O-20

Session 5b) Novel Methodology 2: Technical Development

A new paradigm for fast single-session ^{2}H and ^{13}C IRM NMR

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The main nuclei studied in irm-NMR are ^{2}H and ^{13}C. But to date, there is no NMR equipment able to perform both ^{2}H and ^{13}C isotope analysis. This requires either two spectrometers or frequent probe changes for laboratories working with these two nuclei. This results in either the cost of a second NMR spectrometer or the risk of damage to the equipment and the substantial time required to change the probe. That is why QUAD SYSTEMS created a probe that can measure both ^{2}H and ^{13}C. However, it has its challenges: (i) for ^{13}C, the lock is usually ^{2}H, not ^{19}F as in this case, (ii) for ^{2}H, the tube diameter is much smaller than for the other ^{2}H probes (10 mm versus 5 mm here), which will drastically increase the experimental time to get the same signal to noise ratio. The main question is, can we do ^{2}H and ^{13}C isotope NMR with this probe in a reasonable amount of time? To answer this question, we had to develop a method to decrease the experimental time. We combined the DEFT pulse sequence with the R^{2}D^{2} method (Recovery time Reduction to Decrease experimental time Duration) that we recently developed [1].

With this approach, called R^{2}D^{3}, we were able to record an ethanol ^{2}H spectrum with a trueness and precision under 1% in only 33 min. This reduces experimental time by a factor of 7.2 compared to a regular experiment. Moreover, if the D/H ratios are corrected using a reference spectrum, a factor of 8.4 can be obtained. For ^{13}C, a precision of less than 1‰ was obtained on ethanol, while reducing the experimental time by a factor of 12. However, in this case, the trueness had also to be corrected. Therefore, with R2D3, this 5 mm probe can perform ^{2}H irm-NMR as fast as a 10 mm one, while also being able to perform ^{13}C experiments. The efficiency of this method was evaluated in discriminating between ethanol samples originating from different botanical sources.

In summary, using a dedicated probe and an optimized acquisition method, we were able to perform PSIA ^{2}H and ^{13}C without changing the probe, spectrometer or sample. Isotope profiling of vanillin and the use of a common reference for ^{2}H and ^{13}C are currently under investigation.

References

[1] Sanchez, M., Pontabry, J., Assemat, G., Martinez, A., and Akoka, S. (2024) Recovery time reduction to decrease experimental duration (R^{2}D^{2}): A simple and universal method to accelerate NMR experiments. Talanta 276, 126157.