage.

A list of organizations represented on this committee can be obtained on request to its secretary.

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© The British Standards Institution 2017.
Published by BSI Standards Limited 2017

ISBN 978 0 580 84028 9

ICS 97.195

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 April 2017.

Amendments/corrigenda issued since publication

Date	Text affected
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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 16682

March 2017

ICS 97.195

English Version

**Conservation of cultural heritage - Methods of
measurement of moisture content, or water content, in
materials constituting immovable cultural heritage**

Conservation du patrimoine culturel - Méthodes de
mesurage de la teneur en humidité, ou teneur en eau,
de matériaux constituant un patrimoine culturel
immatériel

Erhaltung des kulturellen Erbes - Verfahren zur
Bestimmung des Feuchte- bzw. Wassergehalts in
Materialien des unbeweglichen kulturellen Erbes

This European Standard was approved by CEN on 25 December 2016.

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Ref. No. EN 16682:2017 E

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European foreword

This document (EN 16682:2017) has been prepared by Technical Committee CEN/TC 346 “Conservation of Cultural Heritage”, the secretariat of which is held by UNI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2017, and conflicting national standards shall be withdrawn at the latest by September 2017.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

The specific field of cultural heritage is characterized by particular needs and in most cases the existing standards devised for normal materials cannot be applied. The moisture content, or the water content, in materials is of primary relevance for the preservation of cultural heritage. High content can be very damaging (e.g. salt dissolution and mobilization, fungal infestation, corrosion, swelling) as well as low content (e.g. salt crystallization, shrinkage, wood cracking) or alternating high/low content. It is therefore important to determine and control this variable to assess the risk of damage and take preventive conservation measures.

Different methods exist to measure moisture content, or water content, in modern building materials, based on different physical or chemical principles but most of them are not applicable to cultural heritage and need to be adapted to this aim.

Generally, non-destructive methods are recommended but their accuracy may be limited. In turn, the most accurate methods require sampling and can only exceptionally be used. Readings taken with non-destructive methods may not be comparable especially because they are expressed in different units. The interpretation of measurements may be obscured by a number of factors (e.g. material, salts, temperature) to which the methods are subject.

This European Standard considers and specifies characteristics, operative methodologies, pros and cons of all methods of measurements and establishes a uniform presentation of data and units. It is addressed to anyone who needs to measure or interpret readings of moisture content, or water content, in building materials (particularly masonry and wood), and in general to whoever is responsible for the preservation and maintenance of heritage buildings.

1 Scope

This European Standard is aimed to inform and assist users in the choice and use of the most appropriate method to obtain reliable measurements of the moisture content, or water content, in wood and masonry (including brickwork, stonework, concrete, gypsum, mortars, etc.) in the specific case of the built cultural heritage.

It provides a basic framework to take and interpret this kind of measurements on the above cultural heritage materials that have undergone weathering, pest attack, salt migration or other transformations over time.

It specifies four absolute methods (i.e. gravimetric, Karl Fischer titration, azeotropic distillation and calcium carbide); explains their characteristics, pros and cons, and gives specifications for the transformation of readings into the same unit to make measurements taken with different methods comparable.

It specifies the three principal relative methods (i.e. electrical resistance, capacitance, and relative humidity in equilibrium with the material), pointing out their characteristics and uncertainties when used in the field of cultural heritage.

In addition, it provides an informative overview of ten other relative methods, their characteristics, pros and cons.

It gives specifications for the calibration of the various methods. It also compares the above methods in relation to their accuracy, sampling requirement, sample size, laboratory or field use, and other problems encountered in the field of cultural heritage to prevent instrument misuse, reduce uncertainties and avoid reading misinterpretation.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 374-1, *Protective gloves against chemicals and micro-organisms - Part 1: Terminology and performance requirements*

EN 420:2003+A1:2009, *Protective gloves - General requirements and test methods*

EN 455-1:2000, *Medical gloves for single use - Part 1: Requirements and testing for freedom from holes*

EN 772-10:1999, *Methods of test for masonry units - Part 10: Determination of moisture content of calcium silicate and autoclaved aerated concrete units*

EN 837-1:1996, *Pressure gauges - Part 1: Bourdon tube pressure gauges - Dimensions, metrology, requirements and testing*

EN 1428:2012, *Bitumen and bituminous binders - Determination of water content in bituminous emulsions - Azeotropic distillation method*

EN 13183-1:2002, *Moisture content of a piece of sawn timber - Part 1: Determination by oven dry method*

EN 13183-2:2002, *Moisture content of a piece of sawn timber - Part 2: Estimation by electrical resistance method*

EN 13183-3:2005, *Moisture content of a piece of sawn timber - Part 3: Estimation by capacitance method*

EN 15758:2010, *Conservation of Cultural Property - Procedures and instruments for measuring temperatures of the air and the surfaces of objects*

EN 15898:2011, *Conservation of cultural property - Main general terms and definitions*

EN 16085:2012, *Conservation of Cultural property - Methodology for sampling from materials of cultural property - General rules*

EN 16096:2012, *Conservation of cultural property - Condition survey and report of built cultural heritage*

EN 16242:2012, *Conservation of cultural heritage - Procedures and instruments for measuring humidity in the air and moisture exchanges between air and cultural property*

EN ISO 10304-1:2009, *Water quality - Determination of dissolved anions by liquid chromatography of ions - Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate (ISO 10304-1:2007)*

EN ISO 11461:2014, *Soil quality - Determination of soil water content as a volume fraction using coring sleeves - Gravimetric method (ISO 11461:2001)*

EN ISO 13130:2011, *Laboratory glassware - Desiccators (ISO 13130:2011)*

EN ISO 14911:1999, *Water quality - Determination of dissolved Li⁺, Na⁺, NH₄⁺, K⁺, Mn²⁺, Ca²⁺, Mg²⁺, Sr²⁺ and Ba²⁺ using ion chromatography - Method for water and waste water (ISO 14911:1998)*

EN ISO 15512, *Plastics - Determination of water content (ISO 15512)*

ISO 760:1978, *Determination of water — Karl Fischer method (General method)*

ISO 3567:2011, *Vacuum gauges — Calibration by direct comparison with a reference gauge*

ISO 5272:1979, *Toluene for industrial use — Specifications*

ISO 5280:1979, *Xylene for industrial use — Specification*

ISO 7183:2007, *Compressed-air dryers — Specifications and testing*

ISO 11465:1993, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method*

ISO 16979:2003, *Wood-based panels — Determination of moisture content*

ISO Guide 34:2009, *General requirements for the competence of reference material producers*

ISO/IEC Guide 98-3:2008, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

EN ISO/IEC 17025:2005, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:2005)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 15898:2011 and the following apply.

3.1

absolute method/measurement

measuring method whose readings can be expressed in terms of SI units

3.2

active sensor

sensor that needs some electrical power supply to operate

3.3

atmospheric pressure

barometric pressure

pressure is the force per unit area exerted by the air column above the measuring point

Note 1 to entry: This is expressed in hPa (hectopascal).

[SOURCE: EN 16242:2012, 3.2]

3.4

bound water

in masonry: water molecules physically or chemically bound to the material; bound water cannot dissolve soluble substances. In wood: water bonded in the cell walls; it is responsible for shrinkage/swelling

3.5

crystallization water

hydration water

water molecules that are part of a hydrated crystal or molecule

3.6

desiccator

equipment in which a desiccant is used to maintain the air as close as possible to the absolutely dry condition

[SOURCE: EN 322:1993, 4.3]

3.7

desiccant drying

method in which drying is obtained with a continuous flow of dry air (from compressed air), or with moisture absorption from a highly hygroscopic substance used as desiccant

[SOURCE: EN 322:1993, 4.3]

3.8

destructive

causing destruction or damage to cultural heritage materials

3.9

dew-point hygrometer

instrument for measuring the temperature at which a cooled parcel of air becomes saturated with water vapour

[SOURCE: EN 16242:2012, 3.4]

3.10

dew-point temperature

temperature to which air is cooled at constant pressure and constant water vapour content in order for saturation to occur

Note 1 to entry: This is expressed in degrees Celsius (°C).

[SOURCE: EN 15758:2010, 3.6]

3.11

dry air

atmospheric air without water vapour

[SOURCE: EN 16242:2012, 3.6]

3.12

dry matter

whole of the substances which do not volatilize under the conditions of drying specified in this standard

[SOURCE: ISO 1026:1982, 2.1]

3.13

equilibrium moisture content

EMC

moisture content at which a material neither loses nor gains moisture from the surrounding atmosphere at given relative humidity and temperature levels

Note 1 to entry: This is expressed in g/kg as the ratio of the mass of water contained in the material and the dry mass of the same material.

[SOURCE: EN 16242:2012, 3.8]

3.14

external sealed box

ESB

box with five thermally insulating and impermeable faces and one missing face, fixed to the surface of a material to create an air pocket in hygrometric equilibrium with the material

3.15

fibre saturation point

moisture content at which no free water is present in wood, while the cell walls are saturated with bound water

3.16

free water

in masonry: liquid water inside pores. In wood: water contained in the cell cavities. It can dissolve soluble substances

3.17

hygrometer

instrument measuring relative humidity

Note 1 to entry: It generally comprises a sensor, which is set in equilibrium with the air, and a system that transforms the signal from the sensor into humidity readings.

[SOURCE: EN 16242:2012, 3.11]

3.18

invasive

requiring the entry of a probe, or other instrument into a cultural heritage material, for diagnostic purposes

3.19

irreversible relative humidity sensor

sensor in which reached relative humidity levels remain permanently marked

3.20

minimally destructive

minimally invasive

damage caused, or the sample taken, are very small and not easily recognizable, so that it can be considered acceptable, or justified in view of the advantages expected from the analysis

3.21

moisture content

MC

amount of water in the material, as determined in accordance with a gravimetric method specified in this European Standard; the method may include the volatile organic compounds that may be lost when the material is heated at moderate temperatures

Note 1 to entry: The MC is expressed as a mass fraction in percent (%).

3.22

oven drying

drying method obtained with a ventilated oven at temperature $103^{\circ}\text{C} \pm 2^{\circ}\text{C}$

[SOURCE: EN 322:1993, 4.2]

3.23

passive sensor

sensor that does not need any electrical power supply to operate

3.24

proxy

material or a specimen that is a substitute or a replacement for something else, under the assumption of identical behaviour of the material under investigation

3.25
relative humidity
RH

ratio of the actual vapour pressure of the air to the saturation vapour pressure

Note 1 to entry: The RH is expressed in percent (%).

[SOURCE: EN 15757:2010, 3.9]

3.26
relative method
relative measurement

measuring method whose readings cannot be expressed in terms of SI units and should be expressed in relation to something else, kept as a reference

Note 1 to entry: The scale depends on the specific choice of the reference; it can also be expressed as a fraction of a given value.

3.27
sample

portion of material, ideally representative, removed from the cultural property for scientific investigation

[SOURCE: EN 16085:2012, 3.2]

3.28
temperature

temperature read on a thermometer which is exposed to air in a position sheltered from direct solar radiation or other energy sources

Note 1 to entry: The temperature is expressed in degrees Celsius (°C).

[SOURCE: EN 15758:2010, 3.1]

3.29
thermometer

instrument to measure temperature which comprises a sensor which is placed in thermal equilibrium with the air (if it measures the air temperature) or the surface, sometimes a probe that contains and protects the sensor, and a system that transforms the input from the sensor into an output expressed in degrees Celsius (°C)

[SOURCE: EN 15758:2010, 3.20]

3.30
vacuum drying

drying method obtained by vacuum pumping to lower the atmospheric pressure to 4 ± 2 hPa in the drying chamber

3.31
vacuum gauge

instrument for measuring gas or vapour pressures that are less than the prevailing atmospheric pressure

[SOURCE: ISO 3529-3:2014, 2.1.2]

3.32

water content

WC

amount of water in the material, as determined in accordance with the Karl Fisher titration specified in this European Standard; this method may include crystallization water

Note 1 to entry: The WC is expressed as a mass fraction in percent (%).

4 Symbols and abbreviations

EFD	Evanescent-Field Dielectrometry
EMC	Equilibrium Moisture Content
ESB	External Sealed Box
IAEA	International Atomic Energy Agency, Vienna
KF	Karl Fischer
KFT	Karl Fischer titration
C-KFT	coulometric KFT
V-KFT	volumetric KFT
MC	Moisture Content expressed on dry basis
MC _w	Moisture Content expressed on wet basis
NIR	Near Infrared
NIRS	Near Infrared Spectroscopy
NMR	Nuclear Magnetic Resonance
OV-KFT	Oven-vaporization KF titration
C-OV-KFT	coulometric oven-vaporization KF titration
V-OV-KFT	volumetric oven-vaporization KF titration
RH	Relative Humidity
SI units	International System of units
SRXTM	Synchrotron-based X-ray Tomographic Microscopy
TDR	Time-Domain Reflectometry
TGA	Thermogravimetric Analysis
VOC	volatile organic compounds
WC	Water content expressed on dry basis
WC _w	Water content expressed on wet basis
ϵ	dielectric constant
m_H	initial (moist) mass of the test sample
m_0	mass of the sample after drying
m_w	mass of water extracted from the sample

5 Moisture and water content in materials

5.1 Moisture content

5.1.1 General

The moisture content (MC) is defined as the ratio between the mass of the water extracted from a material sample and the mass of the dry sample expressed in percent (%), i.e.

$$MC = \frac{m_w}{m_0} \times 100 = \frac{m_H - m_0}{m_0} \times 100 \quad (1)$$

where m_H is the initial mass of the moist sample, m_0 the mass of the sample after it has been dried, and $m_w = m_H - m_0$ the mass of water extracted from the sample (ISO 16979:2003, EN 13183-1:2002, EN 772-10:1999 and EN ISO 11461:2014). MC ranges from 0 (completely dry sample) to a value determined by the material's porosity at saturation.

The MC is specifically measured by gravimetry (ISO 16979:2003) or other methods that comply with the basic Formula (1). From the practical point of view, the oven-drying method assumed as a reference may include volatile organic compounds ¹⁾ (VOC) that may be released when the material is heated at moderate oven temperatures.

The MC is expressed as a ratio of homogeneous quantities and for this reason it is independent of the unit used to express m_H and m_0 . However, in the measuring report the use of the SI unit for mass (i.e. g) is recommended. The moisture content shall be reported to the nearest 0,1 % stating that it is by dry weight.

5.1.2 Dry versus wet mode

The MC defined in Formula (1) is expressed on dry basis, i.e. is referred to the mass m_0 of the sample after it has been dried (e.g. using the gravimetric method in A.2). Other methods exist (e.g. azeotropic distillation in A.5, calcium carbide test in A.6) that allow a direct determination of m_H and m_w but destroy the sample so that it is impossible to determine m_0 with a balance. For these methods the moisture content is usually expressed on wet basis (MC_w) taking the mass of the moist sample m_H as a reference, i.e.:

$$MC_w = \frac{m_w}{m_H} \times 100 \quad (2)$$

Of course $MC > MC_w$. The difference between MC and MC_w makes the comparison between readings obscure. For clarification, this standard recommends the use of the MC on dry basis for all methods.

When a method destroys samples, one of the two following options shall be used:

Option 1: Calculate $m_0 = m_H - m_w$ and proceed with Formula (1).

Option 2: Determine MC_w on wet basis, i.e. Formula (2), and then apply the transformation to dry basis by using Formula (3) that relates MC to MC_w :

$$MC = \left(\frac{MC_w}{100 - MC_w} \right) \times 100 \quad (3)$$

The inverse relationship is

¹⁾ Gases produced by decomposition, reduction and/or other chemical reactions should be excluded.

$$MC_w = \left(\frac{MC}{100 + MC} \right) \times 100 \quad (4)$$

5.1.3 Gravimetric versus volumetric mode

When the MC is measured, it can be expressed as a percentage of the sample, as follows:

- a) gravimetric mode, i.e. the MC is the ratio between the mass of the removed water and the mass of the dried sample (dry basis) or the moist sample (wet basis). The result is expressed in % by mass.
- b) volumetric mode, i.e. the MC is the ratio between the volume of the removed water (its mass in g equals the volume in cm³) and the actual volume of the moist sample. The result is expressed in % by volume.

In order to avoid confusion, it is necessary to specify if the percentage is derived from gravimetric or volumetric determinations. The gravimetric mode is independent from the presence of voids or gradients; the volumetric mode varies with the material density and presence of voids. This is a problem when the material density is not homogeneous. It may vary from point to point (e.g. for deterioration, pests or voids) or change over time, as typically occurs in cultural heritage materials.

5.2 Water content

The water content (WC) consists of all kinds of water present in a material, i.e. free water, bound water and crystallization water, but excludes VOC.

WC is measured by Karl Fischer titration (A.4) that is specific for water (e.g. free water, bound water, crystallization water) (ISO 760:1978).

Although the common use is to express the water content in terms of wet basis, this standard recommends the use of the WC on dry basis for homogeneity reasons, to make comparable the readings with other methods. The WC shall be expressed in terms of dry basis as specified for MC in the previous clause, but substituting WC to MC.

5.3 Comparison between moisture content and water content

When the sample includes VOC or crystallization water in hydrated minerals, the loss of mass after drying is not only due to moisture. The difference in readings when different methods are used may be determined by the presence of VOC or crystallization water (both apparently increase the evaluation of m_w extracted from the sample), the sensitivity of the measuring method to these factors and the operating temperature. One should have clear understanding which type of water is detected and what is the purpose of the measurements: e.g. MC: moisture exchangeable with the environment (wood shrinkage/swelling; soluble salt mobilization, mould), or WC: the total water content including crystallization water. In some cases it is possible to calculate the difference to compare results or to remove the contribution of VOC or crystallization water. In another cases a precise determination of WC may be preferable to a method specific for MC (or vice-versa) because smaller samples are required.

The situation is more complex in the case of electromagnetic forces (e.g. capacitance) or where a microwave beam penetrates inside a material (e.g. microwave, evanescent-field dielectrometry, time-domain reflectometry). The electromagnetic forces or the microwave beam will interact with the H₂O molecules because of their polar structure. However, the interaction is different if the water molecule is part of the material structure (e.g. cellulose chain, crystallization water) or is free to respond to the excitation. As a consequence, all H₂O molecules will respond to the signal, but the strongly bound H₂O molecules will respond more weakly, and the free moisture will respond more strongly. In such a case the instrument reading interpretation is not straightforward.

In the following guidelines MC or WC will represent the amount of water available in a moist sample which can be extracted and measured with one of the measurement methods specified in this standard.

In order to assist the user, each method will specify whether it measures MC or WC, in case this may be relevant. Such methods are considered “absolute” because the readings they provide are representative of the MC, or the WC, and do not depend on anything else (except for observational uncertainties).

When a measurement method cannot be calibrated with a suitable reference standard, the absolute value of the MC (or the WC) remains unknown, but it may be useful to make relative comparisons from one measurement point (at a given time) to another measurement point (at subsequent times) to detect gradients in space or time. Such methods are considered to provide “relative” readings.

6 Absolute and relative methods

6.1 Absolute methods

Absolute methods are measuring methods that comply with the basic definition in Clause 5 and whose readings can be expressed in terms of SI units. The absolute methods are based on the extraction of water from the material sample (including VOC or crystallization water). When sampling is possible, the amount of water in the sample is directly determined by gravimetry, titration, distillation, or gas pressure and is expressed as a percentage of the material sample mass. Sampling is required to measure m_H and m_O or m_w ; the third variable is calculated as a difference between the other two.

The absolute methods considered in this standard are listed in Table 1. The apparatus, the operative methodology, the specific problems met in the field of cultural heritage and the normative recommendations are specified in Annex A.

Table 1 — Absolute methods considered in this standard, as specified in Annex A, and physical or chemical principles used for the determination of m_H , m_O , m_w

Clause	Method	Physical or chemical principle			Specific for
		m_H	m_O	m_w	
A2	gravimetric	weight	desiccation	(calculated)	MC
A4	KF titration	weight	(calculated)	titration	WC
A5	azeotropic distillation	weight	(calculated)	distillation	MC
A6	calcium carbide	weight	(calculated)	gas pressure	MC

All the absolute methods except calcium carbide are designed for laboratory analyses, and only in exceptional circumstances could be used for on-site measurements

6.2 Relative methods

Relative methods are measuring methods that do not directly comply with the basic definition in Clause 5 but refer to other physical mechanisms. Readings cannot be expressed in terms of SI units, but in relation to something else, which used as a reference. Relative methods are especially used to detect differences over space and time, e.g. to assess the relative change of the MC (or WC) in a given point compared to other point without considering the absolute value. The main advantages are that the relative methods afford real time measurements, are suitable for fast on-site observations and especially most of them are non-destructive or minimally invasive.

The relative methods considered in this standard are listed in Table 2. Specifications, related problems and recommendations concerning the field of cultural heritage are reported in Annex B (normative) and Annex C (informative).

Table 2 — Relative methods considered in this standard, as specified in Annexes B and C

Clause	Method	Physical principle
B.2	electrical resistance	Ohm's law
B.3	capacitance	dielectric capacity of water
B.4	equilibrium RH	equilibrium between RH and MC
C.2	microwave	interference of the water molecule with the electromagnetic radiation (microwave frequency)
C.3	evanescent-field dielectrometry (EFD)	interference of the water molecule with the electromagnetic radiation (microwave frequency)
C.4	time-domain reflectometry (TDR)	interference of the water molecule with the electromagnetic radiation (radio frequency)
C.5	nuclear magnetic resonance (NMR)	magnetic moment and resonant frequency of the water molecule
C.6	near infrared spectroscopy (NIRS)	reflection of the near infrared radiation
C.7	ultrasound pulses	speed of ultrasound pulses
C.8	thermography	thermal emission of bodies

Relative methods are based on indirect detection and evaluation of moisture inside a building material. Generally, all relative readings might be transformed into % mass after a calibration with respect to a reference material, performed by comparison with a gravimetric method, or Karl Fischer titration. In the case of materials characterized by VOC or crystallization water, the comparison of MC with WC needs correction. Although automatic conversion or conversion tables provided by manufacturers are useful, any calibration is difficult because of changes that old materials might have undergone, due to ageing, weathering agents, chemical or biological attack.

Further methods that require special safety requirements are listed in Table 3 and reported in Annex D.

Table 3 — Methods that require special safety requirements, as specified in Annex D

Clause	Method	Physical principle
D.2	X-rays	absorption of X-rays
D.3	gamma rays	absorption of gamma rays
D.4	neutron scattering	scattering of neutrons

6.3 Comparison between absolute and relative methods

Absolute and relative methods used to evaluate the MC, or WC, are based on different physical or chemical principles and the readings may not be comparable. For instance, the gravimetric method (A.2) and the Karl Fischer titration (A.4) provide the total mass of water molecules included in a sample, irrespective of how they are spatially distributed within the sample. On the other hand, electrical resistance (B.2) varies with the moisture content and the presence of soluble salts. The capacitance (B.3) and microwave (C.2) methods also respond to the total amount of water molecules, but the output depends on other factors too, including how the molecules are distributed inside the material, the signal being higher for molecules close to the sensor. This constitutes a serious difficulty in the case of uneven distributions and subsurface moisture gradients. The method of relative humidity in equilibrium with the material based on sensors or proxies (B.4) is affected by the hygroscopic features of the materials,

and the presence of salts. This method only provides accurate results in the lower part of the MC range up to the point when some liquid water appears in the pores. Briefly, all methods are affected by a number of disturbing factors that have, case-by-case, different relevance.

For the above uncertainties some instruments providing relative results (relative methods) give readings in arbitrary units useful to assess relative changes in MC or WC. Other instruments, to meet practical needs, provide readings in percentage (%) after calibration with some selected materials. In most cases, such an instrument has reasonable reliability only in establishing if the MC or the WC in a particular part of a cultural heritage material is higher or lower than in another part, i.e. relative readings only. Some manufacturers calibrate their instruments with known modern materials to provide a scale in absolute readings, useful for commercial or building purposes. However, such a calibration may not be appropriate for cultural heritage materials and needs to be verified case-by-case.

7 Taking and handling samples

In principle, all methods based on the analysis of samples are invasive (a portion of material is removed) and destructive (some damage is caused) and should be avoided. All the absolute methods require the extraction of a small portion of material representative of the general properties and composition of the whole building material under investigation. The mass of the sample needed for the measurement, however, may vary from method to method, as specified in Annex A, Table A.1 and Figure A.2.

When sampling is necessary, it shall be made by people with skills and knowledge of the cultural property and shall follow a precise sampling plan in compliance with EN 16085:2012. EN 16085:2012 provides a general methodology and criteria for sampling cultural property materials for their scientific investigation. However, it is not specifically concerned with addressing moisture exchange; for this the following specifications shall be considered.

The sampling plan shall consider that moisture may considerably vary between the surface and deeper layers, in relation to present and past weather events, permanent or temporary shields etc. For the above reasons the sampling plan shall be adapted to the circumstances, and related to the present and past weather (at least 30 days) and the season.

Samples may undergo exchange of moisture with the environment during manipulation and weighing; for this reason samples shall be kept in sealed containers and protected against temperature changes. Small samples are preferred to reduce damage to cultural heritage, although too small sample sizes may lead to increased risk of error. Samples taken from the surface or subsurface layer are not representative of the material interior.

The sampling equipment and the containers for samples shall be inert, i.e. made of aluminium or glass on which the material has no action. They shall be clean, dry, perfectly sealed in order to avoid any exchange of moisture with the environment during the transport or storage.

Direct contact with fingers may also lead to a weight increase due to transfer of grease and/or sweat. Samples shall be handled with impermeable laboratory or medical gloves (e.g. latex, vinyl, polychloroprene, polyisoprene, nitrile, polyurethane, styrene butadiene) complying with EN 420:2003+A1:2009, EN ISO 374-1 or EN 455-1:2000, to avoid noxious contact with hands and avoid absorbing moisture.

8 Calibration

8.1 General

Calibration establishes a metrological traceability through other measurement standards, measuring instruments, or measuring systems having a negligible measurement uncertainty, in compliance with EN ISO/IEC 17025. Calibration is constituted of a chain composed of the measurement unit (i.e. the

instrument), the measurement procedures and the reference material from which the calibration diagram, curve, or table has been produced. This requires operating under the same conditions.

8.2 Instrument calibration

8.2.1 General

All measuring instruments shall be provided with a calibration certificate issued by a laboratory in compliance with EN ISO/IEC 17025. The calibration certificate shall report the correction to the readings of the instrument under calibration and the associated uncertainty in agreement with the ISO/IEC Guide 98-3:2008. The certificate establishes the verified accuracy class of a measuring instrument. The calibration of instruments shall be checked frequently. If it is found to have slipped beyond the manufacturer's specification, the instrument shall be recalibrated.

8.2.2 Calibration for moisture content

Calibration for MC measurements shall be made with the gravimetric method using the most suitable drying method (A.3).

8.2.3 Calibration for water content

Calibration for WC measurements shall be made with the Karl Fischer titration (A.4). Although V-KFT is an absolute method, the titre of the reagent still has to be determined using a standard calibrant (mainly sodium tartrate dihydrate as powder). On the other hand, for C-KFT a titre determination is not required. However, C-KFT requires at least a check of the whole titration system by analysing a certified reference material (performance qualification).

NOTE Taking samples for calibration is a destructive practice and this can constitute a serious problem for cultural heritage materials. However, it can be considered that the calibration needs only a limited number of samples that can justify its use in obtaining a calibration curve. Sampling can be made with the permission and under the strict supervision of a qualified conservation professional.

8.3 Reproducibility

8.3.1 Instruments for absolute measurements

The instruments of the absolute methods in Annex A shall be calibrated in order to provide precise and repetitive MC or WC measurements.

8.3.2 Instruments for relative measurements

The instruments of the relative methods in Annex B and in the informative Annex C and Annex D shall be calibrated in order to provide repetitive readings under the same operating conditions and the same particular reference material. The result of the calibration shall be a calibration certificate including a calibration diagram, a calibration curve, or a calibration table for the reference material. The instrument readings can be easily interpreted and are affected by low uncertainty if the material under investigation is homogeneous and its characteristics are very similar to the reference material used for calibration, e.g. modern building materials or aged materials preserved in good conditions. If the material is not homogeneous or differs from the reference material used for calibration, or has changed physical, chemical or mineralogical properties over time, the readings are affected by a larger uncertainty that makes the interpretation obscure. This may be the case of aged and deteriorate cultural heritage materials. For instance, most relative methods are affected by the presence of internal pores, voids, moisture gradients, deliquescent salts or polar materials.

8.3.3 Comparison between absolute and relative methods

The comparison between absolute and relative methods is easy for a modern building material but may be difficult for cultural heritage materials. In the general case, each calibration is valid for the specific