Agilent 400 MHz Automated Triple Broadband Probe

Versatility and performance

Technical Overview

Introduction

Advantage statement

The Agilent 400 MHz Automated Triple Broadband (ATB) probe is always tuned to three nuclei and eliminates the need to be retuned when samples in different solvents are introduced into the probe. This makes the probe stand out as the perfect choice for automation and hands-off environments that typically observe ^{1}H, ^{19}F and one X nuclei, use different solvents, and require consistently high quality data from sample to sample without moving any mechanical parts.

The ATB probe is the most versatile probe offered at 400 MHz which allows access to all of the features available on the Agilent 400-MR. It is simultaneously tuned to ^{1}H and ^{19}F on the outer coil, and one variable X nuclei (^{31}P to ^{15}N) on the inner coil (four nuclei if one includes lock). It can be used for any combination of single and double-resonance experiments on the 400-MR, and triple-resonance experiments on a 400 MHz Agilent NMR System. Throughput time is reduced as time needed to tune the probe due to solvent changes are eliminated, and mechanical movements are reduced to when the X nuclei is changed.

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Experimental

The following examples of data from phentermine hydrochloride dissolved in two common NMR solvents, deuterated chloroform ($CDCl_3$) and deuterated methanol (CD_3OD), serve as a clear illustration of the probe's tuning design. The data in Figure 1 show that there is effectively no change in the ^{13}C pulse width when different solvents are used.

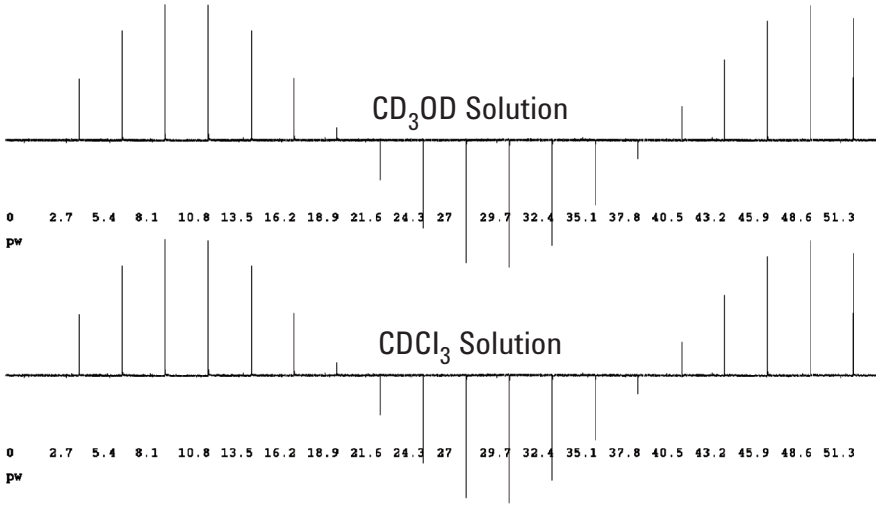

Figure 1. A ^{13}C pulse array using 2.7 µs steps starting at 0 µs collected in CD_3OD (top) $CDCl_3$ (bottom). The 360 degree pulse is identical for both solutions, 38.7 µs. No probe tuning was done between samples, and identical parameters were used to collect the data.

The key to obtaining identical ^{13}C (or other X nuclei) pulse widths, as in Figure 1, is to set the probe tuning to the high side of acetone, as that setting will be midway of CD_3OD and $CDCl_3$. Therefore, the amplitude of RF reflection is nearly identical when switching from CD_3OD to $CDCl_3$ solutions, as shown in Figure 2, which results in the identical pulse width arrays of Figure 1.

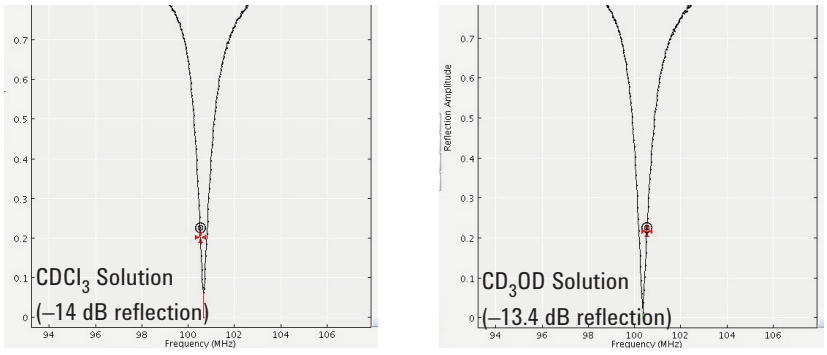

Figure 2. The tune response of the X coil when different solutions with same concentration of phentermine hydrochloride are placed in the probe.

Additionally, the $^{1}H/^{19}F$ coil of the probe is very insensitive to solvent changes and X-nuclei changes, virtually eliminating the requirement to retune the $^{1}H/^{19}F$ coil while obtaining state-of-the-art indirect detection datasets under complete automation. To illustrate this, Figure 3 shows data from the gradient Heteronuclear Single Quantum Coherence (HSQC) experiment acquired with identical acquisition and processing parameters on the same set of samples. In each case, the sample was submitted under automation and no probe tuning was done.

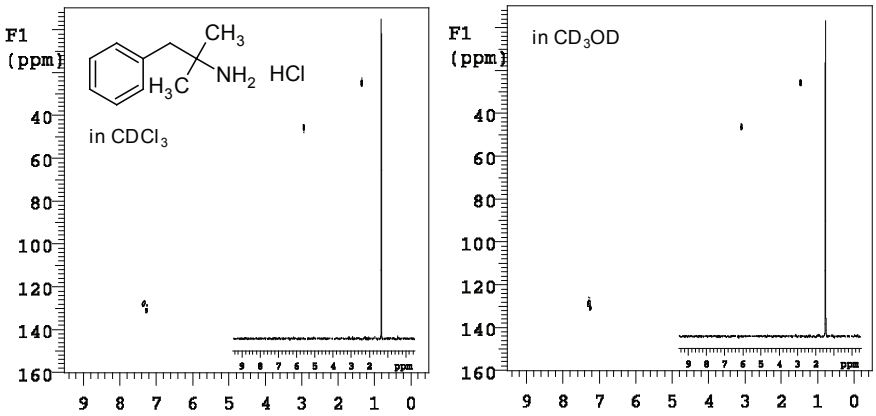

Figure 3. Gradient HSQC data for different solutions but same concentration of phentermine hydrochloride without any probe retuning. Insets are a trace of the methyl resonance showing comparable sensitivity.

Results and Discussion

The simultaneously tuned $^{1}H/^{19}F$ coil sets this probe above other probes as it can be used for double resonance $^{1}H\{^{19}F\}$ and $^{19}F\{^{1}H\}$ experiments on the 400-MR, and triple-resonance $X\{^{1}H/^{19}F\}$ (or any of these combinations) experiments on a 400 MHz Agilent NMR System. Double-resonance $^{19}F\{^{1}H\}$ capability is illustrated by the Heteronuclear Overhauser Enhancement Spectroscopy (HOESY) data in Figure 4, which provides information on the spatial relationship of heteronuclear spins.

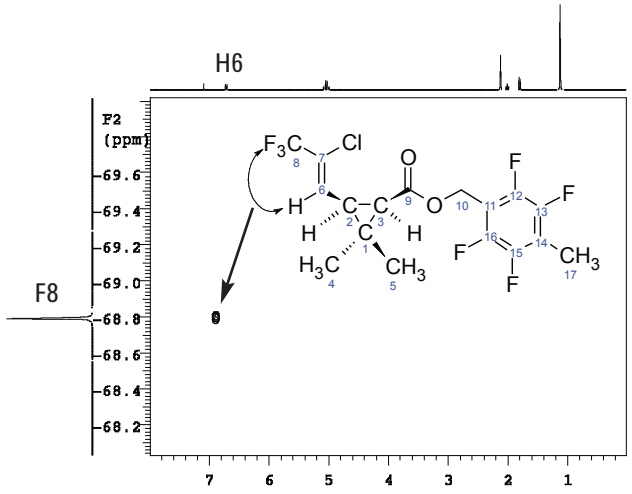

Figure 4. Stereochemical assignment around the C6-C7 double bond established from the spatial relationship of F8 to H6 observed in the HOESY spectrum of tefluthrin.

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