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Benchtop NMR on Paramagnetic Samples

Many organometallic complexes contain paramagnetic ions. These extend the chemical shift range of proton spectra to hundreds of ppm. At high field, such large bandwidths are very challenging to achieve. The Magritek Spinsolve benchtop NMR spectrometer has a special *Paramagnetic* protocol designed to scan such samples. The acquisition bandwidth is 20 kHz corresponding to 480 ppm frequency range. Since paramagnetic ions also reduce the T_1 relaxation time of the sample, the repetition time between scans is reduced to 3 seconds resulting in faster scan times. The examples in this note demonstrate the ability of the *Paramagnetic* protocol to resolve peaks which are not visible in the standard *1D Proton* protocol.

Bis(N-2'-Butylsalicylicylaldiminato)Nickel(II)

In order to demonstrate how paramagnetic ions can extend the spectral range, a sample of bis(n-2'-butylsalicylicylaldiminato)nickel(II) from a third-year chemistry teaching lab was dissolved in $CDCl_3$ and analysed.

Figure 1 shows the spectrum of the sample using the *1D Proton* (red spectrum) and *Paramagnetic* (blue line) protocols. The bandwidth of the *1D Proton* protocol is not sufficient to cover the entire spectral range of the sample while the *Paramagnetic* protocol tells the full story.

Figure 1

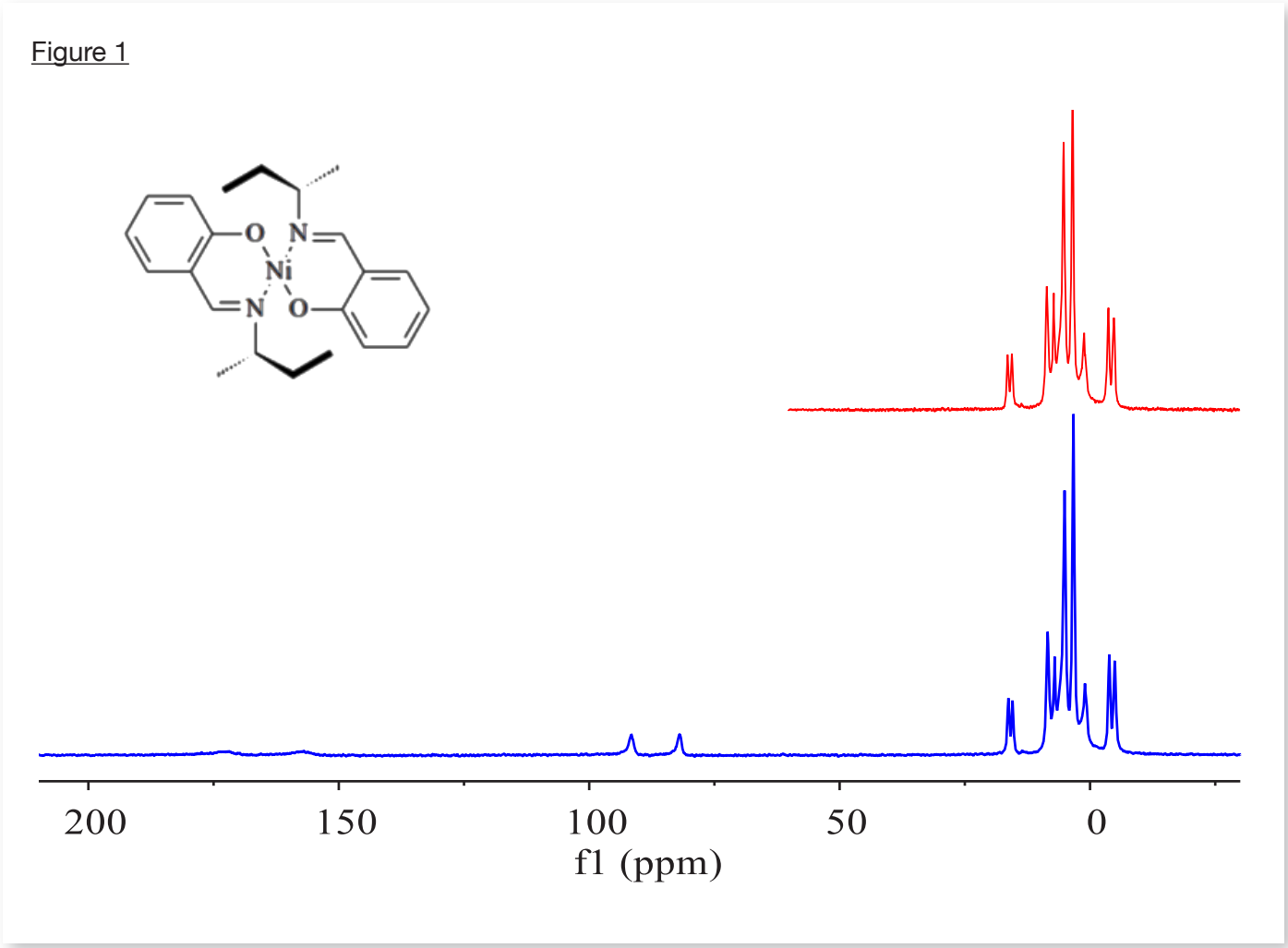

Figure 1: 1H NMR spectrum of bis(n-2'-butylsalicylicylaldiminato)nickel(II) dissolved in $CDCl_3$ using the 1D Proton (red) and Paramagnetic (blue) protocols. Note that the bandwidth of the 1D Proton protocol is insufficient to cover the entire spectrum of the sample.

Cophen

This example from a third-year chemistry experiment demonstrates the reaction of coordinated ligands using the conversion of paramagnetic $[Co(phen)_3]^{2+}$ to diamagnetic $[Co(phen)_3]^{3+}$ complexes.

Figure 2 shows how the chemical shift range differs dramatically between the paramagnetic and diamagnetic cobalt complexes.

Figure 2

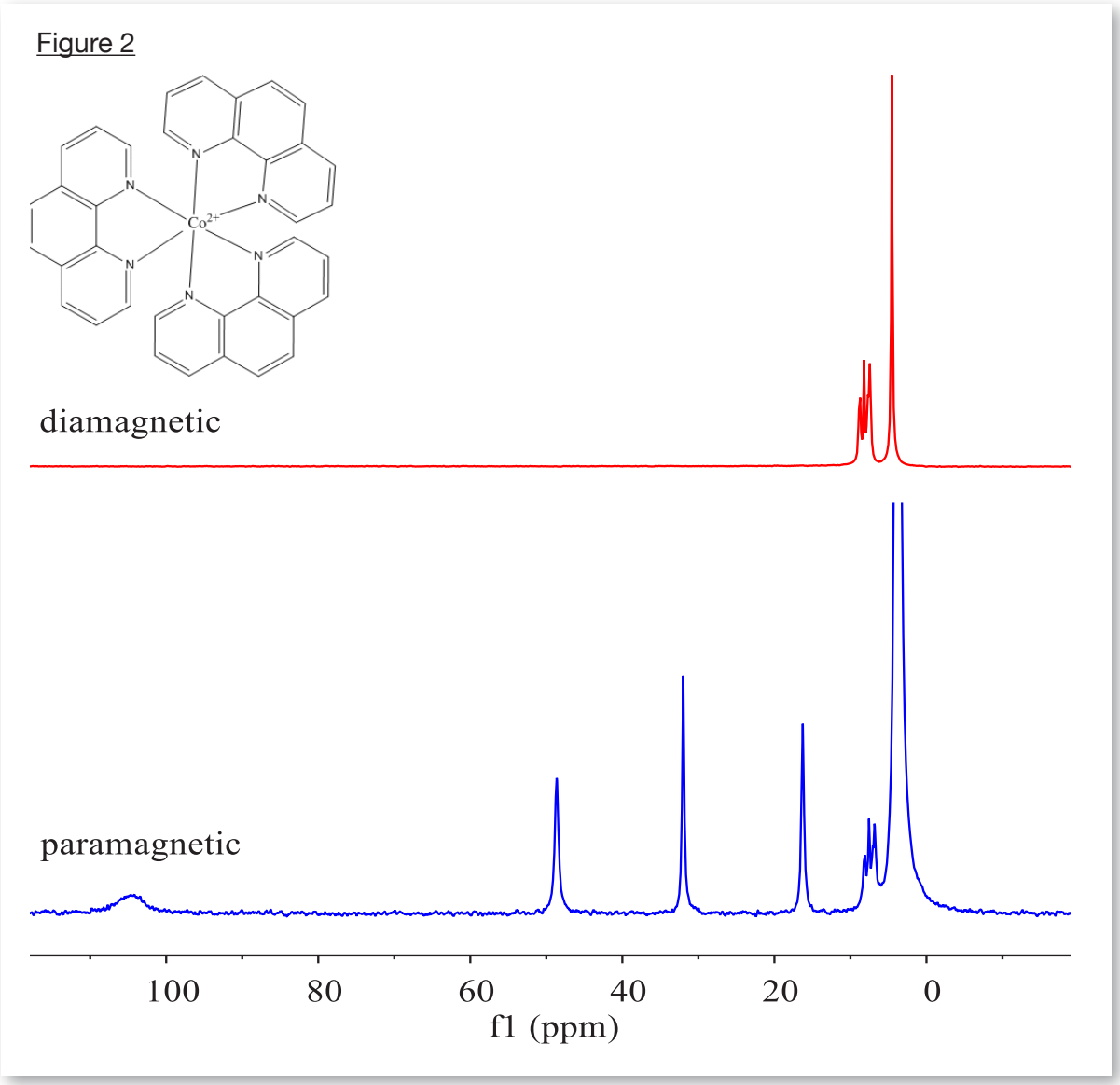

Figure 2: NMR spectrum of diamagnetic and paramagnetic $Co(phen)_3$ dissolved in D_2O, using the Paramagnetic protocol. Note how the chemical shift range extends dramatically for the paramagnetic complex. Spectra courtesy of Paul S Donelly, University of Melbourne.

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Conclusion

Paramagnetic samples are often difficult to obtain NMR spectra from. The specially designed *Paramagnetic* protocol enables the Spinsolve benchtop NMR spectrometer to resolve peaks in wide line spectra, which cannot be resolved with standard methods.

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GERMANY	+49 241 9278 7270	**UNITED STATES**	+1 855 667 6835
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