Supplementary Information

Oxovanadium(V)-Catalyzed Deoxygenative Homocoupling Reaction of Alcohols

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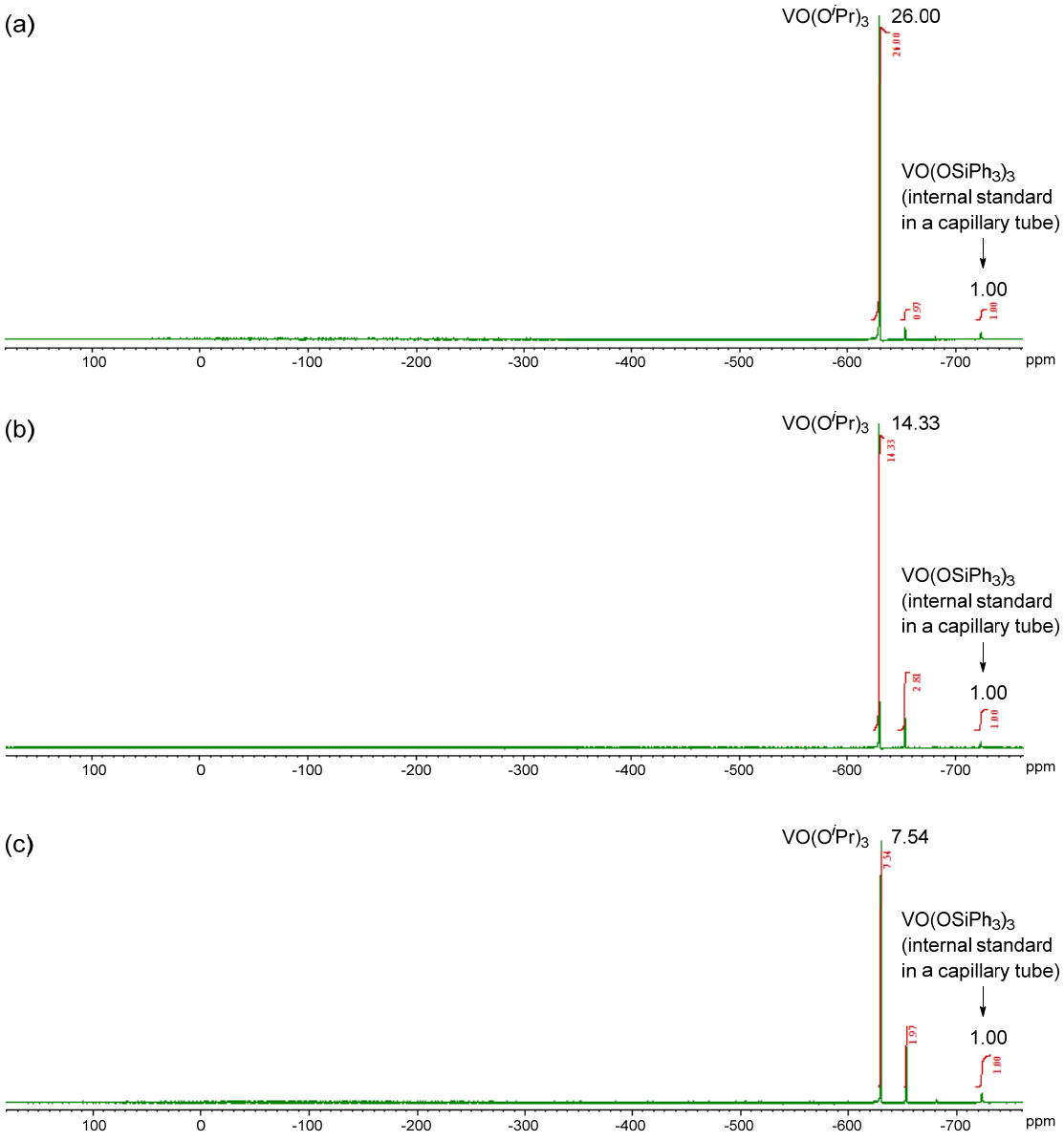
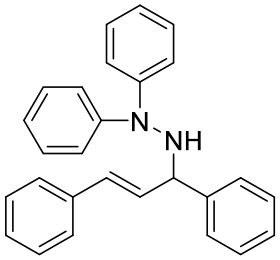


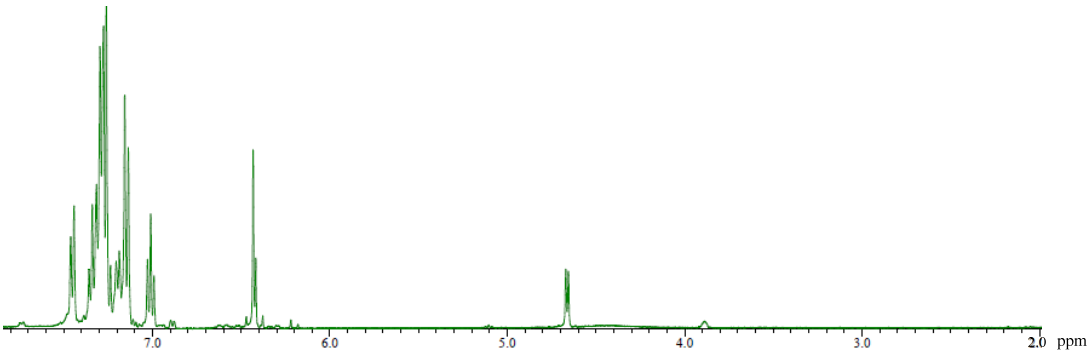
Fig. S1 ^{51}V NMR spectra of VO(O^{i}Pr)$_3$ (4.6 μL, 0.02 mmol) in the presence of 1,2-diphenylhydrazine (36.7 mg, 0.20 mmol), ^{i}Pr$_2$NEt (1.7 μL, 0.01 mmol) and MS3A (25 mg) in toluene-d$_8$ (500 μL): (a) at 20 °C for 10 min; (b) at 100 °C for 1 h; (c) at 100 °C for 12 h.

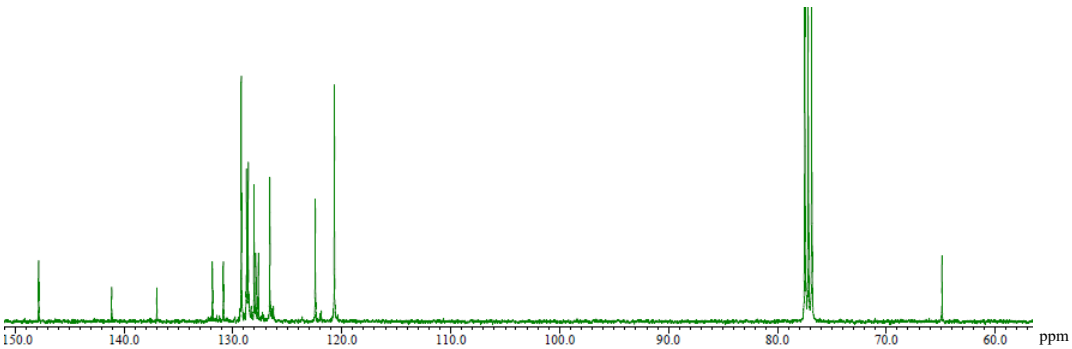
Spectroscopic data of products

(*E*)-2-(1,3-Diphenylallyl)-1,1-diphenylhydrazine (**2a**)

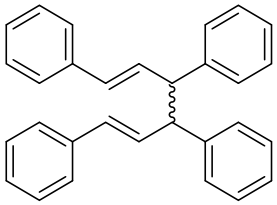


^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46-6.99 (m, 20H), 6.47-6.38 (m, 2H), 4.67 (d, J = 5.9 Hz, 1H), 4.40 (br s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) 147.8, 141.1, 137.0, 131.9, 130.9, 129.2, 128.7, 128.6, 128.1, 127.9, 127.7, 126.6, 122.4, 120.7, 64.9 ppm; HRMS (FAB) *m*/*z* Calcd. for $C_{27}H_{24}N_2$ (M^+), 376.1934; Found, 376.1944.

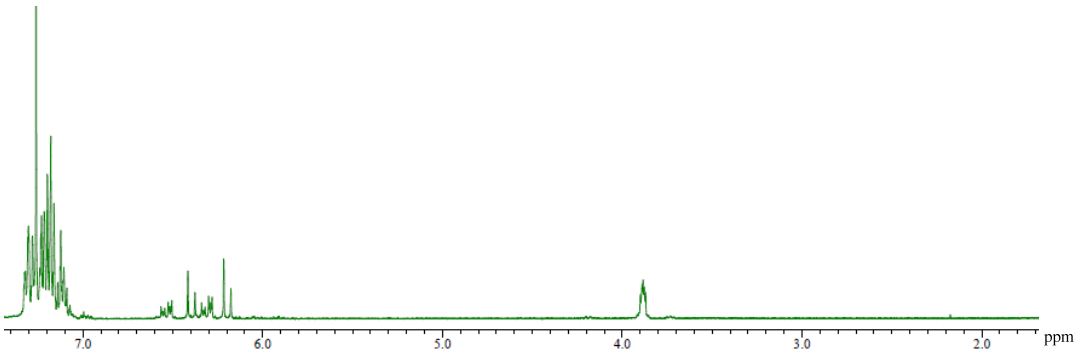


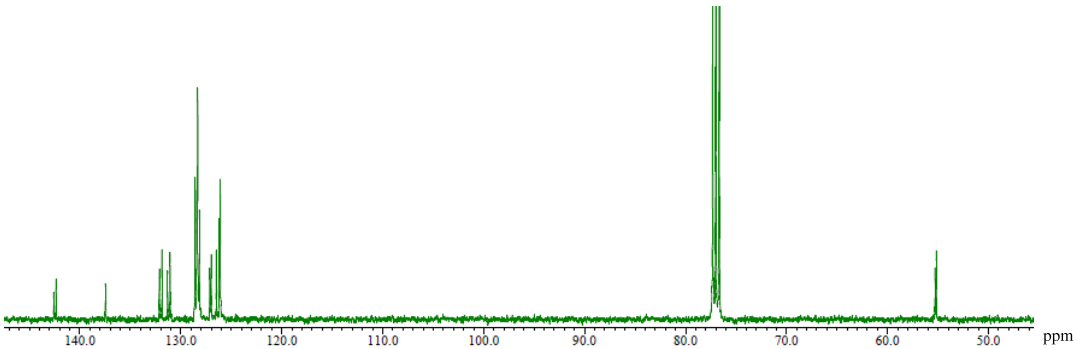


1,3,4,6-Tetraphenyl-1,5-hexadiene [CAS Registry No. 204578-86-9] (**3a**)

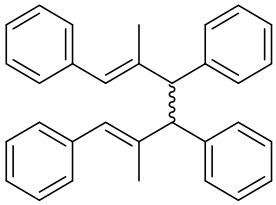


Mixture of stereoisomers: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.07 (m, 20H), 6.57-6.51 (m, 1H), 6.40 (d, J = 15.8 Hz, 1H), 6.34-6.28 (m, 1H), 6.20 (d, J = 15.8 Hz, 1H), 3.92-3.85 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) 142.6, 142.4, 137.5, 137.4, 132.1, 131.9, 131.3, 131.1, 128.6, 128.42, 128.37, 128.35, 128.32, 128.2, 127.1, 127.0, 126.5, 126.2, 126.14, 126.09, 55.3, 55.2 ppm; HRMS (EI) m/z Calcd. for $C_{30}H_{26}$ (M^+), 386.2029; Found, 386.2025.

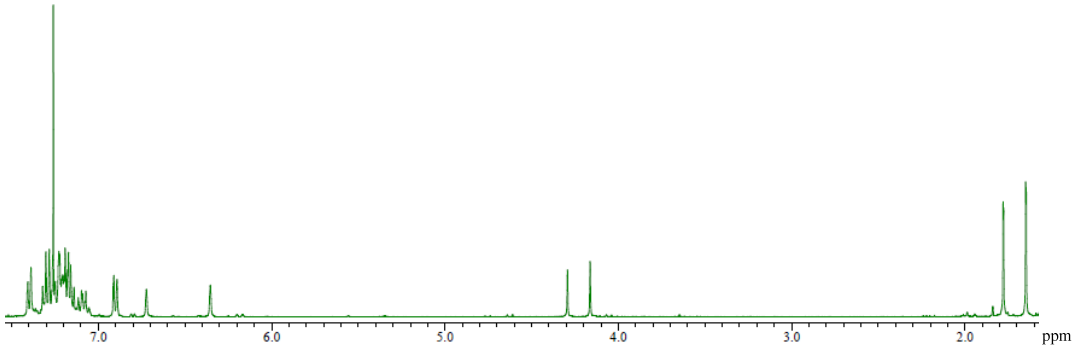


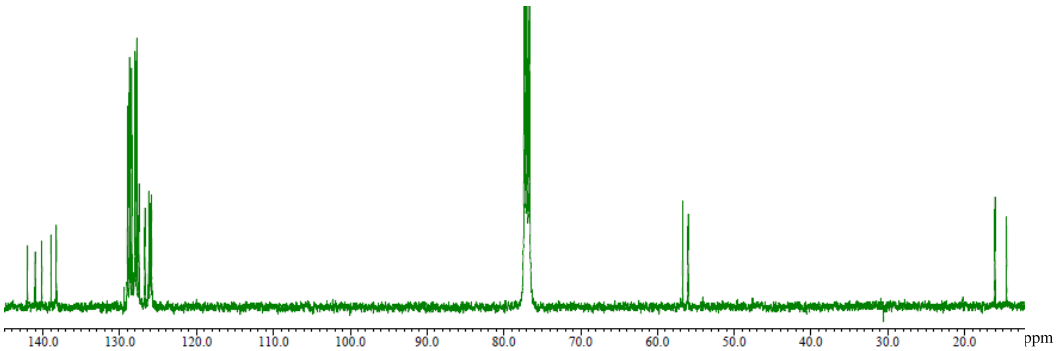


2,5-Dimethyl-1,3,4,6-tetraphenyl-1,5-hexadiene (**3b**)

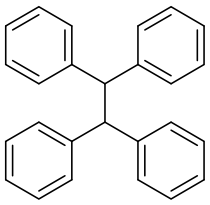


Mixture of stereoisomers: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41-7.05 (m, 18H), 6.91-6.89 (m, 2H), 6.72 (s, 1H), 6.35 (s, 1H), 4.29 (s, 1H), 4.16 (s, 1H), 1.78 (s, 3H), 1.65 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) 142.0, 141.0, 140.2, 139.0, 138.28, 138.26, 129.0, 128.8, 128.7, 128.5, 128.1, 127.99, 127.95, 127.8, 127.5, 126.7, 126.2, 126.1, 126.0, 125.9, 56.7, 56.0, 16.1, 14.5 ppm; HRMS (EI) *m*/*z* Calcd. for $C_{32}H_{30}$ (M^+), 414.2348; Found, 414.2340.

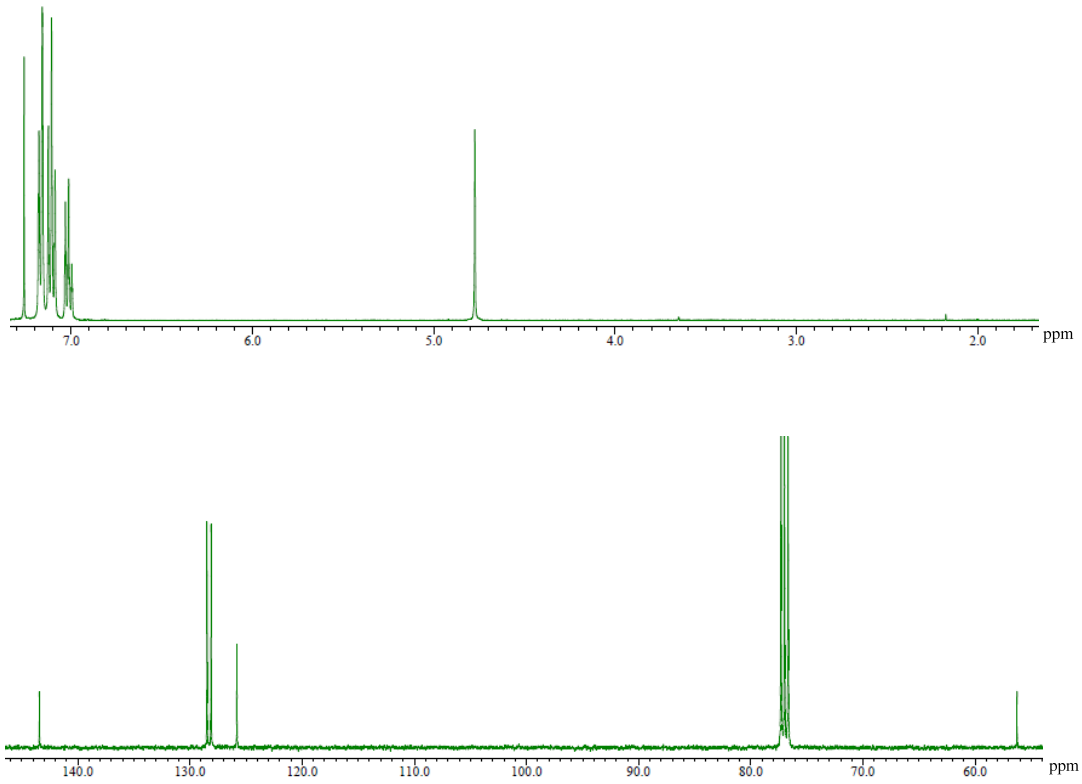




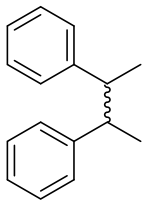
1,1,2,2-Tetraphenyl-ethane [CAS Registry No. 632-50-8] (**3c**)



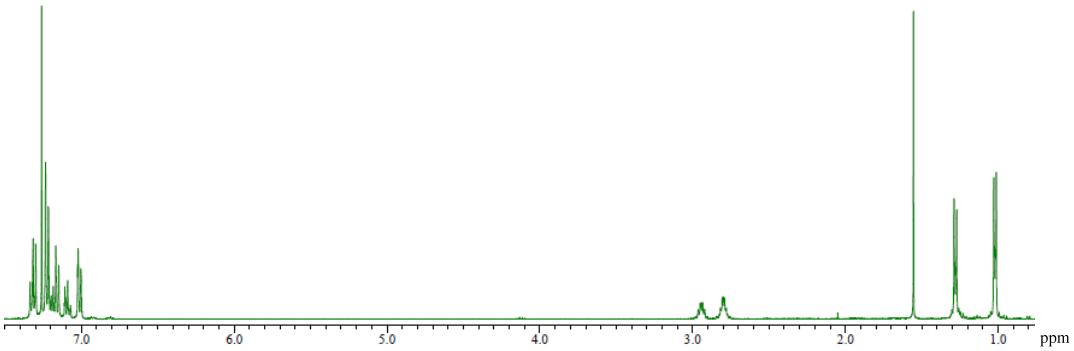
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 (d, J = 7.3 Hz, 8H), 7.11 (t, J = 7.3 Hz, 8H), 7.01 (t, J = 7.3 Hz, 4H), 4.77 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) 143.4, 128.5, 128.1, 125.8, 56.3 ppm; HRMS (EI) m/z Calcd. for $C_{26}H_{22}$ (M^+), 334.1716; Found, 334.1718.

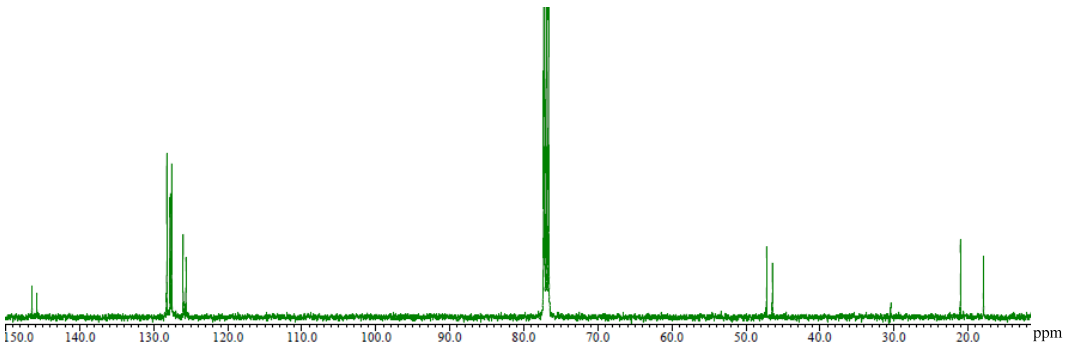


2,3-Diphenyl-buthane [CAS Registry No. 5789-35-5] (**3d**)

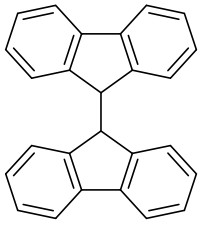


Mixture of stereoisomers: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34-7.00 (m, 20H), 2.98-2.90 (m, 1H), 2.84-2.76 (m, 1H), 1.28 (d, J = 6.9 Hz, 3H), 1.02 (d, J = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) 146.5, 145.8, 128.3, 127.8, 127.7, 127.6, 126.0, 125.7, 47.2, 46.4, 21.0, 17.9 ppm; HRMS (EI) m/z Calcd. for $C_{16}H_{18}$ (M^+), 210.1403; Found, 210.1406.

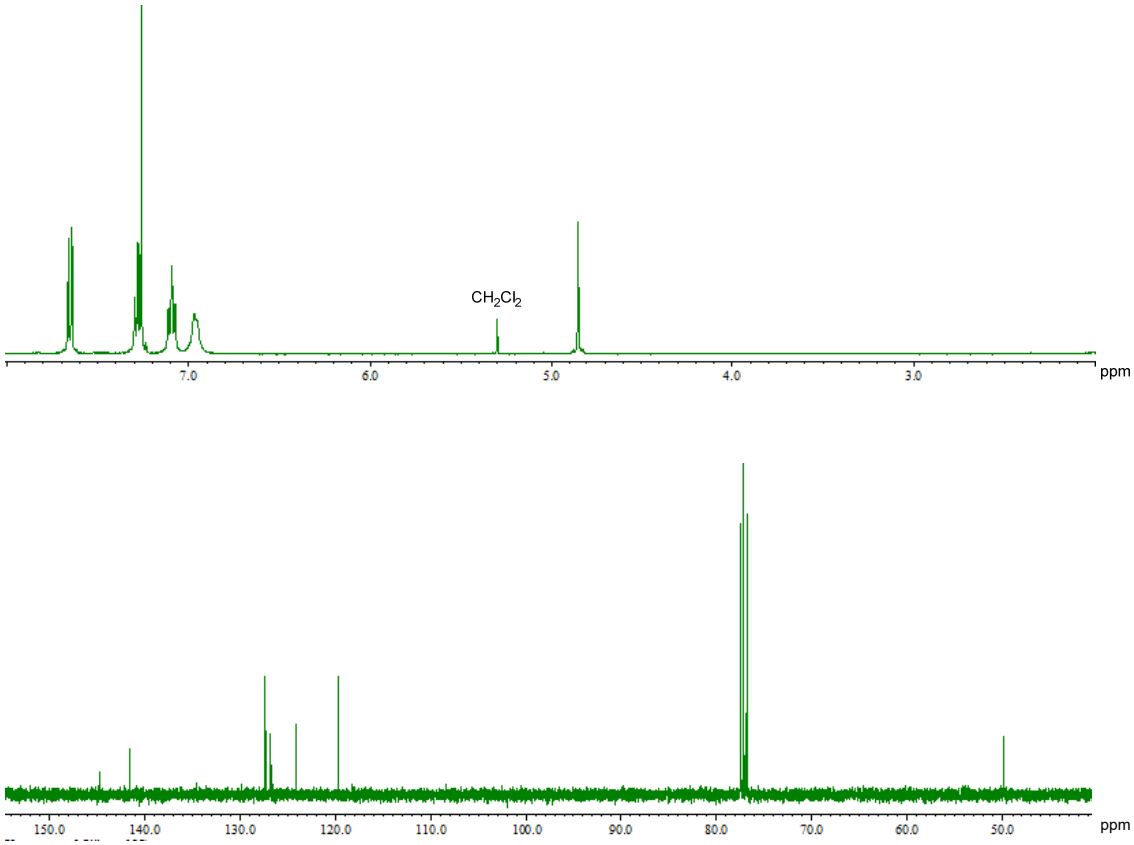




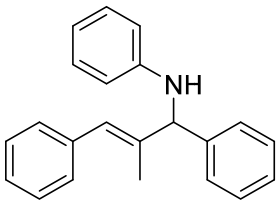
9*H*,9'*H*-9,9'-Bifluorene [CAS Registry No. 1530-12-7] (**3e**)



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69-6.97 (m, 16H), 4.85 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) 144.7, 141,7, 127.4, 126.9, 124.2, 119.8, 49.9 ppm; HRMS (FAB) *m*/*z* Calcd. for $C_{26}H_{18}$ (M^+), 330.1409; Found, 330.1410.



(*E*)-*N*-(1,3-Diphenyl-2-methylallyl)aniline (**5**)



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46-7.15 (m, 12H), 6.78 (s, 1H), 6.72 (t, J = 7.8 Hz, 1H), 6.63 (d, J = 7.8 Hz, 2H), 4.87 (s, 1H), 4.10 (s, 1H), 1.84 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) 147.4, 141.4, 137.7, 137.3, 129.1, 129.0, 128.7, 128.1, 127.60, 127.59, 126.5, 126.4, 117.6, 113.4, 66.3, 15.8 ppm; HRMS (FAB) *m*/*z* Calcd. for $C_{22}H_{21}N$ (M^+), 299.1669; Found,299.1672.