



# Experimental cell for powder X-ray diffractometer

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**X-ray powder diffraction (XRPD)** – essential technique for development of solid-state materials science – adsorbents, catalysts, electromaterials,...

Most frequent users: NIC (D09, D10, D13), IJS, UL-FKKT, UM, UNG, Lek, Krka, Silkem, Calcit,...

Determination of new crystal structures

Phase analysis – quantification

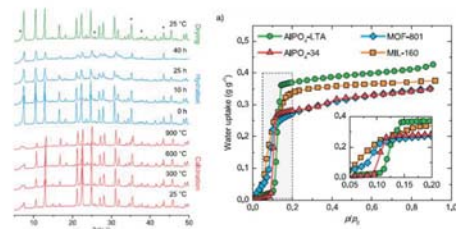
In-situ structure dynamics studies

ADVANCED SCIENCE NEWS  
www.advancesciencenews.com

ADVANCED ENERGY MATERIALS  
www.aem-journal.com

Superior Performance of Microporous Aluminophosphate with LTA Topology in Solar-Energy Storage and Heat Reallocation

Andraž Krajnc, Jure Varlec, Matjaž Mazaj, Alenka Ristič, Nataša Zabukovec Logar,<sup>®</sup> and Gregor Mali<sup>®</sup>

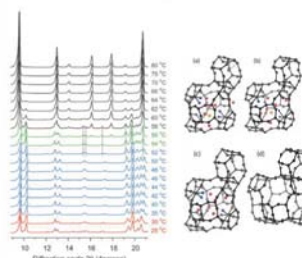


NJC

PAPER

Dehydration of AlPO<sub>4</sub>-34 studied by variable-temperature NMR, XRD and first-principles calculations<sup>†</sup>

Jure Varlec,<sup>1</sup> Andraž Krajnc,<sup>1\*</sup> Matjaž Mazaj,<sup>1</sup> Alenka Ristič,<sup>1</sup> Nataša Zabukovec Logar,<sup>1</sup> Andraž Ovs,<sup>1</sup> Ajo Samsonič,<sup>1</sup> Verica Šušteršič,<sup>1</sup> and Gregor Mali<sup>1,®</sup>



## Current status



**PANalytical X'Pert PRO**  
CuK $\alpha$ 1 – high resolution  
diffractometer  
(2005; D09/FKKT)



**PANalytical X'Pert PRO**  
CuK $\alpha$ 1 – temperature  
programmed high resolution  
diffractometer  
(2008; D09/D10)

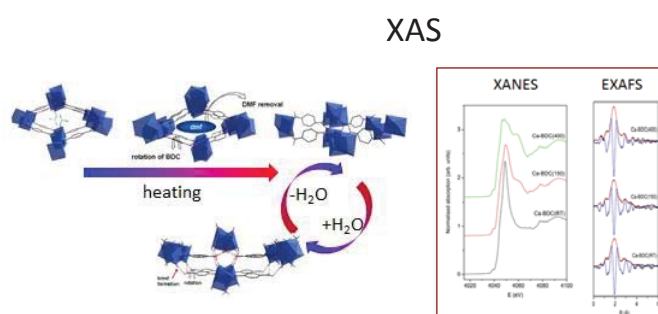
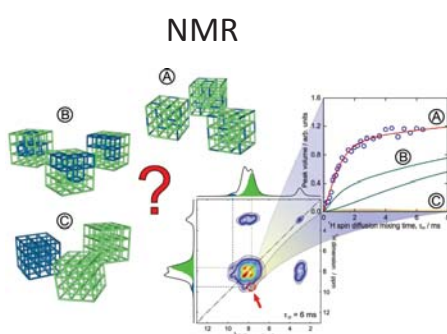


**PANalytical Empyrean**  
Cu, Mo, Ag X-ray source, modular setup, PDF  
analysis  
(2020; D09/D10/D13/FKKT, director's fund )

## Priority goal in materials research field

Understanding of structure functionality on atomic level  
(environmental applications - energy storage/transformation, CO<sub>2</sub>  
capture/conversion,...)

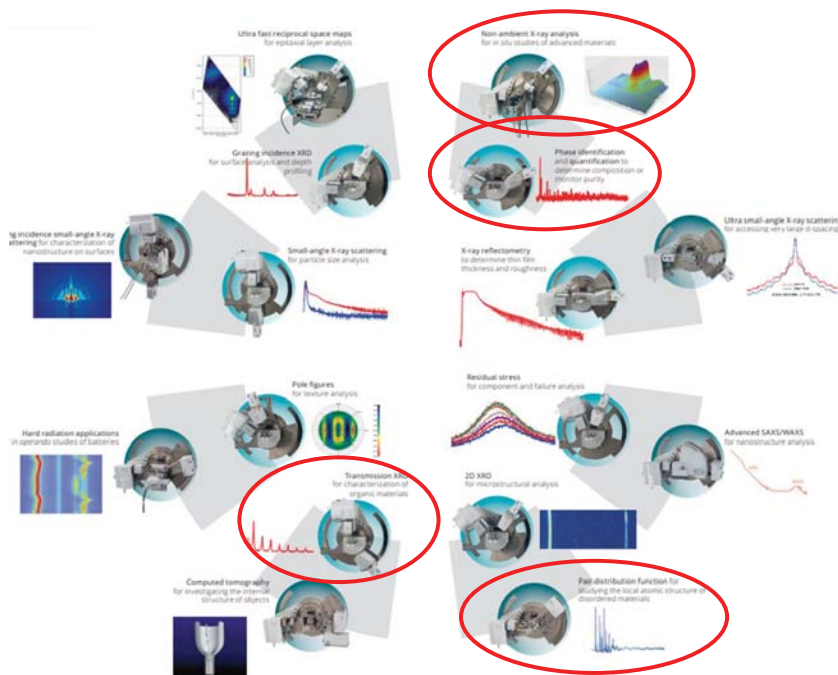
### *In-situ, ex-situ spectroscopic approaches*



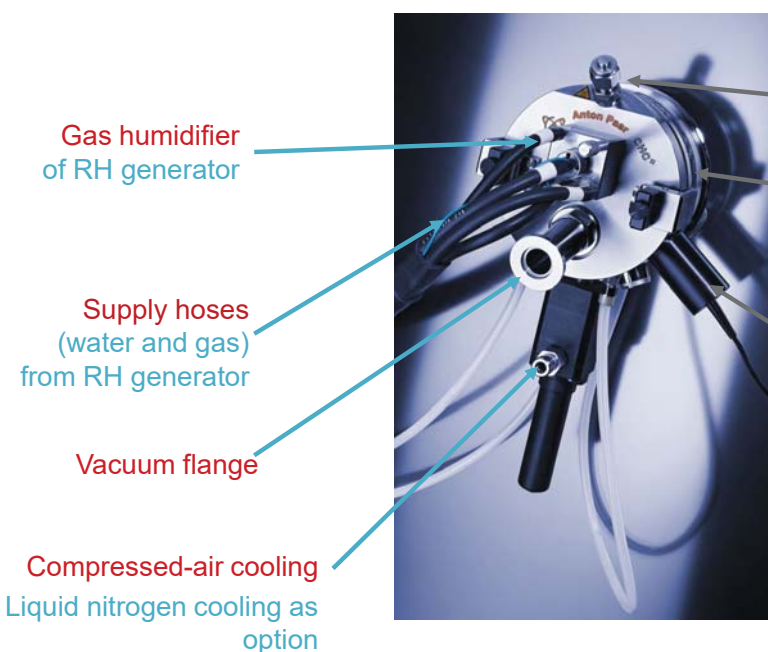


### ...step forward – upgrade scattering/diffraction approaches

PANalytical (Malvern) Empyrean



### CHC plus<sup>+</sup> - Cryo & Humidity Chamber



Connector for reference sensor or gas hose

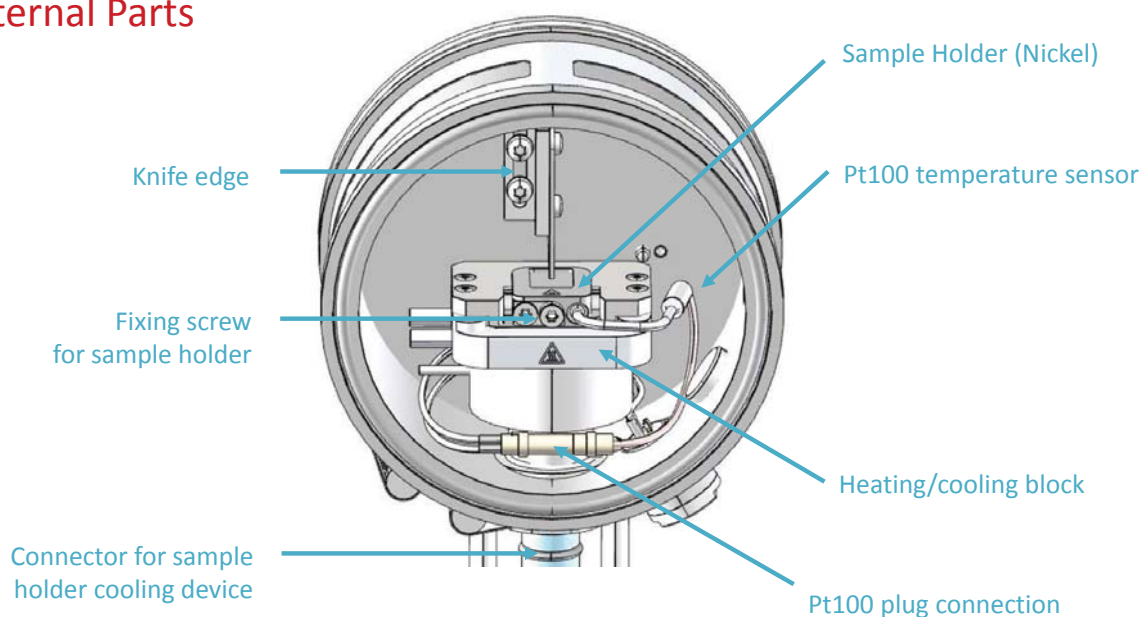
X-ray window Kapton+graphite  
- graphite to avoid condensation  
- Graphite foil easily cracks

Humidity Sensor and gas outlet



## CHC plus<sup>+</sup> - Cryo & Humidity Chamber

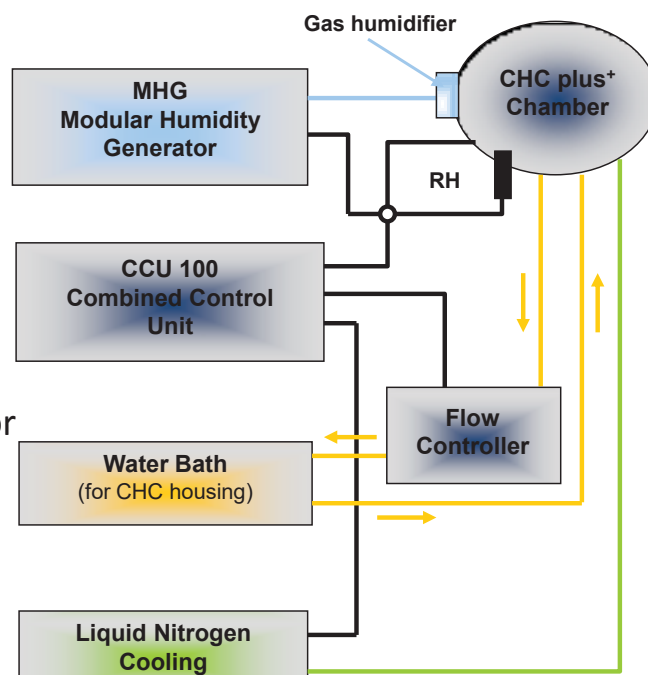
### Internal Parts



## Components for Humidity set-up

### System components:

- CHC plus<sup>+</sup> Cryo & Humidity Chamber with compressed air cooling
- CCU 100 Temperature Controller
- Flow control unit for CHCplus<sup>+</sup>
- MHG Relative Humidity Generator for humidity experiments
- Circulating water bath
- Low Temperature Equipment



Process controller  
*Eurotherm 3504*

Water tank



Supply hoses  
for gas & water

Gas humidifier  
⇒ mounted on *CHCplus lid*

## Operating principle

- two lines of flow-controlled N<sub>2</sub> gas supplied to the humidifier
- water supplied to the humidifier
- one flow of N<sub>2</sub> saturated with water when passing through wet fleece
- Controlled mixing of dry and wet flow provides desired relative humidity

## Cooling Performance

### Liquid Nitrogen Consumption

- LN<sub>2</sub> consumption when permanently at -180 °C: max. 4 L/h
- LN<sub>2</sub> consumption for cyclic heating and cooling: < 4 L/h

### Operating time

For 100 L Dewar vessel:

- max. 25 hours at -180 °C
- > 25 hours for cycling

### CCU 100 Combined Control Unit



## Applications

- XRD studies of crystal structure changes induced by temperature and humidity
- Lattice parameter measurements
- Studies of samples with melting point near or below ambient
- Investigation of polymorphs
- Characterization of pharmaceutical samples, food ingredients and organic materials in general
- Structural studies of building materials

## Application Example *In-situ* XRD study of Trehalose

- Trehalose is a well-known cryoprotectant
  - prevents protein damage during freezing
- Stability was investigated as function of relative humidity and temperature

