Viscosity Measurements of the Ionic Liquid Trihexyl(Tetradecyl)Phosphonium Dicyanamide $[P_{6,6,6,14}][dca]$ Using the Vibrating Wire Technique

Supporting Information

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S.1. Preparation and characterization of trihexyl(tetradecyl)phosphonium dicyanamide[1]

The ionic liquid Trihexyl(tetradecyl)phosphonium dicyanamide ($[P_{6,6,6,14}][dca]$) was prepared following the procedure recommended by MacFarlane et al.[1] A solution of sodium dicyanamide (34.29g; 0.3851mol) in 0.200 dm^3 of distilled water was added to a solution of trihexyl(tetradecyl)phosphonium chloride $[P_{6,6,6,14}]Cl$ (0.100 kg; 0.193 mol) in 0.400 dm^3 of EtOH and left to stir at room temperature for 24 hours. The mixture was then concentrated under vacuum and washed with H_2O (3 × 0.200 dm^3). The organic layer (ionic liquid) was then eluted with CH_2Cl_2 in a column containing celite, decolourizing charcoal and SiO_2 gel. The solvent was then evaporated under vacuum (7 days; pressure approximately equal to 133.3 Pa at 323 K) to obtain the ionic liquid trihexyl(tetradecyl)phosphonium dicyanamide (94.95×10^{-3} kg; 82.3 %), a pale yellow oil.

A total amount of 273.42×10^{-3} kg of this ionic liquid was obtained using the same procedure (yield: 84.1 %).

In Figure S1 the ^{13}C NMR spectral data, obtained on a Bruker Avance 400 Ultrashield spectrometer are shown. Table S1 summarizes the data obtained in the ^{13}C and ^{1}H NMR spectra obtained with that spectrometer.

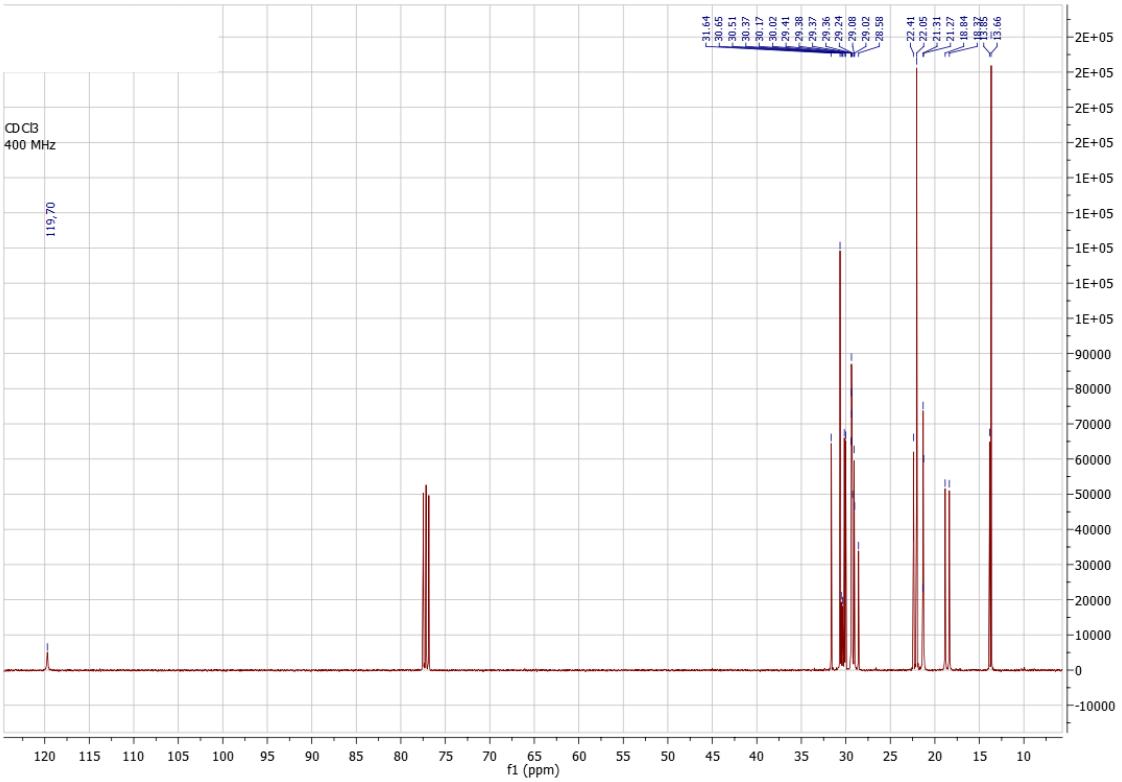

Figure S1. ^{13}C NMR ($CDCl_3$, 100 MHz): $[P_{6,6,6,14}][dca]$ Spectral Data

Table S1. Nuclear magnetic Resonance (NMR) analysis results for the $[P_{6,6,6,14}][dca]$ sample.

^{1}H NMR (400 MHz, $CDCl_3$) δ:	0.84 (m, 12H); 1,27 (m, 40H); 1.47 (m, 8H); 2.13 (m, 8H).
^{13}C NMR (100 MHz, $CDCl_3$) δ:	13.66; 13.85; 18.37; 18.84; 21.27; 21.31; 22.05; 22.41; 28.58; 29.08; 29.24; 29.37; 30.02; 30.17; 30.37; 30.51; 30.65; 31.64; 119.70.

S.2. Vibrating Wire Frequency Response for Different External Noise Levels

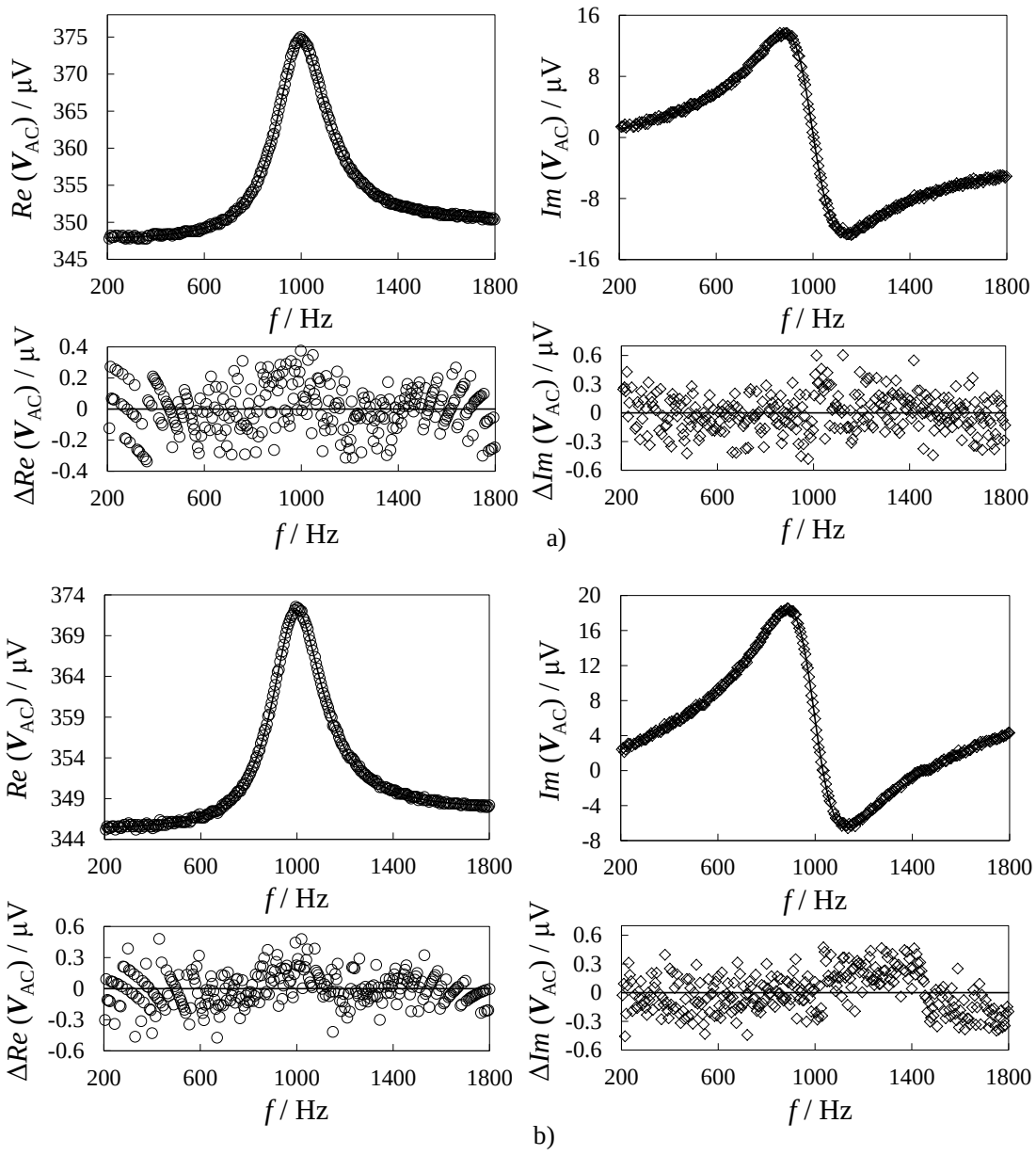

Figure S2. In-phase, $Re(\boldsymbol{V}_{AC})$, and quadrature, $Im(\boldsymbol{V}_{AC})$, experimental voltage curves obtained with a sample of $[P_{6,6,6,14}][dca]$ at 298.20 K and the corresponding fitting eq 4 (straight line). The deviations in the lower plots are the differences between the experimental points and those calculated by eq 4. The experimental curve obtained in experiment b) was carried out with higher external noise than in a). The viscosity obtained differ by less than 0.3 %.

References:

[1] D. R. MacFarlane, S. A. Forsyth, J. Golding, G. B. Deacon, *Green Chemistry*, **2002**, 4, 444–448.