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Green synthesis of furfural from xylose using choline chloride-based deep eutectic solvent and mechanistic insights

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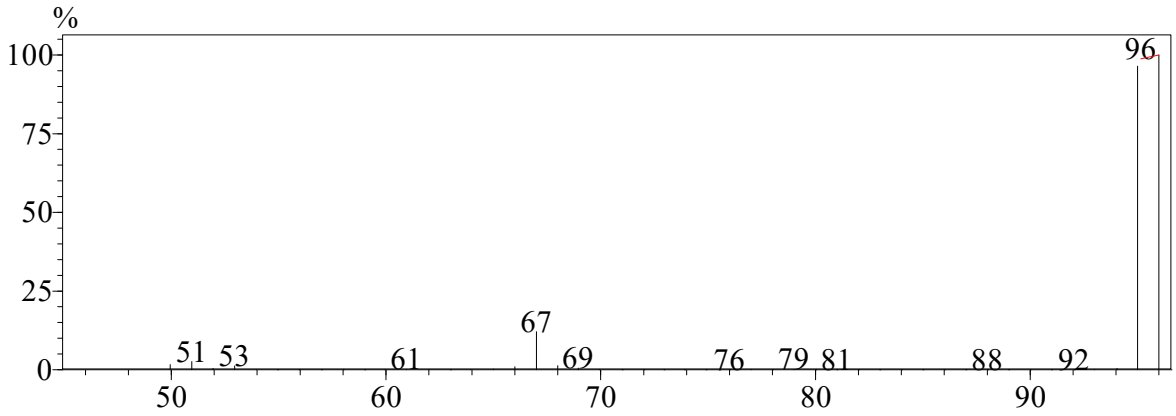

Fig. S1. Mass spectrum of furfural.

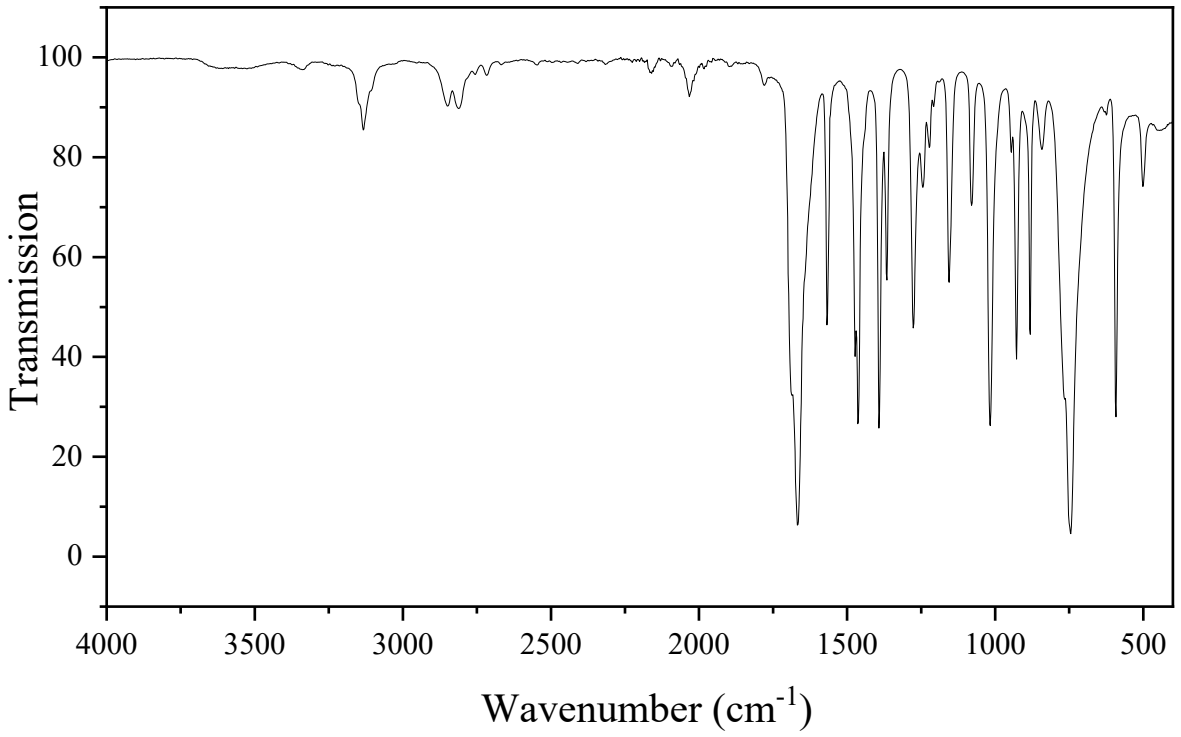

Fig. S2. FTIR Spectrum of furfural.

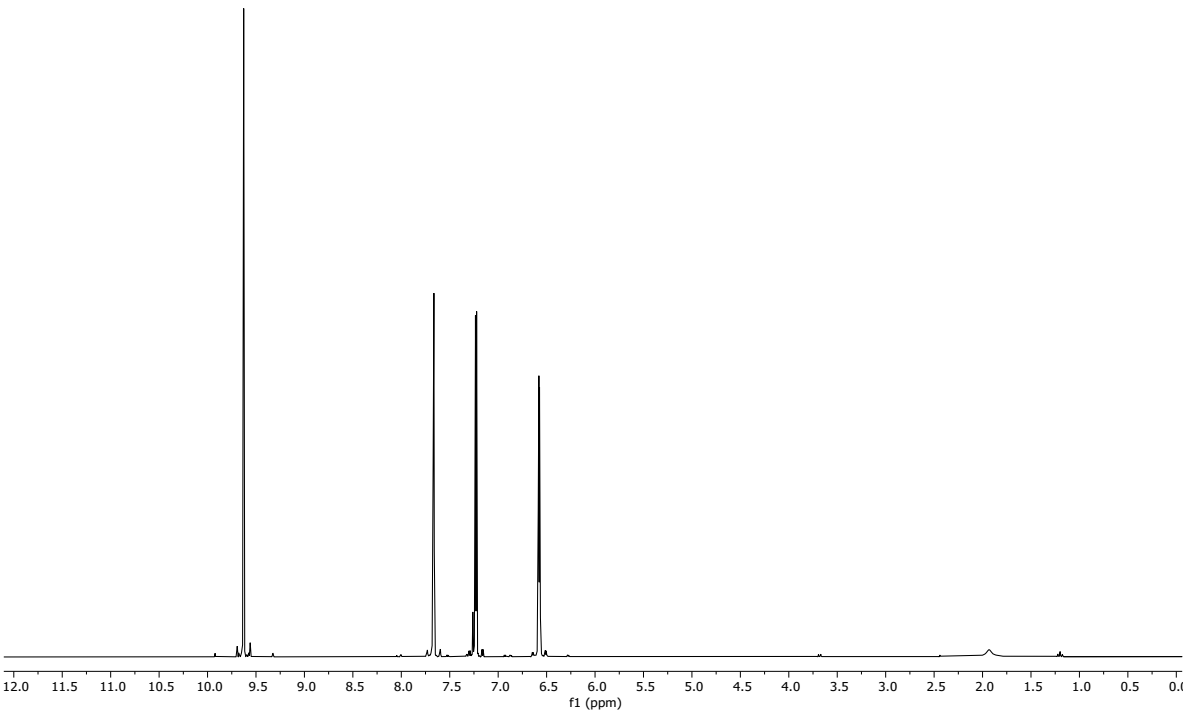

Fig. S3. ^{1}H NMR spectrum (300 MHz; $CDCl_3$) of furfural.

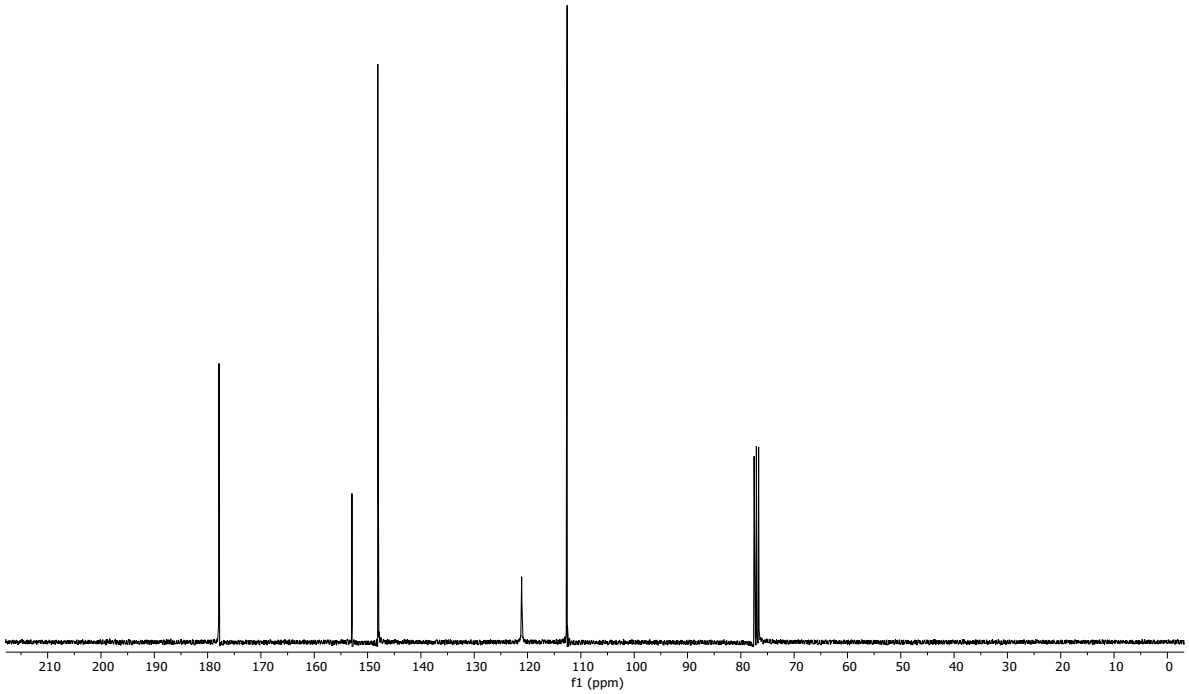

Fig. S4. ^{13}C NMR spectrum (75 MHz; $CDCl_3$) of furfural.

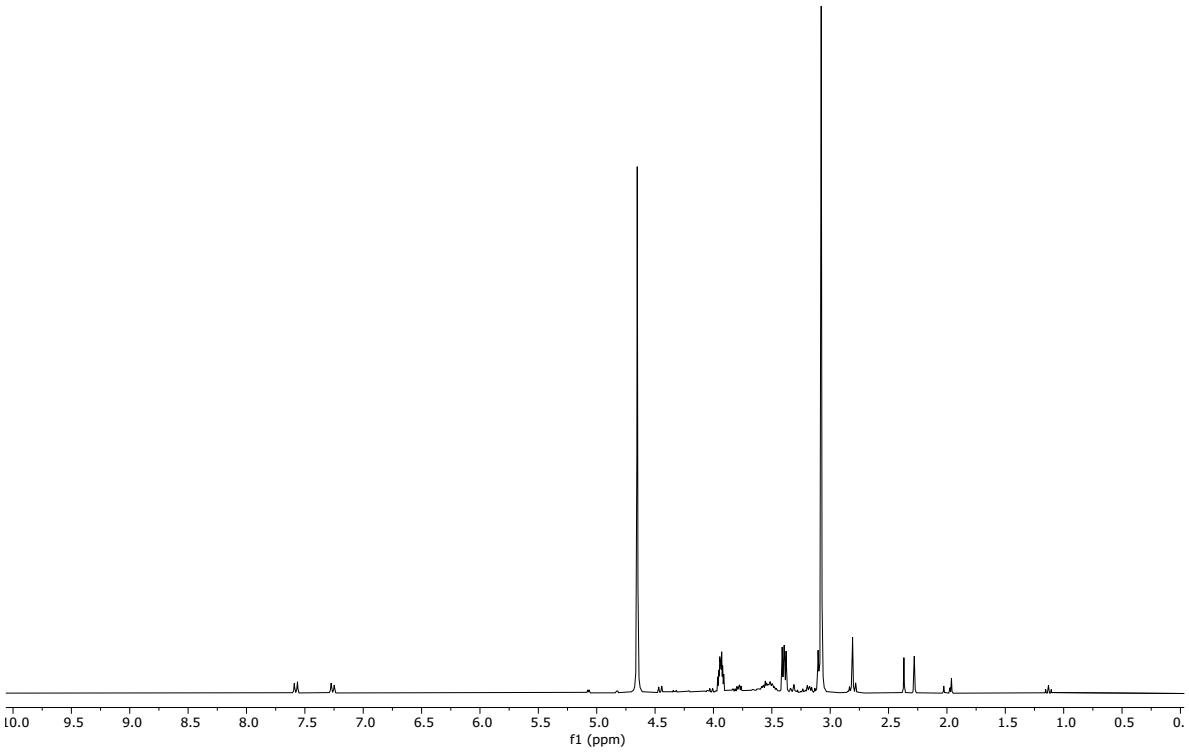

Fig. S5 ^{1}H NMR spectrum (300 MHz; D_2O) of DES.

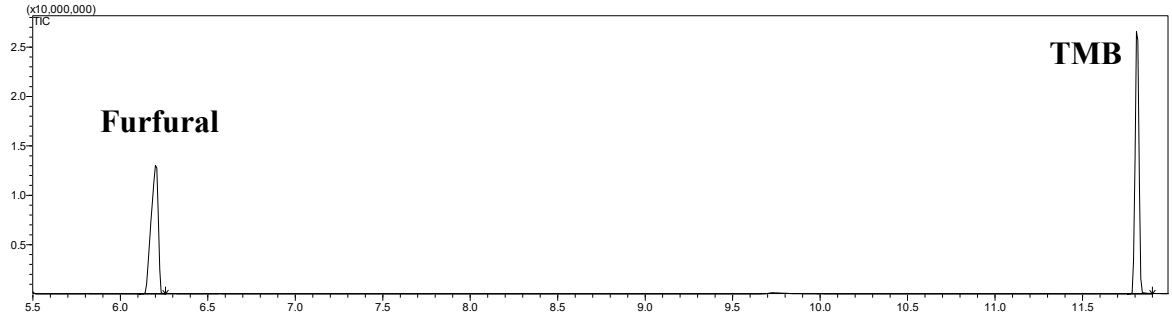

Fig. S6. Typical chromatogram of GC-MS analysis of furfural using TMB as internal standard.

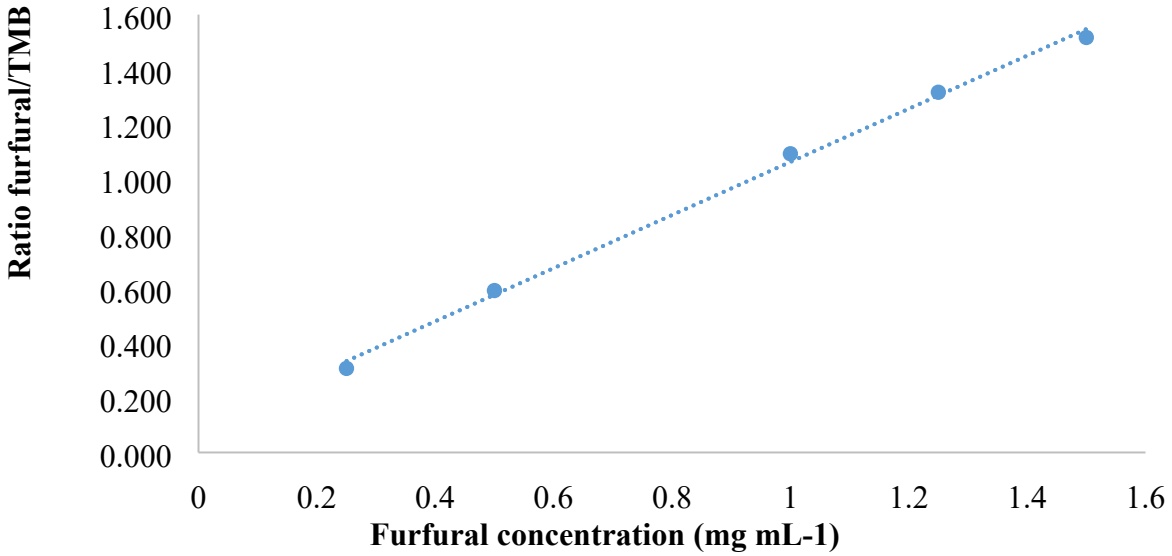

Fig. S7 Furfural calibration curve.

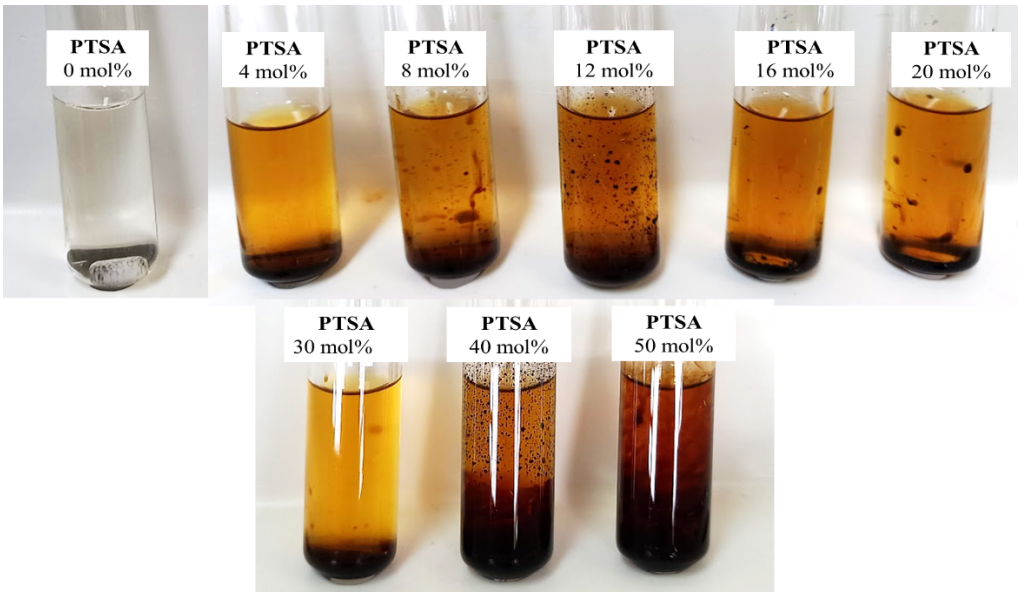

Fig. S8 Images of systems for synthesizing furfural from xylose using different amounts of PTSA.

Fig. S9 DES recovered after reuse.

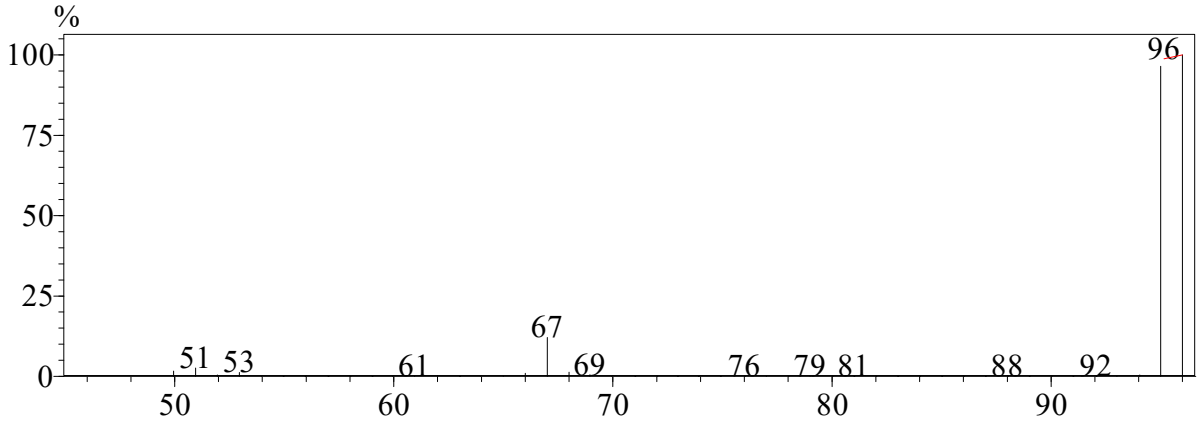

Fig. S10 Mass spectrum of furfural synthesized in D_2O.

DES characterization

In **Fig. S10** it is possible to observe a comparison between the ^{1}H NMR spectrum of the DES used in the synthesis of furfural and the ^{1}H NMR spectra of the pure components of this DES ([Ch]Cl, PTSA and xylose). Note that all signals from the three substances are present in DES. The difference observed is in the chemical shifts of the signals, as can be seen in **Table S1**: the signals from the [Ch]Cl hydrogens were shielded and changed to smaller chemical shifts, as well as to xylose, while the signals from the PTSA hydrogens went to unshielded and switched to larger chemical shifts. This small change in chemical shifts can be explained by hydrogen bond-type interactions between these molecules, which lead to the shielding/unshielding of their hydrogens and the change in their chemical environment.

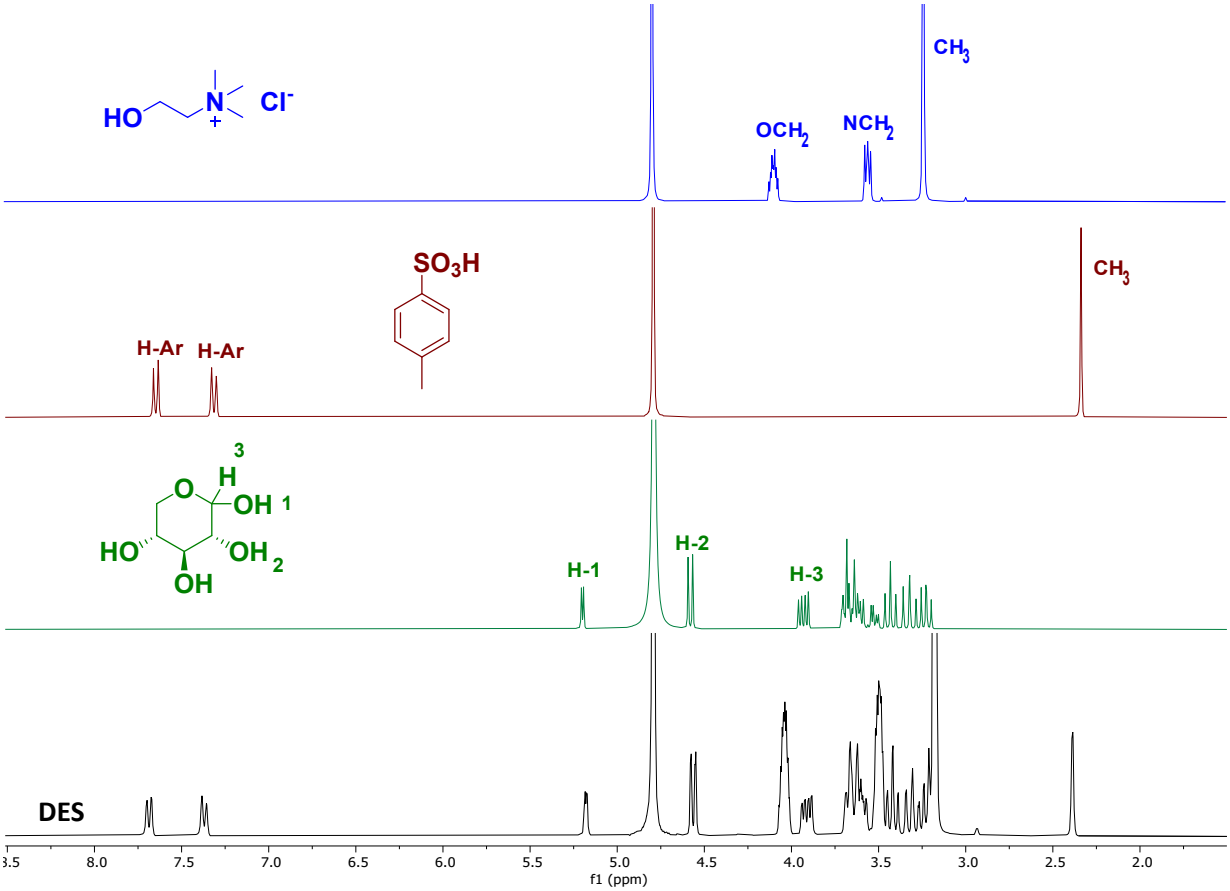

Fig. S11 ^{1}H NMR spectrum (300 MHz; D_2O) of isolated [Ch]Cl, PTSA, xylose and DES.

Table S1 Chemical shifts in ^{1}H NMR of isolated [Ch]Cl, PTSA and xylose signals and in DES.

Hidrogens	δ Isolated (ppm)	δ in DES (ppm)	$\Delta\delta$ (ppm) = $\delta_{DES} - \delta_{isolated}$
[Ch]Cl-CH_3	3.24	3.18	-0.06
[Ch]Cl-NCH_2	3.56	3.50	-0.06
[Ch]Cl-OCH_2	4.10	4.04	-0.06
PTSA-CH_3	2.34	2.39	0.05
PTSA-CH	7.31	7.37	0.06
PTSA-CH	7.64	7.68	0.04
Xylose	3.93	3.92	-0.01
Xylose	4.58	4.56	-0.02
Xylose	5.20	5.18	-0.02