Spinsolve

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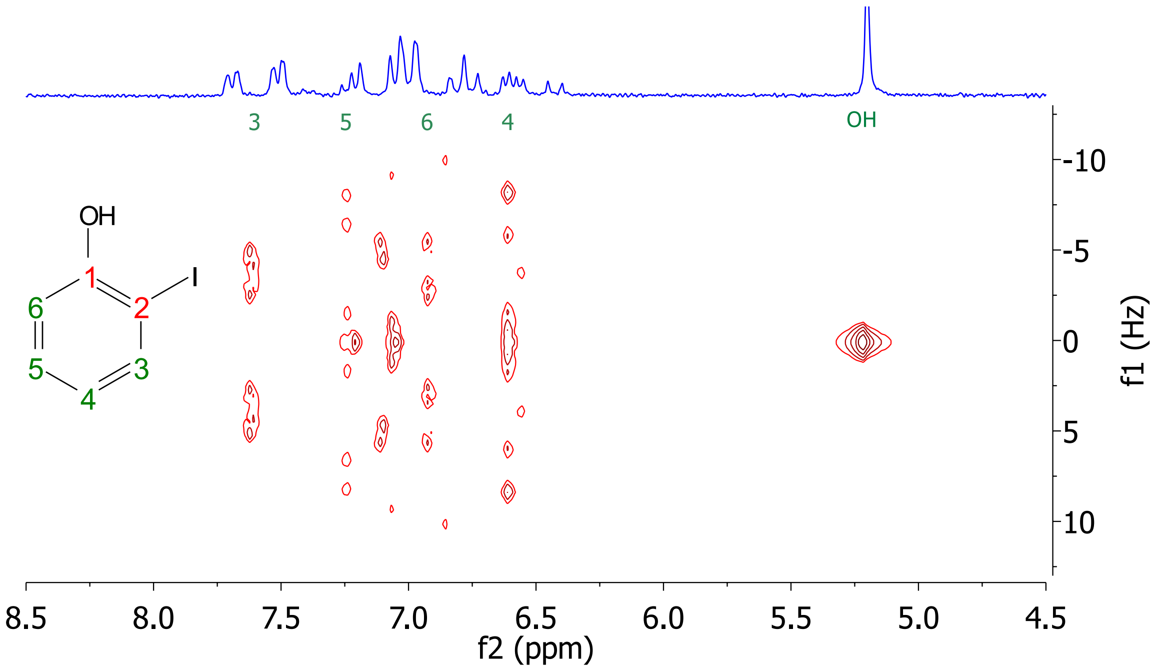
Application Note 4: Aromatic Systems and JRES Experiments

One of the features of working on a lower field instrument is the narrower spectral dispersion which is measured. This means that the spectra of most compounds are susceptible to second order effects of varying degrees. Second order effects arise when the difference in chemical shift becomes similar to the coupling constant. *J*-resolved (JRES) experiments allow the chemical shift to be separated from the *J*-coupling by displaying them in different dimensions. As a result, overlapping peaks can be deconvoluted. Some aromatic compounds are examples where the peaks overlap due to the similarity between the chemical shift and coupling frequencies.



4-Iodophenol has two aromatic proton resonances in the 1D proton NMR spectrum. The spectrum obtained on the Spinsolve (42.5 MHz) contains all of the same information (coupling constant and chemical shift) as the spectrum obtained at 300 MHz, but the multiplets occupy more of the ppm scale, meaning the multiplets appear more spread out (Figure 1).

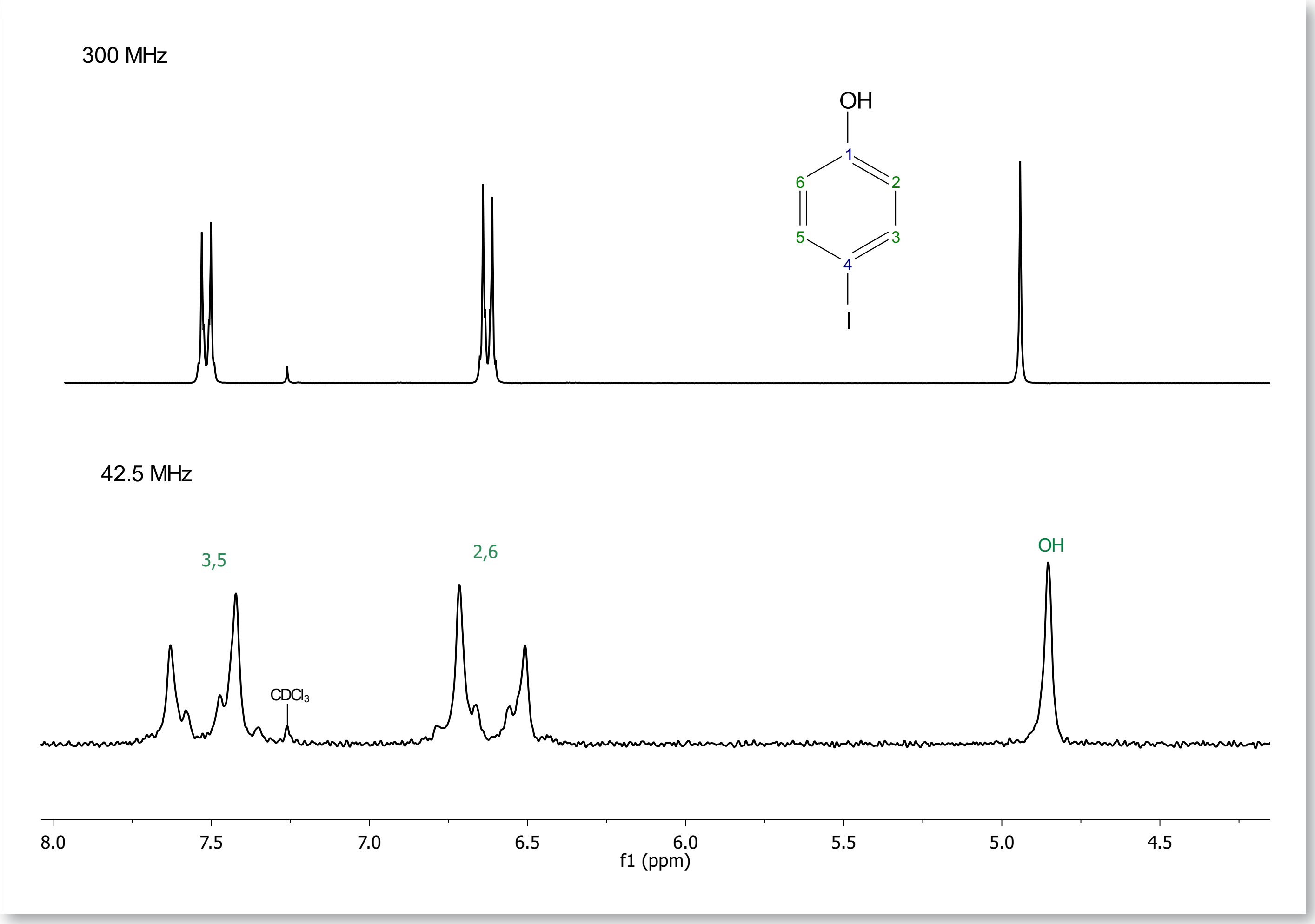


Figure 1: Spinsolve (42.5 MHz) and high field (300 MHz) data for 4-iodophenol.

^{1}H NMR (42.5 MHz, Chloroform-*d*) δ 7.51 (d, *J* = 9.0 Hz, 2H, H-3, H-5), 6.62 (d, *J* = 9.0 Hz, 2H, H-2, H-6).

^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.51 (d, *J* = 8.9 Hz, 2H, H-3, H-5), 6.61 (d, *J* = 8.9 Hz, 2H, H-2, H-6).

Second order effects can make the spectra of compounds that have multiple protons with similar chemical shift and strong coupling quite complicated. An example of this is 2-iodophenol, which has four proton resonances in a similar chemical shift range, with strong *J*-coupling. As a result, the peaks overlap and it is difficult to determine the chemical shift and coupling pattern from the 1D proton NMR spectrum.

For 2-iodophenol: Consider the green and purple regions (Figure 2).

For 42.5 MHz:

$\Delta\delta = 7.01\text{-}6.69\ \text{ppm} = 13.6\ \text{Hz}$

$^{3}J_{\text{H-H}} = 8\ \text{Hz}$

$\therefore\ \Delta\delta = 1.7\ J$

The chemical shift difference and coupling are comparable, which explains why the spectrum is so complicated (Figure 2, bottom).

For 300 MHz:

$\Delta\delta = 7.01\text{-}6.69\ \text{ppm} = 96\ \text{Hz}$

$^{3}J_{\text{H-H}} = 8\ \text{Hz}$

$\therefore\ \Delta\delta = 12\ J$

The difference in chemical shift is much larger than the coupling frequency, so the multiplets are well separated in the spectrum (Figure 2, top).

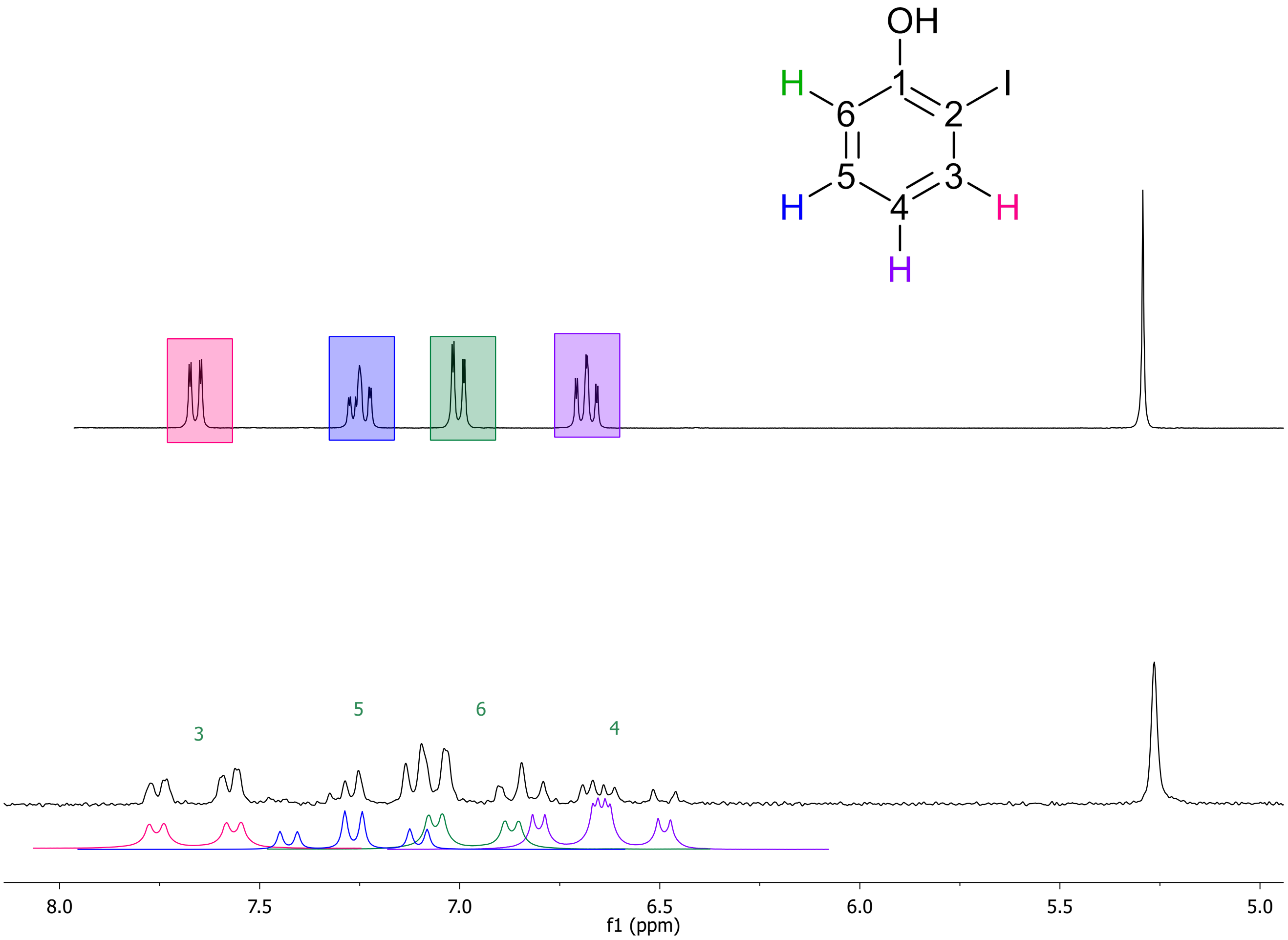


Figure 2: 300 MHz (top) and Spinsolve (bottom) data for 2-iodophenol. The high field data has been overlaid as coloured lines below the Spinsolve data to show the origin of the splitting pattern observed.

The Spinsolve system is capable of performing JRES experiments which separate chemical shift from *J*-coupling. This means all of the information observed in the spectrum from the high field instrument can be resolved (Figure 3).

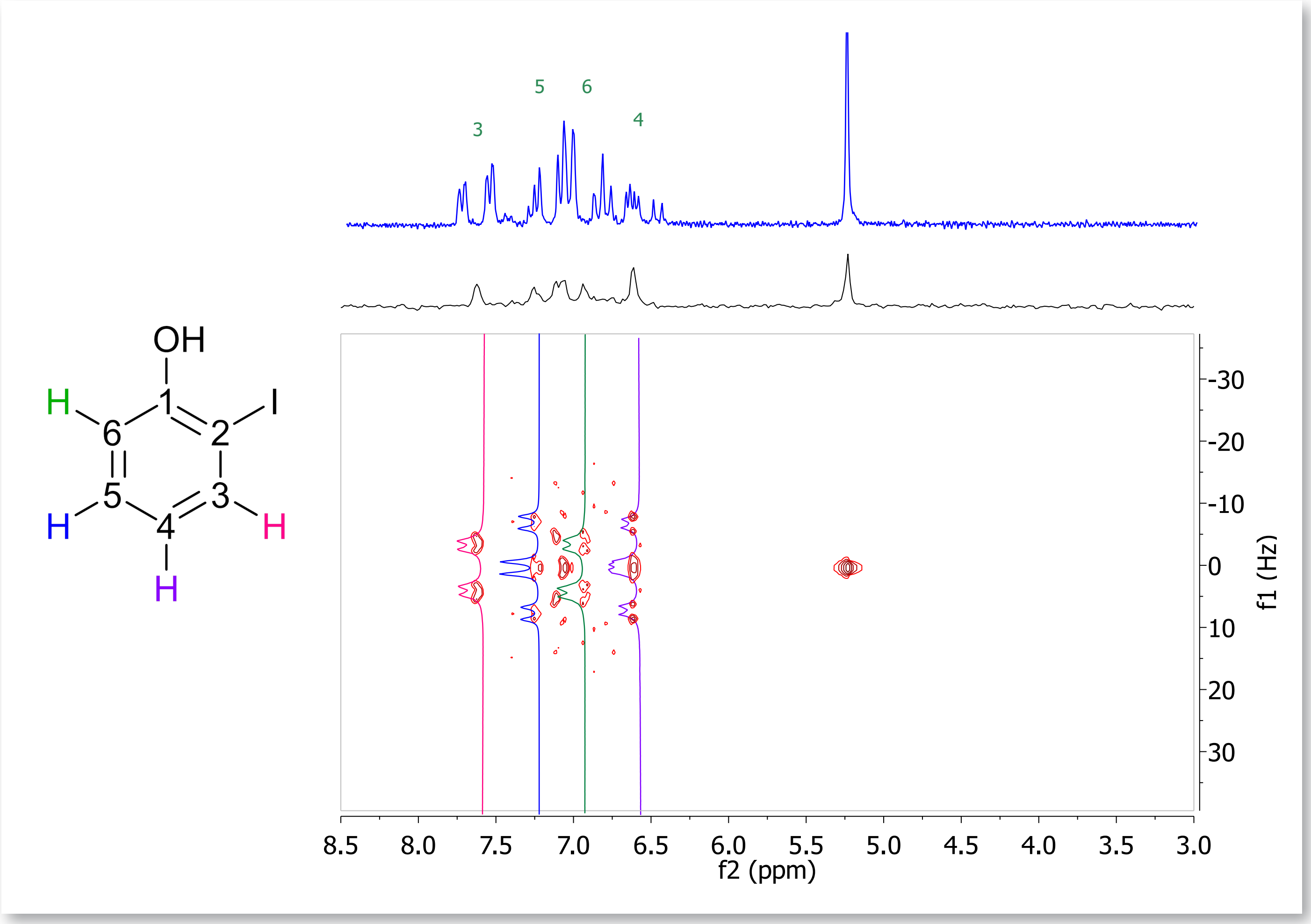


Figure 3: JRES data for 2-iodophenol obtained on a Spinsolve system with the 1D proton NMR above the f2 trace.

The JRES experiment allows the proton NMR spectral data of 2-iodophenol to be reported, which would be extremely difficult given only the 1D proton NMR spectrum.

^{1}H NMR (42.5 MHz, Chloroform-*d*) δ 7.66 (dd, *J* = 7.6, 1.6 Hz, 1H, H-3), 7.23 (td, *J* = 7.3, 1.6 Hz, 1H, H-5), 6.93 (dd, *J* = 8.2, 2.0 Hz, 1H, H-6), 6.60 (td, *J* = 7.5, 2.3 Hz, 1H, H-4), 5.23 (s, 1H, OH).

Conclusion

Working on a 42.5 MHz system means that most spectra will display some second order effects which become more complicated as the difference in chemical shift approaches the magnitude of the *J*-coupling. For compounds with simple spectra such as 4-iodophenol, the 1D proton spectrum contains the same information as that obtained on a high field instrument, but each multiplet occupies more of the ppm scale. For more complicated systems such as 2-iodophenol, the second order effects make the spectrum difficult to interpret. The JRES experiment was able to separate *J*-coupling from chemical shift which allowed the individual resonances to be identified and reported.

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