35Cl - 1H Heteronuclear Correlation MAS NMR Experiments for Probing Pharmaceutical Salts

Dinu Iuga, Emily K. Corlett, Steven P. Brown  
Department of Physics, University of Warwick, Coventry, CV4 7AL, UK

Magnetic Resonance in Chemistry

1. **NMR calculations** (For the calculations, the initial CIF file, the CIF file after geometry optimisation and the magres-files for the full crystal structure and isolated molecules; glycine∙HCl; L-Tyrosine∙HCl; cimetidine∙HCl; amitriptyline∙HCl and lidocaine∙HCl∙H2O)
2. **Raw files from NMR experiments**

**Figure 2 Figure 3 and Figure S1**: 35Cl – 1H (850 MHz) D-HMQC MAS (60 kHz) NMR spectra (Figure 2b and Figure 3 blue spectrum exp. 1; Figure 2a and Figure 3 red spectrum exp. 2; Figure S1 and Figure 3 green spectrum exp. 3).

**Figure 4** 35Cl – 1H (850 MHz) D-HMQC MAS (60 kHz) NMR spectra with different mixing times (Exp 1 – 100 s; Exp 2 – 200 s; Exp 3 – 300 s; Exp 4 – 400 s; Exp 5 – 500 s; Exp 6 – 600 s; Exp 7 – 700 s; Exp 8 – 800 s; Exp 9 – 900 s; Exp 10 – 1000 s)

**Figure 6** 1H (850 MHz) MAS (60 kHz) NMR with background suppresion (Exp 1 - glycine∙HCl; exp 2 - L-Tyrosine∙HCl; exp3 - cimetidine∙HCl; exp 4 - amitriptyline∙HCl and exp 5 - lidocaine∙HCl∙H2O)

**Figure 7** 35Cl (850 MHz) MAS (60 kHz) NMR with WUSRT presaturation of the satellite transitions (Exp 1 - glycine∙HCl; exp 2 - L-Tyrosine∙HCl; exp3 - cimetidine∙HCl; exp 4 - amitriptyline∙HCl and exp 5 - lidocaine∙HCl∙H2O)

**Figure 8** 35Cl-1H (850 MHz) PT-D-HMQC MAS (60 kHz) NMR spectra (Exp 1 - glycine∙HCl; exp 2 - cimetidine∙HCl; exp 3 - amitriptyline∙HCl and exp 4 - lidocaine∙HCl∙H2O)

**Figure S2 a**  35Cl – 1H (850 MHz) D-HMQC MAS (60 kHz) NMR spectra with different 1RCPL and 2RCPL set to 400 s (Exp 1 – 100 s; Exp 2 – 200 s; Exp 3 – 300 s; Exp 4 – 400 s; Exp 5 – 500 s; Exp 6 – 600 s; Exp 7 – 700 s; Exp 8 – 800 s; Exp 9 – 900 s; Exp 10 – 1000 s)

**Figure S2 b**  35Cl – 1H (850 MHz) D-HMQC MAS (60 kHz) NMR spectra with different 2RCPL and 1RCPL set to 400 s (Exp 1 – 100 s; Exp 2 – 200 s; Exp 3 – 300 s; Exp 4 – 400 s; Exp 5 – 500 s; Exp 6 – 600 s; Exp 7 – 700 s; Exp 8 – 800 s; Exp 9 – 900 s; Exp 10 – 1000 s)

1. **Simpson simulation files** (Figure 4b, Figure 5, Figure S3, Figure S4 and Figure S5).