Electronic Supplementary Material (ESI) for Organic and Biomolecular Chemistry

Supporting Information

Copper-Catalyzed Direct Thiolation of Azoles with Aliphatic Thiols

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Experimental Procedures

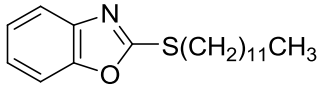
General Remarks:

Column chromatography was carried out on silica gel. Unless noted ^{1}H NMR spectra were recorded on 400 MHz in $CDCl_3$, ^{13}C NMR spectra were recorded on 100 MHz in $CDCl_3$ using TMS as internal standard. IR spectra were recorded on an FT-IR spectrometer and only major peaks are reported in cm^{-1}. Melting points were determined on a microscopic apparatus and were uncorrected. All new compounds were further characterized by HRMS (high resolution mass spectra); copies of their ^{1}H NMR and ^{13}C NMR spectra are provided.

Unless otherwise noted, materials obtained from commercial suppliers were used without further purification. Toluene was dried and distilled from sodium/benzophenone. The known compounds **1a-1c**,[1] **1d-1e**,[2] **1f**,[3] **1h**,[4] **1k**,[5] **1l**,[6] **1n**[7], dodecyl disulfide[8] and bis(dodecylthio)copper[9] were prepared as described in the literature.

General procedure for the preparation of 3

Under air atmosphere, a reaction was charged with heterocycle **1** (0.4 mmol), RSH **2** (0.6 mmol), $Cu(OAc)_2 \cdot H_2O$ (16 mg, 20% mol), CuO (64 mg, 0.8 mmol), and PhMe (2 mL). The mixture stirred at 120 ^{o}C and monitored by TLC. After the completion of the reaction, the residue was purified directly short flash column chromatography on silica gel with hexane/ethyl acetate as an eluent to give the desired corresponding product.



2-(dodecylthio)benzo[*d*]oxazole (3a): silica gel column purification with hexane/ethyl acetate (80/1, v/v); white solid, mp: 23.0-24.2 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, J = 6.8 Hz, 3H), 1.25-1.31 (m, 16H), 1.43-1.50 (m, 2H), 1.78-1.86 (m, 2H), 3.30 (t, J = 7.2 Hz,

(1) Bhor, M. D., Bhanage. B.M.; *Synth. Commun*. **2010**, 40, 1743.

(2) Srivani, A.; Venkateswar Rao, K.T.; Sai Prasad, P.S.; Lingaiah, N.; *J. Mol. Catal. A: Chem.* **2010**, 328 , 119.

(3) Heusen, A.M.; Hoogenbooml, B.E.; Slderius, H. *Tetrahedron Lett*, **1972**, 13, 2369.

(4) Ma, D.W; Xie, S.W.; Xue, P.; Zhang, X.J.; Dong, J.H.; Jiang, Y.W.; *Angew. Chem. Int. Ed.* **2009**, 48, 4222.

(5) Lissel, M.; Schmidt, S.; Neumann, B. *synthesis* **1986**, 5, 382.

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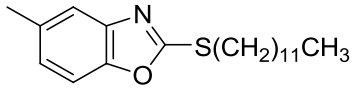
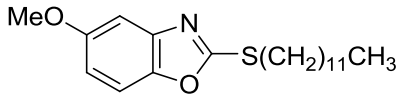
(8) K. Ishizuka, H. Seike, T. Hatakeyama, M. Nakamura, *J. Am. Chem. Soc.*, **2010**, 132, 13117.

(9) Y.- B. Chen, L. Chen, L.-M. Wu, *Chem. Eur. J.*, **2008**, 14, 11069.

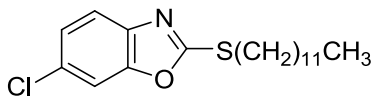
2H), 7.20-7.28 (m, 2H), 7.42 (d, J = 7.6 Hz, 1H), 7.59 (d, J = 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.2, 151.7, 142.0, 124.1, 123.7, 118.3, 109.7, 32.2, 31.9, 29.6, 29.5, 29.4, 29.3, 29.2, 29.0, 28.6, 22.7, 14.1; IR (thin film, cm^{-1}) 2921, 2849, 1503, 1468, 1130, 744; HRMS (ESI) m/z: calcd for $C_{19}H_{29}NOS$ $[M+H]^+$: 320.2043, found: 320.2047.



2-(dodecylthio)-5-methylbenzo[*d*]oxazole (3b): silica gel column purification with hexane/ ethyl acetate (80/1, v/v); white solid, mp: 29.2-31.0 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, J = 6.8 Hz, 3H), 1.26-1.31 (m, 16H), 1.43-1.50 (m, 2H), 1.77-1.85 (m, 2H), 2.43 (s, 3H), 3.29 (t, J = 7.2 Hz, 2H), 7.02 (d, J = 8.0 Hz, 2H), 7.28 (d, J = 8.0 Hz, 2H), 7.38 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.1, 150.0, 142.2, 133.9, 124.6, 118.4, 109.1, 32.2, 31.9, 29.6, 29.5, 29.4, 29.3, 29.2, 29.0, 28.6, 22.7, 21.4, 14.1; IR (thin film, cm^{-1}) 2919, 2850, 1637, 1497, 1152, 805; HRMS (ESI) m/z: calcd for $C_{20}H_{31}NOS$ $[M+H]^+$: 334.2199, found: 334.2191.

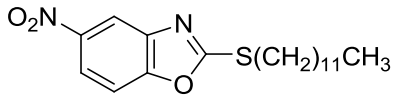


2-(dodecylthio)-5-methoxybenzo[*d*]oxazole (3c): silica gel column purification with hexane/ ethyl acetate (80/1, v/v); white solid, mp: 24.8-26.0 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, J = 6.8 Hz, 3H), 1.26-1.31 (m, 16H), 1.43-1.50 (m, 2H), 1.78-1.85 (m, 2H), 3.29 (t, J = 7.2 Hz, 2H), 3.83 (s, 3H), 6.81 (dd, J = 8.8 , 2.4 Hz, 1H), 7.11 (d, J = 2.4 Hz, 1H), 7.29 (d, J = 9.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.8, 157.1, 146.4, 142.8, 111.6, 109.8, 102.0, 55.9, 32.3, 31.9, 29.6, 29.5, 29.4, 29.3, 29.2, 29.0, 28.6, 22.7, 14.1; IR (thin film, cm^{-1}) 2916, 1636, 1476, 1142, 826; HRMS (ESI) m/z: calcd for $C_{20}H_{31}NO_2S$ $[M+H]^+$: 350.2148, found: 350.2143.

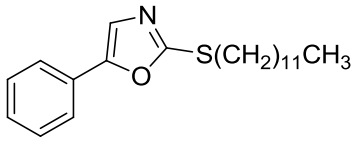


6-chloro-2-(dodecylthio)benzo[*d*]oxazole (3d): silica gel column purification with hexane/ ethyl acetate (80/1, v/v); white solid, mp: 41.2-42.8 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, J = 6.8 Hz, 3H), 1.26-1.30 (m, 16H), 1.45-1.50 (m, 2H), 1.78-1.85 (m, 2H), 3.29 (t, J = 7.2

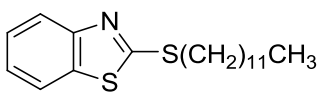
Hz, 2H), 7.24 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.42 (d, *J* = 1.6 Hz, 1H), 7.47 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.1, 151.9, 140.8, 129.3, 124.7, 118.6, 110.4, 32.4, 31.9, 29.7, 29.6, 29.5, 29.4, 29.3, 29.2, 29.0, 28.6, 22.7, 14.1; IR (thin film, cm^{-1}) 2915, 2850, 1504, 1467, 1214, 1140, 817; HRMS (ESI) m/z: calcd for $C_{19}H_{28}ClNOS$ $[M+H]^+$: 354.1653, found: 354.1660.



2-(dodecylthio)-5-nitrobenzo[*d*]oxazole (3e): silica gel column purification with hexane/ ethyl acetate (80/1, v/v); mp: 58.0-59.8 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.8 Hz, 3H), 1.26-1.35 (m, 16H), 1.45-1.52 (m, 2H), 1.82-1.89 (m, 2H), 3.34 (t, *J* = 7.2 Hz, 2H), 7.52 (d, *J* = 9.2 Hz, 1H), 8.22 (dd, *J* = 8.8, 1.6 Hz, 1H), 8.46 (d, *J* = 2.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.4, 155.3, 145.2, 142.5, 119.9, 114.4, 109.7, 32.6, 31.9, 29.6, 29.5, 29.4, 29.3, 29.1, 29.0, 28.6, 22.7, 14.1; IR (thin film, cm^{-1}) 2915, 2851, 2360, 1639, 1527, 1494, 1342, 1108, 819, 736; HRMS (ESI) m/z: calcd for $C_{19}H_{28}ClNOS$ $[M+H]^+$: 365.1893, found: 365.1896.

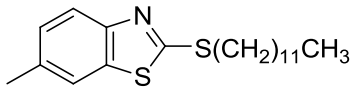


2-(dodecylthio)-5-phenyloxazole (3f): silica gel column purification with hexane/ ethyl acetate (80/1, v/v); white solid, mp: 37.8-39.0 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.8 Hz, 3H), 1.25-1.31 (m, 16H), 1.41-1.48 (m, 2H), 1.75-1.82 (m, 2H), 3.20 (t, *J* = 7.2 Hz, 2H), 7.26-7.31 (m, 2H), 7.37-7.40 (m, 2H), 7.56-7.59 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.1, 152.6, 128.8, 128.1, 127.8, 123.7, 123.0, 32.6, 31.9, 29.6, 29.5, 29.4, 29.3, 29.1, 28.6, 22.7, 14.1; IR (thin film, cm^{-1}) 2920, 2849, 1633, 1476, 1165, 1121, 759, 689; HRMS (ESI) m/z: calcd for $C_{21}H_{31}NOS$ $[M+H]^+$: 346.2199, found: 346.2190.

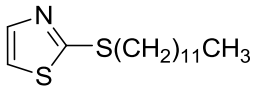


2-(dodecylthio)benzo[*d*]thiazole (3g): silica gel column purification with hexane/ ethyl acetate (80/1, v/v); colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.8 Hz, 3H),

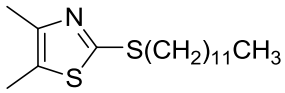
1.26-1.30 (m, 16H), 1.43-1.50 (m, 2H), 1.77-1.84 (m, 2H), 3.33 (t, *J* = 7.2 Hz, 2H), 7.25-7.28 (m, 1H), 7.37-7.41 (m, 1H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.86 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.3, 153.4, 135.1, 125.9, 124.0, 121.4, 120.8, 33.6, 31.9, 29.6, 29.5, 29.4, 29.3, 29.2, 29.1, 28.7, 22.7, 14.1; IR (thin film, cm^{-1}) 2924, 2852, 2360, 1461, 1428, 1239, 996, 755; HRMS (ESI) m/z: calcd for $C_{19}H_{29}NS_2$ $[M+H]^+$: 336.1814, found: 336.1809.



2-(dodecylthio)-6-methylbenzo[*d*]thiazole (3h): silica gel column purification with hexane/ ethyl acetate (80/1, v/v); colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.8 Hz, 3H), 1.26-1.31 (m, 16H), 1.42-1.49 (m, 2H), 1.76-1.84 (m, 2H), 2.44 (s, 3H), 3.31 (t, *J* = 7.2 Hz, 2H), 7.20 (dd, *J* = 8.4, 1.2 Hz, 1H), 7.52 (s, 1H), 7.73 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.0, 151.5, 135.3, 134.1, 127.4, 120.9, 120.7, 33.6, 31.9, 29.6, 29.5, 29.4, 29.3, 29.2, 29.1, 28.7, 22.7, 21.4, 14.1; IR (thin film, cm^{-1}) 2920, 1641, 1446, 994, 813, 728; HRMS (ESI) m/z: calcd for $C_{20}H_{31}NS_2$ $[M+H]^+$: 350.1971, found: 350.1980.

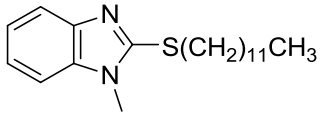


2-(dodecylthio)thiazole (3i): silica gel column purification with hexane/ ethyl acetate (80/1, v/v); colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.8 Hz, 3H), 1.26-1.30 (m, 16H), 1.40-1.47 (m, 2H), 1.71-1.79 (m, 2H), 3.20 (t, *J* = 7.2 Hz, 2H), 7.19 (d, *J* = 3.6 Hz, 1H), 7.65 (d, *J* = 3.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.4, 142.7, 118.5, 34.6, 31.9, 29.6, 29.5, 29.4, 29.3, 29.2, 29.1, 28.7, 22.6, 14.1; IR (thin film, cm^{-1}) 2924, 2853, 2360, 1462, 1388, 1301, 1020, 706; HRMS (ESI) m/z: calcd for $C_{15}H_{27}NS_2$ $[M+H]^+$: 286.1658, found: 286.1656.

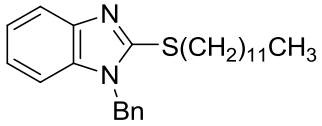


2-(dodecylthio)-4,5-dimethylthiazole (3j): silica gel column purification with hexane/ ethyl acetate (80/1, v/v); colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.8 Hz, 3H), 1.26-1.32 (m, 16H), 1.38-1.43 (m, 2H), 1.68-1.75 (m, 2H), 2.28 (s, 3H), 2.29 (s, 3H), 3.08 (t,

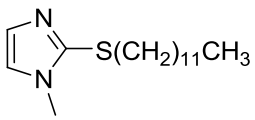
J = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.4, 148.4, 126.4, 34.9, 31.9, 29.6, 29.5, 29.4, 29.3, 29.0, 28.6, 22.6, 14.6, 14.1, 11.2; IR (thin film, cm^{-1}) 2924, 2853, 1642, 1560, 1460, 1420, 1297, 1110, 1020, 723; HRMS (ESI) m/z: calcd for $C_{17}H_{31}NS_2$ $[M+H]^+$: 314.1971, found: 314.1975.



2-(dodecylthio)-1-methyl-1*H*-benzo[d]imidazole (3k): silica gel column purification with hexane/ ethyl acetate (4/1, v/v); white solid, mp: 32.0-33.6 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.8 Hz, 3H), 1.25-1.31 (m, 16H), 1.43-1.50 (m, 2H), 1.75-1.82 (m, 2H), 3.38 (t, *J* = 7.2 Hz, 2H), 3.63 (s, 3H), 7.18-7.20 (m, 3H), 7.65-7.68 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.5, 143.5, 136.7, 121.6, 118.1, 108.2, 32.5, 31.8, 29.8, 29.6, 29.5, 29.4, 29.3, 29.2, 29.1, 28.7, 22.6, 14.0; IR (thin film, cm^{-1}) 2922, 2851, 1462, 1441, 1362, 1275, 1230, 911, 731; HRMS (ESI) m/z: calcd for $C_{20}H_{32}N_2S$ $[M+H]^+$: 333.2359, found: 333.2362.

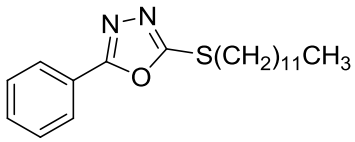


1-benzyl-2-(dodecylthio)-1*H*-benzo[d]imidazole (3l): silica gel column purification with hexane/ ethyl acetate (4/1, v/v); white solid, mp: 47.6-49.2 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.8 Hz, 3H), 1.25-1.30 (m, 16H), 1.40-1.45 (m, 2H), 1.73-1.80 (m, 2H), 3.39 (t, *J* = 7.2 Hz, 2H), 5.27 (s, 2H), 7.10-7.21 (m, 5H), 7.21-7.31 (m, 3H), 7.70 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.5, 143.7, 136.2, 135.7, 128.8, 127.8, 126.8, 121.8, 118.2, 109.0, 47.4, 32.8, 31.9, 29.6, 29.5, 29.4, 29.3, 29.2, 29.1, 28.7, 22.6, 14.1; IR (thin film, cm^{-1}) 2924, 1642, 1440, 731; HRMS (ESI) m/z: calcd for $C_{26}H_{36}N_2S$ $[M+H]^+$: 409.2672, found: 409.2670.

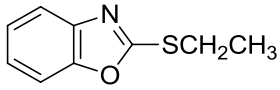


2-(dodecylthio)-1-methyl-1*H*-imidazole (3m): silica gel column purification with hexane/ ethyl acetate (4/1, v/v); colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.8 Hz,

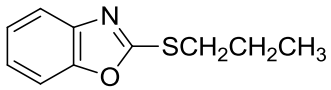
3H), 1.25-1.30 (m, 16H), 1.36-1.41 (m, 2H), 1.61-1.68 (m, 2H), 3.05 (t, *J* = 7.2 Hz, 2H), 3.61 (s, 3H), 6.91 (s, 1H), 7.05 (s, 1H), 7.38 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.1, 129.1, 121.9, 34.3, 33.1, 31.8, 29.7, 29.6, 29.5, 29.4, 29.3, 29.1, 28.6, 22.6, 14.0; IR (thin film, cm^{-1}) 2924, 2853, 1461, 1279, 1124, 1079, 913, 723; HRMS (ESI) m/z: calcd for $C_{16}H_{30}N_2S$ $[M+H]^+$: 283.2202, found: 283.2198.



2-(dodecylthio)-5-phenyl-1,3,4-oxadiazole (3n): silica gel column purification with hexane/ ethyl acetate (80/1, v/v); mp: white solid, 32.4-34.0 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.8 Hz, 3H), 1.26-1.31 (m, 16H), 1.43-1.50 (m, 2H), 1.80-1.87 (m, 2H), 3.29 (t, *J* = 7.2 Hz, 2H), 7.46-7.51 (m, 3H), 7.99-8.01 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.5, 164.5, 131.5, 128.9, 126.6, 123.7, 32.6, 31.9, 29.6, 29.5, 29.4, 29.3, 29.2, 29.0, 28.5, 22.6, 14.1; IR (thin film, cm^{-1}) 2921, 2846, 1637, 1468, 1382, 1186, 1064; HRMS (ESI) m/z: calcd for $C_{20}H_{30}N_2OS$ $[M+H]^+$: 347.2152, found: 347.2147.

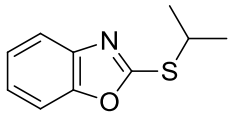


2-(ethylthio)benzo[*d*]oxazol (3o): silica gel column purification with hexane/ ethyl acetate (80/1, v/v); colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.50 (t, *J* = 7.2 Hz, 3H), 3.32 (q, *J* = 14.8 Hz, 2H), 7.20-7.29 (m, 2H), 7.42-7.44 (m, 1H), 7.59-7.61 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.0, 151.7, 142.0, 124.2, 123.7, 118.3, 109.8, 26.6, 14.7; IR (thin film, cm^{-1}) 2930, 2868, 1640, 1451, 1238, 1130, 1096, 924, 743; HRMS (ESI) m/z: calcd for C_9H_9NOS $[M+H]^+$: 180.0478, found: 180.0482.

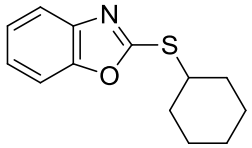


2-(propylthio)benzo[*d*]oxazole (3p): silica gel column purification with hexane/ ethyl acetate (80/1, v/v); colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.08 (t, *J* = 7.2 Hz, 3H), 1.86 (m, 2H), 3.29 (t, *J* = 7.2 Hz, 2H), 7.20-7.29 (m, 2H), 7.41-7.43 (m, 1H), 7.59-7.61 (m, 1H); ^{13}C

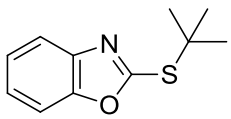
NMR (100 MHz, $CDCl_3$) δ 165.2, 151.7, 141.9, 124.1, 123.7, 118.3, 109.7, 34.1, 22.7, 13.2; IR (thin film, cm^{-1}) 2965, 2873, 1640, 1500, 1452, 1238, 1214, 1130, 1095, 805, 742; HRMS (ESI) m/z: calcd for $C_{10}H_{11}NOS$ $[M+H]^+$: 194.0634, found: 194.0639.



2-(isopropylthio)benzo[*d*]oxazole (3q): silica gel column purification with hexane/ ethyl acetate (80/1, v/v); colorless liquid. ^1H NMR (400 MHz, $CDCl_3$) δ 1.53 (d, J = 6.8 Hz, 6H), 4.04 (m, 1H), 7.21-7.29 (m, 2H), 7.43 (d, J = 7.6 Hz, 1H), 7.60-7.62 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.7, 151.5, 142.0, 124.1, 123.8, 118.4, 109.8, 38.3, 23.3; IR (thin film, cm^{-1}) 2968, 2360, 1648, 1500, 1453, 1237, 1128, 1094, 743; HRMS (ESI) m/z: calcd for $C_{10}H_{11}NOS$ $[M+H]^+$: 194.0634, found: 194.0638.

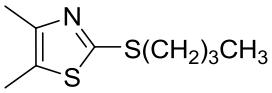


2-(cyclohexylthio)benzo[*d*]oxazole (3r): silica gel column purification with hexane/ ethyl acetate (80/1, v/v); colorless liquid. ^1H NMR (400 MHz, $CDCl_3$) δ 1.35-1.39 (m, 1H), 1.44-1.66 (m, 5H), 1.78-1.83 (m, 2H), 2.19-2.23 (m, 2H), 3.86-3.93 (m, 1H), 7.20-7.29 (m, 2H), 7.41-7.43 (m, 1H), 7.60 (d, J = 7.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.7, 151.6, 142.0, 124.1, 123.7, 118.3, 109.8, 46.0, 33.3, 25.7, 25.5; IR (thin film, cm^{-1}) 3056, 2931, 2854, 1498, 1451, 1238, 1128, 1094, 999, 807, 743; HRMS (ESI) m/z: calcd for $C_{13}H_{15}NOS$ $[M+H]^+$: 234.0947, found: 234.0950.

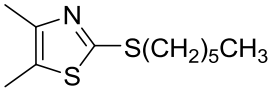


2-(*tert*-butylthio)benzo[*d*]oxazole (3s): silica gel column purification with hexane/ ethyl acetate (80/1, v/v); colorless liquid. ^1H NMR (400 MHz, $CDCl_3$) δ 1.65 (s, 9H), 7.26-7.30 (m, 2H), 7.46-7.48 (m, 1H), 7.64-7.67 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.3, 151.3, 142.0, 124.3, 124.2, 119.0, 109.9, 49.7, 30.9; IR (thin film, cm^{-1}) 2964, 1500, 1452, 1238, 1120, 1089, 806, 743; HRMS (ESI) m/z: calcd for $C_{11}H_{13}NOS$ $[M+H]^+$: 208.0791, found:

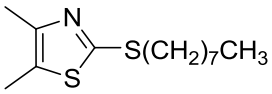
208.0789.



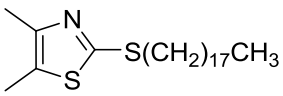
2-(butylthio)-4,5-dimethylthiazole (3t): silica gel column purification with hexane/ ethyl acetate (80/1, v/v); colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.93 (t, J = 7.2 Hz, 3H), 1.41-1.50 (m, 2H), 1.67-1.74 (m, 2H), 2.28 (s, 3H), 2.30 (s, 3H), 3.09 (t, J = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.3, 148.4, 126.4, 34.6, 31.3, 21.8, 14.6, 13.5, 11.2; IR (thin film, cm^{-1}) 2958, 2867, 1560, 1419, 1375, 1297, 1111, 1019; HRMS (ESI) m/z: calcd for $C_9H_{15}NS_2$ $[M+H]^+$: 202.0719, found: 202.0724.



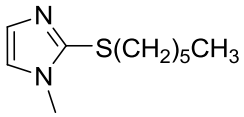
2-(hexylthio)-4,5-dimethylthiazole (3u): silica gel column purification with hexane/ ethyl acetate (80/1, v/v); colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, J = 6.8 Hz, 3H), 1.27-1.33 (m, 4H), 1.39-1.46 (m, 2H), 1.68-1.75 (m, 2H), 2.28 (s, 3H), 2.29 (s, 3H), 3.09 (t, J = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.3, 148.3, 126.4, 34.9, 31.2, 29.2, 28.3, 22.4, 14.6, 13.9, 11.2; IR (thin film, cm^{-1}) 2925, 2856, 1639, 1419, 1375, 1296, 1111, 1020, 726; HRMS (ESI) m/z: calcd for $C_{11}H_{19}NS_2$ $[M+H]^+$: 230.1032, found: 230.1038.



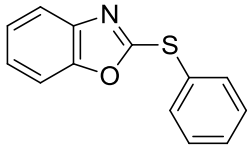
4,5-dimethyl-2-(octylthio)thiazole (3v): silica gel column purification with hexane/ ethyl acetate (80/1, v/v); colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, J = 6.8 Hz, 3H), 1.27-1.33 (m, 8H), 1.38-1.43 (m, 2H), 2.28 (s, 3H), 2.29 (s, 3H), 3.08 (t, J = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.3, 148.3, 126.4, 34.9, 31.7, 29.2, 29.0, 28.6, 22.6, 14.6, 14.0, 11.2; IR (thin film, cm^{-1}) 2924, 2854, 1560, 1461, 1420, 1374, 1296, 1111, 1019, 730; HRMS (ESI) m/z: calcd for $C_{13}H_{23}NS_2$ $[M+H]^+$: 258.1345, found: 258.1343.



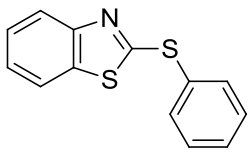
4,5-dimethyl-2-(octadecylthio)thiazole (3w): silica gel column purification with hexane/ ethyl acetate (80/1, v/v); white solid, mp: 36.0-38.0 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, J = 6.8 Hz, 3H), 1.25 (brs, 28H), 1.38-1.43 (m, 2H), 1.67-1.75 (m, 2H), 2.28 (s, 3H), 2.29 (s, 3H), 3.08 (t, J = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.4, 148.4, 126.4, 34.9, 31.9, 29.7, 29.6, 29.5, 29.4, 29.3, 29.2, 29.1, 28.7, 22.7, 14.6, 14.1, 11.3; IR (thin film, cm^{-1}) 2919, 2581, 1560, 1465, 1419, 1020, 909, 734; HRMS (ESI) m/z: calcd for $C_{23}H_{43}NS_2$ $[M+H]^+$: 398.2910, found: 398.2901.



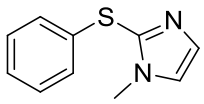
2-(hexylthio)-1-methyl-1*H*-imidazole (3x): silica gel column purification with hexane/ ethyl acetate (4/1, v/v); colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.87 (t, J = 6.8 Hz, 3H), 1.26-1.32 (m, 4H), 1.37-1.44 (m, 2H), 1.61-1.69 (m, 2H), 3.05 (t, J = 7.2 Hz, 2H), 3.61 (s, 3H), 6.91 (s, 1H), 7.05 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.0, 129.1, 121.9, 34.3, 33.1, 31.2, 29.6, 28.2, 22.4, 13.9; IR (thin film, cm^{-1}) 3106, 2927, 2856, 1459, 1414, 1377, 1279, 1124, 914, 728, 685; HRMS (ESI) m/z: calcd for $C_{23}H_{43}NS_2$ $[M+H]^+$: 199.1263, found: 199.1269.



2-(phenylthio)benzo[*d*]oxazole (3y): silica gel column purification with hexane/ ethyl acetate (4/1, v/v); colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21-7.29 (m, 2H), 7.39-7.41 (m, 1H), 7.43-7.47 (m, 3H), 7.59-7.61 (m, 1H), 7.69-7.71 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.2, 151.9, 142.0, 134.4, 129.8, 129.6, 127.2, 124.3, 119.1, 110.0; IR (thin film, cm^{-1}) 3060, 2925, 1799, 1500, 1450, 1237, 1127, 1093, 1024, 925, 804, 743; HRMS (ESI) m/z: calcd for $C_{23}H_{43}NS_2$ $[M+H]^+$: 228.0478, found: 228.0481.

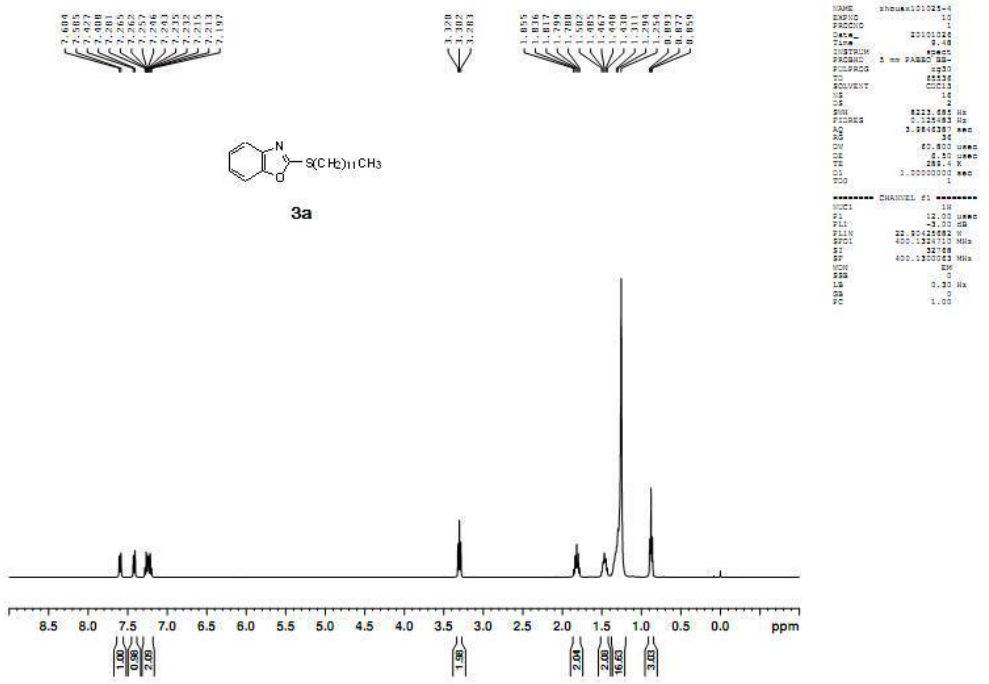


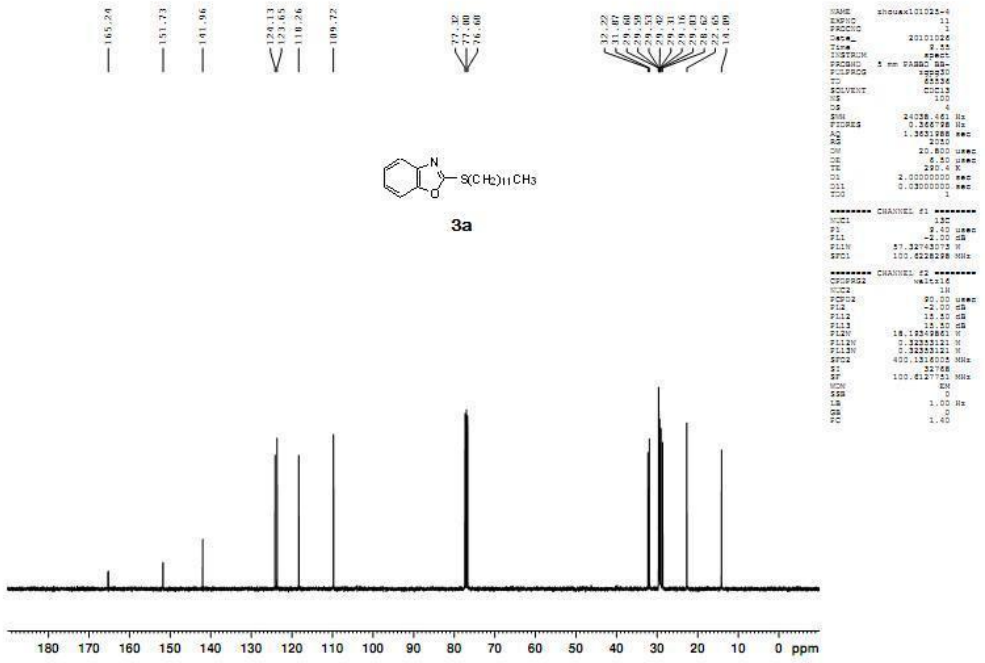
2-(phenylthio)benzo[*d*]thiazole (3z): silica gel column purification with hexane/ ethyl acetate (80/1, v/v); colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (t, J = 7.2 Hz, 1H), 7.40-7.55 (m, 4H), 7.66 (d, J = 8.0 Hz, 1H), 7.76 (d, J = 6.8 Hz, 2H), 7.90 (d, J = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.6, 153.9, 135.3, 130.4, 129.9, 126.1, 124.3, 121.9, 120.7; IR (thin film, cm^{-1}) 3059, 2930, 1580, 1458, 1425, 1309, 1237, 1080, 1004, 753, 690; HRMS (ESI) m/z: calcd for $C_{20}H_{31}NS_2$ $[M+H]^+$: 244.0249, found: 244.0252.

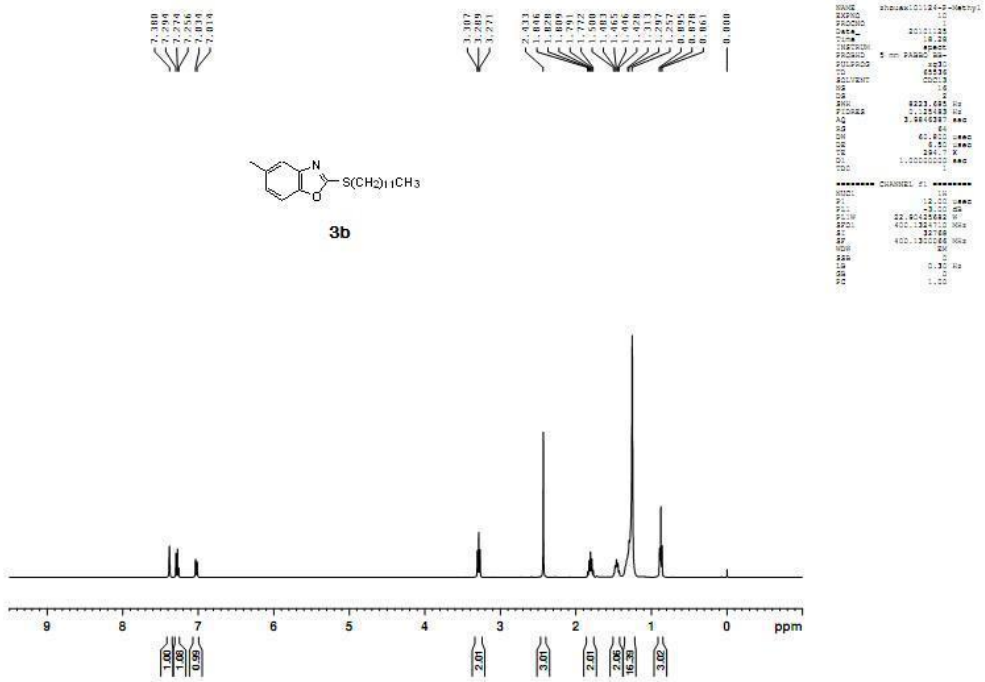


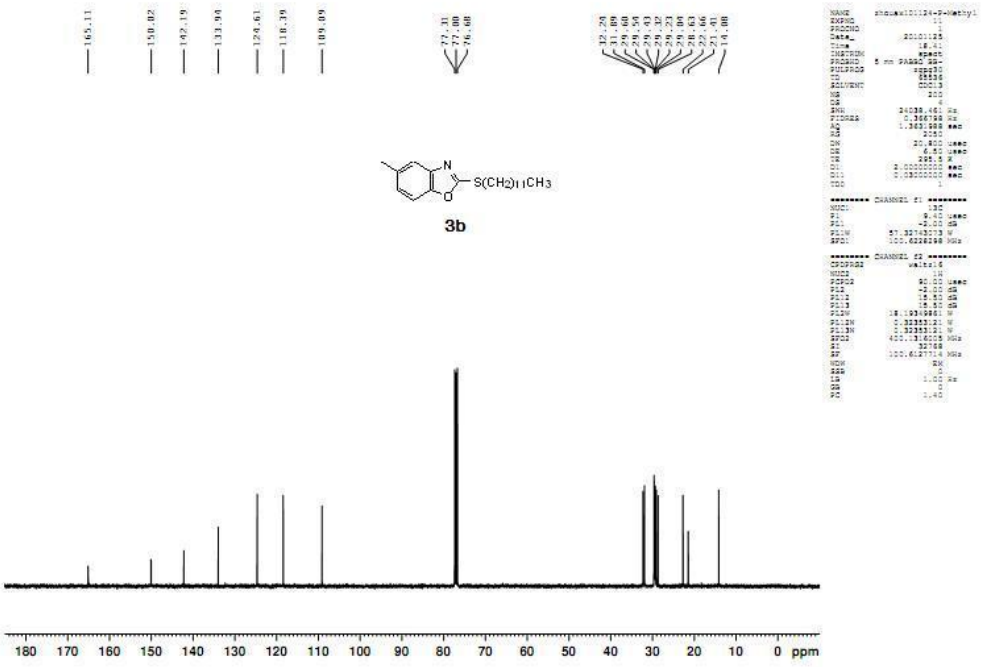
1-methyl-2-(phenylthio)-1*H*-imidazole (3aa): silica gel column purification with hexane/ ethyl acetate (4/1, v/v); colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.61 (s, 3H), 7.06 (d, J = 1.2 Hz, 1H), 7.11-7.17 (m, 4H), 7.22-7.29 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.8, 134.8, 130.0, 129.1, 127.8, 126.4, 123.7, 33.7; IR (thin film, cm^{-1}) 3056, 2946, 1582, 1456, 1411, 1280, 1122, 1081, 1024, 915, 743, 693; HRMS (ESI) m/z: calcd for $C_{20}H_{31}NS_2$ $[M+H]^+$: 191.0637, found: 191.0640.

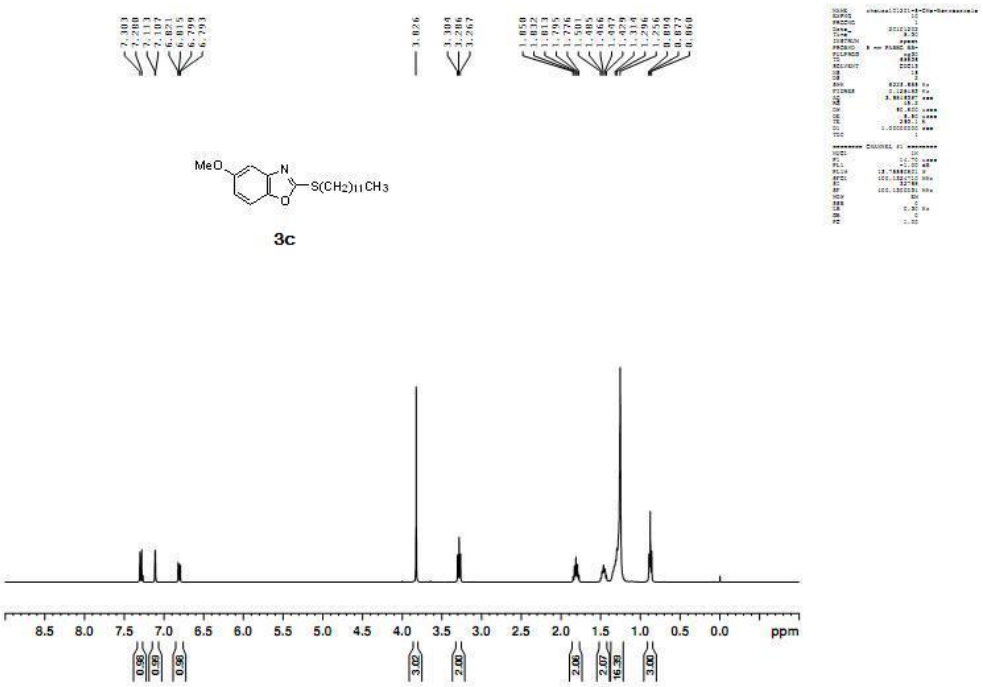
NMR Spectra


7.606
7.585
7.427
7.408
7.261
7.246
7.257
7.296
7.243
7.235
7.232
7.215
7.213
7.197
3.320
3.302
3.283
1.855
1.836
1.817
1.799
1.780
1.485
1.467
1.448
1.430
1.311
1.294
0.893
0.877
0.859
S(CH2)11CH3
3a
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
0.98
2.06
1.98
2.04
2.00
16.63
3.00


165.24
151.73
141.96
124.13
123.65
118.26
109.72
77.32
77.00
76.68
32.29
31.87
29.68
29.59
29.53
29.49
29.31
29.16
29.03
28.82
22.65
14.09
S(CH2)11CH3
3a
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.380
7.296
7.274
7.256
7.034
7.014
3.307
3.289
3.271
2.433
1.846
1.828
1.809
1.791
1.772
1.500
1.483
1.465
1.446
1.428
1.313
1.297
1.257
0.880
0.861
0.000
S(CH2)11CH3
3b
9 8 7 6 5 4 3 2 1 0 ppm
1.00
1.00
0.99
2.01
3.01
2.01
2.06
16.30
3.02


165.11
150.82
142.19
133.93
126.61
118.39
109.09
77.31
77.00
76.68
32.29
31.89
29.60
29.56
29.43
29.32
29.23
29.05
28.63
22.66
21.40
14.08
S(CH2)11CH3
3b
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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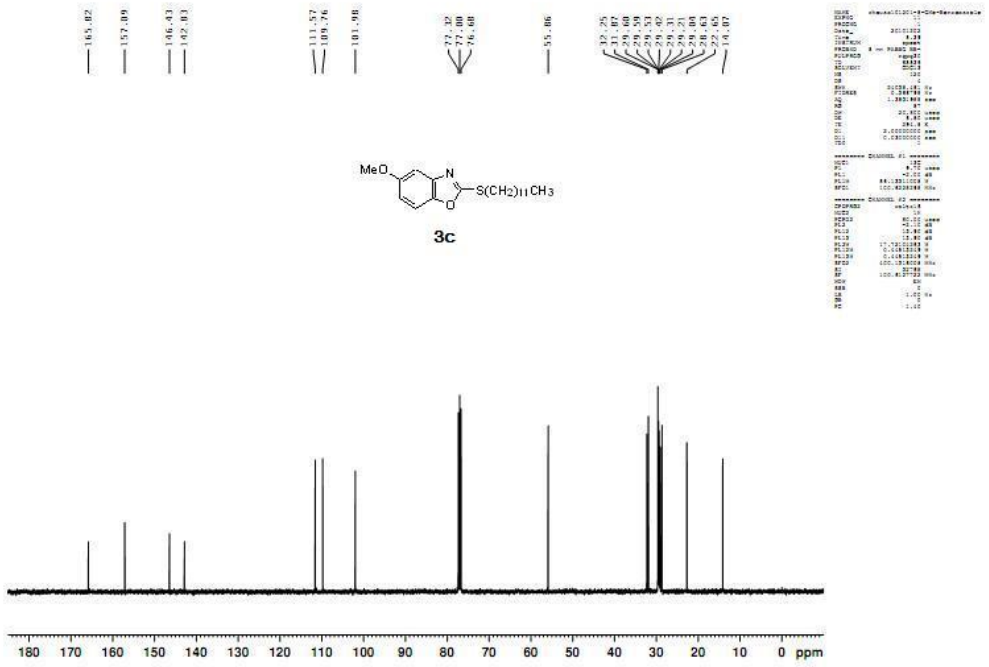
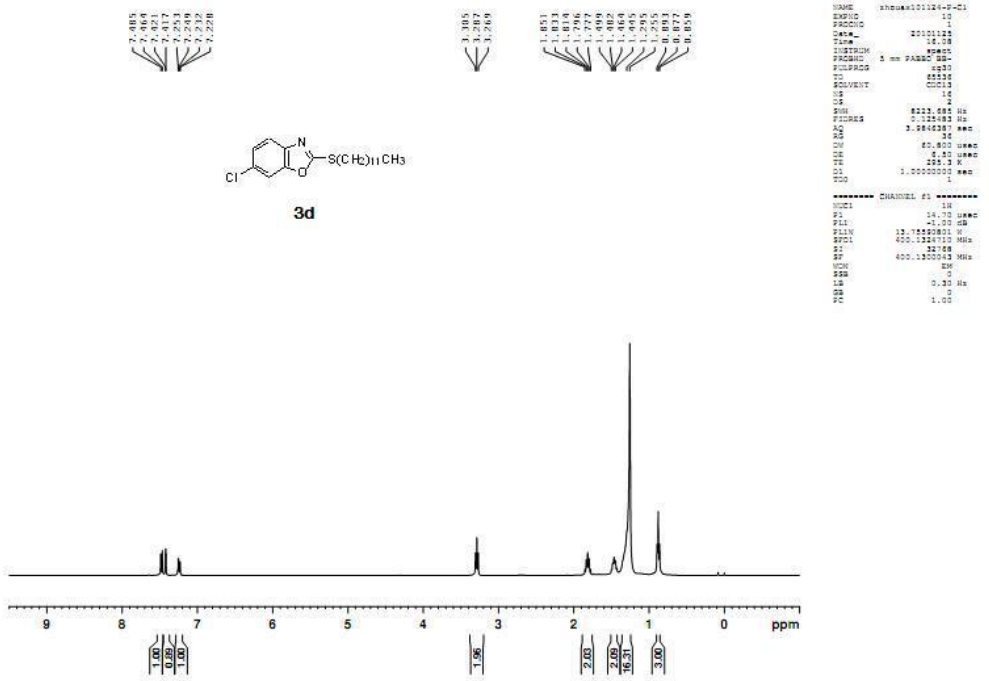
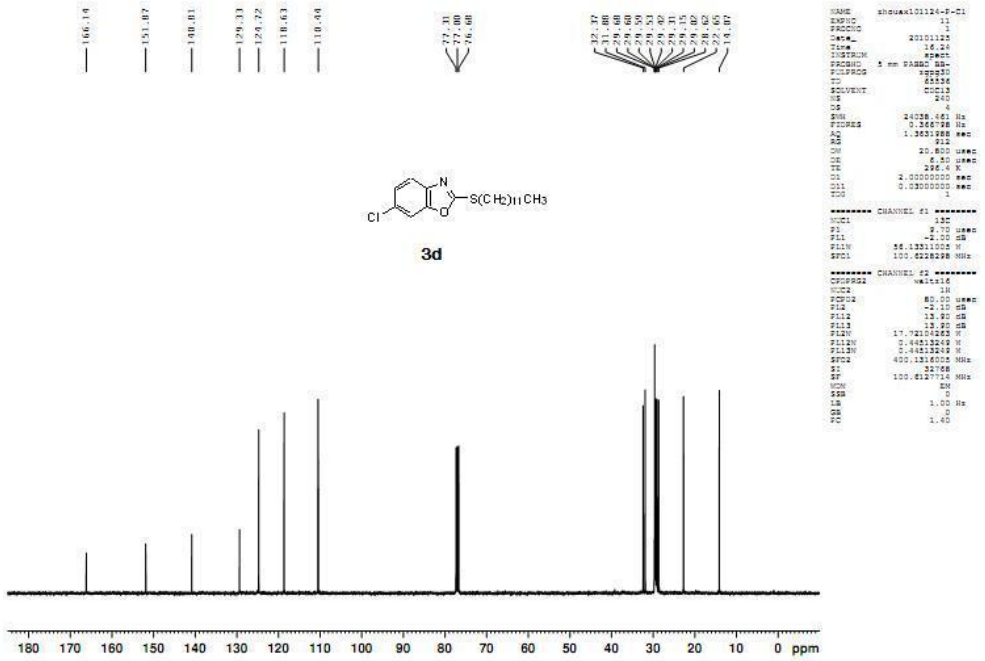
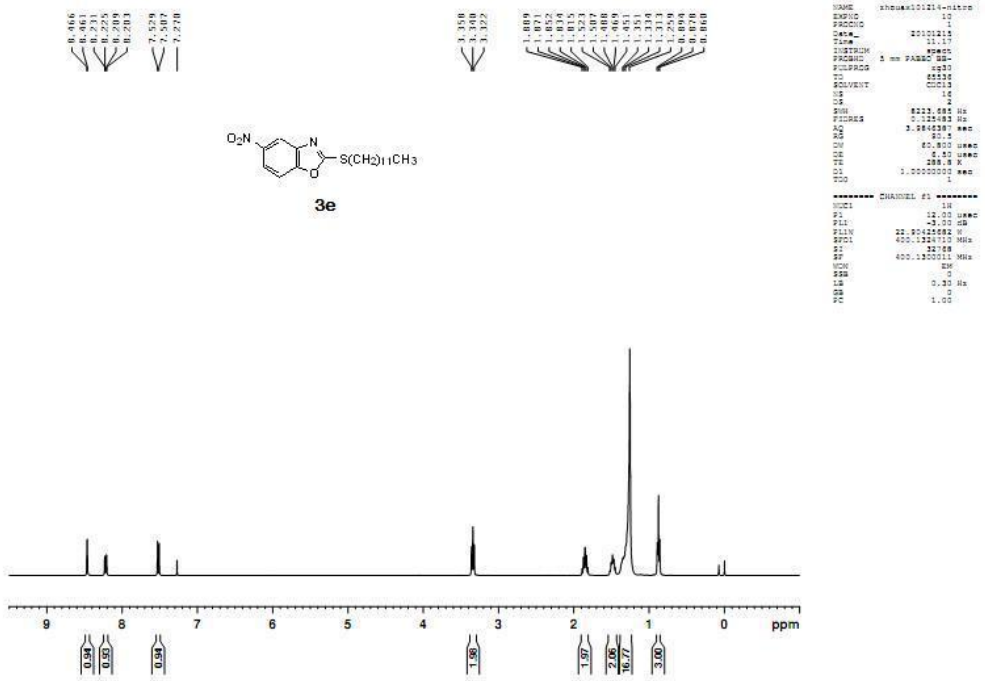
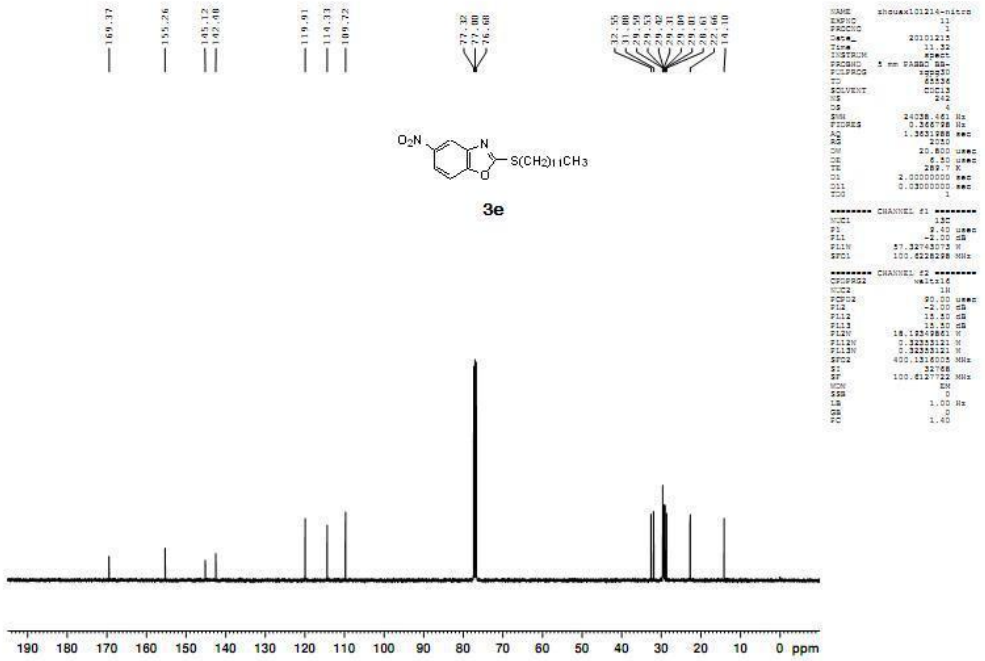
2.280
2.113
2.102
6.821
6.815
6.799
6.793
3.826
3.304
3.286
3.267
1.858
1.832
1.813
1.795
1.776
1.761
1.485
1.466
1.447
1.429
1.416
1.296
1.256
0.899
0.877
0.858
MeO
S(CH2)11CH3
3c
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.98
0.99
0.98
3.00
2.00
2.06
2.07
16.39
3.00

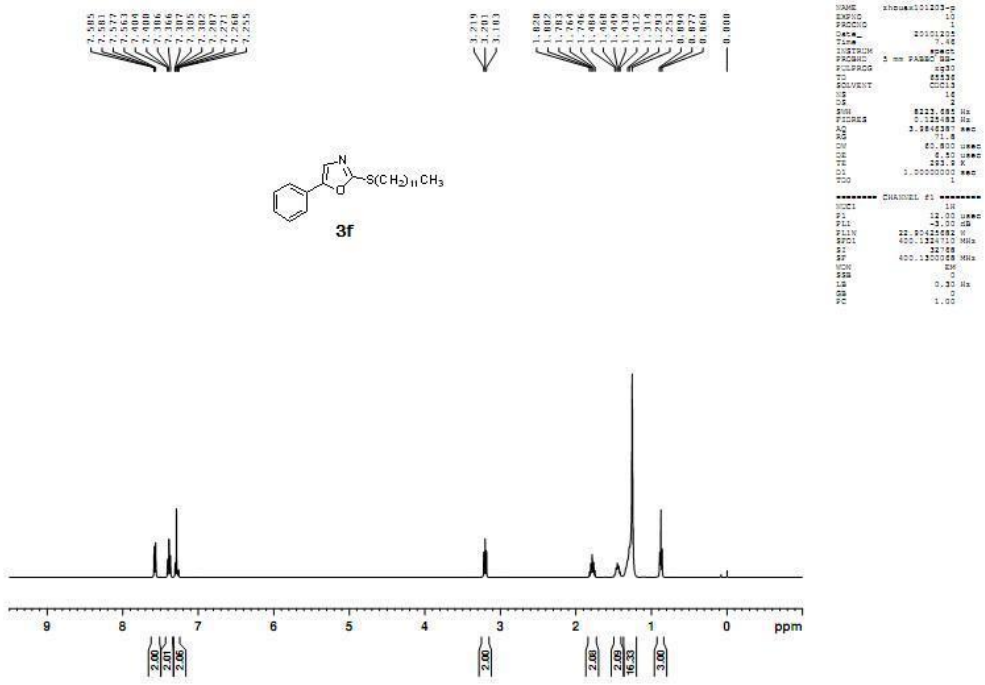

MeO
N
S(CH2)11CH3
O
3c
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

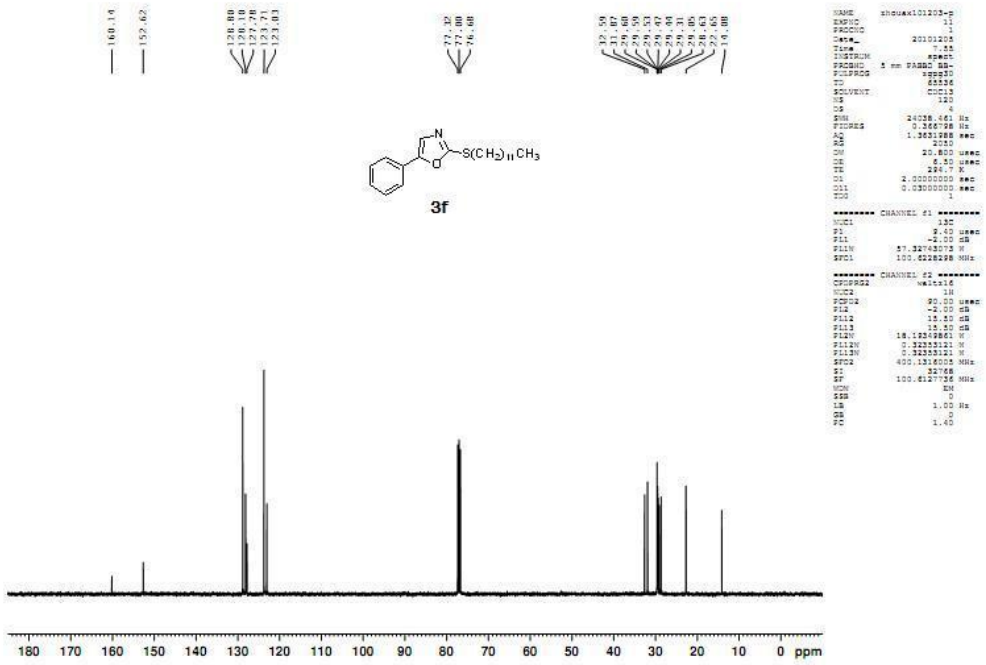

Cl
N
S(CH2)11CH3
O
3d
9 8 7 6 5 4 3 2 1 0 ppm
1.00
0.89
1.00
1.96
2.03
2.00
16.31
3.00

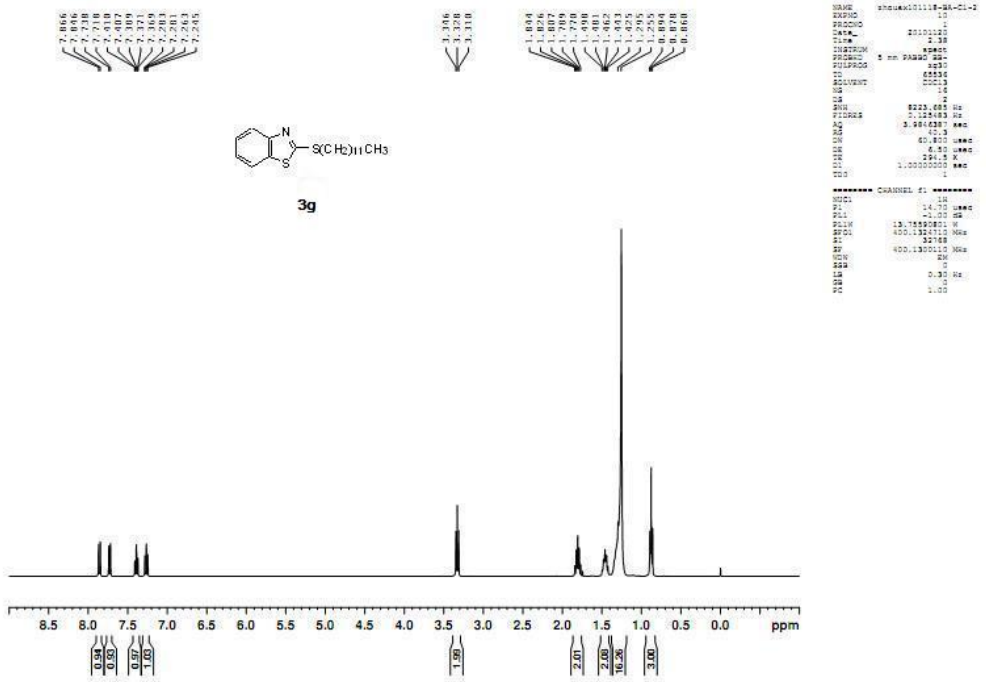

166.14
151.87
140.81
129.33
124.72
118.63
110.44
77.31
77.00
76.68
32.37
31.88
29.68
29.59
29.37
29.32
29.31
29.15
29.10
28.82
22.65
14.07
Cl
N
O
S(CH2)11CH3
3d
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

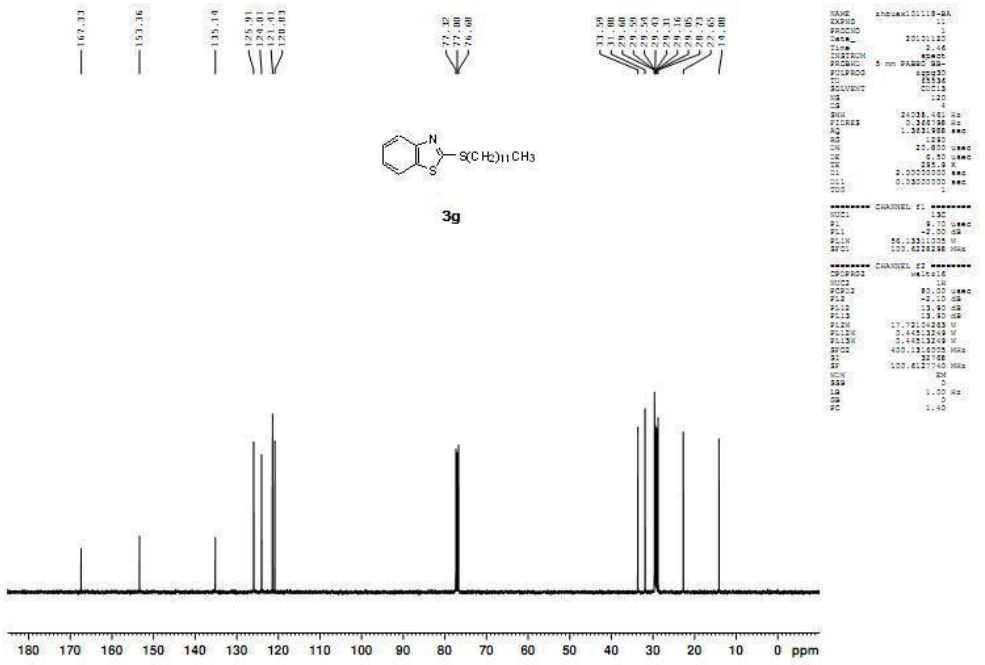

8.466
8.461
8.231
8.225
8.209
8.203
7.529
7.507
7.278
3.358
3.348
3.322
1.889
1.871
1.852
1.834
1.815
1.733
1.507
1.488
1.469
1.451
1.351
1.334
1.313
1.259
0.894
0.878
0.860
O2N
N
O
S(CH2)11CH3
3e
9 8 7 6 5 4 3 2 1 0 ppm
0.94
0.93
0.94
1.98
1.97
2.06
16.77
3.00

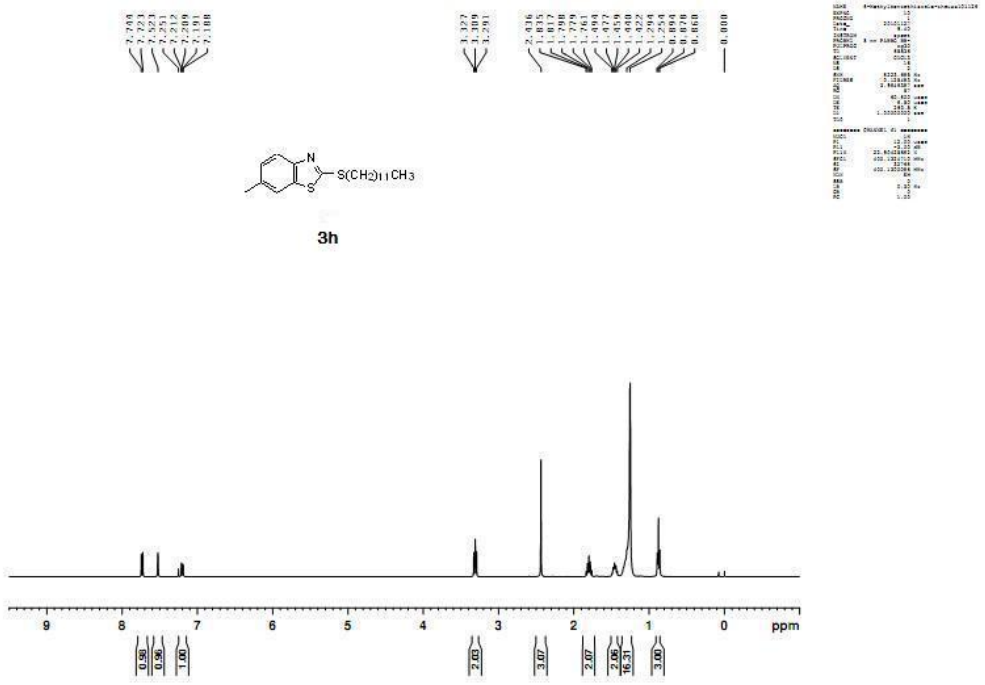

169.37
155.26
145.12
142.48
119.91
114.33
109.72
77.32
77.00
76.68
32.55
31.88
29.55
29.53
29.42
29.31
29.08
28.91
28.61
22.66
14.10
O2N
N
O
S(CH2)11CH3
3e
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

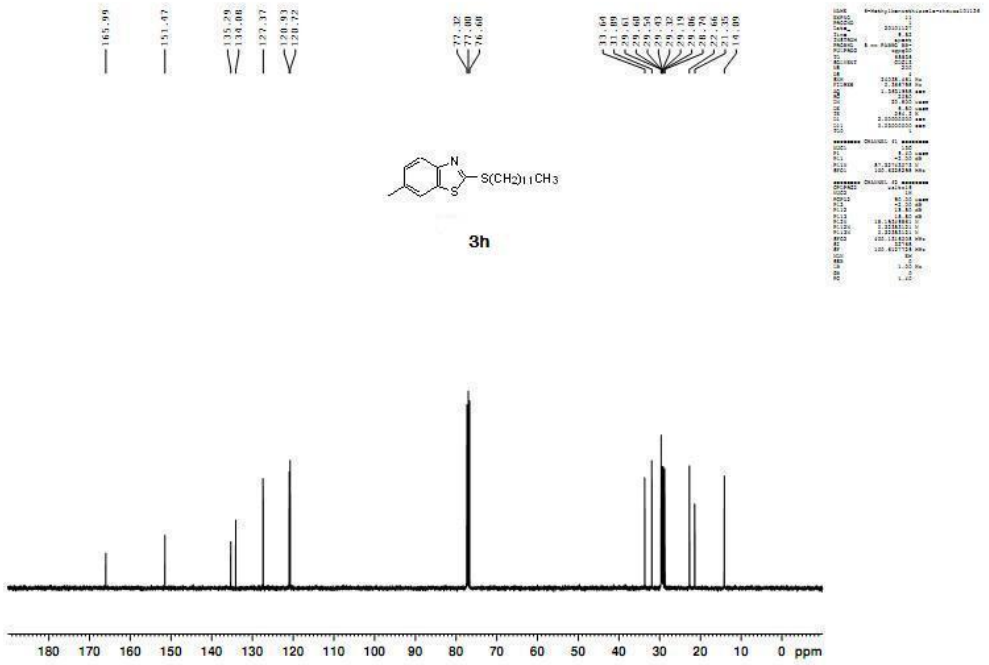

7.585
7.581
7.577
7.563
7.408
7.388
7.386
7.366
7.307
7.305
7.302
7.287
7.271
7.268
7.255
3.219
3.201
3.183
1.828
1.802
1.783
1.764
1.746
1.484
1.468
1.449
1.433
1.417
1.377
1.314
1.293
1.274
0.897
0.879
0.860
0.000
N
O
S(CH2)11CH3
3f
9 8 7 6 5 4 3 2 1 0 ppm
2.00
2.01
2.06
2.00
2.00
2.09
16.33
3.00

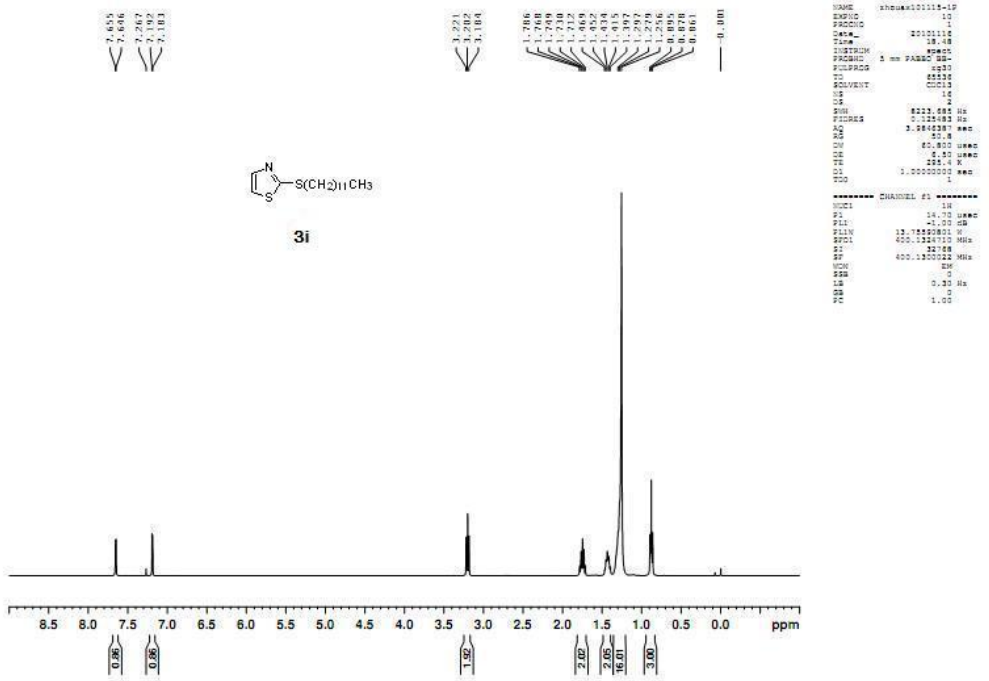

160.14
152.62
128.89
128.10
127.78
123.71
123.03
77.32
77.00
76.68
32.59
31.87
29.68
29.55
29.53
29.47
29.45
29.31
29.26
28.63
22.65
14.08
S(CH2)11CH3
3f
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

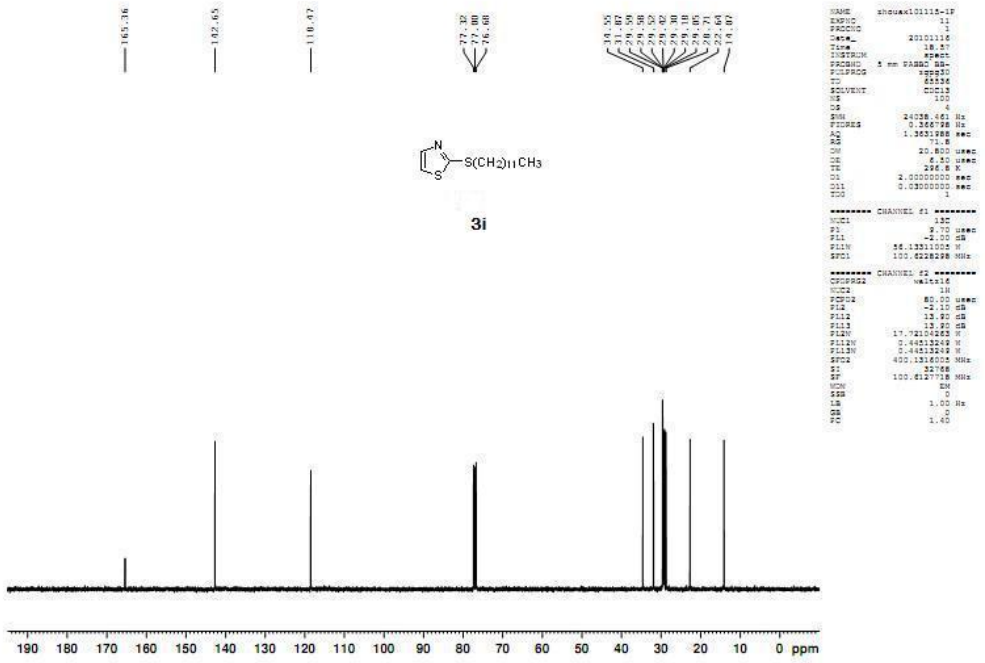

7.866
7.836
7.738
7.718
7.619
7.607
7.369
7.303
7.281
7.263
7.245
3.336
3.328
3.318
1.834
1.816
1.807
1.789
1.778
1.498
1.481
1.462
1.443
1.425
1.295
1.255
0.894
0.878
0.868
S(CH2)11CH3
3g
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.94
0.93
0.97
1.00
1.90
2.01
2.00
16.26
3.00

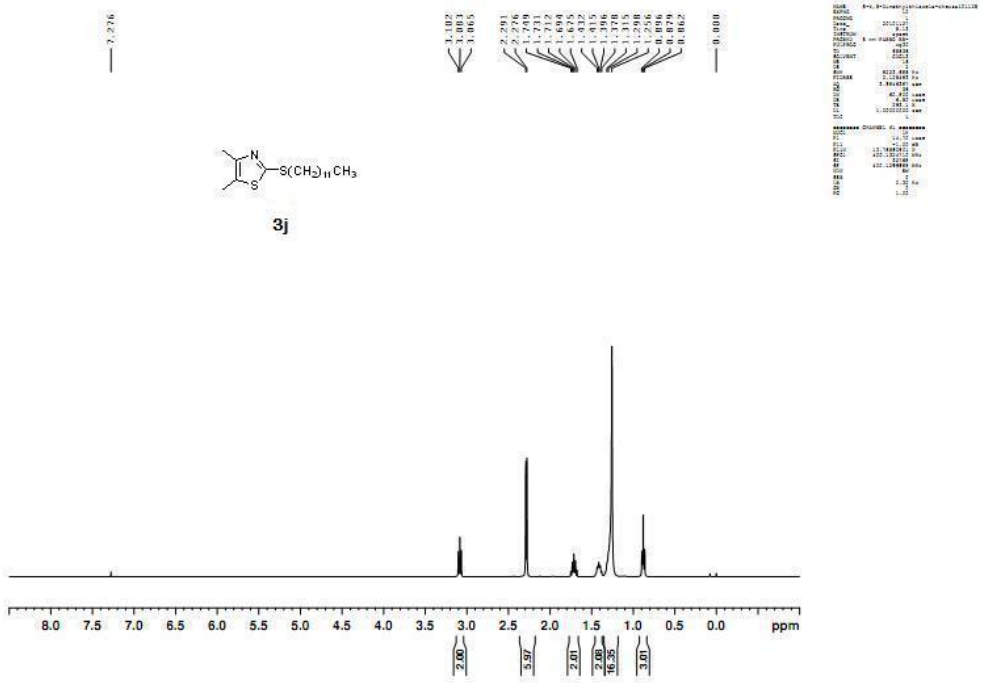

162.33
153.36
135.14
125.91
123.01
121.91
120.83
77.32
77.00
76.68
33.99
31.88
29.58
29.55
29.43
29.31
29.16
29.05
28.73
22.65
14.08
S(CH2)11CH3
3g
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

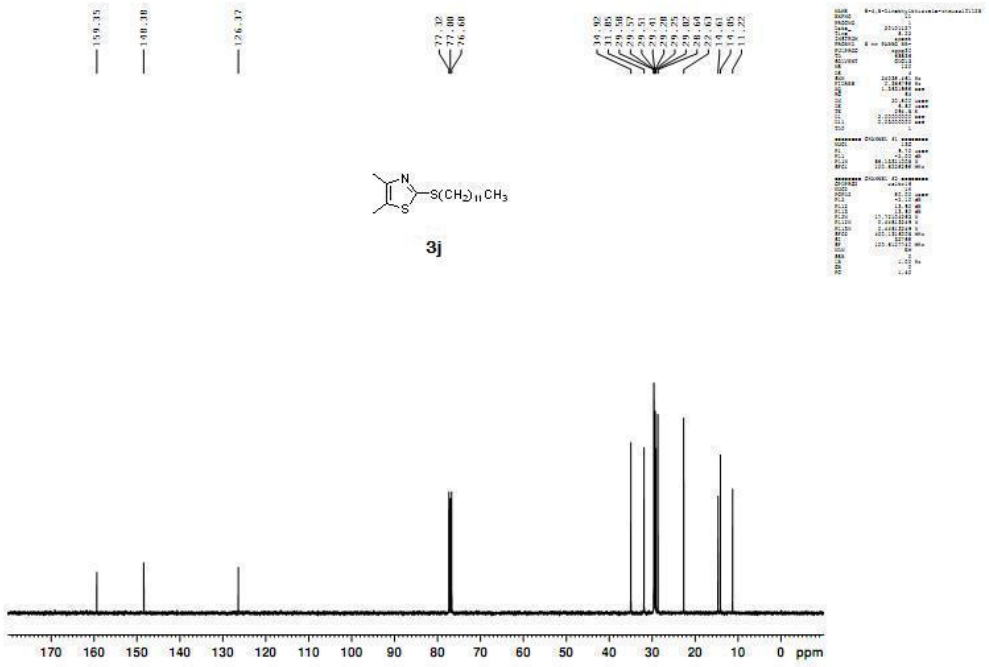

7.730
7.723
7.523
7.517
7.212
7.209
7.191
7.188
3.327
3.309
3.291
2.436
1.835
1.817
1.798
1.779
1.761
1.666
1.477
1.459
1.440
1.422
1.296
1.254
0.894
0.878
0.860
0.000
S(CH2)11CH3
3h
9 8 7 6 5 4 3 2 1 0 ppm
0.98
0.96
1.00
2.03
3.07
2.07
2.06
16.31
3.00

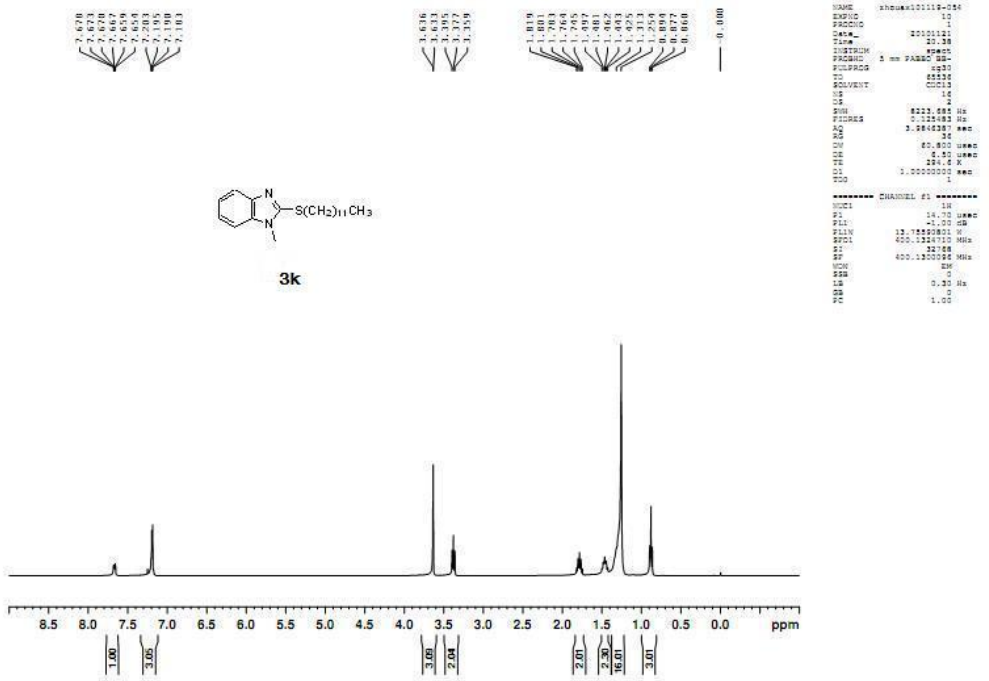

165.99
151.47
135.29
134.06
127.37
120.93
120.72
77.37
77.00
76.68
33.69
31.89
29.61
29.48
29.34
29.32
29.19
29.06
28.74
22.66
21.35
14.09
S(CH2)11CH3
3h
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

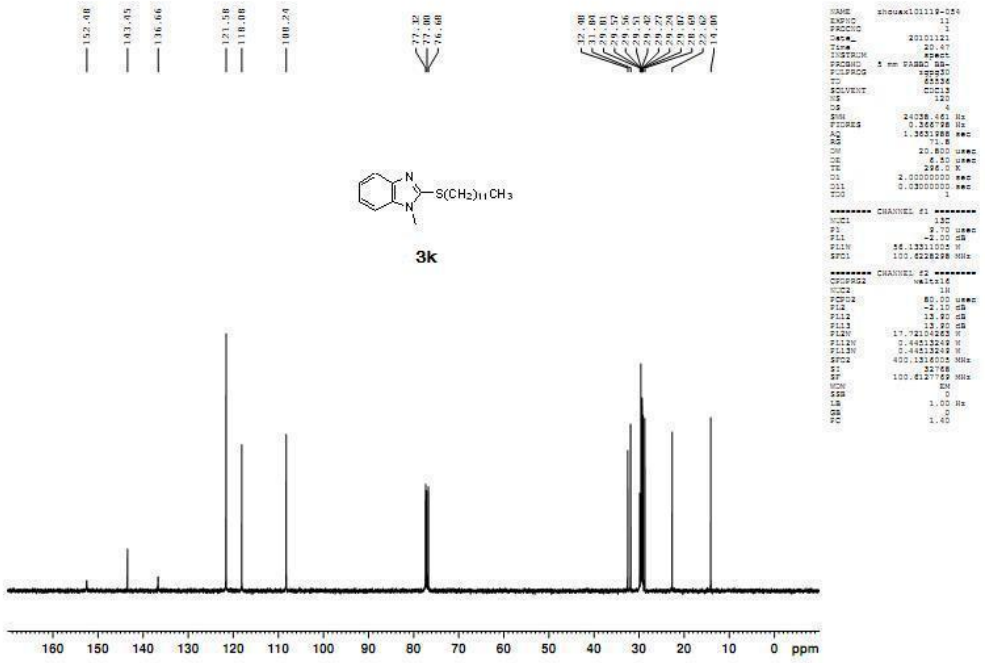

7.655
7.646
7.267
7.192
7.183
3.221
3.202
3.184
1.786
1.768
1.749
1.730
1.712
1.693
1.452
1.434
1.415
1.397
1.387
1.279
1.256
0.895
0.878
0.861
0.000
S(CH2)11CH3
3i
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.96
0.98
1.92
2.07
2.05
16.01
3.00

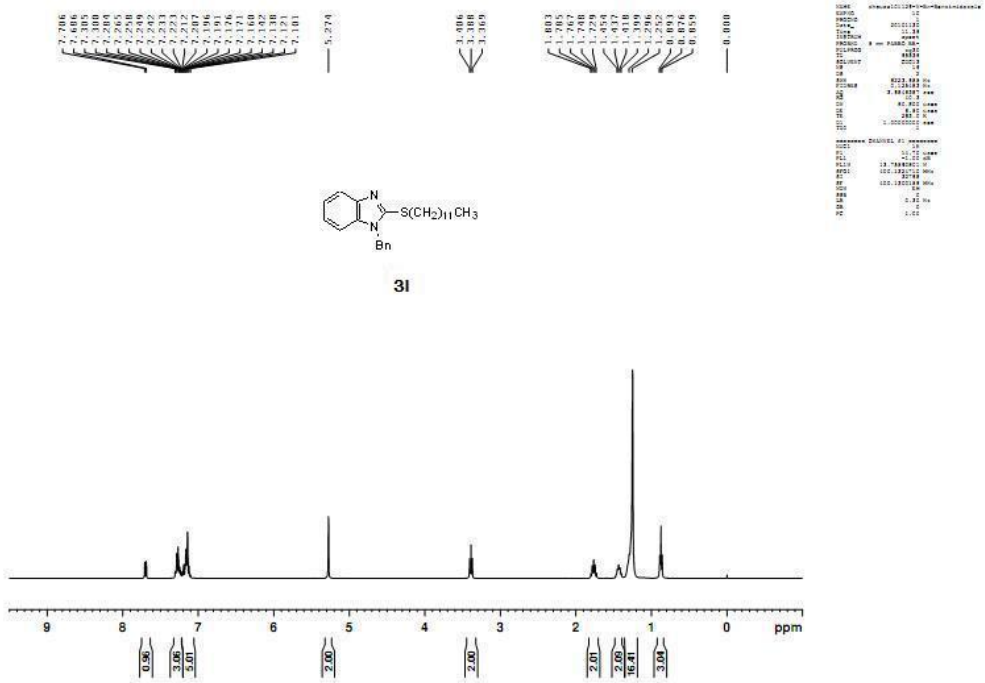

165.36
142.65
118.47
77.32
77.00
76.68
34.55
31.87
29.59
29.58
29.52
29.42
29.38
29.18
29.05
28.71
22.64
14.07
S(CH2)11CH3
3i
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

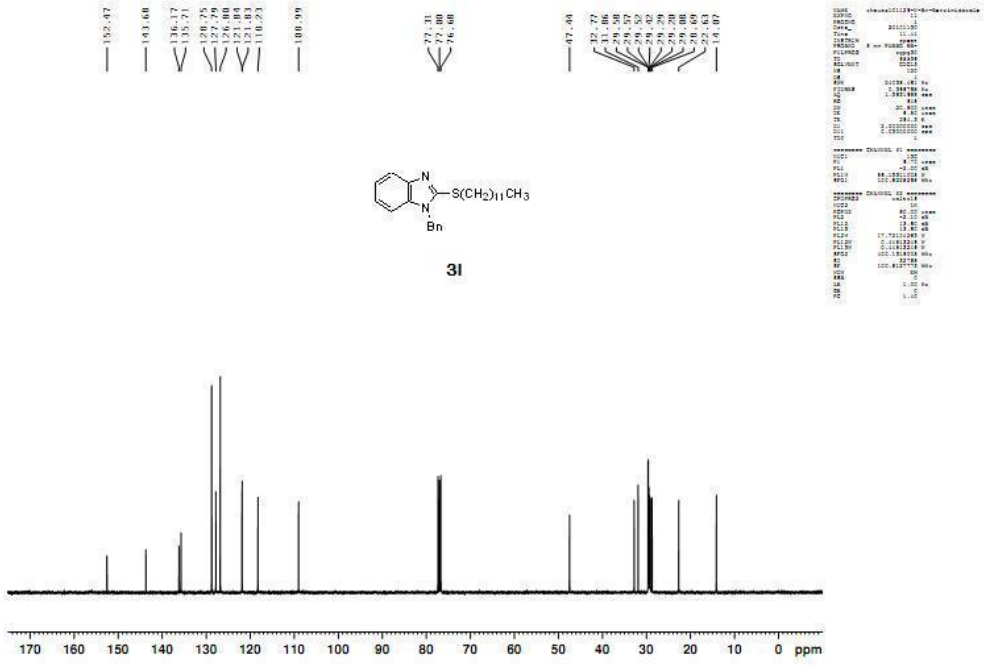

7.276
3.102
3.083
3.065
2.291
2.276
2.259
1.731
1.712
1.694
1.675
1.657
1.415
1.396
1.378
1.315
1.298
1.256
0.896
0.879
0.862
0.000
S(CH2)11CH3
3j
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.00
5.97
2.01
2.08
16.35
3.01

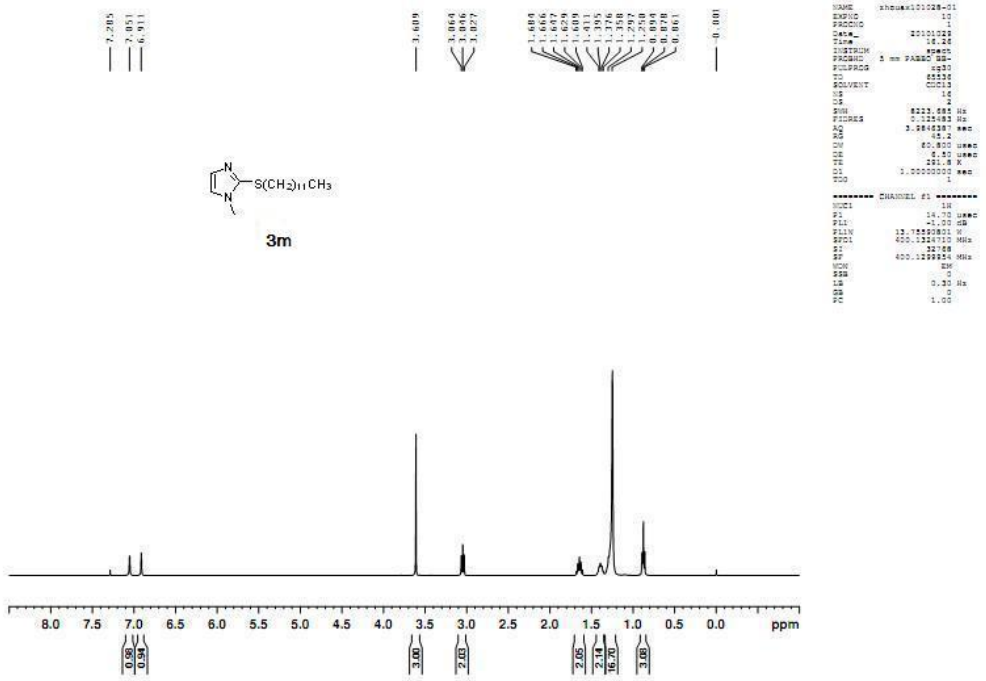

159.35
140.38
126.37
77.32
77.00
76.68
34.92
31.86
29.58
29.57
29.51
29.39
29.38
29.25
29.02
28.63
22.63
14.05
11.22
S(CH2)11CH3
3j
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

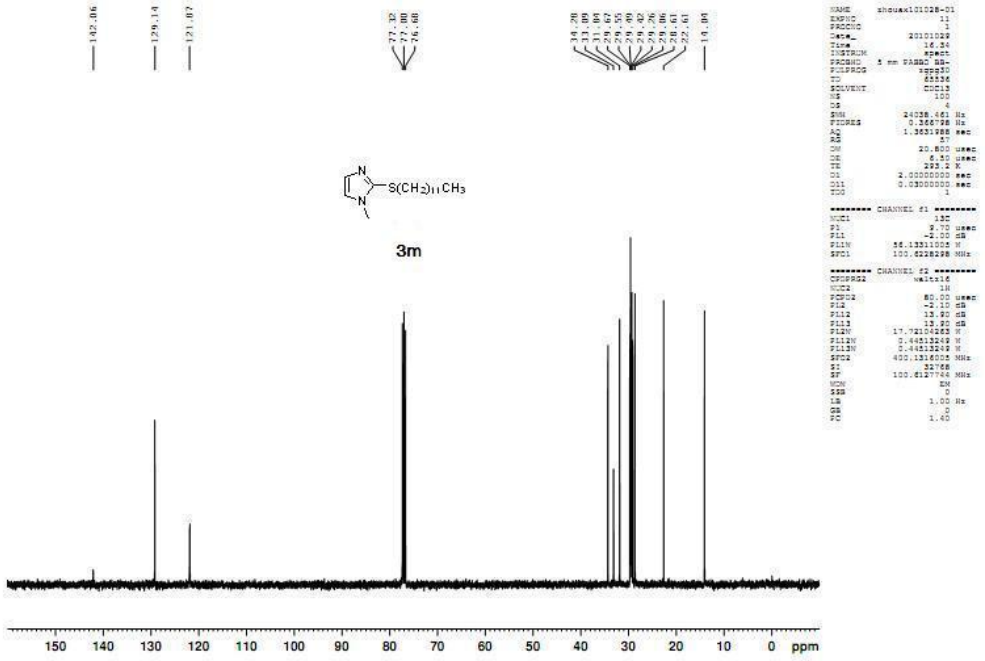

7.678
7.673
7.670
7.667
7.659
7.654
7.203
7.195
7.190
7.183
3.636
3.633
3.395
3.377
3.359
1.819
1.801
1.784
1.764
1.745
1.497
1.481
1.462
1.443
1.425
1.313
1.296
0.894
0.877
0.860
0.000
S(CH2)11CH3
3k
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.00
3.06
3.06
2.04
2.01
2.30
16.01
3.01

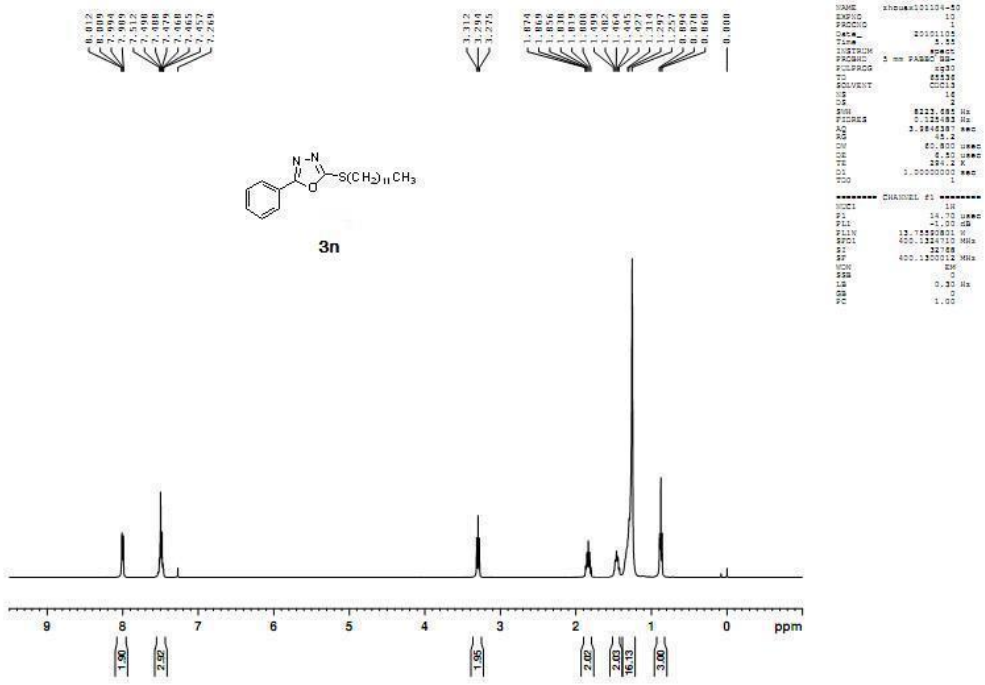

152.48
143.45
136.66
121.58
118.08
109.24
77.32
77.00
76.68
32.00
31.80
29.81
29.57
29.56
29.53
29.42
29.27
29.20
29.00
28.85
22.62
14.06
S(CH2)11CH3
3k
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

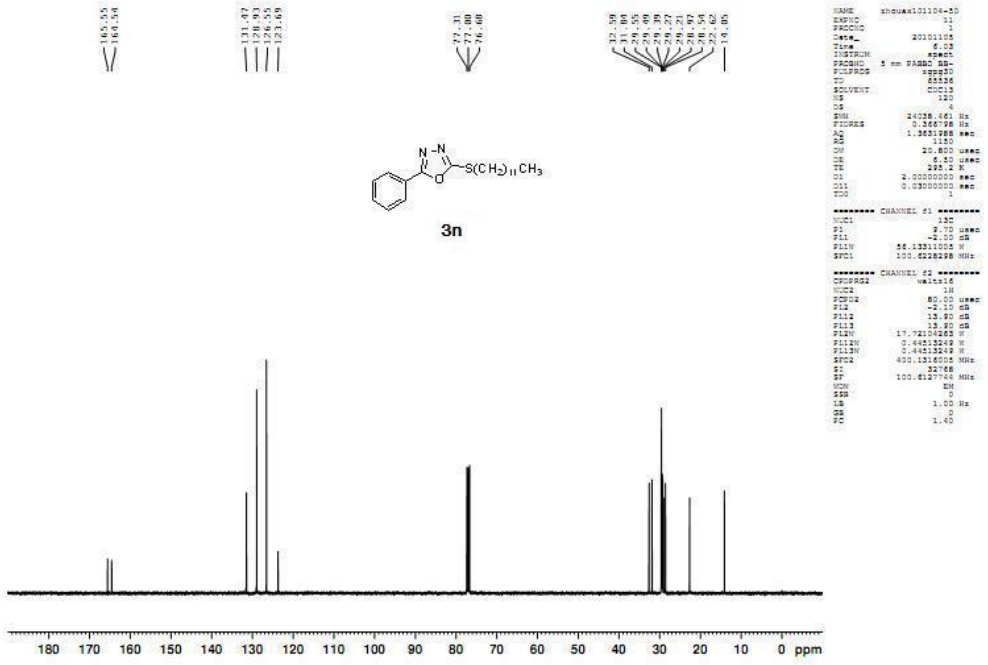

7.706
7.686
7.305
7.288
7.265
7.258
7.249
7.242
7.233
7.212
7.207
7.196
7.191
7.176
7.171
7.160
7.132
7.118
7.121
7.101
5.274
3.006
3.008
3.009
1.803
1.785
1.767
1.738
1.729
1.454
1.437
1.418
1.398
1.296
1.252
0.893
0.876
0.859
0.000
S(CH2)11CH3
Bn
3l
9 8 7 6 5 4 3 2 1 0 ppm
0.96
3.06
3.01
2.00
2.00
2.01
2.00
16.41
3.04

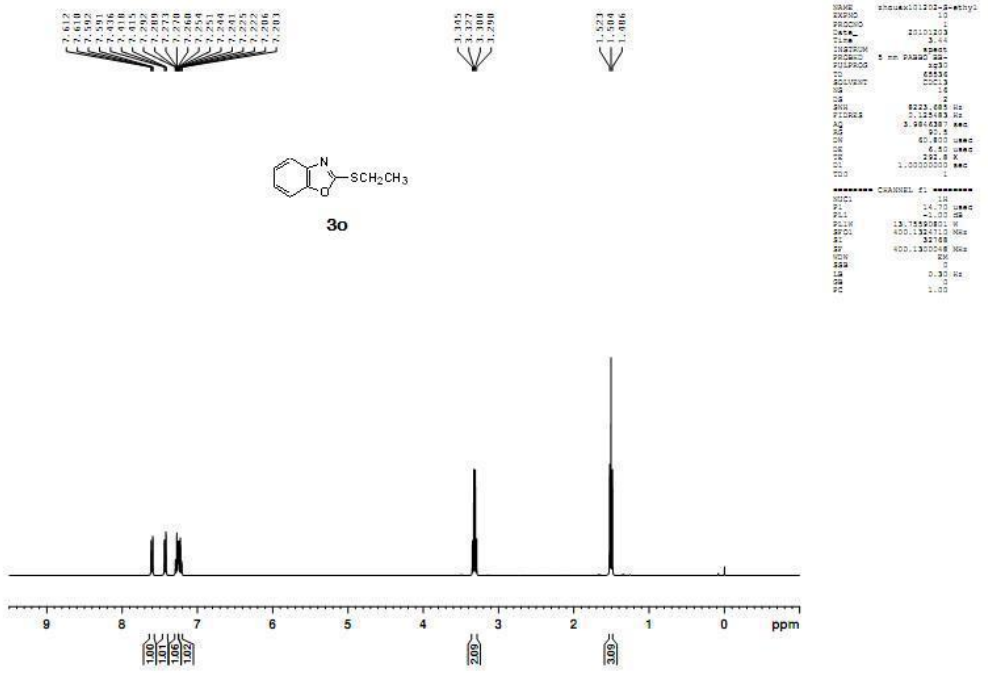

152.47
143.68
136.17
135.71
128.75
127.79
126.88
121.84
121.83
110.23
100.99
77.31
77.00
76.68
47.40
32.77
31.86
29.58
29.57
29.52
29.47
29.39
29.08
29.06
29.03
22.67
14.07
S(CH2)11CH3
N
N
Bn
3l
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

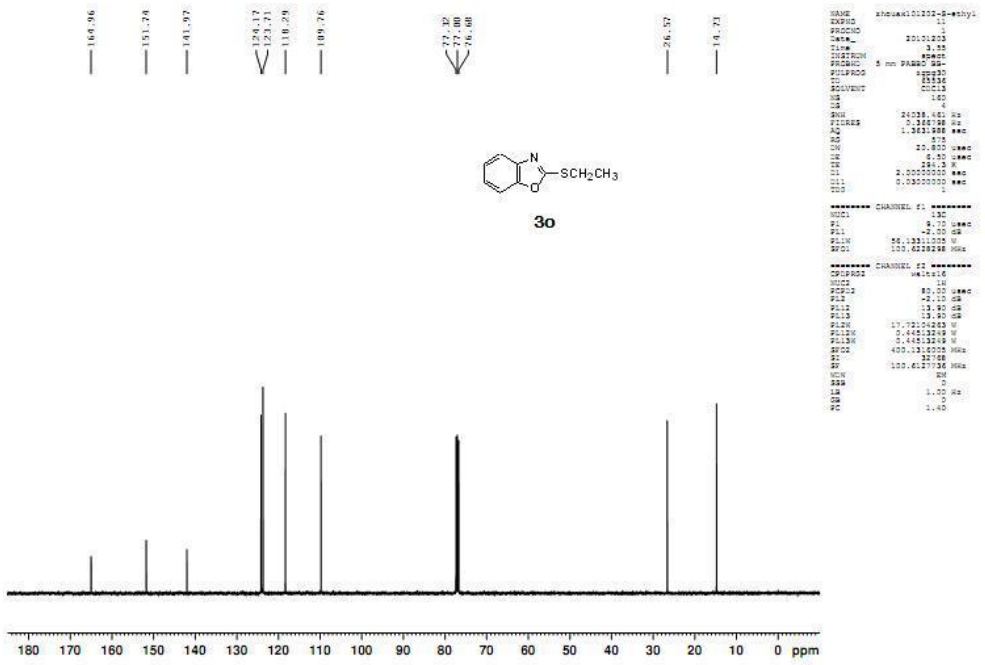

7.285
7.051
6.911
3.609
3.064
3.046
3.027
1.684
1.666
1.647
1.629
1.609
1.411
1.395
1.376
1.358
1.297
1.258
0.894
0.878
0.861
-0.001
S(CH2)11CH3
N
N
3m
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.98
0.94
3.00
2.03
2.05
2.14
16.70
3.00

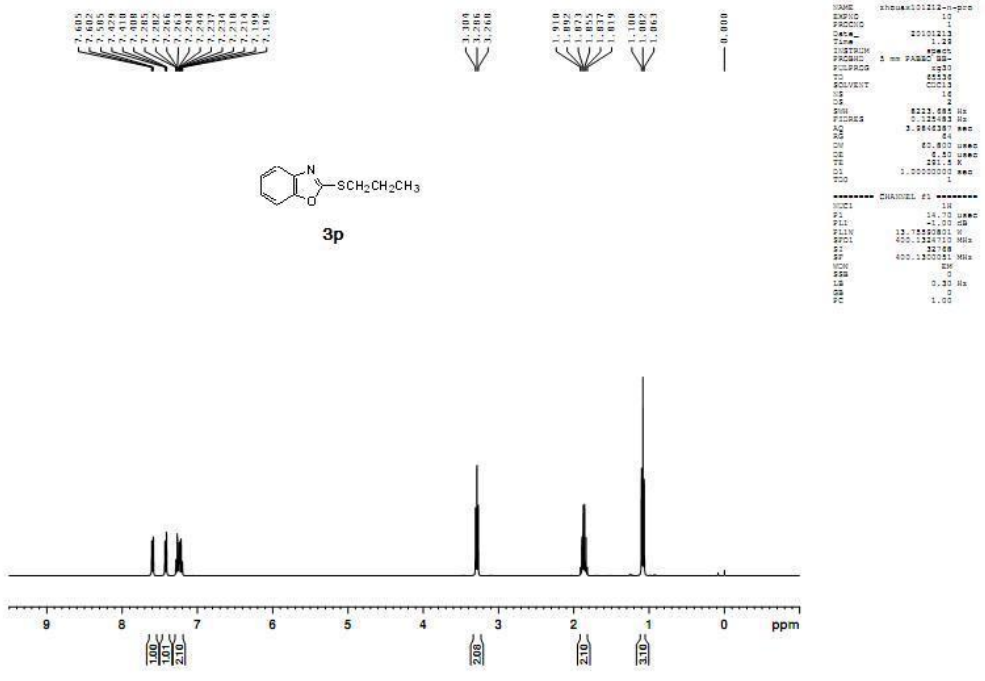

142.86
129.14
121.87
77.32
77.00
76.68
34.28
31.89
31.82
29.55
29.49
29.42
29.36
29.06
24.61
22.61
14.06
S(CH2)11CH3
3m
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

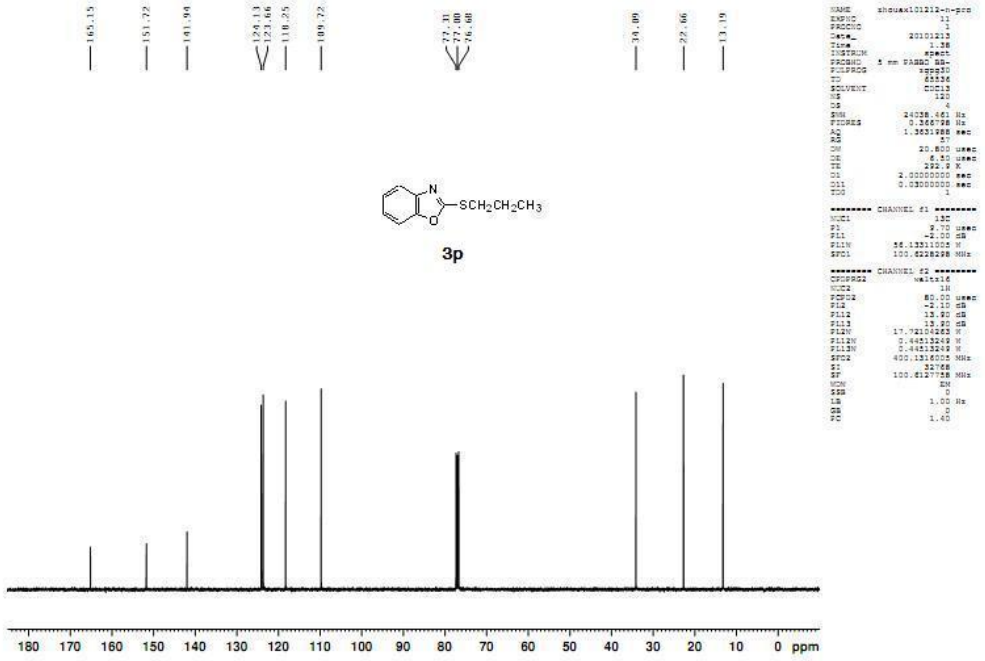

8.012
8.009
7.994
7.909
7.512
7.498
7.488
7.479
7.468
7.465
7.457
7.269
3.312
3.294
3.275
1.874
1.865
1.856
1.838
1.819
1.808
1.795
1.482
1.464
1.445
1.317
1.297
1.257
0.894
0.880
0.868
0.000
S(CH2)11CH3
3n
9 8 7 6 5 4 3 2 1 0 ppm
1.90
2.92
1.95
2.05
2.03
16.13
3.00

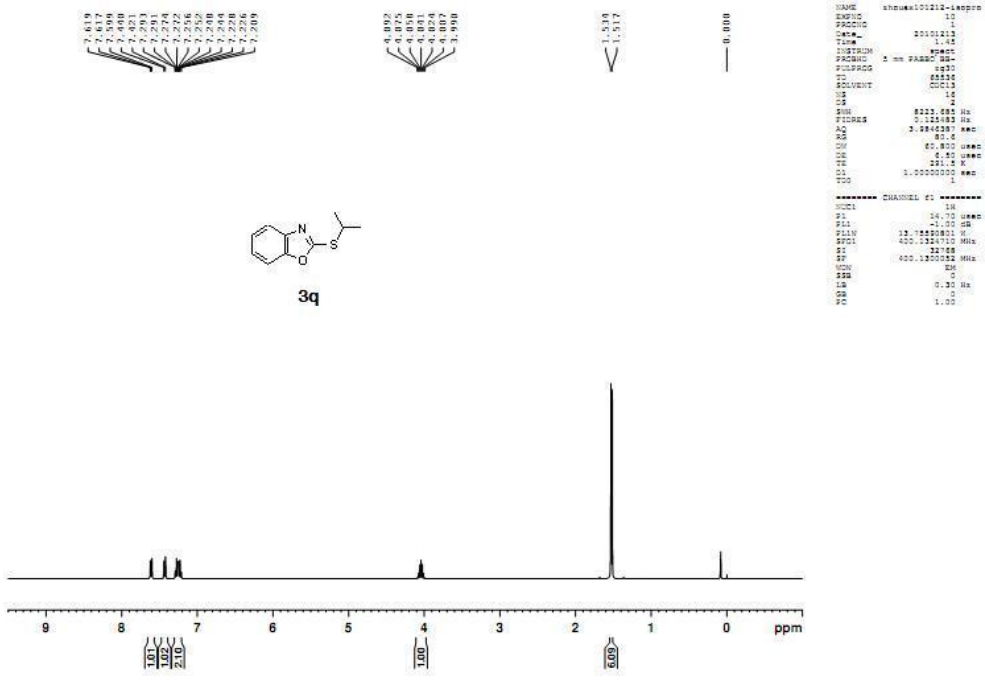

3n
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

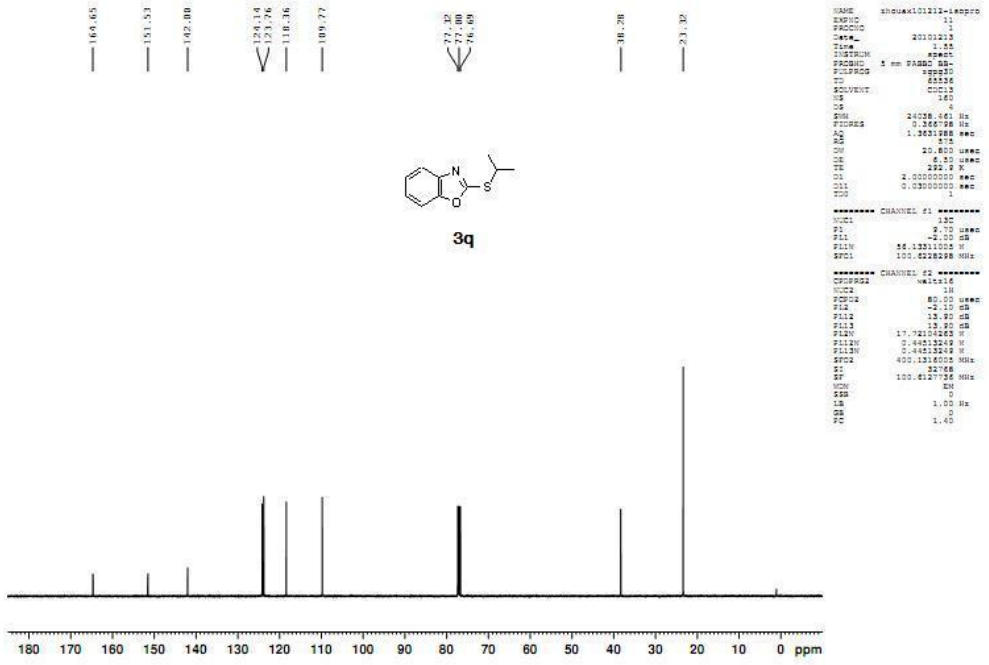

3o
9 8 7 6 5 4 3 2 1 0 ppm

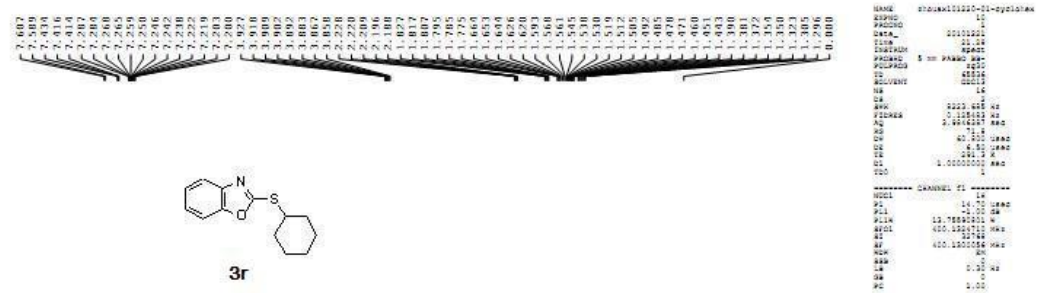

164.96
151.74
141.97
124.17
123.71
118.29
109.76
77.32
77.00
76.68
26.57
14.23
SCH2CH3
3o
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

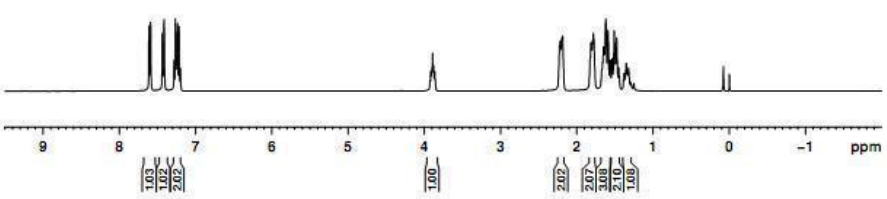

7.605
7.602
7.585
7.429
7.418
7.408
7.285
7.282
7.266
7.263
7.258
7.256
7.237
7.234
7.218
7.216
7.199
7.196
3.304
3.286
3.268
1.910
1.892
1.873
1.855
1.837
1.819
1.100
1.082
1.063
0.000
SCH2CH2CH3
3p
9 8 7 6 5 4 3 2 1 0 ppm
1.00
1.01
2.10
2.08
2.10
3.10

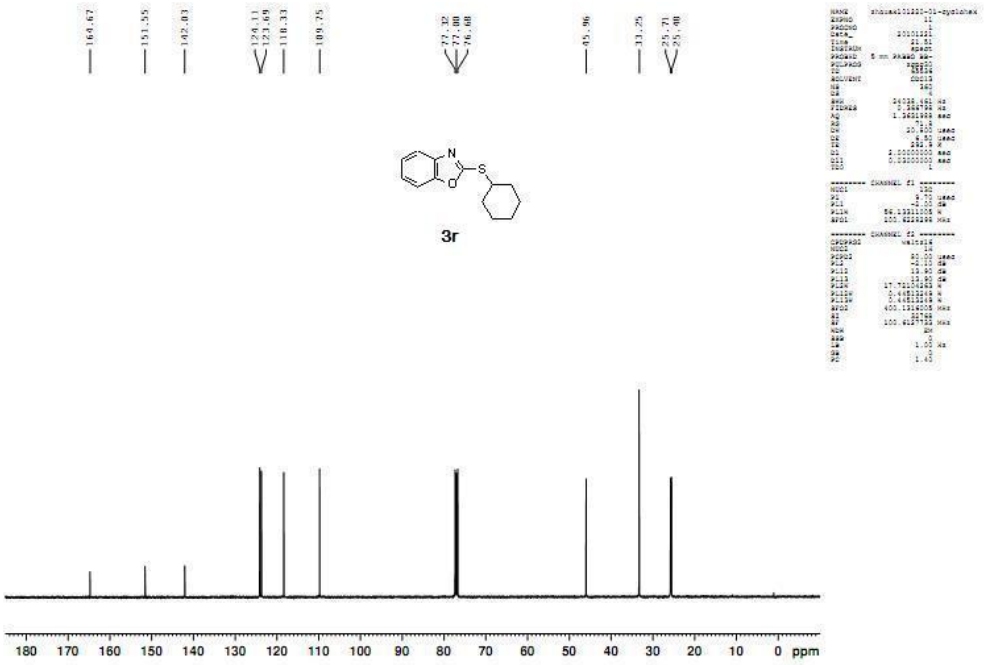

165.15
151.72
141.94
124.13
123.66
118.25
109.72
77.31
77.00
74.68
34.09
22.66
13.19
SCH2CH2CH3
3p
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

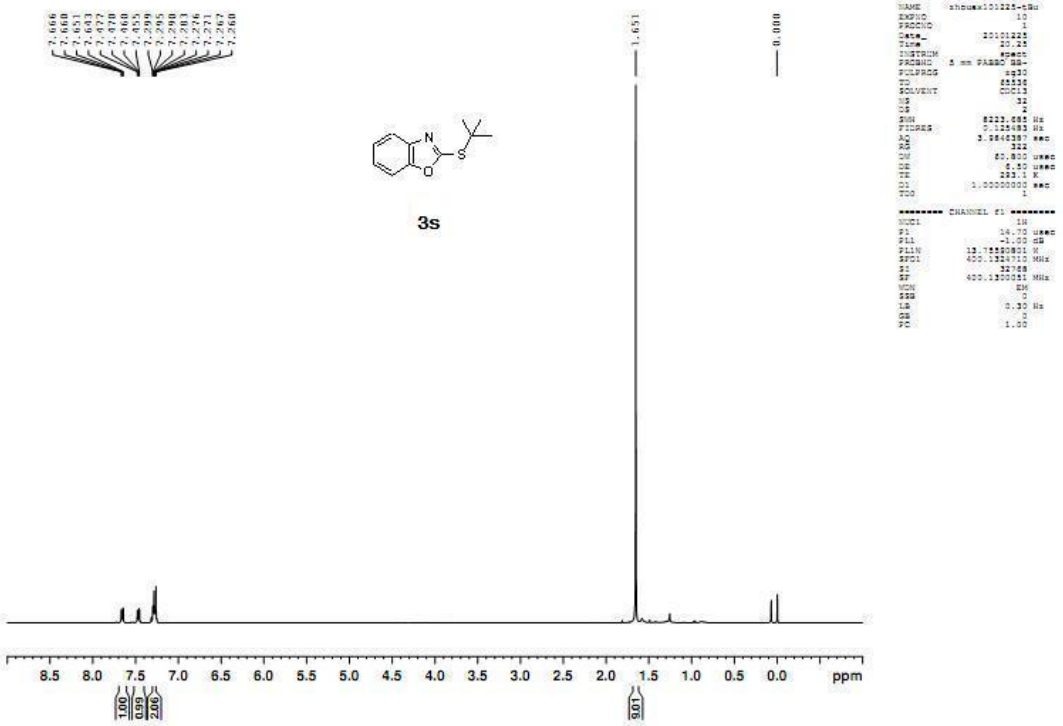

7.619
7.617
7.599
7.490
7.471
7.293
7.291
7.274
7.272
7.256
7.252
7.248
7.244
7.228
7.226
7.209
4.092
4.075
4.058
4.041
4.024
4.007
3.990
1.530
1.517
0.000
3q
9 8 7 6 5 4 3 2 1 0 ppm
1.01
1.02
2.10
1.00
6.09

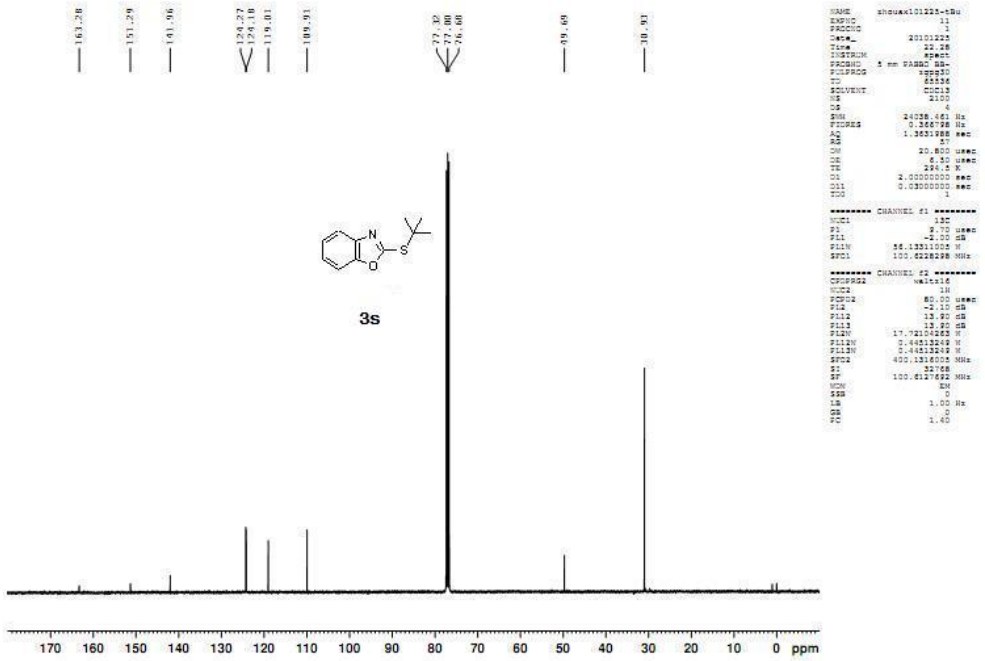

164.65
151.53
142.80
124.14
123.76
118.36
109.77
77.32
77.00
76.69
38.28
23.32
3q
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

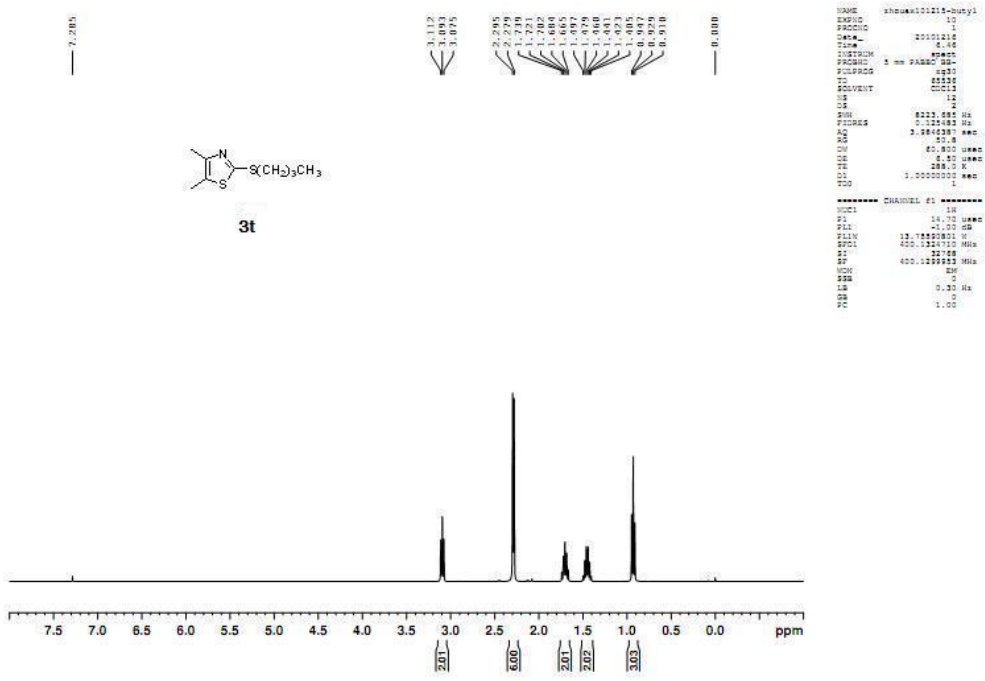

3r

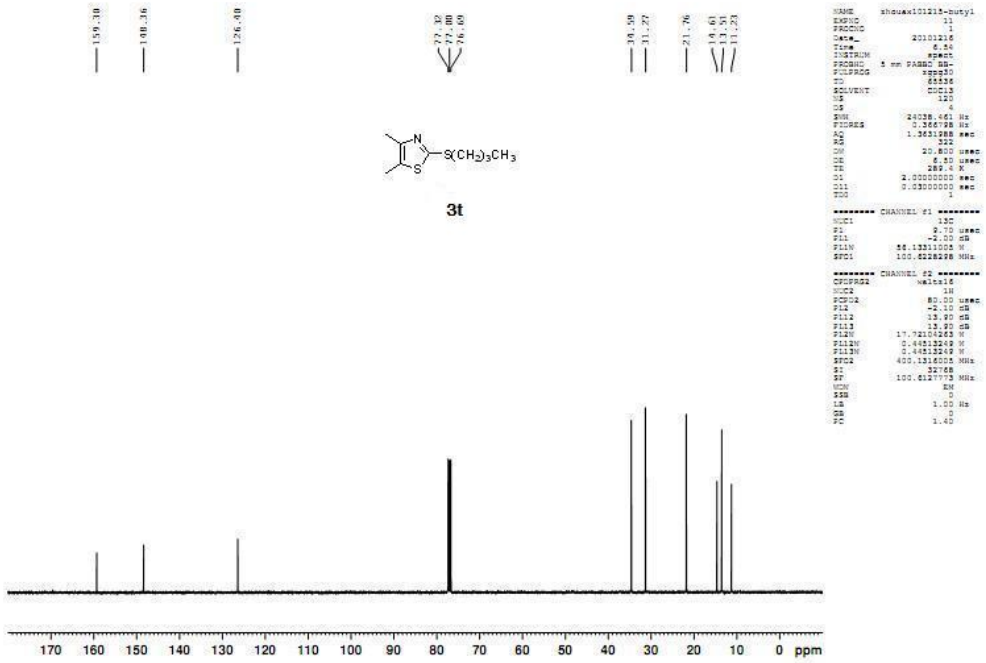

9 8 7 6 5 4 3 2 1 0 −1 ppm
1.03
1.02
2.02
1.00
2.02
2.07
3.08
2.10
1.08

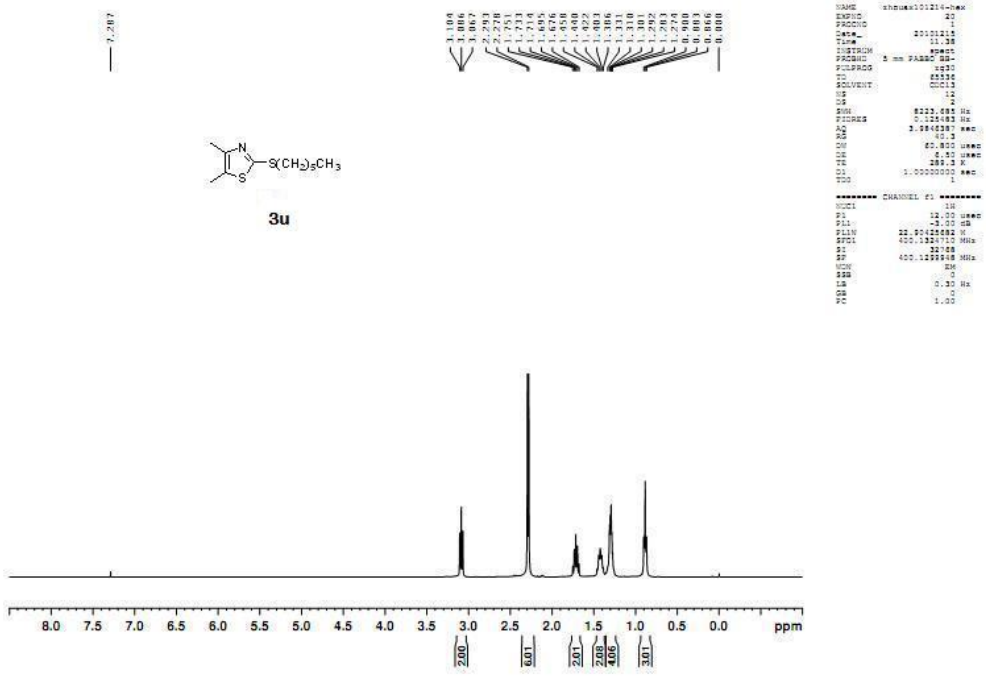

164.67
151.55
142.83
129.11
123.69
118.33
109.75
77.32
77.00
76.68
65.96
33.25
25.71
25.40
3r
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

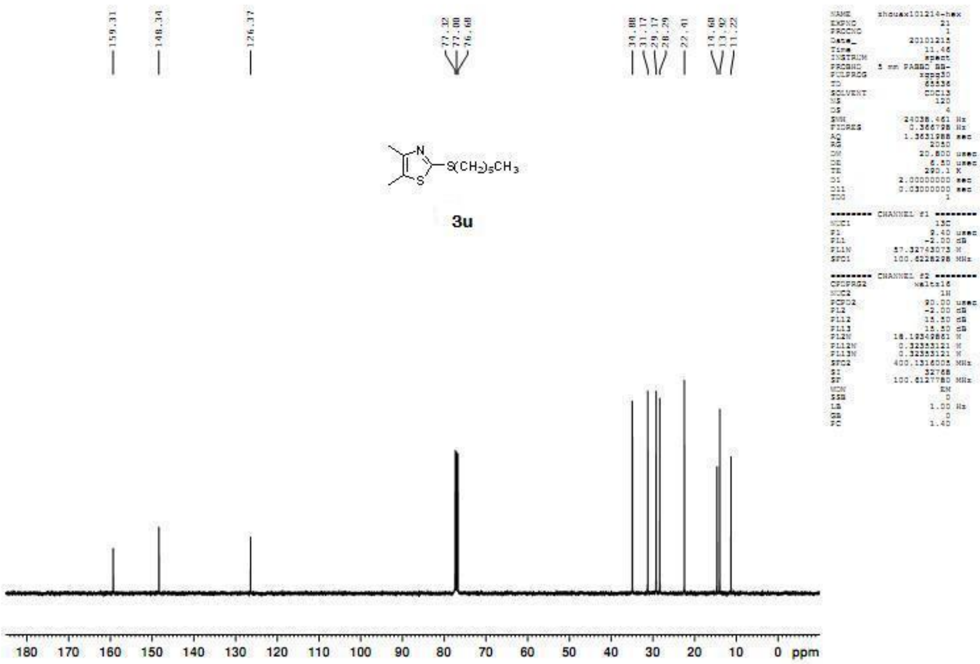

7.666
7.660
7.651
7.643
7.477
7.470
7.468
7.455
7.299
7.295
7.298
7.283
7.276
7.271
7.267
7.260
1.651
0.000
3s
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.00
0.99
2.06
9.01

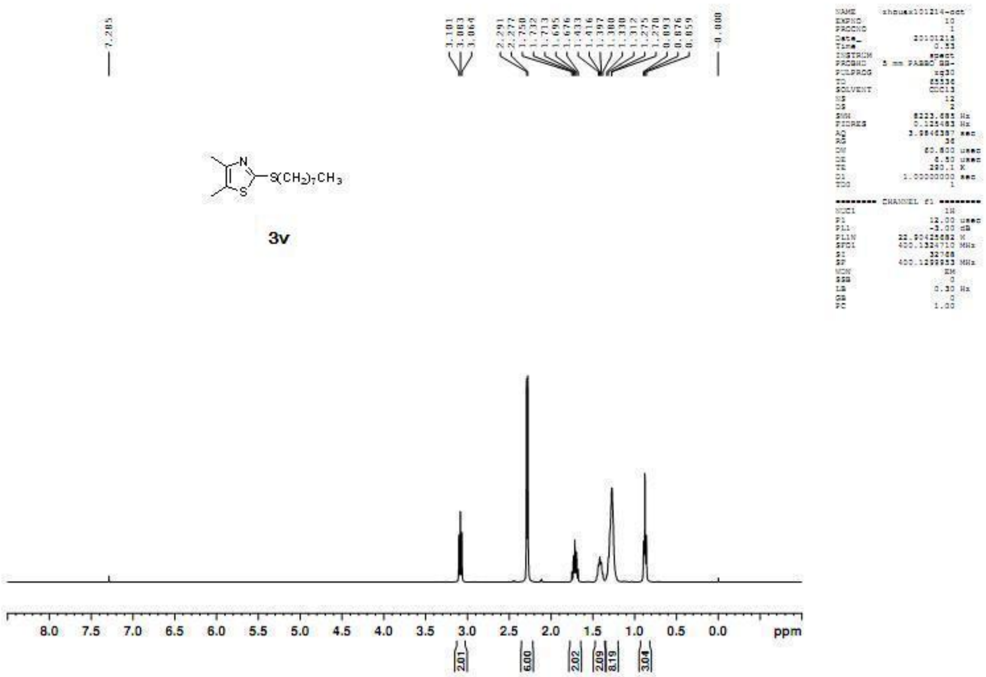

163.28
151.29
141.96
124.27
124.18
119.81
109.91
77.32
77.00
76.68
59.69
30.93
3s
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

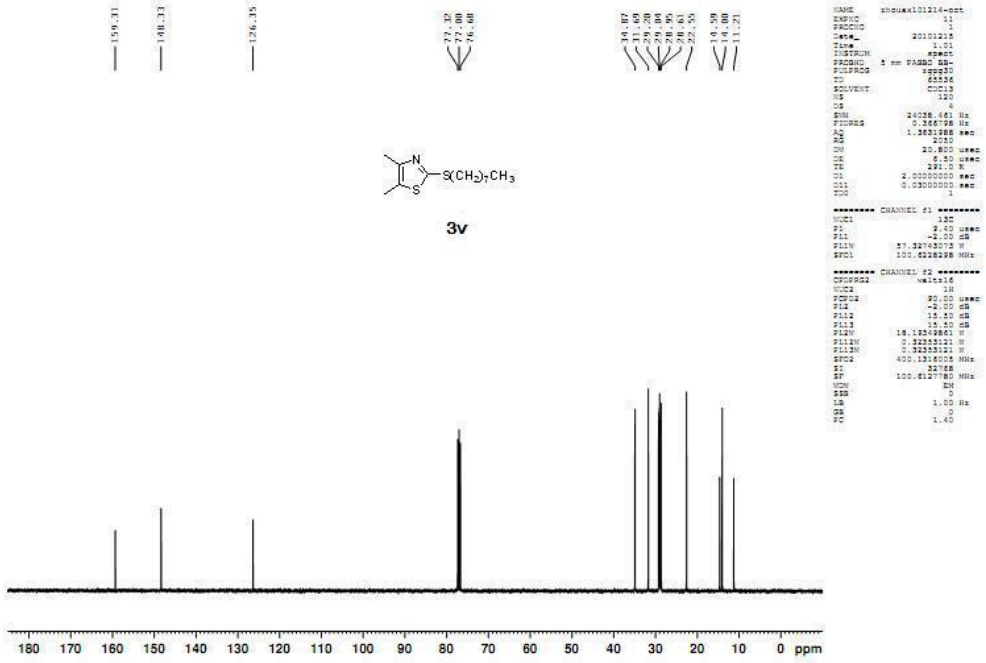

7.285
3.112
3.093
3.075
2.295
2.279
1.739
1.721
1.702
1.686
1.665
1.497
1.479
1.460
1.440
1.423
1.405
0.947
0.929
0.918
0.000
S(CH2)3CH3
3t
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.01
6.00
2.01
2.02
3.03

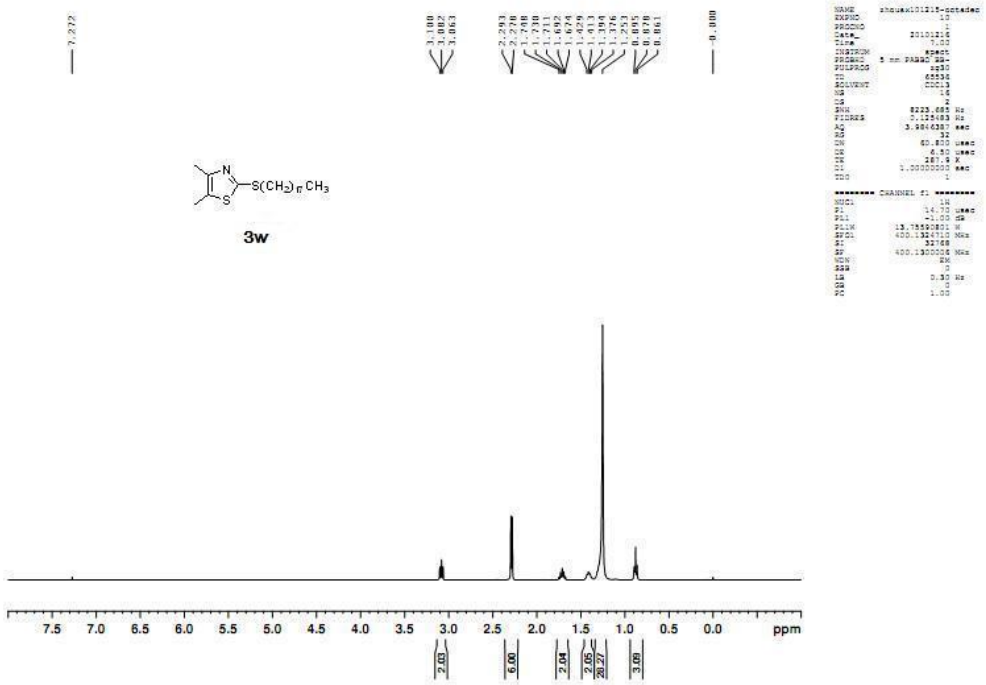

159.30
140.36
126.40
77.32
77.00
76.69
35.59
31.27
21.76
19.61
13.51
11.23
S(CH2)3CH3
3t
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

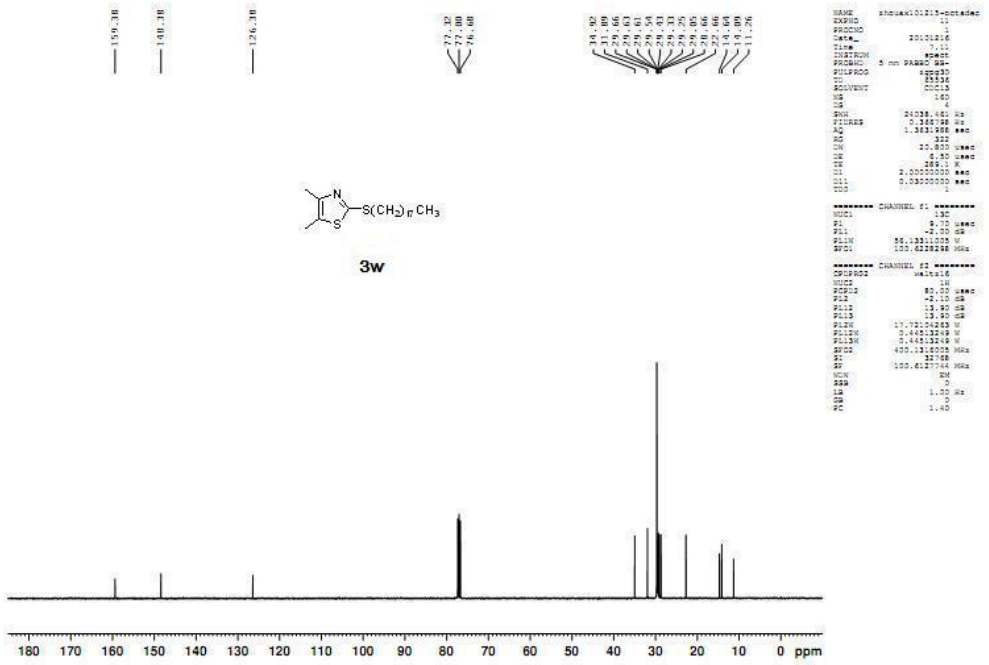

7.287
1.104
1.086
1.067
2.229
2.208
1.751
1.733
1.714
1.695
1.676
1.658
1.440
1.422
1.403
1.386
1.331
1.310
1.291
1.272
1.254
0.980
0.961
0.942
0.923
S(CH2)5CH3
3u
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.00
6.01
2.01
2.08
4.06
3.01

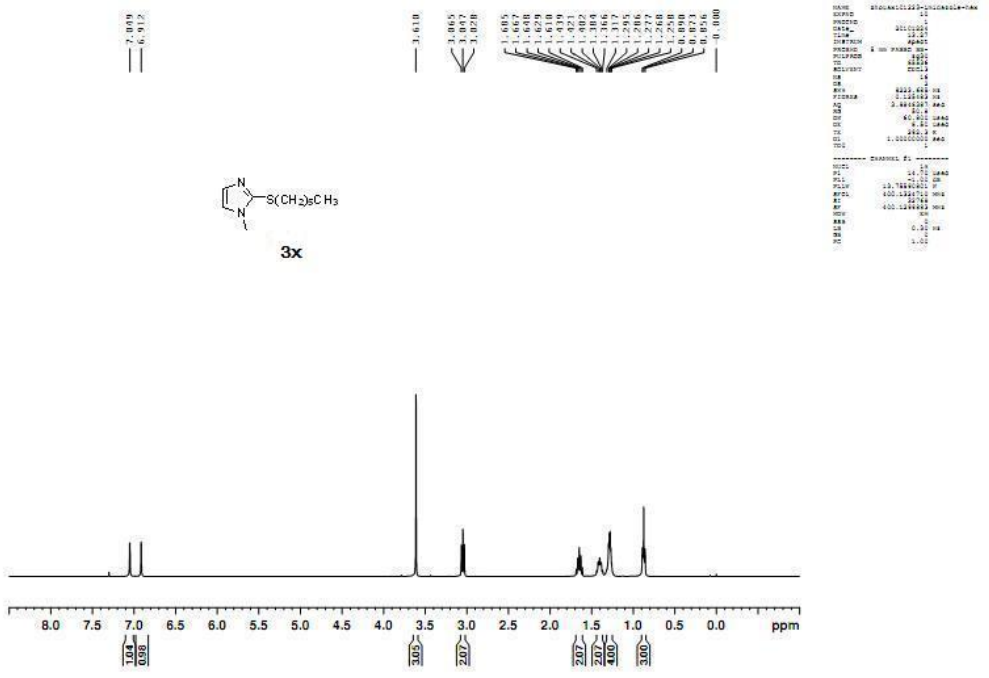

159.31
148.34
126.37
77.32
77.00
76.68
34.88
31.17
29.17
28.29
22.41
14.60
13.92
11.22
N
S
S(CH2)5CH3
3u
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

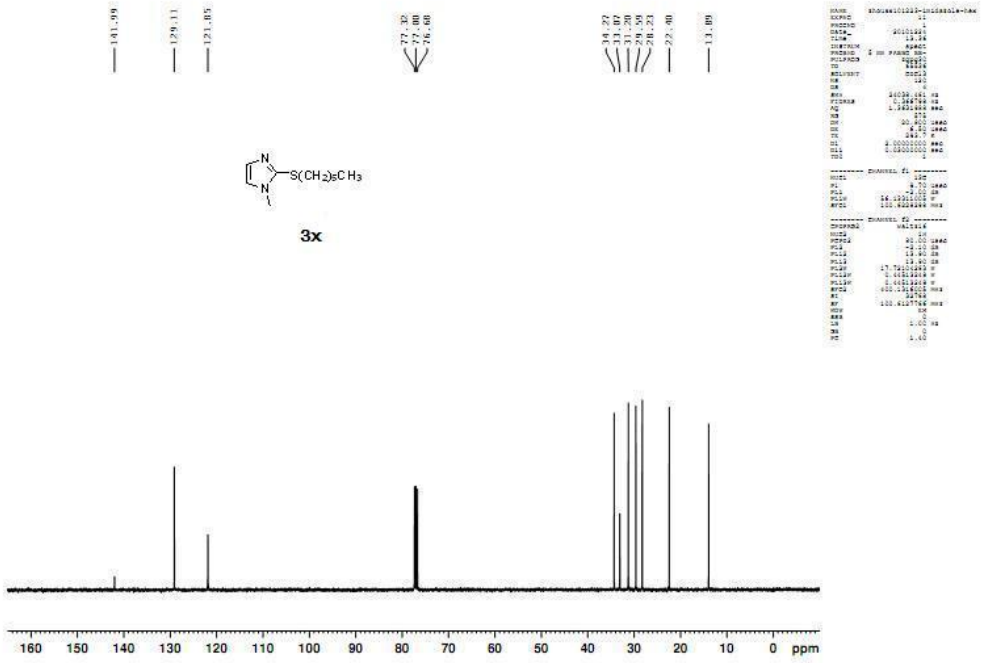

7.285
3.101
3.083
3.064
2.291
2.277
1.750
1.732
1.713
1.695
1.676
1.433
1.416
1.397
1.380
1.330
1.312
1.275
1.270
0.893
0.876
0.859
0.000
N
S
S(CH2)7CH3
3v
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.01
6.00
2.02
2.09
8.19
3.04

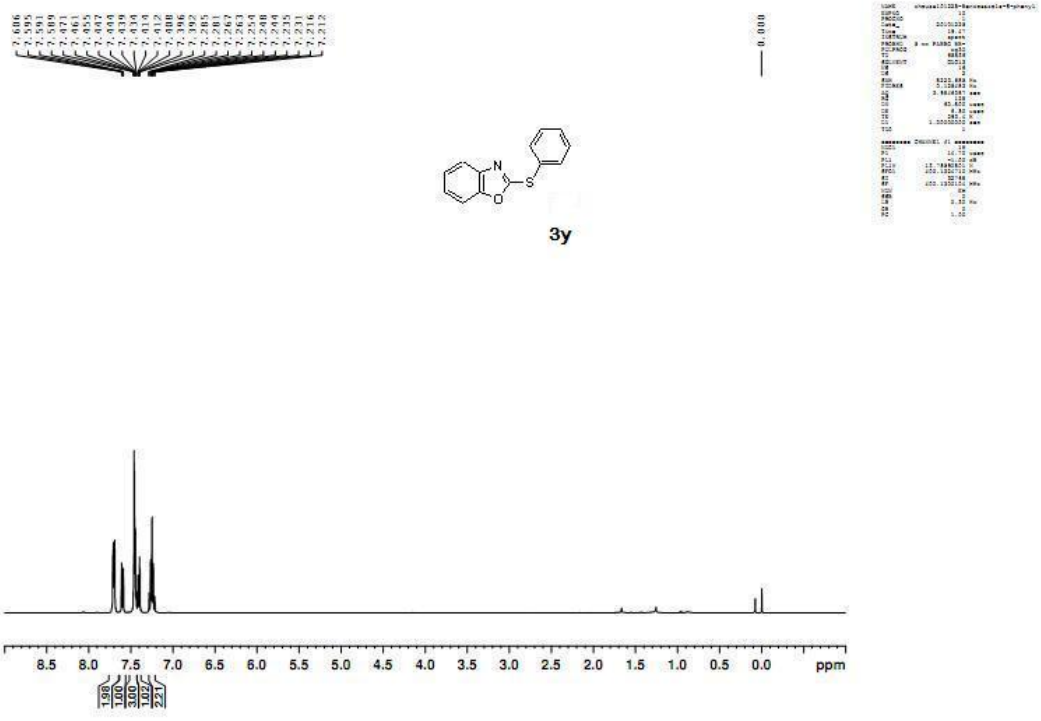

159.31
148.33
126.35
77.32
77.00
76.68
34.87
31.65
29.38
29.06
28.95
28.61
22.55
14.59
14.08
11.21
S(CH2)7CH3
3v
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

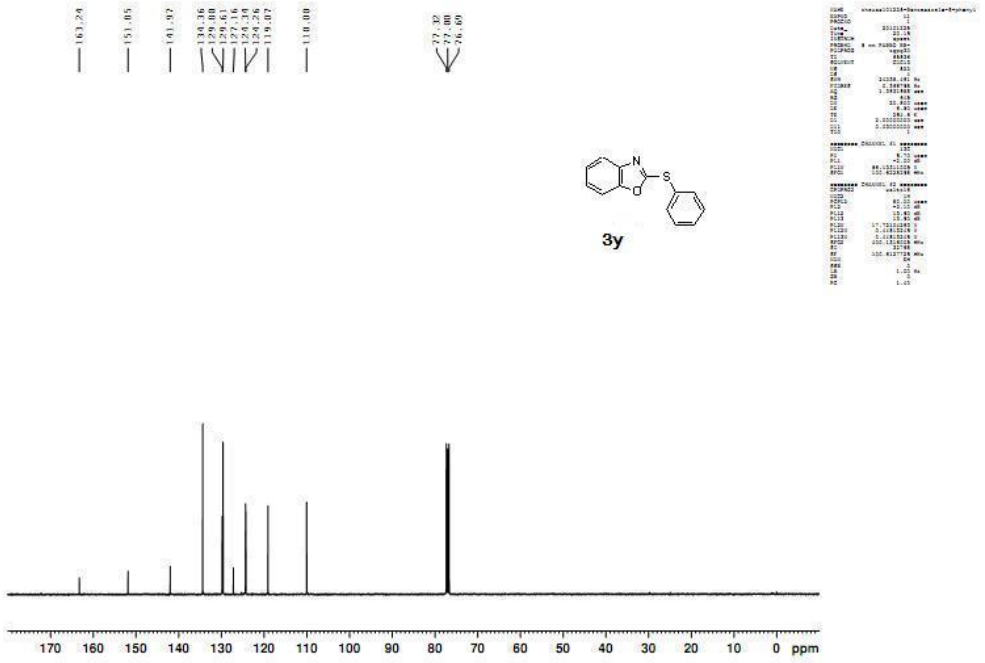

7.272
3.100
3.082
3.063
2.293
2.276
2.258
1.730
1.711
1.692
1.674
1.656
1.413
1.394
1.376
1.293
0.895
0.878
0.861
-0.000
S(CH2)11CH3
3w
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.00
6.00
2.04
2.05
20.27
3.00

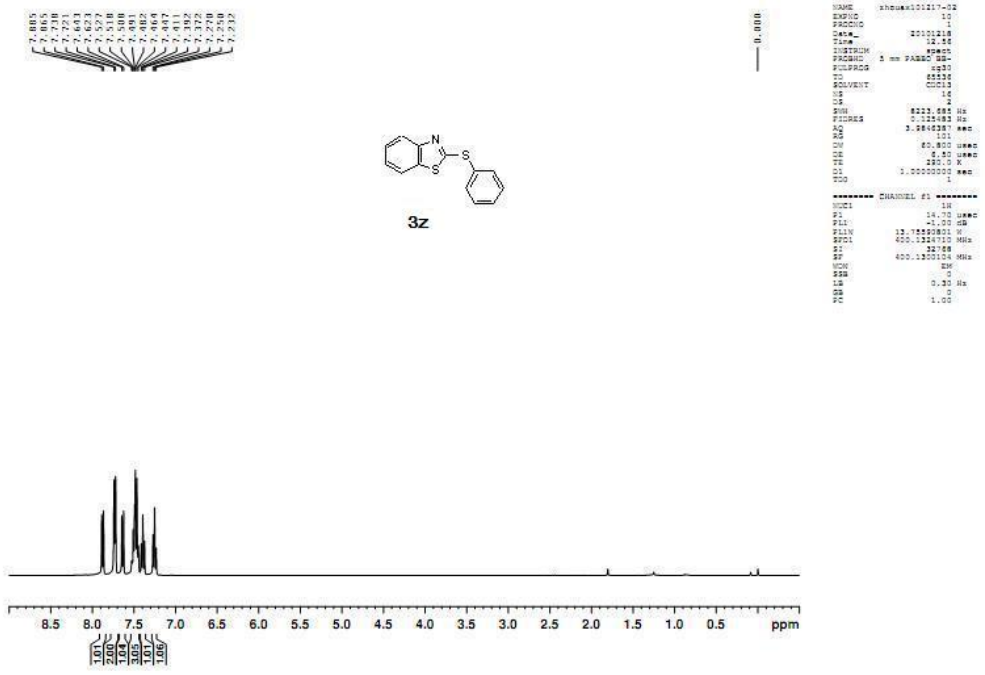

159.38
148.38
126.38
77.32
77.00
76.68
34.92
31.89
29.66
29.63
29.61
29.51
29.43
29.33
29.25
29.16
29.06
22.66
14.09
11.26
S(CH2)11CH3
3w
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

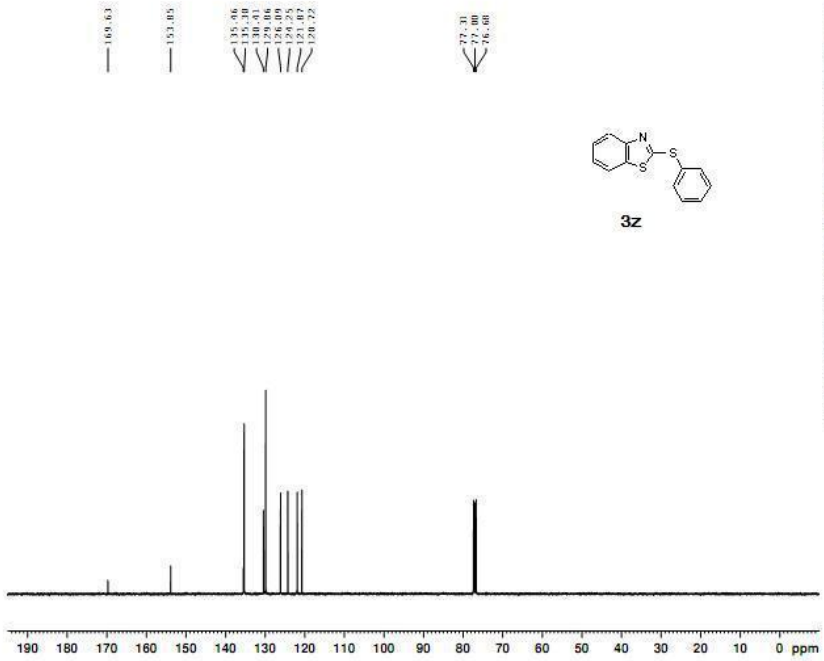

7.039
6.912
3.618
3.065
3.047
3.028
1.685
1.667
1.648
1.628
1.618
1.439
1.421
1.400
1.384
1.366
1.317
1.295
1.286
1.277
1.258
0.898
0.881
0.856
0.000
S(CH2)5CH3
3x
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.04
0.98
3.05
2.07
2.07
2.02
4.00
3.00

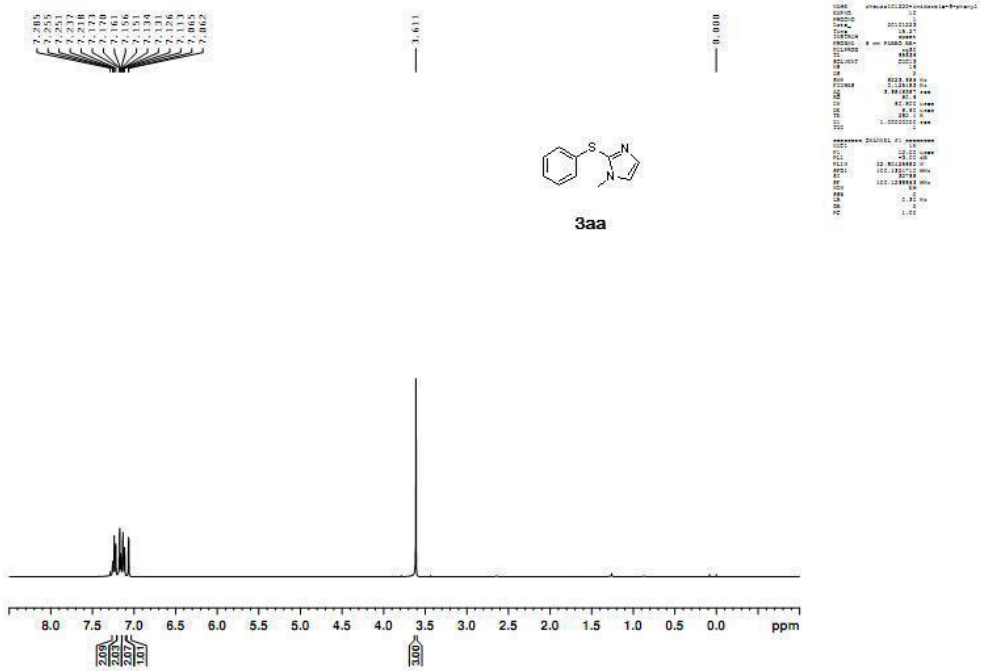

141.99
129.11
121.85
77.32
77.00
76.68
34.27
31.87
31.20
28.99
28.53
22.40
13.89
S(CH2)5CH3
3x
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

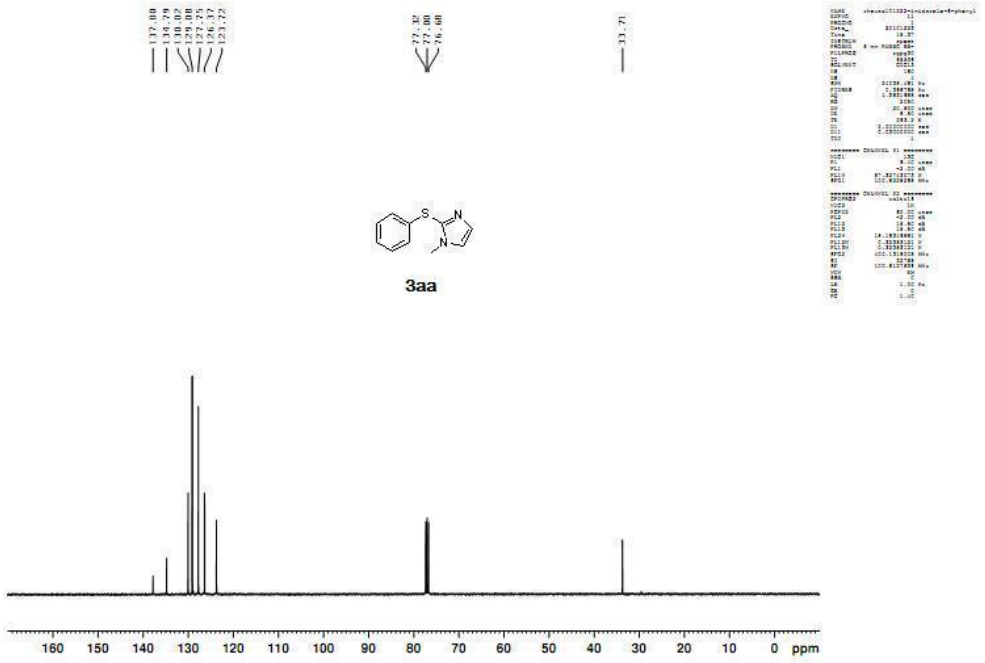

7.606
7.595
7.591
7.589
7.471
7.466
7.455
7.442
7.439
7.434
7.413
7.412
7.408
7.396
7.392
7.285
7.281
7.263
7.254
7.248
7.244
7.233
7.231
7.216
7.212
0.000
3y
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.90
1.00
1.00
1.01
2.21


163.24
151.85
141.92
134.36
129.88
129.61
127.16
126.84
124.26
119.87
110.88
77.32
77.00
76.69
3y
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.885
7.865
7.738
7.721
7.623
7.527
7.518
7.498
7.482
7.464
7.447
7.401
7.392
7.372
7.270
7.250
7.232
0.000
3z
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.01
2.00
1.04
1.05
1.00
1.06


169.63
153.85
135.96
135.30
130.31
129.06
126.09
124.45
121.85
120.72
77.31
77.00
76.68
3z
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.785
7.295
7.251
7.237
7.218
7.173
7.170
7.161
7.156
7.151
7.136
7.131
7.126
7.113
7.065
7.062
3.611
0.000
3aa
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.09
2.03
2.07
1.01
3.00


137.08
134.79
130.02
129.86
127.75
126.37
123.72
77.32
77.00
76.68
33.71
S
N
3aa
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm