Proton Decoupled Deuterium NMR using Scout Scan for Lock

Application Note

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While it is common in NMR spectroscopy of liquids to use a deuterium lock, there are times when this may not be convenient, for example when working with completely protonated solvents, such as with LC-NMR or analysis of biofluids.

Agilent pioneered the use of a technique called Scout for use with samples that don't contain deuterium. In these applications, a Scout Scan uses a single transient, low tipangle pulse to determine the frequencies of the unsuppressed solvent signals. These frequencies are then used, on-the-fly, to compensate for magnet drift or shift changes that occur from use of an LC solvent gradient. (These same frequencies can also be used to calculate the shaped pulses necessary for fully automatic multi-frequency solvent suppression.)

Another application where the Scout Scan technique is particularly useful is for unlocked direct observation of deuterium NMR spectra with proton decoupling. The only difference is that, in this case, the Scout Scan is done using a proton spectrum, while the actual data is acquired on deuterium. (In the case of LC-NMR, both the Scout and the final spectra are on protons.) One might therefore say that this heteronuclear Scout technique is using a proton lock, as the proton signal of the solvent is used to track and compensate for magnetic field drift over very long data acquisitions. Since deuterium is a low gamma nucleus and is therefore much less sensitive to field drift relative to protons, one only needs to do a Scout Scan every 512 transients or so, to provide adequate compensation for magnetic field drift. Agilent has patented the Scout Scan technique for these purposes.

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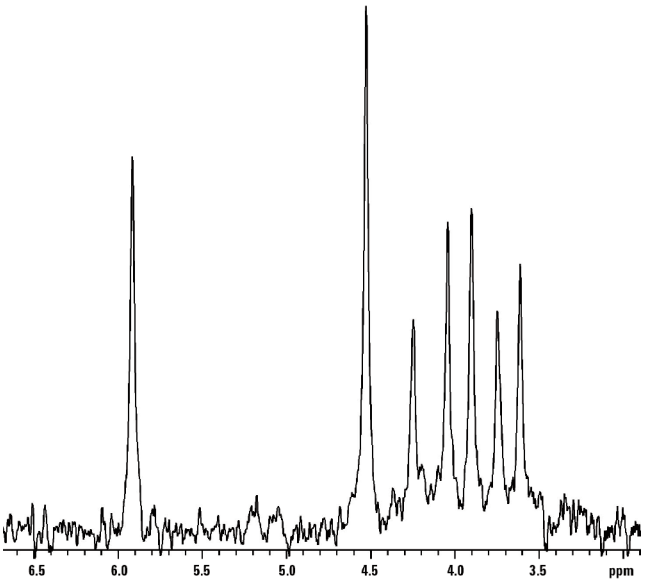

Figure 1. An expansion of a deuterium NMR spectrum of monoacetone glucose.

Figure 1 is an expansion of a deuterium NMR spectrum of monoacetone glucose, a derivative of glucose that is locked into a single conformation. This simplifies the spectrum considerably. The glucose was isolated from 20 mL of human blood, taken 90 minutes after ingestion of enough D_2O to raise the body water enrichment to 0.5%. The total acquisition time was 15 hours, with blocks of 512 deuterium transients collected between proton Scout scans. The data was provided by Shawn C. Burgess, Brian Weis, and Matthew E. Merritt of the University of Texas Southwestern Medical Center. (The work was sponsored by NIH RR 02584).

While it is often believed that deuterium NMR with proton decoupling requires a special NMR console and probe with ^{19}F lock, it is certainly not true. Deuterium NMR using the scout technique for lock can be easily done with any Agilent console and virtually any probe. For many reasons, this is preferable to adding ^{19}F lock capability to the probe and console; furthermore, the use of Scout for lock eliminates the need to add a ^{19}F-containing compound to the sample.

Publications using this setup for deuterium NMR include:

S. C. Burgess, F. M. Jeffrey, C. Storey, A. Milde, N. Hausler, M. E. Merritt, H. Mulder, C. Holm, A. D. Sherry, C. R. Malloy, Effect of murine strain on metabolic pathways of glucose production after brief or prolonged fasting. American Journal of Physiology - Endocrinology & Metabolism. **289**(1):E53-61, 2005 Jul.

S. C. Burgess, N. Hausler, M. Merritt, F. M. Jeffrey, C. Storey, A. Milde, S Koshy, J. Lindner, M. A. Magnuson, C. R. Malloy, A. D. Sherry, Impaired tricarboxylic acid cycle activity in mouse livers lacking cytosolic phosphoenolpyruvate carboxykinase. Journal of Biological Chemistry. **279**(47):48941-9, 2004 Nov 19.

B. C. Weis, D. Margolis, S. C. Burgess, M. E. Merritt, H. Wise, A. D. Sherry, C. R. Malloy, Glucose production pathways by ^{2}H and ^{13}C NMR in patients with HIV-associated lipoatrophy. Magnetic Resonance in Medicine. **51**(4): 649-54, 2004 Apr.

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