MUST HAVE TOOL FOR MATERIAL SCIENCES FOR ENHANCED ANALYTICAL PRODUCTIVITY

CPMAS CryoProbe™ for Material Science

Material science samples often suffer from long experiment times and challenging setups. Quadrupolar and low gamma nuclei are especially demanding of hardware performance, while requiring long spectrometer sessions to achieve adequate signal-to-noise ratios. Two-dimensional spectroscopy, such as MQMAS experiments or 2D homo/hetero-nuclear correlation, proves even more difficult, and often impossible.

The new member of the CPMAS CryoProbe family provides a 3 to 4-fold sensitivity boost, helping you to acquire spectra up to 16x faster compared to regular CPMAS probes. Material science NMR applications which were previously inaccessible by solid-state NMR for reasons of poor sensitivity, particularly for those enquiring low gamma or low natural abundance nuclei are now possible with the help of game-changing enhancement.

Conquering titanium, low-gamma and sensitivity

Titanium is one of the most challenging low-gamma nuclei due to its low natural abundance, relatively large quadrupole moment and low gyromagnetic ratio. Additionally, the two NMR-active isotopes (^{47}Ti/^{49}Ti) are very close in frequency which complicates the spectra through overlapping signals. The new HX CPMAS CryoProbe offers unique possibilities for ^{47}Ti/^{49}Ti NMR spectroscopy, due to the greatly enhanced sensitivity and the larger sample volume of the rotors while still allowing for moderate MAS speeds.

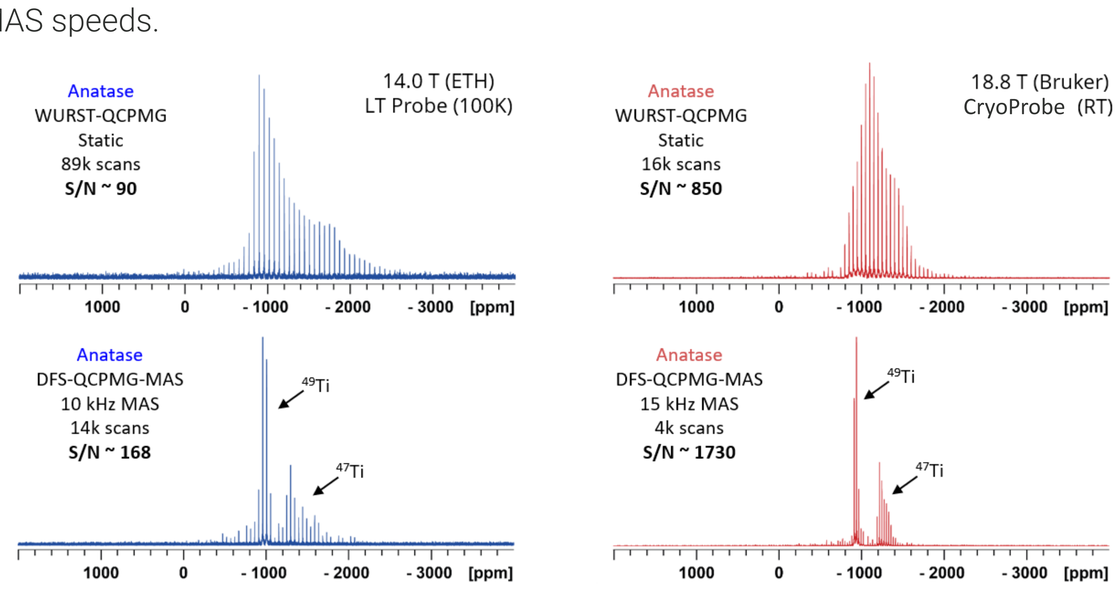
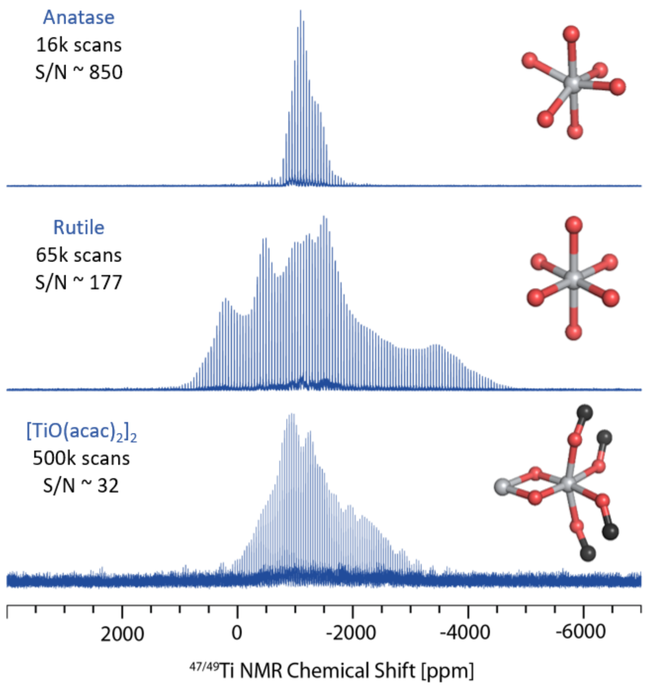


Figure 1: Based on data analysis a signal to noise improvement by more than an order of magnitude was estimated when using the CryoProbe [1].



WURST-QCPMG spectra of TiO_2 – Anatase, Rutile (middle) and the molecular $[TiO(acac)_2]_2$. The insets show the local coordination geometry of Ti and highlight the sensitivity of ^{47}Ti/^{49}Ti NMR spectroscopy towards very subtle changes [1].

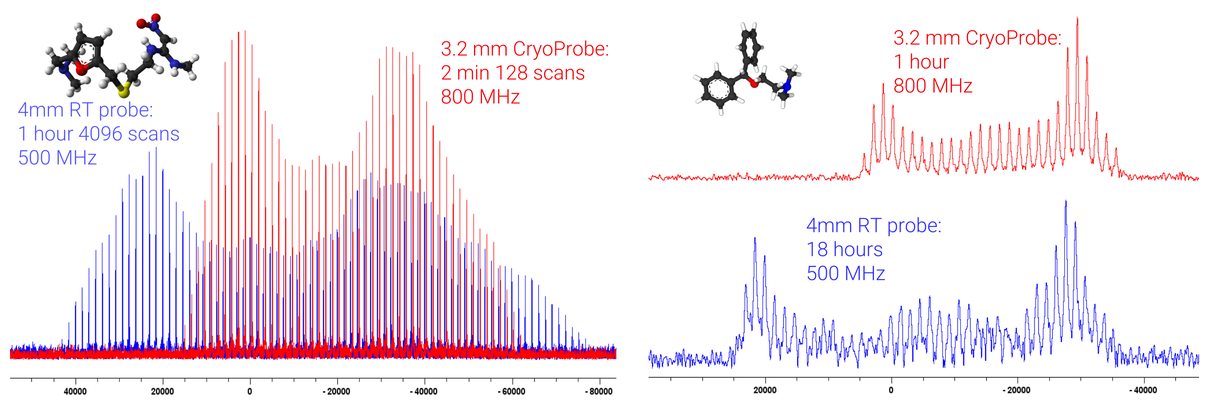
The excellent sensitivity permits the measurement of anatase within minutes, rutile within few hours and even the molecular $[TiO(acac)_2]_2$, with relatively large CQ and low titanium weight loading, was recorded in just 1 day!

[1] L. Lätsch, C. Coopéret, ETH Zurich; manuscript in preparation

Pharmaceuticals: ^{35}Cl wideline QCPMG

More than 50% of APIs are manufactured as HCl salts to stabilize their crystalline forms. The chloride ions in HCl APIs sit in unique environments with intricate hydrogen bonding arrangements reflecting the crystalline molecular structure in its quadrupolar powder pattern, providing a unique spectral fingerprint for each polymorph or pseudo-polymorph containing ^{35}Cl[1]. Since some drug dosage forms contain only a very small amount of the APIs, NMR analysis would require very long acquisition time with conventional NMR probes while the CPMAS CryoProbe enables data acquisition in a reasonable amount of time.

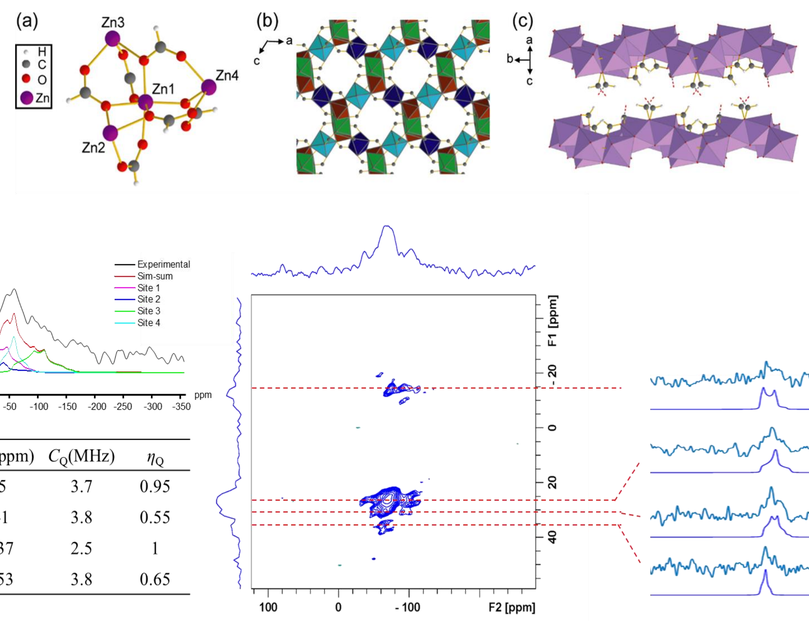
(1) Martine Monette, Solid State NMR | Characterizing Polymorphs and Amorphous Form | Bruker https://www.bruker.com/en/products-and-solutions/mr/nmr-pharma-solutions/solid-state-nmr.html



^{35}Cl WURST-CPMG spectra of a crushed Zantac tablet (left, 150 mg) and Diphenhydramin HCl crushed tablet (right, 25 mg) acquired on a RT probe (blue) and with a CPMAS CryoProbe (red). The time saving with a CryoProbe on a high field instrument is several orders of magnitude.

^{67}Zn-NMR: new tool for characterizing MOF

Yining Huang at University of Western Ontario, Canada, has been very active in the characterization of metal-organic frameworks (MOF). α-Zn-formate or α-$Zn_3(HCOO)_6$, is a microporous MOF that exhibits promising gas adsorption abilities. The low gamma and low natural abundance of ^{67}Zn makes this nucleus extremely challenging with conventional probes. The enhanced sensitivity of CPMAS Cryoprobe allows the acquisition of a 2D MQMAS spectrum. Four ^{67}Zn sites can be resolved and a comparison of the isotropic chemical shift values with those obtained from the DFT calculations allows the assignment of the 4 different signals. To our knowledge, this kind of 2D experiment is not attainable using conventional probes, even at ultra high fields.



	δ_{iso}(ppm)	C_Q(MHz)	η_Q
Site 2	5	3.7	0.95
Site 1	-1	3.8	0.55
Site 4	-37	2.5	1
Site 3	-53	3.8	0.65

Broadband MAS CryoProbe (HX) at 800 MHz

- Enhanced X-channel sensitivities by a factor of >3
- One order of magnitude faster data acquisition and significantly increased productivity
- Solid-state NMR experiments with strong RF fields
- MAS rates up to 20 kHz with variable temperature capabilities
- Standard bore design (compatible to WB)

TECHNOLOGY