

Quantitation in NMR:

Question: What do I need to use for a relaxation delay to obtain quantitative integrations?

Answer: It depends upon how "quantitative" you wish to get, what your excitation pulse-angle is, and on the longest T1 relaxation time in your sample.

If we define "Q" as the "quality" desired for quantitation, often called " M_z/M_0 " (1.0 would mean that the steady state magnetization = the equilibrium magnetization...in reality, Q will always be < 1.0), the relationship can be given by:

$$Q = \frac{1 - e^{-Tr/T1}}{1 - [e^{-Tr/T1} \cdot \text{Cos}(\alpha)]}$$

- Tr is the repetition rate (Acquisition time + Relaxation Delay). On the Varian instrument, Tr = at + d1
- α is the excitation pulse angle. On the Varian instrument, the command "pw(α)" will set the angle to α .

Two more convenient forms of the above equation are shown below:

$$Tr = -\ln\left(\frac{1-Q}{1-Q \cdot \text{Cos}(\alpha)}\right) \cdot T1 \quad \dots \text{or solve for } \alpha \quad \dots \quad \alpha = \cos^{-1}\left(\frac{Q-1+e^{-Tr/T1}}{Q \cdot e^{-Tr/T1}}\right)$$

After choosing on the "quality" of integrations (based on relative relaxation rates), use the known (or estimated) longest T1 in your sample. Using the left equation above, enter the pulse angle (the default in the standard ^1H parameters on our instruments is 30 degrees), and calculate the minimum value of Tr necessary. Or you can use the right equation and enter Tr, the longest T1, and calculate the pulse angle (α) that will give the desired "Q".

Example: We want our integrated intensities to be accurate to within 95% (Q=.95), and we estimate that our T1s would be typical for a non-degassed organic sample (T1=1-3 sec.). Our excitation pulse is 30°. Using the above equation: Q=.95, T1=3.0, $\alpha=30^\circ$...

$$Tr = -\ln\left(\frac{1-.95}{1-(.95 \cdot \text{Cos}(30))}\right) \cdot 3.0$$

This gives Tr = 3.8 Sec.

The default acquisition time is 3.0 sec (at=3.0), so the relaxation delay necessary for 95% relaxation would be 0.8 seconds.