Transfer standard developed at LNE-CETIAT

Bayan TALLAWI

Eric GEORGIN

Pierre SABOUROUX

1st BIOFMET Stakeholders' Workshop

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Introduction and Motivation

Currently, outside the framework of legal metrology, there is only few possibilities to calibrate instruments that measure moisture in solids.

Project of my thesis

BiofMET Project

Eric Georgin   Pierre Sabouroux

Ensure SI traceability of moisture content measurements in solids

Development of a microwave and/or high frequency instrument to measure moisture in biofuels

Wood chips

Biofuels

Objectives:

i. To correlate dielectric permittivity with humidity.

ii. Towards in-line sensor solutions...

Widespread measurement techniques

Moisture content / Moisture water measurement

- Primary techniques
  - (Thermo) gravimetric methods
    - Moisture analyzer
    - Loss-on-drying
    - ,,,
  - Chemical methods
    - Titration Karl Fischer
    - Evolved Evaporated water
    - ,,,
- Secondary techniques
  - Electromagnetic techniques
    - Coaxial cell
    - Resonant cavity
    - Coaxial probe
  - Nuclear electromagnetic resonance techniques
  - …

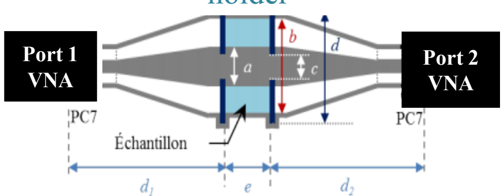
Moisture content/Water content ← **Dielectric permittivity**

Calibration and traceability of measurement

Electromagnetic techniques

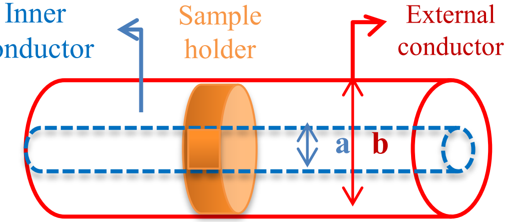
EpsiMu® cell developed at Fresnel Institute

Sample holder



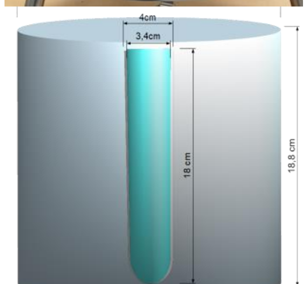
$a = 5,66\ mm$ & $b = 13\ mm$

Coaxial cell developed at *CETIAT*



$a = 8,41\ m$ & $b = 19,4\ mm$

Cylindrical resonant cavity developed at *CETIAT*



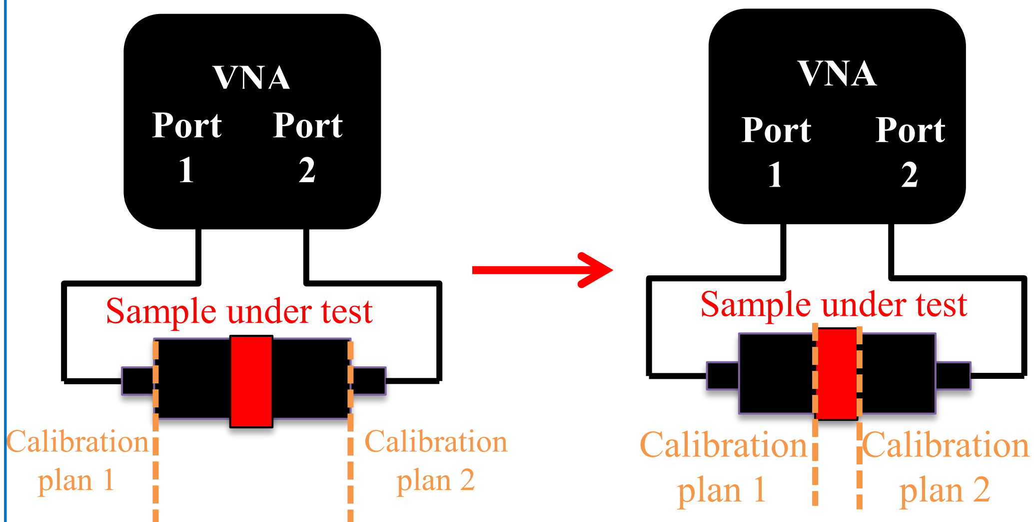
\+  Reflection and transmission coefficients 

- Complex dielectric permittivity
- Complex Magnetic permeability
- Resonance frequency

Coaxial cells : Measurement principle - Transmission measurement

1 **Before De-embedding**

2 **After De-embedding**



$[S_{ij}]_{cell}$ $\quad$ $[S_{ij}]_{SUT}$

3 **Nicholson and Ross protocol**

S_{11}, S_{21} of the sample under test

↓

Reflection coefficient $\Gamma = \dfrac{\sqrt{\frac{\mu_r}{\epsilon_r}} - 1}{\sqrt{\frac{\mu_r}{\epsilon_r}} + 1}$

↓

Transmission coefficient

$$T = e^{-j\frac{2\pi f}{c}\sqrt{\mu_r.\epsilon_r}\, d}$$

↓

Complex permeability μ_r

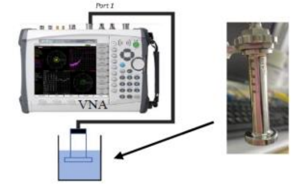
↓

Complex permittivitty ε_r

$$\boldsymbol{\varepsilon_r = j\frac{c}{2\pi f L}\left(\frac{1+\Gamma}{1-\Gamma}\right)\ln\left(\frac{1}{T}\right)}$$

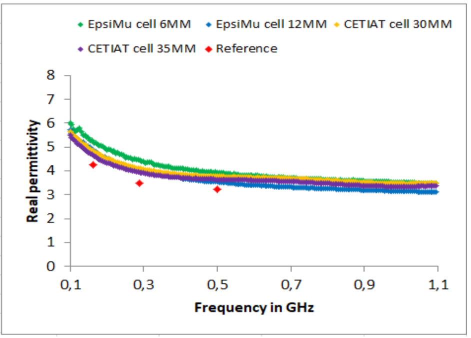
$$\mu_r = j\frac{c}{2\pi L}\left(\frac{1-\Gamma}{1+\Gamma}\right)\ln\left(\frac{1}{T}\right)$$

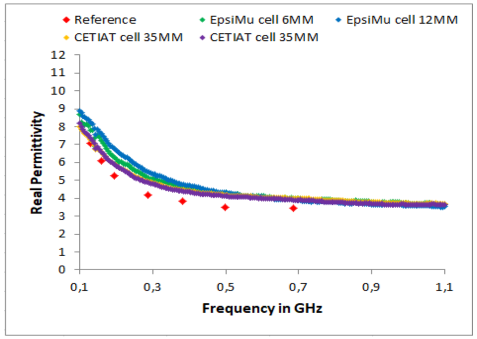
Coaxial cells : Results

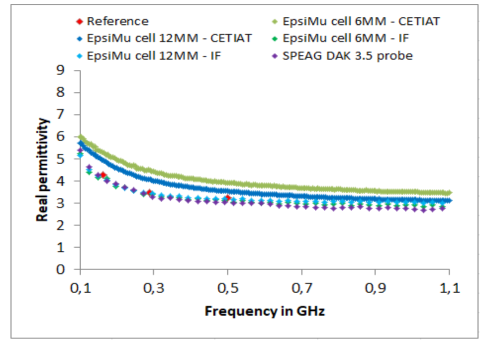


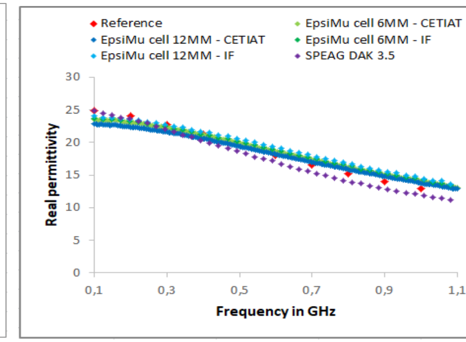
Intra-laboratory comparison at CETIAT

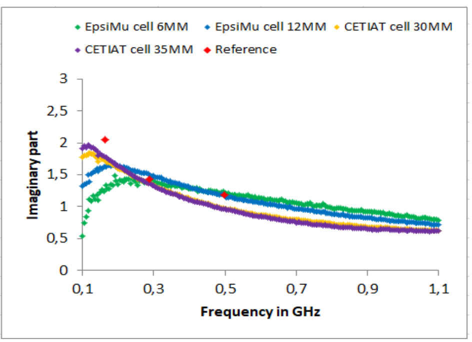
Inter-laboratory comparison between CETIAT and Fresnel Institute

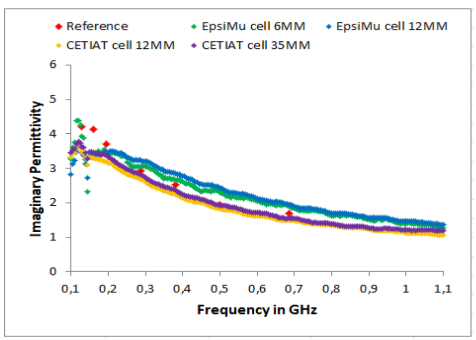


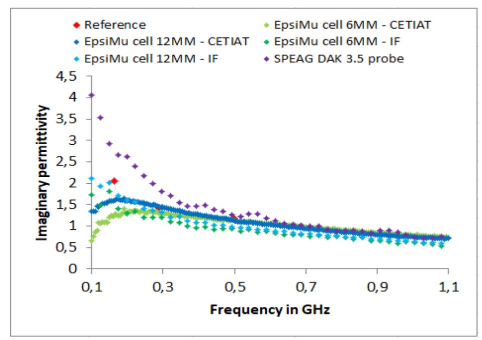


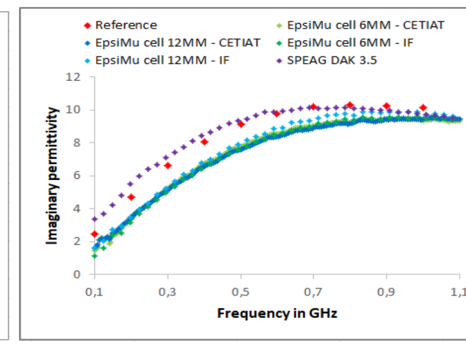












Comparison between the two coaxial cells at 20°C of **Decanol** (a) Real permittivity (b) Imaginary permittivity.

Comparison between the two coaxial cells at 20°C of **Heptanol** (a) Real permittivity (b) Imaginary permittivity.

Inter laboratory comparison for dielectric parameters of **Decanol.**

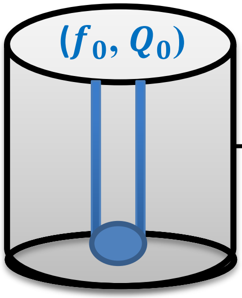
Inter laboratory comparison for dielectric parameters of **Ethanol.**

Cylindrical resonant cavity

Glass tube

Dimensions :
Hight = 180 mm
inner diameter = 36,4 mm
Outer diameter = 40 mm

1 Empty cavity



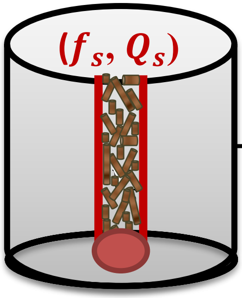
f_0 : Resonant frequency of the empty cavity
Q_0 : Quality factor of the empty cavity

1

2

Vector Network Analyzer
VNA

2 Cavity filled with sample



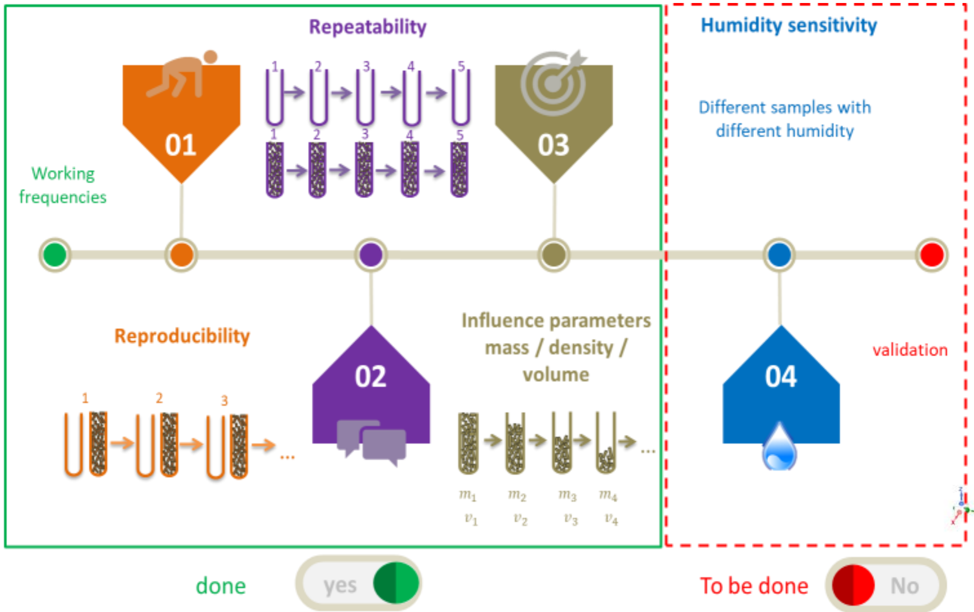
f_s : Resonant frequency of cavity filled with a material
Q_s : Quality factor of the cavity filled with a material

Perturbation theory

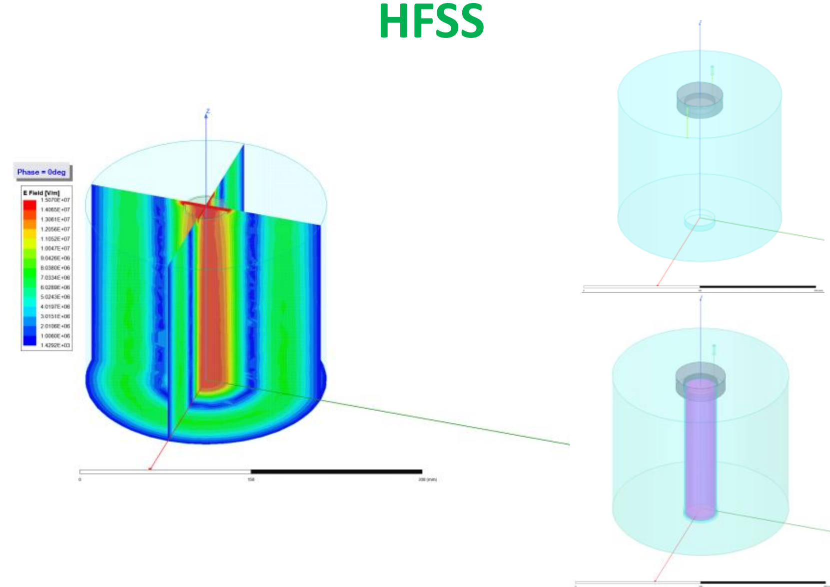
$$\varepsilon' = 1 + \frac{1}{A}\frac{V_c(f_0 - f_s)}{V_s f_s}$$

$$\varepsilon'' = B\frac{V_c}{V_s}\left(\frac{1}{Q_s} - \frac{1}{Q_0}\right)$$

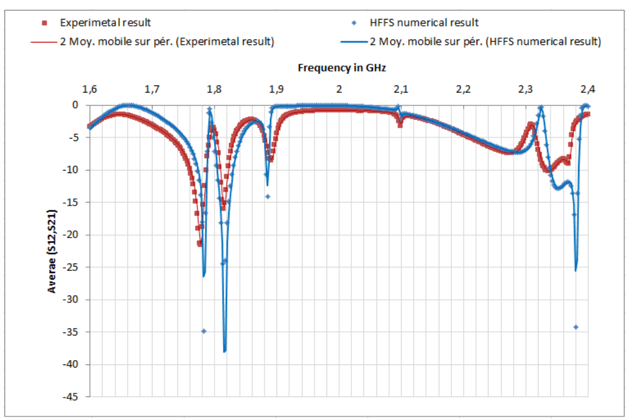
ε' : The real part of the complex dielectric permittivity
ε'' : The imagine part of the complex dielectric permittivity
V_c : The volume of the cavity resonator
V_s : The volume of the test sample
A, B : calibration constants



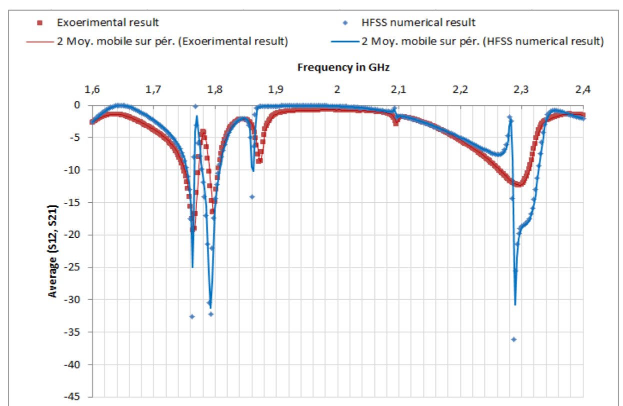
EM simulation and modelisation with HFSS



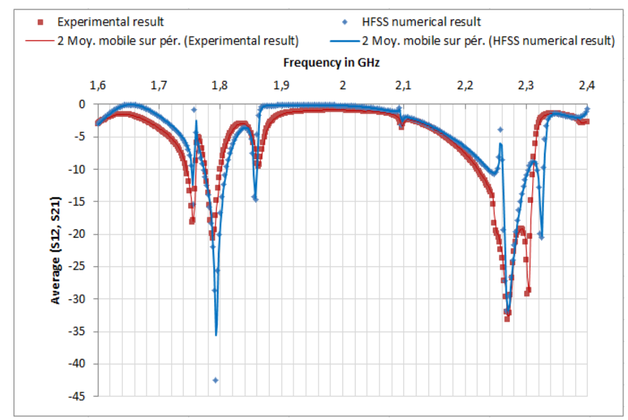
Comparison between experimental and numerical results



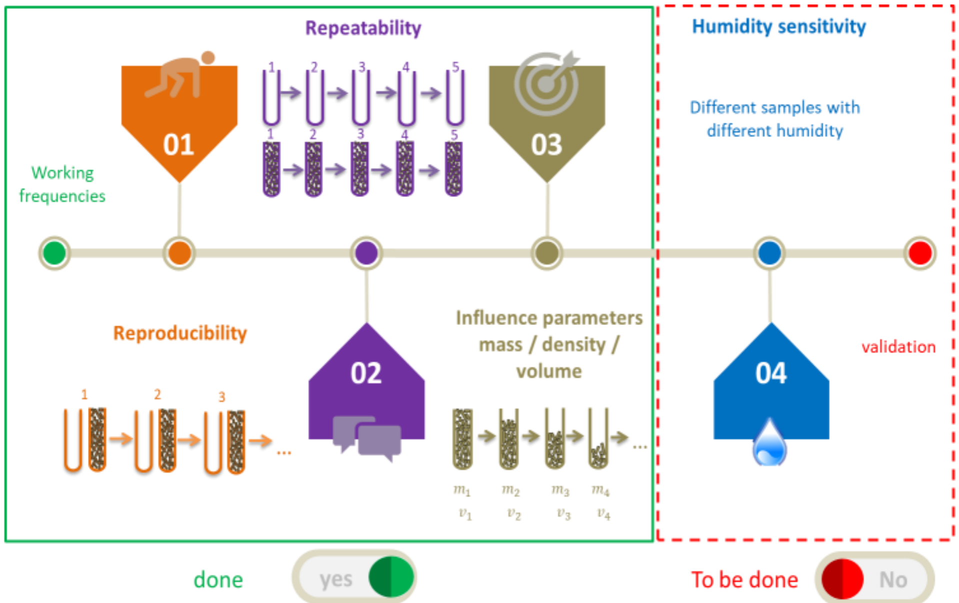
Empty cavity



Empty tube

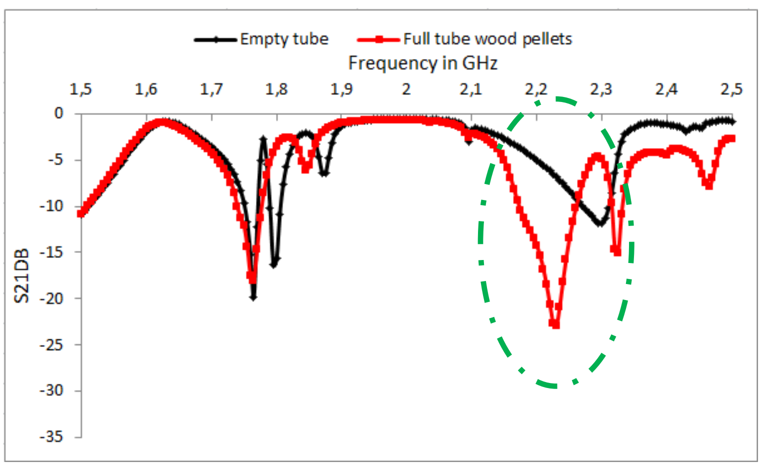


PTFE Cylindrical sample

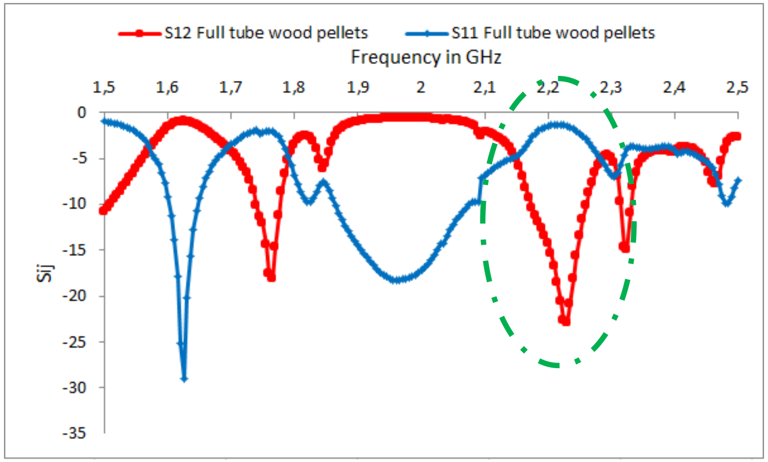


Working frequency band

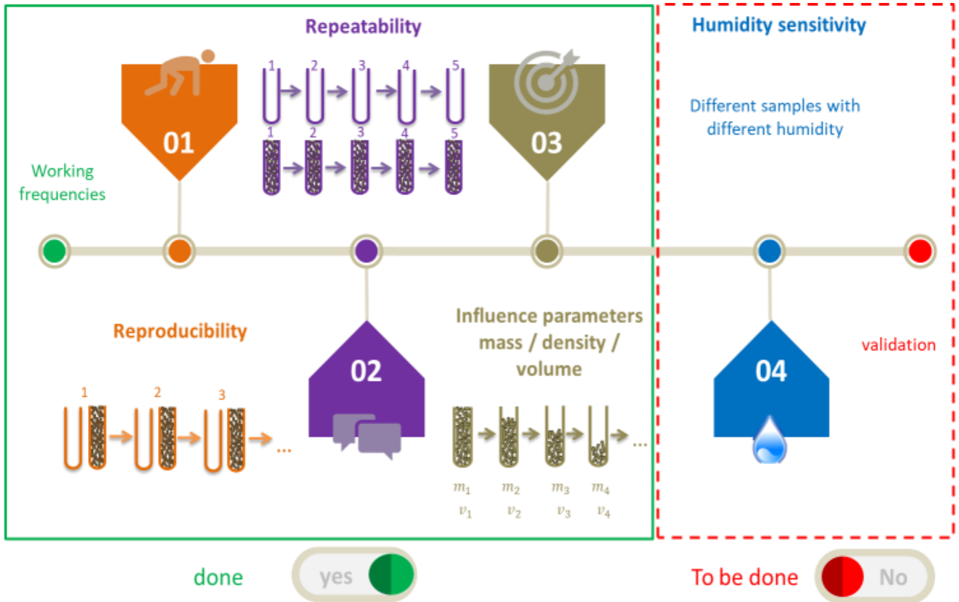
Resonant frequency shift from **2.298**GHz for the empty cavity to **2.222**GHz in the presence of the sample



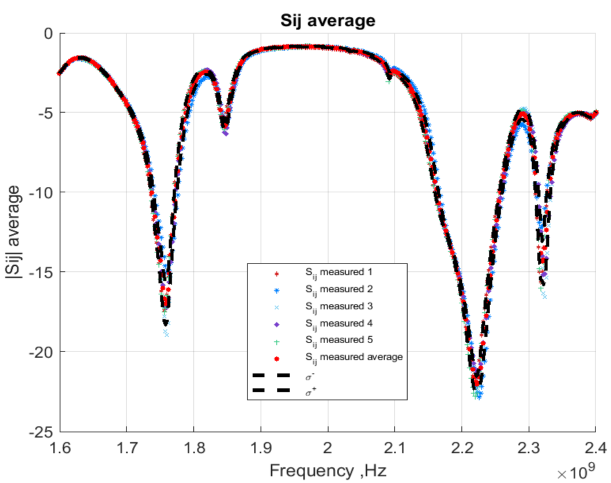
Significant resonance phenomenon in the band [2,1 GHz-2,3 GHz]



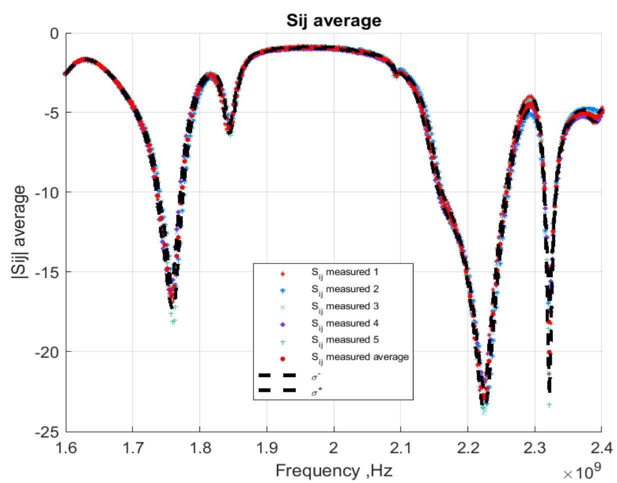
Transmission and reflection phenomena



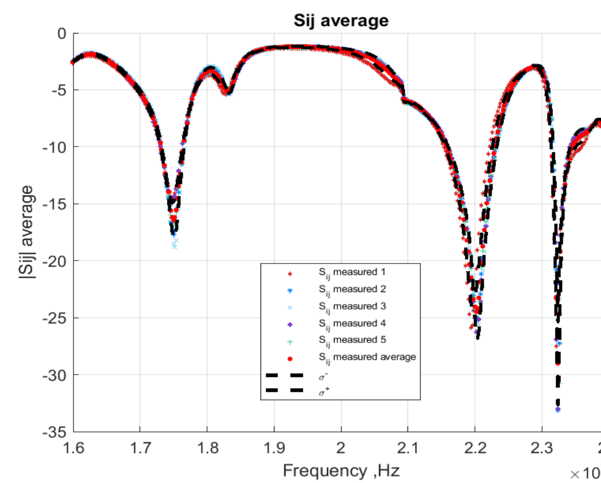
2 Reproductibility



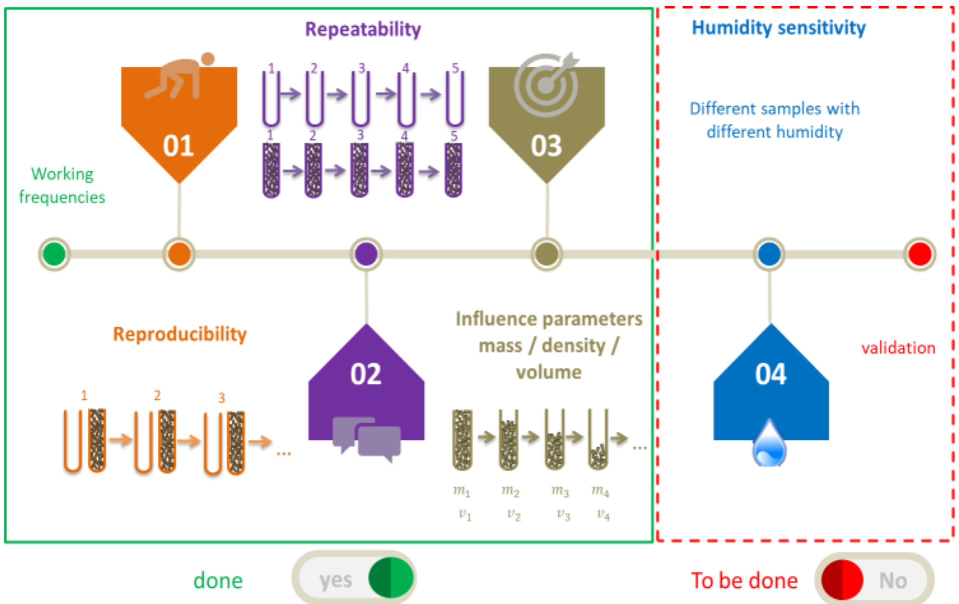
Cogra – CETIAT Wood pellets



DTI wood pellets

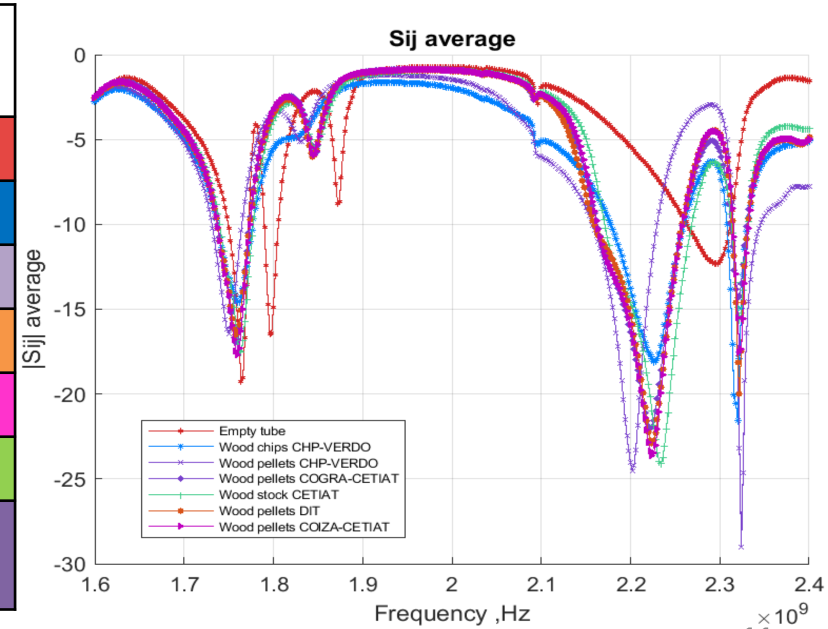


CHP Verdo Wood pellets

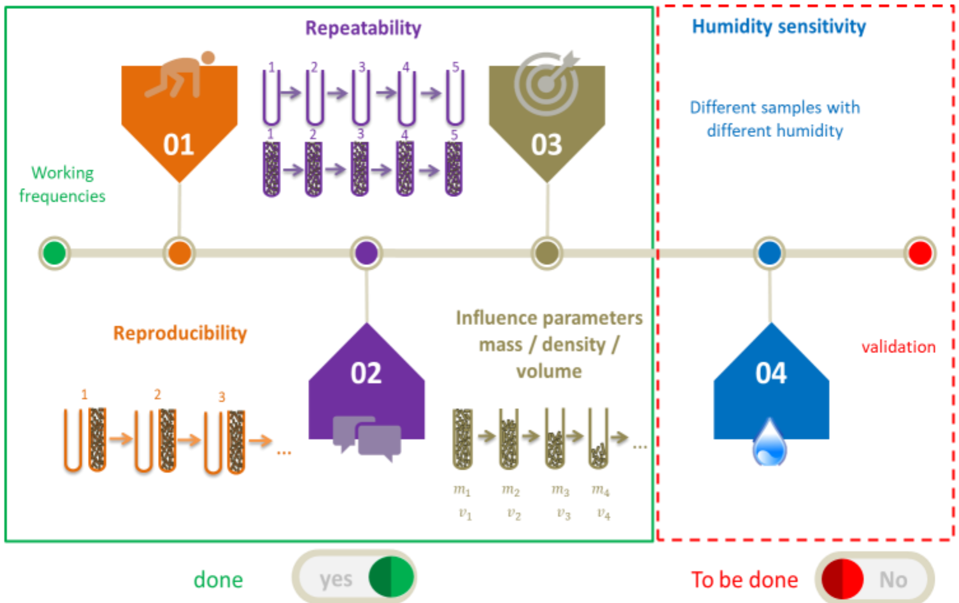


2 Reproductibility

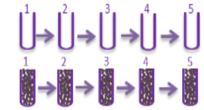
	f_s (GHz)	Deviation (GHz)	$\Delta f = \frac{f_s - f_0}{f_s}$
Empty tube	2,2944	0,0045	-
CHP Verdo Wood chips	2,2232	0,0084	-0,0320
CHP Verdo Wood pellets	2,2008	0,0035	-0,0425
DTI Wood pellets -E	2,2248	0,0020	-0,0313
Colza Wood pellets	2,2332	0,0016	-0,0274
Wood Stock – CETIAT	2,2244	0,0023	-0,0315
Cogra – CETIAT Wood pellets	2,2224	0,0023	-0,0324



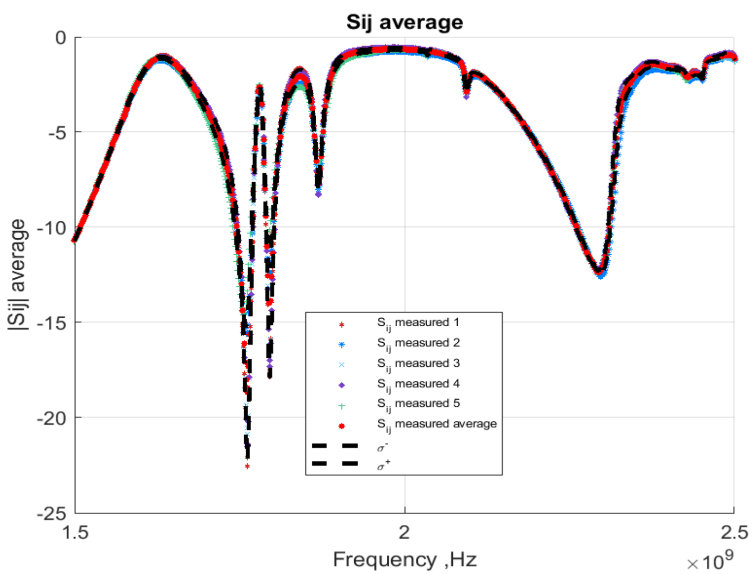
BAYAN TALLAWI
PhD student, CETIAT & Fresnel Institute , France



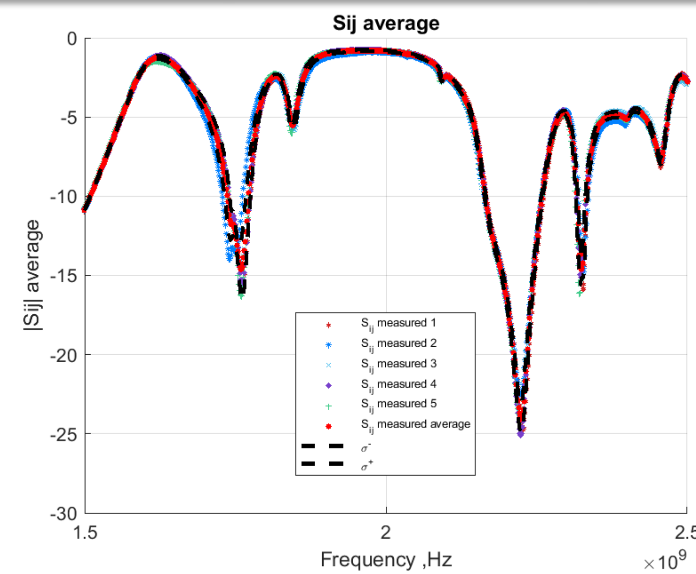
3 Repeatability



	Resonance frequency (GHz)	
	Tube filled with sample	empty tube
N1	2,2225	2,2963
N2	2,2225	2,2975
N3	2,2238	2,2913
N4	2,2225	2,2925
N5	2,2225	2,2940
MEAN	2,2228	2,2943
s	0,0006	0,0026
s^2	3,3800E-07	6,6420E-06



Empty tube



Tube filled with wood pellets

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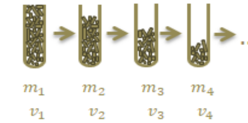
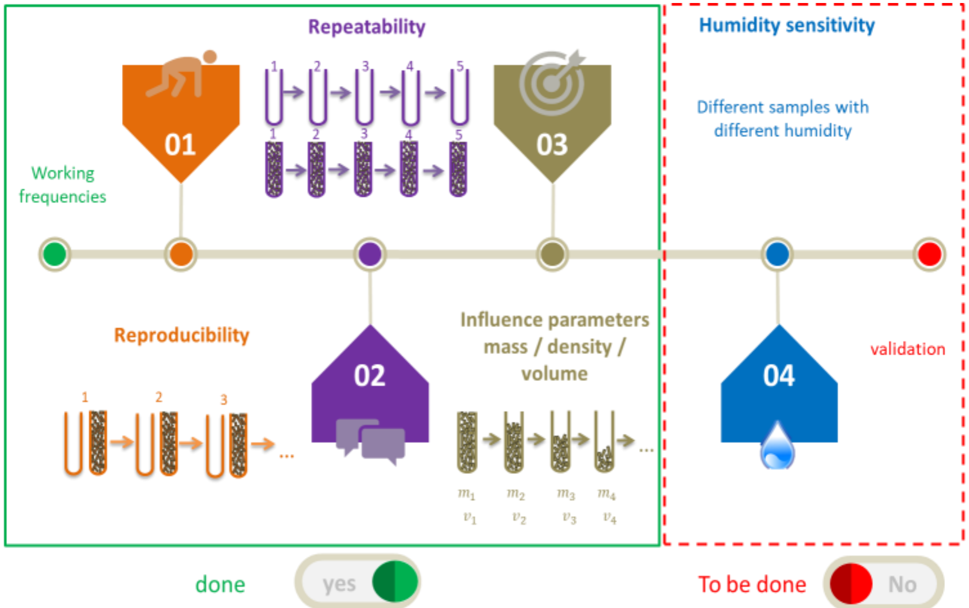
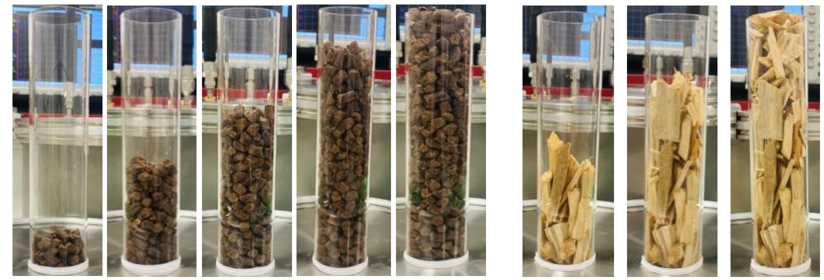
PhD student, CETIAT & Fresnel Institute , France

3 Influence parameters mass/density/volume

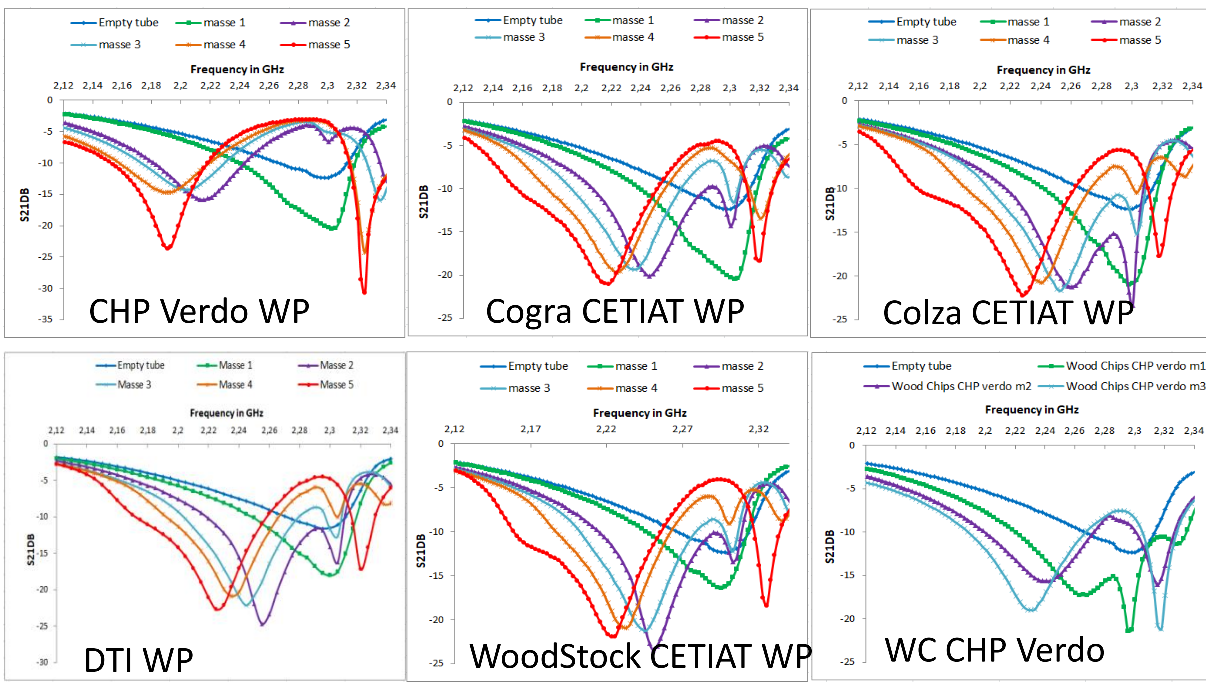


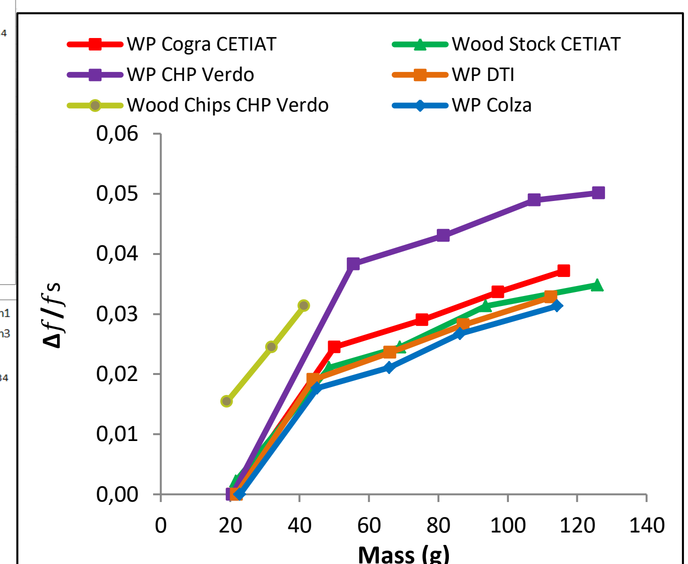




$mass_1$ $mass_2$ $mass_3$ $mass_4$ $mass_5$ $mass_1$ $mass_2$ $mass_3$

v_1 v_2 v_3 v_4 v_5 v_1 v_2 v_3





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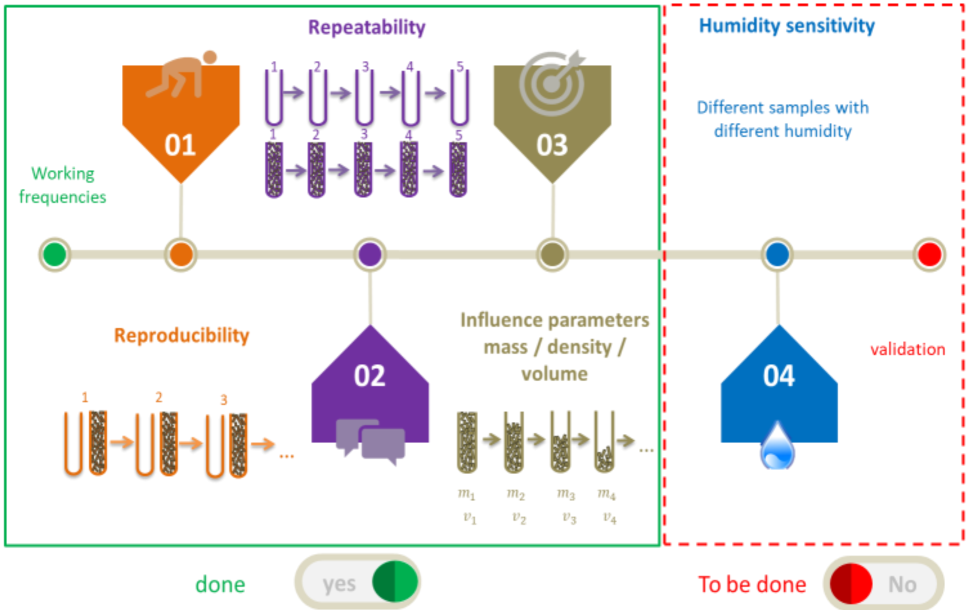
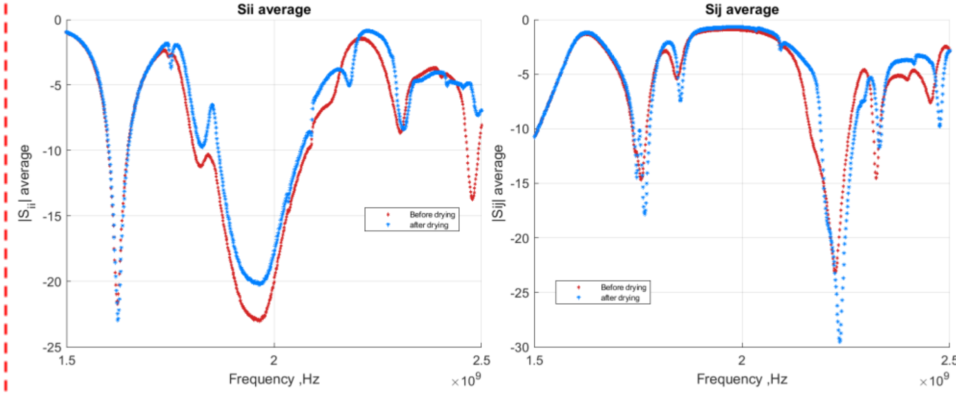


4 Humidity sensitivity (In Progress)



Experiment 1

Moisture content calculation LOD1

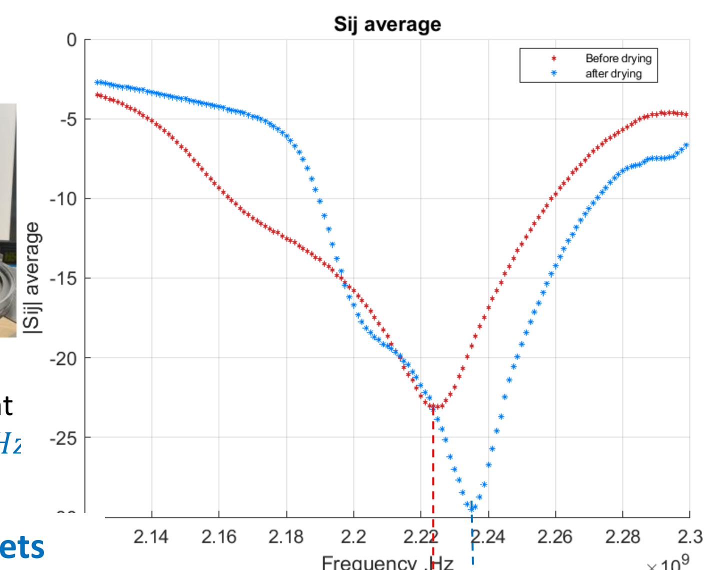
Initial weighing $m_1 = 119{,}12g$

EM Measurement $f_1 = 2{,}225\ GHz$

Put the sample in the oven for 3 hours at 105 °C

Weighing after drying $m_2 = 111{,}96g$

EM Measurement $f_2 = 2{,}235\ GHz$



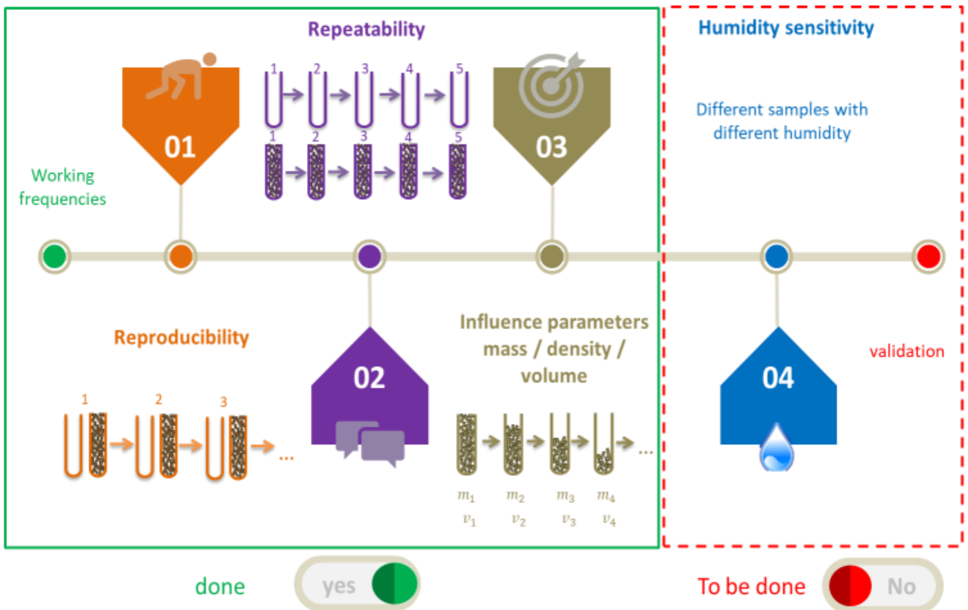
Wet wood pellets
Moisture content = 6,01%

Dry wood pellets

$\Delta f = 0{,}01\ GHz$ for 6% evaporated moisture

2,225 GHz

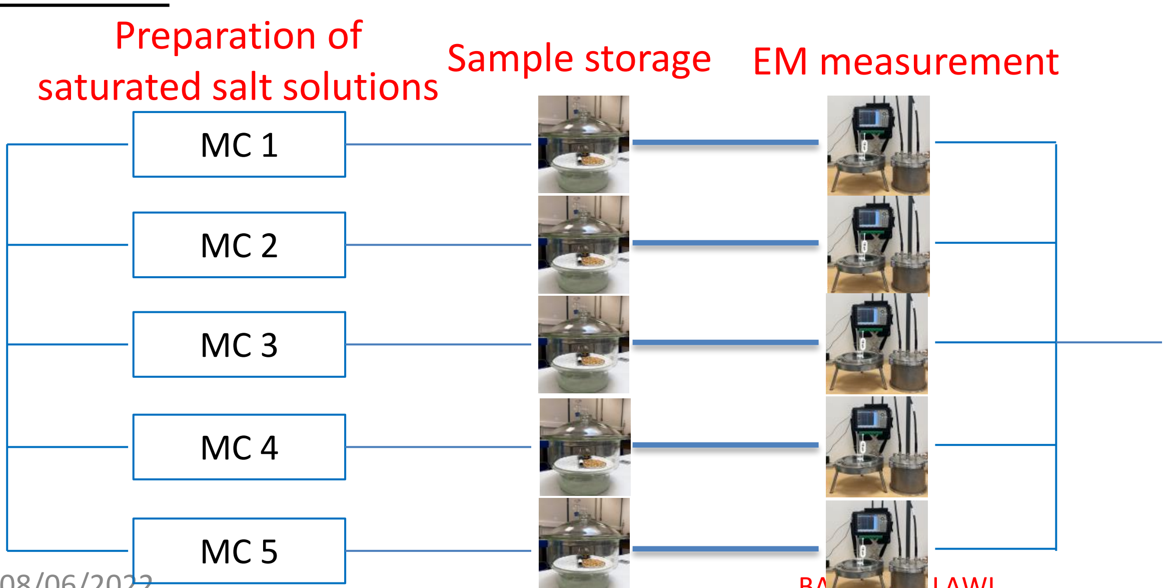
2,235 GHz

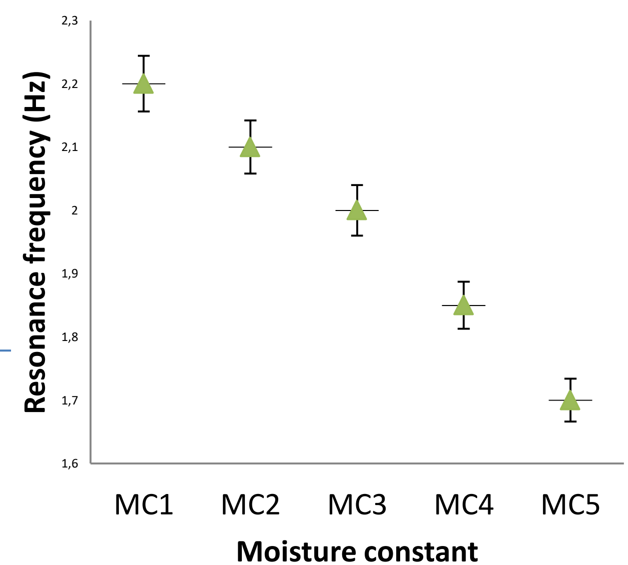


4 Humidity sensitivity (To be done)

- Study the resonant frequency shift for samples with different humidity levels.

Experiment 2





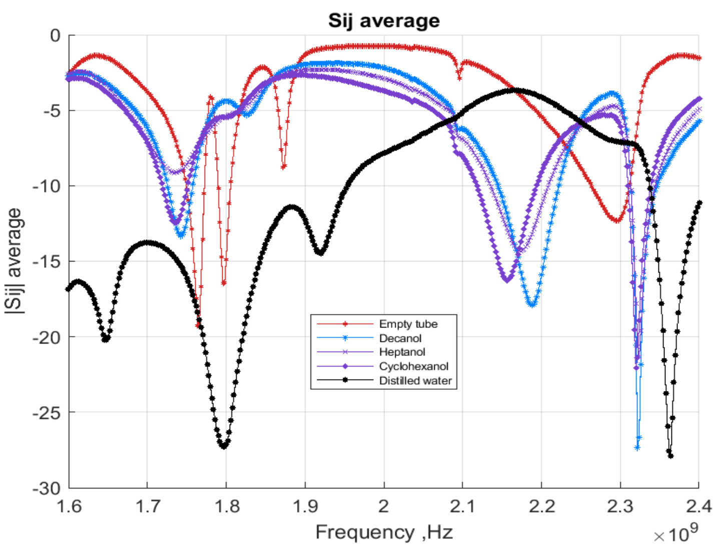
How to calculate dielectric permittivity with Cylindrical resonant cavity?

$$\left(\frac{f_2 - f_1}{f_2}\right) = (\varepsilon'_r - 1)A$$

$$\left(\frac{1}{Q_2} - \frac{1}{Q_1}\right) = \varepsilon'' B$$

The permittivity, more precisely the real dielectric permittivity, is a physical property, which describes the response of a given medium to an applied electric field.

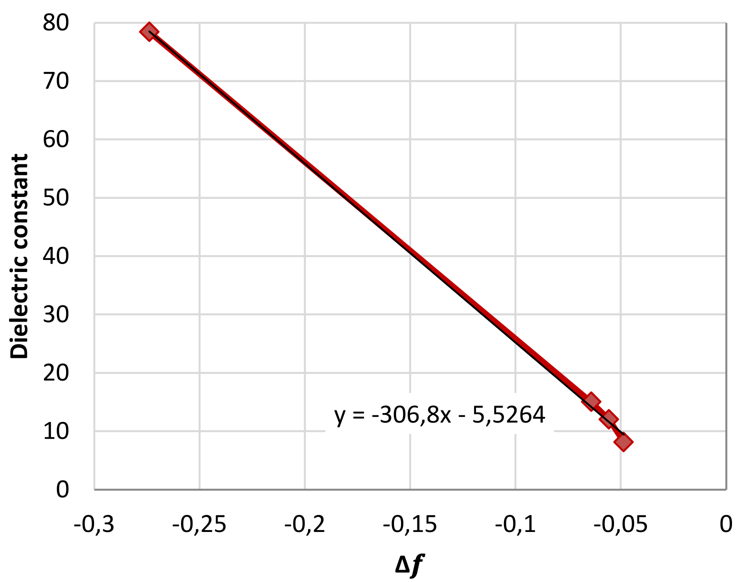
f_1 : *resonance frequency before sample introduction*
f_2 : *resonance frequency after sample introduction*
Q_1: quality factor of the empty caity
Q_2: quality factor of theperturbed cavity
A, B : calibration parameters assumed to be constants and independents of sample properties

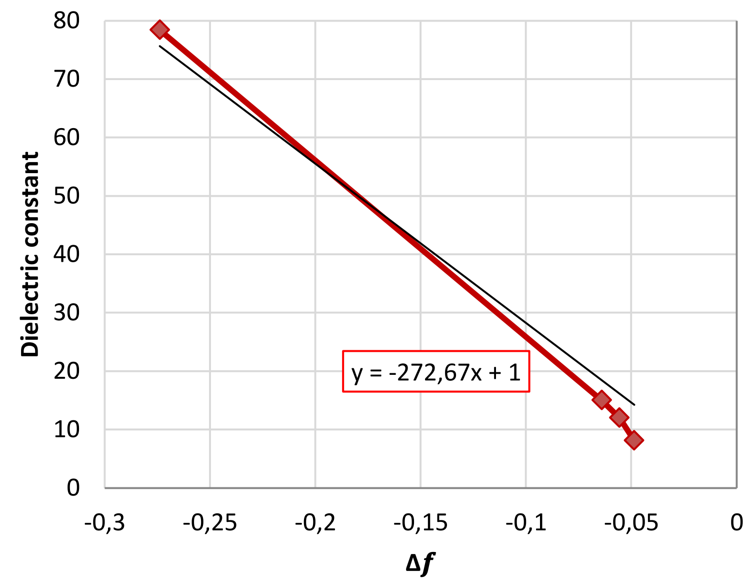


Reference materials	f_r (GHz)	Δf	Deviation (GHz)	Dielectric constant (reference)
Decanol	2,1880	-0,0486	0,0013	8,1
Heptanol	2,1736	-0,0556	0,0020	12
Cyclohexanol	2,1564	-0,0640	0,0008	15
Distilled water	1,8012	-0,2738	0,0108	78,4

How to calculate dielectric permittivity with Cylindrical resonant cavity?

$$\left(\frac{f_2 - f_1}{f_2}\right) = (\varepsilon'_r - 1)A \longrightarrow \varepsilon'_r = 1 + A\left(\frac{f_2 - f_1}{f_2}\right)$$





Linear equation : ε'_r = -272,67Δf + 1

A= -272,67

In progress

BAYAN TALLAWI
PhD student, CETIAT & Fresnel Institute , France

How to ensure the traceability of our measurements to the SI ?

Measured via a VNA

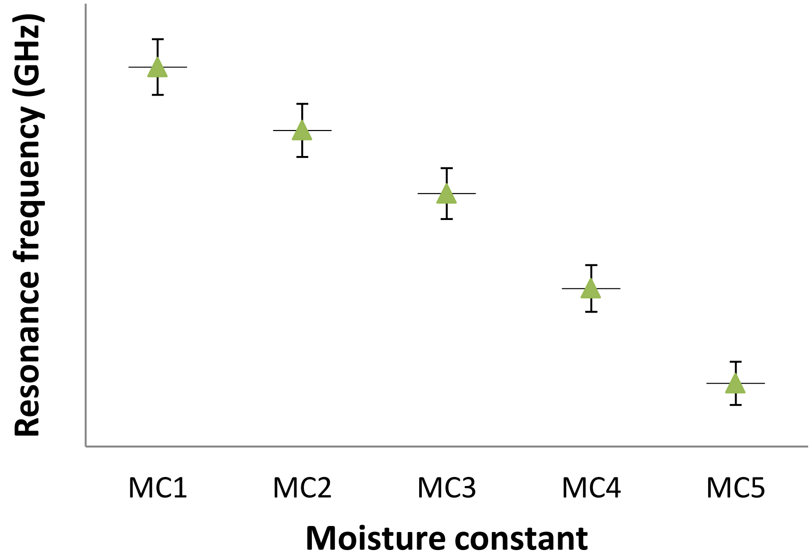
S_{12}Transmission coefficients

Uncertainties estimation of according to the guide EURAMET No. 12

S parameters traceable to SI

Resonance frequency measurement traceable to SI

+ Permittivity measurement traceable to SI



Measured with reference techniques traceable to SI

Calibration curve

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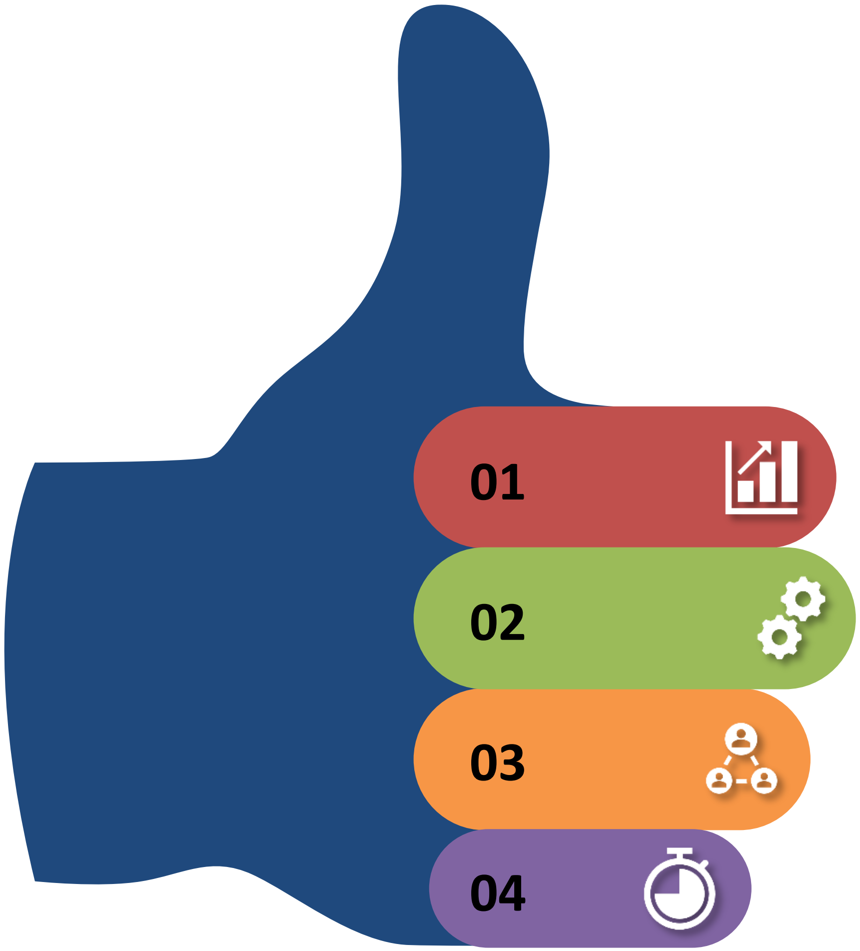
PhD student, CETIAT & Fresnel Institute , France

Advantages of our secondary developed measuring system



01. Reproductible, repeatble and precise

02. Calibrated against a primary methods

03. Non-destructive method

04. fast-measurement (in a minute)

Thank you for your attention

Bayan Tallawi, Eric Georgin and Pierre Sabouroux

CETIAT & Fresnel Institute

France