Low frequency electromagnetic materials characterization best practice guidance

Borut Pinter

Dielectric Permittivity

Polarization *P* describes the dielectric displacement *D* which originates from the response of the material to an external electric field *E*:

$$\vec{P} = \vec{D} - \varepsilon_0 \vec{E} = (\varepsilon_r^* \varepsilon_0 - \varepsilon_0)\vec{E}$$

where:

$$\varepsilon_r^* = \varepsilon_r^{'} - i\varepsilon_r^{''}$$

is complex relative permittivity tensor.

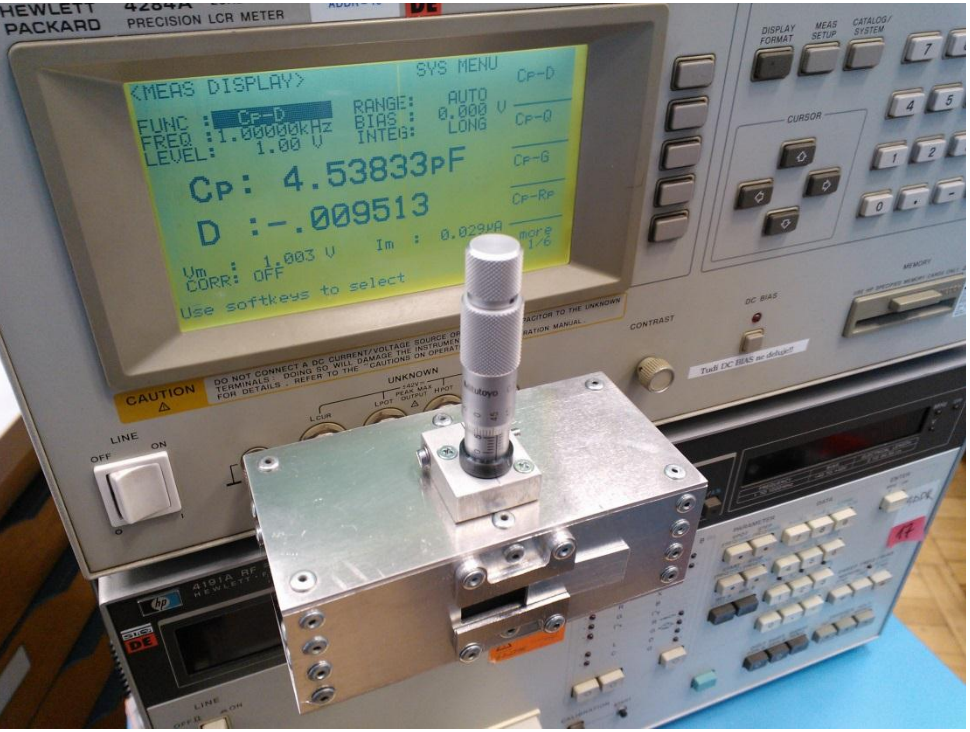
The real part of relative permittivity is the dielectric constant

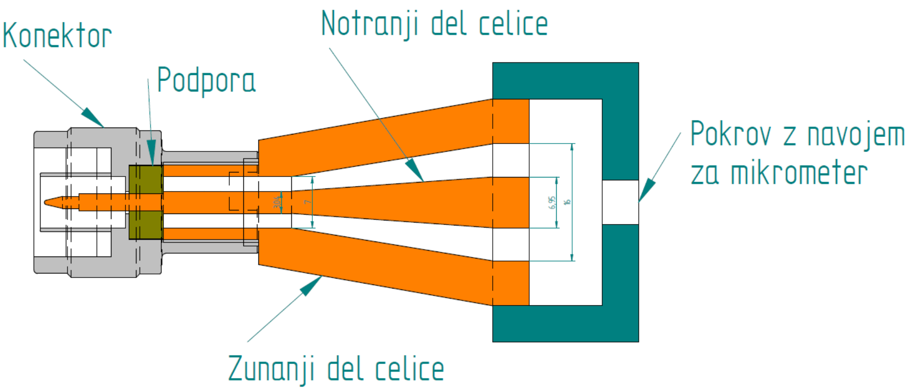
The dielectric loss tangent is calculated as:

$$tg\delta = \frac{\varepsilon_r^{''}}{\varepsilon_r^{'}}$$

Measurement methods for dielectric materials measurements

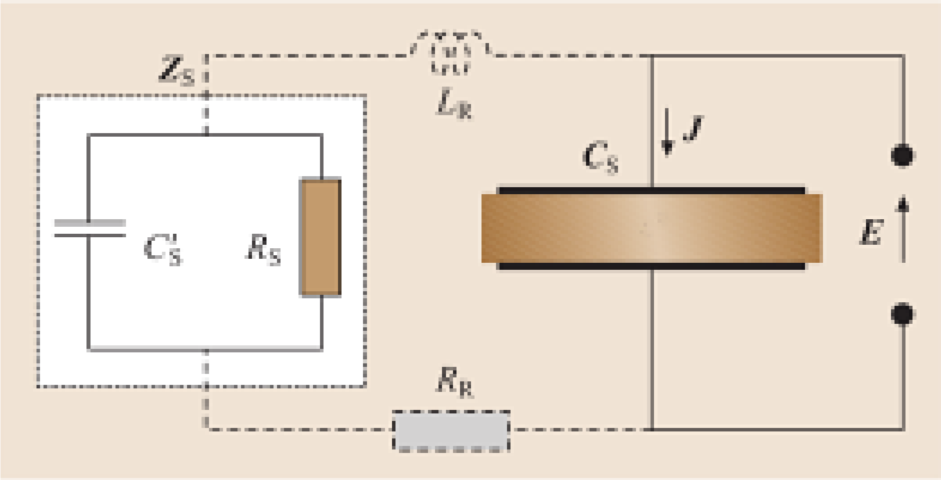
- Impedance measurement using the Four-Terminal Method
- Impedance measurement using Coaxial Line Reflectometry





Four terminal (4TP) method

- The four terminal method is used up to frequencies where sample can be interpreted as a lumped element model

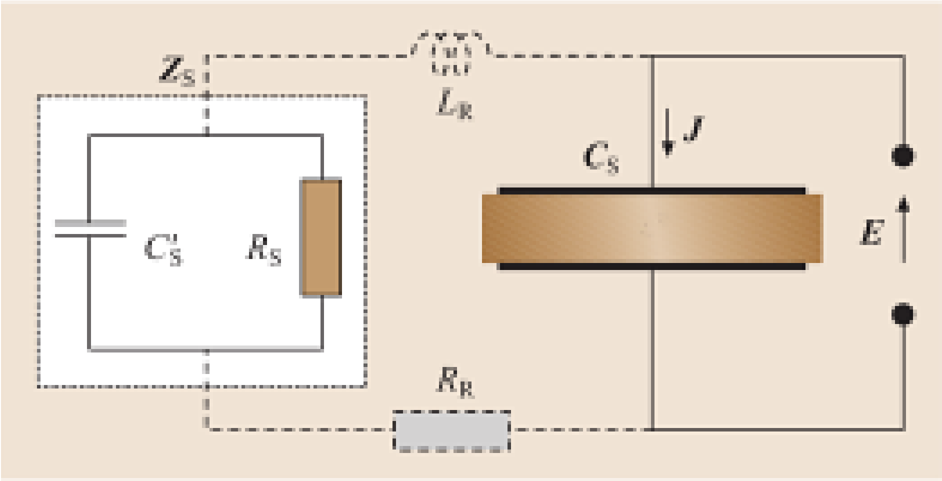


- Dipolar relaxation can be described by an electrical equivalent circuit consisting of capacitance *Cs‘* connected in parallel with resistance *Rs.*
- *Cs‘* and *Rs* can be measured and related to material‘s dielectric properties $\varepsilon_r^{'}$ and $\varepsilon_r^{''}$.

Four terminal (4TP) method

- Interconnecting leads and electrodes introduce serial residual inductance *Lr* and residual resistance *Rr*
- When measuring *Cs* resonance will occur at frequency f_{LC}:

$$f_{LC} = \frac{1}{2\pi\sqrt{L_R C_S}}$$



- At f_{LC} energy is stored in the magnetic field so this resonance is not useful for measurement of dielectric permittivity
- This is most common source of systematic errors in dielectric metrology

Four terminal (4TP) method

- An equivalent complex impedance ***Z**s* can be determined from which *Cs'* and *Rs* can be calculated and then the material's relative complex permittivity determined
- In the low frequency range wave propagation effects can be neglected
- For a capacitor filled with dielectric material resulting in complex capacitance ***C**s*, the permittivity is defined by:

$$\varepsilon_r^*(\omega) = \varepsilon_r^{'}(\omega) - i\varepsilon_r^{''}(\omega) = \frac{\vec{C}s}{C_0}$$

- Here C_0 is capacitance of the empty cell without the dielectric present

Four terminal (4TP) method

- The impedance ***Z****s* of the sample, consistent with equivalent circuit, is defined as:

$$\frac{1}{\vec{Z}_S} = \frac{1}{R_S} + i\omega C_S^{'}$$

$$\varepsilon_r^{'} = \frac{Cs}{C_0}$$

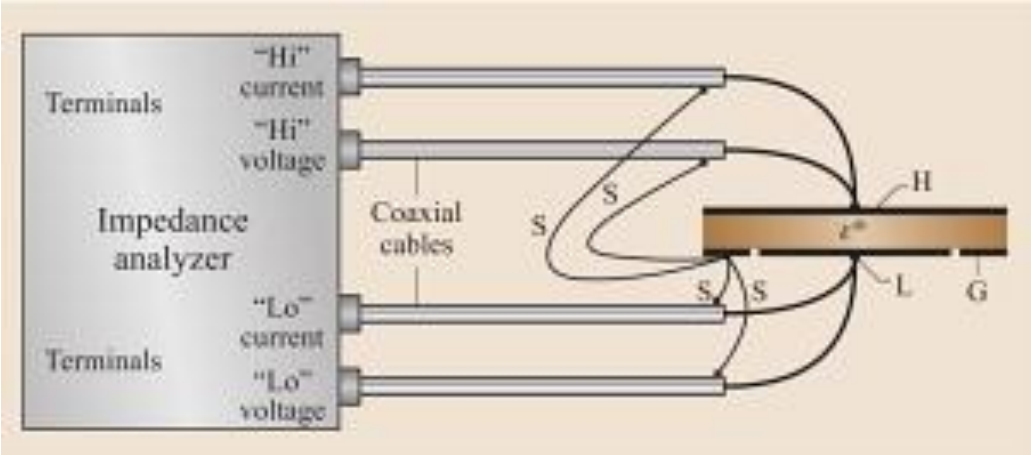
- Direct expressions for $\varepsilon_r^{'}$ and $\varepsilon_r^{''}$ are:

$$\varepsilon_r^{''} = \frac{1}{\omega R_S C_0}$$

- C_0 *can be* determined from specimen geometry or by measurements of reference materials with known dielectric permittivity

Four terminal (4TP) method

- Standard measurement procedures recommend a three-terminal (3T) cell configuration with guard electrode (G)
- Minimizes effects of fringing and stray electric fields



- All cables as short as possible (low *Lr*)
- If no guard electrode is used then shields are shorted together

Coaxial Line Reflectometry

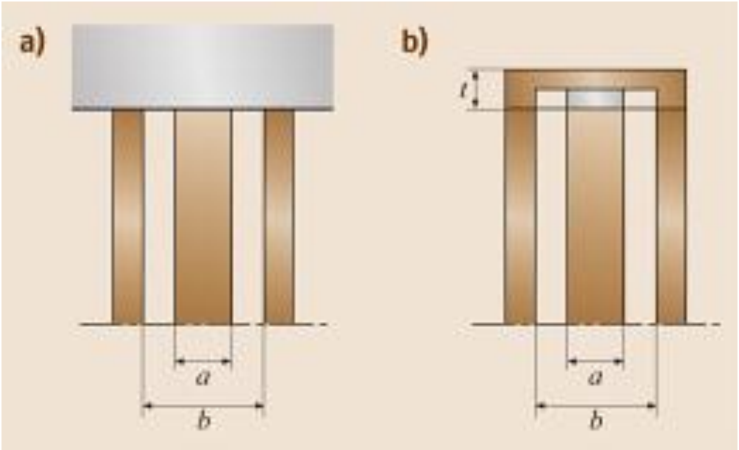
- Impedance at higher frequencies can be determined from the reflection coefficient measurement using a VNA (Vector Network Analyzer) or coaxial RF impedance analyzers
- Reflection coefficient are measured using microwave techniques with precision transmission lines
- A specimen with impedance ***Zs*** terminates a transmission line with characteristic impedance Z_0 which causes mismatch and reflections
- The relation between ***Zs*** and complex reflection coefficient Γ is given by:

$$\vec{\Gamma} = \frac{\vec{Z}_S - Z_0}{\vec{Z}_S + Z_0}$$

assuming the reference plane is at the line/ specimen interface

Coaxial Line Reflectometry

- It follows that when the line is terminated with a short (Z_{short} = 0) $\boldsymbol{\Gamma}$ = -1, for an open termination ($Z_{open} = \infty$) $\boldsymbol{\Gamma}$ = 1, and for ideal load when $\boldsymbol{Z}s = Z_0$, $\boldsymbol{\Gamma}$ = 0. (NB. In practice, fringing capacitances have to be taken into account for the open termination).
- These three terminations are usually used for calibration with the reference plane at the specimen position.
- Coaxial test fixtures can be open-ended or short-ended
- Open-ended for large thick specimens or liquid materials
- Short-ended for thin films, specimens with dimensions comparable to the centre conductor of the probe



Coaxial Line Reflectometry

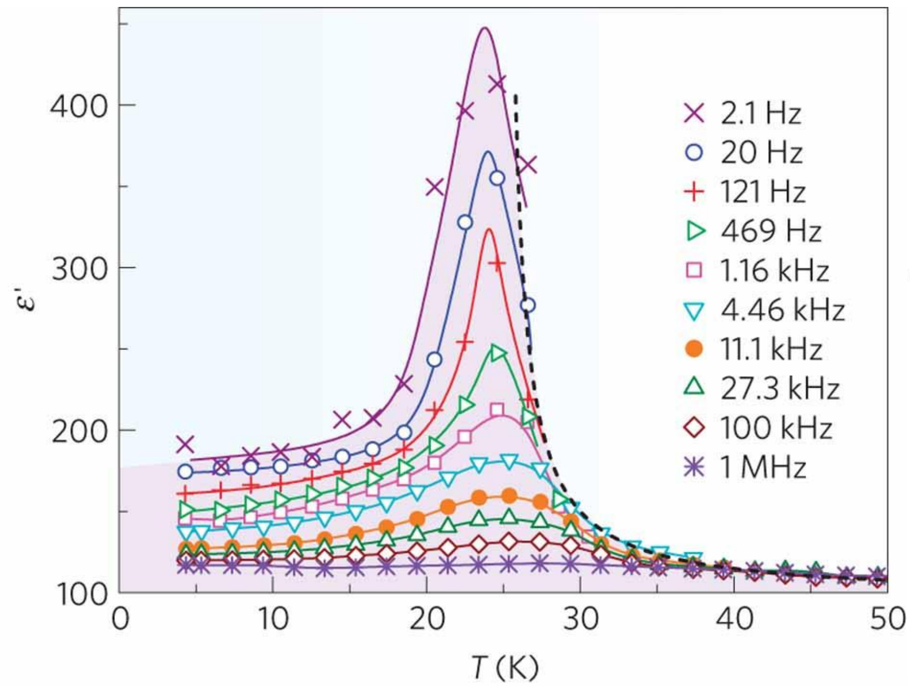
- Dielectric materials with known permittivity are used as references for correcting systematic errors in measuring ***Γ*** because of differences between measurement and calibration configurations
- If the circuit can be described with lumped parameters then $\varepsilon_r^{'}$ and $\varepsilon_r^{''}$ can be obtained from the measured ***Γ*** by:

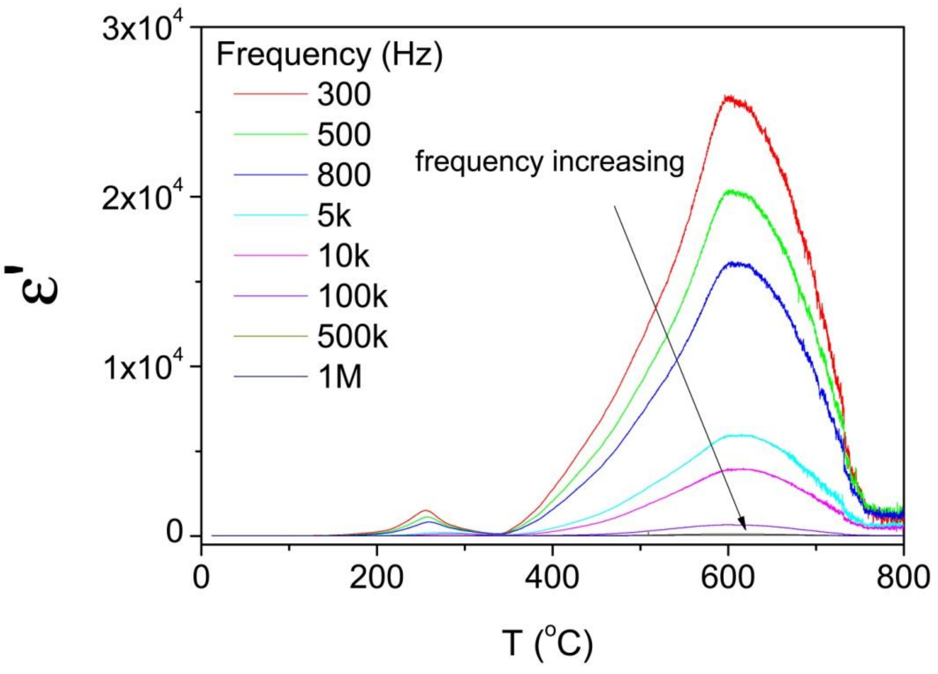
$$\varepsilon_r^{'} = \frac{-2\left|\vec{\Gamma}\right|\sin\varphi}{\omega Z_0 C_0 (1 + 2\left|\vec{\Gamma}\right|\cos\varphi + \left|\vec{\Gamma}\right|^2}$$

$$\tan\delta = \frac{\varepsilon_r^{''}}{\varepsilon_r^{'}} = \frac{1 - \left|\vec{\Gamma}\right|^2}{-2\left|\vec{\Gamma}\right|\sin\varphi}$$

Things to consider

- Permittivity, dielectric loss and relaxation frequency are temperature dependent so temperature has to be monitored and should normally be kept constant (isothermal conditions) during the measurements





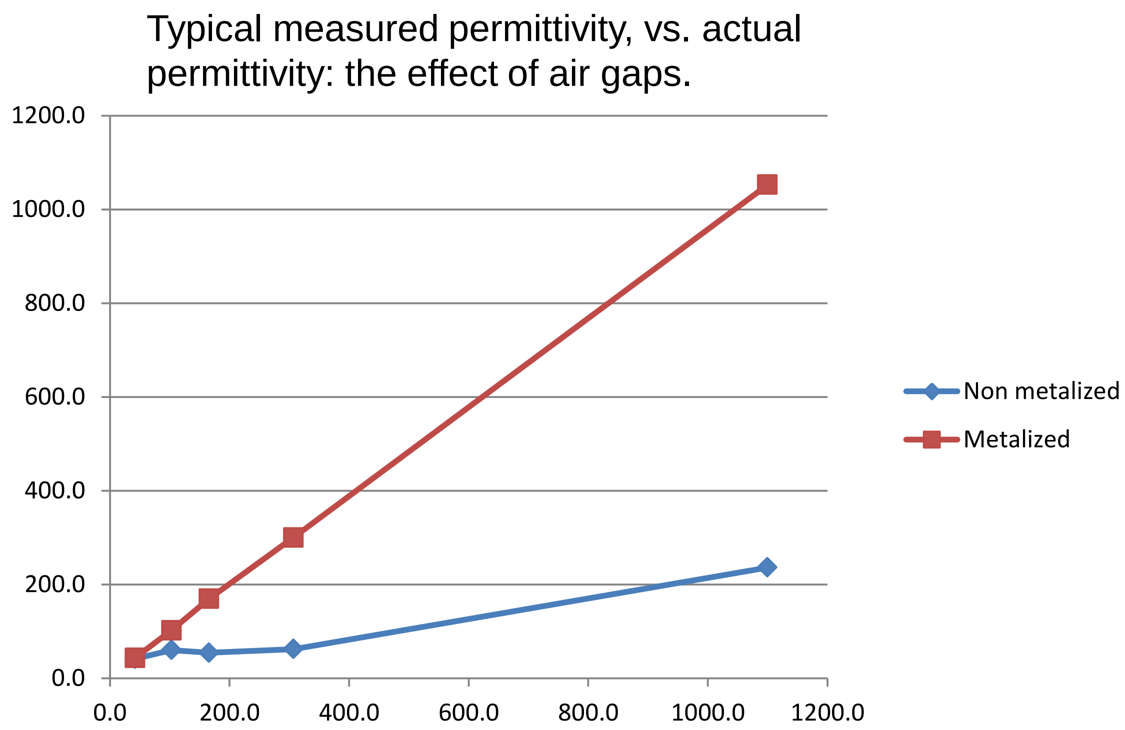
Things to consider

- Sample dimensions have to be accurately measured to minimize errors when calculating permittivity
- Sample faces which come in contact with the electrodes may have to be metalized, especially for high permittivity specimens (metallization performed *after* measuring dimensions)
- Metallization using silver paste, liquid metals (InGa), adhesive cooper foils…

Things to consider

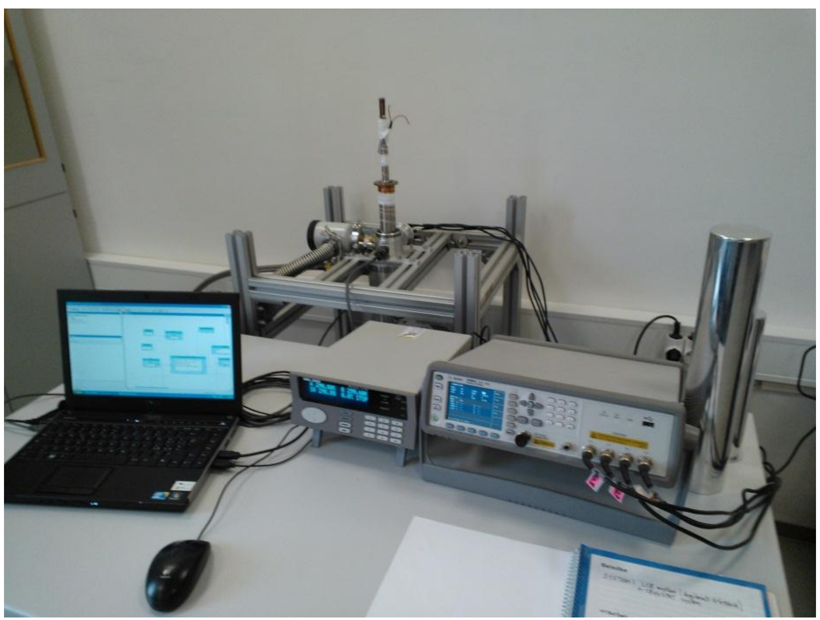
- Air gaps between the electrodes can introduce significant error especially for high permittivity samples

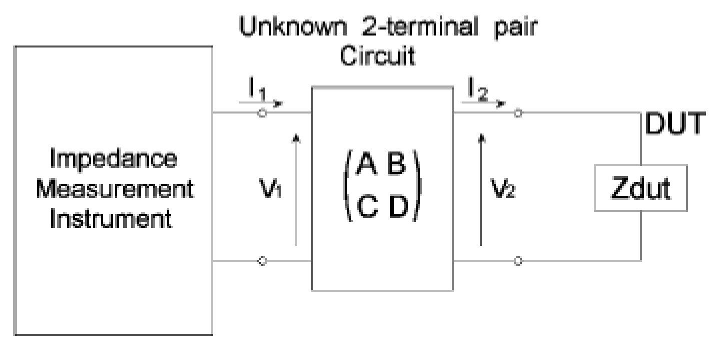
Ref. Value	No Metal	Metal
42,0	41,3	43,3
103,1	59,9	102,1
166	54	169,4
307	62	299,9
1100	236	1053
11000	463	10288



Things to consider

- Long cables from an instrument to a 4TP measuring cell can cause significant errors at higher frequencies (above 500 kHz), especially if the cables are moved.
- This can be corrected by making open, short and known load calibration at the end of the cables
- The load should be chosen so that it‘s impedance is similar to the impedance of the sample (reference materials can be used)





Conclusions

- The 4TP configuration is used for very low frequencies (5 Hz) and typically up to 30 MHz (possibly 100 MHz, but not many instruments are available for that)
- Lumped transmission line reflectometry can be used up to approximately 5 GHz depending on the sample size and line configuration
- Significant errors can arise from instrumental resonances, temperature instability, long cables, air gaps…
- All relevant equipment should be characterized before use: calibration of instruments, characterization of the cell, etc.
- Reference materials are to be recommended, especially for checking on systematic measurement errors and providing confidence in measurements.