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Coupled dielectric spectroscopy and DSC for ferroelectric materials

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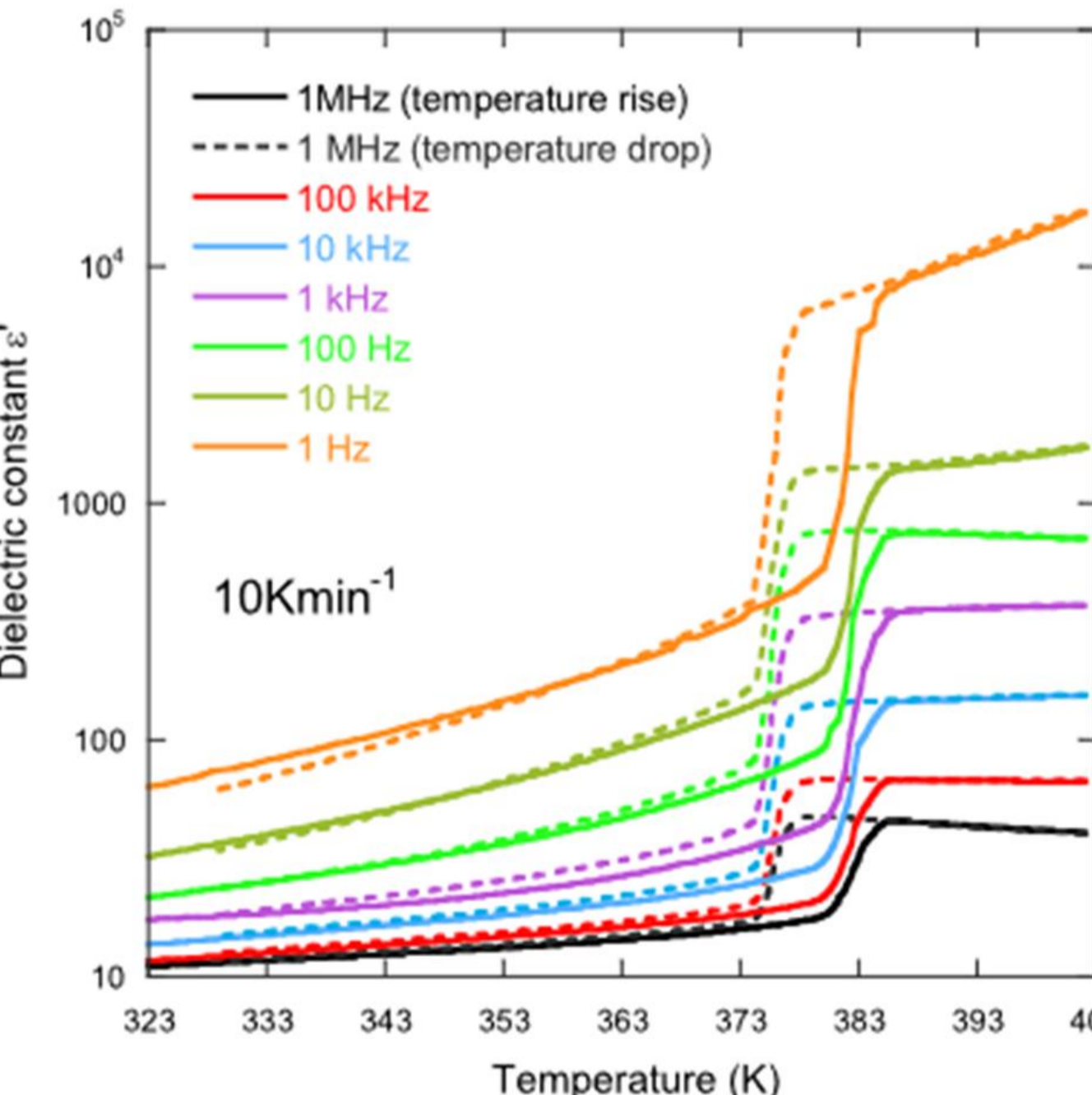
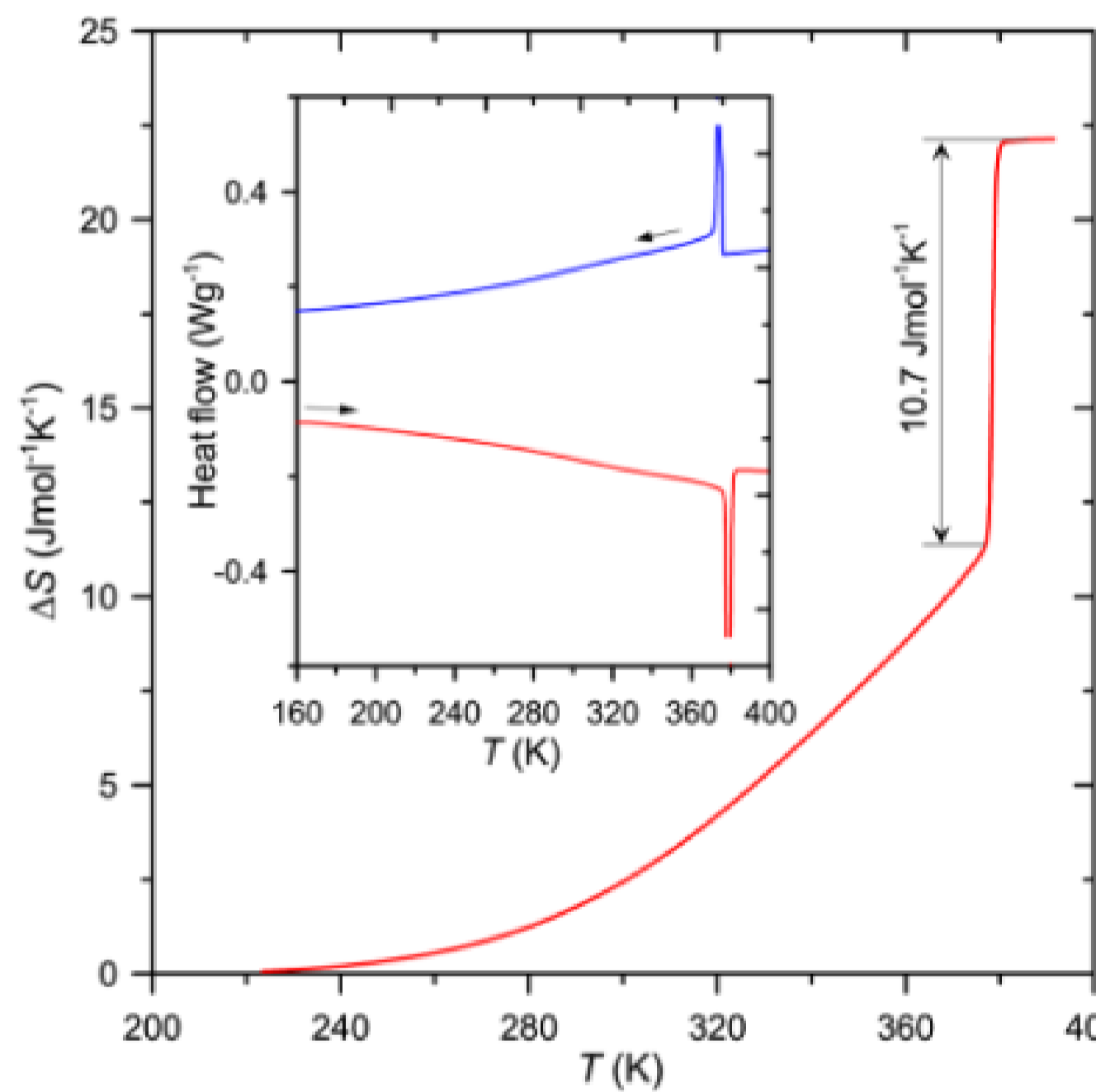
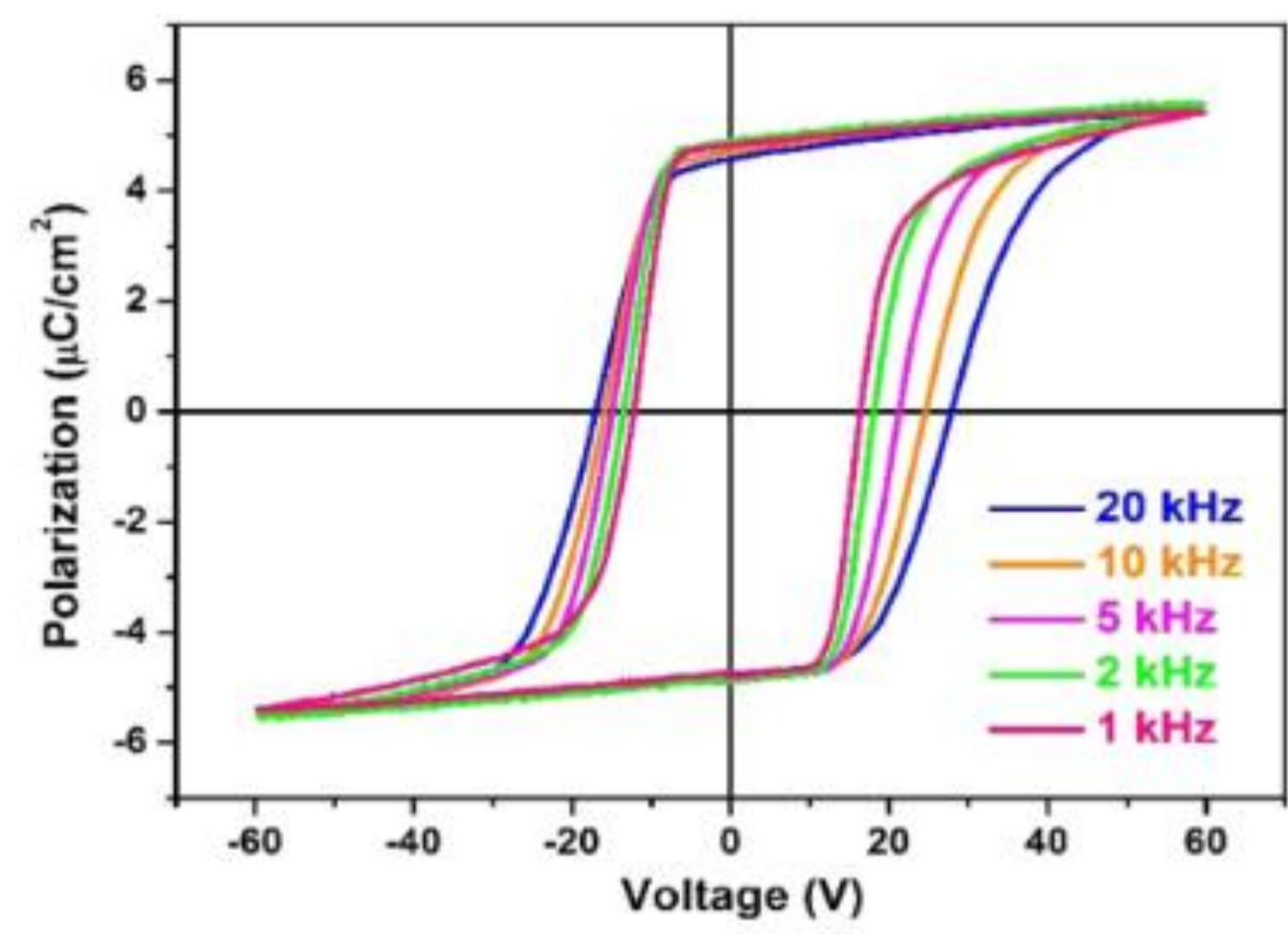
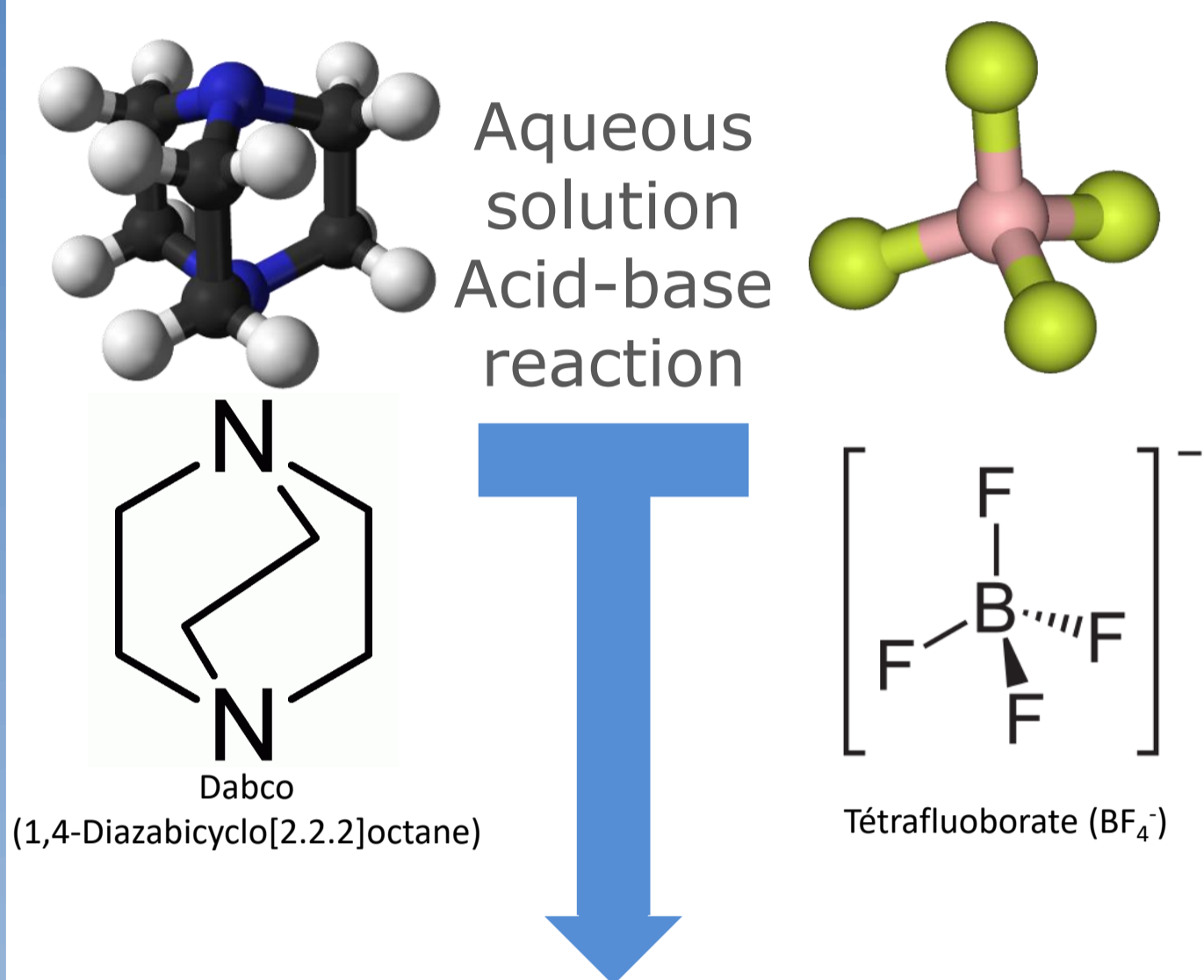
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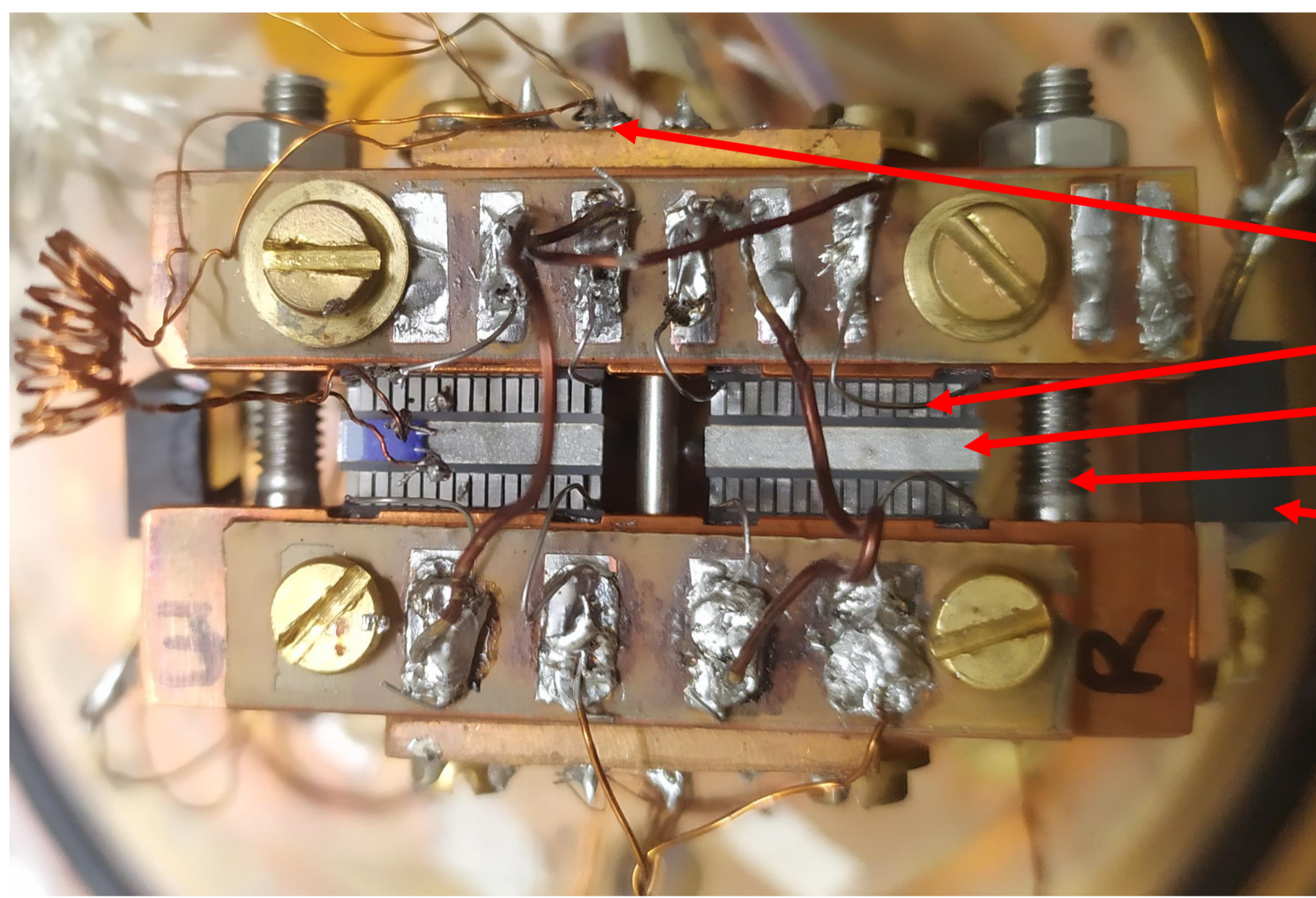
ABSTRACT

We present here a new planar calorimeter coupling dielectric spectroscopy and DSC measurements permitting to link dielectric and thermodynamic behaviors. It is especially suited for sintered ferroelectric pellets or electroactive polymers with transition temperatures from 10 to 140°C and could be applied to thin films with high sensitivity. To show these possibilities, a hybrid molecular ferroelectric material named dabcoHBF₄ is studied and new features are measured in its ferroelectric-paraelectric transition.

Studied material



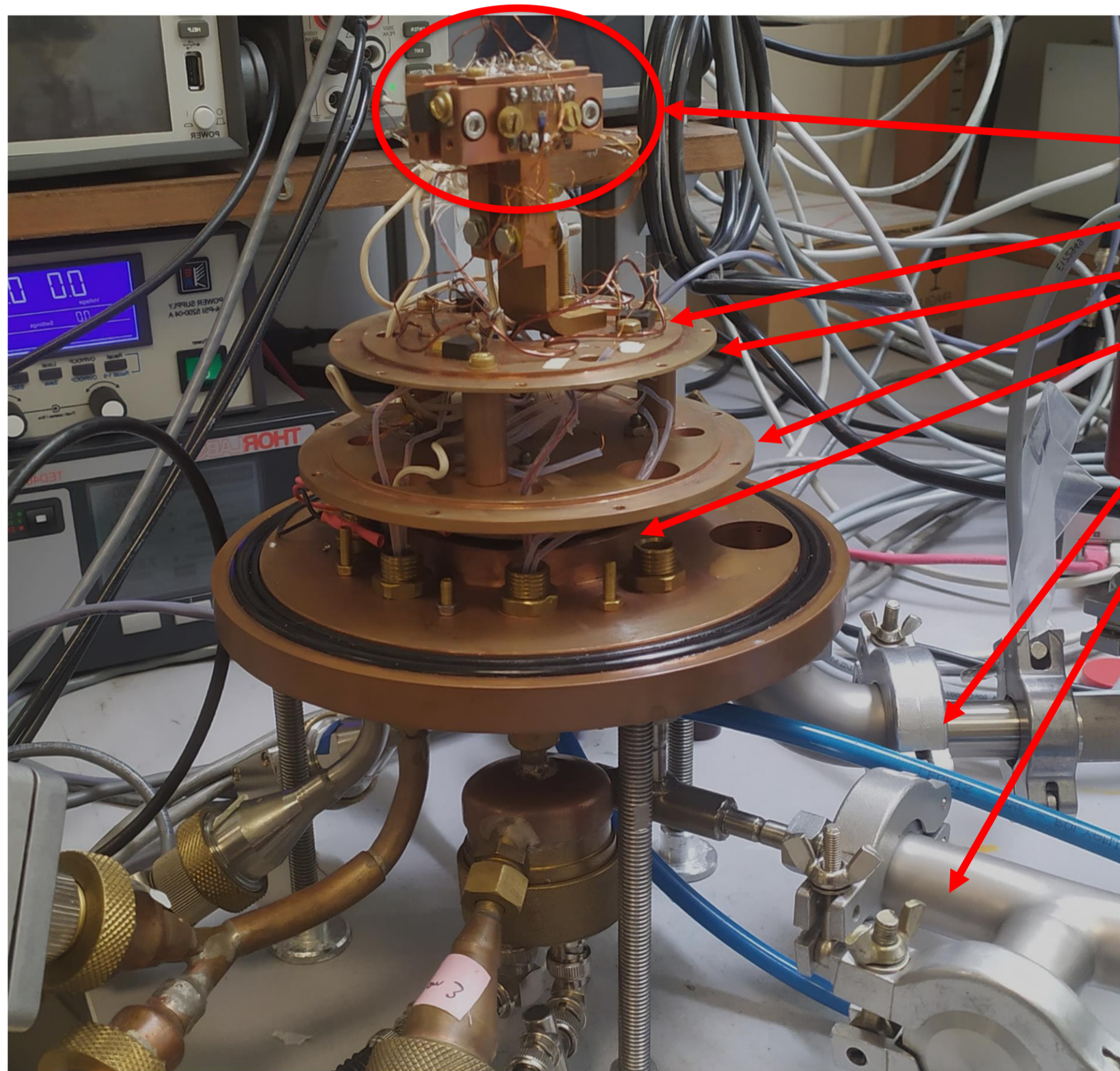
Developped devices



Planar geometry measurement head

- Thermometer Pt1000
- Thermoelectric sensor
- Sample
- Clamping screw
- Heater

- No crucible → lower addenda
- Suited for deposited films, polymer sheets etc.
- Adaptable for electrical measurements



Calorimeter

- Measurement head
- Heaters
- Thermal screens
- Peltier cooler
- Pump
- Controlled pressure gas entry

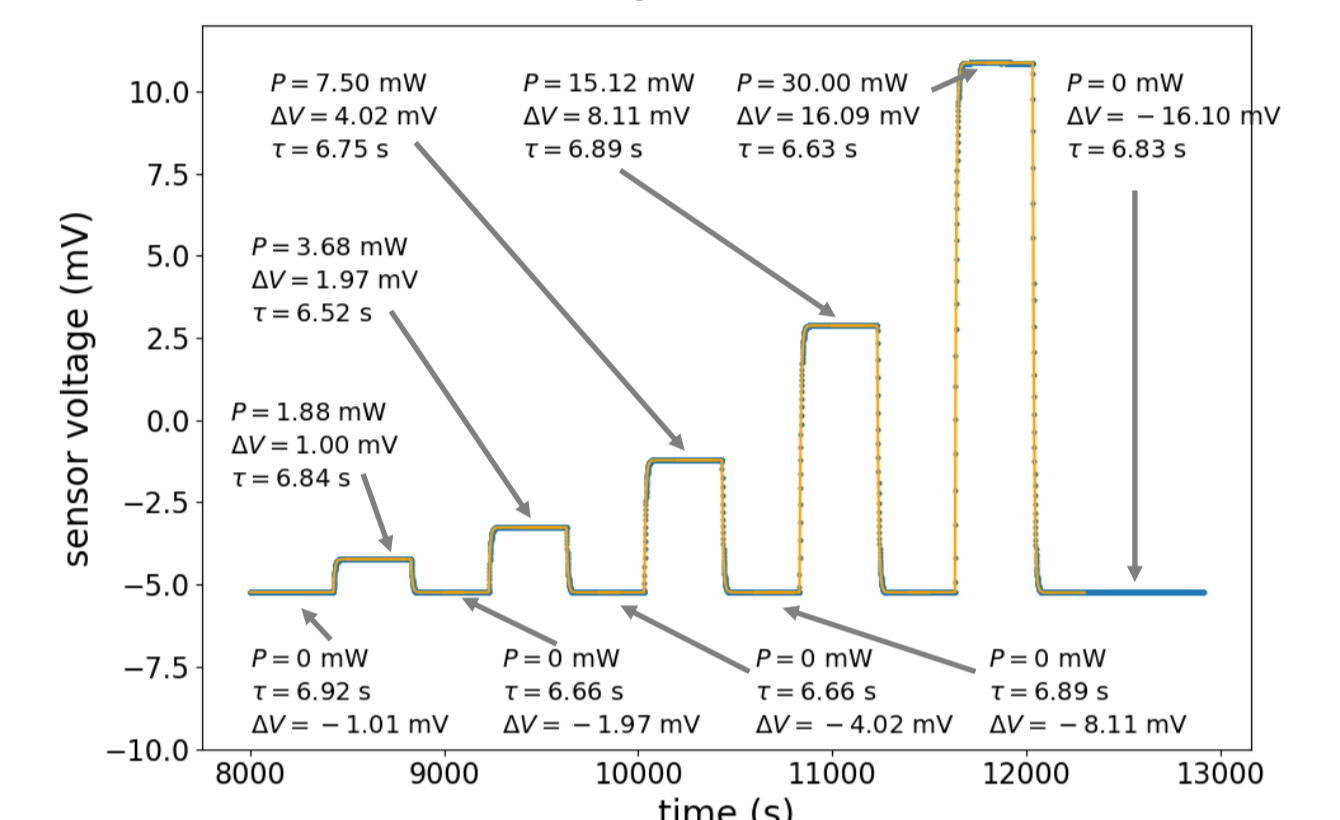
Specifications

- Sensibility ≈ 0,55 V/W
- Time constant = 6s
- Static noise ≈ ±1μW
- κ = 0,20 W/K
- 10 to 150° C
- 0,005 to 3 K/min rate
- Dielectric + DSC measurement
- DC voltage + DSC measurement
- Controlled gas atmosphere

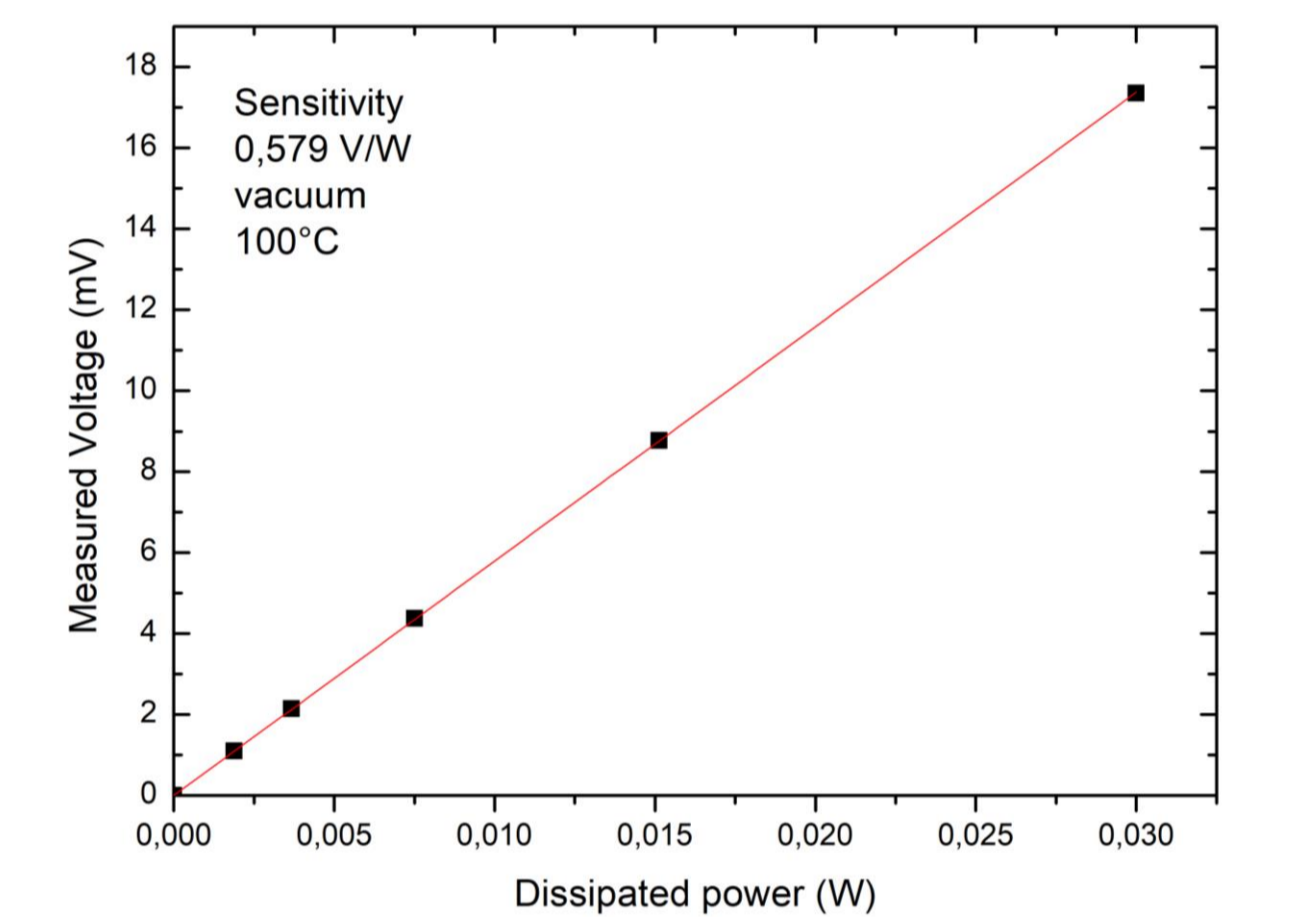
Calibration

Calibration process :

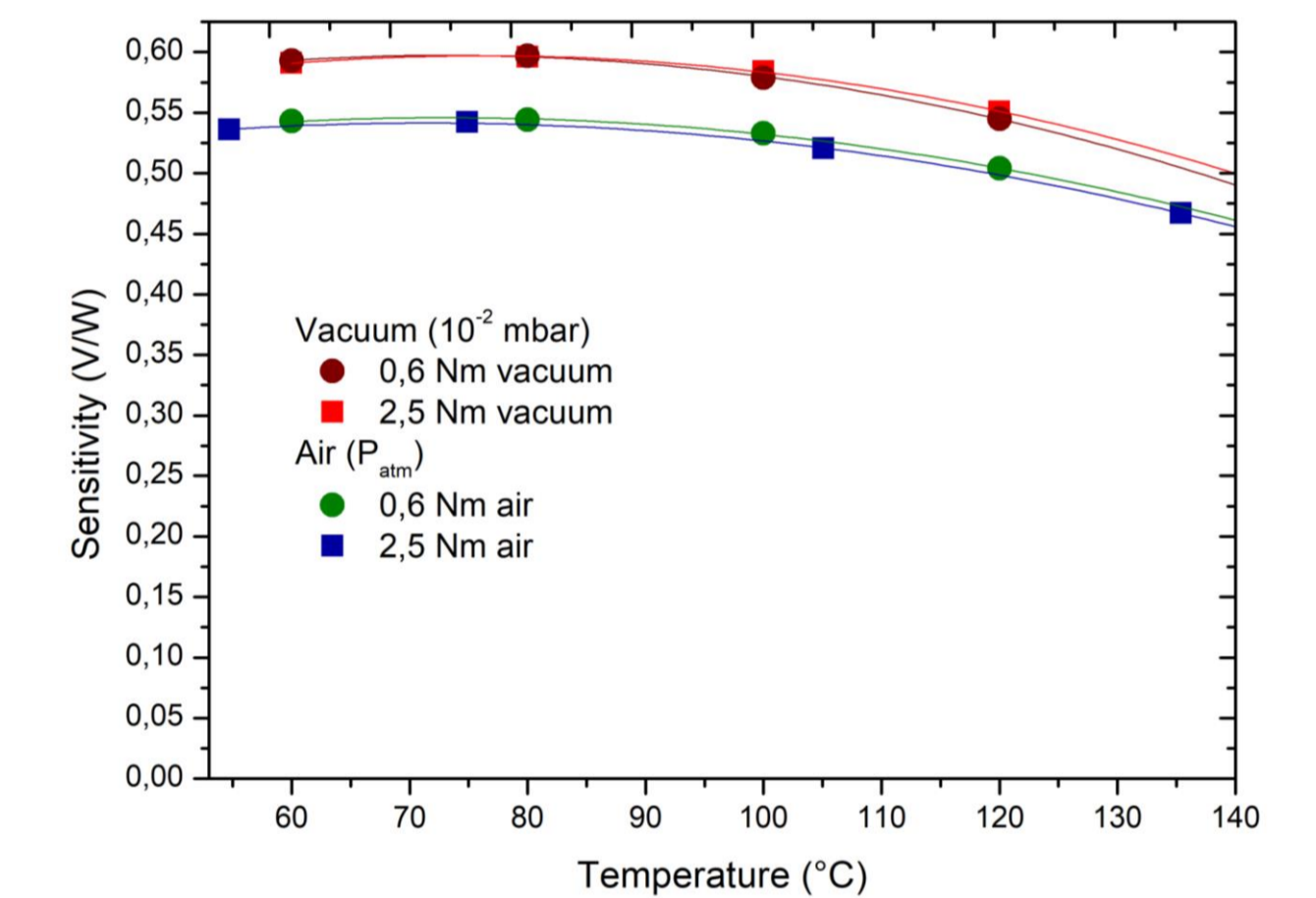
1) Voltage induced by power pulses @ constant Temperature



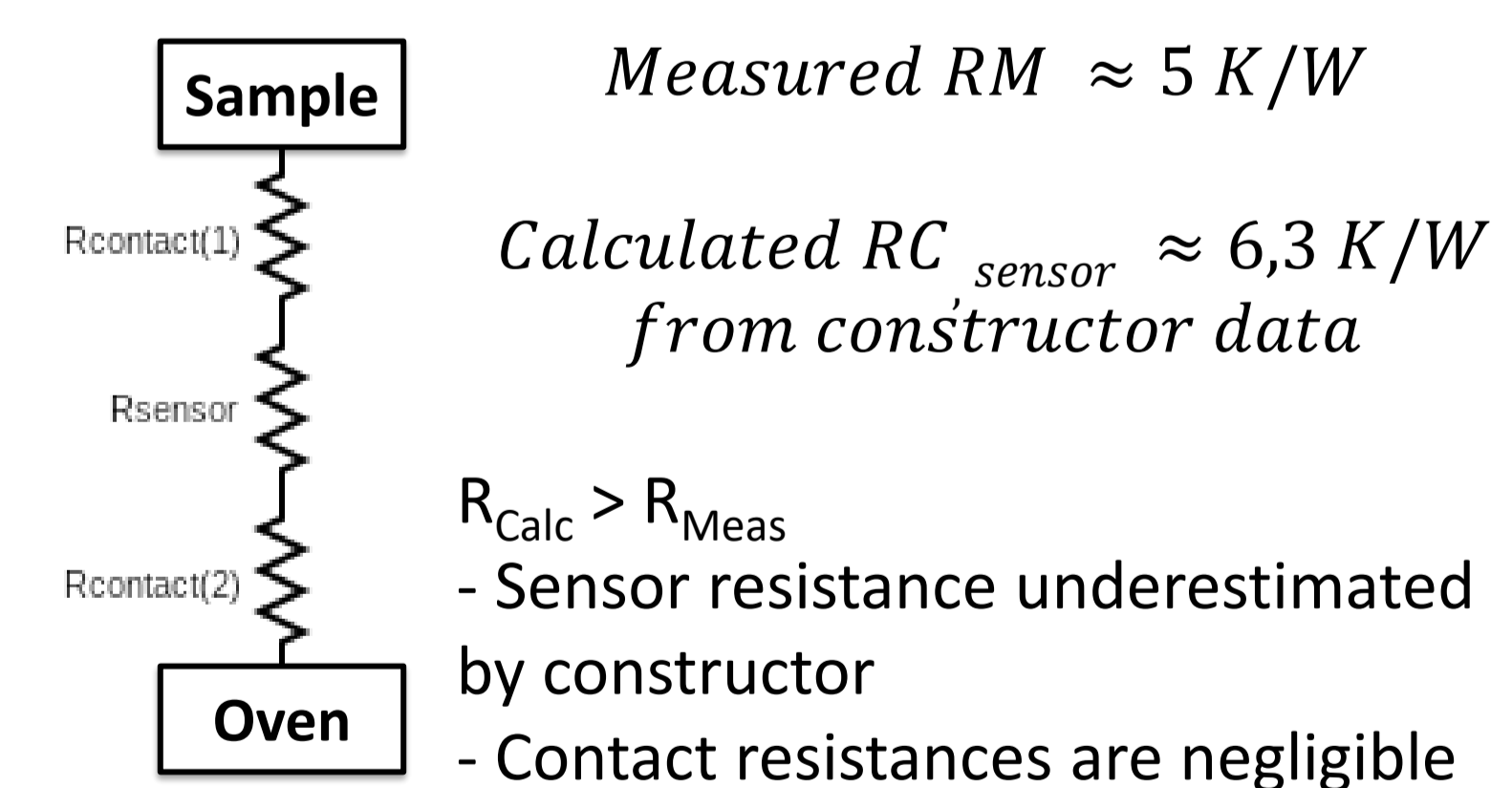
2) Sensitivity S @T_i = slope of U_i vs P



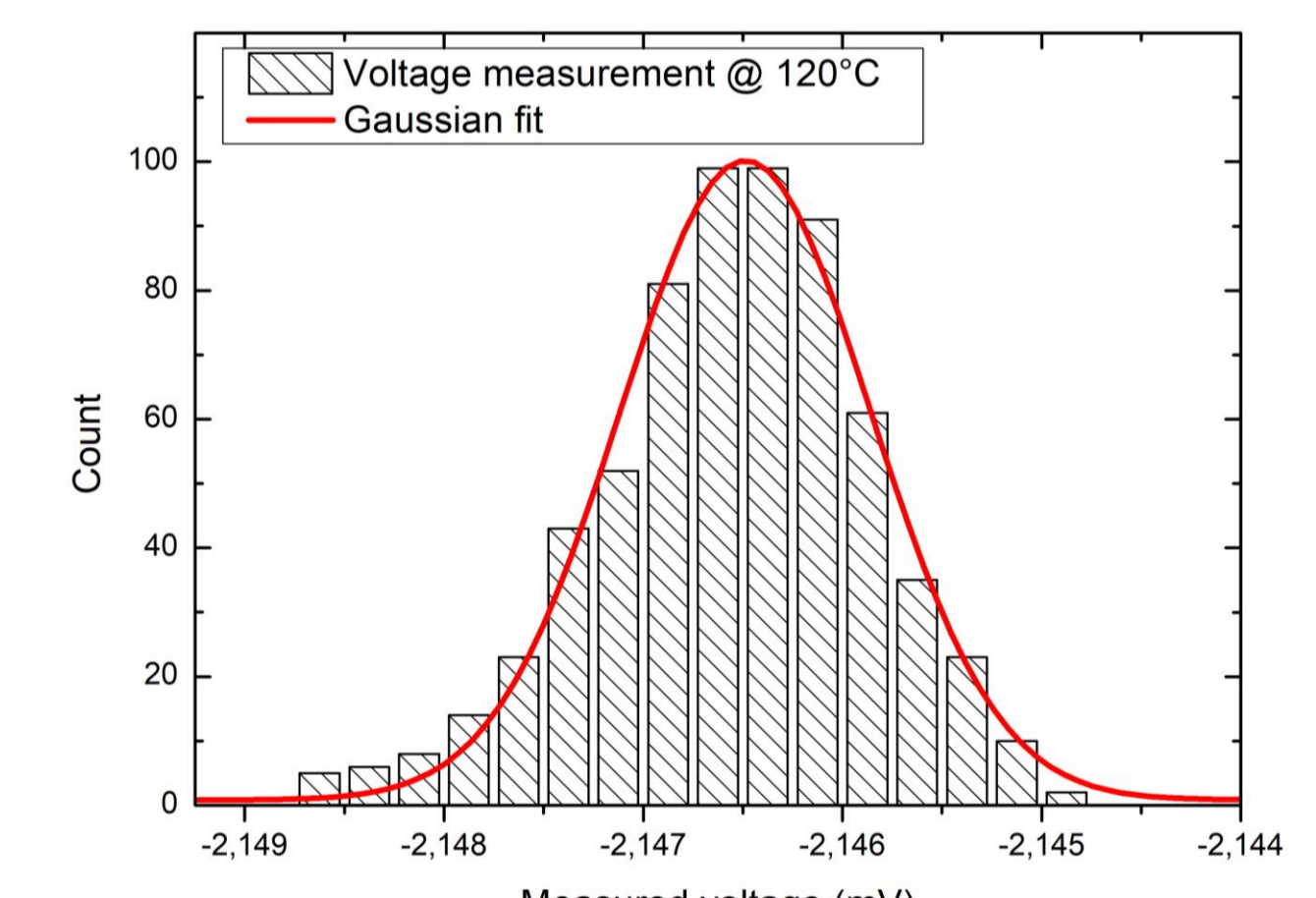
3) Sensitivity S vs T



4) Oven – sample Thermal conductivity Effect of clamping pressure



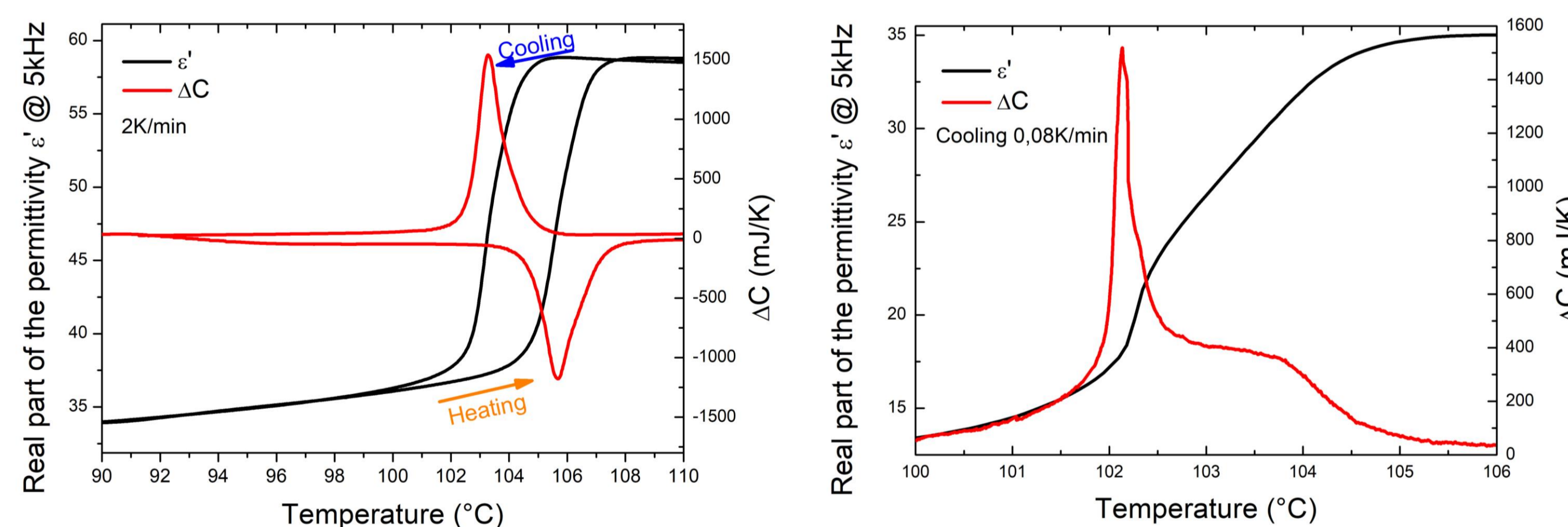
5) Static noise measurement



Noise on measured power : ±1μW

Dielectric-DSC Coupled measurements

Slow scanning rate for phase transition studies



- Dielectric and DSC measurements performed in situ
- New DSC-dielectric correlated structures shown at low heating/cooling rate

Conclusion and perspectives

We successfully developed a new calorimeter based on thermoelectric sensors made for planar samples (such as pellets, thick films or polymer sheets) coupled with dielectric spectroscopy or voltage applications. First, we calibrated it and present its specifications. We used that coupled DSC-dielectric spectroscopy measurement to study a hybrid molecular ferroelectric material named DabcoHBF₄ at low scanning speeds and showed new correlated substructures in the ferroelectric-paraelectric transition.

Now, some of the following steps planned to be made soon to develop that new tool :

- Device optimizations to be made
 - Lower thermal noise
 - Minimize temperature ramps drifts
 - Upgrade cooling power
- New measurements
 - On thin films and polymers
 - With voltage, for electrocaloric effects
 - On liquids and solutions

DabcoHBF₄
 Lead free ferroelectrics
 Aqueous synthesis
 High T_c for organics
 Pr = 4.9 μC/cm²
 Large dielectric constant range

Ref :
 1) P.P. Shi et al. Am. Chem. Soc. 139, 1319–1324 (2018)
 2) A. Olejniczak et al. Crystal Growth & Design, 18, 11, 6488–6496 (2018)
 3) N. Brefuel et al. J. Mat. Science, 56, 18582–18591. (2021)