MOdihMA

MOisture detection in historic MAsonry

Standardization activity in the evaluation of moisture content

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Why CEN TC346 has produced specific standards for cultural heritage

- A number of European Standards (EN) and International Standards (ISO) and EN-ISO joint Standards, provide precise operational protocols and describe selected methods to measure moisture content.
- The above standards are conceived for normal materials, in good conditions, neither aged nor deteriorated, differently from cultural heritage. In addition, they allow to take samples.
- Each of the above standards deals with only one method, specified in the title, without establishing priorities among them.
- This may be confusing because all standards, and therefore all methods, are apparently at the same level, and the user lacks a comprehensive guidance for his/her particular case study.
- Finally, not all methods have been standardized.

EN, ISO & EN-ISO Standards for normal materials

- ISO 16979. *Wood-based panels - Determination of moisture content*.
- EN 13183-1. *Moisture content of a piece of sawn timber - Part 1: Determination by oven dry method.* EN 13183-2. *Part 2: Estimation by electrical resistance method*. EN 13183-3. *Part 3: Estimation by capacitance method*.
- EN-ISO 11461. *Soil quality - Determination of soil water content as a volume fraction using coring sleeves - Gravimetric method*.
- ISO 11465. *Soil quality - Determination of dry matter and water content on a mass basis - Gravimetric method*.
- ISO 12570. *Hygrothermal performance of building materials and products - Determination of moisture content by drying at elevated temperature*.
- ISO 12571. *Hygrothermal performance of building materials and products - Determination of hygroscopic sorption properties*.
- ISO 760. *Determination of water - Karl Fischer method (General method)*.
- EN-ISO 15512. *Plastics - Determination of water content.*
- EN 1428. *Bitumen and bituminous binders - Determination of water content in bituminous emulsions - Azeotropic distillation method*.
- EN 772-10. *Methods of test for masonry units - Part 10: Determination of moisture content of calcium silicate and autoclaved aerated concrete units*.

Camuffo, *Journal of Cultural Heritage* (2018)

EN 16682: 2017

Conservation of cultural heritage. Methods of measurement of Moisture Content, or Water Content, in materials constituting immovable cultural heritage

Scope of EN 16682

- To inform and assist users in the choice and use of the most appropriate method to obtain reliable measurements of MC, or WC, in wood and masonry in the specific case of the built cultural heritage.
- To provide a basic framework to take and interpret measurements on cultural heritage materials.
- To norm 5 absolute methods and 3 relative methods and provide an informative overview of 10 other relative methods, their characteristics, pros and cons, and calibration limits.
- To advice about problems met in the field of cultural heritage to prevent instrument misuse, reduce uncertainties and misinterpretation.

Part 1:
Absolute Methods
in EN 16682

Absolute Methods

- 'Absolute' means that readings can be expressed in terms of the **International System of Units** (SI), e.g. grams, corresponding to the weight of a moist or dry specimen.
- When a reading is expressed in %, the SI **formally disappears**, but the previous measurements in SI have been essential in determining in objective terms the final reading.
- A common problem of the absolute methods is that they are **destructive**, or micro-destructive, because they require samples taken from the object (and this may constitute an insurmountable problem with cultural heritage).
- Finally, absolute methods require laboratory analyses on the samples, which implies a **delayed response**.

Basic Definition

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.

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

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 porosity (*water holding capacity*).

Moisture Content (MC), or Water Content (WC)?

- **MC** is the result of the **Gravimetric** analysis. It may include some loss of weight due to VOC outgassing, especially if the material has been treated with oil, wax, preservatives or include resin. In addition, the drying procedure does not extract all water molecules.

→ **MC** = extracted moisture (not all) + VOC (if any)

- **WC** is the result of the **Karl Fischer Titration** (KFT) that is specific for water. It does not include VOC, but might include crystallization water. KFT detects all water molecules, and therefore the WC is greater than MC.

→ **WC** = all water = all moisture + crystallization water (if any)

- MC and WC are in general different (e.g. WC>MC) and have different names to avoid misunderstanding. Both shall be expressed as % of the mass of the dry sample.

Gravimetric Analysis ⭐⭐⭐

Absolute method, Specific for **MC**

High accuracy, for calibration (when possible)

Requires sampling. Four Drying Options:

1. Ventilated oven (103±2)°C for materials resistant to T=103°C.
2. Vacuum at ambient T (materials outgassing at T=103°C but resistant to low pressure).
3. Compressed air at ambient T and pressure (materials neither resistant to T=103°C nor to low pressure).
4. Adsorption (e.g. SiO_2, $CaCl_2$, LiCl) at ambient T and pressure.

(1) 

(2) 

(3) 

(4) 

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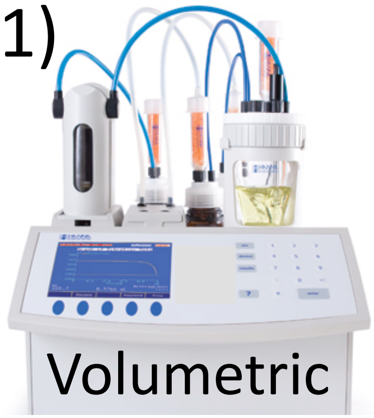
Thermo Gravimetric Analysis (TGA) ☆☆☆

A variant of Gravimetric Analysis, with oven drying record:

- Smaller samples
- Quicker analyses
- Continuous recording showing thresholds, e.g. transition temperature of crystallization water

Karl Fischer Titration (KFT) ☆☆☆

(1)



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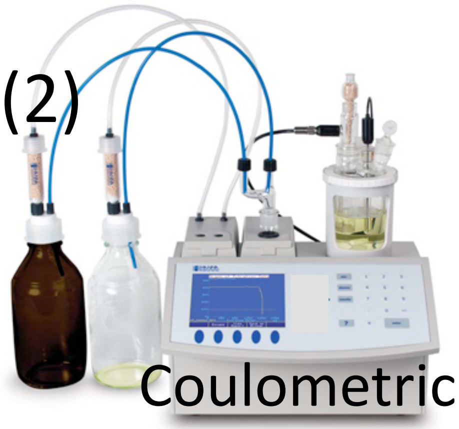

Absolute method, Specific for **WC**
High accuracy, for calibration (when possible). Requires sampling

1. Volumetric (V-KFT) (less sensitive, larger samples).
2. Coulometric (C-KFT)) (more sensitive, smaller samples).
3. oven-vaporization (OV-V-KFT & OV-C-KFT).

Readings traditionally presented in % of the moist sample, shall be transformed into % of the dry sample.

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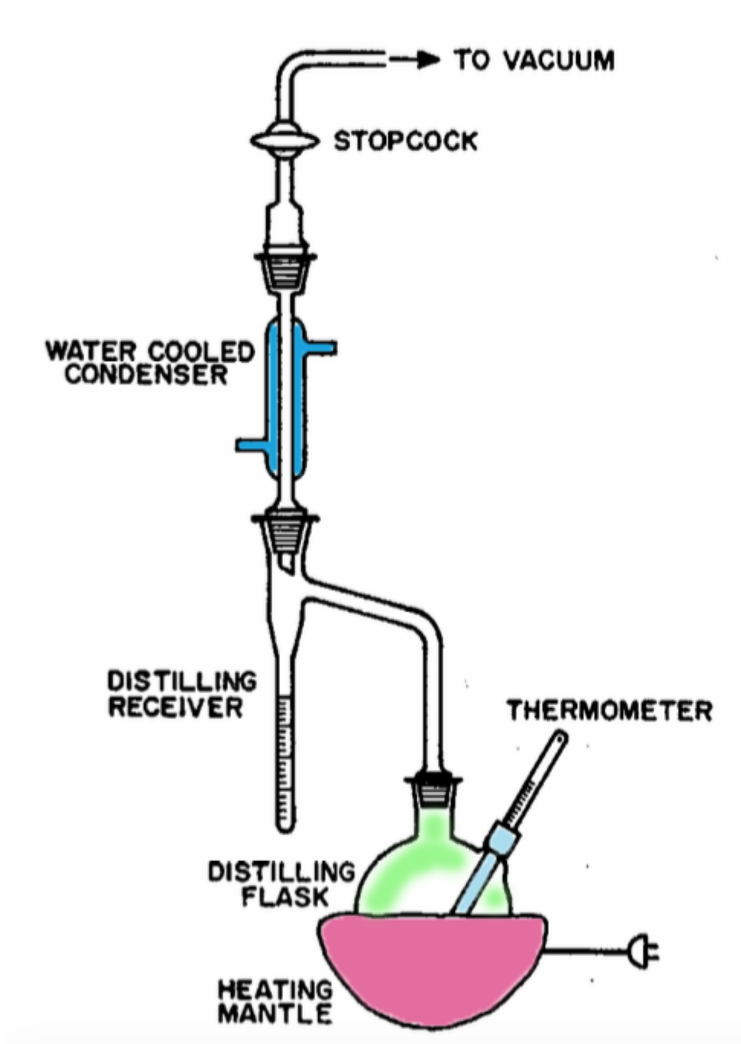
Azeotropic Distillation



Absolute method
Specific for **MC**
Medium accuracy
Requires sampling

This method can be applied when the material does not include VOC soluble in water.

Traditionally presented in % of the moist sample, shall be transformed into % of the dry sample.

Calcium Carbide Test

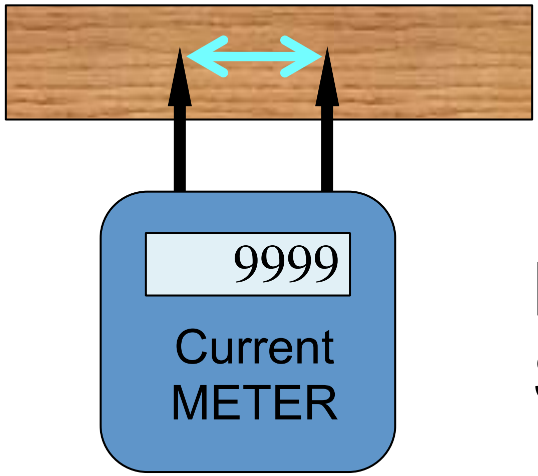
Absolute method
Specific for **MC**
Low accuracy
Requires large samples

Traditionally presented in % of the moist sample, shall be transformed into % of the dry sample.

Part 2:
A few examples of Relative Methods and their problems in EN 16682

Relative Methods

- A ‘relative’ method is a measuring method whose readings cannot be expressed in terms of SI Units, but in relation to something else, kept as a reference. This implies **arbitrary units**.
- Some producers **calibrate the output by comparison** with an absolute method, but the calibration is only true when the tested material and the material used for calibration are absolutely identical. **This is impossible** for aged and deteriorated materials of cultural heritage.
- In general, relative methods are **not destructive**.
- They provide **instantaneous readings**, and are especially useful to detect differences from a point to another (i.e. space gradients) and over time (i.e. trends).



Resistance in Wood

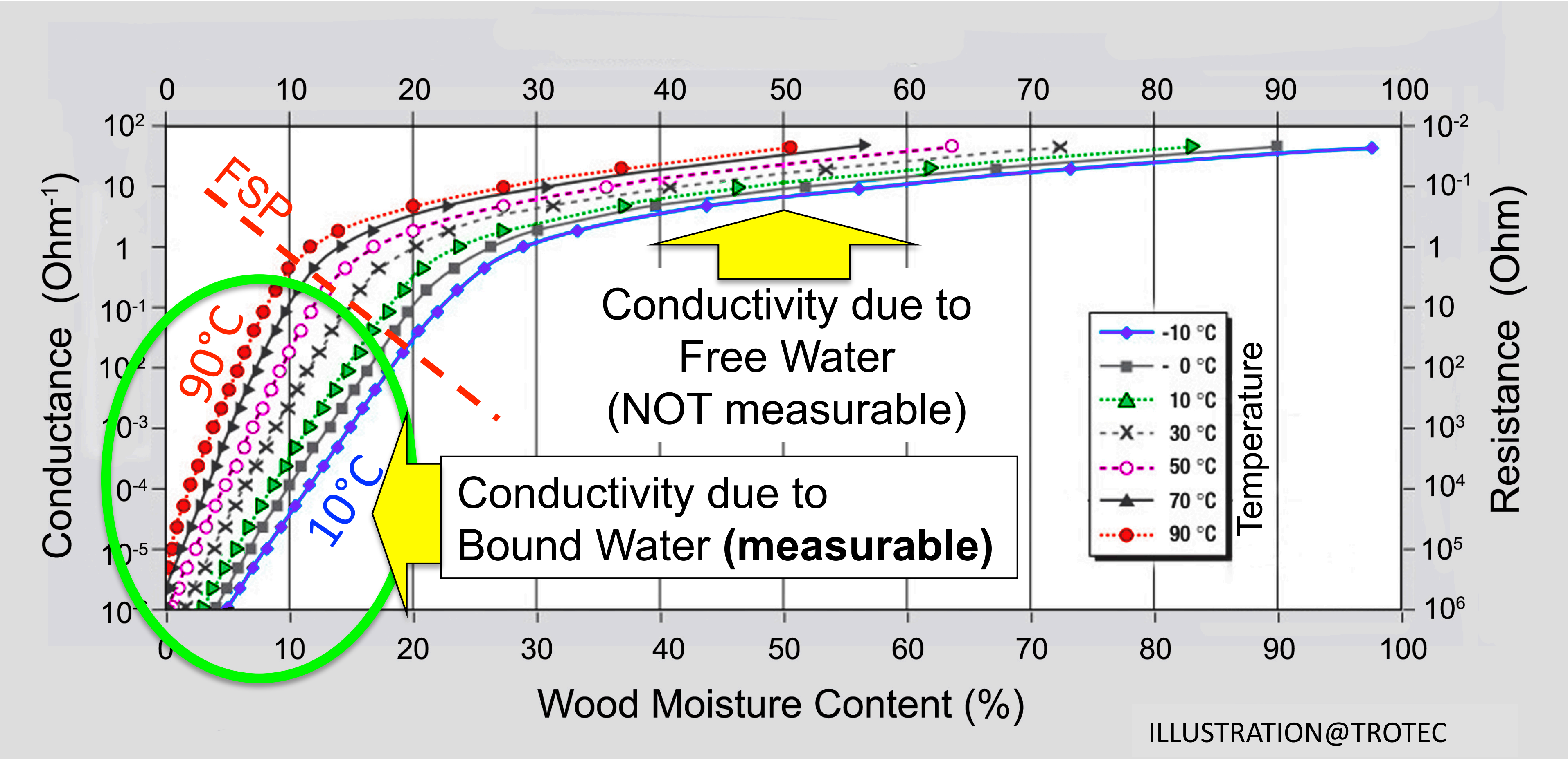
Micro-destructive.
Short penetration.

- Measurements shall be limited to bound water.
- Conductivity is dependent upon the grain orientation.
- Conductivity is strongly dependent upon past treatments with wax, oil, or preservatives that may alter conductivity.
- Cracks, mould rotting, insect tunnelling decrease conductivity and readings are misleading.

Resistance in Wood

Electric properties of Wood vs. MC and T

Only the interval below the Fibre Saturation Point (FSP) i.e. 0 < MC < 15% is measurable



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Resistance in Masonry

- The presence of subsurface layering affects readings.
- A film of salts under dissolution is critical.
- Conductivity is affected by the presence of ions derived from the dissolution of soluble salts.
- Efflorescence and subflorescence strongly affect measurements, which apparently provide higher dampness levels.
- The report shall specify the anions and cations present in the material measured with ion chromatography.

Capacitance in Wood

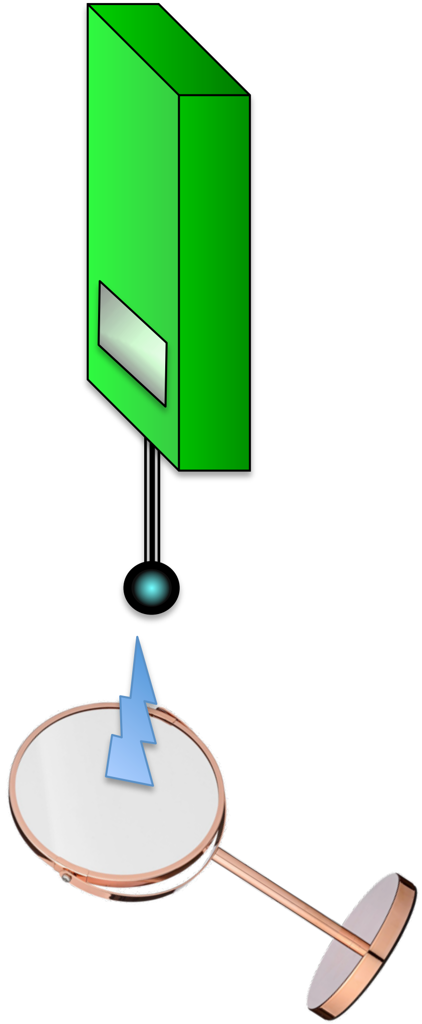
Non-destructive. Short penetration.

- Method affected by moisture gradients.
- The contact electrode can be placed with any grain orientation on the side grain except on the end grain.
- The output is dependent on the wood density.
- Local density changes cause misintrerpretation e.g. knots = damp; subsurface woodworm tunneling = dry.
- Output decreases proportionally to the cumulated volume of voids.
- Output is affected by the proximity to, or penetration into the material.

Capacitance - Departures for deliquescent salts

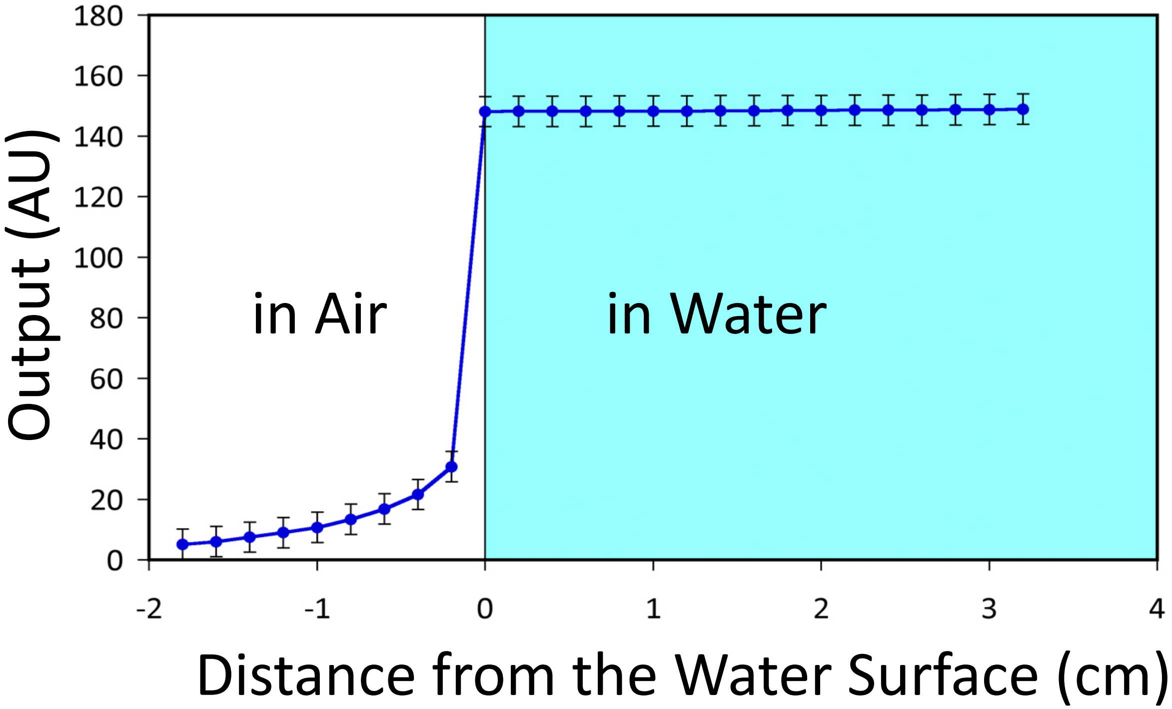
If the wall has some efflorescence, or sub-efflorescence, when the soluble salts approaches the deliquescence point and starts to absorb water molecule, the thin film of moisture that is forming on the crystal will behave like a mirror for the alternative electromagnetic field emitted by the sensor. The output will reach saturation as when the material is full damp, giving a misleading signal.

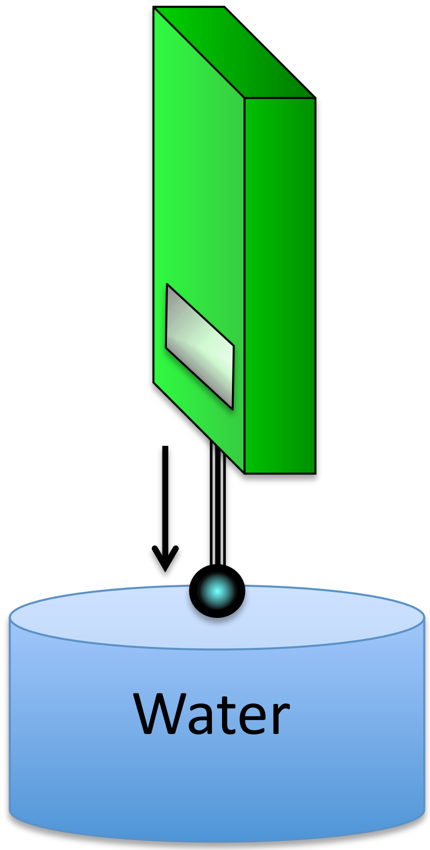
The capacitance method is a hybrid system, and has a number of analogies with microwaves.



Capacitance - Departures for bad contact: approaching a surface

Output when the spherical sensor is approaching to a water surface and is immersed in it.





Camuffo & Bertolin, *E-Preservation Science*, 9, 23-35 (2012)

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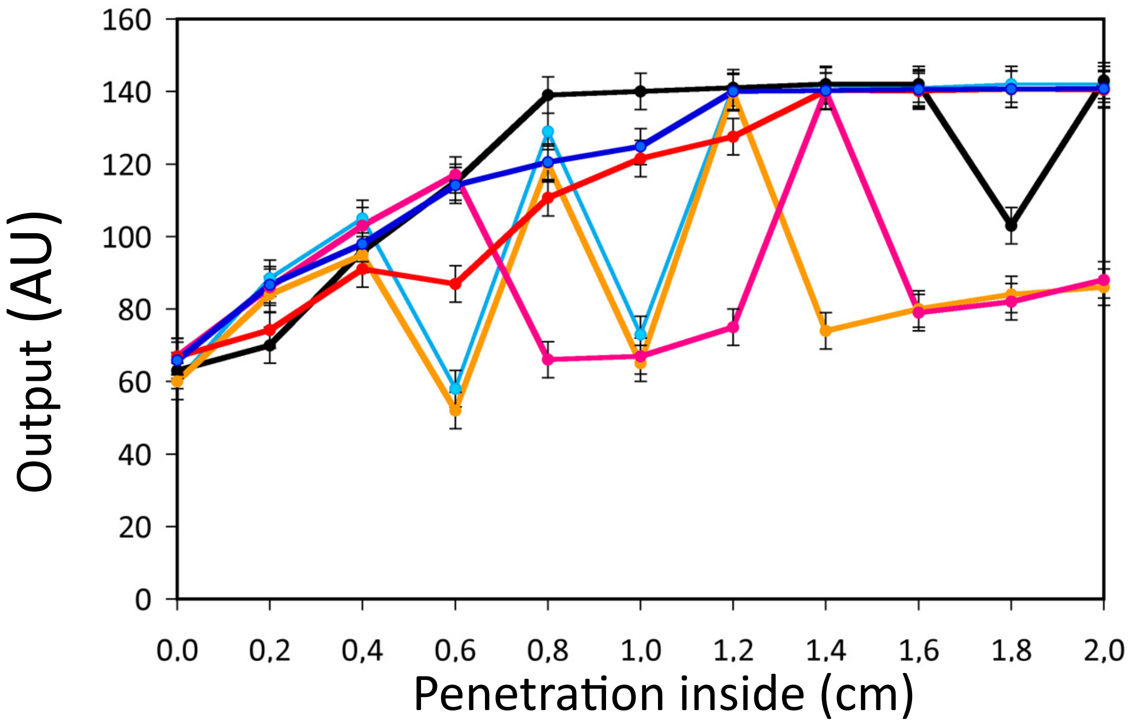
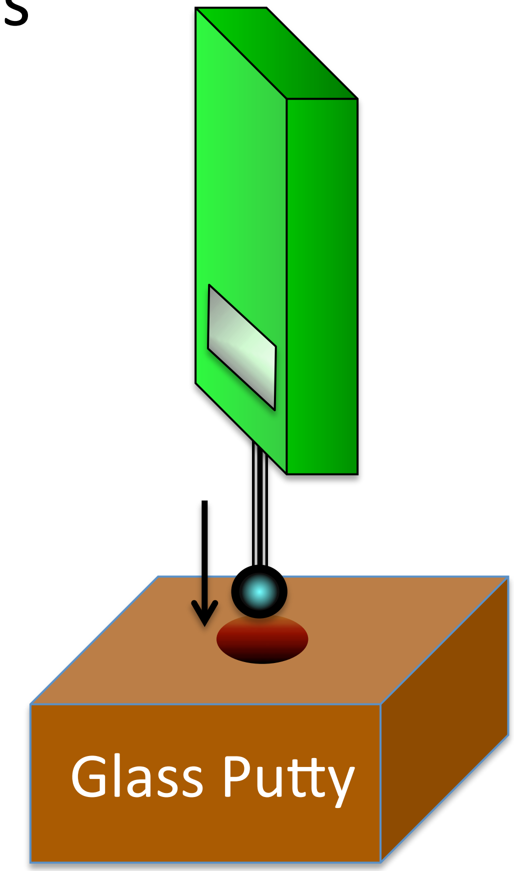
Capacitance - Departures for bad contact: penetration into, or a concave surface

Output when the spherical sensor is pressed against a soft material (glass putty), or is (partially) surrounded by the material

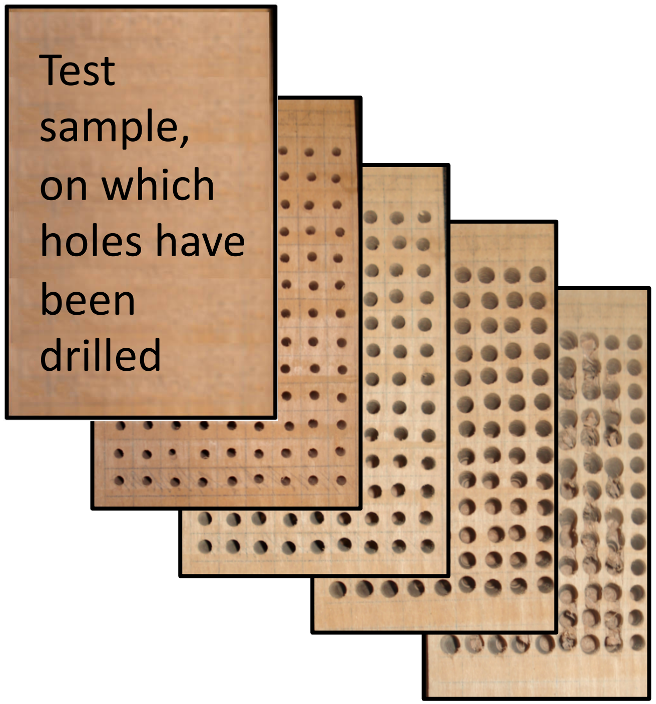


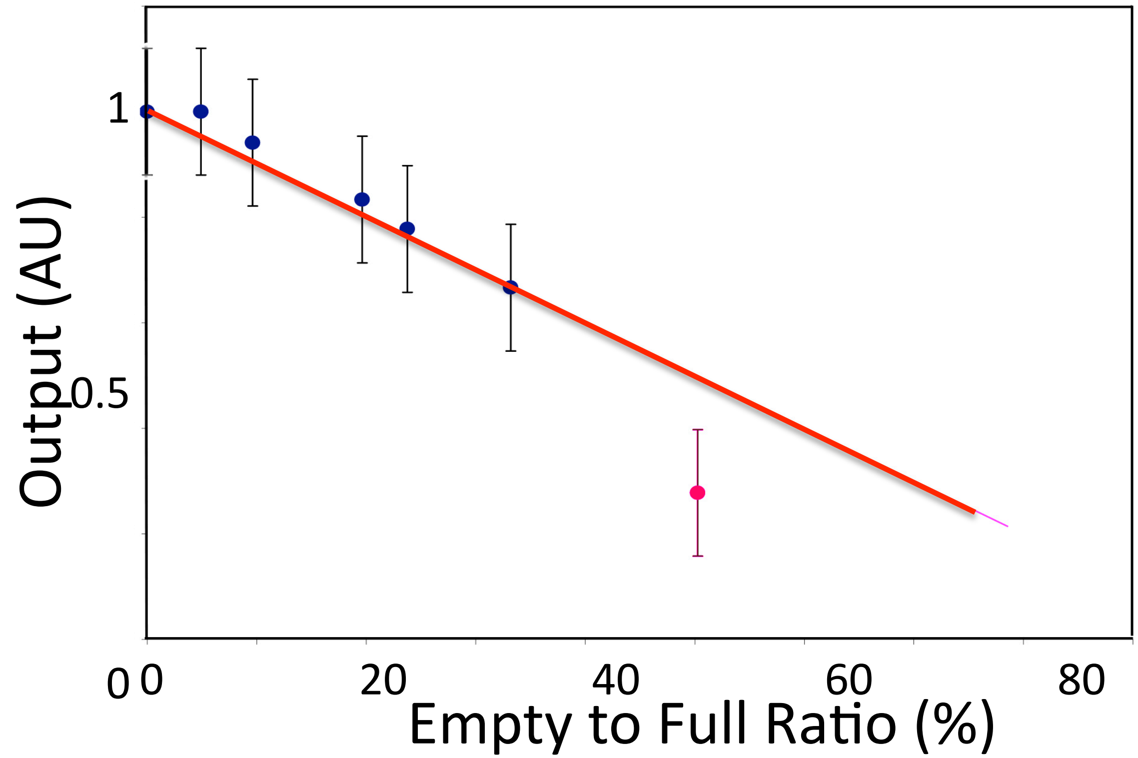


Camuffo & Bertolin, *E-Preservation Science*, 9, 23-35 (2012)

Departures for the presence of subsurface cavities

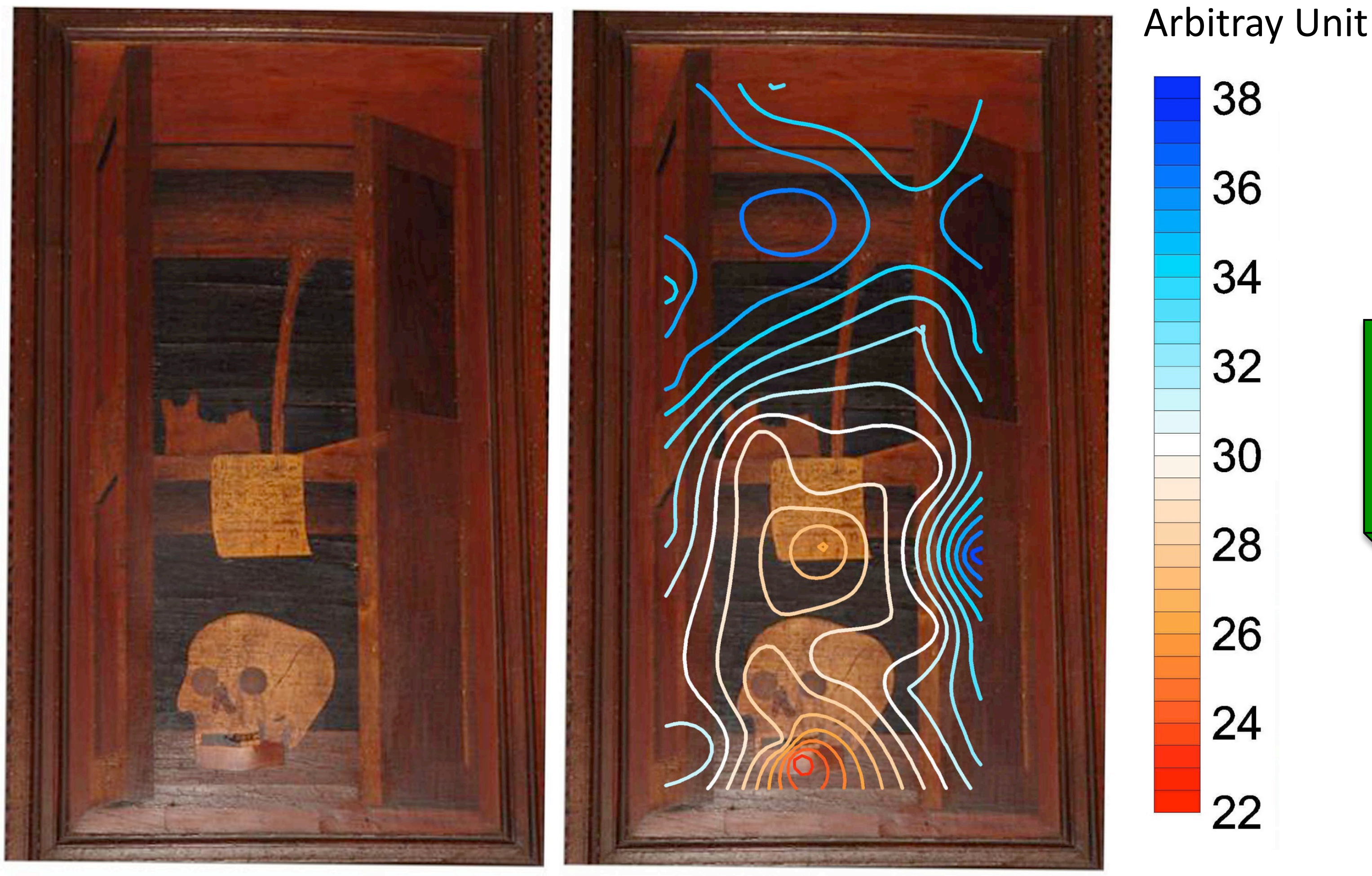
If subsurface cavities exist, a smaller amount of beam is sent back, which is transformed in lower output = drier material. A test with controlled empty to full ratio:

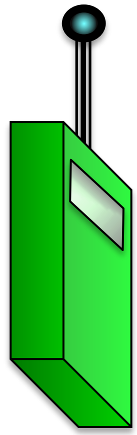




Camuffo & Bertolin, *E-Preservation Science*, 9, 23-35 (2012)

Application: the capacitive method may be used to detect and map insect tunnelling





Camuffo & Bertolin, *E-Preservation Science*, 9, 23-35 (2012)

Capacitance in Masonry

Non-destructive. Short penetration.

- Method affected by gradients
- It is almost independent from the presence of soluble salts, except in the case of deliquescence.
- Deliquescent salts behave as a mirroring surface that will reflect the signal, giving the appearance of a completely damp material.
- The methodology is sensitive to the presence of subsurface layering, cavities, internal discontinuities or metals in the masonry.
- The report shall specify the anions and cations present in the material measured with ion chromatography.

Microwave

Non-destructive.
Deep penetration e.g. 30 cm to 100 cm.
Needs very thick, homogeneous materials

NOTE: The **extinction depth** (ED) is the depth at which the microwave beam is **completely** extinguished within the medium.

If the object is thinner, some back reflection occurs and this will make interference with the incoming beam.

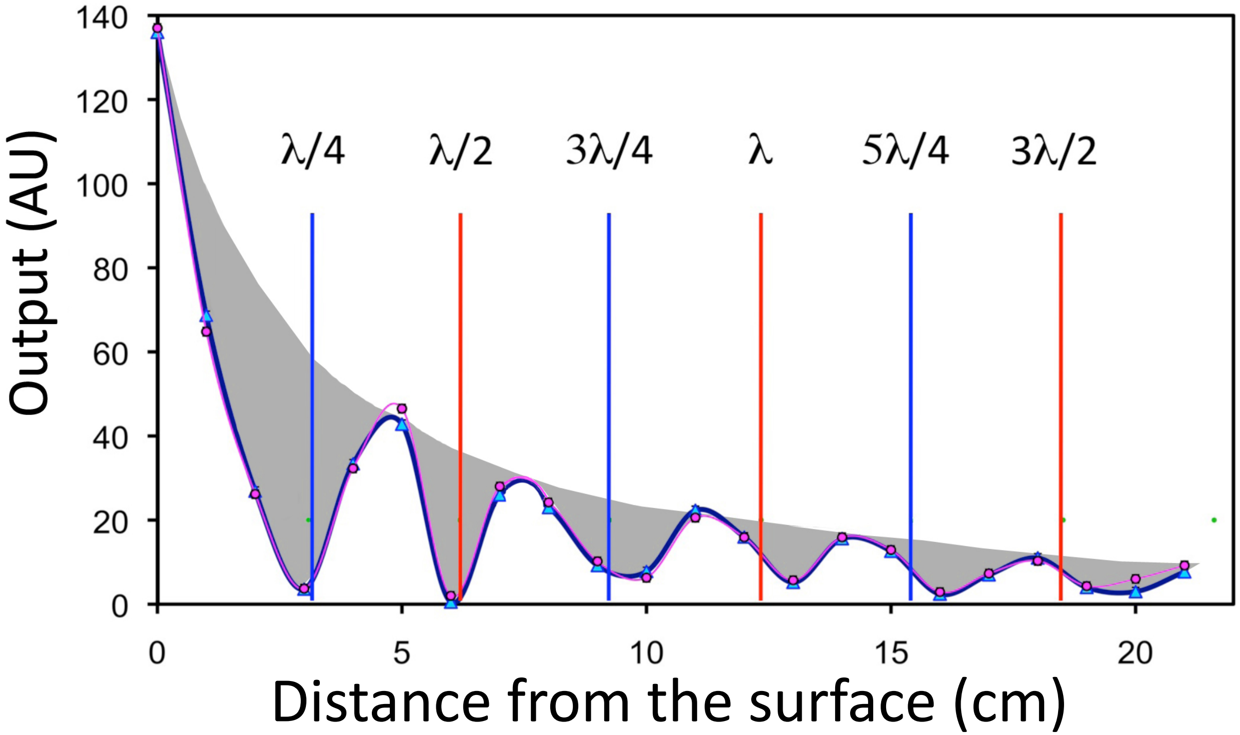
Microwave

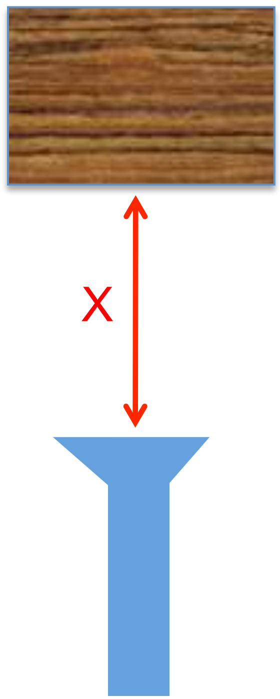
The **extinction depth** (ED) depends on the instrument, material type and density, moisture content and grain orientation in wood (e.g. shorter ED in high MC and longer ED in low MC).

If the path of the MW within the medium is shorter than the extinction depth, the fraction of microwaves that will reach the opposite side of the medium will undergo a back reflection. This will reinforce the backscatter signal, exactly as the material had higher moisture content, or may generate **beatings**.

The medium must be homogeneous. This is a problem for walls with discontinuities, e.g. plaster, bricks, mortar etc.

Microwave: When the sensor is not in contact with the surface



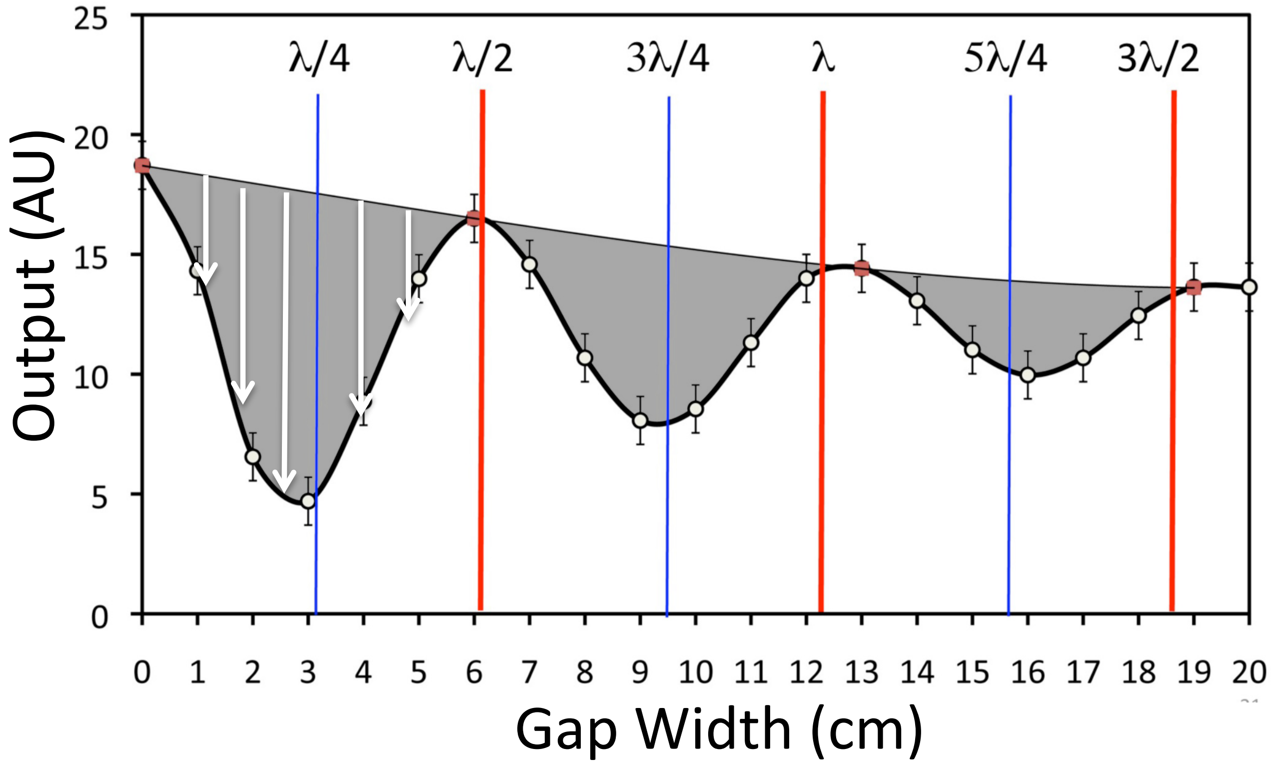


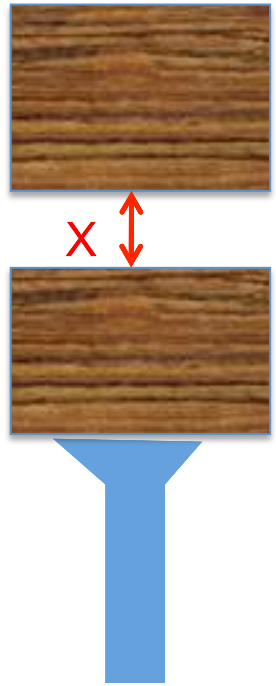
The emitted and reflected beams do interfere between them, causing **interference and beating**. The sensor must be in contact with the medium to avoid misleading readings.

This makes hard to measure rough or non-planar surfaces.

Camuffo & Bertolin, *E-Preservation Science*, 9, 23-35 (2012)

Microwave: Effect of a gap in the medium



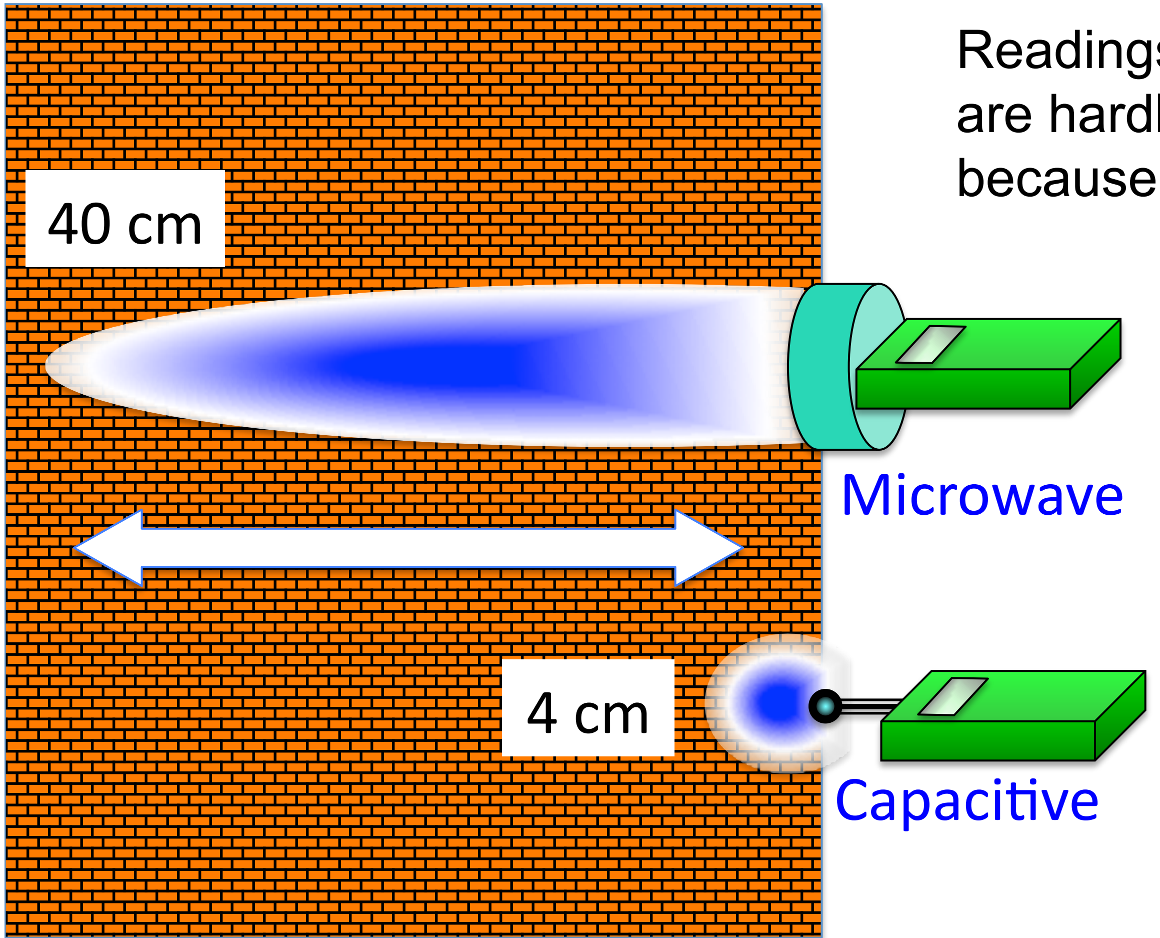


Internal cavities might cause MW beatings and misleading readings with sinusoidal lowering (apparently drier).

Resonant standing waves will form inside the cavity.

Camuffo & Bertolin, *E-Preservation Science*, 9, 23-35 (2012)

Is it possible to detect moisture gradients by comparing different instruments with long and short penetration depth?



Readings taken with different methods are hardly comparable between them because:

- their responses are not linear.
- they haven't the same functional dependence on moisture.
- they do not equally respond to local differences in the physical properties of the material.

A Proxy Method: Irreversible Passive Sensors

Irreversible sensors follow a dynamic equilibrium with the internal Free Water, although with some delay. However, they undergo irreversible transformations at some selected RH thresholds.

Irreversible sensors are based on **soluble salts** that become **deliquescent** when some critical RH levels are reached. These salts have strong affinity for moisture and will absorb relatively large amounts of water from the environment, forming a **liquid solution**.

Once the deliquescence threshold is reached, the salt in the sensor abandons the granular form and the formed **liquid activates some colouring** that remains permanently.

Irreversible sensors react at **some selected, well defined RH thresholds** and provide a useful information about **the highest past humidity level** reached during **the whole sampling period**, e.g. one hour, day, month, or year.

They only **detect the equilibrium moisture**, **do not measure the MC**!

Camuffo, *Microclimate for Cultural Heritage*, Elsevier (2013)

A Proxy Method: The Free Water Sensor

The sensor is **passive**, conceived to be left **unattended** for weeks or months in unmanned buildings, or archaeological remains, to detect if masonry is safe, or damp reaches **critical levels** for mould infestation.

The sensor is composed of a **number of salts**, with **different thresholds**, that will reach equilibrium with the free water inside a masonry. The sensor is initially white, but after use it will have a number of **coloured spots**, that indicate the **maximum humidity that has been reached** during the exposure period. The number of sectors and RH thresholds can be **personalized** for specific problems, on request of the user.

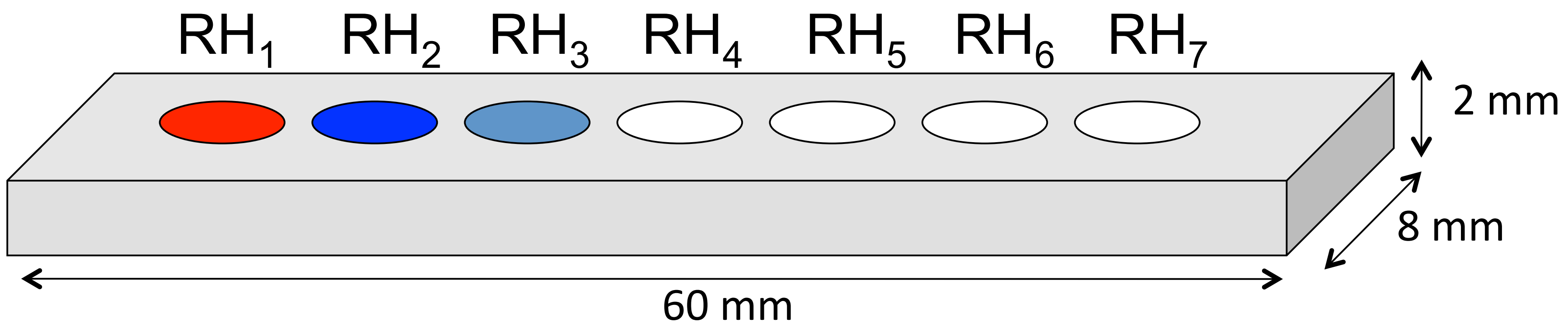
No power supply is needed. This is particularly useful in remote sites.

Its **small size** is convenient for insertion into drilled holes or small cavities.

The sensor is **cheap** and can be used for preliminary investigations.

Camuffo, *Microclimate for Cultural Heritage*, Elsevier (2013)

The Free Water Sensor



The sensor is miniaturized. The envelope is diffusive to water vapour on the bottom layer and has a transparent band on the upper side.

It is divided into specific **sectors** covered with a white paper disc.

Each sector is filled with **deliquescent salts** sensitive to selected RH thresholds (i.e. RH_1, RH_2, RH_3…).

When the humidity reaches a selected deliquescence threshold, the salt will form a solution that will **activate a dye,** thus forming a **colour spot**, specific for each threshold.

Camuffo, *Microclimate for Cultural Heritage*, Elsevier (2013)

Conclusions

- The presentation has been largely incomplete to leave time to some examples. The mentioned standards and the references should be consulted.
- A number of standards exist for particular methods, but none of these is suitable for application to cultural heritage.
- EN16682 has a holistic approach and may be used for a large variety of materials.
- EN 16682 has been produced to assist users to choose the most appropriate instruments, to know pros and cons of each method, to make aware of the specific problems of cultural heritage, to avoid misuse and misinterpretation of readings.

Many thanks for your kind attention!

Disclaimer: Any resemblance with commercial products is purely coincidental. The reproduced figures are solely intended to help understanding.

References

Camuffo, D., and Bertolin C., 2012: Towards standardisation of moisture content measurements in cultural heritage materials. *E-Preservation Science*, 9, 23-35.

Camuffo, D., 2013. *Microclimate for Cultural Heritage - Conservation, Restoration and Maintenance of Indoor and Outdoor Monuments*. Second Edition. Elsevier, New York.

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