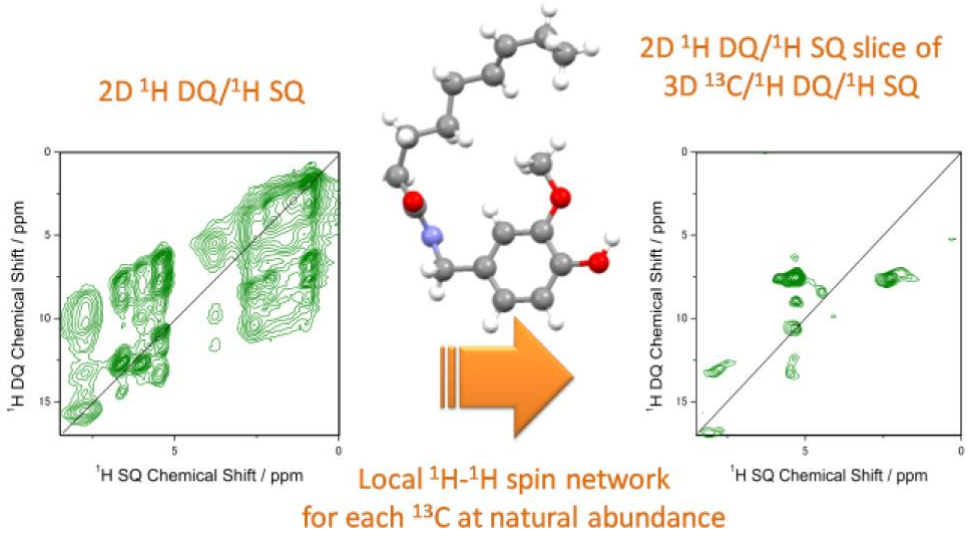
Novel Three-Dimensional Correlation Experiments to Reveal Local Proton Networks under Fast Magic-Angle Spinning at Natural ^{13}C Abundance

Product used : Solid-State Nuclear Magnetic Resonance (NMR)

^{1}H double quantum (DQ)/^{1}H single quantum (SQ) correlation solid-state NMR spectroscopy is widely used to obtain internuclear ^{1}H-^{1}H proximities, especially at fast magic angle spinning (MAS) rate > 60 kHz. However, ^{1}H signals are not well-resolved due to intense ^{1}H-^{1}H homonuclear dipolar interactions even at the attainable maximum MAS frequencies of ~100 kHz to date. We have recently introduced novel three-dimensional (3D) experiments to resolve the ^{1}H DQ/^{1}H SQ correlation peaks using the additional ^{13}C dimension. Although the low natural abundance of ^{13}C (1.1%) significantly reduces the sensitivities, the ^{1}H indirect measurements alleviate this issue and make this experiment possible. The two different implementations of ^{13}C/^{1}H DQ/^{1}H SQ correlations and ^{1}H DQ/^{13}C/^{1}H SQ correlations have been discussed and demonstrated using L-histidine.HCl.H_2O at natural abundance to reveal the local ^{1}H-^{1}H networks nearby each ^{13}C. In addition, the complete ^{1}H resonance assignments have been achieved from a single 3D ^{13}C/^{1}H DQ/^{1}H SQ experiment. We have also demonstrated the applicability of our proposed method on a biologically relevant molecule capsaicin.



The 2D ^{1}H DQ/^{1}H SQ spectrum of capsaicin (left) is too complex due to signal overlaps. On the other hand, the ^{13}C-filtered spectrum (right) yields well-resolved peaks and thus signal assignment is possible. Both spectra were recorded at 70 kHz MAS. Please refer to the original paper for more detail.

Reference: M. Malon, M.K. Pandey and Y. Nishiyama, *J. Phys. Chem. B*, 2017, 121, 8123-8131.

JEOL Ltd. 1-2 Musashino 3-chome Akishima Tokyo 196-8558 Japan Sales Division Tel. +81-3-6262-3560 Fax. +81-3-6262-3577
www.jeol.com ISO 9001 • ISO 14001 Certified

• **AUSTRALIA & NEW ZEALAND** /JEOL(AUSTRALASIA) Pty.Ltd. Suite 1, L2 18 Aquatic Drive - Frenchs Forest NSW 2086 Australia • **BELGIUM** /JEOL (EUROPE) B.V. Planet II, Gebouw B Leuvensesteenweg 542, B-1930 Zaventem Belgium • **BRAZIL** /JEOL Brazil Instrumentos Cientificos Ltda. Av. Jabaquara, 2958 5° andar conjunto 52 ; 04046-500 Sao Paulo, SP Brazil • **CANADA** /JEOL CANADA, INC. 3275 1ere Rue, Local #8 St-Hubert, QC J3Y-8Y6, Canada • **CHINA** /JEOL(BEIJING) CO., LTD. Zhongkeziyuan Building South Tower 2F, Zhongguancun Nansanjie Street No. 6, Haidian District, Beijing, P.R.China • **EGYPT** /JEOL SERVICE BUREAU 3rd Fl. Nile Center Bldg., Nawal Street, Dokki, (Cairo), Egypt • **FRANCE** /JEOL (EUROPE) SAS Espace Claude Monet, 1 Allee de Giverny 78290, Croissy-sur-Seine, France • **GERMANY** /JEOL (GERMANY) GmbH Gute Aenger 30 85356 Freising, Germany • **GREAT BRITAIN & IRELAND** /JEOL (U.K.) LTD. JEOL House, Silver Court, Watchmead, Welwyn Garden City, Herts AL7 1LT, U.K. • **ITALY** /JEOL (ITALIA) S.p.A. Palazzo Pacinotti - Milano 3 City, Via Ludovico il Moro, 6/A 20080 Basiglio(MI) Italy • **KOREA** /JEOL KOREA LTD. Dongwoo Bldg. 7F, 1443, Yangjae Daero, Gangdong-Gu, Seoul, 134-814, Korea • **MALAYSIA** /JEOL(MALAYSIA) SDN.BHD. 508, Block A, Level 5, Kelana Business Center, 97, Jalan SS 7/2, Kelana Jaya, 47301 Petaling Jaya, Selangor, Malaysia • **MEXICO** /JEOL DE MEXICO S.A. DE C.V. Arkansas 11 Piso 2 Colonia Napoles Delegacion Benito Juarez, C.P. 03810 Mexico D.F., Mexico • **RUSSIA** /JEOL (RUS) LLC. Krasnoproletarskaya Street, 16, Bld. 2, 127473, Moscow, Russian Federation • **SCANDINAVIA** /SWEDEN JEOL (Skandinaviska)AB Hammarbacken 6A, Box 716, 191 27 Sollentuna Sweden • **SINGAPORE** /JEOL ASIA PTE.LTD. 2 Corporation Road #01-12 Corporation Place Singapore 618494 • **TAIWAN** /JIE DONG CO., LTD. 7F, 112, Chung Hsiao East Road, Section 1, Taipei, Taiwan 10023 Republic of China • **THE NETHERLANDS** /JEOL (EUROPE) B.V. Lireweg 4, NL-2153 PH Nieuw-Vennep, The Netherlands • **USA** /JEOL USA, INC. 11 Dearborn Road, Peabody, MA 01960, U.S.A.