Epoxiconazole

Epoxiconazole structurally belongs to the class of azoles, bearing a characteristic 1,2,4-triazole moiety. It is a commonly known fungicide which inhibits the metabolism and thus growth of fungal cells. Figure 1 shows the ^{1}H NMR spectrum of a 250 mM Epoxiconazole sample in $CDCl_3$ measured in a single scan taking 10 seconds to acquire.

1D Proton spectrum

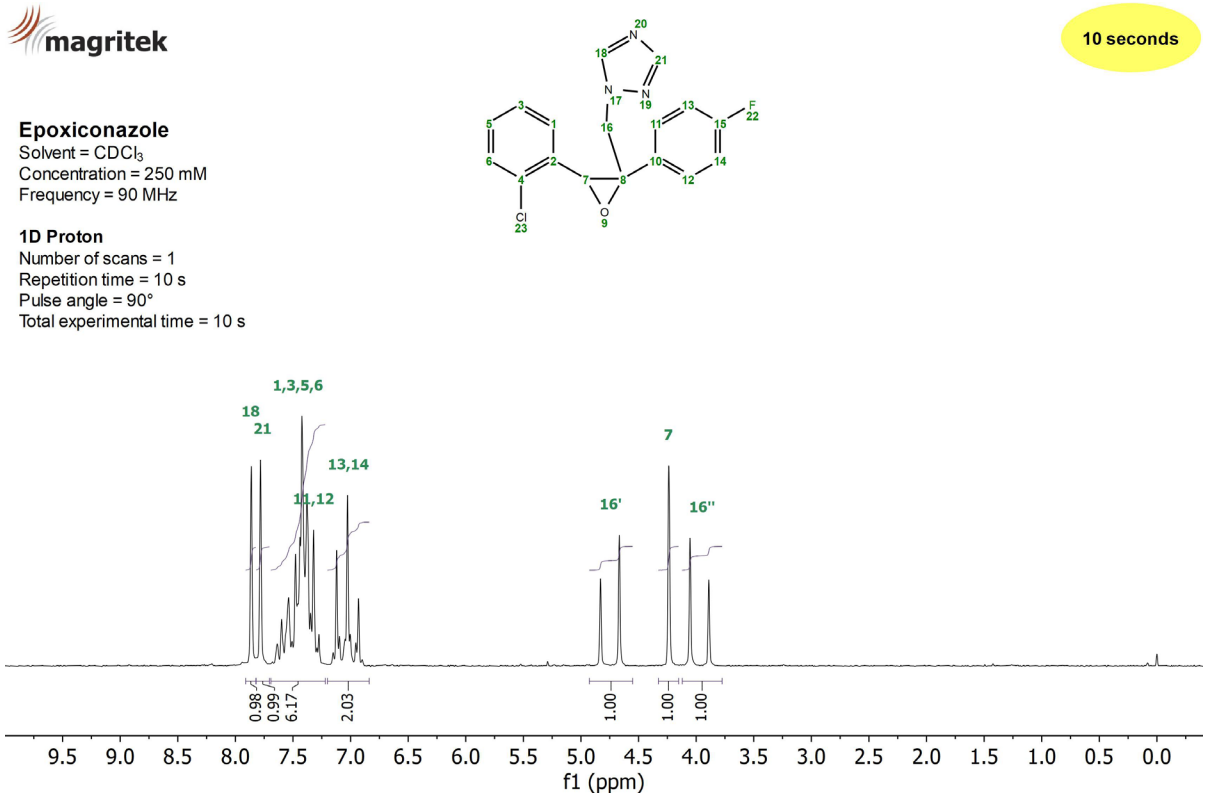

Figure 1: ^{1}H NMR spectrum of a 250 mM Epoxiconazole sample in $CDCl_3$ measured on a Spinsolve 90 MHz system in a single scan.

1D Carbon spectrum

Figure 2 shows the ^{13}C NMR spectrum of 250 mM Epoxiconazole in $CDCl_3$ acquired using NOE polarization transfer from ^{1}H to ^{13}C and ^{1}H decoupling. The 1D Carbon experiment using NOE is sensitive to all ^{13}C nuclei in the sample. It clearly resolves all the expected resonances.

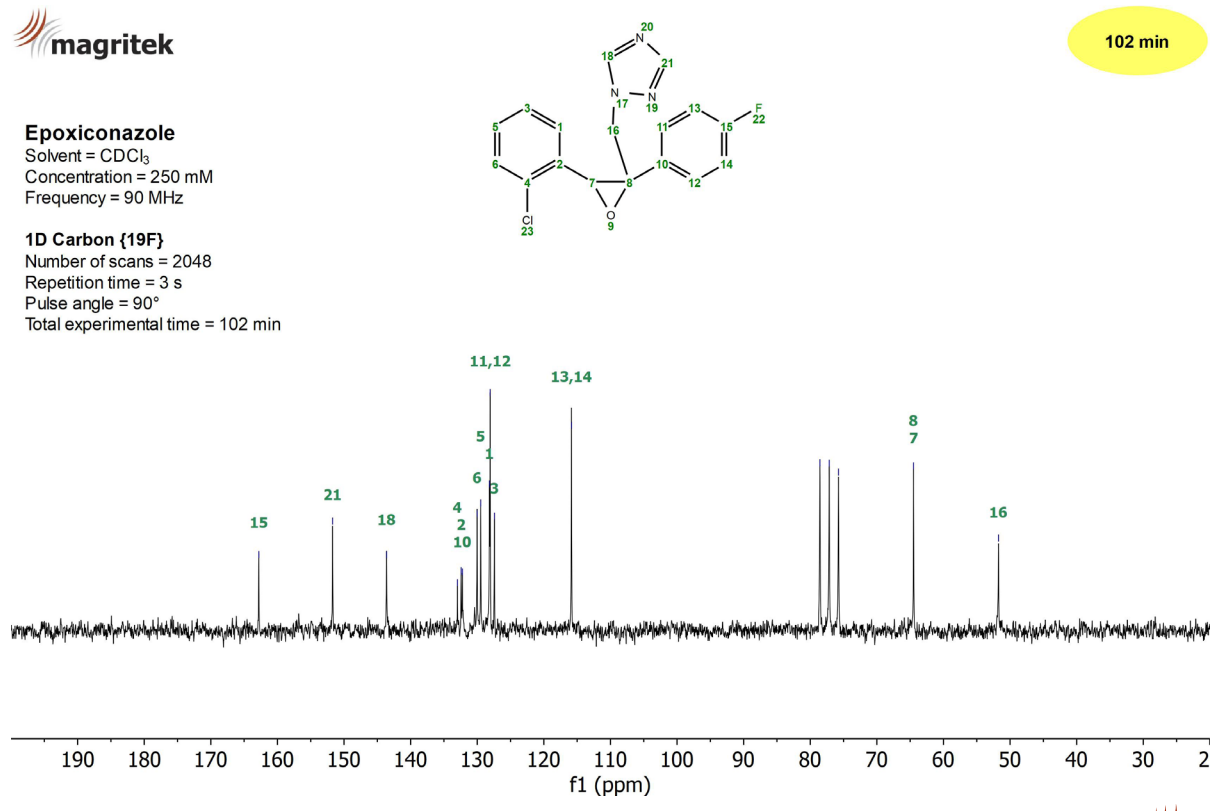

Figure 2: ^{13}C NMR spectrum of a 250 mM Epoxiconazole sample in $CDCl_3$ measured on a Spinsolve 90 MHz system in 102 minutes.

2D COSY spectrum

The 2D COSY experiment allows one to identify coupled ^{1}H nuclei as they generate cross peaks out of the diagonal of the 2D data set. In Figure 3 a large number of cross peaks can be observed nicely. For example, the protons at position 16 and 18 (light blue) couple with each other. Furthermore, proton 7 couples with proton 1 (light green). In addition, the couplings between protons 11,12 and 13,14 (dark blue) can be observed nicely.

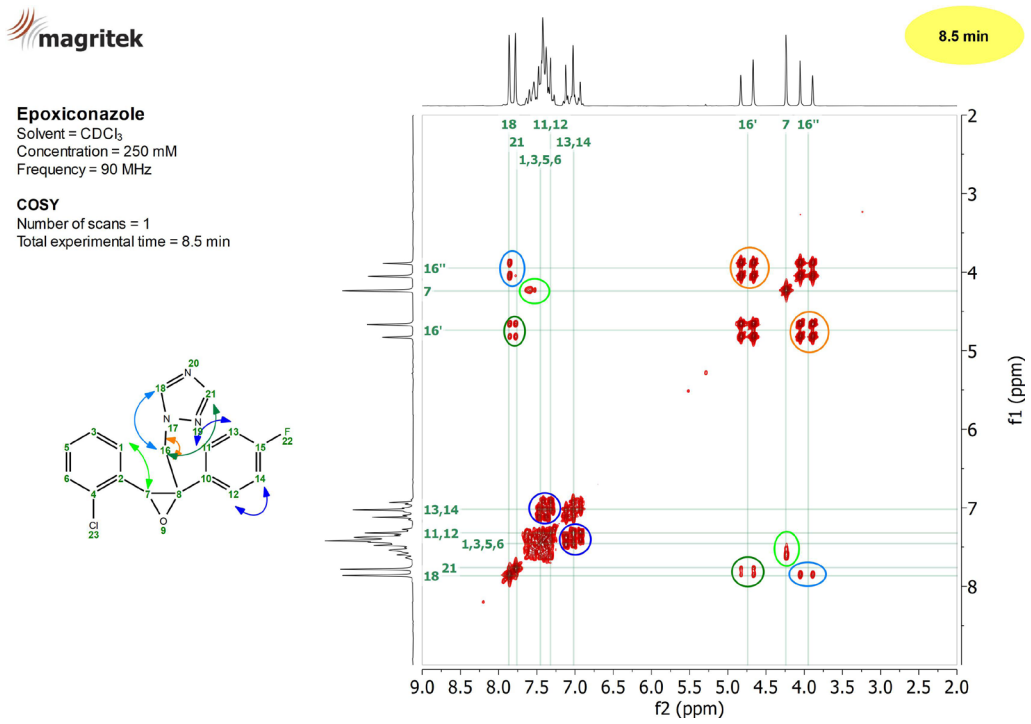

Figure 3: ^{1}H 2D COSY experiment of a 250 mM Epoxiconazole sample in $CDCl_3$ acquired in 8.5 minutes on a Spinsolve 90 MHz system.

2D ^{1}H-^{13}C HSQC-ME

The HSQC is a powerful sequence widely used to correlate the ^{1}H with the one-bond coupled ^{13}C nuclei. The Spinsolve is equipped with a multiplicity edited version (HSQC-ME) of this method. It provides the editing power of the DEPT-135 sequence, which is useful to differentiate between the signals of the CH_2 groups (blue) from the CH and CH_3 groups (red). Figure 4 shows the HSQC-ME spectrum of a 250 mM Epoxiconazole sample in $CDCl_3$ acquired in 4 minutes. The measurement time was optimized applying NUS (non uniform sampling).

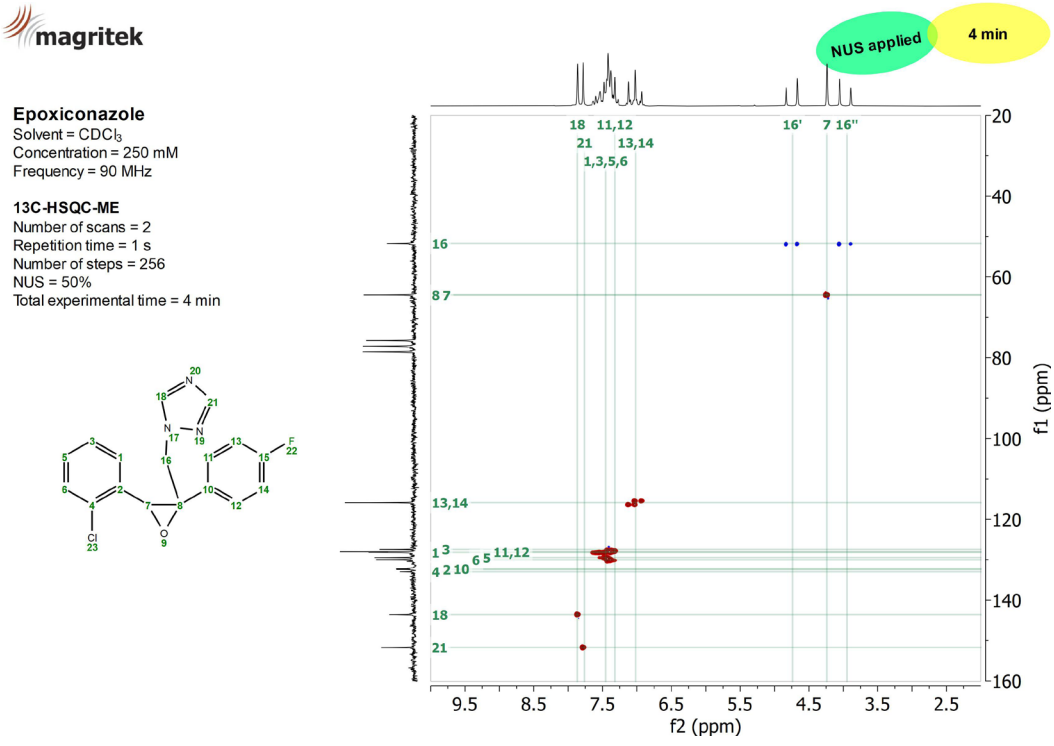

Figure 4: HSQC-ME spectrum of a 250 mM Epoxiconazole sample in $CDCl_3$ showing the correlation between the ^{1}H (horizontal) and ^{13}C (vertical) signals.

2D ^{1}H-^{13}C HMBC

To obtain long-range ^{1}H-^{13}C correlations through two or three bond couplings, the Heteronuclear Multiple Bond Correlation (HMBC) experiment can be used. Figure 5 shows the long-range correlation of proton 7 with carbons 1, 2 and 8 (the sequence shows the correlation with quaternary carbons, too).

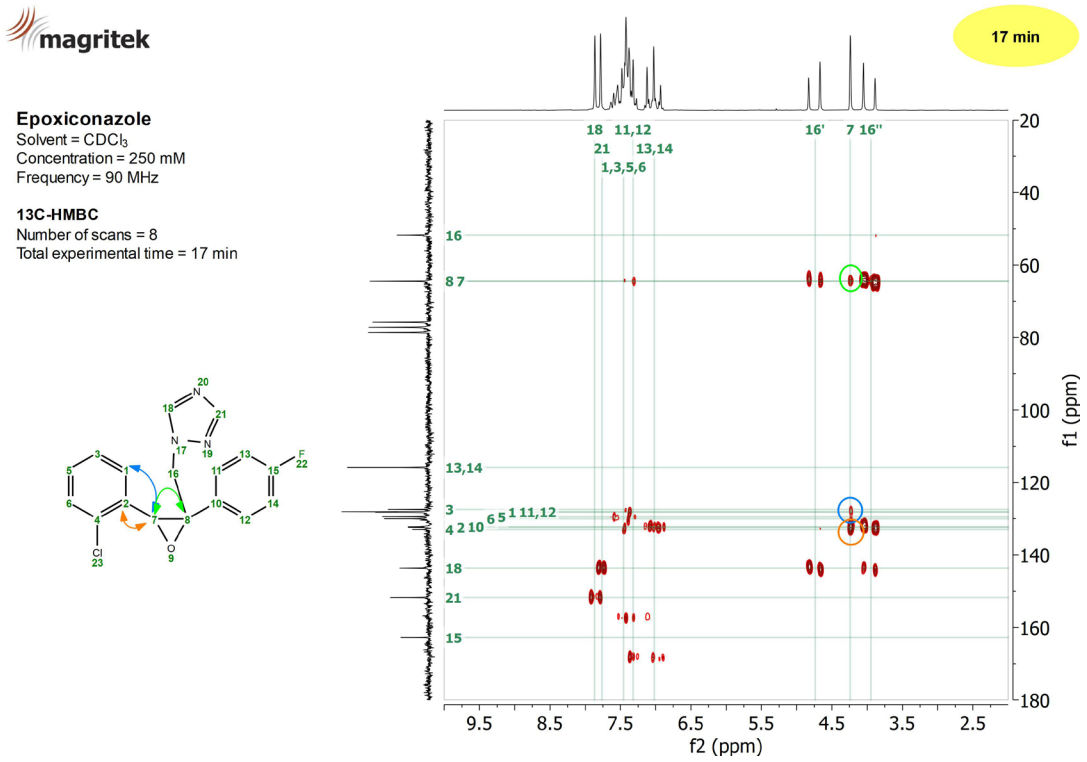

Figure 5: HMBC spectrum of a 250 mM Epoxiconazole sample in $CDCl_3$ showing the long-range couplings between ^{1}H and ^{13}C nuclei.

1D Fluorine spectrum

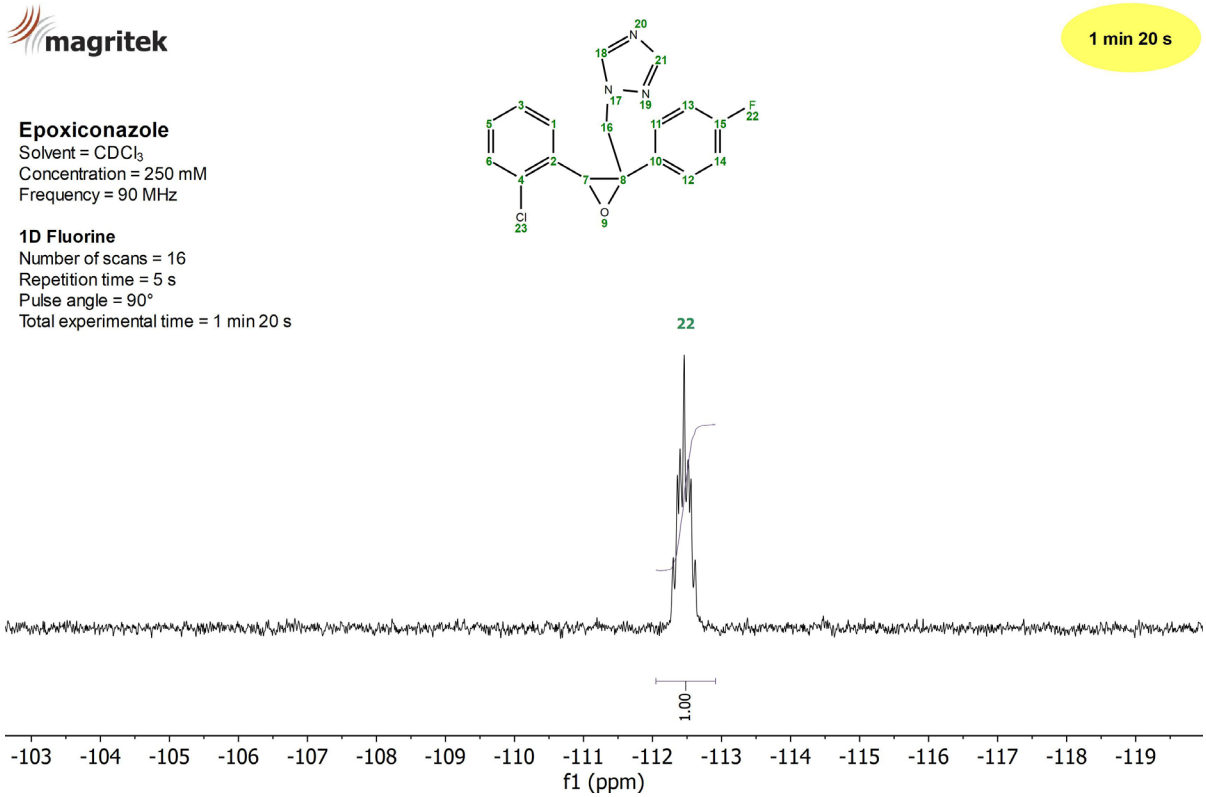

Figure 6: ^{19}F NMR spectrum of a 250 mM Epoxiconazole sample in $CDCl_3$ measured on a Spinsolve 90 MHz system in 16 scans.

2D ^{1}H-^{15}N HMBC

To obtain long-range ^{1}H-^{15}N correlations through two or three bond couplings, the Heteronuclear Multiple Bond Correlation (HMBC) experiment can be used. The top spectrum of Figure 7 shows the full spectral range and depicts nicely all long-range correlations of the three nitrogen nuclei 17, 19 and 20 with the respective protons at positons 16, 18 and 21. The bottom spectum of Figure 7 describes the region between 8.0 and 7.5 ppm in more detail. On the right side the respective traces plots underline the quality of spectral resolution with the shown coupling constants ranging from 8.9 to 14.9 Hz. To note is the great SNR on the ^{15}N trace that indicates even reduced total measurement times to the 3 h 24 min shown.

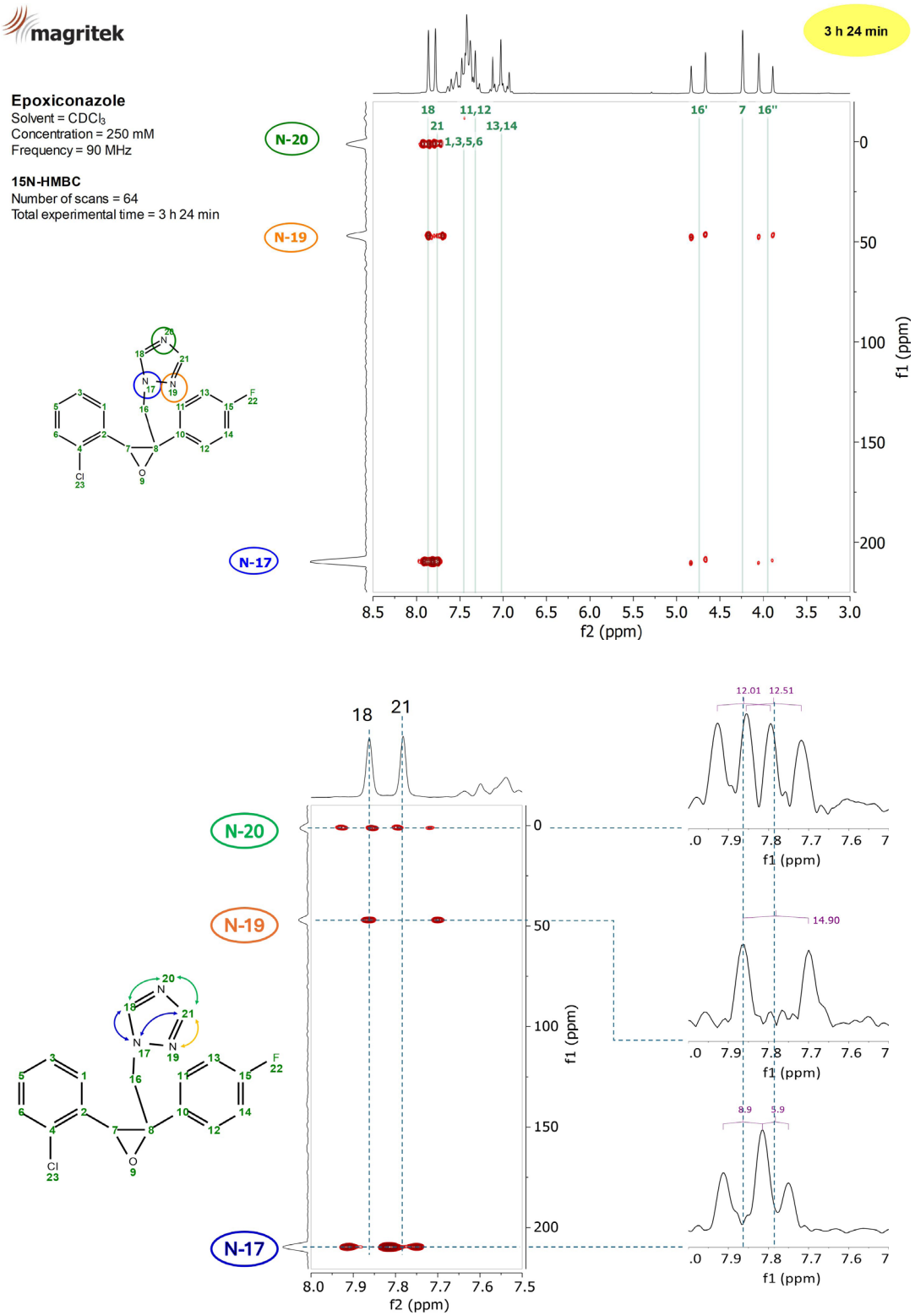

Figure 7: HMBC full spectrum of a 250 mM Epoxiconazole sample in $CDCl_3$ showing the long-range couplings between ^{1}H and ^{15}N nuclei (top); highlight zoom of 1H region between 8.0-7.5 ppm and traces plots for the three ^{15}N nuclei.