# Supplementary Information

## *Oxidative C-C coupling of 2,6-di-tert-butylphenol in aqueous media via catalytically active molybdate surfactants*

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## *General Considerations*

Cumene hydrogen peroxide, *tert*-butyl hydrogen peroxide, CTAB, methyliminodiacetic acid, sodium molybdate, sodium methoxide (25% wt. in MeOH), sodium monochloroacetate, N-dodecylamine, N-tetradecylamine and 2,6-di-*tert*-butylphenol were purchased from Sigma Aldrich and used as received. N-aminodecane was supplied by TCI America and hydrogen peroxide (30% w/v) from Caledon Laboratories. The long chain ligands, $C_nH_{2n+1}N(CH_2COOH)_2$, or $C_n$IDA, were synthesized according to published procedures.[1] Infrared (IR) spectra were recorded with a Perkin Elmer FT-IR System Spectrum GX spectrometer (4,000-600 $cm^{-1}$). Elemental analyses (EA) were performed on an EAS 1108 apparatus from Fisons Instruments SPA. Neutron activation analysis was used to determine the %Mo in the sample using a SLOWPOKE-II reactor at the Royal Military College of Canada. Irradiation took place at an inner irradiation site, at half power, giving a flux of approximately $5x10^{11}$ neutrons · $cm^{-2}$ · $second^{-1}$. A relative analysis was performed, using aqueous ICP-MS NIST Traceable standards. Detection was performed on an EG&G ORTEC High Purity GMX Germanium Detector, with a Beryllium window. Signal processing was performed by an ORTEC digiDART multichannel spectrometer. Ultraviolet-visible spectrometry measurements were taken using a Hewlett Packard 8453 UV-VIS spectrophotometer. Conductivity measurements were performed using an Oakton Con-6 Conductimeter, calibrated to a 1413 µS/cm standard.

## *Preparation of $Na_2MoO_3C_{12}IDA(H_2O)_2$*

A solution of $Na_2MoO_4$ (0.1204 g, 0.4976 mmol) in water (10 mL) was added to a stirring suspension of the acidified long chain iminodiacetic acid, $H_2C_{12}$IDA, (0.1500 g, 0.4977 mmol). The mixture was stirred until the ligand was completely solubilized, giving a clear colourless solution. Concentration of the solution (which was difficult due to the formation of surfactant bubbles), followed by addition of THF resulted in the precipitation of a white solid, identified as $Na_2MoO_9C_{16}H_{33}N$. Anal. calc. (found) (%): C, 36.58 (37.34); H, 6.33 (6.07); N, 2.67 (2.77); Mo, 18.26 (19.61). $^1$H NMR (20°C, 300 MHz, $D_2O$): δ 0.84 (m, 3H, -C$H_3$), 1.26 (bm, 18H, -C$H_2$-), 1.41 (br, 2H, -C$H_2$C$H_2$N), 2.62 (m, 2H, -C$H_2$N), 3.27 (s, 4H NC$H_2$COO). IR (Nujol mull, $cm^{-1}$): ν 3455 (s), 2941 (s), 2858 (s), 2733 (w), 1645 (s), 1466 (s), 1411 (m), 1377 (s), 1239 (w), 1227 (w), 1130 (w), 1095 (m), 1064 (w), 1040 (w), 976 (w), 944 (m), 902 (m), 874 (s), 853 (m), 756 (w), 725 (m), 572 (w).

*Conductivity measurements for determination of the critical micelle concentration (CMC) of* $Na_2MoO_3C_{12}IDA$.

Temperature-corrected conductivity measurements were taken for several solutions of the sample at concentrations between 10 and 0.01 mM. Intersection of the two trendlines on the plot of conductivity vs concentration give a CMC value of 1.7 mM.

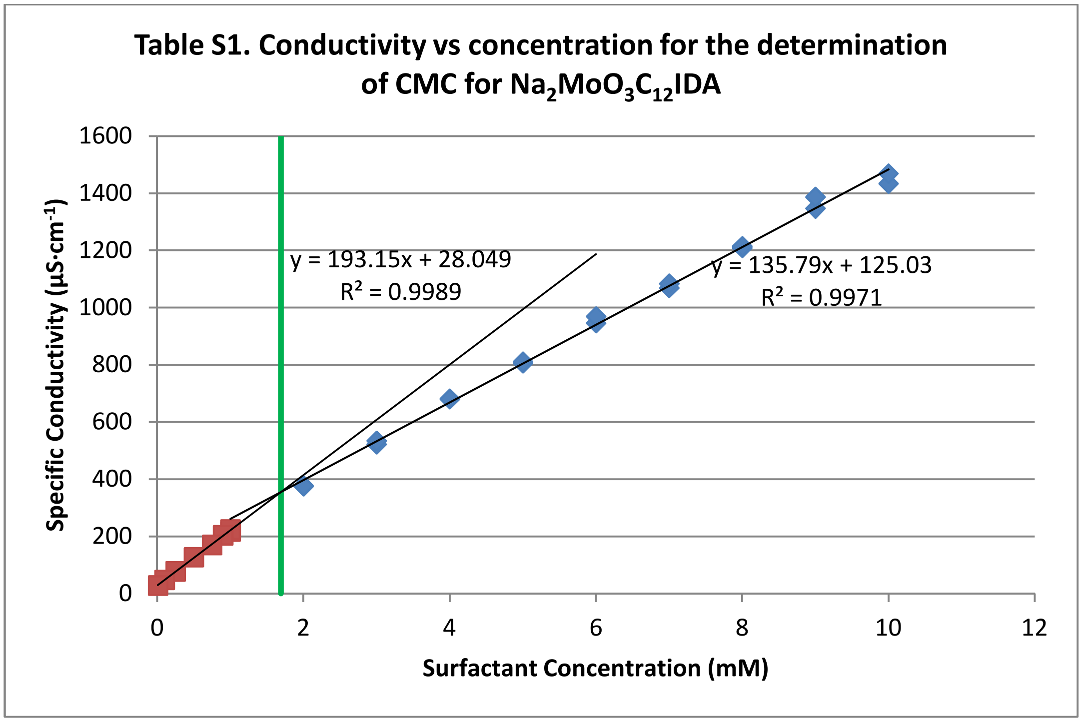

**Table S1. Conductivity vs concentration for the determination of CMC for $Na_2MoO_3C_{12}IDA$**

*Oxidation of 2,6-di-tert-butylphenol (DTBP)*

Several conditions were attempted for the catalysis. Solutions were prepared containing 4.0 mM CTAB and 1.0 mM DTBP with varying amounts of catalyst (as seen in Table 1), most commonly 0.2 mM. To the stirring solution, various equivalents of $H_2O_2$ were added (concentrations reported above are after addition of all reactants). The reactions were allowed to stir, covered, and aliquots taken at different time intervals to be analyzed by UV-VIS spectrophotometry at 418 nm for DPQ ($\varepsilon = 39\ 500\ L\ mol^{-1}\ cm^{-1}$).

*References:*

1) A. Stein, H. P. Gregor and P. E. Spoerri, *J. Am. Chem. Soc.,* 1955, **77**, 191; L. Haggman, C. Lindblad, H. Oskarsson, A.-S. Ullstrom and I. Persson, *J. Am. Chem. Soc.*, 2003, **125**, 3631.