Synthesis of Ionic Liquid/TOP and ionic liquid passivated CdSe Nanoparticles

All reagents were obtained from Sigma-Aldrich and used as received, using standard Schlenk line techniques. In a typical reaction, cadmium acetate dihydrate (0.023 g, 8.6 x 10^-5 moles) was dissolved in 4 g trihexyl(tetradecyl)phosphonium bis 2,4,4-trimethylpentylphosphinate, followed by rigorous degassing before being exposed to an inert atmosphere. The temperature was raised to 240 °C, followed by injection of 2.5 mls of a 1.3 M trioctylphosphine (TOP) selenide solution. The reaction changed colours to dark red/brown over 5 minutes. The temperature dropped to 220 °C, and was maintained between 200 and 220 °C over 30 minutes. The reaction flask was then removed from the heat, allowed to cool to ca. 60 °C where upon which 20 mls of methanol was added, causing an immediate precipitation which was isolated by centrifugation. The precipitate was easily redispersed in toluene, which was centrifuged further to remove solid waste products. The brown/red solution was stored in ambient condition for months with no evidence of agglomeration.

The majority of the paper will report on particles prepared by this method, however, for comparison we have prepared particles using just the ionic liquid (no TOP). In this case, cadmium acetate dihydrate (0.023 g, 8.6 x 10^-5 moles) and selenium powder (0.25 g, 3.25 x 10^-3 moles) were mixed in 4 g trihexyl(tetradecyl)phosphonium bis 2,4,4-trimethylpentylphosphinate, followed by rigorous degassing before being exposed to an inert atmosphere. The temperature was raised to 240 °C, then cooled to room temperature before isolation as described above.
NMR of ionic liquid

$^{31}\text{P}$ solution NMR

$^1\text{H}$ solution NMR

NMR of ionic liquid/TOP capped CdSe

$^{31}\text{P}$ solution NMR

$^1\text{H}$ solution NMR

$^{31}\text{P}$ NMR of ionic liquid capped CdSe
FTIR of ionic liquid (grey) and ionic liquid/TOP capped CdSe (black)
HRTEM images of ionic liquid/TOP capped particles

HRTEM images of ionic liquid capped particles

Cartoon demonstrating suggested modes of ligand coordination.
Absorption spectra of Ionic Liquid capped CdSe