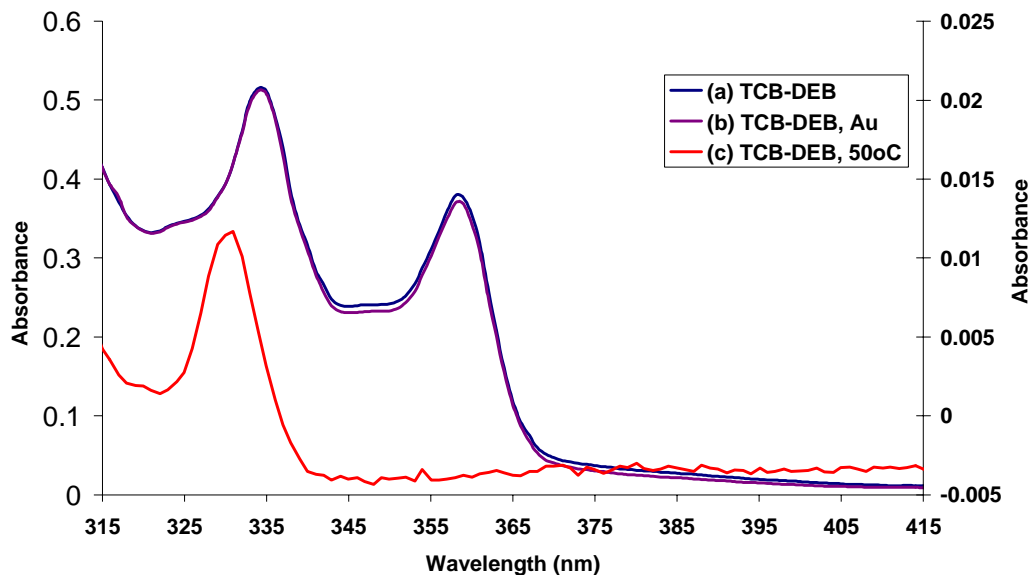
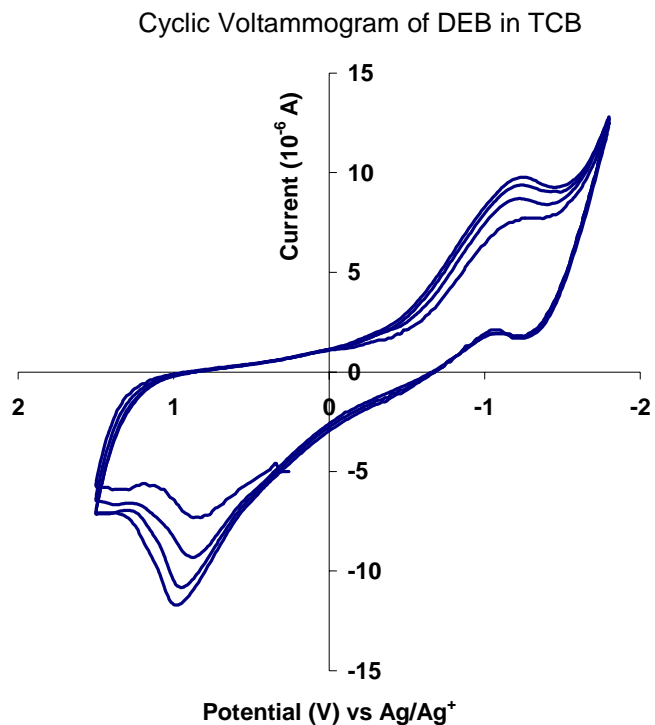


Supplementary Figure 1: Conductance histograms of six compounds (1, 4, 5, 6, 7, 8) and control studies with TCB.

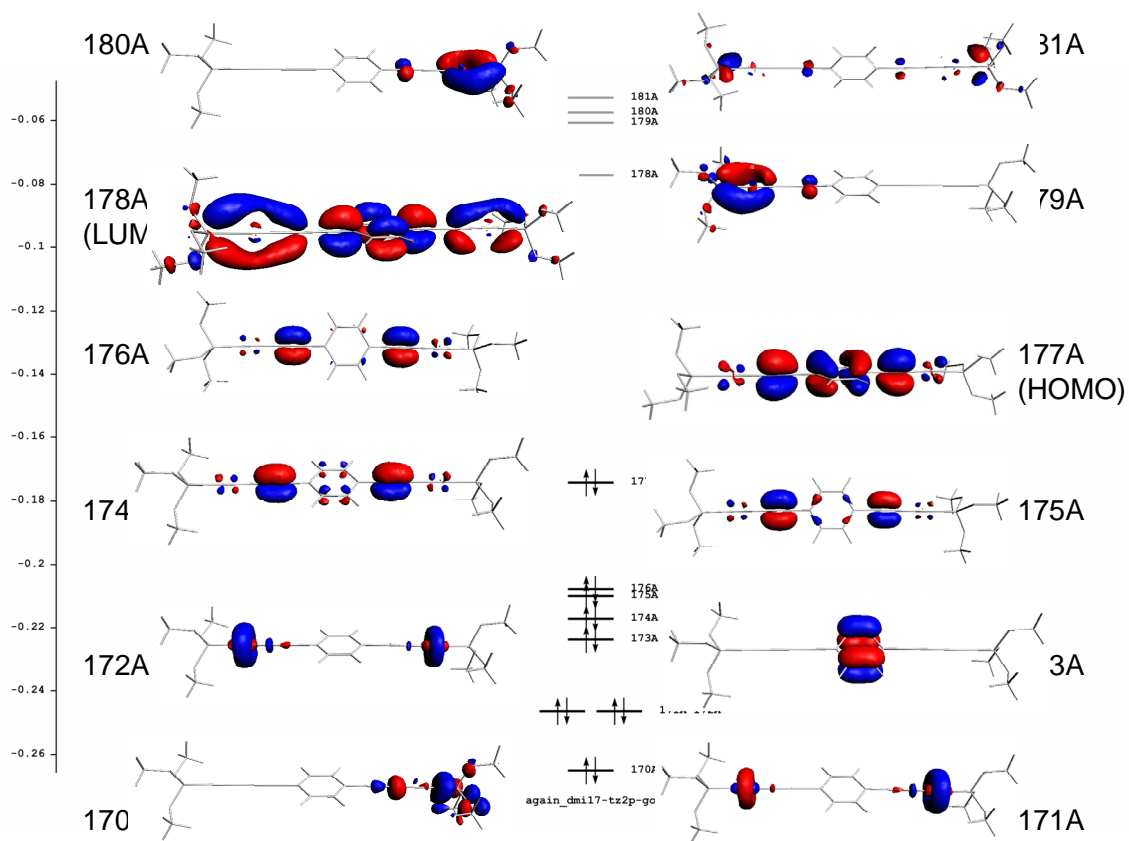
UV-Vis of 1,4-diethynylbenzene in TCB, Effect of Particulate Gold and Heat on available monomer.



Supplementary Figure 2. (a) UV-Vis absorbance of a 0.2mM solution of DEB in TCB (b) after stirring overnight with powdered gold (average particle size ~ 10 microns) (c) after heating to 50°C for 3 hours, black ppt. observed (right hand y-axis) interpreted as deprotonation of the alkylnyl functional groups and subsequent polymerization of DEB in TCB.



Supplementary Figure 3. Cyclic Voltammogram of 0.1M DEB/ 0.01M TBAH solution in TCB; Pt disk working electrode, Pt wire counter electrode and Ag/Ag⁺ reference electrode at a scan rate of 100 mV/s. After 3 cycles an insoluble black film is observed, coating the Pt disk working electrode. This observation in conjunction with an increase in the diffusion limited current at both the reduction and oxidation potentials are indicative of the irreversible 2-D nucleation and growth of poly-phenylacetylene.³⁶ The large potential difference between the observed reduction potential and the oxidation potential may be in part due to the poor mass transport of the DEB monomer in TCB.



Supplementary Figure 4: Expanded MO diagram for **1** from DFT calculations.