An Industrial Approach Towards Traceable Moisture Measurements in Microwave Domain

2nd BIOFMET Workshop

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Outline

1. *Introduction and motivation*
2. *Widespread moisture measurement techniques*
3. *Reference methods used*
4. *Coaxial lines and microwave results*
5. *Cylindrical resonant cavity and microwave results*
6. *Conclusion and perspectives*

1. Introduction and motivation

Energy production

Renewable Energy

Quality control

Pharmaceutical industry

Thermophysical properties

Civil engineering

Shelf life

Food industry

1. Introduction and motivation

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

Thesis project

Eric Georgin ***Pierre Sabouroux***

Ensure SI traceability of moisture content measurements in solids.

Wood chips

Biofuels

Objectives:

i. To correlate dielectric permittivity with humidity.

ii. Towards in-line sensor solutions...

BiofMET project

CETIAT Work in BiofMET :

Development of a microwave and/or high frequency instrument to measure moisture in biofuels.
Coordinated by the DTI

www.biofmet.com

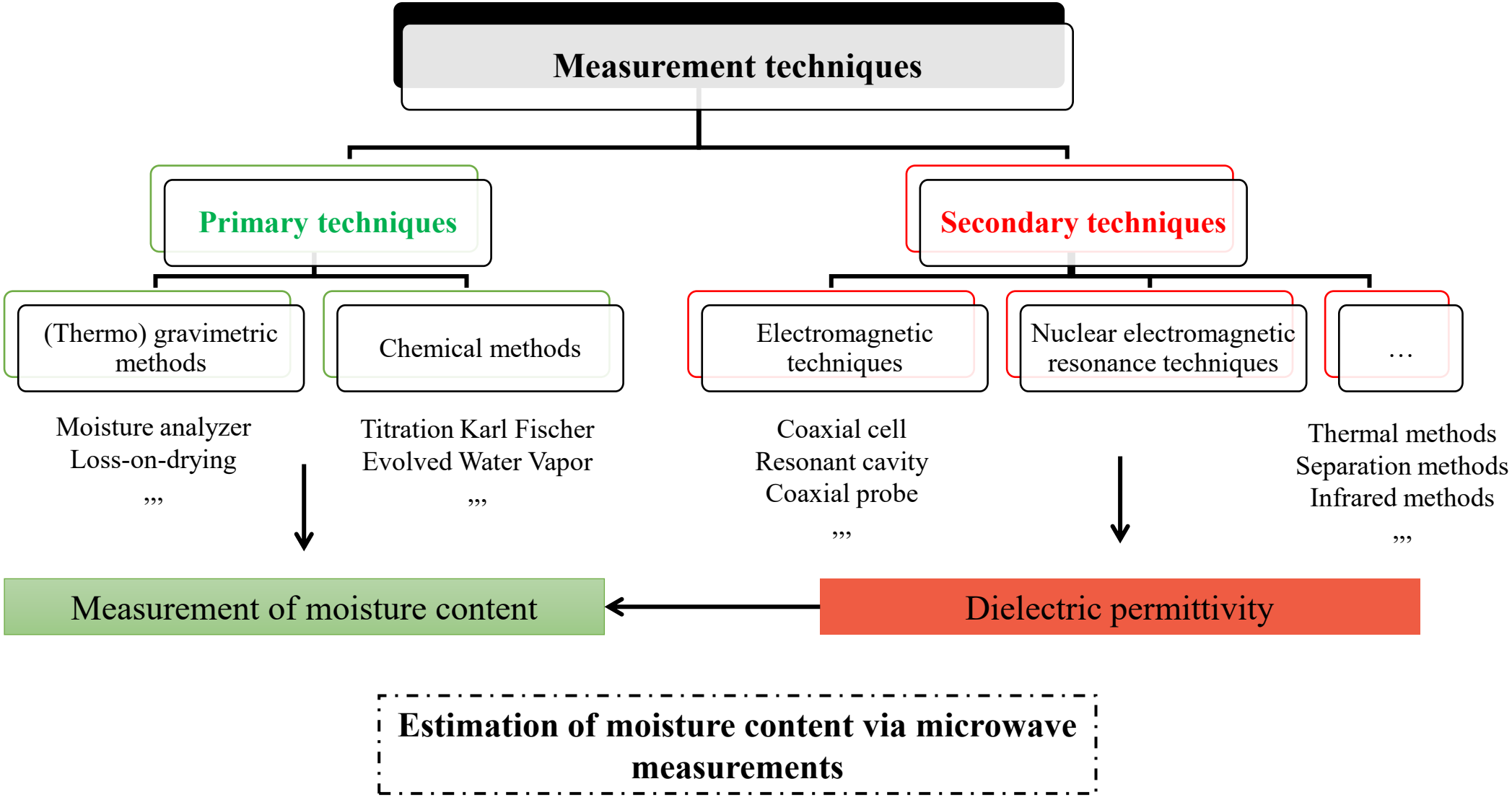
PARTICIPATING EURAMET NMIS AND DIS

CMI (Czech Republic)
DTI (Denmark)
IMBiH (Bosnia and Herzegovina)
INM-RO (Romania)
LNE-CETIAT (France)
PTB (Germany)
UME (Turkey)

OTHER PARTICIPANTS

České Vysoké Učení Technické v Praze (Czech Republic)
Instituto Superior Tecnico (Portugal)
Prometec Tools Oy (Finland)
Université d'Aix-Marseille (France)
Verdo Produktion A/S (Denmark)

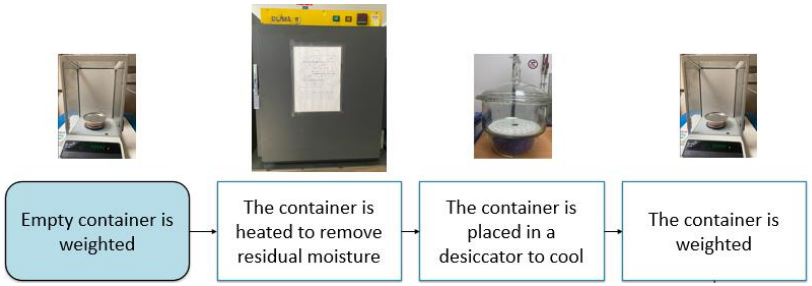
2. *Widespread moisture measurement techniques*



Estimation of moisture content via microwave measurements

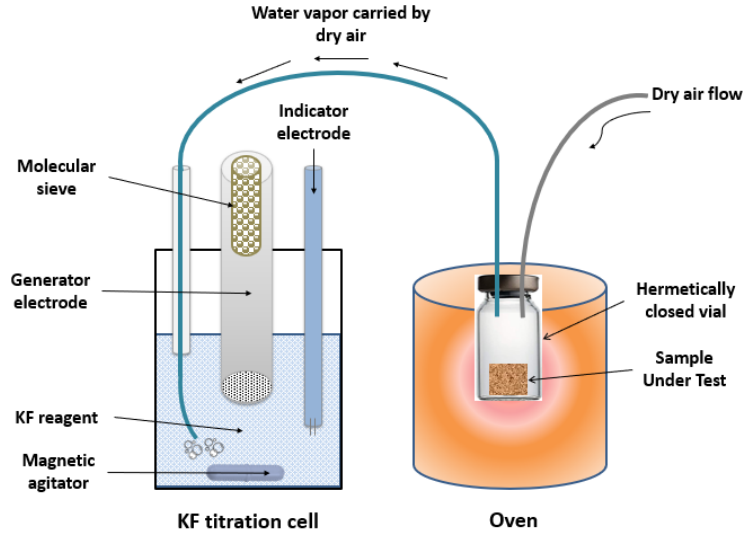
3. Reference methods used

Loss on Drying (LoD)



$$\textbf{Moisture content}\ \left(\frac{g}{100g}\right) = M_C = \frac{m_{LOAD} - m_N}{m_{LOAD} - m_{TARE}}$$

Coulometric Karl Fischer titration



$$ROH + SO_2 + R'N \rightarrow [R'NH]SO_3R$$

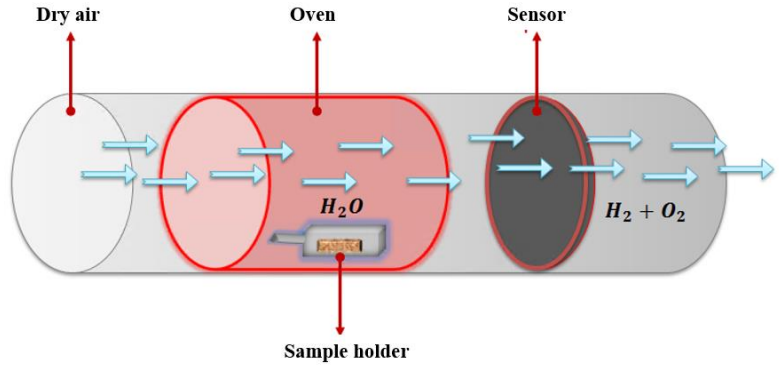
$$[R'NH]SO_3R + H_2O + I_2 + 2 \times R'N \rightarrow 2 \times [R'NH]I + [R'NH]SO_4R$$

Faraday's law

$$m = \frac{M \times \int i(t)dt}{Z \times F}$$

m – mass of water;
M – molar mass (water: 18.016 g/mol)
i(t)dt – electrical charge per time
Z – number of released electrons (2)
F – Faradays constant (96484.56 C/mol)

Evolved Water Vapor (EWV)

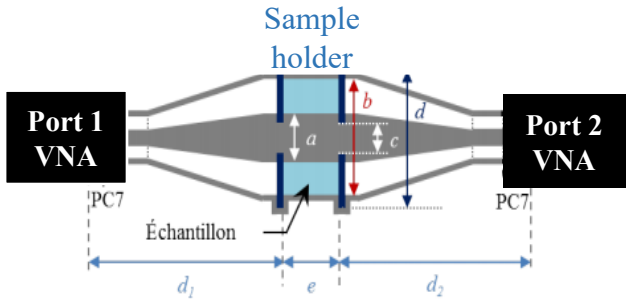


Absorption: $3 \times H_2O + P_2O_5 \rightarrow 2 \times H_3PO_4$

Electrolysis: $4 \times H_3PO_4 \rightarrow 2 \times P_2O_5 + 6 \times H_2 \uparrow + 3 \times O_2 \uparrow$

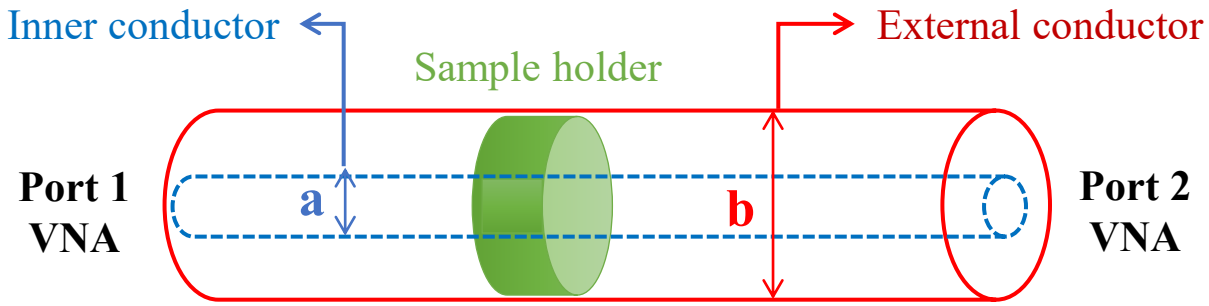
4. *Coaxial lines and microwave results*

EpsiMu® cell



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

Coaxial cell developed at *CETIAT*



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

\+

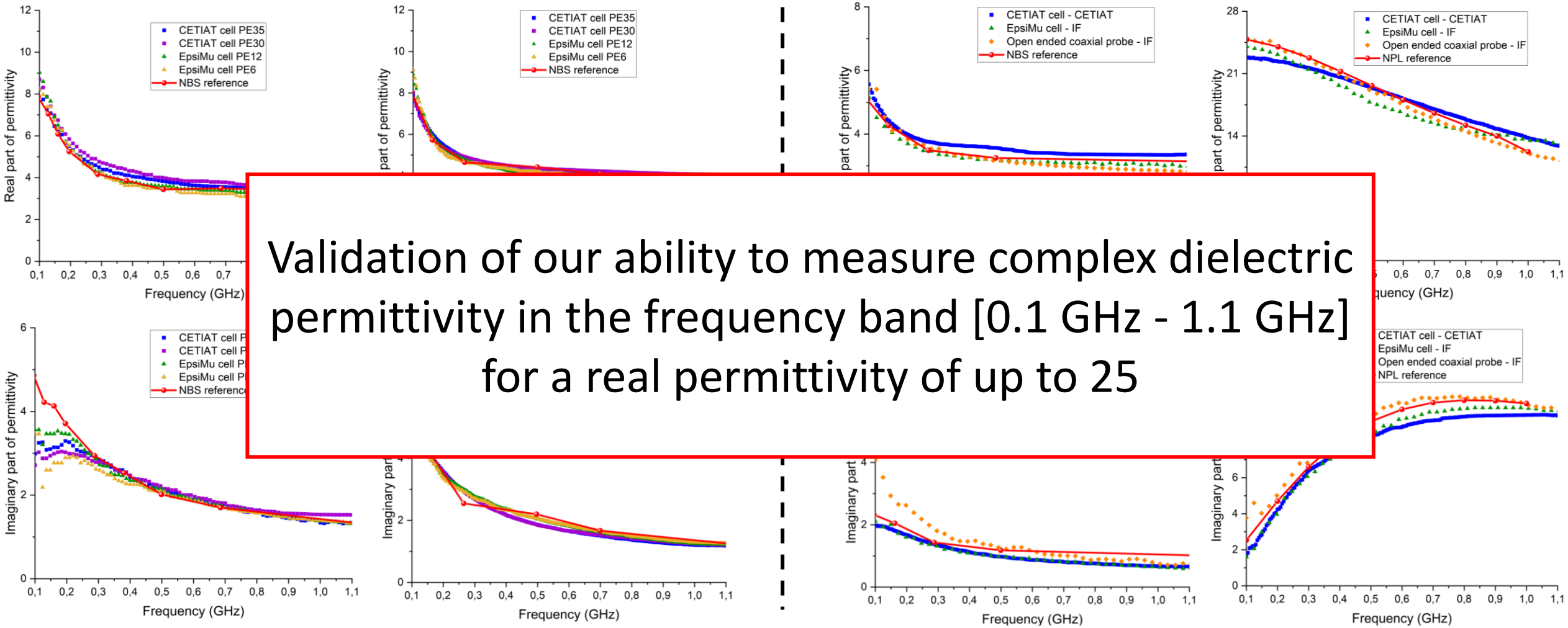
Reflection and transmission coefficients

Complex dielectric permittivity : $\varepsilon_r = j\frac{c}{2\pi fL}\left(\frac{1+\Gamma}{1-\Gamma}\right) ln\left(\frac{1}{T}\right)$

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

4. *Coaxial lines and microwave results*

Heptanol **Cyclohexanol** **Decanol** **Ethanol**



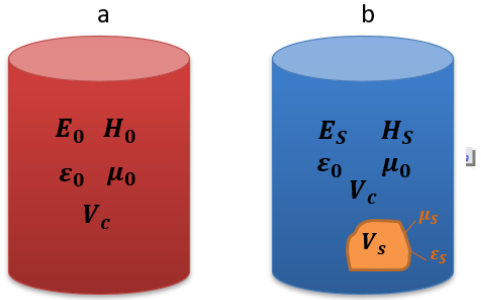
Validation of our ability to measure complex dielectric permittivity in the frequency band [0.1 GHz - 1.1 GHz] for a real permittivity of up to 25

Intralaboratory comparison at 20 °C

Interlaboratory comparison at 20 °C

5. Resonant cavity and microwave results

Fundamental theory



Original cavity Perturbed cavity

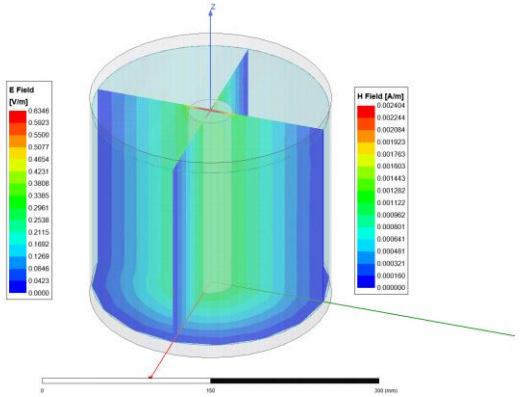
Cavity perturbation method (CPM)

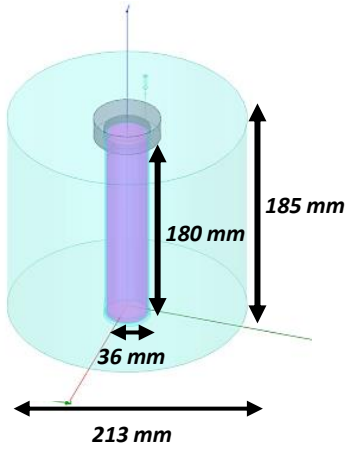
$$\varepsilon_r' = 1 + \frac{2}{C}\left(\frac{f_0 - f_s}{f_s}\right)$$

$$\varepsilon_r'' = \frac{1}{C}\left(\frac{1}{Q_s} - \frac{1}{Q_0}\right)$$

E_0 : Electric field for empty cavity;
H_0 : Magnetic field for empty cavity;
ε_0 : Permittivity of vaccum;
μ_0 : Permeability of vaccum;
V_c : Cavity volume;
E_s : Electric field for perturbed cavity;
H_s : Magnetic field for perturbed cavity;
ε_s : Permittivity of the sample;
μ_s : Permeability of the sample;
V_s : Sample volume;
C : Cell factor(cell constant).

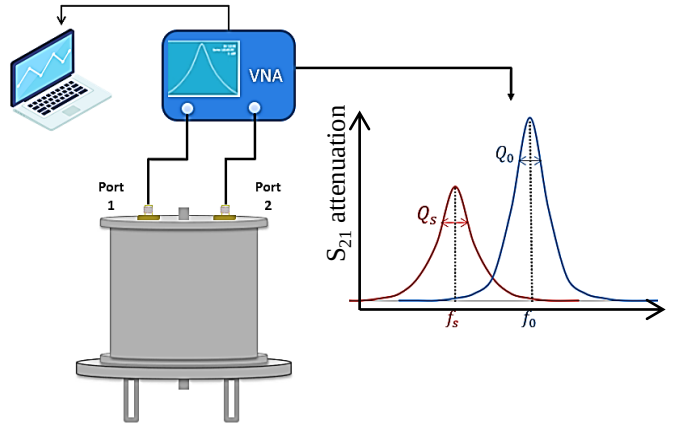
Electromagnetic modelling simulation

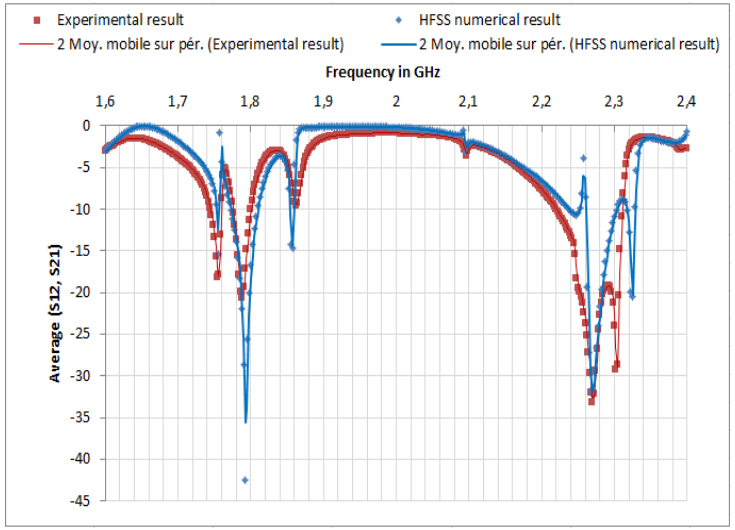




Photos for the developped cavity system

Experimental setup

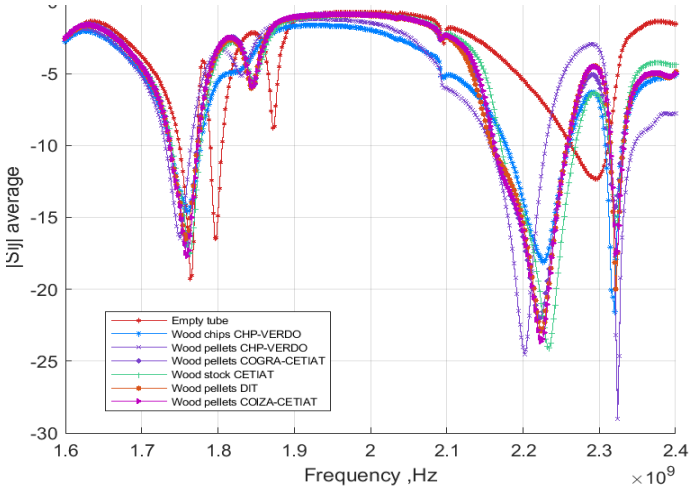




Analytical and experimental microwave spectrum for a Teflon sample

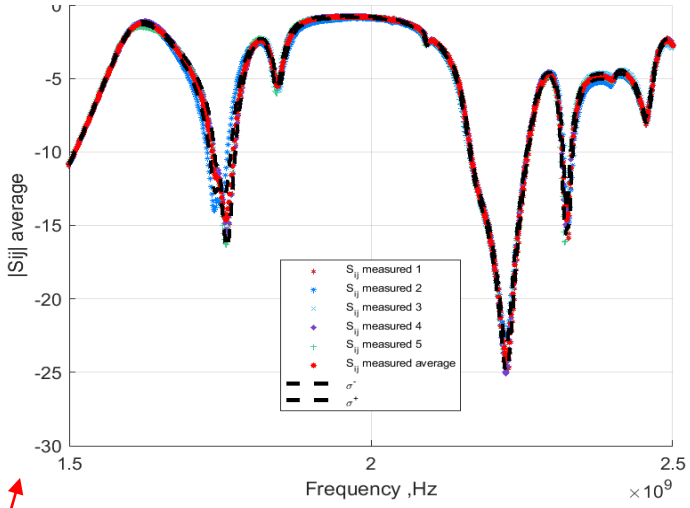
5. Resonant cavity and microwave results

Reproducibility



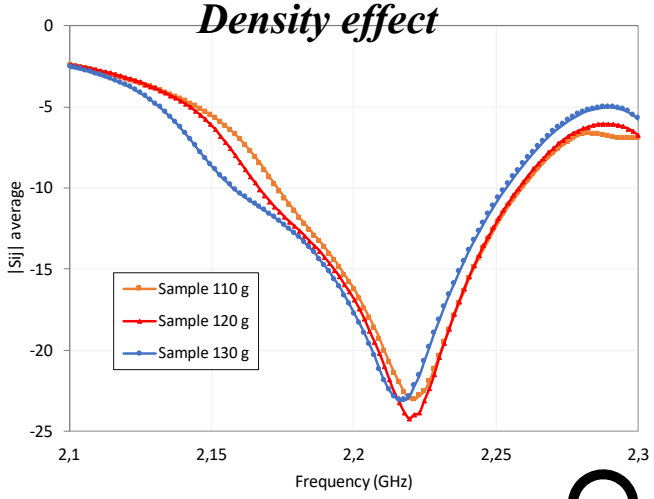
	f_s (GHz)	Standard deviation (GHz)	Δf
CHP Verdo Wood chips	2,2232	0,0084	0,0320
CHP Verdo Wood pellets	2,2008	0,0035	0,0425
DTI Wood pellets	2,2248	0,0020	0,0313
Colza Wood pellets	2,2332	0,0016	0,0274
Wood Stock – CETIAT	2,2244	0,0023	0,0315
Cogra – Wood pellets	2,2224	0,0023	0,0324

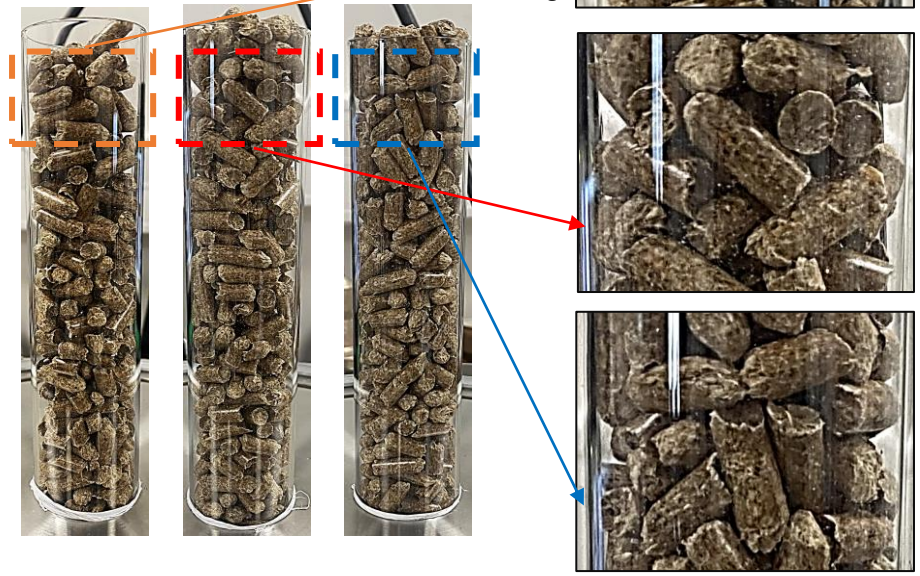
Repeatability



	Resonance frequency (GHz)	
	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
Standard deviation	0,0006	0,0026

Density effect





5. *Resonant cavity and microwave results*

➢ **Online moisture *determination* in industry**

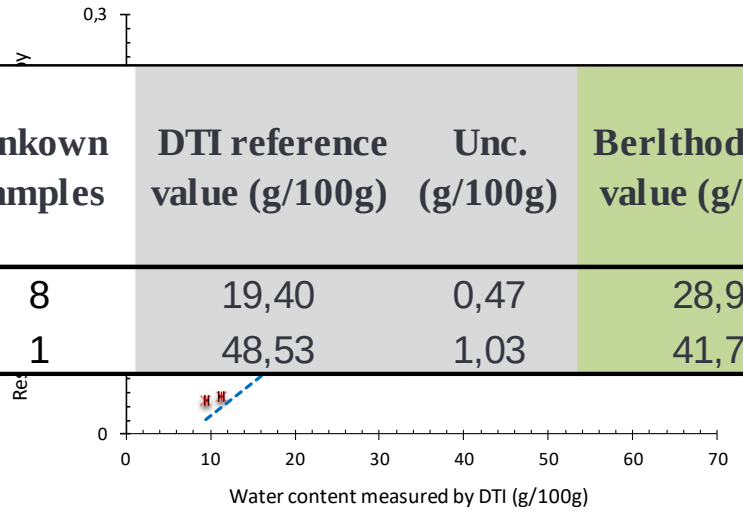
Verdo CHP Berthold X-ray. How to calibrate it?

2 possibilities to calibrate Berthold X-Ray technique

- Using the reference method LoD
- Using the **resonant cavity developped by CETIAT**

Wood chips samples with different humidity levels prepared by DTI

- 2 series of measurements made by CETIAT :
  - 1 at DTI laboratory
  - 1 at Verdo
- Humidity measurements with LoD Reference method by DTI
- Humidity measurements with the Berthold technique of Verdo



Unkown samples	DTI reference value (g/100g)	Unc. (g/100g)	Berlthod given value (g/100g)	Error (Ref. DTI)	CETIAT estimated value (g/100g)	Error (Ref. DTI)
8	19,40	0,47	28,92	49%	20,13	3%
1	48,53	1,03	41,79	14%	47,02	4%

5. *Resonant cavity and microwave results*

➢ **Online moisture *determination* in industry**

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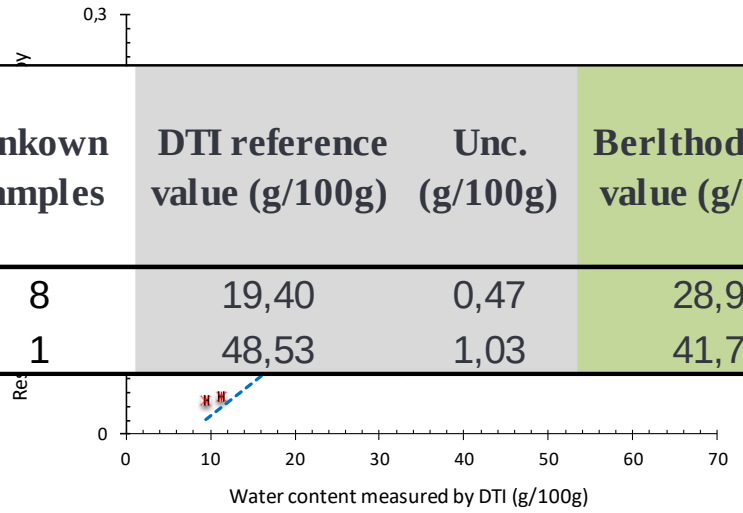

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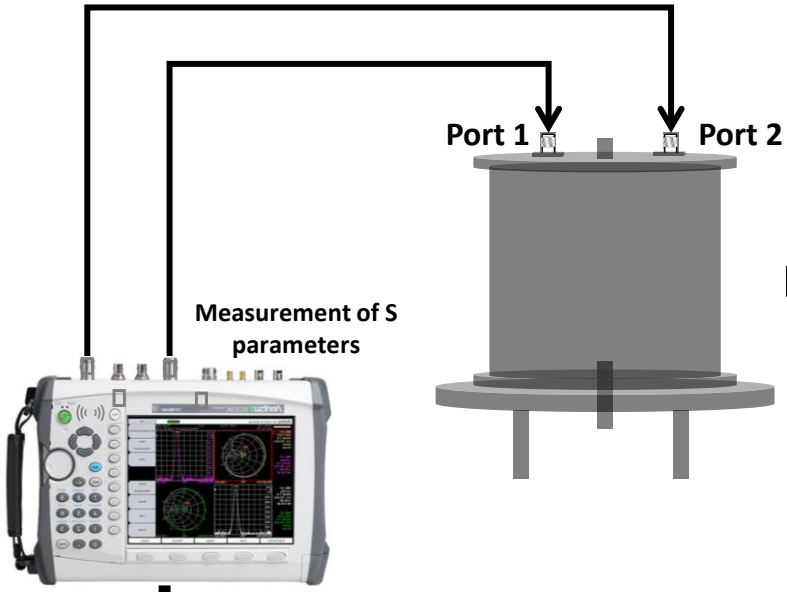
6. Conclusion and perspectives



« VNA calibration» Euramet Guide No 12 - Measurement of S parameters traceable to the SI

Calibrated verification Kit

Measurement model

$$\varepsilon_r' = 1 + C\left(\frac{f_0 - f_s}{f_s}\right)$$

Final measurand ε_r'

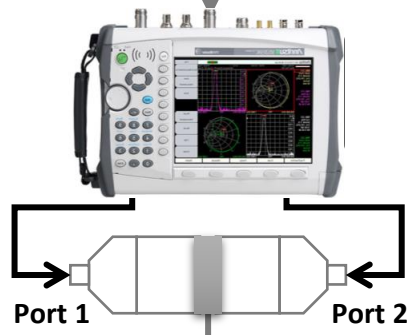
Dimensional aspect

- Cavity volume
- Sample holder volume

Determination of the Cavity constant

Reference Materials with known permittivity

Measurement of permittivity with the coaxial cell developped at CETIAT



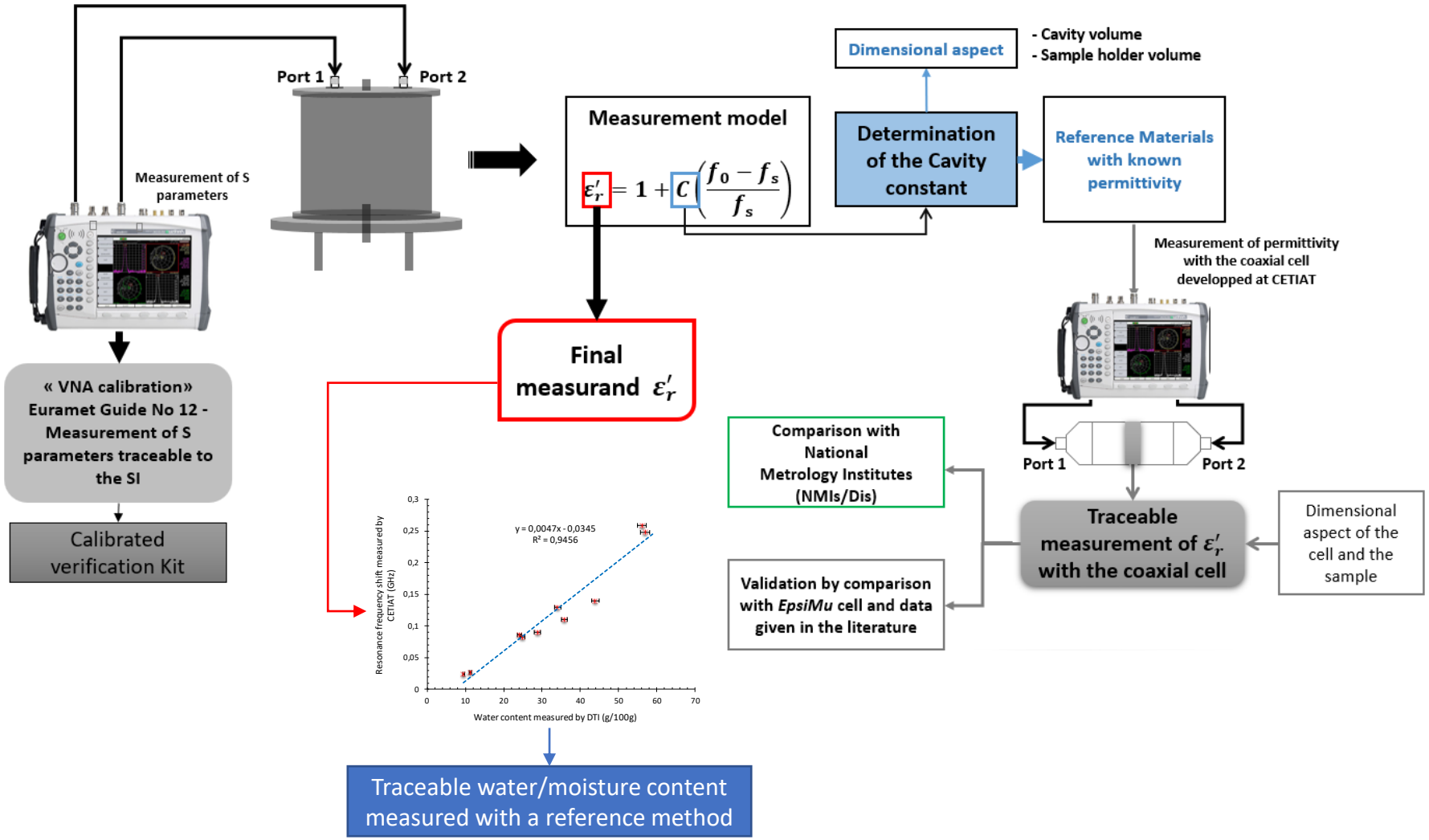
Traceable measurement of ε_r' with the coaxial cell

Dimensional aspect of the cell and the sample

Comparison with National Metrology Institutes (NMIs/Dis)

Validation by comparison with *EpsiMu* cell and data given in the literature

6. Conclusion and perspectives



Thank you for your attention

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France

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