Boron is a good nucleus for direct detection in solution by NMR. Relaxation of the isotopes of boron varies considerably depending on the specific chemistry, and experimental optimization quite important. Typically, the relaxation is directly related to the observed linewidths, providing a fairly straightforward method for optimization of the repetition times.

$^{11}$B is most commonly observed due to its higher magnetogyric ratio (160.4 MHz on a 500) and natural abundance (80.4%). It is spin $\frac{3}{2}$.

$^{10}$B may be better for compounds where asymmetric bonding leads to broader linewidths, as noted in a recent publication. Since $^{10}$B is spin-3, its relaxation in solid materials is so fast (linewidths then are very broad) as to make it undetectable. $^{10}$B spectra therefore have no background from solid probe components (typically boron nitride). This background can be a major issue in $^{11}$B spectra.

$^{10}$B is, however, at least 10$\times$ less sensitive than $^{11}$B, due to its lower magnetogyric ratio (53.7 MHz on a 500) and natural abundance (19.6%). $^{10}$B spectra will therefore take $\geq 10^2$ times longer to obtain than $^{11}$B from the same sample, so sample concentrations must be relatively high for $^{10}$B spectra.

**Experimental Setup:**

Obtain spectra for similar materials, and measure the full width at half-height of the peaks, $\Delta v$. For the narrowest peak, estimate the spin-lattice relaxation, $T_1$, as follows:

$$ T_1 \approx T_2 = \frac{1}{\pi \Delta v} \quad (1) $$

(a) Set $a_0 = 3 \times T_1$ using the narrowest peak of interest in the spectrum of a similar compound.

(b) Set $d_1 = 30 \mu s$

(c) Set $n_s$ very large, and use halt when sufficient scans have been acquired.

(d) If peaks get very narrow — less than 6 Hz (and note that LB adds to the linewidth directly) — switch the pulse sequence to $\text{pulprog} = \text{zg30}$ and $a_0 \sim 1 \times T_1$.

(e) $^1$H decoupling may be desirable for samples with narrow peaks: $\text{pulprog} = \text{zgpg30}$, $a_0 \sim 1 \times T_1$ and make sure to getprosol prior to rga or zg.

To remove background signals in $^{11}$B spectra, acquire an identical spectrum using another tube containing the same amount of the same solvent only. Take the difference of this blank with the $^{11}$B spectrum from the sample.

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