Poster TP-29
Presented at 14th CPSA, Oct. 3-6, 2011
Sheraton Bucks County, Langhorne, PA

A simple and effective tube construct for salty samples in cryoprobes

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Abstract

High ionic (usually salt) concentration seriously reduces the efficiency of cryoprobes (cold probes) degrading sensitivity and extending the 90 degree pulse width, and good quality tune/match can be difficult. There are various solutions which have been proposed, some are quite sophisticated using special tubes (of oval shape, for example – "Bruker" solution) or double smaller concentric, side-by-side inserts ("Agilent" solution). A "poor man's" choice can be simply taking a tube with smaller OD than that of the probe, introducing an air gap and increasing the distance between the body of the sample and the coil itself.

We describe a simple and inexpensive refined version of this latter approach which also utilizes the concept of separating lock and shim. It is most useful for (biological) samples in water but can be beneficial for all samples of high ionic strength. A relatively large OD (typically 4.1 mm) tube is used to carry the sample while the lock solvent is between the walls of the 5mm OD container tube and the insert. Other size combinations are also possible, of course. This tube construct largely resolves the above issues, retains the highest sensitivity, and makes it possible to avoid any dilution of the sample itself, next to additional smaller benefits.

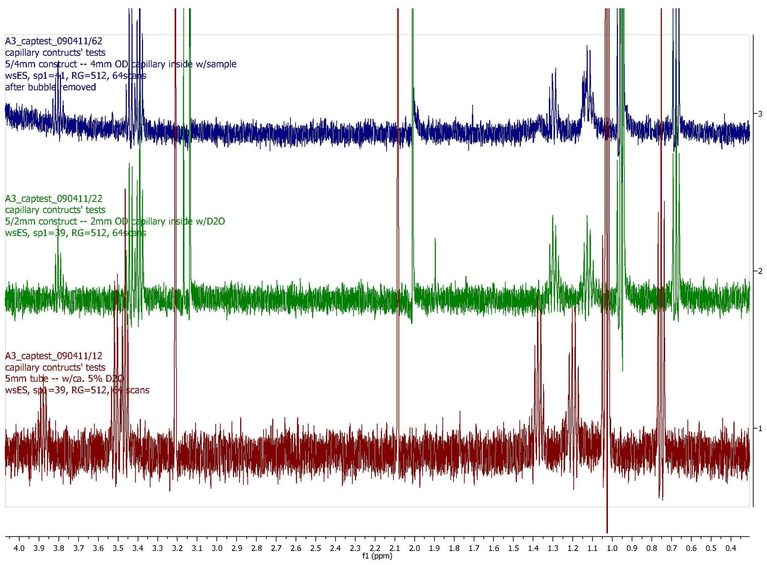


Figure 3.
Results of data acquisition using excitation sculpting on an 500 MHz Bruker Avance-III NMR spectrometer, equipped with a cryo-QNP probehead. The received gain was set to 512 for each acquisition. The detected signals belong to bacterial contamination of the water.

Experimental

Sample Preparation

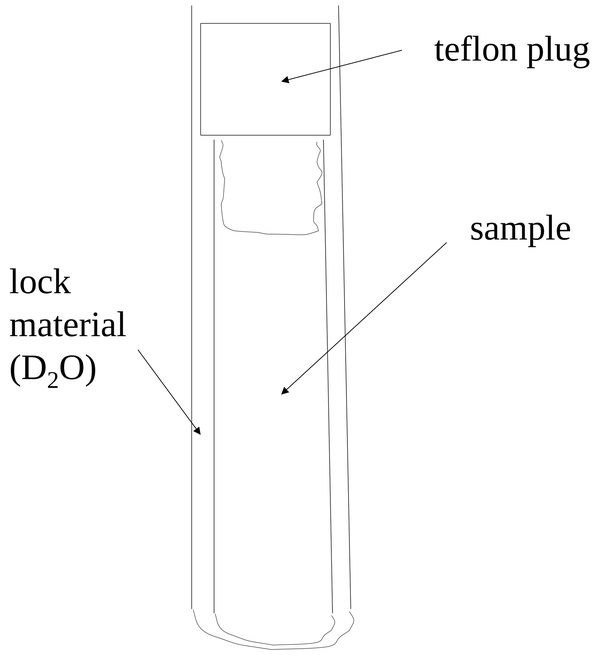
The sample is an artificial high-salt (0.5M NaCl in water) sample split between three tube constructs. One is a conventional 5 mm OD tube with ca. 5% D_2O added (**A**). The second has D_2O in a 2 mm OD insert (**B**), while the third one is the new design with a 4.1 mm OD insert carrying the sample while a small amount of D_2O is between the walls of the two tubes (**C**).

NMR data acquisition

The spectra were collected on Bruker Avance-III, 500 MHz NMR instruments equipped with cryoprobe, with manual activation of the tuning protocol and adjusting the reflection to the best possible condition (see Figure 2.). The optimum 90 degree pulse was calibrated on each sample individually as listed in Table 1. For water suppression the excitation sculpting pulse sequence was applied [1]. The best receiver gain settings, as well as the suitable power settings for the selective pulse for each condition are listed in Table 1. The first three rows are calibrated values for a cryo-QNP (H/P,C,N) probe (default 90 degree pulse is 14.00 us, while SP1 is 42 for non-salty samples), the lower three rows are calibrated values for a TCI (H/C/N) cryoprobe (default values for 90 degree pulse and SP1 are 7.20 us and 43 dB, respectively). The maximum available receiver gain setting is 2050. All spectra were collected at 295K.

References

[1] Hwang, T. and A.J. Shaka. **1994**. Water Supression That Works. Excitation Sculpting Using Arbitrary Waveforms and Pulsed Field Gradients. *J. Magn. Reson.*, 112, 275-279.



	PW90	RG	SP1
A	18.35	912	39
B	19.27	1150	39
C	16.63	1290	41
A	16.05	2050	38
B	17.14	1820	37
C	12.24	2050	40

Table 1.

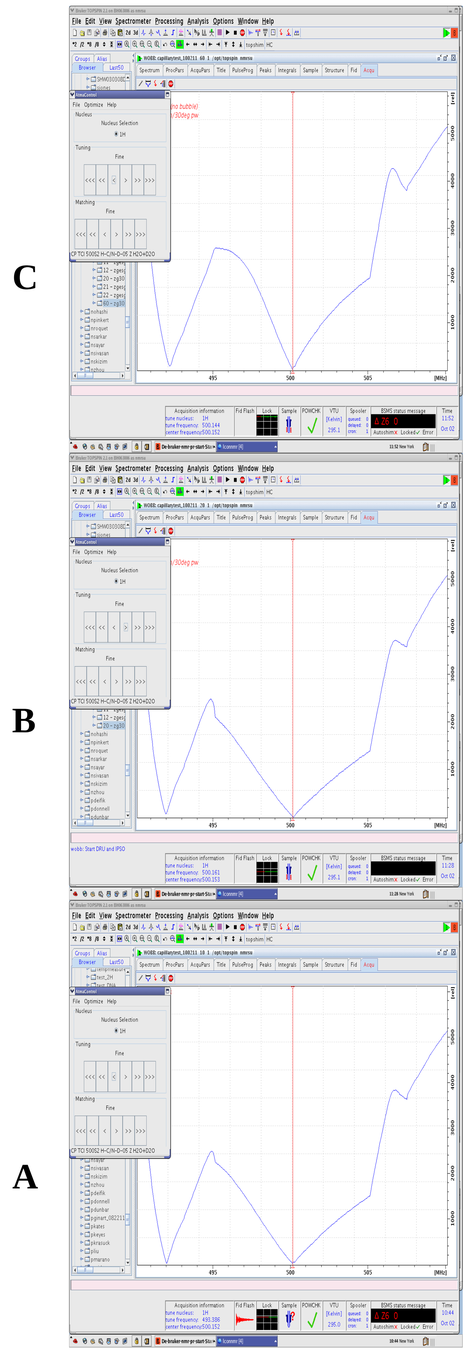


Figure 2.

Conclusion

The simple tube construct with 4.1 mm OD insert introduces better tuning conditions, reduces the actual 90 deg. pulse width, and leads to improved sensitivity in cryoprobes thanks to the insulating effect of the pure lock material between the salty sample and the coil.

This inexpensive tube construct is not only a simple alternative to the more sophisticated, in the same time more complicated and more expensive solutions, but also offers all the advantages of separating lock and shim and allows using the original samples in their native condition.

Acknowledgements

Kind, much forthcoming, and tireless assistance of Frank Bosco (New Era Enterprises, Inc., Vineland, NJ) with glassware, capillaries, and inserts is greatly appreciated.
Elizabeth Zeitler (Frick Chemistry Laboratory) has kindly prepared all the samples for the above tests.