Levofloxacin

Levofloxacin is a commonly known antibiotic drug active against a range of bacterial infections. Structurally Levofloxacin belongs to the group of fluoroquinolones. It is a chiral compound in the (-)-(S) enantiomeric form. Figure 1 shows the ^{1}H NMR spectrum of a 250 mM Levofloxacin sample in DMSO-d_6 measured in a single scan taking 10 seconds to acquire.

1D Proton spectrum

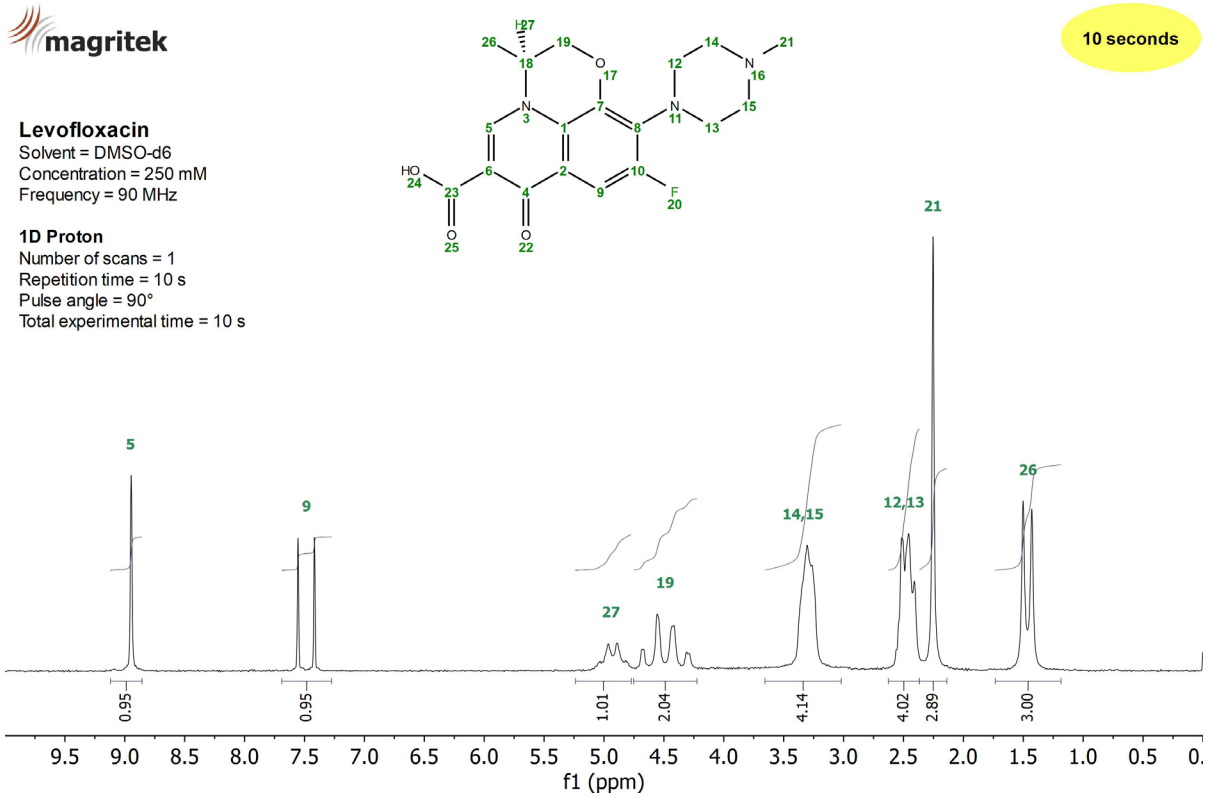

Figure 1: ^{1}H NMR spectrum of a 250 mM Levofloxacin sample in DMSO-d_6 measured on a Spinsolve 90 MHz system in a single scan.

1D Carbon spectrum

Figure 2 shows the ^{13}C NMR spectrum of 250 mM Levofloxacin in DMSO-d_6 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. The spectrum was aquired with both ^{1}H and ^{19}F decopling.

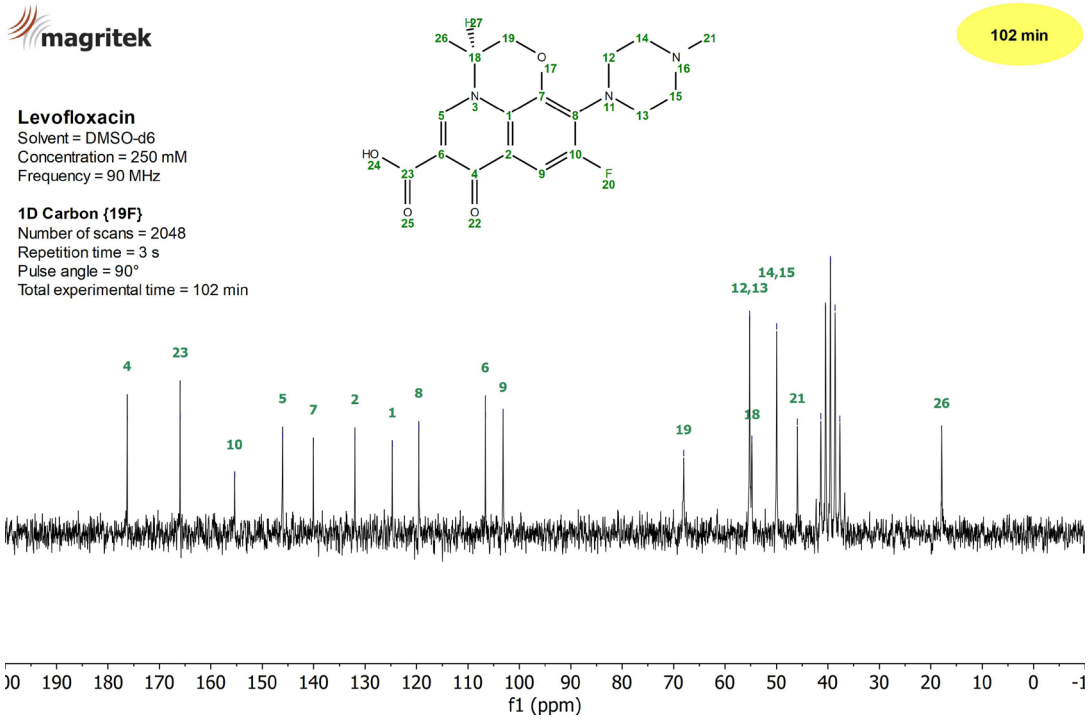

Figure 2: ^{13}C NMR spectrum of a 250 mM Levofloxacin sample in DMSO-d_6 measured on a Spinsolve 90 MHz system in 102 minutes.

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 26 and 19 (light blue) couple with each other. Furthermore, protons 12 and 13 couple with protons 14 and 15 (orange). In addition, the couplings between proton 27 and 5 (dark blue), 19 (dark green) and 26 (light green) can be observed nicely.

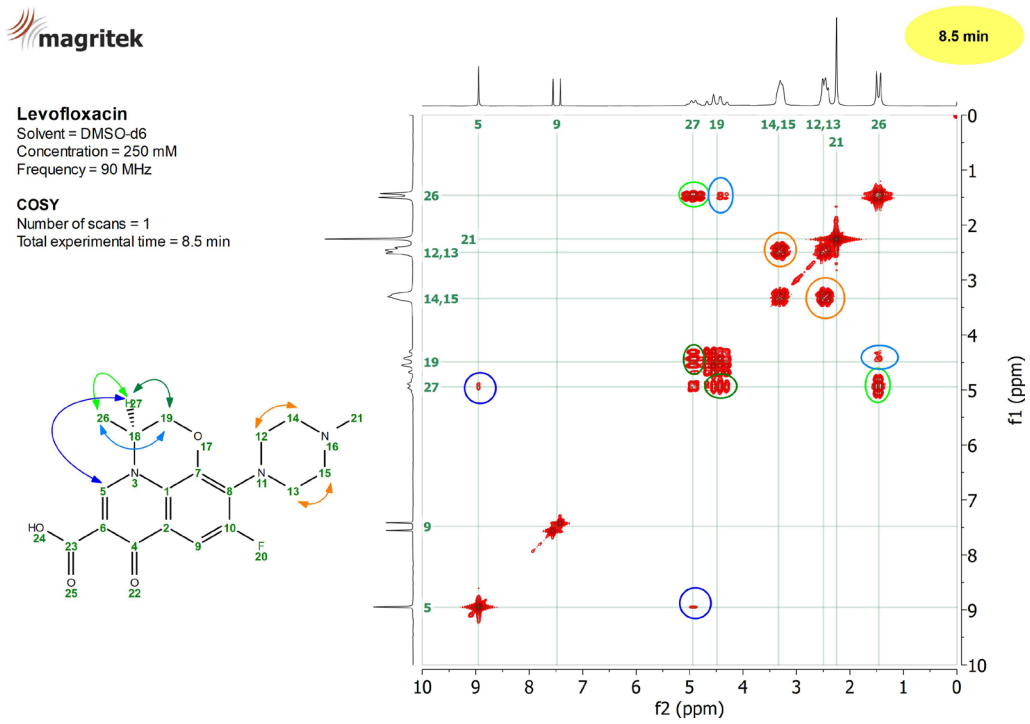

Figure 3: ^{1}H 2D COSY experiment of a 250 mM Levofloxacin sample in DMSO-d_6 acquired in 8.5 minutes on a Spinsolve 90 MHz system.

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 Levofloxacin sample in DMSO-d_6 acquired in 4 minutes. The measurement time was optimized applying NUS (non uniform sampling).

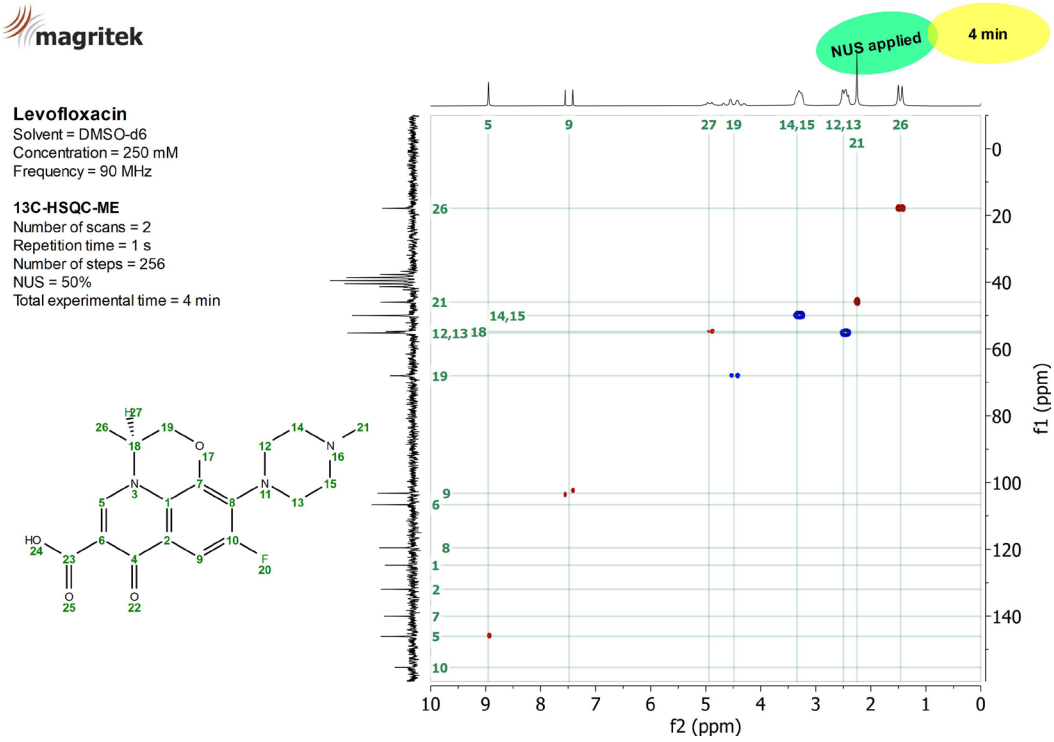

Figure 4: HSQC-ME spectrum of a 250 mM Levofloxacin sample in DMSO-d_6 showing the correlation between the ^{1}H (horizontal) and ^{13}C (vertical) signals.

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 5 with carbons 1, 4, 6, 18 and 23 (the sequence shows the correlation with quaternary carbons, too).

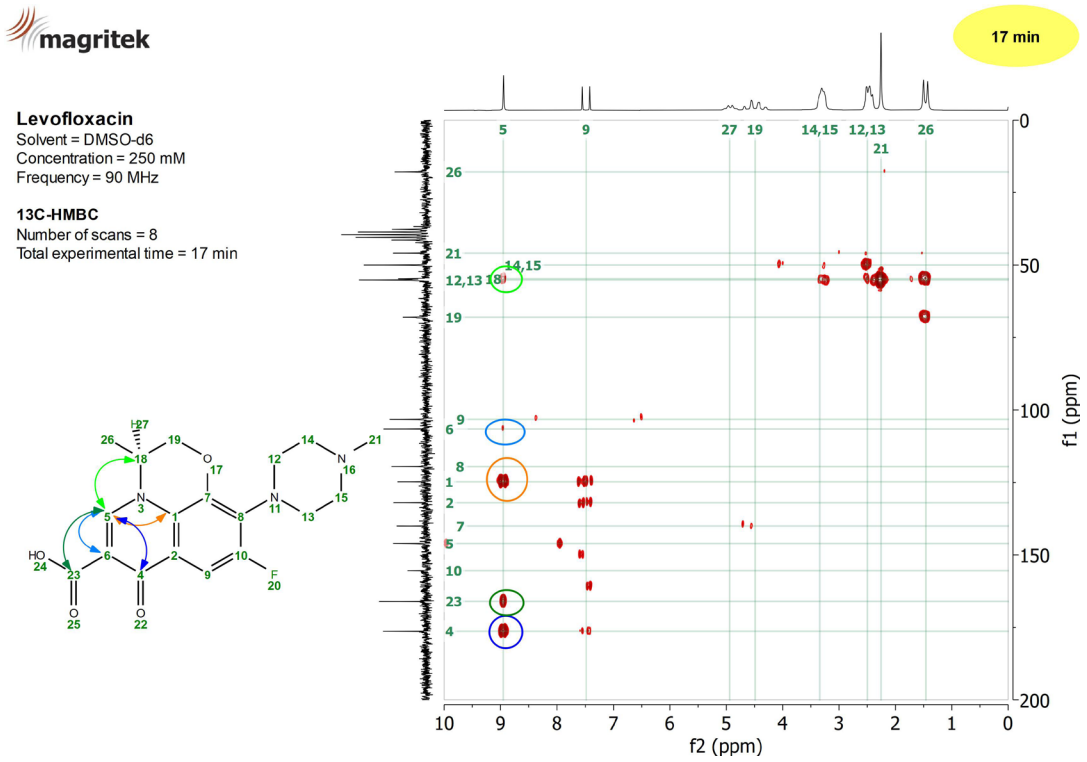

Figure 5: HMBC spectrum of a 250 mM Levofloxacin sample in DMSO-d_6 showing the long-range couplings between ^{1}H and ^{13}C nuclei.

1D Fluorine spectrum

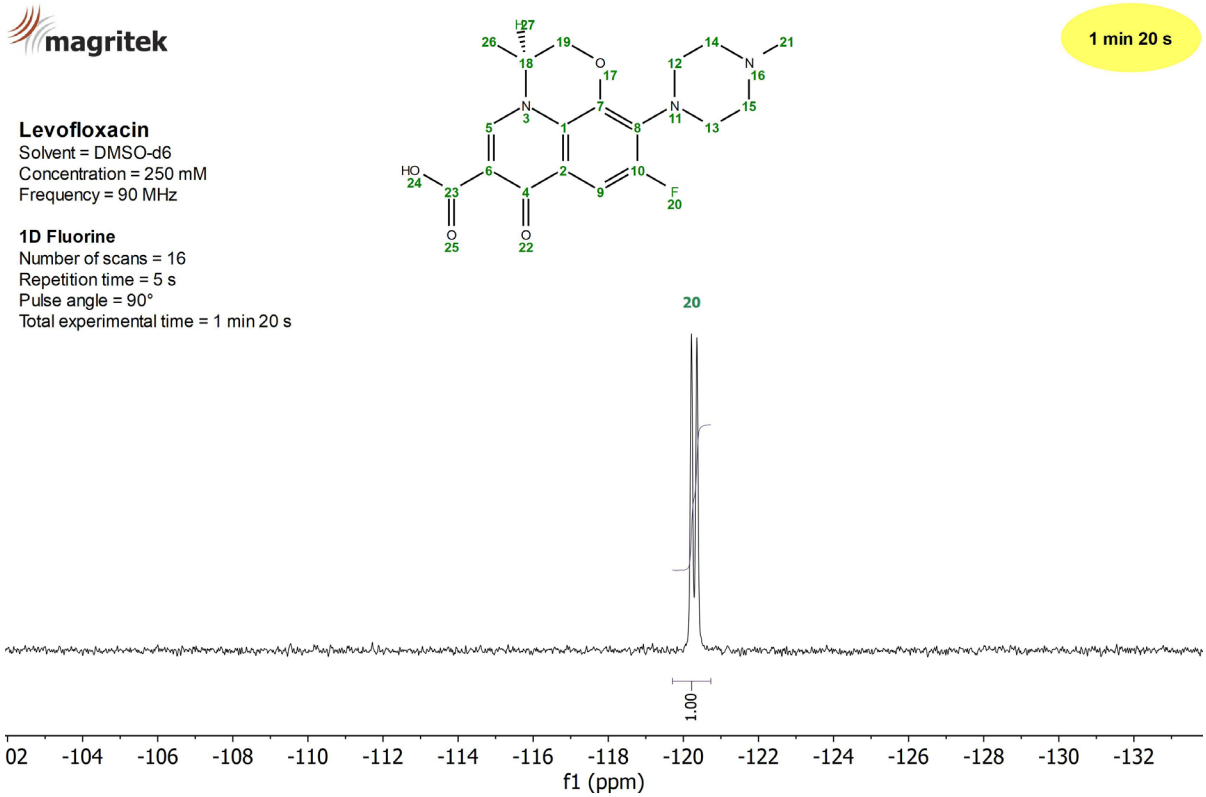

Figure 6: ^{19}F NMR spectrum of a 250 mM Levofloxacin sample in DMSO-d_6 measured on a Spinsolve 90 MHz system in 16 scans.

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. Figure 7 shows the full spectral range and depicts nicely all long-range correlations of the three nitrogen nuclei 3, 11 and 16. Here, nitrogen 3 couples with the protons at positons 5 and 26, nitrogen 11 couples with protons 12 and 13, as well as nitrogen 16 couples with protons 14,15 and 21, respectively. To note is the great SNR on the ^{15}N trace that indicates even reduced total measurement times to the 2 h 17 min shown.

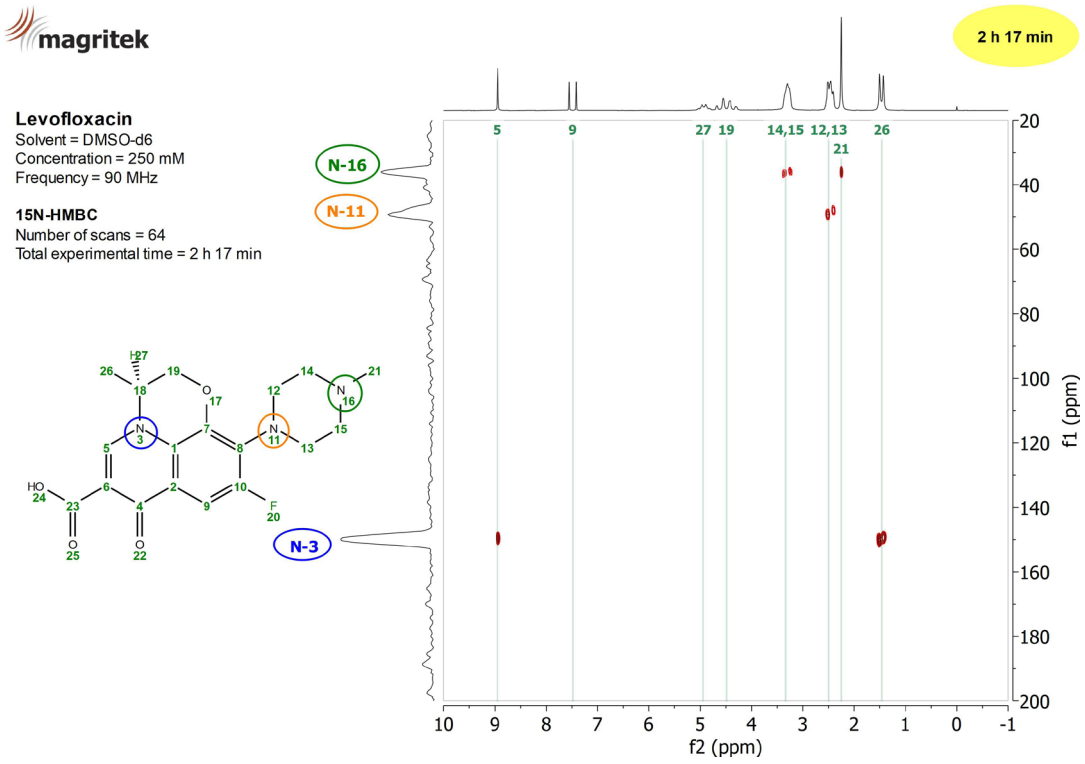

Figure 7: ^{15}N-HMBC spectrum of a 250 mM Levofloxacin sample in DMSO-d_6 showing the long-range couplings between ^{1}H and ^{15}N nuclei.