Probing the Origin of Unexpected Splitting in ^{31}P CP-MAS of Hydrogen-Bonded Ferroelectrics at 21.1 T

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Introduction

Hydrogen bonded compounds such as RbH_2PO_4 (RDP), $NH_4H_2PO_4$ (ADP) have long been considered as model lattices exhibiting paraelectric-to-ferroelectric transitions. The question of pure order/disorder phase transition or presence of other simultaneous electronic instabilities is still unresolved. One approach is to measure the temperature dependence of the isotropic component of the chemical shift δ_{iso} through the phase transition temperature, Tc [1]. We have been studying these systems and our recent results showcase how a small mis-setting in the magic angle of the probe can substantially influence the results.

Experimental

Single crystals were grown by slow evaporation of an aqueous solution of ADP. The ^{31}P CPMAS was carried out on the ultra-wide bore 900 MHz NMR spectrometer available at the NHMFL on a home built 3.2mm ^{1}H-X broadband VT MAS probe. The rotor was packed with both crystaline ADP and powder $Mg_2P_2O_7$ for observing the change in magic angle with temperature.

Results and Discussion

An initial ^{31}P spectrum of $Mg_2P_2O_7$ was taken as reference along ^{31}P CP MAS spectra of ADP. If a low temperature spectrum of $Mg_2P_2O_7$ did not match with its reference (at room temperature), the magic angle was manually adjusted, ensuring its correctness for all temperatures. **Figure 1** shows the ^{31}P CP MAS spectra of ADP without manual magic angle correction and **Figure 2** shows the ^{31}P CP MAS spectra of ADP with the correction.

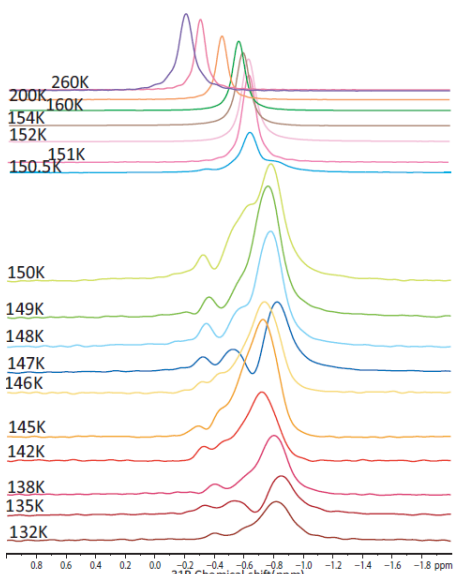
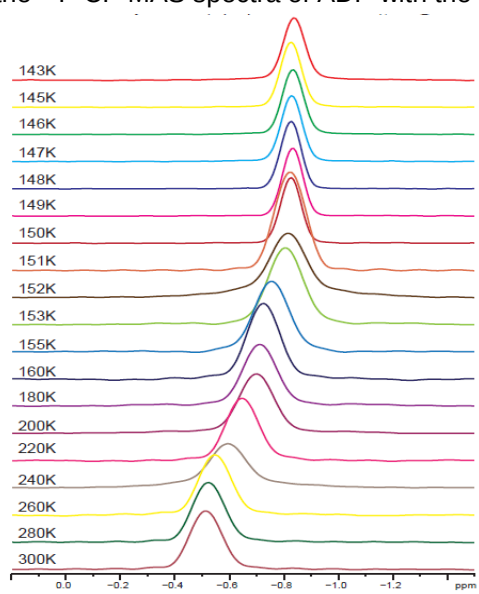


Figure 1: ^{31}P CP-MAS spectra of an ADP



crystal without magic angle correction.**Figure 2**: ^{31}P CP-MAS spectra of an ADP crystal with

Conclusions

While doing low temperature measurements it is clearly crucial to observe any changes in the magic angle. We are now analyzing our samples with a new prospective to better understand the phase transition mechanisms of these model systems.

Acknowledgements

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References

[1] Dalal, N.S.; Bussmann-Holder, A., Ferro-and Antiferroelectricity, Berlin, Heidelberg, Chapter 1 *(2007).*