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REVIEW

TOWARDS STANDARDISATION OF MOISTURE CONTENT MEASUREMENT IN CULTURAL HERITAGE MATERIALS

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Standards developed for industrial or commercial purposes cannot be applied to cultural heritage for two reasons: some methodologies are invasive/destructive; others are not destructive but have readings in arbitrary units and calibration is hardly possible for the specific case of cultural heritage materials that are weathered, chemically degraded, transformed, or affected by fungi or insects. This paper discusses the definition of moisture content for cultural heritage materials and its dependence on the measuring methodology, absolute and relative readings typical of various methodologies, the uncertainties of the most popular methodologies used by conservators and restorers, such as oven-dry weighing, calcium carbide pressure, conductive, capacitive, microwaves, equilibrium relative humidity, and the problem of calibration for cultural heritage materials.

1   Introduction

Temperature and especially relative humidity (RH) are key parameters related to conservation of cultural heritage.[1-4] It is well known that at high RH levels wood, paper and other hydrophilic materials uptake H_2O molecules from the air, increase their moisture content (MC) and swell. On the contrary, at low RH levels these materials release some water molecules, the MC decreases and they shrink. In hydrophilic materials, elevated RH levels are associated with elevated MCs that represent a favourable habitat for moulds. In practice, a dynamic equilibrium is established between the RH level in the air (the 'forcing factor') and the MC in materials (the effect). In some cases, the inverse mechanism is used, taking advantage of the elevated moisture capacity of some materials, e.g. to control RH in display cases by using silica gel or other moisture buffers.

The MC of a selected material is determined by the hygrophilic nature of the material and varies with the ambient RH and the material temperature. However, the direct relationship between RH and MC is complicated by a series of factors, especially under dynamic conditions, e.g. (I) condensation when the surface temperature decreases below the dew point; (II) hygroscopic salts adsorbing water, especially when the ambient RH exceeds the salt deliquescence level; (III) inertia due to heat and moisture diffusion coefficients inside the material when the ambient RH or the object temperature changes; (IV) in the presence of capillary rise, percolation, wetting from falling raindrops or melting snow. Under all of these circumstances the material MC is not simply related to ambient RH and a direct measurement of the MC is necessary.

The European Committee for Standardisation (CEN), Technical Committee for Cultural Heritage (TC346), recently established a standard concerning temperature and RH for conservation of hygroscopic materials, EN 15757: 2010. "*Conservation of cultural property — Specifications for temperature and relative humidity to limit climate-induced mechanical damage in organic hygroscopic materials*". This required two other standards, i.e. on how to measure temperature of air and of cultural heritage objects, EN 15758: 2010 "*Conservation of cultural property - Procedures and instruments for measuring temperatures of the air and the surface of objects*" and a standard on how to measure the relative humidity and the moisture exchange between air and cultural heritage materials, i.e. EN 16242: 2012 "*Conservation of cultural property – Procedures and instruments for measuring humidity in the air and moisture exchanges between air and cultural property*". The next standard will be focused on the measurement of MC in cultural heritage materials, i.e. "*Conservation of cultural property – Procedures and instruments for measuring moisture content in objects and building materials*".

A number of methodologies exist to measure the MC[5-9], but these have been conceived for modern buildings, agriculture, food, commercial or industrial purposes and are regulated by standardisation. However, not all of these methodologies are appropriate for cultural heritage, some of them being invasive or destructive. Some others are neither destructive nor invasive but they can hardly be applied to cultural heritage materials for two reasons: the instrumental readings are in arbitrary units (AU) and need interpretation and calibration to be related to MC[10-14]. In the following we will investigate whether calibration is feasible for cultural heritage materials.

This paper focuses on the most popular methodologies used in the field of cultural heritage. The matter is complex and this paper is intended to clarify various obscure aspects of MC measurement, starting from the definition, units in use, calibration and uncertainties related to instrumental measurements. The study ends by providing scientific support to a standard in preparation for the measurement of MC in cultural heritage materials.

2 The First Problem: Definition of Moisture Content in Materials

Surprisingly, a precise definition of MC in materials is missing in the existing standardisation and we should solve this problem by seeking for a theoretical definition of general use, or by relying on an empirical approach.

2.1 Exchangeable Water and Binding Forces

From the physical point of view, we could establish that "MC is the quantity of moisture found, in a given moment, inside a material as a result of exchange between the material and its environment". However, the statement is vague because "exchange" is not defined and cannot be applied to all material types. A refined definition would be that "MC is the quantity of water of external origin, contained in a non-soluble

material, additional to its chemical composition". This definition seems to be acceptable but is limited to non-soluble materials. When a material has lost its MC, it should be considered 'dehydrated' or simply 'dry, but we will see later that this apparently obvious statement is not clear when some particular hydrated crystals or organic materials are concerned.

The above MC definition refers to water molecules that are not part of the dry composition of the material and are in a way temporarily bound to it. The difference between external water that entered the material from structural water that is an essential part of the material is in the strength of the binding forces.

Binding forces may be weak, medium or strong, and the related water is respectively called 'free water', 'bound water' and 'structural' or 'crystalline water'. From this point of view, one might suppose that exchangeable water, mostly governed by ambient RH, should correspond to the weakest bonds, i.e. 'free water'. However, this is not always true. Wooden artefacts kept indoors have no 'free water' and the equilibrium with the room RH is reached by exchanging molecules of water that are bound to the cell walls via hydrogen bonds, i.e. 'bound water'. The standard methods, which are used to measure the moisture content in wood, allow determining the total amount of bound and free water.[15] Again, some minerals have hydrated or dehydrated crystalline structure depending on some thresholds in temperature and RH, e.g. gypsum ($CaSO_4 \cdot 2H_2O$) and anhydrite ($CaSO_4$), mirabilite ($Na_2SO_4 \cdot 10H_2O$) and thenardite (Na_2SO_4), magnesite ($MgCO_3$) and Mg-carbonates (e.g. $MgCO_3 \cdot 3H_2O$ and $(MgCO_3)_4 \cdot Mg(OH)_2 \cdot 4H_2O$). This means that 'free', 'bound' and even 'structural' water may be exchanged with the environment to reach an equilibrium, with microclimate driving the process. At this point, we should need to abandon any physical definition based on binding forces to investigate other empirical approaches.

2.2 Gravimetrically and Volumetrically Based Definitions

Suppose that we know the amount of moisture m_w absorbed inside a selected material of volume V. The MC can be expressed in one of two forms:

(I) in terms of mixing ratio between m_w and the mass m_d of the dry material, i.e. m_w/m_d,

(II) in terms of density, i.e. the ratio between m_w and the volume into which it is dispersed m_w/V.

The former is called *'gravimetric'* and is independent from how water is distributed inside the material; the latter is 'volumetric' and depends on the spatial distribution of the water molecules. It is possible to establish a clear relationship between the two definitions only for materials with constant and homogeneous density and in the absence of moisture gradients inside the material, which are related to each other by a proportionality coefficient.

Gravimetry is based on the determination, by weighing, of the weight lost by a specimen after it has been fully dried in a ventilated oven at a temperature above boiling, i.e. until it reaches equilibrium with the

oven[16-19]. The loss of weight corresponds to the moisture escaped from the specimen and will be expressed as a percentage of the mass of the oven-dried specimen.

Volumetric determinations are based on interaction between radiation (e.g. electromagnetic field, ultrasounds) or a beam of particles (e.g. neutrons) penetrating the material, with H_2O molecules in the volume investigated. Typical volumetric methods are based on electric conductivity, capacity, microwaves, ultrasounds, EFD, NMR, X or gamma rays, neutron scattering. A crucial point is to verify whether the gravimetric and the volumetric definitions will always provide the same, or at least comparable results.

For commercial or industrial products that are homogeneous, the volumetric and the gravimetric definitions are equivalent, and the choice is subjective. However, in the case of cultural heritage materials the choice is critical because it may lead to misleading conclusions. Let us consider a wooden statue that has its MC in equilibrium with the ambient RH unchanged for a long time and remaining unchanged for the duration of our experiment. Suppose that woodworms infest the statue: the larger the volume of voids, the lower the bulk wooden density. When excavating tunnels, woodworms do remove both moisture and dry wood in the same proportion; however, the external volume of the statue remains the same. If we will measure the MC in gravimetric terms, the MC value is independent from insect tunnelling. If we will measure the MC in volumetric terms, we will find lower MC values. Same as insect tunnelling, mould increases porosity and/or formation of internal cavities in cultural heritage materials, objects or structures (e.g. wood, books, weathered masonry, oxidised metals). The gravimetric definition is preferable.

2.3 Measurable Water and MC Definition Based on Existing Standards

Although we have established that a definition of MC based on the mixing ratio between the mass of water and the mass of dry material is preferable, we have not yet established how to define and distinguish the 'dry' material from the moist one and, consequently, the quantity of absorbed moisture. The starting point is that every physical quantity should be *measurable*; any value should be obtained with a precise quantitative measurement and any definition should be consistent with the result of measurements. The problem is that any measurable quantity depends on the methodology chosen to measure it, and this is a critical factor, especially in the field of standardisation.

In practice, the MC is the moisture we can detect and measure. This basic statement is clear when only one measuring method is available, or when all of the possible measuring methodologies provide identical results. A serious problem arises if different methodologies provide different results, as we will see later.

A number of European standards exist concerning the MC measurement:

(I) EN 322: 1993 "Wood-based panels – Determination of moisture content".

(II) EN 13183-1: 2002 "Moisture content of a piece of sawn timber - Determination by oven dry method".

(III) EN 13183-2: 2002 "Moisture content of a piece of sawn timber- Estimation by electrical resistance method".

(IV) EN 13183-3: 2005 "Moisture content of a piece of sawn timber - Part 3: Estimation by capacitance method".

In practice, EN recommends three methods: weighing (EN 322: 1993 and EN 13183-1: 2002), conductivity[20] (EN 13183-2: 2002) and capacitance[21] (EN 13183-3: 2005). However, in the capacitive and microwave methodology, the electric field generated by the sensor penetrates up to a certain depth inside the material and the molecules of water, that are polar, interfere with the electromagnetic field providing a signal that determines the output. The polarity of the water molecule results in a high dielectric capacity $\varepsilon = 80$, which is considerably higher than the dielectric capacity of materials, generally in the range from 2 to 10. The dielectric capacity of materials is due to the presence of radicals or other polar components of the molecule. Wood includes polar oxygen–hydrogen (-OH) groups and is overall weakly polar. As a consequence, the dry material will generate a background signal, over which the signal due to water molecules is added. The threshold value corresponding to the dry material background is not known, except that oven-dry gravimetry can be used for comparison and calibration using the same specimens under exactly the same conditions.

European standardisation does not specify which methodology is preferable, or if they are equivalent to each other. This is irrelevant for commercial materials because calibration may solve the problem. In the case of cultural heritage, the various methodologies do not provide identical results because weighing is based on gravimetry and is independent from the material density and the internal distribution of moisture; electric resistivity is based on the conductivity of the surface or subsurface layer and is highly affected by surface treatments or soluble salts; capacitance is affected by the spatial distribution of the molecules of water, their distance from the electrodes and the dielectric capacity of the material. In conclusion, we will get three different readings but we cannot say which one is more relevant.

In this respect, ASTM standards are better. The ASTM D4442-07 "*Standard test methods for direct moisture content measurement of wood and wood-based materials*" makes a clear distinction. Oven-dry gravimetry is recognised to provide the highest accuracy and is defined a the 'primary' method, recommended as a reference for calibration of other methodologies. Other methods, e.g. capacitive resistive, distillation, are considered 'secondary' and are allowed in the case that the meters are calibrated by comparison with gravimetry with direct measurement as indicated in the standard. The oven-dry method is intended as the sole primary method. The secondary methods are intended for special purposes or in circumstances where the primary procedure is not desired or justified.

2.4 The Oven-dry Definition

The EN 322: 1993, EN 13183-1:2002, ASTM D4442 - 07 and ASTM D2216 - 10 standards are based on gravimetry, and this methodology should be considered as the most convenient to measure the MC. The above standards don't provide any theoretical definition of the MC. They only establish how to calculate the MC, expressed in percent (%), and the recommended formula constitutes an implicit mathematical definition:

$$MC = (m_H - m_0) / m_0 \times 100,$$

where m_H is the initial mass of the test piece and m_0 the mass of the specimen after ventilated oven drying at a temperature of (103 ± 2) °C until constant mass has been reached. The constant mass at oven equilibrium m_0 is by definition the 'dry' mass m_d, and the difference $m_H - m_0 = m_w$. In practice, the above method and the formula constitute the primary reference.

The definition of MC is strictly related to "the water extractable by oven drying at above boiling temperature until the material has reached equilibrium". This means that the material has lost all moisture, or needs more energy at higher temperature to release other molecules of water, in the case they exist with stronger bounds.

However, at above boiling temperature or/and at low RH, unstable hydrated minerals may slowly dehydrate and organic materials may decompose. For unstable materials, ASTM D2216–10 establishes lower oven temperature (e.g. 60 °C) not harmful to sensitive materials, or recommends the use of desiccators at room temperature.

3 Absolute and Relative Readings

The MC reading obtained after oven-dry gravimetry is expressed in % and constitutes an *absolute reading*. It gives a precise, repeatable quantitative evaluation of the MC. Another methodology that provides *absolute readings* is based on calcium carbide[22], and is regulated by ASTM D4944 -11. This is a subsidiary method for determination of water content from the gas pressure developed by the reaction of calcium carbide with the moisture of the material (e.g. stone, brick, masonry) which is crushed, ground and reduced to coarse powder. The scale of the pressure gauge is calibrated in comparison with gravimetry, and gives the percentage of water in the total mass of wet material, but limited to the particular material used for calibration. The reading in percent of the wet material is slightly different from the percent of the dry material of oven-dry gravimetry. However, it is possible to convert the water content ($m_{w,w}$) obtained on the wet mass basis to the water content (m_w) on the dry mass basis with the equation:

$$m_w = m_{w,w} / (100 - m_{w,w})$$

and return to the basic definition in %. However, the calcium carbide is less precise, limited to some inorganic building materials (e.g. soil, rock, stone, brick, mortar, concrete) and is destructive. For this reason it will be disregarded in the following.

Other methodologies, e.g. electric conductivity or capacitance measurements, microwaves, ultrasound, EFD, thermography, X or gamma rays, neutron scattering, typically expressed in AU, provide *relative readings*, useful to see whether the MC in a given position or a given time is higher or lower than in another, without knowing the actual difference in absolute terms. However, it is sometimes considered that it is possible to pass from relative readings expressed in AU to absolute readings in %, by calibrating the instrument with reference to gravimetry. In the following we will discuss if this is possible for cultural heritage and within which limits.

4 Materials and Methods: Uncertainties of the Most Popular Methodologies

Various methodologies exist to detect quantitative or qualitative levels of MC and there is abundant, although incomplete literature. The most popular methods in use by architects, conservators, restorers and archaeologists are: oven-dry weighing[23], calcium carbide pressure[24,25], conductivity[26-31], capacitance at low or high frequency[10,27,31-35], microwaves[36-38], equilibrium RH[39]. Other methodologies exist for research purposes but they will not be the scope of this paper but are briefly described in the Appendix, e.g. ultrasounds[40-42], thermography[12,43-45], evanescent-field dielectrometry[46] (EFD), nuclear-magnetic resonance[47-50] (NMR), X or gamma rays[51,52], neutron scattering[39]. A comparison of various methodologies is reported in the literature[5-7,9,11,12,31,39,53-55]. Every methodology is differently affected by various factors that increase uncertainties in calibration and during field use.

In the following we will discuss the uncertainties of the most commonly used methodologies, seeking to fill the main knowledge gaps. Tests performed in the laboratory and results of field surveys will be presented and discussed to elucidate problems and typical behaviours of the most popular instruments.

4.1 Conductitivy Measurements

Electrical conductivity varies with the moisture content and subsurface moisture gradients; the accuracy decreases at high moisture contents. Readings are representative of the surface (contact electrodes) or sub-surface layer (implanted nail-pins) up to the depth reached by the pins. Problems can arise in the presence of moisture gradients in the subsurface layer. If the subsurface is wetter compared with the deeper layer, the output is mostly determined by the subsurface layer and less affected by the depth reached by the pins. If the subsurface moisture is homogeneously distributed in depth, the output will slightly increase with the depth to which pins will penetrate. If the interior is moister than the surface, the output will increase as far as the pins will reach inner layers richer in moisture

Conductivity is affected by the presence of ions derived from the dissolution of soluble salts contained inside porous stones or masonry or transported by capillary suction or other mechanisms. Efflorescence and subflorescence strongly affect measurements,

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which apparently provide higher dampness levels depending on the salt type and concentration. This is particularly critical on the front of rising dampness or on the borders of percolations, where the concentration of accumulated salts is elevated and variable.

In wood the conductivity is strongly dependent upon the grain orientation and past treatments with wax or oil that may alter conductivity. In general cracks, mould rotting, insect tunnelling decrease conductivity and the specimen appears drier.

The capacitive, microwave and EFD methodologies are respondent to internal distribution of moisture, but also to material defects and inhomogeneities.

4.2 Capacitance Measurements

The electric field between electrodes decreases with the square of the distance form the electrodes and vanishes at the distance of a few centimetres. Capacitive electrodes are often fed with high-frequency alternative voltage pulses and emit a high-frequency electromagnetic field similar to microwaves (see below).

In practice, the measurement responds to surface and subsurface moisture only and the instrument output is determined by the number of water molecules encountered in the space covered by the electromagnetic field and, in addition, by their distance from the electrodes. Readings may be reliable on materials with a fairly uniform MC, less in the presence of moisture gradients. The output is not linear with MC; the accuracy decreases at high MC. The output is influenced by material type.

In masonry, for a wide range of frequencies, capacitance is almost independent from the presence of soluble salts, except in the case of deliquescence. In the presence of efflorescence, when the RH exceeds the critical level for deliquescence, soluble salts absorb moisture from the atmosphere and dissolve. Laboratory tests with 0.004 g cm^{-2} homogeneously spread water over the surface showed that the thin aqueous solution behaves as a mirroring surface that will reflect the signal, giving the appearance of a completely wet material.

The methodology is sensitive to the presence of cavities, internal discontinuities or metals in the masonry. If a plaster, or a fresco, is locally detached from the wall, the output decreases as if the surface were drier.

In wood, the contact electrode can be placed with any grain orientation on the side grain of wood with little effect on the readings. It should not, however, be applied on the end grain. The output is dependent on the wood density, e.g. wooden species and each species should have a specific calibration or correction factor, and this is usual for commercial timber. However, local density changes are influential, i.e. a knot is interpreted as a local peak in MC and the presence of subsurface voids due to woodworms is interpreted as a local decrease in MC, i.e. dryer wood. Laboratory tests were made to verify the response of a capacitive instrument to voids due to woodworm attack, using blocks of homogeneous wood in which
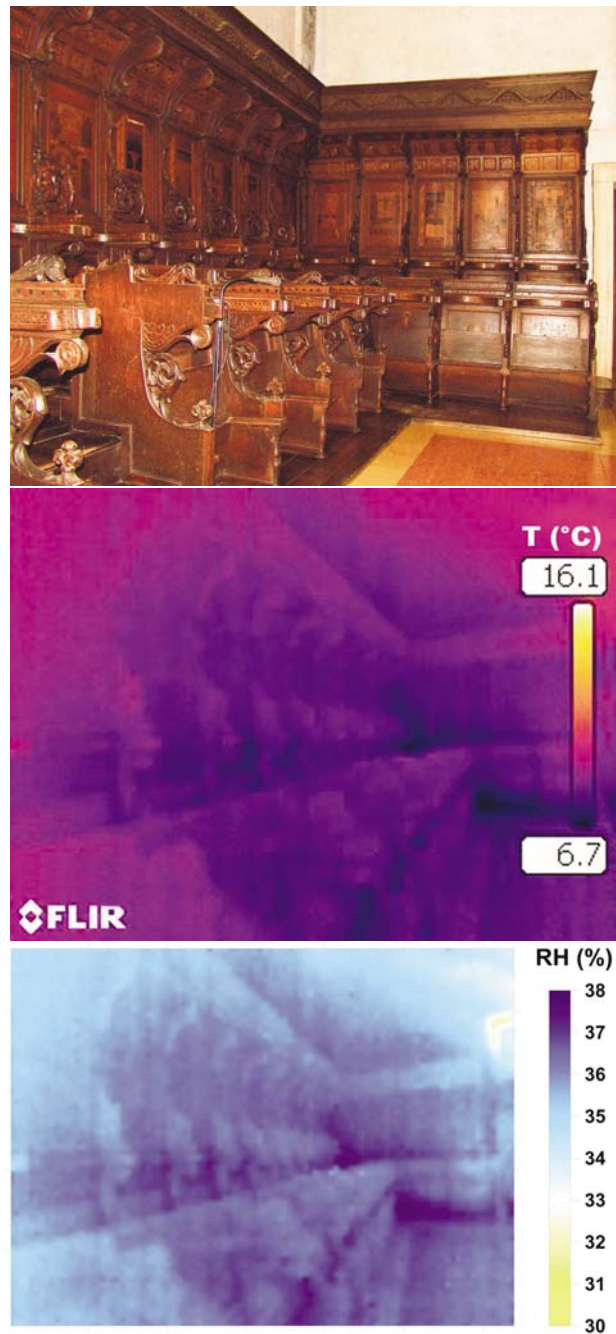

Figure 1: (a) Photograph of a 15th century choir with inlaid wooden stalls. (b) Thermogram (IR image) of the choir showing the homogeneous distribution of surface temperature. (c) Image of the RH within the viscous air sublayer in contact with the wooden choir surface.

voids were simulated by drilling the wood with calibrated drills in a regular grid. To reach general validity, the results were expressed in a non-dimensional form, X, representing the ratio between the volume of internal cavities and the total volume, and Y(X) the ratio of the signal in AU corresponding to the cavity level X, divided by the value for the same wood block in a non drilled part. The result was that readings Y(X) decrease in value from 1 to 0 with an increasing cavity volume from 0 to 1, following the linear equation Y = 1-X. In conclusion, instrument readings decrease with an increasing cumulative volume of voids caused by woodworm.

A wooden surface of the back panel of a wooden choir stall from the 15th century was investigated (Figure 1a), in an unheated monumental church with a stable indoor climate. The room temperature follows a seasonal cycle smoothed by the thermal inertia of the thick walls, and the stalls are in a thermal equilibrium, with the surface temperature homogeneously distributed, as in Figure 1b. The RH in the room is also stable and homogeneous, as shown in Figure
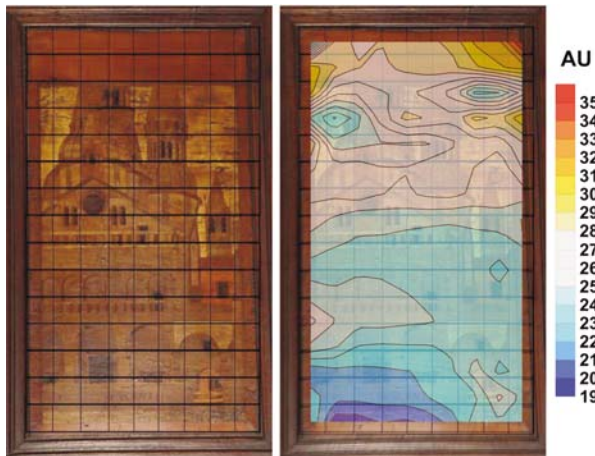
1c. This figure represents the RH within the viscous air sublayer in contact with the wooden choir surface calculated pixel by pixel from the thermal image and the humidity mixing ratio in the room. The stall panel selected for this example represents the urban church of St. Antony in Padua (Figure 2a). The stall panel is composed of a back panel base, 3 cm thick, onto which inlaid decorations are fixed, of 0.15 cm thickness. The capacitance measurement instrument had a spherical probe to make precise spot readings and was moved along a regular grid (cf. the image) on the wooden surface of the panel representing a regularly spaced sampling pattern. As the capacitance instrument averages the signal over the depth of 3 cm, disturbances due to the changes in the wood species used to obtain the decoration is not relevant because they contribute a negligible thickness, i.e. 5% of the total. Also, only a small surface area is in contact with the probe, which the electromagnetic beam will spherically expand into the subsurface layer. The map in Figure 2b, obtained by mapping the readings, is substantially determined by the internal voids produced by woodworms. The scale is in AU: the lowest values, i.e. blue, correspond to lower wood density; the highest values, i.e. red, to a smaller volume of voids.

Figure 2: Map of an inlaid wooden choir stall (15th century) obtained with a capacitance instrument. The stall is in equilibrium with an unheated room at constant temperature and RH, and the map responds to the subsurface insect damage.
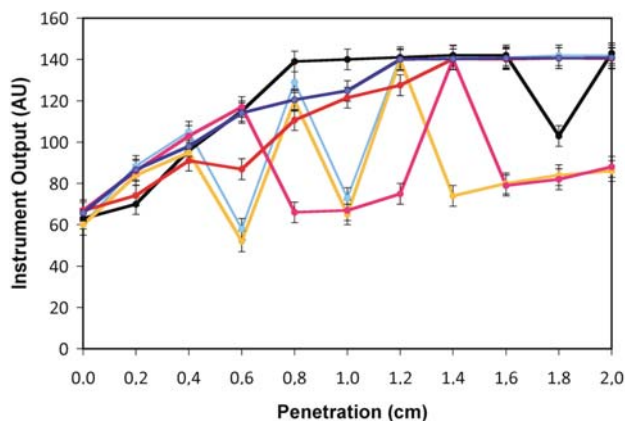

Figure 3: Laboratory test showing the response of a spherical capacitive instrument pushed inside a soft material (stucco).
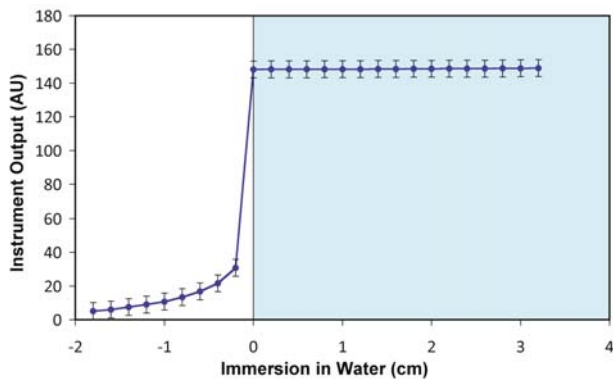

Figure 4: Laboratory test showing the response of a spherical capacitance instrument immersed in water. Negative immersion values (white background) indicate the probe approaching the water surface; positive values (cyan background) indicate actual immersion in water.

The spherical probe might penetrate into concave, rough or soft materials, and in such cases the number of H_2O molecules reached by the sensitive electromagnetic field is increased. A test was made to investigate the instrument response when the probe (sphere diameter 1.2 cm) is progressively pushed inside a soft material, e.g. soft stucco (Figure 3). The output was increasing with the probe penetration until 1/3 of the sphere penetrated inside the material. After this limit, strong instability was observed, with readings widely scattered.

The experiment gave slightly different results when performed with water, fully enveloping the spherical probe. The probe was moved vertically until it reached the water surface and was immersed in it. While approaching the water surface, the probe generated a weak signal; after contact and full immersion, the output was stationary at plateau level (Figure 4).

4.3 Microwave Instruments

The penetration of a microwave beam depends on the instrument, the medium and the MC. The emitting/receiving antenna should be in a close contact with the medium to avoid wave attenuation due to dispersion and interference of the incoming beam with the one reflected by the target object surface.

Although H_2O molecules are more responsive than compounds forming the 'dry' material, the latter cannot be neglected because the material structure might interact with the beam. Measurements are possible only when the object size is larger than the extinction depth of the beam, because the microwave beam should be completely absorbed in the material. If the path of the beam within the medium is shorter than the extinction depth, a fraction of the microwaves that reach the back side of the object will be reflected on the internal surface which reinforces the backscatter signal, as if the material had a higher moisture content.

The microwave beam is exponentially attenuated whilst travelling within a medium. 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, depending on the moisture content and the internal texture, e.g. wood grain angle. The surface and subsurface layers, being closer to the emitter and the detector, are more influential than deeper layers, and the reading interpretation may be difficult in the case of moisture gradients.

A critical problem is the presence of voids in the material that might provide misleading readings. Common examples are a fresco or layer of plaster detached from the brick wall or a deep crack inside a piece of wood. In the case of a microwave beam of the wavelength λ, resonant standing waves might form within the cavity, causing internal reflections and beat superposition. This complex situation makes the detection and the interpretation of such discontinuities uncertain.

A test has been made to investigate the instrument response to the presence of a thin or wide cavity inside the material, e.g. plaster detached from the wall, or a large crack inside a timber structure. The cavity of the width L was formed displacing two mobile vertical slabs of the same material initially in contact, i.e. $L = 0$ (Figure 5a). When the gap is increasingly wider, the back reflection will cause interference with the incoming waves and the output will swing as an attenuated sinusoid giving the apparent features of dryer or moister material. Standing waves (Figure 5b) are created when the gap width is $L = n\,\lambda/2$, where $n = 1, 2, 3\ldots$ where $n = 1$ represents the fundamental mode and $n = 2, 3\ldots$ the harmonic overtones. The peak intensity of maxima (i.e. the less affected levels) occur at $L = n\,\lambda/4$, where $n = 2, 4, 6\ldots$ (i.e. even multiples of $\lambda/4$) represent the beam intensity in the absence of interference when both borders of the gap are at nodal points. The intensity of maxima is progressively reduced whilst the beam penetrates deeper into the material. The backscatter minima will be found at $L = n\,\lambda/4$, where $n = 1, 3, 5\ldots$ (i.e. odd multiples of $\lambda/4$ when the borders of the gap are one at the node and the other one at the antinode).

The probe with the emitter and the receiver should be in perfect contact with a flat surface of the material. This is not always possible because the surface may be rough or curved and the signal will be affected by a distortion that depends upon the (average) distance between the probe and the target surface. A test made in the laboratory shows that, when the probe is placed at increasing distances from the material surface, the signal vanishes at $\lambda/4$,
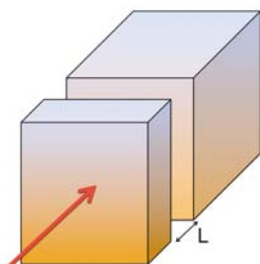
Figure 5a: Apparatus to test the instrument response to the presence of a thin or wide cavity inside the material. The cavity was formed displacing two mobile vertical slabs of the same material initially in contact. The mobile cavity width L is the distance between the two slabs. The red arrow indicates the penetration of the microwave beam.

where λ is the microwave wavelength, and is then followed by a series of smaller peaks and drops spaced $\lambda/8$ between them (Figure 6). If the probe is not perfectly in contact with the surface, even relative comparisons might be misleading.

Another laboratory test was made to measure the response of a microwave instrument with a large penetration depth (e.g. 60 cm), with a thin efflorescence (e.g. 1 mm thick) on a wall. The influence of salts becomes negligible for frequencies higher than 1 GHz and the efflorescence is almost not noticable when RH is below the salt deliquescence level. One might suppose that the efflorescence thickness is very thin, apparently negligible in comparison with the beam penetration depth and the amount of water absorbed in the efflorescence is a very small fraction of the whole moisture content into the bulk material. However, when the RH exceeds the deliquescence level, the thin aqueous film that forms on the surface reflects the signal giving the appearance of a completely damp material.

A popular practice is to measure the subsurface MC with a capacitive instrument (e.g. 2 to 4 cm penetration depth) and the internal MC with a microwave instrument (e.g. 30 to 40 cm penetration depth) in order
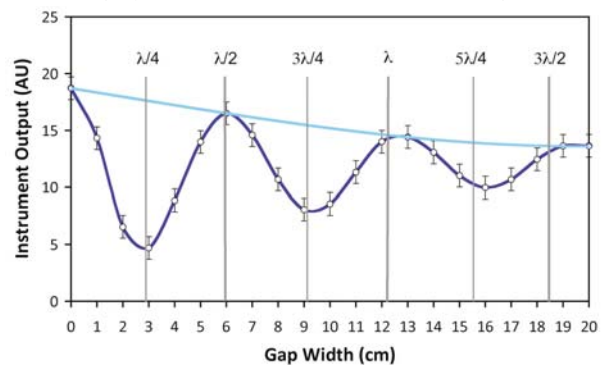

Figure 5b: Output of a microwave instrument (blue line) showing interference between emitted and backscattered waves when a gap with increasing width is created between two mobile slabs inside the material. The natural attenuation of the signal in the absence of interference whilst microwaves are progressing within voids corresponds to the cyan line connecting peaks. Microwave frequency: 2.43 GHz, wavelength $\lambda = 12.345$ cm. Peaks and drops are found at multiples of $\lambda/4$.
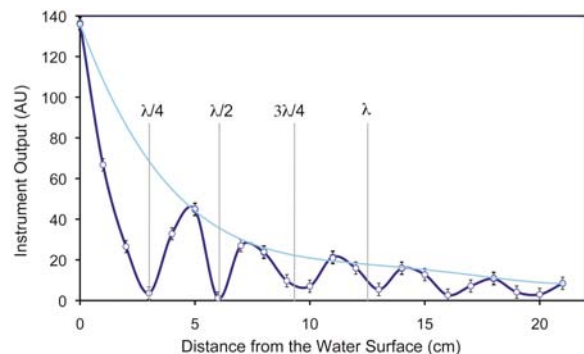

Figure 6: Attenuation of the instrument output whilst increasing the distance of the probe from a water surface (blue line). Microwave frequency: 2.43 GHz, wavelength $\lambda = 12.345$ cm. The output vanishes at $\lambda/4$, $2\lambda/4$, $3\lambda/4$, and then it undergoes an increasing shift and distortion due to other factors. The cyan line indicates the peak envelope.

to determine if the surface layer is drier or moister. It is methodologically incorrect to determine moisture gradients by the cross comparison of readings generated by two instruments with different penetration depths and based on different physical principles. Both instruments provide relative readings in AU, we know that no precise calibration is possible, and that we cannot cross compare independent AU readings although we might find that the manufacturers have expressed both in %, implicitly suggesting that they are comparable.

4.4 Equilibrium RH

This methodology corresponds to the variability of the water vapour pressure before saturation is reached at a given temperature. Water vapour reaches saturation (i.e. RH = 100%) when a film of liquid water forms inside pores, the pores still being empty. It is not possible to provide any information about when the pores are progressively filled with liquid water and the material becomes heavier and heavier, being increasingly soaked with water. Therefore, the range of MC detected by the equilibrium RH methodology is only a small portion of the range covered by gravimetry. However, this methodology is convenient for following MC over time, e.g. drying of a brick wall after flooding (Figure 7). The Southern and Western walls are damp, the Northern wall is rapidly drying, the Western wall is almost stationary at around RH = 75%, typical of NaCl deliquescence level and is in danger of repeating dissolution-crystallisation cycles.
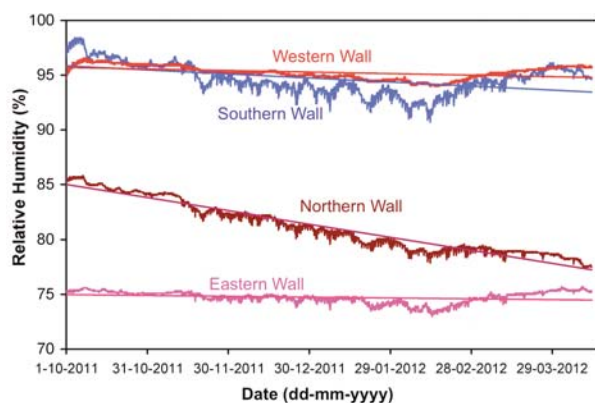
5 Discussion: Calibration of Cultural

Figure 7: Equilibrium RH used to monitor how the brick walls of a church dry over time after the church was flooded by an exceptionally high tide. The straight lines indicate the linear interpolation of the records, showing trends.

Heritage Materials

The only methodology able to provide precise quantitative results is the gravimetric method. This method, obtained by weighing specimens, responds to the total number of free water molecules in the specimen, irrespectively of how they are spatially distributed within it. However, such analysis is invasive/destructive, so gravimetry should be excluded for cultural heritage. On the other hand, various non-destructive methodologies exist to evaluate the MC materials, but

the readings are essentially expressed in AU hardly comparable between them and with gravimetry as well. In order to solve the problem of obscure AU, some instruments are calibrated by comparison with gravimetry, and their scale is expressed in %. However, a number of problems arise as follows.

5.1 Reference Specimens for Calibration

Calibration cannot be done using commercial materials simulating aged cultural heritage materials. Materials in use for commercial purposes have precise chemical and physical properties as a result of selection and/or processing of natural/artificial products, often regulated by standardisation. This is not true for cultural heritage materials. Cultural heritage materials will likely have different physical or chemical properties, the material being aged, altered, deteriorated, with surface or internal transformations, cavities or density losses, caused by corrosion, oxidation, cracking, rotting, insects etc. A virgin material used for such calibration will not be representative of the target heritage object. For instance, ancient wood has chemical and physical properties different from a recently harvested piece of timber due to oxidation, hydrolysis, dehydration, reduction and free radical reactions and macro/micro cavities as a consequence of woodworm or mould[56,57,58] that have a strong impact on subsurface volumetric monitoring. This is particularly relevant e.g. for capacitance sensors with a typical penetration depth of 2-4 cm. Ancient masonry is also made with bricks of particular composition and porosity, and the same can be said for mortar and plaster. In addition, masonry has undergone deterioration weathering and salt impregnation. In conclusion, ancient materials are very particular and/or have been transformed over time. As a consequence, each statue or part of the same statue, each painting or each wall needs its own calibration.

5.2 Repeatability of Calibration

Calibration made with cultural heritage materials is destructive, not repeatable, and is only valid for the particular object used for calibration. Gravimetry requires invasive/destructive sampling, and this is not ethically acceptable. However, one could consider that instrument calibration is needed only once, and needs only a limited number of specimens and the sacrifice of a few samples is justified by the advantage of having a calibration curve that is valid forever. This thought is supported by analogy with medicine, where an invasive analysis may be necessary to save a life. Even in the case that a reasonable calibration curve is obtained, and the MC is homogeneously distributed, the changes caused by time and the environment to an object may lead to local variations of the material response, so that even a relative comparison of readings may become misleading. In addition, every calibration is valid for a specific sample and a limited period of time because the deterioration mechanisms may continue altering the characteristics of the material.

5.3 Real Conditions Differ from Laboratory Conditions

Laboratory calibration under controlled conditions is unrealistic not only due to the particular chemical and physical transformations and decay of aged materials. Specimens used for laboratory calibration are acclimatised in a climate chamber, with moisture homogeneously distributed. In the real world, however, cultural heritage materials are likely affected by subsurface gradients in porosity, density, temperature and moisture, so that the actual conditions will be different from the calibration. Any calibration will have a limited temporal validity and will be affected by large uncertainties in any case.

5.4 Comparing Methodologies Based on Different Physical Principles

It is theoretically incorrect to perform calibrations by comparing methodologies based on different physical principles. This because the measurable physical quantities to which sensors respond are different, specific for each particular method. The measured value depends on the chosen methodology. For instance, the oven-dry gravimetry measures extractable water; the capacitive methodology, microwaves and EFD are respond to electromagnetic interference with radicals or polar molecules. In addition, gravimetry is independent from the spatial distribution of moisture, and the majority of the other methods are not, most of them being based on volumetric detection. The capacitive, microwave and EFD methodologies respond to the presence of H_2O molecules, but the output depends on their spatial distribution and material homogeneity. The comparison of a volumetric with a non-volumetric methodology is possible only in the case of perfectly homogeneous material density and absence of moisture gradients.

6 Conclusions

Existing standards useful to measure the MC in commercial materials cannot be applied to cultural heritage materials for two reasons. One is that the most accurate methodology, i.e. oven-dry gravimetry, is invasive/destructive. The other is that cultural heritage materials are affected by a number of factors that influence the readings, e.g. non-homogeneous density, porosity, chemical reactions, surface treatments and weathering, mould and insect damage.

A precise definition of '*moisture content'*, based on theory, is impossible, and we should use empirical definitions. The most popular definition is the '*extractable water'* after oven drying above the boiling temperature of water, but the methodology is destructive and cannot be used for cultural heritage items. However, it is popularly used for calibration.

Extractable water can be either expressed in *gravimetric* or *volumetric* terms, but in the specific case of cultural heritage materials, with variable subsurface density due to the attack of moulds or insects, due to weathering, the volumetric definition may be misleading, so that only gravimetry is acceptable.

It is necessary to define MC in terms of a *measurable quantity*, but the basic problem is that readings depend on the selected methodology. Every methodology responds to different physical mechanisms and interactions with the H_2O molecules, and in some cases with weaker interactions with molecules, or polar parts of molecules constituting the dry structure of a material. This means that different methodologies may provide similar results, but they cannot be strictly comparable and readings in AU are necessary. Only methods based on gravimetry (that is accurate) and on the calcium carbide (that is less accurate) can provide absolute readings.

In most cases, an instrument can be reasonably reliable only with respect to MC at a particular position of the target surface. If this is higher or lower in another position, or it varies over time, the instrument will provide relative readings in AU only. Some commercially available instruments produce readings in AU only intended for relative comparisons, but this is not very attractive for a user who typically prefers absolute readings expressed in %, as required by EN 322: 1993, EN 13183-1: 2002 and ASTM D4442 – 07, for traditional materials of commercial interest. Other producers, to meet practical requests, provide readings in MC (%) after 'calibration' with selected materials.

All standards make reference to the gravimetric definition and calibration curves are based on it. However, the comparison is between non-homogeneous readings. Gravimetry is related to extractable water. The methodologies based on conductivity, capacitance, microwave, EFD are related to the combined response of H_2O and other polar groups inside material. This makes the cross comparison and calibration of volumetric readings with gravimetry difficult. In conclusion, calibration may help, but only if it is made using the same specimens, and if these are homogeneous both in density and in water distribution. This is not the case of cultural heritage materials that are essentially non-homogeneous and their characteristics continue to change over time.

All methodologies are affected by a number of disturbing factors that have a different relevance. If we are to measure the MC of an object, the experience of the observer and the choice of the instrument are crucial, because the observing methodology might be not convenient for the specific material and its deterioration, or might provide misleading results.

For all of the above reasons it was decided to produce a novel standard, i.e. "Conservation of cultural property – Procedures and instruments for measuring moisture content in objects and building materials" to assist conservators and other professional users in this difficult task.

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8 Appendix: Operating Principles of the Most Popular Methodologies

8.1 Gravimetric: Oven-drying
(See EN 322: 1993 and EN 13183-1: 2002)

The method is based on precise weighing. The specimen is weighed immediately after sampling and when it has reached equilibrium after drying in an oven at a temperature above the water boiling temperature. The loss of weight corresponds to the moisture escaped from the specimen and is expressed as a percentage of the weight of the specimen after drying.

8.2 Calcium Carbide Pressure or Calcium Carbide Bomb
(See ASTM D4944 – 11)

The method is based on the determination of the pressure developed by the acetylene gas released after reaction of calcium carbide crystals with the moisture present in a crushed specimen of the material under investigation. The pressure gauge has a scale in pressure that may be related to the MC of a specific material.

8.3 Electric Conductivity or Resistance (See EN 13183-2: 2002)

The magnitude of the conductivity (or of the resistance), the applied electromotive force, and the current between the electrodes are related by the Ohm law $V = Ri$ where V is the potential difference across the two nails, R the resistance and i the current intensity through the conductor. The conductivity G is the inverse of the resistance, i.e. $G=1/R$. In most cases, the magnitude of conductivity in a material increases with its MC. When a constant electromotive force is applied, in general the intensity of the current between two electrodes increases with the MC. Instruments are based on two electrodes, e.g. two nail-pins, pressed or nailed into the target specimen.

8.4 Electric Capacitance or Dielectricity (see EN 13183-3: 2005)

The water molecule has a high dielectric capacity ε (i.e. ε = 80) compared with that of materials (generally ε ranging from 2 to 8). In a capacitor with plane and parallel plates, the capacitance C is a function of the plate area A, the width d between plates and the dielectric constant ε of the material between the two plates ($C = \varepsilon A/d$). The MC may be determined via its effect on dielectric constant by measuring the capacitance between or in front of conductive plates. Instruments have been developed with probes arran-

ged or shaped in a number of ways, especially in fringe capacitance to utilise the capacitance associated with the edge or outside the perimeter of the plates. The most popular case is constituted of two plates on the same plane. Another popular array is constituted of only one spherical plate ($C= 2 \pi \varepsilon D$ where D is the sphere diameter). Where the moisture is predominantly in the form of free water, the dielectric constant is directly proportional to the MC. The output reflects the change of capacitance determined by water molecules in the specimen.

8.5 Microwave Transmission / Reflection

The microwave 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 frequency. In materials with polar molecules the electric field of microwaves can induce oscillations whilst 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 exhibits a phase shift.

Whilst travelling within the medium for a path length x, the intensity $I(x)$ of the microwave beam decreases following in a first approximation an exponential law, i.e.

$I(x) = I(0) \exp(-\gamma x)$

where $I(0)$ is the initial strength when $x = 0$, and $-\gamma$ is the extinction coefficient determining how the beam intensity attenuates during propagation. The extinction coefficient γ is a complex quantity $\gamma =\alpha + i\beta$, where the real part, i.e. the attenuation constant α, indicates the amplitude reduction of the microwave beam power with increasing path length x, and the imaginary part β, is related to the phase shift caused by the reduction in propagation speed through the medium. For a number of disturbing factors in real materials, it might happen that γ is not constant, but might vary from point to point, and with time too, i.e.

$\gamma = \gamma (x,t)$.

In addition to absorbing or changing 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, or the phase changes, or the back radiation increases, with the MC of the material.

8.6 Equilibrium RH

Hydrophilic materials exchange moisture with the environment until they reach equilibrium. When the ambient RH is increasing the hydrophilic material absorbs moisture, when the RH is decreasing the material gives back moisture to the ambient. When the RH remains unchanged for a sufficiently long time, the material reaches equilibrium and exchanges will stops. The Equilibrium Moisture Content (EMC) is related to the ambient RH and is weakly dependent

on temperature. Vice versa, the equilibrium RH reached in an air pocket inside a small cavity (e.g. a drilled hole or an External Sealed Chamber) is determined by the MC of the material (e.g. masonry, timber).

8.7 Ultrasound Transmission / Reflection

Sound and ultrasound propagate through a material at a velocity and transmittance that are a function of physical material properties, including MC and moisture gradients. The ultrasound velocity decreases with increasing MC, and the transmittance increases. The measurements are based either on transmitted or reflected ultrasounds. Unpredictable uncertainties are generated by structural discontinuities or inhomogeneities (e.g. fractures, changes in density, grain orientation, insect or mould damage) inside the material under investigation.

8.8 Thermography

Thermography is based on the analysis of thermograms (i.e. thermal images) taken with a camera sensitive to the infrared radiation (IR) emitted by bodies in the thermal spectral band of the electromagnetic spectrum. The methodology is useful to localise damp areas cooled for evaporation and in some cases to evaluate evaporation flows; hardly to measure MC. Thermograms may either be taken in an active or a passive way, i.e. the operator heats or does not heat the target surface. The camera is not directly sensitive to the water content, but to temperature changes, i.e. space gradients or time rates, that might be generated by moisture.

8.9 Evanescent-field Dielectrometry (EFD)

EFD measures the parameters of interest, moisture, and salt concentration by estimating the dielectric properties of a wall that is viewed as a mixture of 'dry' material and water. The large difference between the dielectric constant of a dry material (2< ε< 10) and water (ε = 80) allows the detection of water inside a wall. The real part of permittivity depends on the MC, whereas it is nearly independent on the saline content. MC is evaluated through the measurement of a frequency shift.

8.10 Nuclear Magnetic Resonance

With Nuclear Magnetic Resonance (NMR), the water 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.

8.11 Neutron Scattering

Fast neutrons emitted from a radioactive source are launched inside a target material. The fast neutrons gradually loose energy through collisions with other atoms. Hydrogen is the most effective atom to slow down neutrons because they have almost the same mass. 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 MC. A detector of slowed down neutrons counts the number of slow neutrons per second and transforms it into MC.

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10   Endnotes

This paper is intended in support to a new European standard entitled: "Conservation of cultural property – Procedures and instruments for measuring moisture content in objects and building materials" that is in preparation at the European Committee for Standardisation, Technical Committee for Cultural Heritage.