Electronic Supplementary Information

Lewis Base-Catalyzed Diastereoselective [3 + 2] Cycloaddition Reaction of Nitrones with Electron-Deficient Alkenes: An Access to Isoxazolidine Derivatives

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General Information

All reactions were performed under a N_2 atmospheres in oven-dried glassware with magnetic stirring. Unless otherwise stated, all reagents were purchased from commercial suppliers and used without further purification. All solvents were purified and dried according to standard methods prior to use. Reactions were monitored through thin layer chromatography (TLC) on silica gel–precoated glass plates (0.25 mm thickness). Chromatograms were visualized by fluorescence quenching under UV light at 254 nm. Flash column chromatography was performed using flash silica gel (200–300 mesh). ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ using a 300M spectrometer, as indicated. Accurate mass measurements were performed with the ESI-MS technique.

Nitrones were prepared according to the literature procedures.[1]

General Procedure for the [3 + 2] Annulation Reactions

An oven-dried 15 mL of Schlenk tube was charged with Nitrone (0.20 mmol), catalyst (0.04 mmol) and vinyl sulfones (0.24 mmol) at room temperature. Then, 5 mL of CH_2Cl_2 was added and the mixture was stirred at room temperature for 48 h. The reaction mixture was concentrated and the residue was purified by flash column (ethyl acetate/ petroleum ether) to afford the corresponding cycloaddition product.

2-Methyl-3-phenyl-4,5-bis(phenylsulfonyl)isoxazolidine ***(3a)***

99% yield (87.8 mg); white solid; m.p. 171 – 173 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.96 – 7.93 (m, 2H), 7.80 – 7.77 (m, 2H), 7.73 – 7.68 (m, 1H), 7.62 – 7.56 (m, 3H), 7.49 – 7.43 (m, 2H), 7.29 – 7.22 (m, 5H), 5.33 (d, J = 3.6 Hz, 1H), 4.82 (dd, J = 3.6, 8.4 Hz, 1H), 4.02 (d, J = 8.4 Hz, 1H), 2.55 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 137.3, 136.2, 134.5, 134.4, 134.0, 129.5, 129.4, 129.1, 129.0, 128.8, 128.49, 128.48, 91.6, 74.4, 74.2,42.7; HRMS (ESI) calcd for $C_{22}H_{21}NO_5S_2Na^+$ $[M + Na]^+$ 466.0753, found 466.0750; IR (film) ν_{max}: 3065, 2966, 2876, 1585, 1495, 1479, 1448, 1311, 1179, 1152, 1108, 1085, 1061, 1025, 999, 951, 843, 802, 755, 725, 688, 638, 606, 588, 573, 559, 530 cm^{-1}.

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[1] H. Zheng, R. McDonald and D. G. Hall, *Chem. Eur. J.*, 2010, **16**, 5454.

*2-Methyl-4,5-bis(phenylsulfonyl)-3-o-tolylisoxazolidine (**3b**)*

81% yield (74.1 mg); white solid; m.p. 144 – 146 °C; ^{1}H NMR (300 MHz, CDCl$_3$): δ7.91 – 7.88 (m, 2H), 7.80 – 7.77 (m, 2H), 7.72 – 7.46 (m, 4H), 7.51 – 7.46 (m, 3H), 7.18 – 7.09 (m, 3H), 5.24 (d, *J* = 3.9 Hz, 1H), 4.88 (dd, *J* = 3.9, 8.7 Hz, 1H), 4.52 (d, *J* = 8.7 Hz, 1H), 2.55 (s, 3H), 2.45 (s, 3H); ^{13}C NMR (75 MHz, CDCl$_3$): δ 137.5, 136.3, 134.5, 134. 3, 132.0, 130.7, 129.5, 129.4, 129.1, 128.7, 128.3, 128.1, 126.8, 91.9, 74.5, 69.0, 42.4, 19.6; HRMS (ESI) calcd for $C_{23}H_{24}NO_5S_2^+$ $[M + H]^+$ 458.1090, found 458.1086; IR (film) ν_{max}: 3064, 2965, 2921, 1585, 1495, 1448, 1323, 1179, 1152, 1107, 1085, 1061, 999, 954, 864, 843, 794, 754, 722, 687, 632, 608, 588, 559, 503 cm^{-1}.

*2-Methyl-4,5-bis(phenylsulfonyl)-3-m-tolylisoxazolidine (**3c**)*

74% yield (67.7 mg); white solid; m.p. 112 – 114 °C; ^{1}H NMR (300 MHz, CDCl$_3$): δ 7.95 (d, *J* = 7.5 Hz, 2H), 7.79 (d, *J* = 7.5 Hz, 2H), 7.78 – 7.68 (m, 1H), 7.61 – 7.56 (m, 3H), 7.49 – 7.44 (m, 2H), 7.12 – 7.03 (m, 3H), 6.96 (s, 1H), 5.33 (d, *J* = 3.6 Hz, 1H), 4.83 – 4.79 (m, 1H), 3.95 (d, *J* = 8.4 Hz, 1H), 2.55 (s, 3H), 2.25 (s, 3H); ^{13}C NMR (75 MHz, CDCl$_3$): δ 138.4, 137.4, 136.4, 134.5, 134.4, 133.9, 129.8, 129.6, 129.3, 129.1, 129.0, 128.7, 128.6, 125.6, 74.4, 74.3, 42.7; HRMS (ESI) calcd for $C_{23}H_{24}NO_5S_2^+$ $[M + H]^+$ 458.1090, found 458.1085; IR (film) ν_{max}: 3064, 2965, 2923, 2875, 1724, 1609, 1585, 1490, 1479, 1448, 1402, 1325, 1152, 1108, 1086, 1062, 1024, 999, 958, 909, 842, 787, 753, 726, 688, 650, 594, 578, 553, 524 cm^{-1}.

*2-Methyl-4,5-bis(phenylsulfonyl)-3-p-tolylisoxazolidine (**3d**)*

69% yield (63.1 mg); white solid; m.p. 134 – 136 °C; ^{1}H NMR (300 MHz, CDCl$_3$): δ 7.93 (d, *J* = 7.4 Hz, 2H), 7.80 (d, *J* = 7.4 Hz, 2H), 7.73 – 7.68 (m, 1H), 7.62 – 7.55 (m, 3H), 7.50 – 7.46 (m, 2H), 7.15 (d, *J* = 8.0 Hz, 2H), 7.04 (d, *J* = 8.0 Hz, 2H), 5.29 (d, *J* = 3.5 Hz, 1H), 4.78 (dd, *J* = 3.5, 8.4 Hz, 1H), 4.01 (d, *J* = 8.4 Hz, 1H), 2.54 (s, 3H), 2.30 (s, 3H); ^{13}C NMR (75 MHz, CDCl$_3$): δ 138.9, 137.5, 136.3, 134.5, 134.4, 131.0, 129.6, 129.5, 129.4, 129.1, 128.6, 128.4, 91.7, 74.4, 73.9, 42.6; HRMS (ESI) calcd for $C_{23}H_{24}NO_5S_2^+$ $[M + H]^+$ 458.1090, found 458.1085; IR (film) ν_{max}: 3063, 2966, 2922, 2876, 1614, 1585, 1516, 1479, 1448, 1324, 1180, 1152, 1107, 1085, 1061, 1023, 999, 953, 847, 797, 753, 728, 688, 642, 608, 588, 572, 558, 526 cm^{-1}.

*3-(2,4-Dimethylphenyl)-2-methyl-4,5-bis(phenylsulfonyl)-iso-xazolidine (**3e**)*

90% yield (84.9 mg); white solid; m.p. 120 – 122 °C; ^{1}H NMR (300 MHz, CDCl$_3$): δ 7.89 – 7.86 (m, 2H), 7.80 – 7.77 (m, 2H), 7.71 – 7.46 (m,6H), 7.35 – 7.26 (m, 1H), 6.97 – 6.93 (m, 2H), 5.22 (d, *J* = 3.9 Hz, 1H), 4.84 (dd, *J* = 3.9, 8.8 Hz, 1H), 4.51 (d, *J* = 8.8 Hz, 1H), 2.53 (s, 3H), 2.41 (s, 3H), 2.27 (s, 3H); ^{13}C NMR (75 MHz, CDCl$_3$): δ 138.4, 137.5, 137.3, 136.3, 134.4, 134.3, 131.4, 129.4, 129.3, 129.0, 128.8, 128.3, 127.9, 127.5, 92.00, 74.4, 68.8, 42.2, 26.8, 21.0, 19.5; HRMS (ESI) calcd for $C_{24}H_{26}NO_5S_2^+$ $[M + H]^+$ 472.1247, found 472.1242; IR (film) ν_{max}: 3064, 2966, 2923, 1615, 1585, 1506, 1448, 1323, 1152, 1107, 1085, 1061, 999, 950, 848, 820, 799, 752, 688, 610, 591, 575, 559, 504 cm^{-1}.

*3-(3,4-Dimethylphenyl)-2-methyl-4,5-bis(phenylsulfonyl)-isoxazolidine (**3f**)*

87% yield (82.1 mg); white solid; m.p. 130 – 132 °C; ^{1}H NMR (300 MHz, CDCl$_3$): δ 7.95 – 9.92 (m, 2H), 7.81 – 7.78 (m, 2H), 7.72 – 7.67 (m, 1H), 7.63 – 7.54 (m, 3H), 7.49 – 7.44 (m, 2H), 7.00 – 6.94 (m, 2H), 6.91 (s, 1H), 5.30 (d, *J* = 3.6 Hz, 1H), 4.78 (dd, *J* = 3.6, 8.5 Hz, 1H), 3.94 (d, *J* = 8.5 Hz, 1H), 2.53 (s, 3H), 2.19 (s, 3H), 2.15 (s, 3H); ^{13}C NMR (75 MHz, CDCl$_3$): δ 137.5, 137.3, 136.8, 136.3, 134.34, 134.30, 131.2, 129.9, 129.5, 129.4, 129.2, 129.0, 128.6, 125.8, 91.6, 77.2, 74.3, 74.0, 42.6, 19.6, 19.4; HRMS (ESI) calcd for $C_{24}H_{26}NO_5S_2^+$ $[M + H]^+$ 472.1247, found 472.1242; IR (film) ν_{max}: 3064, 2966, 2923, 1585, 1505, 1448, 1323, 1180, 1152, 1107, 1085, 1062, 1024, 1000, 845, 798, 753, 688, 597, 581, 560 cm^{-1}.

*3-(2-Methoxyphenyl)-2-methyl-4,5-bis(phenylsulfonyl)iso-xazolidine (**3g**)*

76% yield (72.0 mg); white solid; m.p. 172 – 174 °C; ^{1}H NMR (300 MHz, CDCl$_3$): δ 7.99 (d, *J* = 7.4 Hz, 2H), 7.78 – 7.67 (m, 3H), 7.61 – 7.48 (m, 3H), 7.41 – 7.33 (m, 3H), 7.19 – 7.13 (m, 1H), 6.85 (t, *J* = 7.4 Hz, 1H), 6.65 (d, *J* = 8.2 Hz, 1H), 5.41 (d, *J* = 3.8 Hz, 1H), 5.09 – 5.05 (m, 1H), 4.59 (d, *J* = 8.9 Hz, 1H), 3.74 (s, 3H), 2.53 (s, 3H); ^{13}C NMR (75 MHz, CDCl$_3$): δ 137.4, 136.6, 134.3, 134.2, 130.1, 129.51, 129.46, 129.1, 128.9, 128.5, 121.1, 120.9, 110.6, 91.3, 73.0, 67.2, 55.3, 42.7; HRMS (ESI) calcd for $C_{23}H_{24}NO_6S_2^+$ $[M + H]^+$ 474.1074, found 474.1037; IR (film) ν_{max}: 3065, 2966, 2843, 1603, 1586, 1497, 1448, 1402, 1325, 1291, 1253, 1152, 1108, 1085, 1052, 1026, 999, 950, 869, 840, 793, 755, 687, 631, 595, 574, 548, 532 cm^{-1}.

*3-(3-Methoxyphenyl)-2-methyl-4,5-bis(phenylsulfonyl)iso-xazolidine (**3h**)*

80% yield (75.8 mg); white solid; m.p. 152 – 154 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.96 – 7.92 (m, 2H), 7.82 – 7.79 (m, 2H), 7.72 – 7.67 (m, 1H), 7.63 – 7.54 (m, 3H), 7.50– 7.45 (m, 2H), 7.15 – 7.09 (m, 1H), 6.92 – 6.91 (m, 1H), 6.83 – 6.77 (m, 2H), 5.32 (d, *J* = 3.5 Hz, 1H), 4.83 (dd, *J* = 3.5, 8.3 Hz, 1H), 3.98 (d, *J* = 8.3 Hz, 1H), 3.77 (s, 3H), 2.56 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 159.7, 140.3, 137.3, 136.1, 135.6, 134.9, 134.5, 134.4, 129.8, 129.7, 129.4, 129.1, 128.5, 128.4, 120.7, 114.9, 113.6, 91.6, 74.4, 74.0, 55.2, 42.7; HRMS (ESI) calcd for $C_{23}H_{24}NO_6S_2^+$ $[M + H]^+$ 474.1074, found 474.1037; IR (film) ν_{max}: 3065, 3004, 2964, 2878, 1603, 1586, 1489, 1448, 1403, 1323, 1261, 1151, 1108, 1085, 1060, 999, 906, 878, 796, 751, 688, 605, 586, 525 cm^{-1}.

*3-(4-Methoxyphenyl)-2-methyl-4,5-bis(phenylsulfonyl)iso-xazolidine (**3i**)*

63% yield (59.7 mg); white solid; m.p. 149 – 151 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.95 – 7.92 (m, 2H), 7.81 – 7.78 (m, 2H), 7.73 – 7.68 (m, 1H), 7.62 – 7.55 (m, 3H), 7.51 – 7.48 (m, 2H), 7.20 (d, *J* = 8.7 Hz, 2H), 6.76 (d, *J* = 8.7 Hz, 2H), 5.30 (d, *J* = 3.6 Hz, 1H), 4.76 (dd, *J* = 3.6, 8.4 Hz, 1H), 3.99 (d, *J* = 8.4 Hz, 1H), 3.77 (s, 3H), 2.54 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 160.0, 137.4, 136.2, 134.5, 134.3, 129.6, 129.5, 129.4, 129.1, 128.5, 125.8, 114.1, 91.6, 74.2, 73.6, 55.2, 42.5; HRMS (ESI) calcd for $C_{23}H_{24}NO_6S_2^+$ $[M + H]^+$ 474.1074, found 474.1040; IR (film) ν_{max}: 3065, 3004, 2961, 2925, 1613, 1585, 1515, 1448, 1310, 1252, 1177, 1152, 1107, 1085, 1061, 1030, 999, 953, 912, 848, 828, 799, 754, 731, 688, 638, 609, 588, 559, 545, 525 cm^{-1}.

*3-(2,3-Dimethoxyphenyl)-2-methyl-4,5-bis(phenylsulfonyl)-isoxazolidine (**3j**)*

77% yield (77.6 mg); white solid; m.p. 128 – 130 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.91 – 7.82 (m, 4H), 7.70 – 7.45 (m, 6H), 7.08 – 6.96 (m, 2H), 6.84– 6.81 (m, 1H), 5.26 (d, *J* =3.8 Hz, 1H), 5.00 (dd, *J* = 3.8, 8.8 Hz, 1H), 4.72 (d, *J* = 8.8 Hz, 1H), 3.93 (s, 3H), 3.83 (s, 3H), 2.55 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 152.5, 148.2, 137.3, 136.4, 134.30, 134.25, 129.4, 129.2, 129.0, 128.6, 126.9, 124.3, 120.4, 112.9, 91.8, 77.2, 73.5, 66.7, 61.1, 55.7, 42.6; HRMS (ESI) calcd for $C_{24}H_{26}NO_7S_2^+$ $[M + H]^+$ 504.1145, found 504.1140; IR (film) ν_{max}: 3064, 2942, 2840, 1587, 1483, 1448, 1324, 1271, 1224, 1152, 1085, 1001, 910, 785, 755, 725, 688, 606, 588, 557, 512 cm^{-1}.

*3-(2,4-Dimethoxyphenyl)-2-methyl-4,5-bis(phenylsulfonyl)-isoxazolidine (**3k**)*

75% yield (75.5 mg); white solid; m.p. 171 – 173 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.99 – 7.96 (m, 2H), 7.79 – 7.76 (m, 2H), 7.72 – 7.67(m, 1H), 7.60 – 7.51 (m, 3H), 7.41 – 7.36 (m, 2H), 7.32 – 7.27 (m, 1H), 6.38 (dd, *J* = 2.4, 8.7 Hz, 1H), 6.21 (d, *J* = 2.4 Hz, 1H), 5.39 (d, *J* = 3.8 Hz, 1H), 5.04 (dd, *J* = 8.7, 3.8 Hz, 1H), 4.47 (t, *J* = 9.8 Hz, 1H), 3.75 (s, 3H), 3.72 (s, 3H), 2.51 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 161.2, 158.6, 137.4, 136.6, 134.2, 134.1, 130.4, 129.5, 129.0, 128.8, 128.5, 113.2, 105.2, 98.2, 91.3, 77.2, 72.6, 67.2, 55.30, 55.26, 42.6, 29.6; HRMS (ESI) calcd for $C_{24}H_{26}NO_7S_2^+$ $[M + H]^+$ 504.1145, found 504.1142; IR (film) ν_{max}: 3064, 2963, 2926, 2853, 1737, 1613, 1587, 1511, 1448, 1422, 1377, 1310, 1265, 1211, 1181, 1149, 1106, 1085, 1033, 937, 798, 755, 733, 687, 636, 609, 585, 545 cm^{-1}.

*2-Methyl-4,5-bis(phenylsulfonyl)-3-(2,3,4-trimethoxyphenyl) isoxazolidine (**3l**)*

78% yield (83.2 mg); white solid; m.p. 132 – 134 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.94 – 7.91 (m, 2H), 7.86 – 7.82 (m, 2H), 7.72 – 7.66 (m, 1H), 7.62 – 7.54 (m, 3H), 7.50 – 7.44 (m, 2H), 7.15 (d, *J* = 8.8 Hz, 1H), 6.60 (d, *J* = 8.8 Hz, 1H), 5.30 (d, *J* = 3.9 Hz, 1H), 5.02 (dd, *J* = 3.9, 8.8 Hz, 1H), 4.54 (d, *J* = 8.8 Hz, 1H), 3.95 (s, 3H), 3.83 (s, 3H), 3.79 (s, 3H), 2.55 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 154.2, 152.7, 141.7, 137.5, 136.5, 134.2, 129.4, 129.2, 129.0, 128.6, 124.0, 118.7, 107.7, 91.6, 77.2, 73.0, 67.5, 61.3, 60.6, 55.9, 42.6; HRMS (ESI) calcd for $C_{25}H_{28}NO_8S_2^+$ $[M + H]^+$ 534.1251, found 534.1252; IR (film) ν_{max}: 3064, 2941, 2840, 1600, 1498, 1469, 1448, 1420, 1310, 1298, 1234, 1152, 1097, 1063, 1023, 962, 903, 798, 753, 725,688, 607, 585, 549 cm^{-1}.

*N,N-Dimethyl-4-(2-methyl-4,5-bis(phenylsulfonyl)isoxazoli-din-3-yl)aniline (**3m**)*

65% yield (63.3 mg); yellow solid; m.p. 121 – 123 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.95 – 7.92 (m, 2H), 7.81 – 7.77 (m, 2H), 7.71 – 7.66 (m, 1H), 7.60 – 7.54 (m, 3H), 7.49 – 7.44 (m, 2H), 7.10 – 7.07 (m, 2H), 6.56 – 6.53 (m, 2H), 5.30 (d, *J* = 3.6 Hz, 1H), 4.76 (dd, *J* = 3.6, 8.4 Hz, 1H), 3.94 (d, *J* = 8.4 Hz, 1H), 2.91 (s, 6H), 2.52 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 150.8, 137.5, 136.4, 134.3, 134.2, 129.5, 129.3, 129.2, 129.1, 129.0, 128.9, 128.5, 120.7, 112.3, 91.7, 74.0, 73.9, 42.5, 40.3; HRMS (ESI) calcd for $C_{24}H_{27}N_2O_5S_2^+$ $[M + H]^+$ 487.1356, found 487.1353; IR (film) ν_{max}: 3065, 2922, 1615, 1584, 1527, 1480, 1448, 1322, 1152, 1106, 1085, 1061, 999, 947, 843, 817, 795, 755, 722, 688, 640, 608, 588, 571, 544 cm^{-1}.

*3-(2-Fluorophenyl)-2-methyl-4,5-bis(phenylsulfonyl)isoxa-zolidine (**3n**)*

51% yield (47.1 mg); white solid; m.p. 167 – 169 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.93 – 7.91 (m, 2H), 7.82 – 7.79 (m, 2H), 7.74 – 7.48 (m, 6H), 7.32 – 7.27 (m, 2H), 6.98 – 6.91 (m, 2H), 5.29 (d, *J* = 3.5 Hz, 1H), 4.74 (dd, *J* = 3.5, 8.3 Hz, 1H), 4.04 (d, *J* = 8.3 Hz, 1H), 2.55 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 164.6, 161.3, 137.3, 136.1, 134.7, 134.4, 130.4, 130.3, 130.1, 130.0, 129.5, 129.2, 128.5, 116.0, 115.7, 91.6, 74.4, 73.2, 42.6; HRMS (ESI) calcd for $C_{22}H_{21}O_5NS_2F^+$ $[M + H]^+$ 462.0840, found 462.0835; IR (film) ν_{max}: 3066, 2963, 2926, 1734, 1607, 1585, 1512, 1479, 1448, 1311, 1266, 1228, 1152, 1108, 1085, 1062, 1024, 1000, 954, 872, 852, 829, 803, 783, 753, 730, 687, 636, 609, 588, 570, 558, 535 cm^{-1}.

*3-(2-Chlorophenyl)-2-methyl-4,5-bis(phenylsulfonyl)isoxa-zolidine (**3o**)*

91% yield (87.0 mg); white solid; m.p. 139 – 141 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.98 – 7.95 (m, 2H), 7.86 – 7.82 (m, 2H), 7.73 – 7.65 (m, 2H), 7.62 – 7.53 (m, 3H), 7.45 – 7.40 (m, 2H), 7.27 – 7.15 (m, 3H), 5.38 (d, *J* = 3.7 Hz, 1H), 4.91 (dd, *J* = 3.7, 8.7 Hz, 1H), 4.80 (d, *J* = 8.7 Hz, 1H), 2.58 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 137.0, 136.1, 134.8, 134.5, 131.1, 130.1, 129.7, 129.6, 129.5, 129.3, 129.2, 128.4, 127.6, 91.3, 74.3, 69.0, 42.5; HRMS (ESI) calcd for $C_{22}H_{21}O_5NS_2Cl^+$ $[M + H]^+$ 478.0544, found 478.0547; IR (film) ν_{max}: 3065, 2965, 2924, 2854, 1585, 1570, 1477, 1448, 1435, 1403, 1325, 1280, 1180, 1153, 1107, 1085, 1062, 1024, 999, 948, 911, 864, 842, 788, 754, 687, 649, 626, 607, 590, 574, 559, 544 cm^{-1}.

*3-(3-Chlorophenyl)-2-methyl-4,5-bis(phenylsulfonyl)iso-xazolidine (**3p**)*

62% yield (59.3 mg); white solid; m.p. 140 – 142 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.95 – 7.92 (m, 2H), 7.83 – 7.81 (m, 2H), 7.76 – 7.49 (m, 6H), 7.30 – 7.17 (m, 3H), 7.07 (s, 1H), 5.31 (d, *J* = 3.3 Hz, 1H), 4.76 – 4.71 (m, 1H), 3.97 (d, *J* = 8.3 Hz, 1H), 2.56 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 137.1, 136.3, 136.0, 134.8, 134.6, 134.5, 130.2, 129.6, 129.5, 129.3, 129.2, 128.6, 128.5, 126.7, 91.6, 74.5, 73.4, 42.7; HRMS (ESI) calcd for $C_{22}H_{21}O_5NS_2Cl^+$ $[M + H]^+$ 478.0544, found 478.0543; IR (film) ν_{max}: 3065, 2965, 2922, 2853, 1733, 1599, 1585, 1493, 1448, 1415, 1324, 1179, 1152, 1086, 1062, 1016, 999, 954, 850, 821, 802, 754, 728, 715, 687, 633, 607, 587, 573, 559, 531 cm^{-1}.

*3-(4-Chlorophenyl)-2-methyl-4,5-bis(phenylsulfonyl)iso-xazolidine (**3q**)*

61% yield (58.3 mg); white solid; m.p. 162 – 164 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.92 – 7.89 (m, 2H), 7.83 – 7.80 (m, 2H), 7.71 – 7.50 (m, 6H), 7.28 – 7.21 (m, 4H), 5.27 (d, J = 3.5 Hz, 1H), 4.73 (dd, J = 3.5, 8.3 Hz, 1H), 4.04 (d, J = 8.3 Hz, 1H), 2.56 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 137.2, 136.1, 135.0, 134.7, 134.5, 132.9, 129.92, 129.55, 129.50, 129.2, 129.1, 128.6, 91.7, 74.5, 73.2, 42.7; HRMS (ESI) calcd for $C_{22}H_{21}O_5NS_2Cl^+$ $[M + H]^+$ 478.0544, found 478.0541; IR (film) ν_{max}: 3065, 2965, 2924, 2878, 1907, 1729, 1599, 1585, 1493, 1448, 1415, 1324, 1179, 1152, 1086, 1062, 1016, 999, 954, 850, 822, 803, 729, 687, 633, 607, 588, 573, 559, 532 cm^{-1}.

*3-(2-Bromophenyl)-2-methyl-4,5-bis(phenylsulfonyl)isoxa-zolidine (**3r**)*

83% yield (86.7 mg); white solid; m.p. 162 – 164 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ7.97 – 7.94 (m, 2H), 7.87 – 7.84 (m, 2H), 7.75 – 7.64 (m, 2H), 7.61 – 7.53 (m, 3H), 7.45 – 7.40 (m, 3H), 7.28 – 7.23 (m, 1H), 7.11 – 7.05 (m, 1H), 5.36 (d, J = 3.6 Hz, 1H), 4.90 (dd, J = 3.6, 8.6 Hz, 1H), 4.82 (d, J = 8.6 Hz, 1H), 2.59 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.1, 136.1, 134.43, 134.41, 133.0, 132.8, 130.3, 130.0, 129.5, 129.3, 129.1, 128.5, 128.3, 125.3, 91.3, 74.4, 71.4, 42.3; HRMS (ESI) calcd for $C_{22}H_{21}O_5NS_2Br^+$ $[M + H]^+$ 522.0039, found 522.0042; IR (film) ν_{max}: 3065, 2965, 2924, 2854, 2256, 1905, 1815, 1585, 1570, 1477, 1448, 1435, 1403, 1325, 1280, 1180, 1153, 1107, 1085, 1062, 1024, 999, 948, 911, 864, 842, 788, 754, 687, 649, 626, 607, 590, 574, 559, 544 cm^{-1}.

*3-(3-Bromophenyl)-2-methyl-4,5-bis(phenylsulfonyl)isoxa-zolidine (**3s**)*

41% yield (42.8 mg); white solid; m.p. 141 – 143 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ7.96 – 7.92 (m, 2H), 7.84 – 7.80 (m, 2H), 7.75 – 7.49 (m, 6H), 7.40 – 7.33 (m, 2H), 7.19 – 7.11 (m, 2H), 5.31 (d, J = 3.5 Hz, 1H), 4.72 (dd, J = 3.5, 8.3 Hz, 1H), 3.94 (d, J = 8.3 Hz, 1H), 2.55 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 137.0, 136.5, 136.0, 134.8, 134.5, 132.2, 131.3, 130.4, 129.5, 129.5, 129.2, 128.5, 127.1, 122.5, 91.5, 74.5, 73.4, 58.4, 42.7; HRMS (ESI) calcd for $C_{22}H_{21}O_5NS_2Br^+$ $[M + H]^+$ 522.0039, found 522.0042; IR (film) ν_{max}: 3064, 2963, 2925, 1724, 1584, 1477, 1448, 1432, 1323, 1180, 1152, 1107, 1085, 1062, 1024, 999, 953, 881, 791, 753, 727, 687, 607, 593, 577, 552, 524 cm^{-1}.

*3-(4-Bromophenyl)-2-methyl-4,5-bis(phenylsulfonyl)isoxa-zolidine (**3t**)*

62% yield (64.8 mg); white solid; m.p. 174 – 176 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.92 – 7.89 (m, 2H), 7.83 – 7.80 (m, 2H), 7.74 – 7.64 (m, 2H), 7.61 – 7.50 (m, 4H), 7.41 – 7.37 (m, 2H), 7.21 – 7.18 (m, 2H), 5.26 (d, *J* = 3.5 Hz, 1H), 4.72 (dd, *J* = 3.5, 8.3 Hz, 1H), 4.03 (d, *J* = 8.3 Hz, 1H), 2.56 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.2, 136.0, 134.7, 134.5, 133.4, 132.0, 130.2, 129.6, 129.5, 129.2, 128.6, 123.2, 91.7, 74.4, 73.2, 42.7; HRMS (ESI) calcd for $C_{22}H_{21}O_5NS_2Br^+$ $[M + H]^+$ 522.0039, found 522.0034; IR (film) ν_{max}: 3065, 2966, 2877, 1585, 1489, 1448, 1411, 1324, 1179, 1153, 1108, 1085, 1070, 1012, 999, 954, 849, 819, 802, 754, 728, 687, 631, 607, 588, 572, 559, 523 cm^{-1}.

*3-(Biphenyl-4-yl)-2-methyl-4,5-bis(phenylsulfonyl)isoxazo-lidine (**3u**)*

75% yield (77.9 mg); white solid; m.p. 77 – 79 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.93 – 7.79 (m, 2H), 7.83 – 7.80 (m, 2H), 7.74 – 7.68 (m, 1H), 7.61 – 7.52 (m, 5H), 7.50 – 7.42 (m, 6H), 7.39 – 7.33 (m, 3H), 5.33 (d, *J* = 3.6 Hz, 1H), 4.84 (dd, *J* = 3.6, 8.4 Hz, 1H), 4.09 (d, *J* = 8.4 Hz, 1H), 2.59 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 141.9, 140.3, 137.4, 136.3, 134.5, 134.4, 133.1, 129.8, 129.6, 129.4, 129.1, 128.9, 128.8, 128.6, 128.4, 127.6, 127.5, 127.0, 91.7, 74.4, 73.8, 42.7; HRMS (ESI) calcd for $C_{28}H_{26}NO_5S_2^+$ $[M + H]^+$ 520.1247, found 520.1249; IR (film) ν_{max}: 3062, 2964, 2924, 2874, 1584, 1487, 1448, 1412, 1324, 1311, 1276, 1263, 1179, 1152, 1107, 1085, 1061, 1024, 1008, 999, 948, 854, 830, 800, 765, 752, 723, 687, 627, 606, 588, 559, 528 cm^{-1}.

*2-Methyl-3-(naphthalen-2-yl)-4,5-bis(phenylsulfonyl)isoxa-zolidine (**3v**)*

99% yield (97.7 mg); white solid; m.p. 143 – 145 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.97 (d, *J* = 7.5 Hz, 2H), 7.79 – 7.70 (m, 6H), 7.62 – 7.57 (m, 3H), 7.49 – 7.45 (m, 4H), 7.40 – 7.35 (m, 2H), 5.37 (d, *J* = 3.6 Hz, 1H), 4.90 (dd, *J* = 3.6, 8.3 Hz, 1H), 4.18 (d, *J* = 3.6 Hz, 1H), 2.59 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 137.3, 136.3, 134.5, 134.4, 133.5, 132.9, 131.4, 129.6, 129.3, 129.2, 128.9, 128.6, 128.5, 127.9, 127.7, 126.6, 126.3, 124.9, 91.7, 74.4, 74.2, 42.8; HRMS (ESI) calcd for $C_{26}H_{24}NO_5S_2^+$ $[M + H]^+$ 494.1090, found 494.1091; IR (film) ν_{max}: 3061, 3025, 2966, 2878, 1814, 1602, 1584, 1509, 1479, 1448, 1323, 1152, 1107, 1084, 1024, 999, 970, 898, 865, 822, 799, 752, 687, 651, 629, 607, 589, 572, 533, 480 cm^{-1}.

General Procedure for the Synthesis of 4

The product **3e** (47.1 mg, 0.1 mmol) and K_2CO_3 (13.8 mg, 0.1 mmol) was dissolved in 1 mL THF, the mixture was stirred at rt for 12 h. Once starting material was consumed (monitored by TLC), the mixture was concentrated to dryness. The residue was purified through flash column chromatography (EtOAc/PE) to afford the corresponding product **4** as a white semi-solid, 24.0 mg, 73% yield.

*3-(2,4-dimethylphenyl)-2-methyl-5-(phenylsulfonyl)-2,3-dihydroisoxazole (**4e**)*

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.56 – 7.33 (m, 4H), 7.23 – 7.11 (m, 2H), 6.93 – 6.57 (m, 3H), 5.20 (d, *J* = 1.6 Hz, 1H), 2.97 (s, 3H), 2.23 (s, 3H), 2.19 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 152.2, 140.5, 137.7, 135.8, 132.1, 131.4, 131.0, 128.4, 128.2, 126.9, 126.7, 117.3, 71.7, 47.4, 20.6, 18.7; HRMS (ESI) calcd for $C_{18}H_{20}NO_3S^+$ $[M+H]^+$ 330.1158, found 330.1154; IR (film) ν_{max} 2922, 1688, 1610, 1447, 1317, 1148, 807, 757, 723, 688, 615, 557 cm^{-1}.

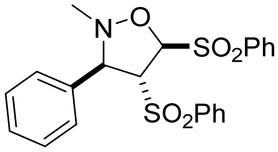
General Procedure for the Synthesis of 5

The product **3e** (47.1 mg, 0.1 mmol) and *m*CPBA (20.3 mg, 0.1 mmol) was dissolved in 2 mL DCM, the mixture was stirred at rt for 12 h. Once starting material was consumed (monitored by TLC), the mixture was concentrated to dryness. The residue was purified through flash column chromatography (EtOAc/PE) to afford the corresponding product **5** as a white semi-solid, 20.1 mg, yield 67%.

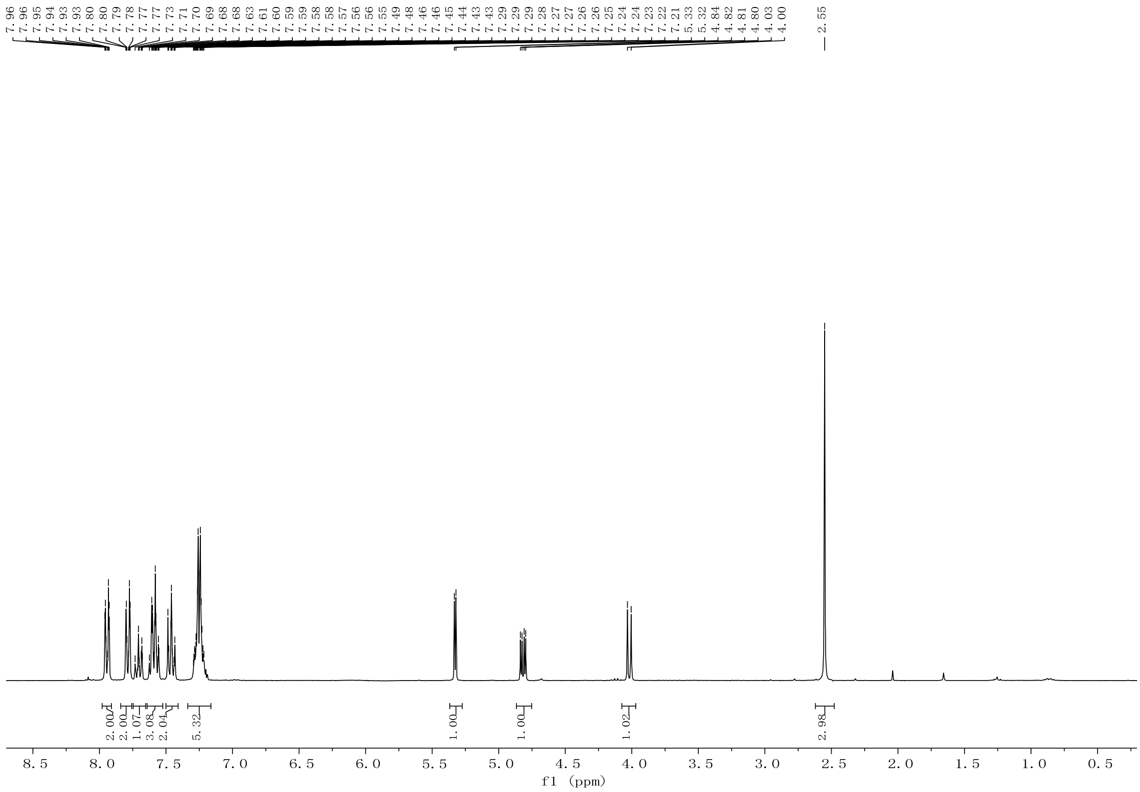
*(E)-3-(2,4-dimethylphenyl)-2-(phenylsulfonyl)acrylaldehyde (**5**)*

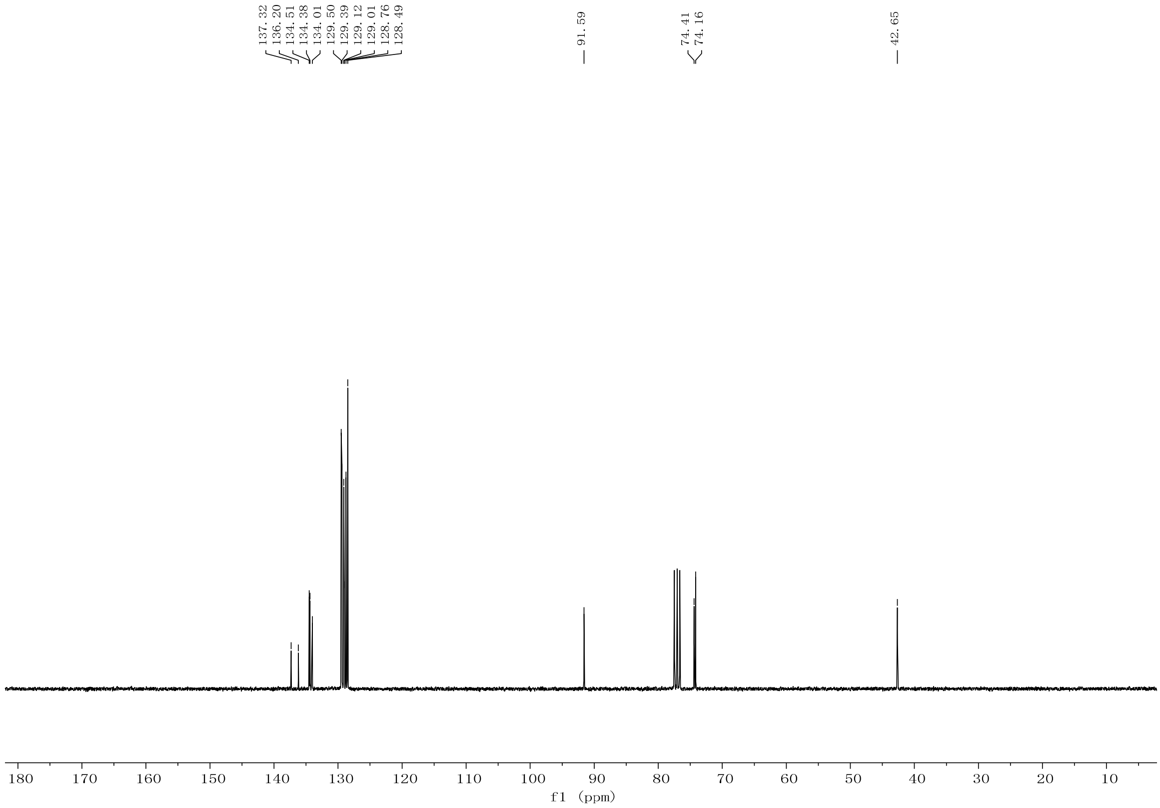
^{1}H NMR (300 MHz, $CDCl_3$) δ 9.64 (s, 1H), 8.98 (s, 1H), 8.21 – 7.88 (m, 2H), 7.74 – 7.45 (m, 3H), 7.21 – 6.98 (m, 3H), 2.45 (s, 3H), 2.38 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 184.8, 153.0, 143.1, 139.8, 139.4, 138.7, 133.4, 131.7, 131.5, 128.7, 128.6, 126.8, 126.7, 21.2, 19.8; HRMS (ESI) calcd for $C_{17}H_{16}O_3SNa^+$ $[M+Na]^+$ 323.0712, found 323.0710; IR (film) ν_{max} 2922, 1684, 1587, 1447, 1318, 1248, 1154, 1101, 825, 749, 717, 687, 649, 589, 536 cm^{-1}.

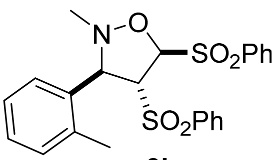
^{1}H and ^{13}C NMR Spectra of All Cycloadducts



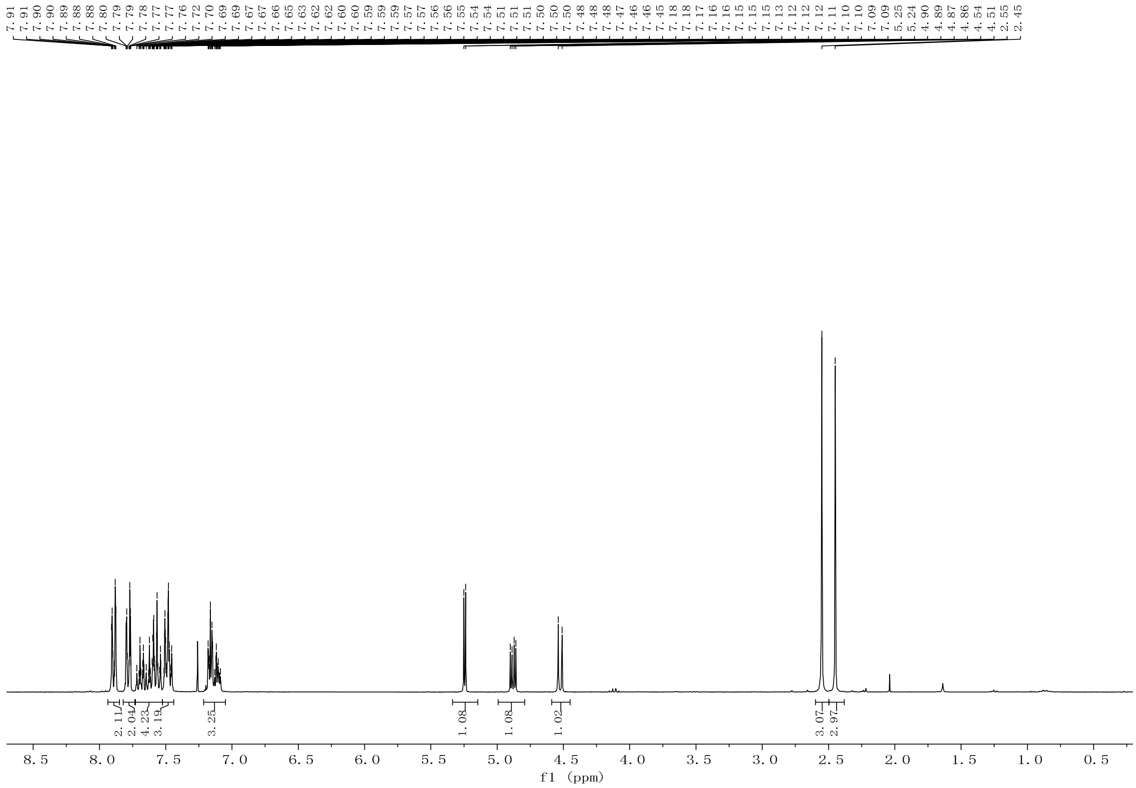
3a

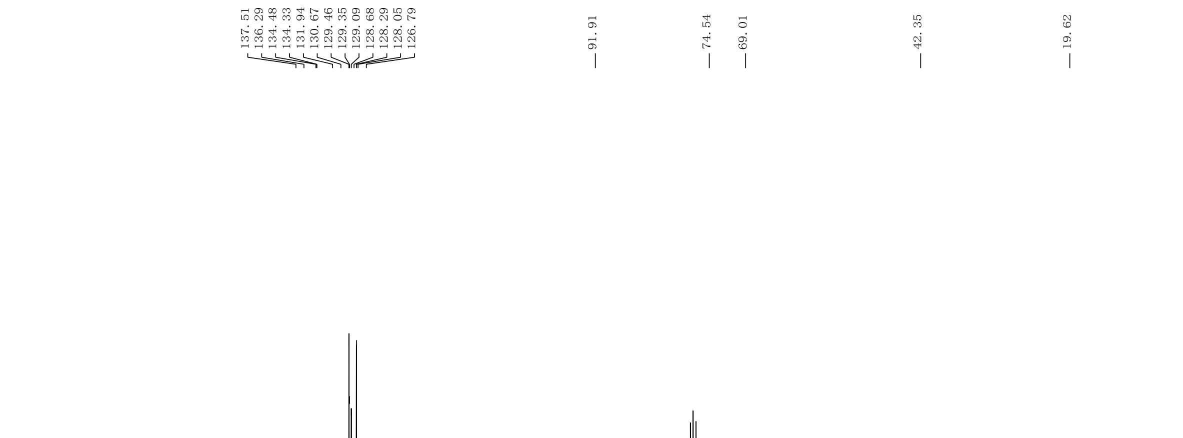


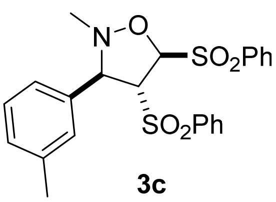



N
O
SO2Ph
SO2Ph

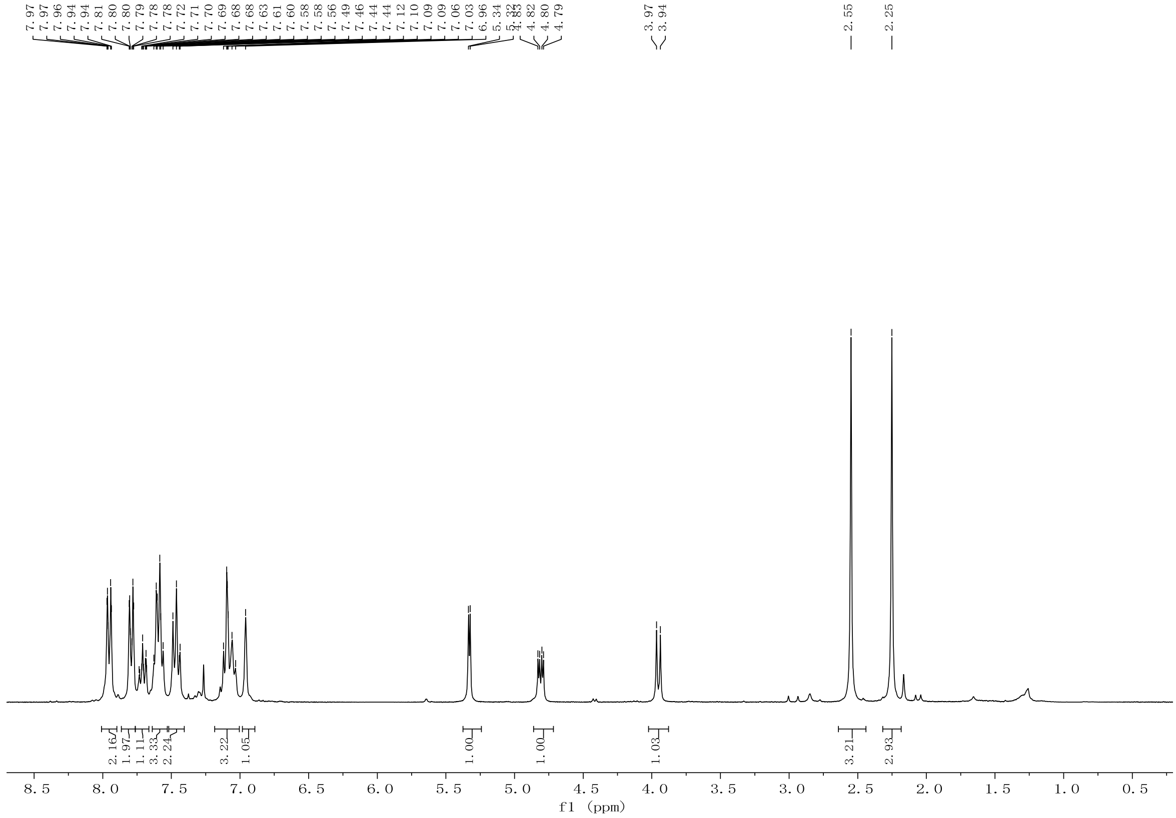
3b

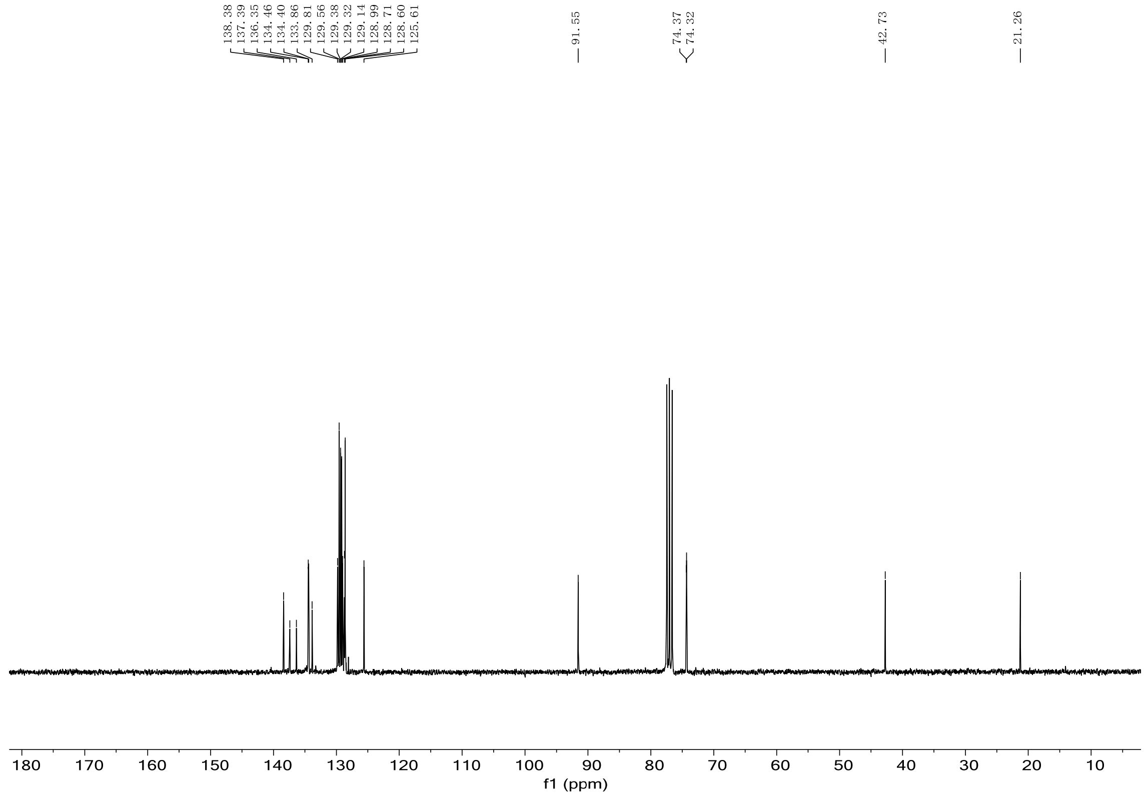

7.91 7.91 7.90 7.89 7.88 7.80 7.79 7.79 7.78 7.77 7.76 7.72 7.70 7.69 7.69 7.67 7.67 7.66 7.65 7.63 7.62 7.62 7.60 7.60 7.59 7.59 7.59 7.57 7.57 7.56 7.56 7.55 7.54 7.54 7.51 7.51 7.51 7.50 7.50 7.48 7.48 7.47 7.46 7.46 7.45 7.18 7.18 7.17 7.16 7.16 7.15 7.15 7.15 7.13 7.12 7.12 7.12 7.11 7.10 7.10 7.09 5.25 5.24 4.90 4.89 4.87 4.86 4.54 4.51 2.55 2.45
2.11 2.04 4.23 3.19 3.25 1.08 1.08 1.02 3.07 2.97
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
f1 (ppm)

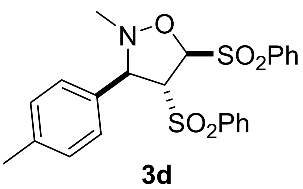

137.51 136.29 134.48 134.33 131.94 130.67 129.46 129.35 129.09 128.68 128.29 128.05 126.79
91.91
74.54
69.01
42.35
19.62
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


N
O
SO2Ph
SO2Ph

3c

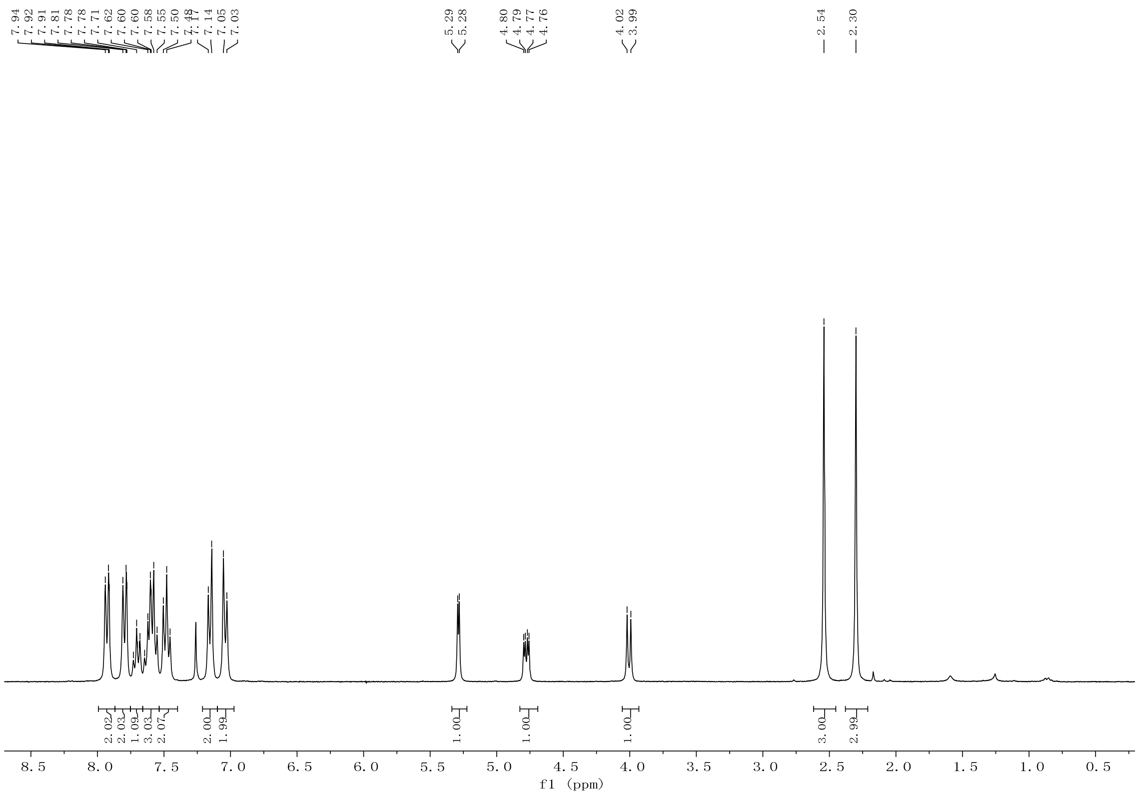

7.97 7.97 7.96 7.94 7.94 7.81 7.80 7.80 7.79 7.78 7.78 7.72 7.71 7.70 7.69 7.68 7.68 7.63 7.61 7.60 7.58 7.58 7.56 7.49 7.46 7.44 7.44 7.12 7.10 7.09 7.09 7.06 7.03 6.96 5.34 5.32 4.83 4.82 4.80 4.79
3.97 3.94
2.55
2.25
2.16 1.97 1.11 3.33 2.24 3.22 1.05 1.00 1.00 1.03 3.21 2.93
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


138.38 137.39 136.35 134.46 134.40 133.86 129.81 129.56 129.38 129.32 129.14 128.99 128.71 128.60 125.61
91.55
74.37 74.32
42.73
21.26
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

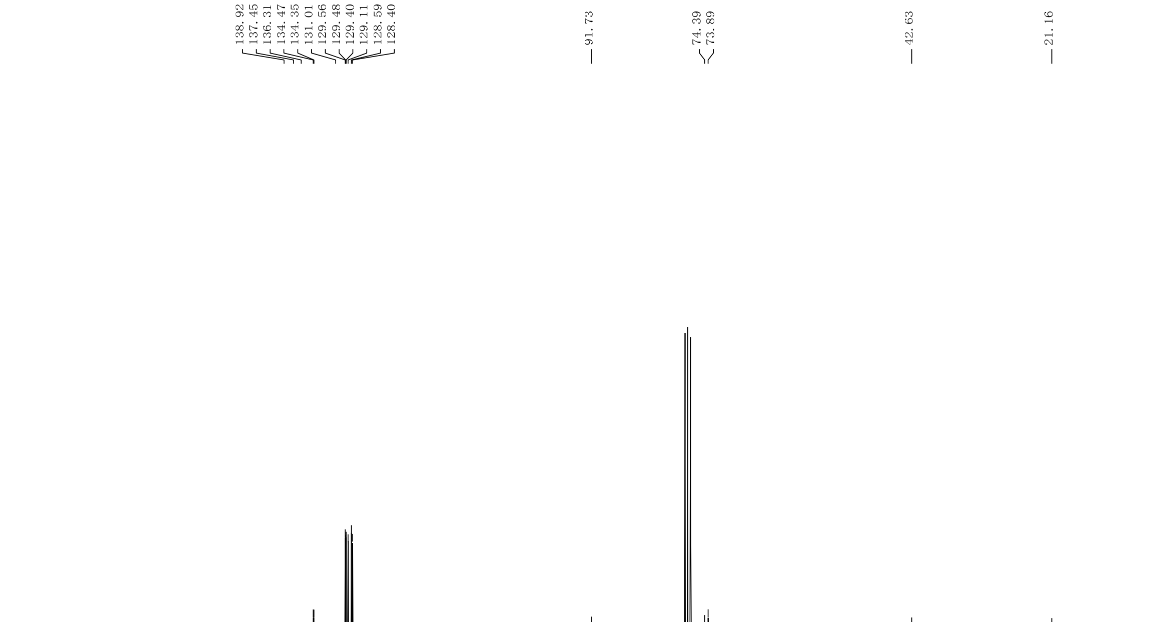


N
O
SO_2Ph
SO_2Ph

3d



7.94 7.92 7.91 7.81 7.78 7.78 7.71 7.62 7.60 7.60 7.58 7.55 7.50 7.18 7.17 7.14 7.05 7.03
5.29 5.28
4.80 4.79 4.77 4.76
4.02 3.99
2.54
2.30
2.02 2.03 1.09 3.03 2.07 2.00 1.99
1.00 1.00 1.00 3.00 2.99
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
f1 (ppm)



138.92 137.45 136.31 134.47 134.35 131.01 129.56 129.48 129.40 129.11 128.59 128.40
91.73
74.39 73.89
42.63
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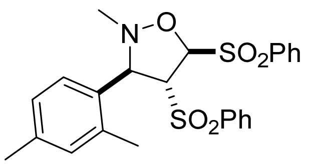
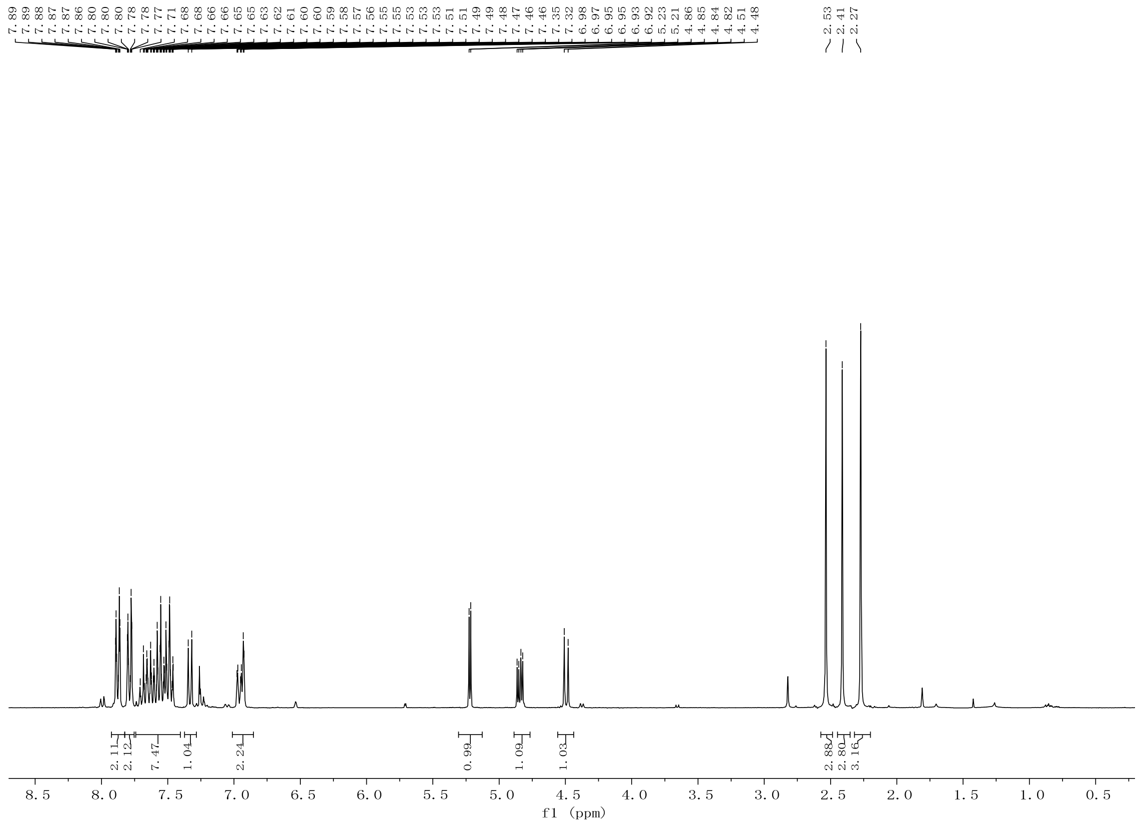
f1 (ppm)

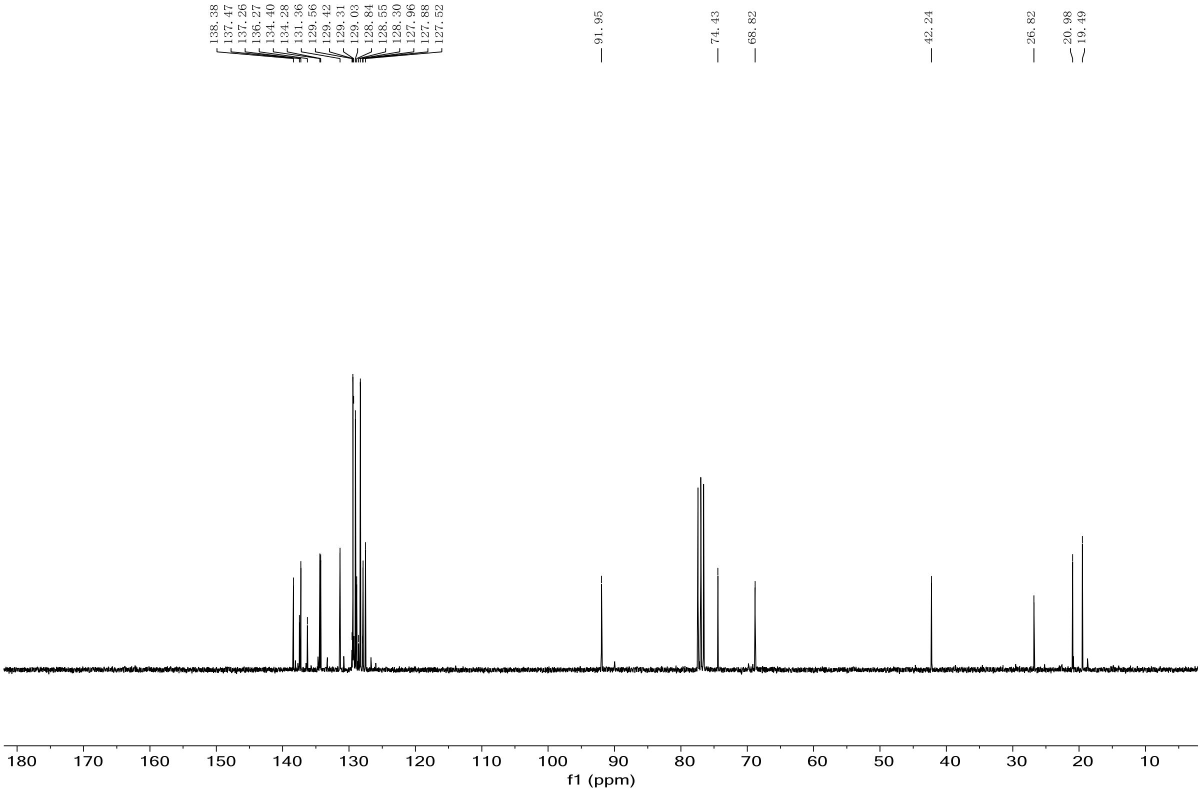


N
O
SO2Ph
SO2Ph

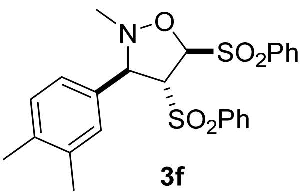
3e



7.89 7.89 7.88 7.87 7.87 7.86 7.80 7.80 7.80 7.78 7.78 7.77 7.71 7.68 7.66 7.66 7.65 7.65 7.63 7.62 7.61 7.60 7.60 7.59 7.58 7.57 7.56 7.55 7.55 7.53 7.53 7.53 7.51 7.51 7.49 7.49 7.48 7.47 7.46 7.35 7.32 6.98 6.97 6.95 6.95 6.93 6.92 5.23 5.21 4.86 4.85 4.84 4.82 4.51 4.48
2.53 2.41 2.27
2.11 2.12 7.47 1.04 2.24 0.99 1.09 1.03 2.88 2.80 3.16
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
f1 (ppm)

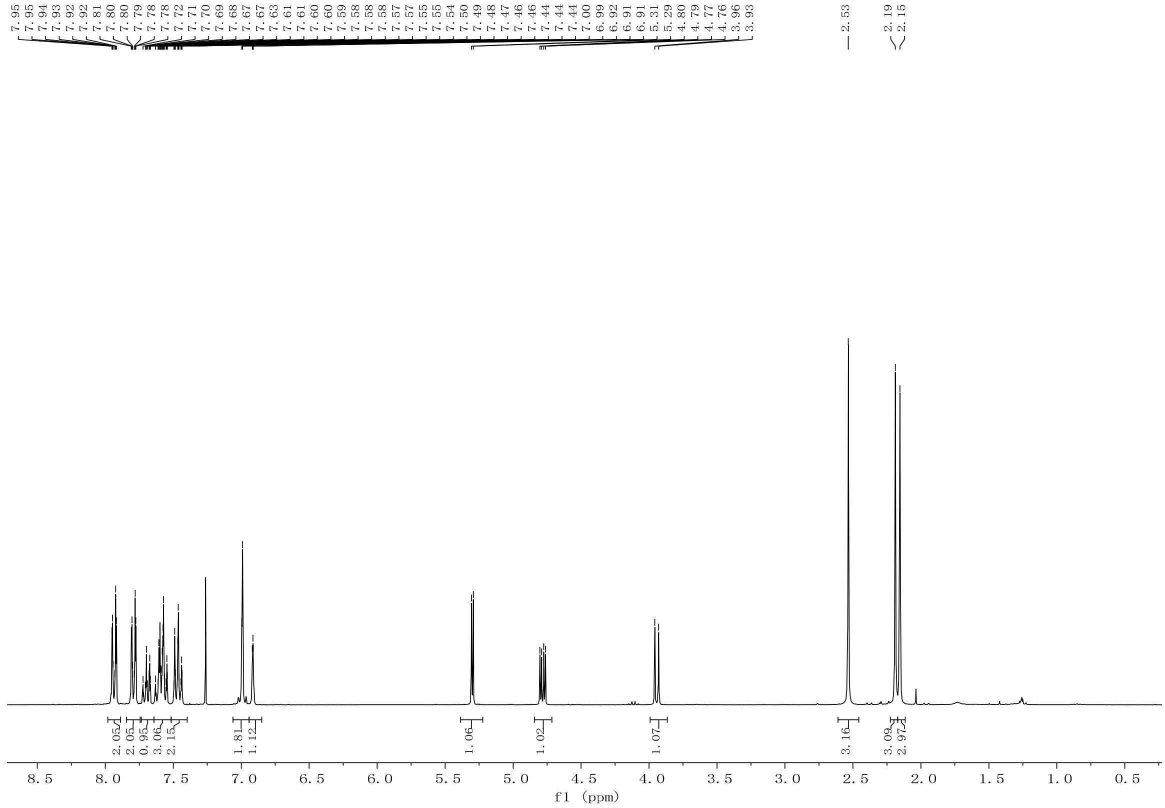


138.38 137.47 137.26 136.27 134.40 134.28 131.36 129.56 129.42 129.31 129.03 128.84 128.55 128.30 127.96 127.88 127.52
91.95
74.43 68.82
42.24
26.82 20.98 19.49
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)



N
O
SO2Ph
SO2Ph

3f



7.95
7.95
7.94
7.93
7.92
7.92
7.81
7.80
7.80
7.79
7.78
7.78
7.72
7.71
7.70
7.69
7.68
7.67
7.67
7.63
7.61
7.61
7.60
7.60
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7.57
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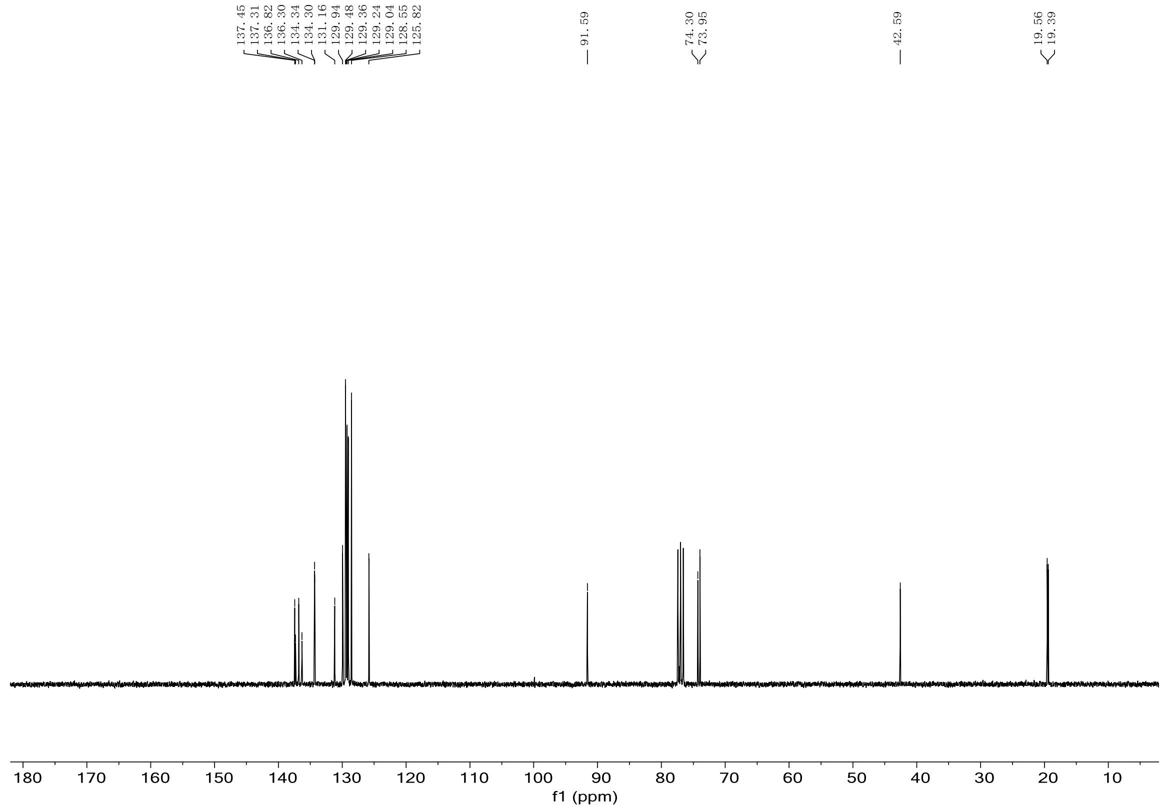
7.55
7.55
7.54
7.50
7.49
7.48
7.47
7.46
7.46
7.44
7.44
7.44
7.00
6.99
6.92
6.91
6.91
5.31
5.29
4.80
4.79
4.77
4.76
3.96
3.93
2.53
2.19
2.15
2.05
2.05
0.95
3.06
2.15
1.81
1.12
1.06
1.02
1.07
3.16
3.09
2.97
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)



137.45
137.31
136.82
136.30
134.34
134.30
131.16
129.94
129.48
129.36
129.24
129.04
128.55
125.82
91.59
74.30
73.95
42.59
19.56
19.39
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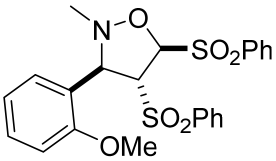
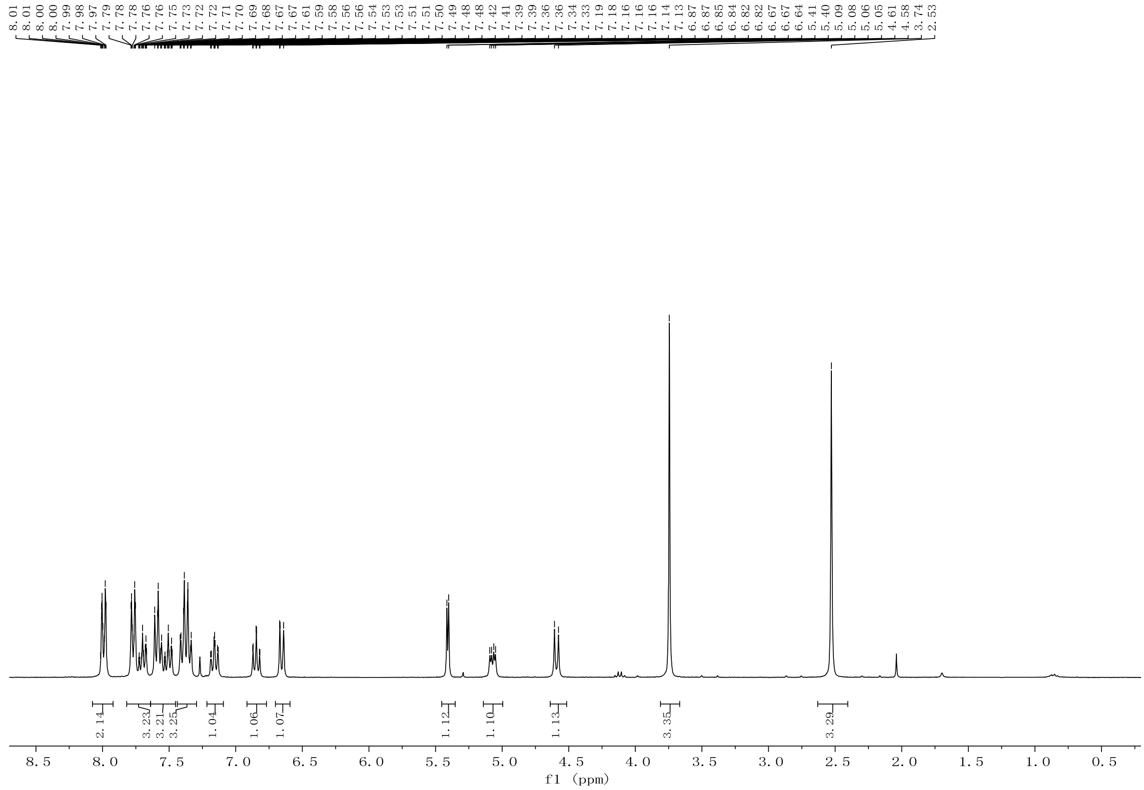
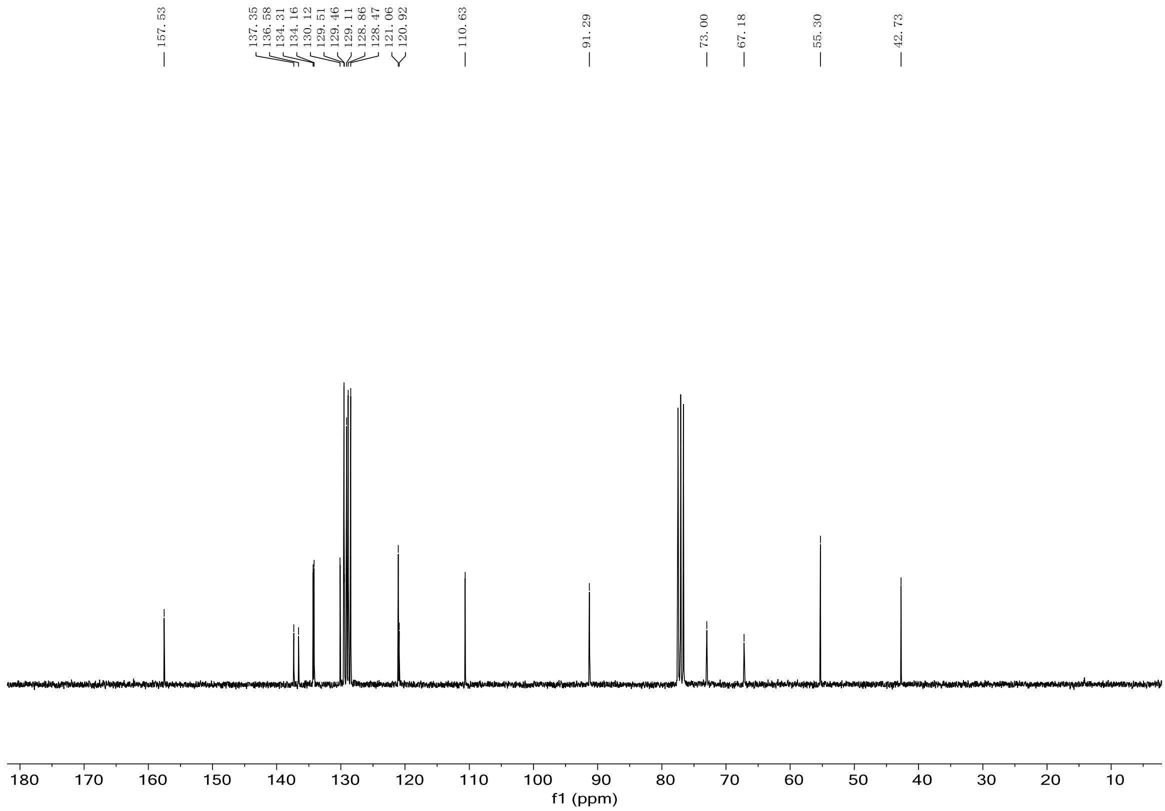
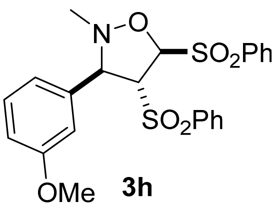
30
20
10
f1 (ppm)



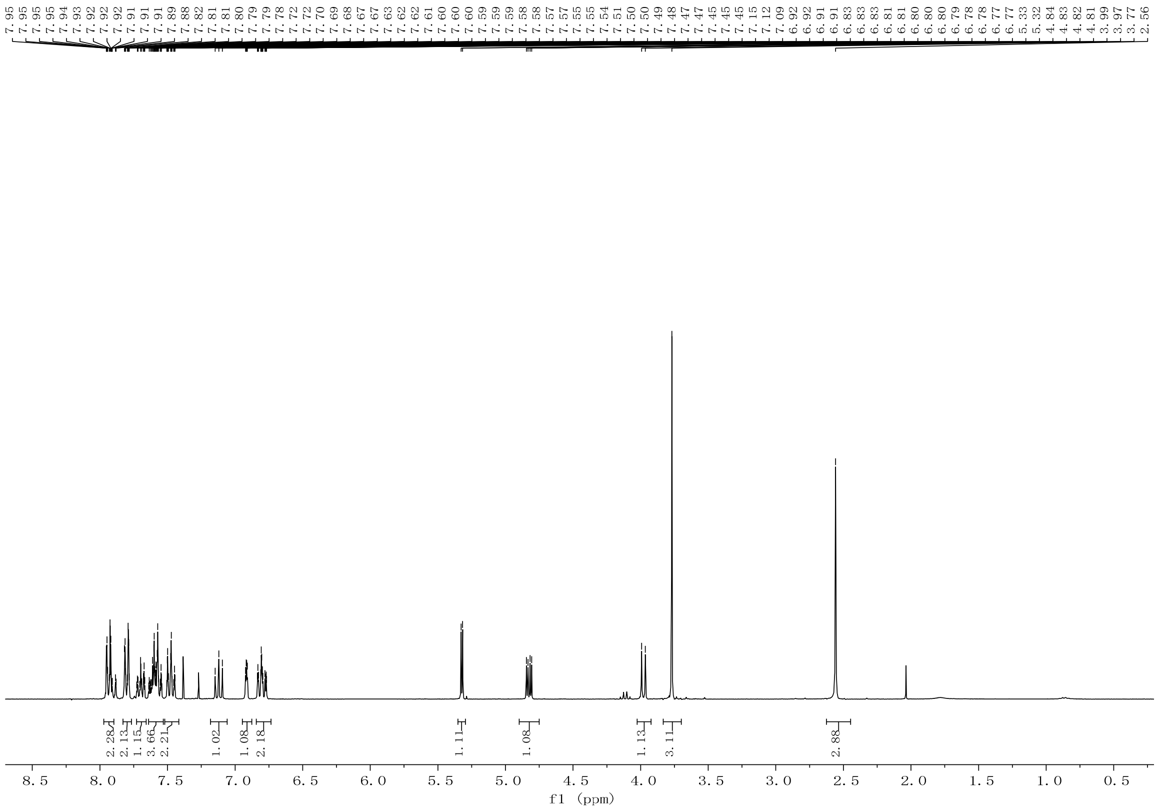
3g

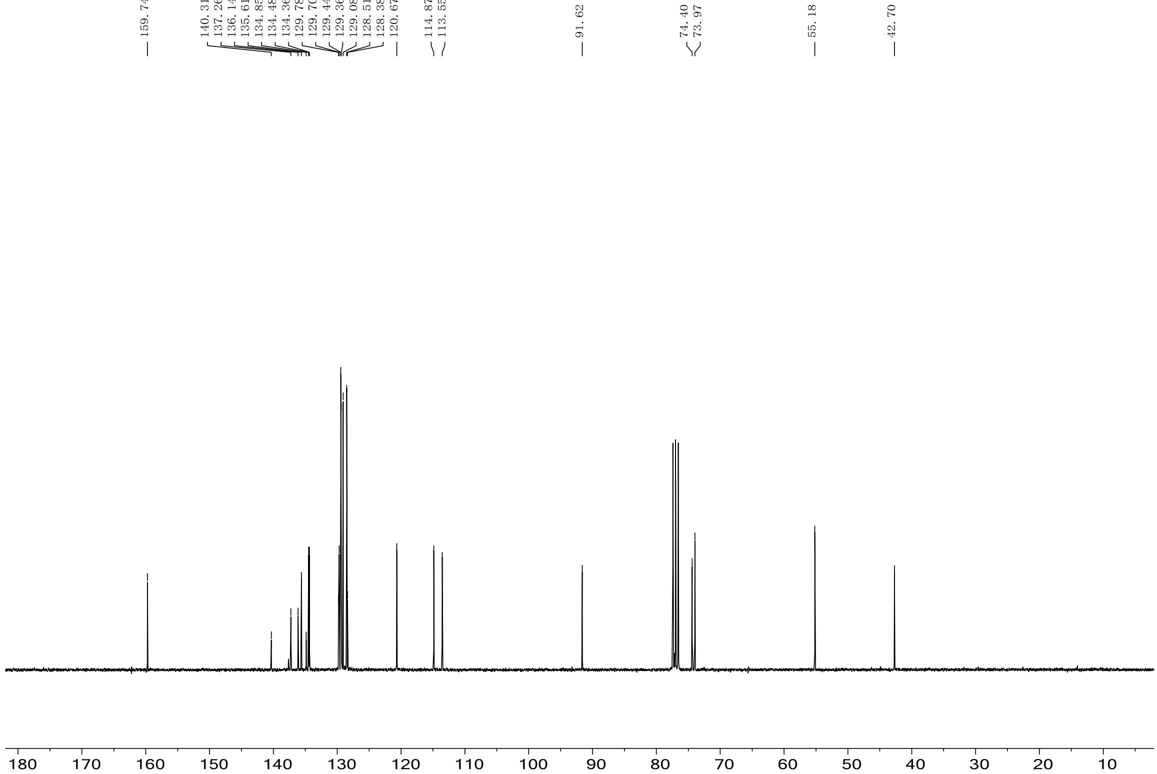


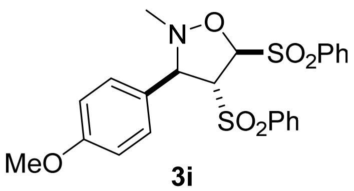



N
O
SO2Ph
SO2Ph
OMe

3h

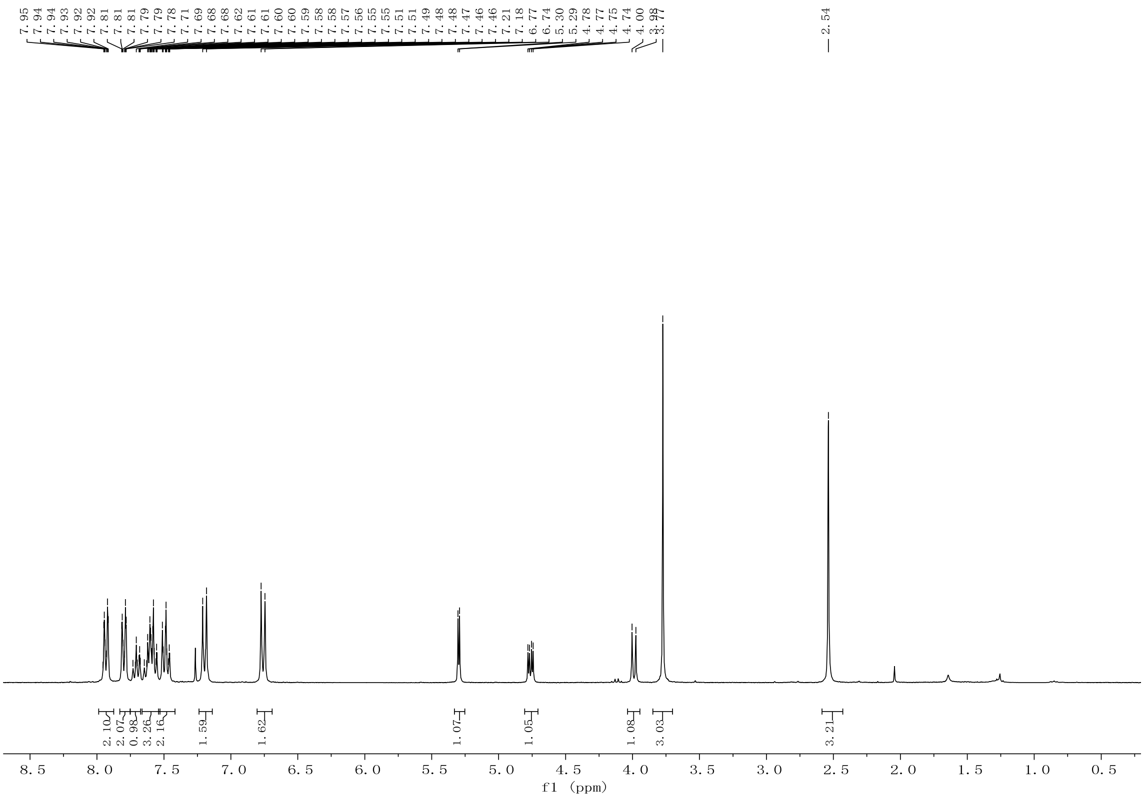

7.95 7.95 7.95 7.94 7.93 7.92 7.92 7.92 7.91 7.91 7.91 7.89 7.88 7.82 7.81 7.81 7.80 7.79 7.79 7.78 7.72 7.72 7.70 7.69 7.68 7.67 7.67 7.63 7.62 7.62 7.61 7.60 7.60 7.60 7.59 7.59 7.59 7.58 7.58 7.57 7.57 7.55 7.55 7.54 7.51 7.50 7.49 7.48 7.47 7.47 7.45 7.45 7.45 7.15 7.12 7.09 6.92 6.92 6.91 6.91 6.83 6.83 6.83 6.81 6.81 6.80 6.80 6.80 6.79 6.78 6.78 6.77 6.77 5.33 5.32 4.84 4.83 4.82 4.81 3.99 3.97 3.77 2.56
2.28
2.13
1.15
3.66
2.21
1.02
1.08
2.18
1.11
1.08
1.13
3.11
2.88
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
f1 (ppm)


159.74
140.31 137.26 136.14 135.61 134.85 134.48 134.36 129.78 129.70 129.44 129.36 129.08 128.51 128.38 120.67
114.87 113.55
91.62
74.40 73.97
55.18
42.70
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

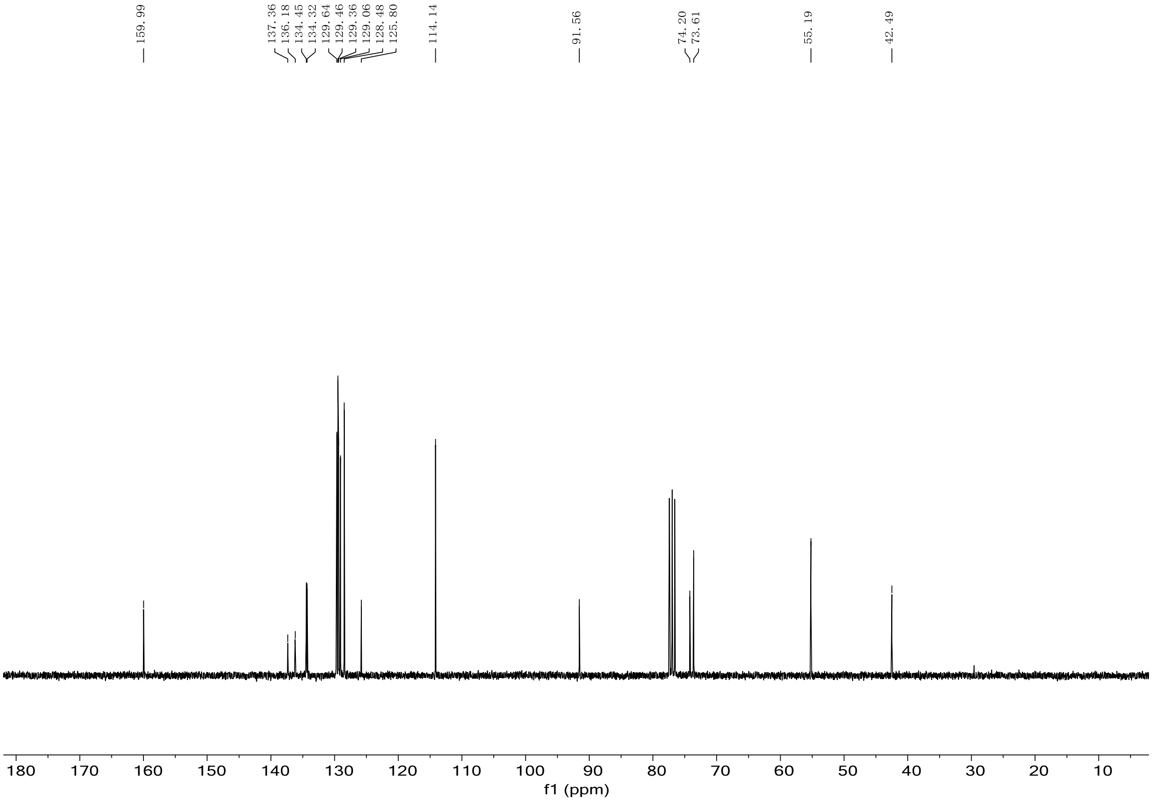


N
O
SO2Ph
SO2Ph
MeO

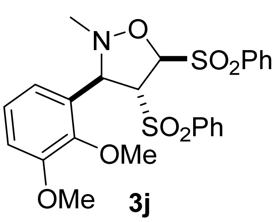
3i



7.95 7.94 7.94 7.93 7.92 7.92 7.81 7.81 7.81 7.79 7.79 7.78 7.71 7.69 7.68 7.62 7.61 7.61 7.60 7.60 7.59 7.58 7.58 7.57 7.56 7.55 7.55 7.51 7.51 7.49 7.48 7.48 7.47 7.46 7.46 7.21 7.18 6.77 6.74 5.30 5.29 4.78 4.77 4.75 4.74 4.00 3.98 3.77
2.54
2.10 2.07 0.98 3.26 2.16 1.59 1.62 1.07 1.05 1.08 3.03 3.21
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
f1 (ppm)

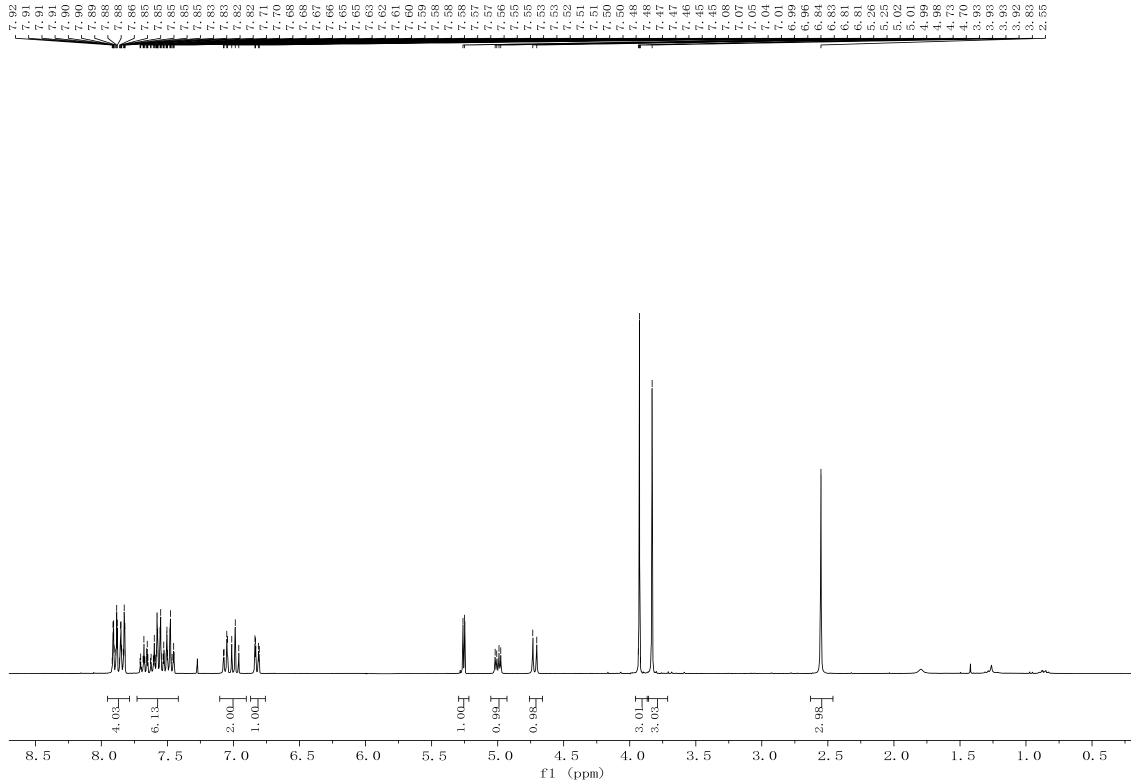


159.99
137.36 136.18 134.45 134.32 129.64 129.46 129.36 129.06 128.48 125.80
114.14
91.56
74.20 73.61
55.19
42.49
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

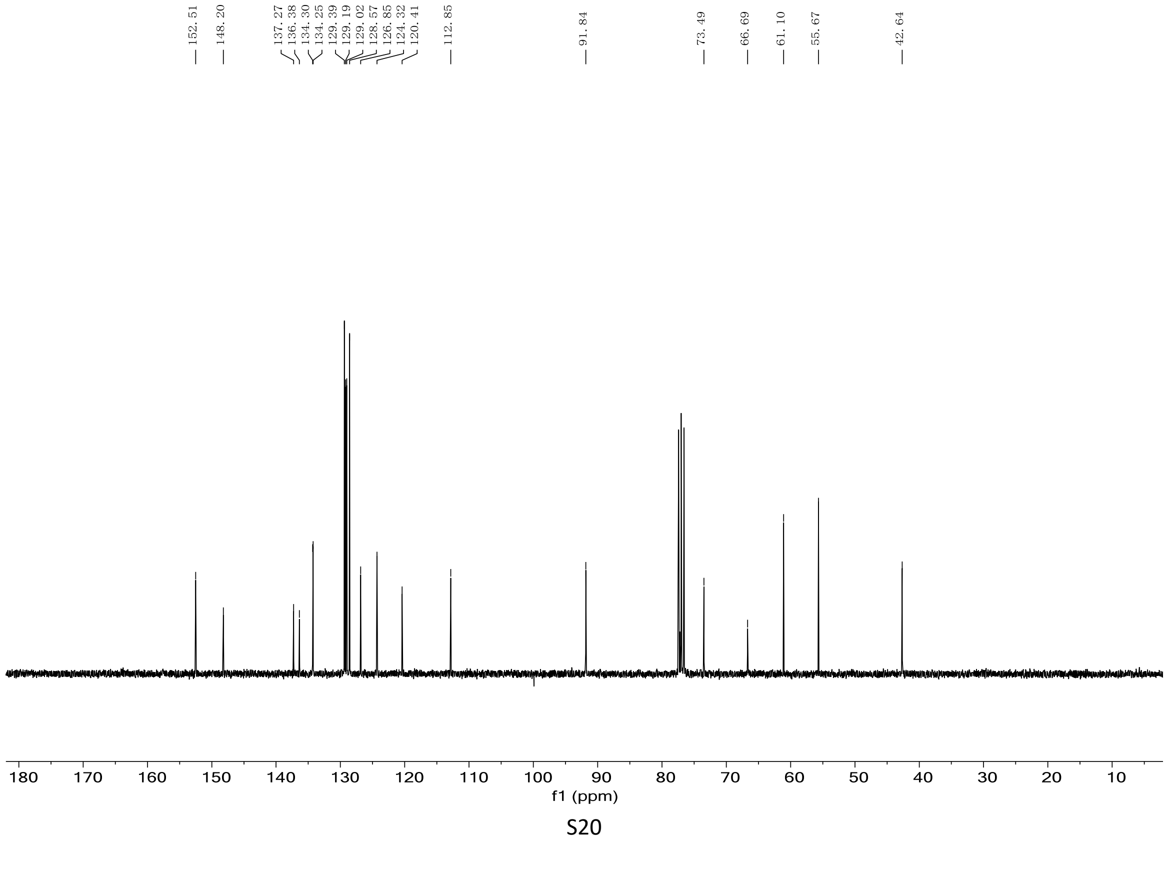


N
O
SO2Ph
SO2Ph
OMe
OMe

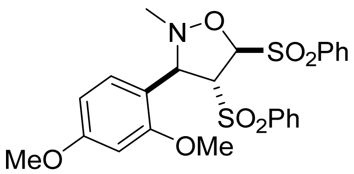
3j



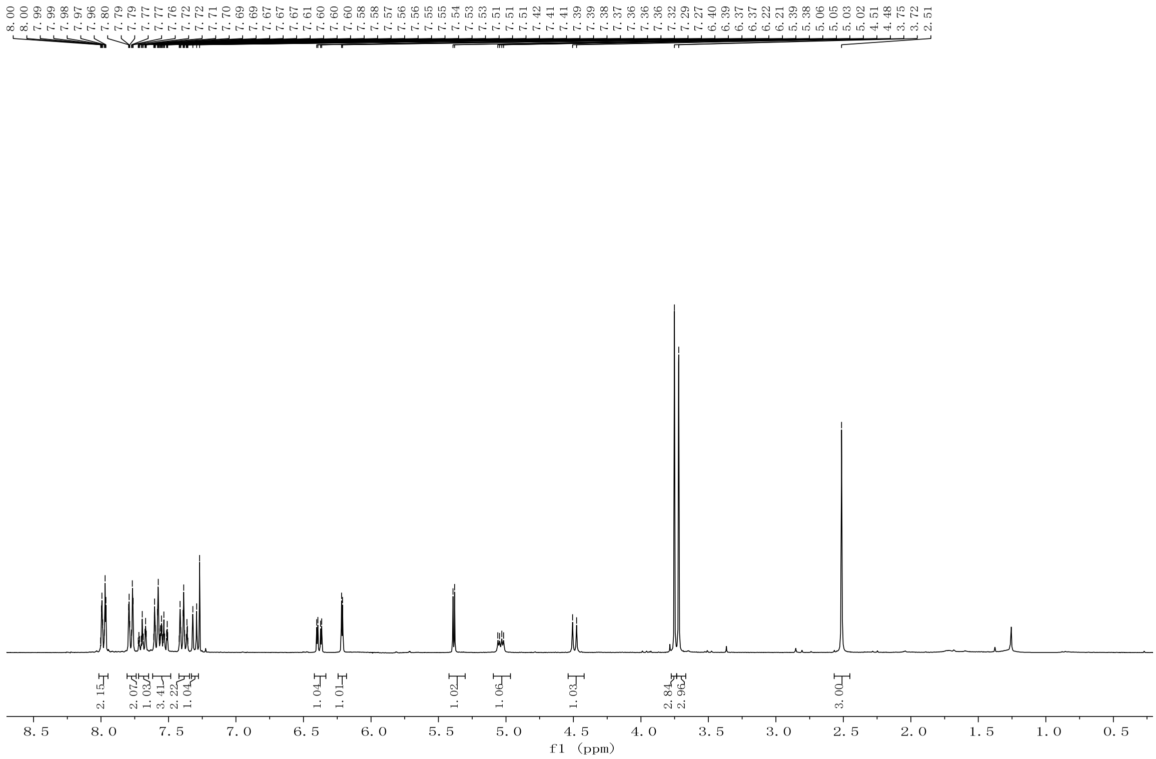
7.92 7.91 7.91 7.91 7.90 7.90 7.89 7.88 7.88 7.86 7.85 7.85 7.85 7.85 7.85 7.83 7.83 7.82 7.82 7.71 7.70 7.68 7.68 7.67 7.66 7.65 7.65 7.63 7.62 7.61 7.60 7.59 7.58 7.58 7.58 7.57 7.57 7.56 7.55 7.55 7.53 7.53 7.52 7.51 7.51 7.50 7.50 7.48 7.48 7.47 7.47 7.46 7.45 7.45 7.08 7.07 7.05 7.04 7.01 6.99 6.96 6.84 6.83 6.81 6.81 5.26 5.25 5.02 5.01 4.99 4.98 4.73 4.70 3.93 3.93 3.92 3.83 2.55
4.03
6.13
2.00
1.00
1.00
0.99
0.98
3.01
3.03
2.98
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
f1 (ppm)

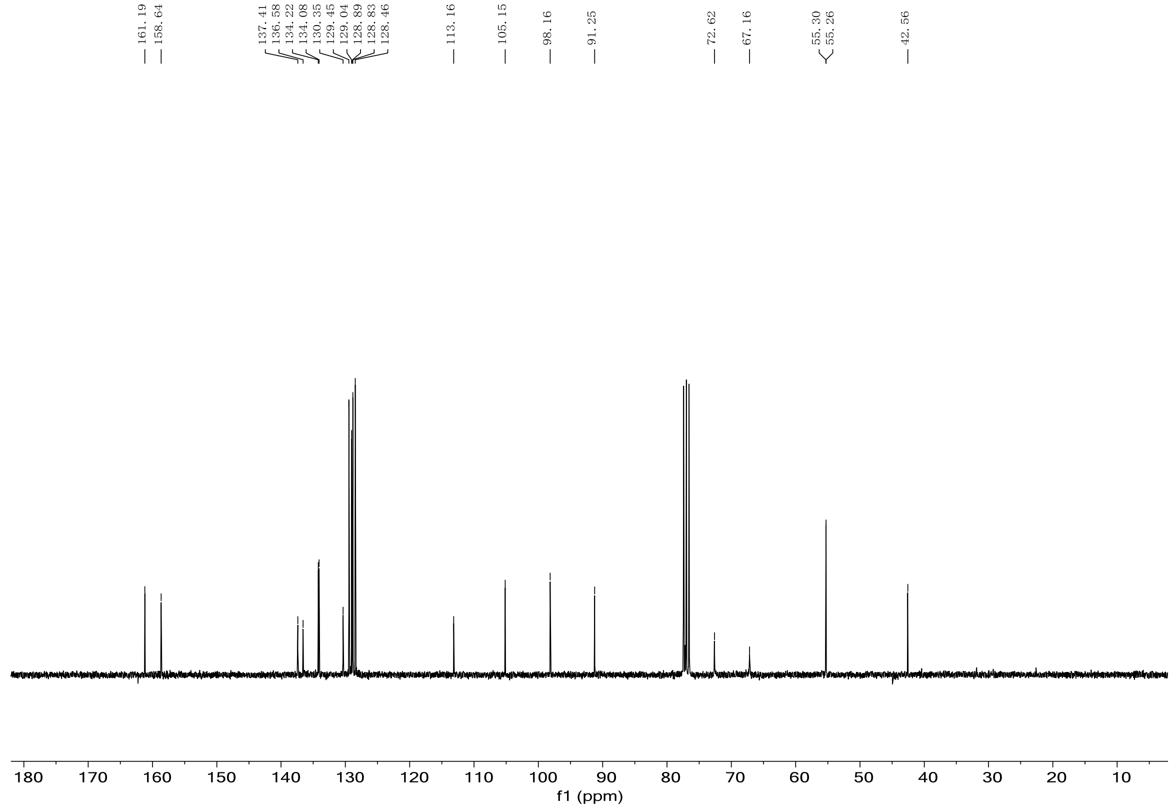


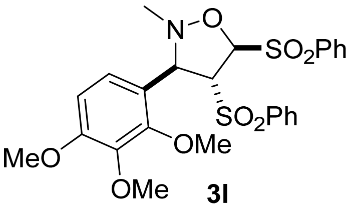
152.51
148.20
137.27 136.38 134.30 134.25 129.39 129.19 129.02 128.57 126.85 124.32 120.41
112.85
91.84
73.49
66.69
61.10
55.67
42.64
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

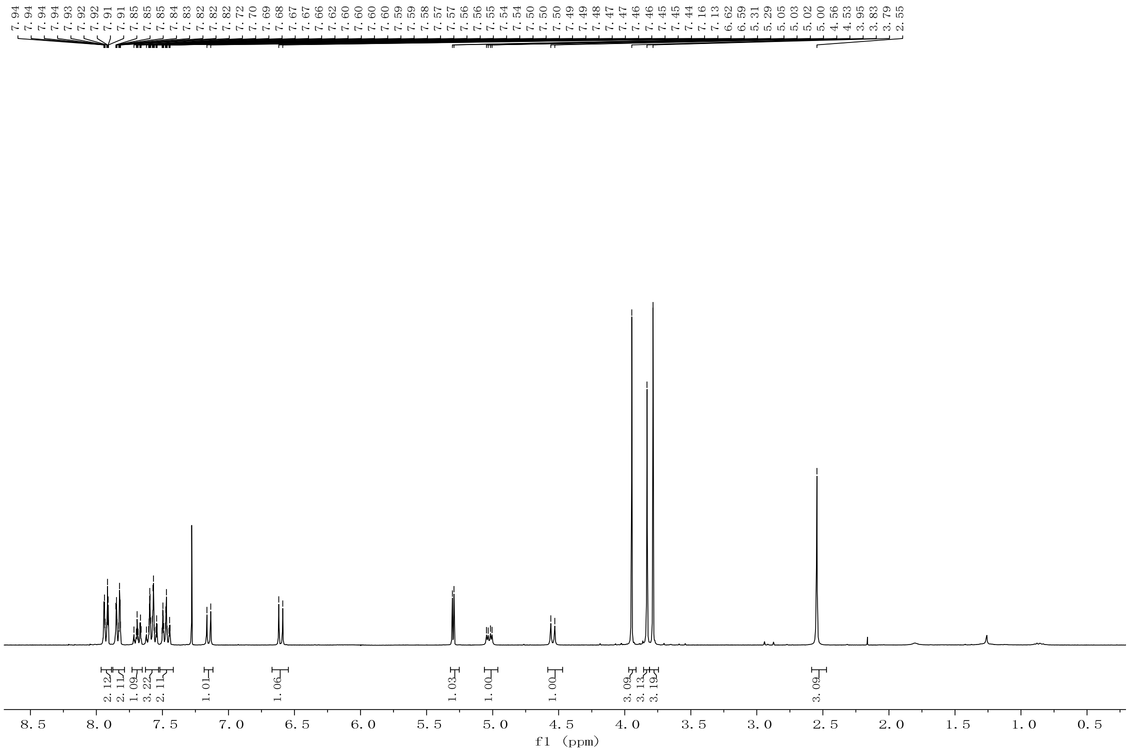


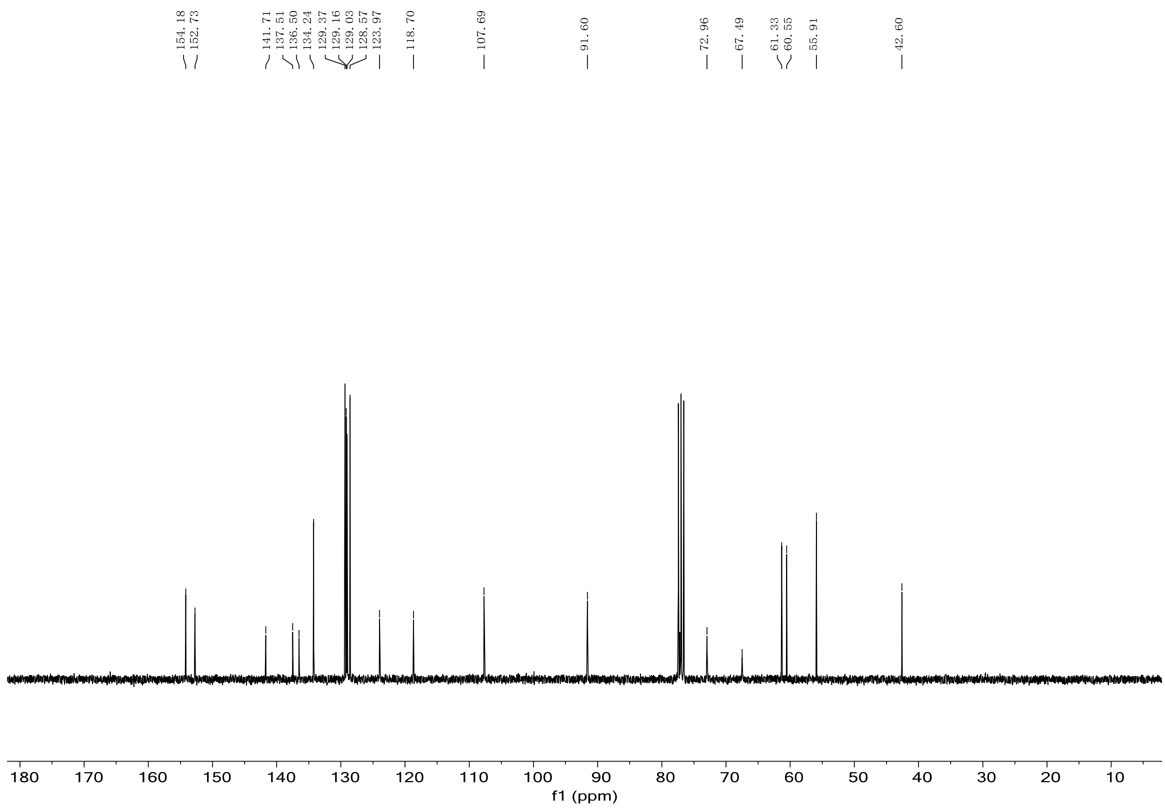
3k

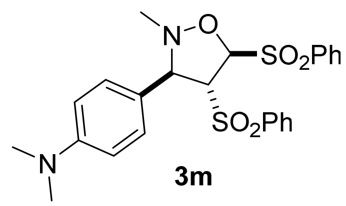




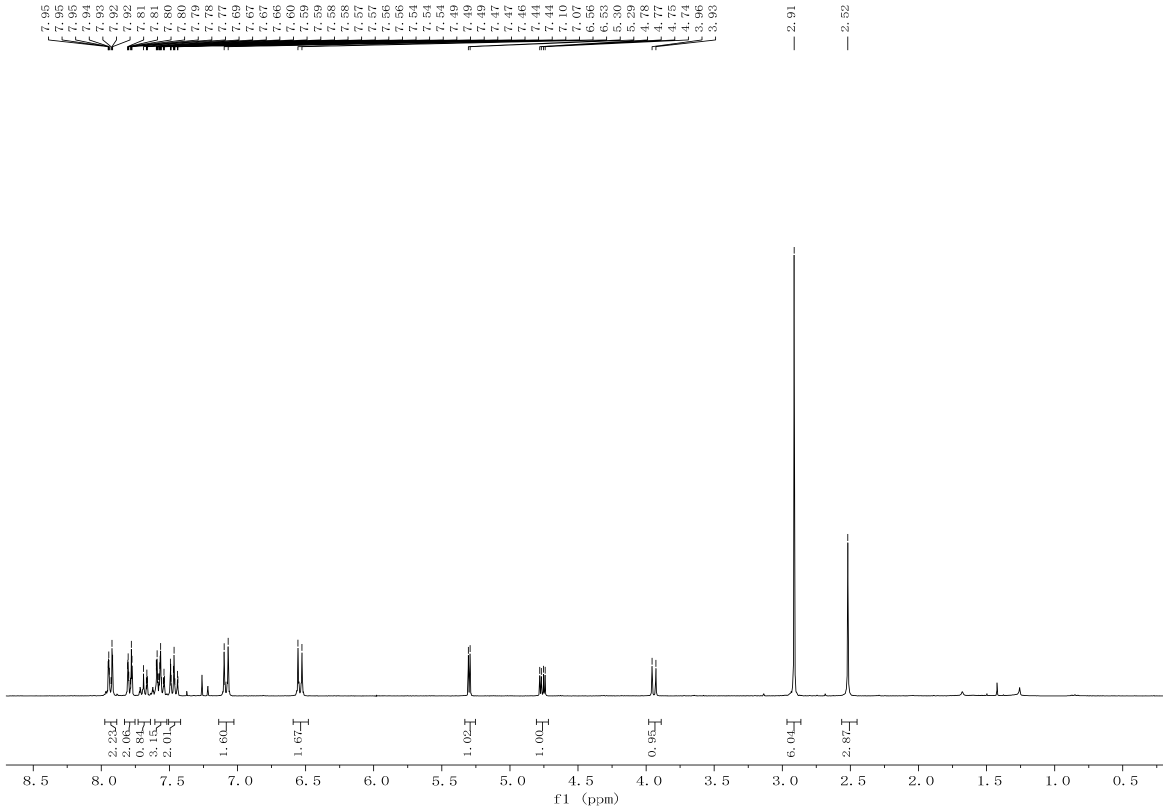

N
O
SO2Ph
SO2Ph
MeO
OMe
OMe
3l

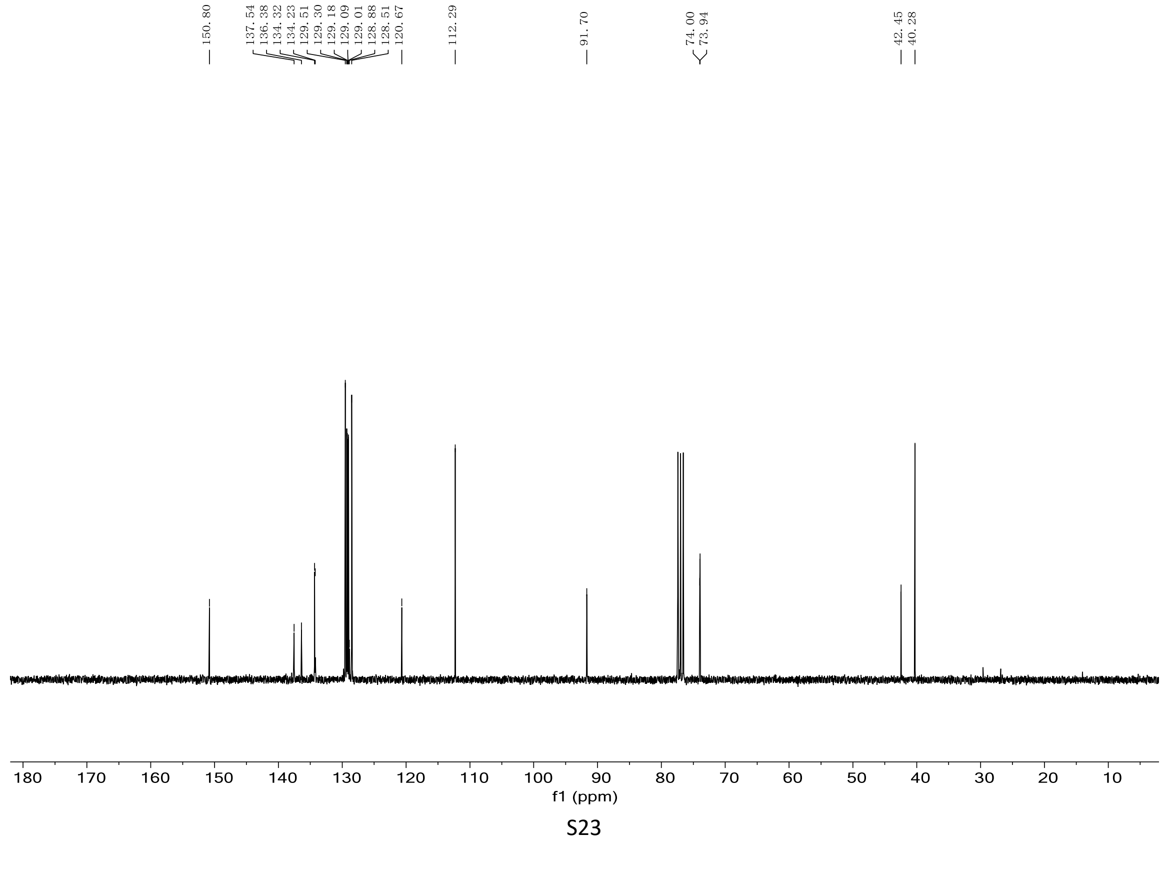

7.94 7.94 7.94 7.93 7.92 7.92 7.91 7.85 7.85 7.85 7.84 7.83 7.82 7.82 7.72 7.70 7.69 7.68 7.67 7.67 7.66 7.62 7.60 7.60 7.60 7.59 7.59 7.58 7.57 7.57 7.56 7.56 7.55 7.54 7.54 7.50 7.50 7.50 7.49 7.49 7.48 7.47 7.47 7.46 7.46 7.45 7.45 7.44 7.16 7.13 6.62 6.59 5.31 5.29 5.05 5.03 5.02 5.00 4.56 4.53 3.95 3.83 3.79 2.55
2.12 2.11 1.09 3.22 2.11 1.01 1.06 1.03 1.00 1.00 3.09 3.13 3.19 3.09
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
f1 (ppm)

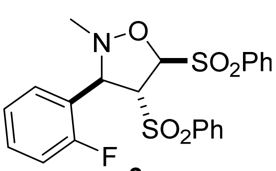

154.18 152.73 141.71 137.51 136.50 134.24 129.37 129.16 129.03 128.57 123.97 118.70 107.69 91.60 72.96 67.49 61.33 60.55 55.91 42.60
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


SO2Ph
SO2Ph
N
O

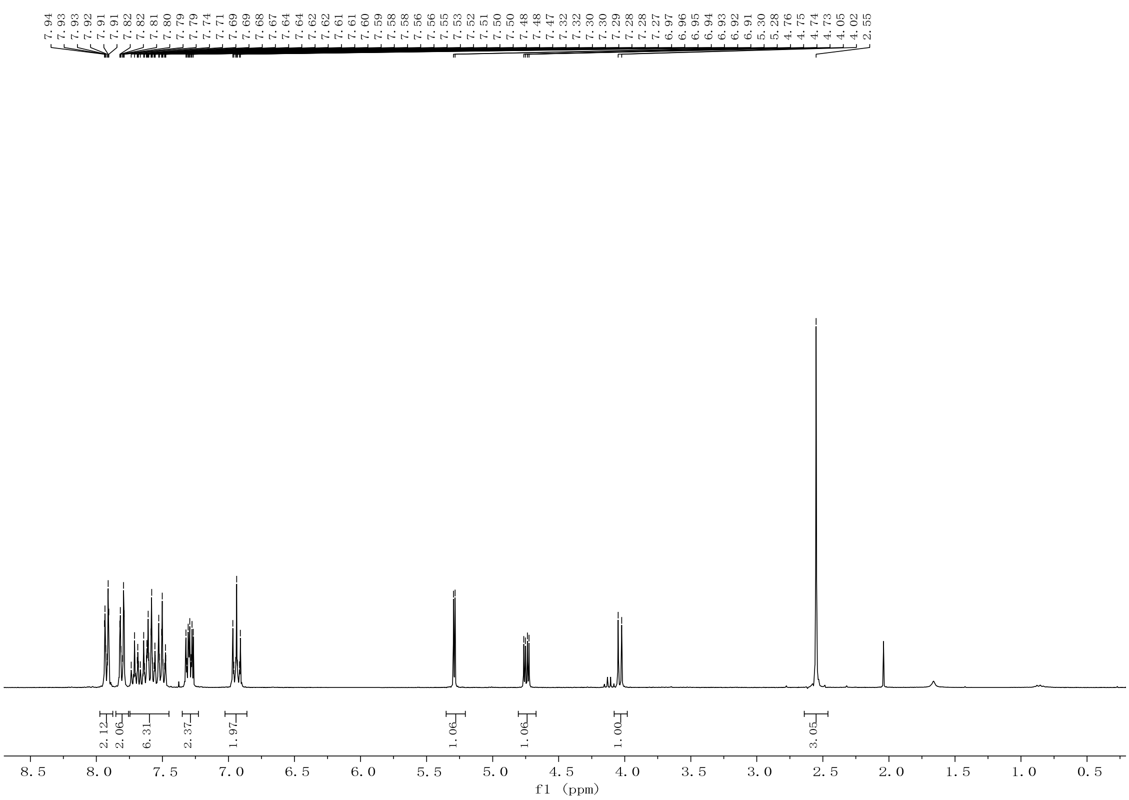
3m

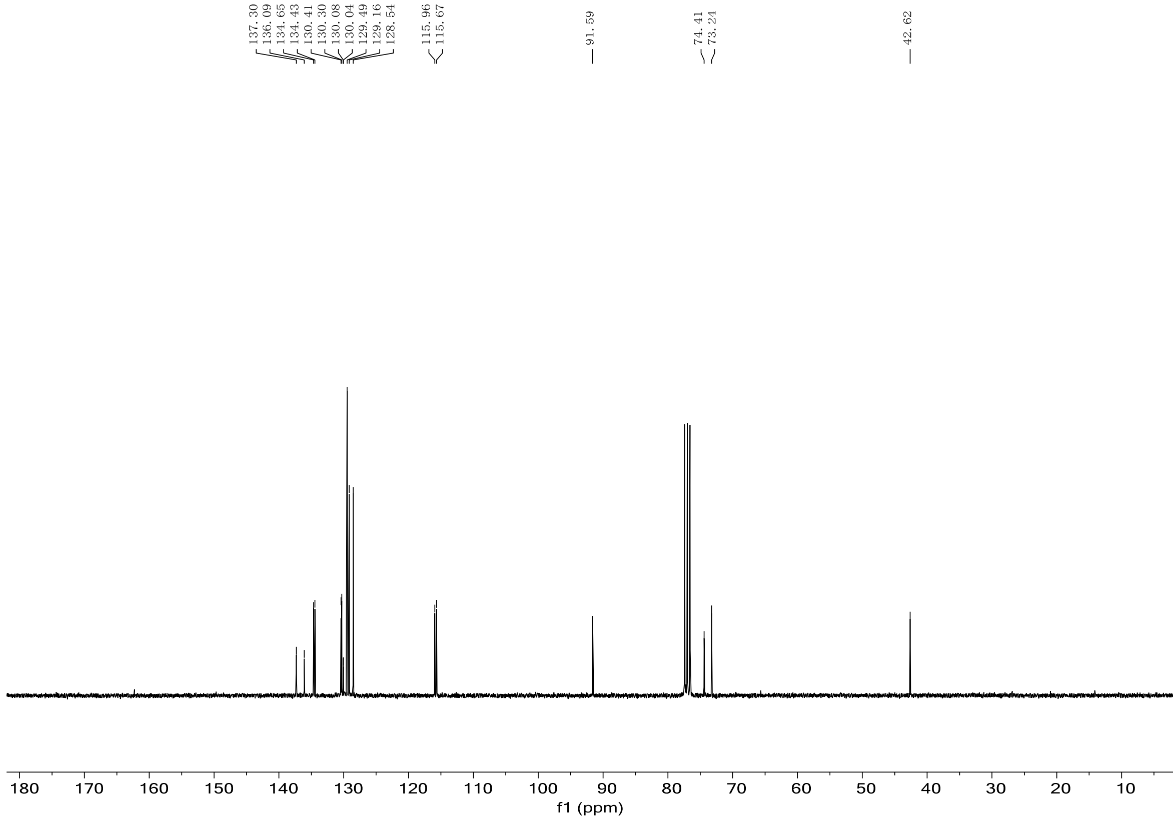

7.95
7.95
7.95
7.94
7.93
7.92
7.92
7.81
7.81
7.80
7.80
7.79
7.78
7.77
7.69
7.67
7.67
7.66
7.60
7.59
7.59
7.58
7.58
7.57
7.57
7.56
7.56
7.54
7.54
7.54
7.49
7.49
7.49
7.47
7.47
7.46
7.44
7.44
7.10
7.07
6.56
6.53
5.30
5.29
4.78
4.77
4.75
4.74
3.96
3.93
2.91
2.52
2.23
2.06
0.84
3.15
2.01
1.60
1.67
1.02
1.00
0.95
6.04
2.87
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
f1 (ppm)

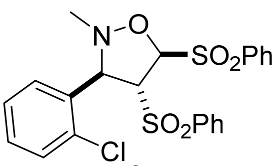

150.80
137.54
136.38
134.32
134.23
129.51
129.30
129.18
129.09
129.01
128.88
128.51
120.67
112.29
91.70
74.00
73.94
42.45
40.28
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


N
O
SO2Ph
SO2Ph
F

3n

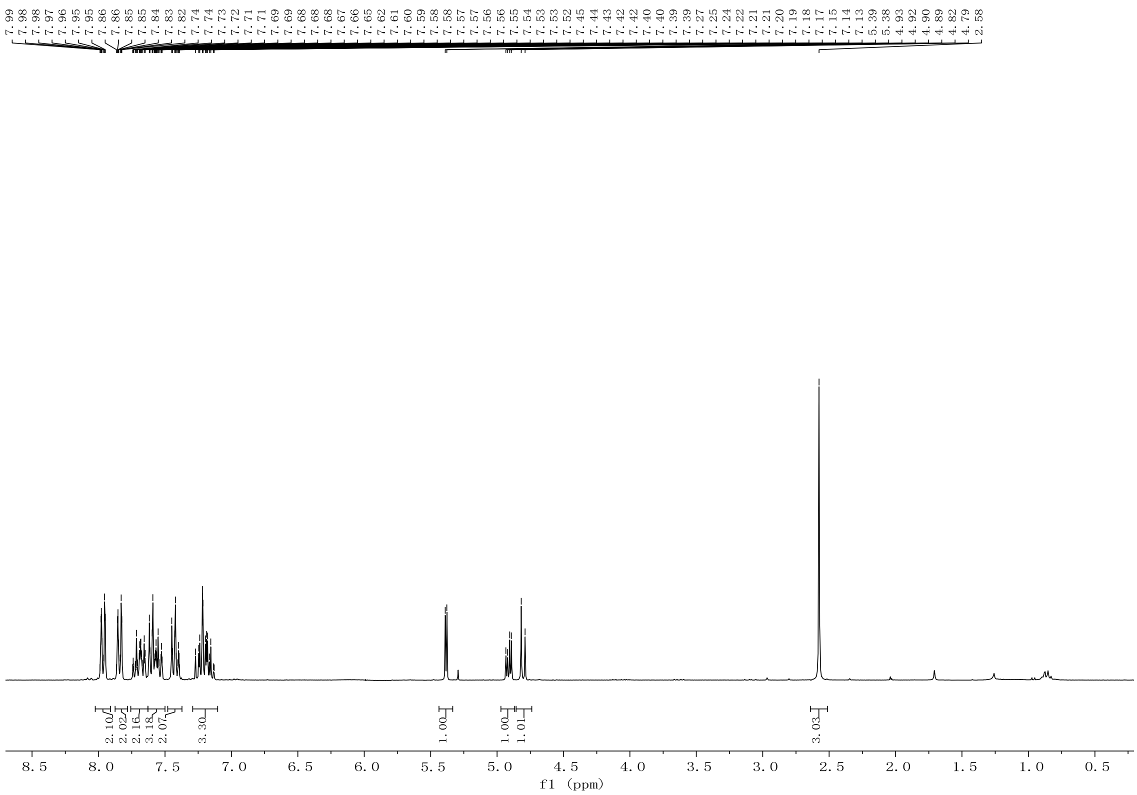

7.94 7.93 7.93 7.92 7.91 7.91 7.82 7.82 7.81 7.80 7.79 7.74 7.71 7.69 7.68 7.67 7.64 7.62 7.62 7.61 7.61 7.60 7.59 7.58 7.58 7.56 7.55 7.53 7.52 7.51 7.50 7.48 7.47 7.32 7.32 7.30 7.30 7.29 7.28 7.28 7.27 6.97 6.96 6.95 6.94 6.93 6.92 6.91 5.30 5.28 4.76 4.75 4.74 4.73 4.05 4.02 2.55
2.12
2.06
6.31
2.37
1.97
1.06
1.06
1.00
3.05
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
f1 (ppm)


137.30 136.09 134.65 134.43 130.41 130.30 130.08 130.04 129.49 129.16 128.54
115.96 115.67
91.59
74.41 73.24
42.62
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

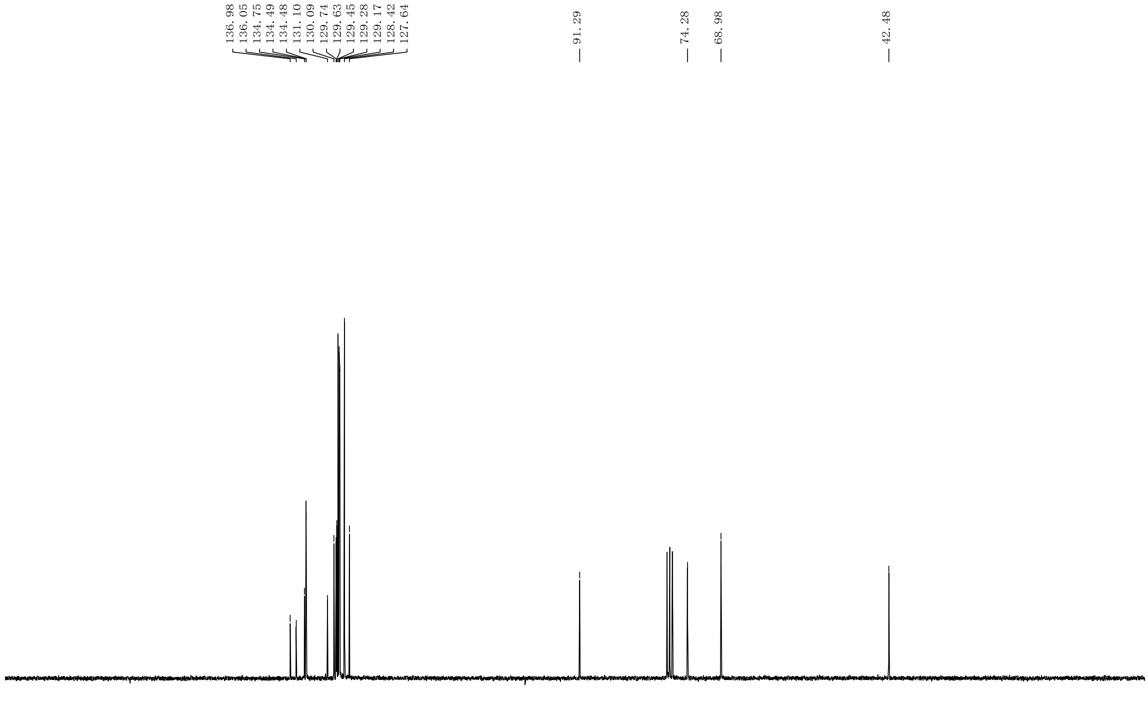


N
O
SO2Ph
SO2Ph
Cl

3o



7.99 7.98 7.98 7.97 7.96 7.95 7.95 7.86 7.86 7.85 7.85 7.84 7.83 7.82 7.74 7.74 7.73 7.72 7.71 7.71 7.69 7.69 7.68 7.68 7.68 7.67 7.66 7.65 7.62 7.61 7.60 7.59 7.58 7.58 7.57 7.57 7.56 7.55 7.54 7.53 7.53 7.52 7.45 7.44 7.43 7.42 7.42 7.40 7.40 7.39 7.39 7.27 7.25 7.24 7.22 7.21 7.20 7.19 7.18 7.17 7.15 7.14 7.13 5.39 5.38 4.93 4.92 4.90 4.89 4.82 4.79 2.58
2.10
2.02
2.16
3.18
2.07
3.30
1.00
1.00
1.01
3.03
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
f1 (ppm)



136.98 136.05 134.75 134.49 134.48 131.10 130.09 129.74 129.63 129.45 129.28 129.17 128.42 127.64
91.29
74.28
68.98
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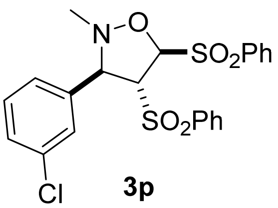
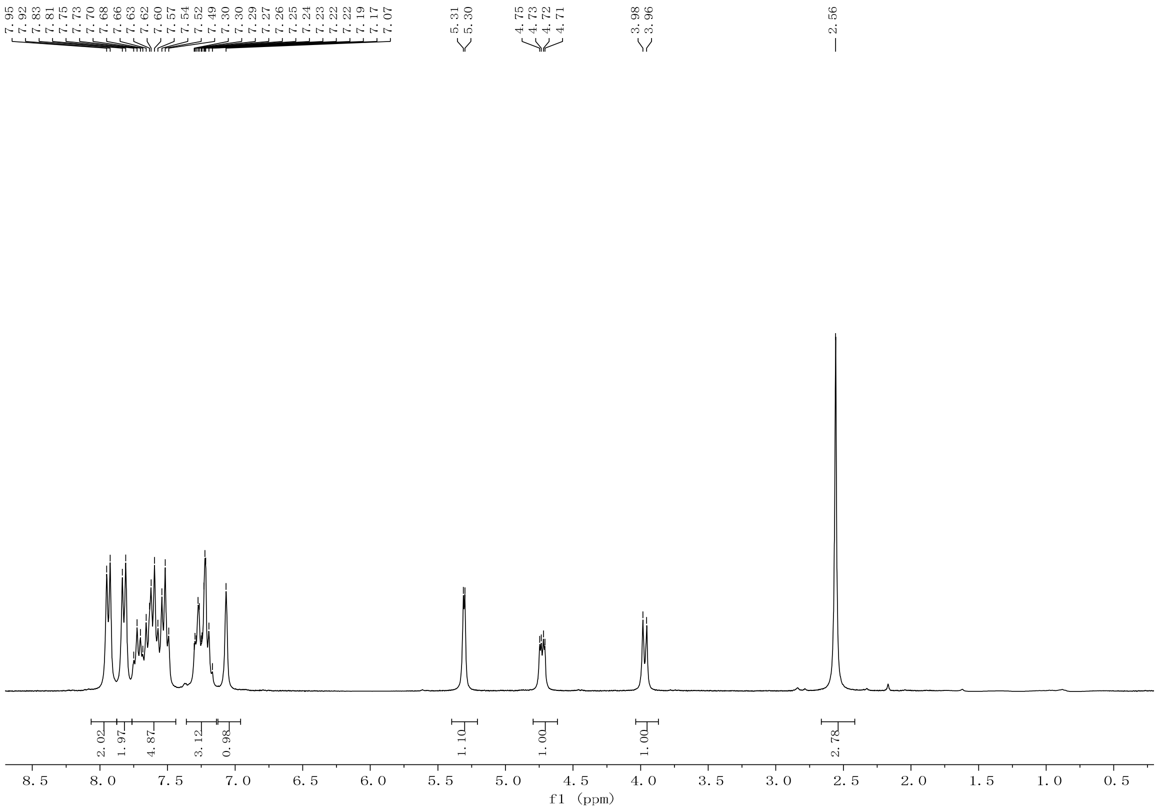
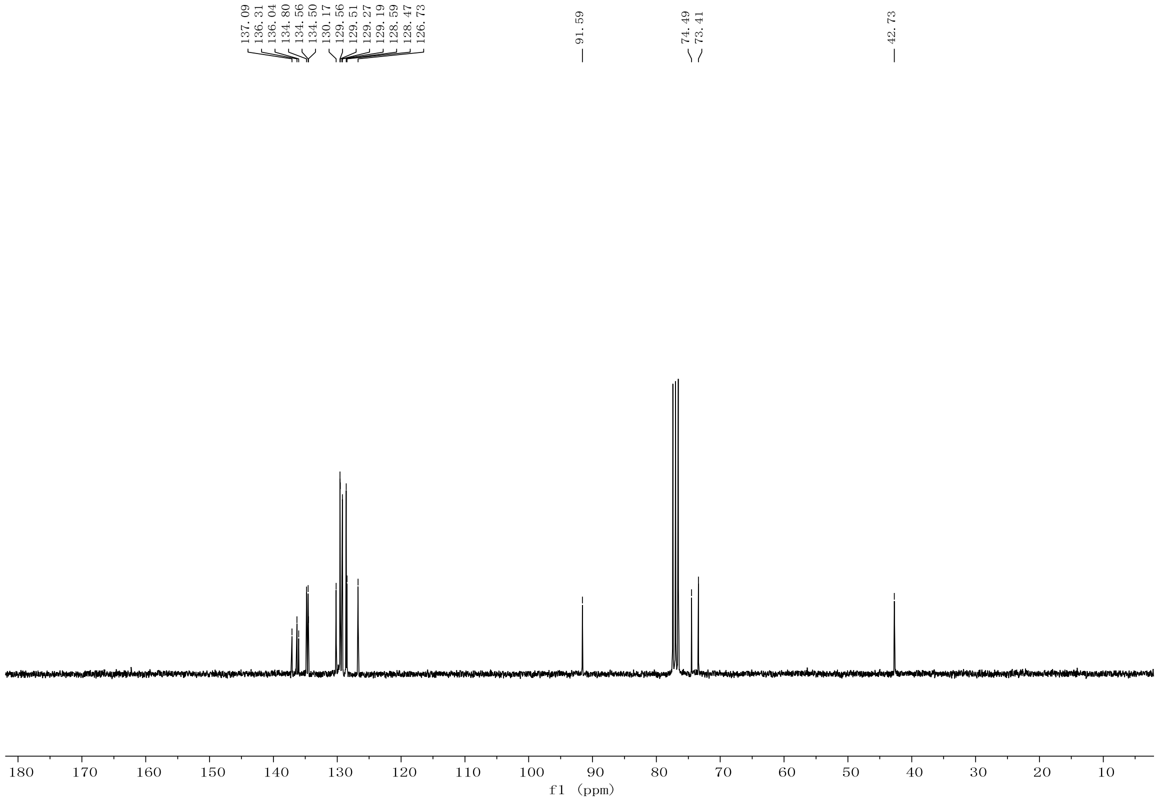
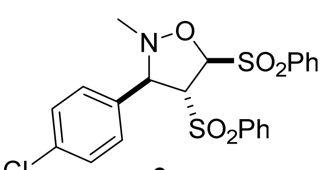
f1 (ppm)



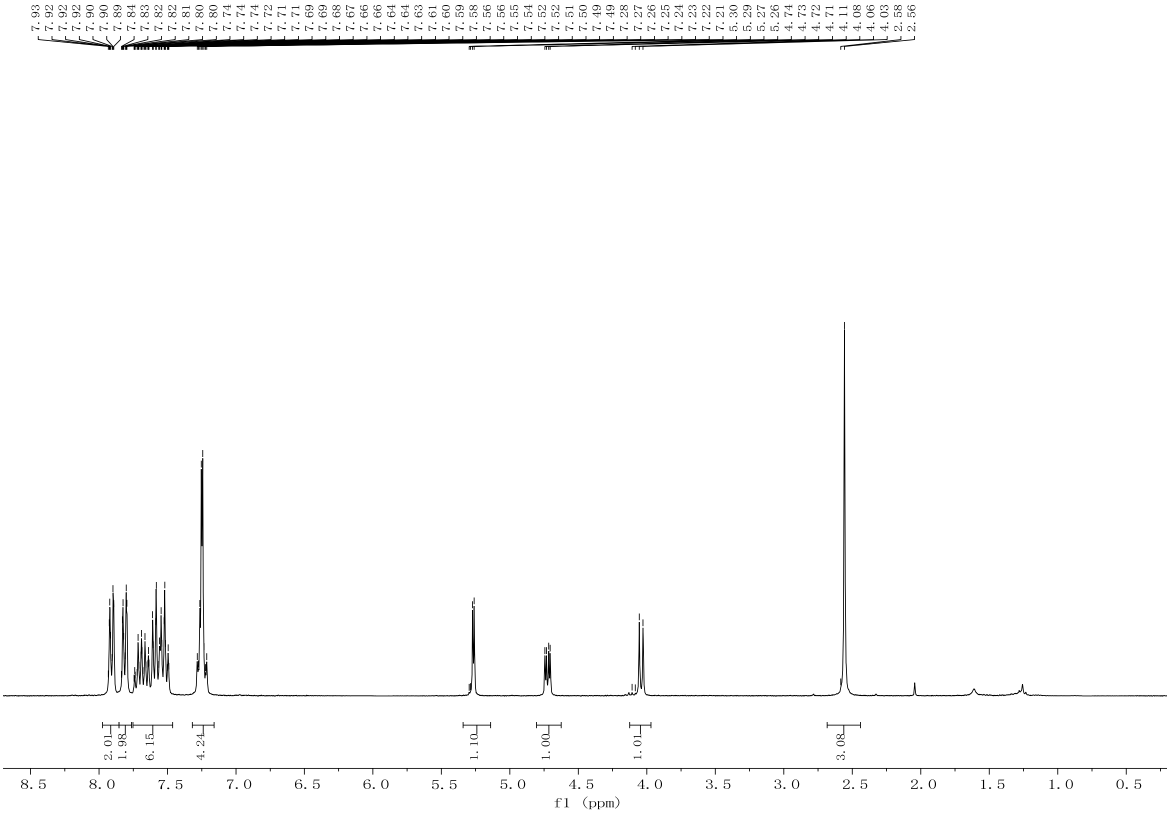
3p

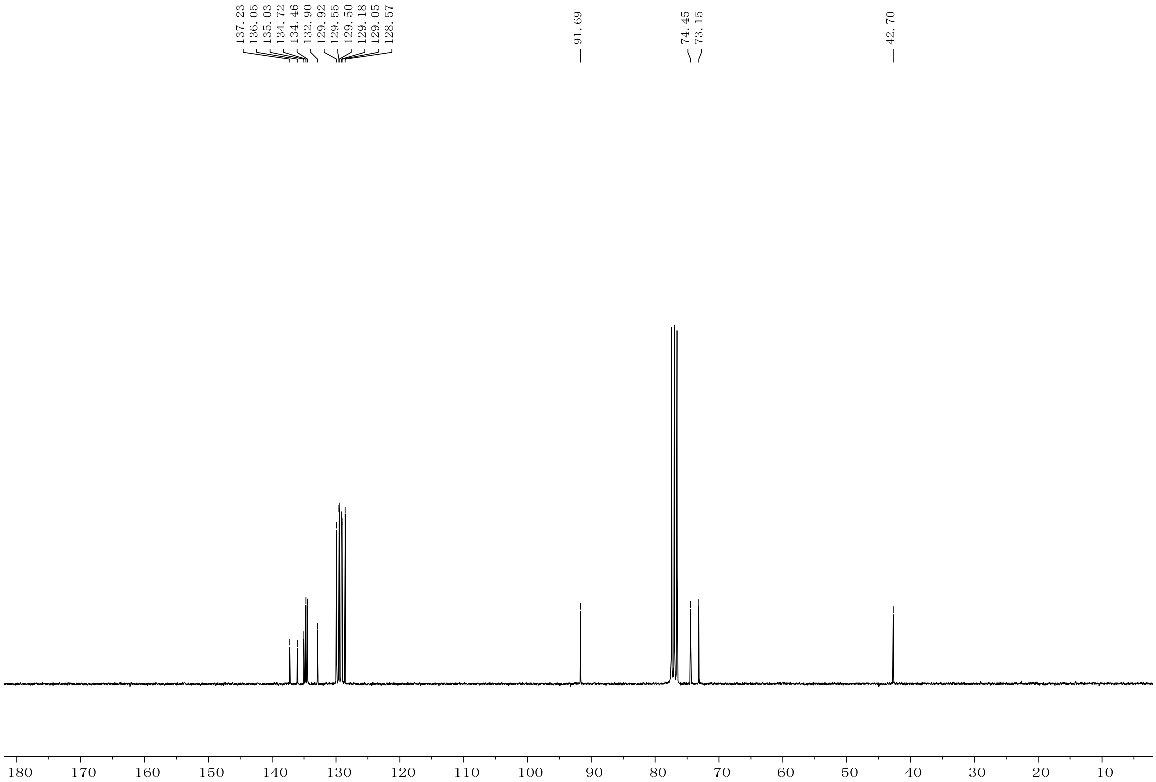

7.95 7.92 7.83 7.81 7.75 7.73 7.70 7.68 7.66 7.63 7.62 7.60 7.57 7.54 7.52 7.49 7.30 7.30 7.29 7.27 7.26 7.25 7.24 7.23 7.22 7.22 7.19 7.17 7.07
5.31 5.30
4.75 4.73 4.72 4.71
3.98 3.96
2.56
2.02 1.97 4.87 3.12 0.98
1.10
1.00
1.00
2.78
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

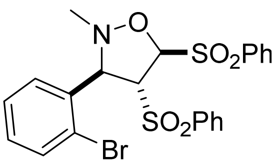

137.09 136.31 136.04 134.80 134.56 134.50 130.17 129.56 129.51 129.27 129.19 128.59 128.47 126.73
91.59
74.49 73.41
42.73
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


N
O
SO2Ph
SO2Ph
Cl

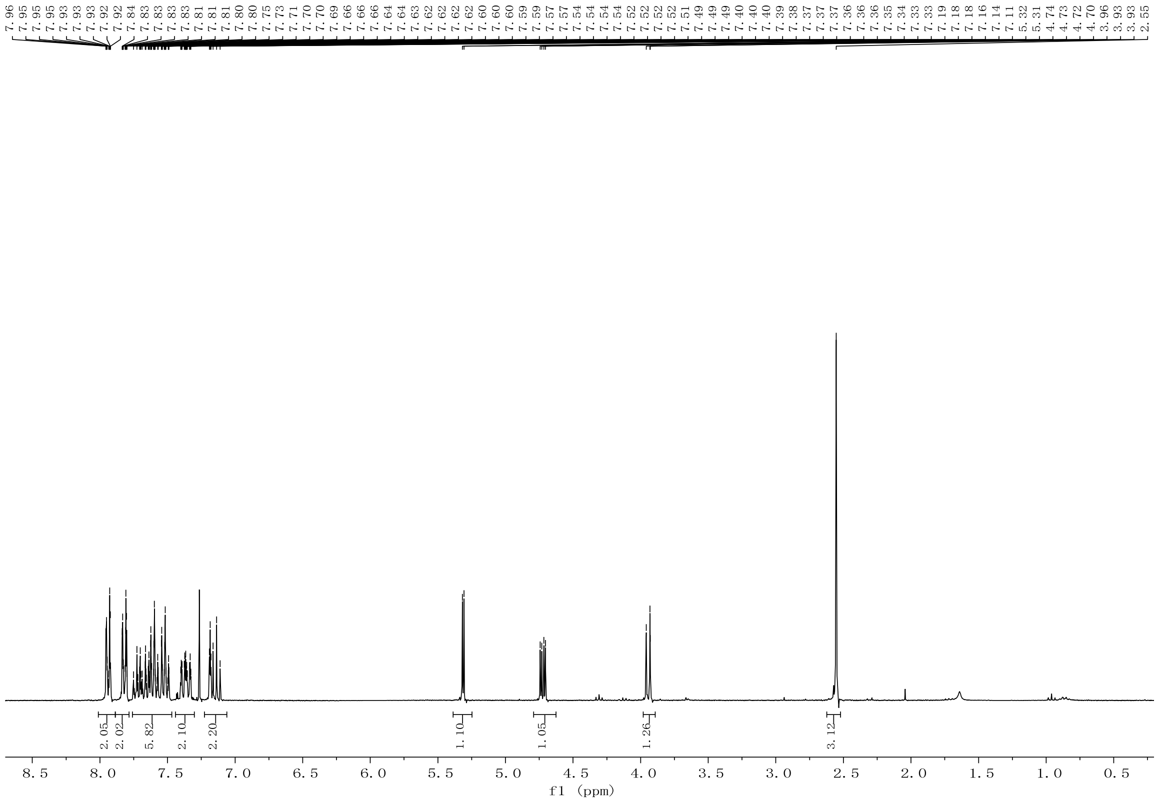
3q

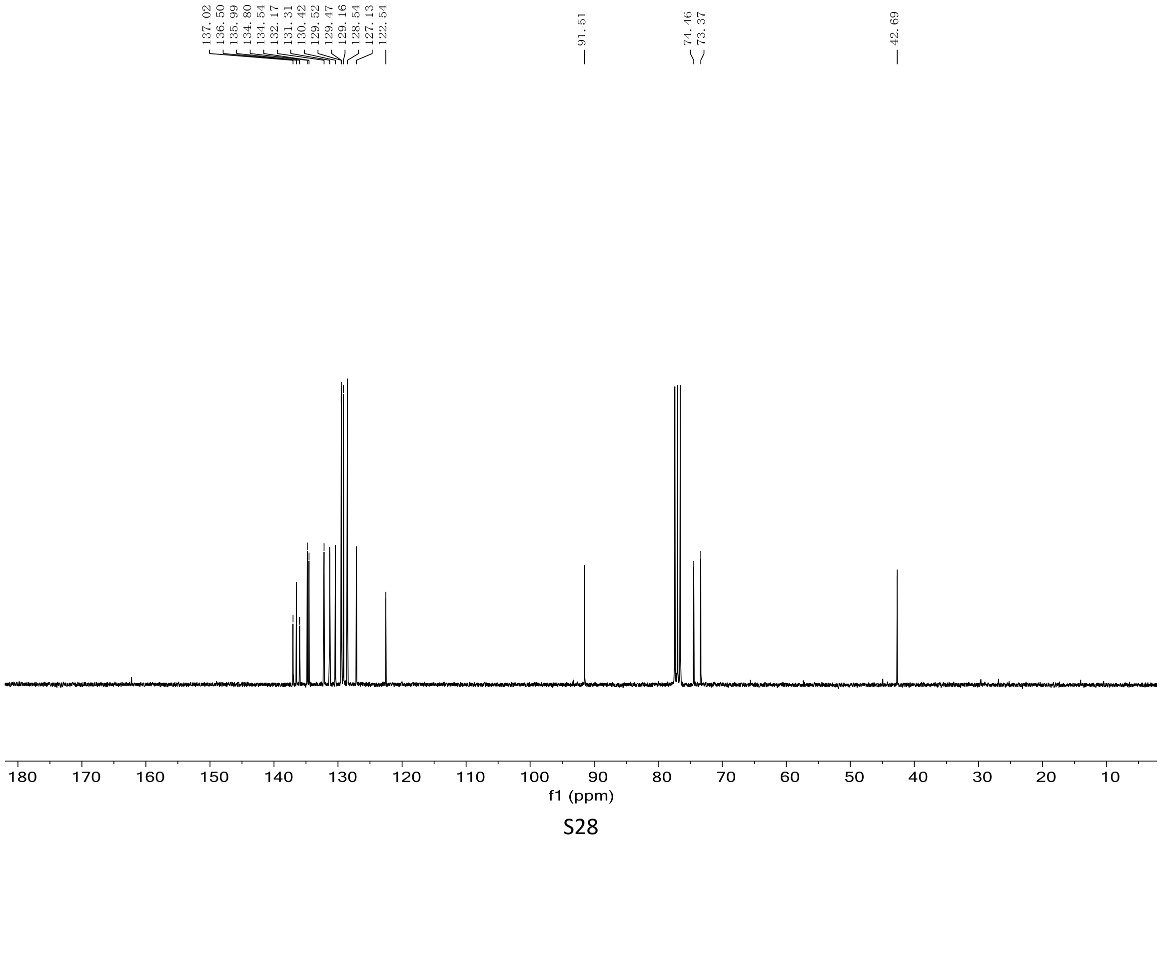

7.93 7.92 7.92 7.90 7.90 7.89 7.84 7.83 7.82 7.82 7.81 7.80 7.80 7.74 7.74 7.74 7.72 7.71 7.71 7.69 7.68 7.67 7.66 7.66 7.64 7.63 7.61 7.60 7.59 7.58 7.56 7.56 7.55 7.54 7.52 7.52 7.51 7.50 7.49 7.49 7.28 7.27 7.26 7.25 7.24 7.23 7.22 7.21 5.30 5.29 5.27 5.26 4.74 4.73 4.72 4.71 4.11 4.08 4.06 4.03 2.58 2.56
2.01 1.98 6.15 4.24 1.10 1.00 1.01 3.08
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
f1 (ppm)

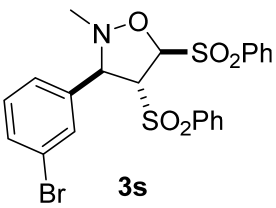

137.23 136.05 135.03 134.72 134.46 132.90 129.92 129.55 129.50 129.18 129.05 128.57
91.69
74.45 73.15
42.70
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)



3r

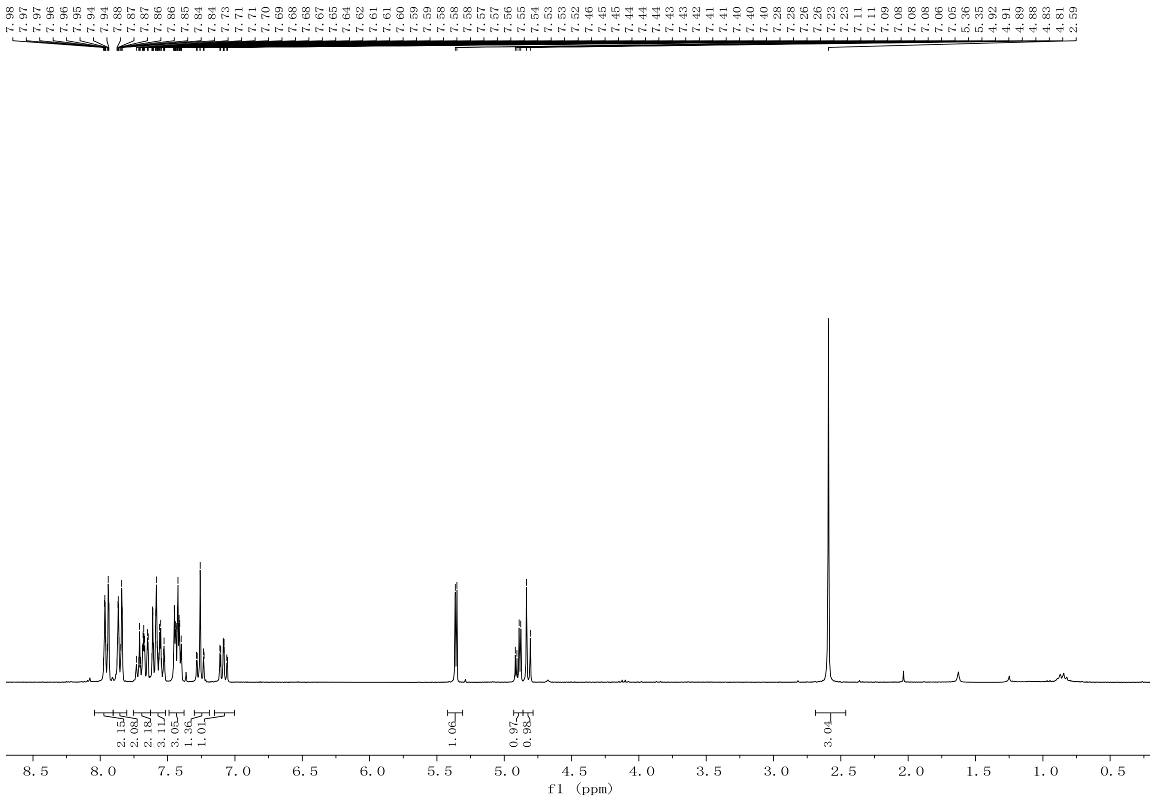




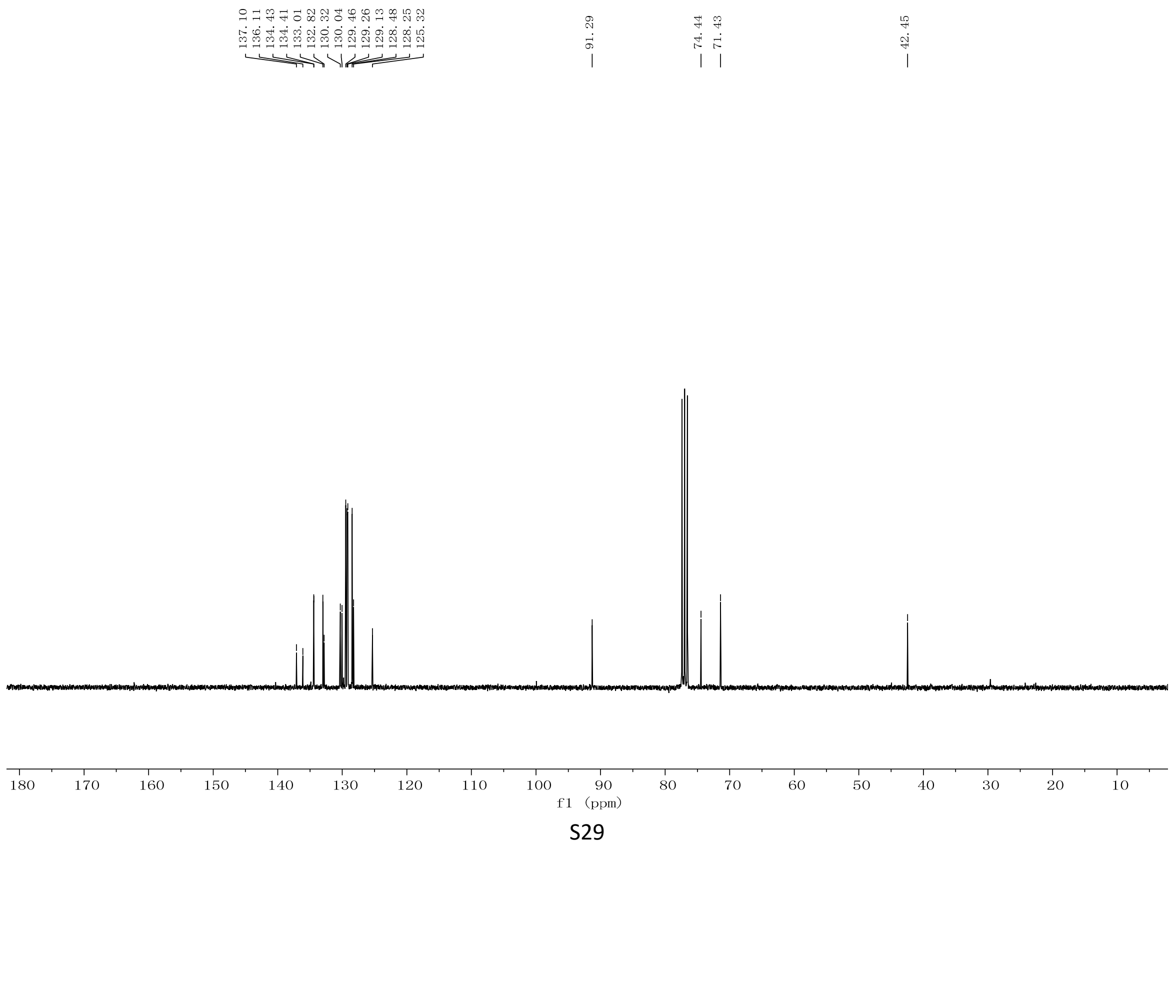


N
O
SO2Ph
SO2Ph
Br

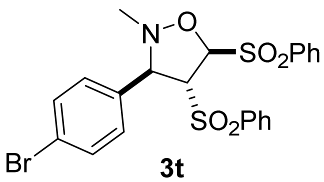
3s



7.98 7.97 7.97 7.96 7.96 7.95 7.94 7.94 7.88 7.87 7.87 7.86 7.86 7.85 7.84 7.84 7.73 7.71 7.71 7.70 7.69 7.68 7.68 7.67 7.65 7.64 7.62 7.61 7.61 7.60 7.59 7.59 7.58 7.58 7.57 7.57 7.56 7.55 7.54 7.53 7.53 7.52 7.46 7.45 7.45 7.44 7.44 7.43 7.43 7.42 7.41 7.41 7.40 7.40 7.40 7.28 7.28 7.26 7.26 7.23 7.23 7.11 7.11 7.09 7.08 7.08 7.08 7.06 7.05 5.36 5.35 4.92 4.91 4.89 4.88 4.83 4.81 2.59
2.15 2.08 2.18 3.11 3.05 1.36 1.01 1.06 0.97 0.98 3.04
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
f1 (ppm)

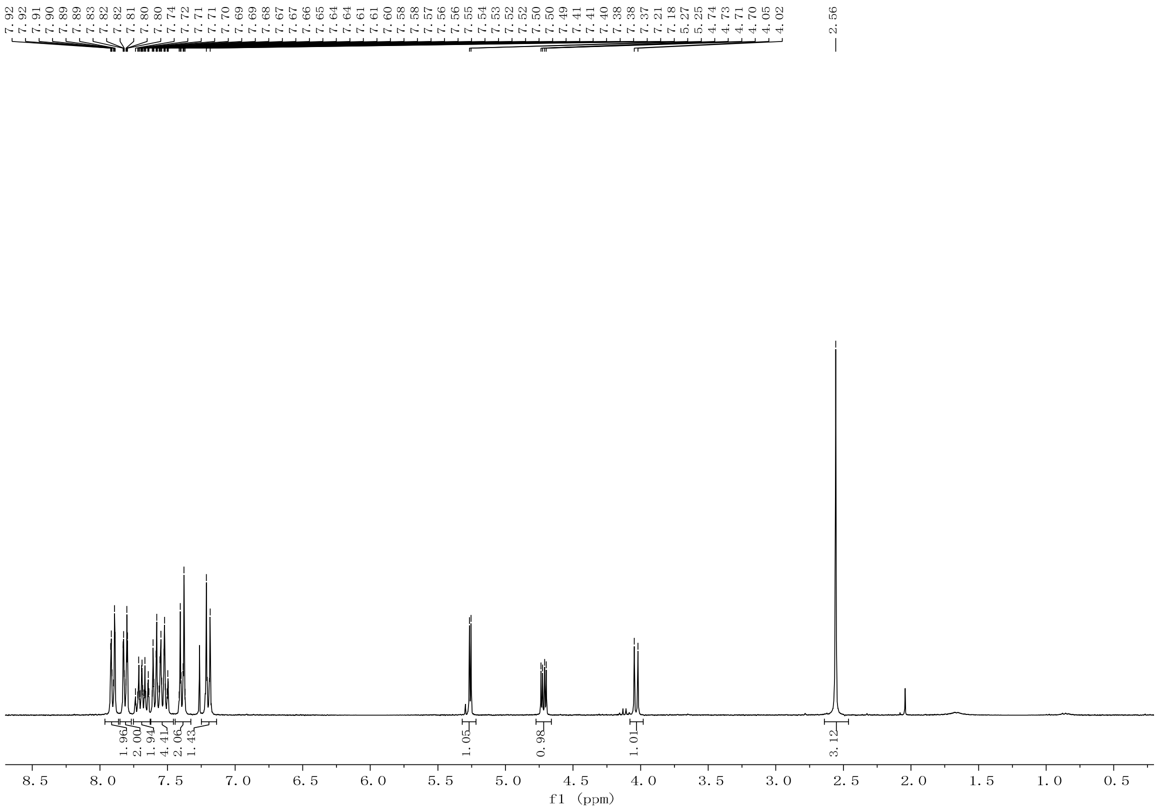


137.10 136.11 134.43 134.41 133.01 132.82 130.32 130.04 129.46 129.26 129.13 128.48 128.25 125.32
91.29
74.44 71.43
42.45
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

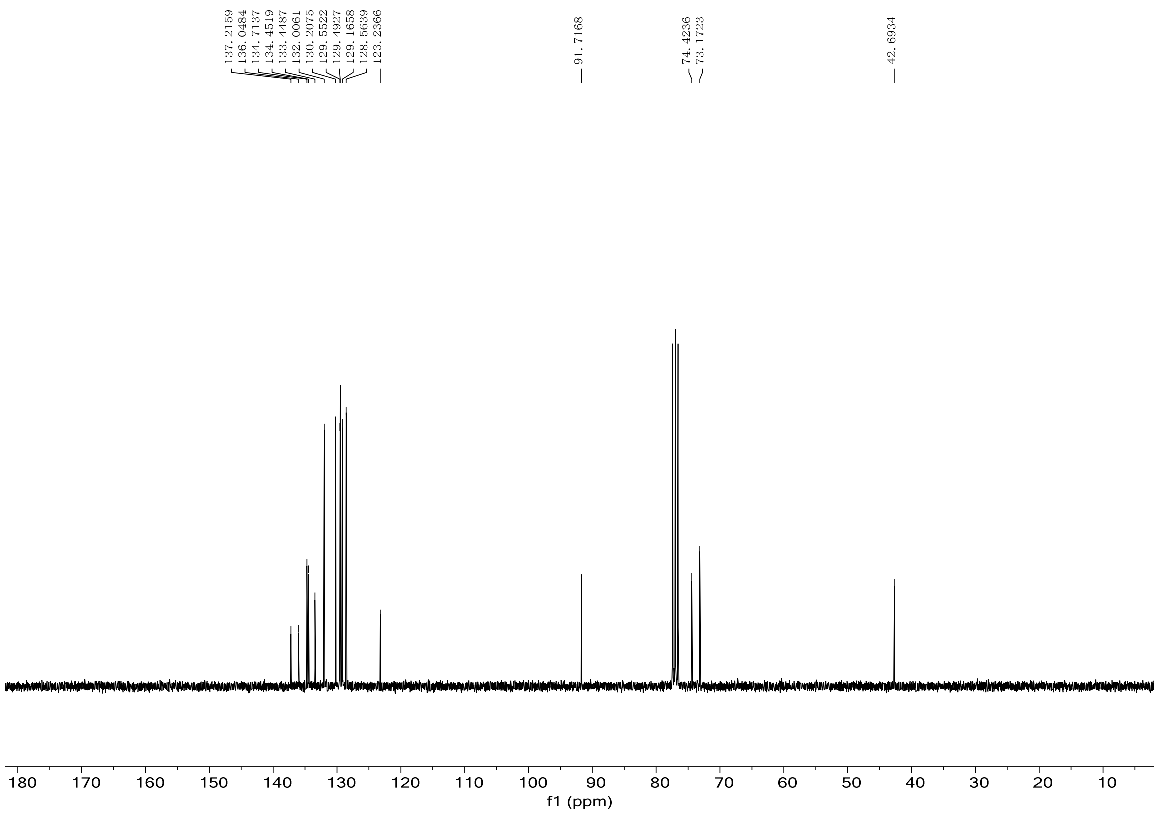


N
O
SO2Ph
SO2Ph
Br

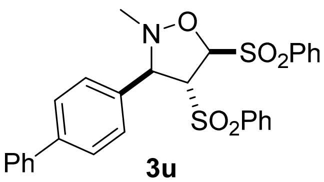
3t



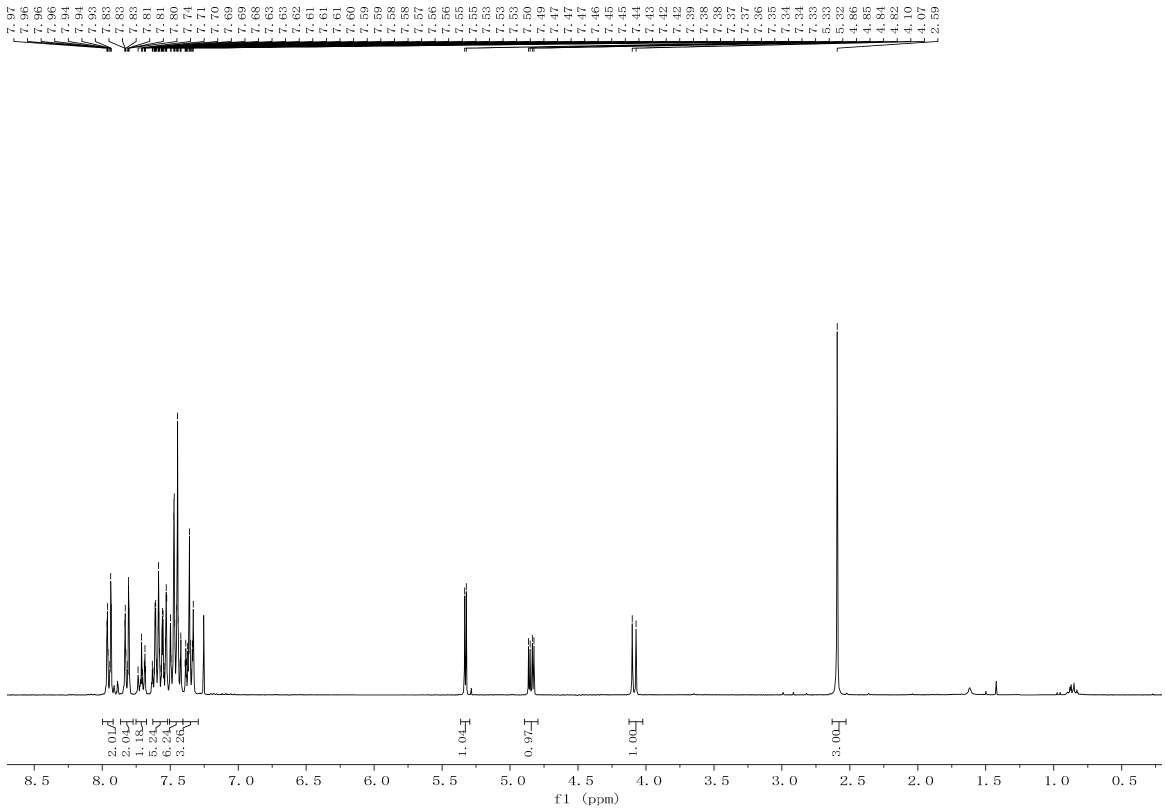
7.92 7.92 7.91 7.90 7.89 7.89 7.83 7.82 7.82 7.81 7.80 7.80 7.74 7.72 7.71 7.71 7.70 7.69 7.69 7.68 7.67 7.67 7.66 7.65 7.64 7.64 7.61 7.61 7.60 7.58 7.58 7.57 7.56 7.56 7.55 7.54 7.53 7.52 7.52 7.50 7.50 7.49 7.41 7.41 7.40 7.38 7.38 7.37 7.21 7.18 5.27 5.25 4.74 4.73 4.71 4.70 4.05 4.02
2.56
1.96 2.00 1.94 4.41 2.06 1.43
1.05
0.98
1.01
3.12
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
f1 (ppm)



137.2159 136.0484 134.7137 134.4519 133.4487 132.0061 130.2075 129.5522 129.4927 129.1658 128.5639 123.2366
91.7168
74.4236 73.1723
42.6934
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


N
O
SO2Ph
SO2Ph
Ph

3u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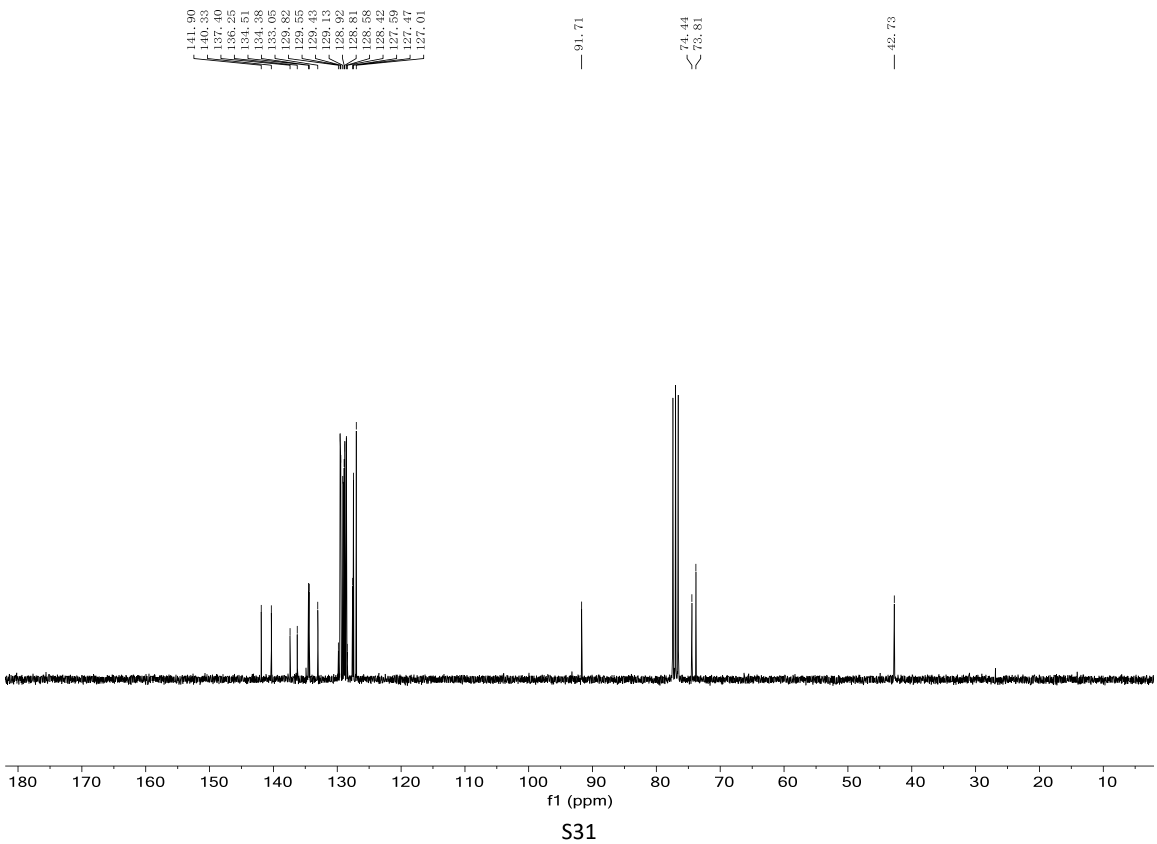
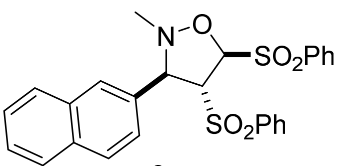
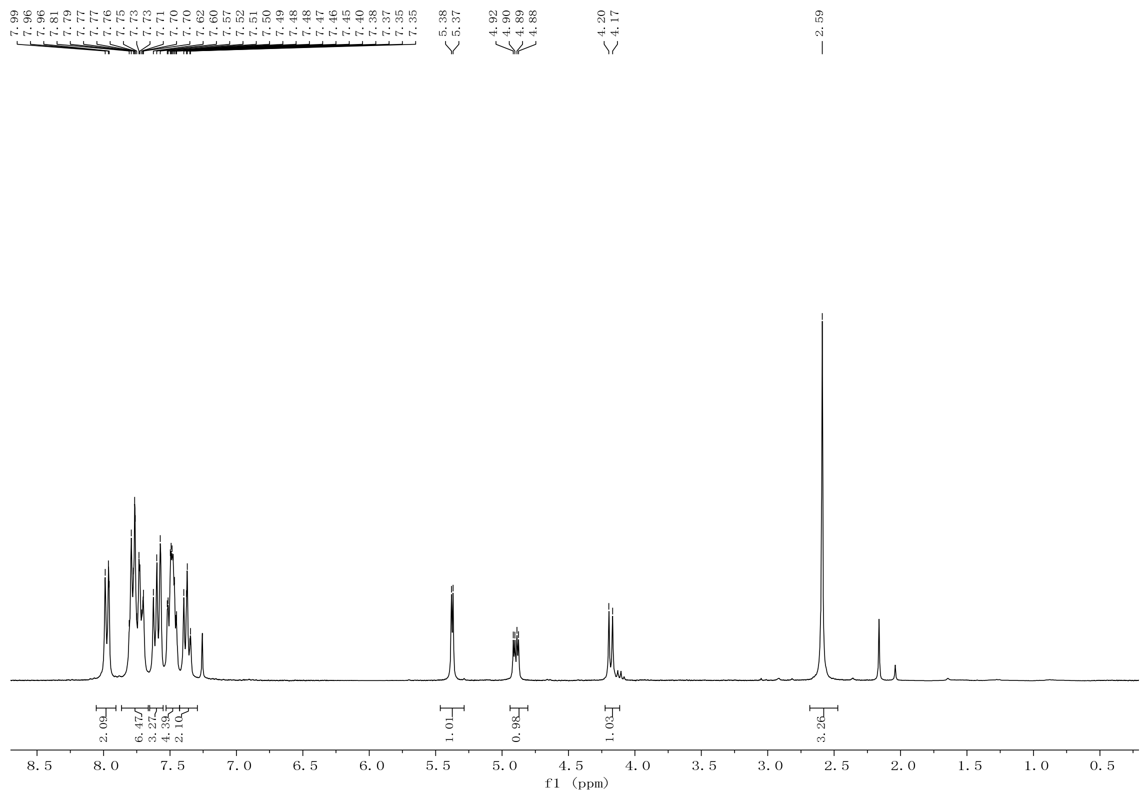
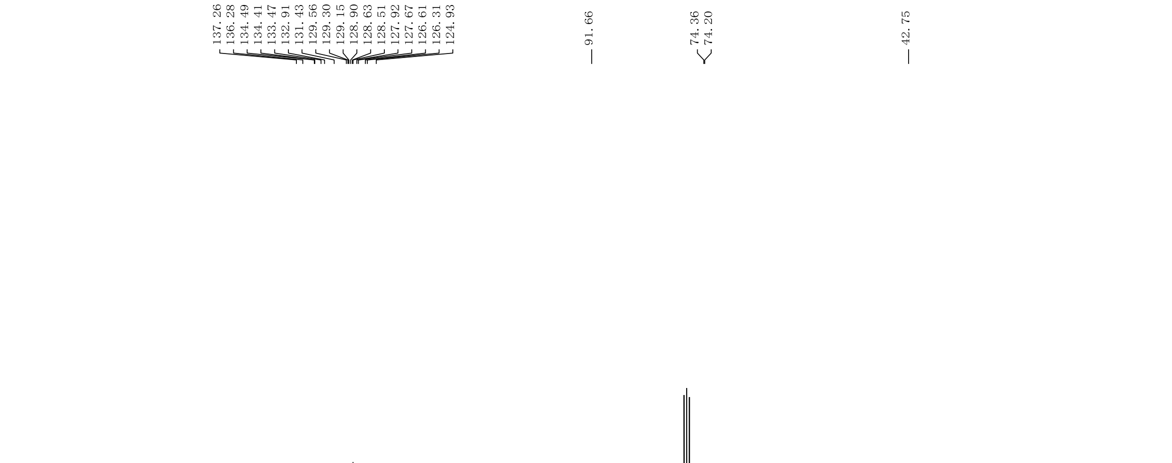
2.04
1.18
5.24
6.24
3.26
1.04
0.97
1.00
3.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

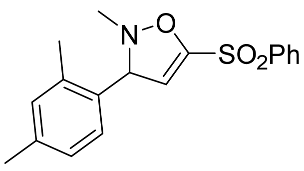

141.90
140.33
137.40
136.25
134.51
134.38
133.05
129.82
129.55
129.43
129.13
128.92
128.81
128.58
128.42
127.59
127.47
127.01
91.71
74.44
73.81
42.73
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


N
O
SO2Ph
SO2Ph

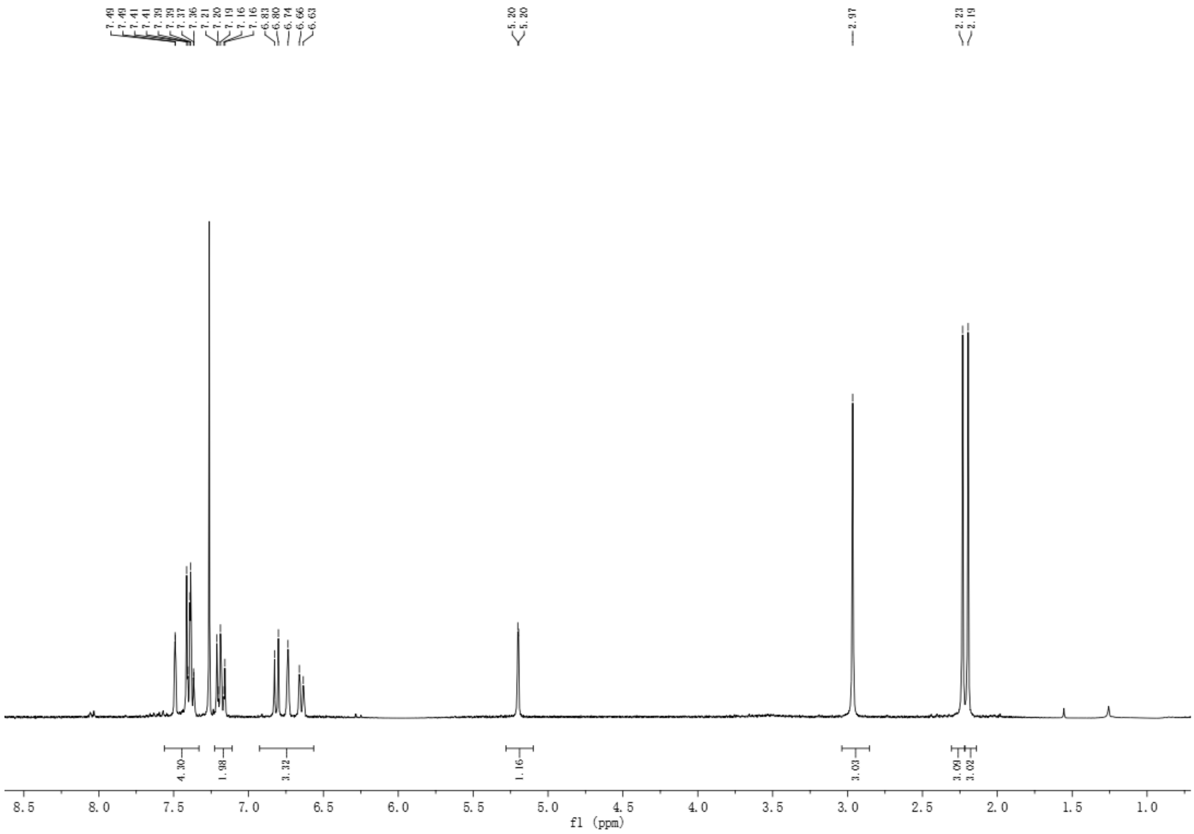
3v


7.99 7.96 7.96 7.81 7.79 7.77 7.77 7.76 7.75 7.73 7.73 7.71 7.70 7.70 7.62 7.60 7.57 7.52 7.51 7.50 7.49 7.48 7.48 7.47 7.46 7.45 7.40 7.38 7.37 7.35 7.35
5.38 5.37
4.92 4.90 4.89 4.88
4.20 4.17
2.59
2.09
6.47
3.27
4.39
2.10
1.01
0.98
1.03
3.26
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
f1 (ppm)

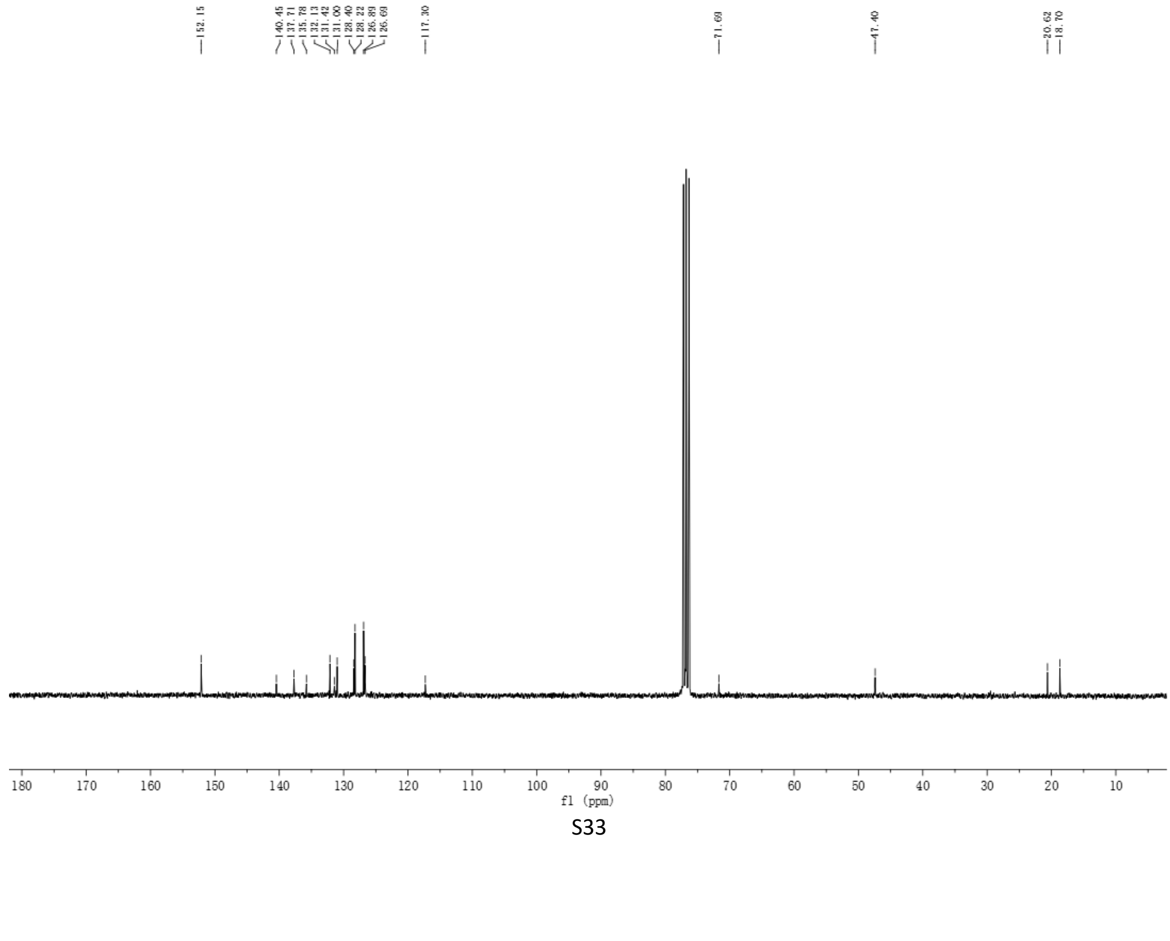

137.26 136.28 134.49 134.41 133.47 132.91 131.43 129.56 129.30 129.15 128.90 128.63 128.51 127.92 127.67 126.61 126.31 124.93
91.66
74.36 74.20
42.75
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)



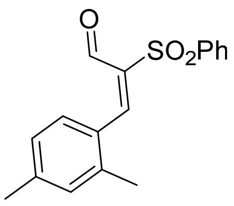
4



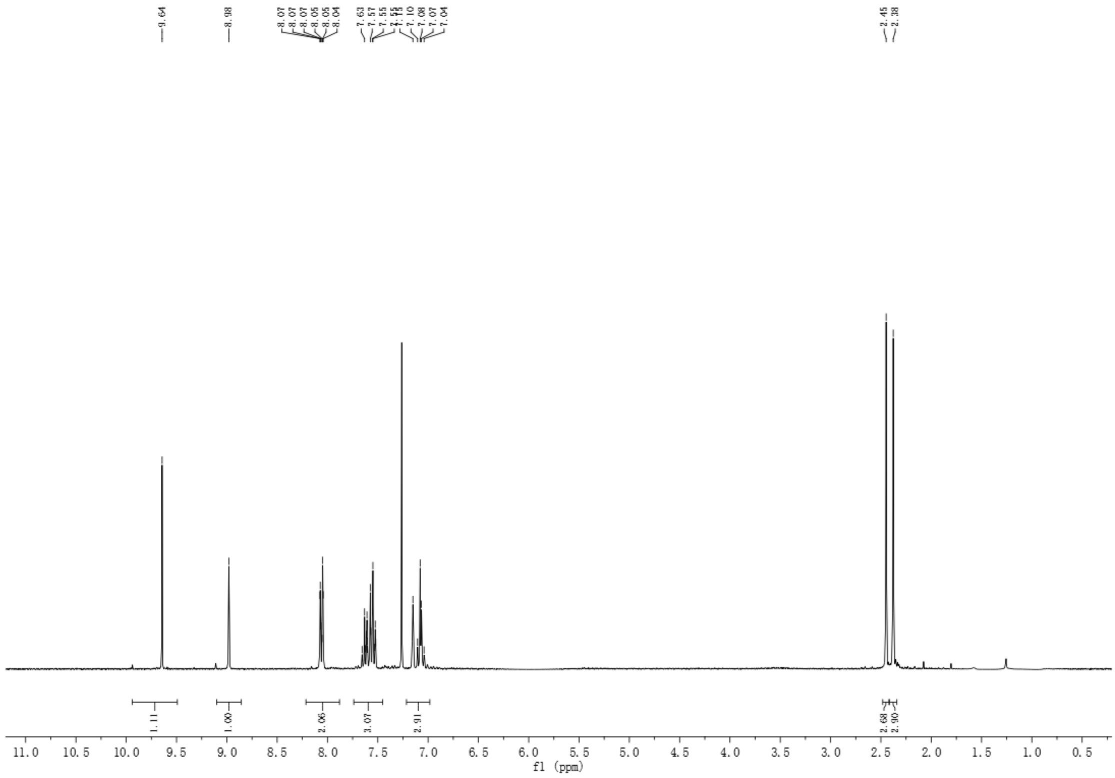
7.49
7.49
7.41
7.39
7.39
7.37
7.36
7.21
7.20
7.19
7.16
7.16
6.83
6.80
6.74
6.66
6.63
5.20
5.20
2.97
2.23
2.19
4.30
1.98
3.32
1.16
3.03
3.09
3.02
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f1 (ppm)

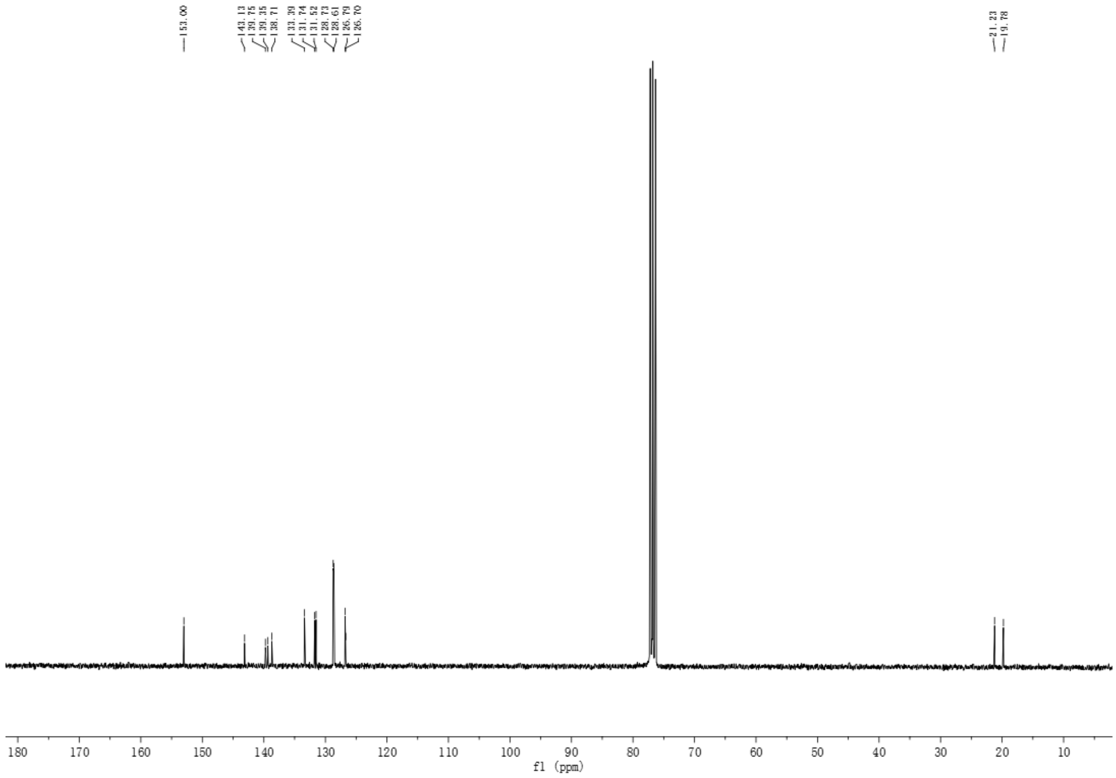


152.15
140.45
137.71
135.78
132.13
131.42
131.00
128.40
128.22
126.89
126.69
117.30
71.69
47.40
20.62
18.70
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)



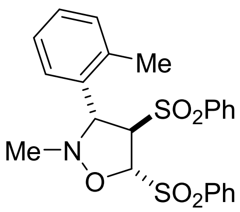
5





X-Ray Crystallographic Data

Crystallographic data for **3b** has been deposited with the Cambridge Crystallographic Data Centre as. These data can be obtained free of charge via www.ccdc.cam. ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.



3b

≡

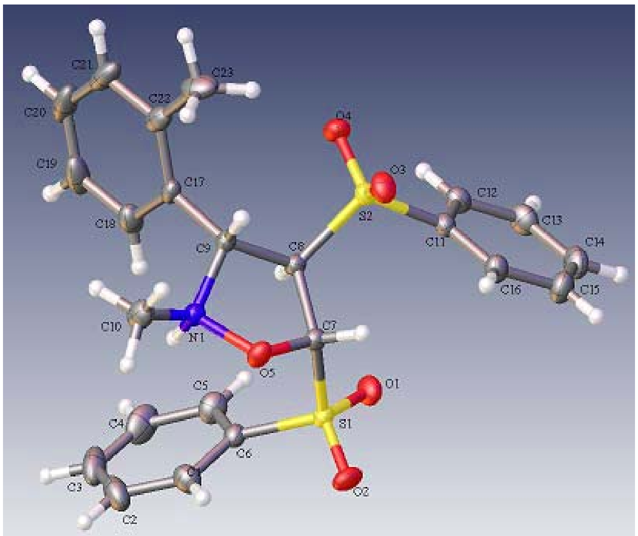


Table 1. Crystal data and structure refinement for 3**b**.

Identification code	**3b**	
Empirical formula	$C_{23}H_{23}NO_5S_2$	
Formula weight	458.55	
Temperature	173.1500 K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P^{-1}	
Unit cell dimensions	a = 10.184(2) Å	alpha= 104.70(3)°.
	b = 10.710(2) Å	beta= 103.61(3)°.
	c = 11.083(2) Å	gamma = 102.46(3)°.
Volume	1086.6(4) $Å^3$	
Z	2	
Density (calculated)	1.401 Mg/m^3	

Absorption coefficient	0.281 mm^{-1}
F(000)	482
Crystal size	0.504 x 0.428 x 0.31 mm^3
Theta range for data collection	3.189 to 27.497°.
Index ranges	-13<=h<=13, -13<=k<=13, -14<=l<=14
Reflections collected	14612
Independent reflections	4958 [R(int) = 0.0234]
Completeness to theta = 26.000°	99.5 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.8925
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4958 / 0 / 282
Goodness-of-fit on F^2	1.101
Final R indices [I>2sigma(I)]	R1 = 0.0373, wR2 = 0.0912
R indices (all data)	R1 = 0.0397, wR2 = 0.0928
Extinction coefficient	n/a
Largest diff. peak and hole	0.348 and -0.794 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **3b**. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
S1	3474(1)	3934(1)	7819(1)	19(1)
S2	1962(1)	915(1)	9267(1)	20(1)
O1	2078(1)	3966(1)	7868(1)	29(1)
O2	4648(1)	5139(1)	8478(1)	29(1)
O3	3115(1)	1003(1)	10358(1)	28(1)

O4	878(1)	-349(1)	8584(1)	29(1)
O5	5120(1)	2380(1)	8310(1)	22(1)
N1	4531(1)	1135(1)	7161(1)	20(1)
C1	4701(2)	3583(2)	5878(2)	30(1)
C2	4670(2)	3123(2)	4584(2)	42(1)
C3	3406(3)	2472(2)	3615(2)	44(1)
C4	2150(2)	2275(2)	3908(2)	43(1)
C5	2153(2)	2712(2)	5205(2)	30(1)
C6	3435(2)	3361(1)	6174(1)	21(1)
C7	3907(2)	2677(1)	8562(1)	19(1)
C8	2720(1)	1331(1)	8054(1)	17(1)
C9	3519(2)	305(1)	7610(1)	19(1)
C10	5729(2)	626(2)	7061(2)	31(1)
C11	1151(2)	2154(2)	9810(1)	21(1)
C12	-299(2)	1885(2)	9279(2)	25(1)
C13	-952(2)	2811(2)	9784(2)	31(1)
C14	-168(2)	3980(2)	10809(2)	33(1)
C15	1279(2)	4242(2)	11322(2)	32(1)
C16	1953(2)	3331(2)	10829(2)	26(1)
C17	2658(2)	-1003(1)	6536(2)	22(1)
C18	2070(2)	-979(2)	5279(2)	31(1)
C19	1314(2)	-2178(2)	4259(2)	42(1)
C20	1147(2)	-3388(2)	4506(2)	46(1)
C21	1723(2)	-3415(2)	5753(2)	41(1)
C22	2501(2)	-2233(2)	6798(2)	29(1)
C23	3139(2)	-2319(2)	8128(2)	40(1)

Table 3. Bond lengths [Å] and angles [°] for **3b**.

S1-O1	1.4421(12)
S1-O2	1.4397(13)
S1-C6	1.7580(15)
S1-C7	1.8255(15)
S2-O3	1.4410(13)
S2-O4	1.4368(13)
S2-C8	1.8019(15)
S2-C11	1.7629(16)
O5-N1	1.4811(16)
O5-C7	1.4117(17)
N1-C9	1.4740(18)
N1-C10	1.4557(19)
C1-C2	1.383(2)
C1-C6	1.390(2)
C2-C3	1.370(3)
C3-C4	1.378(3)
C4-C5	1.393(3)
C5-C6	1.382(2)
C7-C8	1.540(2)
C8-C9	1.5520(19)
C9-C17	1.510(2)
C11-C12	1.389(2)
C11-C16	1.389(2)
C12-C13	1.385(2)
C13-C14	1.384(3)
C14-C15	1.385(3)
C15-C16	1.384(2)
C17-C18	1.390(2)

C17-C22	1.403(2)
C18-C19	1.395(2)
C19-C20	1.375(3)
C20-C21	1.379(3)
C21-C22	1.401(2)
C22-C23	1.500(3)
O1-S1-C6	109.24(8)
O1-S1-C7	106.85(7)
O2-S1-O1	119.55(7)
O2-S1-C6	107.72(8)
O2-S1-C7	105.73(7)
C6-S1-C7	107.11(7)
O3-S2-C8	106.96(7)
O3-S2-C11	108.09(7)
O4-S2-O3	119.41(8)
O4-S2-C8	105.58(7)
O4-S2-C11	108.28(7)
C11-S2-C8	108.04(7)
C7-O5-N1	103.46(10)
C9-N1-O5	100.56(10)
C10-N1-O5	104.86(11)
C10-N1-C9	113.10(12)
C2-C1-C6	118.91(17)
C3-C2-C1	120.17(18)
C2-C3-C4	120.84(17)
C3-C4-C5	120.14(18)
C6-C5-C4	118.44(17)
C1-C6-S1	118.98(12)
C5-C6-S1	119.55(12)
C5-C6-C1	121.48(15)

O5-C7-S1	110.23(10)
O5-C7-C8	107.11(11)
C8-C7-S1	113.75(10)
C7-C8-S2	114.94(10)
C7-C8-C9	101.48(11)
C9-C8-S2	109.45(10)
N1-C9-C8	99.62(11)
N1-C9-C17	111.00(11)
C17-C9-C8	117.15(12)
C12-C11-S2	119.09(12)
C16-C11-S2	119.36(12)
C16-C11-C12	121.36(14)
C13-C12-C11	118.99(15)
C14-C13-C12	120.22(15)
C13-C14-C15	120.20(16)
C16-C15-C14	120.49(16)
C15-C16-C11	118.73(15)
C18-C17-C9	119.34(14)
C18-C17-C22	120.51(15)
C22-C17-C9	120.11(14)
C17-C18-C19	120.48(18)
C20-C19-C18	119.47(19)
C19-C20-C21	120.23(17)
C20-C21-C22	121.82(18)
C17-C22-C23	122.73(15)
C21-C22-C17	117.49(17)
C21-C22-C23	119.78(17)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2$ x 10^3) for **3b**. The anisotropic displacement factor exponent takes the form: -2p2[h2 a*2U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
S1	24(1)	15(1)	19(1)	3(1)	8(1)	6(1)
S2	23(1)	19(1)	18(1)	6(1)	8(1)	6(1)
O1	32(1)	30(1)	34(1)	12(1)	18(1)	18(1)
O2	38(1)	16(1)	26(1)	3(1)	4(1)	0(1)
O3	33(1)	35(1)	21(1)	12(1)	10(1)	17(1)
O4	31(1)	20(1)	33(1)	6(1)	14(1)	1(1)
O5	18(1)	18(1)	25(1)	1(1)	4(1)	3(1)
N1	20(1)	17(1)	22(1)	3(1)	7(1)	4(1)
C1	29(1)	34(1)	31(1)	12(1)	14(1)	10(1)
C2	57(1)	51(1)	40(1)	23(1)	33(1)	29(1)
C3	79(2)	45(1)	24(1)	14(1)	23(1)	37(1)
C4	56(1)	42(1)	22(1)	4(1)	-4(1)	19(1)
C5	28(1)	32(1)	26(1)	6(1)	3(1)	10(1)
C6	25(1)	19(1)	20(1)	6(1)	8(1)	9(1)
C7	21(1)	17(1)	17(1)	4(1)	6(1)	5(1)
C8	19(1)	16(1)	15(1)	3(1)	6(1)	4(1)
C9	22(1)	16(1)	18(1)	4(1)	8(1)	5(1)
C10	24(1)	27(1)	43(1)	6(1)	16(1)	9(1)
C11	24(1)	22(1)	19(1)	7(1)	10(1)	7(1)
C12	24(1)	28(1)	23(1)	8(1)	8(1)	5(1)
C13	23(1)	39(1)	35(1)	14(1)	12(1)	13(1)
C14	37(1)	36(1)	35(1)	11(1)	19(1)	20(1)
C15	35(1)	29(1)	29(1)	1(1)	11(1)	10(1)
C16	24(1)	27(1)	23(1)	4(1)	8(1)	7(1)

C17	21(1)	19(1)	24(1)	0(1)	11(1)	3(1)
C18	29(1)	33(1)	24(1)	1(1)	11(1)	0(1)
C19	30(1)	52(1)	26(1)	-8(1)	12(1)	-5(1)
C20	30(1)	33(1)	52(1)	-18(1)	20(1)	-7(1)
C21	31(1)	18(1)	68(1)	-1(1)	25(1)	2(1)
C22	23(1)	20(1)	46(1)	6(1)	18(1)	7(1)
C23	41(1)	28(1)	62(1)	25(1)	22(1)	15(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **3b**.

	x	y	z	U(eq)
H1	4072	1301	6358	24
H1A	5554	4034	6538	36
H2	5508	3256	4371	51
H3	3396	2159	2749	53
H4	1299	1850	3239	52
H5	1312	2571	5414	36
H7	4108	3050	9512	22
H8	1978	1316	7300	21
H9	4032	110	8373	23
H10A	6426	1279	6910	47
H10B	5417	-204	6346	47
H10C	6135	468	7862	47
H12	-822	1095	8595	30
H13	-1920	2647	9433	37
H14	-613	4592	11155	40
H15	1801	5036	12001	39
H16	2923	3503	11173	31

H18	2180	-158	5117	38
H19	927	-2159	3418	51
H20	644	-4190	3831	55
H21	1591	-4242	5905	50
H23A	4151	-1998	8367	60
H23B	2859	-3241	8110	60
H23C	2817	-1774	8759	60

Table 6. Torsion angles [°] for **3b**.

S1-C7-C8-S2	-115.81(10)
S1-C7-C8-C9	126.21(10)
S2-C8-C9-N1	-155.34(9)
S2-C8-C9-C17	84.96(13)
S2-C11-C12-C13	174.65(12)
S2-C11-C16-C15	-174.49(13)
O1-S1-C6-C1	163.13(12)
O1-S1-C6-C5	-16.38(15)
O1-S1-C7-O5	167.76(9)
O1-S1-C7-C8	47.45(12)
O2-S1-C6-C1	31.84(14)
O2-S1-C6-C5	-147.67(13)
O2-S1-C7-O5	-63.88(11)
O2-S1-C7-C8	175.81(10)
O3-S2-C8-C7	-52.77(12)
O3-S2-C8-C9	60.62(11)
O3-S2-C11-C12	-146.23(12)
O3-S2-C11-C16	28.79(14)
O4-S2-C8-C7	179.06(10)

O4-S2-C8-C9	-67.54(11)
O4-S2-C11-C12	-15.54(14)
O4-S2-C11-C16	159.48(12)
O5-N1-C9-C8	50.46(12)
O5-N1-C9-C17	174.57(11)
O5-C7-C8-S2	122.14(11)
O5-C7-C8-C9	4.15(13)
N1-O5-C7-S1	-97.22(10)
N1-O5-C7-C8	27.01(13)
N1-C9-C17-C18	-46.24(18)
N1-C9-C17-C22	131.46(14)
C1-C2-C3-C4	0.6(3)
C2-C1-C6-S1	179.50(13)
C2-C1-C6-C5	-1.0(2)
C2-C3-C4-C5	-1.5(3)
C3-C4-C5-C6	1.1(3)
C4-C5-C6-S1	179.62(13)
C4-C5-C6-C1	0.1(2)
C6-S1-C7-O5	50.80(11)
C6-S1-C7-C8	-69.51(12)
C6-C1-C2-C3	0.7(3)
C7-S1-C6-C1	-81.49(14)
C7-S1-C6-C5	99.00(14)
C7-O5-N1-C9	-49.59(12)
C7-O5-N1-C10	-167.13(12)
C7-C8-C9-N1	-33.47(12)
C7-C8-C9-C17	-153.16(12)
C8-S2-C11-C12	98.35(13)
C8-S2-C11-C16	-86.63(13)
C8-C9-C17-C18	67.21(18)

C8-C9-C17-C22	-115.09(15)
C9-C17-C18-C19	177.62(14)
C9-C17-C22-C21	-178.31(14)
C9-C17-C22-C23	1.0(2)
C10-N1-C9-C8	161.75(12)
C10-N1-C9-C17	-74.13(16)
C11-S2-C8-C7	63.38(12)
C11-S2-C8-C9	176.77(9)
C11-C12-C13-C14	-0.5(2)
C12-C11-C16-C15	0.4(2)
C12-C13-C14-C15	1.1(3)
C13-C14-C15-C16	-0.9(3)
C14-C15-C16-C11	0.2(3)
C16-C11-C12-C13	-0.3(2)
C17-C18-C19-C20	0.4(3)
C18-C17-C22-C21	-0.6(2)
C18-C17-C22-C23	178.68(15)
C18-C19-C20-C21	-0.1(3)
C19-C20-C21-C22	-0.7(3)
C20-C21-C22-C17	1.0(2)
C20-C21-C22-C23	-178.34(16)
C22-C17-C18-C19	-0.1(2)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for **3b** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)