ELECTRONIC SUPPLEMENTARY INFORMATION FOR

Preparation of 1-Aryl-Substituted Isoindoline Derivatives by Sequential Morita-Baylis-Hillman and Intramolecular Diels-Alder Reactions

Kristen Nicole Clary, Masood Parvez and Thomas George Back*

Department of Chemistry, University of Calgary, Calgary, AB, Canada, T2N 1N4

Contents

Characterization data for **11b-11i**	ESI 2-4
Characterization data for **12d, 12e, 12f and 12i**	ESI 4-5
Characterization data for **13d**	ESI 6
Characterization data for **14e, 14f** and **14i**	ESI 6-7
Characterization data for **15d**	ESI 7
Characterization data for **16f** and **16i**	ESI 7-8
Characterization data for **17d**	ESI 8
^{1}H and ^{13}C NMR spectra for **11-17**	ESI 9-31
X-Ray crystallographic data for **11b**	ESI 32-33
X-Ray crystallographic data for **11d**	ESI 34-35
References for X-ray crystallography	ESI 35

Supplementary Material (ESI) for Organic & Biomolecular Chemistry
This journal is (c) The Royal Society of Chemistry 2009

2-Benzenesulfonyl-1-(2-chlorophenyl)-7a-(toluene-4-sulfonyl)-2,3,3a,4,5,7a-hexahydro-1*H*-isoindole (11b). Colourless crystals, mp 168-170 ˚C (from ethyl acetate); IR (film) 1348, 1309, 1170, 1139 cm^{-1}; ^{1}H NMR (400 MHz) δ 7.91 (d, *J* = 7.7 Hz, 2 H), 7.69-7.57 (m, 5 H) 7.43-7.40 (m, 1 H), 7.29-7.21 (m, 3 H), 7.16-7.10 (m, 2 H), 5.81 (s, 1 H), 5.80 (m, 1 H), 5.22 (dd, *J* = 10.2, 2.3 Hz, 1 H), 3.97 (dd, *J* = 9.0, 7.9 Hz, 1 H), 3.65 (dd, *J* = 9.2, 1.9 Hz, 1 H), 2.77 (crude q, *J* = 9.2 Hz, 1 H), 2.42 (s, 3 H), 1.91-1.82 (m, 1 H), 1.75-1.68 (m, 2 H), 1.49-1.40 (m, 1 H); ^{13}C NMR (50 MHz) δ 145.2, 136.9, 136.3, 135.7, 133.3, 132.3, 131.4, 131.1, 129.7, 129.3, 129.1, 128.5, 126.5, 121.5, 77.9, 63.3, 54.6, 38.5, 26.0, 22.8, 21.8; MS (*m/z*, %) 527 (<1), 372 (100), 230 (88), 77 (53); HRMS calc'd for $C_{20}H_{19}{}^{35}ClNO_2S$ (M^+ - Ts): 372.0825. Found: 372.0805. Anal. calc'd for $C_{27}H_{26}ClNO_4S_2$: C, 61.4; H, 5.0; N, 2.65. Found: C, 61.4; H, 5.0; N, 2.5.

2-Benzenesulfonyl-1-(4-methoxyphenyl)-7a-(toluene-4-sulfonyl)-2,3,3a,4,5,7a-hexa-hydro-1*H*-isoindole (11c). White solid, mp 179-181 ˚C (from ethyl acetate); IR (film) 1352, 1248, 1165, 1143 cm^{-1}; ^{1}H NMR (300 MHz) δ 7.68 (d, *J* = 7.1 Hz, 2 H), 7.65-7.60 (m, 1 H), 7.60-7.50 (m, 4 H), 7.23 (d, *J* = 7.9 Hz, 2 H), 7.08 (d, *J* = 8.5 Hz, 2 H), 6.75 (d, *J* = 8.9 Hz, 2 H), 5.76 (ddd, *J* = 10.1, 5.6, 2.6 Hz, 1 H), 5.20 (s, 1 H), 4.92 (dd, *J* = 10.2, 1.6 Hz, 1 H), 3.77 (s, 3 H), 3.73 (dd, *J* = 9.3, 2.4 Hz, 1 H), 3.52 (dd, *J* = 9.4, 3.0 Hz, 1 H), 2.60-2.52 (m, 1 H), 2.40 (s, 3 H), 1.90-1.79 (m, 1 H), 1.69-1.57 (m, 2 H), 1.45-1.34 (m, 1 H); ^{13}C NMR (75 MHz) δ 159.0, 145.0, 135.8, 134.5, 133.0, 132.1, 130.7, 129.9, 129.7, 129.1, 128.9, 128.3, 122.5, 113.3, 76.3, 66.2, 55.1, 53.9, 37.8, 24.8, 22.0, 21.6; MS (*m/z*, %) 523 (10), 368 (100), 367 (85). HRMS calc'd for $C_{28}H_{29}NO_5S_2$: 523.1487. Found: 523.1519. Anal. calc'd for $C_{28}H_{29}NO_5S_2$: C, 64.2; H, 5.6; N, 2.7. Found: C, 64.1; H, 5.8; N, 2.6.

2-Benzenesulfonyl-3-phenyl-1,2,3,6,7,7a-hexahydroisoindole-3a-carbonitrile (11d). White solid, mp 176-178 ˚C (from ethyl acetate); IR (film) 2233, 1348, 1171, 1086 cm^{-1}; ^{1}H NMR (300 MHz) δ 7.67 (d, *J* = 7.6 Hz, 2 H), 7.53 (t, *J* = 7.2 Hz, 1 H), 7.41 (t, *J* = 7.9 Hz, 1 H), 7.33-7.24 (m, 6 H), 5.70 (dt, *J* = 9.8, 3.7 Hz, 1 H), 5.29 (d, *J* = 10.1 Hz, 1 H), 4.73 (s, 1 H), 3.85 (dd, *J* = 10.1, 7.6 Hz, 1 H), 3.47 (dd, *J* = 10.1, 7.4 Hz, 1 H), 2.88-2.79 (m, 1 H), 2.07-1.96 (m, 2 H), 1.84-1.73 (m, 1 H), 1.63-1.54 (m, 1 H); ^{13}C NMR (75 MHz) δ 138.0, 137.0, 132.8, 132.0, 128.8, 128.7, 128.4, 127.4, 127.2, 122.7, 118.9, 70.9, 50.0, 48.2, 39.2, 20.9, 19.6; MS (*m/z*, %) 364 (70), 118 (100), 91 (45). HRMS calc'd for

This journal is (c) The Royal Society of Chemistry 2009

$C_{21}H_{20}N_2O_2S$: 364.1245. Found: 364.1252. Anal. calc'd for $C_{21}H_{20}N_2O_2S$: C, 69.2; H, 5.5; N, 7.7. Found: C, 69.1; H, 5.8; N, 7.6.

2-Benzenesulfonyl-3-(2-chlorophenyl)-1,2,3,6,7,7a-hexahydroisoindole-3a-carbonitrile (11e). Obtained as an inseparable mixture of two diastereoisomers (ca. 1:1) that cocrystallized to afford white sugar-like crystals, mp 179-186 ˚C (from ethyl acetate); IR (film) 2243, 1352, 1176, 752, 714 cm^{-1}; ^1H NMR (300 MHz) δ 7.9-7.2 (m, 2 x 9 H, both isomers), 5.83 (dt, *J* = 10.1, 3.8, Hz, 1 H), 5.58 (m, 1 H), 5.55 (s, 1 H), 5.27 (d, *J* = 7.5 Hz, 1 H), 5.22 (s, 1 H), 5.12 (d, *J* = 10.1 Hz, 1 H), 3.88 (dd, *J* = 9.8, 7.8 Hz, 1 H), 3.73 (dd, *J* = 10.9, 7.3 Hz, 1 H), 3.62 (dd, *J* = 10.9, 6.9 Hz, 1 H), 3.32 (t, *J* = 9.7 Hz, 1 H), 2.92 (m, 1 H), 2.42 (m, 1 H), 2.1-1.65 (m, 2 x 4 H, both isomers); ^{13}C NMR (75 MHz) both isomers: δ 136.2, 136.1, 135.1, 134.5, 133.7, 133.2, 133.0, 132.3, 131.6, 130.4, 129.9, 129.6, 129.5, 129.45, 129.4, 129.1, 128.4, 128.0, 127.7, 127.2, 126.6, 122.5, 120.8, 120.1, 118.5, 67.1, 67.0, 51.5, 49.5, 47.1, 44.4, 41.9, 38.9, 20.93, 20.88, 20.1, 18.4; MS (*m/z*, %) 398 (14), 152 (100), 125 (55). HRMS calc'd for $C_{21}H_{19}ClN_2O_2S$: 398.0856. Found: 398.0838. Anal calc'd for $C_{21}H_{19}ClN_2O_2S$: C, 63.2; H, 4.8; N, 7.0. Found: C, 63.0; H, 4.85; N, 6.8.

2-Benzenesulfonyl-3-(4-chlorophenyl)-1,2,3,6,7,7a-hexahydroisoindole-3a-carbonitrile (11f). White solid, mp 178-180 ˚C (from ethyl acetate); IR (film):2239, 1343, 1174 cm^{-1}; ^1H NMR (300 MHz) δ 7.66 (d, *J* = 7.1 Hz, 2 H), 7.56 (tt, *J* = 7.4, 1.2 Hz, 1 H), 7.44 (t, *J* = 7.4 Hz, 2 H), 7.29-7.20 (m, 4 H), 5.69 (dt, *J* = 9.9, 3.7 Hz, 1 H), 5.24 (d, *J* = 9.9 Hz, 1 H), 4.66 (s, 1 H), 3.86 (dd, *J* = 10.3, 7.5 Hz, 1 H), 3.44 (dd, *J* = 10.3, 7.2 Hz, 1 H), 2.78 (m, 1 H), 2.12-1.90 (m, 2 H), 1.81-1.70 (m, 1 H), 1.56-1.46 (m, 1 H); ^{13}C NMR (75 MHz) δ 137.5, 135.7, 134.7, 133.1, 132.3, 129.0, 128.7, 128.6, 127.7, 127.4, 122.1, 118.8, 70.3, 50.1, 48.1, 39.2, 21.0, 19.6; MS (*m/z*, %) 398 (30), 152 (100), 125 (47). HRMS calc'd for $C_{21}H_{19}ClN_2O_2S$: 398.0856. Found: 398.0841. Anal calc'd for $C_{21}H_{19}ClN_2O_2S$: C, 63.2; H, 4.8; N, 7.0. Found: C, 63.2; H, 4.8; N, 7.0.

2-Benzenesulfonyl-3-(4-cyanophenyl)-1,2,3,6,7,7a-hexahydroisoindole-3a-carbonitrile (11g). White solid, mp 160-164 ˚C (from ethyl acetate); IR (film) 2228, 1357, 1174 cm^{-1}; ^1H NMR (300 MHz) δ 7.72-7.60 (m, 5 H), 7.52-7.45 (m, 4 H), 5.77 (dt, *J* = 9.8, 3.7 Hz, 1 H), 5.27 (d, *J* = 9.9 Hz, 1 H), 4.73 (s, 1 H), 3.92 (dd, *J* = 10.4, 7.3 Hz, 1 H), 3.48 (dd, *J* = 10.4, 6.9, Hz, 1 H), 2.82-2.74 (m, 1 H), 2.15-1.94 (m, 2 H), 1.84-1.73

This journal is (c) The Royal Society of Chemistry 2009

(m, 1 H), 1.56-1.41 (m, 1 H); ^{13}C NMR (75 MHz) δ 142.4, 137.3, 133.3, 132.7, 132.2, 129.1, 128.1, 127.4, 121.7, 118.5, 118.3, 112.8, 70.5, 50.5, 48.2, 39.5, 21.1, 19.7; MS (*m/z*, %) 389 (20), 248 (25), 143 (100), 116 (51). HRMS calc'd for $C_{22}H_{19}N_3O_2S$: 389.1198. Found: 389.1187.

4-(2-Benzenesulfonyl-7a-cyano-2,3,3a,4,5,7a-hexahydro-1*H*-isoindol-1-yl)-benzoic acid methyl ester (11h). White solid, mp 176-179 ˚C (from ethyl acetate); IR (film) 2226, 1717, 1283, 1165 cm^{-1}; ^{1}H NMR (400 MHz) δ 8.02 (d, *J* = 8.4 Hz, 2 H), 7.70 (d, *J* = 7.3 Hz, 2 H), 7.59 (t, *J* = 7.4 Hz, 1 H), 7.47 (t, *J* = 7.8 Hz, 2 H), 7.38 (d, *J* = 8.3 Hz, 2 H), 5.77 (dd, *J* = 9.8, 3.8 Hz, 1 H), 5.32 (d, *J* = 9.3 Hz, 1 H), 4.79 (s, 1 H), 3.93 (s, 3 H), 3.91 (dd, *J* = 10.3, 7.4 Hz, 1 H), 3.52 (dd, *J* = 10.3, 7.1 Hz, 1 H), 2.87-2.79 (m, 1 H), 2.15-1.96 (m, 2 H), 1.84-1.76 (m, 1 H), 1.63-1.54 (m, 1 H); ^{13}C NMR (75 MHz) δ 166.5, 142.0, 137.8, 135.9, 133.0, 132.3, 130.6, 129.7, 128.9, 127.4, 127.3, 122.3, 70.7, 52.1, 50.2, 48.2, 39.5, 21.0, 19.7; MS (*m/z*, %) 422 (67), 281 (21), 176 (100). HRMS calc'd for $C_{23}H_{22}N_2O_4S$: 422.1300. Found: 422.1283. Anal calc'd for $C_{23}H_{22}N_2O_4S$: C, 65.4; H, 5.25; N, 6.6. Found: C, 65.0; H, 5.3; N, 6.5.

2-Benzenesulfonyl-3-(1-naphthyl)-1,2,3,6,7,7a-hexahydroisoindole-3a-carbonitrile (11i). White solid, mp 199-201 ˚C (from ethyl acetate); IR (film) 2235, 1348, 1165 cm^{-1}; ^{1}H NMR (400 MHz) δ 7.95 (d, *J* = 8.4 Hz, 1 H), 7.91 (d, *J* = 8.0 Hz, 1 H), 7.83 (d, *J* = 8.2 Hz, 1 H), 7.69 (d, *J* = 7.5 Hz, 2 H), 7.60-7.50 (m, 4 H), 7.44 (t, *J* = 7.7 Hz, 2 H), 7.39 (t, *J* = 8.0, 1 H), 5.77 (dt, *J* = 9.8, 3.8 Hz, 1 H), 5.70 (s, 1 H), 5.47 (d, *J* = 9.9 Hz, 1 H), 3.99 (dd, *J* = 9.9, 7.7 Hz, 1 H), 3.60 (dd, *J* = 9.9, 8.2 Hz, 1 H), 3.00-2.94 (m, 1 H), 2.17-2.06 (m, 2 H), 1.91-1.84 (m, 1 H), 1.77-1.68 (m, 1 H); ^{13}C NMR (75 MHz) δ 137.9, 133.7, 132.8, 131.9, 131.0, 129.24, 129.20, 128.8, 127.4, 126.4, 125.8, 125.4, 125.0, 122.9, 122.5, 119.0, 66.3, 50.0, 48.2, 39.8, 20.9, 19.4; MS (*m/z*, %) 414 (20), 168 (100), 141 (45). HRMS calc'd for $C_{25}H_{22}N_2O_2S$: 414.1402. Found: 414.1387. Anal calc'd for $C_{25}H_{22}N_2O_2S$: C, 72.4; H, 5.35; N, 6.8. Found: C, 72.4; H, 5.5; N, 6.7.

2-Benzenesulfonyl-3-phenyl-1,2,3,6-tetrahydroisoindole-3a-carbonitrile (12d). White solid, mp 185-187 ˚C (from ethyl acetate); IR (film):2217 1344, 1162, 959 cm^{-1}; ^{1}H NMR (300 MHz) δ 7.62-7.53 (m, 3 H), 7.46-7.33 (m, 7 H), 6.07-6.03 (m, 1 H), 5.82 (s, 1 H), 5.60 (d, *J* = 9.7 Hz, 1 H), 4.52-4.35 (m, 2 H), 4.29 (s, 1 H), 2.87-2.69 (m, 2 H); ^{13}C NMR (75 MHz) δ 137.5, 134.2, 133.0, 129.9, 129.3, 129.1, 128.9, 128.3, 128.1,

This journal is (c) The Royal Society of Chemistry 2009

127.7, 121.1, 119.2, 117.3, 72.0, 51.3, 47.8, 26.5; MS (*m/z*, %) 362 (28), 246 (62), 221 (80), 219 (80), 116 (100). HRMS calc'd for $C_{21}H_{18}N_2O_2S$: 362.1089. Found: 362.1076. Anal calc'd for $C_{21}H_{18}N_2O_2S$: C, 69.6; H, 5.0; N, 7.7. Found: C, 69.25; H, 5.05; N, 7.6.

2-Benzenesulfonyl-3-(2-chlorophenyl)-1,2,3,6-tetrahydroisoindole-3a-carbonitrile (12e). Colourless needles; mp 228-229 ˚C (from ethyl acetate); IR (film) 2224, 1352, 1167, 1081, 1038 cm^{-1}; ^1H NMR (300 MHz) δ 7.85-7.82 (m, 1 H), 7.72 (d, *J* = 7.3 Hz, 2 H), 7.59 (t, *J* = 7.5 Hz, 1 H), 7.49 (t, *J* = 7.5 Hz, 2 H), 7.43-7.40 (m, 1 H), 7.34-7.29 (m, 2 H), 6.09-5.97 (m, 1 H), 5.78 (d, *J* = 9.3 Hz, 2 H), 5.02 (s, 1 H), 4.58-4.45 (m, 1H), 4.33 (d, *J* = 14.0 Hz, 1 H), 2.88-2.58 (m, 2 H); ^{13}C NMR (50 MHz) δ 136.2, 133.3, 132.8, 132.7, 131.0, 130.1, 129.4, 129.2, 129.09, 129.05, 128.0, 126.9, 121.5, 119.6, 117.2, 66.6, 51.6, 48.4, 26.3; MS (*m/z*, %) 396 (4), 280 (17), 219 (100), 116 (100). HRMS calc'd for $C_{21}H_{17}ClN_2O_2S$: 396.0699. Found: 396.0704. Anal calc'd for $C_{21}H_{17}ClN_2O_2S$: C, 63.55; H, 4.3; N, 7.1. Found: C, 63.3; H, 4.35; N, 6.9.

2-Benzenesulfonyl-3-(4-chlorophenyl)-1,2,3,6-tetrahydroisoindole-3a-carbonitrile (12f). White solid, mp 181-183 ˚C (from ethyl acetate); IR (film) 2230, 1350, 1167, 1091 cm^{-1}; ^1H NMR (400 MHz) δ 7.65-7.55 (m, 3 H), 7.46 (t, *J* = 7.7 Hz, 2 H), 7.38-7.27 (m, 4 H), 6.10-5.99 (m, 1 H), 5.81 (s, 1 H), 5.55 (d, *J* = 9.6 Hz, 1 H), 4.47 (dd, *J* = 13.7, 2.5 Hz, 1 H), 4.38 (d, *J* = 13.7 Hz, 1 H), 4.23 (s, 1 H), 2.75 (m, 2 H); ^{13}C NMR (75 MHz,) δ 137.1, 135.1, 133.3, 133.0, 130.2, 129.1, 129.4, 128.9, 128.6, 127.8, 121.4, 118.9, 117.1, 71.3, 51.4, 47.6, 26.5; MS (*m/z*, %) 396 (17), 280 (51), 255 (100), 116 (75). HRMS calc'd for $C_{21}H_{17}N_2O_2SCl$: 396.0699. Found: 396.0690. Anal calc'd for $C_{21}H_{17}N_2O_2SCl$: C, 63.55; H, 4.3; N, 7.1. Found: C, 63.7; H, 4.1; N, 7.0.

2-Benzenesulfonyl-3-(1-naphthyl)-1,2,3,6-tetrahydroisoindole-3a-carbonitrile (12i). White solid, mp 225-227 ˚C (from ethyl acetate); IR (film) 2243, 1357, 1171 cm^{-1}; ^1H NMR (300 MHz) δ 7.91-7.85 (m, 3 H), 7.81 (d, *J* = 8.2 Hz, 1 H), 7.54-7.48 (m, 5 H), 7.41-7.31 (m, 3 H), 5.99 (dt, *J* = 9.8, 2.8 Hz, 1 H), 5.85 (m, 1 H), 5.51 (d, *J* = 9.8 Hz, 1 H), 5.37 (s, 1 H), 4.55 (s, 2 H), 2.77 (m, 2 H); ^{13}C NMR (75MHz) δ 137.4, 133.7, 133.0, 131.1, 129.74, 129.72, 129.5, 129.4, 129.3, 128.9, 127.8, 126.6, 125.5, 125.2, 121.7, 121.1, 119.7, 117.4, 66.0, 51.5, 49.3, 26.4; MS (*m/z*, %) 412 (6), 296 (33), 269 (55), 154 (77), 116 (100). HRMS calc'd for $C_{25}H_{20}N_2O_2S$: 412.1245. Found 412.1241. Anal calc'd for $C_{25}H_{20}N_2O_2S$: C, 72.8; H, 4.9; N, 6.8. Found: C, 72.5; H, 5.0; N, 6.6.

This journal is (c) The Royal Society of Chemistry 2009

2-Benzenesulfonyl-7-methyl-3-phenyl-1,2,3,6-tetrahydroisoindole-3a-carbonitrile (13d). White solid, mp 189-191 ˚C (from ethyl acetate); IR (film) 2227, 1360, 1167 cm^{-1}; ^{1}H NMR (400 MHz) δ 7.62 (d, *J* = 7.3 Hz, 2 H), 7.55 (t, *J* = 7.5 Hz, 1 H), 7.44 (d, *J* = 7.8 Hz, 2 H), 7.40 (d, *J* = 8.3 Hz, 2 H), 7.32 (t, *J* = 7.1 Hz, 3 H), 6.03 (ddd, *J* = 9.6, 4.5, 2.3 Hz, 1 H), 5.58 (d, *J* = 9.6 Hz, 1 H), 4.55 (d, *J* = 13.9 Hz, 1 H), 4.40 (d, *J* = 13.9 Hz, 1 H), 4.19 (s, 1 H), 2.84 (d, *J* = 22.8 Hz, 1 H), 2.57 (dd, *J* = 22.6, 4.2 Hz, 1 H), 1.73 (s, 3 H); ^{13}C NMR (75 MHz) δ 137.5, 134.2, 133.0, 130.1, 129.1, 129.0, 128.8, 128.3, 128.2, 127.8, 122.6, 119.4, 117.7, 71.6, 49.3, 48.9, 32.1, 18.9; MS (*m/z*, %) 376 (2), 246 (75), 141 (50), 130 (72), 91 (49), 77 (100). HRMS calc'd for $C_{22}H_{20}N_2O_2S$: 376.1245. Found: 376.1236.

2-Benzenesulfonyl-1-(2-chlorophenyl)-2,3-dihydro-1*H*-isoindole (14e). White solid, mp 164-167 ˚C (from ethyl acetate); IR (film):1350, 1167, 1094 cm^{-1}; ^{1}H NMR (400 MHz) δ 7.83 (d, *J* = 7.4 Hz, 2 H), 7.55-7.53 (m, 1 H), 7.51-7.42 (m, 2 H), 7.40-7.29 (m, 2 H), 7.23-7.15 (m, 5 H), 7.00(d, *J* = 7.6 Hz, 1 H), 6.45 (d, *J* = 2.4 Hz, 1 H), 4.99 (dd, *J* = 13.4, 2.9 Hz, 1 H), 4.84 (d, *J* = 13.4 Hz, 1 H); ^{13}C NMR (75 MHz) δ 140.1, 139.7, 137.3, 134.8, 132.8, 132.1, 129.9, 129.6, 129.1, 128.9, 128.2, 127.7, 127.3, 123.4, 122.5, 65.9, 54.3; MS (*m/z*, %) 369 (2), 258 (52), 228 (100). HRMS calc'd for $C_{20}H_{16}ClNO_2S$: 369.0590. Found: 369.0574.

2-Benzenesulfonyl-1-(4-chlorophenyl)-2,3-dihydro-1*H*-isoindole (14f). Off white solid, mp 119-121 ˚C (from ethyl acetate); IR (film) 1353, 1164, 1097 cm^{-1}; ^{1}H NMR (300 MHz) δ 7.63 (d, *J* = 7.2 Hz, 2 H), 7.51-7.46 (m, 1 H), 7.40-7.35 (m, 2 H), 7.22-7.10 (m, 7 H), 6.84 (d, *J* = 7.3 Hz, 1 H), 5.89 (s, 1 H), 4.84 (s, 2 H); ^{13}C NMR (75MHz) δ 140.4, 140.2, 138.3, 135.0, 133.7, 132.6, 129.0, 128.9, 128.7, 128.2, 128.1, 127.2, 123.6, 122.5, 68.7, 54.0; MS (*m/z*, %) 369 (1), 258 (33), 228 (100), 77 (93). HRMS calc'd for $C_{20}H_{16}ClNO_2S$: 369.0590. Found 369.0580.

2-Benzenesulfonyl-1-(1-naphthyl)-2,3-dihydro-1*H*-isoindole (14i). White solid, mp 159-161 ˚C (from ethyl acetate); IR (film) 1353, 1164, 1097 cm^{-1}; ^{1}H NMR (400 MHz) δ 7.93 (d, *J* = 8.0 Hz, 1 H), 7.83 (d, *J* = 7.9 Hz, 1 H), 7.75 (d, *J* = 8.1 Hz, 1 H), 7.55 (d, *J* = 8.1 Hz, 2 H), 7.44 (t, *J* = 8.1 Hz, 2 H), 7.41-7.32 (m, 3 H), 7.29-7.19 (m, 4 H), 7.09 (t, *J* = 7.4 Hz, 1 H), 6.85 (d, *J* = 7.7 Hz, 1 H), 6.67 (s, 1 H), 5.10 (d, *J* = 14.6 Hz, 1 H), 5.05 (d, *J* = 14.6 Hz, 1 H); ^{13}C NMR δ 141.3, 138.1, 136.5, 134.6, 134.1, 132.3,

This journal is (c) The Royal Society of Chemistry 2009

130.8, 128.9, 128.7, 128.5, 128.12, 128.08, 127.2, 126.2, 125.5, 125.3, 123.14, 123.11, 122.6, 67.1, 54.2; MS (*m/z*, %) 385 (21), 258 (53), 242 (85), 77 (100). HRMS calc'd for $C_{24}H_{19}NO_2S$: 385.1137. Found: 385.1121. Anal calc'd for $C_{24}H_{19}NO_2S$: C, 74.8; H, 5.0; N, 3.6. Found: C, 74.65; H, 5.0; N, 3.7.

2-Benzenesulfonyl-4-methyl-1-phenyl-2,3-dihydro-1*H*-isoindole (15d). White solid, mp 180-182 ˚C (from ethyl acetate); IR (film) 1337, 1167, 1094 cm^{-1}; ^1H NMR (400 MHz) δ 7.65 (d, *J* = 8.5 Hz, 2 H), 7.49 (t, *J* = 7.4 Hz, 1 H), 7.38 (t, *J* = 7.7 Hz, 2 H), 7.28-7.18 (m, 5 H), 7.11 (t, *J* = 7.5 Hz, 1 H), 7.05 (d, *J* = 7.3 Hz, 1 H), 6.73 (d, *J* = 7.4 Hz, 1 H), 5.98 (s, 1 H), 4.89-4.76 (m, 2 H), 2.30 (s, 3 H); ^{13}C NMR (75 MHz) δ 141.8, 140.7, 138.6, 134.1, 132.5, 132.4, 128.8, 128.7, 128.5, 128.4, 127.7, 127.2, 120.9, 69.9, 53.4, 18.7; MS (*m/z*, %) 349 (2), 272 (47), 208 (100), 77 (85). HRMS calc'd for $C_{21}H_{19}NO_2S$: 349.1137. Found: 349.1125. Anal calc'd for $C_{21}H_{19}NO_2S$: C, 72.2; H, 5.5; N, 4.0. Found: C, 71.7; H, 5.2; N, 4.0.

2-Benzenesulfonyl-3-(4-chlorophenyl)-2,3-dihydro-isoindol-1-one (16f). White solid, mp 166-168 ˚C (from ethyl acetate); IR (film) 1729, 1362, 1281, 1162, 1086 cm^{-1}; ^1H NMR (300 MHz) δ 7.89 (d, *J* = 7.6 Hz, 1 H), 7.69 (d, *J* = 7.5 Hz, 2 H), 7.59-7.48 (m, 3 H), 7.39 (t, *J* = 7.9 Hz, 2 H), 7.23 (d, *J* = 8.4 Hz, 2 H), 7.16 (d, *J* = 7.7 Hz, 1 H), 7.02 (d, *J* = 8.5 Hz, 2 H), 6.21 (s, 1 H); ^{13}C NMR (75 MHz,) δ 166.2, 145.9, 138.8, 135.4, 134.5, 133.8, 129.4, 129.3, 129.0, 128.8, 128.6, 127.9, 127.8, 124.9, 123.7, 64.6; MS (*m/z*, %) 383 (<1), 319 (80), 242 (100). HRMS calc'd for $C_{20}H_{14}ClNO_3S$: 383.0383. Found: 383.0389.

2-Benzenesulfonyl-1-(1-naphthyl)-2,3-dihydro-isoindol-1-one (16i). White solid, mixture of two rotamers, mp 182-183 ˚C (from ethyl acetate); IR (film):1729, 1362, 1295, 1176, 1095 cm^{-1}; ^1H NMR (400 MHz; signals from both rotamers are given) δ 8.54 (d, *J* = 8.5 Hz), 8.03 (m), 7.97 (d, *J* = 8.2 Hz), 7.95-7,87 (m,), 7.84-7.68 (m), 7.61 (dt, *J* = 14.4, 7.5 Hz), 7.52 (m), 7.50-7.44 (m), 7.41 (t, *J* = 7.8 Hz), 7.32-7.27 (m), 7.23-7.09 (m), 6.93 (t, *J* = 7.8 Hz), 6.76 (d, *J* = 7.2 Hz), 6.68 (t, *J* = 7.6 Hz), 6.56 (s), 6.50 (d, *J* = 8.6 Hz); when heated to 100 ˚C, coalescence of numerous signals was observed, while cooling back to room temperature restored the original spectrum; ^{13}C NMR (75 MHz; signals from both rotamers are given) δ 166.9, 166.6, 147.2, 145.9, 138.6, 137.9, 134.6, 134.4, 134.11, 134.09, 133.8, 133.1, 133.0, 132.1, 131.2, 130.5, 129.6, 129.3,

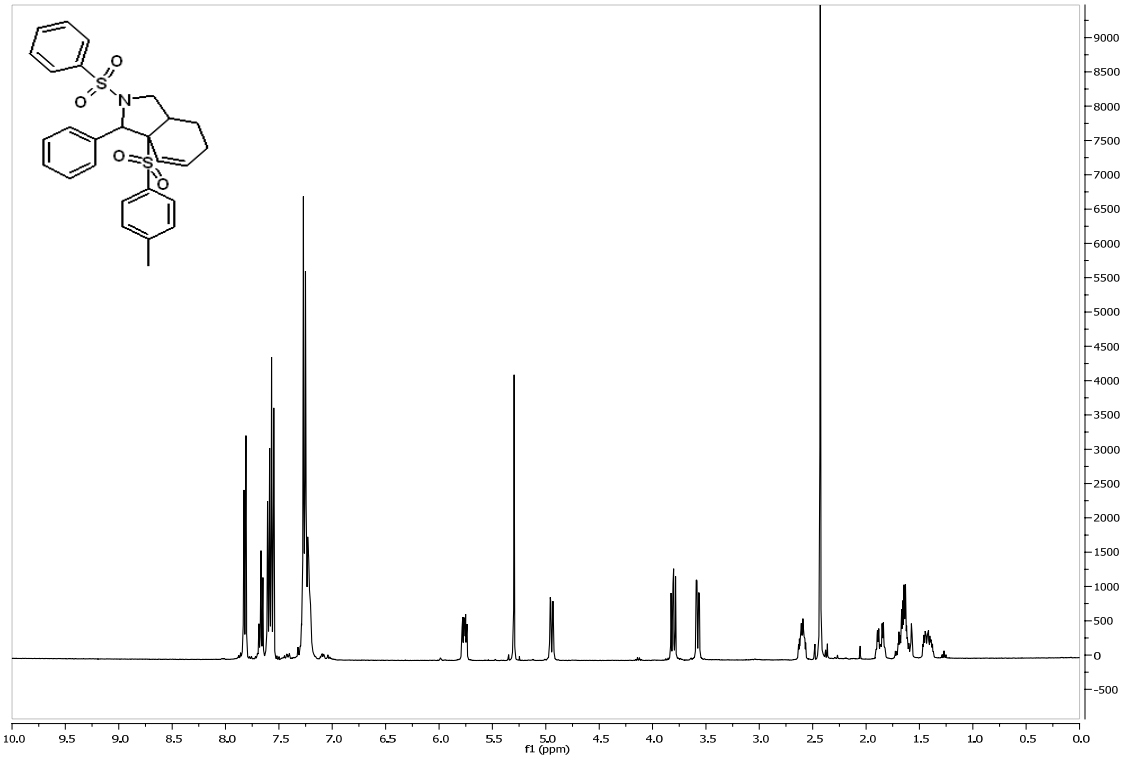
This journal is (c) The Royal Society of Chemistry 2009

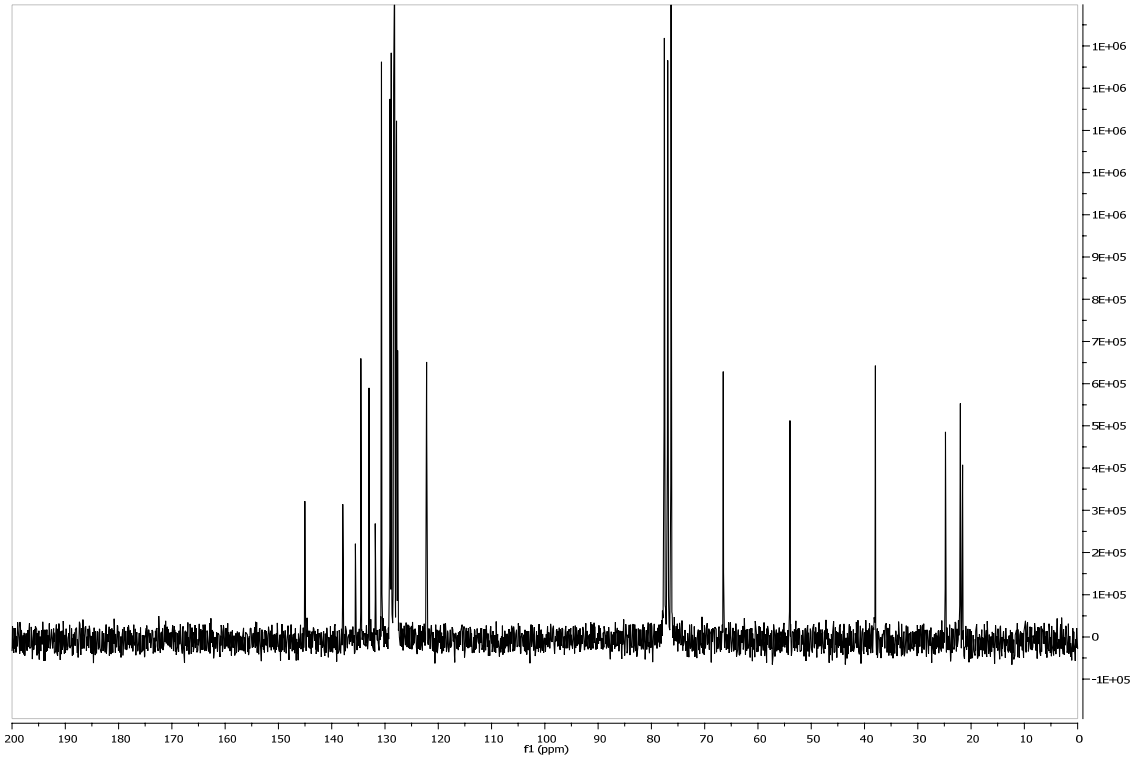
129.2, 128.8, 128.6, 128.3, 128.0, 127.25, 127.17, 126.22, 126.19, 125.4, 125.2, 125.10, 125.06, 123.9, 123.4, 123.1, 122.6, 122.3, 68.5, 60.5; MS (*m/z*, %) 399 (15), 335 (14), 258 (100). HRMS calc'd for $C_{24}H_{17}NO_3S$: 399.0929. Found: 399.0926.

2-Benzenesulfonyl-4-methyl-1-phenyl-2,3-dihydro-isoindol-1-one (17d). White solid, mp 162-164 ˚C (from ethyl acetate); IR (film): 1733, 1362, 1190, 1162, 1100 cm^{-1}; ^1H NMR (400 MHz) δ 7.62 (dd, *J* = 8.5, 1.2 Hz, 2 H), 7.54-7.49 (m, 1 H), 7.41 (t, *J* = 7.6 Hz, 1 H), 7.36-7.29 (m 3 H), 7.25-7.21 (m, 3 H), 7.09-7.07 (m, 2 H), 6.97 (d, *J* = 7.6 Hz, 1 H), 6.18 (s, 1 H), 2.71 (s, 3 H); ^{13}C NMR (75 MHz) δ 167.1, 147.0, 139.4, 138.9, 137.1, 133.9, 133.5, 130.9, 128.7, 128.6, 128.5, 128.1, 128.0, 125.9, 121.1, 64.8, 17.5; MS (*m/z*, %): 363 (<1), 299 (43), 222 (100), 77 (60). HRMS calc'd for $C_{21}H_{17}NO_3S$: 363.0929. Found: 363.0918.

This journal is (c) The Royal Society of Chemistry 2009

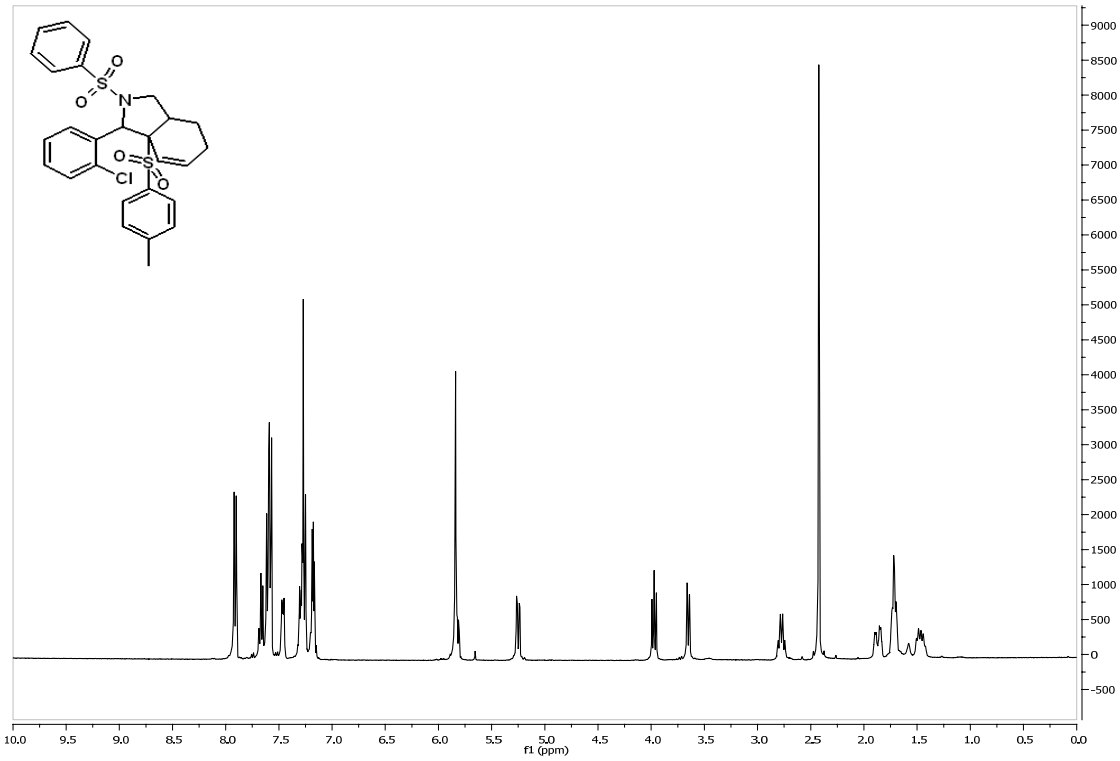
^{1}H and ^{13}C NMR Spectra of **11a**

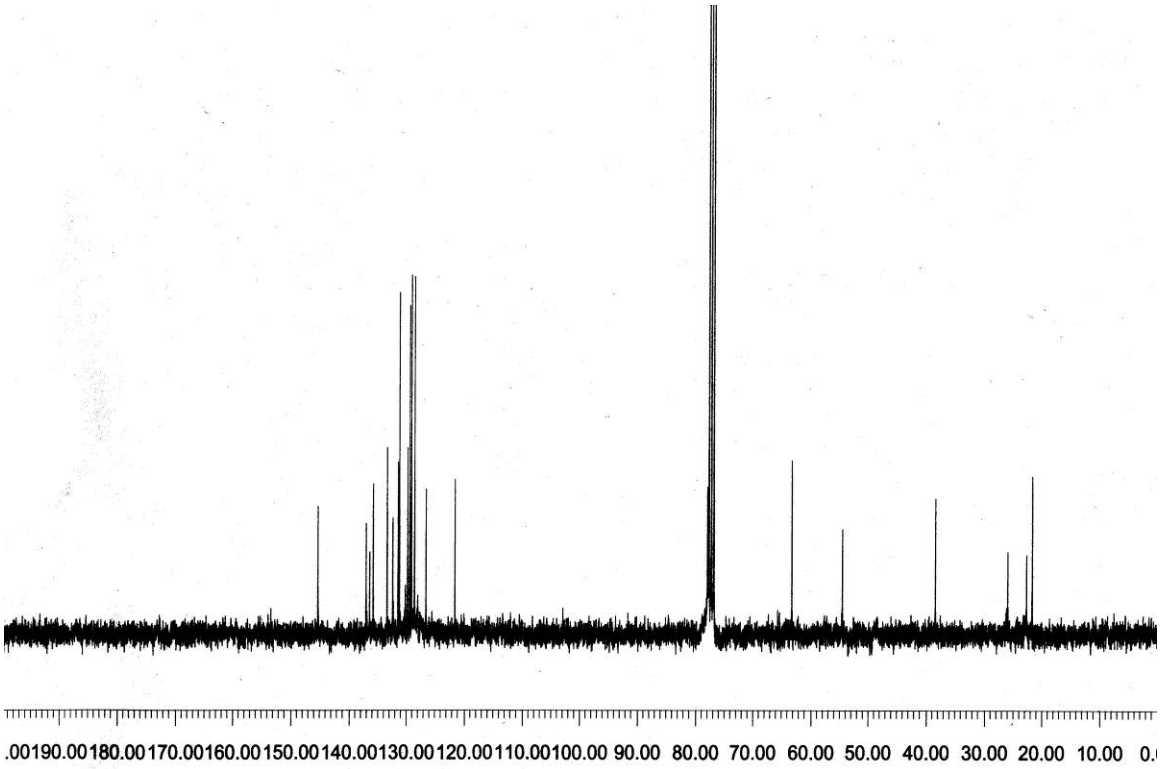




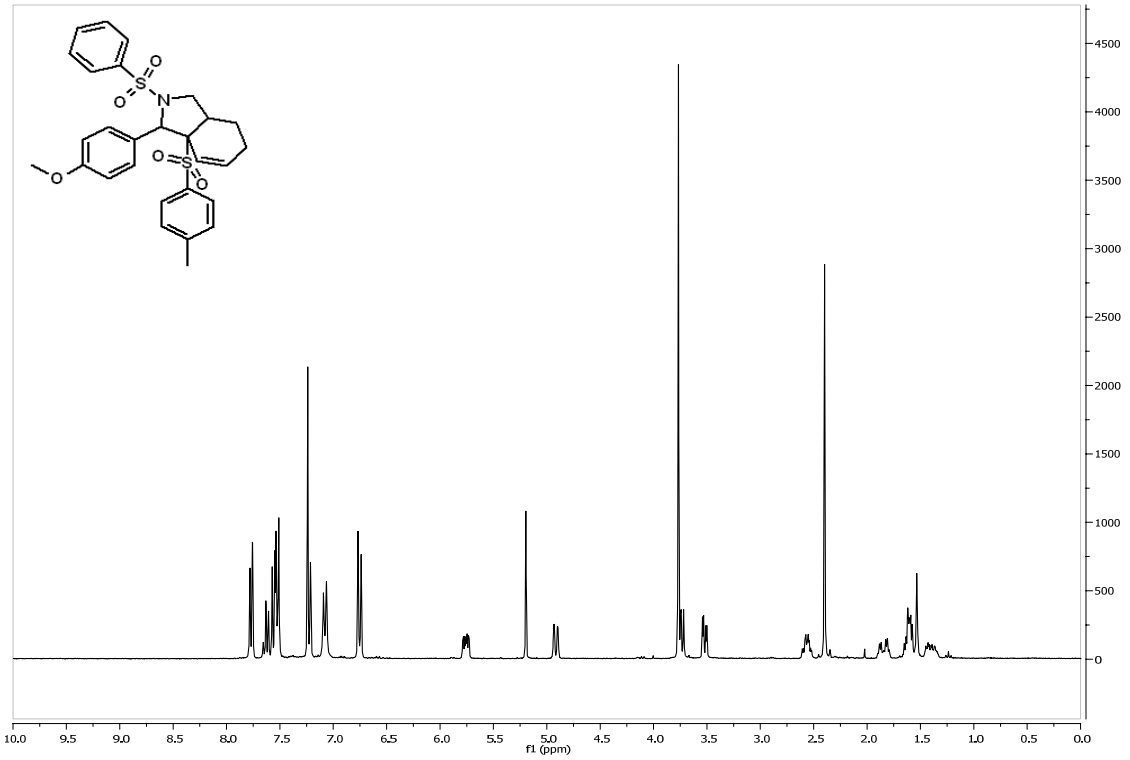
This journal is (c) The Royal Society of Chemistry 2009

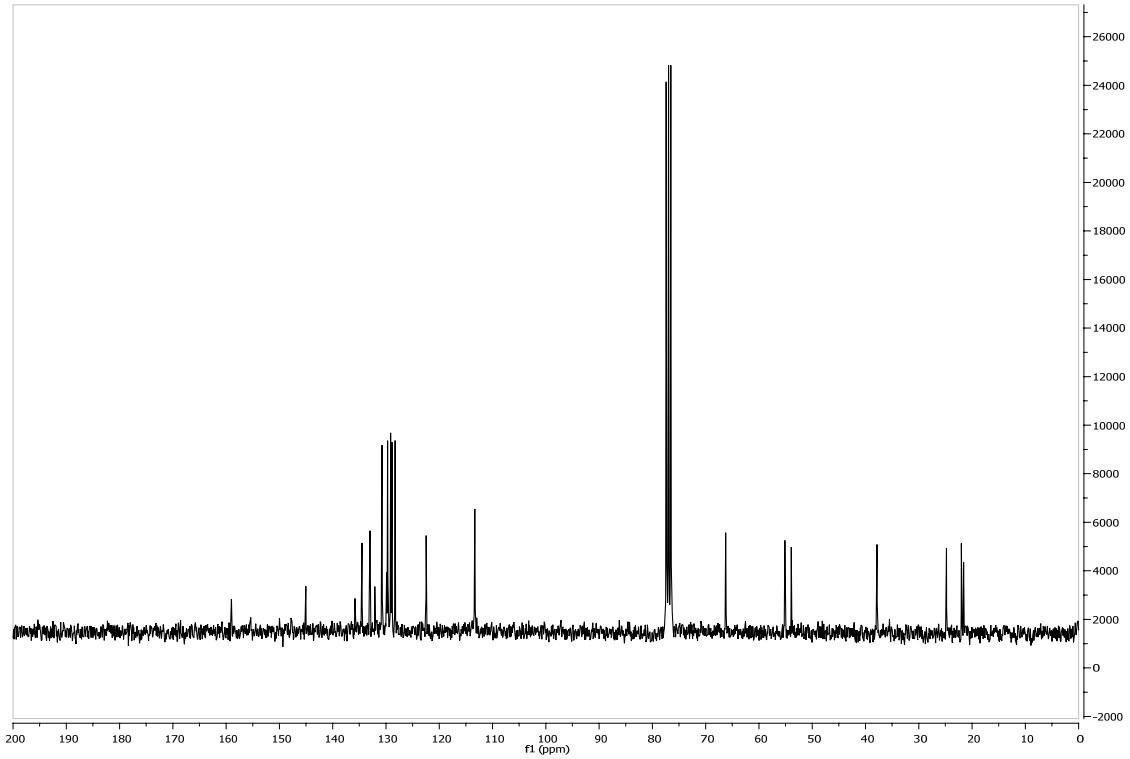
^{1}H and ^{13}C NMR Spectra of **11b**





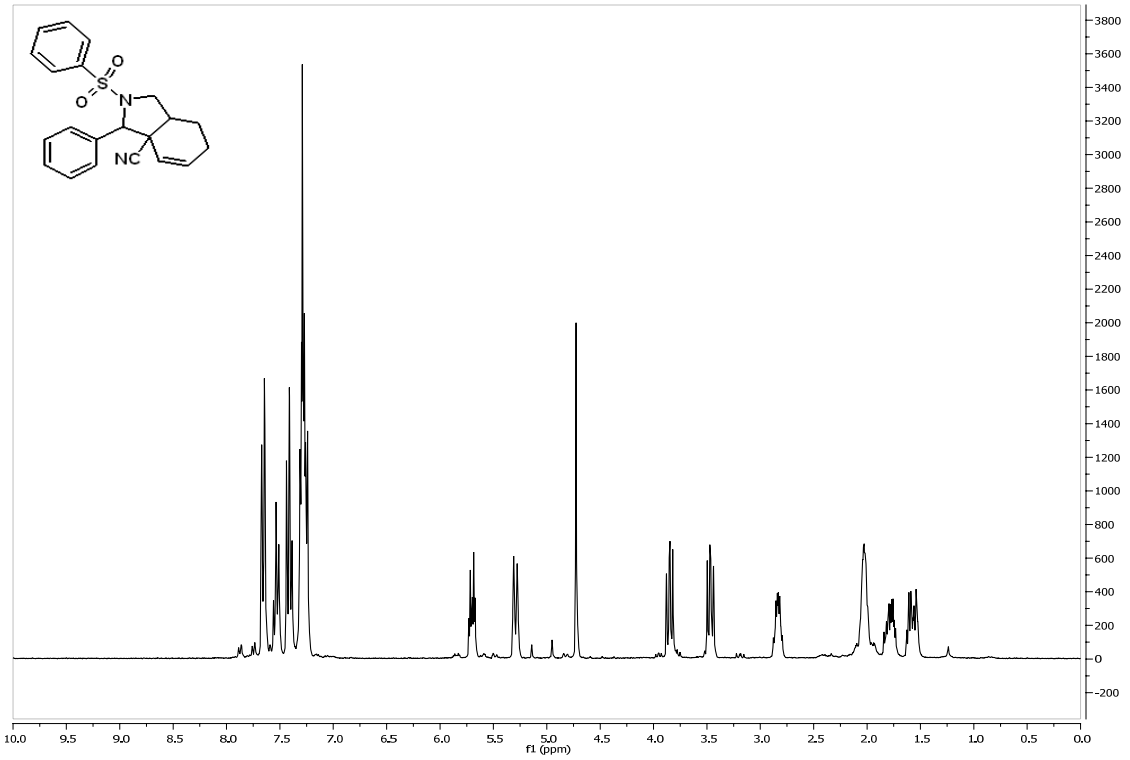
^{1}H and ^{13}C NMR Spectra of **11c**

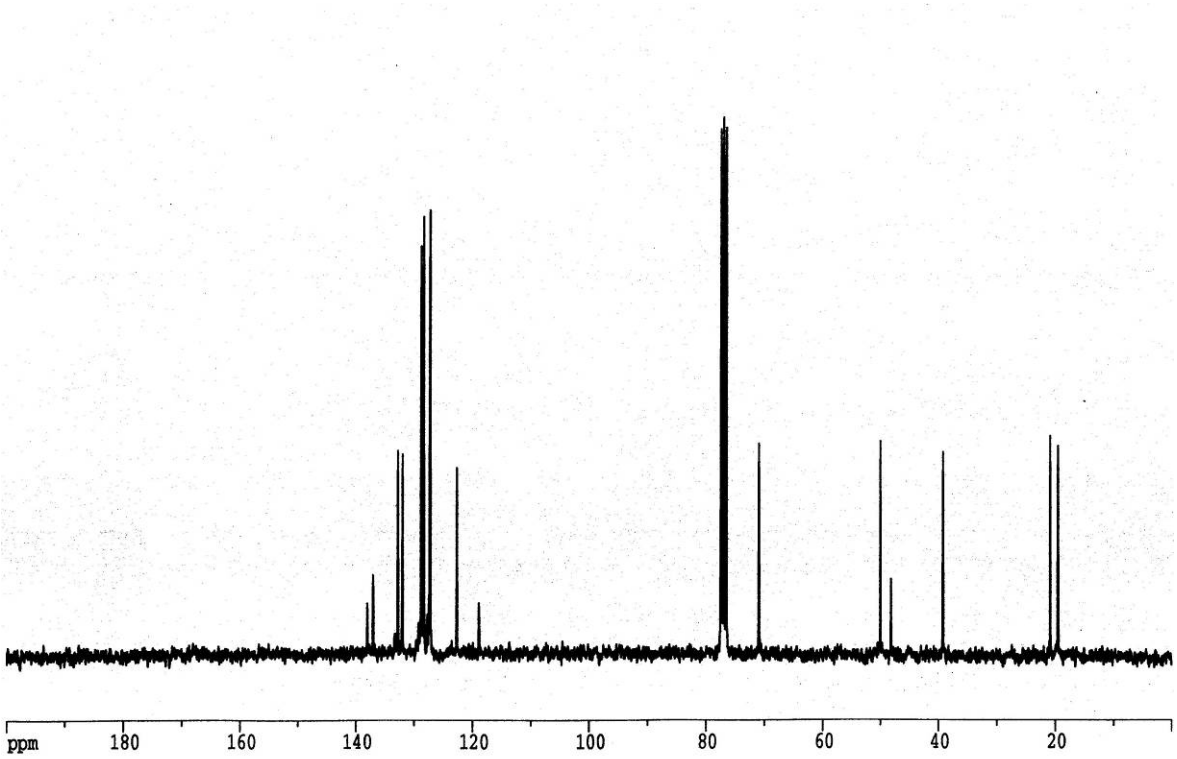




This journal is (c) The Royal Society of Chemistry 2009

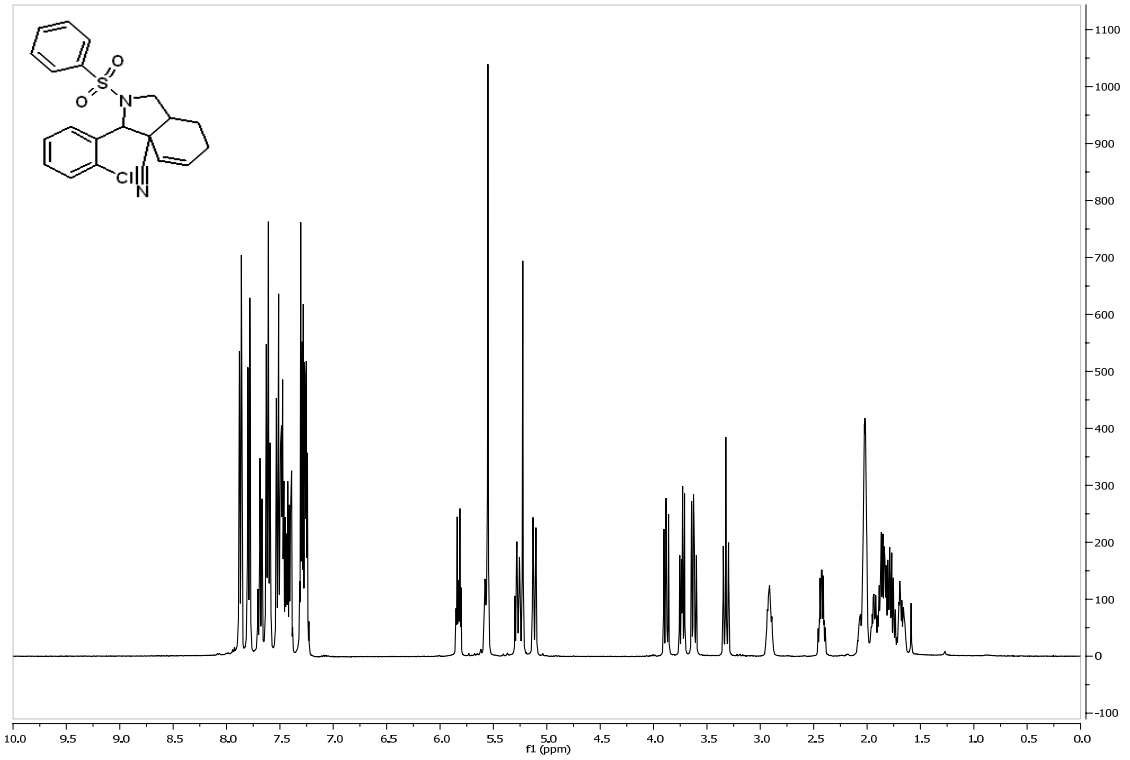
^{1}H and ^{13}C NMR Spectra of **11d**

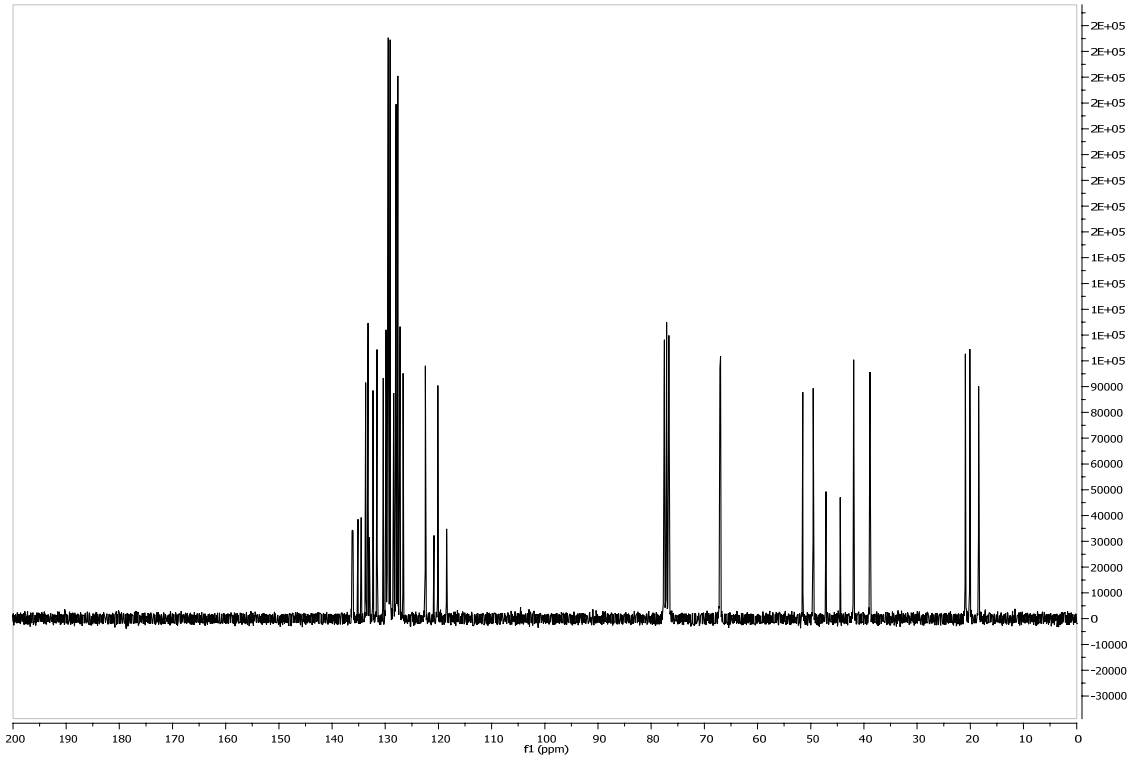




Supplementary Material (ESI) for Organic & Biomolecular Chemistry
This journal is (c) The Royal Society of Chemistry 2009

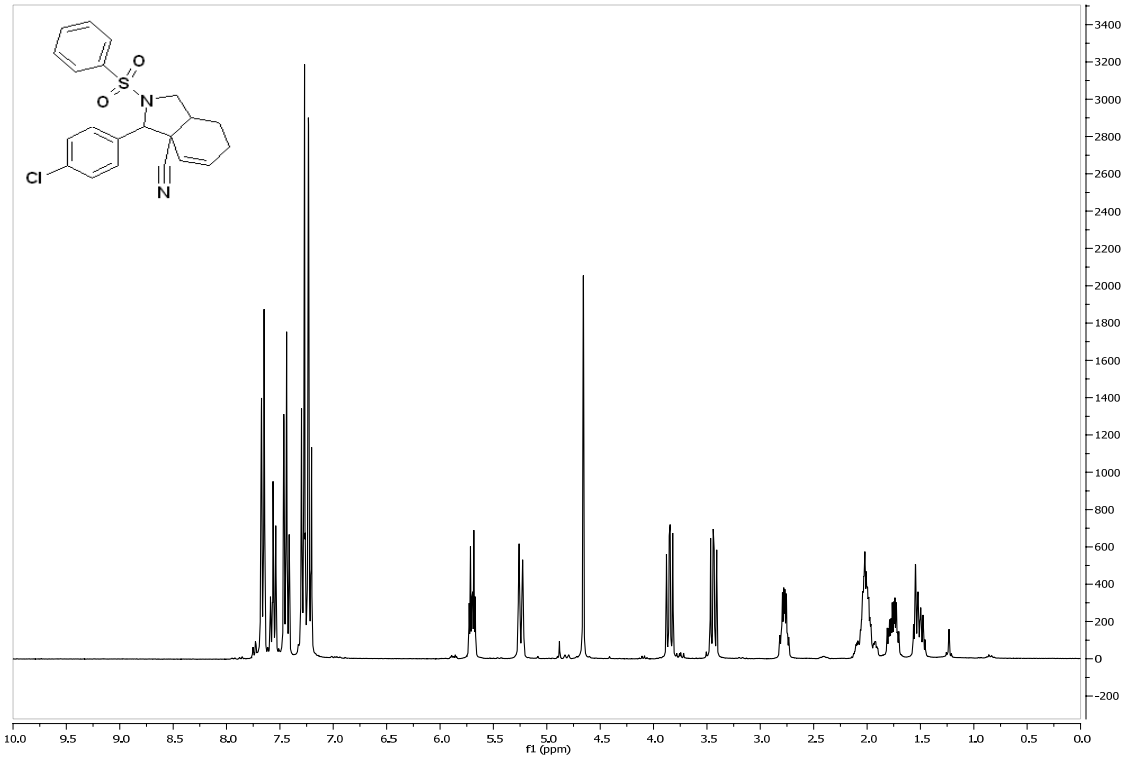
^{1}H and ^{13}C NMR Spectra of **11e**

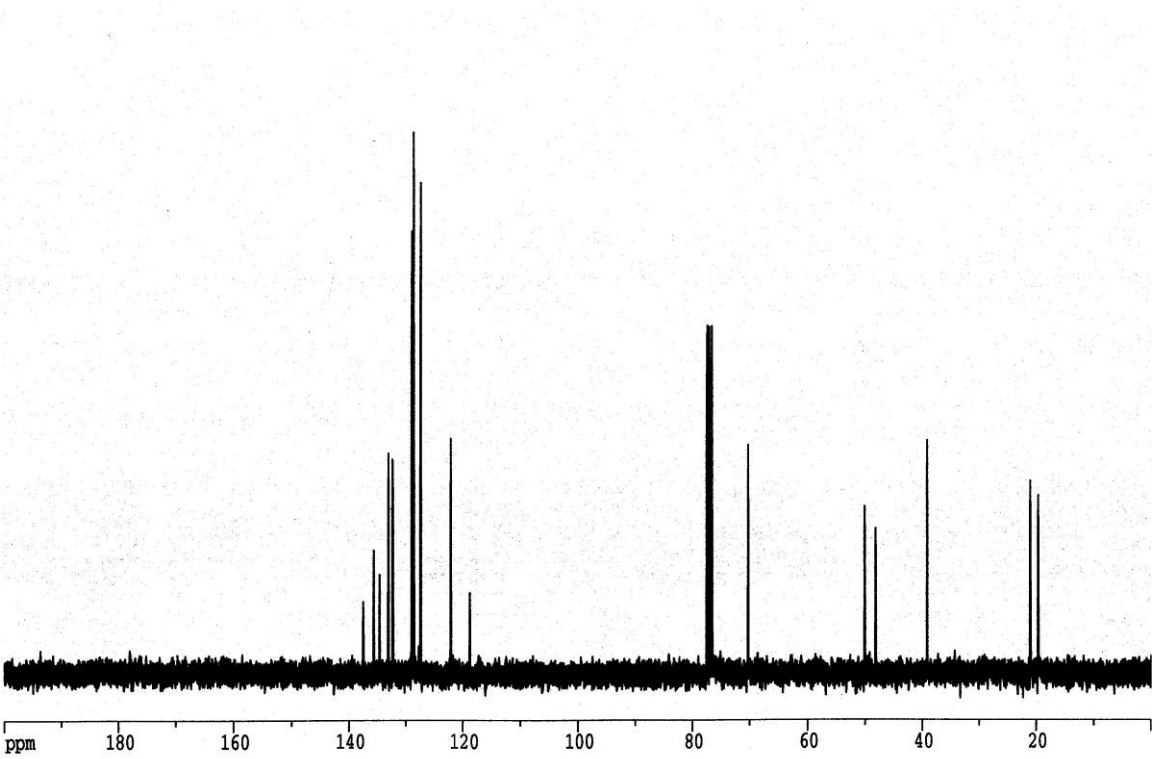




This journal is (c) The Royal Society of Chemistry 2009

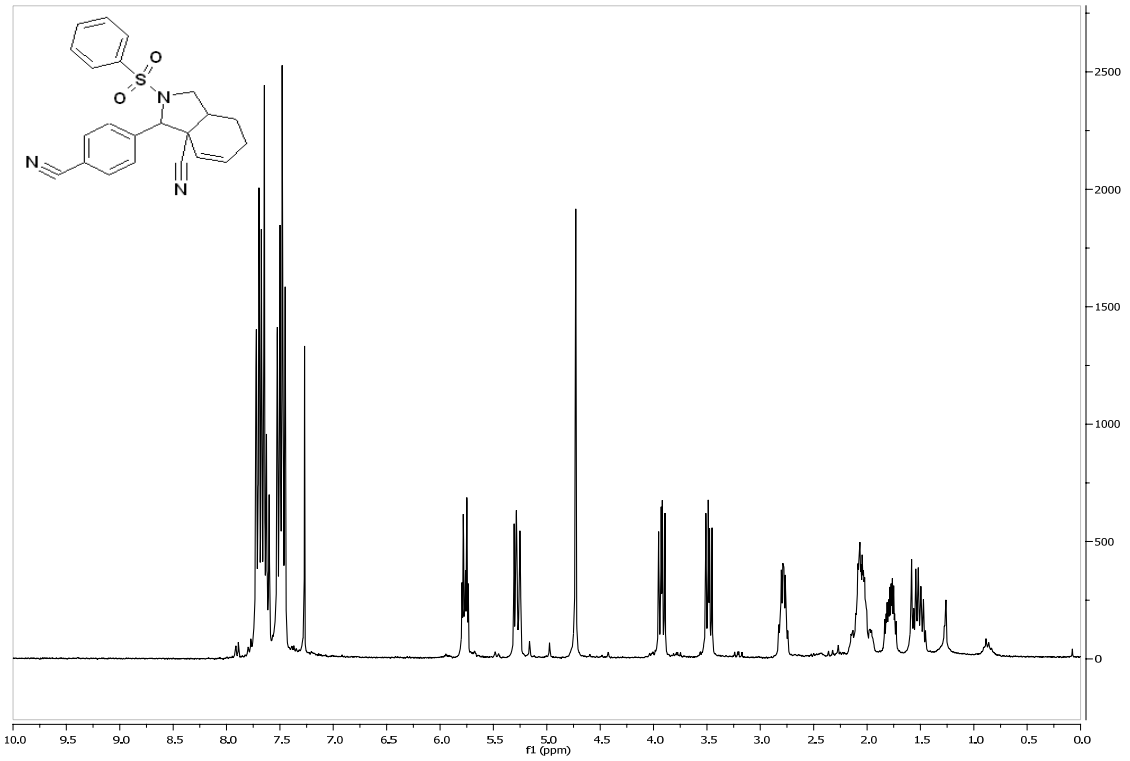
^{1}H and ^{13}C NMR Spectra of **11f**

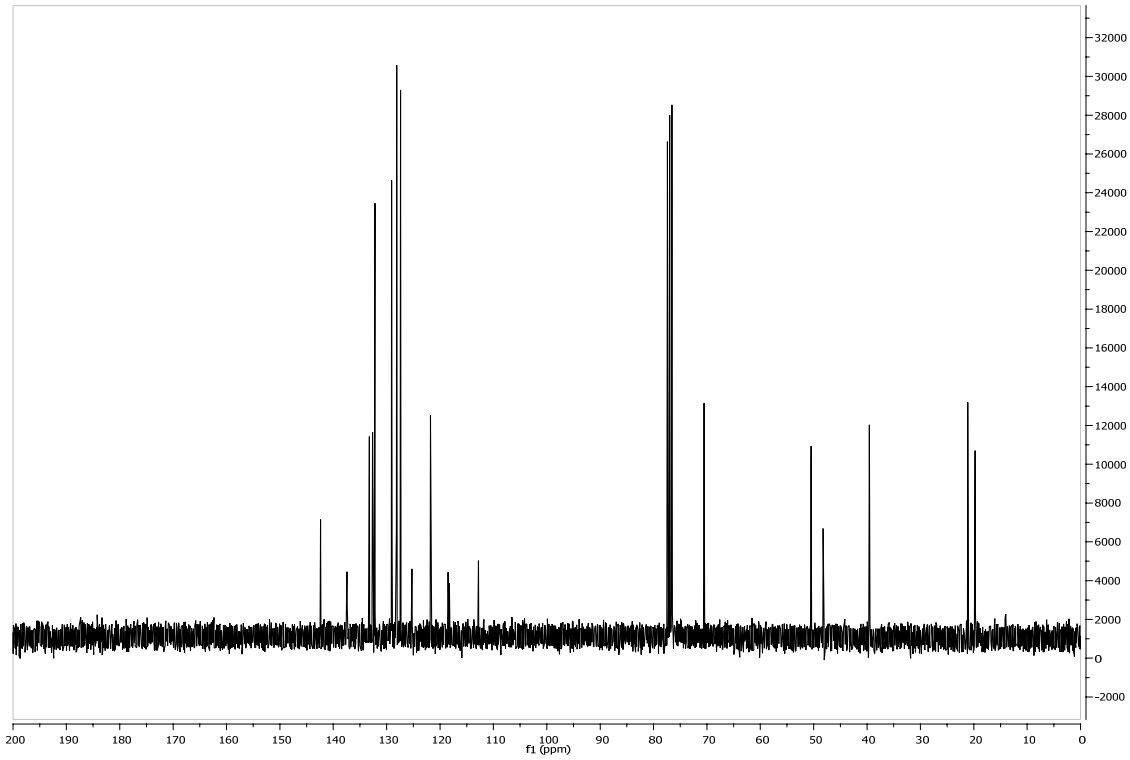




This journal is (c) The Royal Society of Chemistry 2009

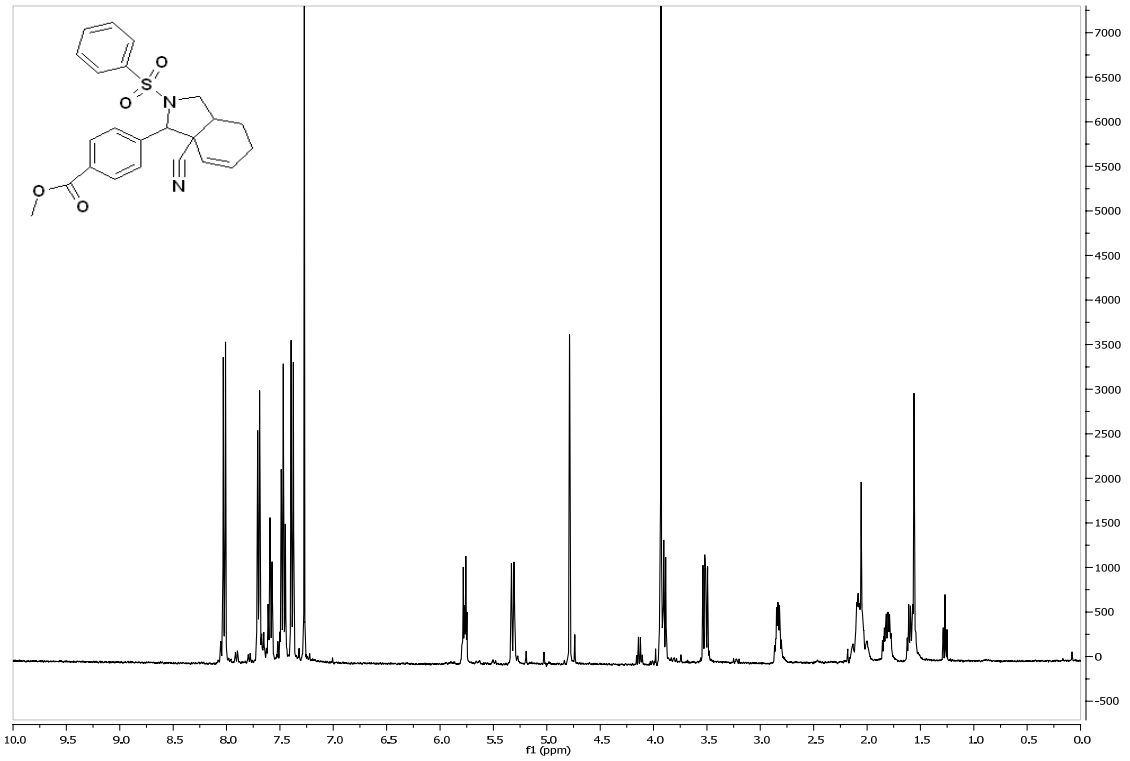
^{1}H and ^{13}C NMR Spectra of **11g**

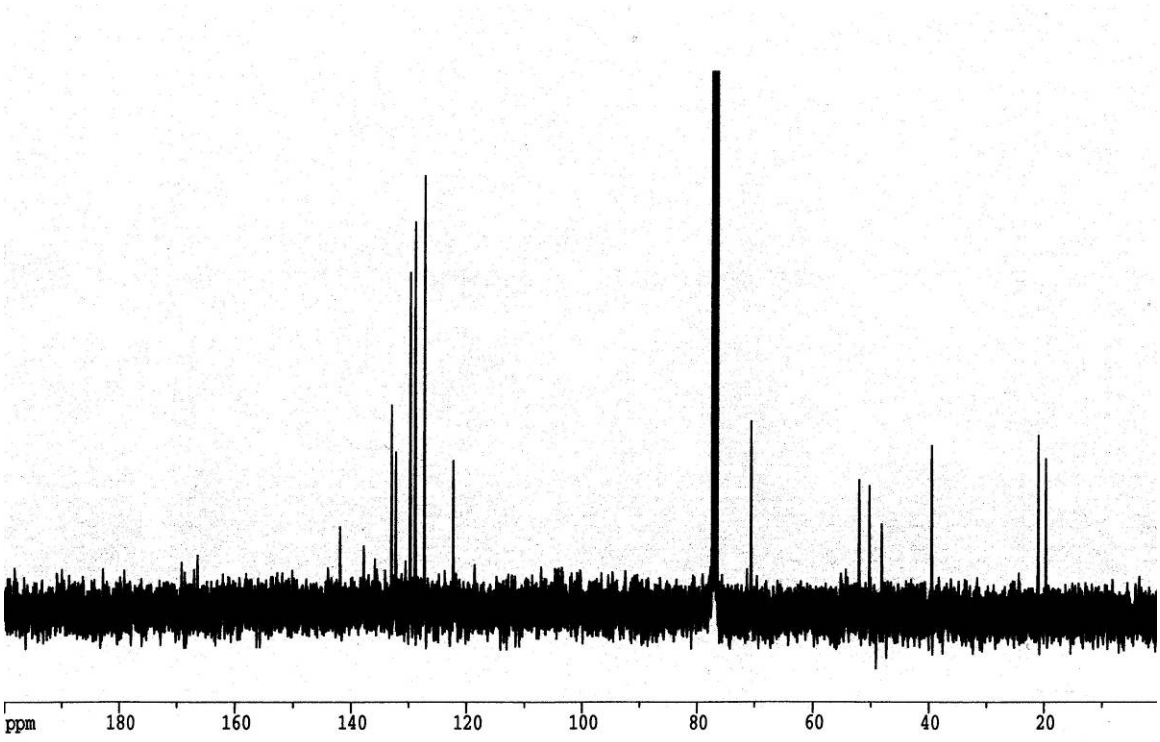




This journal is (c) The Royal Society of Chemistry 2009

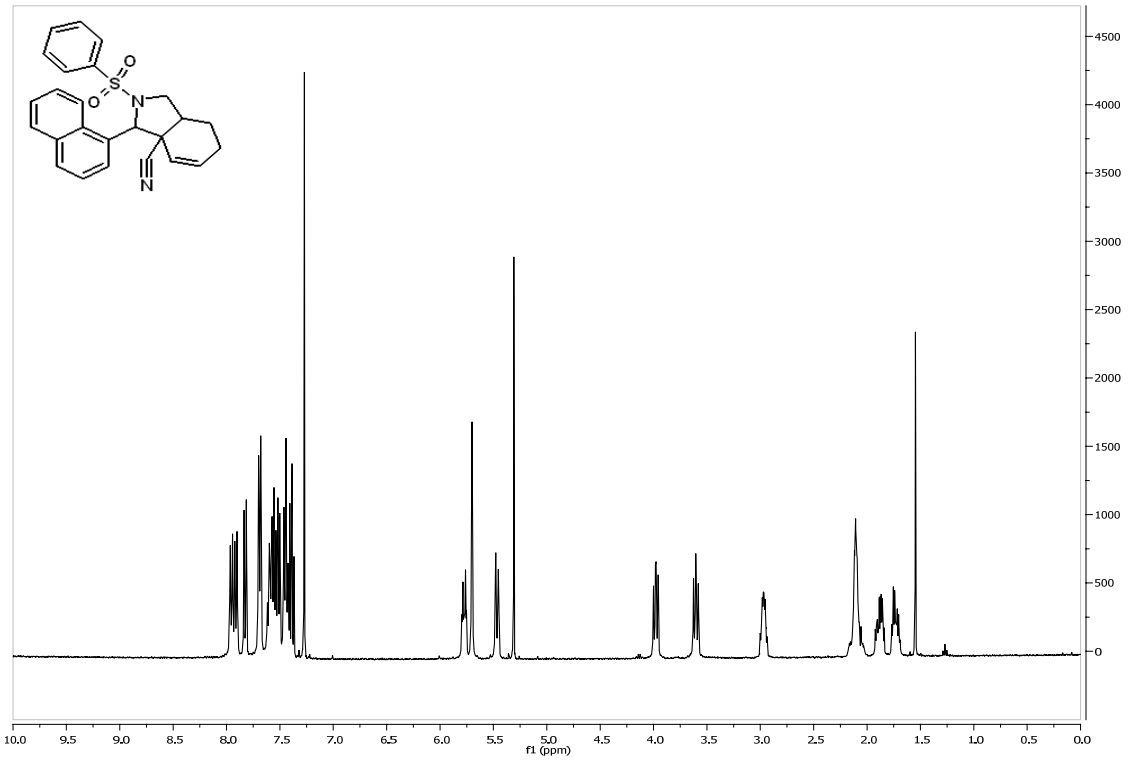
^{1}H and ^{13}C NMR Spectra of **11h**

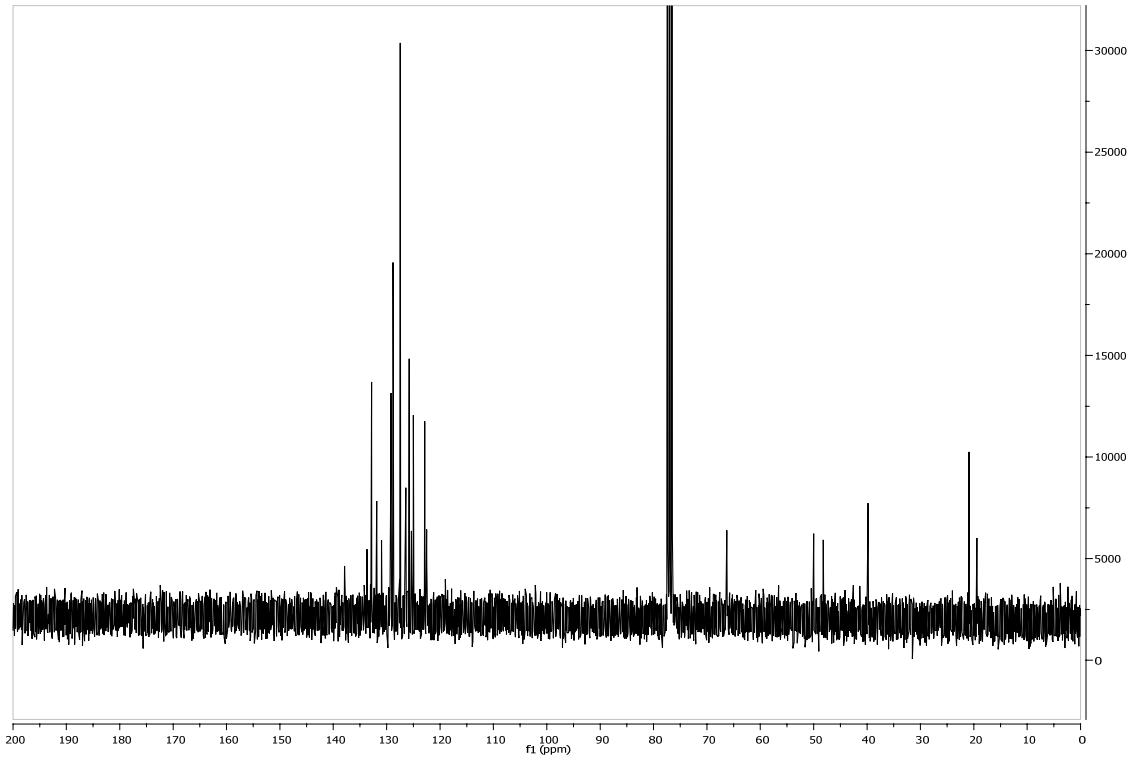




This journal is (c) The Royal Society of Chemistry 2009

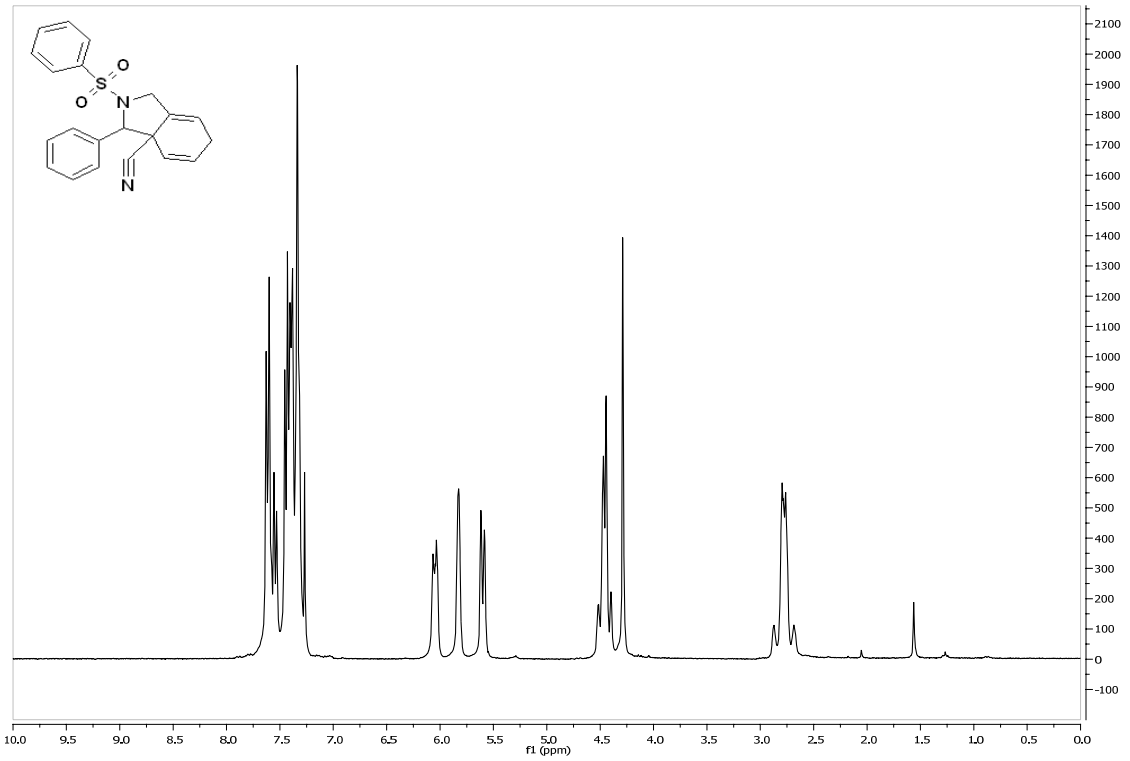
^{1}H and ^{13}C NMR Spectra of **11i**

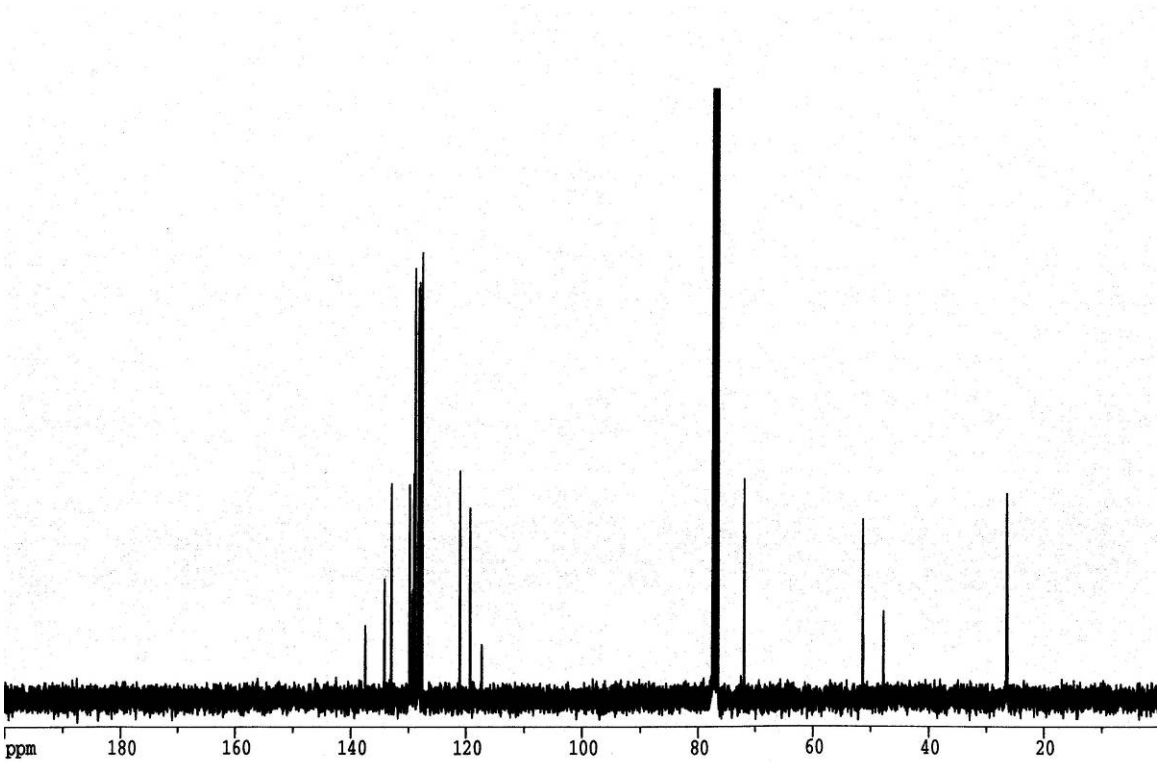




This journal is (c) The Royal Society of Chemistry 2009

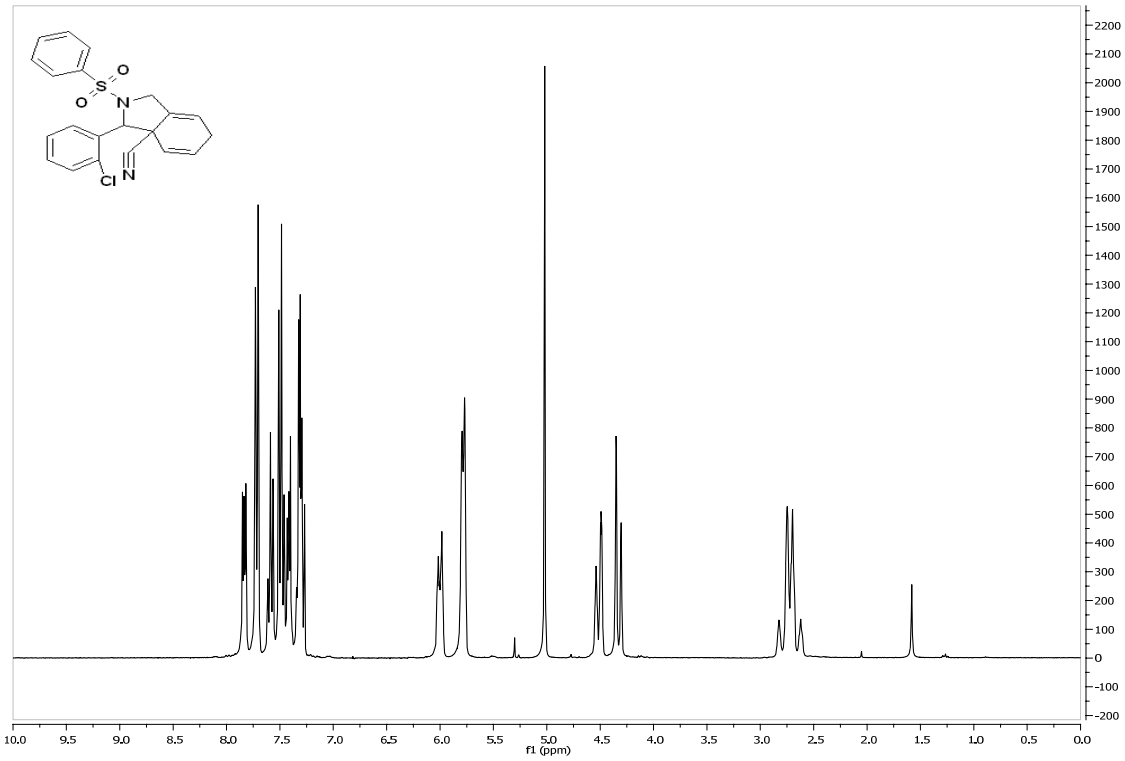
^{1}H and ^{13}C NMR Spectra of **12d**

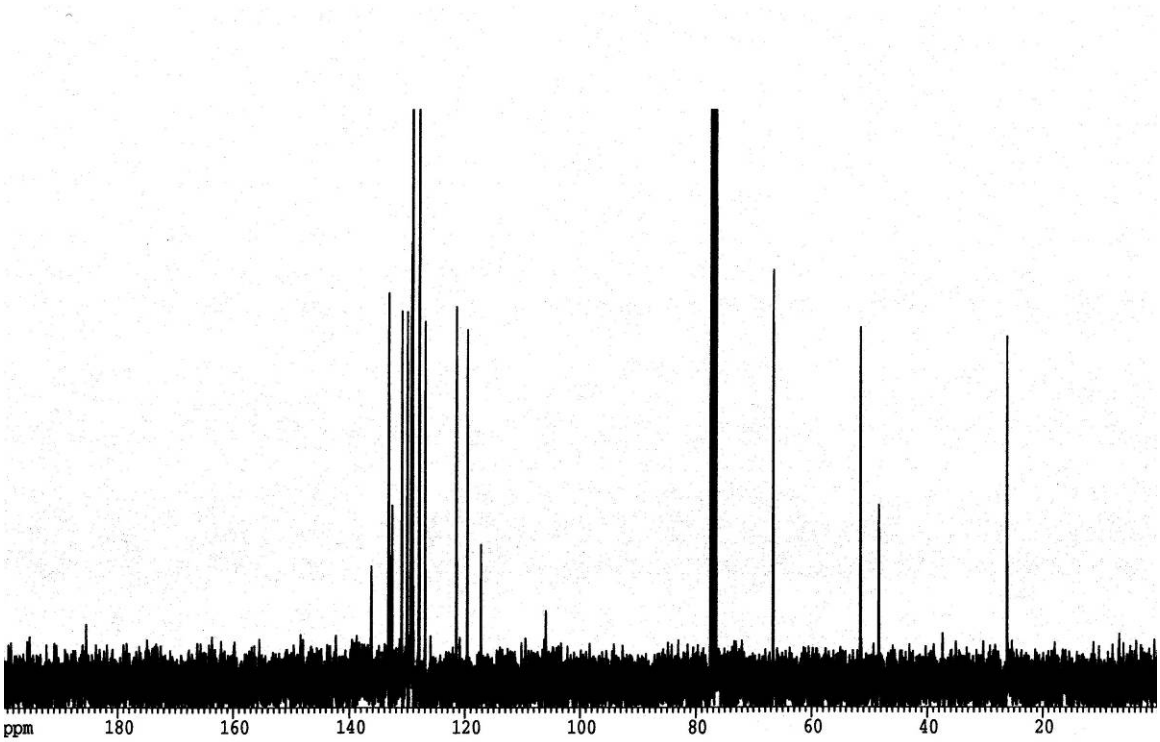




This journal is (c) The Royal Society of Chemistry 2009

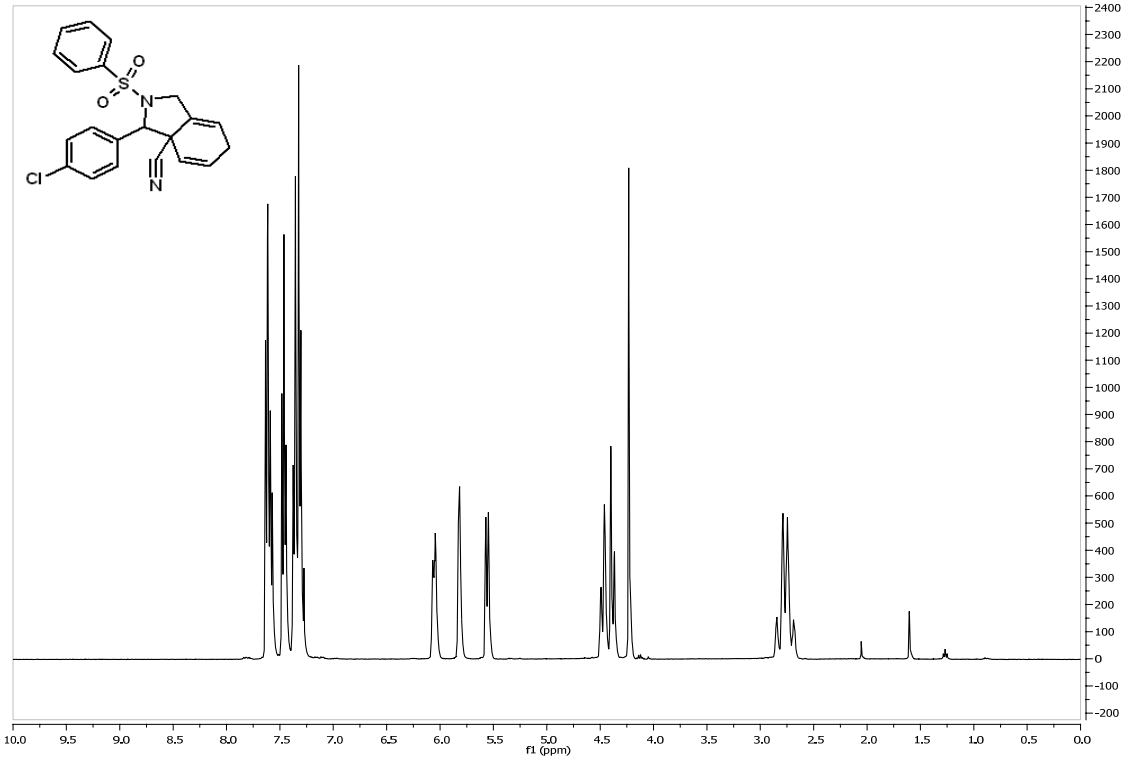
^{1}H and ^{13}C NMR Spectra of **12e**

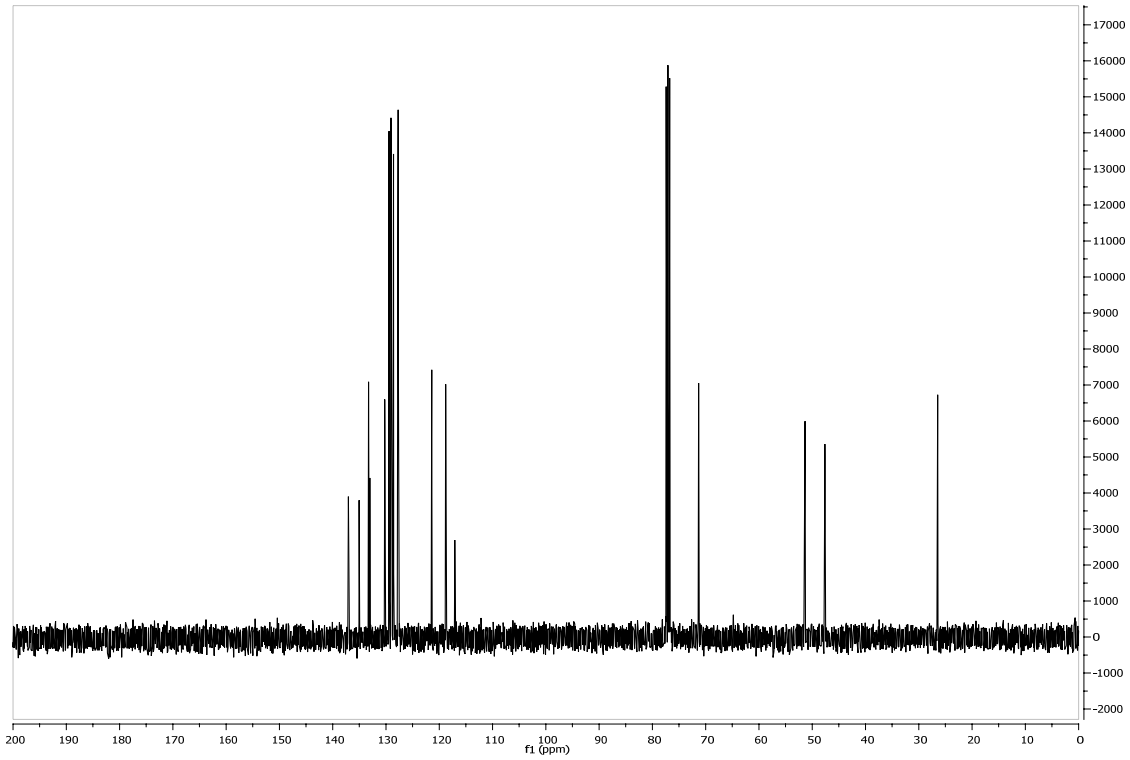




This journal is (c) The Royal Society of Chemistry 2009

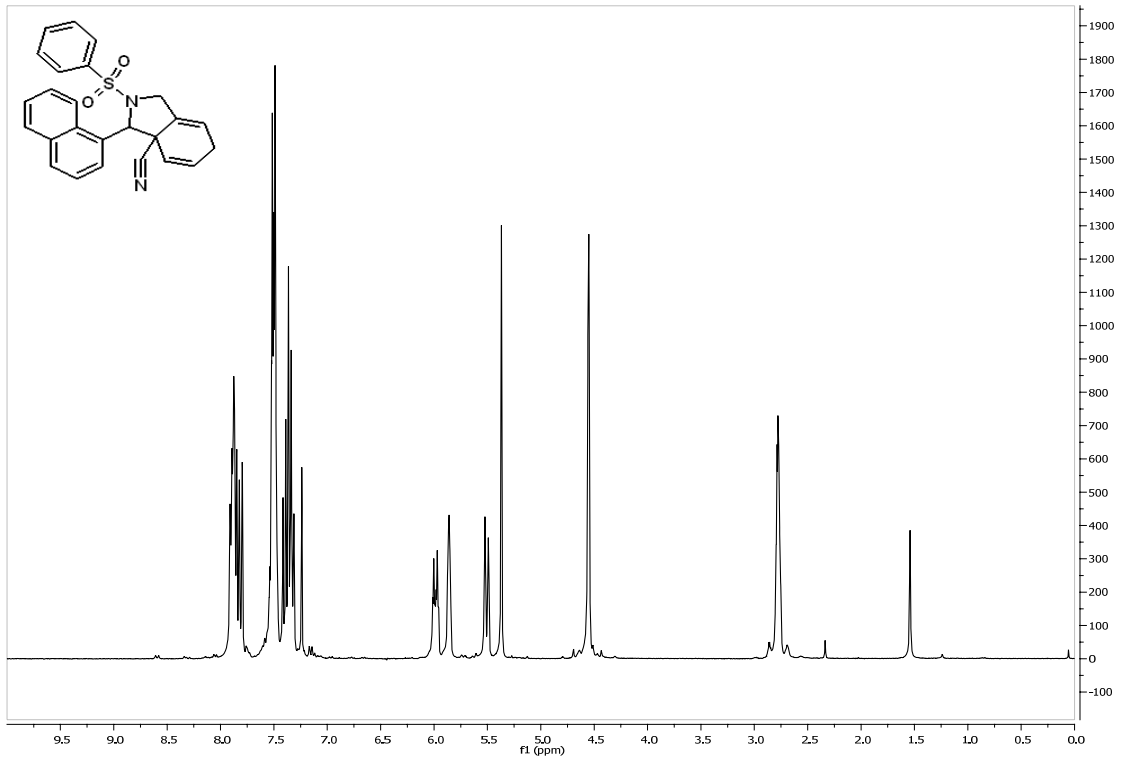
^{1}H and ^{13}C NMR Spectra of **12f**

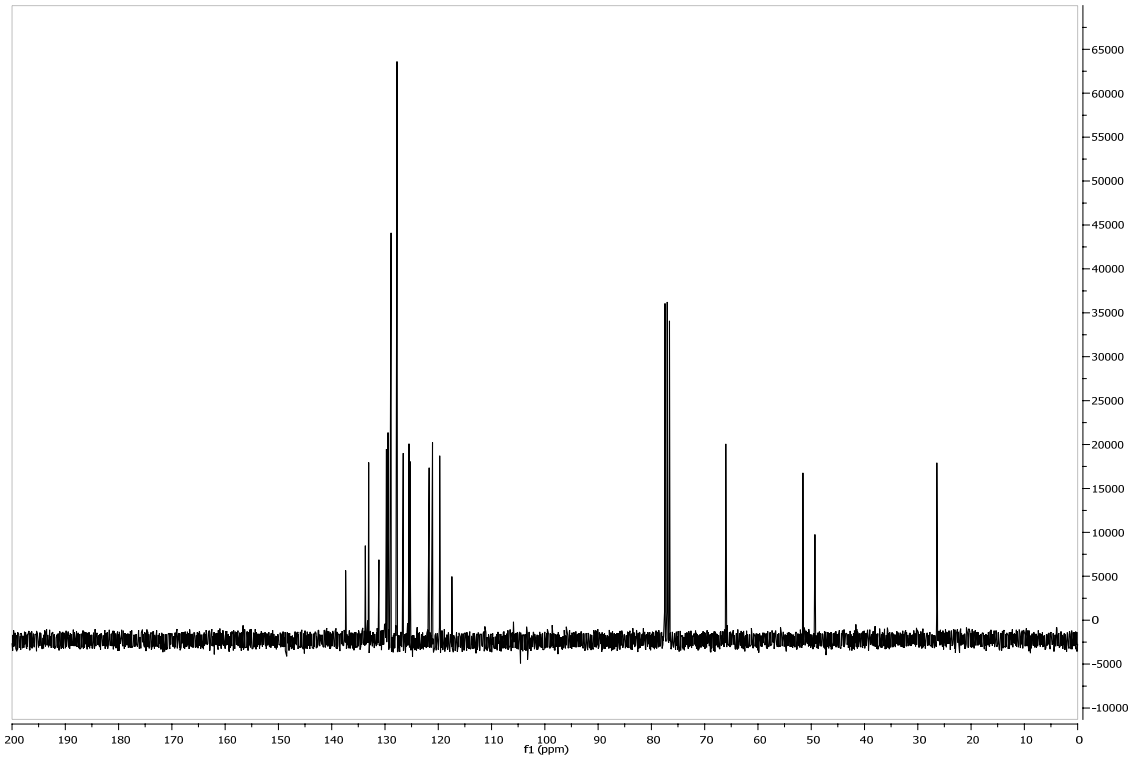




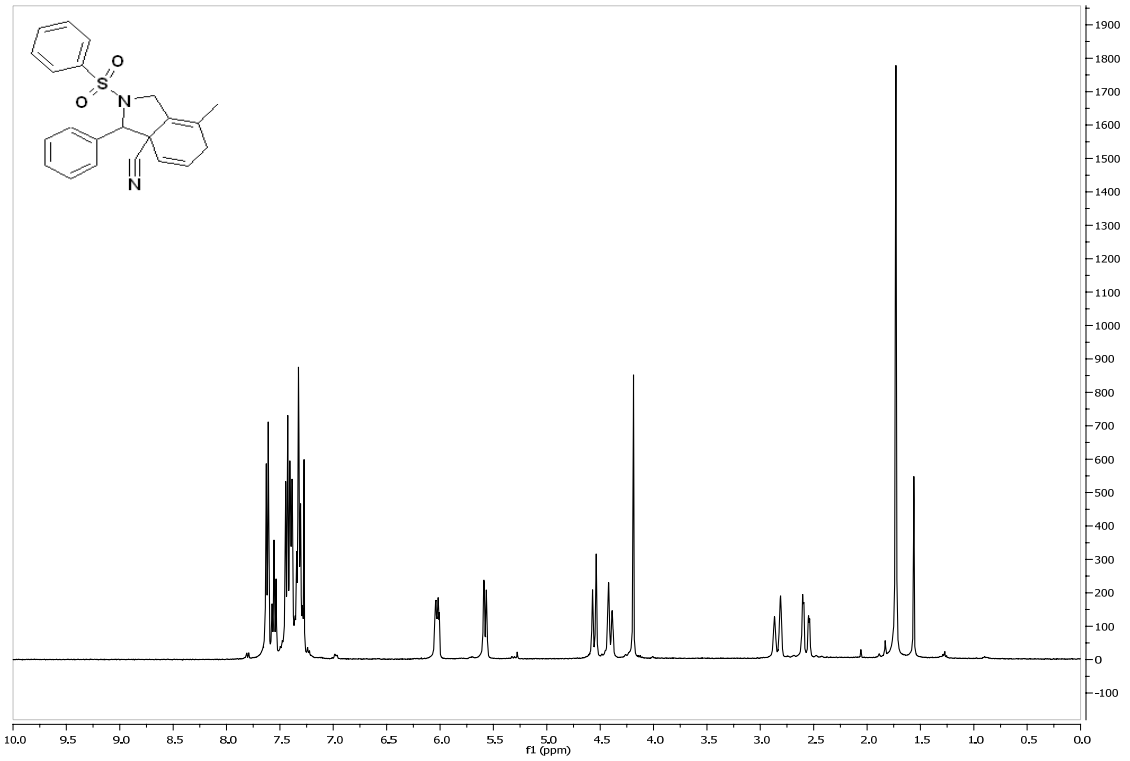
This journal is (c) The Royal Society of Chemistry 2009

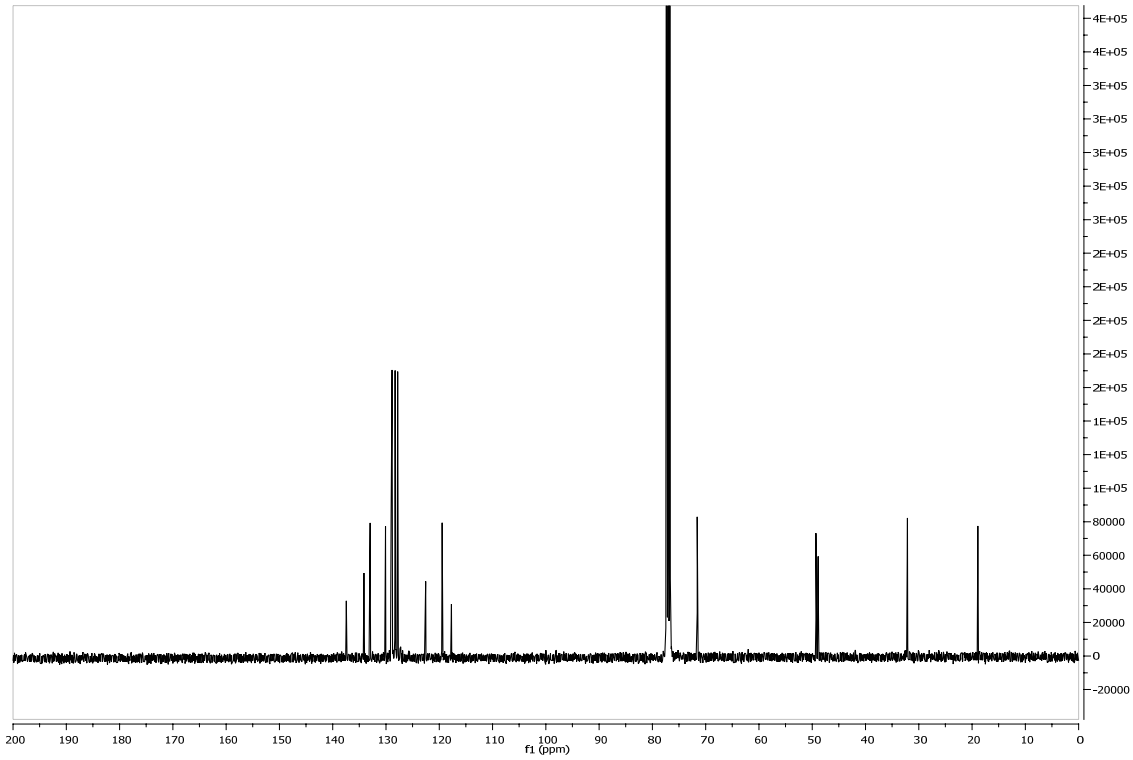
^{1}H and ^{13}C NMR Spectra of **12i**



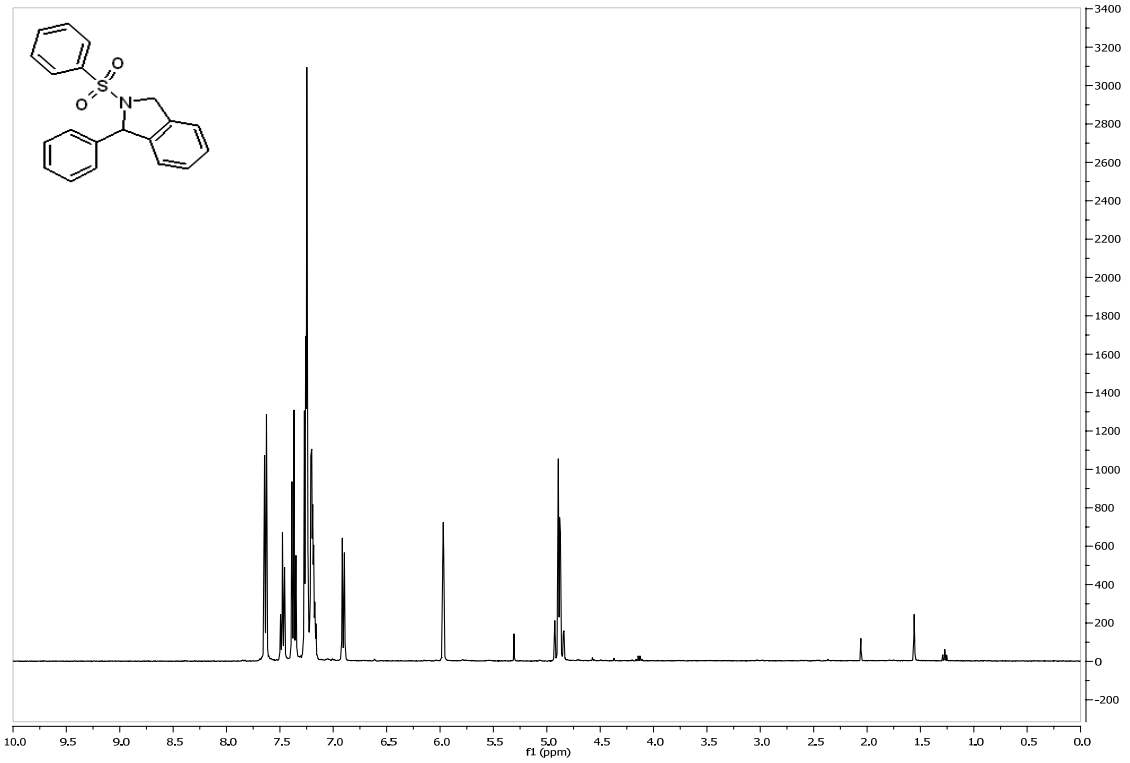


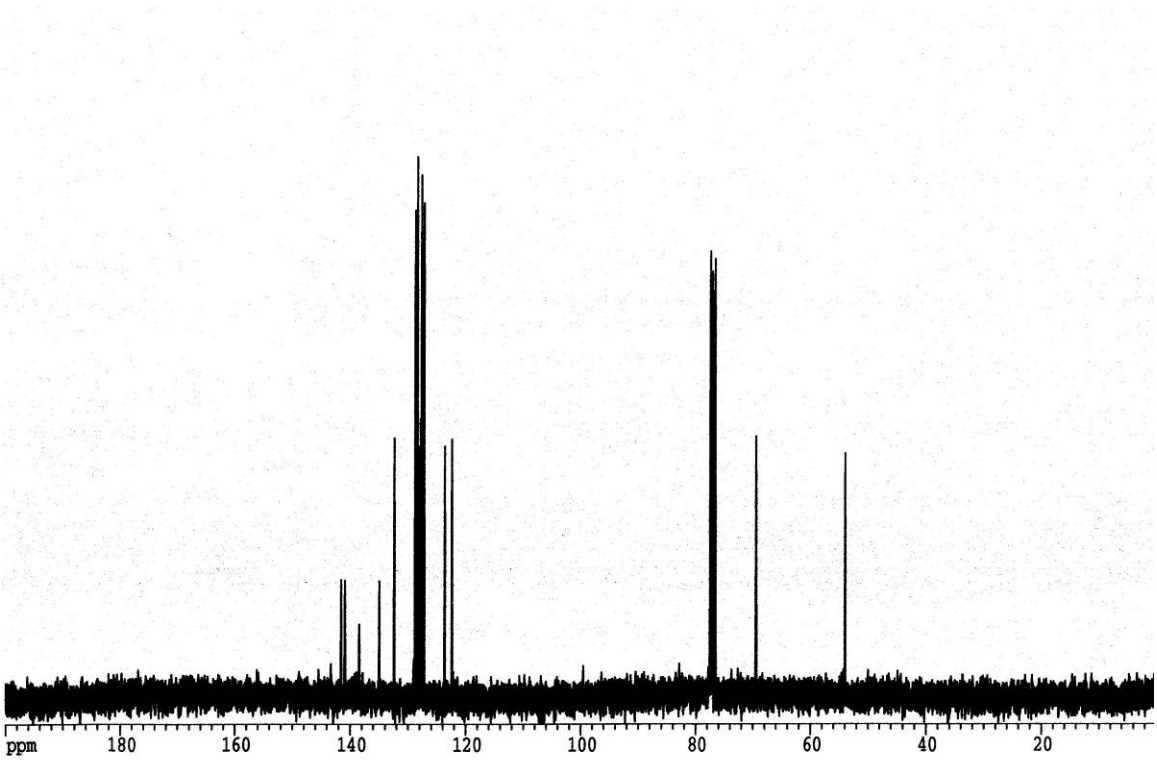
^{1}H and ^{13}C NMR Spectra of **13d**





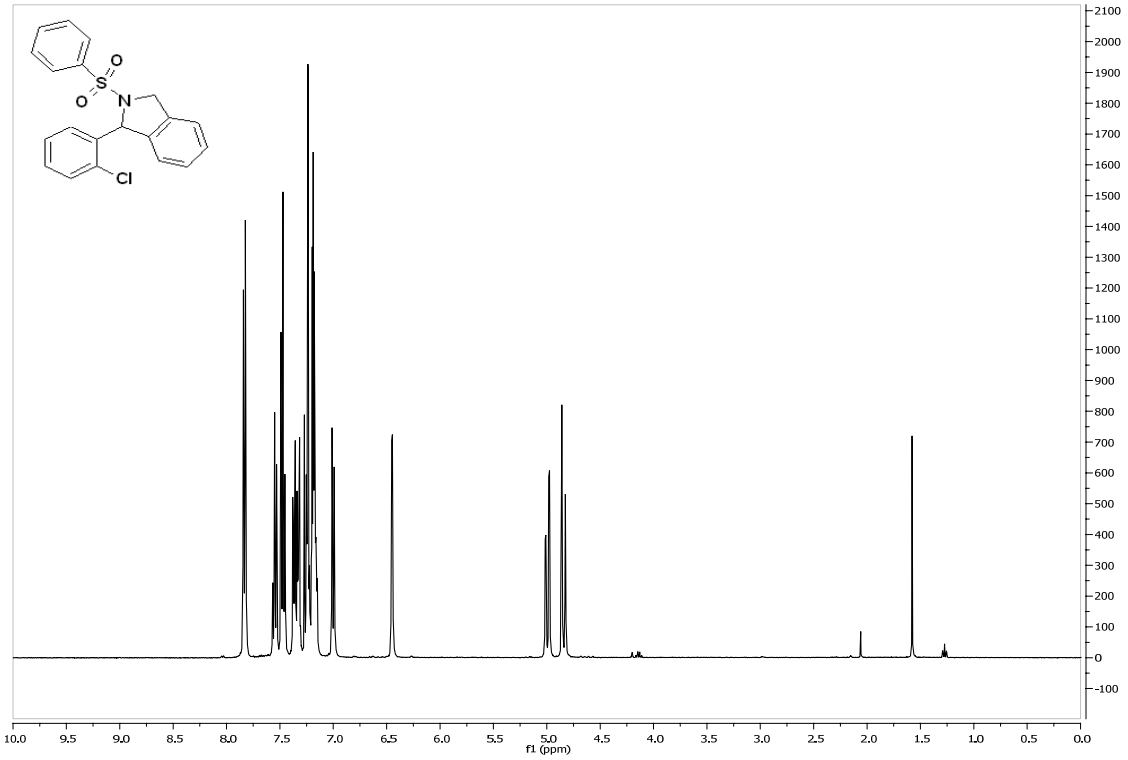
^{1}H and ^{13}C NMR Spectra of **14d**

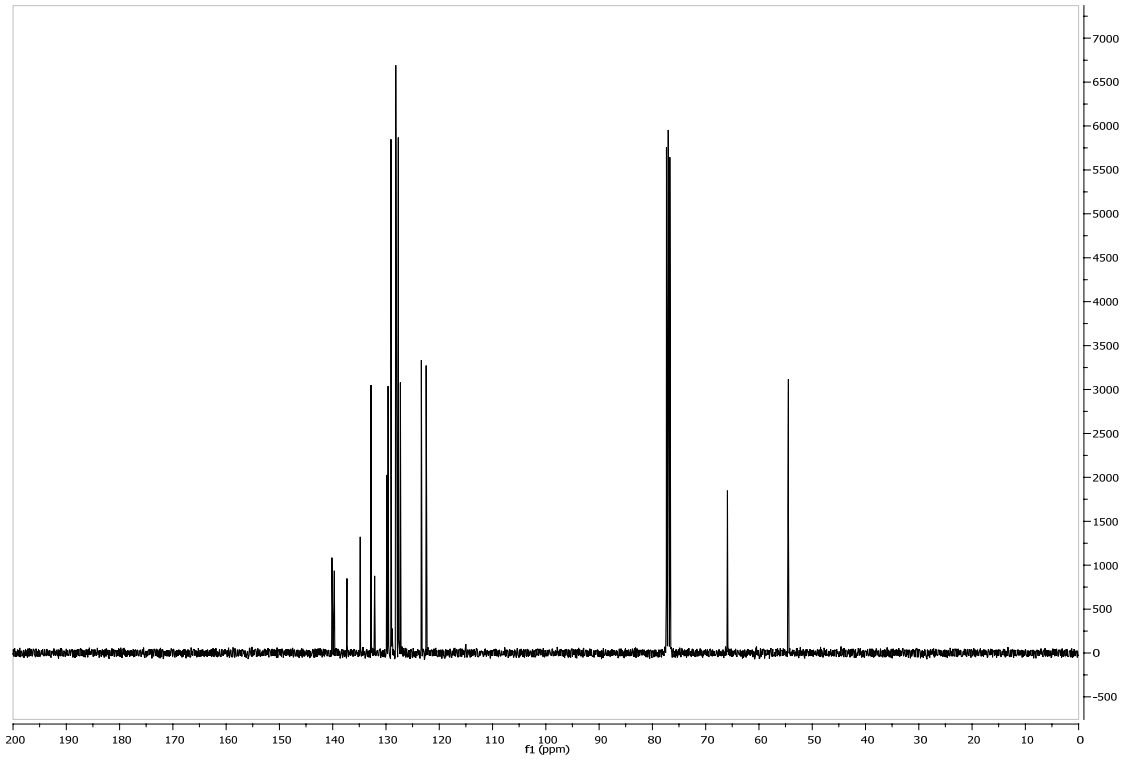




This journal is (c) The Royal Society of Chemistry 2009

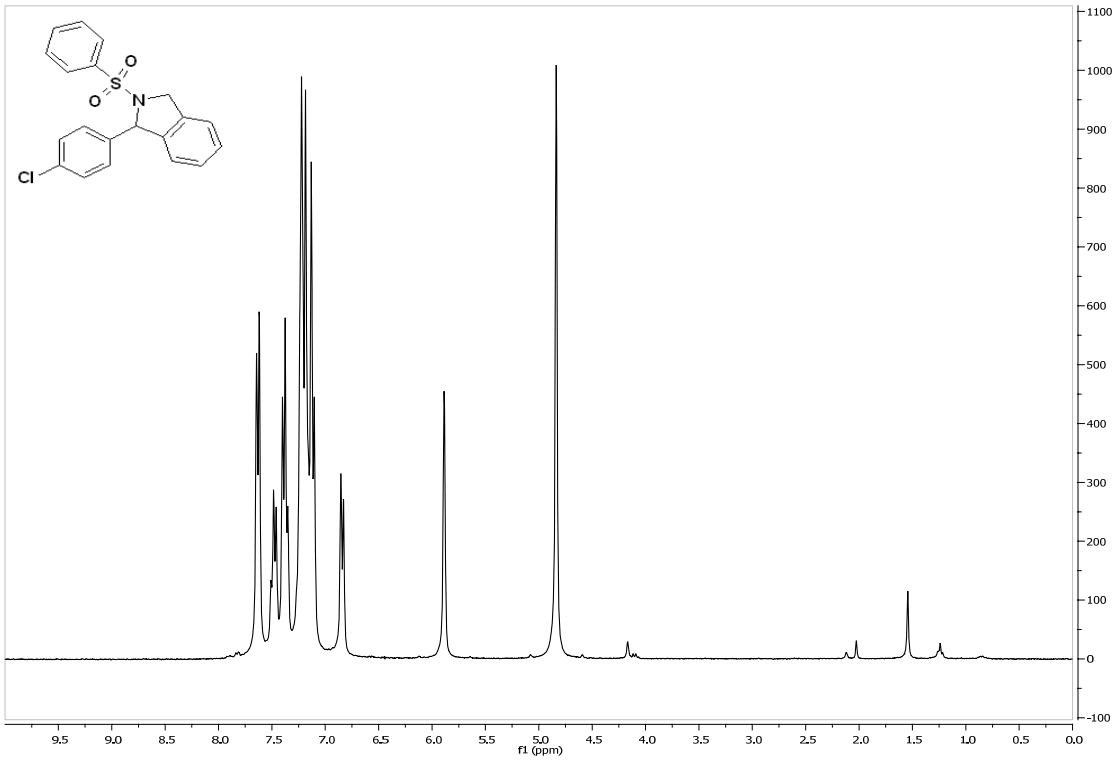
^{1}H and ^{13}C NMR Spectra of **14e**

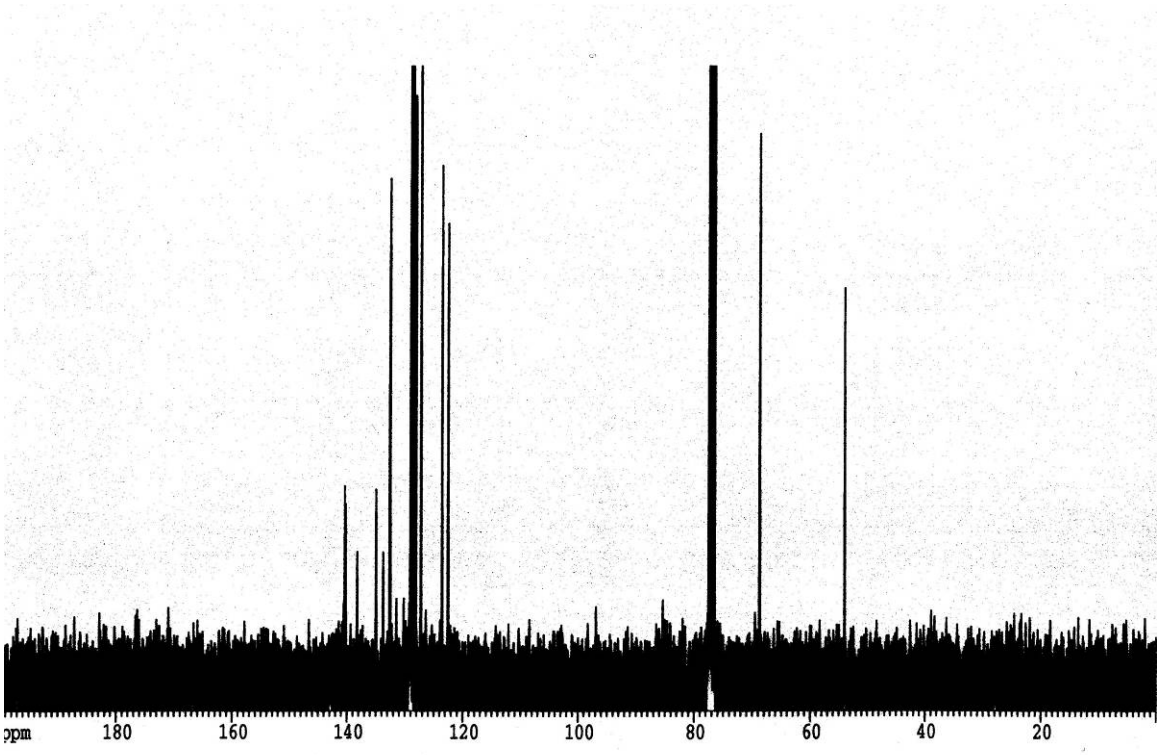




This journal is (c) The Royal Society of Chemistry 2009

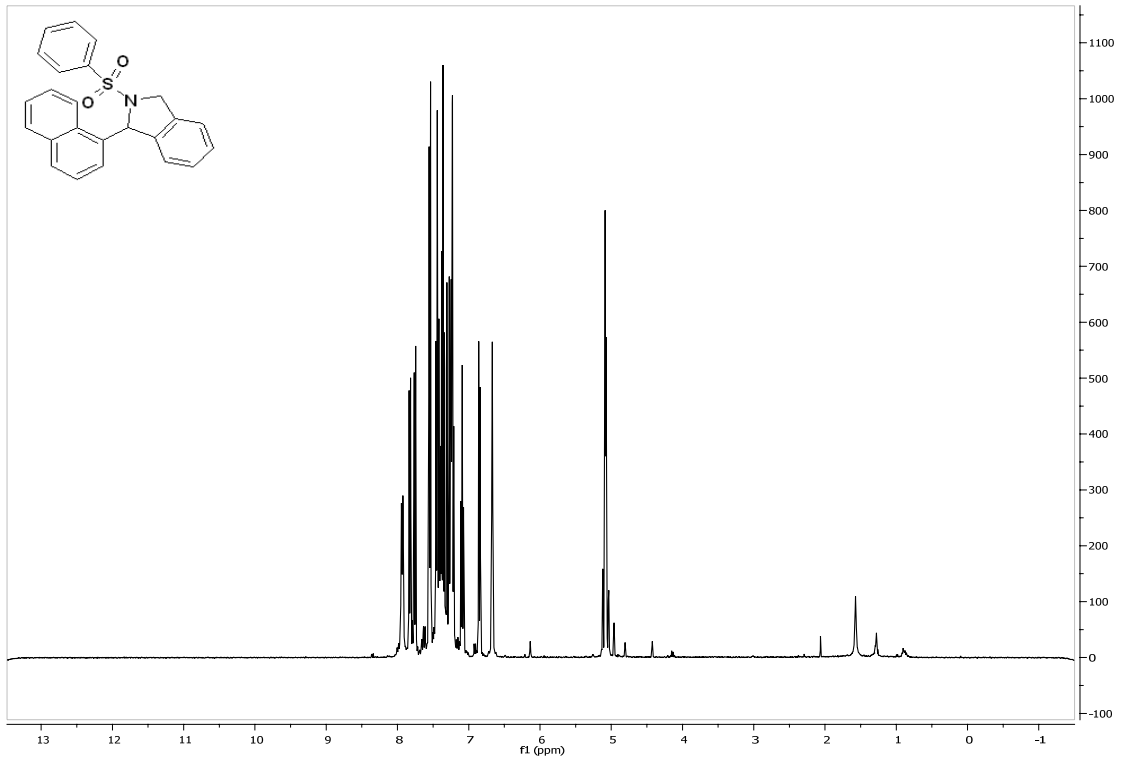
^{1}H and ^{13}C NMR Spectra of **14f**

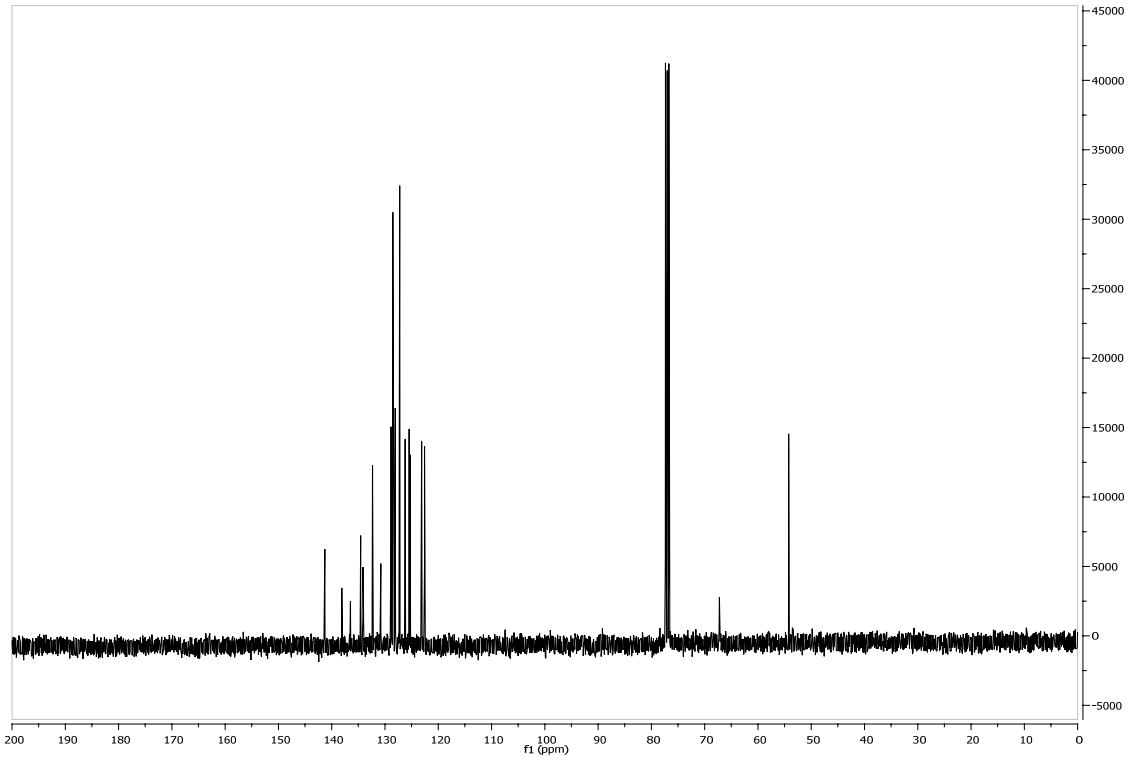




This journal is (c) The Royal Society of Chemistry 2009

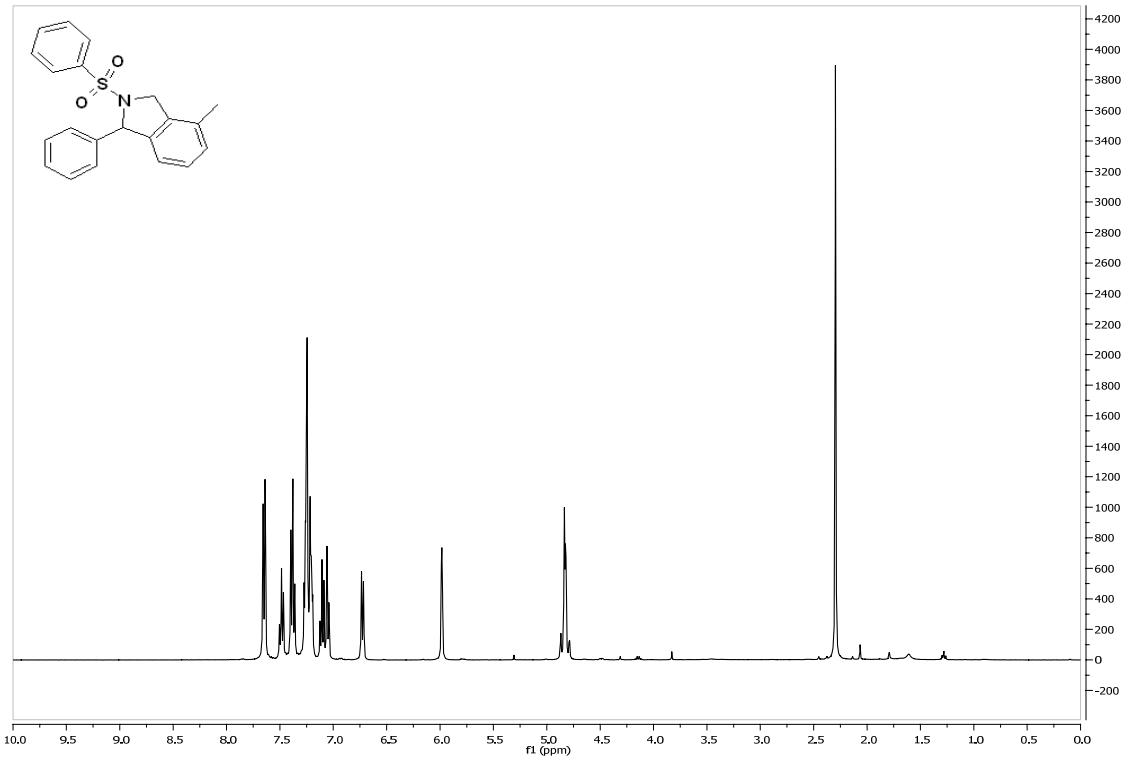
^{1}H and ^{13}C NMR Spectra of **14i**

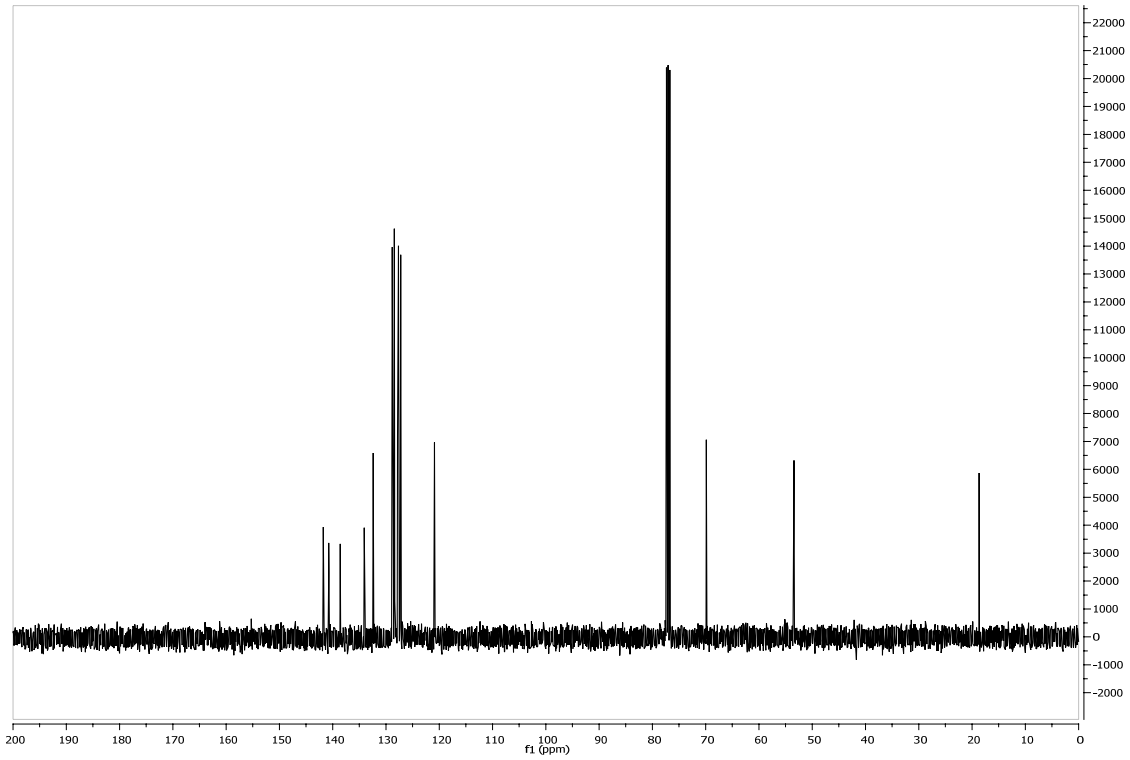




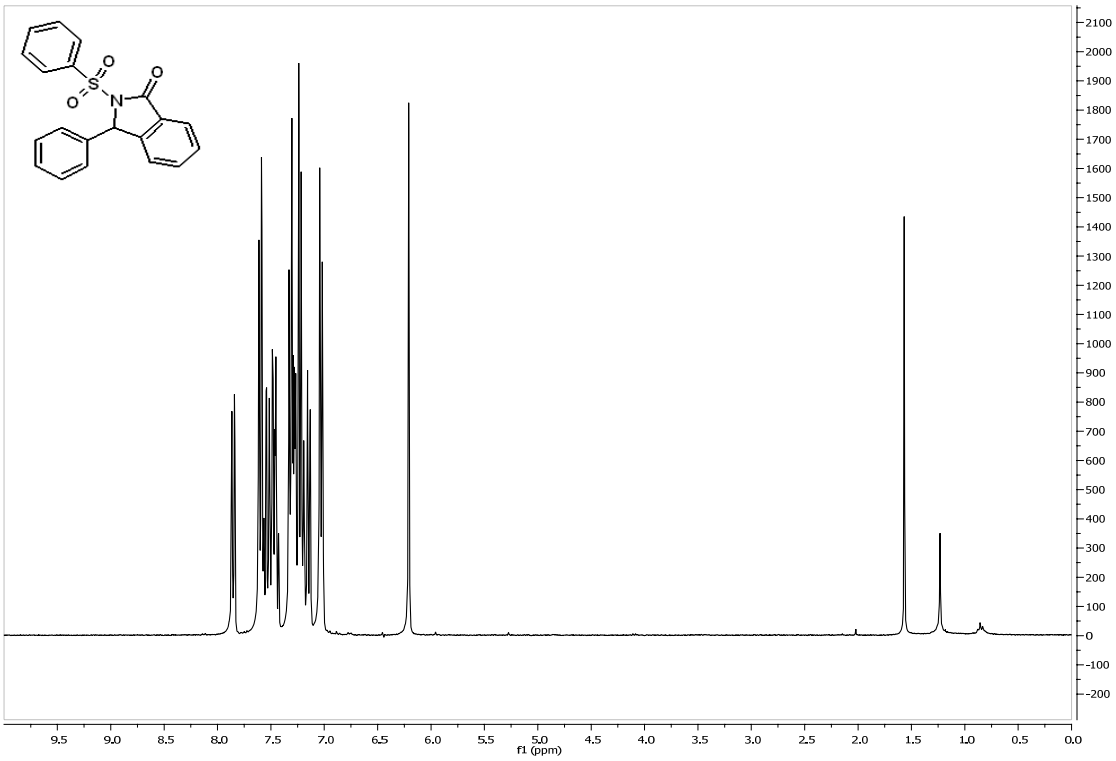
This journal is (c) The Royal Society of Chemistry 2009

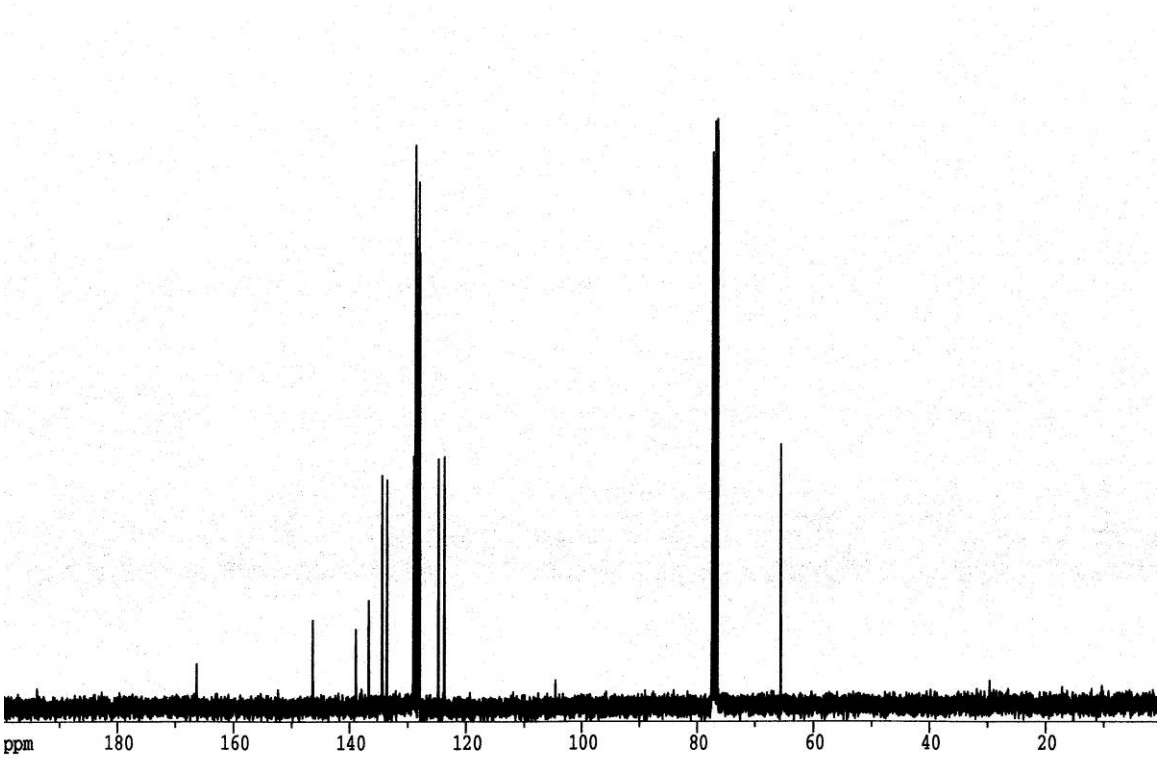
^{1}H and ^{13}C NMR Spectra of **15d**





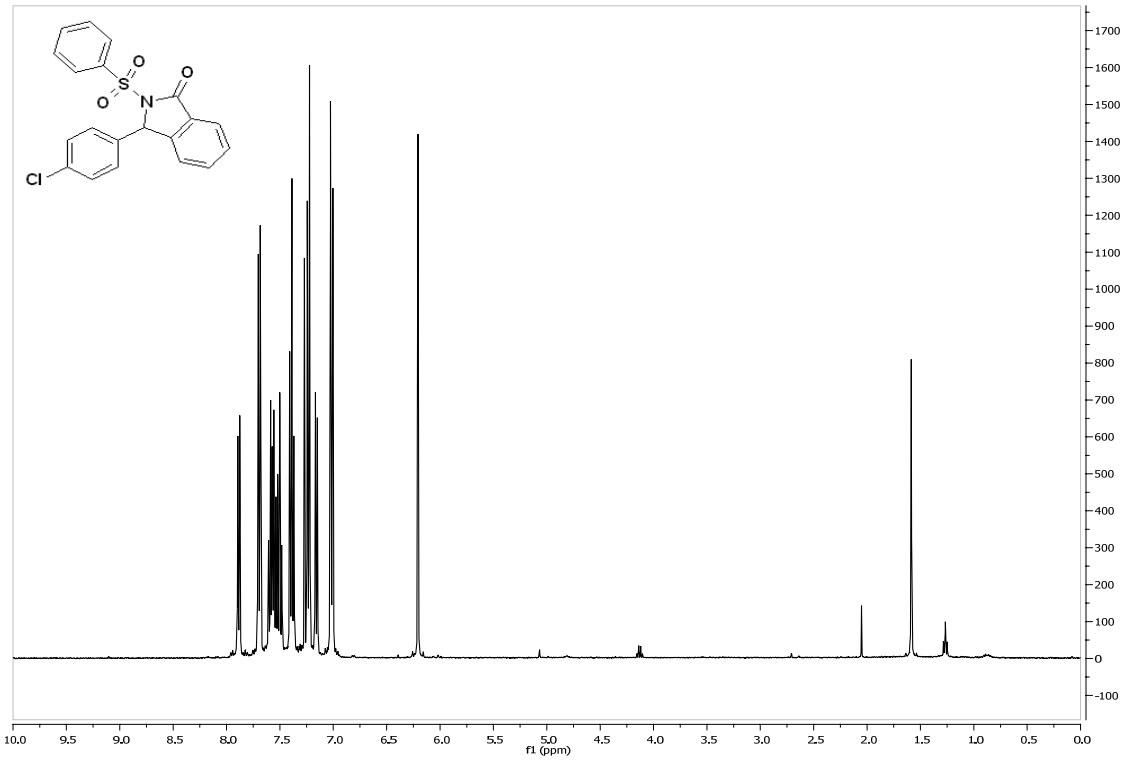
^{1}H and ^{13}C NMR Spectra of **16d**

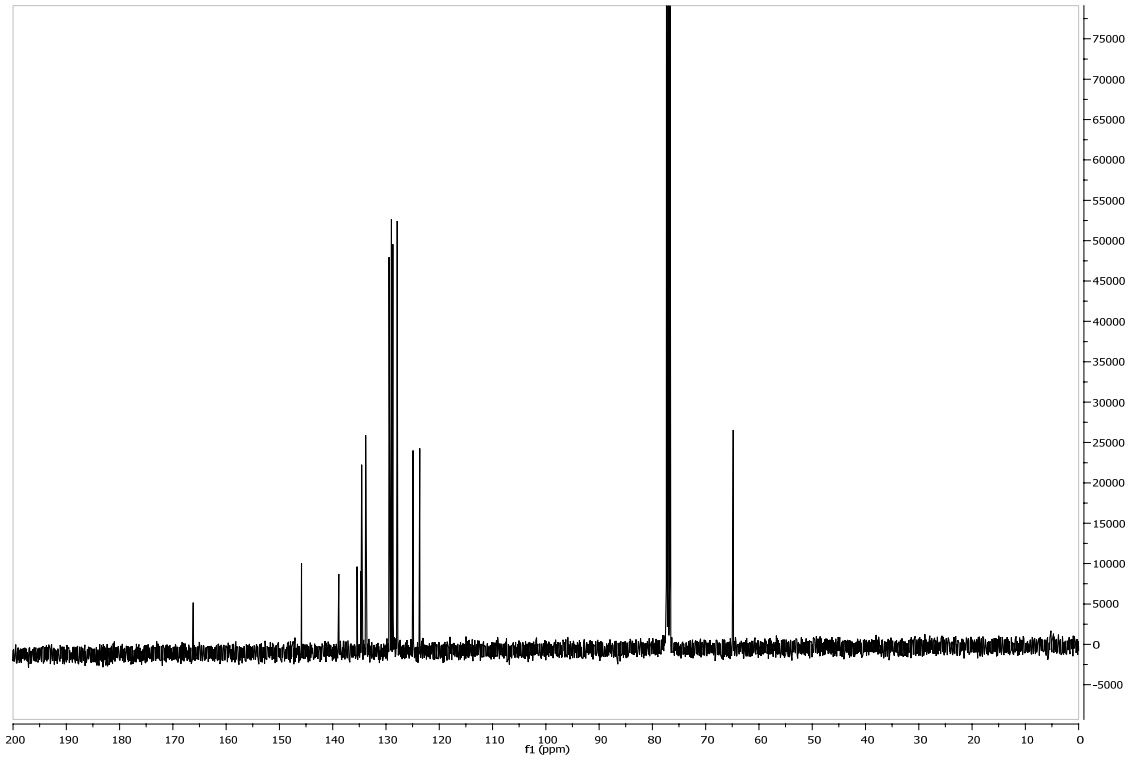




This journal is (c) The Royal Society of Chemistry 2009

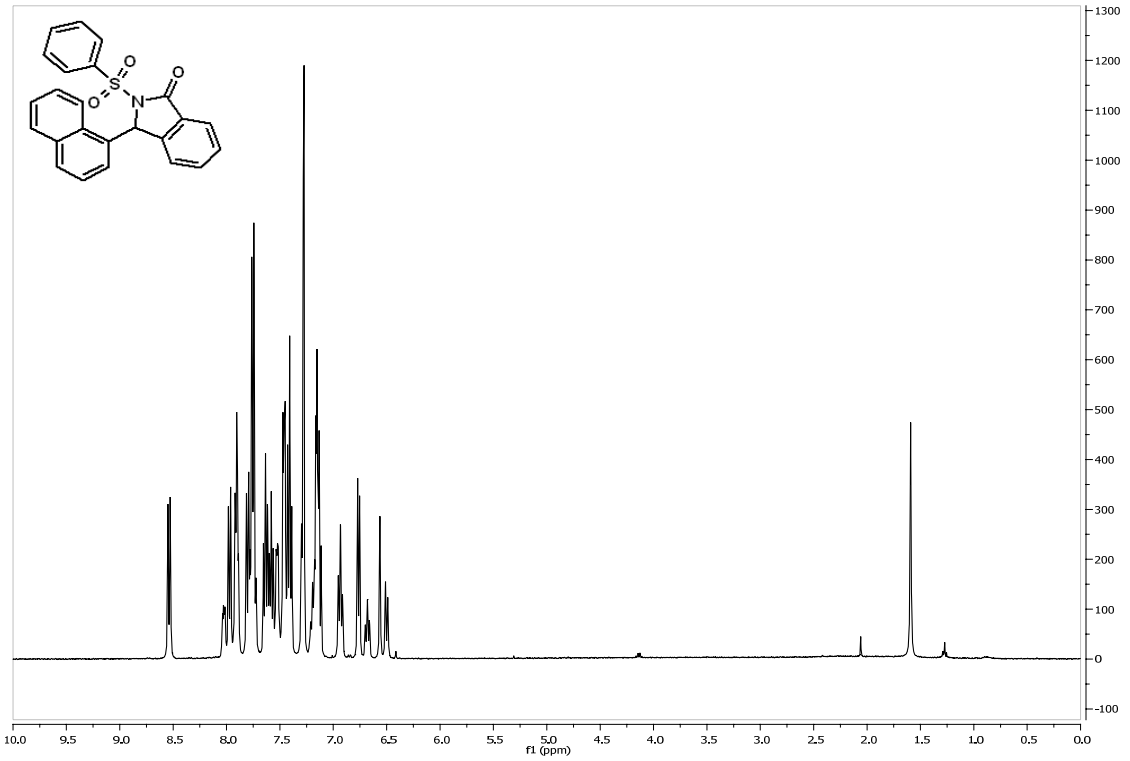
^{1}H and ^{13}C NMR Spectra of **16f**

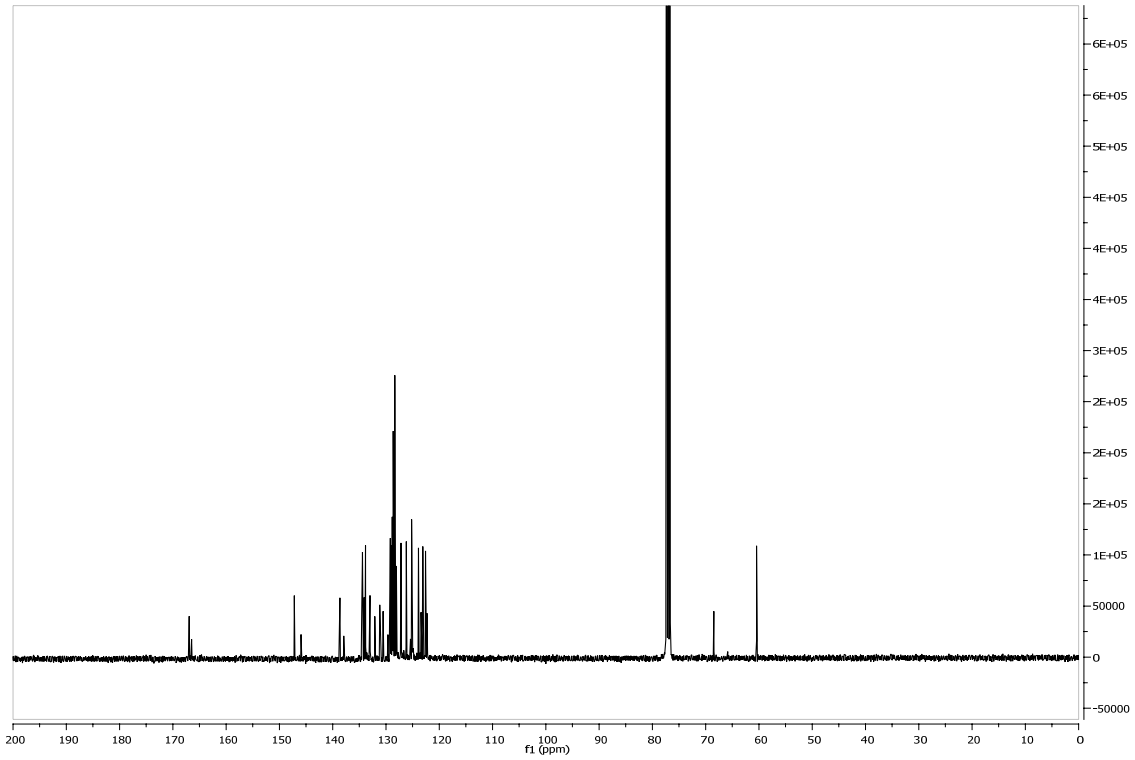




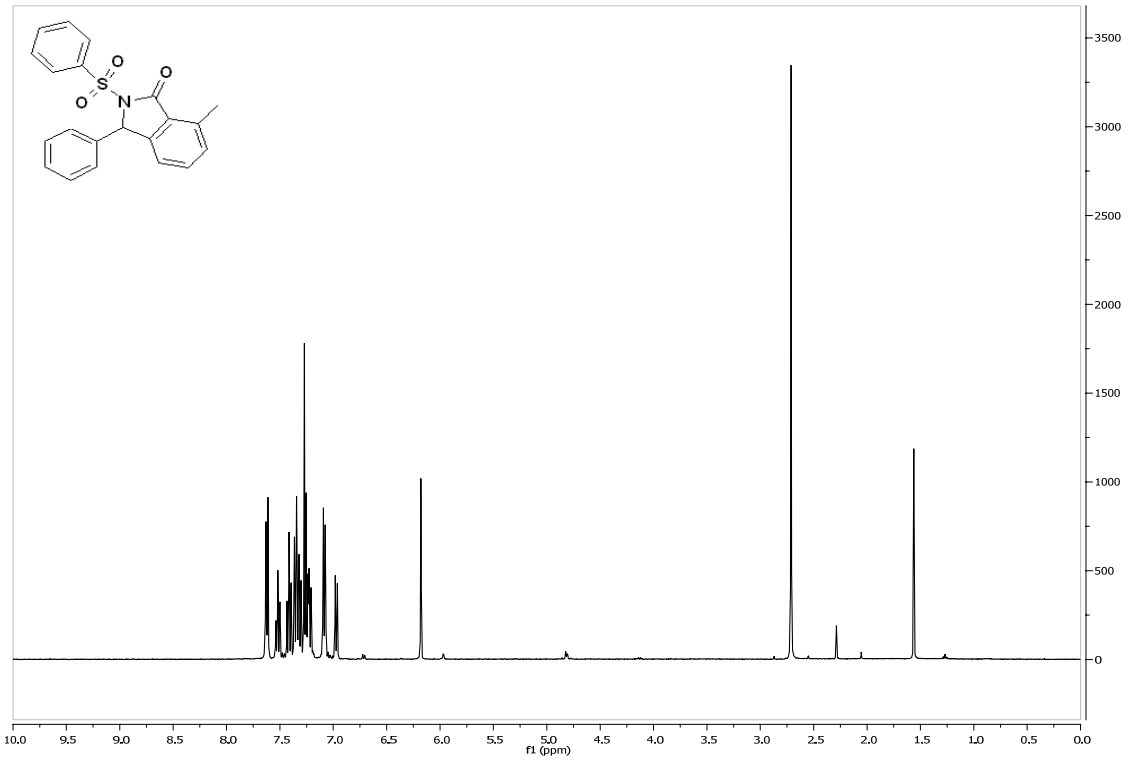
This journal is (c) The Royal Society of Chemistry 2009

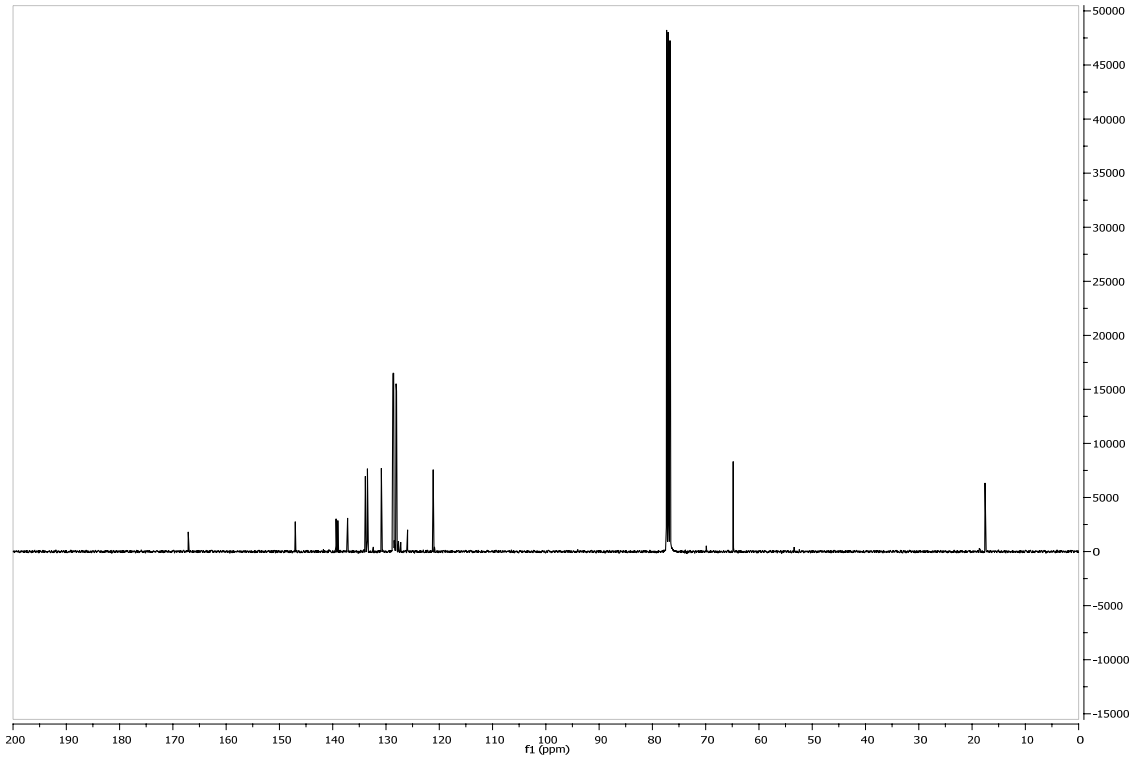
^{1}H and ^{13}C NMR Spectra of **16i**





^{1}H and ^{13}C NMR Spectra of **17d**





X-Ray Crystallographic Data for 11b.

A colorless prismatic crystal of C_{27} H_{26} Cl N O_4 S_2 was coated with Paratone 8277 oil (Exxon) and mounted on a glass fiber. All measurements were made on a Nonius KappaCCD diffractometer with graphite monochromated Mo-Kα radiation. Details of crystal data and structure refinement have been provided in Table 1. The data were collected[1] using ω and φ scans. The data were corrected for Lorentz and polarization effects and for absorption using multi-scan method[2].

The structure was solved by the direct methods[3] and expanded using Fourier techniques[4]. The non-hydrogen atoms were refined anisotropically. The H-atoms were included at geometrically idealized positions and were not refined. The final cycle of full-matrix least-squares refinement using SHELXL97[5] converged with unweighted and weighted agreement factors, R = 0.039 and wR = 0.103 (all data), respectively, and goodness of fit, S = 1.03. The weighting scheme was based on counting statistics and the final difference Fourier map was essentially featureless. The figure was plotted with the aid of ORTEPII[6].

Table 1. Crystal data and structure refinement for C_{27} H_{26} Cl N O_4 S_2.

Empirical formula	C_{27} H_{26} Cl N O_4 S_2	
Formula weight	528.06	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 9.968(3) Å	α= 86.210(17)°.
	b = 11.772(3) Å	β= 72.991(15)°.
	c = 12.075(4) Å	γ = 65.948(17)°.
Volume	1235.0(6) $Å^3$	
Z	2	
Density (calculated)	1.420 Mg/m^3	
Absorption coefficient	0.359 mm^{-1}	
F(000)	552	
Crystal size	0.16 x 0.14 x 0.10 mm^3	

This journal is (c) The Royal Society of Chemistry 2009

Theta range for data collection	3.6 to 27.5°.
Index ranges	-12<=h<=12, -15<=k<=15, -15<=l<=15
Reflections collected	10359
Independent reflections	5589 [R(int) = 0.026]
Completeness to theta = 27.5°	98.9 %
Absorption correction	Multi-scan method
Max. and min. transmission	0.9650 and 0.9448
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5589 / 0 / 316
Goodness-of-fit on F^2	1.03
Final R indices [I>2sigma(I)]	R1 = 0.039, wR2 = 0.093
R indices (all data)	R1 = 0.057, wR2 = 0.103
Largest diff. peak and hole	0.34 and -0.41 e.$Å^{-3}$

This journal is (c) The Royal Society of Chemistry 2009

X-Ray Crystallographic Data for 11d.

A colorless prismatic crystal of $C_{21}H_{20}N_2O_2S$ was coated with Paratone 8277 oil (Exxon) and mounted on a glass fiber. All measurements were made on a Nonius KappaCCD diffractometer with graphite monochromated Mo-Kα radiation. Details of crystal data and structure refinement have been provided in Table 1. The data were collected[1] using ω and φ scans. The data were corrected for Lorentz and polarization effects and for absorption using multi-scan method[2].

The structure was solved by the direct methods[3] and expanded using Fourier techniques[4]. The non-hydrogen atoms were refined anisotropically. The H-atoms were located from a difference map and were included at geometrically idealized positions in a riding mode. The final cycle of full-matrix least-squares refinement using SHELXL97[5] converged with unweighted and weighted agreement factors, R = 0.0474 and wR = 0.1263 (all data), respectively, and goodness of fit, S = 1.016. The weighting scheme was based on counting statistics and the final difference Fourier map was essentially featureless. The Figure was plotted with the aid of ORTEP-3 for Windows[6].

Table 2. Crystal data and structure refinement for $C_{21}H_{20}N_2O_2S$.

Empirical formula	$C_{21}H_{20}N_2O_2S$	
Formula weight	364.45	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P b c a	
Unit cell dimensions	a = 15.706(2) Å	α= 90°.
	b = 9.048(6) Å	β= 90°.
	c = 25.308(9) Å	γ = 90°.
Volume	3596(3) $Å^3$	
Z	8	
Density (calculated)	1.346 Mg/m^3	
Absorption coefficient	0.198 mm^{-1}	
F(000)	1536	
Crystal size	0.26 x 0.21 x 0.16 mm^3	

This journal is (c) The Royal Society of Chemistry 2009

Theta range for data collection	3.53 to 27.48°.
Index ranges	-20<=h<=20, -11<=k<=11, -32<=l<=32
Reflections collected	7329
Independent reflections	4073 [R(int) = 0.0438]
Completeness to theta = 27.48°	98.5 %
Absorption correction	Multi-scan method
Max. and min. transmission	0.9690 and 0.9503
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4073 / 0 / 235
Goodness-of-fit on F^2	1.016
Final R indices [I>2sigma(I)]	R1 = 0.0474, wR2 = 0.1068
R indices (all data)	R1 = 0.0824, wR2 = 0.1263
Largest diff. peak and hole	0.234 and -0.432 e.$Å^{-3}$

References for X-ray Crystallography

1. Otwinowski, Z. & Minor, W. (1997). *Methods Enzymol*. **276**, 307.

2. Hooft, R. (1998). *COLLECT*. Nonius BV, Delft. The Netherlands.

3. Altomare, A., Cascarano, M., Giacovazzo, C.& Guagliardi, A. (1993). *SIR92*. J. Appl. Cryst., **26**, 343

4. Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. & Smits, J.M.M. (1994). The *DIRDIF-94* program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

5. Sheldrick, G.M. (1997). *SHELXL97*. University of Göttingen, Germany.

6. Johnson, C.K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

This journal is (c) The Royal Society of Chemistry 2009