

# The Agilent 500 MHz OneNMR Probe

## Technical Overview

### Sample Dielectric Tolerance

#### Advantage Statement

The Agilent 500 MHz OneNMR probe is very tolerant of the differences in the dielectric properties of common NMR solvents, thereby eliminating the need for sample tuning in the routine  $^1\text{H}$  and  $^{13}\text{C}$  environment. This feature allows high-quality data collection on typical organic chemistry samples without the cost in time, wear and tear, and complexity required to actively tune the probe for each sample.

#### Introduction

NMR samples interact electromagnetically with the RF coils in the NMR probe. The magnitude of this interaction is proportional to the dielectric constant of the sample. When placed into the probe, samples with a high dielectric constant such as ionic solutions, couple strongly to the RF coils, increasing the capacitance of the circuit and thereby changing the tuning of the probe. Unless the probe is re-tuned for this new condition, the length of the 90 degree pulse width can suffer dramatically. Conversely, a probe tuned appropriately for a high dielectric sample will not perform as well if the sample has a comparatively low dielectric constant, for example, chloroform.



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The performance cost for running the NMR system in a poorly tuned state is significant for a typical NMR probe. To demonstrate this effect, a standard 500 MHz 5 mm Dual Broadband probe was tuned accurately on an organic chemistry sample dissolved in deuteriochloroform. Carbon spectra were acquired to establish baseline performance for the 90 degree pulse width and sensitivity. An aqueous 200 mM NaCl sample was then inserted into the probe and the system was tuned to this sample. Using this tune setting, the chloroform sample was returned to the magnet, and the pulse width and sensitivity data were once again collected. The results are shown in Table 1.

Table 1. Relative Probe Performance. 500 MHz 5 mm Dual Broadband Probe

	90 degree pulse width <sup>13</sup> C	<sup>1</sup> Signal-to-Noise <sup>13</sup> C
Probe tuned to chloroform	10.75 $\mu$ s	1.00
Probe tuned to 200 mM salt	15.00 $\mu$ s	0.59

<sup>1</sup>Both signal-to-noise measurements were made using the pulse width and power levels calibrated for the accurately tuned sample.

The NMR probe performance was degraded significantly by the poor tuning condition. Moreover, if a user attempted to operate the NMR in this condition using default system calibrations, the pulse width expected to yield a 90 degrees tip angle would actually yield only 64.5 degrees of spin rotation. An attempt was made to acquire HSQC data under these conditions but, as would be predicted based on the effective tip angles, the experiment failed to yield useful information.

As expected for the Dual Broadband probe configuration, the <sup>1</sup>H coil is not as strongly coupled to the sample. Similar experiments carried out on the proton channel yielded a pulse width increase of 5.1% with a concomitant loss in S/N ratio of 3.7%.

Until now, the only means available to correct this situation was to retune the NMR probe for every sample. Since tuning the probe is a mechanical operation, long-term considerations of wear and tear must be considered. Manual tuning is time consuming and prohibits the use of the spectrometer in an automated fashion. The Agilent ProTune accessory allows automated probe tuning, but this entails adding mechanical complexity to the system and more work is required when changing probes.

## The 500 MHz OneNMR probe

The Agilent 500 MHz OneNMR probe is very forgiving with respect to sample dielectric constant. Repeating the experiments described above with the OneNMR probe demonstrates the outstanding sample tolerance of this system. In this case, the carbon pulse widths were observed to change by only 8.6% and the <sup>13</sup>C sensitivity, even using the default pulse calibrations, was still 94% of optimum (Table 2).

Table 2. Relative Probe Performance. 5 mm OneNMR Probe

	90 degree pulse width – <sup>13</sup> C	<sup>2</sup> Signal-to-Noise <sup>13</sup> C
Probe tuned to chloroform	6.95 μs	1.00
Probe tuned to 200 mM salt	7.55 μs	0.94

<sup>2</sup>Both signal-to-noise measurements were made using the pulse width and power levels calibrated for the accurately tuned sample.

Given the <sup>13</sup>C performance results presented above and the excellent <sup>1</sup>H specifications of the Agilent OneNMR probe, one might anticipate that the proton channel would suffer from this type of intentionally mis-optimized tune experiment. This is not the case.

When the same worst-case set of tuning experiments were repeated using the high frequency channel on the 500 MHz OneNMR probe, the performance changes between the well-tuned probe and the poorly tuned probe were negligible. The proton 90 degree pulse width increased by 5.9%, while the S/N ratio decreased by only 5.3%.

These experiments plainly display the superior ability of the OneNMR probe to remain effectively tuned even with a large change in the dielectric constant of the sample.

## Practical Application of the OneNMR Probe's Sample Tolerance

The ability of the OneNMR probe to accept a wide range of solvents with minimal change in probe tuning means that, for routine organic chemistry applications at 500 MHz, the OneNMR probe can be used to collect high quality data without adjustment of the tuning circuit from sample-to-sample.

The typical NMR solvents used in organic chemistry do not represent a large range of dielectric constants. Chloroform is on the lower end of the scale with water on the upper side of the series (Figure 1).

Given this situation, one could easily tune the 500 MHz OneNMR probe to the middle of the frequency shift band and simply leave it there. The full range of organic NMR solvents would then be available for use without any need to retune the probe while maintaining essentially all of the excellent performance of the OneNMR probe.

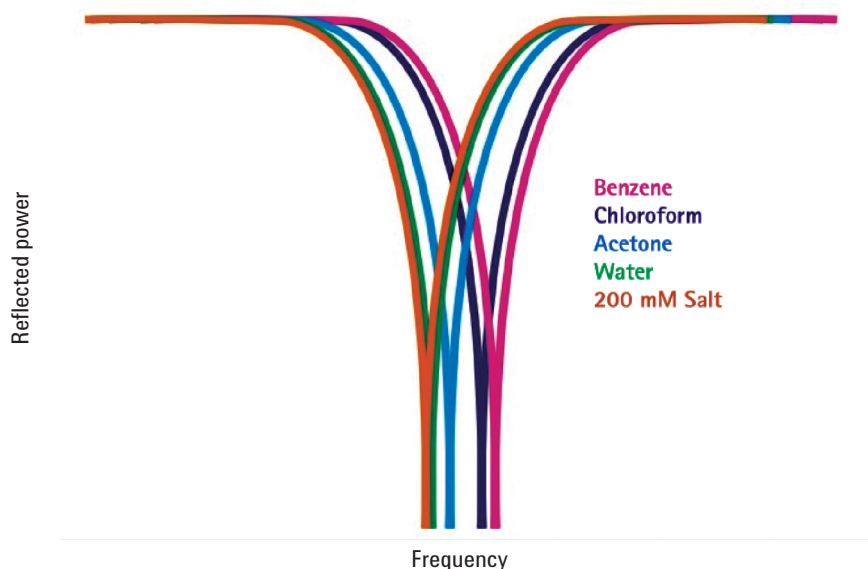


Figure 1. Relative frequency shifts for various solvents as measured in the Agilent OneNMR probe.

The HSQC experiment is a cornerstone NMR experiment for organic chemistry. It is also one of the more challenging experiments with respect to the quality of the NMR pulses used to collect the data. This makes it a perfect test experiment to demonstrate the ability of the OneNMR probe to yield high quality data without the need for careful tuning adjustments.

Figure 2 displays two gHSQC data sets obtained on a mixture of two alkaloids in deuteriochloroform using the 500 MHz OneNMR probe.

These data show that running a demanding 2-D experiment without tune optimization has little effect on sensitivity. In fact, comparison of the first increment of two adiabatic HSQC experiments with the probe tuned versus detuned as described above yielded a S/N change of less than 9%.

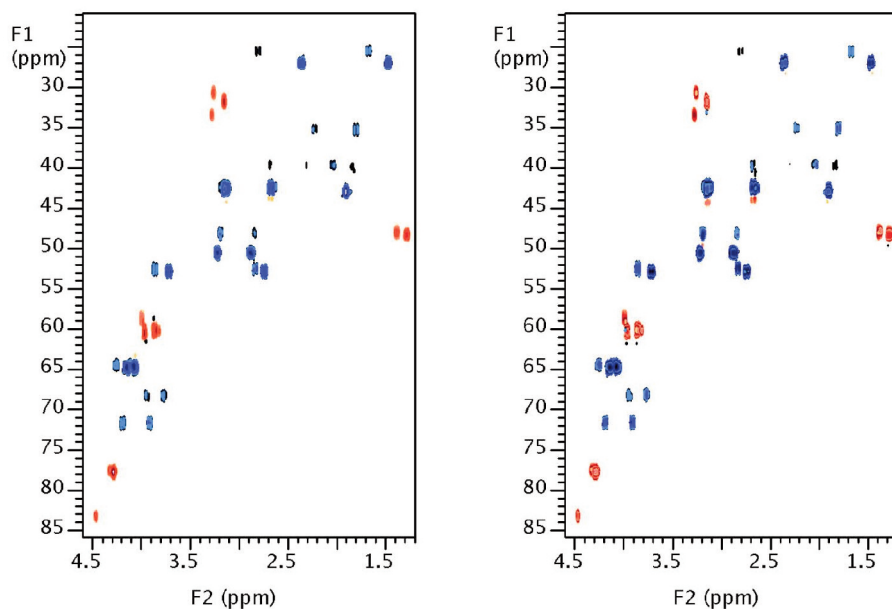


Figure 2. gHSQC data spectra acquired on a mixture of two alkaloids in deuteriochloroform using the OneNMR probe. The data in the left panel were obtained with the RF coils carefully tuned to the sample. The data in the right panel were obtained on the same sample but with both the proton and carbon RF coils tuned on a sample of 200 mM NaCl in  $D_2O$ . No attempt was made to compensate for the mis-optimization of the RF pulses in the second experiment; the pulse widths, power levels, and parameterization used for each experiment were identical. The experiment time for each data set was less than 5 minutes.

## Conclusions

These results clearly demonstrate that data collection of routine organic chemistry spectra using the Agilent 500 MHz OneNMR probe can be accomplished without sample-to-sample tuning. This procedure impacts neither the functionality of the NMR system nor the ability of the chemist to collect practical, interpretable NMR data. Furthermore, the ability to operate the OneNMR probe without sample-specific tuning saves the user time, wear and tear on the system, and mechanical complexity.

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