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Revolution NMR Announces Memorandum of Understanding with Battelle on behalf of the Pacific Northwest National Laboratory for Development of High Pressure NMR

Revolution NMR LLC is pleased to announce the signing of a Memorandum of Understanding (MOU) with Battelle on behalf of the Pacific Northwest National Laboratory (PNNL) for the further development of high pressure Magic Angle Spinning rotors for solid state NMR. Under the terms of the MOU, Revolution plans to support Battelle in preparing a proposal for additional development funding. If Battelle is successful in obtaining the additional funding, Revolution NMR intends to participate in a Cooperative Research and development Agreement (CRADA) with Battelle and will provide components and technical assistance. If the CRADA is executed, Revolution NMR will also have the opportunity to license the CRADA-developed technology for commercialization.
Please contact us for further information. Former PNNL staff member Flaviu Turcu will present a talk on High Pressure MAS NMR in the Hardware and Software Session starting at 4:00 on Thursday. We encourage you to attend.

High Pressure MAS NMR

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Despite its wide spread application, the technique of Magic Angle Spinning (MAS) [1, 2] at pressure exceeding 70 bars has not been possible until recently [3, 4]. Using a pensile type ceramic rotor sleeve, we have developed a new modular design (Figure 1) that can be applied for different rotor diameter sizes. Based on this design, high-pressure MAS rotors that are fully compatible with commercial available probes (6 to 9.5 mm standard MAS probes) have been developed in our laboratory. A high pressure loading/reaction device (Figure 2) for *in situ* sealing and re-opening of the high pressure MAS rotor is also developed.

As illustrated in Figure 1, the new design consists of four key plastic components, i.e., two-components that fit into the top end and another two that fit into the bottom end of a standard ceramic MAS rotor. Using a 7.5 mm alumina-reinforced zirconia ceramic MAS rotor sleeve, a high pressure MAS rotor capable of 300 bar at a sample spinning rate of 3.5 kHz, or 150 bar at 8 kHz spinning rate, was successfully developed. This development sets a new milestone in high pressure MAS NMR. Another distinct advantage of the current design over our already published design (3,4) is the large active sample volume. For example, for the 7.5 mm high pressure MAS rotor, an active sample volume of over 400 μL was obtained.

High-Pressure MAS NMR capability is used for applications like geological carbon sequestration (GCS), sub-surface microbiology and catalytic reactions. As examples of its many possible applications, *in situ* ^{13}C and ^{25}Mg MAS NMR studies of the reaction products and intermediates associated with GCS using different kind of minerals, i.e., brucite ($MgOH$), forsterite (Mg_2SiO_4), reacted with supper critical CO_2 and H_2O at 50 to 70°C are carried out and the results will be reported. A typical result is illustrated in Figure 3, illustrating reaction intermediates, products and the dynamic conversion of the reactants to products can all be obtained in a single time sequence experiment.

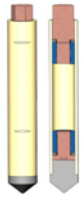

Figure1.

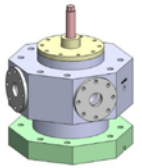

Figure 2.

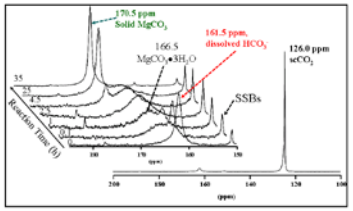

Figure 3. The in situ ^{13}C-SP MAS NMR spectrum of Mg_2OH_4 reacted with 150 bar $scCO_2$ and H_2O as a function of the reaction time. The sample spinning rate was 2.1 kHz.

References:
1. E.R. Andrew, A. Bradbury, R. G. Eades, Nature 182, 1659, (1959).
2. I.J. Lowe, Phys. Rev. Lett. 2 (1959) 285.
3. D.W. Hoyt, J.A. Sears, RVF Turcu, K.M. Rosso, J.Z. Hu, US Patent Office, E-16894, July 2011
4. D.W. Hoyt, RVF Turcu, J.A. Sears, K.M. Rosso, S.D. Burton, A. Felmy, J.Z. Hu., J. Magn. Reson. 212, 378-385, (2011)

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