Supporting Information

Regioselective Intermolecular [2+2]-Cycloaddition of α-Iodo Unsaturated Ketones Promoted by Diisobutylaluminum Hydride

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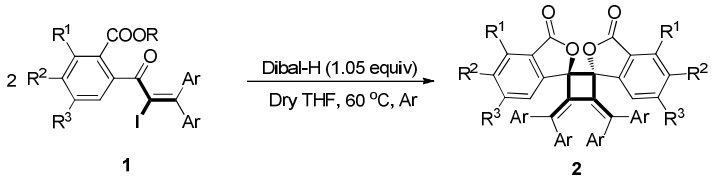
General Remarks

Column chromatography was carried out on silica gel (200-300 meshes). Conversion was monitored by thin layer chromatography (TLC). ^{1}H NMR spectra were recorded on 400 MHz in chloroform-d solution and ^{13}C NMR spectra were recorded on 100 MHz in chloroform-d solution. IR spectra were recorded on a FT-IR spectrometer and only major peaks are reported in cm^{-1}. All products were further characterized by high resolution mass spectra (HRMS); copies of their ^{1}H NMR and ^{13}C NMR spectra are provided in the Supporting Information. The commercial solvents were distilled over CaH_2.

Synthetic Procedures and Spectral Data

All starting materials were prepared according to previously reported procedures.[1]

Synthesis of substituted cyclobutane derivatives:



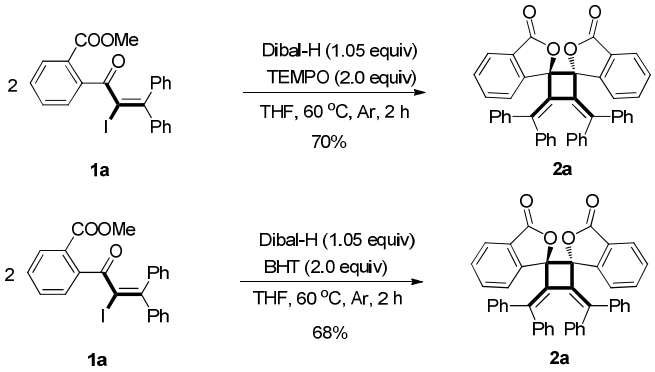
1 **2**

To a solution of 2-(2-iodo-3,3-diarylacryloyl)-benzoates (**1**) (0.20 mmol) in anhydrous THF (2.0 mL) was added 1.05 equiv diisobutylaluminum hydride (Dibal-H) under argon atmosphere. Subsequently, the reaction mixture was stirred for 2 h at 60 ^{o}C. When the reaction was completed, the reaction mixture was quenched by addition of saturated ammonium chloride solution and extracted with ethyl acetate (3 x 15 mL), washed with water, saturated brine, dried over Na_2SO_4 and evaporated under reduced pressure. The residue was purified by chromatography on silica gel to afford corresponding cyclobutane products **2**.

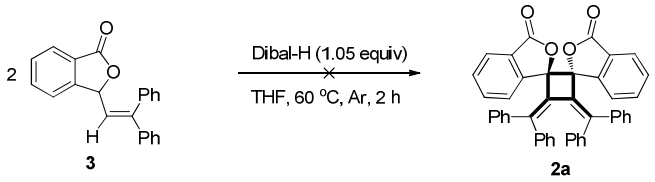
General procedure for inhibition experiments:

To a solution of methyl-2-(2-iodo-3,3-diphenylacryloyl)benzoate (**1a**) (0.20 mmol) and 2.0 equiv 2,2,6,6-tetramethylpiperidinooxy (TEMPO) or 2.0 equiv 2,6-di-tert-butyl-4-methylphenol (BHT) in anhydrous THF (2.0 mL) was added 1.05 equiv diisobutylaluminum hydride (Dibal-H) under argon atmosphere. Subsequently, the reaction mixture was stirred for 2 h at 60 ^{o}C. When the reaction was completed, the

reaction mixture was quenched by addition of saturated ammonium chloride solution and extracted with ethyl acetate (3 x 15 mL), washed with water, saturated brine, dried over Na_2SO_4 and evaporated under reduced pressure. The residue was purified by chromatography on silica gel to afford the corresponding cyclobutane product **2a** in 70% and 68% yield, respectively.



General procedure for mechanism investigation:[2]

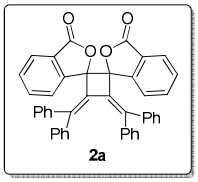


To a solution of 3-(2,2-diphenylvinyl)isobenzofuran-1(3H)-one (**3**) (0.20 mmol) in anhydrous THF (2.0 mL) was added 1.05 equiv diisobutylaluminum hydride (Dibal-H) under argon atmosphere. Subsequently, the reaction mixture was stirred for 2 h at 60 ^{o}C. When the reaction was determined by TLC analysis, the desired product **2a** was not obtained.

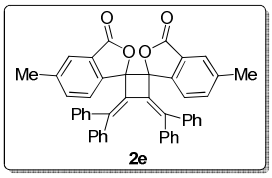
Reference:

(1) H.-T. Zhu, X. Dong, L.-J. Wang, M.-J. Zhong, X.-Y. Liu and Y.-M. Liang, *Chem.Commun.*, 2012, **48**, 10748;

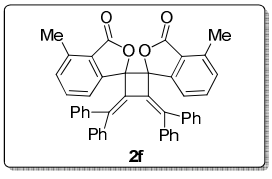
(2) H.-T. Zhu, M.-J. Fan, D.-S. Yang, X.-L. Wang, S. Ke, C.-Y. Zhang and Z.-H. Guan, *Org. Chem. Front.*, 2015, **2**, 506.



3,4-bis(diphenylmethylene)-3',3''*H*-dispiro[cyclobutane-1,1':2,1''-diisobenzofuran]-3',3''-dione (2a) The crude product was purified by flash chromatography (Hexanes:EtOAc = 4:1) to provide (90.5 mg, 73%) as a yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.87 (d, *J* = 8.0 Hz, 2H), 7.62 (t, *J* = 7.2 Hz, 2H), 7.55 (d, *J* = 7.6 Hz, 2H), 7.40 (t, *J* = 7.6 Hz, 2H), 7.11-7.08 (m, 2H), 7.01-6.96 (m, 10H), 6.84-6.79 (m, 8H). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm 168.4, 146.9, 141.6, 139.9, 138.7, 134.1, 132.6, 129.5, 129.4, 128.9, 128.3, 128.2, 127.8, 127.4, 126.2, 124.9, 124.3, 92.3. IR (neat, cm^{-1}): 3061, 1775, 1597, 1466, 1287, 1062, 972, 726. HRMS (ESI) Calcd for $C_{44}H_{28}NaO_4$: M+Na = 643.1880. Found: 643.1853.

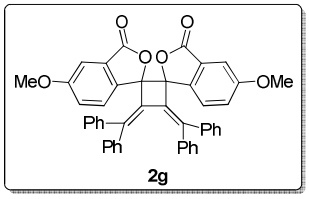


3,4-bis(diphenylmethylene)-5',5''-dimethyl-3',3''*H*-dispiro[cyclobutane-1,1':2,1''-diisobenzofuran]-3',3''-dione (2e) The crude product was purified by flash chromatography (Hexanes:EtOAc = 4:1) to provide (92.1 mg, 71%) as a yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.71 (d, *J* = 8.0 Hz, 2H), 7.41 (dd, *J* = 7.6, 0.8 Hz, 2H), 7.33 (d, *J* = 0.8 Hz, 2H), 7.12-7.08 (m, 2H), 7.01-6.95 (m, 10H), 6.84-6.79 (m, 8H), 2.38 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm 168.6, 144.4, 141.3, 140.0, 139.8, 138.8, 135.2, 132.8, 129.5, 129.0, 128.2, 128.1, 127.7, 127.4, 126.4, 124.8, 123.9, 92.3, 21.2. IR (neat, cm^{-1}): 3058, 1762, 1592, 1442, 1282, 1159, 984, 745. HRMS (ESI) Calcd for $C_{46}H_{32}NaO_4$: M+Na = 671.2193. Found: 671.2173.

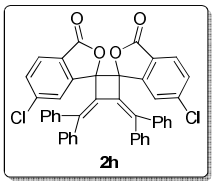


3,4-bis(diphenylmethylene)-4',4''-dimethyl-3',3''*H*-dispiro[cyclobutane-1,1':2,1''-diisobenzofuran]-3',3''-dione (2f) The crude product was purified by flash chromatography (Hexanes:EtOAc = 4:1) to provide (68.7 mg, 53%) as a yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.72 (d, *J* = 7.6 Hz, 2H), 7.52 (t, *J* = 7.6 Hz, 2H), 7.18-7.12 (m, 4H), 7.03-6.98 (m, 5H), 6.95-6.81 (m, 5H), 6.79-6.76 (m, 8H), 2.44 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm 168.5, 148.2, 140.9, 139.9, 138.9, 138.7, 133.8, 133.0, 130.8, 129.6, 128.9, 128.1, 128.0, 127.5, 127.3, 123.6, 121.6,

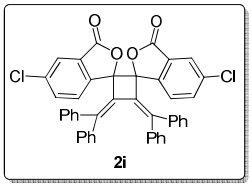
91.1, 17.2. IR (neat, cm^{-1}): 3037, 1759, 1590, 1432, 1292, 1133, 964, 765. HRMS (ESI) Calcd for $C_{46}H_{32}NaO_4$: M+Na = 671.2193. Found: 671.2198.



3,4-bis(diphenylmethylene)-5',5''-dimethoxyl-3',3''*H*-dispiro[cyclobutane-1,1':2,1''-diisobenzofunan]-3',3''-dione (2g) The crude product was purified by flash chromatography (Hexanes:EtOAc = 2:1) to provide (87.1 mg, 64%) as a yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.66 (d, *J* = 8.4 Hz, 2H), 7.14-7.11 (m, 4H), 7.09-6.95 (m, 12H), 6.84-6.80 (m, 8H), 3.78 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm 168.5, 160.9, 141.6, 139.9, 138.9, 138.8, 133.0, 129.5, 129.0, 128.2, 128.1, 127.8, 127.7, 127.4, 125.3, 122.6, 107.0, 92.6, 55.7. IR (neat, cm^{-1}): 3056, 1773, 1619, 1491, 1280, 1022, 981, 734. HRMS (ESI) Calcd for $C_{46}H_{32}$ NaO_6: M+Na = 703.2091. Found: 703.2100.

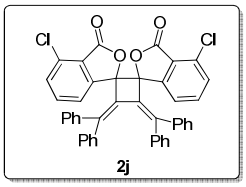


3,4-bis(diphenylmethylene)-6',6''-dichloro-3',3''*H*-dispiro[cyclobutane-1,1':2,1''-diisobenzofunan]-3',3''-dione (2h) The crude product was purified by flash chromatography (Hexanes:EtOAc = 4:1) to provide (100.5 mg, 73%) as a yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.76-7.74 (m, 2 H), 7.43 (dd, *J* = 8.4, 3.6 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.12-7.09 (m, 2H), 7.05-6.94 (m, 10H), 6.88-6.80 (m, 8H). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm 167.1, 147.6, 142.6, 140.7, 139.5, 138.4, 132.0, 130.4, 129.6, 129.0, 128.6, 128.5, 127.9, 127.4, 126.1, 124.9, 124.8, 92.0. IR (neat, cm^{-1}): 3057, 1775, 1591, 1443, 1236, 1071, 986, 741. HRMS (ESI) Calcd for $C_{44}H_{26}Cl_2NaO_4$: M+Na = 711.1100. Found: 711.1109.

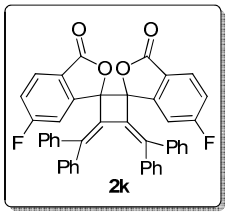


3,4-bis(diphenylmethylene)-5',5''-dichloro-3',3''*H*-dispiro[cyclobutane-1,1':2,1''-diisobenzofunan]-3',3''-dione (2i) The crude product was purified by flash chromatography (Hexanes:EtOAc = 4:1) to provide (77.1 mg, 56%) as a yellow

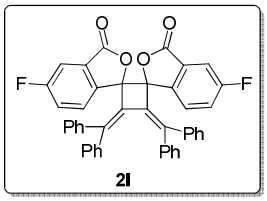
solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.72 (d, *J* = 8.4 Hz, 2H), 7.52 (dd, *J* = 8.0 Hz, 2.0 Hz, 2H), 7.47 (d, *J* = 1.6 Hz, 2H), 7.12 (t, *J* = 7.6 Hz, 2H), 7.04-6.94 (m, 10H), 6.84-6.80 (m, 8H). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm 166.8, 144.0, 142.5, 139.5, 138.5, 135.9, 134.2, 132.2, 129.5, 129.0, 128.5, 128.2, 127.9, 127.4, 125.8, 124.7, 92.5. IR (neat, cm^{-1}): 3057, 1778, 1592, 1443, 1244, 1074, 982, 736. HRMS (ESI) Calcd for $C_{44}H_{26}Cl_2NaO_4$: M+Na = 711.1100. Found: 711.1077.



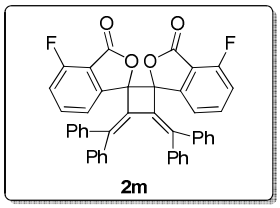
3,4-bis(diphenylmethylene)-4',4''-dichloro-3',3''*H*-dispiro[cyclobutane-1,1':2,1''-diisobenzofunan]-3',3''-dione (2j) The crude product was purified by flash chromatography (Hexanes:EtOAc = 4:1) to provide (93.4 mg, 67%) as a yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.74 (d, *J* = 7.6 Hz, 2H), 7.50 (t, *J* = 7.6 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 7.14 (t, *J* = 7.6 Hz, 2H), 7.05 (d, *J* = 7.6 Hz, 4H), 7.01 (d, *J* = 7.2 Hz, 2H), 6.98-6.92 (m, 4H), 6.82-6.78 (m, 8H). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm 165.1, 148.4, 142.4, 139.6, 138.4, 134.9, 132.5, 132.4, 130.8, 129.6, 128.9, 128.6, 128.5, 127.7, 127.3, 123.1, 123.0, 91.2. IR (neat, cm^{-1}): 3032, 1770, 1583, 1442, 1240, 1068, 980, 745. HRMS (ESI) Calcd for $C_{44}H_{26}Cl_2NaO_4$: M+Na = 711.1100. Found: 711.1086.



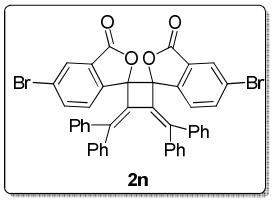
3,4-bis(diphenylmethylene)-6',6''-difluoro-3',3''*H*-dispiro[cyclobutane-1,1':2,1''-diisobenzofunan]-3',3''-dione (2k) The crude product was purified by flash chromatography (Hexanes:EtOAc = 4:1) to provide (94.9 mg, 72%) as a yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.52 (dd, *J* = 8.4, 4.8 Hz, 2H), 7.47 (dd, *J* = 7.6, 2.0 Hz, 2H), 7.14-7.05 (m, 10 H), 7.03-6.93 (m, 4 H), 6.86-6.80 (m, 8 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm 167.0, 166.2 (d, *J* = 255.4 Hz), 149.1 (d, *J* = 10.5 Hz), 142.5, 139.5, 138.5, 129.6, 129.0, 128.53, 128.47, 127.9, 127.4, 117.9 (d, *J* = 24.2 Hz), 111.7 (d, *J* = 14.7 Hz), 91.8. IR (neat, cm^{-1}): 3058, 1776, 1603, 1482, 1273, 1075, 992, 731. HRMS (ESI) Calcd for $C_{44}H_{26}F_2NaO_4$: M+Na = 679.1691. Found: 679.1697.



3,4-bis(diphenylmethylene)-5',5''-difluoro-3',3''*H*-dispiro[cyclobutane-1,1':2,1''-diisobenzofunan]-3',3''-dione (2l) The crude product was purified by flash chromatography (Hexanes:EtOAc = 4:1) to provide (74.9 mg, 57%) as a yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.75 (q, *J* = 4.0 Hz, 2H), 7.26 (dd, *J* = 6.4, 2.4 Hz, 2H), 7.25-7.16 (m, 2H), 7.15-7.08 (m, 2H), 7.03-6.95 (m, 10H), 6.86-6.81 (m, 8H). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm 167.0 (d, *J* = 4.0 Hz), 163.2 (d, *J* = 249.5 Hz), 142.5, 141.2, 139.6, 138.6, 129.6, 129.0, 128.5, 128.4, 127.8, 127.4, 121.9 (d, *J* = 23.8 Hz), 111.1 (d, *J* = 23.7 Hz), 92.7. IR (neat, cm^{-1}): 3075, 1768, 1599, 1485, 1263, 1093, 910, 747. HRMS (ESI) Calcd for $C_{44}H_{26}F_2NaO_4$: M+Na = 679.1691. Found: 679.1686.

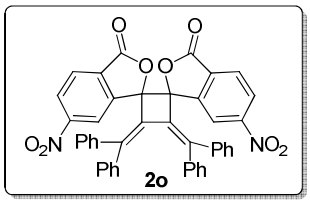


3,4-bis(diphenylmethylene)-4',4''-difluoro-3',3''*H*-dispiro[cyclobutane-1,1':2,1''-diisobenzofunan]-3',3''-dione (2m) The crude product was purified by flash chromatography (Hexanes:EtOAc = 4:1) to provide (87.1 mg, 66%) as a yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.61-7.55 (m, 4H), 7.16-7.12 (m, 2H), 7.05 (d, *J* = 8.0 Hz, 4H), 7.08-6.97 (m, 3H), 6.96-6.93 (m, 5H), 6.85-6.79 (m, 8H). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm 164.2 (d, *J* = 2.7 Hz), 158.7 (d, *J* = 263.3 Hz), 148.4, 142.4, 139.6, 138.5, 129.6, 129.0, 128.6, 128.5, 127.8, 127.4, 120.6 (d, *J* = 4.3 Hz), 116.4 (d, *J* = 18.5 Hz), 114.1 (d, *J* = 14.3 Hz), 92.2. IR (neat, cm^{-1}): 3053, 1747, 1586, 1453, 1244, 1090, 913, 745. HRMS (ESI) Calcd for $C_{44}H_{26}F_2NaO_4$: M+Na = 679.1691. Found: 679.1697.

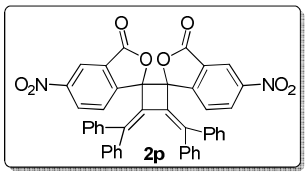


3,4-bis(diphenylmethylene)-5',5''-dibromo-3',3''*H*-dispiro[cyclobutane-1,1':2,1''-diisobenzofunan]-3',3''-dione (2n) The crude product was purified by flash chromatography (Hexanes:EtOAc = 4:1) to provide (122.4 mg, 78%) as a yellow

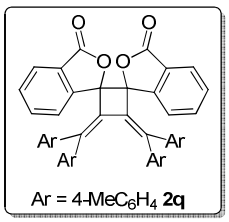
solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.69-7.63 (m, 6H), 7.13 (t, *J* = 7.6 Hz, 2H), 7.05-6.94 (m, 10H), 6.82 (t, *J* = 8.0 Hz, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm 166.6, 144.6, 142.5, 139.5, 138.5, 137.1, 132.1, 129.6, 129.0, 128.6, 128.5, 128.4, 127.9, 127.8, 127.4, 126.0, 123.7, 92.5. IR (neat, cm^{-1}): 3062, 1768, 1598, 1417, 1208, 1100, 987, 742. HRMS (ESI) Calcd for $C_{44}H_{26}Br_2Na\ O_4$: M+Na = 799.0090. Found: 799.0101.



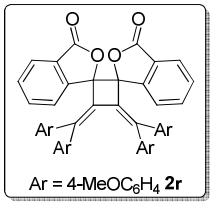
3,4-bis(diphenylmethylene)-6',6''-dinitro-3',3''*H*-dispiro[cyclobutane-1,1':2,1''-diisobenzofunan]-3',3''-dione (2o) The crude product was purified by flash chromatography (Hexanes:EtOAc = 3:1) to provide (78.4 mg, 54%) as a yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 8.55 (s, 2H), 8.17 (d, *J* = 8.4 Hz, 2H), 7.61 (d, *J* = 8.4 Hz, 2H), 7,07-6.91(m, 16H), 6.83 (t, *J* = 7.6 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm 165.7, 151.3, 146.1, 144.0, 139.1, 138.3, 131.3, 131.2, 129.7, 129.1, 129.0, 128.7, 128.0, 127.4, 126.1, 125.0, 120.3, 92.7. IR (neat, cm^{-1}): 3058, 1780, 1535, 1337, 1241, 1037, 990, 702. HRMS (ESI) Calcd for $C_{44}H_{26}N_2NaO_8$: M+Na = 733.1581. Found: 733.1602.



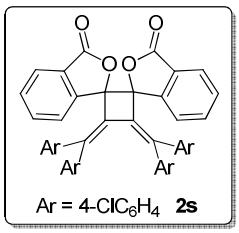
3,4-bis(diphenylmethylene)-5',5''-dinitro-3',3''*H*-dispiro[cyclobutane-1,1':2,1''-diisobenzofunan]-3',3''-dione (2p) The crude product was purified by flash chromatography (Hexanes:EtOAc = 3:1) to provide (80.7 mg, 56%) as a yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 8.41 (dd, *J* = 8.4, 2.0 Hz, 2H), 8.27 (d, *J* = 1.6 Hz, 2H), 7.97 (d, *J* = 8.8 Hz, 2H), 7.11-7.08 (m, 2H), 7.04-6.99 (m, 6H), 6.93 (d, *J* = 7.6 Hz, 4H), 6.88-6.81 (m, 8H). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm 165.5, 150.3, 149.1, 143.9, 139.1, 138.3, 131.3, 129.7, 129.1, 129.0, 128.9, 128.6, 128.2, 128.1, 127.5, 126.2, 120.4, 92.9. IR (neat, cm^{-1}): 3080, 1785, 1614, 1527, 1444, 1242, 987, 727. HRMS (ESI) Calcd for $C_{44}H_{26}N_2NaO_8$: M+Na = 733.1581. Found: 733.1585.



3,4-bis(di-*p*-tolylmethylene)-3',3''*H*-dispiro[cyclobutane-1,1':2,1''-diisobenzofuran]-3',3''-dione (2q) The crude product was purified by flash chromatography (Hexanes:EtOAc = 4:1) to provide (113.8 mg, 84%) as a yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.84 (d, *J* = 7.6 Hz, 2H), 7.61-7.57 (m, 2H), 7.53 (d, *J* = 7.6 Hz, 2H), 7.40-7.37 (m, 2H), 6.81-6.76 (m, 8H), 6.67 (d, *J* = 7.6 Hz, 4H), 6.58 (d, *J* = 8.0 Hz, 4H), 2.18 (s, 6H), 2.15 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm 168.7, 147.2, 141.0, 138.1, 138.0, 137.4, 136.1, 133.9, 131.8, 129.5, 129.2, 129.0, 128.3, 127.8, 126.3, 124.7, 124.5, 92.5, 21.1, 21.0. IR (neat, cm^{-1}): 3019, 1772, 1610, 1509, 1284, 1065, 974, 726. HRMS (ESI) Calcd for $C_{48}H_{36}NaO_4$: M+Na = 699.2506. Found: 699.2499.

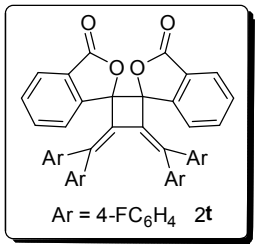


3,4-bis(di-(4-methoxyphenyl)methylene)-3',3''*H*-dispiro[cyclobutane-1,1':2,1''-diisobenzofunan]-3',3''-dione (2r) The crude product was purified by flash chromatography (Hexanes:EtOAc = 1:1) to provide (117.3 mg, 79%) as a yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.79 (d, *J* = 8.0 Hz, 2H), 7.56 (t, *J* = 7.6 Hz, 2H), 7.51 (d, *J* = 7.6 Hz, 2H), 7.35 (t, *J* = 7.6 Hz, 2H), 6.85 (d, *J* = 8.8 Hz, 4H), 6.75 (d, *J* = 8.4 Hz, 4H), 6.48 (d, *J* = 8.8 Hz, 4H), 6.33 (d, *J* = 9.2 Hz, 4H), 3.68 (s, 6H), 3.67 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm 168.8, 160.1, 159.4, 139.9, 133.8, 132.9, 131.4, 131.0, 130.6, 129.2, 126.6, 124.7, 113.2, 112.5, 92.9, 55.2, 55.1. IR (neat, cm^{-1}): 3024, 1769, 1594, 1463, 1234, 1047, 969, 721. HRMS (ESI) Calcd for $C_{48}H_{36}NaO_8$: M+Na = 763.2302. Found: 763.2296.



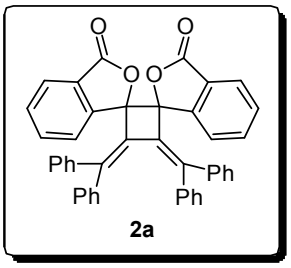
3,4-bis(di-(4-chlorophenyl)methylene)-3',3''*H*-dispiro[cyclobutane-1,1':2,1''-diisobenzofunan]-3',3''-dione (2s) The crude product was purified by flash chromatography (Hexanes:EtOAc = 4:1) to provide (107.8 mg, 71%) as a yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.72 (d, *J* = 8.0 Hz, 2H), 7.59-7.54 (m, 4H), 7.40 (t, *J* = 7.6 Hz, 2H), 6.94-6.85 (m, 12H), 6.73 (d, *J* = 8.0 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm 168.1, 144.8, 140.1, 137.7, 136.4, 135.1, 134.9, 134.4, 134.2, 130.7, 130.4, 129.9, 128.1, 128.0, 126.6, 125.1, 124.7, 92.6. IR (neat, cm^{-1}): 3063,

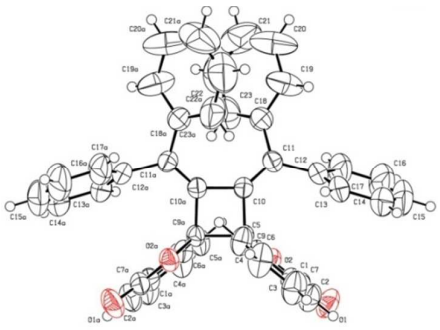
1778, 1593, 1488, 1283, 1092, 971, 785. HRMS (ESI) Calcd for $C_{44}H_{24}O_4NaCl_4$: M+Na = 779.0321. Found: 779.0336.



3,4-bis(di-(4-fluorophenyl)methylene)-3',3''*H*-dispiro[cyclobutane-1,1':2,1''-diiso benzofunan]-3',3''-dione (2t) The crude product was purified by flash chromatography (Hexanes:EtOAc = 4:1) to provide (124.1 mg, 89%) as a yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.74 (d, *J* = 8.0 Hz, 2H), 7.57-7.54 (m, 4H), 7.41-7.37 (m, 2H), 6.98-6.95 (m, 4H), 6.77-6.76 (m, 4H), 6.67-6.60 (m, 8H). ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm 168.2, 162.9 (d, *J* = 249.2 Hz), 162.7 (d, *J* = 248.5 Hz), 145.1, 139.9, 135.7, 135.6, 134.4, 134.3, 134.1, 133.5, 131.2 (d, *J* = 8.2 Hz), 130.9 (d, *J* = 8.4 Hz), 129.8, 128.5, 126.9, 126.5, 125.1, 124.7, 114.9 (d, *J* = 21.7 Hz), 114.7 (d, *J* = 21.8 Hz), 92.7. IR (neat, cm^{-1}): 2934, 1767, 1590, 1499, 1220, 1147, 976, 731. HRMS (ESI) Calcd for $C_{44}H_{24}O_4NaF_4$: M+Na = 715.1503. Found: 715.1510.

Crystallographic data of 2a





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alpha=90 beta=90 gamma=90

Temperature: 291 K

	Calculated	Reported
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Hall group	-P 2n 2ab	-P 2n 2ab
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Correction method= # Reported T Limits: Tmin=0.929 Tmax=1.000 AbsCorr = MULTI-SCAN

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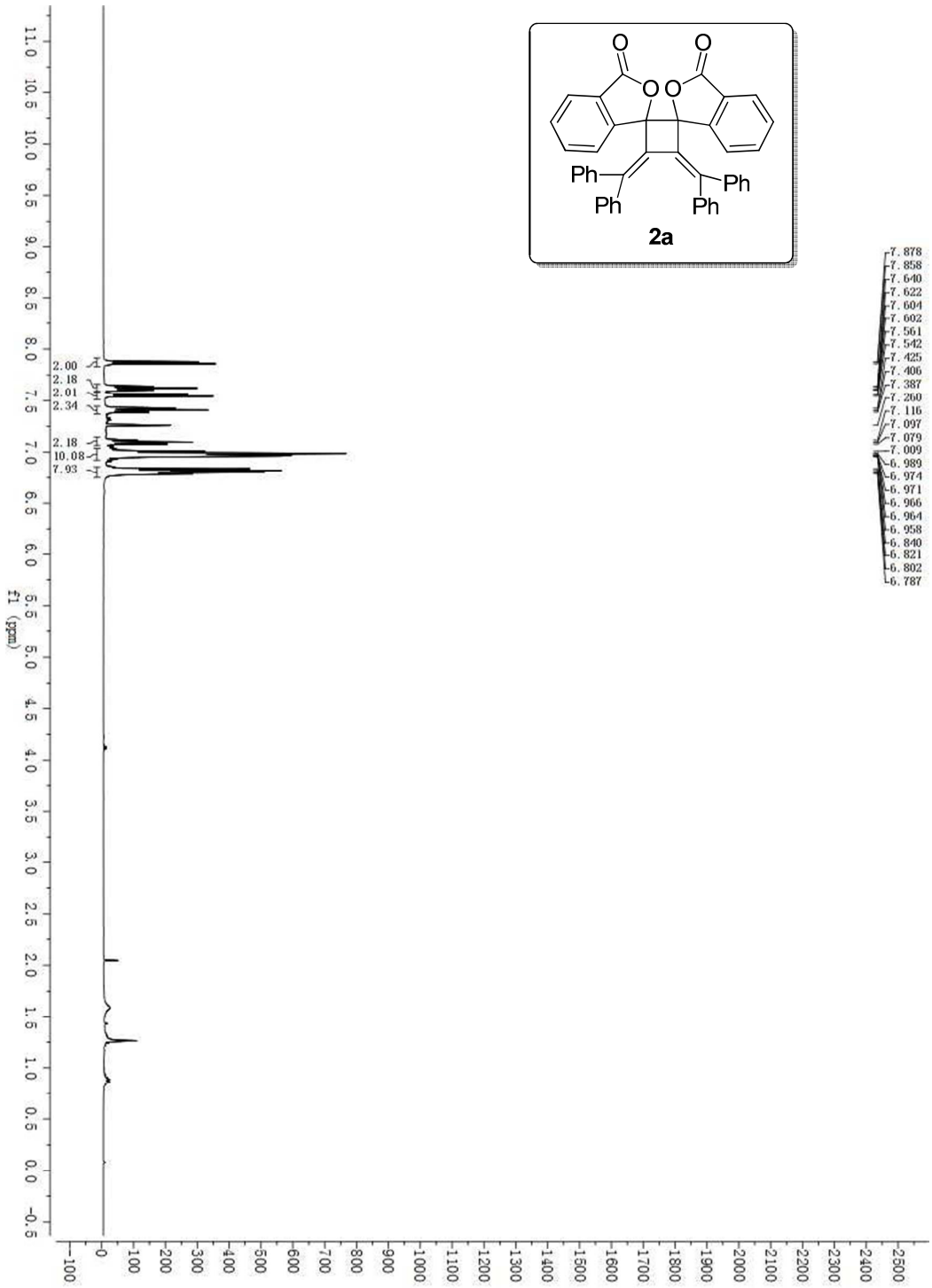
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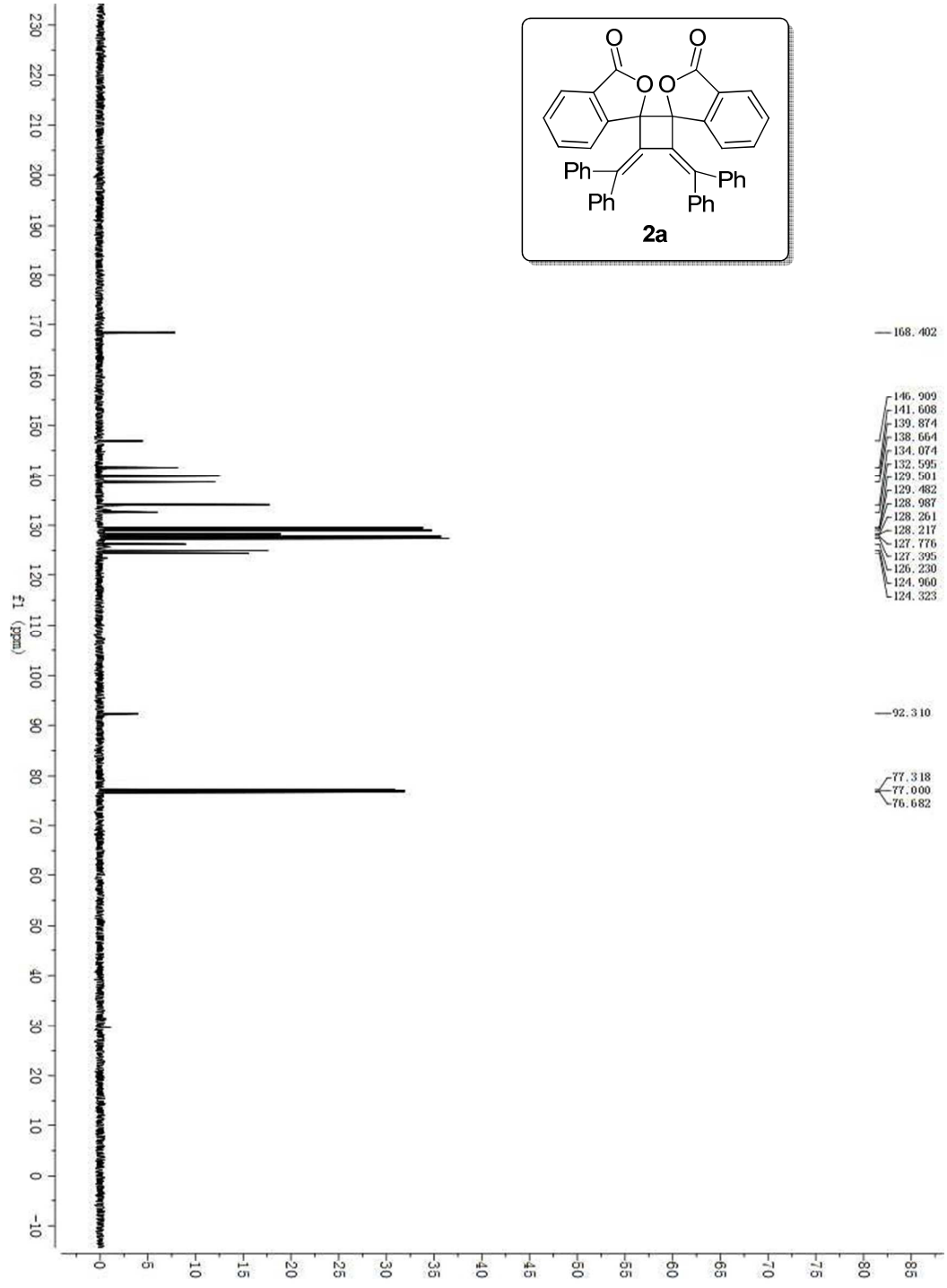
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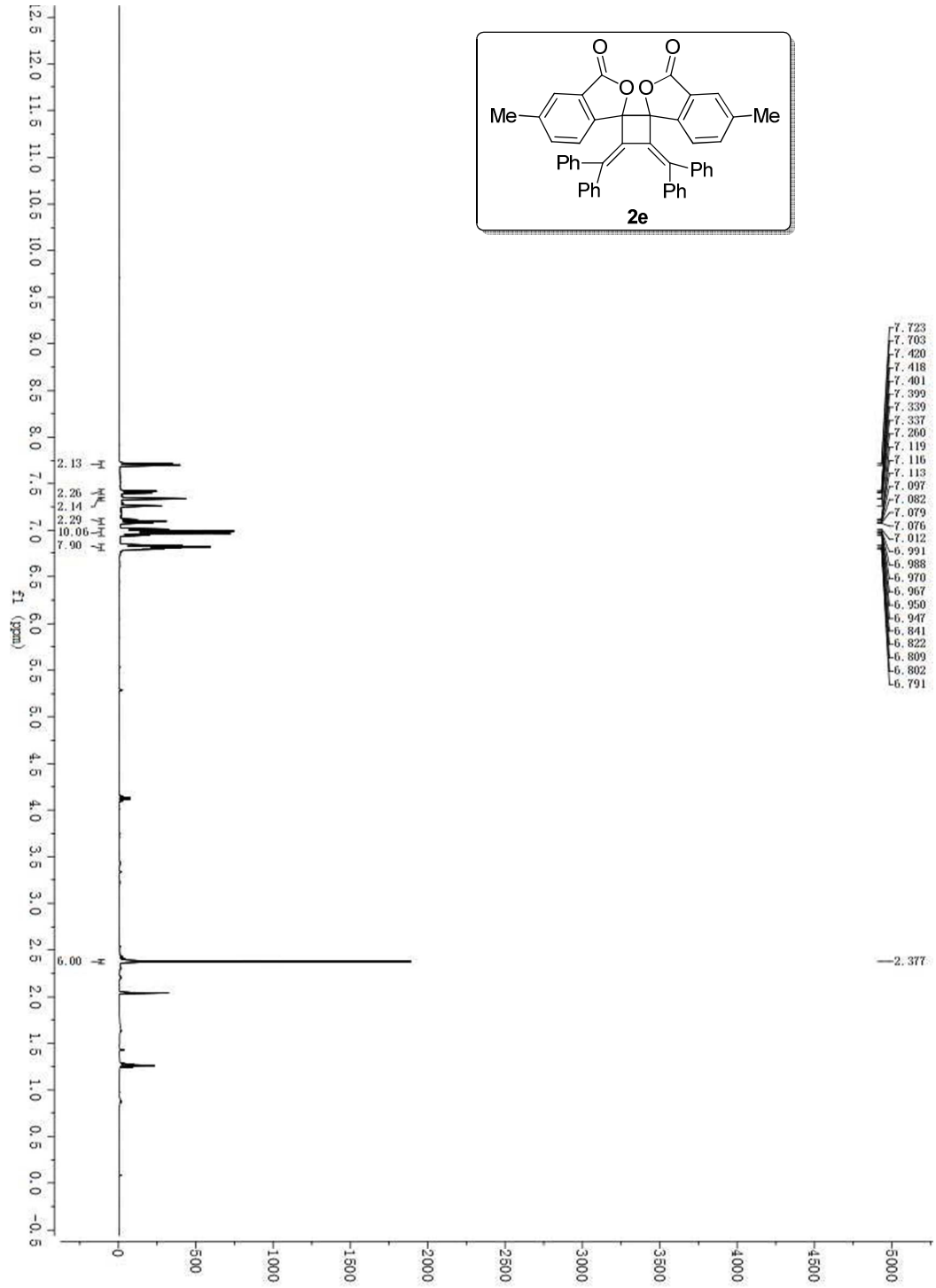
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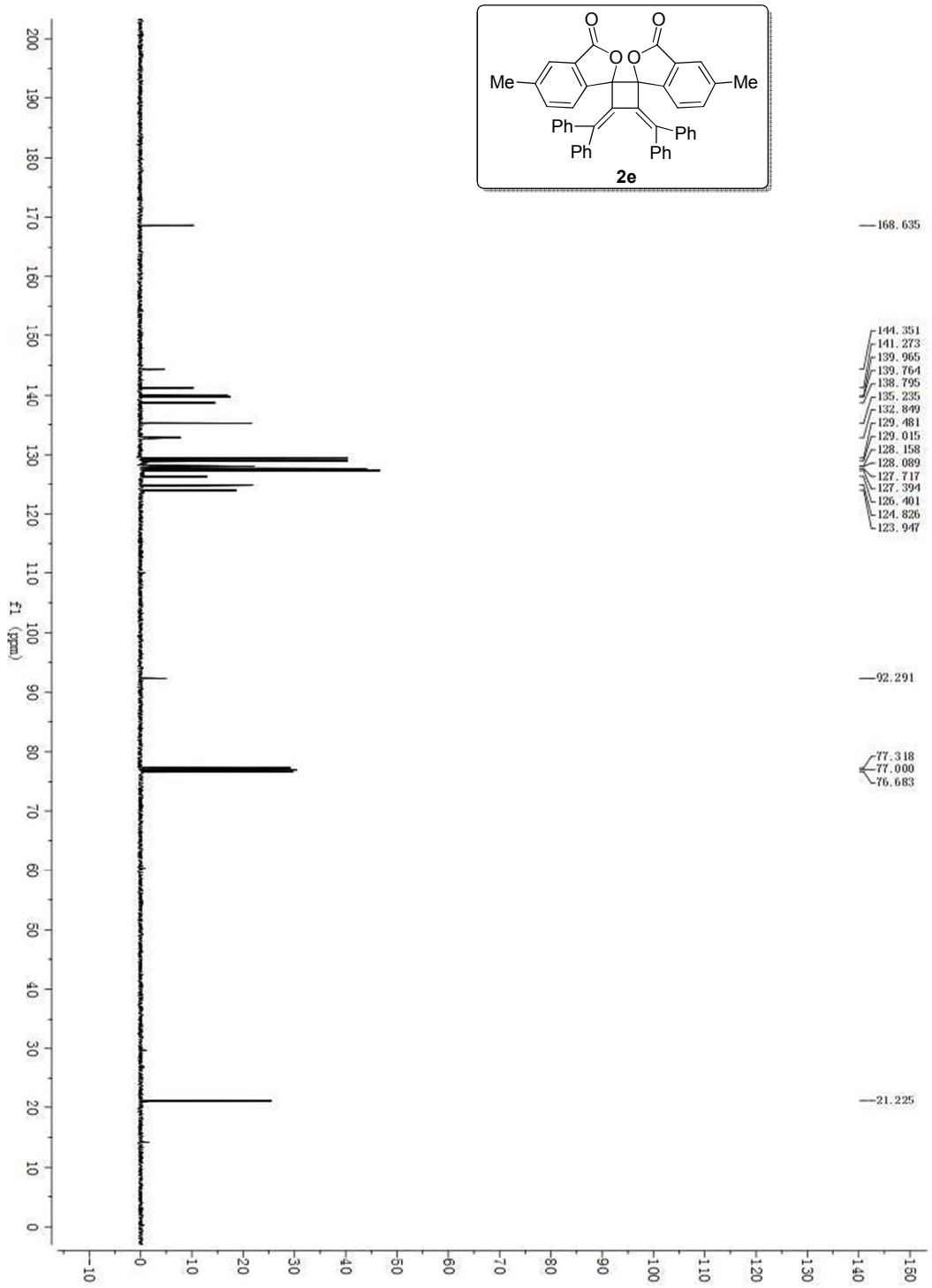
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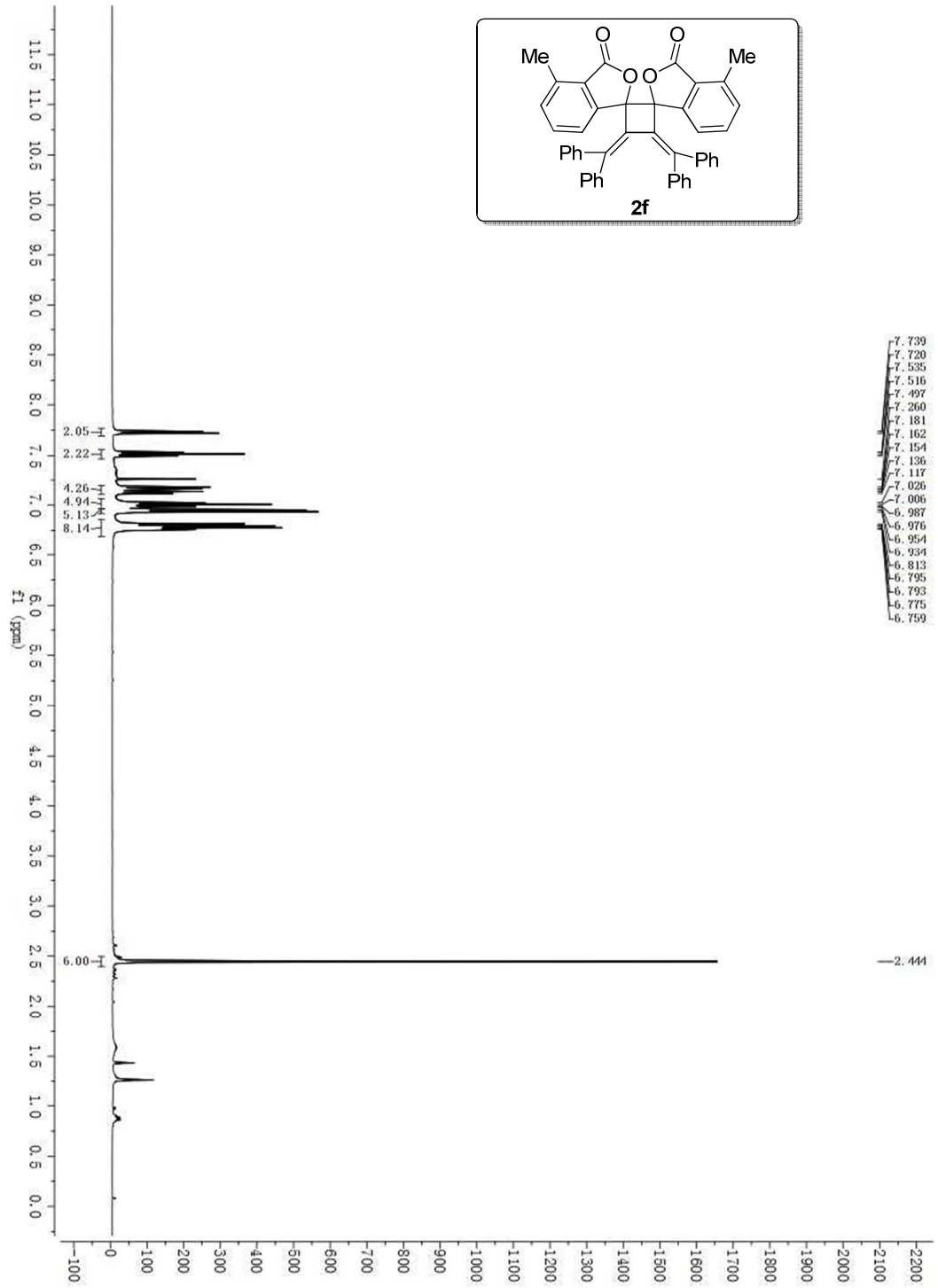
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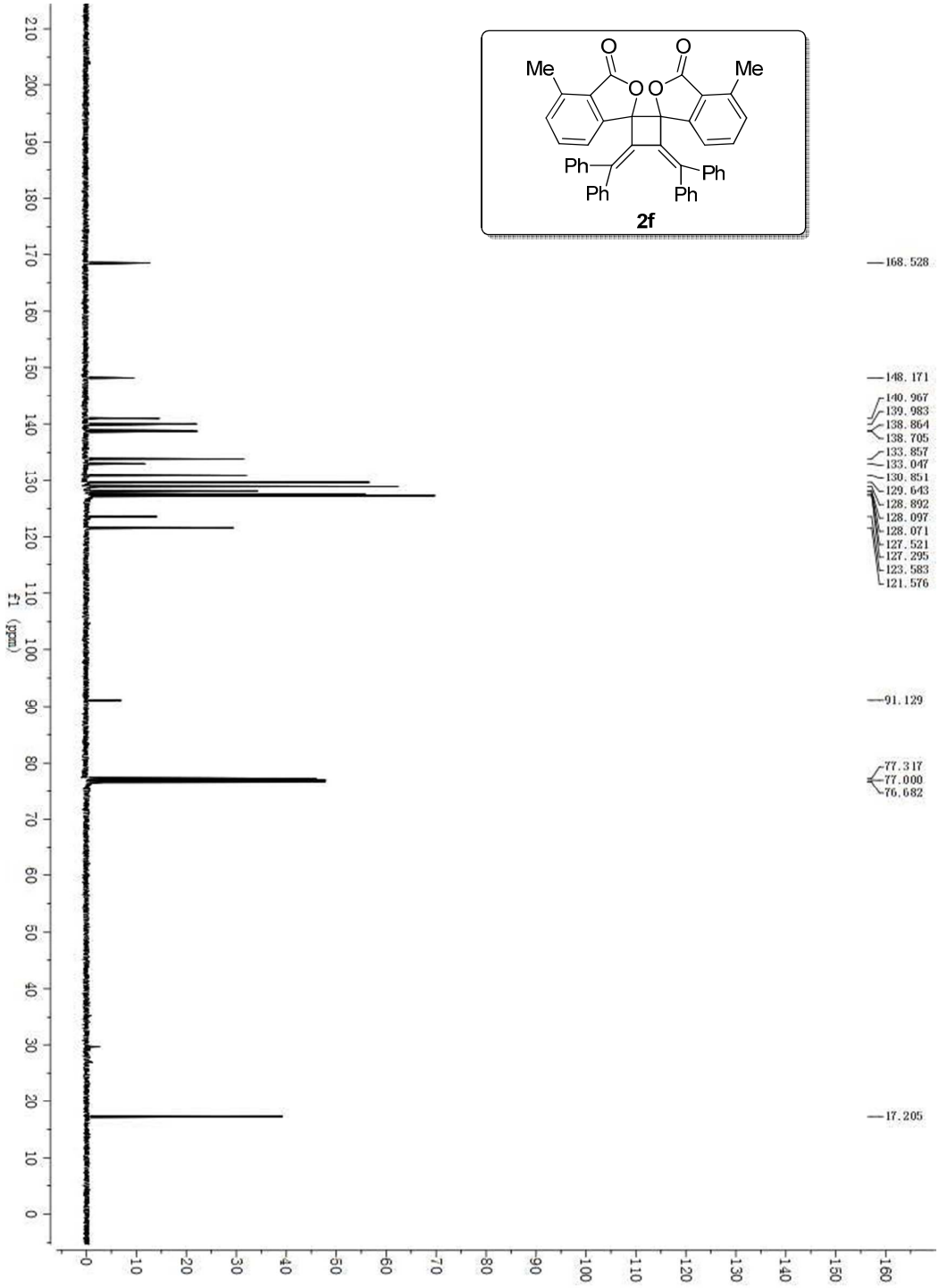
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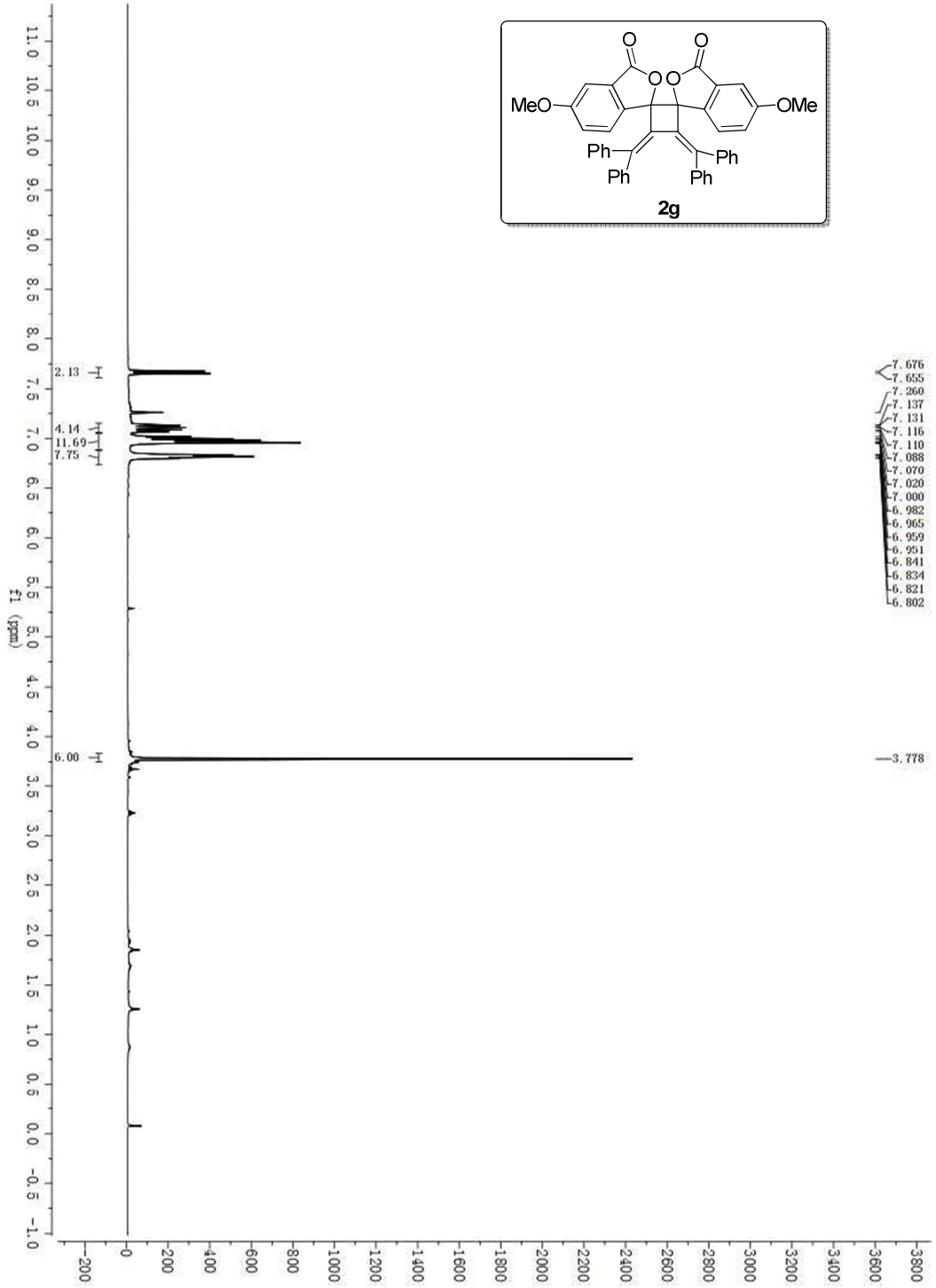
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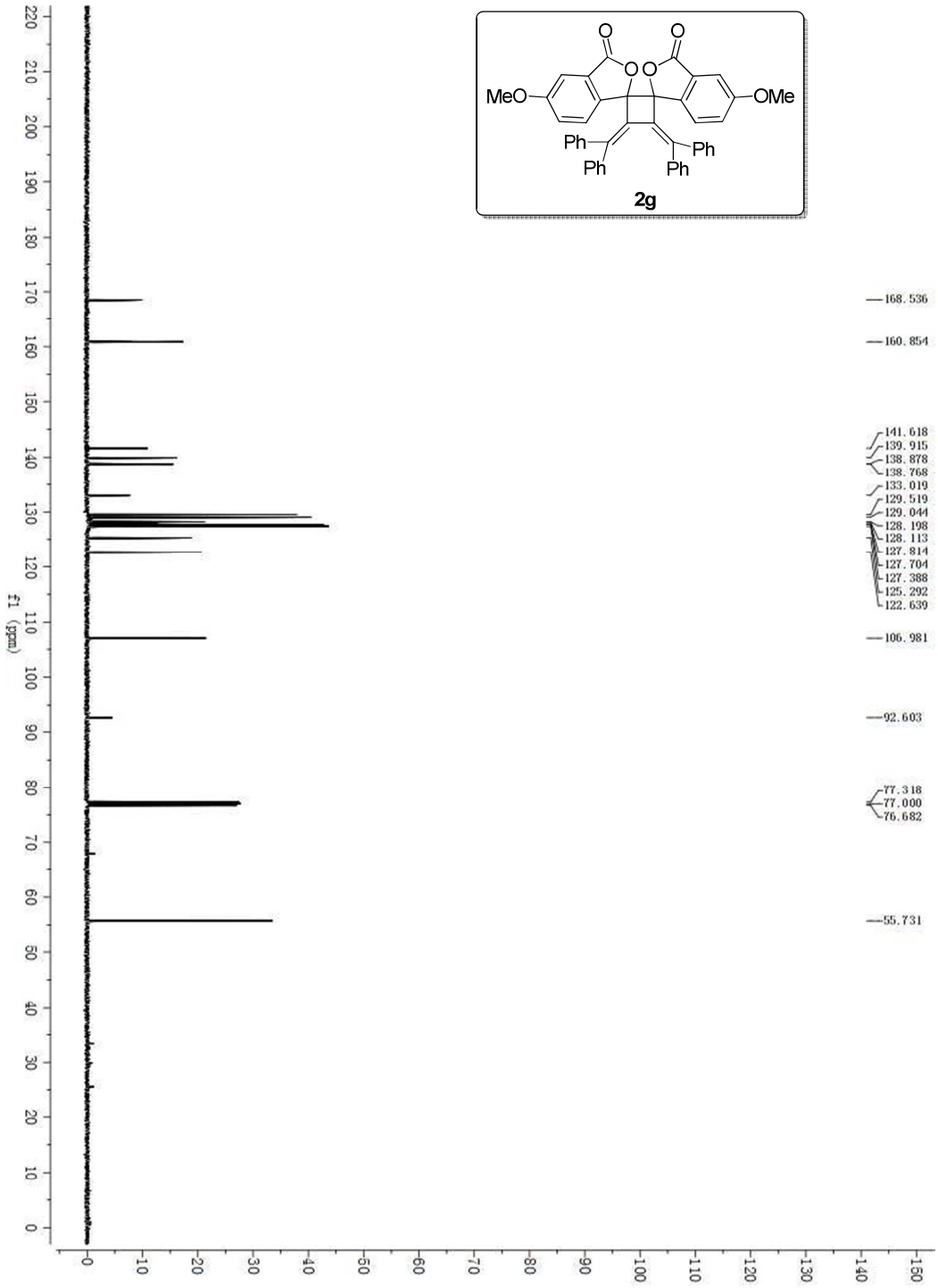
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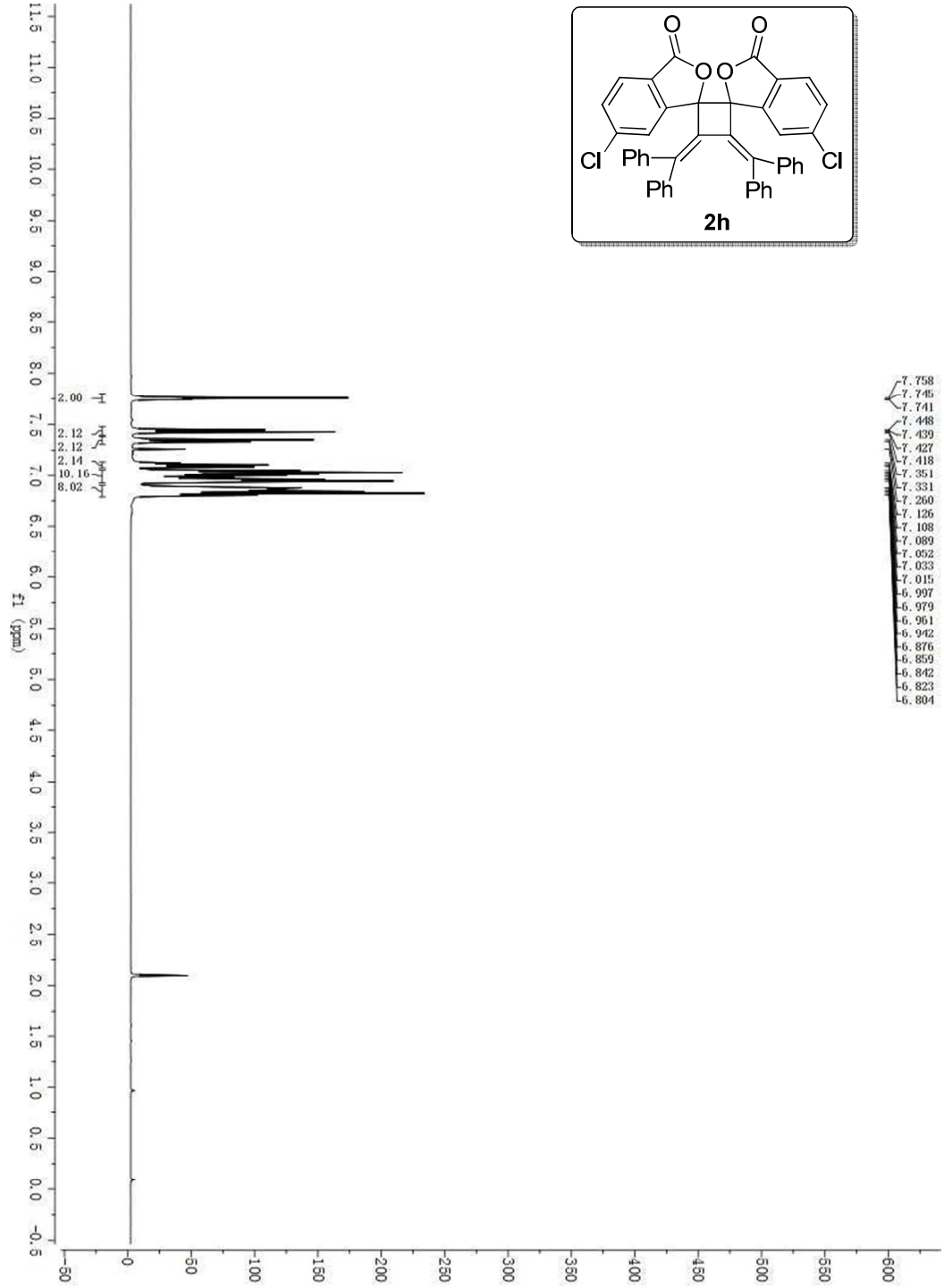
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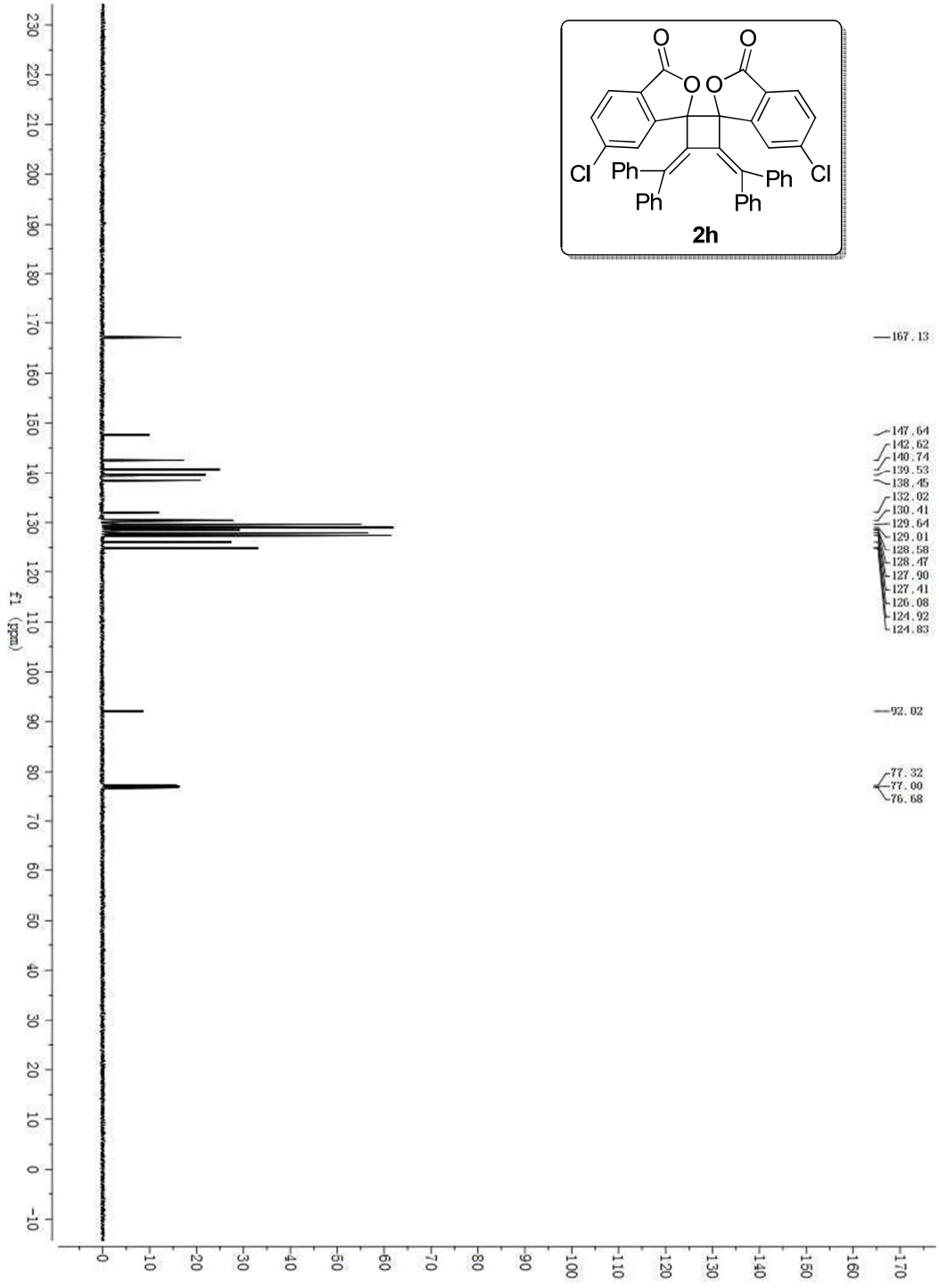
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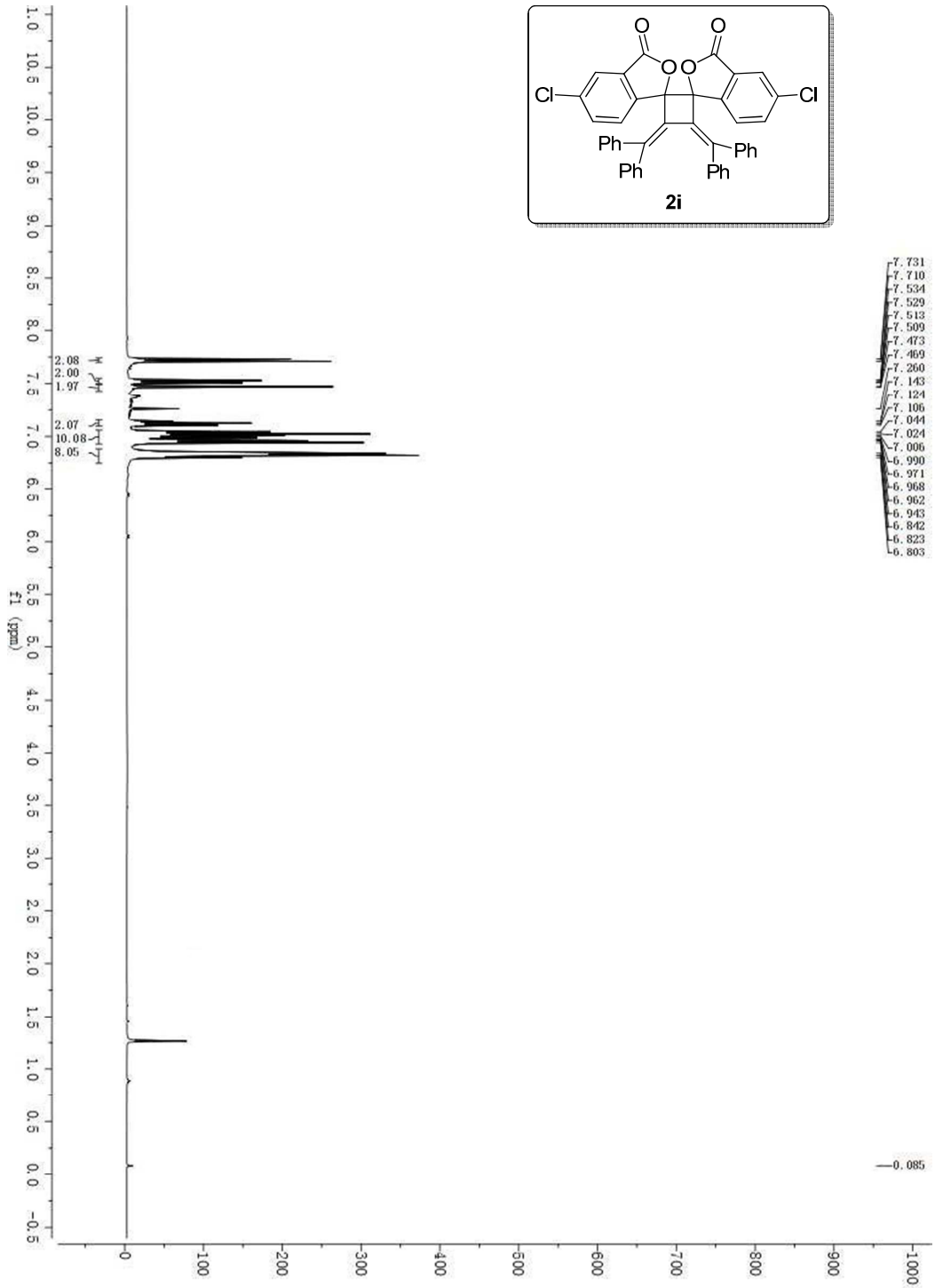
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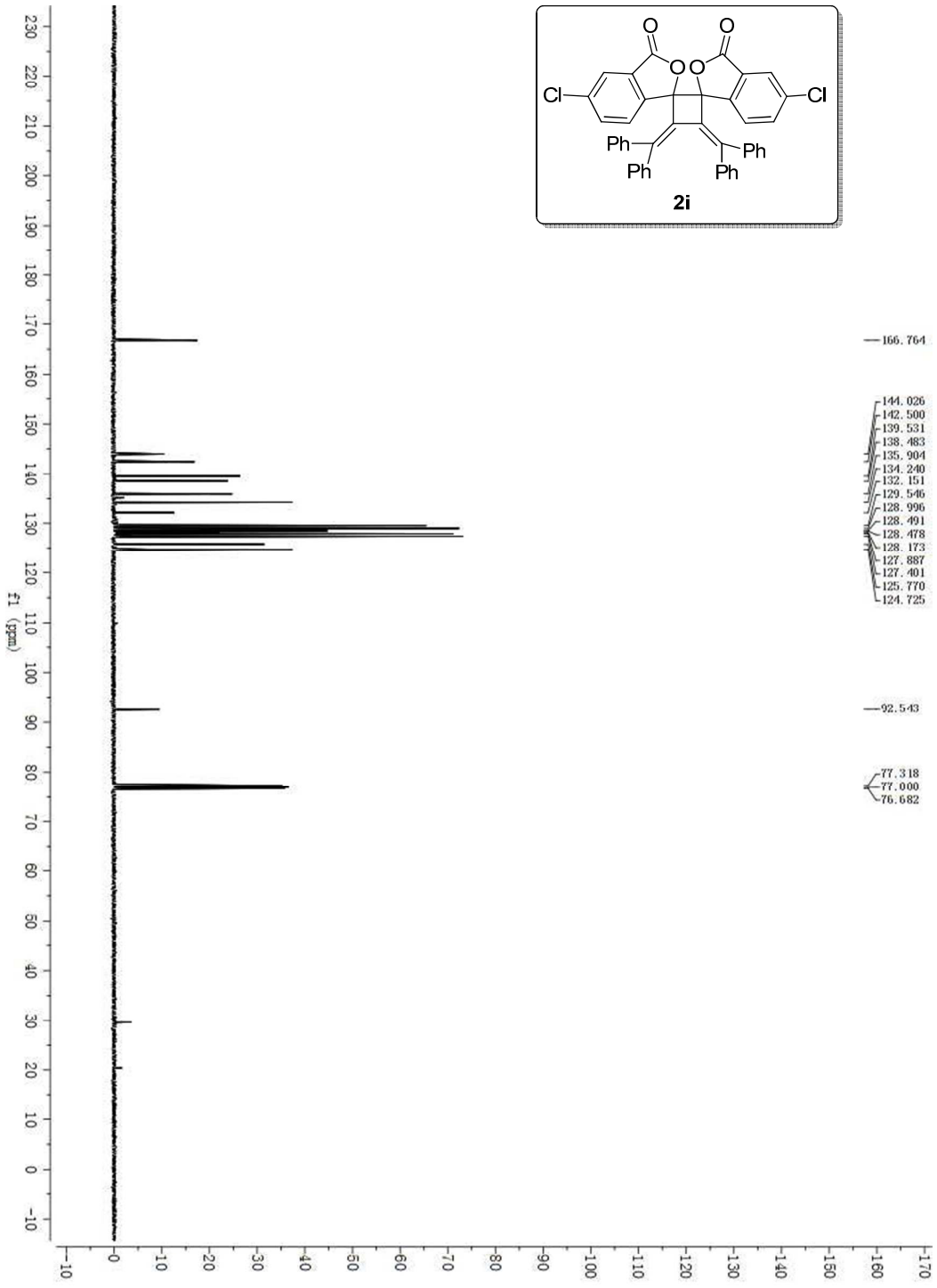
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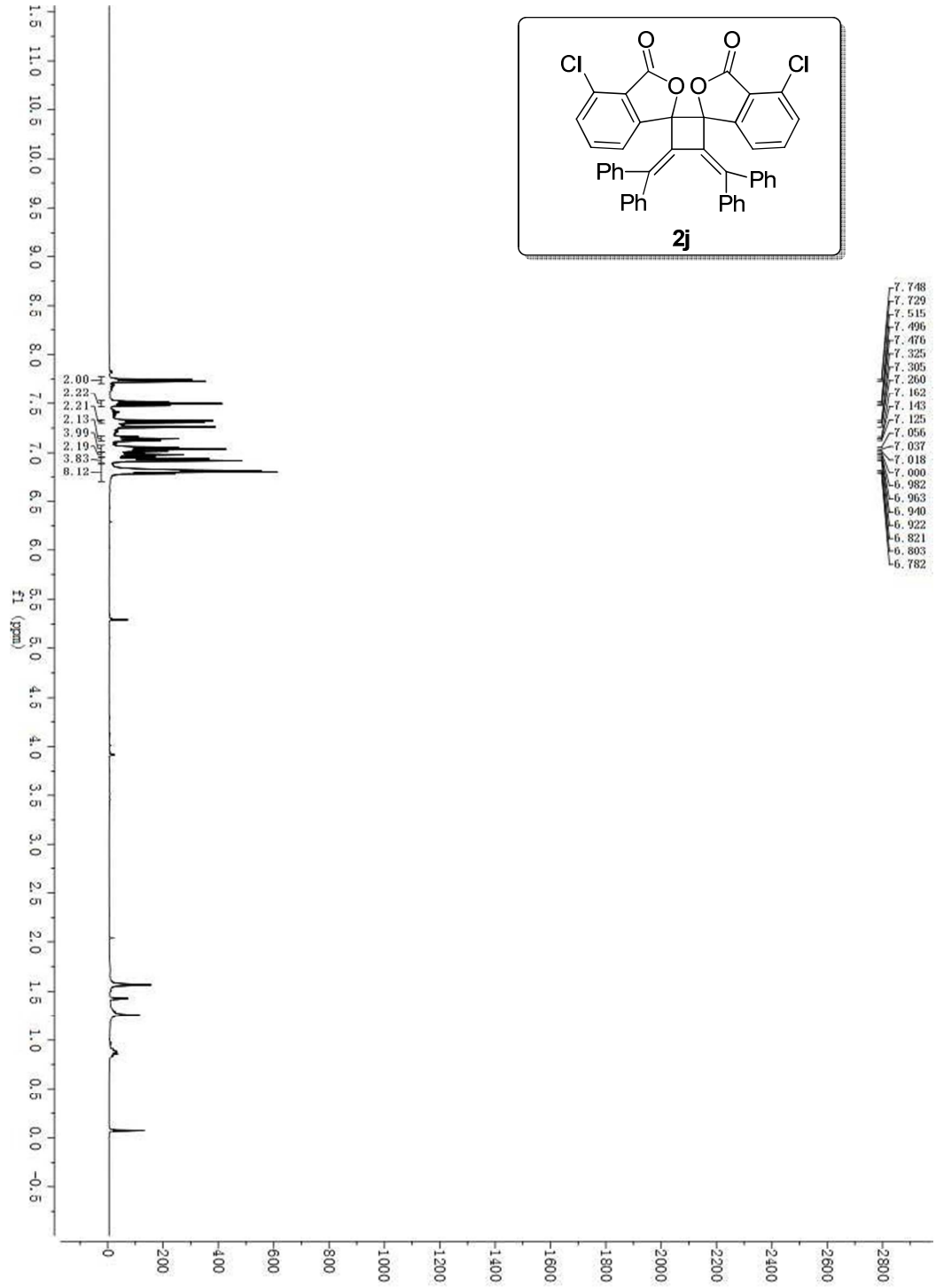
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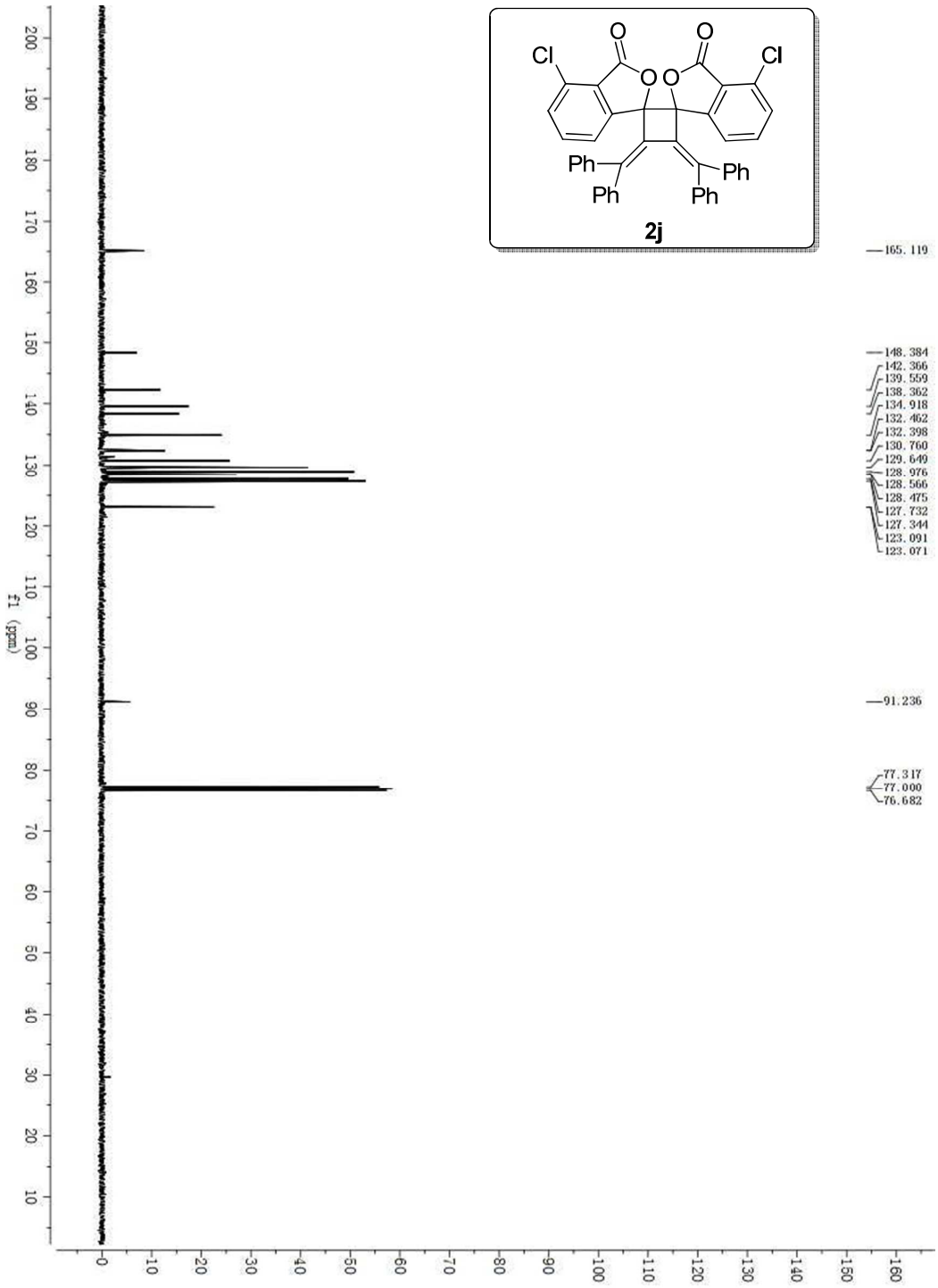
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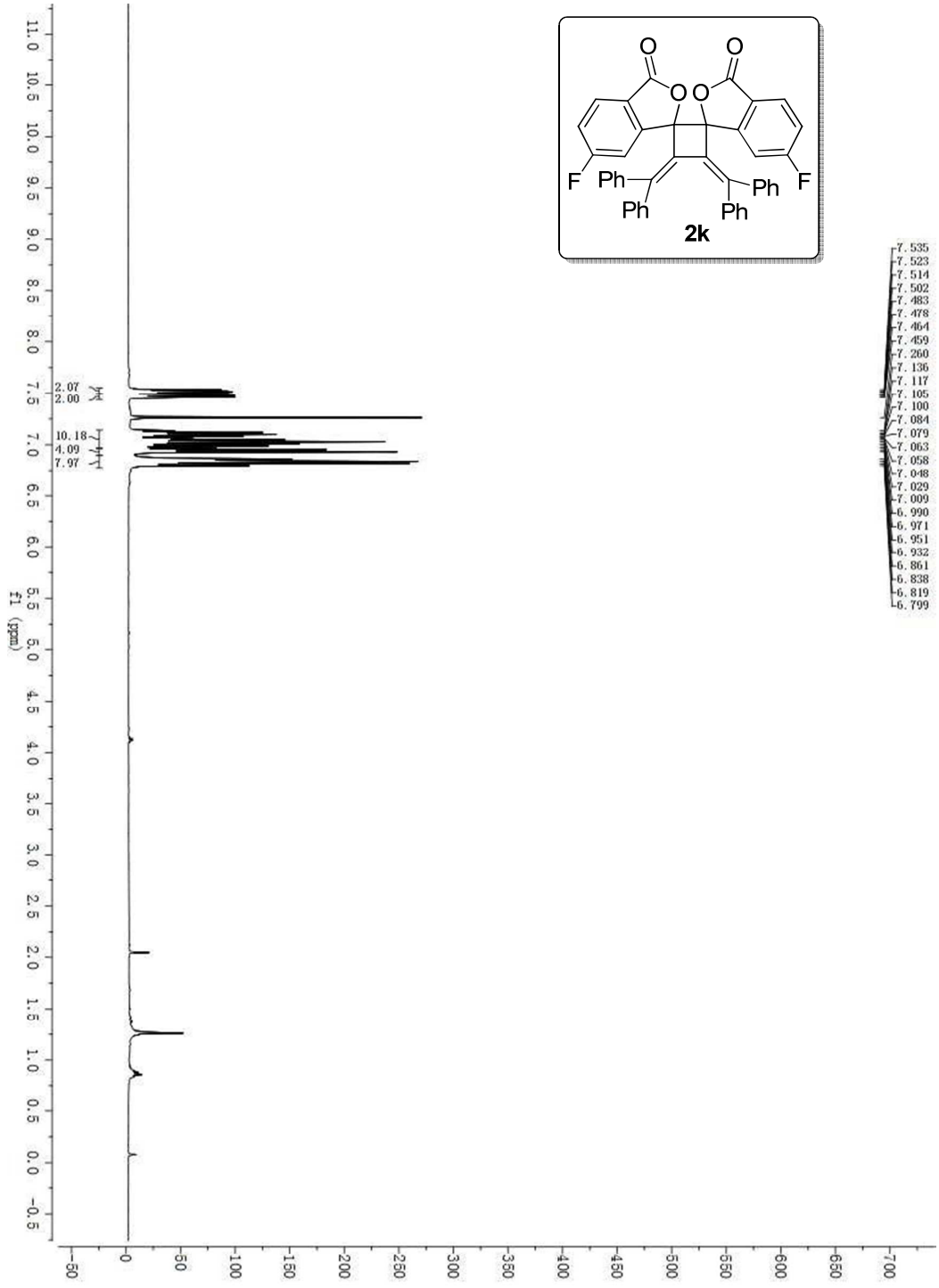
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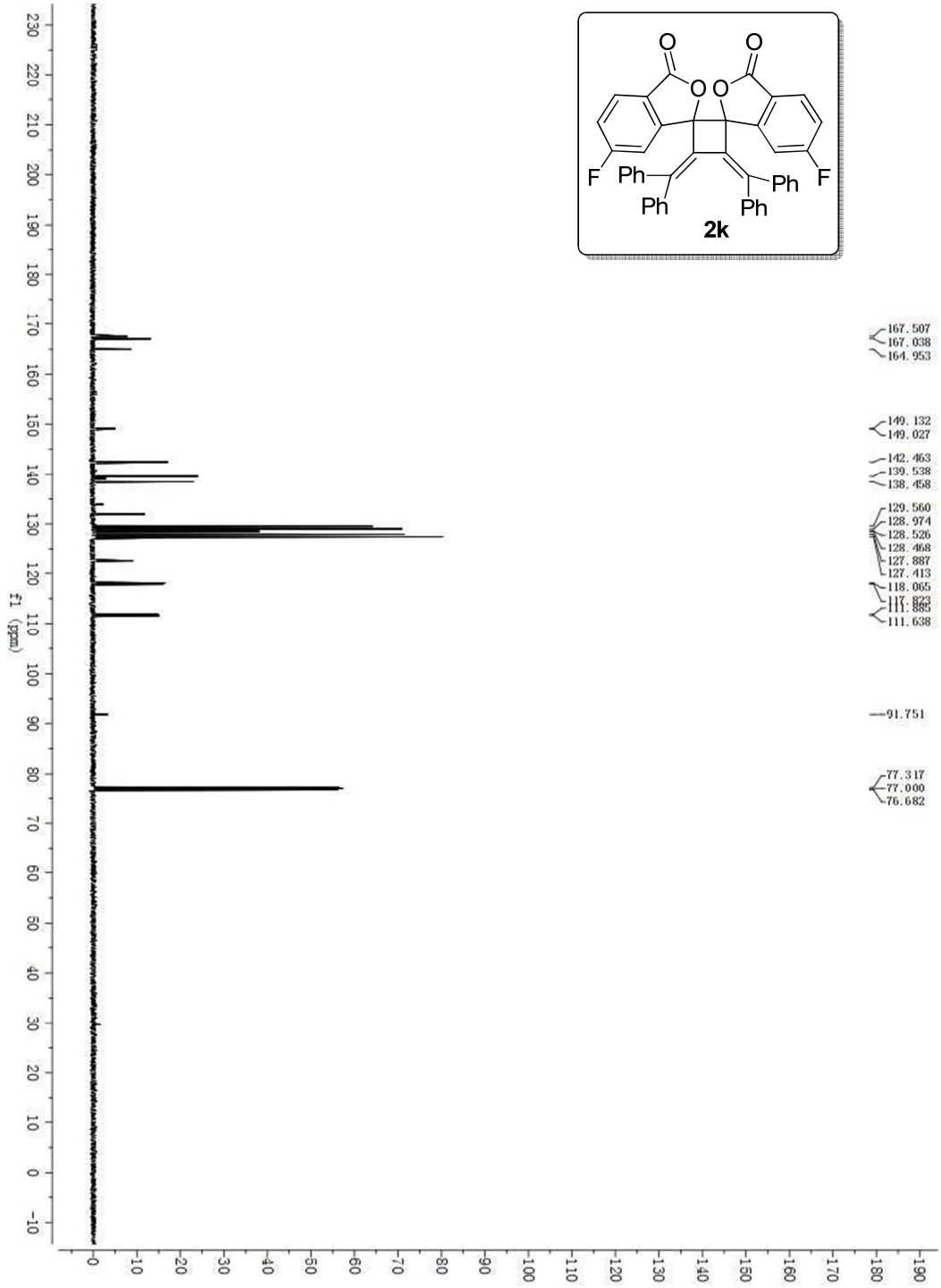
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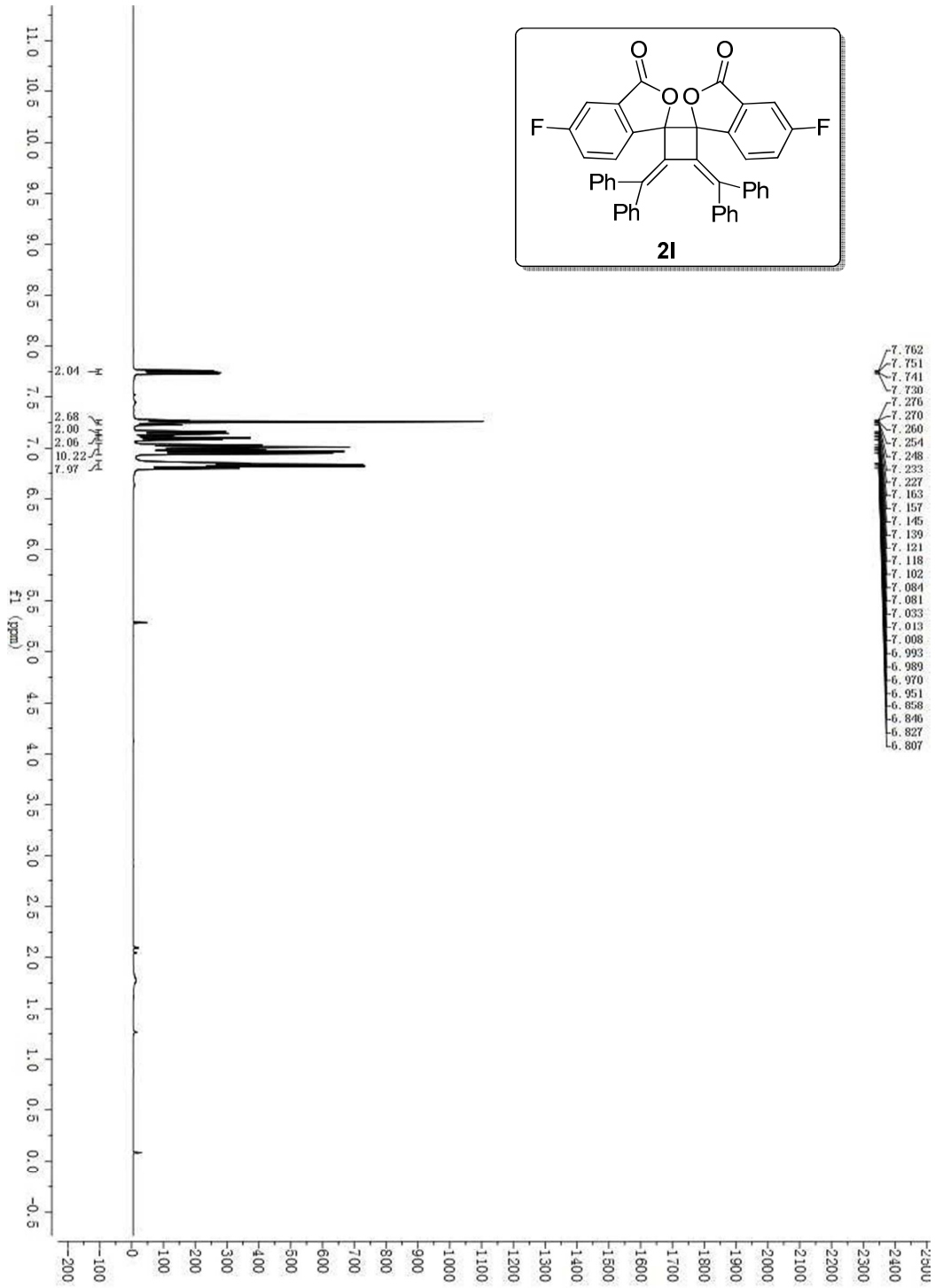
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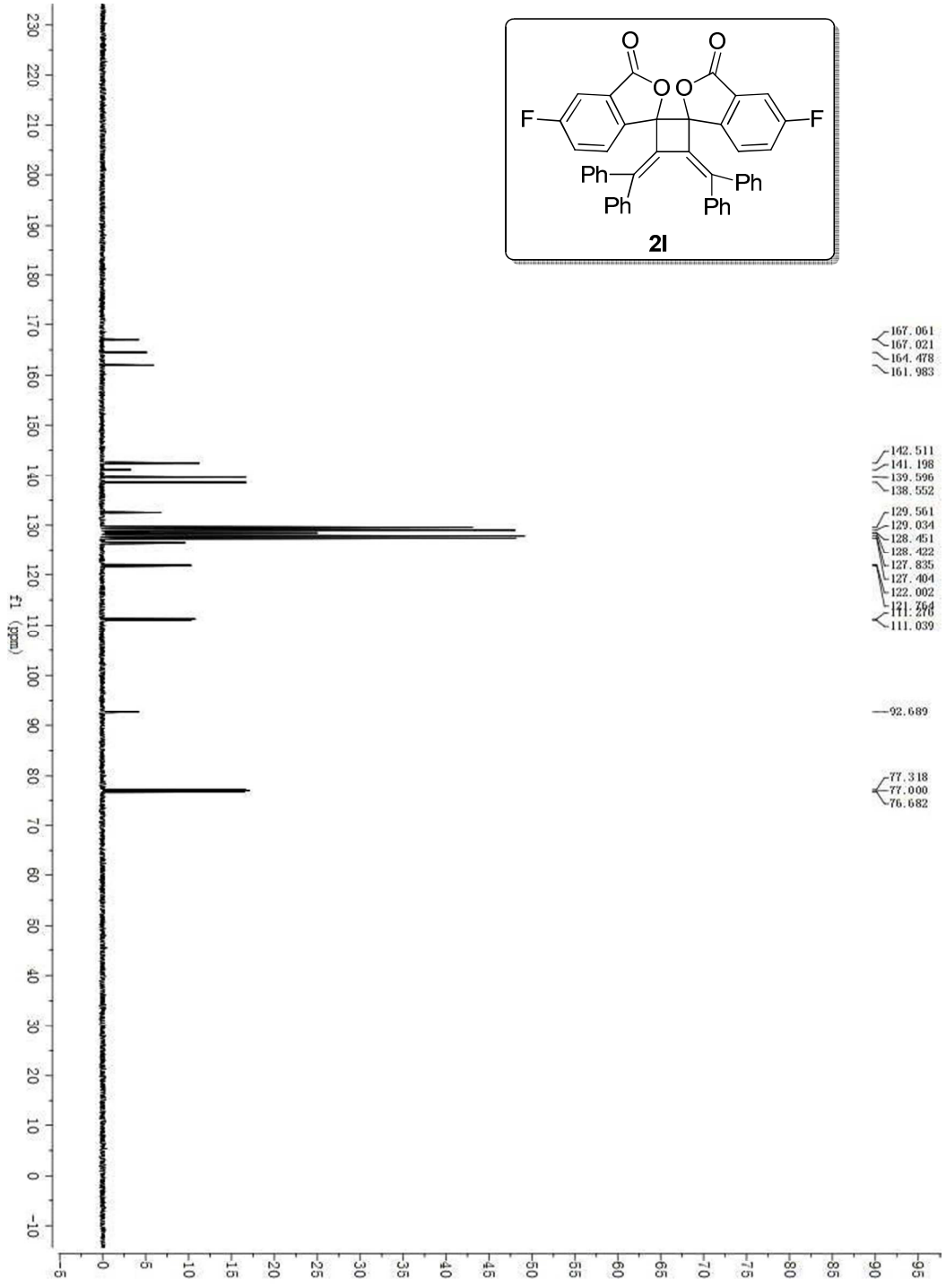
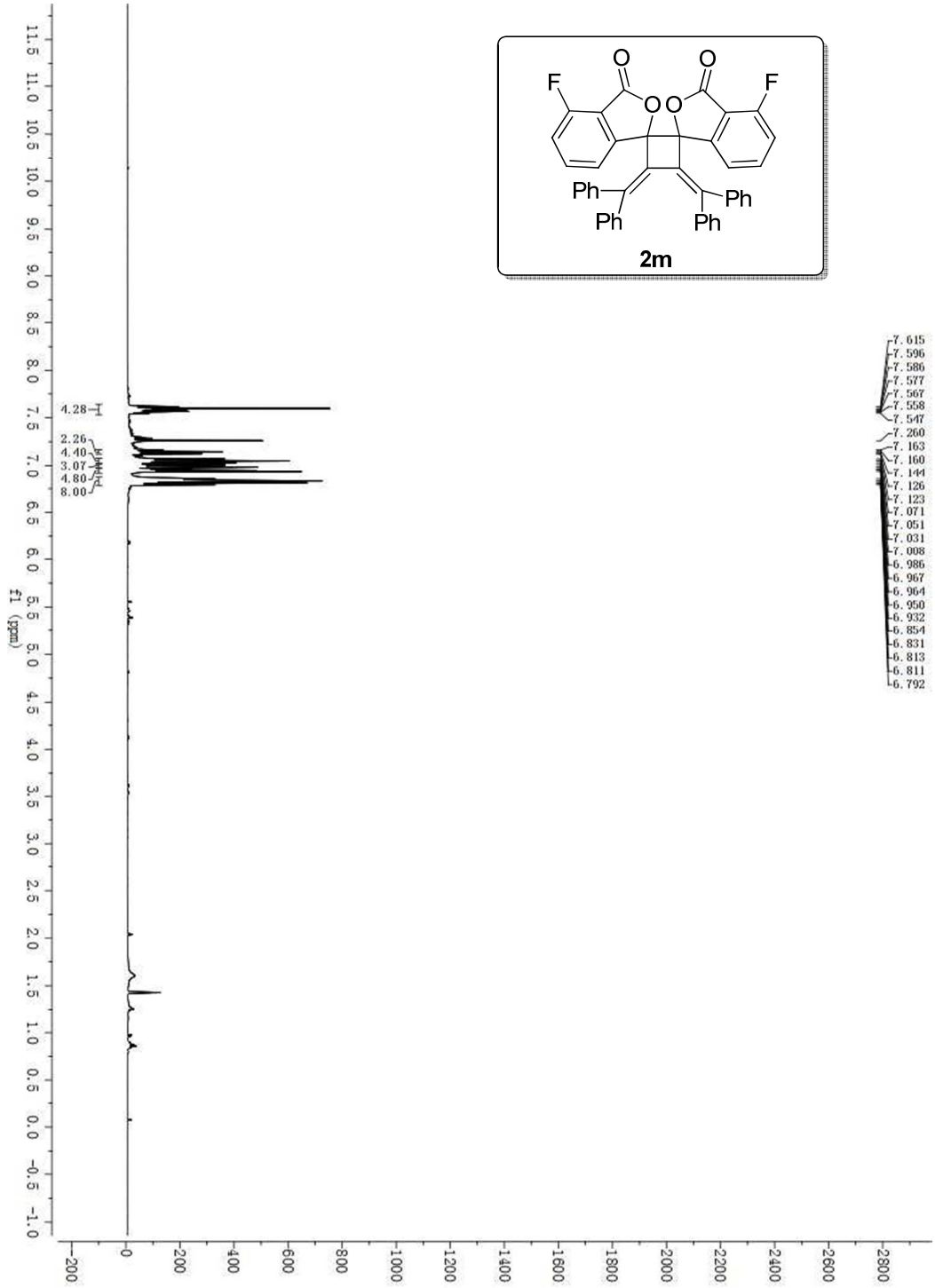
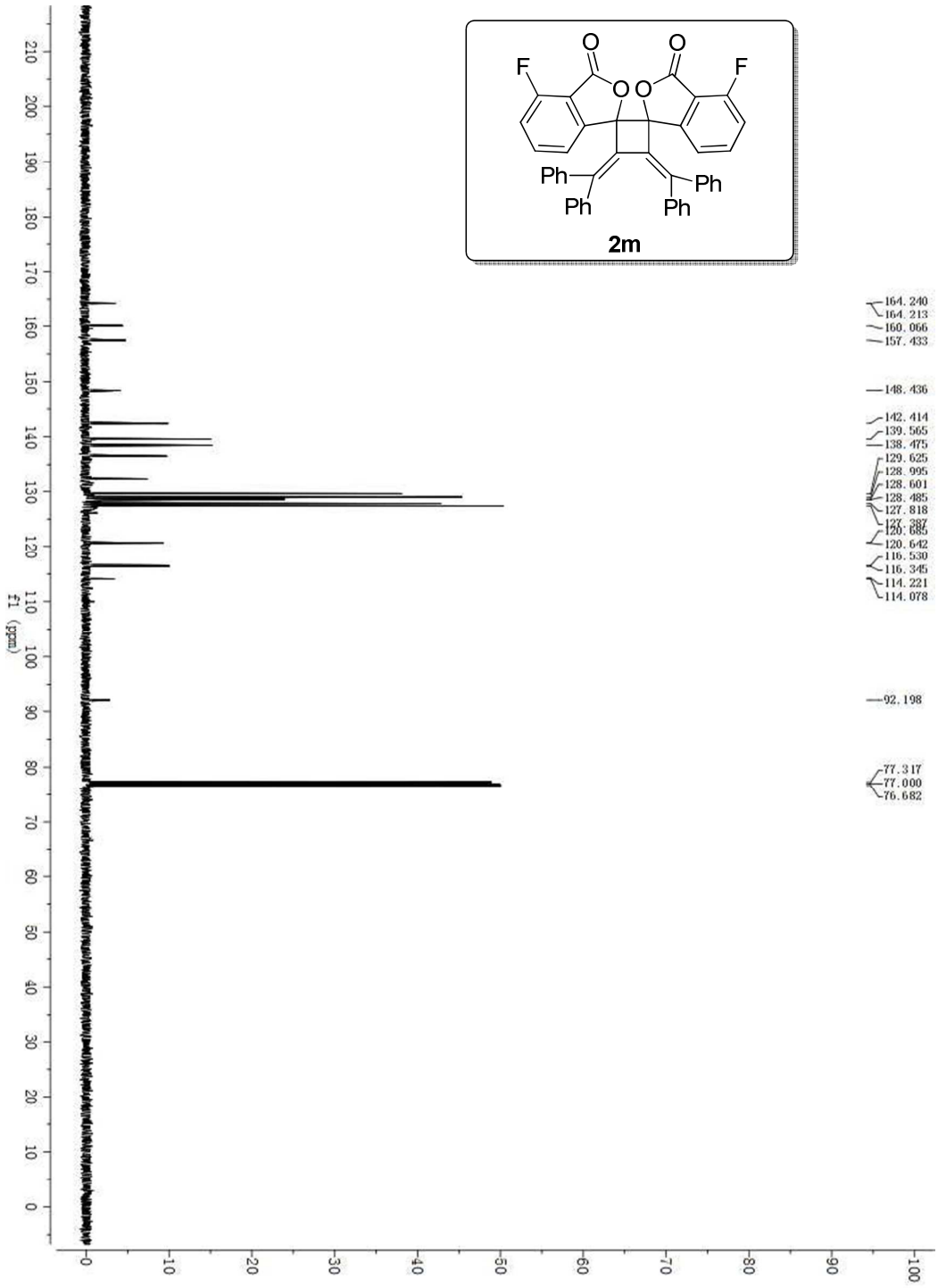
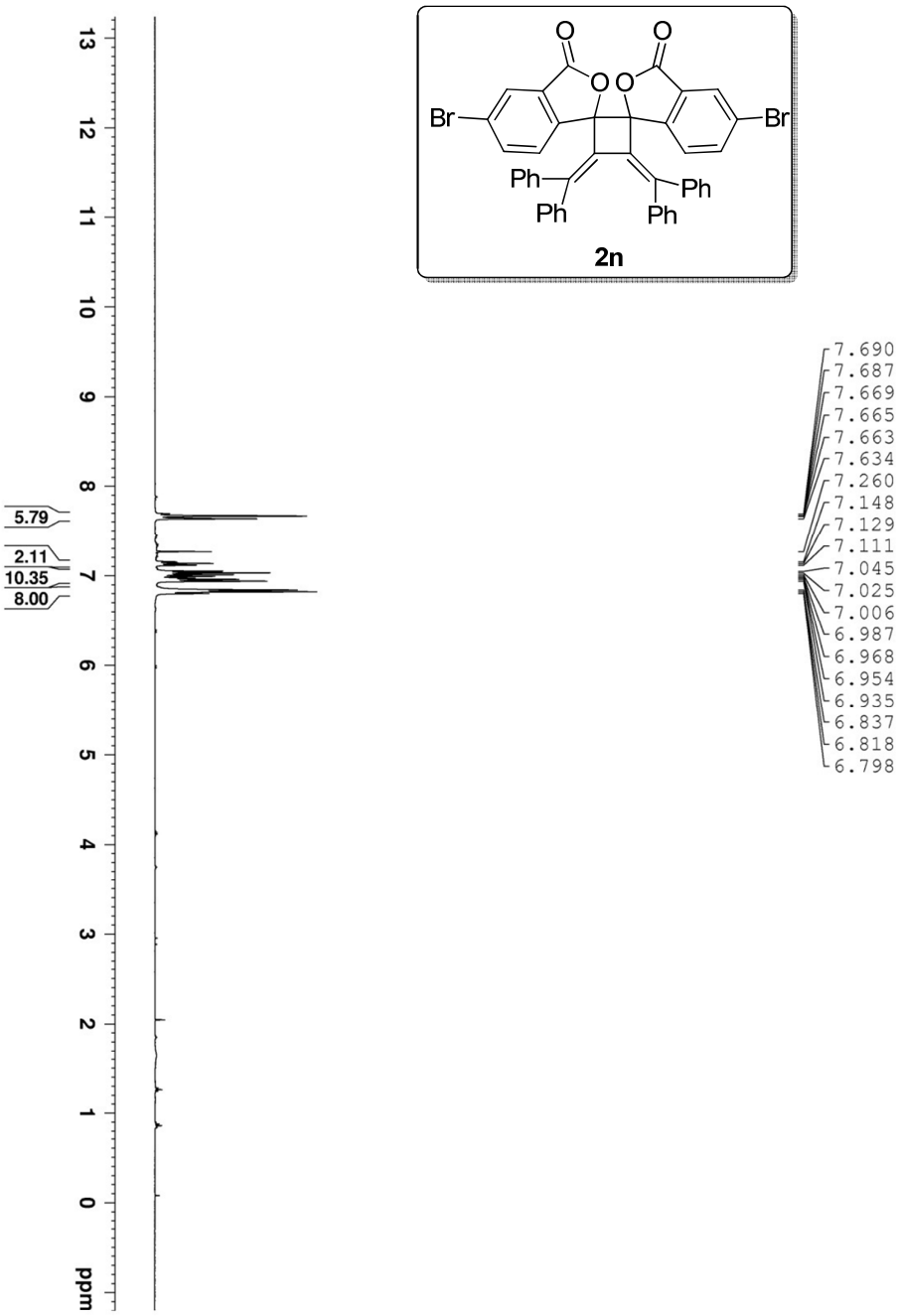
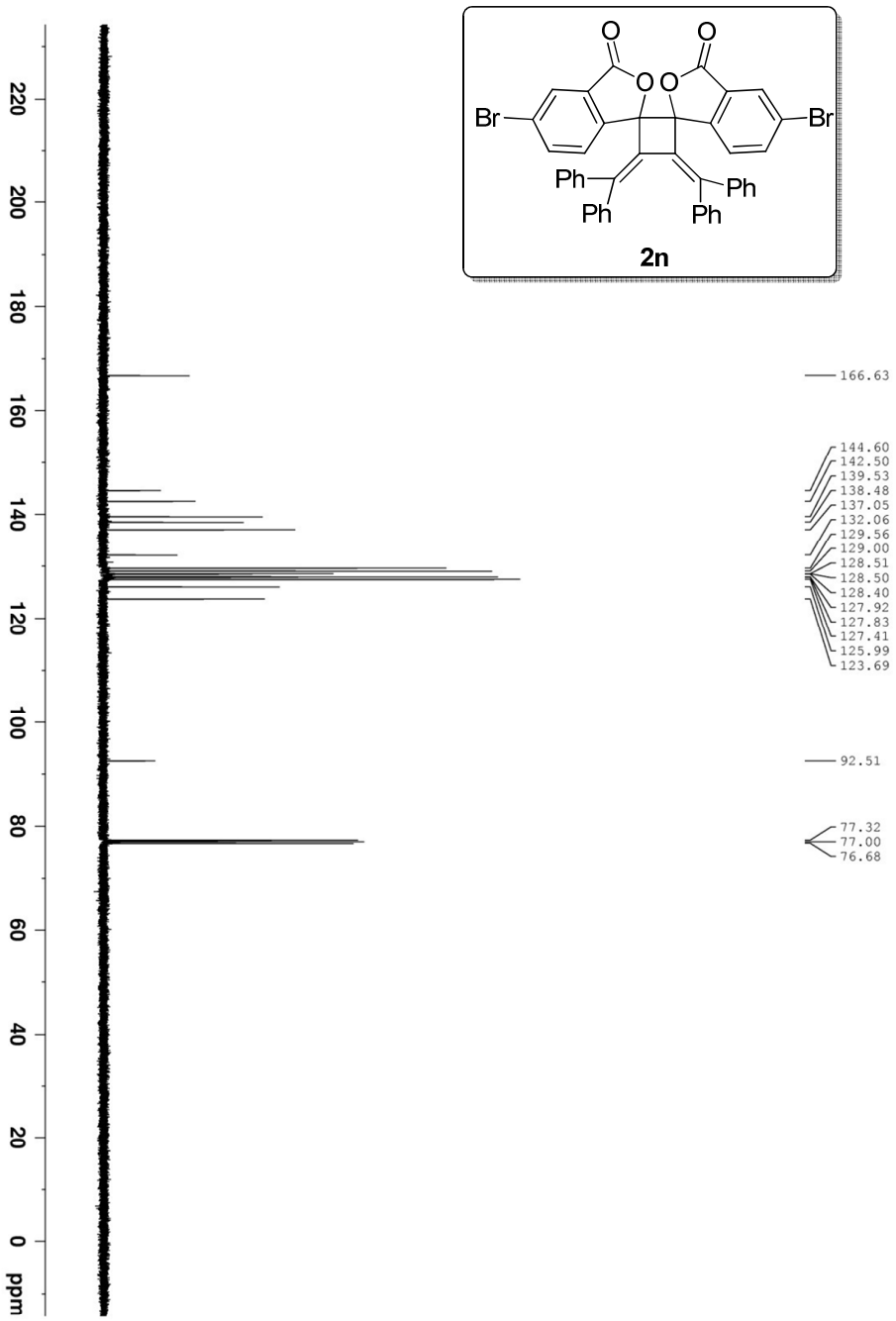
6.807
f1 (ppm)

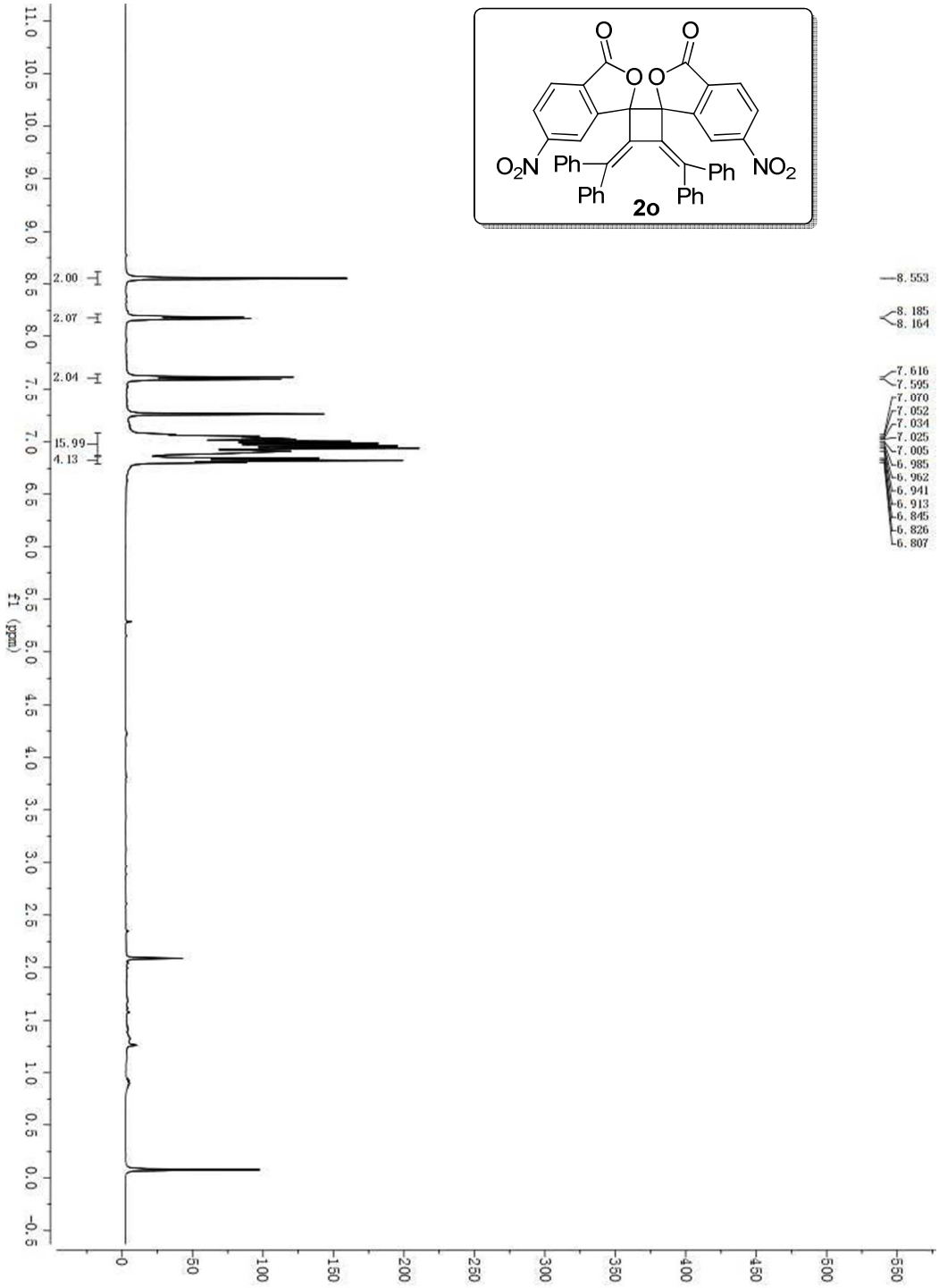

O
O
O
O
F
F
Ph
Ph
Ph
Ph
2l
167.061
167.021
164.478
161.983
142.511
141.198
139.596
138.552
129.561
129.034
128.451
128.422
127.835
127.404
122.002
121.764
111.276
111.039
92.689
77.318
77.000
76.682
f1 (ppm)

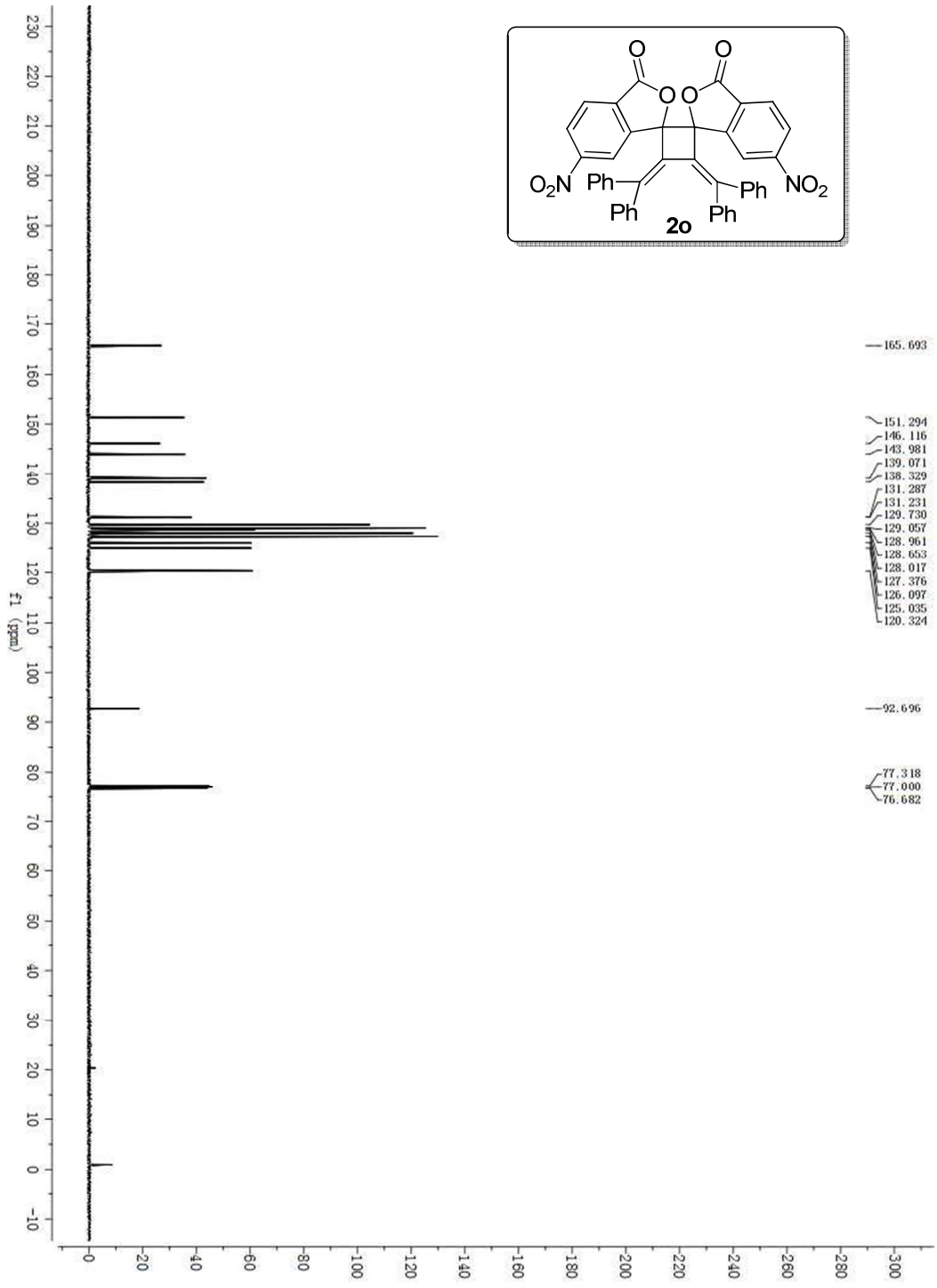

F
O
O
O
O
F
Ph
Ph
Ph
Ph
2m
4.28
2.26
4.40
3.07
4.80
8.00
7.615
7.596
7.586
7.577
7.567
7.558
7.547
7.260
7.163
7.160
7.144
7.126
7.123
7.071
7.051
7.031
7.008
6.986
6.967
6.964
6.950
6.932
6.854
6.831
6.813
6.811
6.792
f1 (ppm)

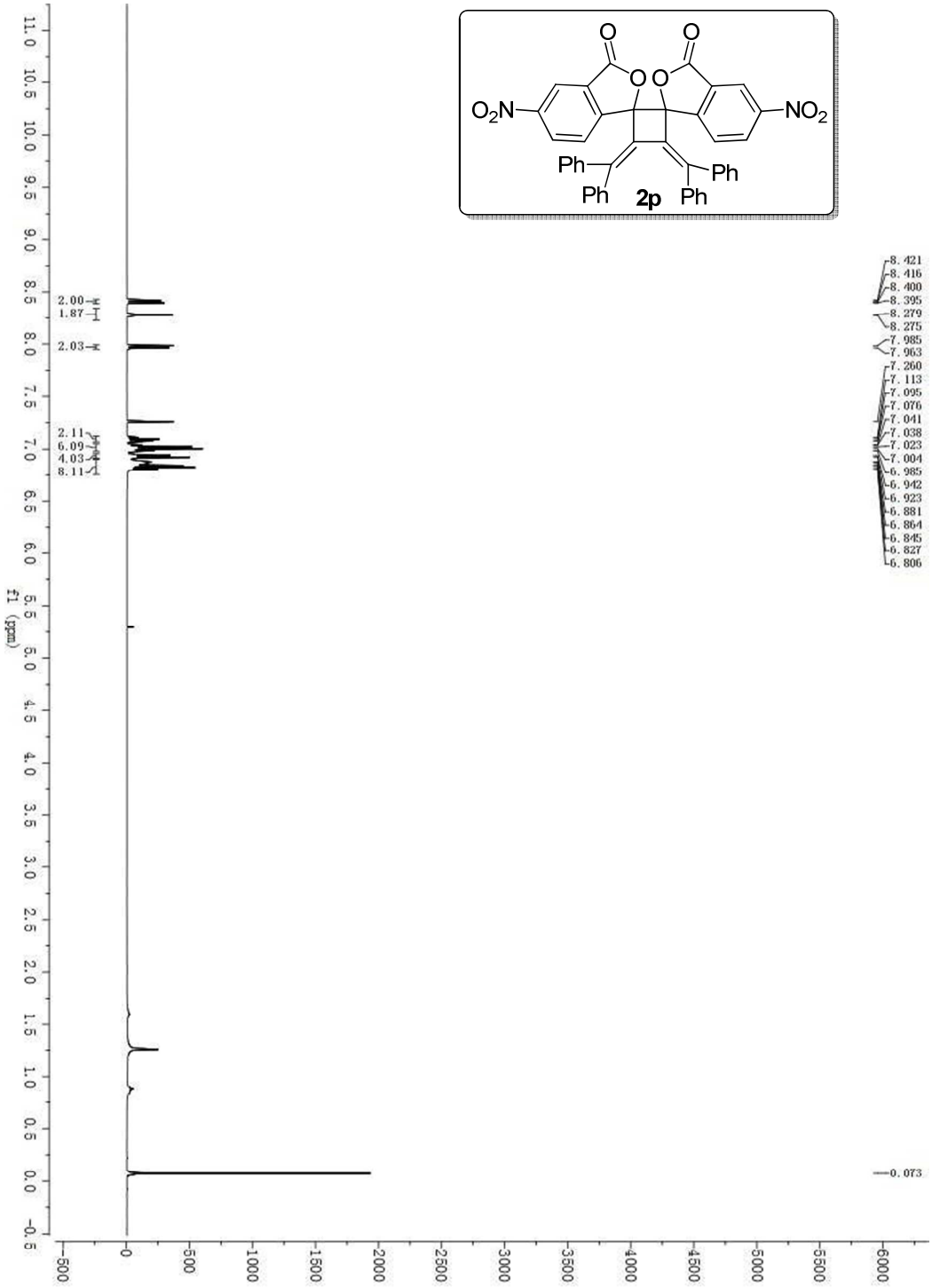

O
O
F
F
O
O
Ph
Ph
Ph
Ph
2m
164.240
164.213
160.066
157.433
148.436
142.414
139.565
138.475
129.625
128.995
128.601
128.485
127.818
127.387
120.685
120.642
116.530
116.345
114.221
114.078
92.198
77.317
77.000
76.682
f1 (ppm)

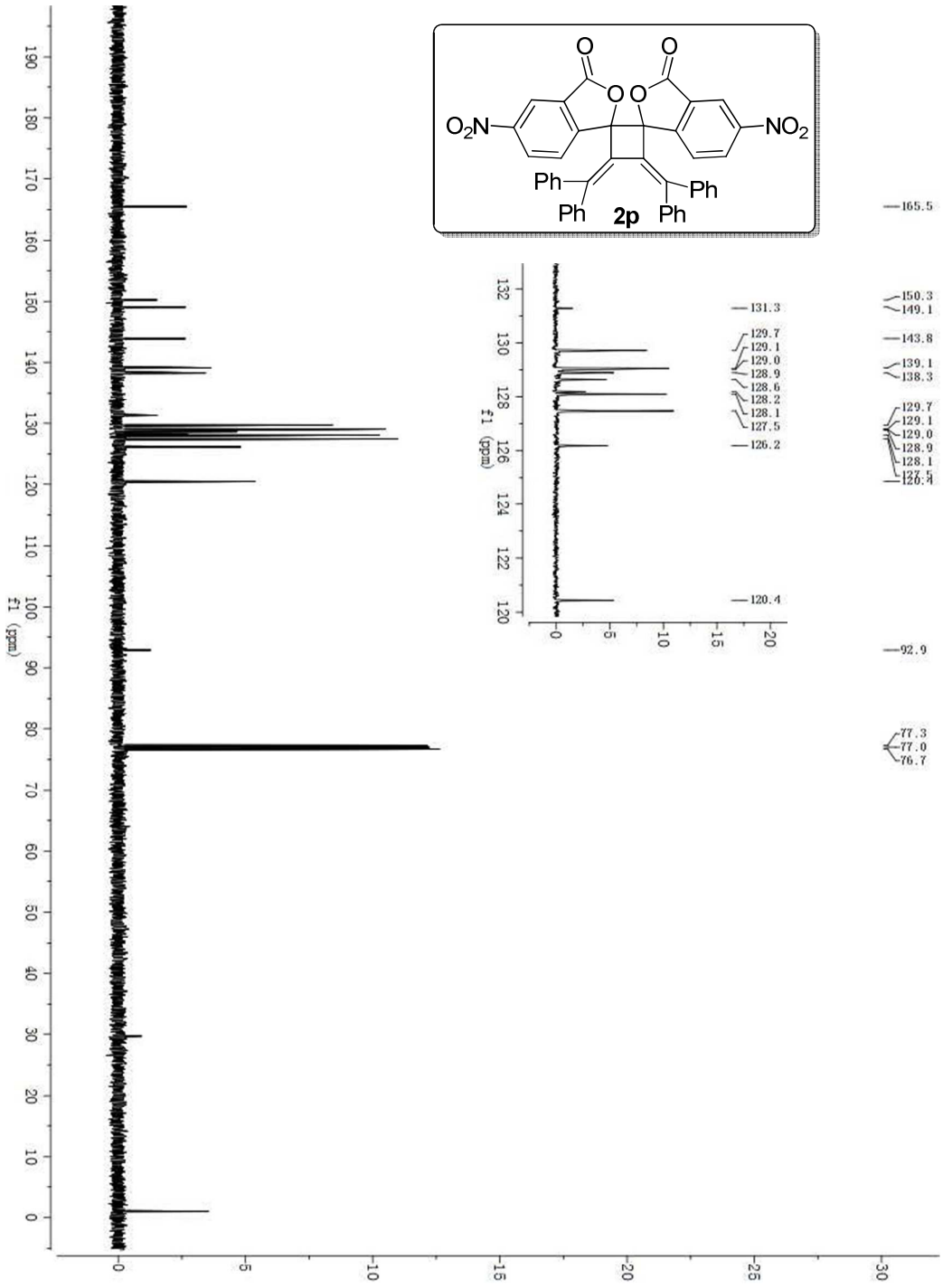

O
O
O
O
Br
Br
Ph
Ph
Ph
Ph
2n
7.690
7.687
7.669
7.665
7.663
7.634
7.260
7.148
7.129
7.111
7.045
7.025
7.006
6.987
6.968
6.954
6.935
6.837
6.818
6.798
5.79
2.11
10.35
8.00
ppm

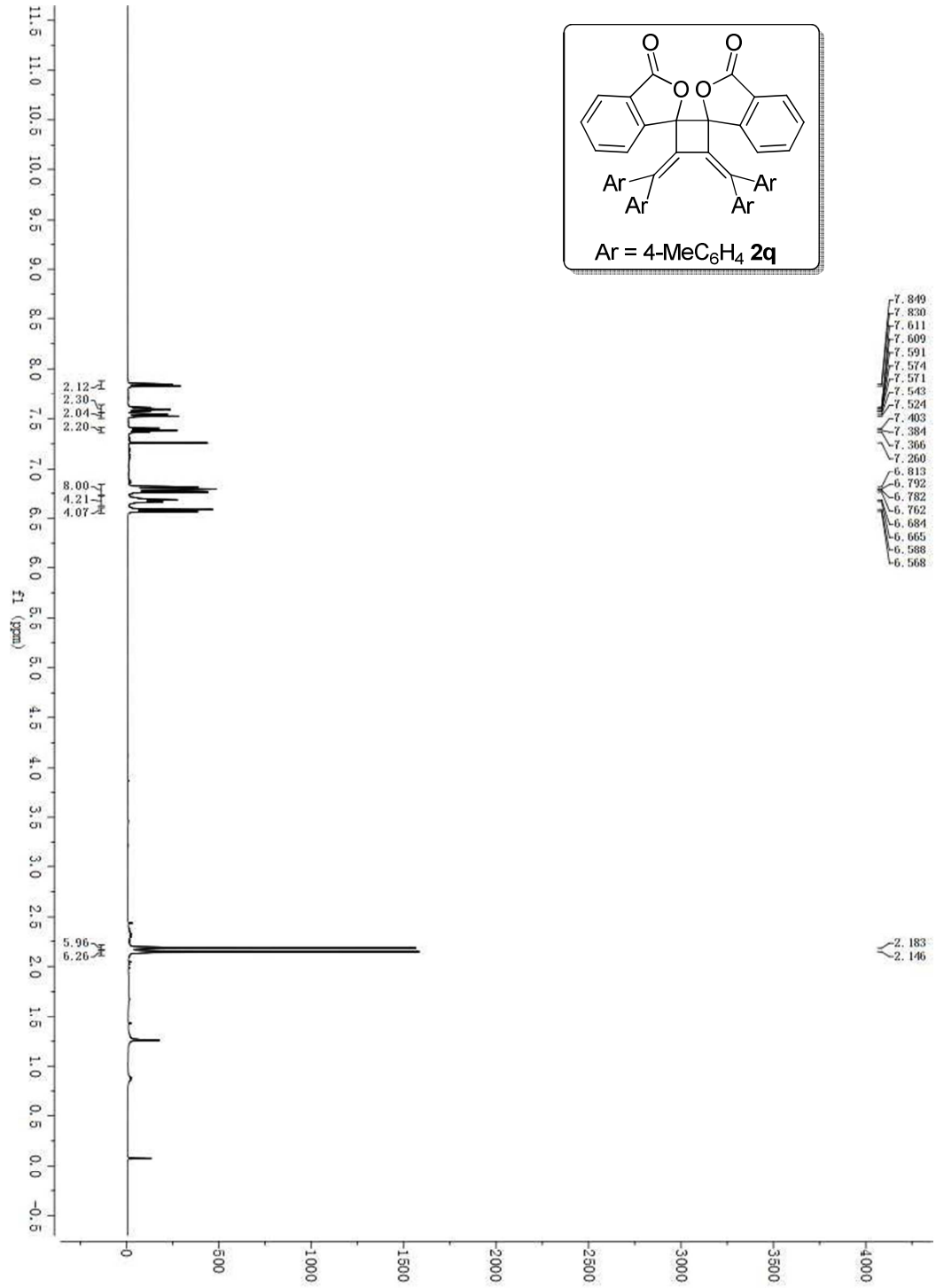

O
O
O
O
Br
Br
Ph
Ph
Ph
Ph
2n
166.63
144.60
142.50
139.53
138.48
137.05
132.06
129.56
129.00
128.51
128.50
128.40
127.92
127.83
127.41
125.99
123.69
92.51
77.32
77.00
76.68
220
200
180
160
140
120
100
80
60
40
20
0
ppm

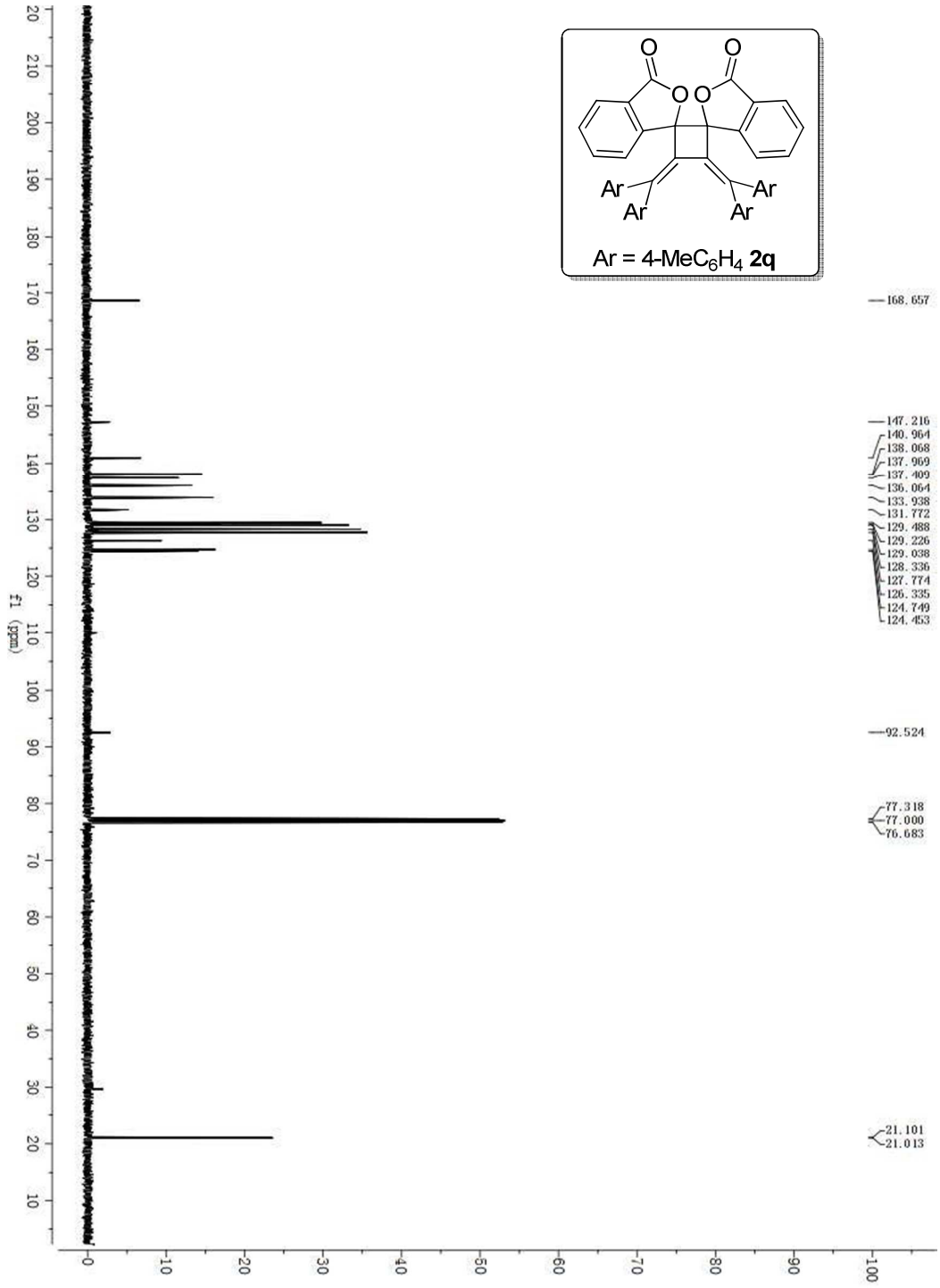

O
O
O
O
O_2N
Ph
Ph
Ph
Ph
NO_2
2o
2.00
2.07
2.04
15.99
4.13
8.553
8.185
8.164
7.616
7.595
7.070
7.052
7.034
7.025
7.005
6.985
6.962
6.941
6.913
6.845
6.826
6.807
f1 (ppm)

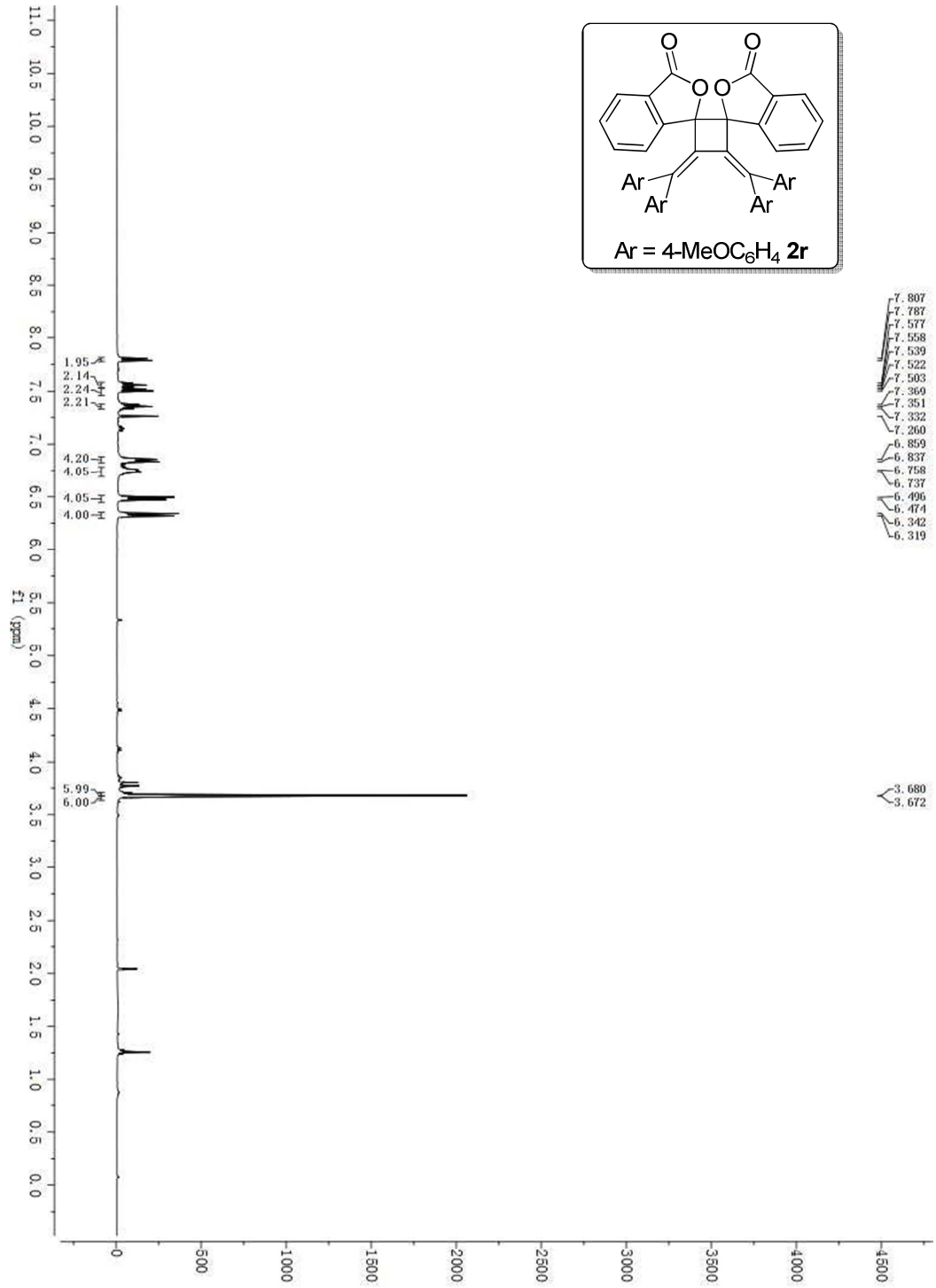

O
O
O
O
O2N
Ph
Ph
Ph
Ph
NO2
2o
165.693
151.294
146.116
143.981
139.071
138.329
131.287
131.231
129.730
129.057
128.961
128.653
128.017
127.376
126.097
125.035
120.324
92.696
77.318
77.000
76.682
f1 (ppm)

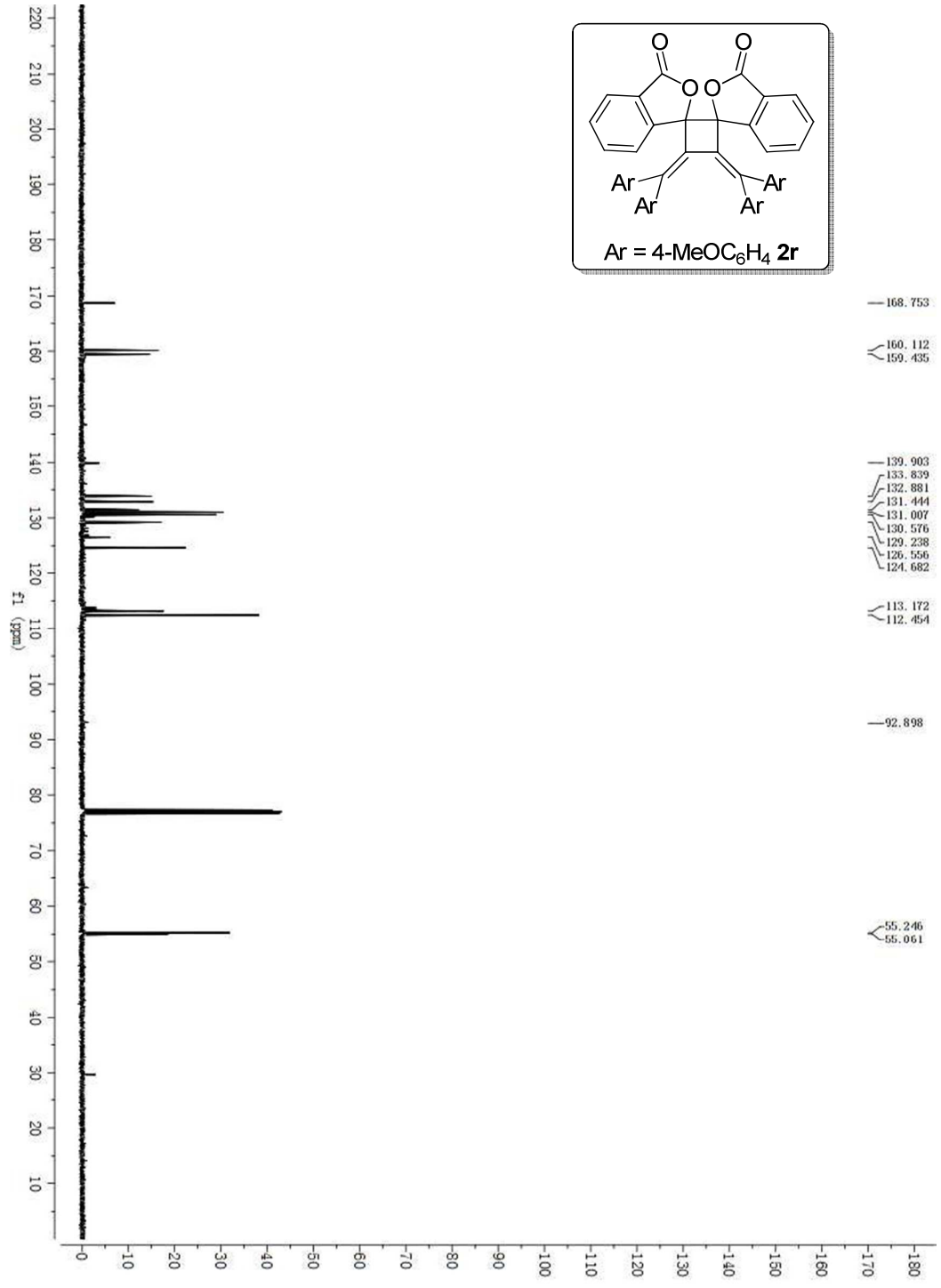

O
O
O
O
O2N
NO2
Ph
Ph
Ph
Ph
2p
8.421
8.416
8.400
8.395
8.279
8.275
7.985
7.963
7.260
7.113
7.095
7.076
7.041
7.038
7.023
7.004
6.985
6.942
6.923
6.881
6.864
6.845
6.827
6.806
0.073
2.00
1.87
2.03
2.11
6.09
4.03
8.11
f1 (ppm)

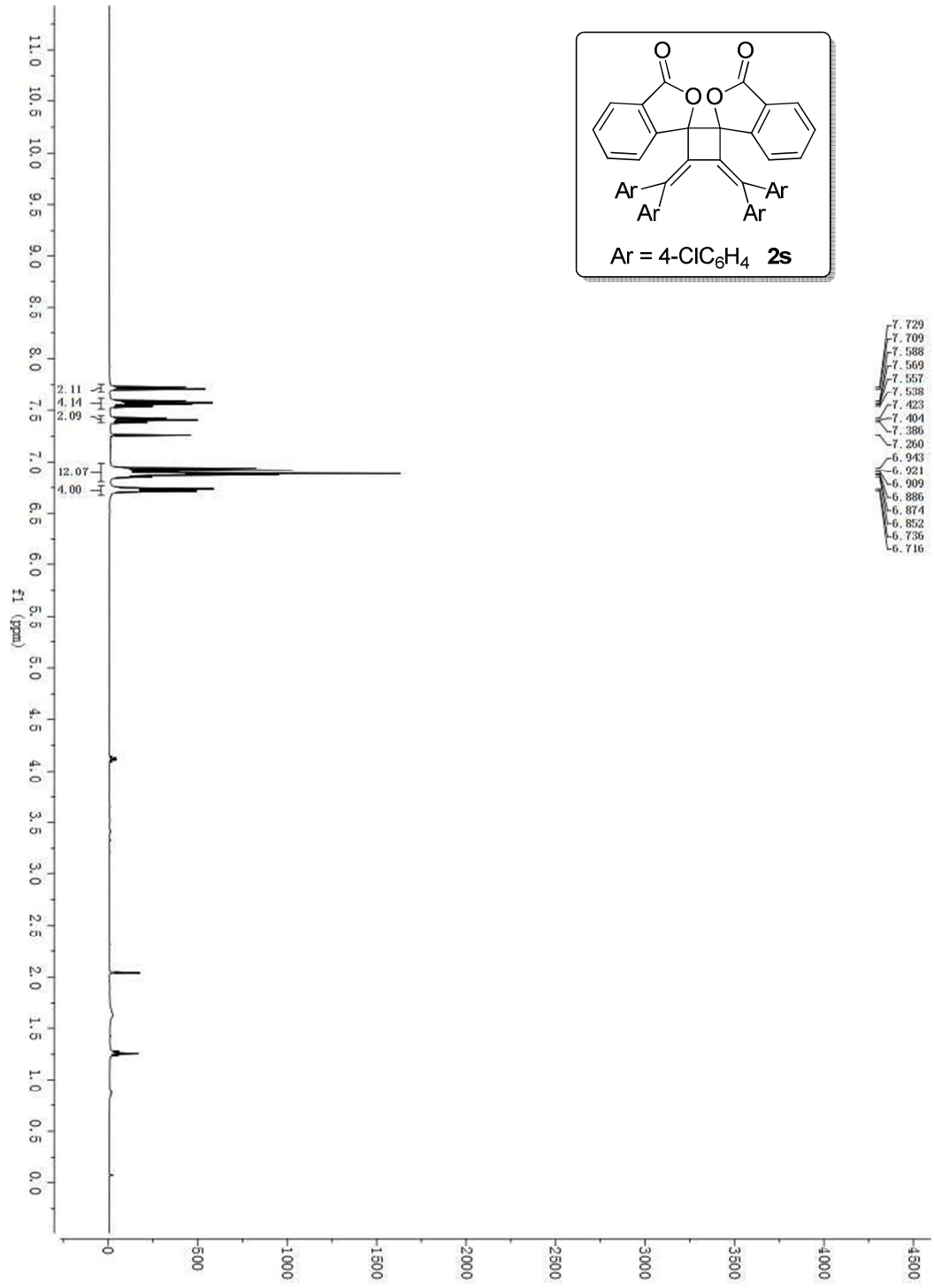

O
O
O
O
O2N
NO2
Ph
Ph
Ph
Ph
2p
165.5
150.3
149.1
143.8
139.1
138.3
131.3
129.7
129.1
129.0
128.9
128.6
128.2
128.1
127.5
126.2
120.4
92.9
77.3
77.0
76.7
f1 (ppm)

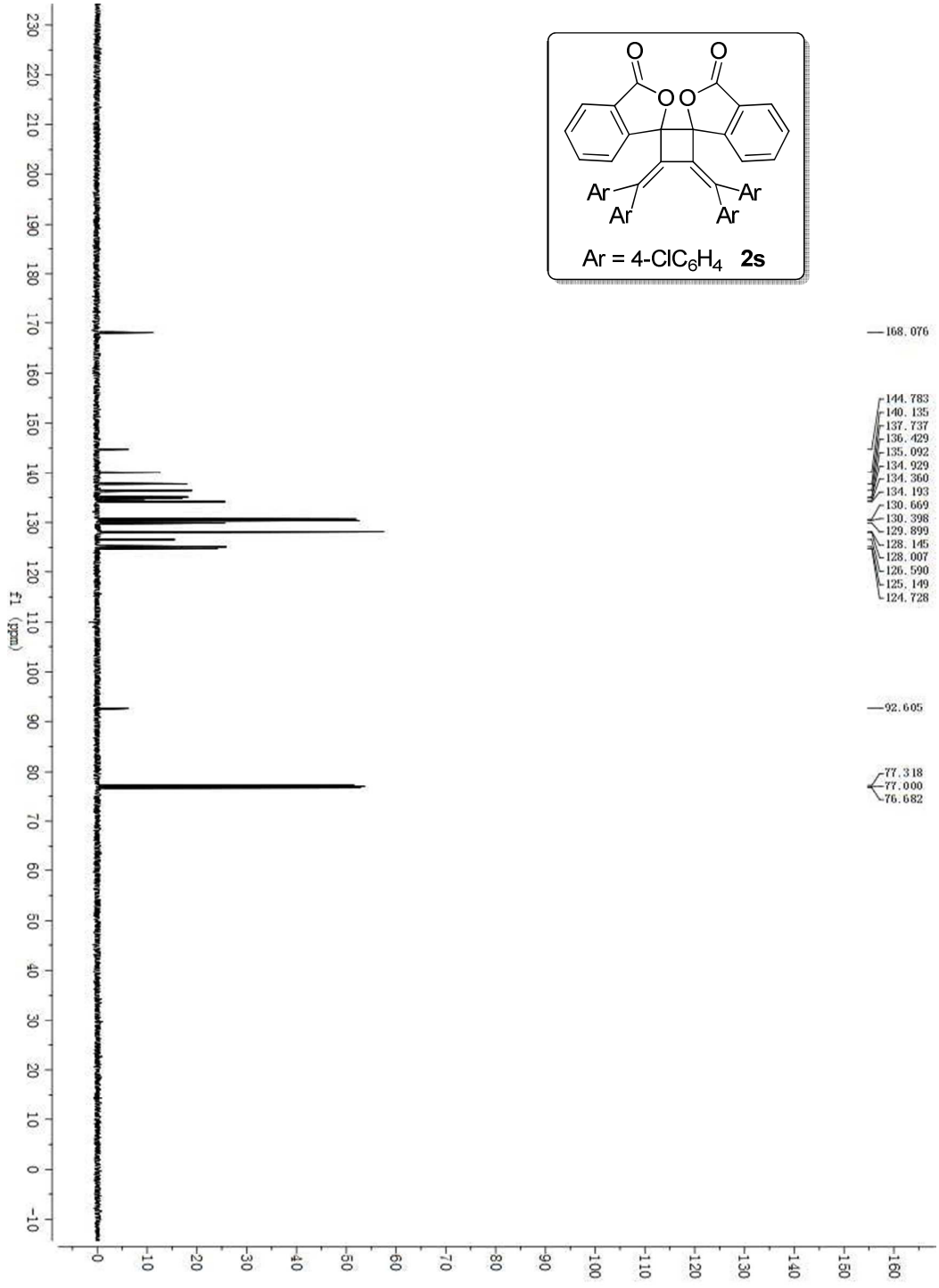

Ar = 4-MeC6H4 2q
7.849
7.830
7.611
7.609
7.591
7.574
7.571
7.543
7.524
7.403
7.384
7.366
7.260
6.813
6.792
6.782
6.762
6.684
6.665
6.588
6.568
2.183
2.146
2.12
2.30
2.04
2.20
8.00
4.21
4.07
5.96
6.26
f1 (ppm)

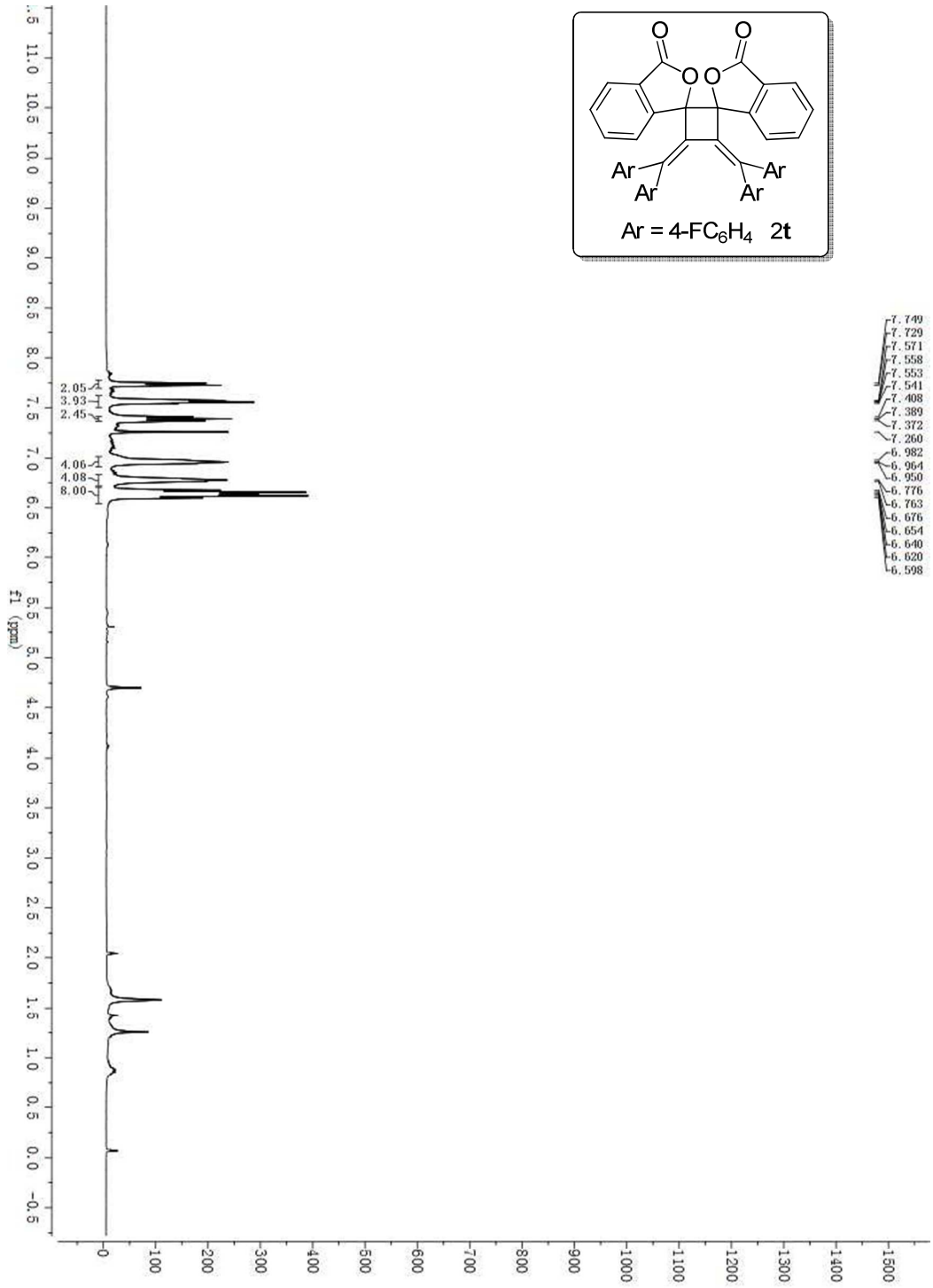

Ar = 4-MeC6H4 2q
168.657
147.216
140.964
138.068
137.969
137.409
136.064
133.938
131.772
129.488
129.226
129.038
128.336
127.774
126.335
124.749
124.453
92.524
77.318
77.000
76.683
21.101
21.013
f1 (ppm)

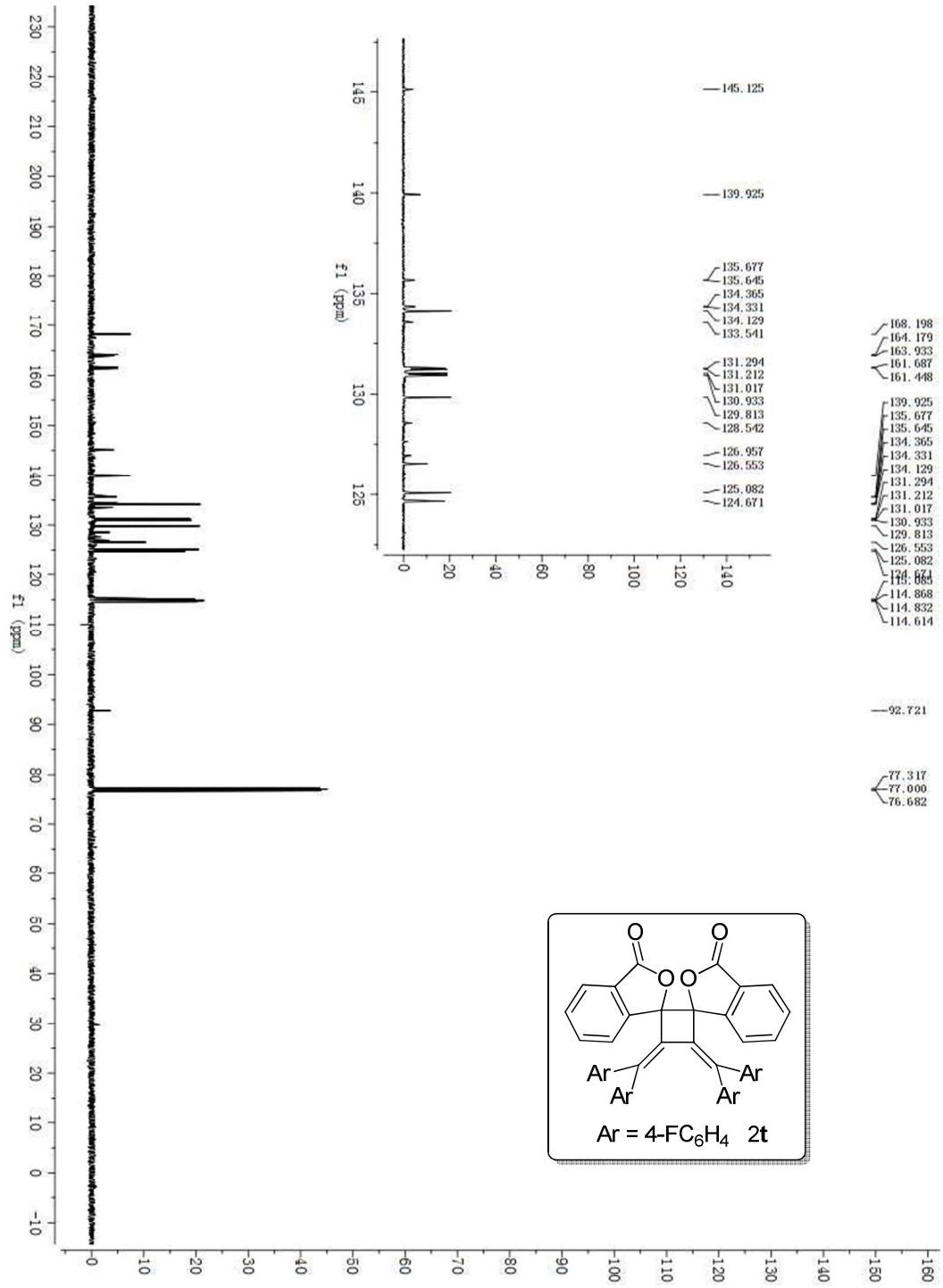

Ar = 4-MeOC6H4 2r
1.95
2.14
2.24
2.21
4.20
4.05
4.05
4.00
5.99
6.00
7.807
7.787
7.577
7.558
7.539
7.522
7.503
7.369
7.351
7.332
7.260
6.859
6.837
6.758
6.737
6.496
6.474
6.342
6.319
3.680
3.672
f1 (ppm)


Ar = 4-MeOC6H4 2r
168.753
160.112
159.435
139.903
133.839
132.881
131.444
131.007
130.576
129.238
126.556
124.682
113.172
112.454
92.898
55.246
55.061
f1 (ppm)


Ar = 4-ClC6H4 2s
2.11
4.14
2.09
12.07
4.00
7.729
7.709
7.588
7.569
7.557
7.538
7.423
7.404
7.386
7.260
6.943
6.921
6.909
6.886
6.874
6.852
6.736
6.716
f1 (ppm)


Ar = 4-ClC6H4 2s
168.076
144.783
140.135
137.737
136.429
135.092
134.929
134.360
134.193
130.669
130.398
129.899
128.145
128.007
126.590
125.149
124.728
92.605
77.318
77.000
76.682
f1 (ppm)


Ar = 4-FC6H4 2t
2.05
3.93
2.45
4.06
4.08
8.00
7.749
7.729
7.571
7.558
7.553
7.541
7.408
7.389
7.372
7.260
6.982
6.964
6.950
6.776
6.763
6.676
6.654
6.640
6.620
6.598
f1 (ppm)


145.125
139.925
135.677
135.645
134.365
134.331
134.129
133.541
131.294
131.212
131.017
130.933
129.813
128.542
126.957
126.553
125.082
124.671
168.198
164.179
163.933
161.687
161.448
139.925
135.677
135.645
134.365
134.331
134.129
131.294
131.212
131.017
130.933
129.813
126.553
125.082
114.868
114.832
114.614
92.721
77.317
77.000
76.682
f1 (ppm)
Ar
Ar
Ar
Ar
Ar = 4-FC6H4 2t