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

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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).