

Supporting Information

Phthalide Synthesis through Dehydrogenated Lactonization of C(sp³)-H Bond by Photoredox Catalysis

Hui Liu,^a Chao Liu,^a Shanyi Chen,^a Qihong Lai,^a Yulin Lin,^a Zhixiong Cai,^a
Mingqiang Huang,^a and Shunyou Cai^{*a,b}

^a Fujian Provincial Key Laboratory of Modern Analytical Science and Separation
Technology, School of Chemistry, Chemical Engineering and Environment
Minnan Normal University, Zhangzhou, 363000, China.

^b Guangdong Provincial Key Laboratory of Chemical Genomics, School of Chemical
Biology and Biotechnology, Peking University Shenzhen Graduate School, Shenzhen,
518055, China.

E-mail: caishy05@mnnu.edu.cn

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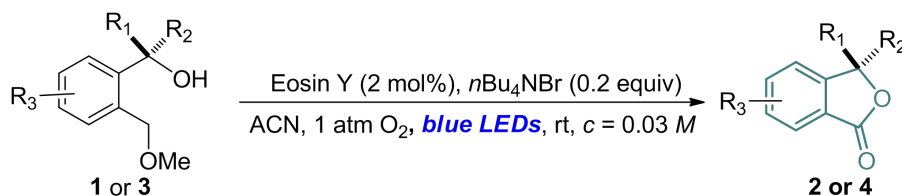
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Materials and methods

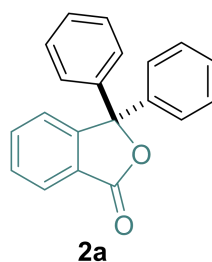
All the chemicals were purchased commercially, and used without further purification. Thin-layer chromatography (TLC) was conducted with 0.25 mm Tsingdao silica gel plates (60F-254) and visualized by exposure to UV light (254 nm) or stained with potassium permanganate. Flash column chromatography was performed using Tsingdao silica gel (60, particle size 0.040–0.063 mm). Reagents were purchased at the highest commercial quality and used without further purification, unless otherwise stated. ^1H NMR spectra were recorded on JEOL spectrometers (at 400 MHz) and were reported relative to deuterated solvent signals. Data for ^1H NMR spectra were reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz) and integration. ^{13}C NMR spectra were recorded on JEOL Spectrometers (at 100 MHz). Data for ^{13}C NMR spectra were reported in terms of chemical shift. Mass spectrometric data were obtained using Bruker Apex IV RTMS. The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad.

General procedure for phthalide synthesis



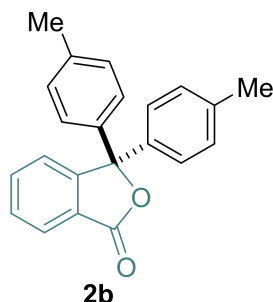
A flame-dried round bottom flask was equipped with magnetic stir bar and charged with 2-(2-(methoxymethyl) phenyl) propan-2-ol **1** or **3** (0.098 mmol, 1.0 equiv), Eosin Y (0.0049 mmol, 0.02 equiv), *n*Bu₄NBr (0.0196 mmol, 0.2 equiv), and ACN (3.0 mL). The reaction mixture was irradiated by blue LEDs under a balloon oxygen atmosphere at room temperature until the starting material disappeared from the TLC. After that the reaction mixture was directly concentrated under reduced pressure and the crude residue was purified by silica gel column chromatography using hexane/EtOAc (4/1 to EtOAc) to afford the desired pure product **2** or **4** in 60-90% yields.

¹H, ¹³C, and ¹⁹F spectra data of compounds **2a-2y**, **4a-4q**

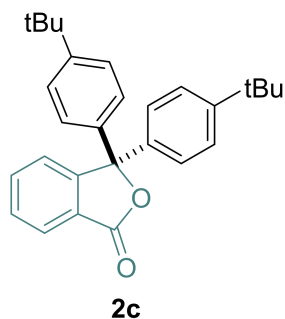


3,3-diphenylisobenzofuran-1(3H)-one (2a): ¹H NMR (400 MHz, CDCl₃) δ 7.96-7.91 (d, *J* = 8.0 Hz, 1H), 7.85-7.64 (t, *J* = 8.0 Hz, 1H), 7.60-7.50 (m, 2H), 7.40-7.25 (m, 10H); ¹³C NMR (100 MHz, CDCl₃) δ 169.9, 152.0, 140.9, 134.3, 129.5, 128.7, 128.6, 127.2, 126.1, 125.6, 124.3, 91.8. These data are consistent with literature values, see: L. Mahendar and G. Satyanarayana, *J. Org. Chem.*, 2016, **81**,

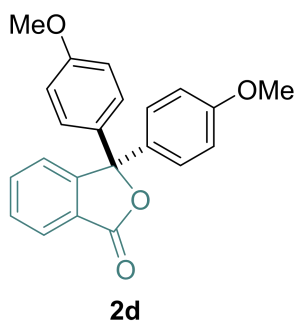
7685. (White solid, 259.6 mg, 92% isolated yield)



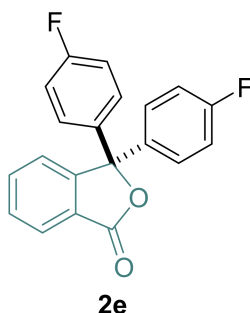
3,3-di-*p*-tolylisobenzofuran-1(3*H*)-one (2b): ^1H NMR (400 MHz, CDCl_3) δ 7.95-7.89 (d, $J = 7.2$ Hz, 1H), 7.86-7.60 (t, $J = 7.2$ Hz, 1H), 7.58-7.48 (m, 2H), 7.28-7.18 (d, $J = 8.4$ Hz, 4H), 7.16-7.05 (d, $J = 8.4$ Hz, 4H), 2.32 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 170.0, 152.4, 138.4, 138.1, 134.2, 129.3, 129.2, 127.2, 126.1, 125.6, 124.2, 91.9, 21.2; HRMS calculated for $\text{C}_{22}\text{H}_{19}\text{O}_2$ ($\text{M} + \text{H}^+$): 315.1385, found: 315.1379. (Yellow oil, 34.0 mg, 90% isolated yield)



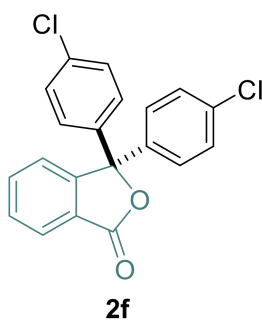
3,3-bis(4-*tert*-butylphenyl)isobenzofuran-1(3*H*)-one (2c): ^1H NMR (400 MHz, CDCl_3) δ 7.98-7.90 (d, $J = 7.6$ Hz, 1H), 7.68-7.65 (m, 1H), 7.60-7.52 (m, 2H), 7.36-7.30 (d, $J = 8.4$ Hz, 4H), 7.28-7.25 (d, $J = 7.6$ Hz, 4H), 1.29 (s, 18H); ^{13}C NMR (100 MHz, CDCl_3) δ 170.0, 152.5, 151.5, 137.9, 134.0, 129.2, 126.9, 125.9, 125.7, 125.4, 124.2, 91.7, 34.6, 31.3; HRMS calculated for $\text{C}_{28}\text{H}_{31}\text{O}_2$ ($\text{M} + \text{H}^+$): 399.2324, found: 399.2318. (Pale yellow oil, 34.1 mg, 91% isolated yield)



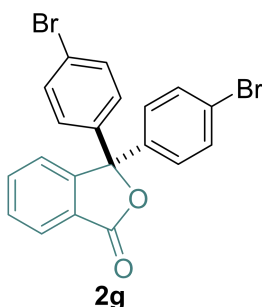
3,3-bis(4-methoxyphenyl)isobenzofuran-1(3H)-one (2d): ^1H NMR (400 MHz, CDCl_3) δ 7.98-7.90 (d, $J = 7.2$ Hz, 1H), 7.71-7.65 (m, 1H), 7.56-7.50 (m, 2H), 7.28-7.20 (d, $J = 8.4$ Hz, 4H), 6.86-6.82 (d, $J = 8.4$ Hz, 4H), 3.79 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 159.7, 152.6, 134.1, 133.1, 132.3, 129.2, 128.6, 126.0, 125.6, 124.0, 113.7, 91.7, 55.3; These data are consistent with literature values, see: L. Mahendar and G. Satyanarayana, *J. Org. Chem.*, 2016, **81**, 7685. (Yellow oil, 26.5 mg, 93% isolated yield)



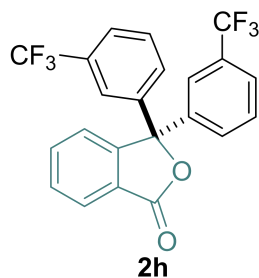
3,3-bis(4-fluorophenyl)isobenzofuran-1(3H)-one (2e): ^1H NMR (400 MHz, CDCl_3) δ 7.98-7.92 (d, $J = 8.0$ Hz, 1H), 7.76-7.62 (m, 1H), 7.62-7.55 (m, 2H), 7.35-7.28 (m, 4H), 7.10-6.98 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.4, 162.8 (d, $J = 247.2$ Hz), 151.7, 136.6, 134.5, 129.7, 129.1, 126.3, 125.4, 124.0, 115.6 (d, $J = 22$ Hz), 90.7; ^{19}F NMR (376 MHz, CDCl_3) δ -112.7; These data are consistent with literature values, see: D. M. Knauss, and J. T. Bender, *J. Polym. Sci. Part A: Polym. Chem.*, 2002, **40**, 3046. (Pale yellow oil, 28.3 mg, 89% isolated yield)



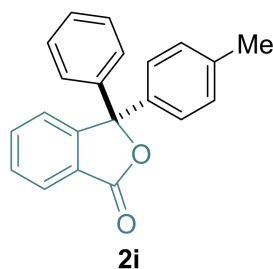
3,3-bis(4-chlorophenyl)isobenzofuran-1(3H)-one (2f): ^1H NMR (400 MHz, CDCl_3) δ 7.98-7.92 (d, J = 8.0 Hz, 1H), 7.76-7.70 (m, 1H), 7.62-7.55 (m, 1H), 7.54-7.50 (d, J = 7.6 Hz, 1H), 7.38-7.28 (d, J = 8.8 Hz, 4H), 7.26-7.20 (d, J = 8.4 Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.3, 151.2, 139.1, 135.0, 134.6, 129.9, 128.9, 128.5, 126.4, 125.4, 123.9, 90.5; These data are consistent with literature values, see: Y. Nakamura, S. Yoshida, and T. Hosoya, *Chem. Lett.*, 2017, **46**, 858. (White solid, 29.6 mg, 93% isolated yield)



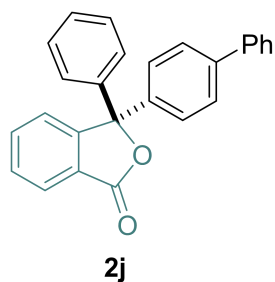
3,3-bis(4-bromophenyl)isobenzofuran-1(3H)-one (2g): ^1H NMR (400 MHz, CDCl_3) δ 7.98-7.92 (d, J = 8.0 Hz, 1H), 7.78-7.70 (m, 1H), 7.65-7.54 (m, 1H), 7.52-7.48 (d, J = 8.0 Hz, 1H), 7.46-7.44 (d, J = 8.0 Hz, 4H), 7.22-7.16 (d, J = 8.8 Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.2, 151.1, 139.5, 134.6, 131.9, 129.9, 128.8, 126.4, 125.4, 123.9, 123.2, 90.6; HRMS calculated for $\text{C}_{20}\text{H}_{12}\text{Br}_2\text{NaO}_2$ ($\text{M} + \text{Na}^+$): 464.9102, found: 464.9096. (White solid, 26.2 mg, 91% isolated yield)



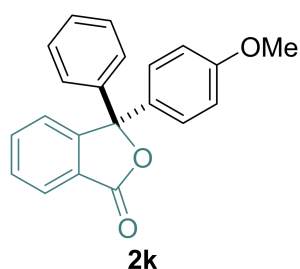
3,3-bis(3-(trifluoromethyl)phenyl)isobenzofuran-1(3H)-one (2h): ^1H NMR (400 MHz, CDCl_3) δ 8.04-7.99 (d, $J = 7.6$ Hz, 1H), 7.80-7.76 (m, 1H), 7.72-7.68 (m, 3H), 7.60-7.58 (m, 3H), 7.56-7.49 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 168.9, 150.4, 141.5, 131.5 (q, $J = 62.6$ Hz), 130.6, 129.5, 126.7, 126.0, 125.4, 125.1 (q, $J = 271.1$ Hz), 123.9, 123.6, 90.2; ^{19}F NMR (376 MHz, CDCl_3) δ -62.5; HRMS calculated for $\text{C}_{22}\text{H}_{13}\text{F}_6\text{O}_2$ ($\text{M} + \text{H}^+$): 423.0819, found: 423.0814. (White solid, 26.7 mg, 93% isolated yield)



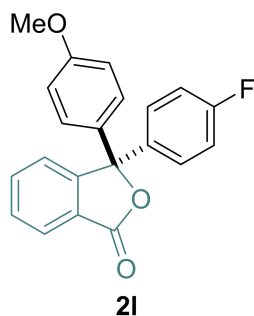
3-phenyl-3-(p-tolyl)isobenzofuran-1(3H)-one (2i): ^1H NMR (400 MHz, CDCl_3) δ 7.98-7.90 (d, $J = 8.0$ Hz, 1H), 7.72-7.68 (m, 1H), 7.62-7.50 (m, 2H), 7.40-7.28 (m, 5H), 7.24-7.15 (d, $J = 8.4$ Hz, 2H), 7.14-7.08 (d, $J = 8.4$ Hz, 2H), 2.33 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.9, 152.2, 141.1, 138.5, 138.0, 134.2, 129.3, 129.2, 128.5, 127.2, 127.1, 126.1, 125.6, 124.8, 124.2, 91.8, 21.2; HRMS calculated for $\text{C}_{21}\text{H}_{17}\text{O}_2$ ($\text{M} + \text{H}^+$): 301.1228, found: 301.1223. (Pale yellow oil, 22.4 mg, 79% isolated yield)



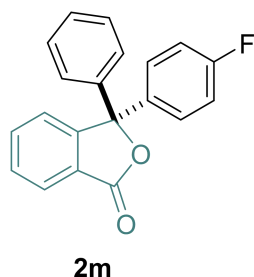
3-([1,1'-biphenyl]-4-yl)-3-phenylisobenzofuran-1(3H)-one (2j): ^1H NMR (400 MHz, CDCl_3) δ 8.00-7.90 (d, $J = 7.2$ Hz, 1H), 7.88-7.70 (m, 1H), 7.68-7.50 (m, 6H), 7.48-7.28 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.8, 152.0, 141.5, 140.9, 140.3, 139.9, 134.3, 129.5, 128.9, 128.7, 128.6, 127.8, 127.6, 127.3, 127.2, 127.1, 126.2, 125.6, 124.3, 91.7; HRMS calculated for $\text{C}_{26}\text{H}_{19}\text{O}_2$ ($\text{M} + \text{H}^+$): 363.1385, found: 363.1379. (Pale yellow oil, 25.1 mg, 88% isolated yield)



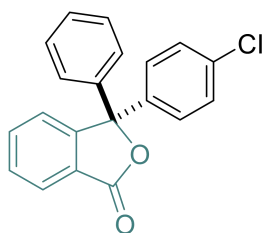
3-(4-methoxyphenyl)-3-phenylisobenzofuran-1(3H)-one (2k): ^1H NMR (400 MHz, CDCl_3) δ 7.96-7.92 (d, $J = 8.0$ Hz, 1H), 7.72-7.68 (m, 1H), 7.58-7.52 (m, 2H), 7.38-7.30 (m, 5H), 7.26-7.18 (d, $J = 8.4$ Hz, 2H), 6.88-6.82 (d, $J = 8.4$ Hz, 2H), 3.80 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.9, 159.7, 152.3, 141.2, 137.6, 134.2, 132.9, 129.3, 128.8, 128.5, 126.9, 126.1, 125.7, 124.1, 113.8, 91.8, 55.4; HRMS calculated for $\text{C}_{21}\text{H}_{17}\text{O}_3$ ($\text{M} + \text{H}^+$): 317.1177, found: 317.1172. (Brown oil, 20.9 mg, 71% isolated yield)



3-(4-fluorophenyl)-3-(4-methoxyphenyl)isobenzofuran-1(3H)-one (2l): ^1H NMR (400 MHz, CDCl_3) δ 7.98-7.92 (d, $J = 8.0$ Hz, 1H), 7.72-7.68 (m, 1H), 7.60-7.50 (m, 2H), 7.36-7.32 (m, 2H), 7.24-7.18 (d, $J = 8.8$ Hz, 2H), 7.06-6.98 (m, 2H), 6.88-6.80 (d, $J = 8.8$ Hz, 2H), 3.80 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.7, 162.5 (d, $J = 270.0$ Hz), 159.8, 152.2, 134.3, 132.7, 129.5, 129.1, 128.6, 126.2, 125.6, 124.0, 115.6, 115.4, 113.9, 91.2, 55.4; ^{19}F NMR (376 MHz, CDCl_3) δ -113.2; HRMS calculated for $\text{C}_{21}\text{H}_{16}\text{FO}_3$ ($\text{M} + \text{H}^+$): 335.1083, found: 335.1078. (Brown oil, 25.5 mg, 82% isolated yield)

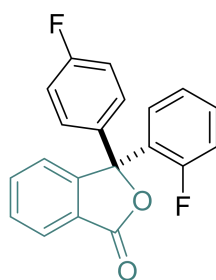


3-(4-fluorophenyl)-3-phenylisobenzofuran-1(3H)-one (2m): ^1H NMR (400 MHz, CDCl_3) δ 7.98-7.92 (d, $J = 7.6$ Hz, 1H), 7.76-7.69 (m, 1H), 7.60-7.52 (m, 2H), 7.40-7.36 (m, 5H), 7.38-7.30 (m, 2H), 7.08-6.98 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.6, 162.7 (d, $J = 250.0$ Hz), 151.9, 140.8, 136.8, 134.4, 129.6, 129.3, 129.2, 128.7, 127.0, 126.3, 125.6, 124.2, 115.6 (d, $J = 20.0$ Hz), 91.3; ^{19}F NMR (376 MHz, CDCl_3) δ -112.9; HRMS calculated for $\text{C}_{20}\text{H}_{14}\text{FO}_2$ ($\text{M} + \text{H}^+$): 305.0978, found: 305.0972. (Pale yellow solid, 23.6 mg, 88% isolated yield)



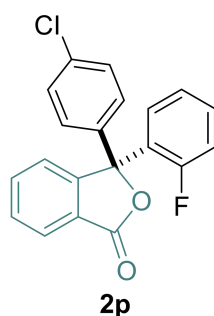
2n

3-(4-chlorophenyl)-3-phenylisobenzofuran-1(3H)-one (2n): ^1H NMR (400 MHz, CDCl_3) δ 7.98-7.92 (d, $J = 8.0$ Hz, 1H), 7.76-7.68 (m, 1H), 7.60-7.52 (m, 2H), 7.38-7.22 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.6, 151.6, 139.6, 134.4, 129.6, 128.8, 128.7, 128.6, 127.1, 126.3, 124.1, 91.1; Commercial Sources: CAS 102159-84-2. (Pale yellow solid, 25.6 mg, 89% isolated yield)

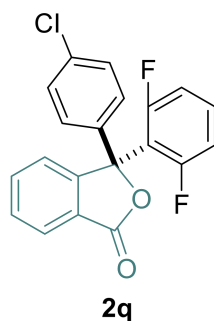


2o

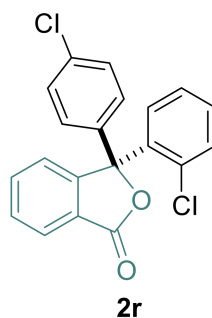
3-(2-fluorophenyl)-3-(4-fluorophenyl)isobenzofuran-1(3H)-one (2o): ^1H NMR (400 MHz, CDCl_3) δ 8.00-7.94 (d, $J = 7.6$ Hz, 1H), 7.78-7.72 (m, 3H), 7.62-7.58 (t, $J = 6.4$ Hz, 1H), 7.56-7.48 (t, $J = 7.6$ Hz, 1H), 7.40-7.30 (m, 1H), 7.28-7.22 (t, $J = 8.4$ Hz, 2H), 7.20-7.12 (m, 1H), 7.10-6.98 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.3, 163.0 (d, $J = 247.3$ Hz), 160.0 (d, $J = 248.2$ Hz), 151.2, 135.8, 134.6, 131.1 (d, $J = 10.0$ Hz), 129.9, 128.7, 127.7, 126.2, 125.7, 124.5 (d, $J = 14.4$ Hz), 124.4, 124.2, 117.0 (d, $J = 21.1$ Hz), 115.7 (d, $J = 21.1$ Hz), 89.0; ^{19}F NMR (376 MHz, CDCl_3) δ -107.7, -112.7; HRMS calculated for $\text{C}_{20}\text{H}_{13}\text{F}_2\text{O}_2$ ($\text{M} + \text{H}^+$): 323.0883, found: 323.0878. (Colorless oil, 25.2 mg, 86% isolated yield)



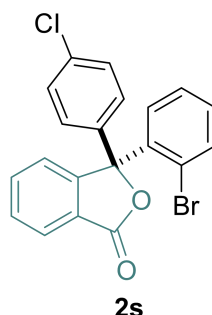
3-(4-chlorophenyl)-3-(2-fluorophenyl)isobenzofuran-1(3H)-one (2p): ^1H NMR (400 MHz, CDCl_3) δ 7.98-7.94 (d, $J = 7.6$ Hz, 1H), 7.78-7.72 (d, $J = 3.6$ Hz, 2H), 7.64-7.57 (m, 1H), 7.52-7.44 (m, 1H), 7.42-7.34 (m, 1H), 7.32-7.26 (d, $J = 7.6$ Hz, 2H), 7.24-7.18 (d, $J = 7.6$ Hz, 2H), 7.16-7.10 (m, 1H), 7.08-7.02 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.2, 160.1 (d, $J = 248.2$ Hz), 150.9, 138.5, 134.9, 134.7, 131.2, 130.0, 128.8, 128.0, 127.8, 126.3, 124.5, 124.4, 124.3 (d, $J = 12.5$ Hz), 116.9 (d, $J = 22.1$ Hz), 88.9, 68.0; ^{19}F NMR (376 MHz, CDCl_3) δ -107.7; HRMS calculated for $\text{C}_{20}\text{H}_{13}\text{ClFO}_2$ ($\text{M} + \text{H}^+$): 339.0588, found: 339.0582. (Colorless oil, 21.2 mg, 87% isolated yield)



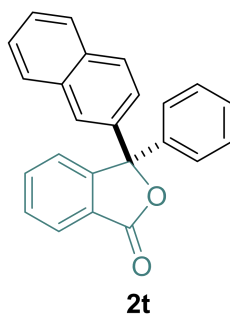
3-(4-chlorophenyl)-3-(2,6-difluorophenyl)isobenzofuran-1(3H)-one (2q): ^1H NMR (400 MHz, CDCl_3) δ 7.94 (d, $J = 8.0$ Hz, 1H), 7.70-7.65 (m, 1H), 7.62-7.56 (m, 2H), 7.38-7.28 (m, 5H), 6.90-6.82 (m 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.1, 160.8 (d, $J = 253.0$ Hz), 160.7 (d, $J = 253.0$ Hz), 150.9, 139.7, 134.6, 134.4, 131.4, 129.8, 128.9, 126.6, 125.9, 125.0, 124.5, 113.0 (d, $J = 26.8$ Hz), 87.3; ^{19}F NMR (376 MHz, CDCl_3) δ -104.2; HRMS calculated for $\text{C}_{20}\text{H}_{12}\text{ClF}_2\text{O}_2$ ($\text{M} + \text{H}^+$): 357.0494, found: 357.0488. (Pale yellow oil, 21.5 mg, 84% isolated yield)



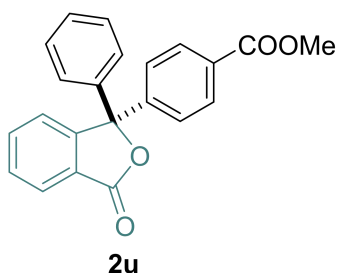
3-(2-chlorophenyl)-3-(4-chlorophenyl)isobenzofuran-1(3H)-one (2r): ^1H NMR (400 MHz, CDCl_3) δ 7.98-7.94 (d, $J = 7.6$ Hz, 1H), 7.74-7.60 (m, 2H), 7.59-7.56 (m, 1H), 7.54-7.40 (d, $J = 7.6$ Hz, 1H), 7.36-7.22 (m, 4H), 7.20-7.18 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.5, 151.1, 139.2, 136.8, 134.5, 134.4, 134.3, 132.4, 130.6, 129.9, 129.3, 128.9, 127.2, 126.6, 126.4, 125.6, 124.8, 90.8; HRMS calculated for $\text{C}_{20}\text{H}_{13}\text{Cl}_2\text{O}_2$ ($\text{M} + \text{H}^+$): 355.0292, found: 355.0287. (Pale yellow oil, 28.7 mg, 87% isolated yield)



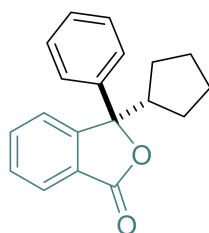
3-(2-bromophenyl)-3-(4-chlorophenyl)isobenzofuran-1(3H)-one (2s): ^1H NMR (400 MHz, CDCl_3) δ 7.98-7.92 (d, $J = 7.6$ Hz, 1H), 7.74-7.56 (m, 4H), 7.32-7.16 (m, 8H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.5, 151.2, 139.4, 138.0, 136.1, 134.4, 134.3, 130.8, 130.0, 129.9, 129.0, 127.3, 127.1, 126.4, 125.6, 124.9, 123.6, 91.5; HRMS calculated for $\text{C}_{20}\text{H}_{13}\text{BrClO}_2$ ($\text{M} + \text{H}^+$): 398.9787, found: 398.9782. (Pale yellow viscous oil, 24.4 mg, 85% isolated yield)



3-(naphthalen-1-yl)-3-phenylisobenzofuran-1(3H)-one (2t): ^1H NMR (400 MHz, CDCl_3) δ 8.00-7.98 (d, $J = 7.6$ Hz, 1H), 7.90-7.72 (d, $J = 8.8$ Hz, 5H), 7.70-7.64 (m, 1H), 7.62-7.56 (m, 1H), 7.54-7.48 (m, 2H), 7.46-7.40 (m, 1H), 7.40-7.30 (m, 5H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.9, 151.9, 140.7, 138.2, 134.3, 133.0, 132.7, 129.6, 128.7, 128.6, 128.4, 127.6, 127.3, 126.9, 126.7, 126.2, 125.8, 125.7, 125.1, 124.4, 122.3, 91.9; HRMS calculated for $\text{C}_{24}\text{H}_{17}\text{O}_2$ ($\text{M} + \text{H}^+$): 337.1228, found: 337.1223. (Yellow oil, 16.9 mg, 70% isolated yield)

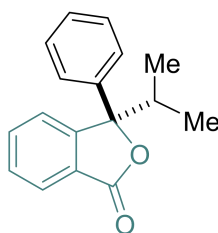


methyl-4-(3-oxo-1-phenyl-1,3-dihydroisobenzofuran-1-yl)benzoate (2u): ^1H NMR (400 MHz, CDCl_3) δ 8.02-7.98 (m, 3H), 7.72-7.70 (m, 1H), 7.62-7.58 (m, 2H), 7.54-7.42 (d, $J = 8.8$ Hz, 2H), 7.38-7.24 (m, 5H), 3.90 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.5, 166.5, 151.3, 145.8, 140.2, 134.4, 130.3, 129.8, 129.7, 128.9, 128.7, 127.2, 127.0, 126.3, 125.5, 124.2, 91.2, 52.3; HRMS calculated for $\text{C}_{22}\text{H}_{17}\text{O}_4$ ($\text{M} + \text{H}^+$): 345.1127, found: 345.1121. (Pale yellow oil, 25.3 mg, 89% isolated yield)



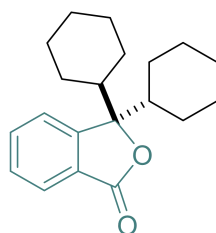
2v

3-cyclopentyl-3-phenylisobenzofuran-1(3H)-one (2v): ^1H NMR (400 MHz, CDCl_3) δ 7.92-7.89 (d, $J = 6.0$ Hz, 1H), 7.69-7.54 (m, 4H), 7.52-7.46 (s, 1H), 7.39-7.30 (s, 2H), 7.28-7.25 (d, $J = 7.2$ Hz, 1H), 3.12-3.00 (m, 1H), 1.70-1.42 (m, 7H), 1.30-1.22 (d, $J = 7.2$ Hz, 1H), 1.10-1.02 (d, $J = 8.0$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 170.6, 153.6, 141.2, 134.3, 129.0, 128.7, 127.8, 125.7, 125.4, 124.8, 122.0, 91.3, 48.3, 27.8, 26.8, 25.9, 25.6; HRMS calculated for $\text{C}_{19}\text{H}_{19}\text{O}_2$ ($\text{M} + \text{H}^+$): 279.1385, found: 279.1379. (Colorless oil, 20.3 mg, 72% isolated yield)



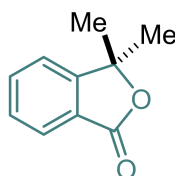
2w

3-isopropyl-3-phenylisobenzofuran-1(3H)-one (2w): ^1H NMR (400 MHz, CDCl_3) δ 7.88-7.84 (d, $J = 7.2$ Hz, 1H), 7.68-7.62 (m, 1H), 7.60-7.52 (m, 3H), 7.50-7.46 (t, $J = 14.8$ Hz, 1H), 7.40-7.32 (m, 2H), 7.30-7.24 (m, 1H), 2.82-2.72 (m, 1H), 1.00-0.92 (d, $J = 6.8$ Hz, 3H), 0.80-0.70 (d, $J = 6.8$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 170.5, 152.7, 140.7, 134.3, 129.0, 128.7, 127.9, 125.8, 125.6, 124.8, 122.2, 92.8, 36.9, 17.4, 16.4; These data are consistent with literature values, see: Y. Nakamura, S. Yoshida, and T. Hosoya, *Chem. Lett.*, 2017, **46**, 858. (Yellow oil, 16.1 mg, 68% isolated yield)



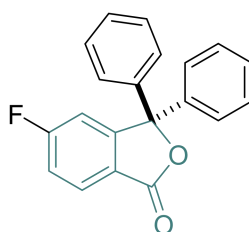
2x

3,3-dicyclohexylisobenzofuran-1(3H)-one (2x): ^1H NMR (400 MHz, CDCl_3) δ 7.88-7.80 (d, $J = 7.6$ Hz, 1H), 7.65-7.60 (m, 1H), 7.50-7.42 (m, 1H), 7.38-7.32 (d, $J = 8.0$ Hz, 1H), 2.14-2.02 (m, 2H), 1.74-1.62 (m, 11H), 1.48-1.38 (m, 2H), 1.28-1.18 (m, 5H), 1.16-1.12 (m, 3H), 0.80-0.72 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 171.1, 151.3, 133.5, 128.6, 127.8, 125.5, 122.0, 94.8, 41.6, 26.7, 26.6, 26.4, 26.3, 26.2; HRMS calculated for $\text{C}_{20}\text{H}_{27}\text{O}_2$ ($\text{M} + \text{H}^+$): 299.2011, found: 299.2005. (Yellow oil, 22.6 mg, 83% isolated yield)



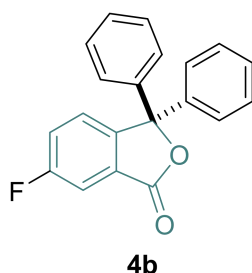
2y

3,3-dimethylisobenzofuran-1(3H)-one (2y): ^1H NMR (400 MHz, CDCl_3) δ 7.89-7.85 (d, $J = 7.6$ Hz, 1H), 7.69-7.64 (m, 1H), 7.54-7.49 (m, 1H), 7.42-7.39 (d, $J = 7.6$ Hz, 1H), 1.67 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.9, 155.0, 134.2, 129.0, 125.8, 125.3, 120.7, 85.5, 27.4.; These data are consistent with literature values, see: S. Zhang, L. Li, H. Wang, Q. Li, W. Liu, K. Xu, and C. Zeng, *Org. Lett.*, 2018, **20**, 252. (Yellow oil, 17.8 mg, 78% isolated yield)

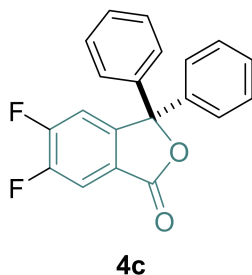


4a

5-fluoro-3,3-diphenylisobenzofuran-1(3H)-one (4a): ^1H NMR (400 MHz, CDCl_3) δ 8.00-7.92 (q, $J = 4.8$ Hz, 1H), 7.38-7.30 (m, 11H), 7.28-7.22 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 168.6, 166.5 (d, $J = 254.9$ Hz), 154.8 (d, $J = 9.6$ Hz), 140.3, 128.9, 128.7, 128.6, 128.4, 127.1, 117.8 (d, $J = 24.0$ Hz), 111.7 (d, $J = 24.9$ Hz), 91.1; ^{19}F NMR (376 MHz, CDCl_3) δ -101.7; HRMS calculated for $\text{C}_{20}\text{H}_{14}\text{FO}_2$ ($\text{M} + \text{H}^+$): 305.0978, found: 305.0972. (Pale yellow solid, 26.0 mg, 92% isolated yield)

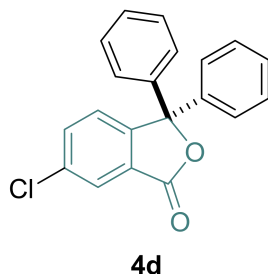


6-fluoro-3,3-diphenylisobenzofuran-1(3H)-one (4b): ^1H NMR (400 MHz, CDCl_3) δ 7.62-7.52 (m, 2H), 7.48-7.35 (m, 1H), 7.34-7.28 (d, $J = 3.2$ Hz, 10H); ^{13}C NMR (100 MHz, CDCl_3) δ 168.6, 163.3 (d, $J = 249.1$ Hz), 147.7, 140.6, 128.8, 128.7, 127.7, 127.1, 126.0, 122.2 (d, $J = 23.9$ Hz), 112.4 (d, $J = 24.0$ Hz), 91.8; ^{19}F NMR (376 MHz, CDCl_3) δ -110.9; HRMS calculated for $\text{C}_{20}\text{H}_{14}\text{FO}_2$ ($\text{M} + \text{H}^+$): 305.0978, found: 305.0972. (Colorless solid, 25.7 mg, 91% isolated yield)

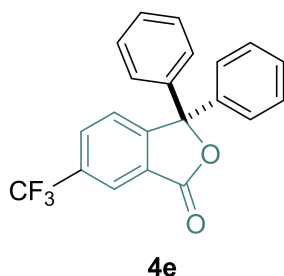


5,6-difluoro-3,3-diphenylisobenzofuran-1(3H)-one (4c): ^1H NMR (400 MHz, CDCl_3) δ 7.76-7.69 (m, 1H), 7.39-7.33 (m, 7H), 7.32-7.27 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 167.8, 154.5 (d, $J = 243.0$ Hz), 149.7 (d, $J = 163.9$ Hz), 149.6 (d, $J = 164.7$ Hz), 140.0, 129.0, 128.8, 127.0, 122.0 (d, $J = 6.7$ Hz), 114.6 (d, $J = 19.2$ Hz),

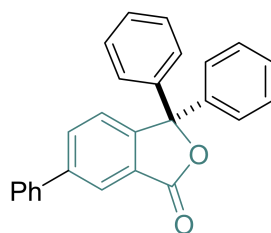
113.5 (d, $J = 20.2$ Hz), 91.3; ^{19}F NMR (376 MHz, CDCl_3) δ -124.1, -132.9; HRMS calculated for $\text{C}_{20}\text{H}_{13}\text{F}_2\text{O}_2$ ($\text{M} + \text{H}^+$): 323.0883, found: 323.0878. (Pale yellow solid, 27.9 mg, 89% isolated yield)



6-chloro-3,3-diphenylisobenzofuran-1(3H)-one (4d): ^1H NMR (400 MHz, CDCl_3) δ 7.90 (d, $J = 1.6$ Hz, 1H), 7.68-7.62 (m, 1H), 7.52-7.50 (d, $J = 8.4$ Hz, 1H), 7.38-7.28 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3) δ 168.4, 150.3, 140.4, 135.8, 134.5, 128.8, 128.7, 127.4, 127.1, 125.9, 125.5, 91.8; These data are consistent with literature values, see: Y. Nakamura, S. Yoshida, and T. Hosoya, *Chem. Lett.*, 2017, **46**, 858. (Pale brown solid, 26.4 mg, 87% isolated yield)

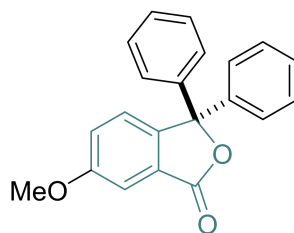


3,3-diphenyl-6-(trifluoromethyl)isobenzofuran-1(3H)-one (4e): ^1H NMR (400 MHz, CDCl_3) δ 8.25 (s, 1H), 7.99-7.92 (d, $J = 8.4$ Hz, 1H), 7.76-7.60 (d, $J = 8.4$ Hz, 1H), 7.40-7.28 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3) δ 168.3, 155.1, 139.9, 132.5, 132.2, 131.1, 129.0, 128.8, 127.1, 125.2 (q, $J = 288.5$ Hz), 125.1, 124.7, 92.1; ^{19}F NMR (376 MHz, CDCl_3) δ -62.4; HRMS calculated for $\text{C}_{21}\text{H}_{14}\text{F}_3\text{O}_2$ ($\text{M} + \text{H}^+$): 355.0946, found: 355.0940. (Pale brown solid, 26.3 mg, 92% isolated yield)



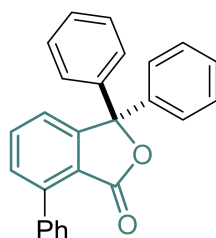
4f

3,3,6-triphenylisobenzofuran-1(3H)-one (4f): ^1H NMR (400 MHz, CDCl_3) δ 8.15 (d, $J = 0.8$ Hz, 1H), 7.92-7.88 (q, $J = 9.6$ Hz, 1H), 7.68-7.58 (m, 3H), 7.50-7.46 (t, $J = 14.8$ Hz, 2H), 7.42-7.28 (m, 12H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.8, 150.8, 143.1, 140.9, 139.3, 133.4, 129.2, 128.7, 128.6, 128.3, 127.3, 127.2, 126.4, 124.6, 124.3, 91.7. HRMS calculated for $\text{C}_{26}\text{H}_{19}\text{O}_2$ ($\text{M} + \text{H}^+$): 363.1385, found: 363.1379. (Pale yellow solid, 26.5 mg, 76% isolated yield)



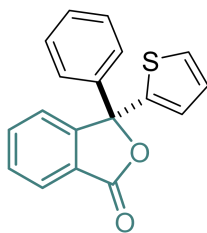
4g

6-methoxy-3,3-diphenylisobenzofuran-1(3H)-one (4g): ^1H NMR (400 MHz, CDCl_3) δ 7.40-7.30 (m, 11H), 7.08-7.03 (d, $J = 8.4$ Hz, 1H), 6.92 (s, 1H), 7.26 (s, 0.84H), 3.87 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.5, 164.7, 154.6, 141.0, 128.6, 128.5, 127.6, 127.2, 117.9, 116.3, 108.8, 91.0, 56.0; HRMS calculated for $\text{C}_{21}\text{H}_{17}\text{O}_3$ ($\text{M} + \text{H}^+$): 317.1177, found: 317.1172. (Pale yellow solid, 24.9 mg, 88% isolated yield)



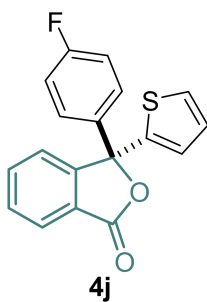
4h

3,3,7-triphenylisobenzofuran-1(3*H*)-one (4h): ¹H NMR (400 MHz, CDCl₃) δ 7.73-7.68 (m, 1H), 7.58-7.52(m, 3H), 7.49-7.43 (m, 4H), 7.40-7.32 (m, 10H); ¹³C NMR (100 MHz, CDCl₃) δ 168.5, 153.3, 143.1, 141.2, 136.4, 133.9, 131.2, 129.7, 128.6, 128.5, 128.1, 127.3, 123.2, 121.5, 90.0; HRMS calculated for C₂₆H₁₉O₂ (M + H⁺): 363.1386, found: 363.1379. (White solid, 18.6 mg, 62% isolated yield)



4i

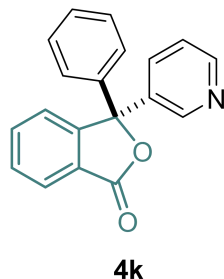
3-phenyl-3-(thiophen-2-yl)isobenzofuran-1(3*H*)-one (4i): ¹H NMR (400 MHz, CDCl₃) δ 7.98-7.92 (d, *J* = 7.6 Hz, 1H), 7.78-7.68 (m, 1H), 7.66-7.62 (d, *J* = 8.0 Hz, 1H), 7.60-7.55 (m, 1H), 7.52-7.46 (m, 2H), 7.42-7.32 (m, 4H), 6.98-6.92 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 169.3, 152.1, 144.6, 140.5, 134.4, 129.8, 128.9, 128.6, 127.9, 127.3, 126.7, 126.4, 126.1, 125.2, 123.8, 88.7; HRMS calculated for C₁₈H₁₃O₂S (M + H⁺): 293.0636, found: 293.0630. (Purple solid, 25.3 mg, 90% isolated yield)



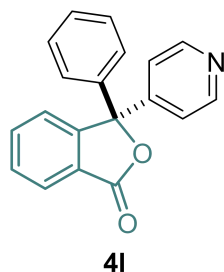
4j

3-(4-fluorophenyl)-3-(thiophen-2-yl)isobenzofuran-1(3*H*)-one (4j): ¹H NMR (400 MHz, CDCl₃) δ 7.95-7.92 (d, *J* = 8.0 Hz, 1H), 7.75-7.69 (m, 1H), 7.62-7.55 (m, 2H), 7.47-7.41 (m, 2H), 7.35-7.32 (m, 1H), 7.06-6.99 (m, 2H), 6.97-6.90 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 169.2, 162.9 (d, *J* = 247.3 Hz), 151.0, 144.4, 140.5, 134.6, 129.9, 128.5, 127.9, 127.5, 126.8, 126.3, 125.2, 123.7, 115.5 (d, *J* = 21.1 Hz), 88.7;

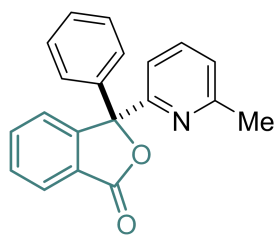
^{19}F NMR (376 MHz, CDCl_3) δ -115.1; HRMS calculated for $\text{C}_{18}\text{H}_{12}\text{FO}_2\text{S}$ ($\text{M} + \text{H}^+$): 311.0543, found:311.0537. (Purple solid, 23 mg, 84% isolated yield)



3-phenyl-3-(pyridin-3-yl)isobenzofuran-1(3H)-one (4k): ^1H NMR (400 MHz, CDCl_3) δ 8.60 (s, 2H), 8.00-7.96 (d, $J = 8.0$ Hz, 1H), 7.77-7.68 (m, 2H), 7.63-7.58 (m, 2H), 7.39-7.28 (m, 7H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.3, 151.0, 149.9, 148.3, 139.9, 136.9, 135.0, 134.6, 129.9, 129.0, 128.8, 126.9, 126.4, 125.4, 124.1, 123.4, 90.0; HRMS calculated for $\text{C}_{19}\text{H}_{14}\text{NO}_2$ ($\text{M} + \text{H}^+$): 288.1025, found: 288.1019. (Pale yellow liquid, 16.8 mg, 56% isolated yield)

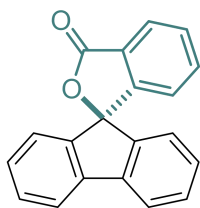


4-phenyl-3-(pyridin-4-yl)isobenzofuran-1(3H)-one (4l): ^1H NMR (400 MHz, CDCl_3) δ 8.62-8.58 (d, $J = 5.6$ Hz, 2H), 8.00-7.96 (d, $J = 7.2$ Hz, 1H), 7.78-7.72 (t, $J = 7.6$ Hz, 1H), 7.65-7.58 (m, 2H), 7.39-7.25 (m, 7H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.2, 150.5, 150.1, 150.0, 139.2, 134.6, 130.0, 129.2, 128.8, 127.3, 126.5, 125.5, 124.0, 121.4, 90.1; HRMS calculated for $\text{C}_{19}\text{H}_{14}\text{NO}_2$ ($\text{M} + \text{H}^+$): 288.1025, found: 288.1019. (Yellow liquid, 15.3 mg, 53% isolated yield)



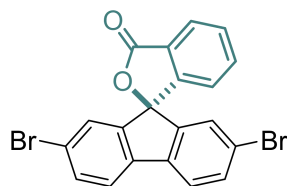
4m

3-(6-methylpyridin-2-yl)-3-phenylisobenzofuran-1(3H)-one (4m): ^1H NMR (400 MHz, CDCl_3) δ 8.24-8.19 (d, $J = 7.6$ Hz, 1H), 7.90-7.86 (d, $J = 8.0$ Hz, 1H), 7.73-7.68 (m, 1H), 7.62-7.51 (m, 3H), 7.44-7.40 (m, 2H), 7.32-7.25 (m, 3H), 7.07-7.03 (dd, $J_1 = 6.8$ Hz, $J_2 = 1.2$ Hz, 1H), 2.53 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 170.0, 158.7, 158.0, 151.8, 140.8, 137.1, 134.0, 129.3, 128.4, 128.3, 126.7, 126.2, 125.2, 125.1, 122.4, 117.2, 90.3, 24.6; HRMS calculated for $\text{C}_{20}\text{H}_{16}\text{NO}_2$ ($\text{M} + \text{H}^+$): 302.1182, found:302.1176. (Pale yellow liquid, 16.8 mg, 55% isolated yield)



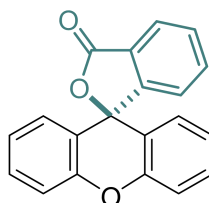
4n

3'H-spiro[fluorene-9,1'-isobenzofuran]-3'-one (4n): ^1H NMR (400 MHz, CDCl_3) δ 8.06-8.00 (d, $J = 6.8$ Hz, 1H), 7.74-7.70 (d, $J = 7.6$ Hz, 2H), 7.62-7.48 (m, 2H), 7.46-7.38 (m, 2H), 7.28-7.18 (m, 2H), 7.08-6.98 (d, $J = 7.6$ Hz, 2H), 6.96-6.94 (t, $J = 6.8$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 170.7, 150.6, 142.8, 140.8, 134.8, 130.5, 129.6, 128.5, 126.0, 125.7, 124.6, 122.3, 120.6, 91.9; Commercial Sources: CAS 82111-99-7. (Brown solid, 23.1 mg, 89% isolated yield)



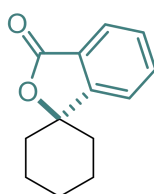
4o

2,7-dibromo-3'*H*-spiro[fluorene-9,1'-isobenzofuran]-3'-one (4o): ^1H NMR (400 MHz, CDCl_3) δ 8.10-8.04 (t, $J = 7.6$ Hz, 1H), 7.64-7.60 (m, 2H), 7.58-7.52 (t, $J = 6.8$ Hz, 4H), 7.14 (s, 2H), 6.98-6.92 (d, $J = 6.8$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.9, 149.0, 144.5, 138.7, 135.2, 133.8, 130.2, 128.0, 126.1, 125.7, 122.6, 122.3, 122.0, 90.6; HRMS calculated for $\text{C}_{20}\text{H}_{11}\text{Br}_2\text{O}_4$ ($\text{M} + \text{H}^+$): 440.9126, found: 440.9120. (Pale yellow solid, 30.9 mg, 74% isolated yield)



4p

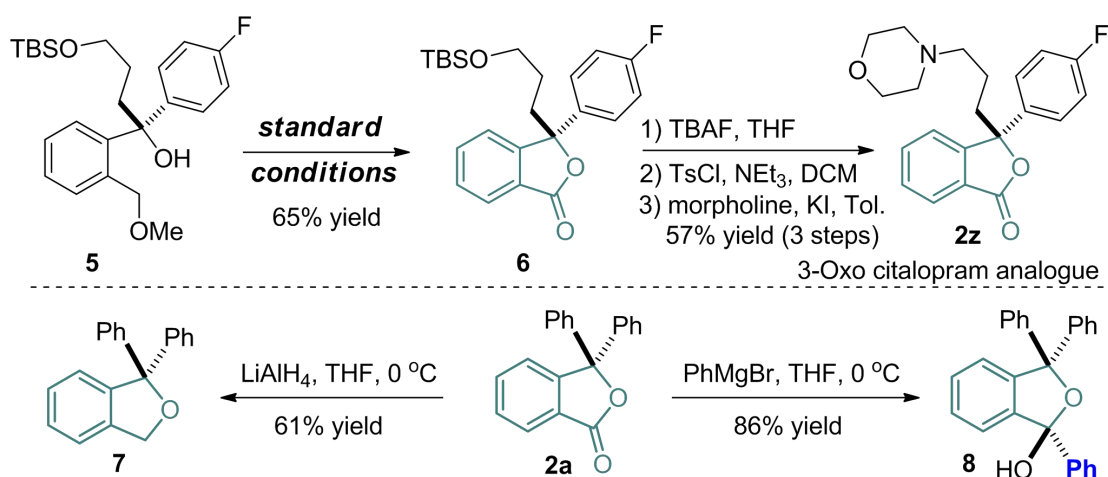
3'*H*-spiro[isobenzofuran-1,9'-xanthen]-3-one (4p): ^1H NMR (400 MHz, CDCl_3) δ 8.06-8.02 (d, $J = 7.2$ Hz, 1H), 7.68-7.62 (m, 2H), 7.45-7.39 (m, 2H), 7.38-7.28 (d, $J = 8.8$ Hz, 2H), 7.18-7.14 (d, $J = 7.6$ Hz, 1H), 7.08-7.02 (m, 2H), 6.88-6.82 (d, $J = 7.6$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.6, 153.6, 151.2, 135.2, 130.7, 129.8, 128.0, 126.1, 125.2, 123.9, 123.7, 118.9, 117.2, 88.4; HRMS calculated for $\text{C}_{20}\text{H}_{13}\text{O}_3$ ($\text{M} + \text{H}^+$): 301.0864, found: 301.0859. (Purple solid, 20.3 mg, 72% isolated yield)



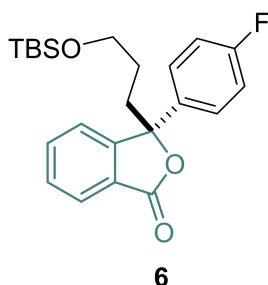
4q

3'*H*-spiro[cyclohexane-1,1'-isobenzofuran]-3'-one (4q): ^1H NMR (400 MHz, CDCl_3) δ 7.90-7.84 (d, $J = 7.2$ Hz, 1H), 7.68-7.62 (m, 1H), 7.54-7.48 (m, 1H), 7.42-7.36 (d, $J = 7.2$ Hz, 1H), 1.92-1.72 (m, 10H); ^{13}C NMR (100 MHz, CDCl_3) δ 170.2, 155.0, 134.0, 129.0, 125.9, 125.6, 121.0, 87.1, 36.4, 24.8, 22.4; These data are consistent with literature values, see: S. Zhang, L. Li, H. Wang, Q. Li, W. Liu, K. Xu, and C. Zeng, *Org. Lett.*, 2018, **20**, 252. (Pale yellow solid, 11.2 mg, 60% isolated yield)

Illustrative synthetic transformations

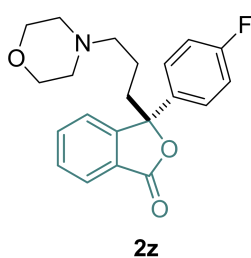


^1H , ^{13}C , and ^{19}F spectra data of compounds **6**, **2z**, **7-8**, **11**

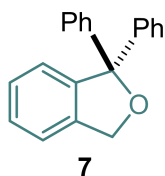


3-(3-((tert-butyldimethylsilyloxy)propyl)-3-(4-fluorophenyl)isobenzofuran-1(3*H*))-one (6): ^1H NMR (400 MHz, CDCl_3) δ 7.82-7.78 (d, $J = 7.2$ Hz, 1H), 7.60-7.54 (m,

1H), 7.44-7.35 (m, 3H), 6.98-6.92 (m, 2H), 3.48-3.43 (t, $J = 6.0$ Hz, 2H), 2.46-2.38 (m, 1H), 2.21-2.16 (m, 1H), 1.42-1.35 (m, 1H), 1.24-1.12 (s, 3H), 0.76 (s, 9H), -0.12 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.9, 162.5 (d, $J = 248.3$ Hz), 152.8, 137.5, 134.4, 129.3, 127.0, 126.0, 125.4, 122.1, 115.8 (d, $J = 21.1$ Hz), 89.7, 62.3, 36.8, 29.7, 27.1, 25.9, 25.7, 18.3, -3.4, -5.2; ^{19}F NMR (376 MHz, CDCl_3) δ -113.4; HRMS calculated for $\text{C}_{23}\text{H}_{29}\text{O}_3\text{FNaSi}$ ($\text{M} + \text{Na}^+$): 423.1768, found: 423.1762. (Pale yellow oil, 217.8 mg, 65% isolated yield)

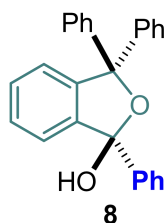


3-(4-fluorophenyl)-3-(3-morpholinopropyl)isobenzofuran-1(3H)-one (2z): ^1H NMR (400 MHz, CDCl_3) δ 7.92-7.88 (d, $J = 7.2$ Hz, 1H), 7.72-7.64 (m, 1H), 7.58-7.48 (m, 4H), 7.08-6.98 (m, 2H), 3.68-3.64 (t, $J = 4.6$ Hz, 4H), 2.55-2.46 (m, 1H), 2.38-2.28 (m, 6H), 2.26-2.24 (m, 1H), 1.52-1.42 (m, 1H), 1.35-1.22 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.9, 162.5 (d, $J = 247.2$ Hz), 152.7, 136.2, 134.5, 129.4, 126.9, 126.8, 126.1, 125.4, 122.0, 115.8 (d, $J = 21.1$ Hz), 89.5, 66.8, 58.2, 53.5, 38.0, 20.7; ^{19}F NMR (376 MHz, CDCl_3) δ -113.7; HRMS calculated for $\text{C}_{21}\text{H}_{23}\text{O}_3\text{NF}$ ($\text{M} + \text{H}^+$): 356.1656, found: 356.1662. [Pale yellow solid, 18mg, 57% isolated yield (3 steps)]

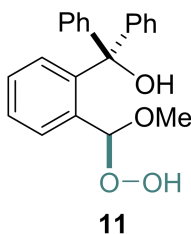


1,1-diphenyl-1,3-dihydroisobenzofuran (7): ^1H NMR (400 MHz, CDCl_3) δ 7.35-7.20 (m, 12H), 4.38 (s, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 144.7, 144.2, 139.7,

128.1, 127.8, 127.5, 127.43, 123.9, 121.2, 93.1, 71.6, 68.1; HRMS calculated for $C_{20}H_{17}O$ ($M + H^+$): 273.1279, found: 273.1274. (Pale yellow solid, 34.7 mg, 61% isolated yield)

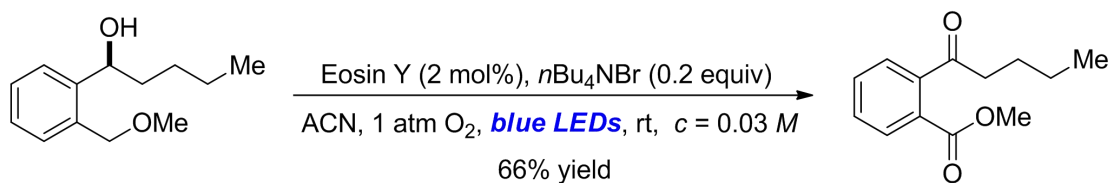


1,3,3-triphenyl-1,3-dihydroisobenzofuran-1-ol (8): 1H NMR (400 MHz, $CDCl_3$) δ 7.60-7.52 (m, 3H), 7.42-7.38 (m, 2H), 7.36-7.24 (m, 13H), 7.20 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.0, 144.6, 143.4, 142.7, 142.5, 137.6, 134.3, 128.8, 128.5, 128.4, 128.3, 128.2, 127.9, 127.8, 127.6, 127.1, 126.5, 124.1, 115.4, 108.0, 93.1; HRMS calculated for $C_{26}H_{20}O_2Na$ ($M + Na^+$): 387.1361, found: 387.1353. (white solid, 65.7 mg, 86% isolated yield)



(2-(hydroperoxy(methoxy)methyl)phenyl)diphenylmethanol (11): 1H NMR (400 MHz, $CDCl_3$) δ 7.72-7.70 (d, $J = 7.6$ Hz, 1H), 7.40-7.25 (m, 11H), 7.24-7.16 (m, 3H), 6.71-6.66 (d, $J = 8.0$ Hz, 1H), 5.95 (s, 1H), 4.30-4.20 (m, 1H), 3.12 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.6, 146.5, 144.4, 135.0, 129.8, 128.8, 128.7, 128.5, 128.2, 128.1, 128.0, 127.6, 127.5, 127.4, 106.8, 82.7, 57.4. HRMS calculated for $C_{21}H_{20}NaO_4$ ($M + Na^+$): 359.1260, found: 359.1253. (Colorless oil, 28.4 mg, 91% isolated yield)

Investigation of the secondary alcohol



methyl 2-pentanoylbenzoate : ^1H NMR (400 MHz, CDCl_3) δ 7.91-7.88 (m, 1H), 7.59-7.42 (m, 2H), 7.38-7.35 (d, $J = 7.6$ Hz, 1H), 3.9 (s, 3H), 2.83-2.78 (t, $J = 7.6$ Hz, 2H), 1.76-1.68 (m, 2H), 1.44-1.34 (m, 2H), 0.96-0.88 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 206.0, 167.3, 143.4, 134.0, 132.1, 129.9, 129.7, 126.3, 52.6, 42.6, 26.2, 22.4, 14.0; These data are consistent with literature values, see: Lu, B.; Zhao, M.; Ding, G.; Xie, X.; Jiang, L.; Ratovelomanana-Vidal, V.; Zhang, Z. *ChemCatChem*. 2017, **9**, 3989.

Fluorescence quenching experiments

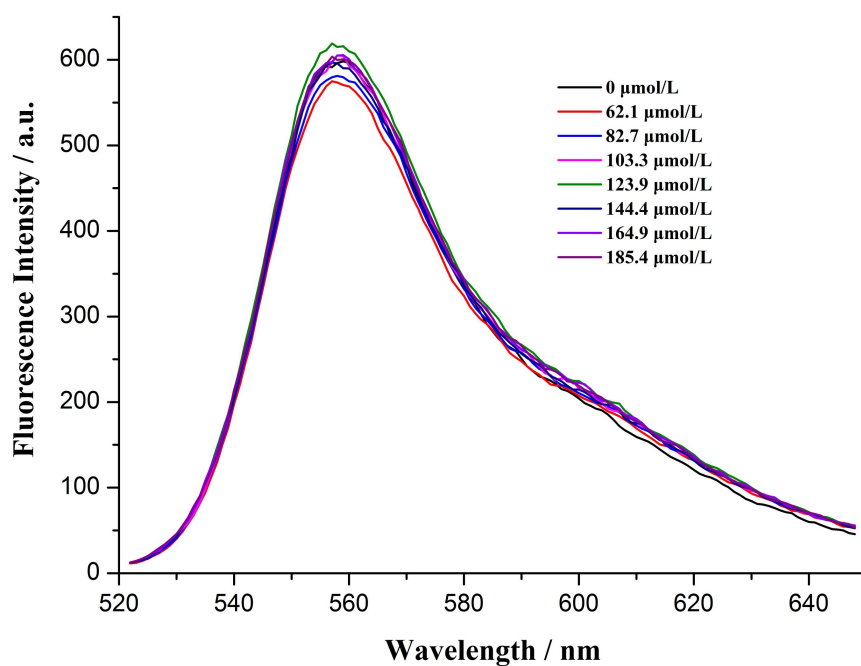


Figure 1. Fluorescence spectra of Eosin Y with different concentrations of tetrabutylammonium bromide

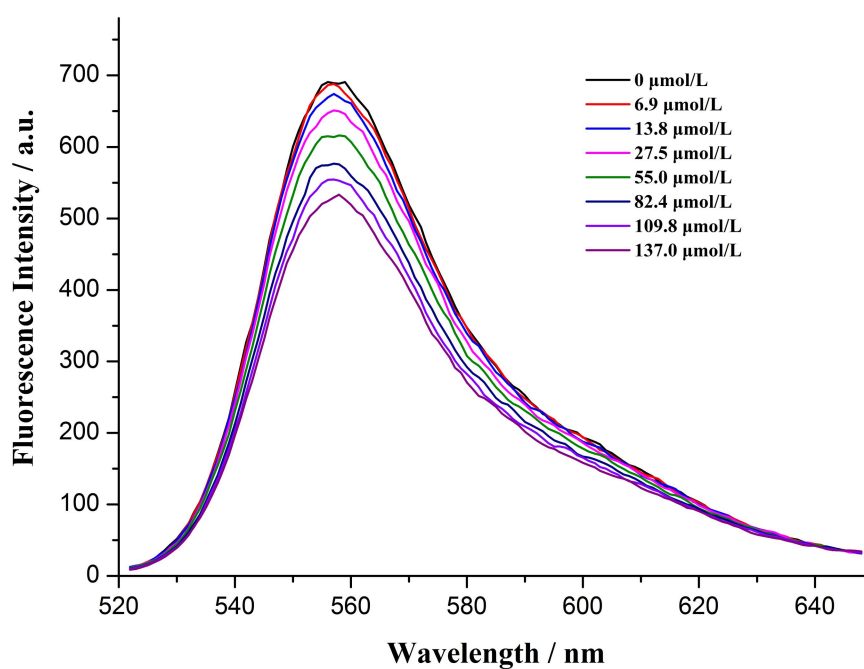


Figure 2. Fluorescence spectra of Eosin Y with different concentrations of substrate **1a**

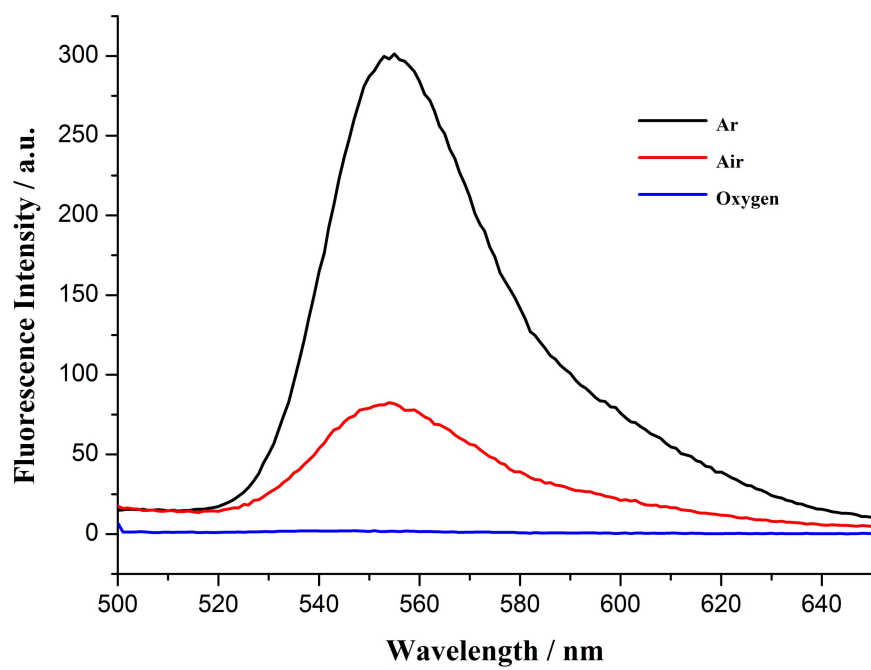


Figure 3. Fluorescence spectra of Eosin Y with different concentrations of O₂

CV curve of the substrate 1a

Cyclic voltammetry (CV) was performed using an Epsilon electrochemical workstation (a BASi three-electrode cell system): glassy carbon electrode as the working electrode, Pt wire as the counter electrode, Ag/AgCl (KCl, 3 M) electrode as the reference electrode, and ferroceniumferrocene (Fc⁺/Fc) as the internal standard. Scan rate: 100 mV s⁻¹ (in the range -2.2 to +2.5 V). Bu₄NPF₆ (0.1 M MeCM) was used as the supporting electrolyte.

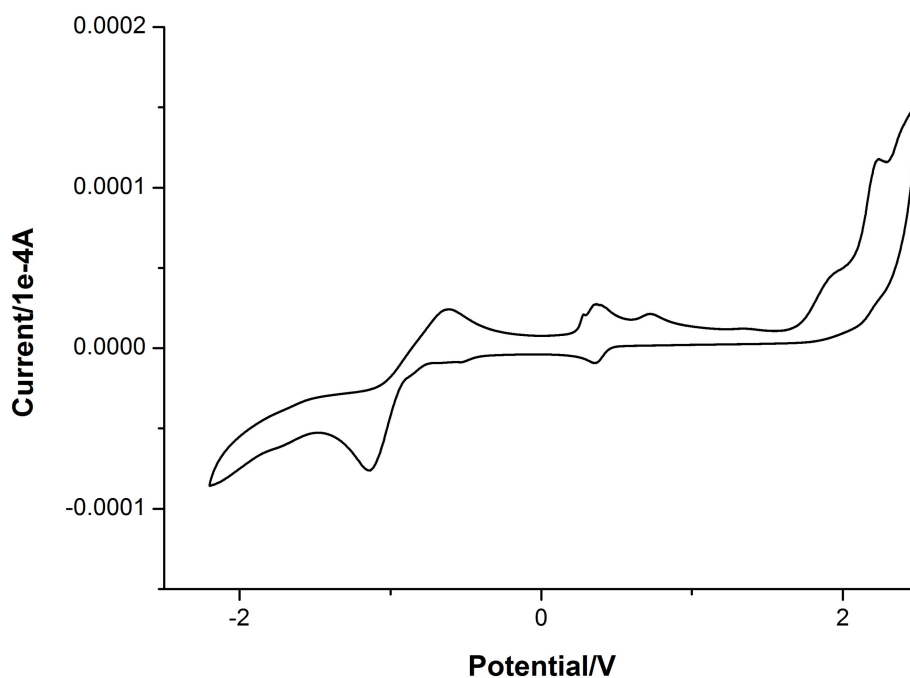
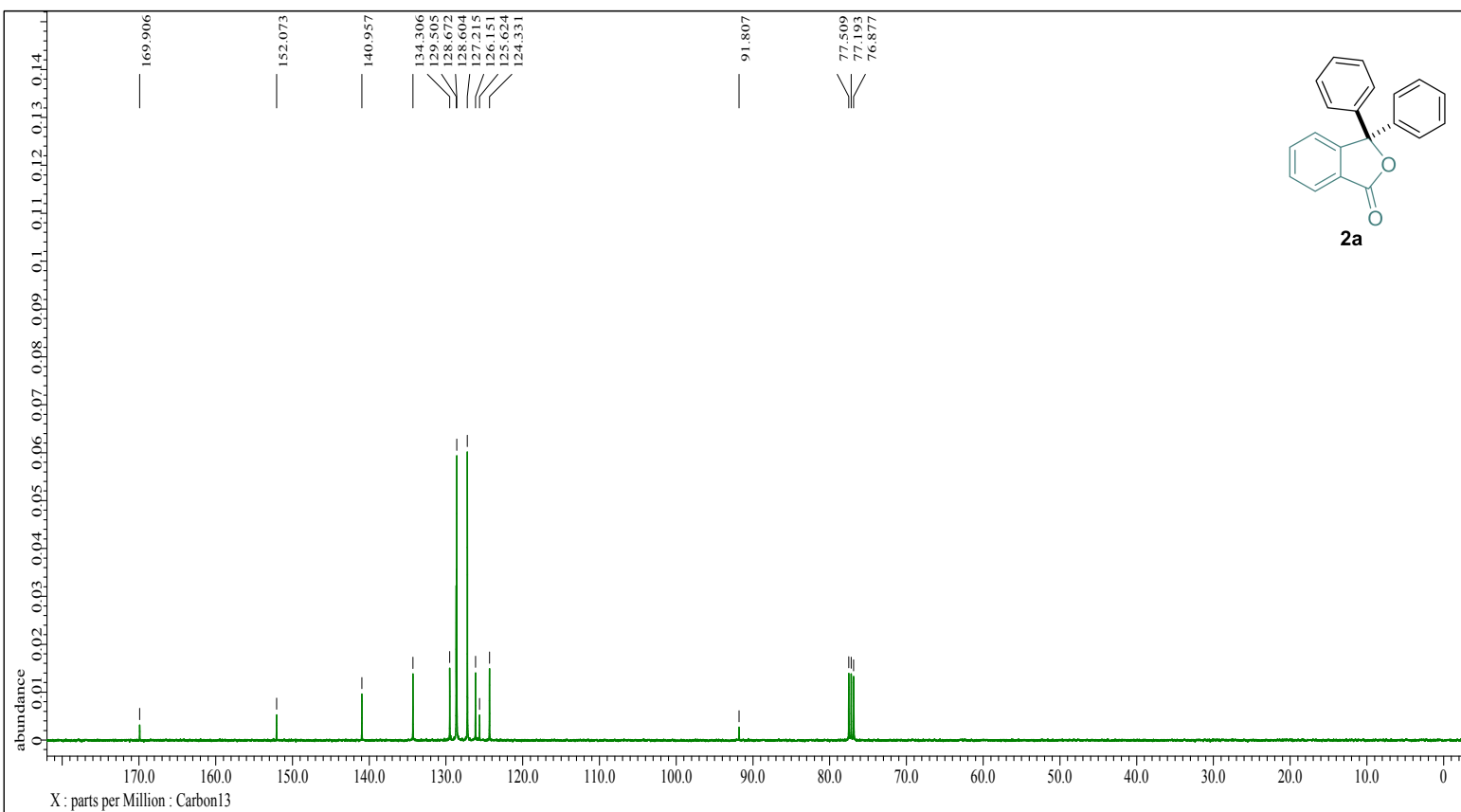
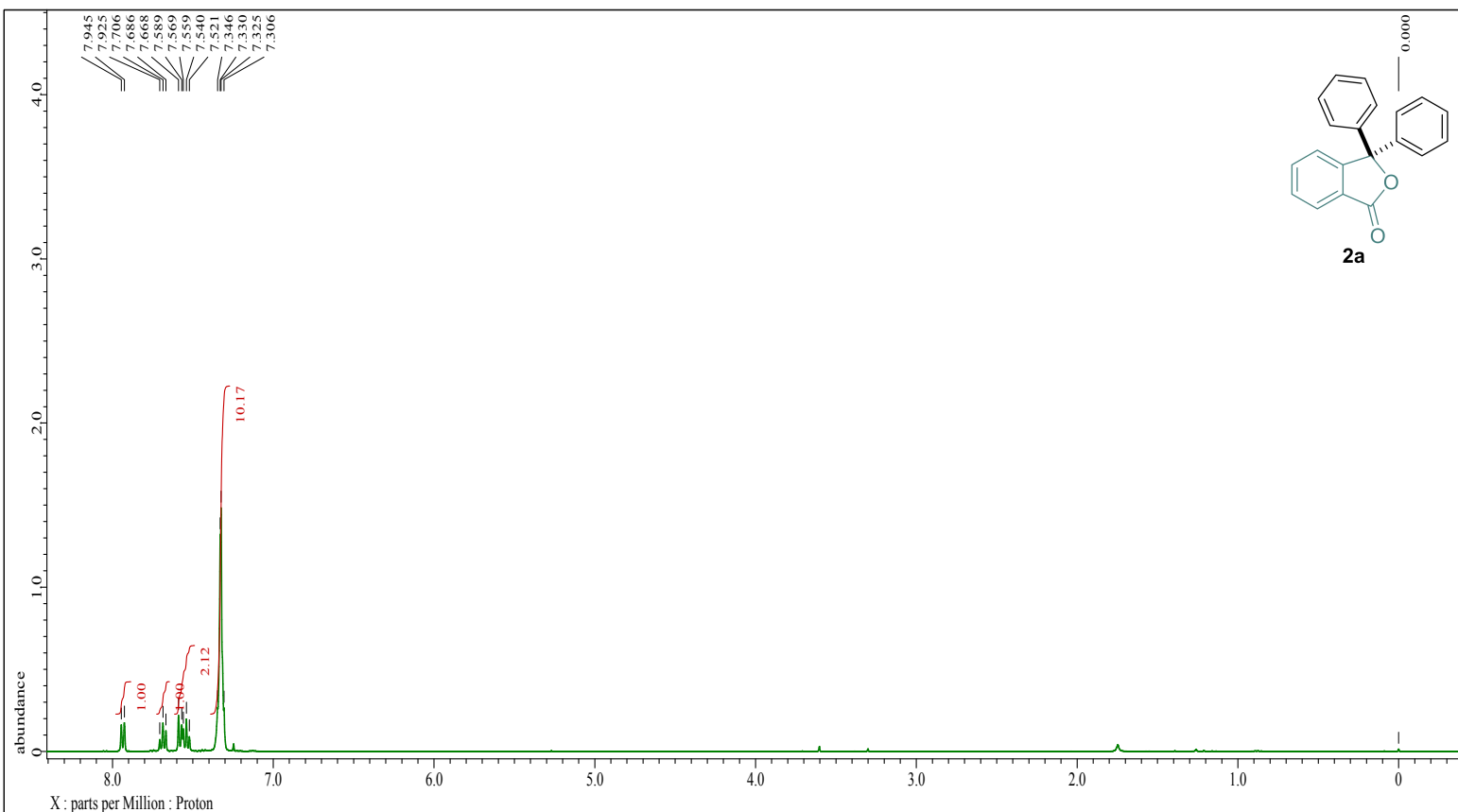
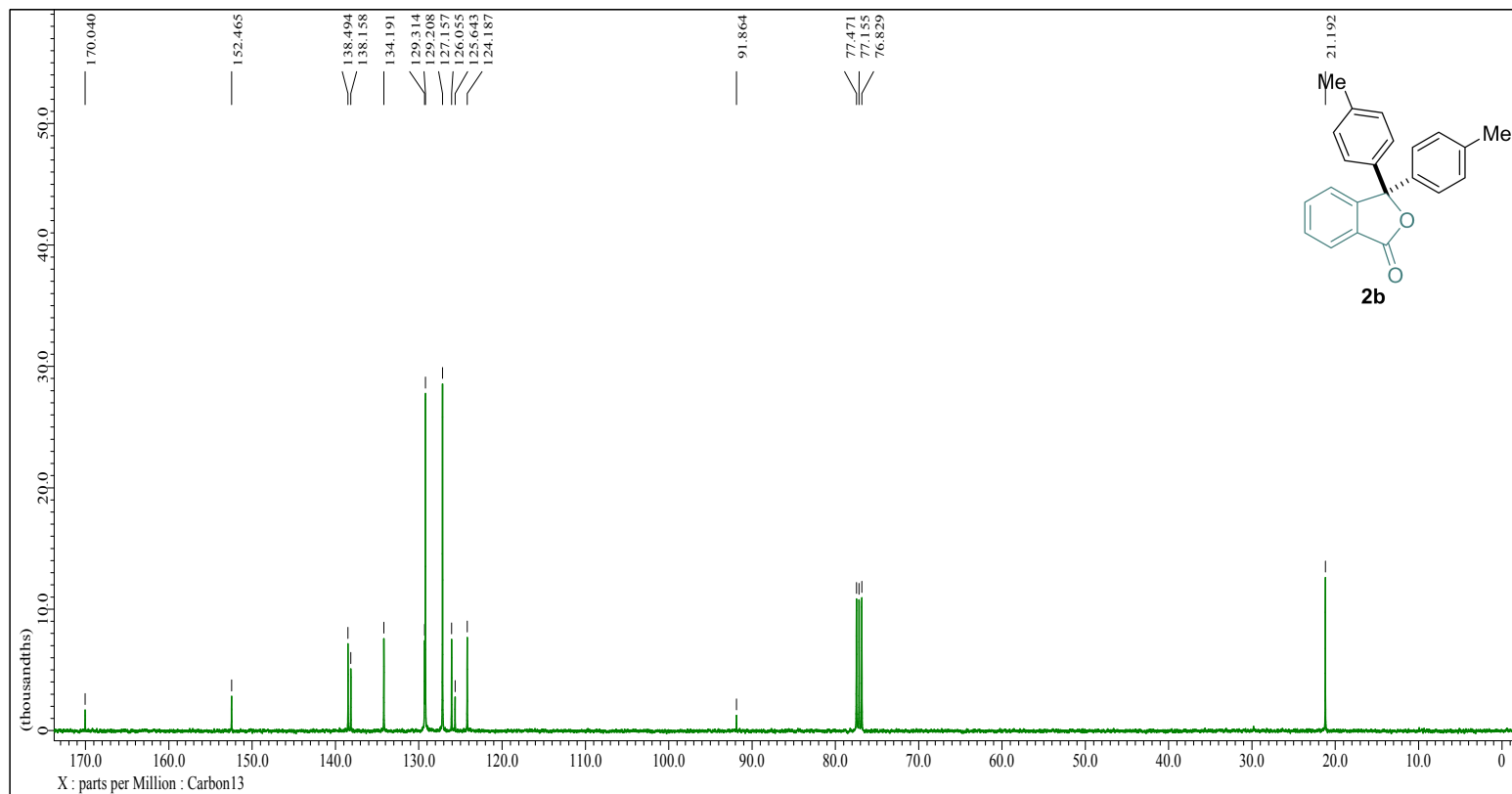
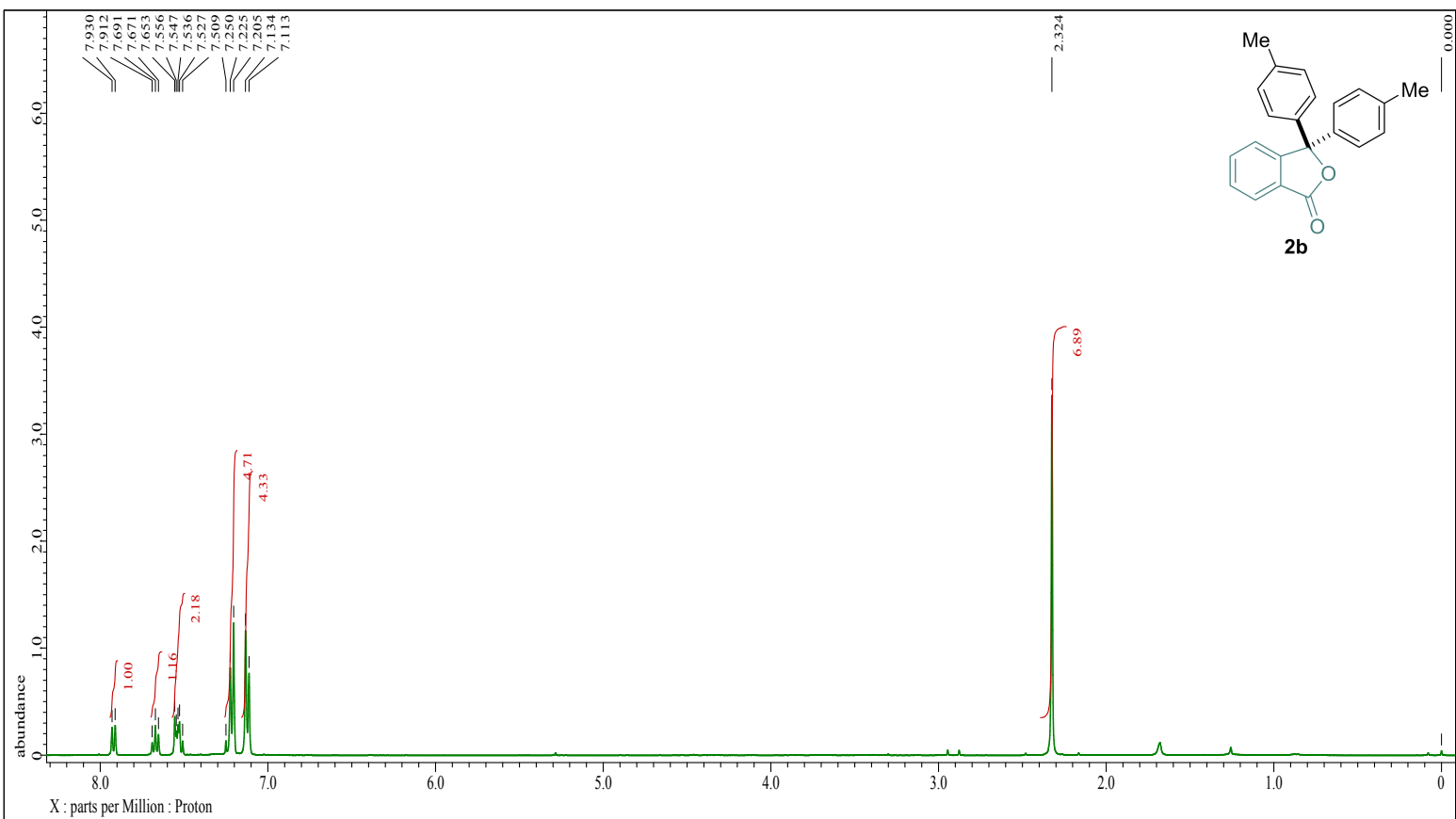
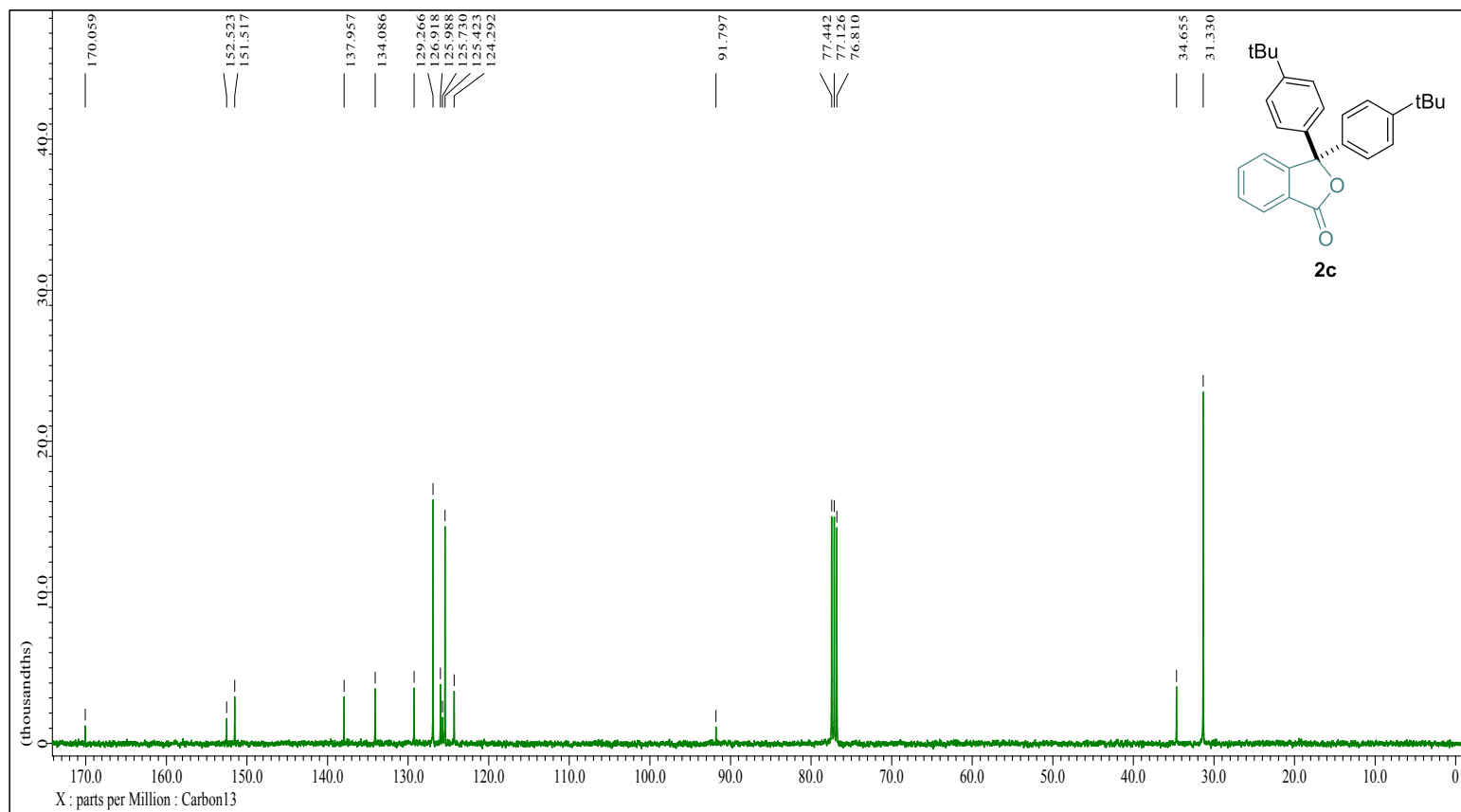
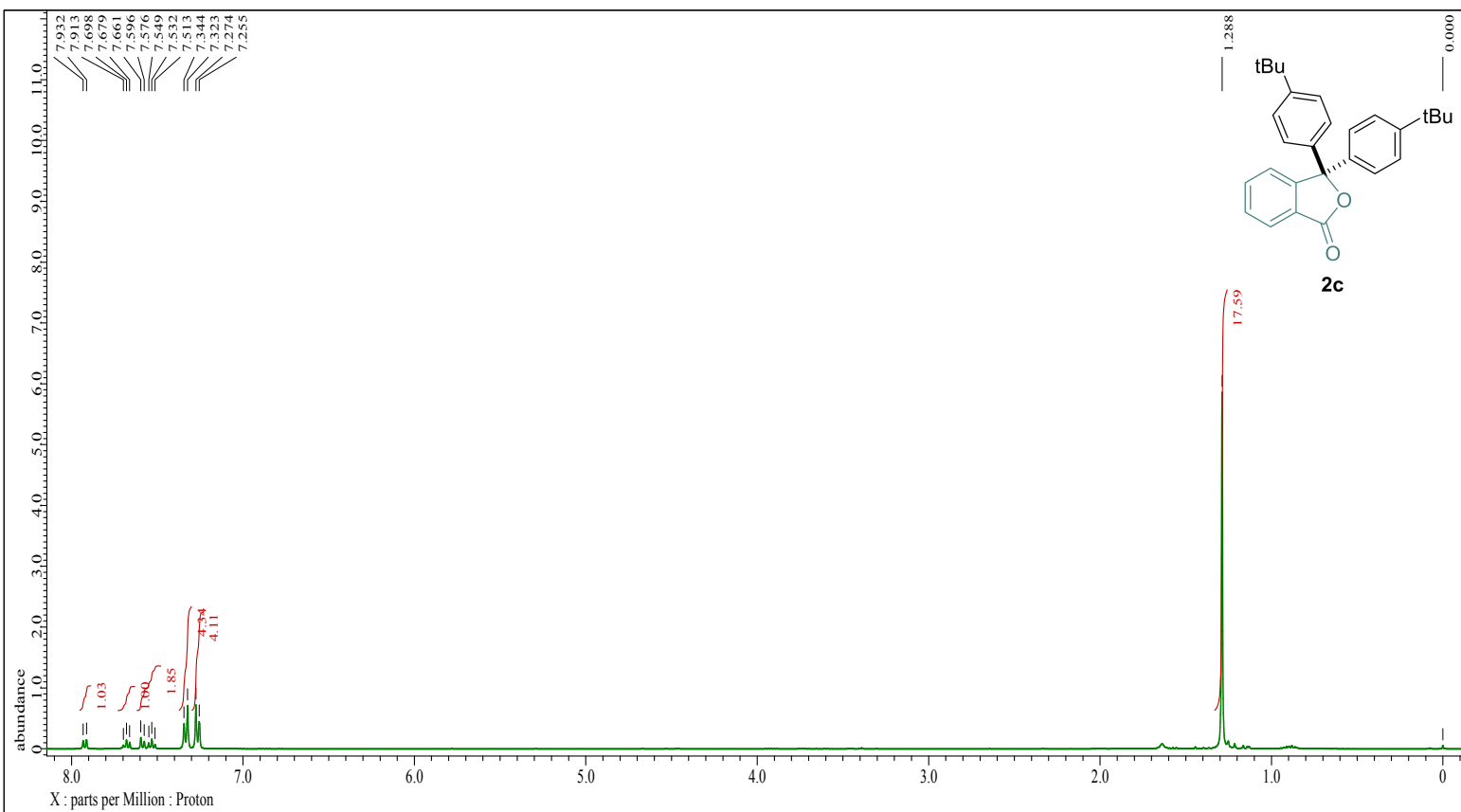


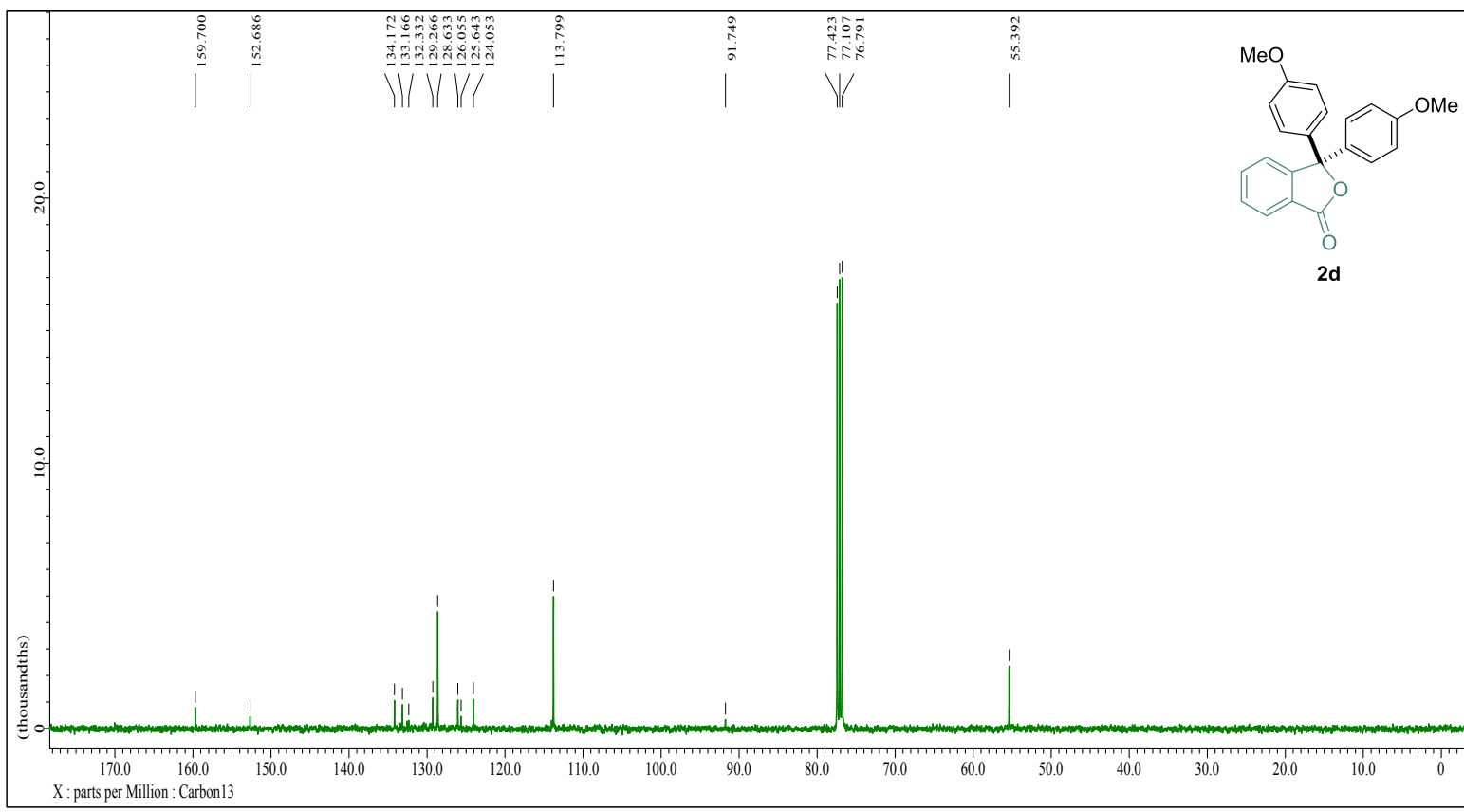
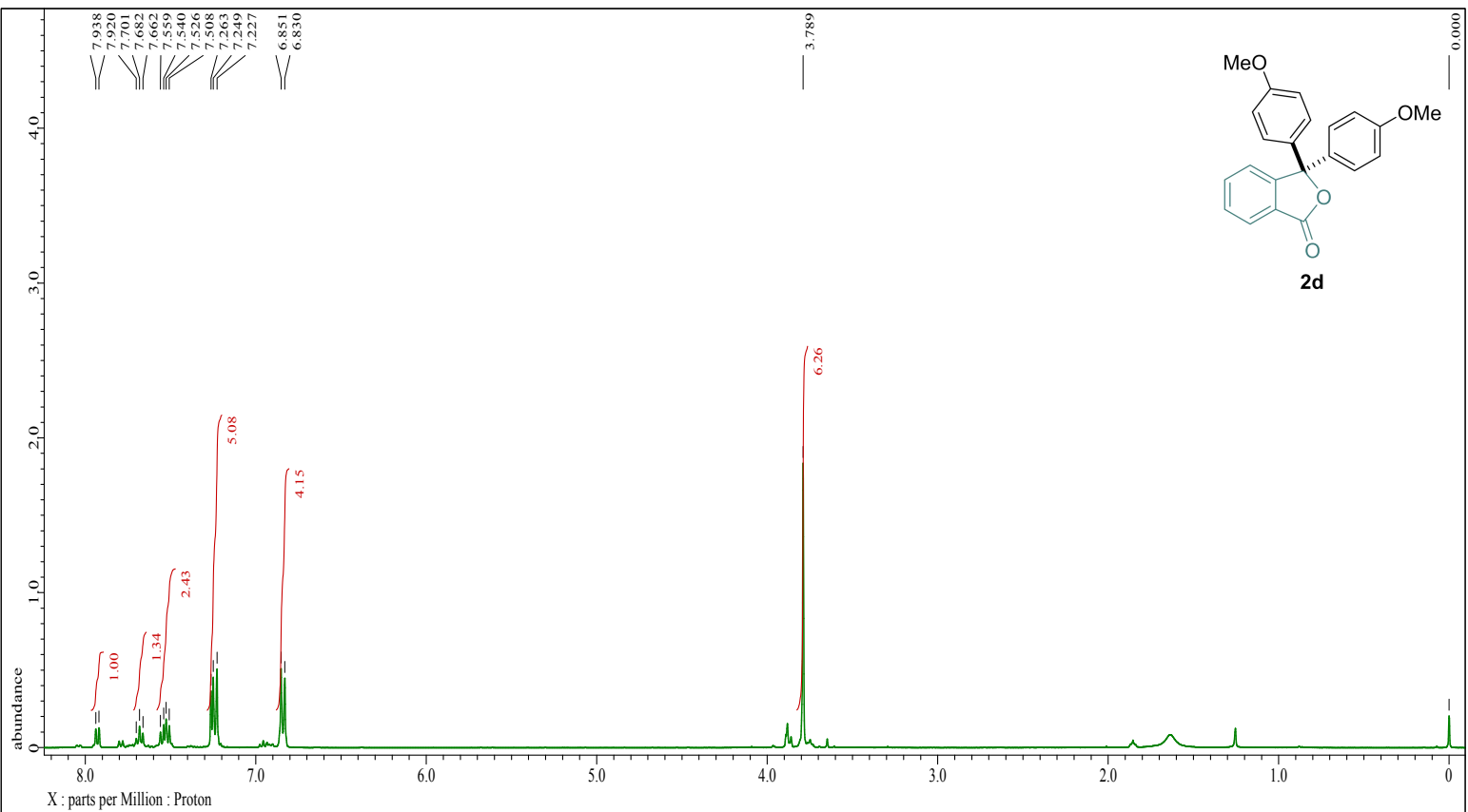
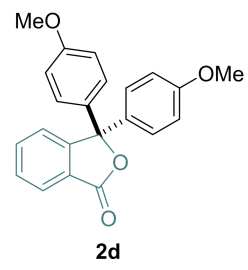
Figure 4. Cyclic voltammetry of **1a** (MeCN, scan rate: 100 mV s⁻¹, ferrocene was used as internal standard).

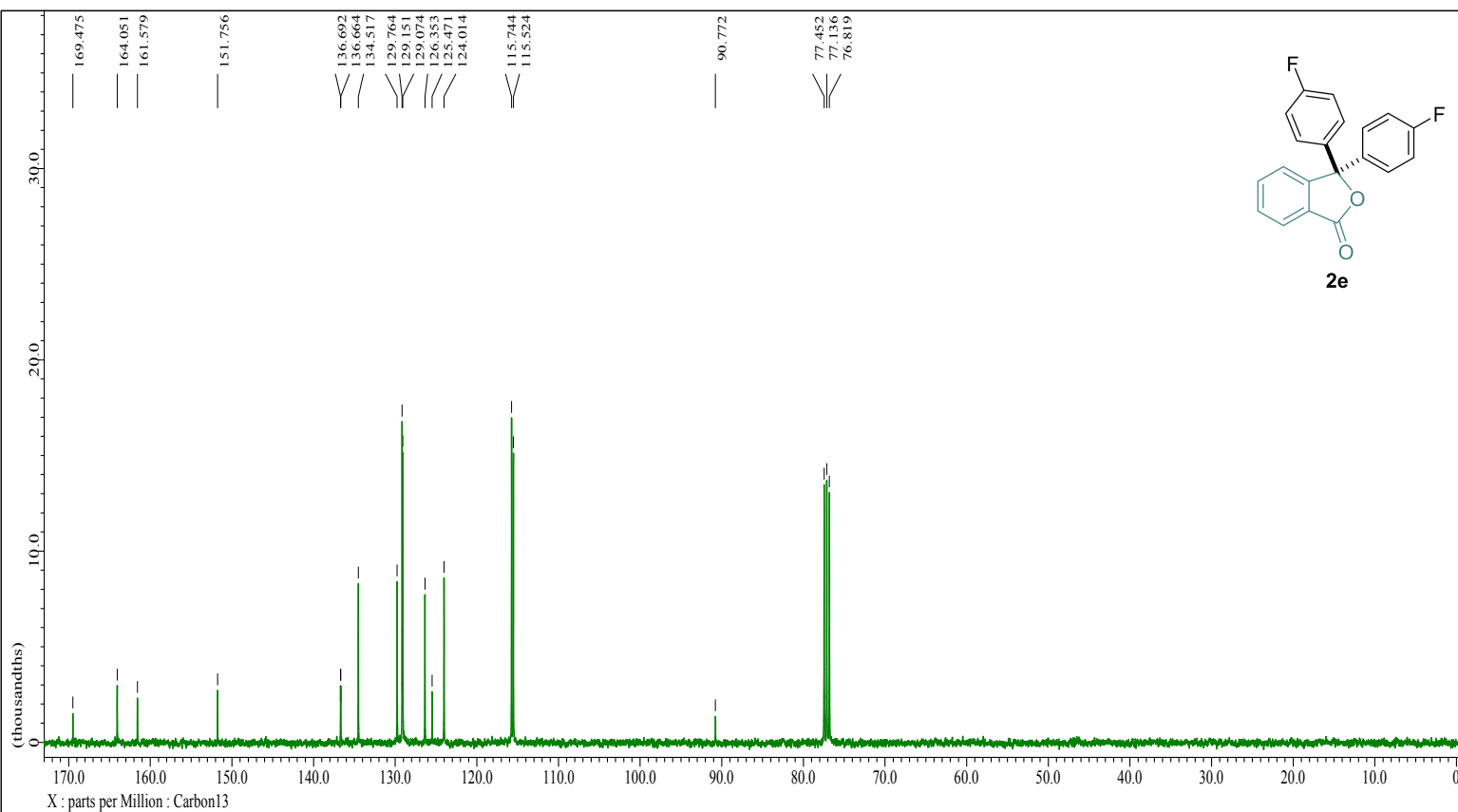
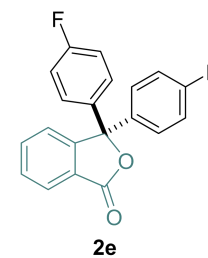
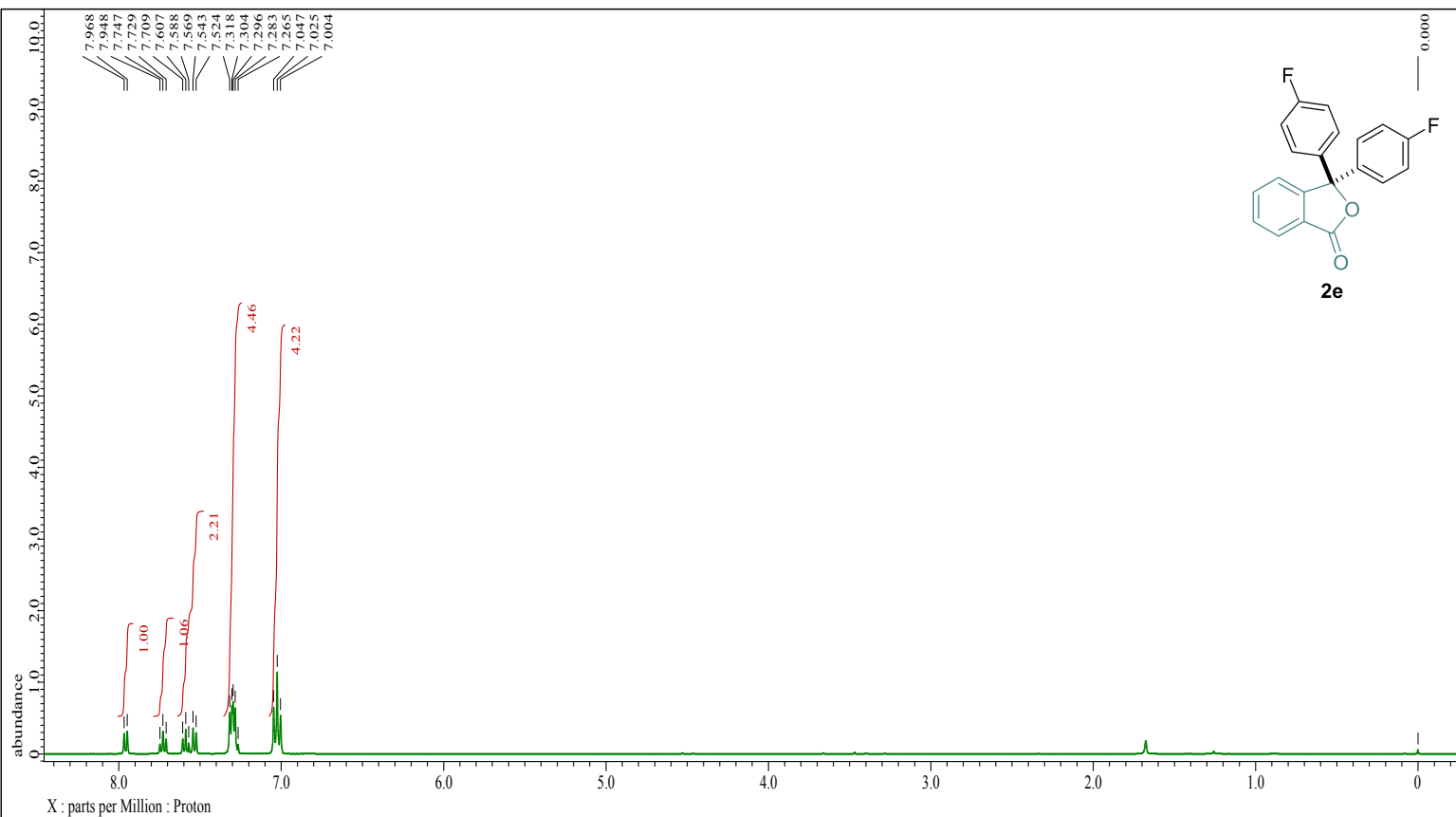
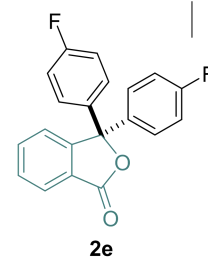
Copies of ^1H NMR, ^{13}C NMR and ^{19}F NMR spectra

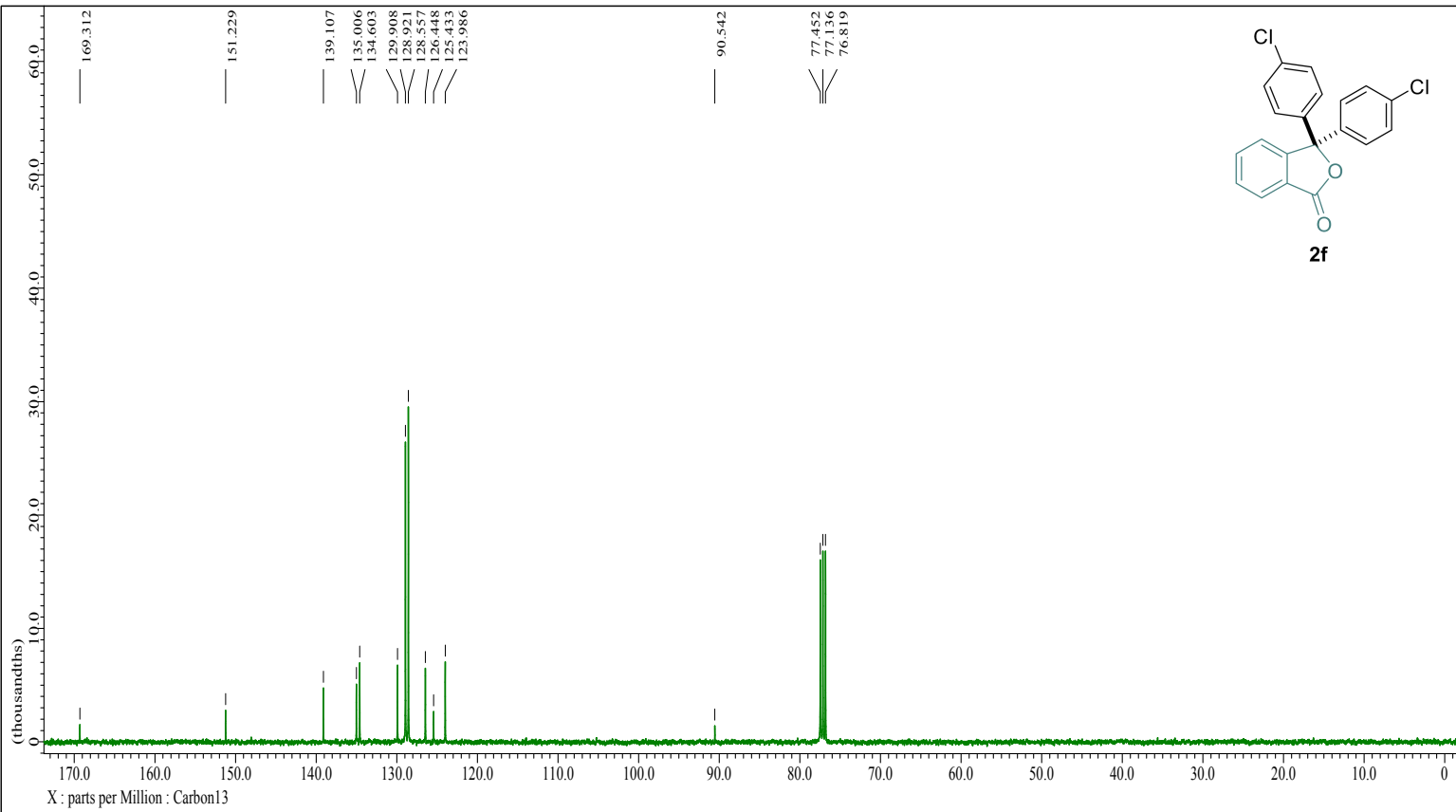
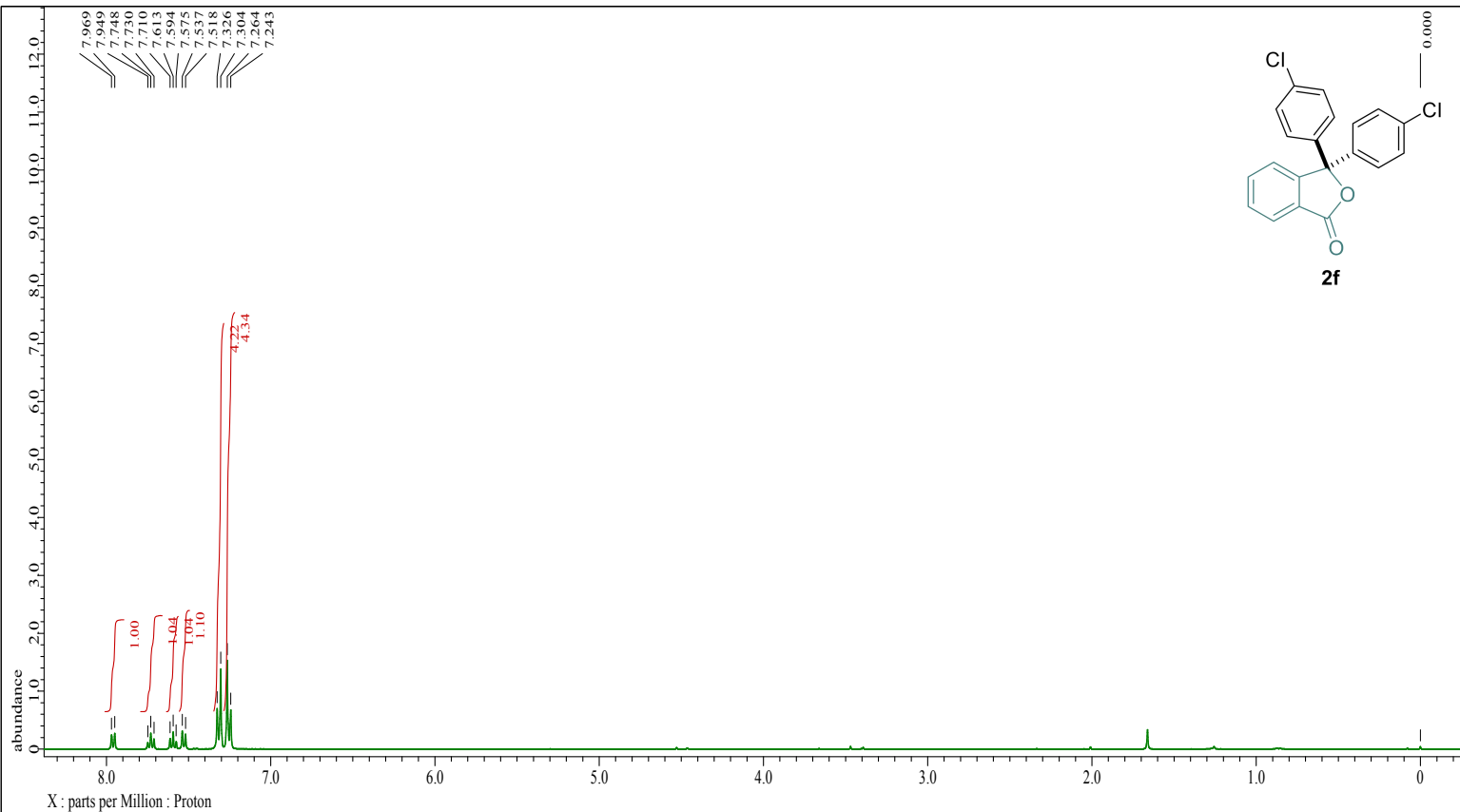
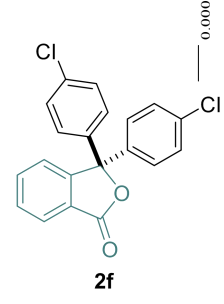


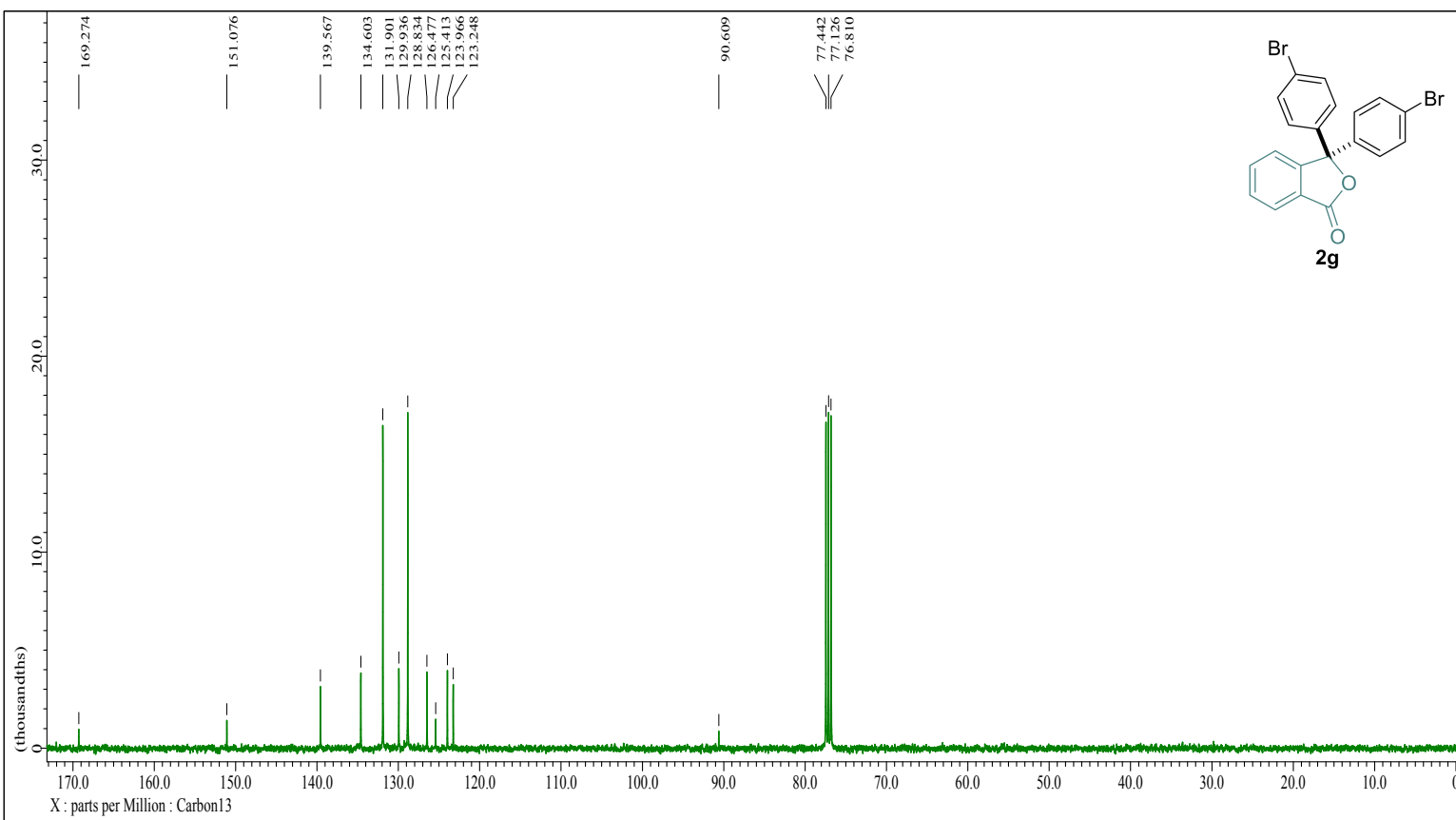
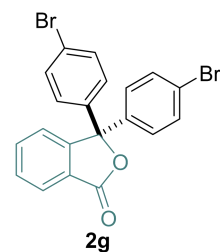
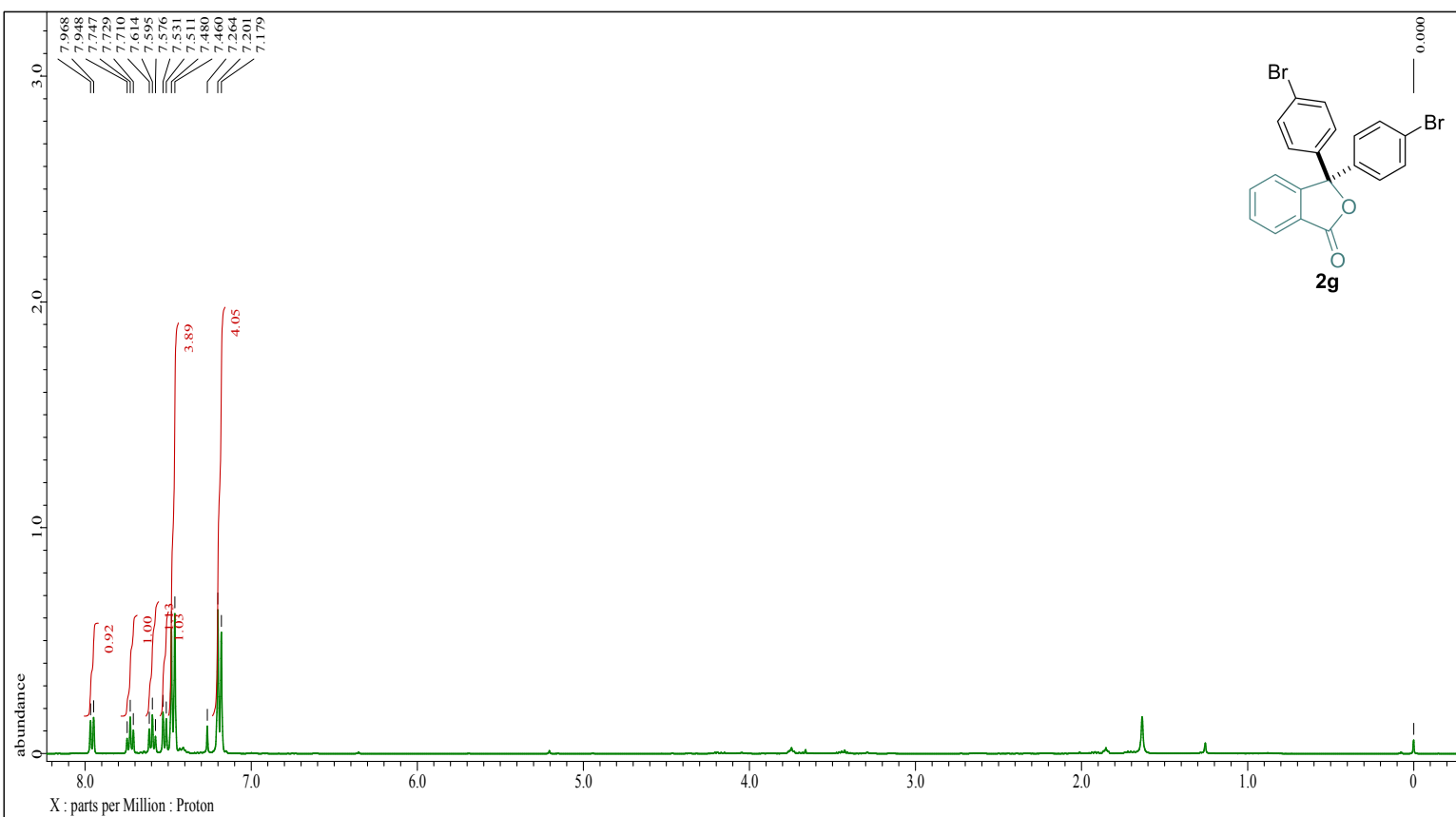
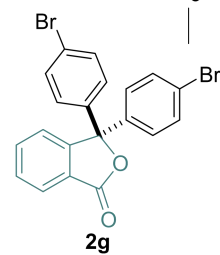


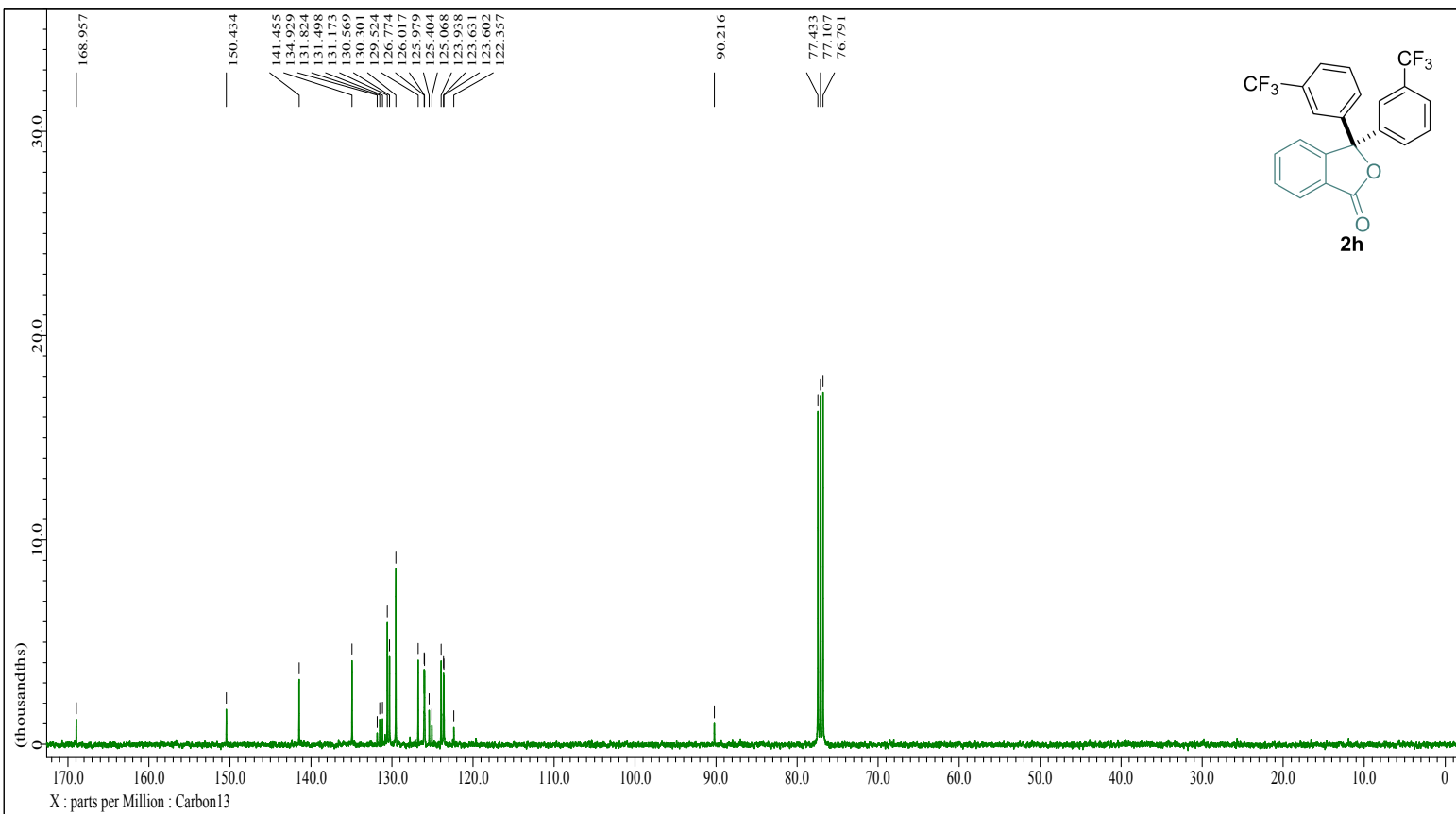
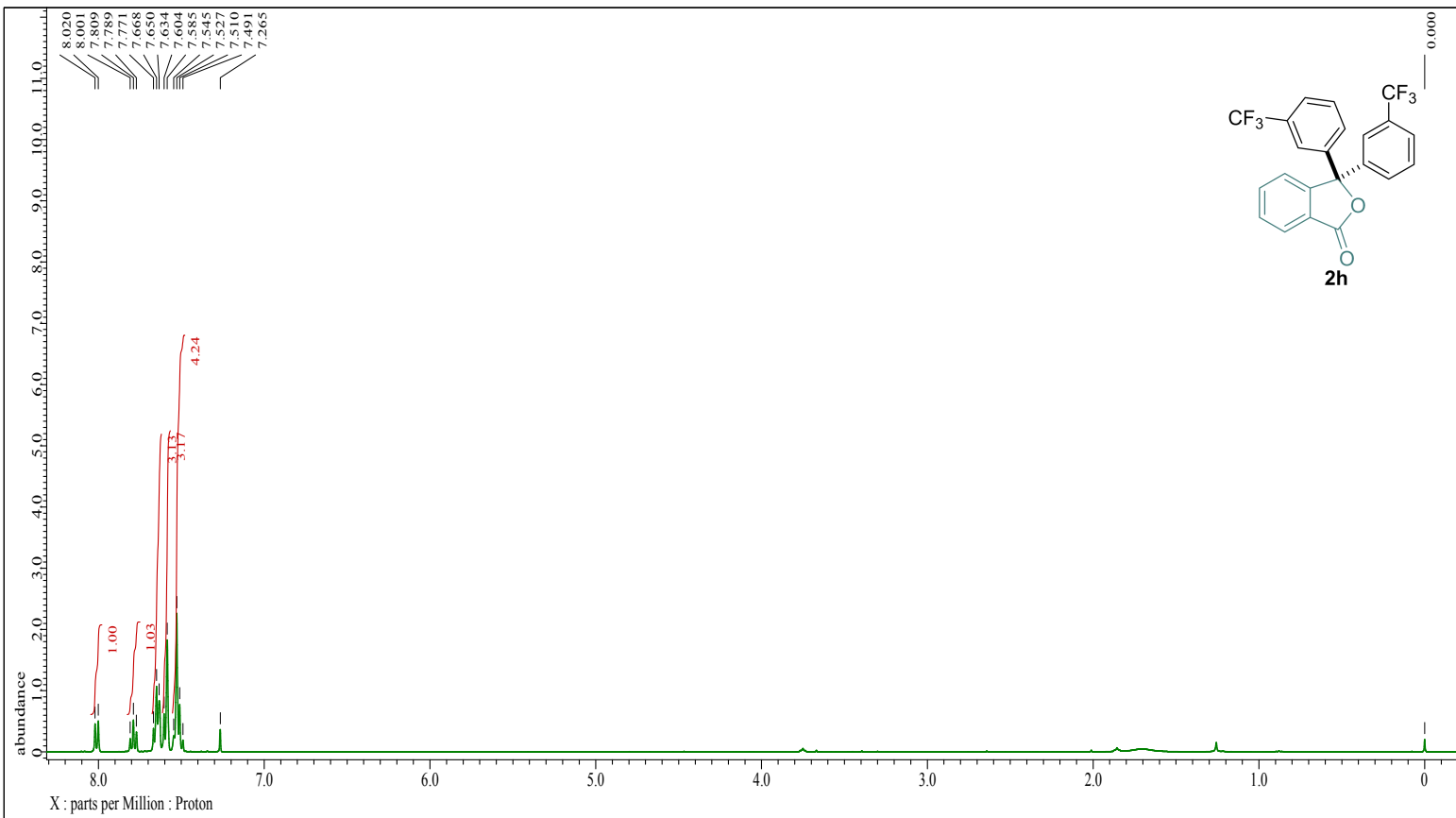


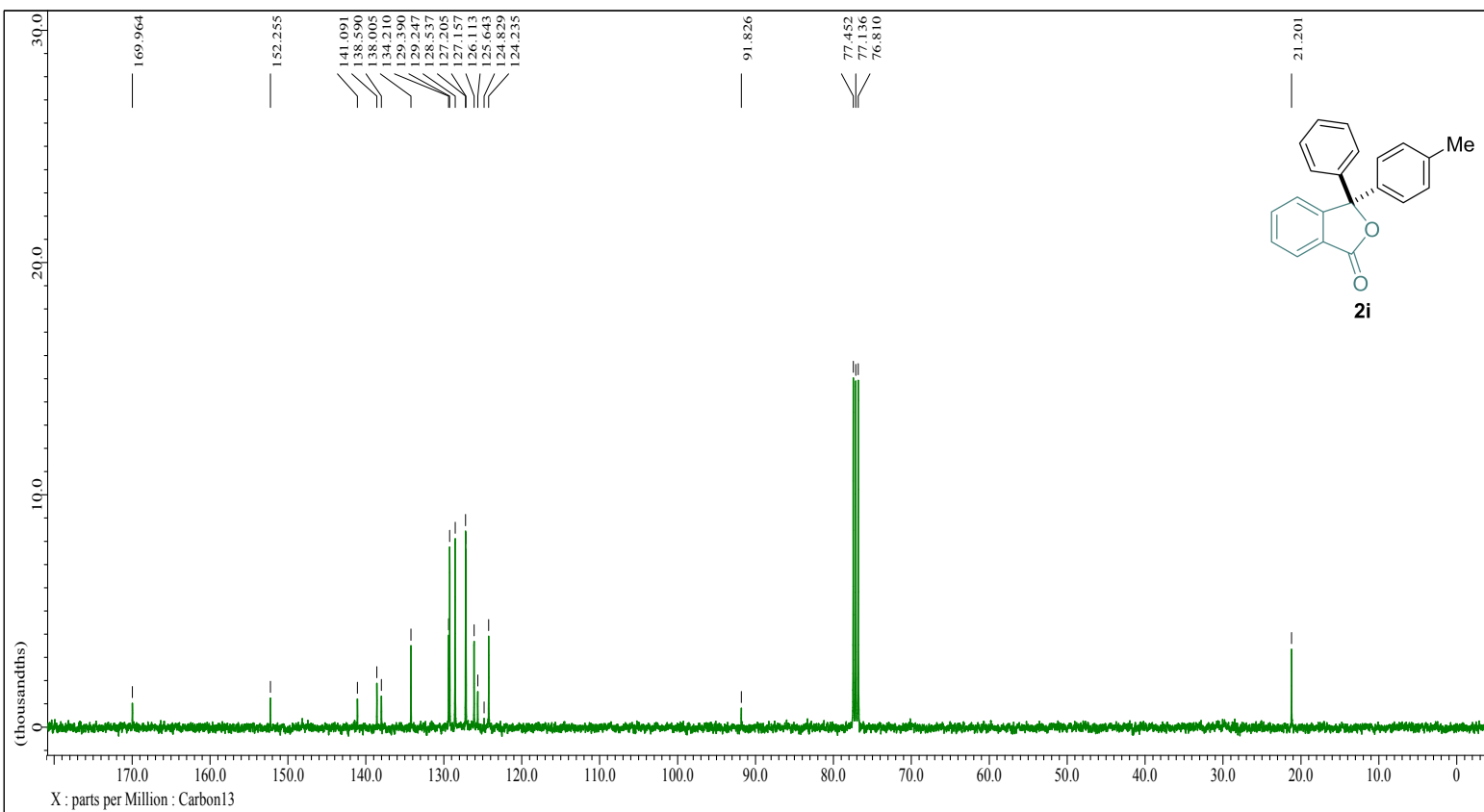
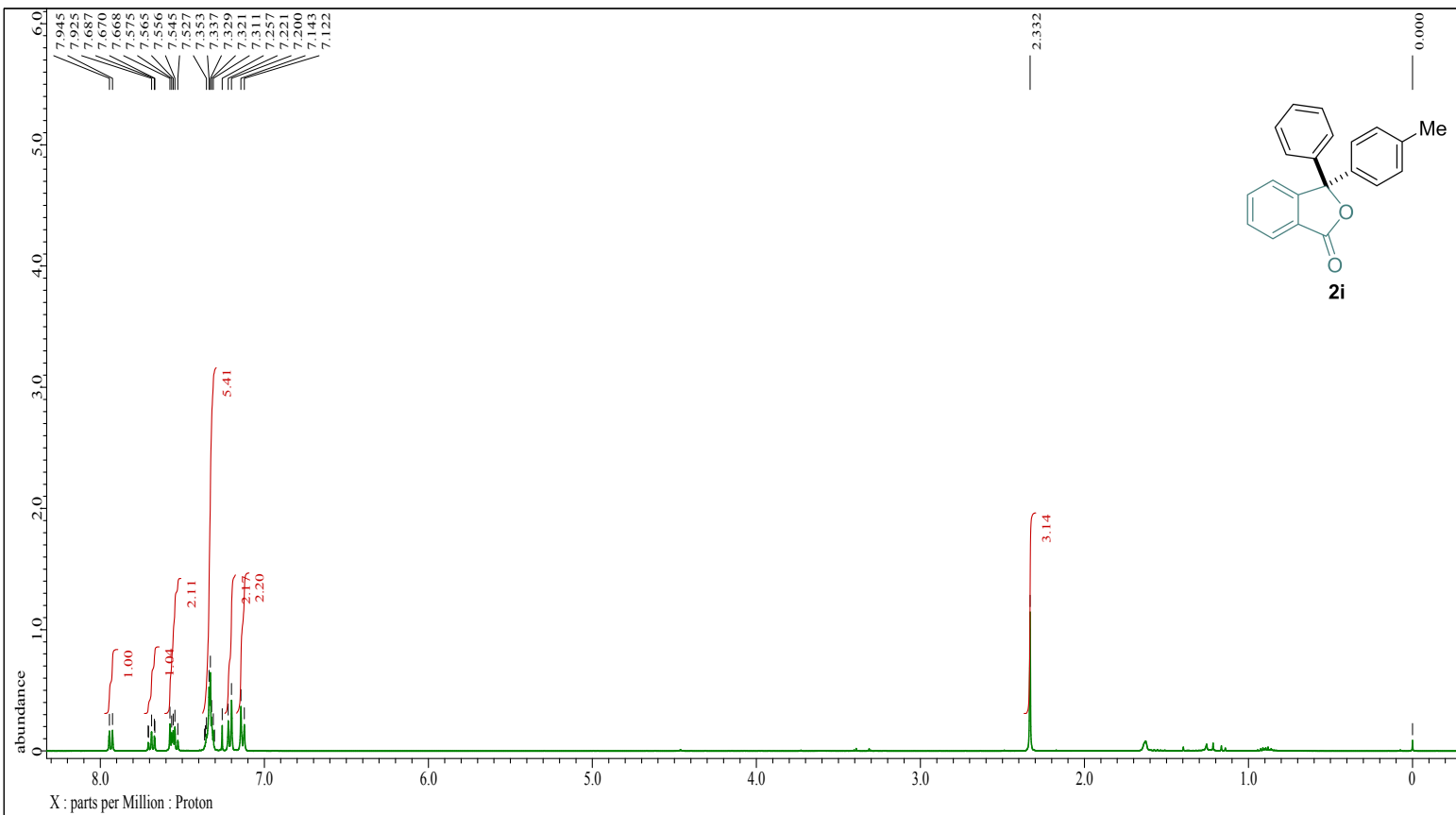


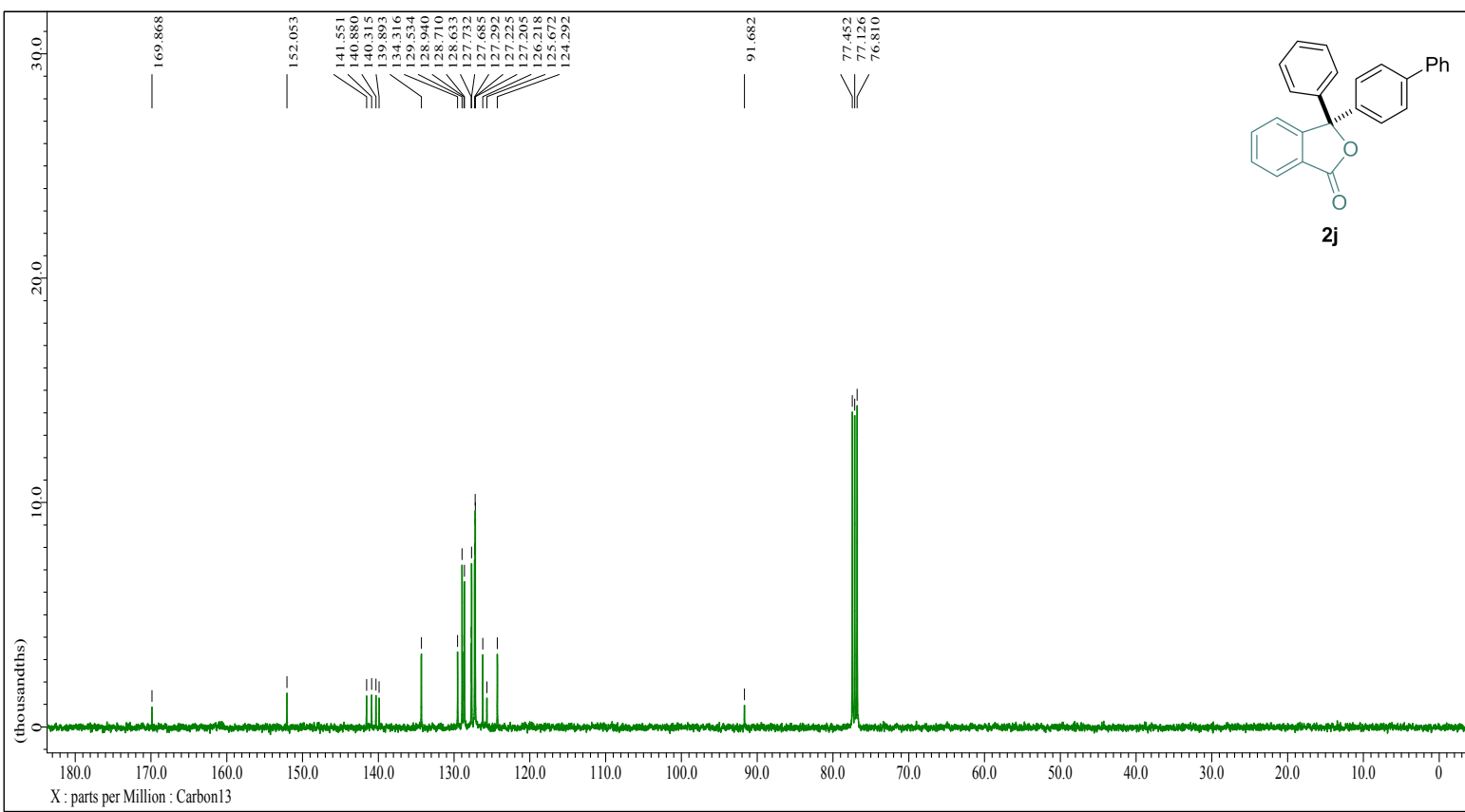
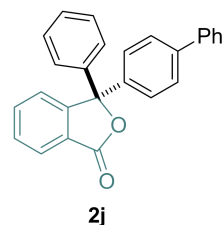
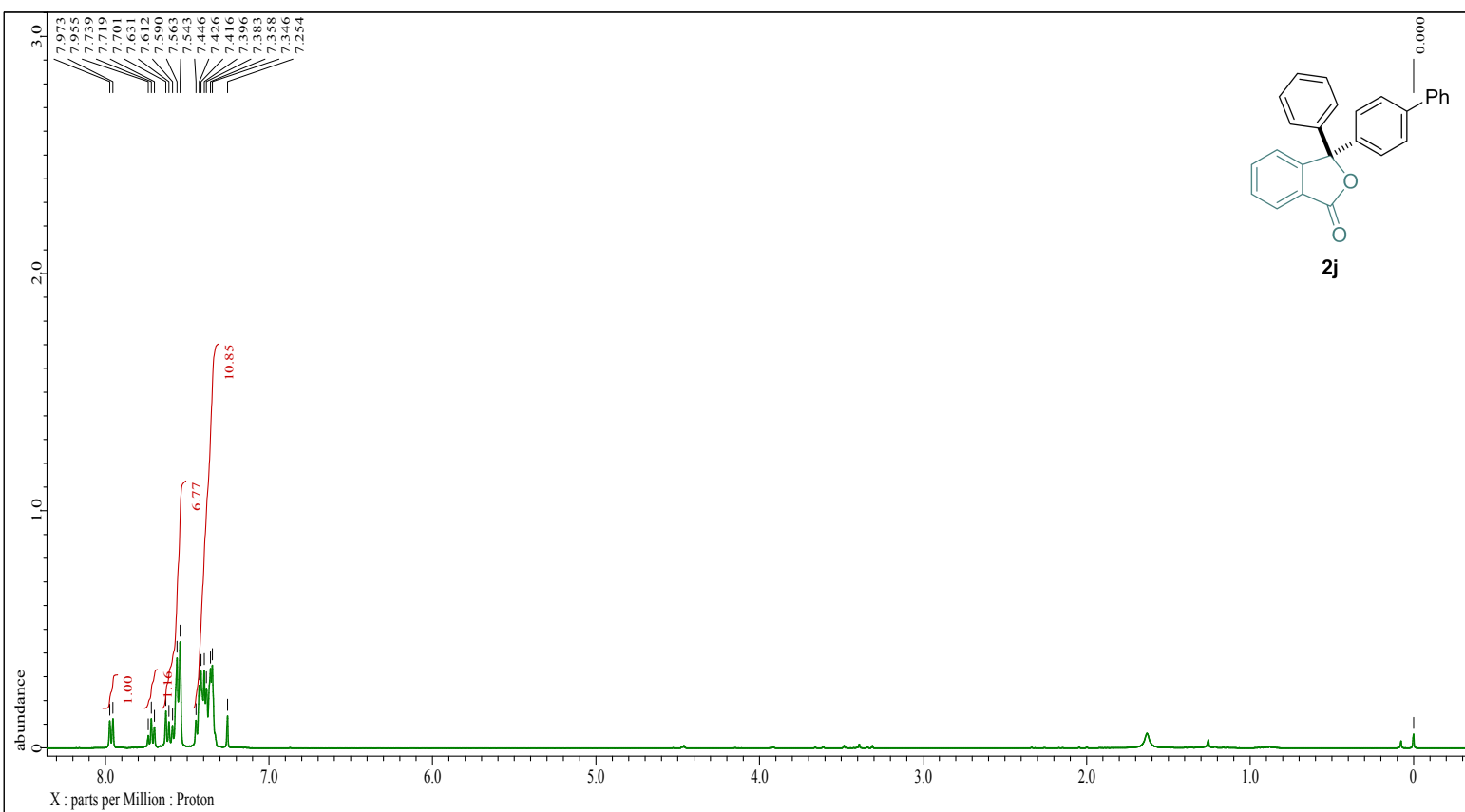
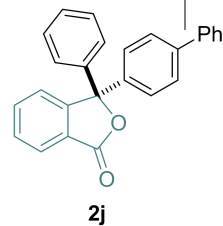


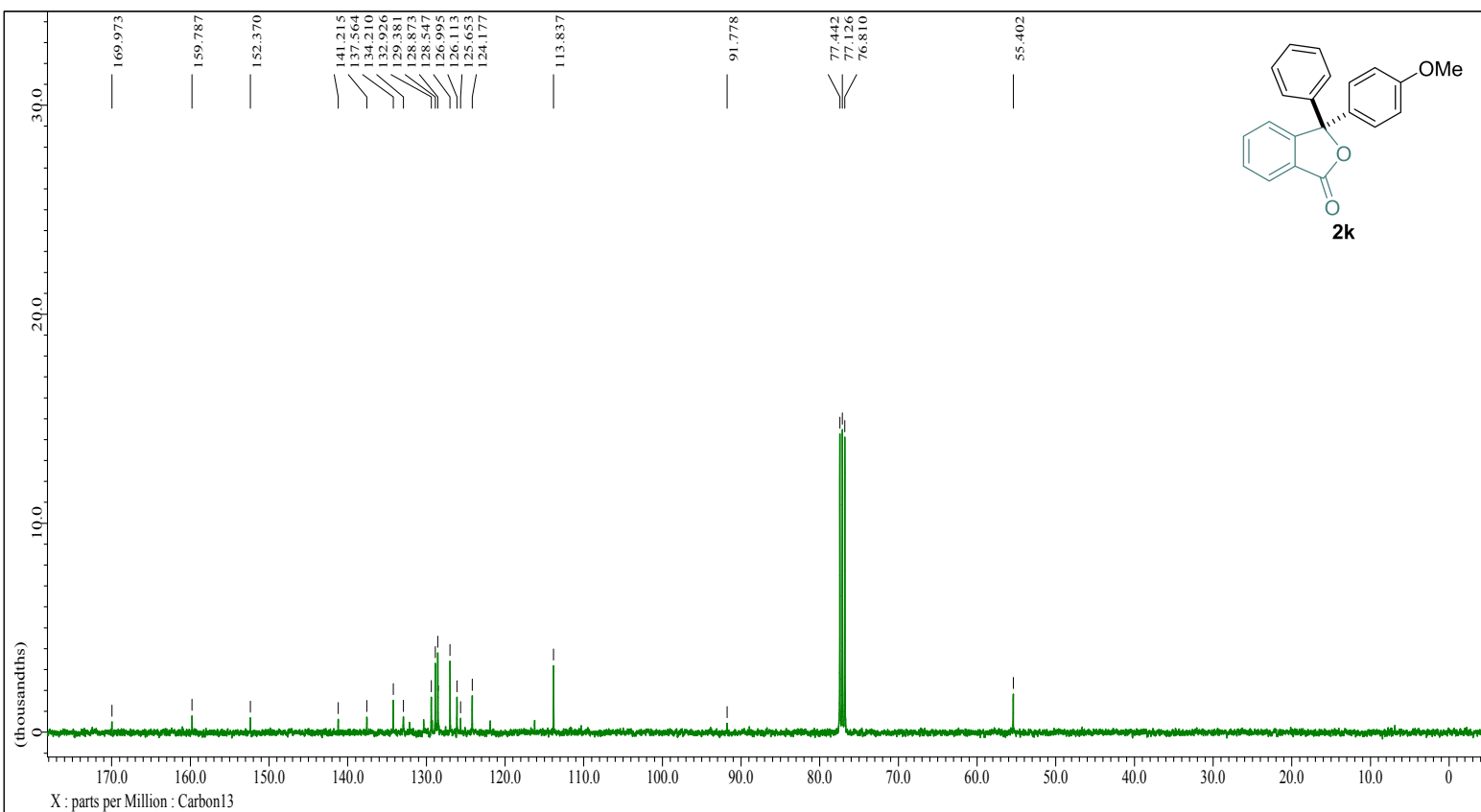
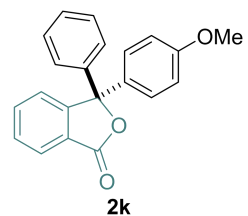
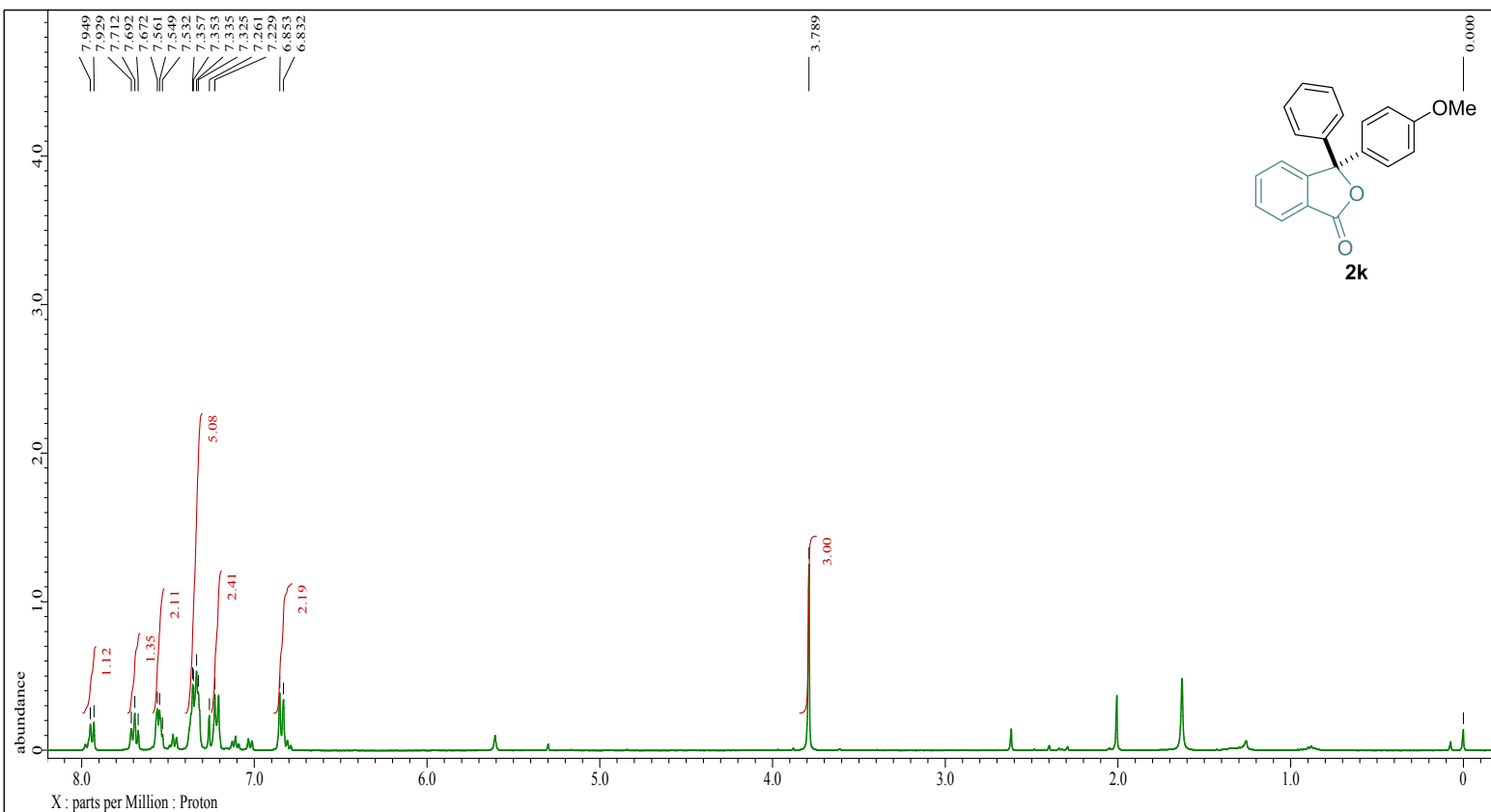
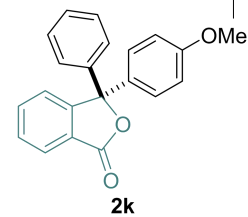


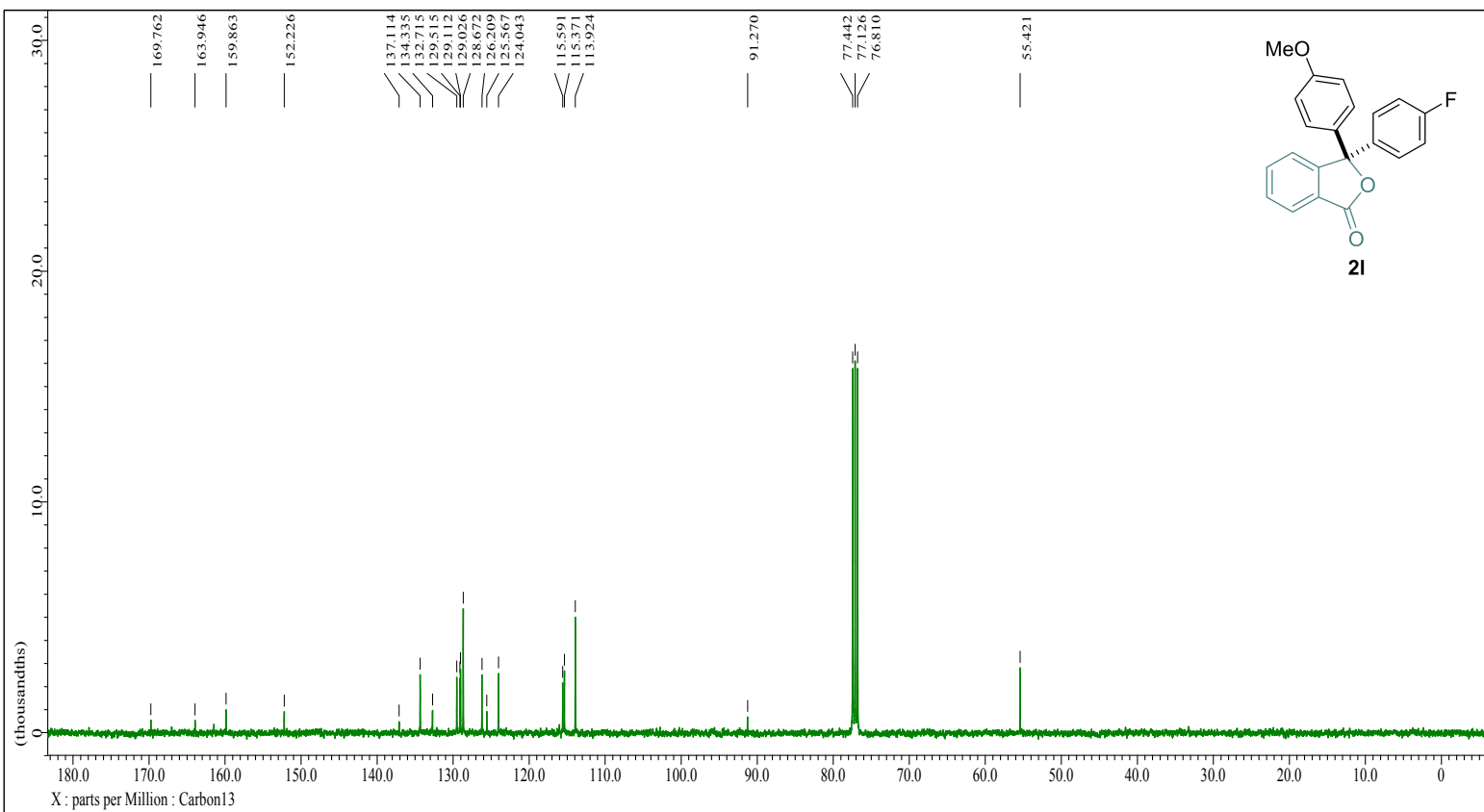
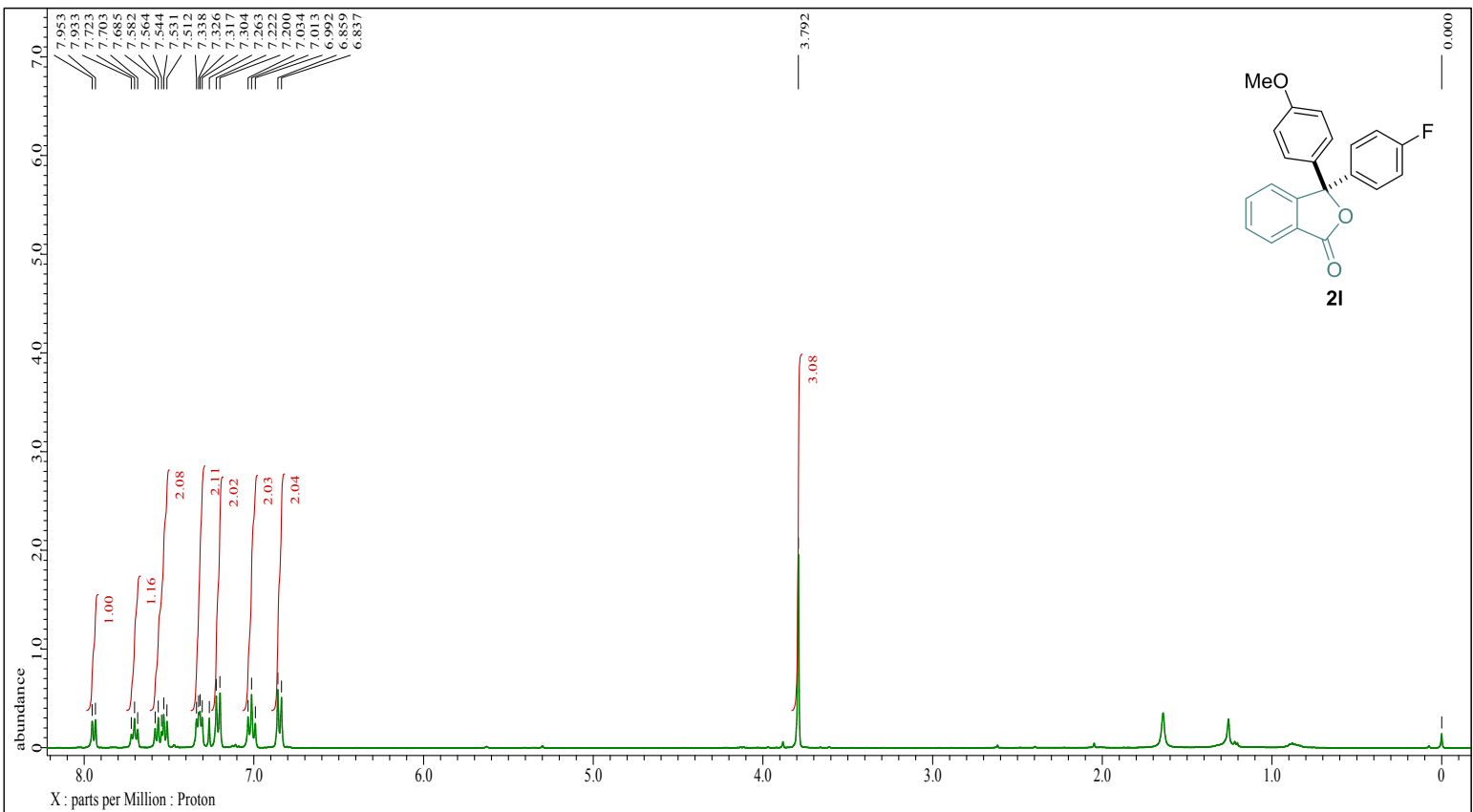


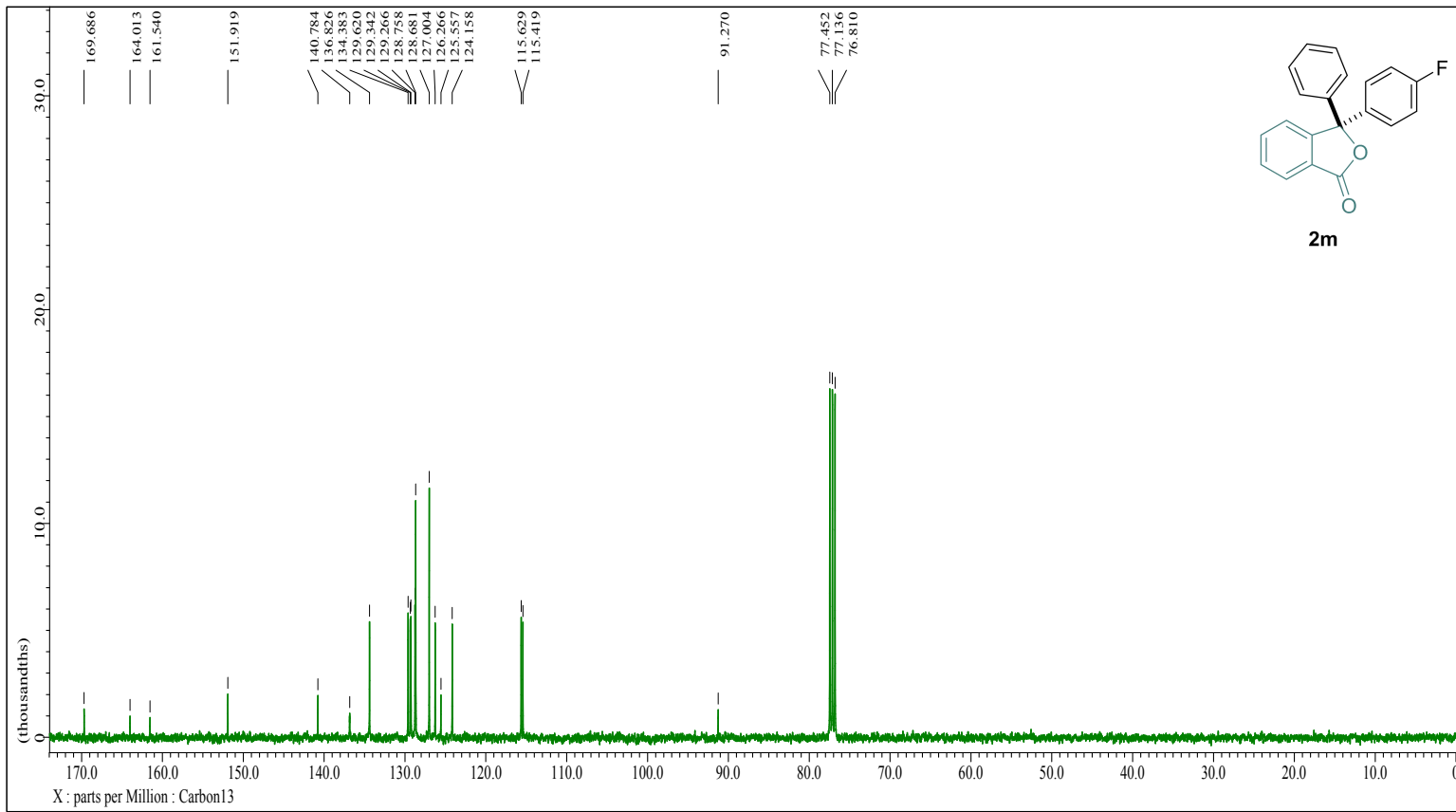
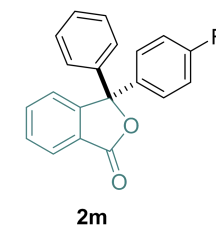
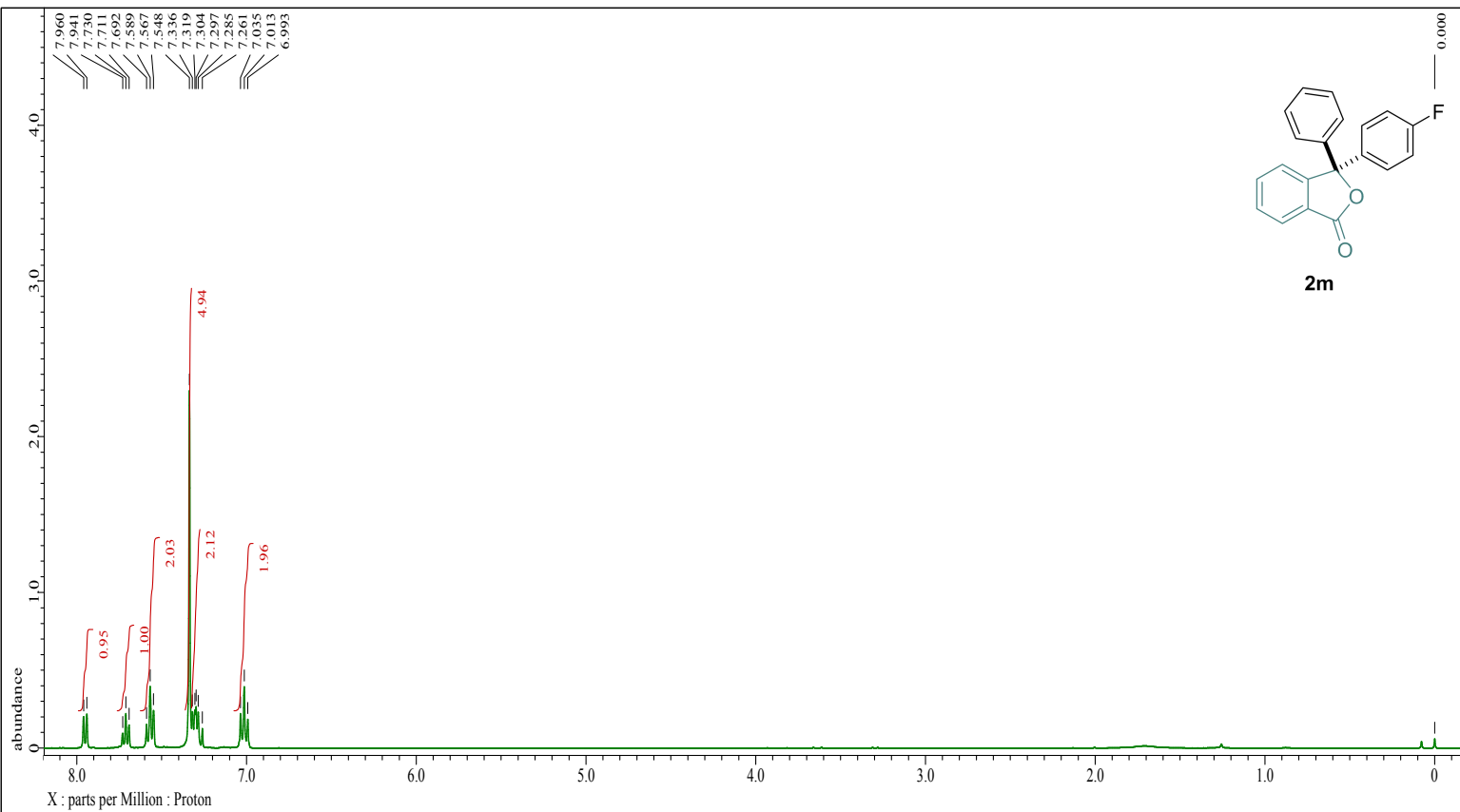
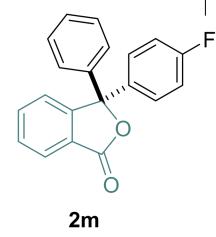


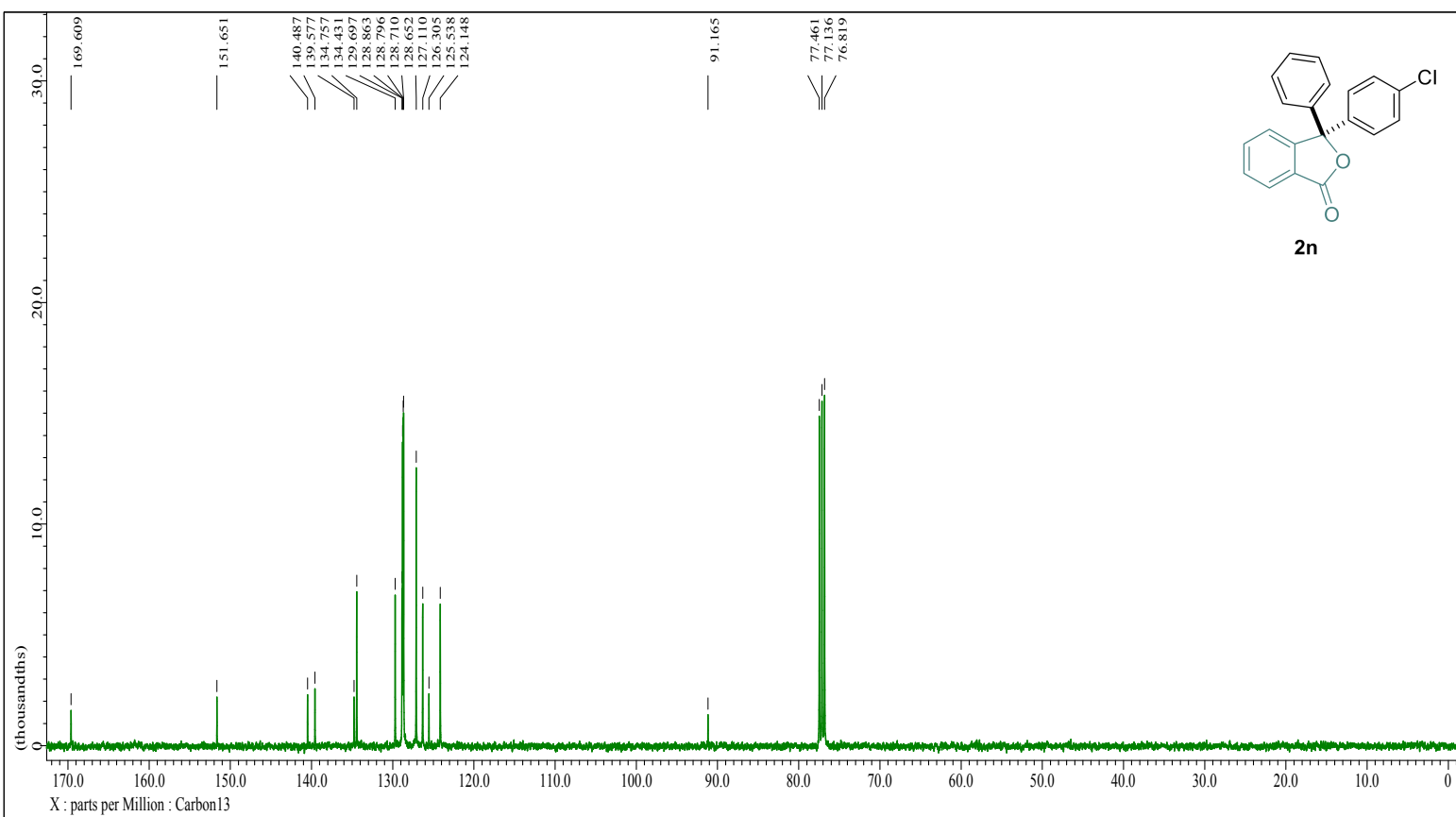
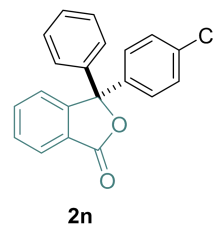
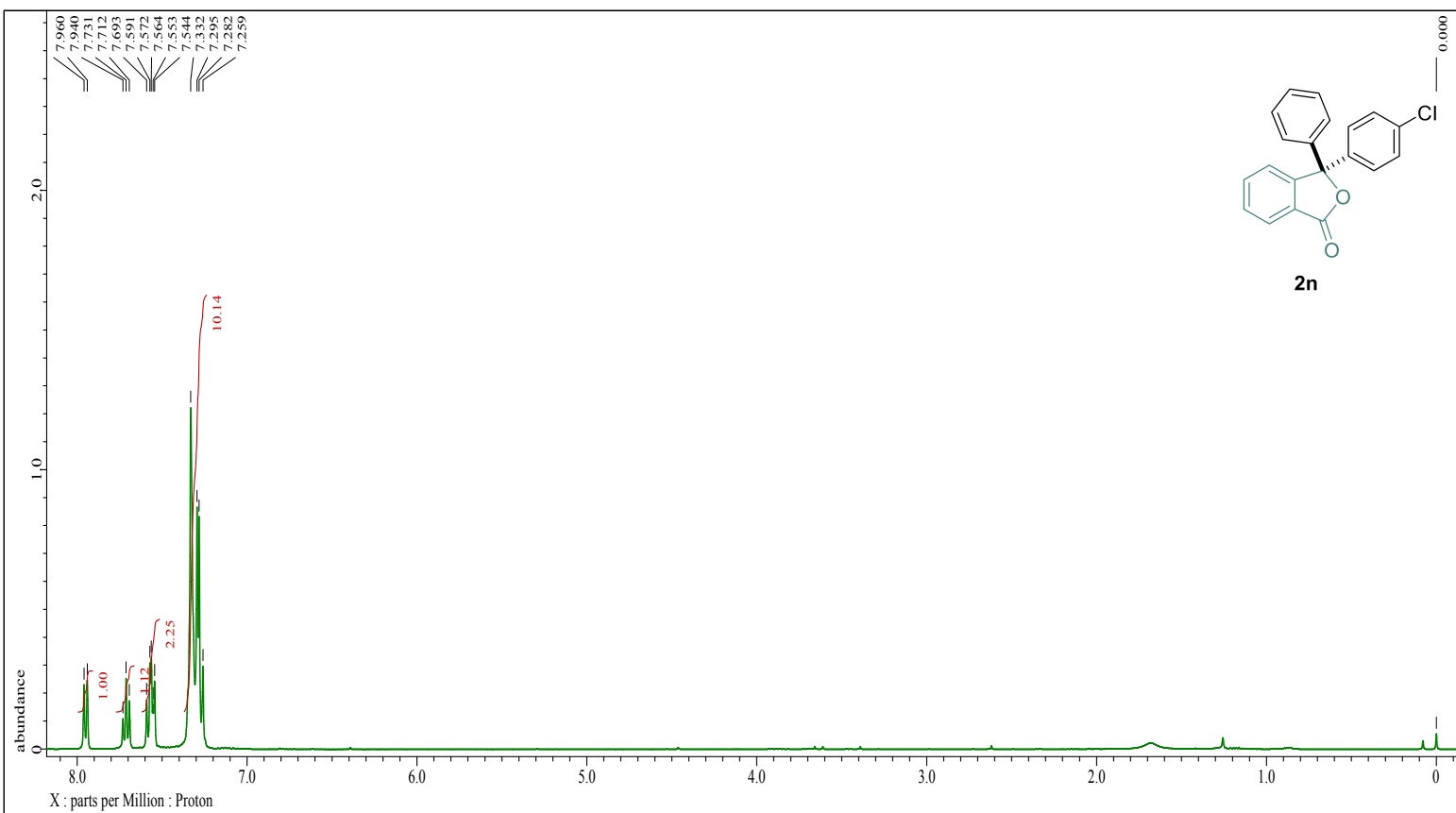
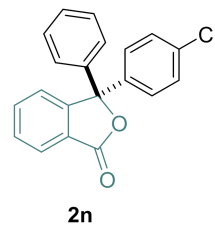


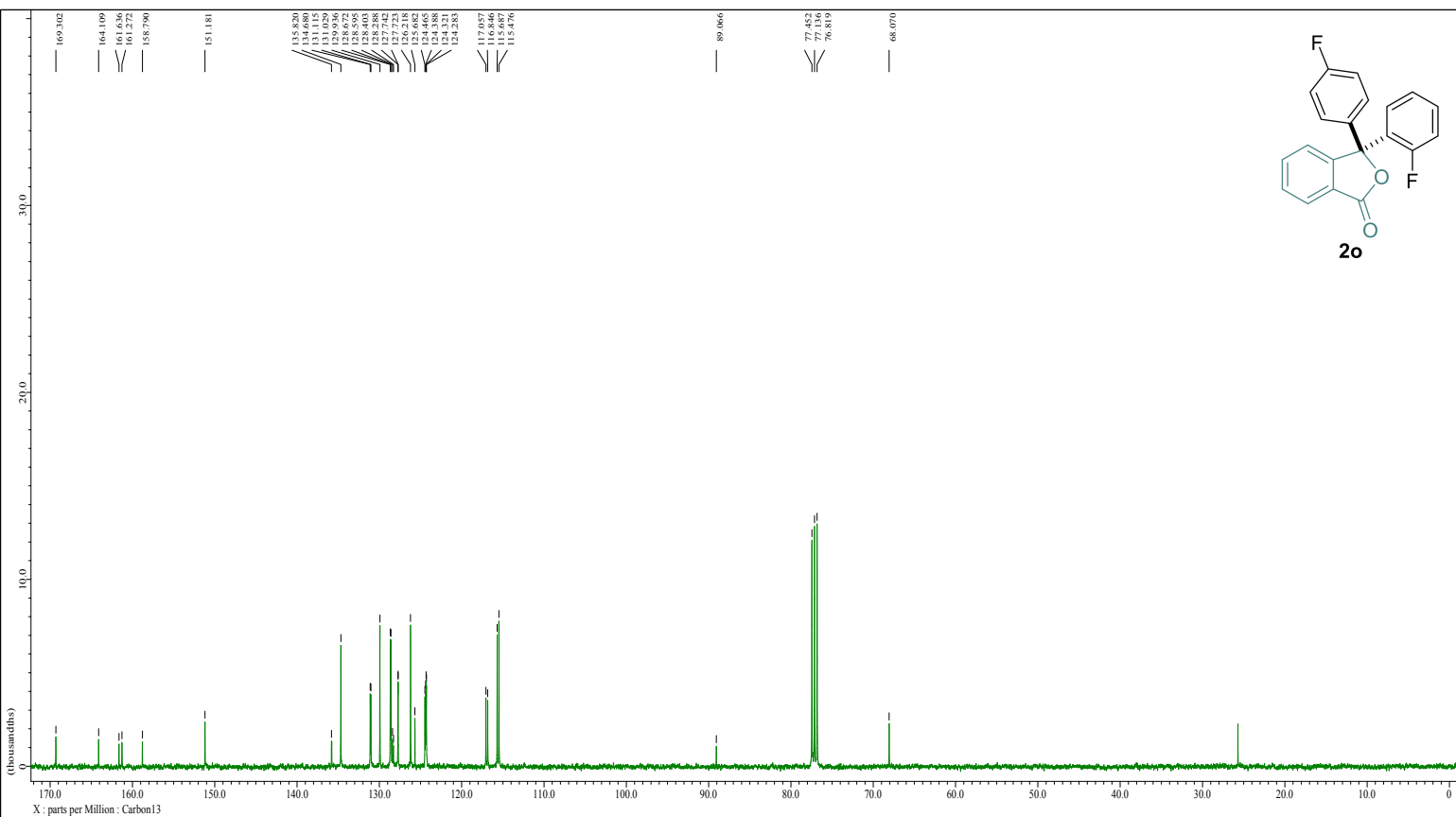
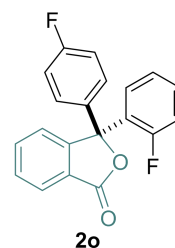
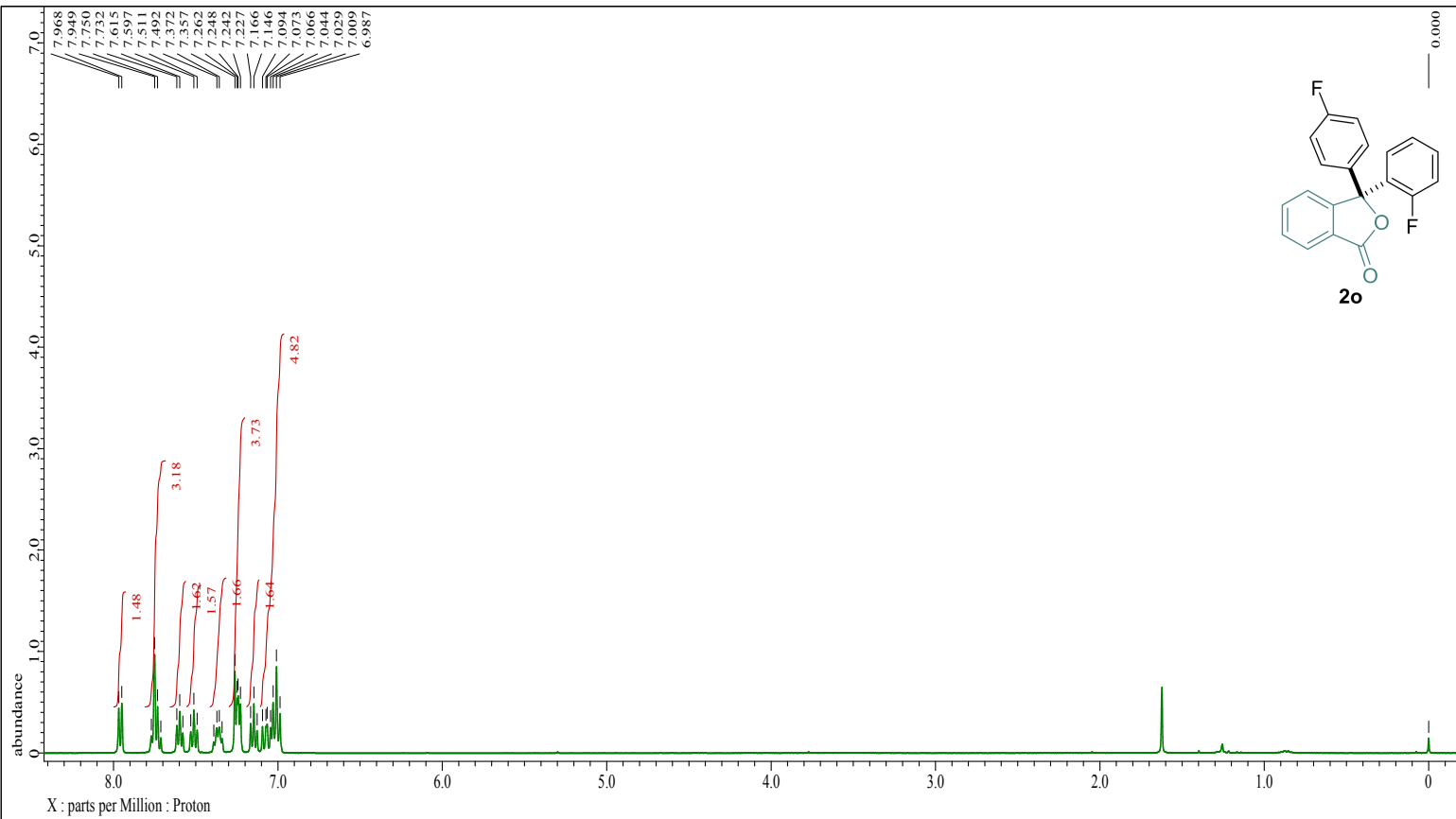
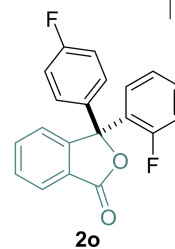


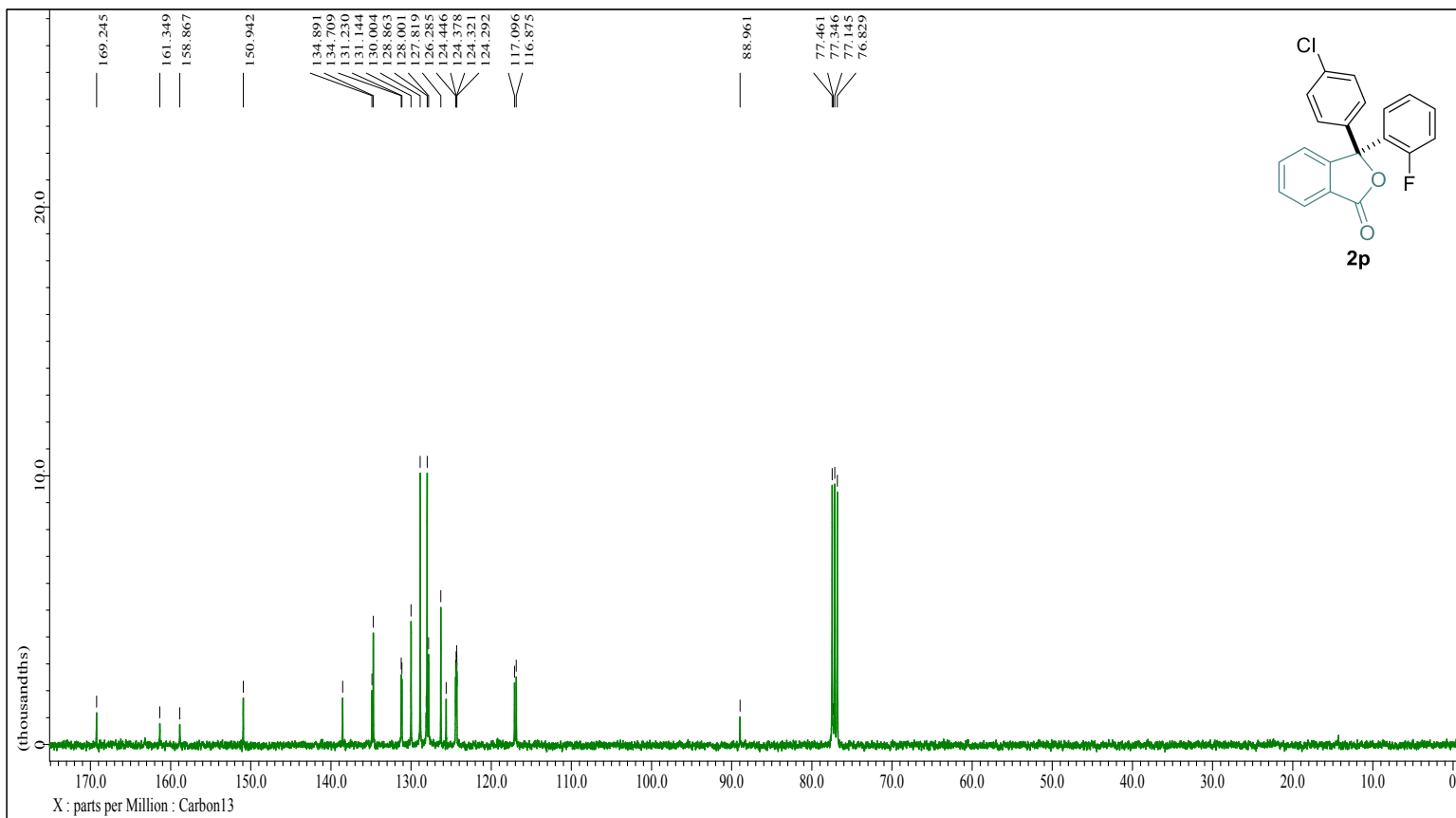
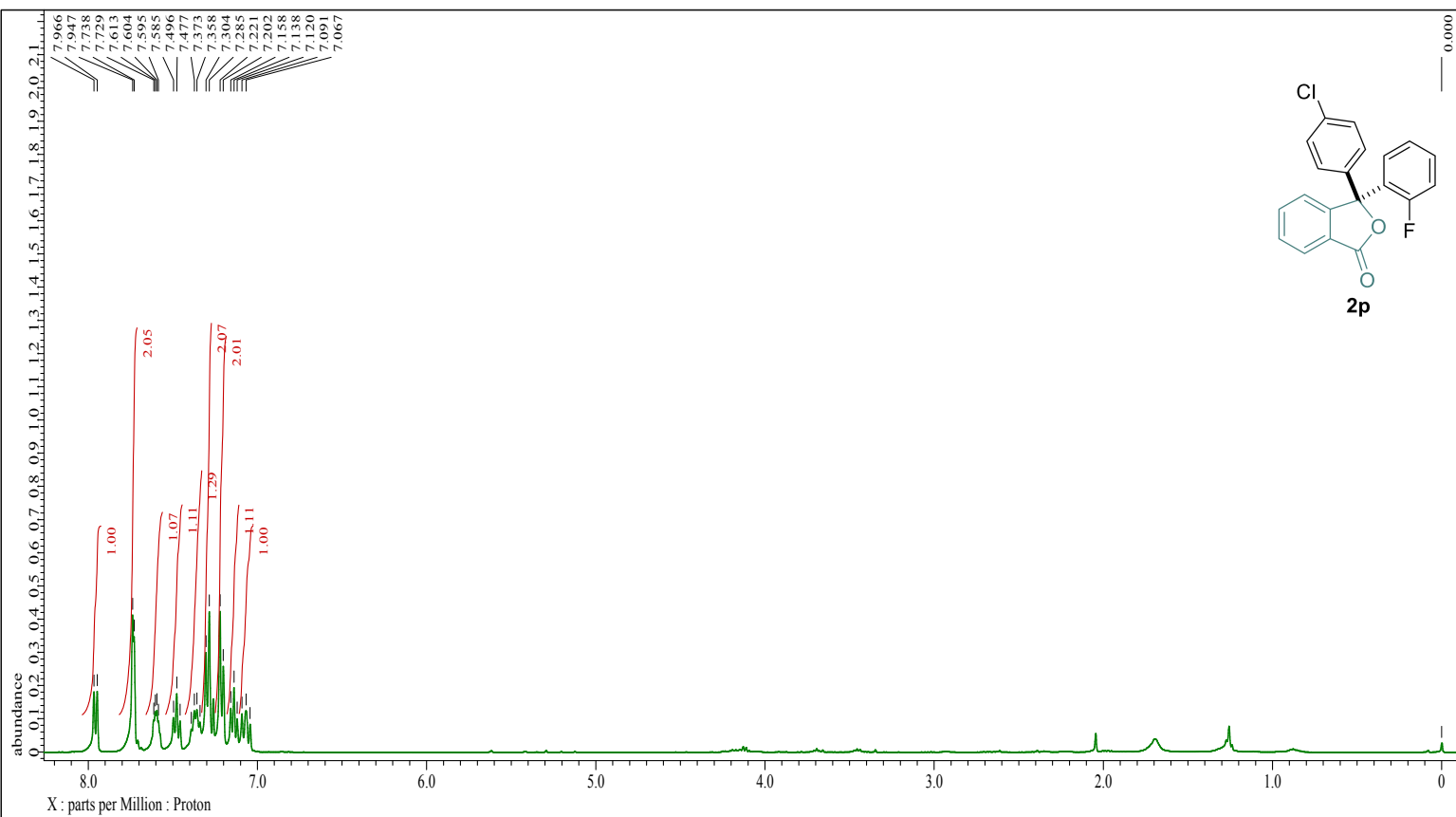
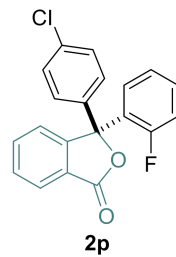


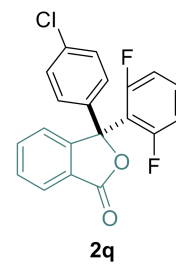




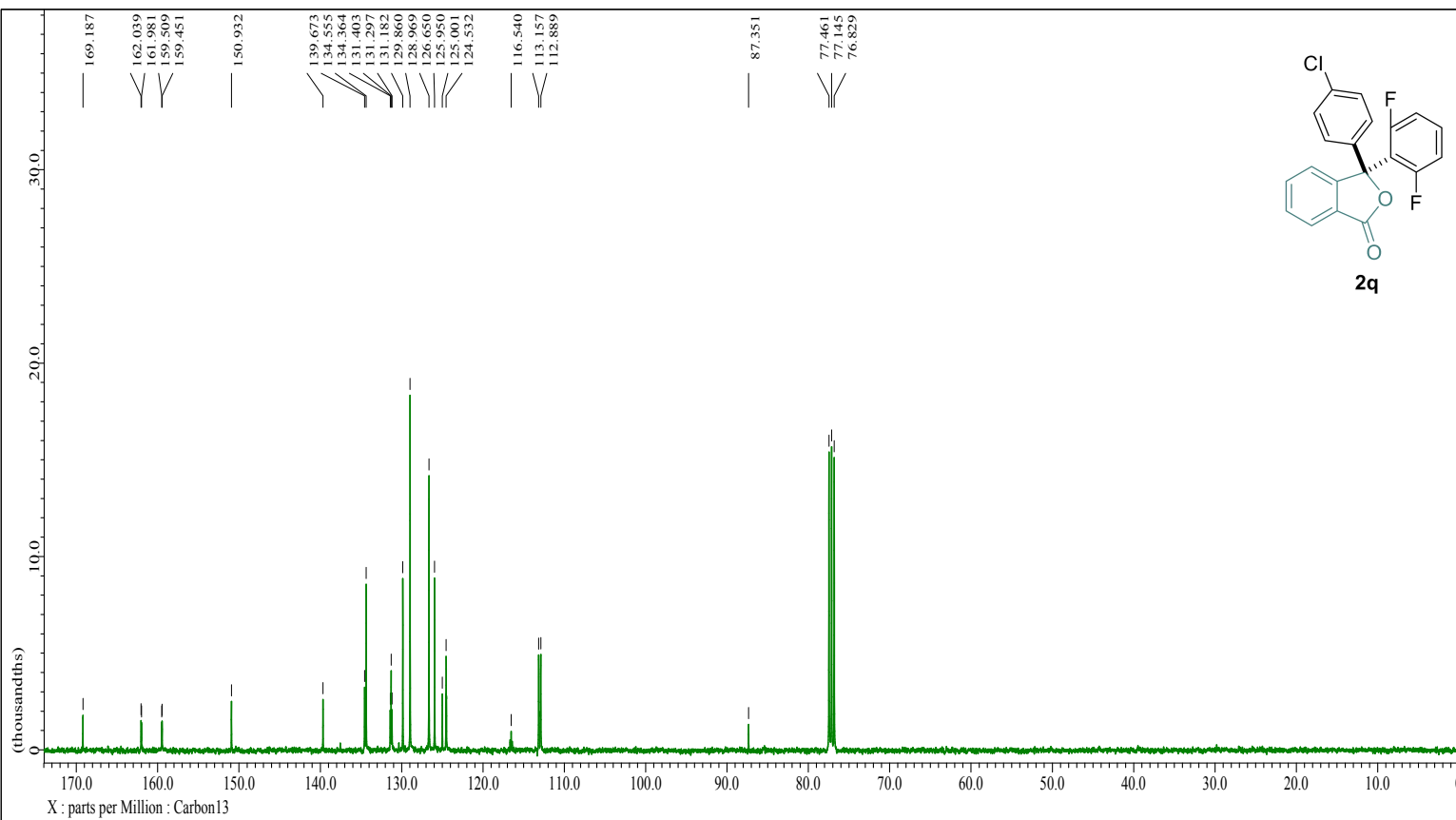
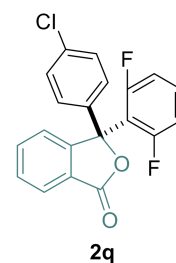
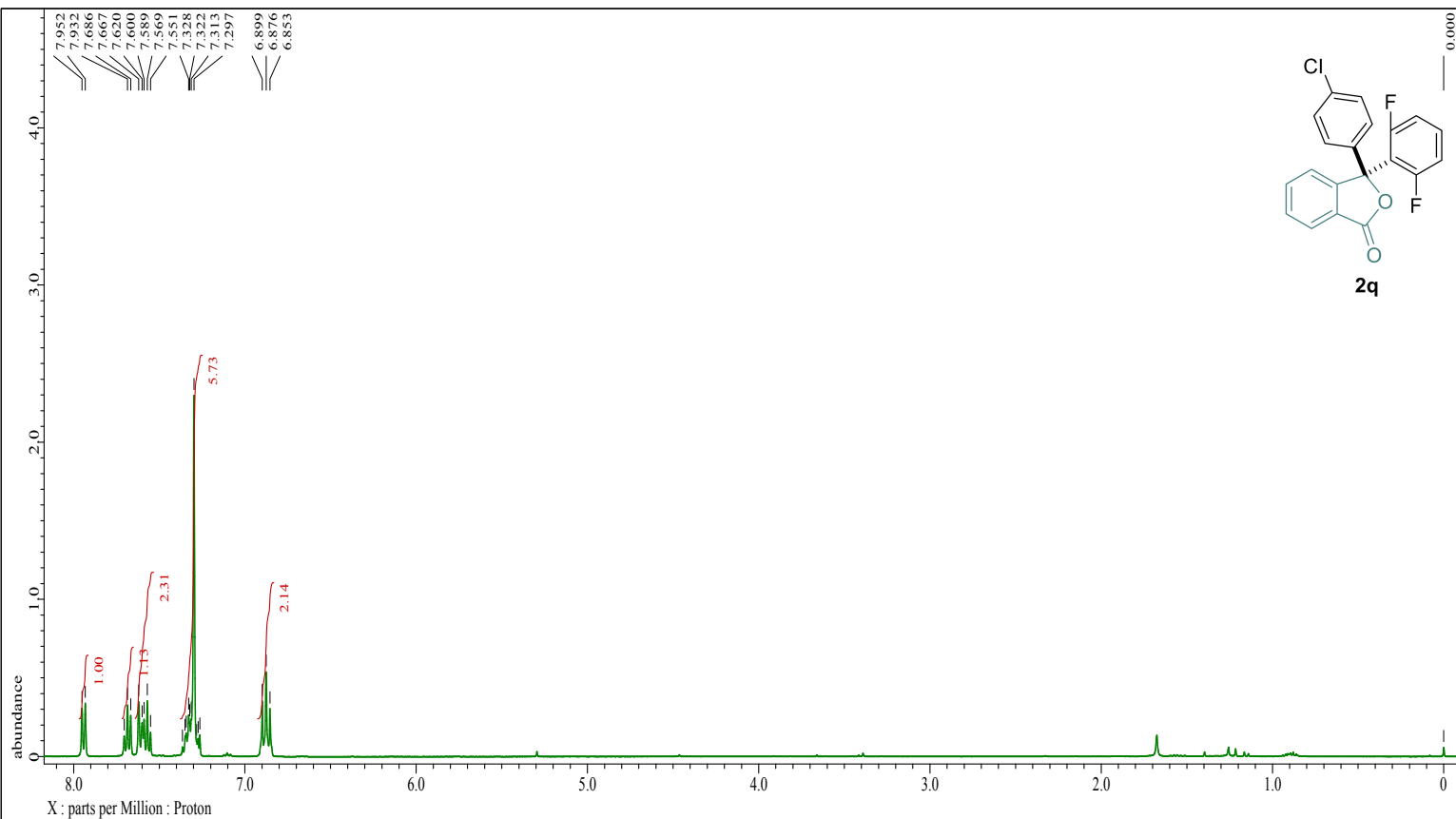


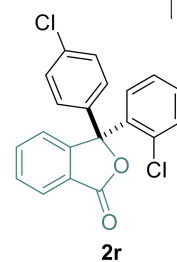




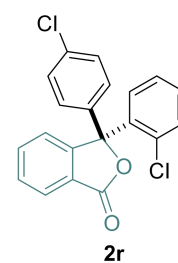
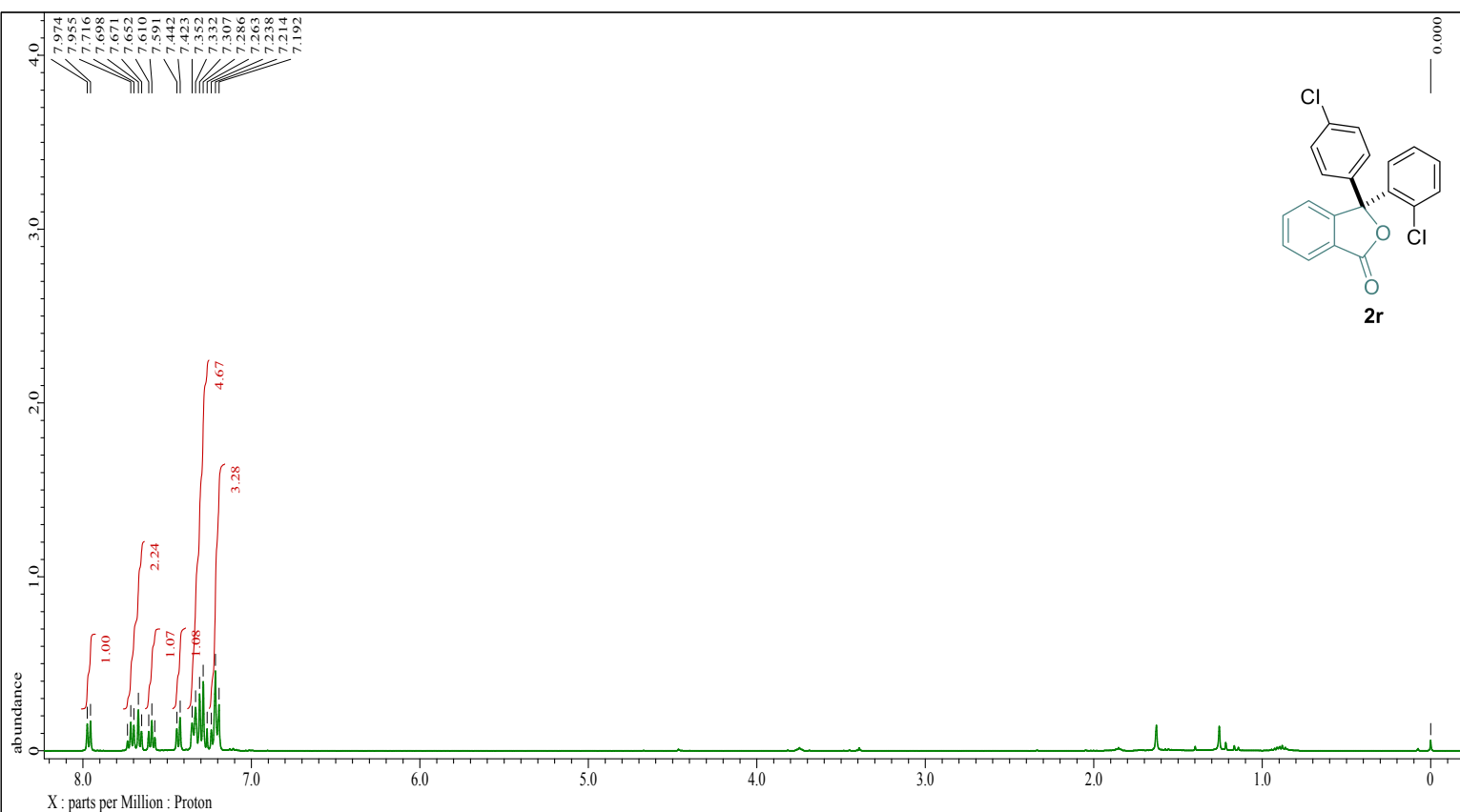


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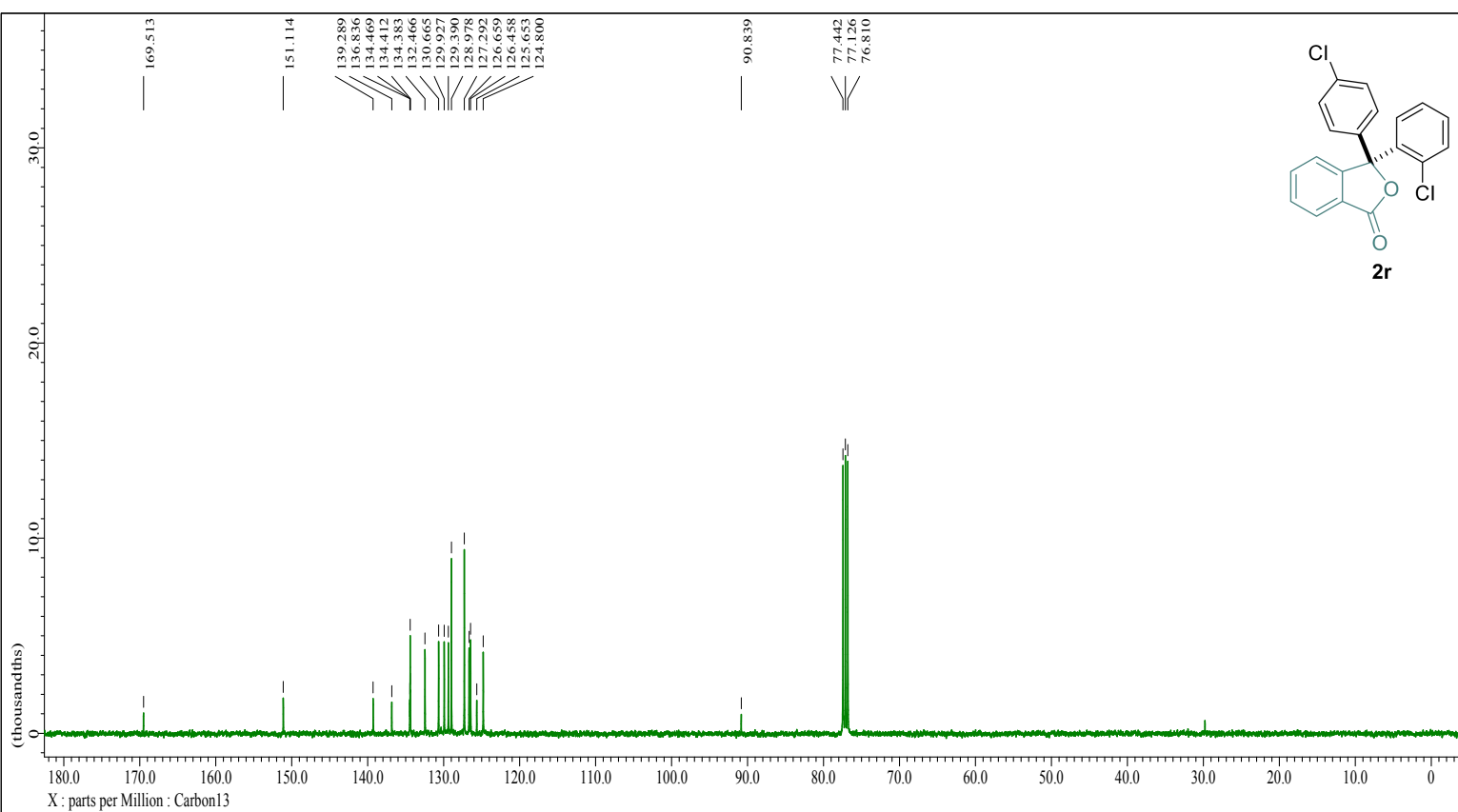


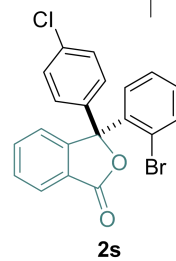


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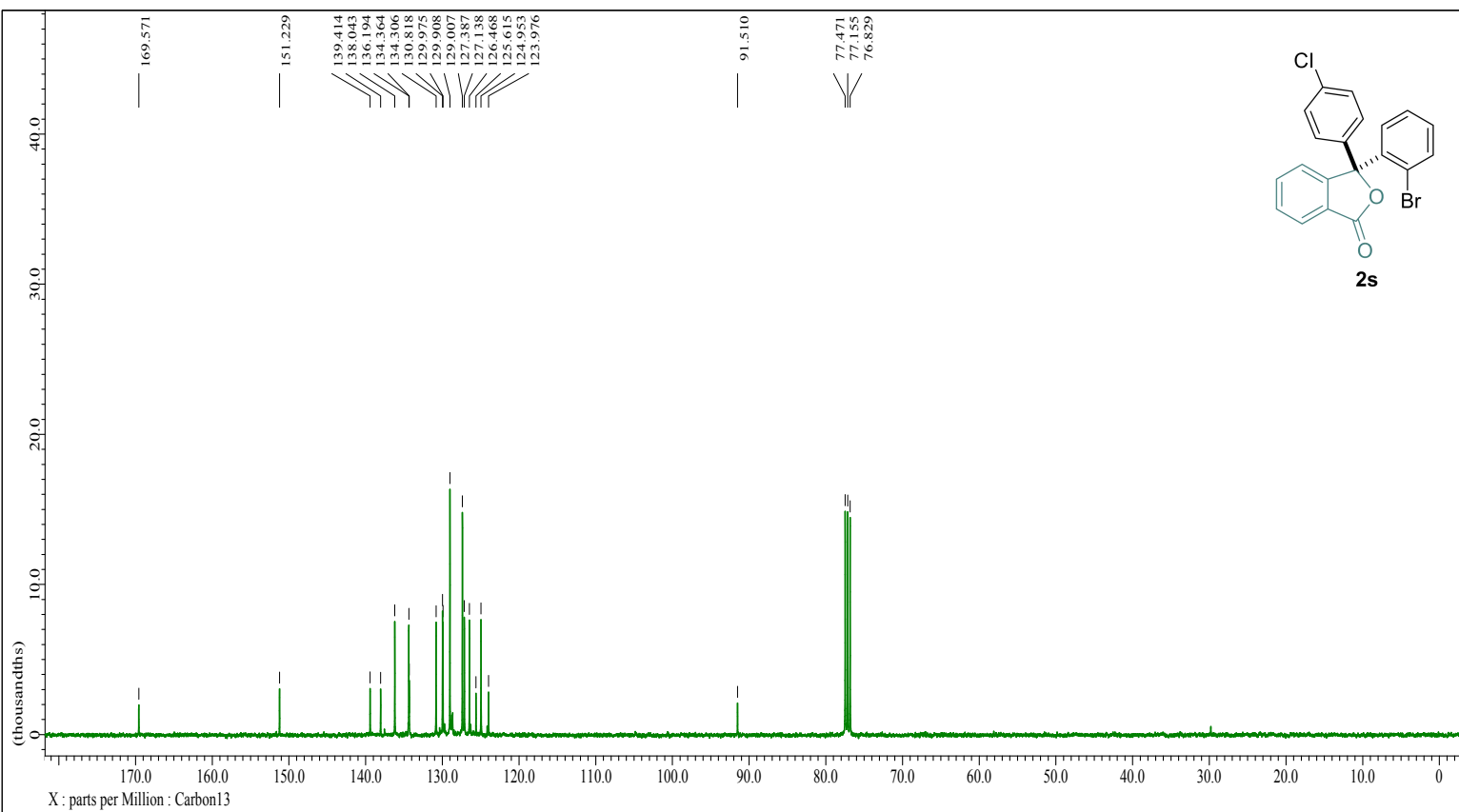
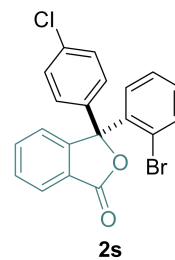
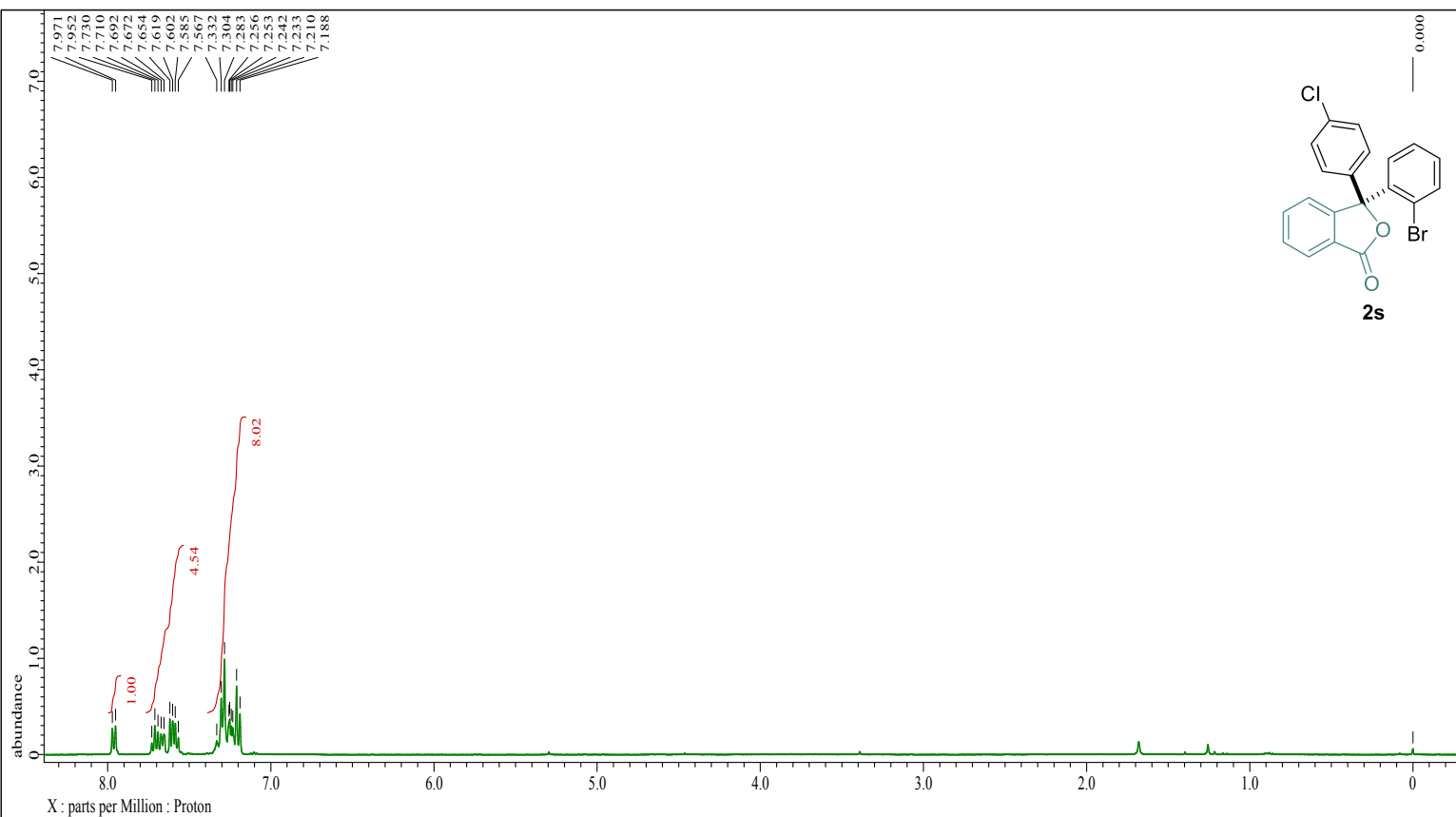


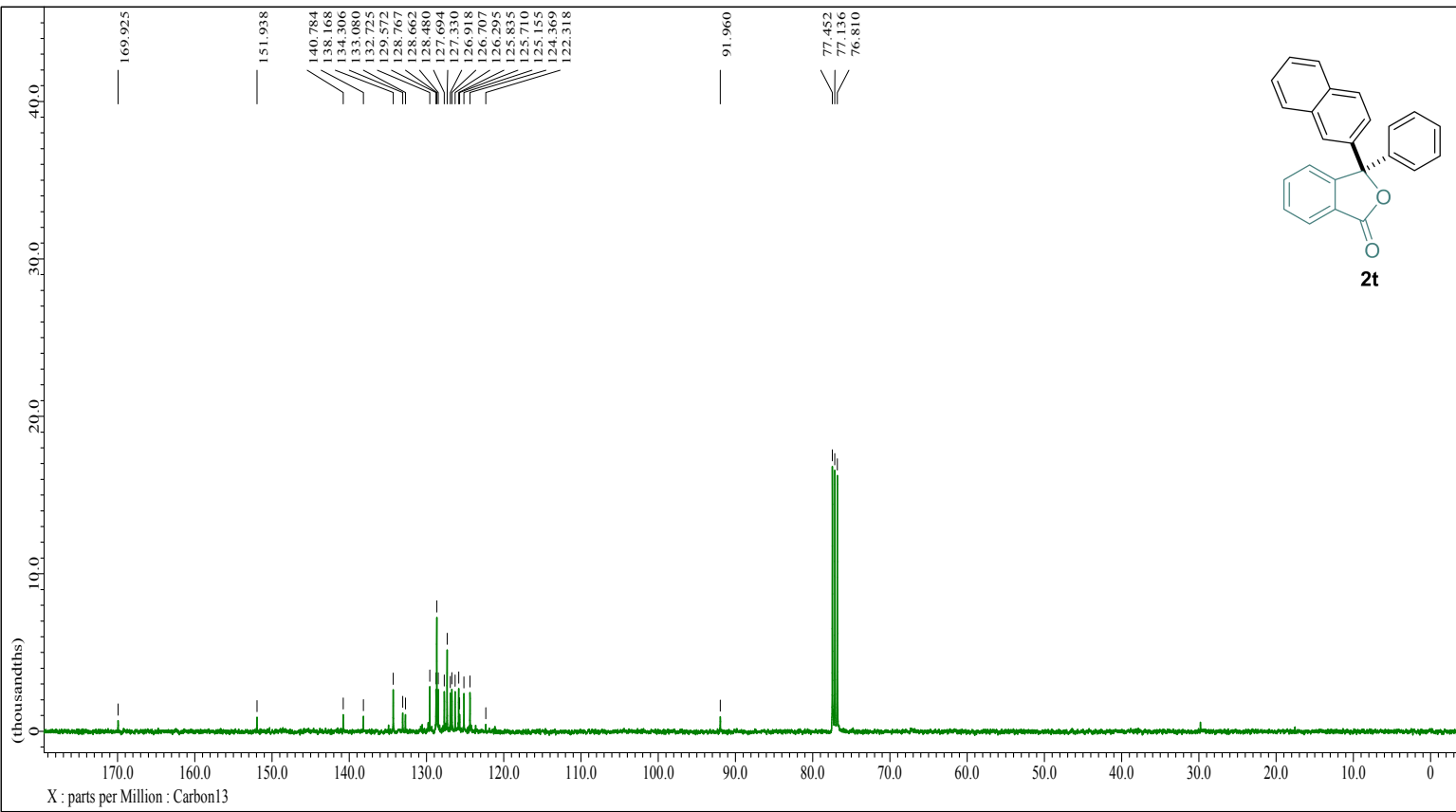
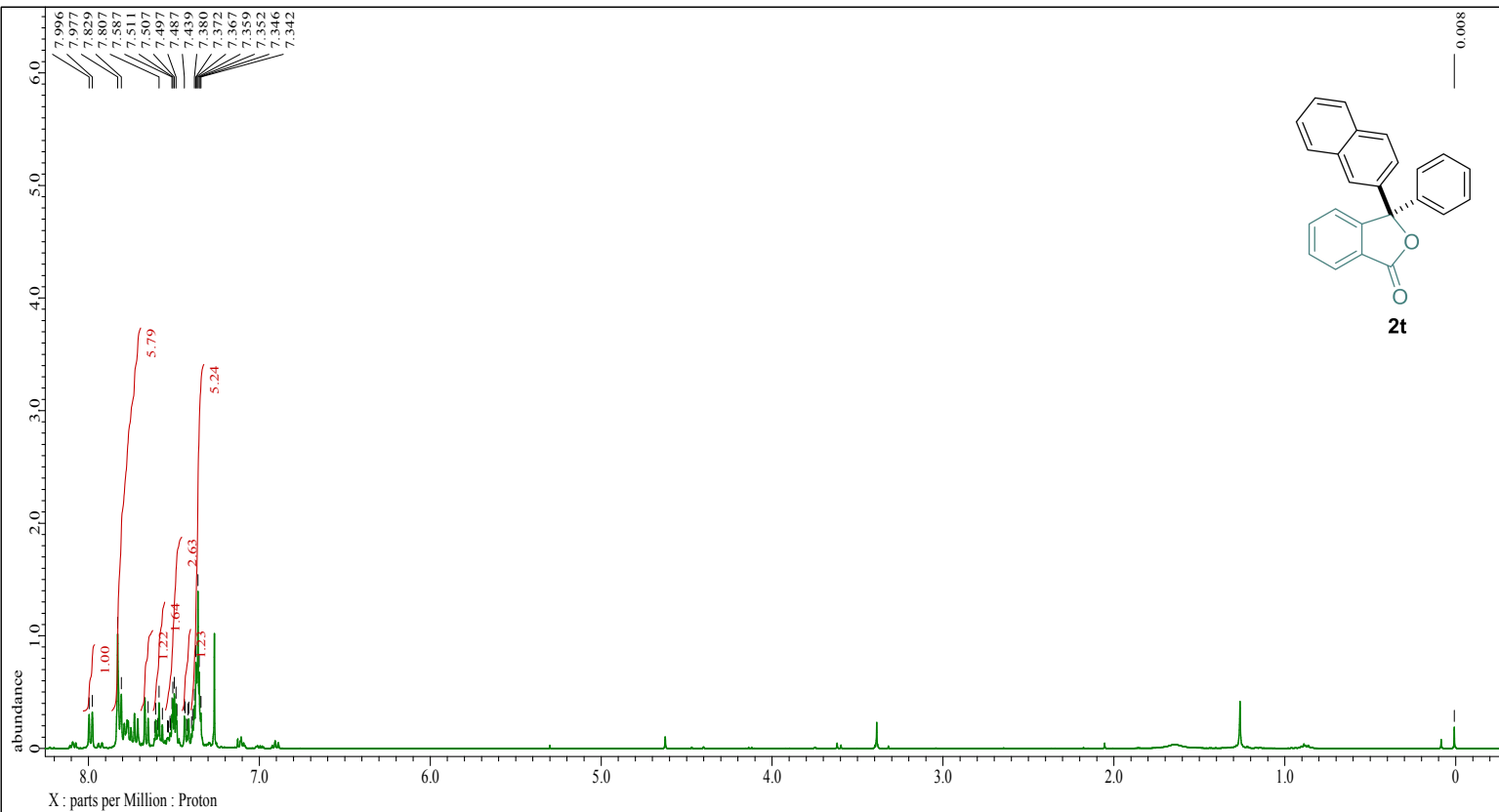
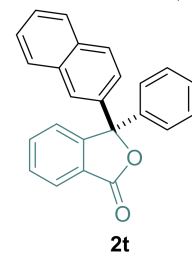
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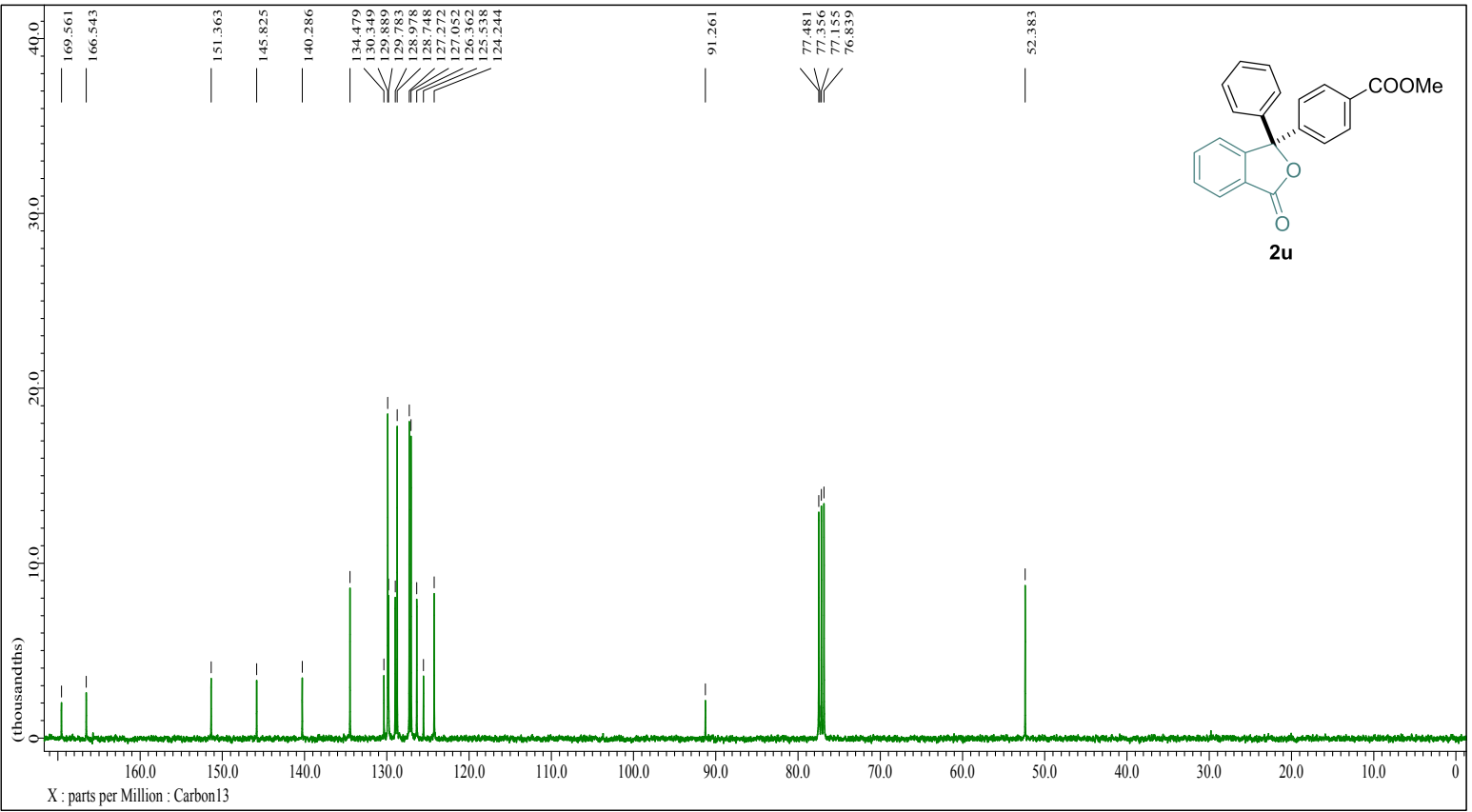
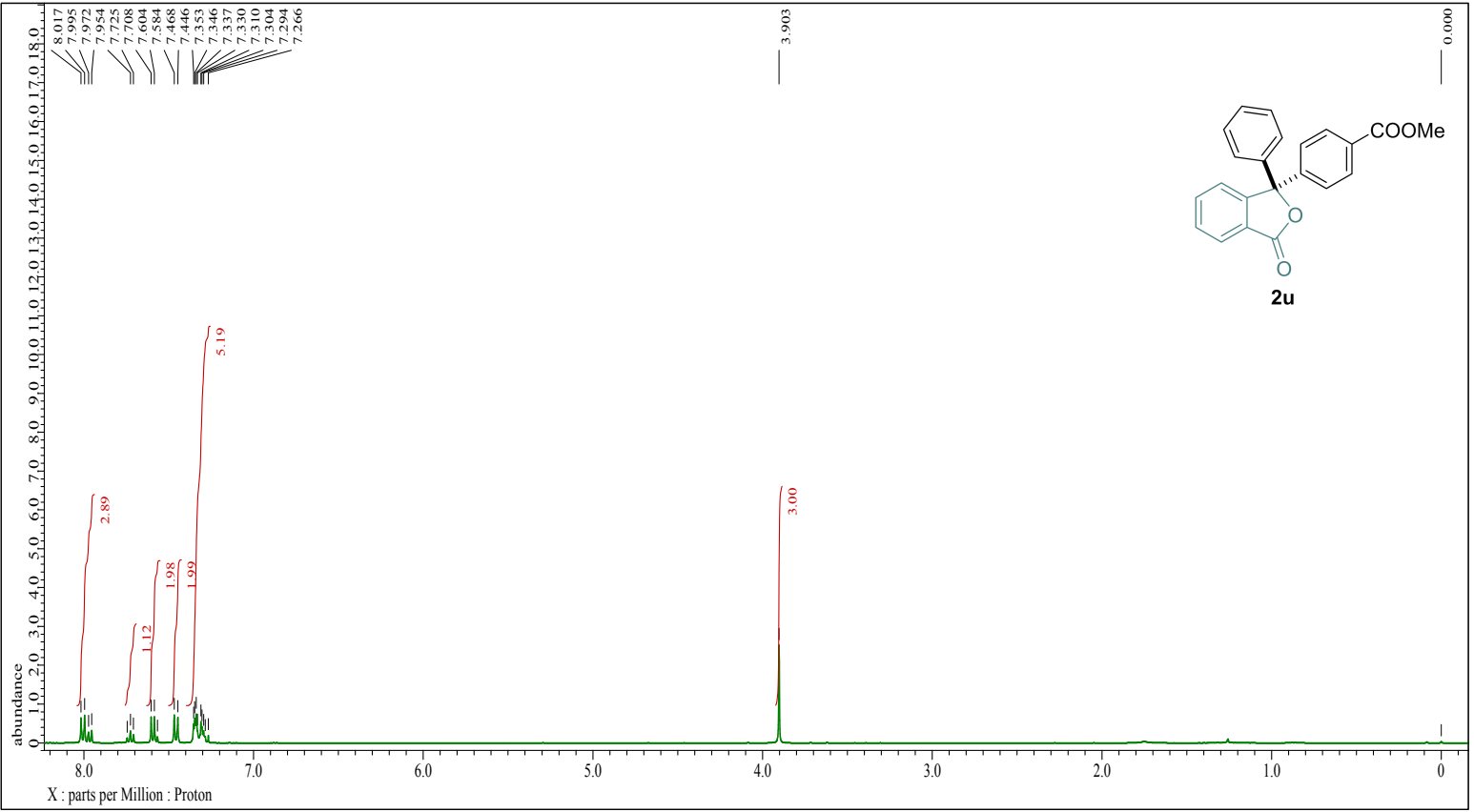


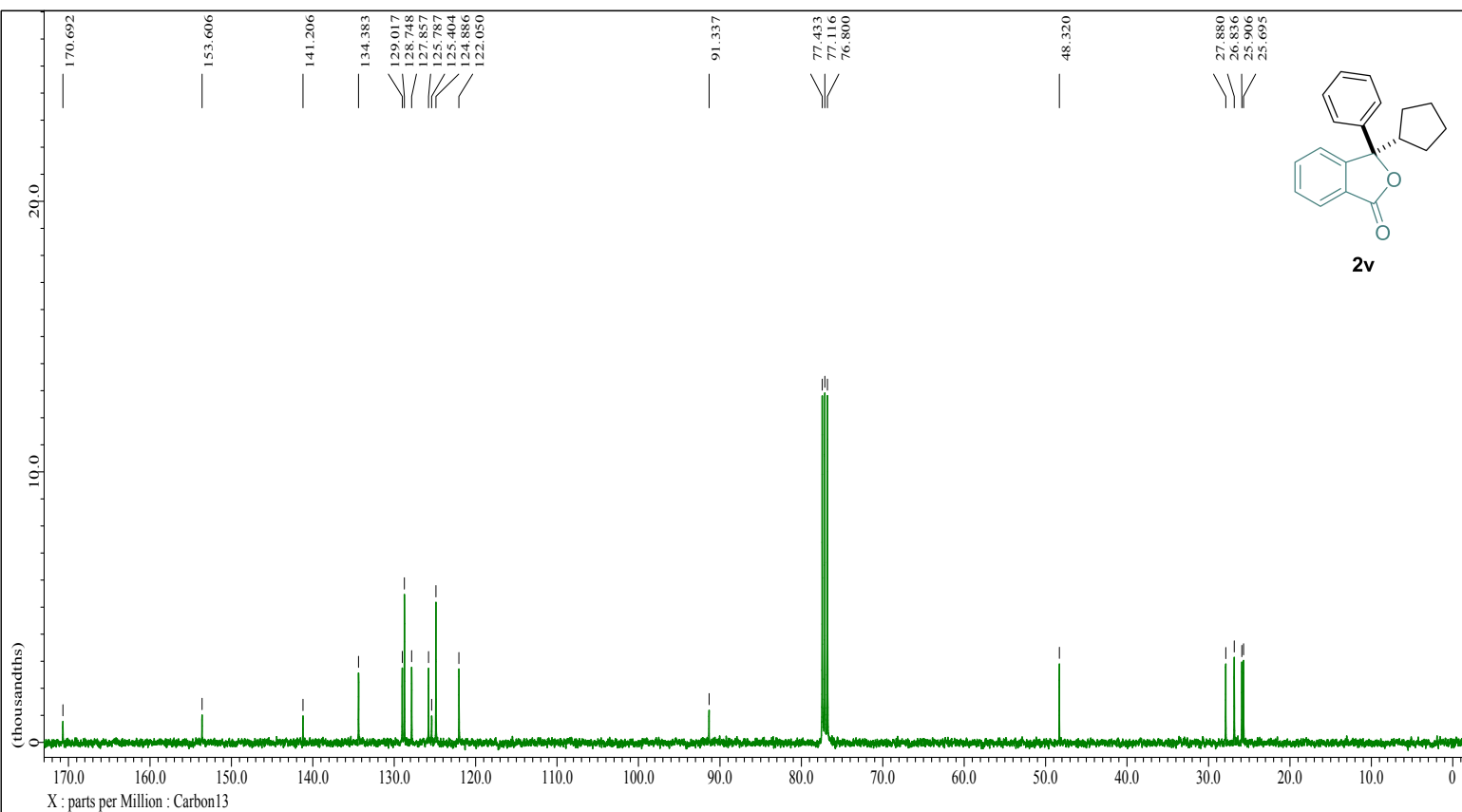
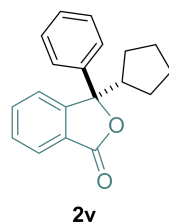
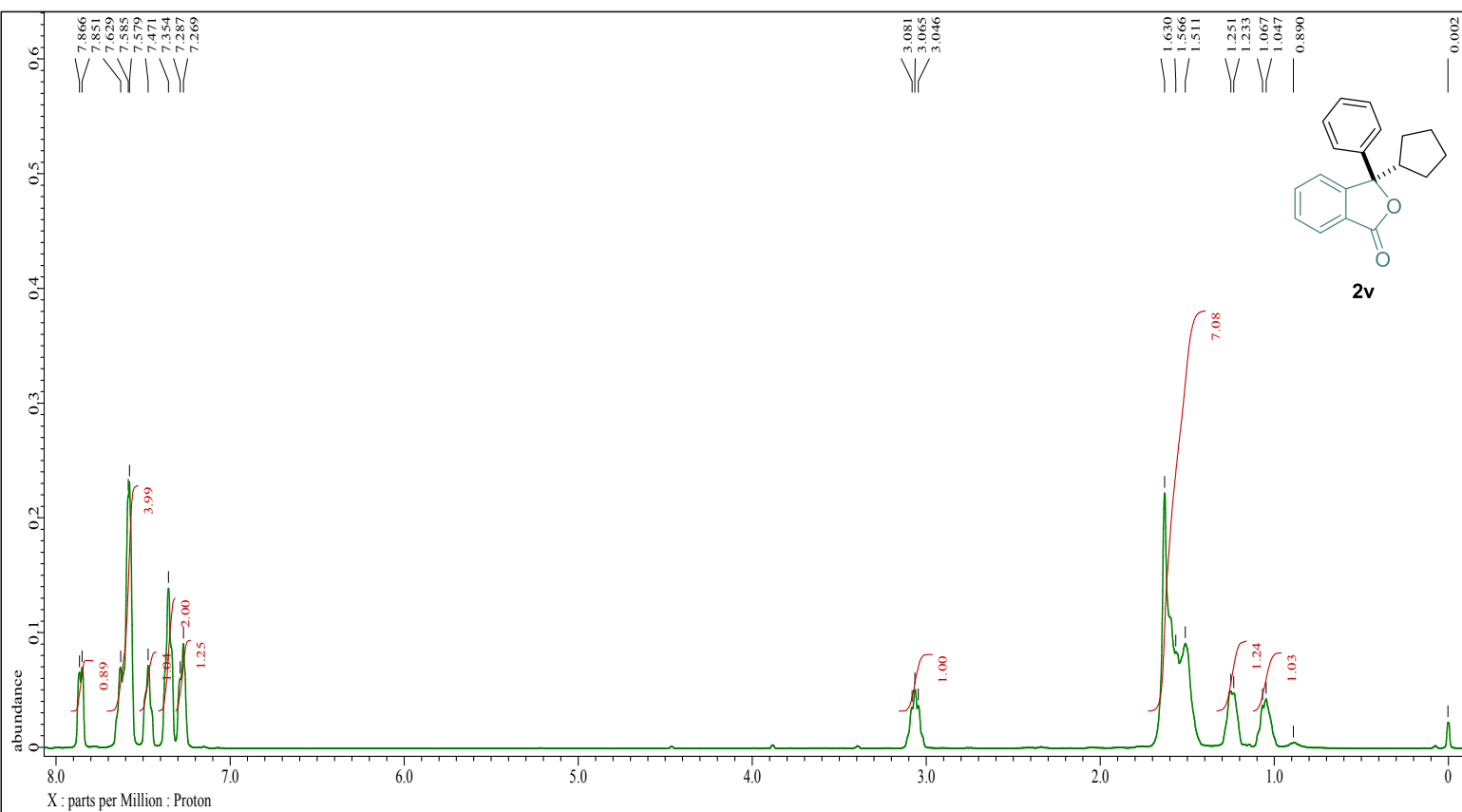
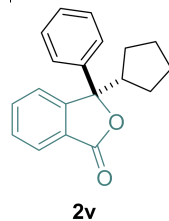


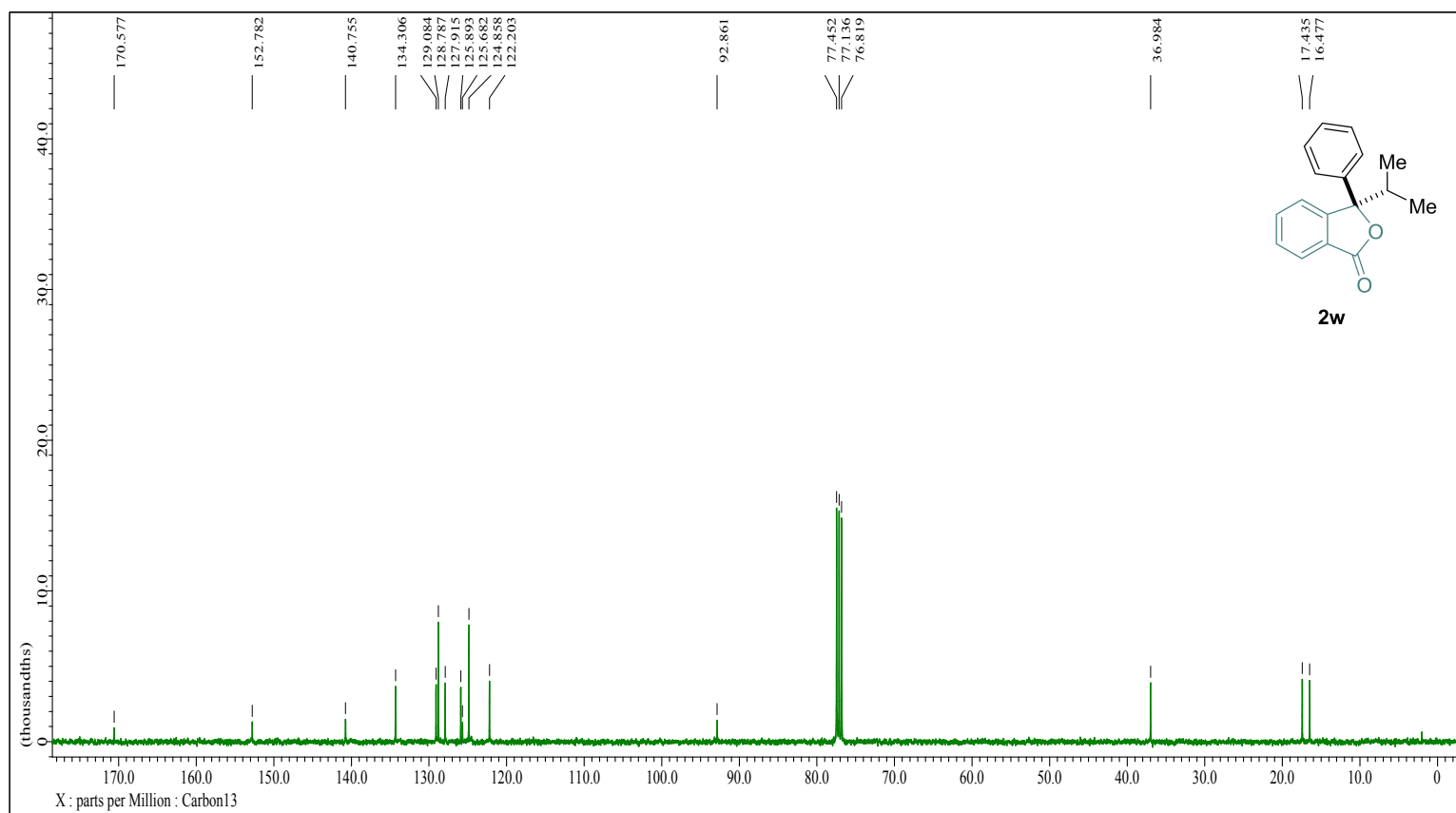
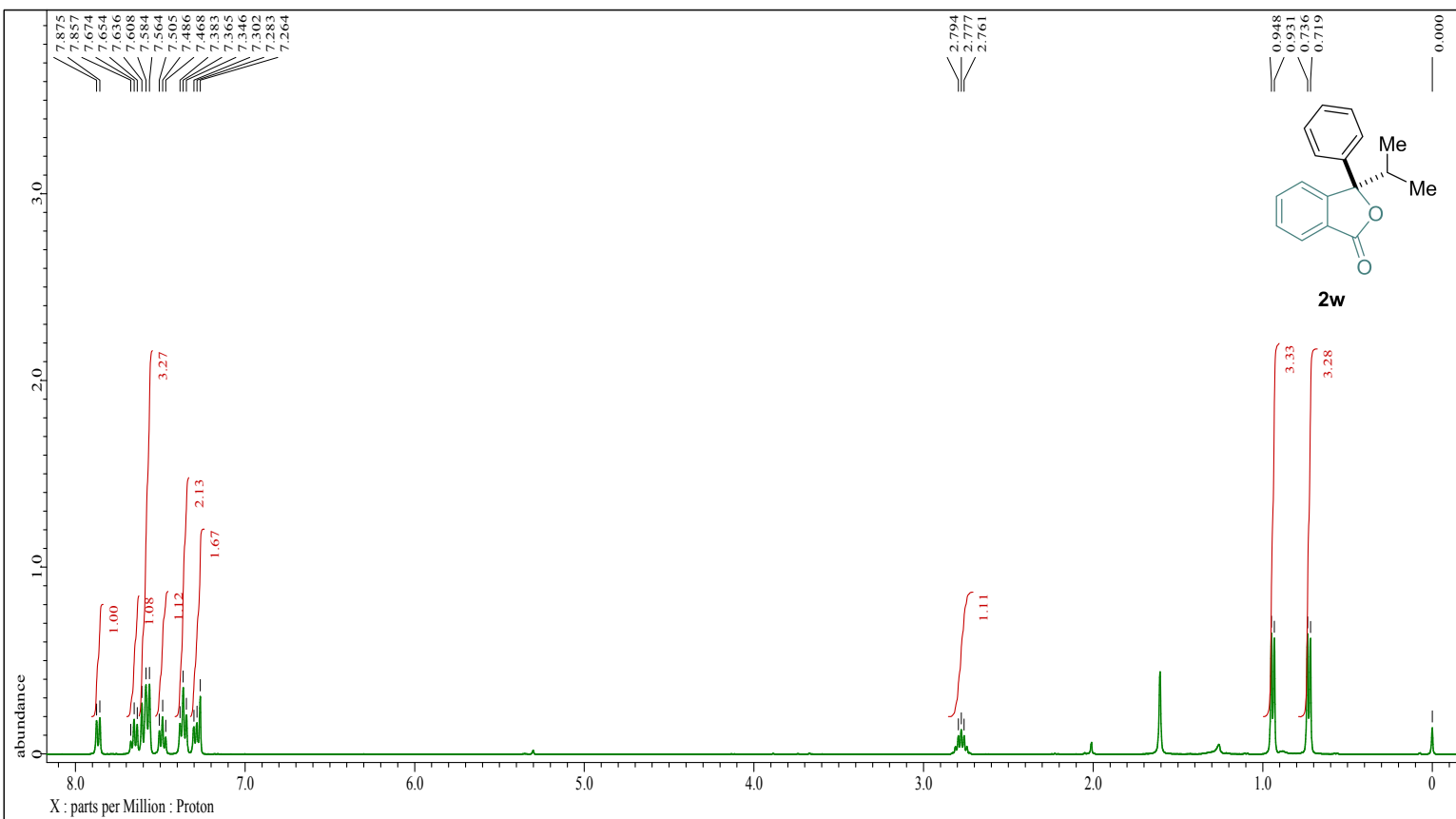
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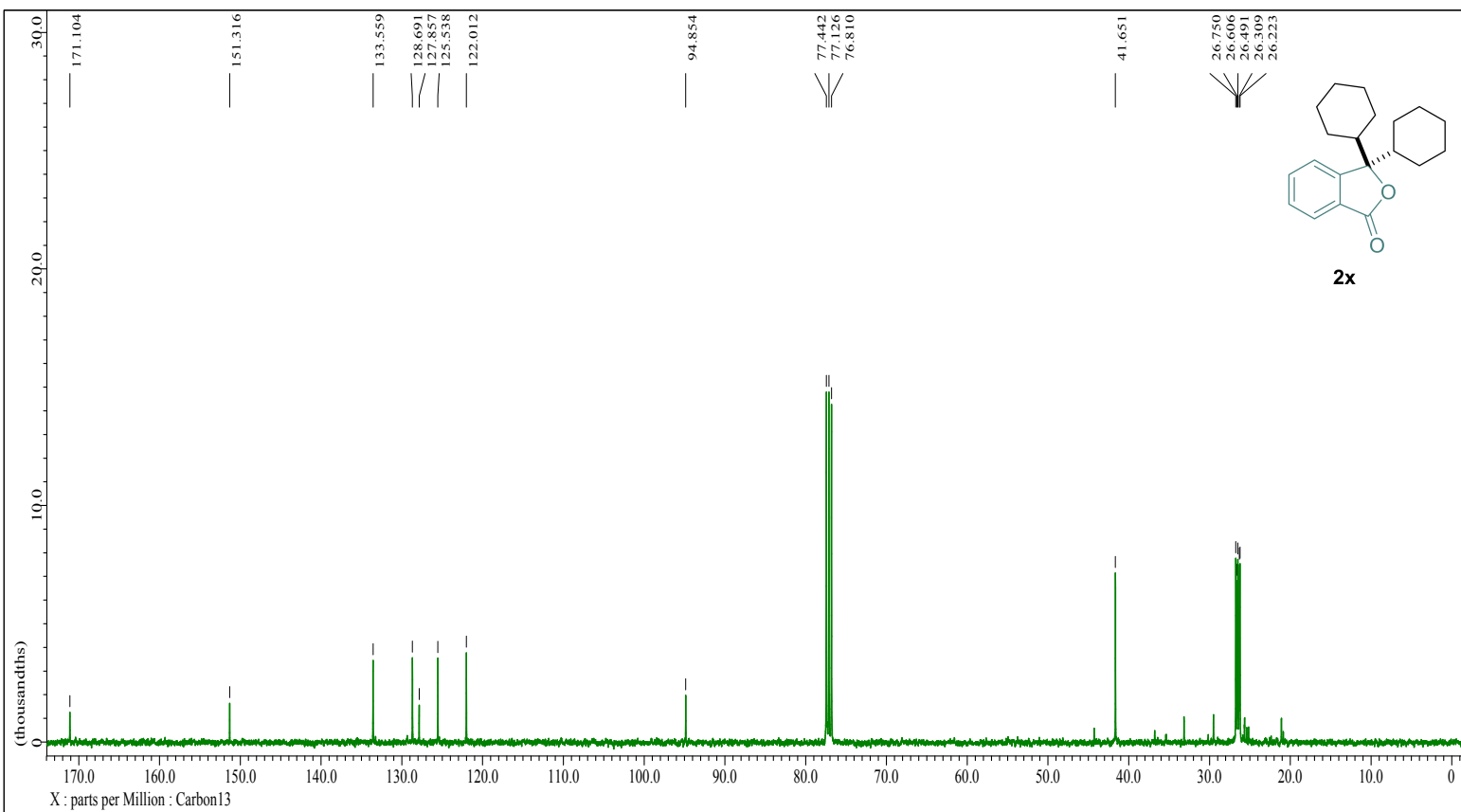
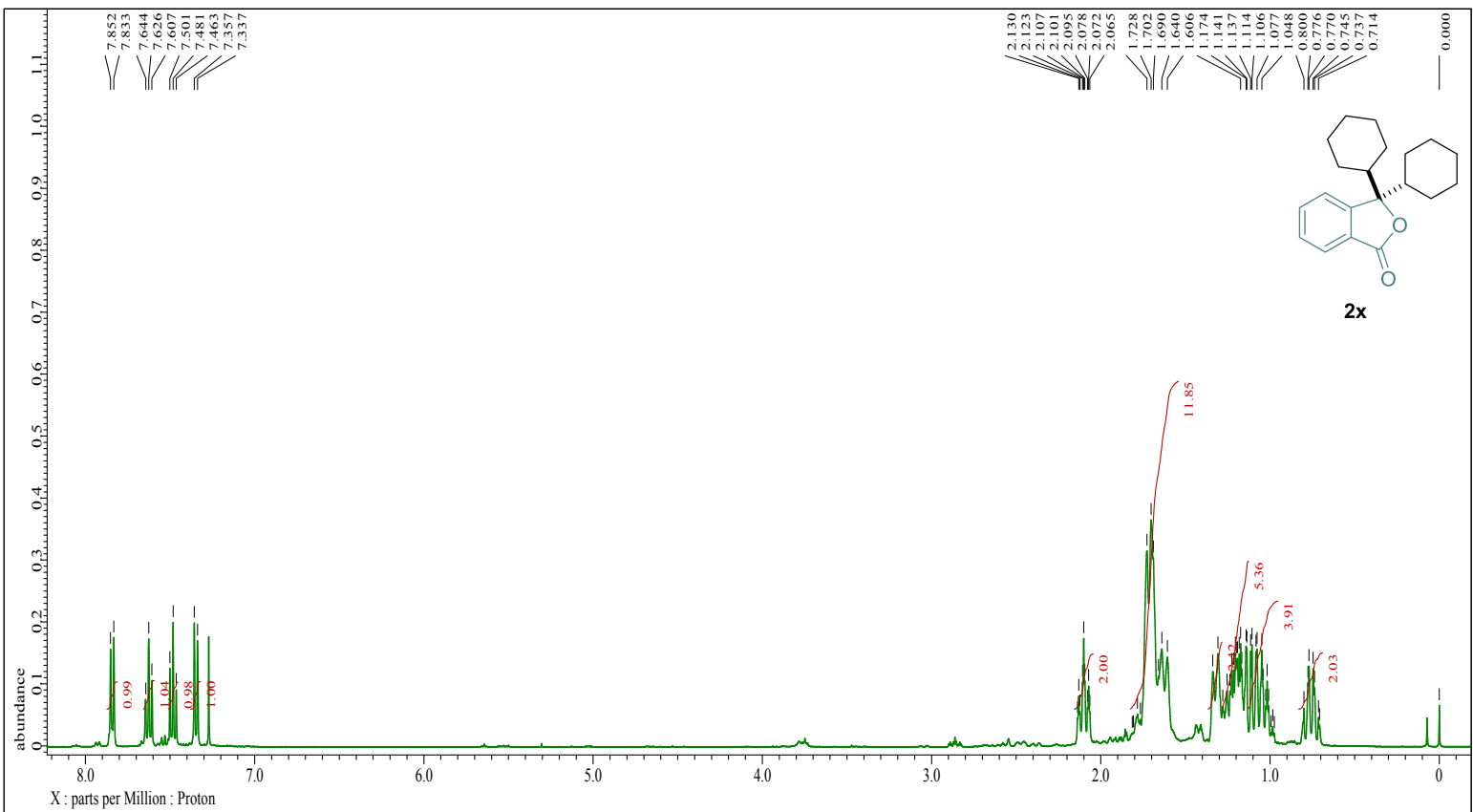


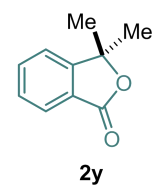




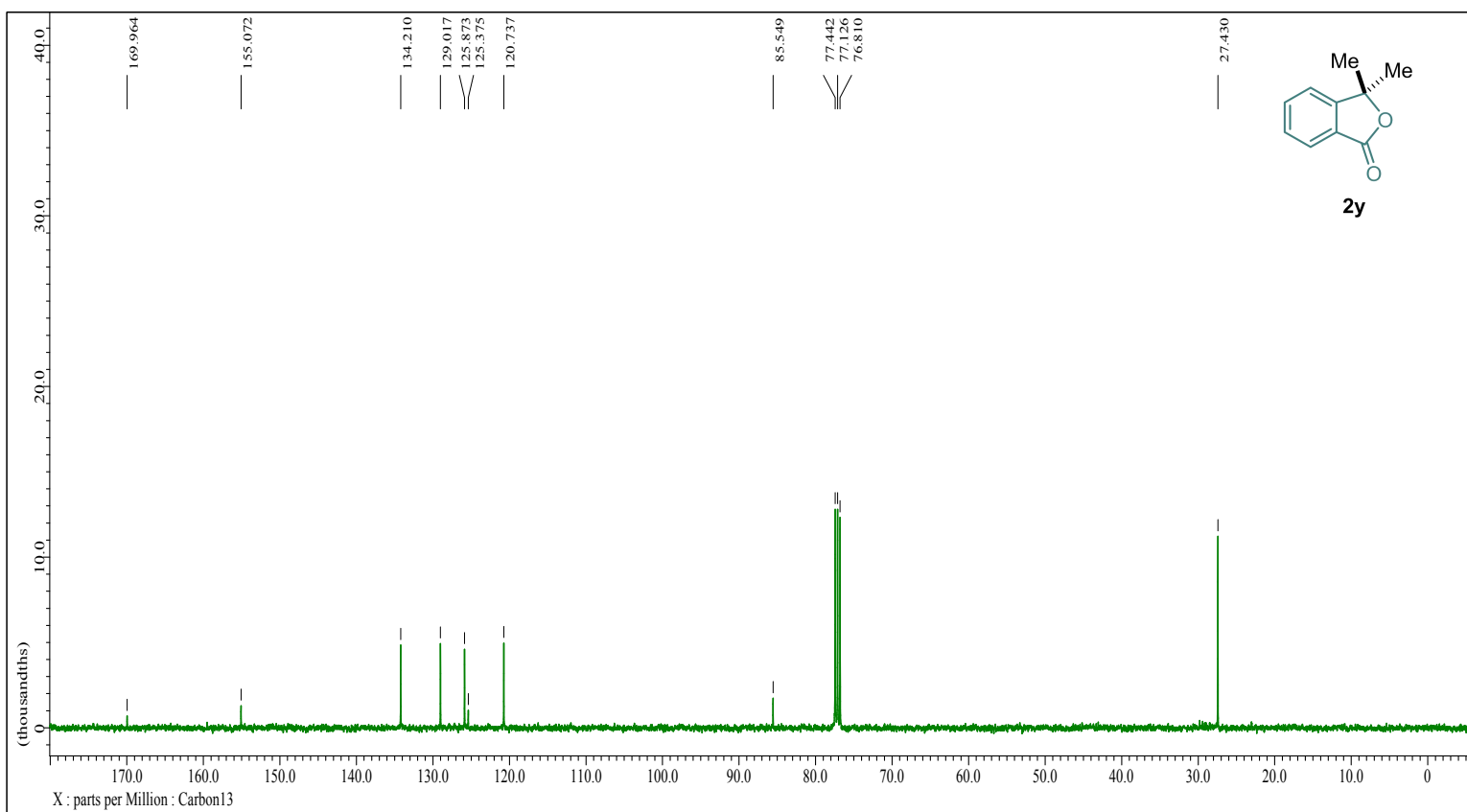
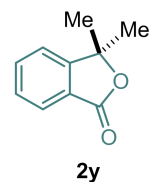
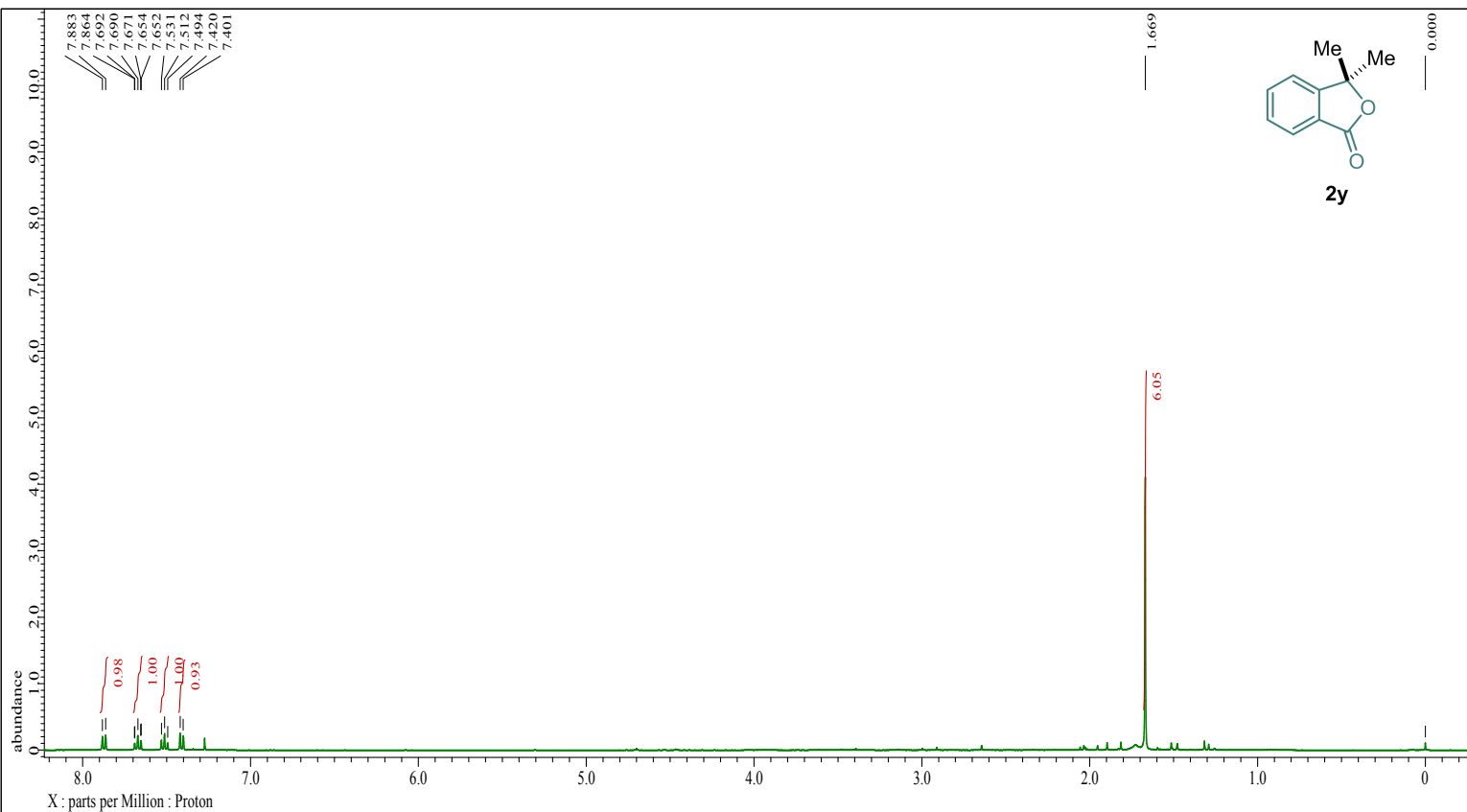


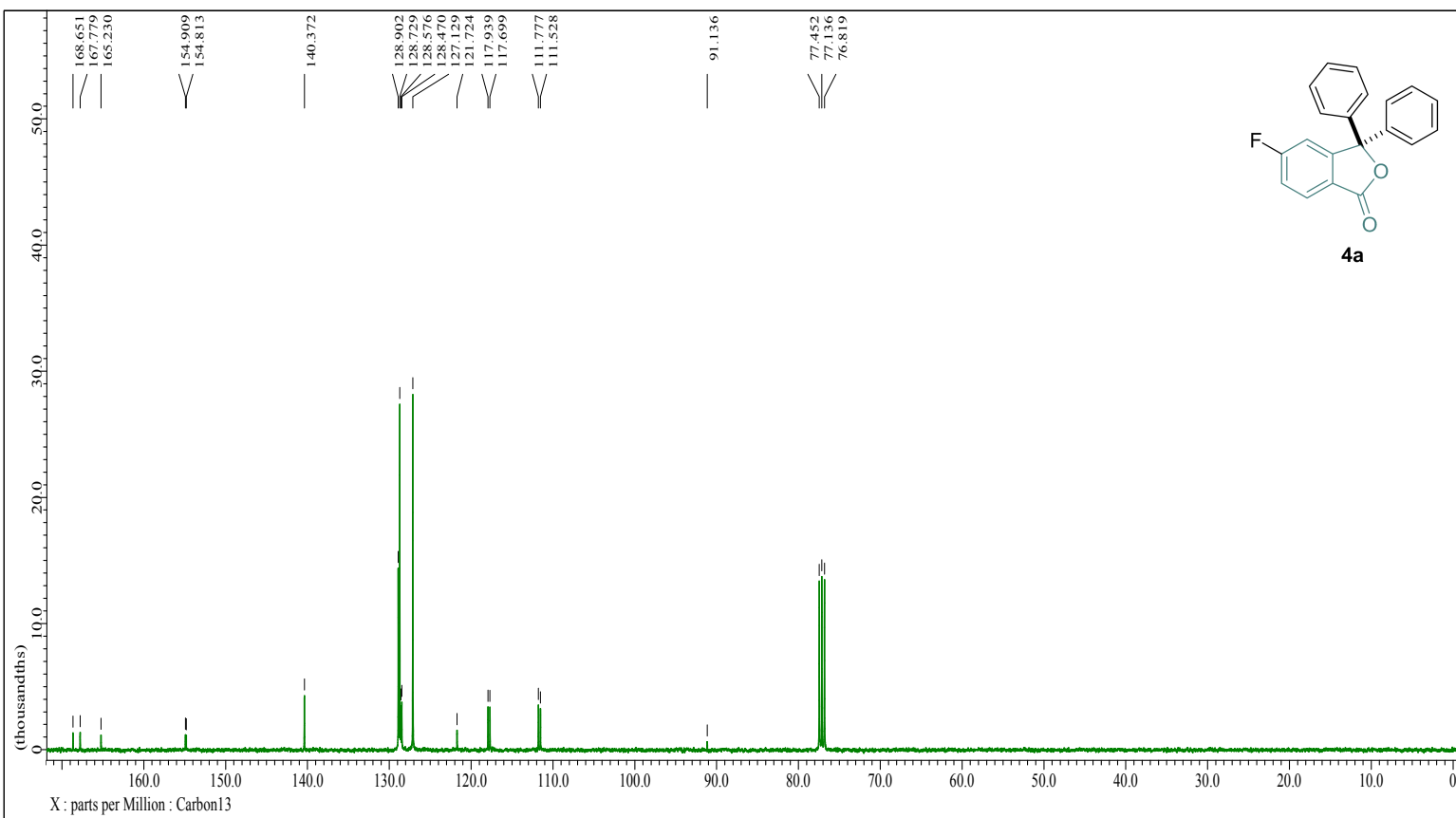
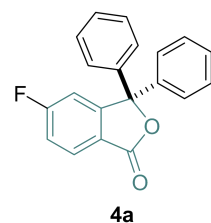
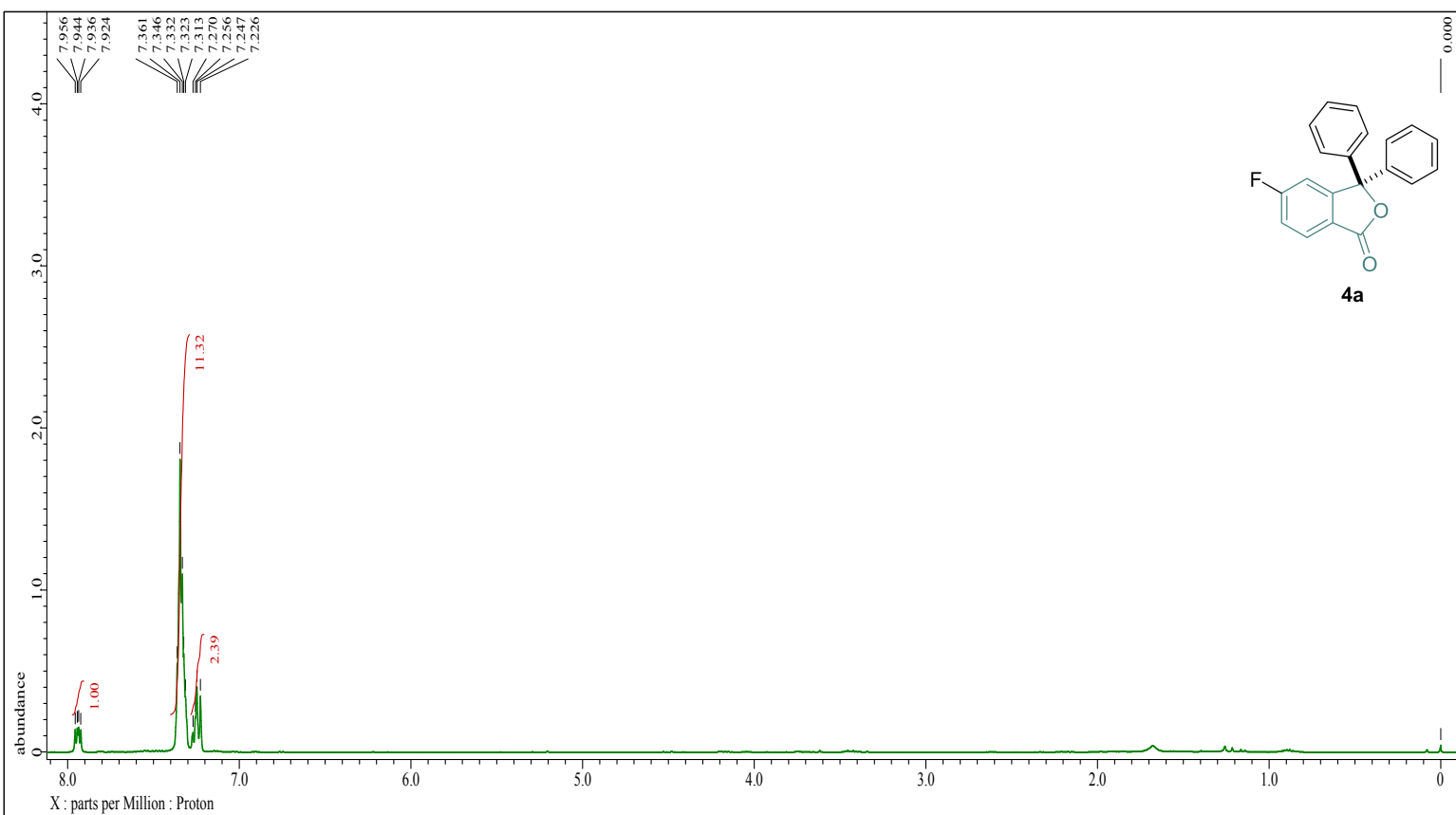
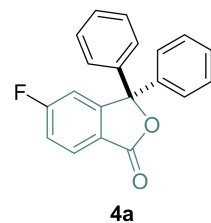


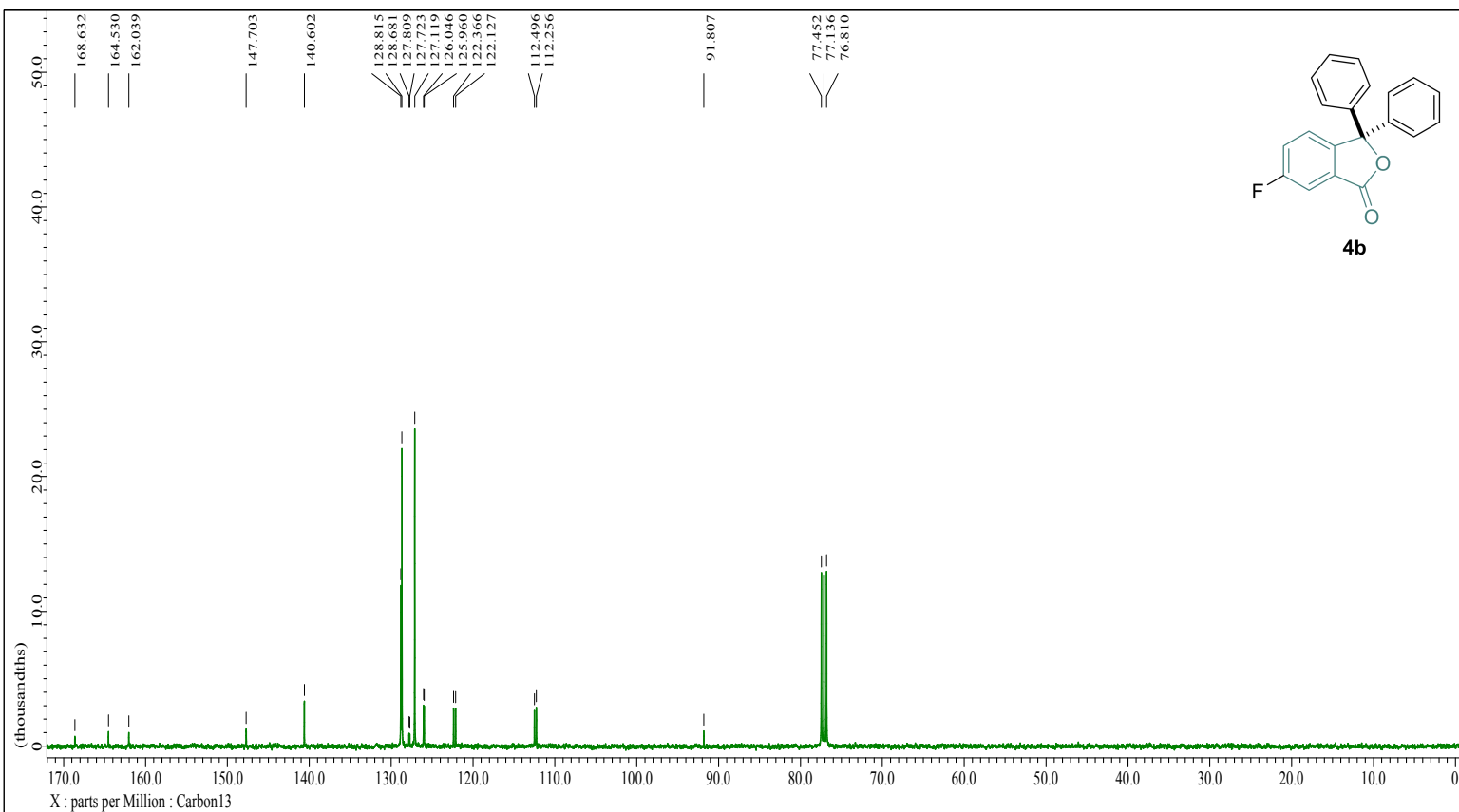
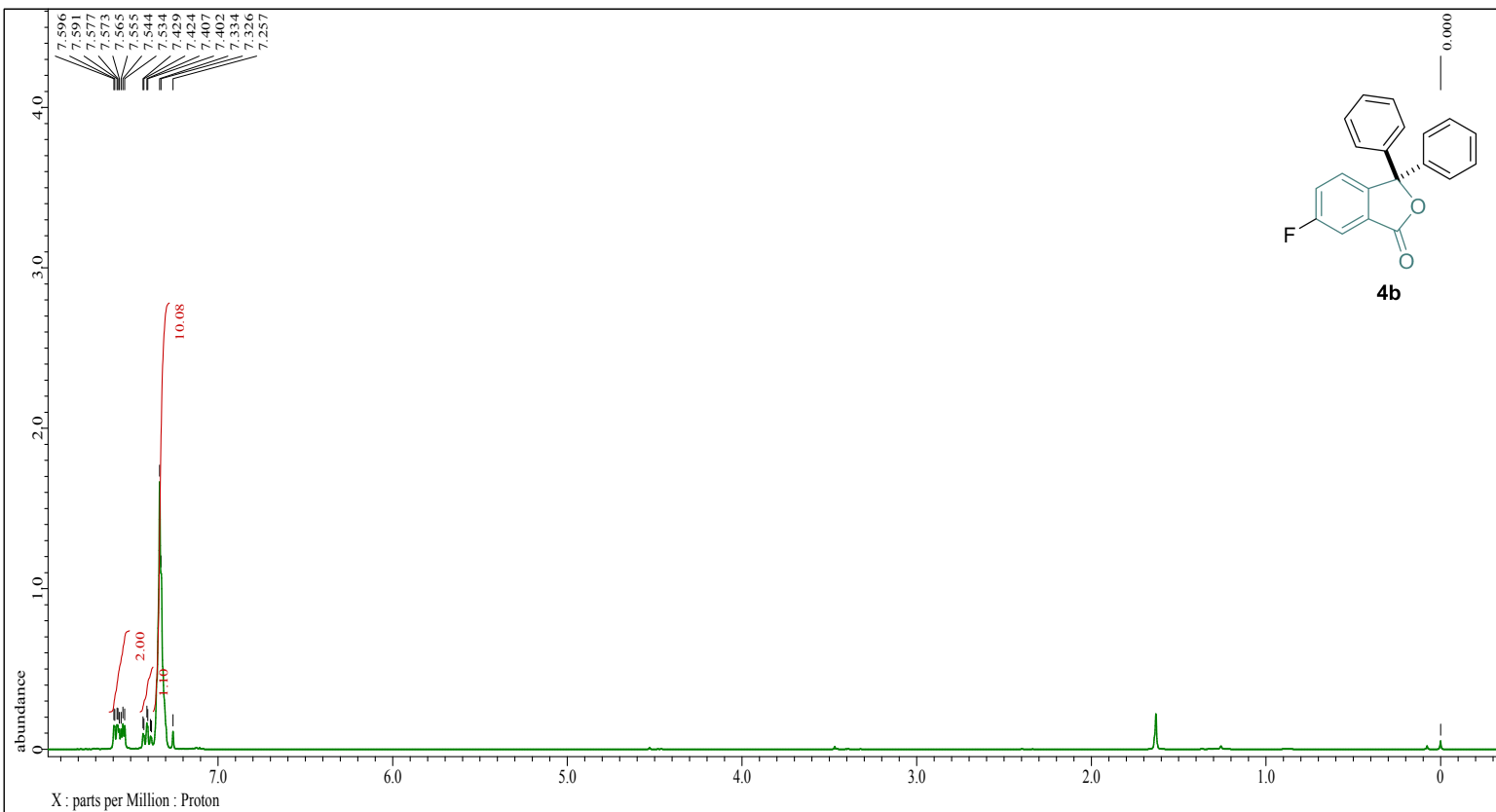


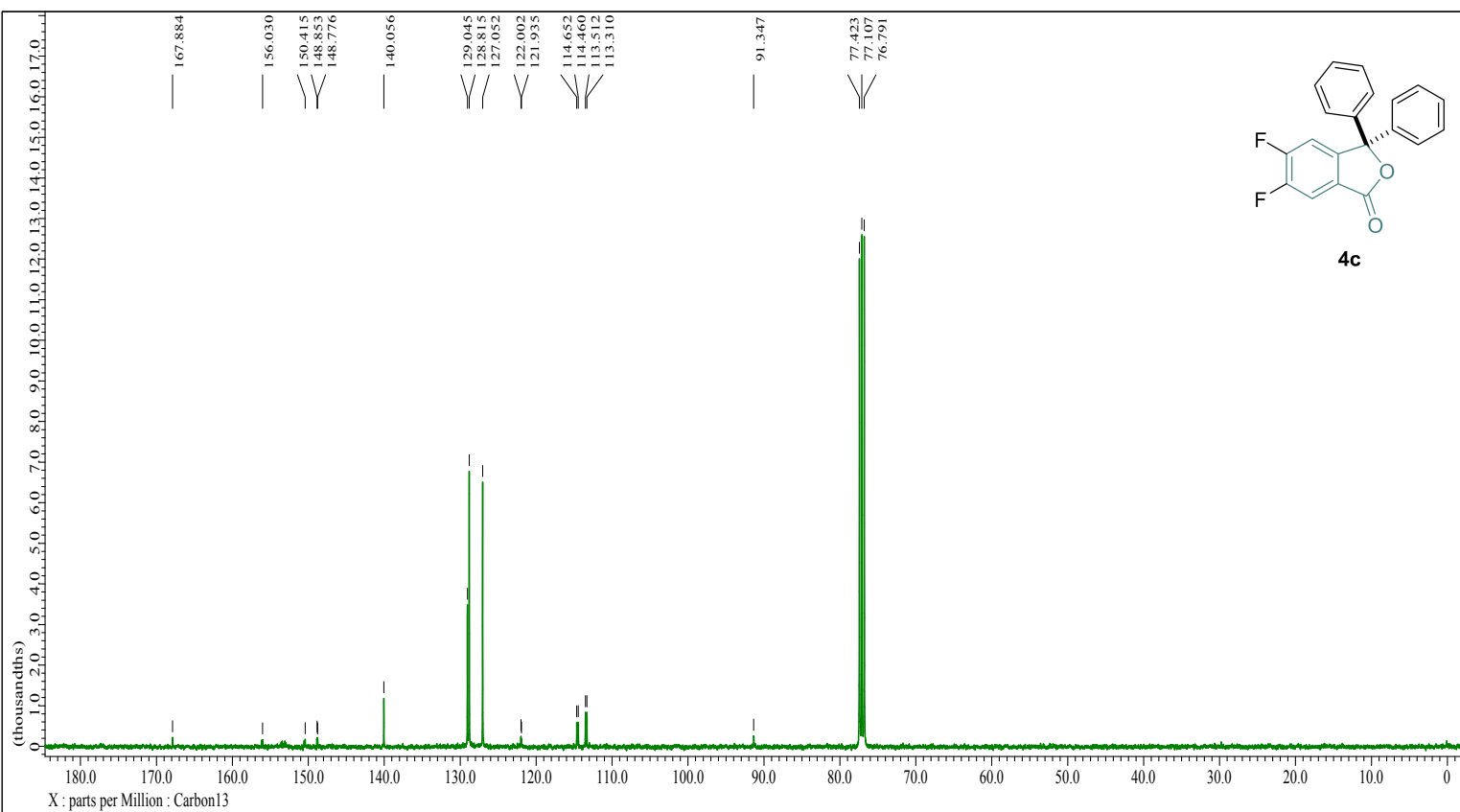
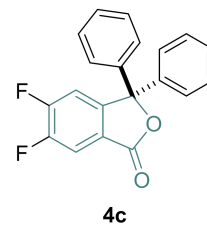
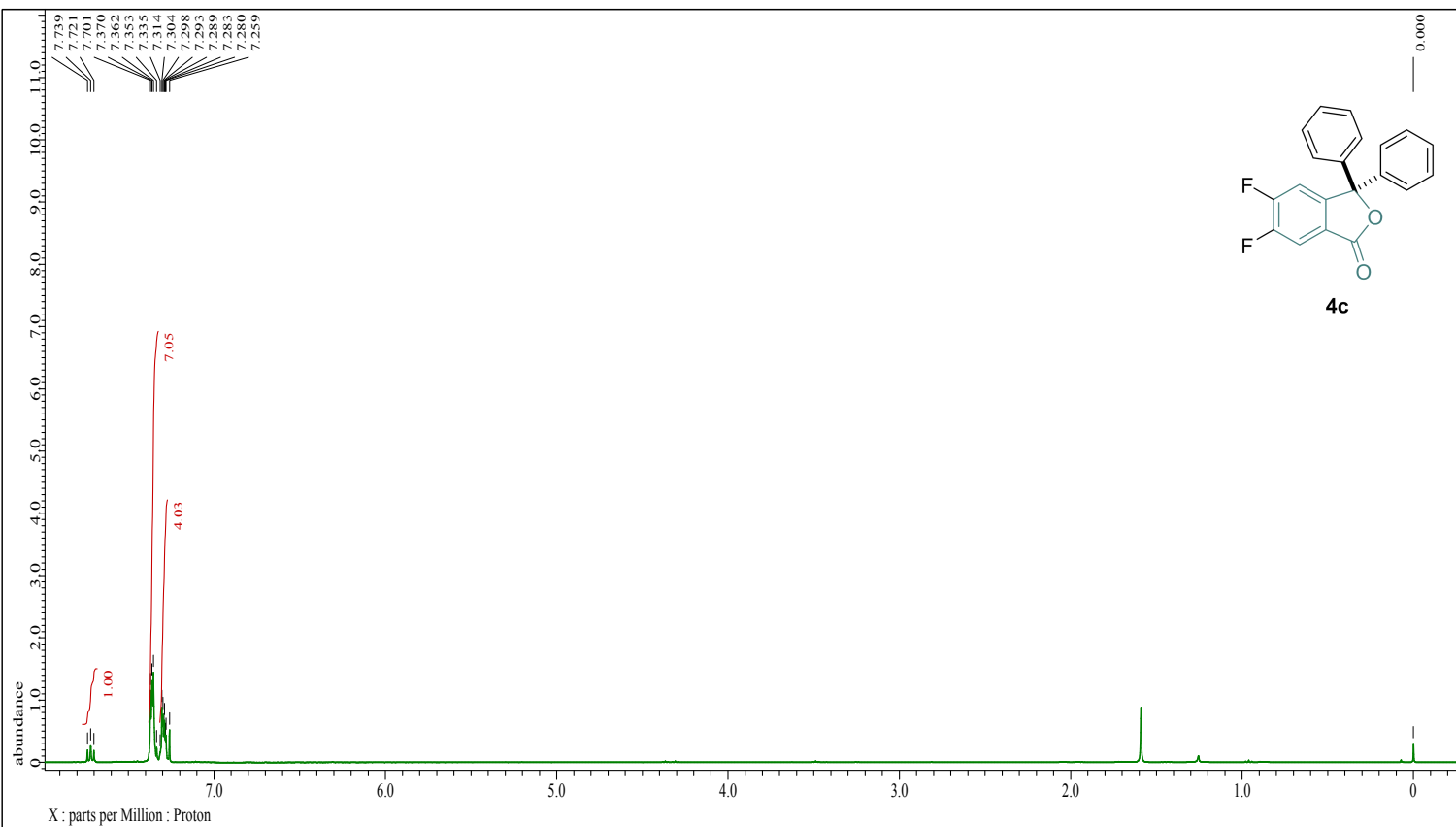
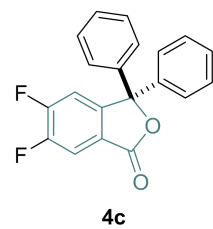


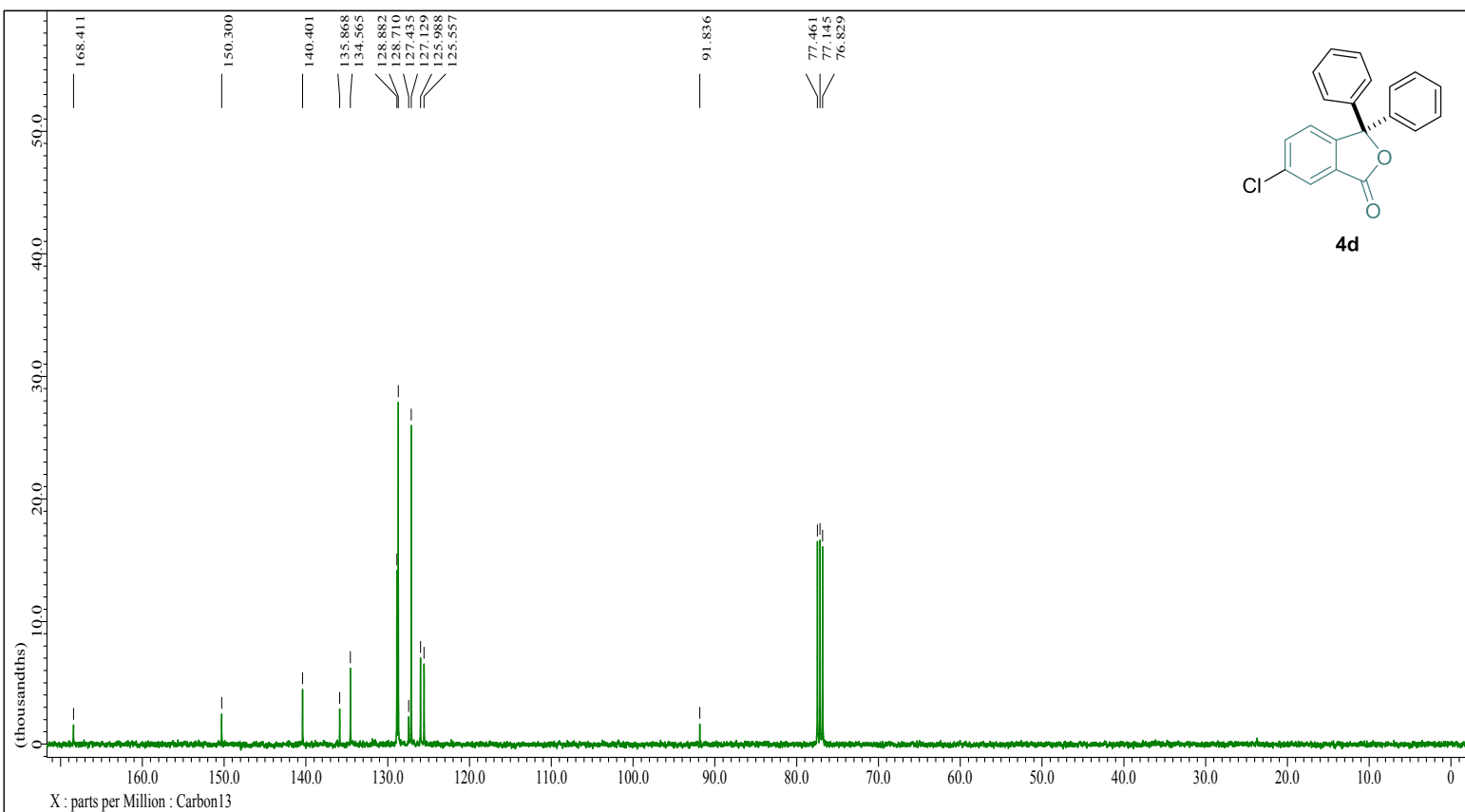
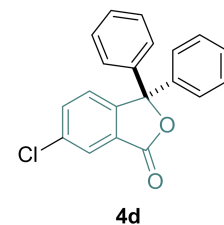
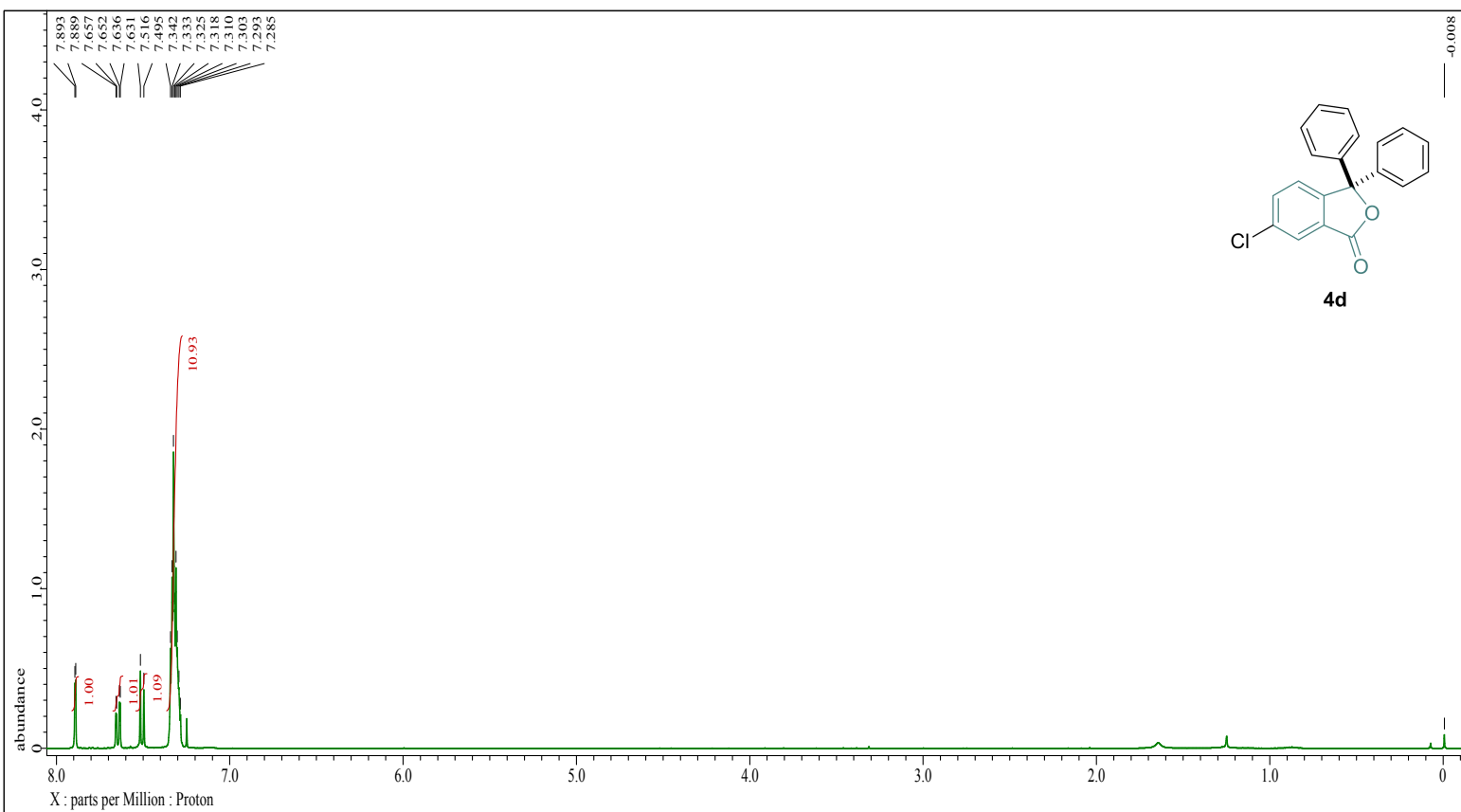
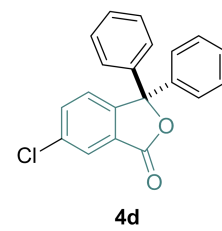
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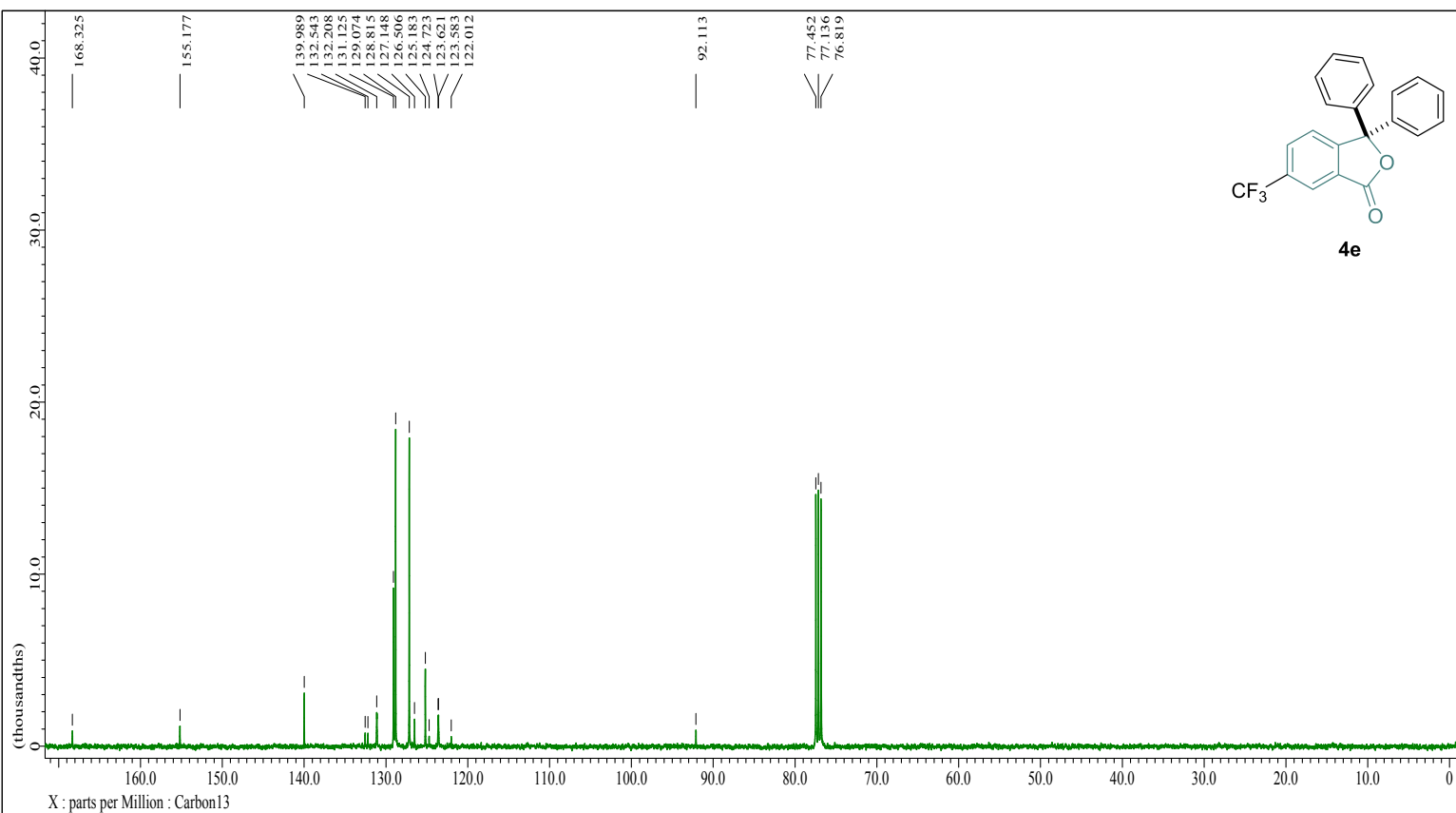
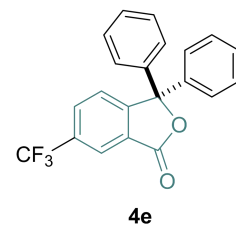
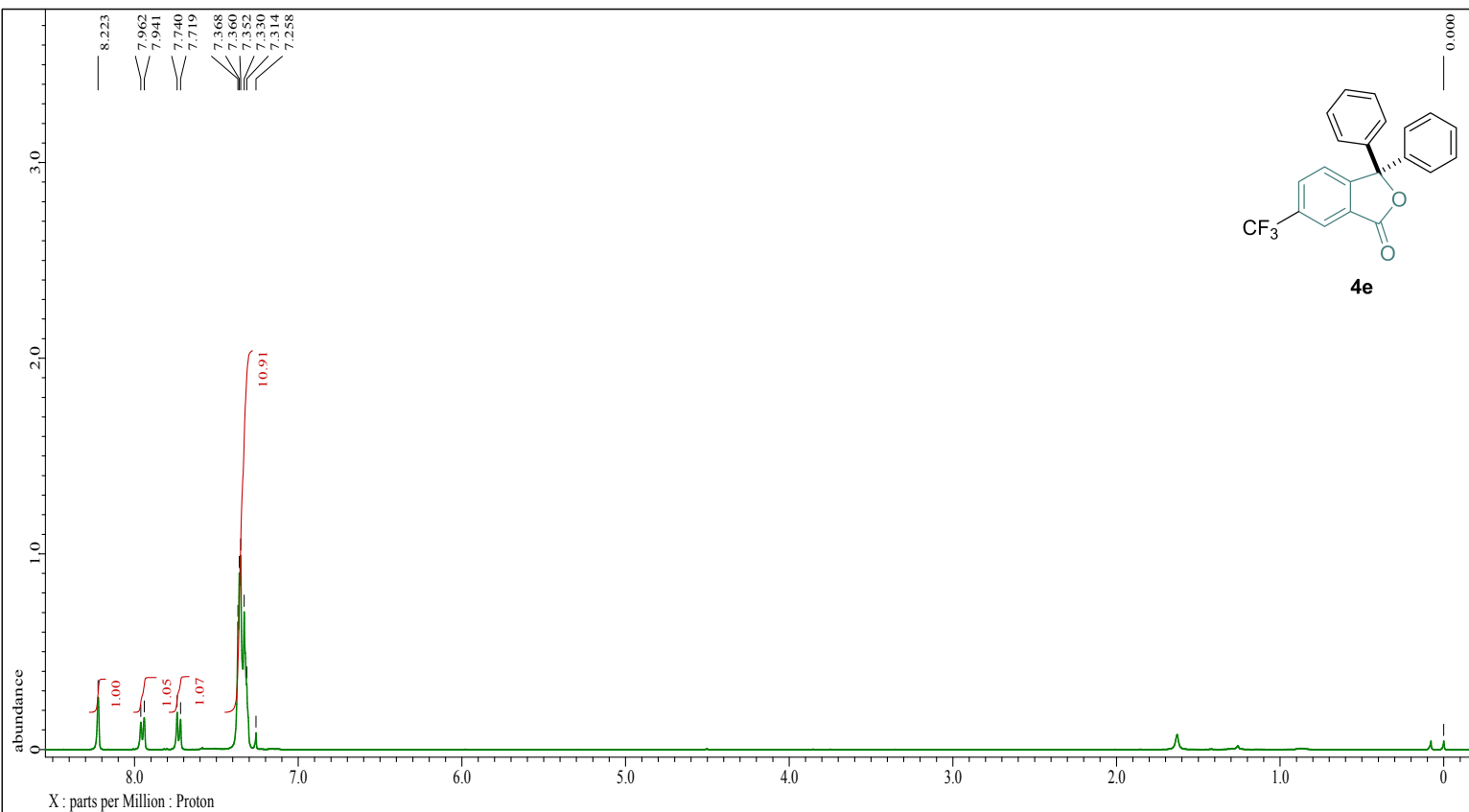
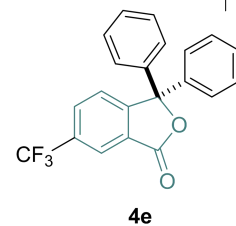


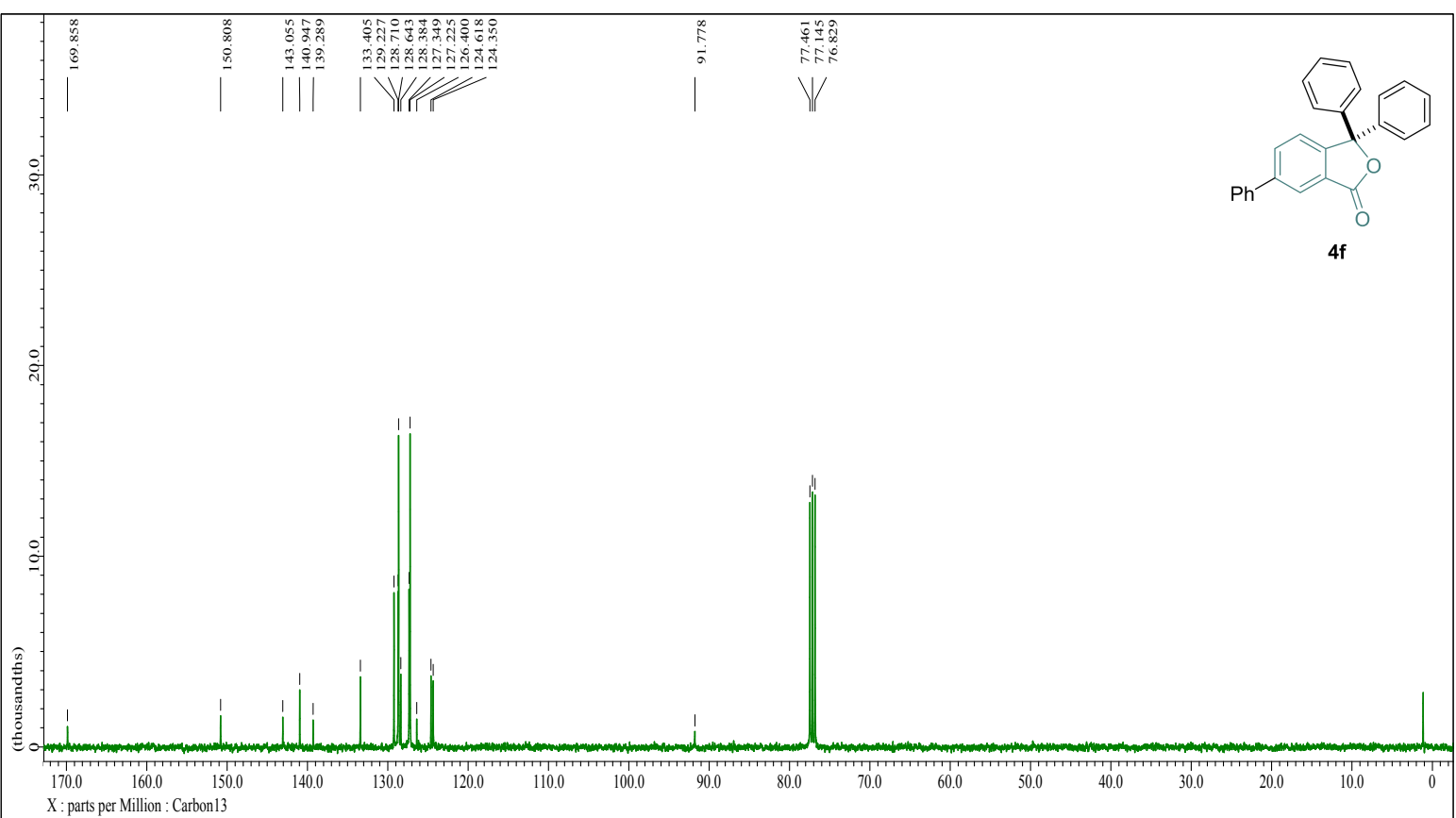
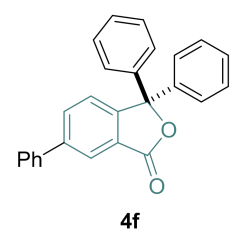
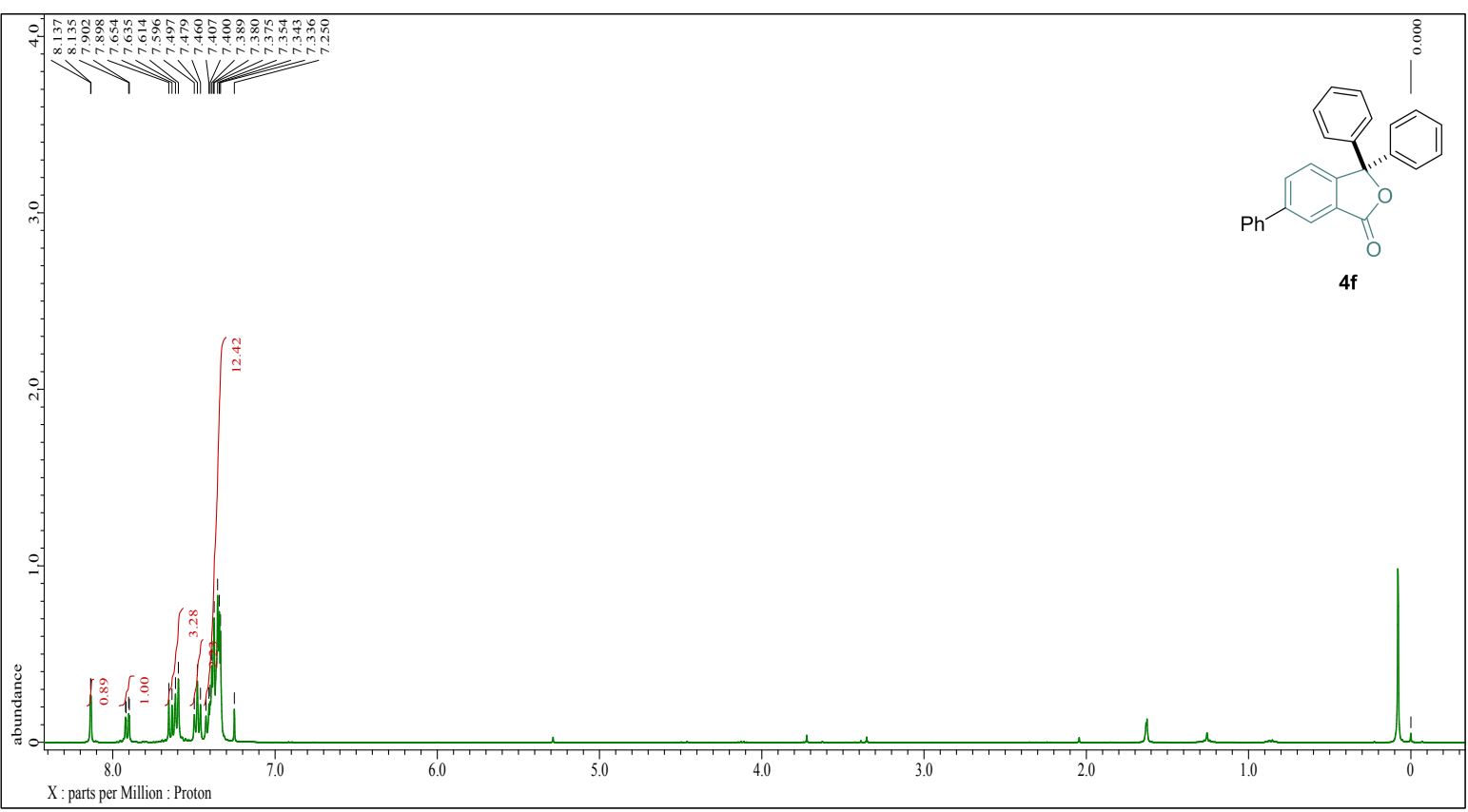
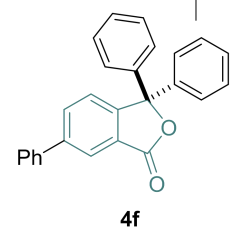


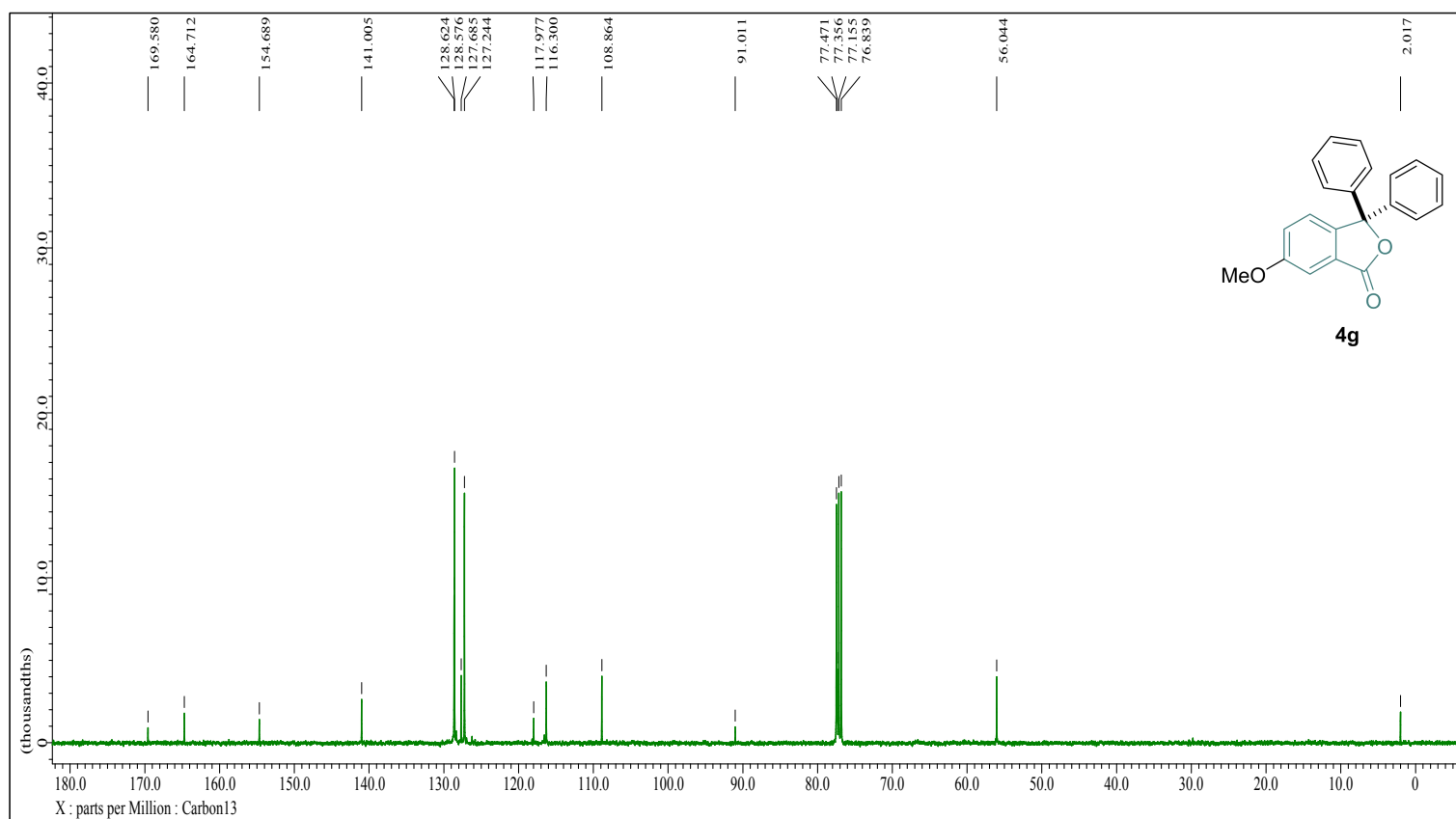
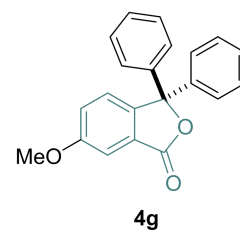
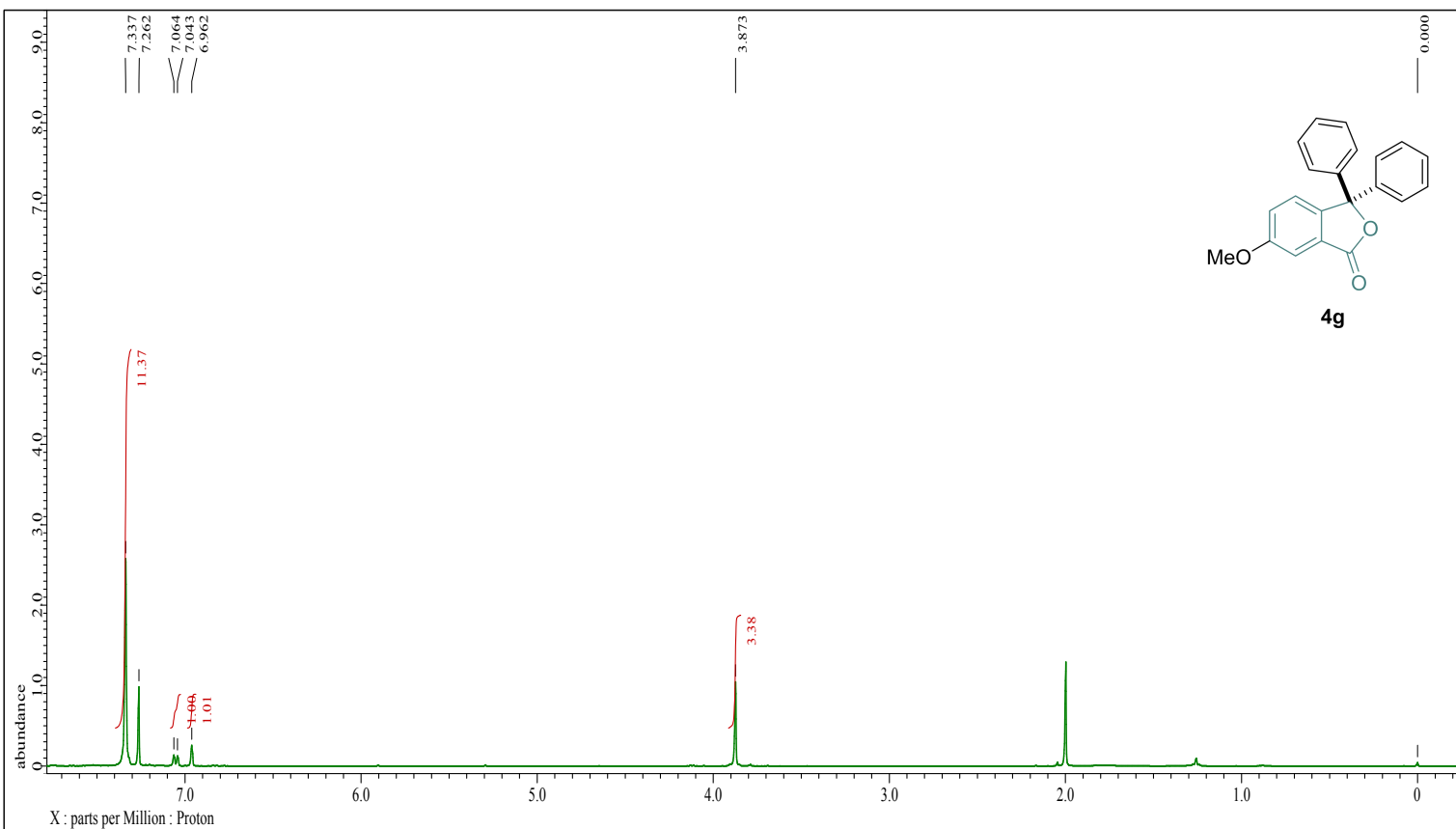
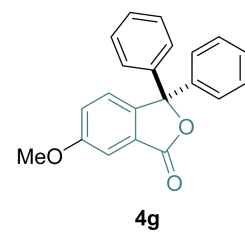


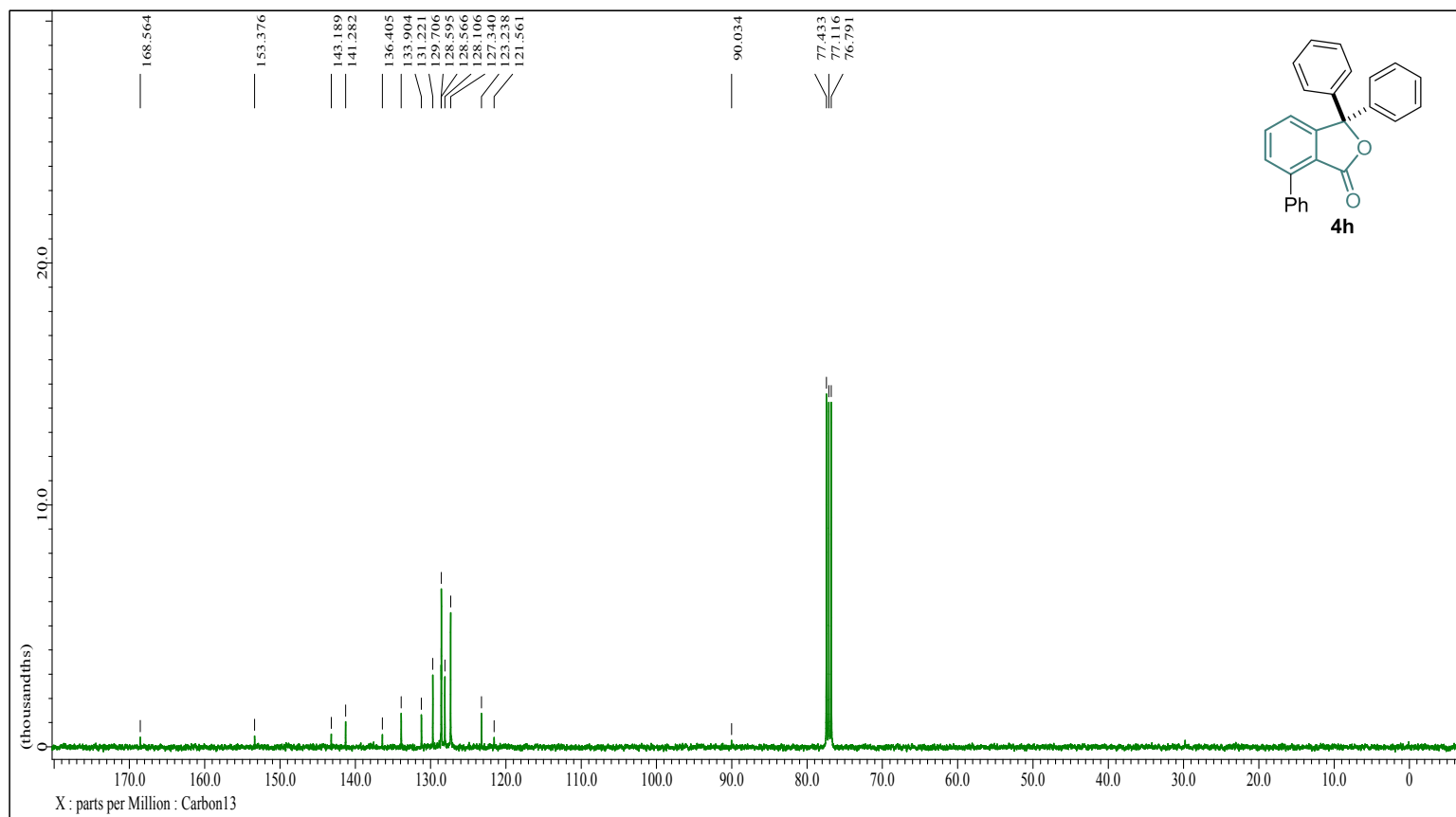
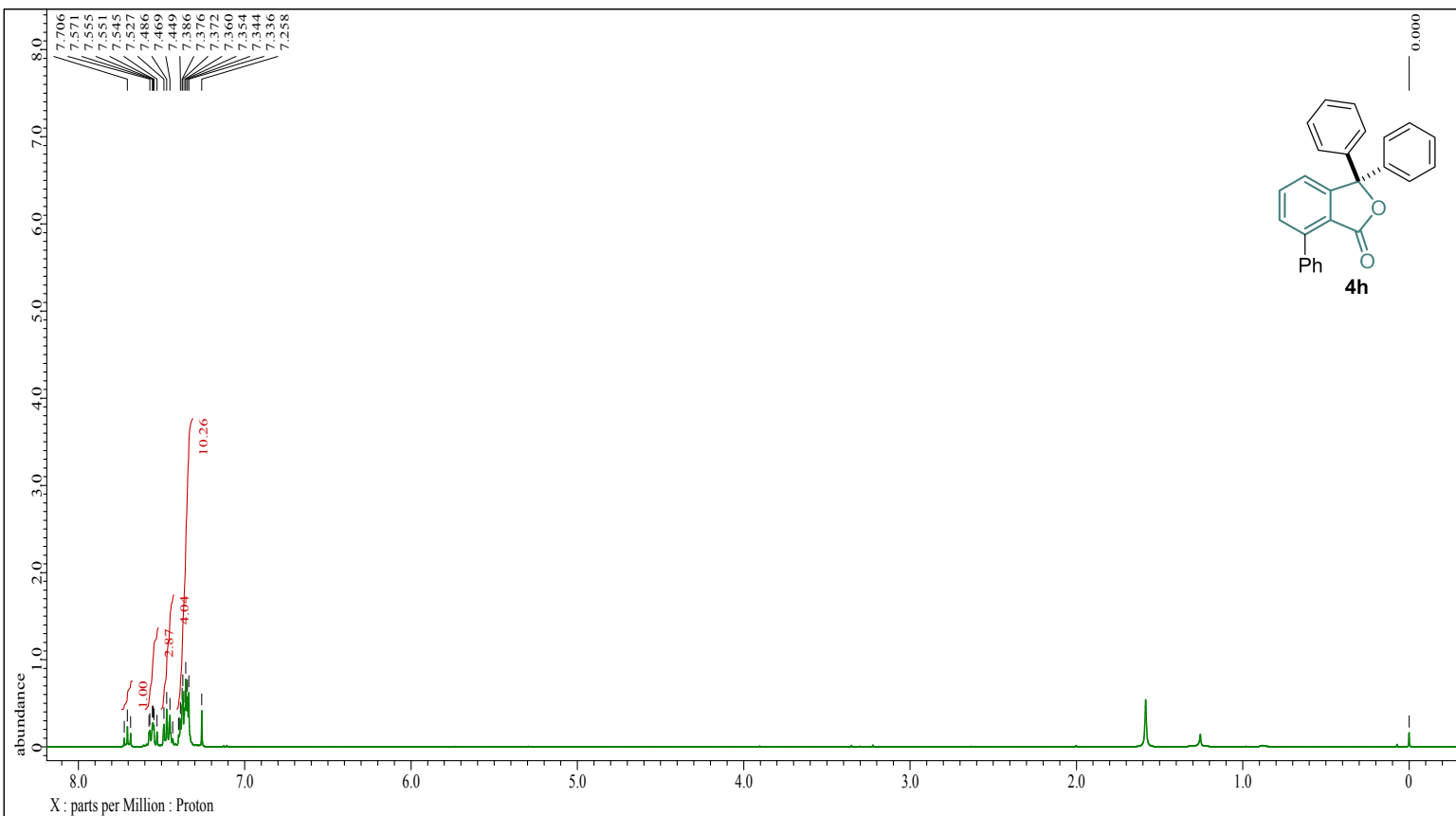


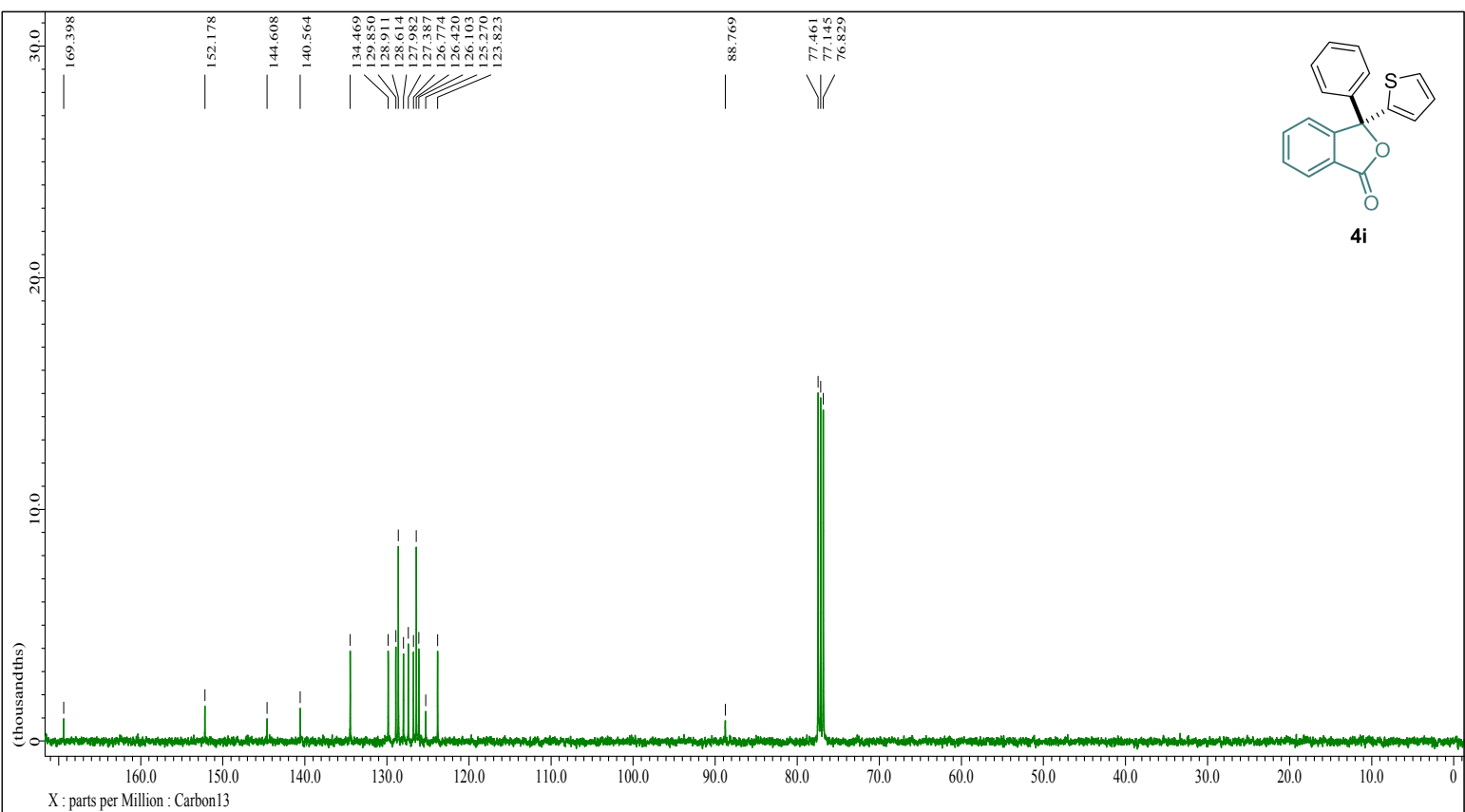
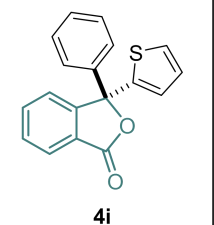
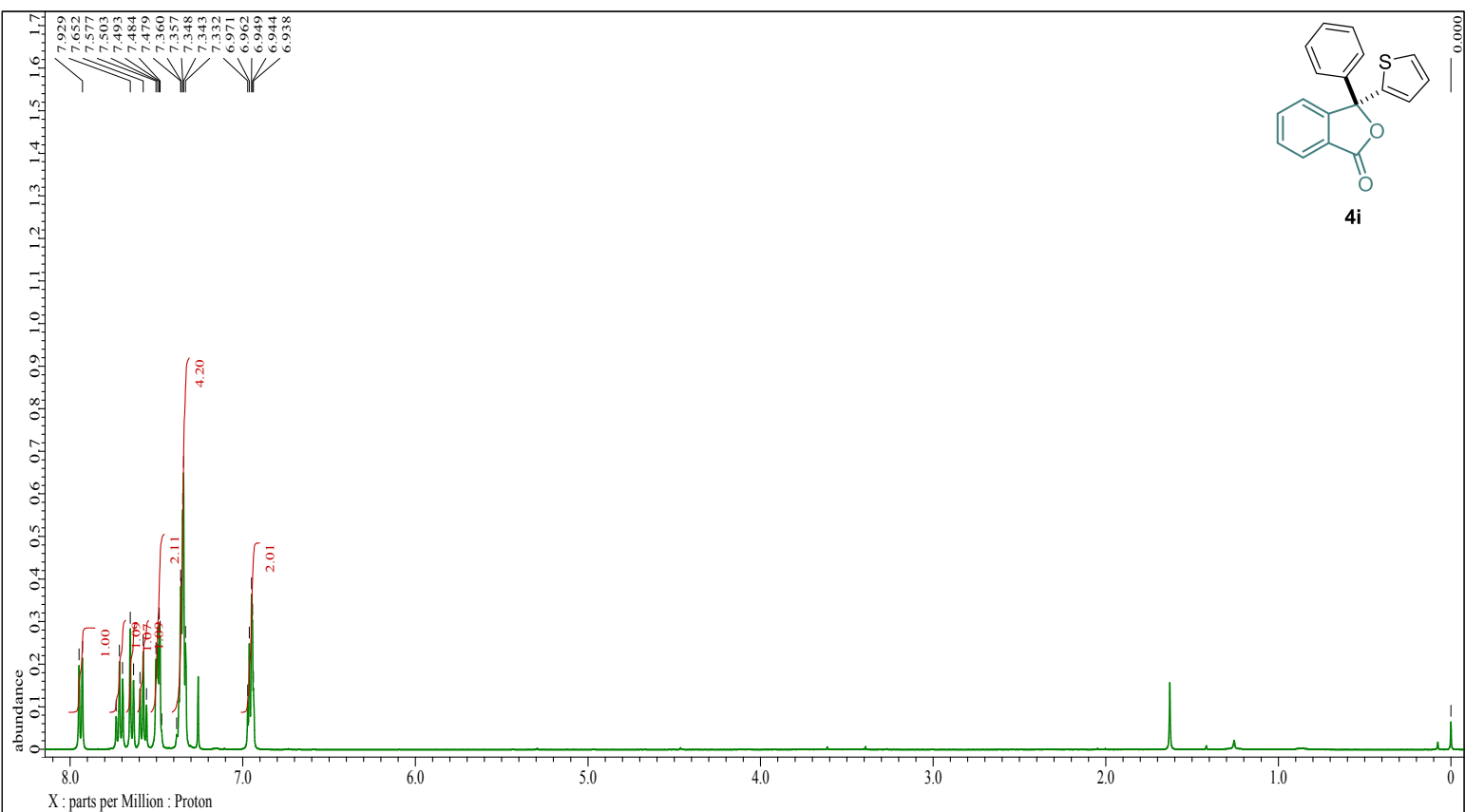
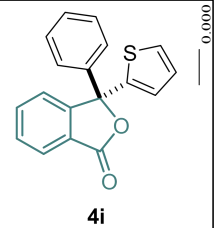


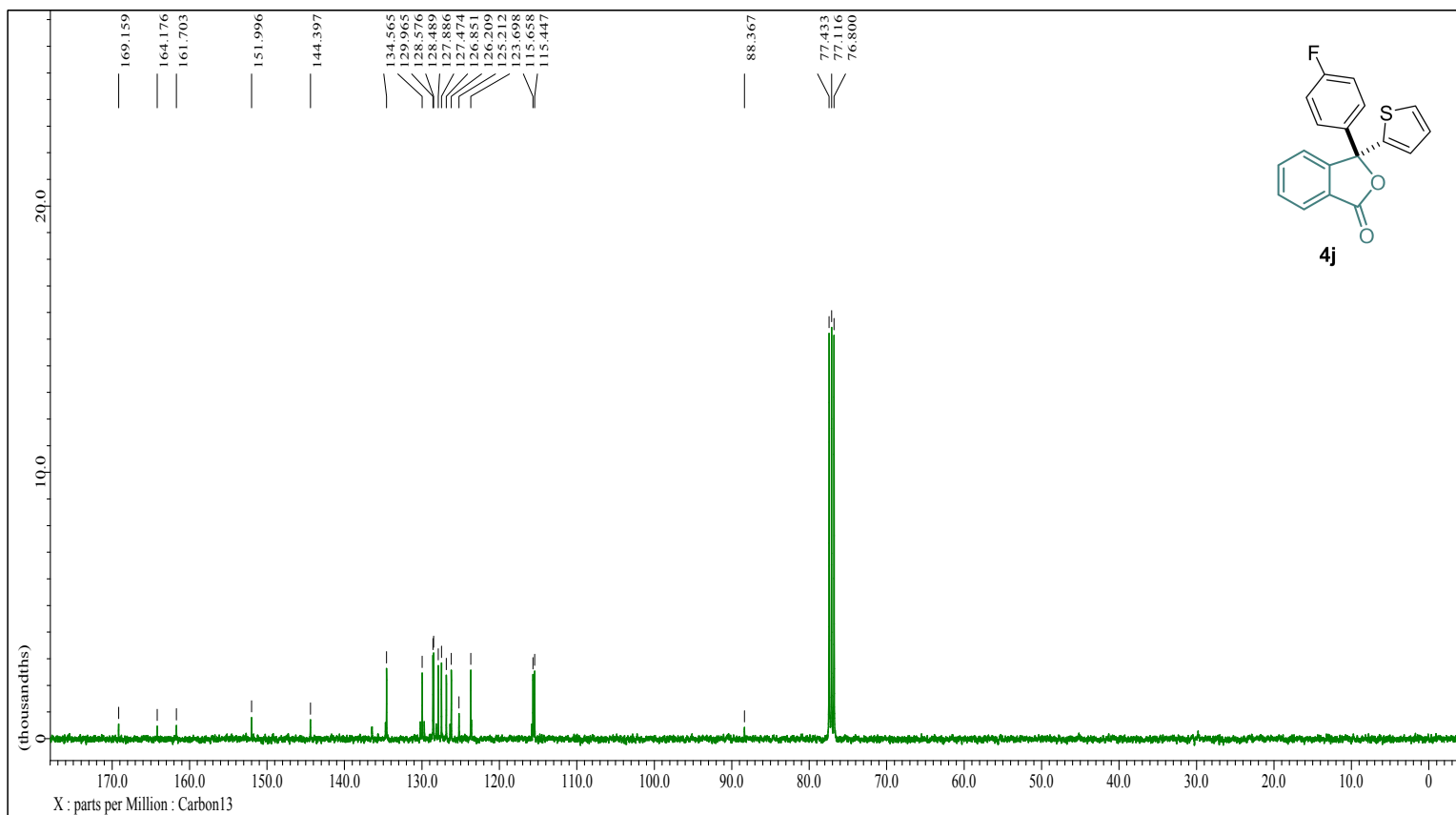
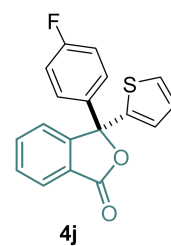
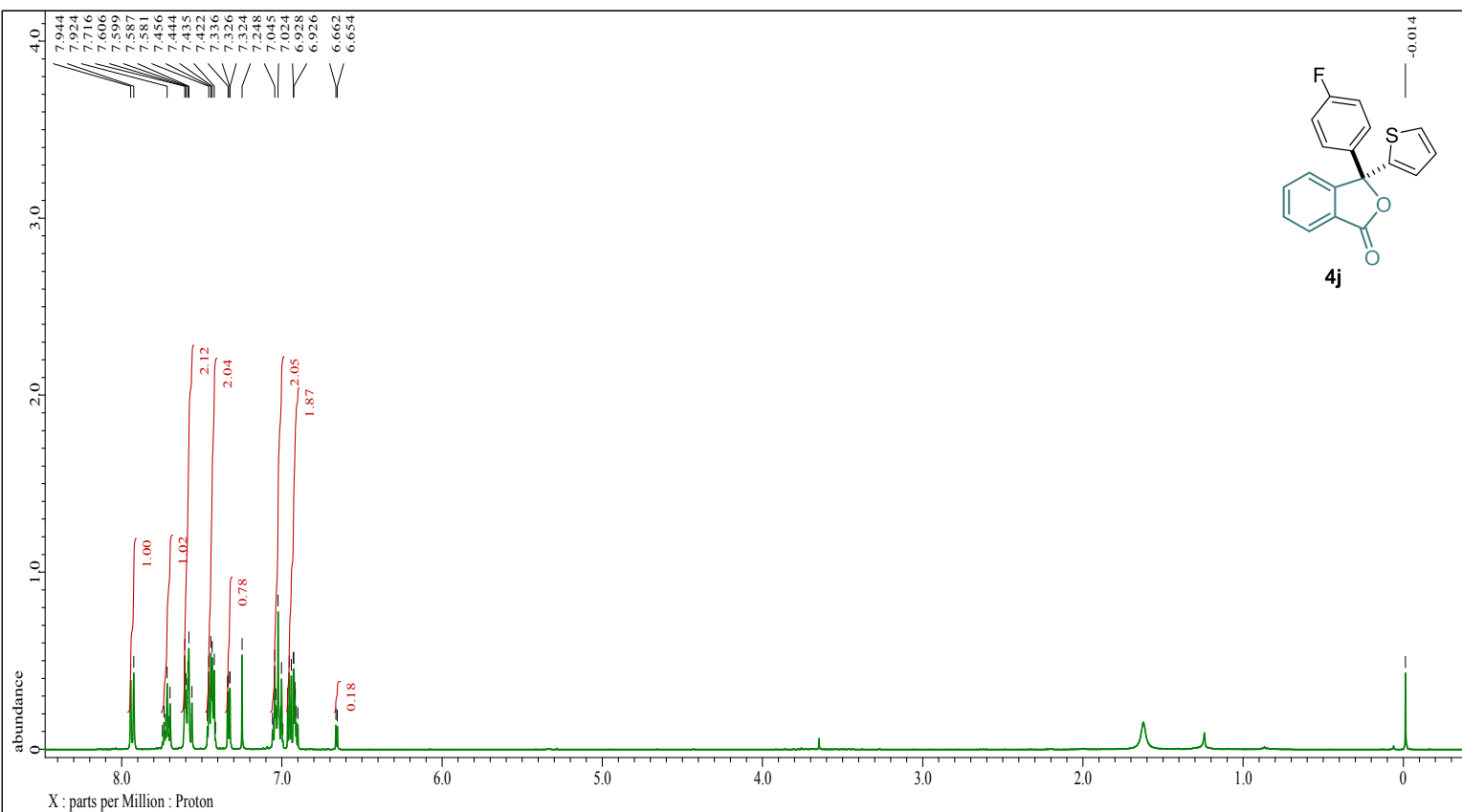
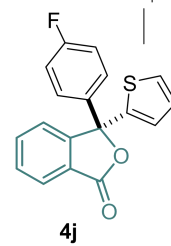


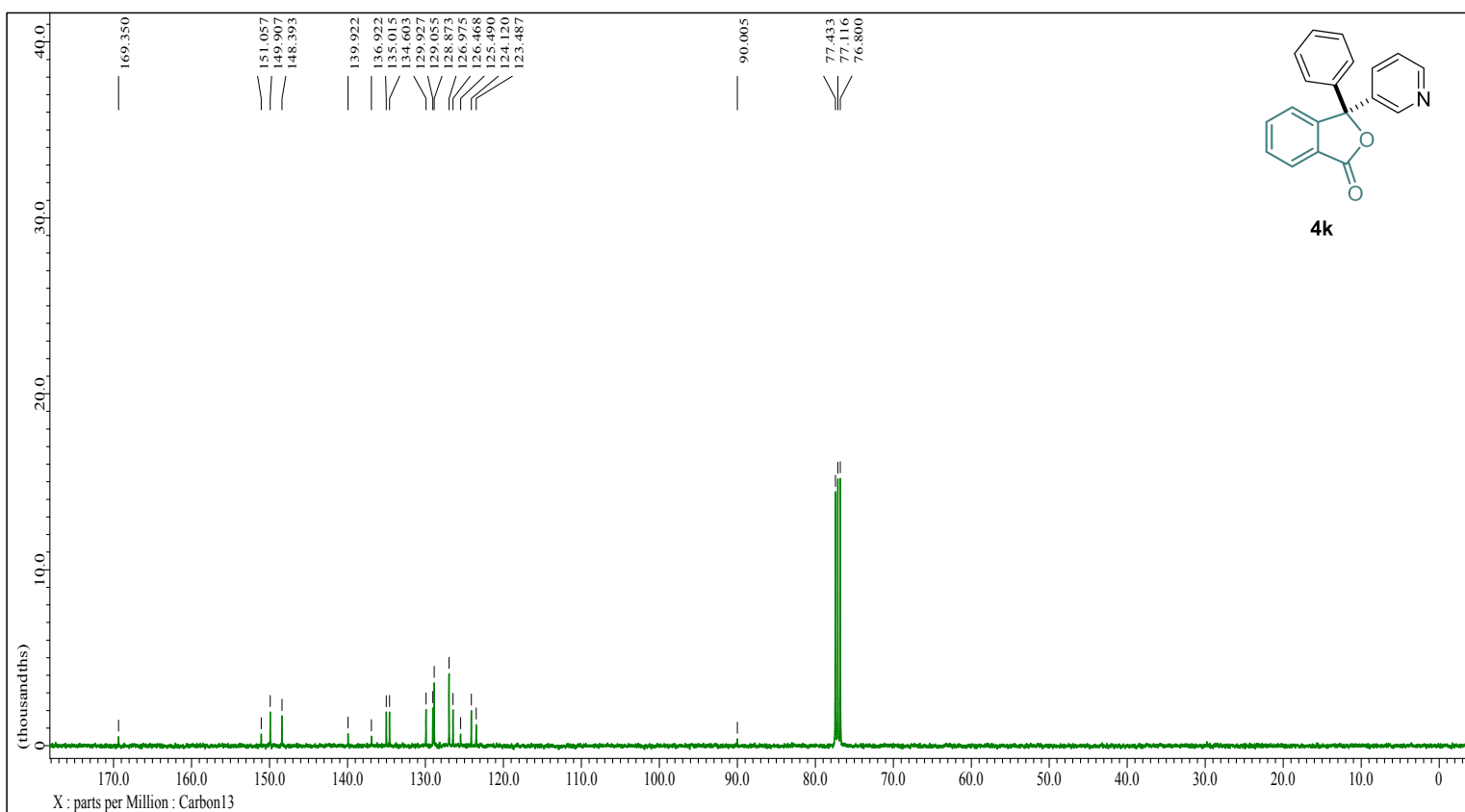
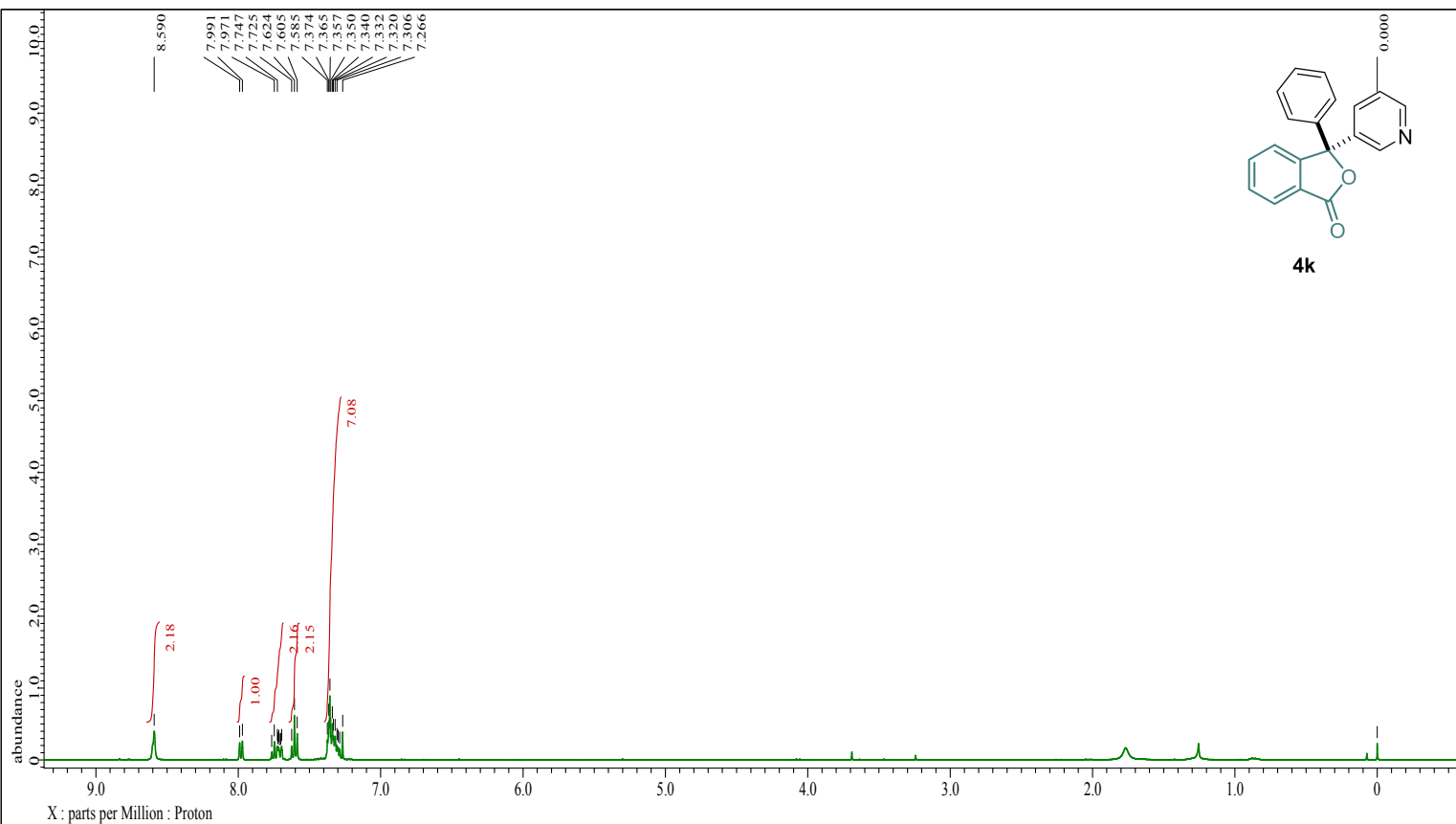
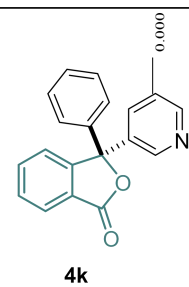


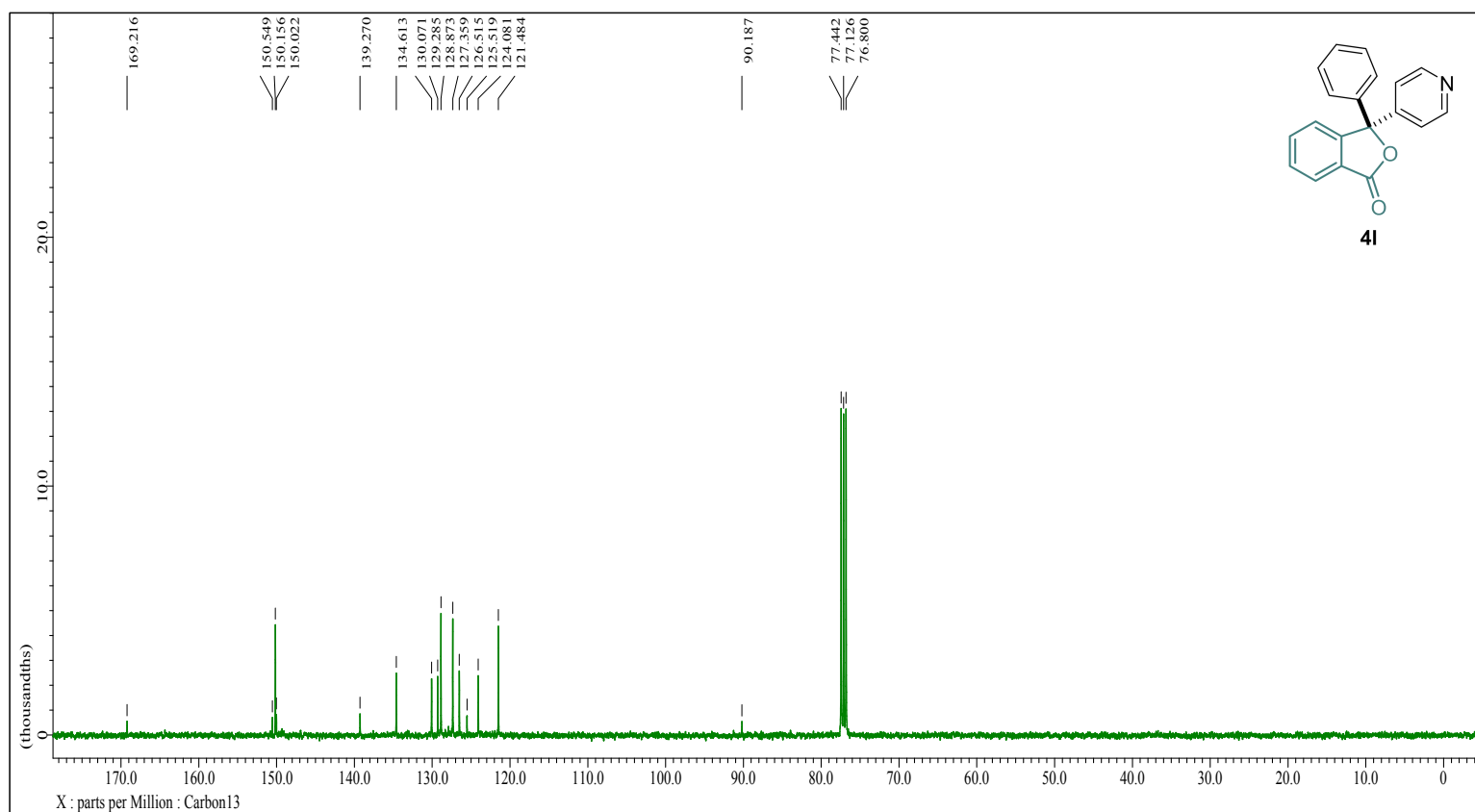
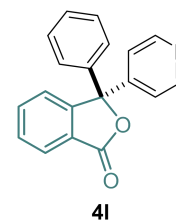
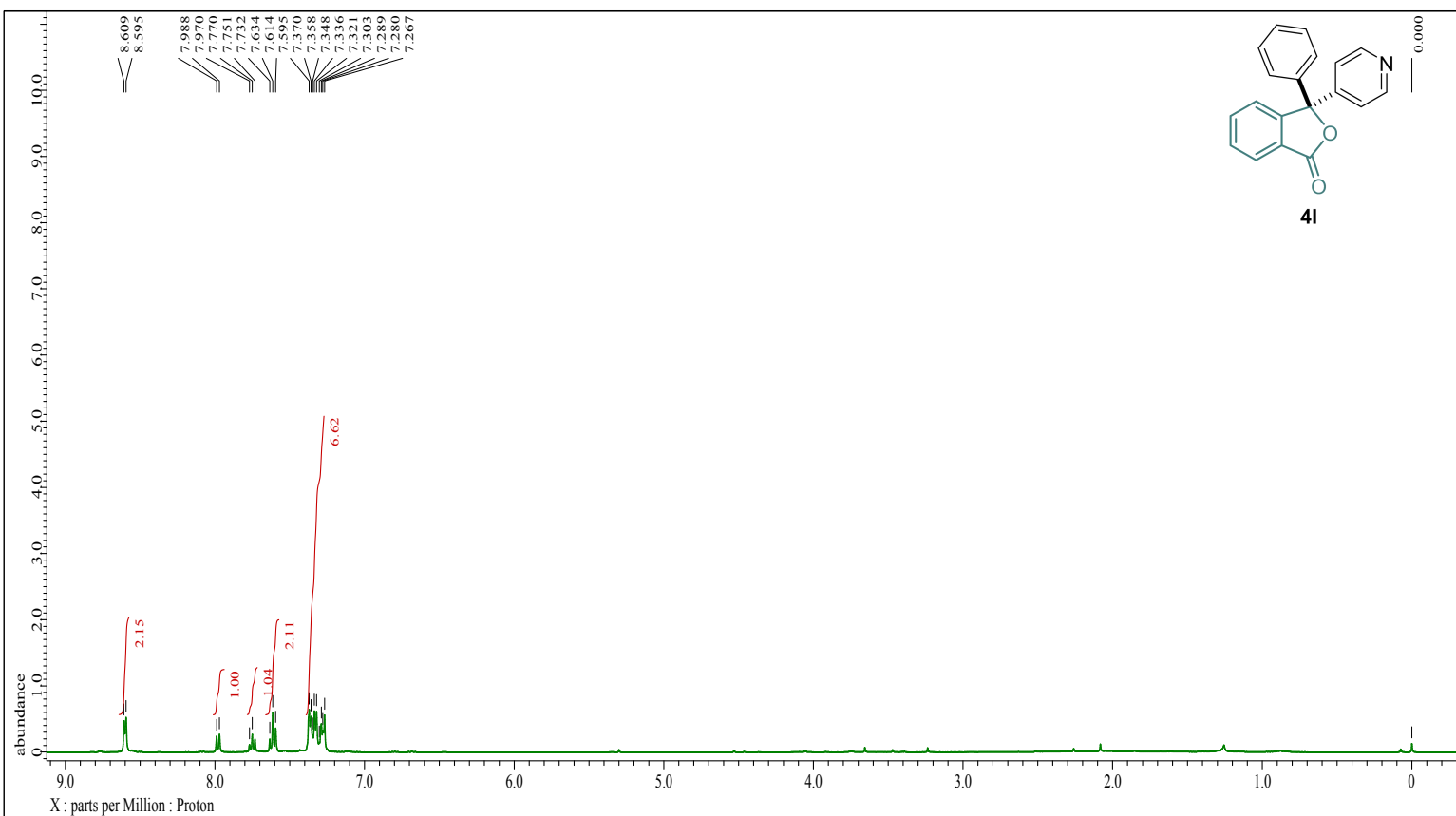
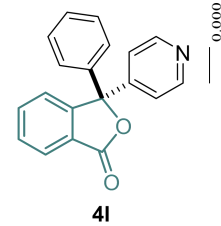


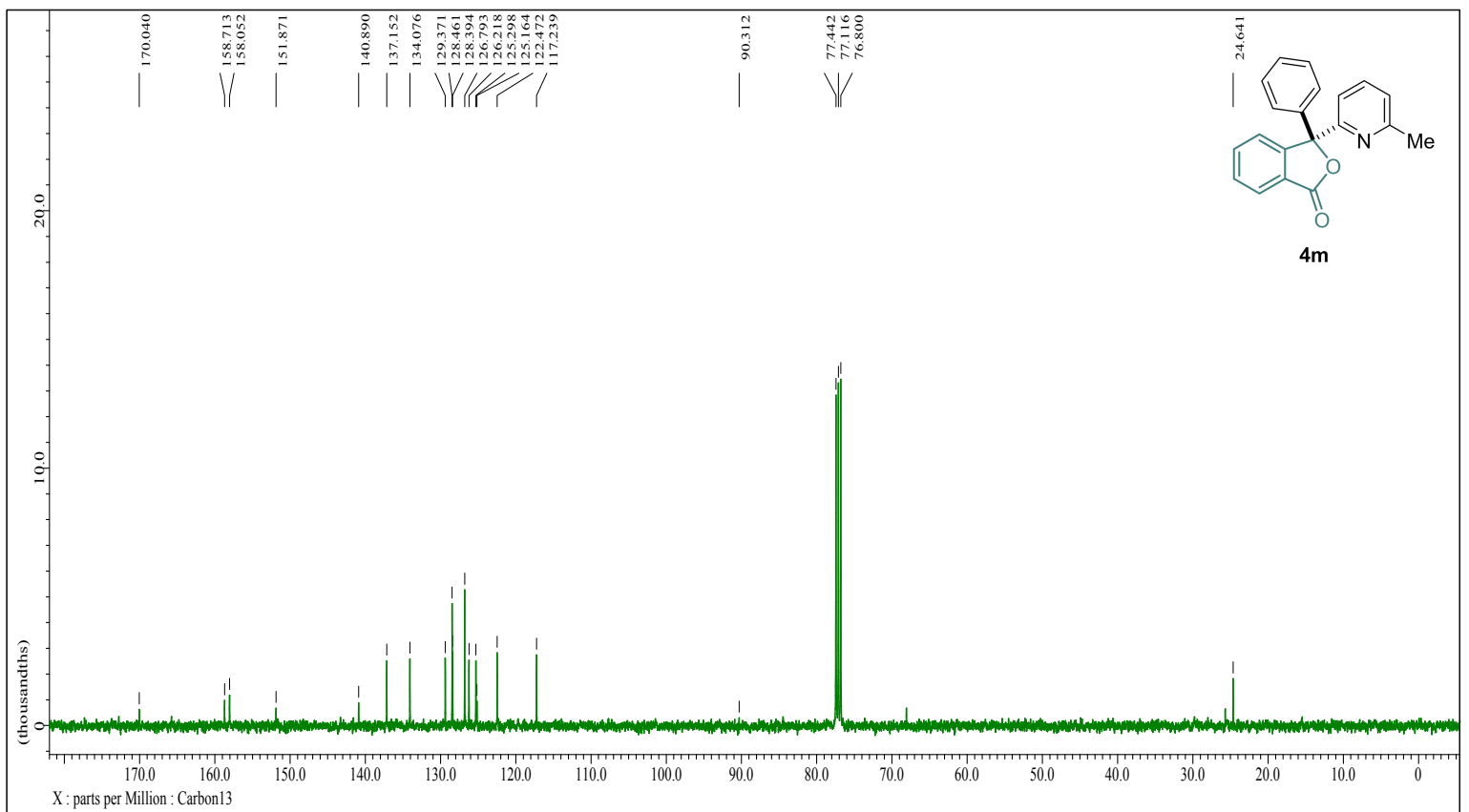
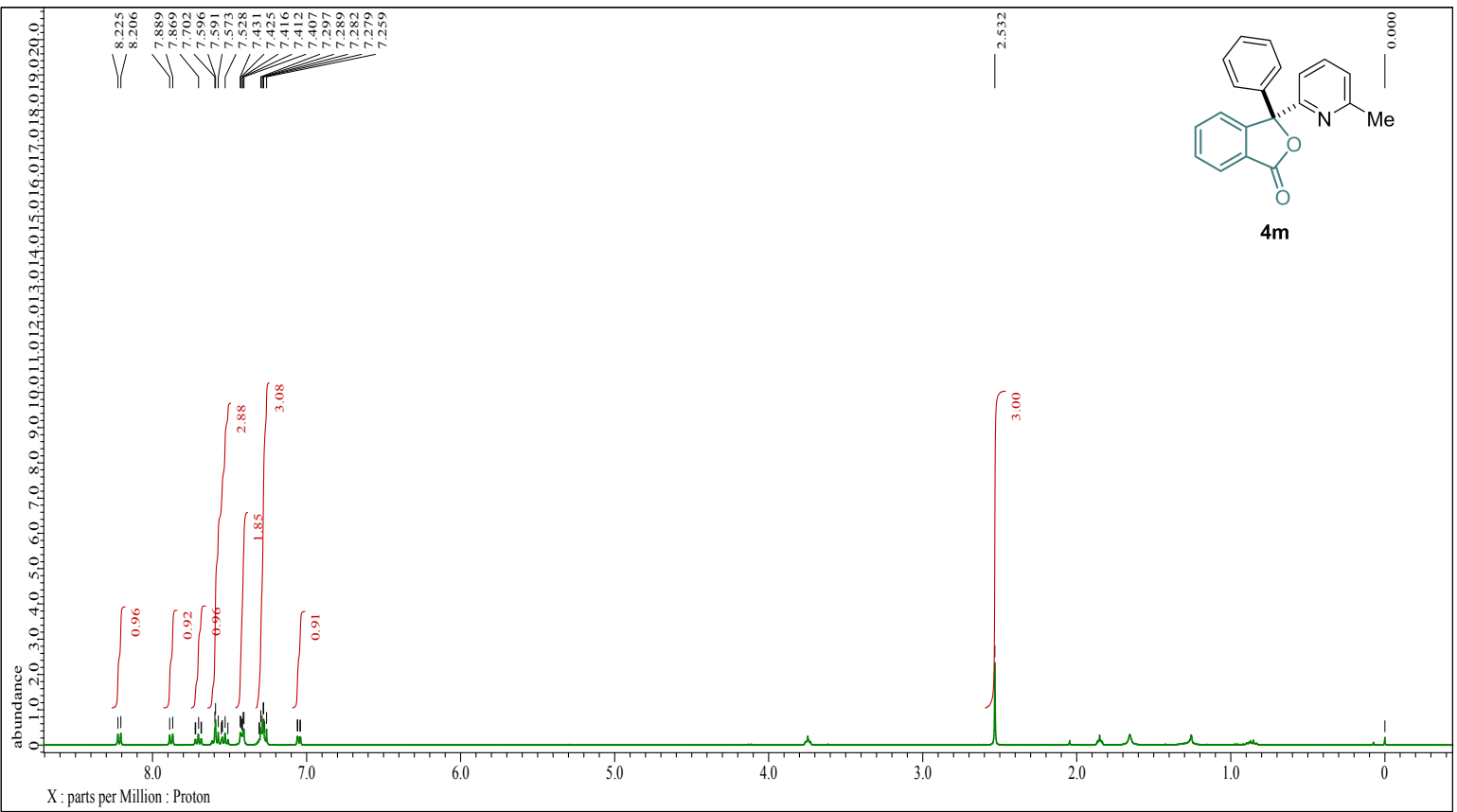


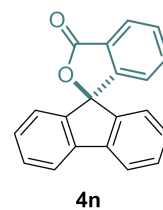




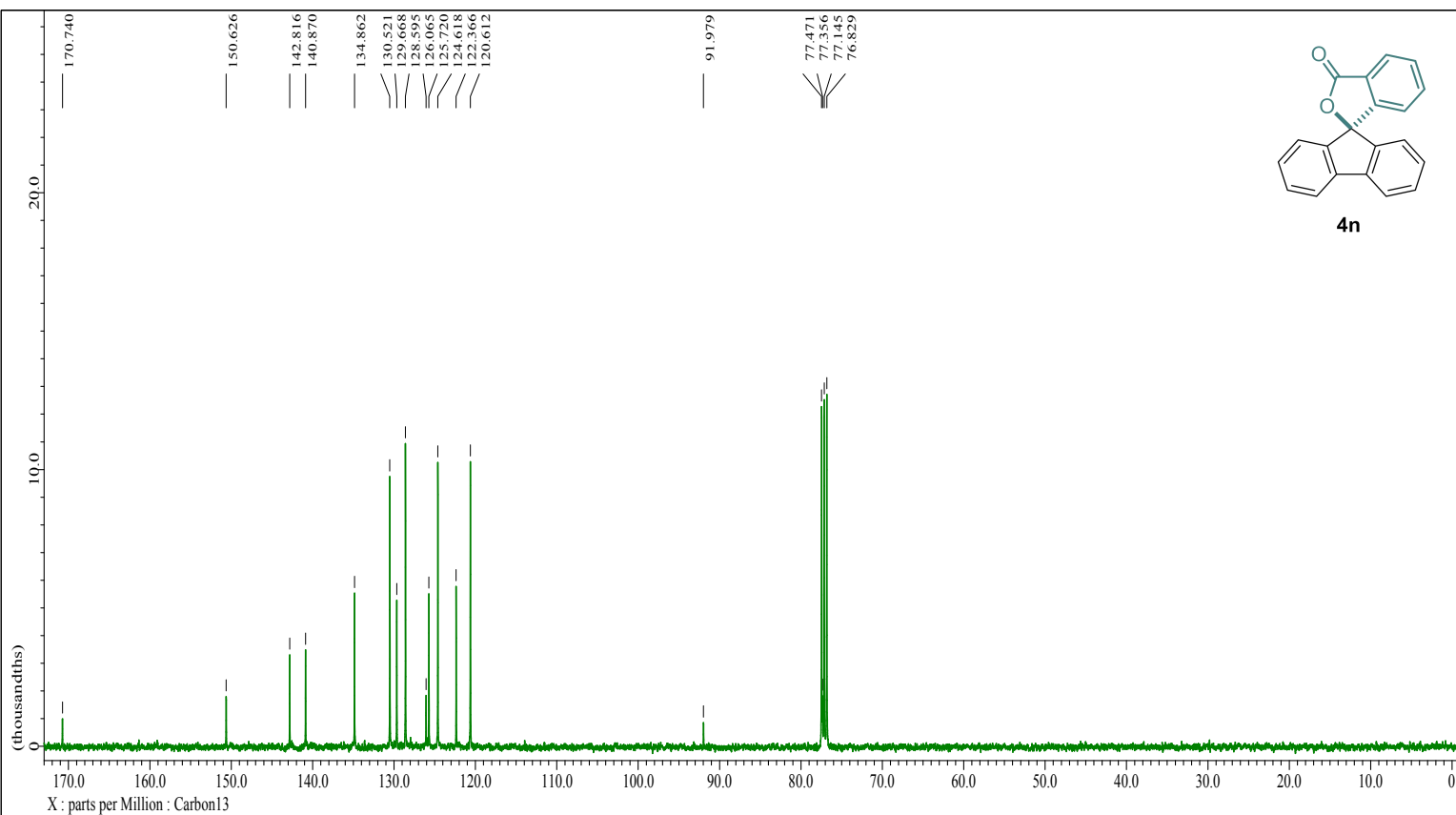
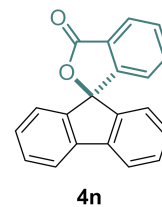
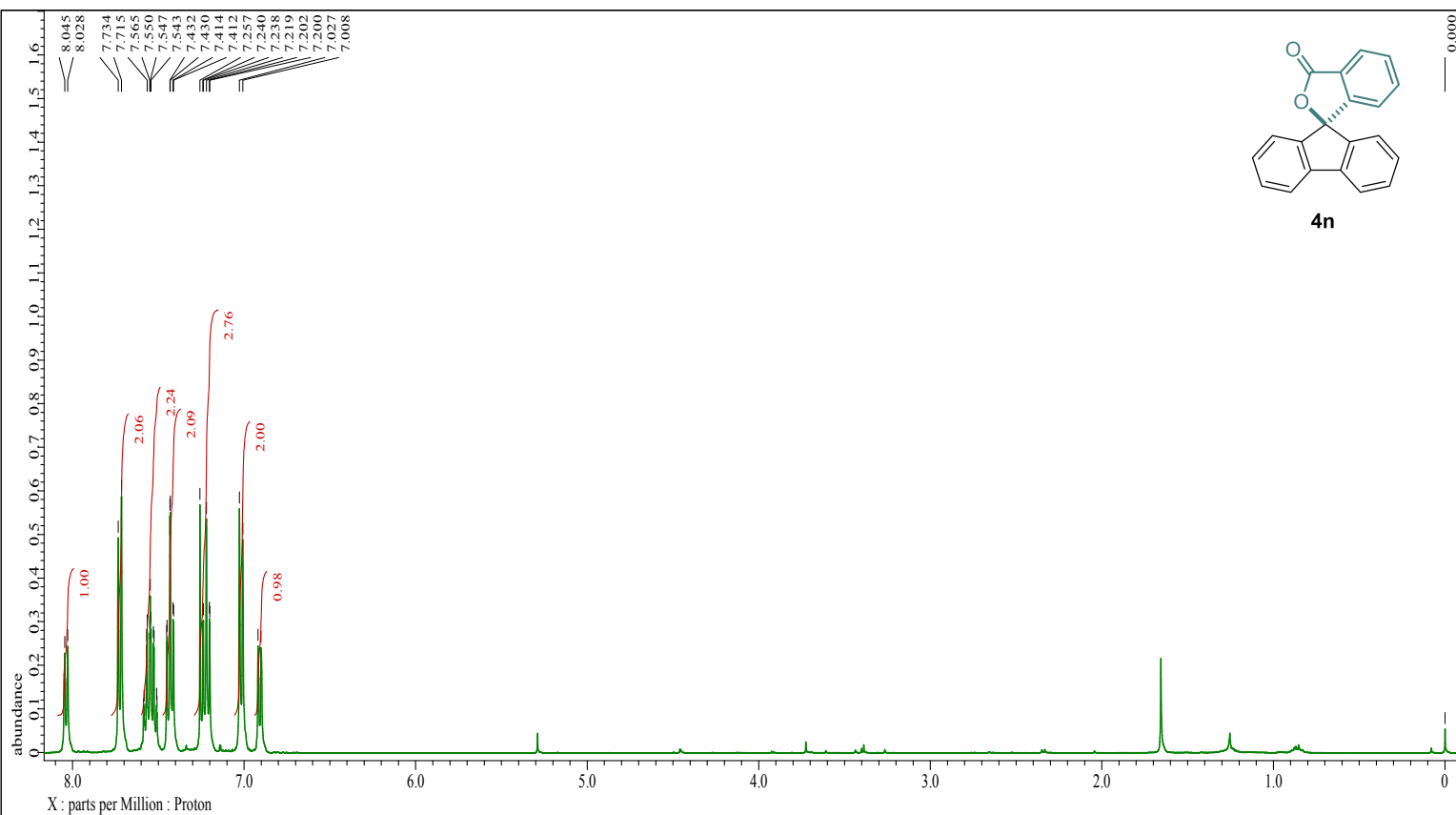


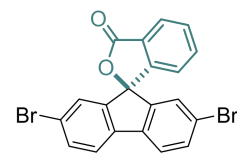






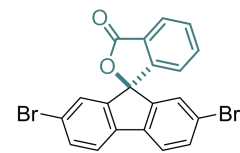
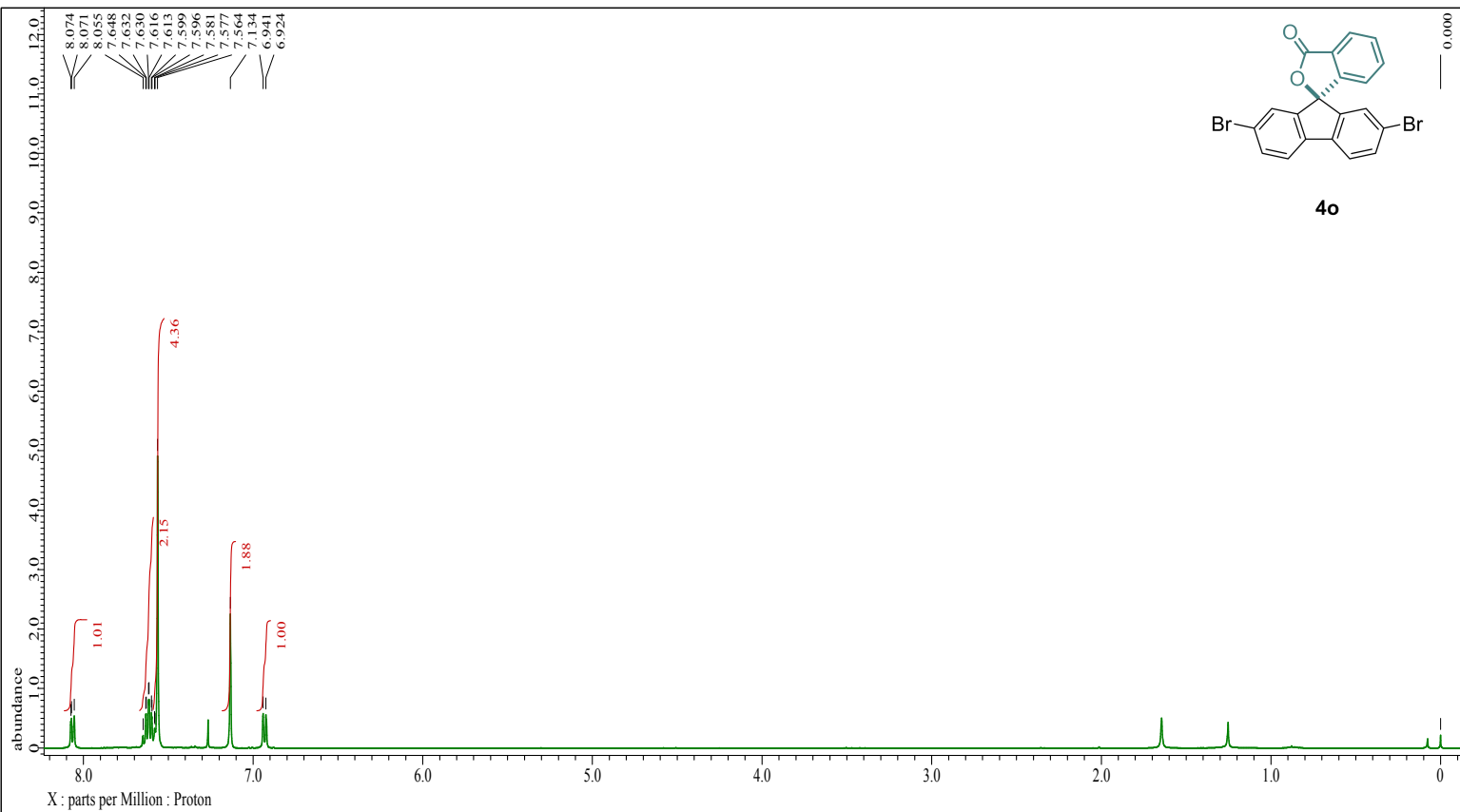
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