16. X-Nucleus Decoupling on Athena (AC-300a)

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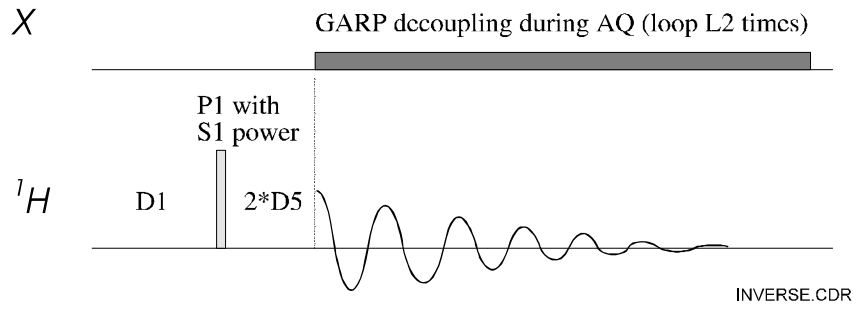
I. Discussion

There may often be times when X-nucleus coupling interferes with assignments in ^1H spectra, or where selective or broadband decoupling of the X-nucleus would help with ^1H assignments, especially for ^1H-X correlation. Broadband and selective decoupling of X-nuclei (specifically ^{31}P and ^{19}F—^{13}C is also feasible, but not likely except for an enriched compound) can now be performed on Athena (AC-300a). Decoupling an X-nucleus while observing ^1H is often referred to in NMR literature as an "inverse" or "indirect" experiment. "Inverse" refers to the opposite sense of the experiment: normally one decouples ^1H and observes X. Here the inverse is performed. "Indirect" specifically refers to performing ^1H-X heterocorrelation experiments (HETCOR, or XHCORR type), where ^1H coupled modulations are observed through the X nucleus. The indirect experiment instead observed X coupled modulations on the ^1H; observing ^1H gives ~10 better sensitivity.

Athena can do selective 1D ^1H-X correlation spectroscopy: e.g., selectively decouple on ^{31}P, and observe which ^1H is affected.

II. Critical Parameters—Broadband Decoupling

INVH1G.AU



O1 – center of X-nucleus spectrum

O2 – center of ^1H spectrum (use O1 from jobfile for normal ^1H for your solvent)

P* – X-nucleus pulses (lots of them!!); if experiment doesn't seem to be decoupling well, use INVP90DE.AU to calibrate X-nucleus 90° pulse width, and see NMR Director

S1 – = 0H

D1 – = repetition delay

D5 – = AQ/2 ***don't change if using normal ^1H SW***

L2 – number of GARP loops ***don't change if using normal ^1H SW***

III. Acquisition of Inverse Spectra

A. Initial Measurements

- take an X-nucleus spectrum as normal
- write down frequency for center of nuclei of interest (use EP, center arrow, enter **O1** and write down value shown)

B. Hardware Changes for Inverse Experiments

1. switch Pneumatic unit from COMPUTER CONTROL to nucleus to be decoupled.
2. switch mode plug inside right panel (looking from front) from NORMAL to INVERSE.
3. attach unlabeled/red-ring BNC cable (normally attached at TRANSMITTER position on preamp box) directly to BB BNC cable on probe using a straight-through BNC connector
4. move yellow BNC cable labeled DECOUPLER from DECOUPLER IN position to TRANSMITTER position on preamp box
5. insert 5 dB attenuation to EXTERNAL ATTENUATOR BNC connection on X-nucleus transmitter (located at far-right—looking from behind—in upper panel on back of console

C. Software Setup for Broadband Decoupling

- for ^{31}P decoupling, enter **RJ INVP31.1DJ**
  RC INVP31.CON

  for ^{19}F decoupling, enter **RJ INVF19.1DJ**
- change **O1** to value noted above as center frequency for X-nuclei wanted to decouple
- change **O2** to O1 value used for ^{1}H jobfile for solvent used (e.g., O1 from ACETONE.1DJ)
- enter **AU INVH1G.AU**

D. Switching Hardware Back to Normal Detection

All steps must be correctly done, or normal detection will not work!!!

1. switch Pneumatic unit back to COMPUTER CONTROL
2. switch mode plug back to NORMAL
3. move yellow BNC cable labeled DECOUPLER back to DECOUPLER IN position on preamp box
4. re-attach unlabeled/red-ring BNC cable to TRANSMITTER position on preamp box, and probe BB BNC cable back to X-nucleus filter box
5. remove 5 dB attenuation from EXTERNAL ATTENUATOR BNC connection; make sure small BNC is connected through both side of EXTERNAL ATTENUATOR connection.