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The nuclei being radio frequency (RF) irradiated are of a different isotope than the nuclei being observed. The splitting reduces the sensitivity of signals that are already weak, and the large splitting causes an overlap of lines which results in complex spectra.

- 1. Create new experiment.
- 2. Inject the sample.
- 3. Set the following acquisition parameters:

PL1 : F1 channel – High power level for 13C transmitter pulse
P0 : F1 channel – 45° <sup>13</sup>C transmitter pulse
PL14 : F2 channel – Power level for CW/HD decoupling
D1 : 2 s – relaxation delay
D11 : 30 ms – delay for disk I/O
TD : 4 K
SW : 500 Hz
O1 : On resonance of <sup>13</sup>C signal
O2 : 50 Hz offset from <sup>1</sup>H signal
NS : 1
RG : Receiver gain for correct ADC input

- 4. Lock signal on appropriate solvent.
- 5. Shim.
- 6. Set up the spectrometer.
- 7. Acquire data.
- 8. Process the data.
- 9. Set the following processing parameters:

SI : 4 K or more; use zero-filling to ensure enough data points to obtain accurate values for the residual splittings
BC\_mod : "quad"
WDW : "EM"
LB : 2 Hz
FT: Fourier Transform
Phase correction : adjust the phase to pure absorption

## **Baseline correction** : ABS **Referencing** : set the TMS signal to 0 ppm

- 10. Reset the parameters once finished.
- 11. Eject the sample and replace it with the standard.
- 12. Lock signal on specified solvent.