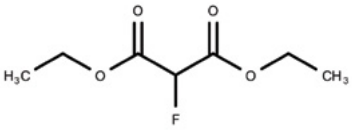
^{1}H, ^{19}F and ^{13}C analysis in under two minutes using X–Pulse

Application Note 9

Introduction

Nuclear Magnetic Resonance (NMR) spectroscopy is an invaluable analytical technique; the information from an NMR spectrum complements the information obtained from other types of molecular spectroscopy and in many cases it offers unique diagnostic information about the sample material.

Benchtop NMR performs well when a quick analysis of a high-concentration sample is needed, demonstrated using the example of diethylfluoromalonate.

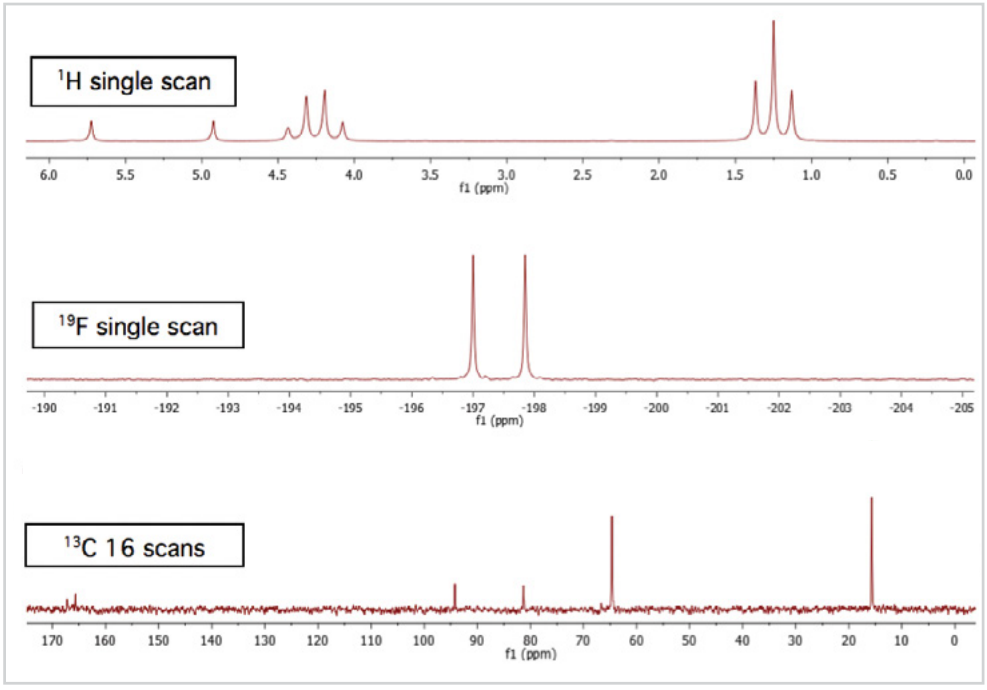


Diethylfluoromalonate is liquid at room temperature, and can be analysed neat in a standard NMR tube

The **X–Pulse** 60 MHz NMR spectrometer has two frequency channels: one acquires spectra of ^{1}H and ^{19}F and the other, ^{13}C. The probe can be manually tuned and matched for optimal performance. In this example, the first channel was tuned to ^{1}H and then the pulse width was lengthened to ensure effective radiofreqency (RF) transmission for the ^{19}F experiment.

Method

0.4 mL of diethylfluoromalonate was pipetted into a standard 5 mm NMR tube and placed in the **X–Pulse** 60 MHz spectrometer with no temperature preconditioning. The sample was run unlocked and data was processed with Mestrelab's Mnova software.



Results

The results can be seen in the following scans.

Note that the spectra are approximately referenced. 1Hz exponential apodization was applied to the ^{13}C spectra.

Figure 1. *Total acquisition time for three spectra = approximately 75 seconds.*

Acquiring a greater number of scans provides spectra with better signal to noise ratio. This is particularly noticeable for low-sensitivity nuclei such as ^{13}C. The quaternary carbon doublet (split by the fluorine) signals at 166 ppm can be compared in Figure 1 and Figure 2.

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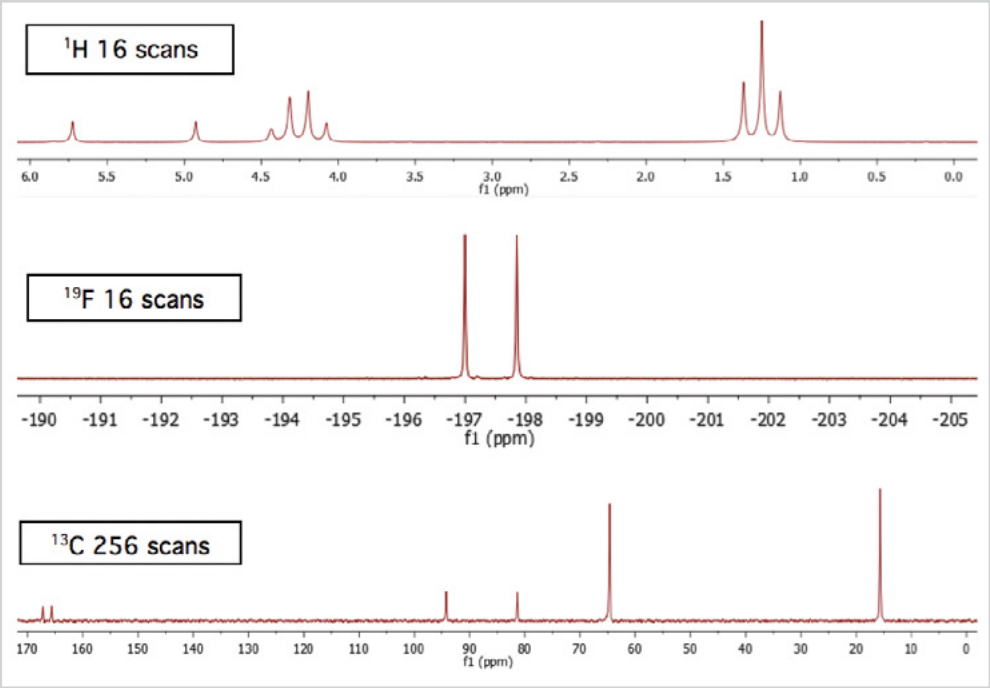

Figure 2. *Total acquisition time for the three spectra = approximately 30 minutes.*

Conclusion

This application note shows that for a sample of high enough concentration, it is possible to rapidly acquire meaningful 1D spectra of different nuclei on a benchtop NMR spectrometer.

visit **nmr.oxinst.com/x–pulse** for more information or email **magres@oxinst.com**

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