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

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National foreword

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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

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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of an identical text or by endorsement, at the latest by September 2017, and conflicting national standards shall be withdrawn at the latest by September 2017.

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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°C ± 2°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.

$$MC = \frac{m_w}{m_0} \times 100 = \frac{m_H - m_0}{m_0} \times 100 \tag{1}$$

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.:

$$MC_w = \frac{m_w}{m_H} \times 100 \tag{2}$$

Of course MC > MC_w. The difference between MC and MC_w makes the comparison between readings obscure. For clarification, this standard recomends 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:

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

The inverse relationship is

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

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

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 cm^3) 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 H_2O 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 H_2O molecules will respond to the signal, but the strongly bound H_2O 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_0 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_0, m_w

Clause	Method	Physical or chemical principle			Specific for
		m_H	m_0	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

case study for which calibration has been made and may hold for a limited time. It can only be extended with difficulty to other apparently similar cultural heritage materials.

Some examples to elucidate the difference between absolute and relative methods, or to assist the user in the choice of the most convenient method in relation to the material conditions and other key variables (e.g. accuracy needed, possibility of removing samples, allowed sample size) are reported in Annex A (Figures A.1, A.2 and A.3) and Annex B (Figures B.1, B.2 and B.3).

9 Use of existing European Standards concerning modern building materials

A number of European Standards exist on specific methods to measure the moisture content in modern building materials, as listed in Clause 2. However, the fact that a standard exists does not mean that the method is directly applicable to the specific field of cultural heritage as the above standards may need to be modified, or their use may require modifications, or their application could be limited or even impossible.

The absolute methods are destructive or invasive, being based on extracting and analysing samples. Relative humidity in equilibrium with the material (drilled cavity) is also destructive. Electrical resistance is minimally destructive when small pins are used. Most of the relative methods are non-destructive and suitable for non-aged materials, but cannot be applied to quantitative moisture measurements when a material is aged, degraded and its physical or chemical properties have changed, e.g. by salt contamination, increased porosity or insect tunnelling, fungal infestation, surface coverings for past treatments. In these cases, commercial instruments supplied with absolute scales by the manufacturer should be considered indicative rather than absolute.

In principle, all methods and standards that may cause damage to heritage materials should ethically be rejected; however, some exceptions can be made, under advice and strict supervision of a qualified conservation professional, when it is absolutely necessary to know the MC (or the WC).

In the field of cultural heritage, the existing standards for modern building materials may be applied only to a limited number of cases, within some specific restrictions, or with certain different clauses. This standard makes a comparison of the different methods, specifies what use should be made of these standards and what changes should be applied for the specific field of cultural heritage.

10 Test report

The test report shall be made in compliance with EN 16085:2012 and EN 16096:2012. In addition, the report shall specify:

a) the method and the instrument used for the measurement of the MC (or the WC), making reference to the respective clause and subclause of this European Standard;

b) if material samples were taken or not;

   1) if samples were taken (absolute methods): the report shall include all details about the method used for sampling (in accordance with EN 16085:2012); the number of samples, the exact location of the sampling points and any other detail useful for sample identification;

   2) if no samples were taken (relative methods): the report shall clearly indicate the exact location of the points where instrument readings were taken;

c) the result of the measurement;

   1) for absolute methods: the MC (or the WC) measurements shall be expressed as a percentage (%) of the dry mass of the test portion;

2)   for relative methods: the instrument readings shall be reported with indication of the scale;

d)   the environmental information necessary to interpret data, e.g. date, hour, air temperature; surface temperature of the material; RH of the air (temperature and RH measured following EN 15758:2010 and EN 16242:2012). The report shall also specify the general weather conditions of the day and the 30 days before the measurement.

e)   in the case of masonry, the report shall report the anions and cations present in the material; they shall be measured with ion chromatography (EN ISO 14911:1999; EN ISO 10304-1:2009).

Annex A
(normative)

Absolute methods

A.1 Generalities

This annex is normative and establishes the operational procedures that shall be applied for heritage materials and gives a guideline concerning the main difficulties met with observations and the readings interpretation.

The methods included in this annex provide absolute readings and have been chosen for their applicability and good comparability of results.

The absolute methods require the extraction of samples. This is a destructive practice and shall be used only in exceptional circumstances and always in compliance with EN 16085:2012. The number and size of samples shall be considered in order to obtain a reliable result while respecting the cultural value of the heritage material. Samples shall be taken and handled as recommended in Clause 7. When samples need to be dried, the flow diagram in Figure A.1 assists in the choice of the drying method.

Table A.1 and Table A.2 provide a general overview and a comparison between absolute methods, will report the characteristics of the various methods, their experimental uncertainties, the sample size and some notes concerning their specific use with wood or masonry.

Figure A.2 and Figure A.3 constitute two fundamental decision trees, based on the above Tables. Figure A.2 illustrates the flow chart for the choice of the most convenient absolute method in relation to the possibility of removing small, medium or large samples. Figure A.3 illustrates the flow for the choice of the most convenient absolute method in relation to the required accuracy level.

Table A.1, Table A.2, Figure A.2 and Figure A.3 are useful to assist the user in the choice of the most convenient method and in the interpretation of readings of MC (or WC) in the field of cultural heritage.

A.2 Gravimetric method

A.2.1 General

The gravimetric method is a direct, absolute method of estimating the moisture content (including the volatile compounds released at moderate temperatures) in materials. It provides the amount of moisture removed from the sample during drying.

The method is based on the determination of the loss of mass of a sample weighed before and after drying as measured with an analytical balance. The loss of mass can be associated with the water mass removed from the sample and is commonly expressed as a percentage of the final mass (of the dried sample).

The method is independent of the material type. The general procedures are specified in EN 13183-1:2002 for wood, EN 772-10:1999 for masonry, ISO 11465:1993 for minerals. Cultural heritage materials, however, may require specific drying procedures as specified in Clause A.3.

The gravimetric method is particularly relevant because it is able to provide precise quantitative results. It should be used as a reference for calibrating instruments from other methods, provided material of the same type as the material under investigation, or very similar to it, is used.

The main limitation of this method is that it is destructive and generally not suitable for cultural heritage. In principle, the total weight loss after drying is determined, not just the moisture content. In

addition to moisture, other volatile components of the sample and/or decomposition products may be determined. As a consequence, the most suitable drying method (as described in A.3) shall be considered and applied.

A.2.2 Apparatus

a)   Analytical balance: shall have at least 4 digits (0,1 mg resolution), readable to 0,1 % of the mass of the test sample. The balance shall be accurately calibrated, placed in a room with stable relative humidity and temperature and properly operated;

b)   weighing boats (or other sample containers): shall be inert, airtight, non-corrodible, of capacity suitable to the mass of the test sample;

c)   dryer: ventilated oven or other drying devices as specified in A.3;

d)   temperature, pressure or humidity controlling instruments as appropriate (as specified in A.3.4, A.3.5 and A.3.6).

A.2.3 Procedure

The gravimetric method is based on the following steps:

a)   a moist test sample shall be taken from the building material, as in Clause 7;

b)   the initial mass of the moist test sample m_H shall be determined with the analytical balance;

c)   the moist test sample shall be dried with one of the drying procedures described in A.3 until constant weight has been reached;

d)   the mass of the sample after drying m_0 shall be determined with the analytical balance;

e)   the mass of water m_w extracted from the sample shall be determined as a difference $m_H - m_0$:

f)   the ratio m_w/m_0 shall be computed and expressed in percentage (%) of the dry mass, as specified in Clause 5;

g)   the test report shall be produced in accordance with Clause 10.

A.3 Drying procedures in the gravimetric method

A.3.1 General

This drying method specified by EN 13183-1:2002 for wood, or EN 772-10:1999 for masonry, is based on oven drying, at (103 ± 2) °C. However, cultural heritage materials may include materials not resistant to oven-drying temperature; thus the weight loss might be due to something other than just moisture.

In some cases materials may be heat resistant but covered with varnish or impregnated with organic substances that volatilize when exposed to oven temperature (e.g. polychrome wood or wood impregnated with wax). In such cases the change of mass of the oven-dried sample includes the loss of volatile organic compounds (VOC) and does not correspond to the water loss.

In general, crystallization water is released from 170 °C to 200 °C but some hydrated minerals included in masonry may release it at lower temperatures.

In order to avoid misleading results, three alternative methods for desiccation are recommended (i.e. oven, vacuum and desiccant drying), depending on the response of the material sample to temperature

and/or pressure. Reducing the pressure would allow the applied drying temperature to be reduced (e.g. as low as 40 °C).

Sample drying shall be made with one of the following clauses: A.3.2 and A.3.6 when the sample is resistant to oven temperature; Clauses A.3.3 to A.3.5 shall be used to avoid material decomposition, reduction and/or other chemical reactions or material transformations that may affect the dry sample weight. This is illustrated in the flow diagram in Figure A.1.
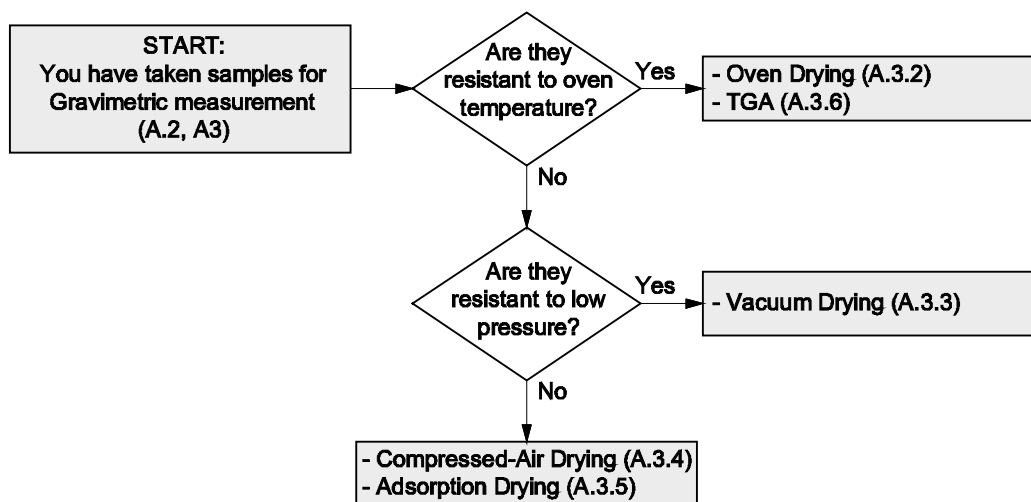
Figure A.1 — Flow diagram to assist in the choice of the drying method. Numbers in brackets refer to the Clause numbers

A.3.2 Oven drying

A.3.2.1 General

This drying method applies to heat resistant materials, thermostable up to (103 ± 2) °C (e.g. untreated wood, limestone, mortar, bricks, concrete).

A.3.2.2 Apparatus

The ventilated oven shall have the following characteristics:

a) drying temperature shall be (103 ± 2) °C under normal atmospheric pressure;

b) the temperature shall be uniform at all points in the oven;

c) the oven ventilation shall consist of a slow current of dry air entering at a rate of 10 000 cm^3 hr^{-1} to 40 000 cm^3 hr^{-1}, as appropriate;

d) the air introduced into the oven shall be dry (e.g. using a drier, or compressed air, or nitrogen);

e) the forced air ventilation shall not spread the sample in the oven, with the risk of causing a significant weight loss.

A.3.2.3 Drying procedure

a) The sample shall be dried in a ventilated oven at (103 ± 2) °C until a constant mass has been reached. Constant mass is defined as being when consecutive readings made at 4-h intervals differ by less than 0,5 %;

b) the cooling down from oven to room temperature shall be performed in a controlled manner, the same for all samples, using a stopwatch.

NOTE   Even at oven temperature not all water is removed because equilibrium is established between the samples and the relative humidity of the surrounding air. For this reason one can assume a residual moisture content in the oven-dried sample of up to 0,5 %.

A.3.3 Vacuum drying

A.3.3.1 General

This drying method applies to materials not resistant to oven temperature, but resistant to low pressure.

A.3.3.2 Apparatus

a) Vacuum pump;

b) vacuum-desiccator for general laboratory purposes in compliance with EN ISO 13130:2011;

c) vacuum gauge in compliance with ISO 3567:2011.

A.3.3.3 Drying procedure

a) A sample shall be taken in accordance with Clause 7;

b) the air pressure in the chamber shall be lowered with a vacuum pump pumping down to (4 ± 2) hPa (i.e. below the water vapour saturation pressure at ambient temperature);

c) the chamber pressure shall be monitored with a vacuum gauge;

d) the system shall be kept at ambient temperature above the freezing point of water (i.e. $t > 0$ °C);

e) once the sample has been dried, further operations and calculations shall follow EN 13183-1:2002.

A.3.4 Compressed-air drying

A.3.4.1 General

This drying method applies to materials neither resistant to oven temperatures nor resistant to low pressures.

In this method, the sample is dried placing it inside a cell where a continuous flow of clean dry air, obtained from compressed air, removes the moisture released by the sample.

This method requires longer times to dry the sample in comparison with oven or vacuum drying, but does not cause thermal stress or vacuum-induced damage to the material.

As the drying rate is slow and depends on the material type and sample size, it is necessary to report on a graph the mass of the sample in the desiccant cell over time and to stop the process when the mass change in successive readings is within 1 %.

A.3.4.2 Apparatus

a) Compressed-air dryer in compliance with ISO 7183:2007;

b) Chilled-mirror dew point hygrometer in compliance with EN 16242:2012.

A.3.4.3 Drying procedure

a) The sample shall be placed inside the drying cell and dried until constant mass has been reached;

b) The dew point temperature of the dry air blowing from the compressor shall be lower than −30 °C;

c) The dry air shall flow at a rate ranging between 10 000 cm^3 hr^{-1} and 40 000 cm^3 hr^{-1}, as appropriate;

d) The forced ventilation of dry air from the compressor shall not spread the sample in the oven, with the risk of causing a significant weight loss;

e) Once the sample has been dried, operations and calculations shall follow EN 13183-1:2002.

A.3.5 Adsorption drying

A.3.5.1 General

This drying method applies to materials neither resistant to oven temperatures nor resistant to low pressures and constitutes an alternative to compressed-air drying.

In this method the sample is dried placing it inside a desiccator provided with an efficient desiccant.

As the drying rate is slow and depends on the material type and sample size, it is necessary to report on a graph the mass of the sample in the desiccant cell over time and to stop the process when the mass change in successive readings is within 1 %.

A.3.5.2 Apparatus

a) Desiccator for general laboratory purposes. It is composed of a sealable glass enclosure, usually designed so the drying agent rests in a lower chamber and the sample is placed in the upper part on a plate containing holes to enable internal diffusion of water vapour. The glass desiccator shall comply with EN ISO 13130:2011;

b) Desiccant. The desiccant shall have high affinity for water and strong hygroscopic properties, e.g. silica gel (SiO_2), phosphorus pentoxide (P_4O_{10}), barium monoxide (BaO), magnesium perchlorate ($Mg(ClO_4)_2$), anhydrous calcium chloride ($CaCl_2$), activated alumina (Al_2O_3), aluminium silicate ($AlNa_{12}SiO_5$), lithium chloride (LiCl);

c) Humidity capacitance sensor or a chilled-mirror dew point hygrometer (as specified in EN 16242:2012).

A.3.5.3 Drying procedure

a) The sample shall be placed inside the drying cell and dried until constant mass has been reached;

b) After desiccation, the sample shall be in equilibrium with the RH inside the desiccator, which shall be lower than 11,30 %. The desiccation of the sample is made in one or two steps, as follows:

   1) The first step lowers the MC of the sample to, or in proximity of, the required threshold. An efficient desiccant shall be used in the glass desiccator chamber. Desiccants shall be used in excess in comparison with removable moisture. Depending on the salts that are used, the presence of undissolved crystals at equilibrium indicates that the RH level is below the deliquescence level of the salt used, e.g. RH ≤ 32 % for $CaCl_2$, although the exact level is unknown and some unknown residual moisture is left in the sample. A relative humidity

capacitance sensor or a chilled-mirror dew point hygrometer shall be used to verify if RH ≤ 11,30 % ± 3 %. If it is not, or the measurement is not possible, the next step applies;

2) The second step lowers the equilibrium to the required interval, i.e. $0 < RH \leq 11,30$. Lithium chloride (LiCl) should be used as desiccant, being characterized by RH = 11,30 % ± 3 % deliquescence level over the temperature range 0 °C to 35 °C. The presence of undissolved crystals of LiCl at equilibrium indicates that the internal desiccator RH has reached 11,30 % ± 3 %. However, some residual moisture may remain in the samples, even with the strongest desiccants.

c) Once the sample has been dried, operations and calculations shall follow EN 13183-1:2002.

A.3.6 Thermo-gravimetric analysis (TGA)

A.3.6.1 General

This method may be considered a sophisticated variety of oven drying, especially useful because it requires very small samples and makes fast measurements. The thermobalance automatically records the change in weight of the sample while it is being heated in a flow of controlled atmosphere. Moisture is continuously removed from the sample and the weight reduction is recorded until the sample has reached a constant weight. The record is made in function of both time and temperature.

TGA is useful to detect at what temperature water is released (e.g. to see whether there is crystallization water or to see when water is produced by degradation such as condensation reactions), but also to monitor directly degradation (e.g. emission of CO_2, NH_3 and other gases).

TGA can be combined with selective detection methods like Fourier transform infrared spectrometry (FTIR), or mass spectrometry (MS), which would allow quantifying the release of water only.

A.3.6.2 Apparatus

a) microbalance;

b) furnace (generally made of resistance heaters and temperature sensors);

c) source of pure, low pressure, dry nitrogen gas;

d) programmer/controller;

e) computer or data acquisition system.

A.3.6.3 Procedure

a) a sample shall be taken in accordance with Clause 7;

b) the mass of the moist sample shall be determined with the analytical microbalance that remains outside the oven;

c) the TGA shall be performed by gradually raising the temperature of a sample in the oven;

d) oven temperature shall be monitored via a thermocouple;

e) the change in weight of the sample shall be tracked with a microgram balance;

f)   data shall be recorded as weight percent (%) versus temperature (°C) to illustrate thermal transitions in the sample, e.g. loss of water by evaporation, decomposition and release of VOC, water of hydration in inorganic materials;

g)   a test report shall be produced in accordance with Clause 10.

A.4 Karl Fischer titration

A.4.1 General

The Karl Fischer titration (KFT) is a precise analytical method to determine the water content of solid and liquid samples.

In contrast to gravimetry, azeotropic distillation and calcium carbide, which are typical for moisture (MC), the KFT is a water-specific method (WC). It can be more accurate than gravimetry because in the oven drying and weight loss method volatiles other than water may be lost, which will be misinterpreted in terms of higher MC. This may be particularly relevant for materials treated with wax, oil, or varnishes.

The method is based on the direct determination of the initial mass of the moist test sample m_H and the mass of the water m_w that is extracted from the sample with KFT.

The material sample is dissolved in an anhydrous solvent acting as working medium. If the solid is not soluble, a solvent is used to leach the moisture out of the solid. Once the water is transferred into the solvent and carried in the titration cell, it is measured by chemical titration, using a KF reagent.

Titration can be made in two basic ways, i.e. volumetric (V-KFT, see A.4.4) that is less sensitive and requires larger samples, and coulometric (C-KFT, see A.4.5) that is more sensitive and requires smaller samples.

In the case a higher temperature is necessary to extract water from the sample, the KFT takes advantage of vaporization in a heated oven (A.4.6), and the vapour is carried to the titration cell by a carrier gas, e.g. dry air or nitrogen.

The water extraction from the sample shall comply with EN ISO 15512, ISO 760, ISO Guide 34 and EN ISO/IEC 17025.

The mass of removed samples is generally small. The KFT works over a wide WC range from part-per-million up to 50 %. The analysis is rapid (tens of minutes) and accurate (up to 5 ppm).

The results of KFT determinations are generally reported in percent (%) of the moist sample. In order to make comparable these values with those obtained with other methods, all measurements shall be transformed in WC with the transformation to dry basis using Formula (3) as specified in 5.1.2, but with WC instead of MC.

A.4.2 Apparatus

In general, the KFT apparatus consists of usual chemical laboratory equipment. Several KF titration instruments exist, differing in their design and concept, their ease of use and handling. Simple instruments consist of a burette and an indication system. Titrators carry out the titration automatically, register the weight of the sample being determined, adapt the titration rate to the amount of water in the sample and print out the titration results.

This clause reports the most general features; some specific ones for V-KFT, C-KFT and oven-vaporization KFT will be reported in A.4.4.2, A.4.5.2 and A.4.6.2, respectively.

a)   Titration cell. The titration cell consists of a smaller compartment with a cathode immersed in the anode solution of the main compartment. The cell shall be a glass vessel kept tightly closed by

plastic seals. The size of the cell should not be larger than absolutely necessary because the unused volume is a source of extraneous moisture;

b) Magnetic stirring mechanism. This mechanism shall be included in the cell to ensure a rapid mixing of the reagent;

c) Pipettes and syringes. Pipettes shall be made of glass. Plastic syringes with calibrated cylinder for the volumetric addition of small sample volumes (<5 cm^3) can be also used;

d) Solvents and KF reagents shall be used as indicated by the manuals produced by manufacturers and the KF reagent manuals.

A.4.3 Procedure

The KF method is based on the direct determination of the initial mass of the moist test sample m_H and the mass of the water m_w extracted from the sample. The mass of the dry sample, or the value of WC on the dry basis, shall be computed as specified in Clause 5.

The procedure to determine m_w shall be as specified in Clause 5. This Clause reports the most general features; some specific ones for V-KFT, C-KFT and oven-vaporization OV-KFT will be reported in A.4.4.3, A.4.5.3 and A.4.6.3, respectively.

Additional precautions:

a) Titration cell. The penetration of atmospheric moisture shall be kept to a minimum. The cell shall be hermetically sealed when in use, and shall only briefly be opened in order to add the sample or to replenish the solvent. Before use, the cell shall be rinsed with methanol and dried. The cell shall be completely disassembled if it will be unused for long periods of time;

b) Pipettes and syringes. Glass pipettes shall be used to add working medium and reagent into the cell because they ensure an accurate addition and a better protection against infiltration of moisture. Before use, pipettes and syringes shall be rinsed with methanol and dried;

c) Solvents and KF reagents (titrants). Solvents and reagents shall have low moisture content and contain no impurities. A commonly used titration solvent is methanol, which has a high polarity and considerable capacity to extract water. Some KF reagents tend to precipitate, which could significantly change the range of the titre. This may happen although the reagent is still "good" according to the "best before" date on the bottle. For this reason fresh reagents shall be used;

d) Side reactions. KFT is specific for WC if no side reactions take place with the solvent or the KF reagent. This means that no water should be released in side reactions, nor should the sample consume or release iodine. Side-reactions are possible, which either consume water or iodine. Most (unwanted and interfering) side reactions can be suppressed by suitable measures. The most important interfering substances are: carbonates, hydroxides and oxides; aldehydes and ketones; strong acids; metal peroxides; reducing agents; strong oxidising agents;

e) The user shall consult the manuals produced by manufacturers and the KF reagent manuals, which describe possible side reactions and how to circumvent them for a suitable choice of solvents and KF titration reagents;

f) Test report. After the water content of the material sample has been determined, a test report shall be produced in accordance with Clause 10.

A.4.4 Volumetric KF titration (V-KFT)

A.4.4.1 General

The water content of the material sample is measured by V-KFT of water with an iodine solution. The method determines the amount of water mw extracted from the sample, considering that its mass expressed in g equals its volume in cm^3.

The volumetric analysis takes advantage of the fact that iodine contained in KF reagents reacts quantitatively and selectively with water. The quantification is based on the principle that 1 mol of iodine (254 g) reacts with 1 mol of water (18 g). The end point is indicated by the appearance of the brown colour of free iodine. Iodine is consumed as long as there is any water present in the solution

$$2H_2O + SO_2 + I_2 \rightarrow H_2SO_4 + 2HI$$
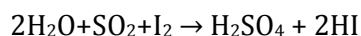
The addition of pyridine or imidazole binds and prevents the loss of sulphur dioxide, and the methanol acts as the solvent medium. If a substance reacts with the KF reagents and releases water or consumes iodine; this may cause large measurement errors. V-KFT needs a proper titre determination.

The water content on wet basis WC_w shall be determined from the amount titrated according to the equation

$$WC_w\left(\%\right) = \frac{Titration\ volume\left(cm^3\right) \times Titre\left(g_{H2O}\,cm^{-3}\right)}{Sample\ mass\left(g\right)} \times 100 \tag{A.1}$$

where the titration volume (cm^3) multiplied by the titre ($g_{H2O}\ cm^{-3}$) represents the mass (g) of water m_w extracted from the sample, and the sample mass is the initial mass of the moist test sample m_H. In order to make comparable these values with those obtained with other methods, all measurements shall be transformed in WC with the transformation to dry basis using Formula (3) as specified in 5.1.2, but with WC instead of MC.

V-KFT is less sensitive, but more robust than C-KFT. High water contents are preferably determined by volumetric titration. Volumetric KF also has the advantages that solid or pasty samples can be introduced directly into the titration vessel and the analysis can be carried out with a variety of suitable organic solvents specially adapted for use with a particular sample.

A.4.4.2 Apparatus

As specified in A.4.2, but with specific reference to devices for volumetric titration.

A.4.4.3 Procedure

a) The sample of interest should be more or less dissolved. When dissolution is impossible, the sample shall be ground and well homogenized (at least 90 % of the particles should be smaller than 100 μm);

b) There are different commercially available solvents and references but the choice shall be made following to the production manual;

c) Volumetric titrations normally require 20-50 cm^3 of reagent;

d) Drawbacks that shall be controlled are instrumental drift, tightness of all tubing connections and the titration cell;

e) Laboratory staff should keep the KF solvent consumption in the range 5 to 10 $mm^3\ min^{-1}$. This depends on the amount of sample and the WC of the sample;

f) If only small samples with rather low water content (<5 %) are available, the drift may have an impact on the scatter of the measurement results (method repeatability);

NOTE   1 cm^3 of liquid corresponds to 1 ml (mL); 1 mm^3 to 1 μl (μL).

A.4.5 Coulometric KF titration (C-KFT)

A.4.5.1 General

The water content of the material sample is measured by C-KFT of water with electrolysis. The method determines the mass of water m_w extracted from the sample from the number of electric charges used in the electrolysis. C-KFT is based on the electrochemical production of iodine, which reacts with an equivalent amount of water. C-KFT is more sensitive than V-KFT (usually a factor of 10). Coulometric titrations normally require 100 cm^3 of reagent.

With coulometric titration, electrolytic oxidation is achieved by adding the sample to an iodide ion solution, instead of the iodine contained in the KF reagent, and used as the titration agent for volumetric titration. Electrolytic oxidation produces iodine at the anode. The water content m_w is determined from the electrical charge (in coulombs) required for electrolysis, as specified in Formula (A.2).

The detector circuit maintains a constant current between the two detector electrodes during titration.

The electrical current releases the stoichiometrically corresponding amount of iodine from the iodide-containing KF reagent by electrolysis. According to Faraday's law of electrolysis the mass of the converted substance, in this case the water present in the test sample is:

$$m_w\left(g\right) = \frac{Molar\ mass\left(gmol^{-1}\right) \times Electrical\ charge\left(A \cdot s\right)}{z \times Faraday\ constant} \times 100 \tag{A.2}$$

where the molar mass of water is 18 g mol^{-1}; the electrical charge is expressed by the product of the current intensity (A) of the electrolysis by the operation time (s); z is the number of exchanged electrons (z = 2 for iodine: 2 I$^-$ – 2 e$^-$ → I$_2$) and F the Faraday constant (electrochemical equivalent), i.e. 1 F = 96485 coulomb mol^{-1}.

The instrument measures the time and the current flow that are required to reach the titration end point. The product of (time × current) is directly proportional to the amount of iodine generated and therefore to the amount of water determined.

In order to make comparable these values with those obtained with other methods, all measurements shall be transformed in WC with the transformation to dry basis using Formula (3) as specified in 5.1.2, but with WC instead of MC.

A.4.5.2 Apparatus

As specified in A.4.2, plus a detector circuit to measure time and current intensity at the electrodes.

A.4.5.3 Procedure

a) Dissolved or dispersed samples shall be introduced into the titration cell using a syringe. The sample shall be introduced rapidly (e.g. to avoid the penetration of external air humidity).

b) C-KFT can be run in two different modes, with and without diaphragm. The cell with the diaphragm uses two solutions, one in the cathode chamber the other in the anode chamber. The cell without diaphragm uses one solution that contains all the reagents needed for KFT. Reference shall be made to the instruction manual. The major drawback is that some samples precipitate on the electrode, which may lead to too long titrations (end point not found). The use of a diaphragm may not always improve the situation and in such cases oven-vaporization C-KFT should be preferred.

c) Coulometric water determination shall be mainly used for the determination of small amounts of water (WC from part-per-million up to 2 %). Large amounts of water may require longer time and/or may exceed the water capacity of a KF reagent that could lead to incorrect results. Side-reactions are possible and the producer manual shall be carefully consulted.

d) The septum of the titration cell shall be regularly checked for tightness and cleanliness.

e) The C-KFT system requires proper handling and tightness of tubing connections and titration cell.

A.4.6 Oven-vaporization KF titration (OV-KFT)

A.4.6.1 General

This method uses higher temperatures to facilitate the KFT of water from the sample. Many materials release their water slowly or at higher temperatures, so they are not suitable for direct KFT at ambient temperatures.

This problem can be avoided at higher temperatures by using a moisture vaporization method followed by KFT, in general in combination with coulometric KFT (C-KFT) that is more sensitive, but it could also be combined with volumetric KFT (V-KFT).

Oven-vaporization C-KFT allows a kind of thermogravimetric analysis (TGA) but the water content is directly monitored versus temperature, instead of the weight loss.

The main advantage of OV-KFT is automation, i.e. the ability to run several samples in a row. The main disadvantage is the larger instrument drift (gas flow between 50 and 100 cm^3 min^{-1}). While V-KFT may require about 5 min titration time, oven-vaporization C-KFT may require up to 30 min or even longer. The achievable drift is usually double that of V-KFT (10 mm^3 min^{-1} to 20 mm^3 min^{-1}). For very small sample sizes containing only little water, this might sum up in the total amount of water and the scatter of the results. There is the possibility that not all water is transferred into the titration cell with a little underestimation of the WC.

NOTE   1 cm^3 of fluid corresponds to 1 ml (mL); 1 mm^3 to 1 µl (µL).

A.4.6.2 Apparatus

An oven (as specified in A.3.2.2), a dry carrier gas, e.g. nitrogen gas cylinder (as specified in A.3.5.2), plus an instrument that graphically monitors the water content by KF titration versus temperature (as specified in A.4.2).

A.4.6.3 Temperature

a) The material sample under investigation shall be heated in an oven located upstream the titration cell and the released water is transferred by a dry carrier gas flow to the cell where it shall be determined by KFT. As only water enters the titration cell and the sample itself does not come into contact with the KF reagent, unwanted side reactions and matrix effects are minimized.

b) The gas used for transferring the water (and all other volatiles) into the titration cell shall be absolutely dry. Dry nitrogen should be preferred because it does not oxidize the sample.

c) Attention shall be paid that the applied heat in the oven could cause condensation reactions (e.g. between amino acids and carbohydrates), in particular at higher temperatures (>130 °C).

d) Instruments that graphically monitor the water content versus temperature are useful, but the following shall be considered:

e) Stable materials generate a single peak of water release;

f) Materials with crystalline water generate two or more peaks depending on the type of hydrate (some organic hydrate materials do not have any free water on the surface, which may lead to only one peak). When the oven temperature does not reach the threshold for crystallization water, an underestimate of the WC will result. However, this is not a problem but an advantage, when only the moisture exchangeable with the environment is of interest.

g) Unstable materials generate a defined peak at lower temperatures and a broad peak at higher temperatures (or even a steadily increase of the baseline).

h) For the above reasons, the temperature programme shall be carefully chosen: neither too slow (that requires too long measurement times, i.e. instrument drift sums up), nor too fast (that risks to overlook the moment when sample degradation might take place).

A.4.7 KF titration of selected materials

a) **Wood.** The water content depends to a large extent on the storage conditions. An external extraction method with dry methanol shall be used combined with the oven-vaporization-KF method at temperatures up to 190 °C. Water determination by the OV-KFT always yields higher moisture contents than the drying oven method, which can never determine all the water.

b) **Masonry.** Moisture vaporization shall be combined with KFT, either V-KFT or C-KFT is generally used for substances with high WC. Samples shall be crushed to a fine powder. During this process care shall be used to prevent exchanges of moisture with the environment. Fractional measurement shall be achieved using the moisture vaporization method. At heating temperature from 100 °C to 200 °C free and bound water are released; from 500 °C to 1 000 °C crystallization water. The temperature for vaporization shall be set according to the type of sample and the purpose of the measurement. Specific indications for the main types of materials constituting masonry and dissolved salts as follows:

   1) **Limestone and marble.** Calcium carbonate does not dissolve in the solvent. It is dispersed and the moisture content shall be determined by V-KFT. In general, for carbonates that are stable when heated, the moisture content shall be measured using OV-KFT.

   2) **Bricks and ceramics.** As ceramics do not dissolve in organic solvents, either V-KFT or C-KFT shall be used in combination with oven vaporization. The heating temperature and other conditions for moisture vaporization shall be determined with reference to the type of sample.

   3) **Plaster.** The sample shall be placed in the titration vessel with methanol, heated to 50 °C and titrated to dryness with a titrant solution (conditioning).

   4) **Concrete.** Direct titration is not possible. The water is chemically bonded, while the oxides in cement cause interference by reacting with the KF reagent to produce water. Crystallization water shall be measured with vaporization from 800 °C to 1 000 °C, followed by KFT.

   5) **Gypsum** ($CaSO_4 \bullet 2 H_2O$) releases some crystallization water at 128 °C with a transition to $CaSO_4 \bullet 1/2 H_2O$; at 163 °C it becomes anhydrous ($CaSO_4$). If the total WC is needed (i.e. including crystallization water), the analysis should be made with C-KFT in association with oven vaporization, at temperature above 200 °C. If the MC but no crystallization water is required, gravimetry at moderate or low temperatures shall be used.

   6) **Inorganic salts.** Salts in masonry may contain moisture or crystallization water. Alkali halides crystallize out without any water of crystallization, but usually contain moisture that is only released slowly. Examples are: NaCl, KCl, NH4Cl, KI, CsI and KF. Alkali silicates cannot be

titrated directly. They react like alkali hydroxides and release water on neutralization: OV-KF method should be used.

7) **Inorganic hydroxides and carbonates.** Masonry might contain such minerals. During the KFT methyl sulphuric acid and hydriodic acid are formed that react with these compounds to form salts and release water. The water content in hydroxides and carbonates shall be determined by the OV-KF method.

A.5 Azeotropic distillation

A.5.1 General

This method is based on the direct determination of the initial mass of the moist test sample m_H and the mass of the water m_w in the sample that will be extracted via azeotropic distillation. The mass of the dry sample, or the value of MC on the dry basis, shall be computed as specified in Clause 5.

This method is specific for MC of organic materials. It is based on the entrainment of the water in the form of vapour by a volatile solvent immiscible with water, condensation and separation in a reflux trap, recovery and measurement of the volume of condensed water in a graduated tube.

This method shall be applied when wood contains volatile oils not soluble in water. In case the sample contains high amount of water-soluble volatile organic compounds the MC overestimates the WC and a correction is needed to remove the contribution of volatiles to compare the results with other methods.

Following EN 1428:2012, the moisture content is given as a percentage of the moist sample MC_w as follows:

$$MC_w = \frac{m_w}{m_H} \times 100 \tag{A.3}$$

where m_w is the mass of the water extracted by distillation from the sample (expressed in grams and therefore, equal to the volume of water read in millilitres on the graduated collecting tube) and m_H is the initial mass of the material sample.

In order to make comparable the results of this method with the other absolute methods, the values of MC_w determined with azeotropic distillation shall be transformed in MC with the transformation to dry basis using Formula (3) as specified in 5.1.2.

The moisture content shall be reported to the nearest 0,1 %.

Main advantages of this method are: relatively short distillation time (about 1 h); no influence of environmental humidity; low cost and easy to handle. Main disadvantages are: limited reading accuracy; contamination of other substances (e.g. glycerol and alcohol) that may distil with water.

A.5.2 Apparatus

The apparatus consists of usual laboratory equipment, and in particular:

a) analytical balance (as in A.2.2);

b) distiller composed of the following items connected together by means of glass joints: conical flask made of glass (capacity 500 cm³ at least), moisture trap, reflux condenser, receiver (composed of collection vessel and a graduated tube in 0,1 cm³ divisions) that will be connected to the flask and the condenser;

c) connecting tubes;

d) electric heating apparatus, with a temperature control system, or a boiling water bath;

e)   magnetic stirrer;

f)   glass pipettes or plastic syringes;

g)   solvents.

A.5.3 Procedure

The method is based on the direct determination of the initial mass of the moist test sample m_H and the mass of the water m_w extracted from the sample. The mass of the dry sample, or the value of MC on the dry basis, shall be computed as specified in Clause 5. Specific steps as follows.

a)   the mass of the moist sample m_H shall be determined with the analytical balance;

b)   the glassware shall be accurately rinsed and dried to avoid contamination or water remaining adherent to the glass surface;

c)   the azeotropic distillation shall follow the operational procedures specified in EN 1428:2012. The moisture contained in a wood sample (approximately 100 g) shall be thermally extracted by azeotropic distillation with an organic solvent, usually xylene (in accordance with ISO 5280:1979), or toluene (in accordance with ISO 5272:1979) that acts as a carrier. The azeotropic distillation shall occur at a temperature < 100°C. The condensed organic solvent and water shall be continuously separated. After the condensed solvent will return to the flask, the condensed water will be collected in a graduated trap where it shall be measured;

d)   the mass of water m_w extracted by distillation from the sample shall be read on the graduated tube;

e)   after the water content of the material sample has been determined, a test report shall be produced in accordance with Clause 10.

A.6 Calcium carbide test

A.6.1 General

Also called "speedy moisture meter" or "calcium carbide bomb".

This method is based on the direct determination of the initial mass of the moist test sample m_H and the mass of the water m_w in the sample that will react with calcium carbide (CaC_2) producing acetylene gas (C_2H_2). The mass of the dry sample, or the value of MC on the dry basis, shall be computed as specified in Clause 5. The method is generally used to measure on the site the moisture content of soil, masonry and cementitious materials.

A sample is inserted into a sealed container with calcium carbide (CaC_2). The moisture content is determined by chemical reaction and measurement of the pressure of the gas produced. The method provides fast measurements and is used on-site. This test is usually accurate to within 2 % of the MC measured with gravimetry. If more accurate readings are required, the gravimetric method should be used instead.

The calcium carbide method requires that sampled material is ground to a fine powder and mixed with calcium carbide in a sealed vessel. Calcium carbide will react with the free moisture present within the powder and produce acetylene gas (C_2H_2). The chemical reaction is

$$CaC_2 + 2H_2O \rightarrow C_2H_2 + Ca(OH)_2$$
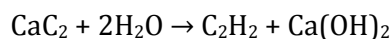
The amount of gas released is directly proportional to the amount of moisture present in the material and is insensitive to salt contamination.

Calcium carbide pressure meters shall be calibrated in accordance with the manufacturer's instructions. Pressure readings are absolute (expressed in Pa) and can be converted into % by mass with the aid of tables. In addition, specific correction factors need to be determined for certain materials.

A.6.2 Apparatus

Measuring instruments and pressure gauges are built with different characteristics for some selected ranges of pressure and MC. The apparatus is portable and consists of:

a) Analytical balance having 2 000 g capacity and 0,1 g accuracy;

b) Stainless steel vessel for the sample and reagent, with attached pressure gauge. The vessel shall have a clamp capable of resisting the pressure generated;

c) Pressure gauge for the measurement in compliance with EN 837-1:1996;

d) 3 to 6 stainless steel balls with various diameters from 5 mm to 15 mm;

e) Calcium carbide reagent in compliance with the material Safety Data Sheet of the European Chemicals Agency (ECHA). It shall be kept in the sealed glass phials (with indicated expiry date) of the supplier until use. Calcium carbide is a hazardous substance and shall be packed and transported according to international transport regulations (EU-Regulation No 1272:2008);

f) Metal scoop to measure and insert the calcium carbide reagent into the cup;

g) Conversion chart for moisture content based on individual calibration versus gravimetric determination (% by mass) made for the specific material under investigation.

A.6.3 Procedure

a) As the procedure involves a potentially dangerous chemical reaction, measurements shall be performed by qualified personnel expert in handling and storing flammable, explosive and toxic substances; all the operational instructions of the manufacturer manual shall be carefully followed. All components of the whole apparatus shall be clean and dry. Sample preparation and measurements shall be made outdoors, or in a well ventilated room;

b) Sampling. Materials being tested for residual moisture shall not be sampled using methods that will generate frictional heat, e.g. drilling, grinding. Care shall be taken that the sample may contain certain compounds or chemicals that may react unpredictably with the reagent and give erroneous results;

c) Preparing the sample. Samples shall be crushed to small lumps using impermeable laboratory or medical gloves (EN 420:2003+A1:2009, EN ISO 374-1 or EN 455-1:2000). It is important to crush the sample in a very short time to avoid the crushed grains exchanging moisture with the surrounding air. The use of a mesh sieve to separate the coarse grains from the fine powder to be analysed is not recommended because it may affect the moisture content of grains;

d) Weighing the sample. The exact mass specified by the manufacturer shall be determined. For materials with moisture content higher than the gauge limit, only half the weight of sample suggested in the manufacturer's reference table shall be used; then the readings shall be multiplied by two;

e) Adding the sample to the vessel. Care shall be taken not to allow the moist sample to come into contact with the reagent during this operation;

f)   Reagent. Calcium carbide (CaC_2) shall be finely pulverized to produce acetylene gas in the amount of at least 0,15 m^3 per kilogram of CaC_2;

g)   Adding the reagent to the cap cavity and sealing the vessel clamp. The amount of reagent shall be considered in proportion of quantity of the moisture that will react. If the amount of reagent is too small, the MC will be underestimated. The internal pressure that will be exerted shall not exceed 150 kPa. Conversion tables furnished by the manufacturer shall be consulted to relate the pressure to the volume of the vessel and the amount of moisture that will react;

h)   Mixing the sample with the reagent. The instrument shall be shaken with a rotating motion so that the steel spheres will continue to break down the lumpy sample, favour mixing and permit the calcium carbide to react with all available free moisture which allows the heat generated to dissipate without risk for the observer or damage for the instrument. Shaking shall be continued at least for 2 min or as indicated by the manufacturer as a function of the vessel size, sample weight and moisture content of the sample (that should be approximately estimated in advance). It is crucial that shaking is continued for the specified time;

i)   Taking the pressure reading. When the needle of the pressure gauge stops moving, the dial shall be read at eye level;

j)   Releasing the pressure. With the cap of the instrument pointed away from the operator, and away from open flame or any source of ignition, the gas pressure shall be slowly released;

k)   For optimum performance the instrument and sample should be at 20 °C. If the instrument is used at very low temperatures, the chemical reaction may be incomplete, providing low readings. In such a case a number of preliminary readings should be performed in quick succession to raise the operating temperature of the instrument. When the readings stabilize, the final result shall be kept for the MC;

l)   Removing the sample and reagent and cleaning all apparatus. If any material has remained in the cap or has remained lumpy, the reading is misleading and the test shall be repeated from the beginning;

m)   Conversion of readings. The conversion of readings from pressure to moisture content shall be made. Calcium carbide pressure moisture meters are commercially available and shall be calibrated in accordance with the manufacturer's instructions. Moisture content based on the dry mass shall be obtained using a conversion chart or an individual calibration. If the sample contains volatile components other than water, or include hydrated salts, the MC readings may be affected. Correction factors for selected materials shall be established by plotting pressure readings versus the gravimetric method to obtain a calibration plot. The MC obtained from the conversion is usually expressed as a % of the mass of the moist test sample on wet basis, and shall be converted to MC with the transformation to dry basis using Formula (3) as specified in 5.1.2.

n)   Test report. After the moisture content of the material sample has been determined, a test report shall be produced in accordance with Clause 10.

WARNING — Some of the procedures included in this method entail the use of potentially hazardous chemicals. It is emphasized that all appropriate safety precautions shall be taken.

Table A.1 — Comparison between absolute methods: characteristics

	Gravimetric methods	Karl Fischer titration	Azeotropic distillation	Calcium carbide test
Reference standards	ISO 16979:2003 EN 13183–1:2002	ISO 760:1978 EN ISO 15512	EN 1428:2012	
Material	Wood, Masonry	Wood, Masonry	Wood	Masonry
Specific for	MC	WC	MC	MC
Invasive/destructive	Yes	Yes	Yes	Yes
Sample size	Small TGA: very small	C-KFT: small V-KFT: medium	Small	Large
Accuracy	Very accurate; useful for calibration of other methods	Very accurate; useful for calibration of other methods	Accurate determinations	Less accurate determinations
Method sensitive to:	Water mass	Water mass	Water mass	Water mass
Penetration depth	Selectable depth	Selectable depth	Selectable depth	Selectable depth
Time response	Medium-term (day)	Short-term (hour)	Short-term (hour)	Short-term (hour)
Lab/on-site measurements	Laboratory	Laboratory	Laboratory	On-site
Spot/continuous time monitoring	Spot	Spot	Spot	Spot
Notes	Select oven drying, vacuum drying or desiccant drying depending on material characteristics	Select titration type, KF reagents and oven temperature. V-KFT: high water content. C-KFT: low water content. OV- KFT: when evaporation needs high temperatures	Method recommended when wood contains volatile oils	Method limited to masonry

Table A.2 — Comparison between absolute methods: experimental uncertainties and notes

| Gravimetric methods	Accurate method. It can be used for calibration of other methods Attention shall be paid to the most suitable drying method.
Karl Fischer titration	Accurate method. It can be used for calibration of other methods. Attention shall be paid to KF reactant and temperature to avoid possible interference and side reactions. Possible to measure volatile compounds or contaminants.
Azeotropic distillation	It provides precise quantitative results for each sample measured. If the number of sampling locations is limited, the results may not be representative for the entire material due to variability of moisture content across the sampling area. If wood contains water soluble substances, they can be extracted along with water and the apparent weight of water is increased.
Calcium carbide test	Method with large uncertainties. Each particular material needs a specific calibration curve to relate pressure readings to the moisture content.
All methods	Because a sample has been removed, it is impossible to measure change of moisture content over time at the same point. If the number of sampling locations is limited, the results may be not representative for the entire material due to variability of moisture content across the sampling area.
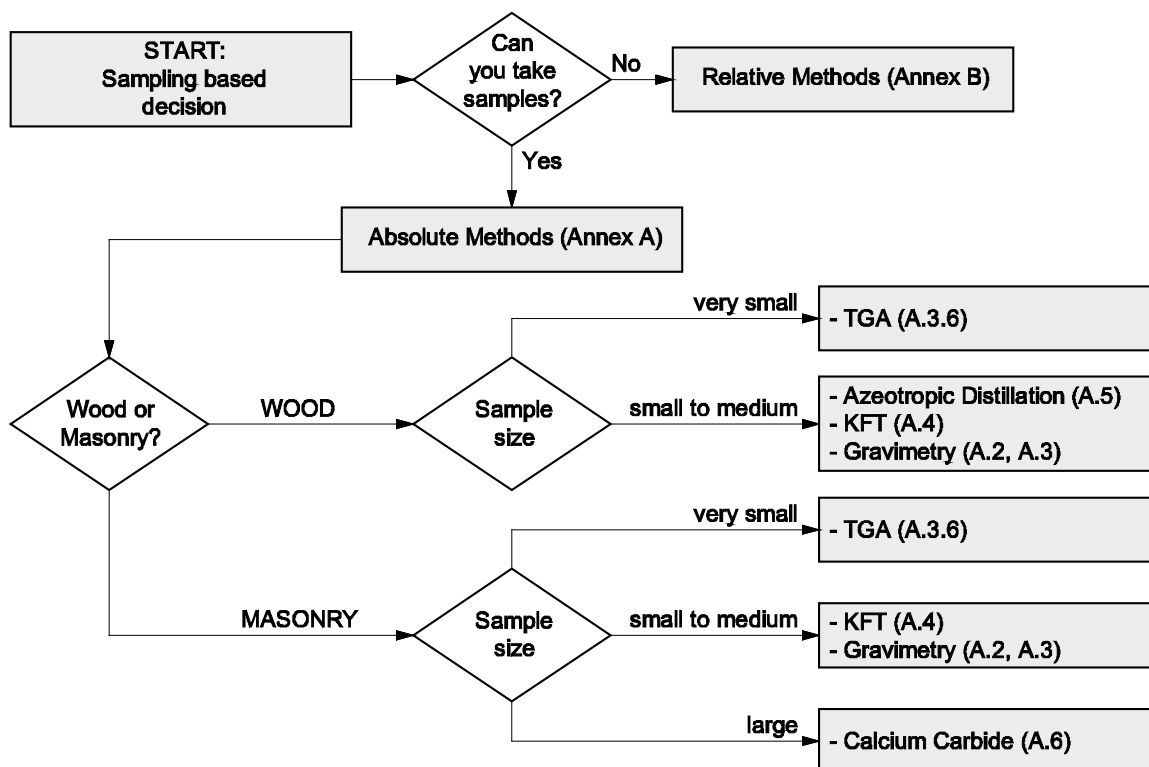
Figure A.2 — Decision tree: choice of the most convenient absolute method in relation to the possibility of removing small, medium or large samples. Numbers in brackets refer to the clause numbers
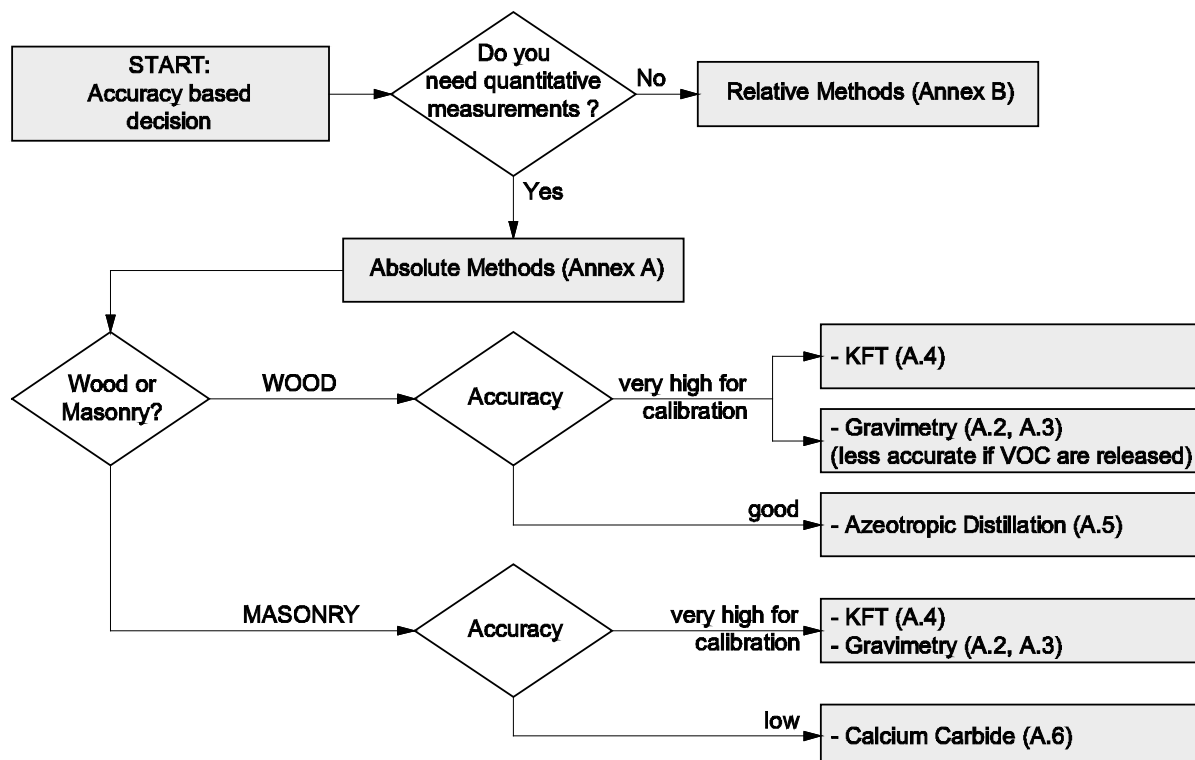

Figure A.3 — Decision tree: choice of the most convenient absolute method in relation to the required accuracy level. Numbers in brackets refer to the clause numbers

Annex B
(normative)

Relative methods

B.1 Generalities

This annex is normative and aims to provide specifications concerning relative methods that have been chosen for their easy applicability although the results are not directly comparable.

This annex establishes the operational procedures and provides guidelines concerning the main problems occurred during on-site observations and interpretation of readings.

The use of these methods is limited to the detection of moisture and comparison of changes over space or over time.

The following relative methods require contact of electrodes or other sensors with the material surface.

a) The use of sensors in contact with the material surface, i.e. capacitance, electrical resistance (only contact without pin insertion) and RH in equilibrium with the material–external sealed box, is generally safe, but shall in any case be made under supervision of a qualified conservation professional.

b) The method of electrical resistance with insertion of pins is (minimally) invasive/destructive and should only exceptionally be used under the strict control of a qualified conservation professional.

c) The method of reaching RH in equilibrium with the material (drilled cavity) is invasive/destructive and should only exceptionally be used under the strict control of a qualified conservation professional. This method is illustrated in the flow diagram in Figure B.1.

The number of observations shall be considered in order to obtain a reliable result and at the same time respect the cultural value of the heritage material. With non-invasive, fast response sensors it is possible to repeat the measurement at different points on the same material to obtain more representative MC values.

In order to provide a general overview and a cross comparison between relative methods, Table B.1 and Table B.2 will report the characteristics of the various methods, their experimental uncertainties and some notes concerning their specific use with wood or masonry. Figure B.2 and Figure B.3 elucidate the possibility of doing a reliable calibration and the quality of measurements obtained with resistance or capacitance methods in wood and masonry. Table B.1, Table B.2, Figure B.1, Figure B.2 and Figure B.3 are useful to assist the user selecting the most convenient method and interpreting the MC readings in the field of cultural heritage.

B.2 Electrical resistance (conductance)

The magnitude of the resistance, the applied electromotive force, and the current between the electrodes are related by Ohm's law. In most cases, the resistance in a material decreases when its moisture content increases. When a constant electromotive force is applied, in general, the intensity of the current between two electrodes increases with the moisture content.

EN 13183-2:2002 refers to normal wood and cannot be applied to cultural heritage wood if this material has been attacked by pests (e.g. fungi or tunnelling by wood-boring insects), or has been previously treated with substances that alter the surface or subsurface conductivity.

The method can be applied to stone and masonry, but it should be noticed that contamination with electrolytes (usually salts in cultural property) is common in most applications and makes the resistance method highly, and commonly, vulnerable to misinterpretation. If the ambient RH has exceeded the threshold for deliquescence of salts, the conductivity is increased very much.

B.2.1 Apparatus. Instruments are based on two or more electrodes, e.g. pins, pressed or nailed into the material under investigation. Instruments with insulated pins can measure moisture content at various penetration depths.

B.2.2 Procedure. The electrical resistance method shall follow the procedures specified in EN 13183-2:2002 and the producer manual.

B.2.3 Test report. Readings are relative in arbitrary units; they might be tentatively transformed into % after calibration for the specific material made by comparison to gravimetry.

Although automatic conversion or conversion tables provided by the manufacturers are useful, any calibration is difficult because of the changes that historic materials might have undergone, due to ageing, salt contamination, chemical or biological attack.

The test report shall be produced in accordance with Clause 10.

B.3 Capacitance (dielectric)

A capacitor is an electronic component that stores electric charge and is usually made of two plates that are separated by a dielectric material. The plates accumulate electric charge when connected to a power source. The amount of electric charge is proportional to the dielectric properties of the material. The dielectric response of the material increases in proportion to the moisture content. The water molecule has a high dielectric constant ε (ε approximately 80) compared with other materials (generally $2 < \varepsilon < 8$). The moisture content may be determined via its effect on the dielectric constant.

The method can be applied to stone and masonry, but it should be noticed that contamination with electrolytes (usually salts in cultural property) is common in most applications and makes the capacitance method highly, and commonly, vulnerable to misinterpretation. Blistering or subsurface inhomogeneities may cause multiple reflections and bias. If the ambient RH has exceeded the threshold for deliquescence of salts, the capacity is increased very much and in particular the film of absorbed water that forms on the surface will act as a mirror causing saturation of the signal.

B.3.1 Apparatus. Instruments based on capacitance have been developed in a number of ways, to make specific measurements. Instruments exist with two plates both lying in the same plane to be put into contact with the surface under investigation. This sensor is suitable for objects or structures having a plane surface. Other sensor types with diverse, complex shapes (e.g. combs, wires, spheres) have been built for specific purposes and to operate even with objects having non-planar surfaces.

When using a high frequency alternating voltage in the order of GHz the electrical field has properties similar to microwaves (see later) and the instrument can be considered a hybrid method. For this reason the material under investigation shall not include metals or traces of past metal coatings, e.g. gilding.

B.3.2 Procedure. The capacitance method shall follow the operative procedures specified in EN 13183-3:2005, and the manufacturer manual.

B.3.3 Test report. Readings are relative to arbitrary units; they might be tentatively transformed into % after calibration for the specific material made by comparison with gravimetry.

Although automatic conversion or conversion tables provided by the manufacturers are useful, any calibration is difficult because of the changes that historic materials might have undergone, due to ageing, salt contamination, chemical or biological attack.

The test report shall be produced in accordance with Clause 10.

B.4 Relative humidity in equilibrium with the material

B.4.1 General

This method is based on measuring the RH in a small air pocket in equilibrium with the moisture content of the building material. An active or passive RH sensor or a proxy is used to measure the equilibrium RH. The method is particularly used to control dampness in masonry.

The RH inside the cavity is governed by the MC of the material as well as by the presence of salts and the sorption characteristics of the material. The relationship between the RH in the void and the MC in the substrate is nonlinear as a result of moisture absorption/ desorption hysteresis.

The method is possibly appropriate for very wet/very dry situations (e.g. concrete drying where it is commonly used) but it is unsuitable and commonly misused when to determining MC variations in heritage applications.

In masonry, the presence of soluble salts is irrelevant until the RH threshold for salt deliquescence is reached. After this threshold is exceeded, the deliquescent salt starts to form a solution and keeps the equilibrium RH constant.

The air pocket may be internal or external to the material, as specified in the next two clauses.

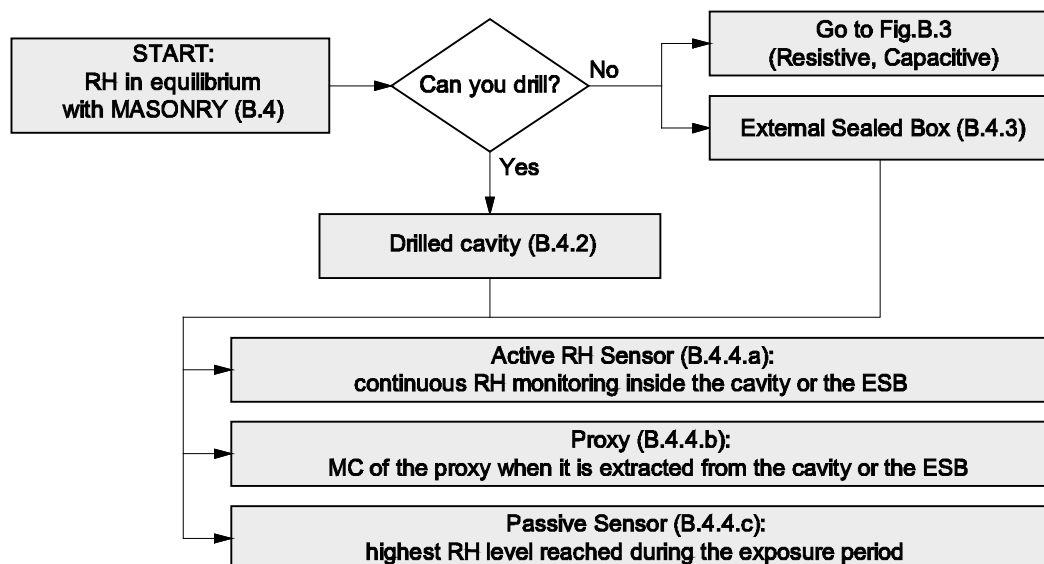The method is illustrated in Figure B.1.

Figure B.1 — Flow diagram illustrating the method of the relative humidity in equilibrium with the material. Numbers in brackets refer to the clause numbers

B.4.2 Drilled cavity

The air pocket is a cavity that has been obtained by drilling the building material and will be representative of the moisture inside the material.

Once the active or passive sensor or the proxy has been inserted into the cavity, the opening of the cavity shall be carefully sealed with an impermeable plug that should prevent exchanges of moisture between the cavity and the free air without altering the internal microclimate equilibrium.

This method is invasive and should be performed under the strict control of a qualified conservation professional.

B.4.3 External sealed box (ESB)

The air pocket is obtained by applying to the surface an external sealed box (ESB) that is representative of the surface/sub-surface moisture content of the building material. The ESB is fixed to the material surface to reach equilibrium with it. Active, or passive RH sensors, or proxies shall be placed in the ESB to measure the RH of this air pocket.

The ESB has a missing side to allow a free moisture exchange between the material surface and the sensor; all other box sides in contact with the external air shall be thermally insulated, and the box shall be sealed to the surface.

The presence of an ESB may alter the free exchange of heat and moisture between the air and the material, in comparison with the free material surface, especially in the presence of thermal radiation.

This method is not invasive.

B.4.4 Apparatus

Either in the drilled cavity or the ESB, the equilibrium RH in the internal cavity can be directly or indirectly measured with one of the following methods:

a) Active RH sensors: capacitive or resistive sensors as specified in EN 16242:2012. Temperature sensor shall be in compliance with EN 15758:2010. The active RH sensor will provide a continuous indication of the RH reached inside the entrapped air pocket, which is in equilibrium with the moisture content of the material. The output should be recorded in a data logger or other recording device.

b) Proxies: consist of a material with hygroscopic properties similar to the building element under investigation. Proxies will provide spot readings representative of the situation when they are extracted from the cavity.

c) Passive Sensors: consist of a combination of hygroscopic salts and dyes that reveal the EMC in the salts, or that some selected RH thresholds have been reached. Passive sensors will provide readings representative of the highest humidity levels reached while they were kept in the cavity.

B.4.5 Procedure

a) The air pocket shall be obtained with a drilled cavity as specified in B.4.1, or with an external sealed box as in B.4.2. The diameter of the drilled cavity shall have the minimum size useful to insert the sensor, and the depth shall be representative of the internal layer under consideration. The ESB shall be made with a highly insulating material, and never the box shall be exposed to direct solar radiation or be in contact with other heat sources.

b) The active or the passive sensor or the proxy shall be inserted into the cavity or the sealed box. The cavity opening shall be carefully sealed to avoid moisture exchanges with the external environment.

c) Capacitive RH sensors cannot be used for humidities continuously exceeding 80 % as moisture absorption by the polymer substrate will falsify readings. It is advisable to include a temperature sensor to obtain more complete information. The RH and temperature sensors should be connected to a datalogger to obtain a continuous set of readings.

d) Proxies shall be extracted at scheduled times for the readings that will be made via gravimetry or other methods as specified in Annex A. An alternative is to provide the proxy with electrodes and monitor the changes of electric resistance or electric capacity.

e) Passive sensors shall be used in accordance with the manufacturer's operating manual.

f) A test report shall be produced. Readings are relative, but might be tentatively transformed into quantitative values after calibration for the specific material made by comparison with gravimetry or KF titration. The test report shall be produced in accordance with Clause 10.

Table B.1 — Comparison between relative methods: characteristics

	Electrical resistance	**Capacitance**	**RH in equilibrium with the material** **Drilled cavity/ESB**
Reference standards	EN 13183–2:2002 EN ISO 14911:1999 EN ISO 10304-1:2009	EN 13183–3:2005 EN ISO 14911:1999 EN ISO 10304-1:2009	EN 16242:2012 EN ISO 14911:1999 EN ISO 10304-1:2009
Material	Wood, Masonry	Wood, Masonry	Masonry
Invasive/ destructive	Partially, i.e. micro invasive/ destructive	No	ESB: No Drilled cavity: Yes
Recommended use for cultural heritage	Possible use with surface (contact) electrodes. Exceptional use of pins under supervision of a qualified conservation professional.	Useful for thin layer monitoring	ESB: may be used if installation is non-destructive Drilled cavity: exceptional use under supervision of a qualified conservation professional.
Method sensitive to:	moisture gradients and other factors (e.g. soluble salts)	moisture gradients and other factors (e.g. polar molecules, deliquescence)	Equilibrium between RH and MC
Penetration depth	Sub-surface	Sub-surface	ESB: surface Drilled cavity: selectable depth
Time response	Fast response (seconds)	Fast response (seconds)	medium- to long-term (week)
Lab/on-site measurements	On-site	On-site	On-site
Spot/ continuous time monitoring	Spot or continuous monitoring	Spot or continuous monitoring	Spot or continuous monitoring
Pros	**Wood and Masonry:** Special pins to measure sub-surface gradients. **Masonry,** Range upper limit: pore saturation	**Wood**: Not invasive **Masonry**: Not invasive; Weak or no dependence on the salt content (except for deliquescence).	Time change relative readings

	Electrical resistance	Capacitance	RH in equilibrium with the material Drilled cavity/ESB
Cons	**Wood**: Influenced by density (e.g. fungal or insect infestation, knots); surface coverings, moisture gradients; grain orientation; temperature. Range limited from EMC = 7 % to fibre saturation point. **Masonry**: Influenced by salts, moisture gradients; temperature.	**Wood**: Influenced by density (e.g. fungal or insect infestation, knots); surface coverings, moisture gradients. Critical factor: metals (e.g. nails) inside wood. **Masonry**: Salt deliquescence; Influenced by moisture gradients. Critical factors: layering; metals (e.g. nails) inside masonry.	Drilled cavity not recommended except in special circumstances under supervision of a qualified conservation professional. **Masonry**: Upper RH limit for measurements: deliquescence threshold of salt contaminants or 80 %.

Table B.2 — Comparison between relative methods: experimental uncertainties and notes

Electrical Resistance	Each particular object needs a specific calibration curve to relate arbitrary readings to the moisture content. However, calibration may not be representative of the material under investigation.
	This method is uncertain for substances that have a very high conductivity in the dry state, or in the presence of deliquescent salts.
	Problems may arise in the presence of density changes or moisture gradients in the subsurface layer.
	The accuracy depends on how the method is applied and the moisture content.
	The method exhibits appreciable temperature dependence, but this can be corrected with temperature measurement and calibration equations.
	The output is highly affected if the surface is contaminated with conductive materials, e.g. metals.
Capacitance	Each particular object needs a specific calibration curve to relate arbitrary readings to the moisture content.
	The output is determined by the number of water molecules and the distance from the sensor. Therefore, the result is strongly affected by the moisture content of the surface and subsurface zone.
	Problems can arise in the presence of density changes. Measurements are more reliable for materials with uniform moisture content than in the presence of moisture gradients.
	The output is not linear with moisture content and is influenced by the material type, in particular it increases with the density of the material, or in the presence of deliquescent salts.
	The accuracy decreases at high moisture contents.
	The output is highly affected if the surface is contaminated with conductive materials, e.g. metals.
RH in equilibrium with the material **Drilled cavity/ESB**	Each particular object needs a specific calibration curve to relate moisture content and measurements.
	Additional uncertainties are caused by inhomogeneity in the material. Numerous calibrations are needed.
	Each proxy has somewhat different characteristics and shall be individually calibrated. The response of the proxy changes over time (drift) and needs frequent calibrations.
	The accuracy decreases at high moisture contents.
	The presence of soluble salts affects the equilibrium.
	The ESB might affect the surface temperature.

WOOD: Resistance (B.2), Capacitance (B.3)

WOOD conditions	Calibration	Measurement
- not treated with wax or oil - not attacked by pests	possible →	good
- treated with wax or oil - not attacked by pests	difficult →	poor
- attacked by pests	impossible →	misleading
- metals inside	impossible →	misleading

Figure B.2 — Possibility of doing a reliable calibration and quality of measurements obtained with resistance or capacitance methods in wood. Numbers in brackets refer to the clause numbers

MASONRY: Resistance (B.2), Capacitance (B.3)

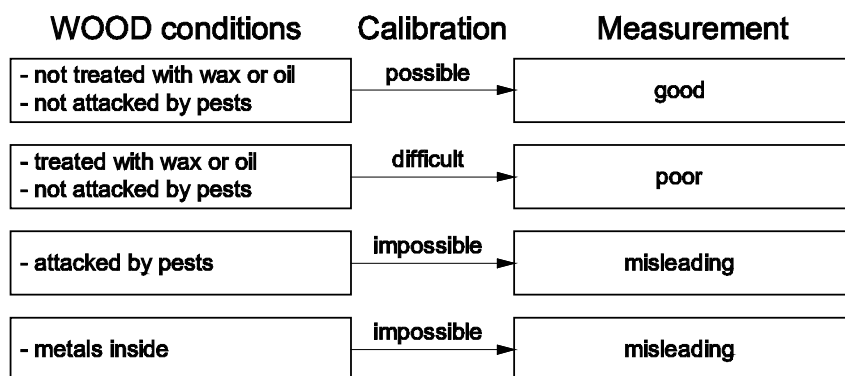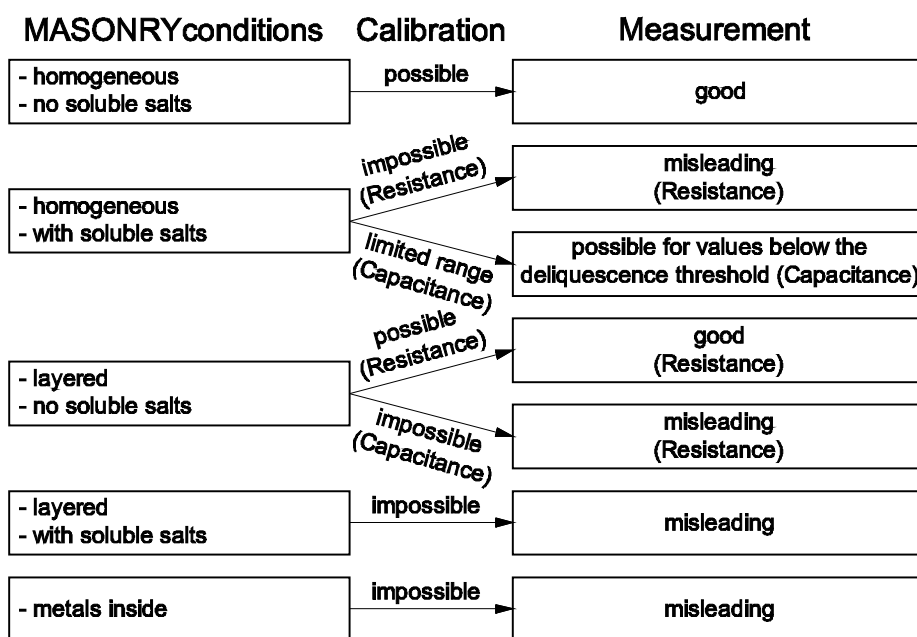| MASONRY conditions	Calibration	Measurement
- homogeneous - no soluble salts	possible →	good
- homogeneous - with soluble salts	impossible (Resistance) →	misleading (Resistance)
	limited range (Capacitance) →	possible for values below the deliquescence threshold (Capacitance)
- layered - no soluble salts	possible (Resistance) →	good (Resistance)
	impossible (Capacitance) →	misleading (Resistance)
- layered - with soluble salts	impossible →	misleading
- metals inside	impossible →	misleading

Figure B.3 — Possibility of doing a reliable calibration and quality of measurements obtained with resistance or capacitance methods in masonry. Numbers in brackets refer to the clause numbers

Annex C
(informative)

Other relative methods

C.1 Generalities

This annex is informative and concerns relative methods not covered by European Standards.

In order to provide a general overview and a cross comparison between relative methods, Table C.1 and Table C.2 will report the characteristics of the various methods, their experimental uncertainties and some notes concerning their specific use with wood or masonry. Table C.1 and Table C.2 are useful to assist the user selecting the most appropriate method and interpreting MC readings in the field of cultural heritage.

C.2 Microwave

This method is based on the dielectric property of the water molecule. An antenna assembly radiates an alternating electromagnetic field into the material in the order of GHz, i.e. in the microwave region. In materials with polar molecules (e.g. wood) the electric field of microwaves can induce oscillations while travelling through the medium. During this process, the travelling microwave beam, interacting with the material and the water molecules, loses energy, decreasing in power intensity, and has a phase shift.

While travelling within the medium, the intensity of the microwave beam decreases following an exponential law. However, for a number of disturbing factors in real materials, the response may vary from point to point, and also with time. In addition to absorbing energy or changing the phase, polar molecules reflect back a fraction of the energy from the incoming beam. As the interaction with the water molecules is stronger than the interaction with the other molecules of the material, the transmitted intensity decreases, the phase changes, and the back radiation increases, as the MC increases.

The method can be applied either to the transmitted beam intensity, or the phase shift, or the backscatter. It is easier to operate with the backscatter because emitter and detector are assembled together, and the operation of emitting and receiving is made from the same side, a particularly relevant factor when dealing with large structures.

The size of the material under investigation should be larger than the extinction depth of the beam, because the microwave beam should be completely extinguished within the material to avoid back reflection and interference.

The absorption increases with the moisture content, but the microwave frequency varies from material to material, and even within the same material. The part of the beam that reaches the opposite surface of the structure is reflected back and affects the reading. This limits the use to thick walls (e.g. 40 cm to 100 cm depending on the beam penetration depth and material type). It is rarely possible to use this method reliably on wood.

The microwave sensor should be in contact with the material surface. If there is no contact or the surface is rough, resonant standing waves are formed between the material surface and the sensor, generating misleading readings. The signal rapidly falls when the sensor is located at increasing distances from the surface under investigation.

If a cavity inside the building material is found (e.g. a fresco detached from the wall or hollowed bricks) resonant standing waves are formed in the cavity, affecting readings.

In the case of multi-layered structures (e.g. frescoes, plaster on brickworks), the dispersion or the back reflection of the microwave at every discontinuity between successive layers may be dominant. In practice, the building structure should be fully homogeneous, and this is rare in heritage buildings.

The influence from salts becomes negligible at frequencies higher than 1 GHz. However, if the salts reach the deliquescence level, and water molecules start to be absorbed, they act as a mirror for the incoming beam saturating the output.

The material under investigation should not include metals or traces of past metal coating, e.g. gilding, because metals reflect microwaves and affect readings.

The use is possible only in a limited number of cases where the wood thickness is larger than the beam extinction (e.g. 50 cm to 120 cm depending on the beam penetration depth for insect tunnelling, moisture content and wooden species).

The microwave propagation is not the same along the axial, radial and tangential direction of wood. The radial and tangential components are quite similar, but the axial component is different. As a consequence, measurements taken parallel and perpendicular to the grains may differ.

Lumber or squared log size should exceed the extinction depth of the signal to avoid back reflection of microwaves.

The beam intensity decreases with increasing moisture content, but varies with the microwave frequency, varies from material to material, and even within the same material, depending on the internal texture, wood grain angle, fungal infestation, insect tunnelling. A locally reduced wood density (e.g. insect tunnelling) might be interpreted as dry spots.

Readings are in arbitrary units, sometimes converted into % by mass. The output might be tentatively transformed into % gravimetric mode after calibration for the specific material made by comparison with gravimetry. Although automatic conversion or conversion tables provided by the manufacturers are useful, any calibration is difficult because of the changes that old materials might have undergone, due to ageing, chemical or biological attack. The test report should be produced in accordance with Clause 10.

C.3 Evanescent-field dielectrometry

Evanescent-field dielectrometry (EFD) is a recent diagnostic method based on dielectric spectroscopy at 1 GHz to 1,5 GHz microwave frequency. The measuring instrument is a portable resonant microwave device for mapping in a non-destructive way the moisture content and salinity on walls up to a depth of 2 cm to 3 cm in real time. It detects the MC and the salt concentration in frescoes and walls by estimating the dielectric properties of a wall that is viewed as a "binary" dielectric mixture consisting of bulk material and water, for the contrast between the dielectric constant ε of a dry wall (e.g. $\varepsilon < 4$ for mortar, plaster, brick) and water (ε approximately 80).

The material under investigation should not include metals or traces of past metal coatings, e.g. gilding.

Readings are relative, but might be tentatively transformed into quantitative values after calibration for the specific material made by comparison with gravimetry or KF titration. The method measures MC in the range 0 % – 20 % and provides semiquantitative information about the presence of soluble salts in terms of a salinity index from 0 to 10. The test report should be produced in accordance with Clause 10.

C.4 Time-domain reflectometry

Time-domain reflectometry (TDR) is based on the measurement of the time between transmission and reception of a radio signal. The system consists of a radio frequency transmitter (which emits a short pulse of electromagnetic energy), a directional antenna, and sensitive radio frequency receiver.

TDR's sensitivity to changes in the dielectric constant of material between two conductors has been adapted to the measurement of moisture content.

As seen in B.1, a small change in moisture content will have a significant effect on the bulk dielectric constant of the air-material-water medium. The speed of the electromagnetic radiation in this medium depends on this constant. So the apparent dielectric constant is obtained by measuring the time for a voltage pulse to travel along the probe and return.

The material under investigation should not include metals or traces of past metal coatings, e.g. gilding.

Readings are relative, but might be tentatively transformed into quantitative values after calibration for the specific material made by comparison with gravimetry or KF titration. The test report should be produced in accordance with Clause 10.

C.5 Nuclear magnetic resonance

The nuclear magnetic resonance (NMR) gives precise data about moisture content and distribution. The method is based on the angular momentum of the positively charged protons and their resultant magnetic moment. Hydrogen atoms immersed in a magnetic field behave as dipoles with energy levels related to their orientation within the field. The material under investigation is exposed to a large magnetic field; the hydrogen atoms within the material are then excited by a pulse of radio waves, and subsequently relax back to their normal state, emitting a characteristic signal (relaxation signal).

The measurement of the relaxation signal allows the number of hydrogen atoms present to be evaluated and, with appropriate calibration for the material, this can allow an absolute measurement of the water content. With NMR, the H_2O molecules contained in a material are subjected to static and oscillating magnetic fields at right angles to each other. NMR can distinguish molecules within materials based on their molecular mobility, i.e. the distance they move in a given time. The molecular mobility of free water is appreciably higher than that of bound water and so NMR can be used to provide an indication of the concentrations of water in "free" and "bound" states.

NMR can be performed in different ways, either destructively or non-destructively:

a) Portable NMR sensors should be used to investigate large objects on the site avoiding sampling. Quantitative moisture distribution mapping, or detecting moisture uptake and dynamics in wooden objects, frescoes, stones and masonry can be obtained. These investigations are non-destructive.

b) "Classic" NMR in the laboratory uses samples that are placed in NMR glass tubes (5 mm to 20 mm diameter) with a few milligrams of sampled material (micro-destructive).

c) MRI (magnetic resonance imaging) allows rather large pieces into an NMR instrument cavity; objects or samples size is limited by the cavity size.

Readings are relative, but can be transformed into quantitative values. The test report should be produced in accordance with Clause 10.

C.6 Near-infrared spectroscopy (NIRS)

NIR spectroscopy has been applied for measurement of MC, since overtone and combination bands of the hydroxy O-H group of water are pronounced in the NIR region of the spectrum, especially from 1,450 nm and 1,940 nm. NIRS operates in diffuse reflection and is usable for surfaces only (non-destructive). However, NIRS may operate with ground powder of samples of the building material (invasive/destructive).

NIRS is based on reflected radiation coming from the sample surface and is representative of the surface only. NIR reflection is affected by surface roughness and other nonlinear effects by water filled pores on the surface or incidence angle.

NIRS can detect the water content and activity in a wide range of materials, and over a large range of water content. The method is fast, non-destructive and non-contact (except for ground samples) and can be used for the real-time measurement of moisture, usable on site or in the laboratory.

In general, NIRS is a repeatable method, with high signal-to-noise ratio but needs precise calibration. Calibration should preferably be made with Karl Fischer titration; alternatively with a gravimetric method.

Readings are relative, but might be tentatively transformed into quantitative values after calibration for the specific material made by comparison with gravimetry or KF titration. The test report should be produced in accordance with Clause 10.

C.7 Ultrasound pulses

This method is based on the transmission or reflection of ultrasonic pulses. It measures the propagation of ultrasonic pulses through a material to detect either or both the time of travel and any change of intensity for a given distance. The propagation velocity is determined by measuring the length and the travel time (time-of-flight) of the ultrasonic wave.

Ultrasonic pulses propagate through a material at a velocity and with a transmittance that are a function of physical material properties, e.g. elastic modulus and density, including moisture content. The ultrasonic pulse velocity increases with MC and the transmittance decreases. The instrument can be used either in transmitted or reflected mode.

Readings are in arbitrary units; sometimes they may be converted into MC %, by mass or volume. Although conversion tables provided by the manufacturers are useful, calibration is difficult. The test report should be produced in accordance with Clause 10.

C.8 Thermography

The thermal imaging camera detects radiation in the infrared (IR) range of the electromagnetic spectrum (roughly in the 9 μm to 14 μm spectral band) and produces images of this radiation. The camera does not respond to visible light. It only captures IR radiation either emitted by a surface (related to the surface absolute temperature by the Stefan–Boltzmann and Planck laws), or reflected by a surface.

A modern camera is composed of an optical system, thermally controlled detector, amplifier, signal processor, input/output ports, including data storage devices, and display. The output is converted into a visual image, called a thermogram, or may be converted into a temperature map of the surface under investigation, when emissivity and environmental radiant temperature are known. Thermograms make areas of higher or lower surface temperature visible.

The camera is not directly sensitive to the water content, but to thermal radiation and may detect differences in temperature over space and over time, which might be generated by moisture. For this reason, the method cannot be calibrated.

As local differences in thermal conduction may alter the surface temperature and other different physical processes may interact with each other, it is sometimes misleading to think about the coldest areas as moist. Moisture improves thermal conductivity and forms a thermal bridge: for instance, thermal conductivity may be dominant over evaporation cooling and a damp area may appear colder or warmer, depending on the temperature on the other side of the same wall. This may lead to misleading interpretations.

Thermography may be used under stationary conditions (temperature not changing over time) or dynamic conditions (temperature increasing or decreasing). The approach may be passive (i.e. no deliberate heating), or active (i.e. deliberate warming of the surface under observation with heaters), as follows:

a) In the passive approach under stationary conditions, the assumption is that, at an evaporating surface, the latent heat subtracted from the surface might make it colder.

b) In the passive approach under dynamic conditions, the assumption is that a material shows higher thermal inertia in the presence of higher MC.

c) In the active approach under dynamic conditions, the assumption is that the surface is artificially warmed or cooled to monitor surface temperature differences while the heat is travelling through the sub-surface layer made inhomogeneous by an uneven moisture distribution or structural features.

Thermography cannot be used to measure quantitative values of MC but is a useful tool to localize damp zones. All of the above approaches may be useful to point out localized zones with higher MC, e.g. to detect percolation and the source of leakage. However, the information may be misleading. If a quantitative MC is needed, the investigation should be continued with other methods.

The test report should be produced in accordance with Clause 10.

Table C.1 — Comparison between other relative methods: characteristics

	Microwave	Evanescent-Field Dielectrometry (EFD)	Time-Domain Reflectometry (TDR)	Nuclear Magnetic Resonance (NMR)	Near Infrared Spectrometry (NIRS)	Ultrasound Pulses	Thermography
Material	Masonry (Wood)	Masonry	Masonry	Wood, Masonry	Wood, Masonry	Wood, Masonry	Wood, Masonry
Invasive destructive	No	No	No	No/Yes	No	No	No
Method sensitive to:	Space distribution of H_2O molecules, deliquescence, gradients, voids and other factors	Space distribution of H_2O molecules, gradients, voids and other factors	Space distribution of H_2O molecules, gradients, voids and other factors	Space distribution of H_2O molecules, gradients, voids and other factors	Absorbance bands typical of O-H bonds of water	Space distribution of H_2O molecules, gradients, voids and other factors	Space distribution of H_2O molecules, gradients, voids and other factors
Penetration depth	Core	Sub-surface	Sub-surface	Core	Surface (reflectance mode)	Core	Surface
Time response	Very fast (seconds)	Very fast (seconds)	Very fast (seconds)	Very fast (seconds)	Very fast (seconds)	Very fast (seconds)	Fast (minutes)
Lab/on-site measurements	On-site	On-site/ laboratory	On-site/ laboratory	On-site/ laboratory	On-site/ laboratory	On-site/ laboratory	On-site/ laboratory
Spot/ continuous time monitoring	Spot	Spot	Spot / Continuous monitoring	Spot	Spot	Spot	Spot
Pros	**Masonry:** Weak or no dependence on the salt content (except for deliquescence)	Large structures; Possible scanning at different depths using different probes	Weak dependence on the salt content.	Accurate determination of the amount of H_2O molecules. Imaging of the H_2O molecule distribution	Fast, non destructive. Accurate determination of small or large amounts of moisture	Not relevant	**Masonry**: Weak dependence on the salt content.
Cons	**Wood and Masonry:** Only plane surfaces and homogeneous mediums. Critical factors: thickness; metals. layering; internal cavities **Wood**: method Influenced by grain orientation; critical factor: very large thickness; **Masonry**: critical factor: salt deliquescence	Dependent on the material physical properties	Dependent on the material physical properties. Low measured volume. Need of perfect contact	Influenced by other H atoms	Representative of the surface only, affected by nonlinear effects	Influenced by: structural discontinuities, density changes, and moisture gradients. **Wood:** Influenced by grain orientation;	**Wood**: Required active approach, low sensitivity to MC **Masonry**: Environmental conditions affect the measurement

Table C.2 — Comparison between other relative methods: experimental uncertainties and notes

Microwave	Each particular material needs specific calibration to relate arbitrary readings to the moisture content. However, calibration may not be representative of heritage materials.
	The penetration of the microwave beam depends on the instrument, the medium and the moisture content.
	The sensor shall be in contact with the medium. The signal falls rapidly when the sensor is at some distance from the surface under investigation.
	Although the water molecules respond much more than other (polar) molecules constituting the "dry" material, nevertheless the material structure might interact with the beam.
	The extinction coefficient increases with the moisture content, but varies with the microwave frequency, varies from material to material, and even within the same material. The intensity of the microwave beam decreases from the surface to the deeper layers and the interpretation of readings may be difficult in the case of moisture gradients.
	The size of the material under investigation should be larger than the extinction depth of the beam.
	Internal voids or inhomogeneities (e.g. cracks) might provide back or multiple reflections and misleading readings.
	The result may vary with the skill of the operator.
Evanescent-Field Dielectrometry (EFD)	See Microwaves
Time-Domain Reflectometry (TDR)	Each particular material needs specific calibration to relate arbitrary readings to the moisture content. However, calibration may not be representative of heritage materials.
	The sensor shall be in contact with the medium. The signal falls rapidly when the sensor is at some distance from the surface under investigation.
	Although the water molecules respond much more than other (polar) molecules constituting the "dry" material, nevertheless the material structure might interact with the beam.
	Internal voids or inhomogeneities (e.g. cracks) might provide back or multiple reflections and misleading readings.
Nuclear Magnetic Resonance (NMR)	Can be sensitive to organic matter content.
	The output is relative, but can be transformed into quantitative values after calibration for the specific material made by comparison with gravimetry or KFT. Calibration holds for the particular item from which samples have been taken, not for other cultural heritage items apparently similar.
	Care should be taken to minimize health risks.
Near Infrared Spectrometry (NIRS)	Method generally sensitive and repeatable.
	It is affected by surface roughness, water filled pores and incidence angle.
	The output can be transformed into quantitative values after calibration for the specific material made by comparison with KFT. Calibration holds for the particular item from which samples have been taken, not for other cultural heritage items apparently similar.

Ultrasound Pulses	The speed of the sound waves varies with the moisture content, but varies also from material to material, and even within the same material, depending on the internal texture, e.g. fungal infestation, insect tunnelling, wood grain angle. Interpretation of the readings may be difficult in the case of moisture gradients.
	Each particular material needs a specific calibration curve. However, calibration may not be representative of (deteriorated) heritage materials.
	The sensor shall be in contact with the medium.
	Internal voids or inhomogeneities (e.g. cracks) might provide back or multiple reflections and misleading readings.
	The result may vary with the skill of the operator.
Thermography	Method to find damp zones and for semiquantitative information.
	The method is difficult to apply and active/passive thermograms are difficult to interpret.
	The result can depend on the physical conditions of the material surface and on a variety of environmental factors (e.g. present and recent solar radiation, cloud cover, wind, precipitation, air temperature and humidity, reflection or shadow from nearby bodies).
	On-site instrumentation should be high quality.
	Relative measurement only: results cannot be calibrated.

Annex D

(informative)

Methods with special safety requirements

D.1 Generalities

This annex is informative and concerns methods with special safety requirements that should comply with safety laws and IAEA regulations for protection against ionizing radiation and for the safety of radiation sources.

Measurements should be performed by qualified and certified personnel complying with ISO 20807:2004, ISO 9712:2012 and ISO/IEC 17024:2012.

In order to provide a general overview and a cross comparison between the methods listed in this annex, Table D.1 and Table D.2 will report the characteristics of the various methods, their experimental uncertainties and some notes concerning their specific use with wood or masonry. Table D.1 and Table D.2 are useful to assist the user in the choice of the most convenient method and in the interpretation of readings of MC in the field of cultural heritage.

D.2 X-ray

Radiography is a non-destructive imaging technique that uses X-rays to view the internal structure of an object. When the object is irradiated with an X-ray beam, the beam attenuation is due to the interaction with the material and the absorption by water. The assumption is made that the density of the dry material remains relatively constant, and that the only variable is the moisture content. The quantitative determination of the moisture content is based on the logarithmic subtraction of a reference image (e.g. sample at dry state) from images of the wet sample. The distribution and amount of moisture can be detected and measured with a scale of grey, or with the intensity of light passing through the radiography, or by contrast variation.

The general principle requires the sample to be exposed to a beam of radiation, with a sensor placed behind the material to measure the level of attenuation caused by the material and its internal moisture. This attenuation can be related to the moisture content within the material; this requires calibration of the material for absolute moisture contents to be measured. The method can be applied to analyse flows in porous materials.

The test method is non-destructive.

Synchrotron X-ray tomography is based on the detection of either the attenuation or the phase shift of the beam transmitted through a sample. While radiography measures images for a single orientation of the sample, tomography measures images for many different angular positions. Synchrotron-based X-ray tomographic microscopy (SRXTM) is nowadays a powerful method for non-destructive, high-resolution investigations of a broad kind of materials. High-brilliance and high-coherence third generation synchrotron radiation facilities allow micrometer and sub-micrometer, quantitative, three-dimensional imaging.

Readings in arbitrary units. They might be converted into % by mass after calibration for the specific material. The test report should be produced in accordance with Clause 10.

D.3 Gamma rays

Similar to X-ray but using more energetic radiation: gamma rays. The attenuation of a beam of gamma rays penetrating a material depends both on the material characteristics and the water content. The number of gamma photons is reduced according to an exponential attenuation law. The attenuation coefficient increases with increasing WC, reducing the beam intensity. Measurement of the attenuation gives an indication of the WC.

The mass density of a porous material (e.g. porous stone, clay brick, cement) can be measured by counting the number of gamma photons that are back-scattered to a detector by collisions with atoms of the material. The intensity of a beam of monoenergetic gamma emitted from a radioactive source (e.g. Cesium 137, Cobalt 60 and Americium 241) is attenuated depending on the material and its density. The assumption is made that the density of the dry material remains relatively constant, and that the only variable is the moisture content. Changes in wet material density are measured by the gamma transmission method and the WC is determined from this density change. The percentage of fractional change in the linear attenuation coefficient of the gamma beam due to presence of water is nearly equal to the WC of the sample and makes possible a calibration or conversion tables.

If measurements are made at two different gamma ray energies, attenuation equations may be solved simultaneously to provide both water content and material bulk density. By using the dual gamma method the accuracy of water content measurements improves compared to when bulk density is assumed to remain constant.

Readings are in arbitrary units. They might be converted into % by mass after calibration for the specific material. The test report should be produced in accordance with Clause 10.

D.4 Neutron scattering

Fast neutrons emitted from a radioactive source are launched inside a material. The fast neutrons gradually lose energy through collisions with other atoms. Hydrogen is the most effective atom to slow down neutrons because the masses of neutrons and hydrogen atoms are similar. Water molecules are the most widespread chemical compound including Hydrogen atoms, and are responsible for most of the slowing down. The number of slow neutrons is linearly related to the WC. A detector counts the number of slow neutrons per second and transforms it into moisture content. Synchrotron tomography provides accurate spatial resolution.

Readings in arbitrary units. They might be converted into % after calibration for the specific material. The test report should be produced in accordance with Clause 10.

Table D.1 — Comparison between methods with special safety requirements: characteristics

	X Ray	Gamma Rays	Neutrons
Material	Masonry, Wood	Masonry	Masonry
Invasive/ destructive	No	No	No
Method sensitive to:	Volumetric distribution of H_2O molecules and other factors	Volumetric distribution of H_2O molecules and other factors	Volumetric distribution of H atoms, in H_2O and other molecules and other factors
Absolute/relative readings	Relative	Relative	Relative
Penetration depth	Whole body	Whole body	Whole body
Time response	Fast response (seconds)	Fast response (seconds)	Fast response (seconds)
Lab/on-site measurements	On-site/ laboratory	On-site/ laboratory	On-site/ laboratory
Spot/ continuous time monitoring	Spot	Spot	Spot
Recommended use for cultural heritage	Large objects/ structures	Large objects/ structures	Large objects/ structures
Pros	Imaging Small to large structures; possible scanning at different depths	Very large structures; possible scanning at different depths	Readings can be directly related to moisture in masonry. Very large structures
Cons	Stringent requirements regarding protection against radiation. Dependent on the material physical properties	Stringent requirements regarding protection against radiation. Dependent on the material physical properties.	Stringent requirements regarding protection against radiation. Dependent on the material physical properties

Table D.2 — Comparison between methods with special safety requirements: Experimental Uncertainties and Notes

X Ray	Large variations in material density can occur and cause departures or limit spatial resolution. On-site instrumentation is difficult to use. Calibration is required for each specific material.
Gamma Rays	Large variations in material density can occur and cause departures or limit spatial resolution. On-site instrumentation is difficult to use. Calibration is required for each specific material.
Neutrons	Calibration may be difficult. Inadequate depth resolution makes measurement of absolute moisture content difficult. Absolute moisture measurement is difficult due to large, unpredictable proportions of fast neutrons escaping into the atmosphere. Interpretation of results can be difficult because the actual volume that is gauged by the detector is never precisely known. The gauged volume varies inversely with water content and water nearer the source has a greater effect on the shape of the volume.

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