Electronic Supplementary Material (ESI) for Chemical Communications

Metal Free: Bu_4NI-Catalyzed C–H Oxidation for *t*-Butyl Perester and its Combination with the Kharasch–Sosnovsky Reaction

Wei Wei, Chao Zhang, Yuan Xu, and Xiaobing Wan*

Key Laboratory of Organic Synthesis of Jiangsu Province, College of Chemistry, Chemical Engineering and Materials Science , Soochow (Suzhou) University, Suzhou 215123, P. R. China

List of contents

.

General Information

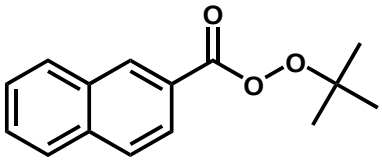
All manipulations were carried out under air atmosphere. Bu_4NI and TBHP (70% aqueous solution) were purchased from Sigma-Aldrich. Column chromatography was generally performed on silica gel (300-400 mesh) and reactions were monitored by thin layer chromatography (TLC) using UV light to visualize the course of the reactions. The ^1H NMR (400 MHz) and ^{13}C NMR (100 MHz) data were recorded on Varian 400 M spectrometers using $CDCl_3$ as solvent at room temperature. The chemical shifts (δ) are reported in ppm and coupling constants (*J*) in Hz. ^1H NMR spectra was recorded with tetramethylsilane (δ= 0.00 ppm) as internal reference; ^{13}C NMR spectra was recorded with $CDCl_3$ (δ = 77.00 ppm) as internal reference. IR, MS, and HRMS were performed by the State-authorized Analytical Center in Soochow University.

General procedures for products 3a-3w.

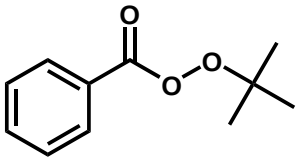
Aldehyde (0.5 mmol, 1.0 equiv), 2.0 mL H_2O, Bu_4NI (0.1 mmol, 20 mol %) and TBHP (3.0 equiv, 70% aqueous solution) were added to a tube under air. The reaction mixture was heated in an oil bath at 40 ^oC for 24 h. The reaction mixture was extracted with ethyl acetate. It was then removal of the organic solvent in vacuum and followed by flash silica gel column chromatographic purification afforded product with Petroleum/Ethyl acetate mixtures.

General procedures for products 4a-4p.

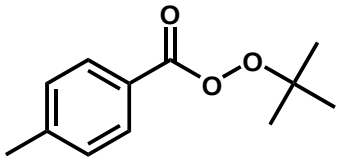
Aldehyde (0.5 mmol, 1.0 equiv), 5.0 mL CH_2Cl_2, Bu_4NI (0.1 mmol, 20 mol %) and TBHP (3 equiv, 70% aqueous solution) were added to a tube under air. The reaction mixture was heated in an oil bath at 40 ^oC for 24 h, then copper bromide (0.025 mmol, 5 mol %), olefin (5 mmol, 10.0 equiv) were added and stirred in an bath at 40 ^oC for 36 h. It was then removal of the organic solvent in vacuum and followed by flash silica gel column chromatographic purification afforded product with Petroleum/Ethyl acetate mixtures.



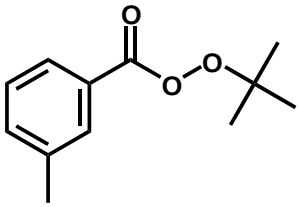
***t*-butyl naphthalene-2-carboperoxoate (3a).**[1] ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.46 (s, 9H), 7.51-7.59 (m, 2H), 7.85 (m, 2 H), 7.93 (m, 2H), 8.51 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 26.2, 83.9, 124.3, 124.7, 126.8, 127.7, 128.4, 128.4, 129.1, 130.6, 132.2, 135.4, 164.4; HRMS (EI) *m/z* $[M]^+$ Calcd for $C_{15}H_{16}O_3$: 244.1099, found: 244.1096; IR (KBr, cm^{-1}): ν 1743.



***t*-butyl benzoperoxoate (3b).**[2] ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.42 (s, 9H), 7.46 (m, 2H), 7.59 (m, 1H), 7.96 (d, *J* = 7.7 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 26.1, 83.9, 127.5, 128.5, 129.0, 133.3, 164.3; HRMS (EI) *m/z* $[M]^+$ Calcd for $C_{11}H_{14}O_3$: 194.0943, found: 194.0940; IR (KBr, cm^{-1}): ν 1757.

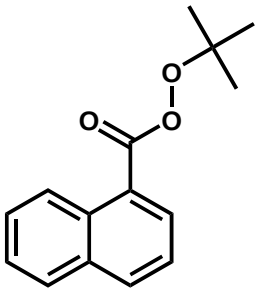


***t*-butyl 4-methylbenzoperoxoate (3c).** ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.41 (s, 9H), 2.41 (s, 3H), 7.25 (d, *J* = 8.1 Hz, 2H), 7.85 (d, *J* = 8.1 Hz, 2H); ^{13} C NMR ($CDCl_3$, 100 MHz): δ 21.6, 26.1, 83.7, 124.7, 129.0, 129.2, 144.1, 164.4; HRMS (EI) *m/z* $[M]^+$ Calcd for $C_{12}H_{16}O_3$: 208.1099, found: 208.1100; IR (KBr, cm^{-1}): ν 1757.

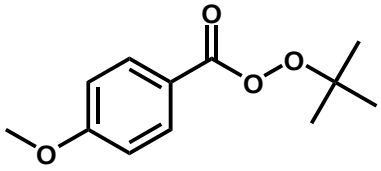


***t*-butyl 3-methylbenzoperoxoate (3d).** ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.42 (s, 9H), 2.40 (s, 3H) 7.32-7.40 (m, 2H) 7. 38-7.40 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 21.2, 26.2, 83.8, 126.1, 127.5, 128.4, 129.5, 134.0, 138.4, 164.5; HRMS (EI) *m/z*

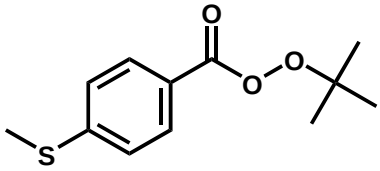
$[M]^+$ Calcd for $C_{12}H_{16}O_3$: 208.1099, found: 208.1104; IR (KBr, cm^{-1}): ν 1758.



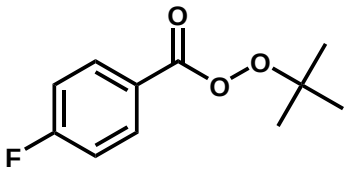
***t*-butyl naphthalene-1-carboperoxoate (3e).**[1] ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.46 (s, 9H), 7.43-7.56 (m, 2H), 7.62 (t, *J* = 7.7 Hz, 1H), 7.87 (d, *J* = 8.2 Hz, 2H), 7.97-8.02 (m, 2H), 8.65 (d, *J* = 8.6 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 26.2, 83.8, 124.3, 125.0, 125.2, 126.4, 127.9, 128.5, 128.8, 130.8, 133.3, 133.6, 165.5; HRMS (EI) *m/z* $[M]^+$ Calcd for $C_{15}H_{16}O_3$: 244.1099, found: 244.1100; IR (KBr, cm^{-1}): ν 1756.



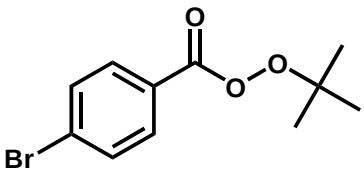
***t*-butyl 4-methoxybenzoperoxoate (3f).**[1] ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.41 (s, 9H), 3.86 (s, 3H), 6.94 (d, *J* = 9.0 Hz, 2H), 7.92 (d, *J* = 9.0 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 26.2, 52.4, 83.7, 113.8, 119.7, 131.1, 163.6, 164.2; HRMS (EI) *m/z* $[M]^+$ Calcd for $C_{12}H_{16}O_4$: 224.1049, found: 224.1050; IR (KBr, cm^{-1}): ν 1748.



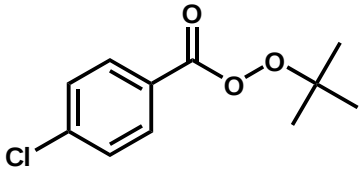
***t*-butyl 4-(methylthio)benzoperoxoate (3g).** ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.41 (s, 9H), 2.51 (s, 3H), 7.26 (d, *J* = 8.4 Hz, 2H), 7.85 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 14.6, 26.1, 83.8, 123.2, 124.9, 129.2, 146.2, 164.1; HRMS (EI) *m/z* $[M]^+$ Calcd for $C_{12}H_{16}O_3S$: 240.0820, found: 240.0817; IR (KBr, cm^{-1}): ν 1748.



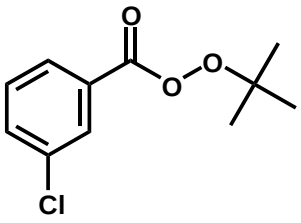
***t*-butyl 4-fluorobenzoperoxoate (3h).** ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.42 (s, 9H), 7.15 (m, 2H), 7.97-8.01 (m, 2H,); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 26.1, 84.0, 115.7, 115.9, 123.8, 131.6, 131.7, 163.4, 164.5, 167.0; HRMS (EI) *m/z* $[M]^+$ Calcd for $C_{11}H_{13}FO_6$: 212.0849, found: 212.0851; IR (KBr, cm^{-1}): ν 1760.



***t*-butyl 4-bromobenzoperoxoate (3i).** ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.41 (s, 9H), 7.61 (d, *J* = 8.3 Hz, 2H), 7.82 (d, *J* = 8.3 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 26.2, 84.1, 126.5, 128.5, 130.6, 132.0, 163.7; HRMS (EI) *m/z* $[M]^+$ Calcd for $C_{11}H_{13}BrO_3$: 272.0048, found: 272.0042; IR (KBr, cm^{-1}): ν 1766.

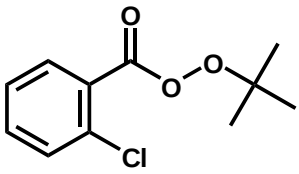


***t*-butyl 4-chlorobenzoperoxoate (3j).**[3] ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.42 (s, 9H), 7.44 (d, *J* = 8.4 Hz, 2H), 7.90 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 26.2, 84.1, 126.0, 129.0, 130.4, 139.8, 163.5; HRMS (EI) *m/z* $[M]^+$ Calcd for $C_{11}H_{13}ClO_3$: 228.0553, found: 228.0553; IR (KBr, cm^{-1}): ν 1753.

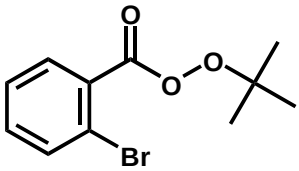


***t*-butyl 3-chlorobenzoperoxoate (3k).**[1] ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.42 (s,

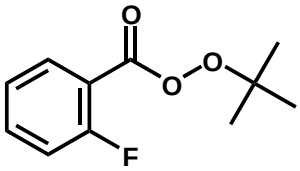
9H), 7.40-7.44 (m, 1H) 7. 56-7.58 (m, 1H), 7.84-7.92 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 26.2, 84.3, 127.2, 129.1, 129.3, 129.9, 133.4, 134.7, 163.2; HRMS (EI) *m/z* $[M]^+$ Calcd for $C_{11}H_{13}ClO_3$: 228.0553, found: 228.0555; IR (KBr, cm^{-1}): ν 1761.



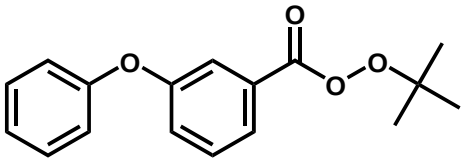
***t*-butyl 2-chlorobenzoperoxoate (3l).**[3] ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.42 (s, 9H), 7.14-7.19 (m, 1H) 7. 32-7.36 (m, 1H), 7.45-7.46 (m, 2H), 7.70 (d, *J* = 7.6 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 26.2, 84.1, 126.7, 128.3, 130.7, 130.9, 132.8, 163.9; HRMS (EI) *m/z* $[M]^+$ Calcd for $C_{11}H_{13}ClO_3$: 228.0553, found: 228.0555; IR (KBr, cm^{-1}): ν 1770.



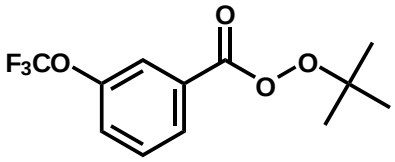
***t*-butyl 2-bromobenzoperoxoate (3m).** ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.43 (s, 9H), 7.34-7.41 (m, 2H) 7. 62-7.67 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 26.2, 84.2, 120.8, 127.2, 130.6, 130.7, 132.8, 133.9, 164.4; HRMS (EI) *m/z* $[M]^+$ Calcd for $C_{11}H_{13}BrO_3$: 272.0048, found: 272.0047; IR (KBr, cm^{-1}): ν 1770.



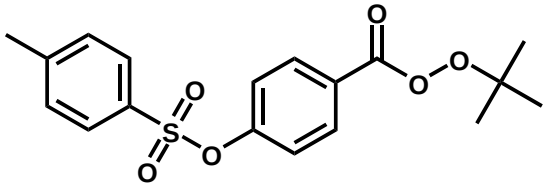
***t*-butyl 2-fluorobenzoperoxoate (3n).** ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.42 (s, 9H), 7.14-7.19 (m, 1H) 7. 23-7.27 (m, 1H), 7.54-7.59 (m, 1H), 7.89-7.93 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 26.0, 84.0, 116.0, 116.2, 124.3, 131.9, 134.8, 134.9, 159.7, 162.3, 162.7; HRMS (EI) *m/z* $[M]^+$ Calcd for $C_{11}H_{13}FO_3$: 212.0849, found: 212.0852; IR (KBr, cm^{-1}): ν 1757.



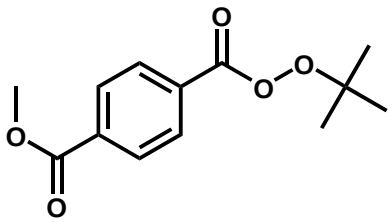
***t*-butyl 3-phenoxybenzoperoxoate (3o).** ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.39 (s, 9H), 7.02 (d, *J* = 7.7 Hz, 2H) 7.12-7.21 (m, 2H) 7.34-7.42 (m, 3H) 7.59 (s, 1H) 7.68 (d, *J* = 7.7 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 26.1, 84.0, 118.9, 119.1, 123.2, 123.5, 129.2, 129.9, 130.0, 156.2, 157.5, 163.7; HRMS (EI) *m/z* $[M]^+$ Calcd for $C_{17}H_{18}O_4$:286.1205, found: 286.1205; IR (KBr, cm^{-1}): ν 1760.



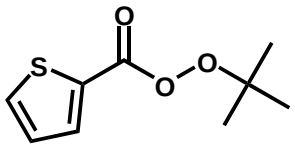
***t*-butyl 3-(trifluoromethoxy)benzoperoxoate (3p).** Yield: 58%; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.43 (s, 9H), 7.45-7.55 (m, 2H) 7.80 (s, 1H) 7. 90-7.92 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 26.1, 84.4, 119.0, 121.6, 125.8, 127.4, 129.6, 130.2, 149.2, 163.0; HRMS (ESI) *m/z* $[M+NH_4]^+$ Calcd for $C_{12}H_{17}F_3NO_4$: 296.1100, found: 296.1104; IR (KBr, cm^{-1}): ν 1765.



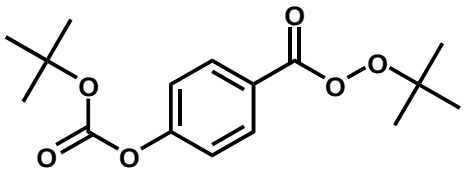
***t*-butyl 4-(tosyloxy)benzoperoxoate (3q).** ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.40 (s, 9H), 2.46 (s, 3H), 7.10 (d, *J* = 8.7 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H) , 7.72 (d, *J* = 8.2 Hz, 2H), 7.91 (d, *J* = 8.7 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 21.7, 26.1, 84.2, 122.6, 126.3, 128.4, 129.9, 130.8, 131.9, 145.8, 153.1, 163.2; HRMS (ESI) *m/z* $[M+NH_4]^+$ Calcd for $C_{18}H_{24}NO_6S$: 382.1319, found: 382.1317; IR (KBr, cm^{-1}): ν 1753.



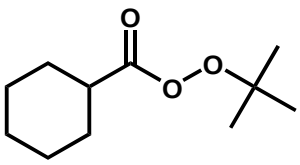
methyl 4-(*t*-butylperoxycarbonyl)benzoate (3r). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.43 (s, 9H), 3.96 (s, 3H), 8.02 (d, *J* = 8.3 Hz, 2H), 8.13 (d, *J* = 8.3 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 26.2, 52.5, 84.2, 129.0, 129.7, 131.4, 134.2, 163.5, 165.9; HRMS (EI) *m/z* $[M]^+$ Calcd for $C_{13}H_{16}O_5$:252.0998, found: 252.0999; IR (KBr, cm^{-1}): ν 1754, 1716.



***t*-butyl thiophene-2-carboperoxoate (3s).** ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.40 (s, 9H),7.13-7.15 (m, 1H), 7.61-7.63 (m, 1H), 7.80-7.82 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 26.1, 84.2, 127.8, 129.2, 132.8, 133.6, 160.3; HRMS (EI) *m/z* $[M]^+$ Calcd for $C_9H_{12}O_3S$: 200.0507, found: 200.0504; IR (KBr, cm^{-1}): ν 1747.

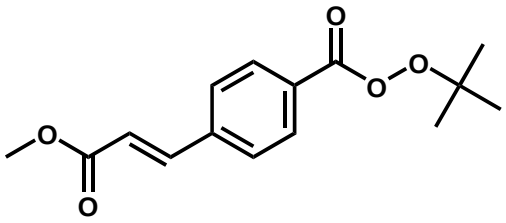


***t*-butyl 4-(*t*-butoxycarbonyloxy)benzoperoxoate (3t).** Yield: 44%; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.41 (s, 9H), 1.57 (s, 9H), 7.28 (d, *J* = 8.7 Hz, 2H), 7.99 (d, *J* = 8.7 Hz, 2H); ^{13}C NMR ($CDCl_3$,100 MHz): δ 26.2, 27.6, 84.0, 84.2, 121.5, 124.9, 130.7, 150.9, 154.8, 163.6; HRMS (EI) *m/z* $[M]^+$ Calcd for $C_{16}H_{22}O_6$: 310.1416, found: 310.1422; IR (KBr, cm^{-1}): ν 1754.

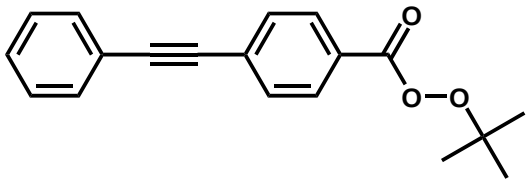


***t*-butyl cyclohexanecarboperoxoate (3u).** ^{1}H NMR ($CDCl_3$, 400 MHz): δ

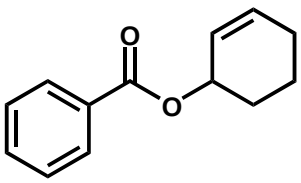
1.26-1.29 (m, 2H), 1.32 (s, 9H), 1.48-1.92 (m, 8H), 2.33-2.41 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 25.3, 25.5, 26.1, 29.0, 41.1, 83.2, 173.1; HRMS (EI) *m/z* $[M+NH_4]^+$ Calcd for $C_{11}H_{24}NO_3$: 218.1751, found: 218.1743; IR (KBr, cm^{-1}): ν 1772.



(E)-methyl 3-(4-(*t*-butylperoxycarbonyl)phenyl)acrylate (3v). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.43 (s, 9H), 3.83 (s,3H), 6.53 (d, *J* = 16.1 Hz, 1H), 7.61 (d, *J* = 8.3 Hz, 2H), 7.71 (d, *J* = 16.1 Hz, 1H), 7.98 (d, *J* = 8.3 Hz, 2H); ^{13}C NMR ($CDCl_3$,100 MHz): δ 26.1, 51.8, 84.1, 120.5, 128.0, 128.7, 129.6, 138.9, 143.0, 163.7, 166.7; HRMS (EI) *m/z* $[M]^+$ Calcd for $C_{15}H_{18}O_5$: 278.1154, found: 278.1154; IR (KBr, cm^{-1}): ν 1754.

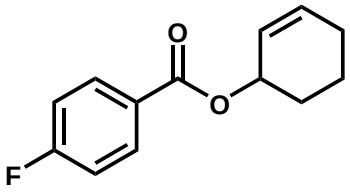


***t*-butyl 4-(phenylethynyl)benzoperoxoate (3w).** ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.41 (s, 9H), 7.34-7.35 (m, 3H), 7.53-7.55 (m, 2H), 7.59 (d, *J* = 8.2 Hz, 2H), 7.92 (d, *J* = 8.2 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 26.0, 83.9, 88.2, 92.7 122.34 126.7, 128.3, 128.4 128.7 128.9 131.5, 163.7; HRMS (EI) *m/z* $[M]^+$ Calcd for $C_{19}H_{18}O_3$: 294.1256, found: 294.1224; IR (KBr, cm^{-1}): *v* 1750.

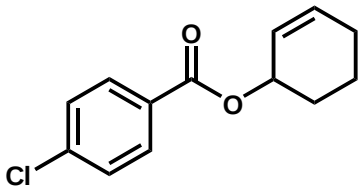


cyclohex-2-enyl benzoate (4a).[4] ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.67-1.75 (m, 1H), 1.82-1.91 (m, 2H), 1.94-2.07 (m, 2H), 2.11-2.17 (m, 1H), 5.51 (m, 1H), 5.82-5.85 (m, 1H), 5.98-6.03 (m, 1H), 7.41-7.45 (m, 2H), 7.52-7.56 (m, 1H),

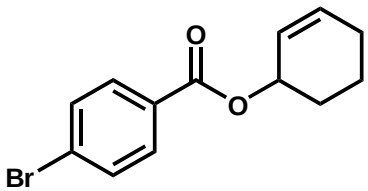
8.04-8.07 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.9, 24.9, 28.3, 68.5, 125.6, 128.2, 129.5, 130.7, 132.7, 132.8, 166.2; HRMS (ESI) *m/z* $[M+Na]^+$ Calcd for $C_{13}H_{14}NaO_2$: 225.0886, found: 225.0885; IR (KBr, cm^{-1}): ν 1715.



cyclohex-2-enyl 4-fluorobenzoate (4b). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.67-1.75 (m, 1H), 1.78-1.91 (m, 2H), 1.94-2.17 (m, 3H), 5.50 (m, 1H), 5.81-5.84 (m, 1H), 5.99-6.03 (m, 1H), 7.01-7.12 (m, 2H), 8.05-8.08 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.9, 24.9, 28.3, 68.7, 115.2, 115.4, 125.5, 132.0, 132.1, 132.9, 164.3, 165.2, 166.9; HRMS (ESI) *m/z* $[M+Na]^+$ Calcd for $C_{13}H_{13}FNaO_2$: 243.07918, found: 243.07882; IR (KBr, cm^{-1}): ν 1716.

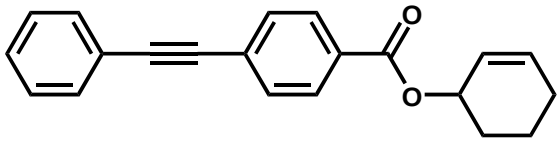


cyclohex-2-enyl 4-chlorobenzoate (4c). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.67-1.75 (m, 1H), 1.79-1.91 (m, 2H), 1.93-2.17 (m, 3H), 5.49 (m, 1H), 5.80-5.84 (m, 1H), 5.99-6.03 (m, 1H), 7.40 (d, *J* = 8.5 Hz, 2H), 7.98 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.8, 24.8, 28.3, 68.8, 125.4, 128.5, 129.1, 130.9, 132.9, 139.0, 165.2; HRMS (ESI) *m/z* $[M+Na]^+$ Calcd for $C_{13}H_{13}ClNaO_2$: 259.0496, found: 259.0502; IR (KBr, cm^{-1}): ν 1716.

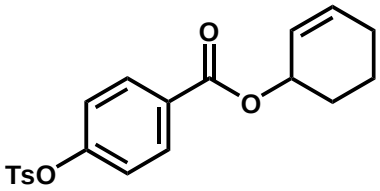


cyclohex-2-enyl 4-bromobenzoate (4d). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.67-1.74 (m, 1H), 1.78-1.91 (m, 2H), 1.93-2,17 (m, 3H), 5.49 (m,1H), 5.80-5.83 (m,1H), 5.99-6.03 (m,1H), 7.56 (d, *J* = 8.5 Hz, 2H), 7.91 (d, *J* = 8.5 Hz, 2H); ^{13}C

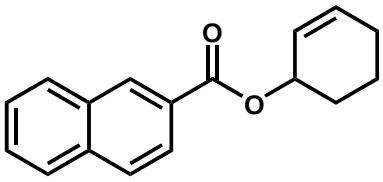
NMR (100 MHz, $CDCl_3$) δ 18.8, 24.9, 28.3, 68.9, 125.4, 127.8, 129.6, 131.1, 131.5, 133.0, 165.4; HRMS (ESI) *m/z* $[M+Na]^+$ Calcd for $C_{13}H_{13}BrNaO_2$: 302.9991, found: 302.0995; IR (KBr, cm^{-1}): ν 1717.



cyclohex-2-enyl 4-(phenylethynyl)benzoate (4e). ^1H NMR (400 MHz, $CDCl_3$) δ 1.69-1.74 (m, 1H), 1.84-1.91 (m, 2H), 1.95-2.18 (m, 3H), 5.51 (m, 1H), 5.82-5.84 (m, 1H), 6.01-6.03 (m, 1H), 7.36-7.37 (m, 3H), 7.54-7.59 (m, 4H), 8.03 (d, *J* = 8.1 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.9, 24.9, 28.4, 69.8, 88.7, 92.2, 122.7, 125.6, 127.8, 128.4, 128.7, 129.5, 130.1, 131.4, 131.7, 133.0, 165.6; HRMS (ESI) *m/z* $[M+H]^+$ Calcd for $C_{21}H_{19}O_2$: 303.1385, found: 303.1382; IR (KBr, cm^{-1}): ν 1709.

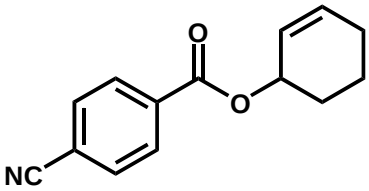


cyclohex-2-enyl 4-(tosyloxy)benzoate (4f). ^1H NMR (400 MHz, $CDCl_3$) δ 1.66-1.73 (m, 1H), 1.78-1.85 (m, 2H), 1.91-2.15 (m, 3H), 2.44 (s, 3H), 5.47 (m, 1H), 5.79-5.81 (m, 1H), 5.98-6.01 (m, 1H), 7.05 (d, *J* = 8.6 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.70 (d, *J* = 8.2 Hz, 1H), 7.99 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.7, 21.6, 24.8, 28.2, 68.9, 122.2, 125.2, 128.4, 129.5, 129.8, 132.0 131.2, 133.1, 145.6, 152.7, 164.9; HRMS (ESI) *m/z* $[M+Na]^+$ Calcd for $C_{20}H_{20}NaO_5S$: 395.0924, found: 395.0924; IR (KBr, cm^{-1}): *v* 1712.

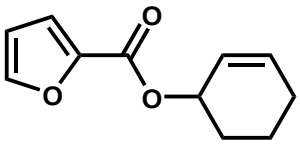


cyclohex-2-enyl 2-naphthoate (4g). ^1H NMR (400 MHz, $CDCl_3$) δ 1.69-1.78

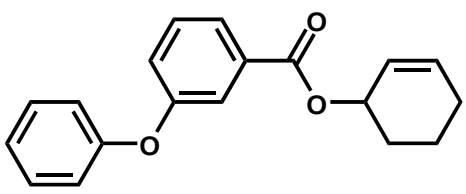
(m, 130H), 1.85-1.97 (m, 2H), 1.99-2.09 (m, 2H), 2.15-2.20 (m, 1H), 5.58 (m, 1H), 5.87-5.91 (m, 1H), 6.02-6.06 (m, 1H), 7.51-7.60 (m, 2H), 7.87 (m, 2H), 7.95 (m, 1H), 8.08 (m, 1H), 8.61 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.0, 25.0, 28.4, 68.8, 125.3, 125.8, 126.5, 127.7, 128.0, 128.0, 128.1, 130.9, 132.5, 132.9, 135.4, 166.4; HRMS (ESI) *m/z* $[M+Na]^+$ Calcd for $C_{17}H_{16}NaO_2$: 275.1043, found: 275.1042; IR (KBr, cm^{-1}): *v* 1711.



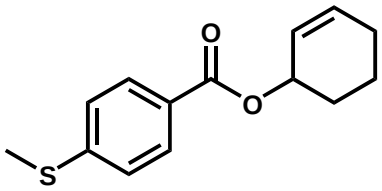
cyclohex-2-enyl 4-cyanobenzoate (4h). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.69-1.74 (m, 1H), 1.81-1.92 (m, 2H), 1.96-2.19 (m, 3H), 5.53 (m, 1H), 5.81-5.84 (m, 1H), 6.03-6.06 (m, 1H), 7.74 (d, *J* = 7.7 Hz, 2H), 8.15 (d, *J* = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.7, 24.8, 28.2, 69.5, 116.1, 118.0, 124.9, 130.0, 132.1, 133.5, 134.5, 164.4; HRMS (ESI) *m/z* $[M+Na]^+$ Calcd for $C_{14}H_{13}NNaO_2$: 250.0838, found: 250.0849; IR (KBr, cm^{-1}): *v* 1710.



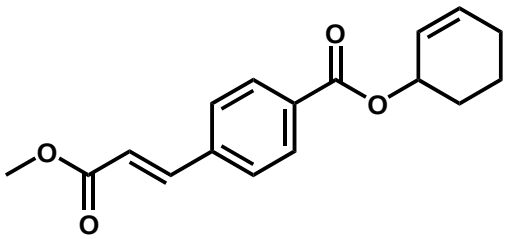
cyclohex-2-enyl furan-2-carboxylate (4i). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.65-1.72 (m, 1H), 1.79-1.89 (m, 2H), 1.93-2.15 (m, 3H), 5.49 (m, 1H), 5.79-5.81 (m, 1H), 5.99-6.03 (m, 1H), 6.49-6.51 (m, 1H), 7.17-7.18 (m, 1H), 7.57 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.9, 24.9, 28.3, 68.8, 111.7, 117.7, 125.3, 133.2, 145.0, 146.1, 158.5; HRMS (ESI) *m/z* $[M+Na]^+$ Calcd for $C_{11}H_{12}NaO_3$: 215.0679, found: 215.07; IR (KBr, cm^{-1}): ν 1722.



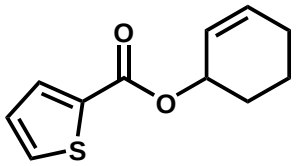
cyclohex-2-enyl 3-phenoxybenzoate (4j). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.66-1.73 (m, 1H), 1.79-1.88 (m, 2H), 1.92-2.16 (m, 3H), 5.49 (m, 1H), 5.80-5.83 (m, 1H), 5.98-6.01 (m, 1H), 7.00-7.01 (m, 2H), 7.11-7.19 (m, 2H), 7.33-7.41 (m, 3H), 7.70 (m, 1H), 7.79-7.81(m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.8, 24.9, 28.3, 68.8, 118.9, 119.9, 123.1, 123.6, 124.4, 125.5, 129.6, 129.8, 132.6, 133.0, 156.8, 157.2, 165.6; HRMS (ESI) *m/z* $[M+Na]^+$ Calcd for $C_{19}H_{18}NaO_3$: 317.1148, found: 317.1148; IR (KBr, cm^{-1}): *v* 1715.



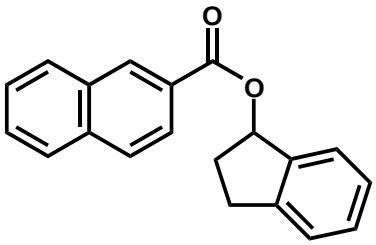
cyclohex-2-enyl 4-(methylthio)benzoate (4k). Yield: 63%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.66-1.74 (m, 1H), 1.80-1.90 (m, 2H), 1.93-2.17 (m, 3H), 2.50 (s, 3H), 5.49 (m, 1H), 5.81-5.84 (m, 1H), 5.98-6.02 (m, 1H), 7.23 (d, *J* = 8.5 Hz, 2H), 7.95 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.8 18.9, 24.9, 28.4, 68.4, 124.8, 125.7, 126.8, 129.8, 132.7, 145.1, 165.9; HRMS (ESI) *m/z* $[M+Na]^+$ Calcd for $C_{14}H_{16}NaO_2S$: 271.0763, found: 271.0765; IR (KBr, cm^{-1}): *v* 1708.



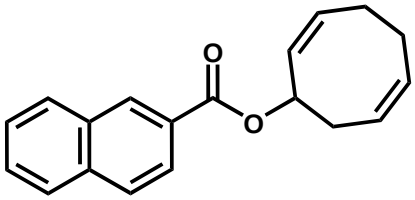
(E)-cyclohex-2-enyl 4-(2-methoxyvinyl)benzoate (4l). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.68-1.75 (m, 1H), 1.80-1.90 (m, 2H), 1.94-2.18 (m, 3H), 3.82 (s, 3H), 5.51 (m, 1H), 5.82-5.85 (m, 1H), 6.00-6.04 (m, 1H), 6.52 (d, *J* = 16.1 Hz, 1H), 7.57 (d, *J* = 8.3 Hz, 2H), 7.70 (d, *J* = 16.1 Hz, 1H), 8.06 (d, *J* = 8.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.8, 24.9, 28.3, 51.8, 68.8, 119.9, 125.4, 127.8, 130.0, 132.0, 133.0, 138.3, 143.4, 165.4, 166.9; HRMS (ESI) *m/z* $[M+H]^+$ Calcd for $C_{17}H_{19}O_4$: 287.1278, found: 287.1289; IR (KBr, cm^{-1}): *v* 1713.



cyclohex-2-enyl thiophene-2-carboxylate (4m). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.66-1.73 (m, 1H), 1.81-1.90 (m, 2H), 1.92-2.16 (m, 3H), 5.47 (m, 1H), 5.80-5.83 (m, 1H), 5.98-6.02 (m, 1H), 7.08-7.10 (m, 1H), 7.53-7.54 (m, 1H), 7.79-7.80 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.8, 24.9, 28.3, 68.9, 125.4, 127.6, 132.1, 133.0, 133.1, 134.4, 161.9; HRMS (ESI) *m/z* $[M+Na]^+$ Calcd for $C_{11}H_{12}NaO_2S$: 231.04502, found: 231.04459; IR (KBr, cm^{-1}): *v* 1703.

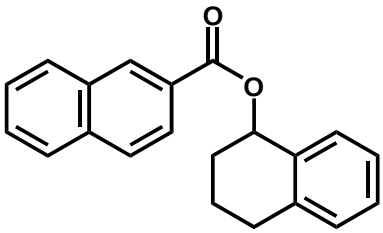


2,3-dihydro-1H-inden-1-yl 2-naphthoate (4n). ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.27-2.34 (m, 1H), 2.64-2.73 (m, 1H), 2.94-3.02 (m, 1H), 3.19-3.27 (m, 1H), 6.51-6.54 (m, 1H), 7.27-7.34 (m, 3H), 7.50-7.58 (m, 3H), 7.85-8.08 (m, 4H), 8.60 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 30.2, 32.4, 79.0, 124.8, 125.2, 125.7, 126.5, 126.7, 127.6, 127.6, 128.0, 128.0, 128.9, 129.2, 131.0, 132.3, 135.4, 141.1, 144.4, 166.6; HRMS (ESI) *m/z* $[M+Na]^+$ Calcd for $C_{20}H_{16}NaO_2$: 311.10425, found: 311.10365; IR (KBr, cm^{-1}): *v* 1699.

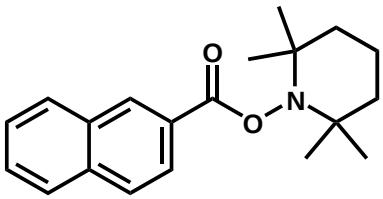


(2Z,6Z)-cycloocta-2,6-dienyl 2-naphthoate (4o). ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.22-2.38 (m, 2H), 2.55-2.65 (m, 3H), 2.88-2.97 (m, 2H), 5.61-5.75 (m, 4H), 6.28-6.30 (m, 1H), 7.52-1.60 (m, 2H), 7.06-7.88 (m, 2H), 7.95-8.09 (m, 2H), 8.62 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 27.9, 28.0, 33.9, 73.1, 125.2, 125.3, 126.6, 127.7, 128.0, 128.2, 129.2, 129.2, 129.3, 129.6, 129.8, 131.0, 132.4, 135.4, 166.0; HRMS (ESI) *m/z* $[M+Na]^+$ Calcd for $C_{19}H_{18}NaO_2$: 301.1199, found: 301.1200; IR (KBr,

cm^{-1}): *v* 1718.



1,2,3,4-tetrahydronaphthalen-1-yl 2-naphthoate (4p). ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.91-2.18 (m, 4H), 2.81-3.00 (m, 2H), 6.33 (m, 1H), 7.19-7.26 (m, 4H), 7.40-7.58 (m, 3H), 7.86-8.09 (m, 4H), 8.61 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.1, 29.1, 29.3, 70.8, 125.4, 126.1, 126.5, 127.7, 127.8, 128.0, 128.1, 128.2, 129.1, 129.3, 129.6, 131.1, 132.4, 134.7, 135.5, 138.1, 166.4; HRMS (ESI) *m/z* $[M+Na]^+$ Calcd for $C_{21}H_{18}NaO_2$: 325.1199, found: 325.1195; IR (KBr, cm^{-1}): *v* 1711.

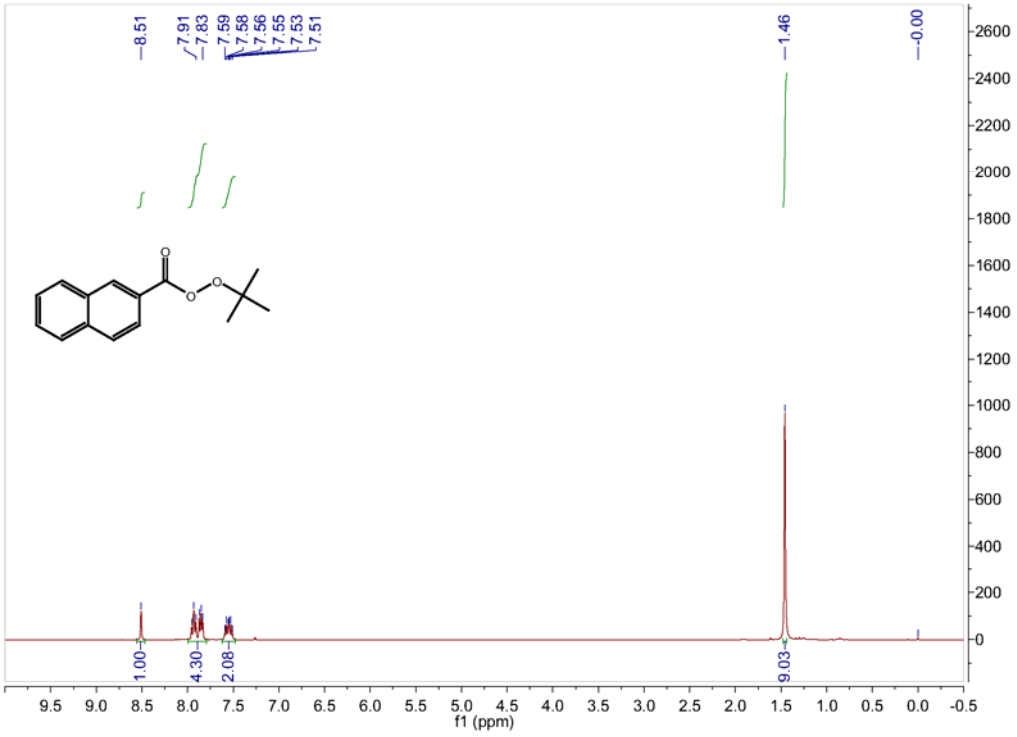


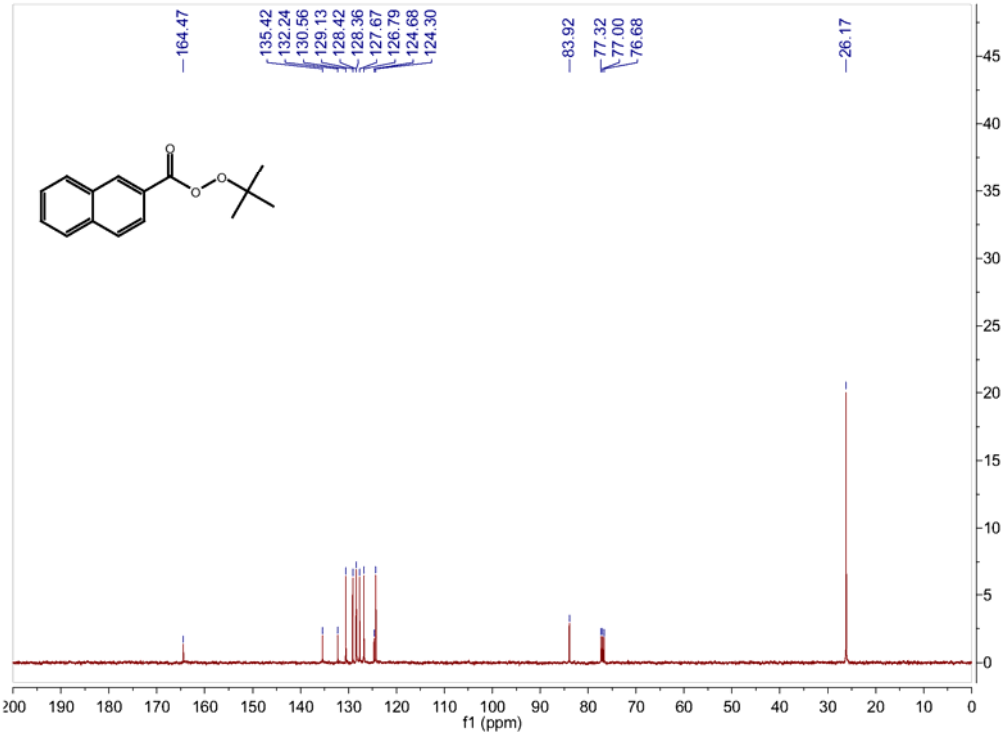
2,2,6,6-tetramethylpiperidin-1-yl 2-naphthoate (5). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.16 (s, 6H), 1.33 (s, 6H), 1.47-1.85 (m, 6H), 7.53-7.62 (m, 2H), 7.88-7.91 (m, 2H), 7.97-8.11 (m, 2H), 8.64 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 17.0, 20.9, 32.0, 39.0, 60.4, 125.2, 126.6, 127.7, 129.3, 130.9, 132.5, 135.4, 166.5; HRMS (EI) *m/z* $[M]^+$ Calcd for $C_{20}H_{25}NO_2$:311.1885, found: 311.1884; IR (KBr, cm^{-1}): *v* 1736,.

References:

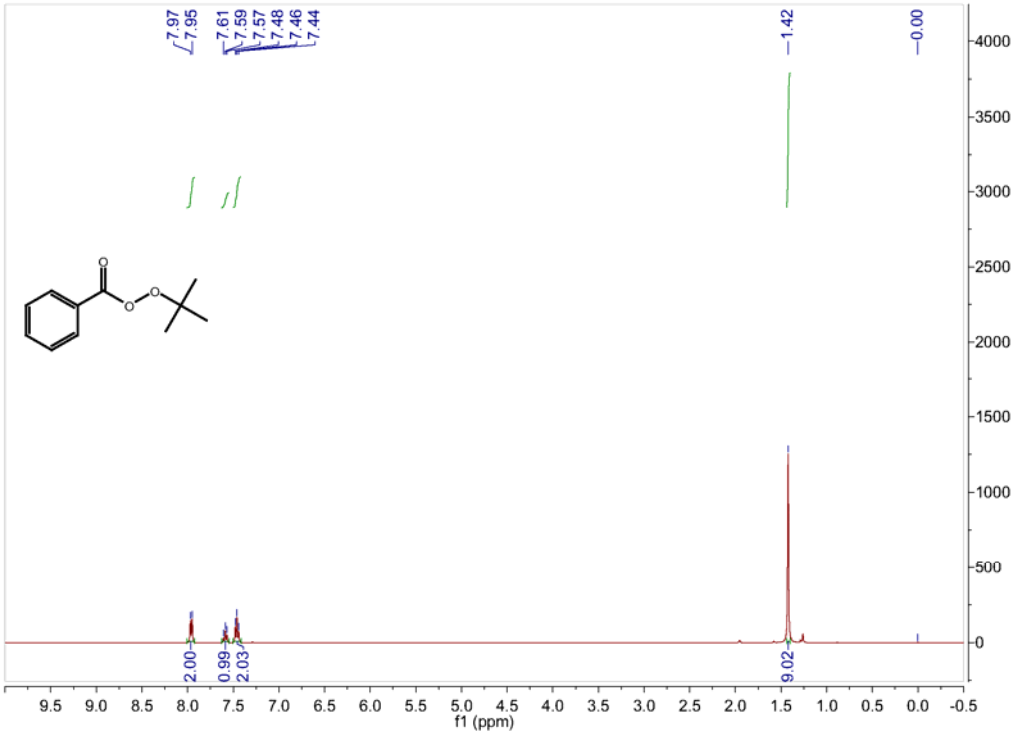
1. Hamada, Y.; Mizuno, A.; Ohno, T.; Shioiri, T. *Chem. Pharm. Bull.* **1984**, *32*, 3683.
2. Ahn, C.; Correia, R.; DeShong, P. *J. Org. Chem.* **2002**, *67*, 1751.
3. Andrus, M, B; Chen, X. *Tetrahedron.* **53**, *48*, 16229.
4. Fache, F.; Piva, O. *Synlett.* **2002**, *12*, 2035.

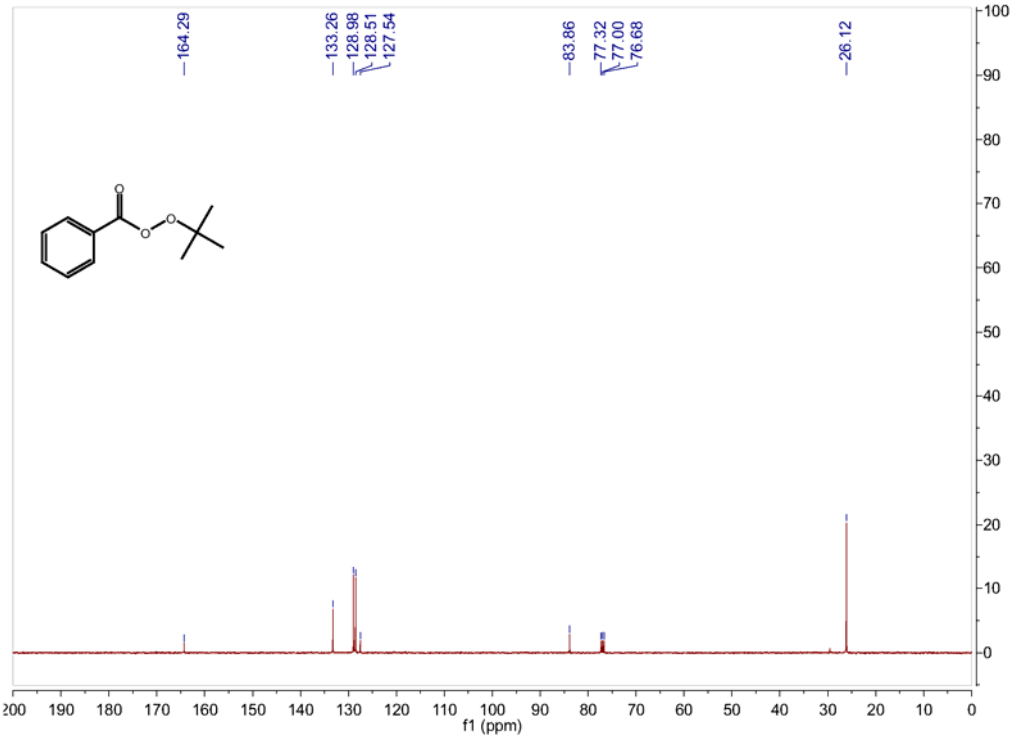
3a


8.51
7.91
7.83
7.59
7.58
7.56
7.55
7.53
7.51
1.46
0.00
1.00
4.30
2.08
9.03
f1 (ppm)

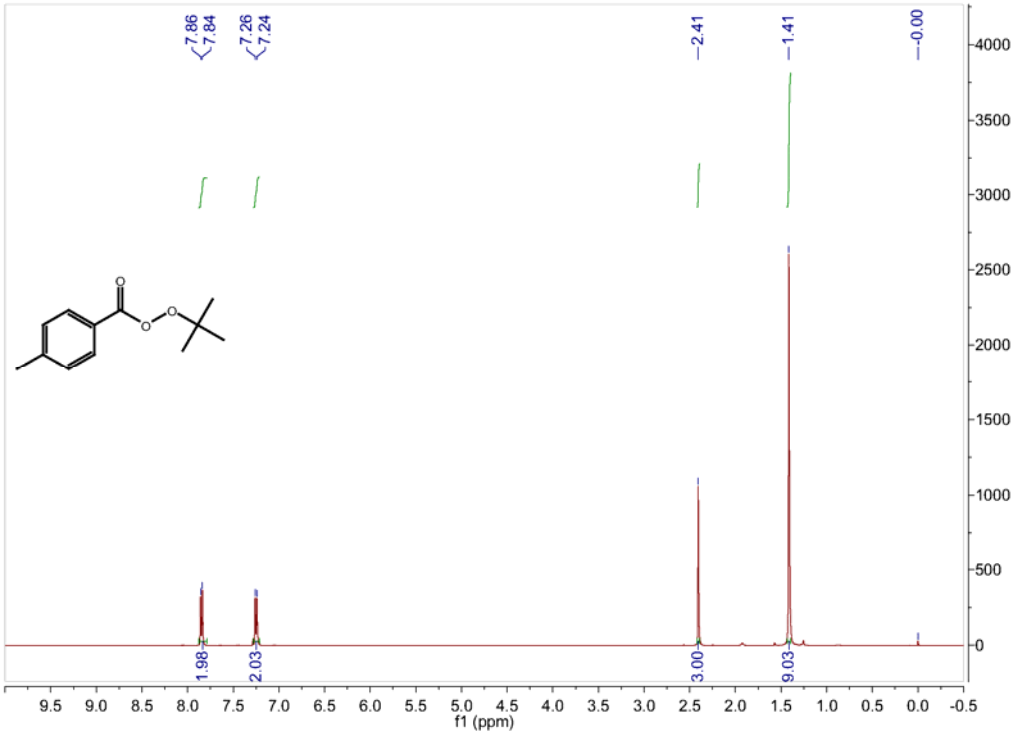

164.47
135.42
132.24
130.56
129.13
128.42
128.36
127.67
126.79
124.68
124.30
83.92
77.32
77.00
76.68
26.17
f1 (ppm)

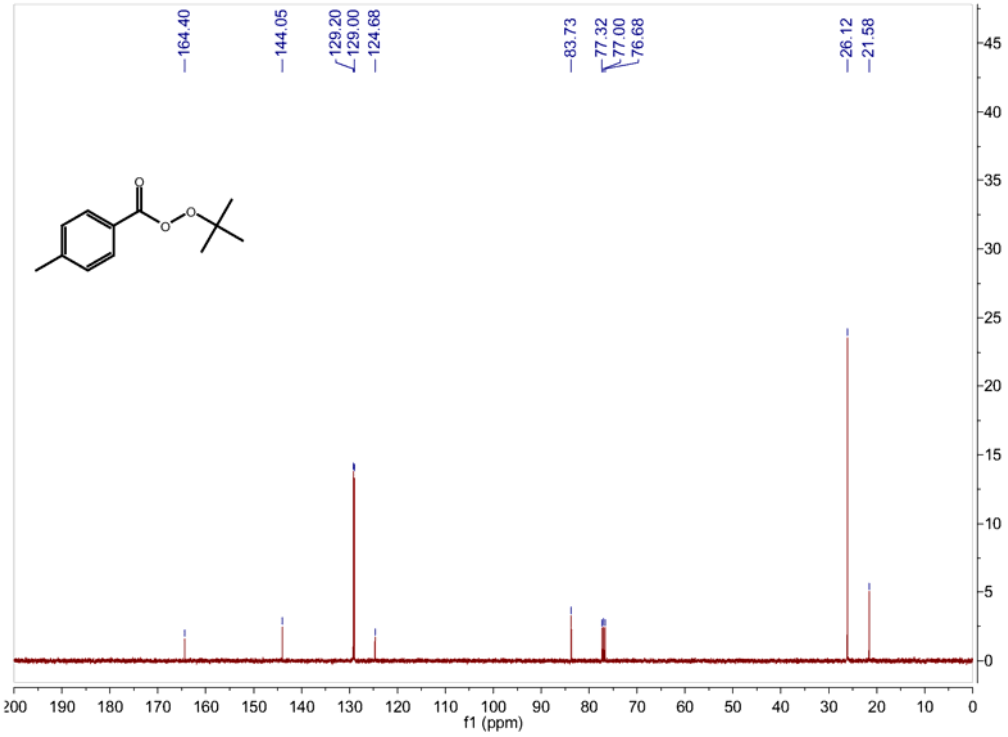
3b


7.97
7.95
7.61
7.59
7.57
7.48
7.46
7.44
1.42
0.00
2.00
0.99
2.03
9.02
f1 (ppm)

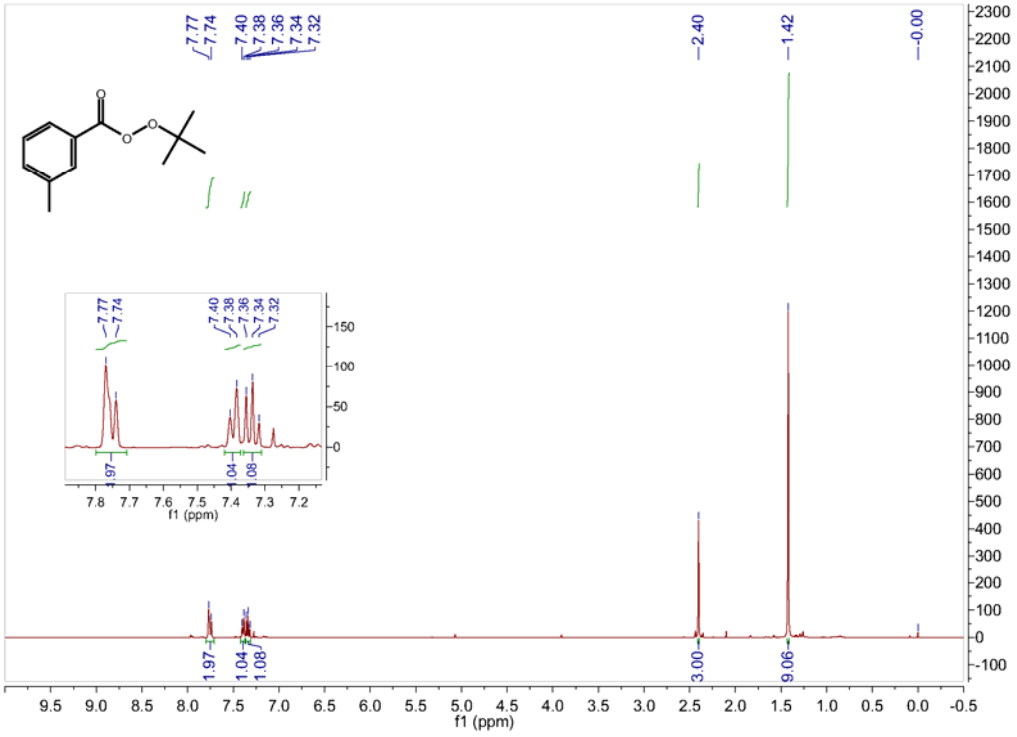

164.29
133.26
128.98
128.51
127.54
83.86
77.32
77.00
76.68
26.12
f1 (ppm)

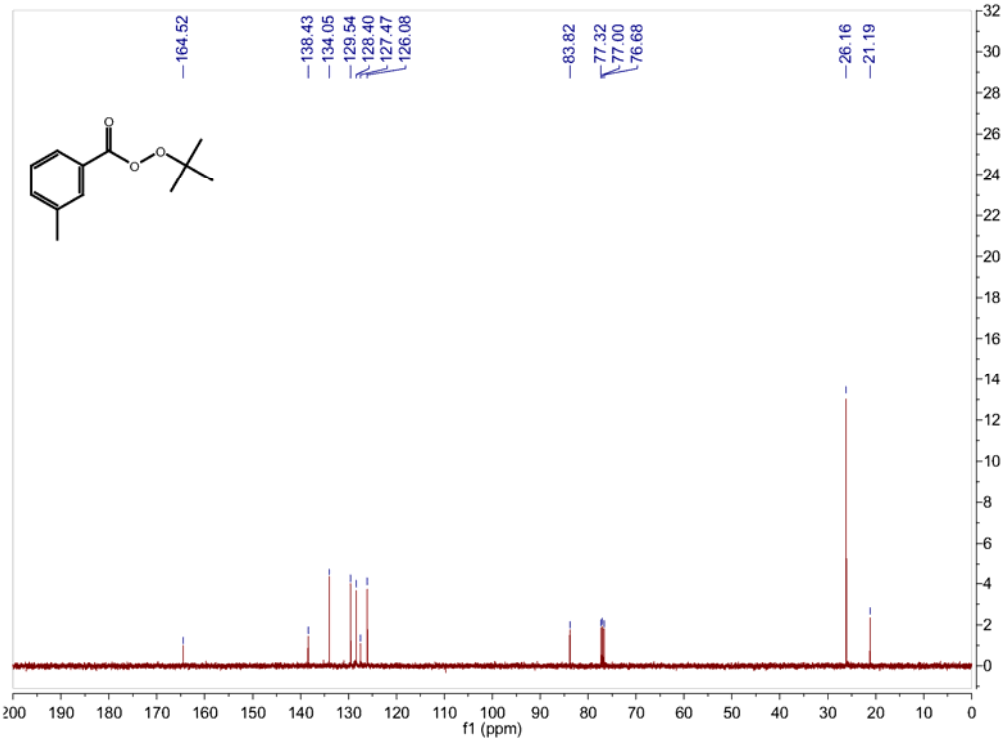
3c


7.86
7.84
7.26
7.24
2.41
1.41
0.00
1.98
2.03
3.00
9.03
f1 (ppm)

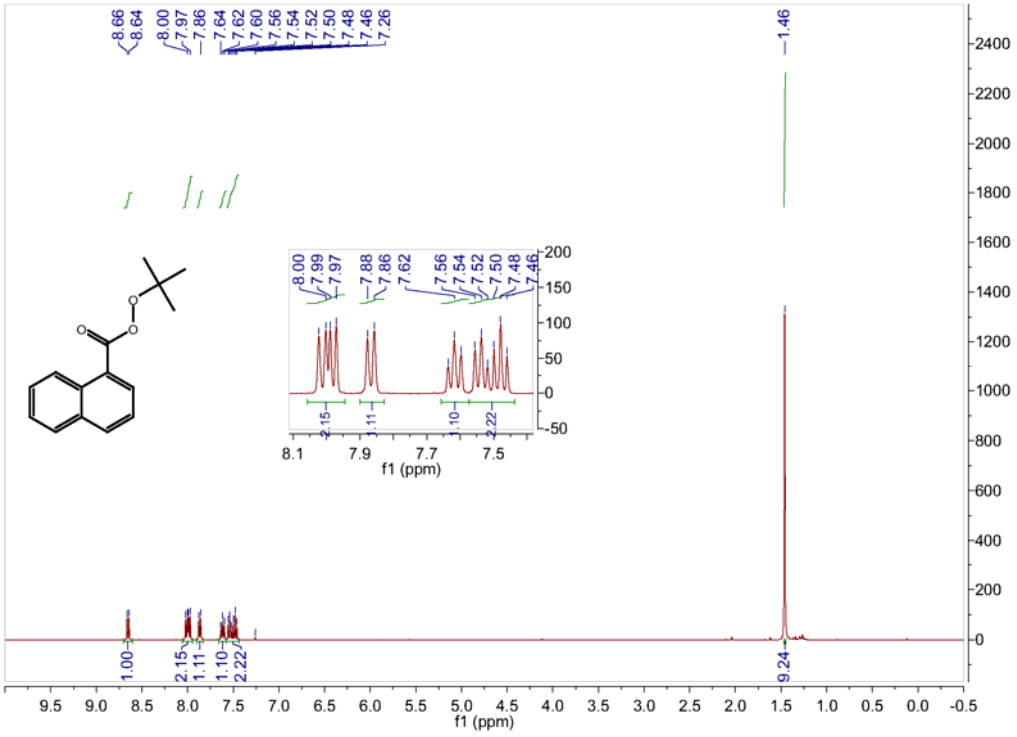

164.40
144.05
129.20
129.00
124.68
83.73
77.32
77.00
76.68
26.12
21.58
f1 (ppm)

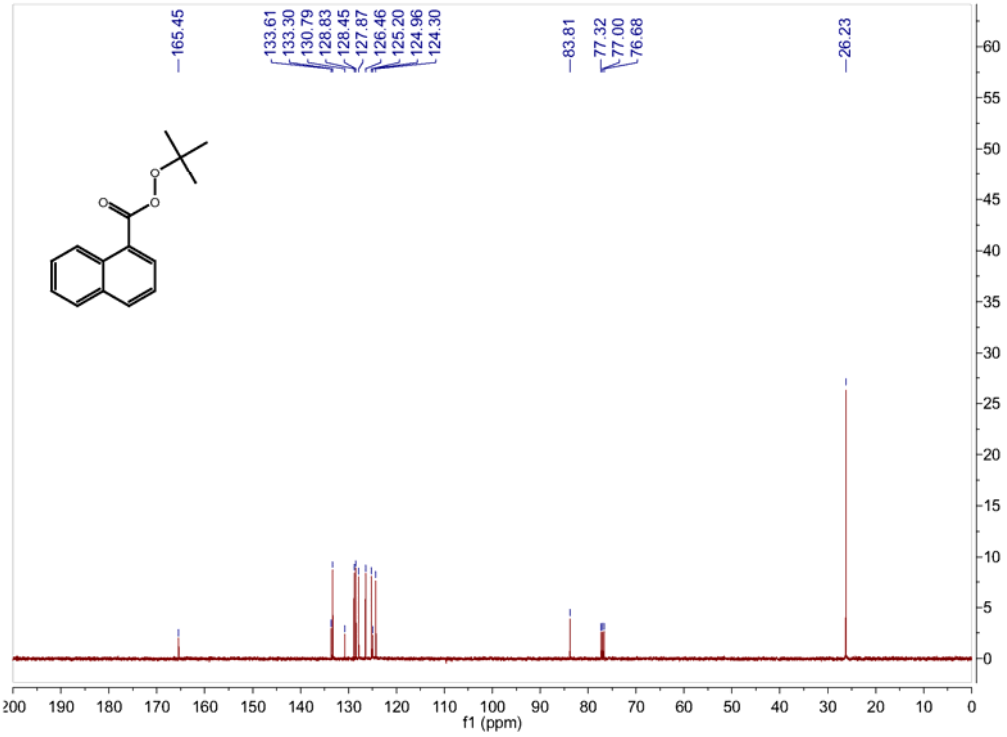
3d


7.77
7.74
7.40
7.38
7.36
7.34
7.32
2.40
1.42
0.00
1.97
1.04
1.08
3.00
9.06
f1 (ppm)

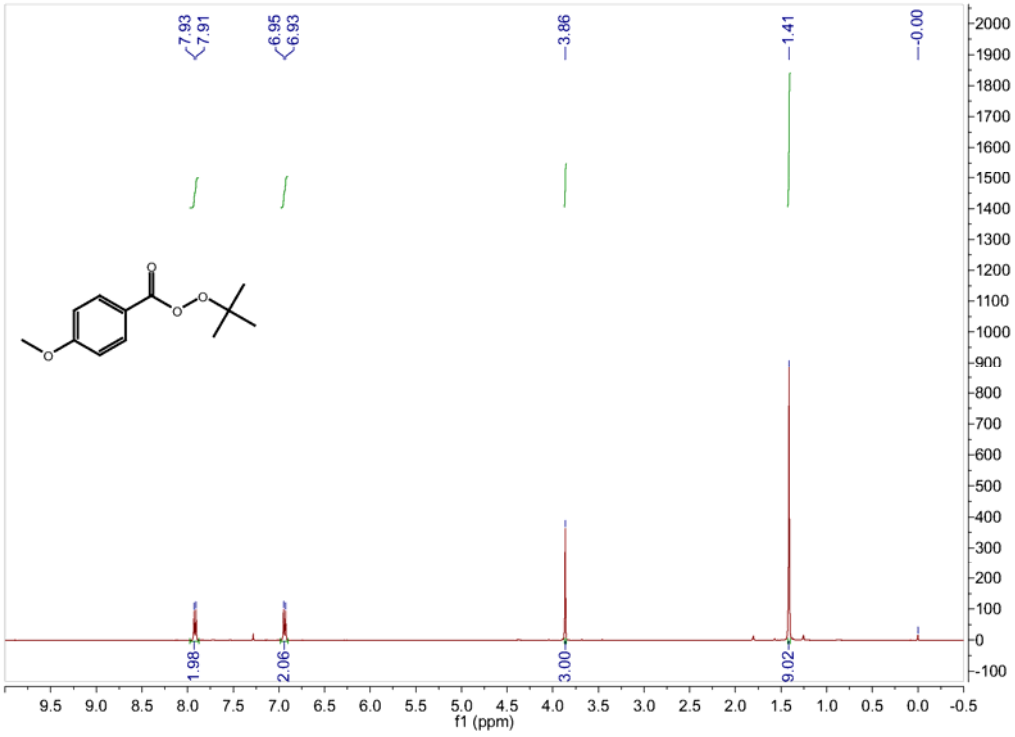

164.52
138.43
134.05
129.54
128.40
127.47
126.08
83.82
77.32
77.00
76.68
26.16
21.19
f1 (ppm)

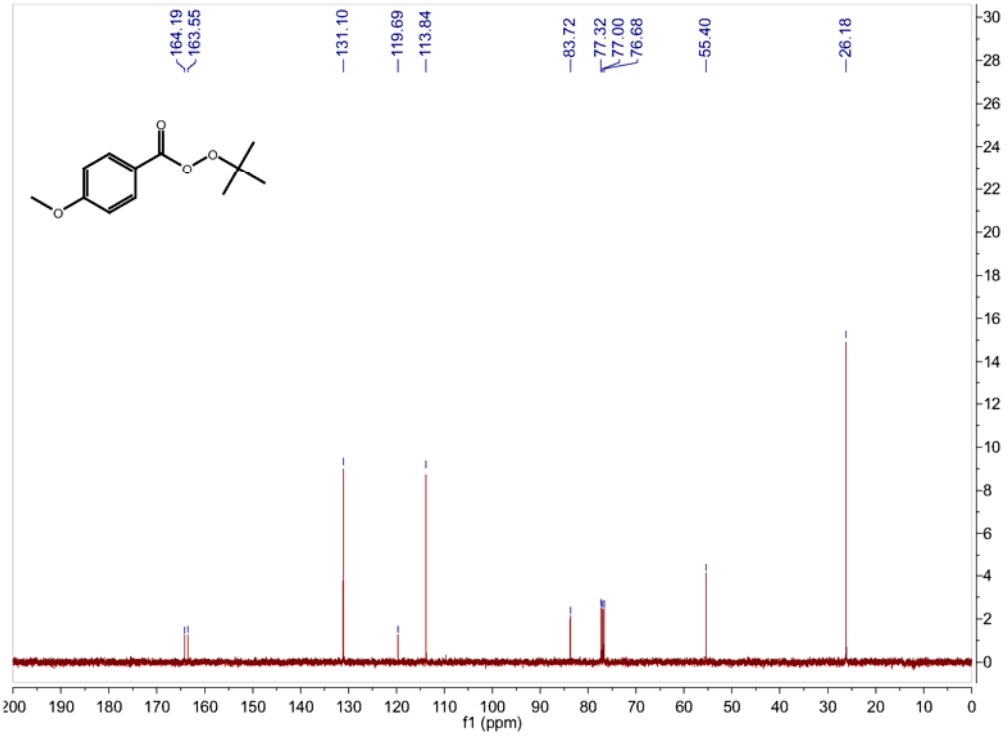
3e


8.66
8.64
8.00
7.97
7.86
7.64
7.62
7.60
7.56
7.54
7.52
7.50
7.48
7.46
7.26
1.46
1.00
2.15
1.11
1.10
2.22
9.24
f1 (ppm)

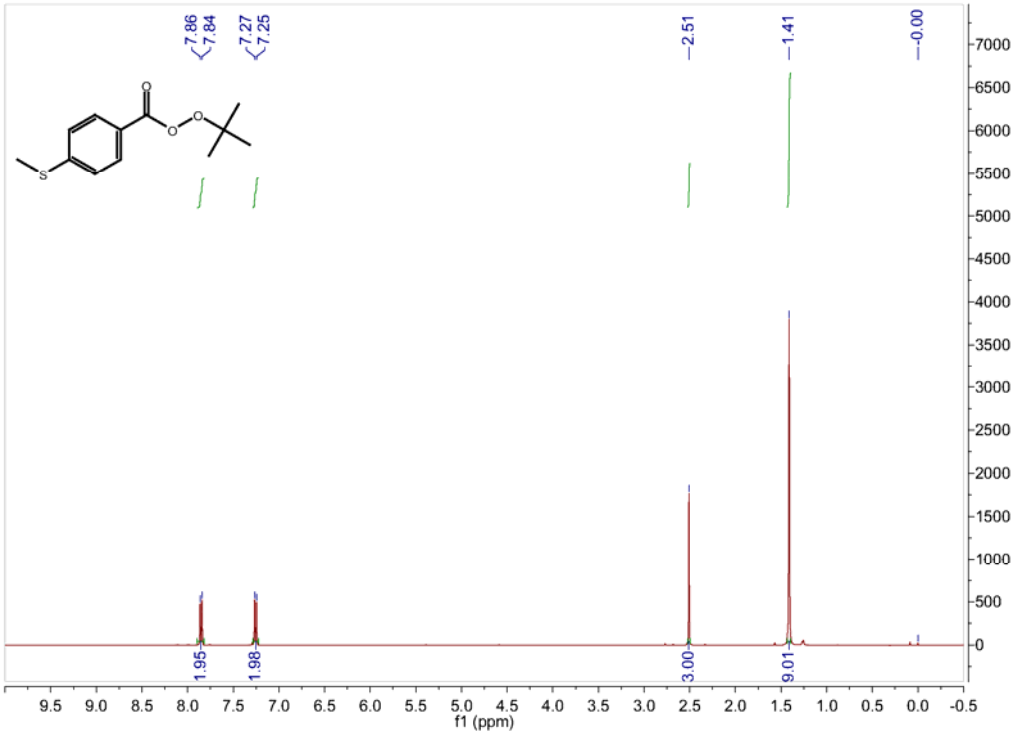

165.45
133.61
133.30
130.79
128.83
128.45
127.87
126.46
125.20
124.96
124.30
83.81
77.32
77.00
76.68
26.23
f1 (ppm)

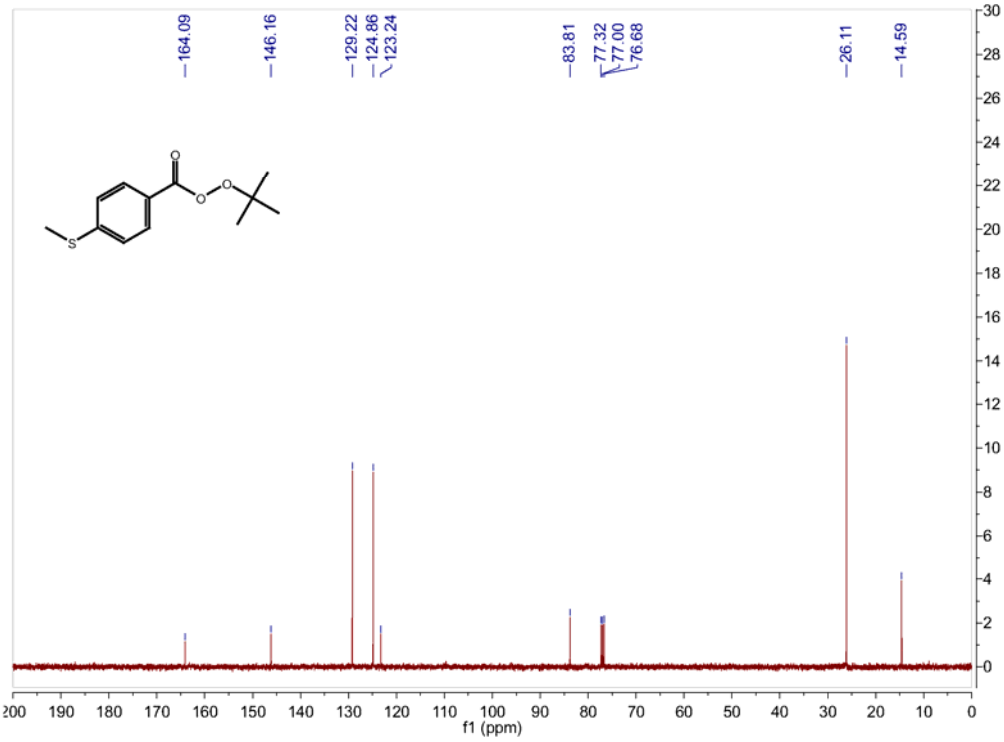
3f


7.93
7.91
6.95
6.93
3.86
1.41
0.00
1.98
2.06
3.00
9.02
f1 (ppm)

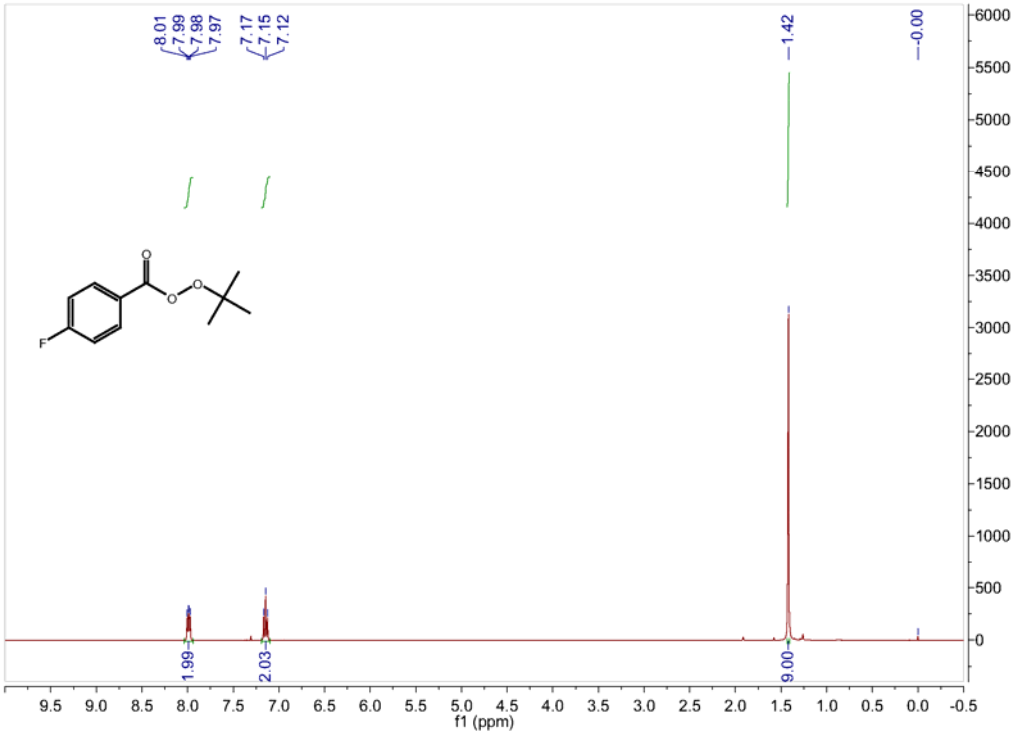

164.19
163.55
131.10
119.69
113.84
83.72
77.32
77.00
76.68
55.40
26.18
f1 (ppm)

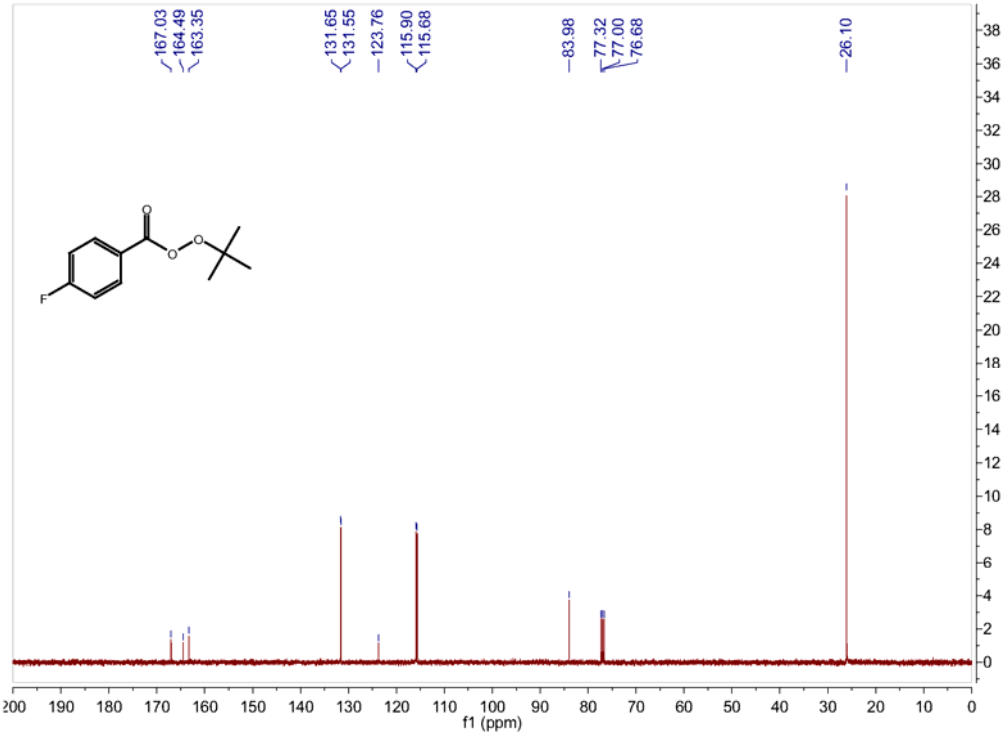
3g


7.86
7.84
7.27
7.25
2.51
1.41
0.00
1.95
1.98
3.00
9.01
f1 (ppm)

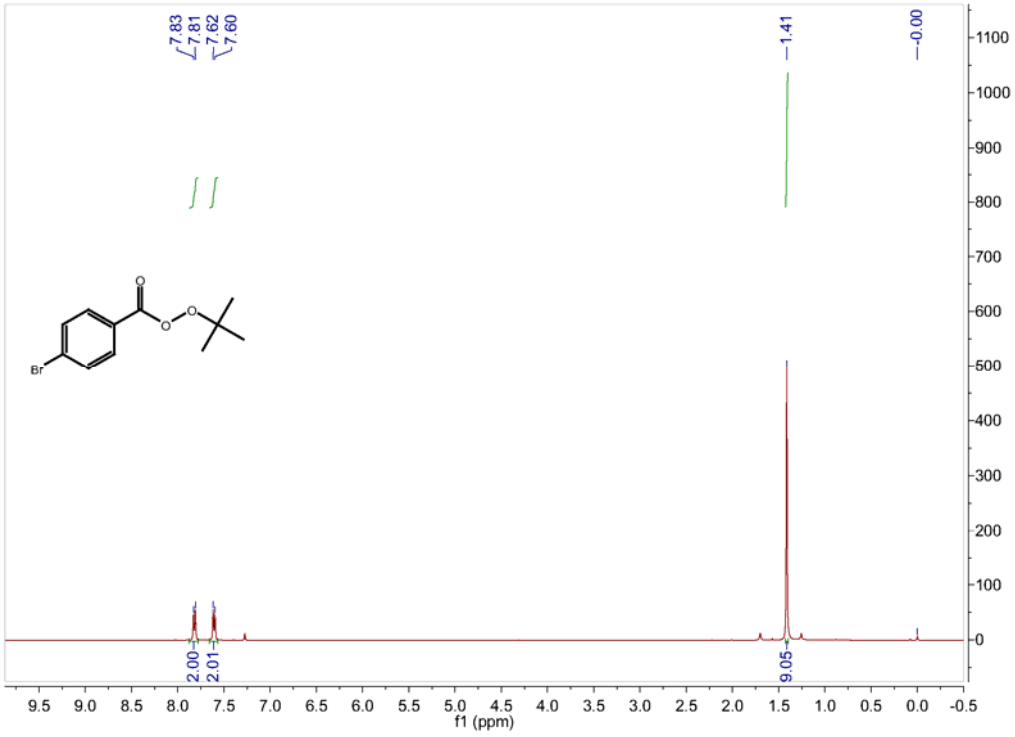

164.09
146.16
129.22
124.86
123.24
83.81
77.32
77.00
76.68
26.11
14.59
f1 (ppm)

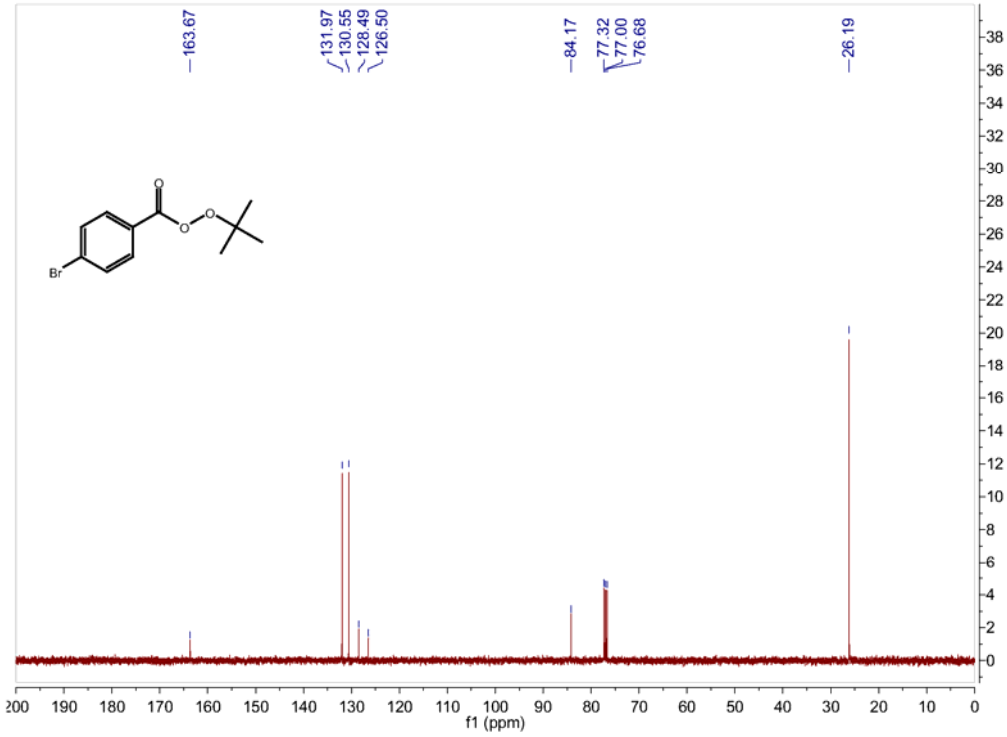
3h


8.01
7.99
7.98
7.97
7.17
7.15
7.12
1.42
0.00
1.99
2.03
9.00
f1 (ppm)

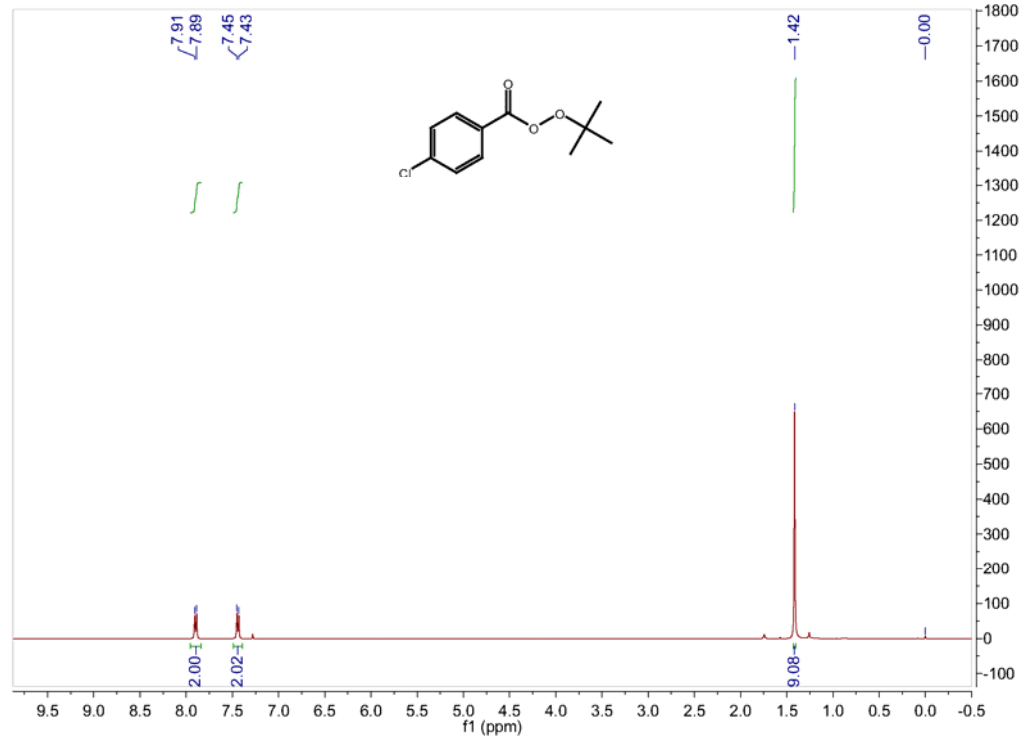

167.03
164.49
163.35
131.65
131.55
123.76
115.90
115.68
83.98
77.32
77.00
76.68
26.10
f1 (ppm)

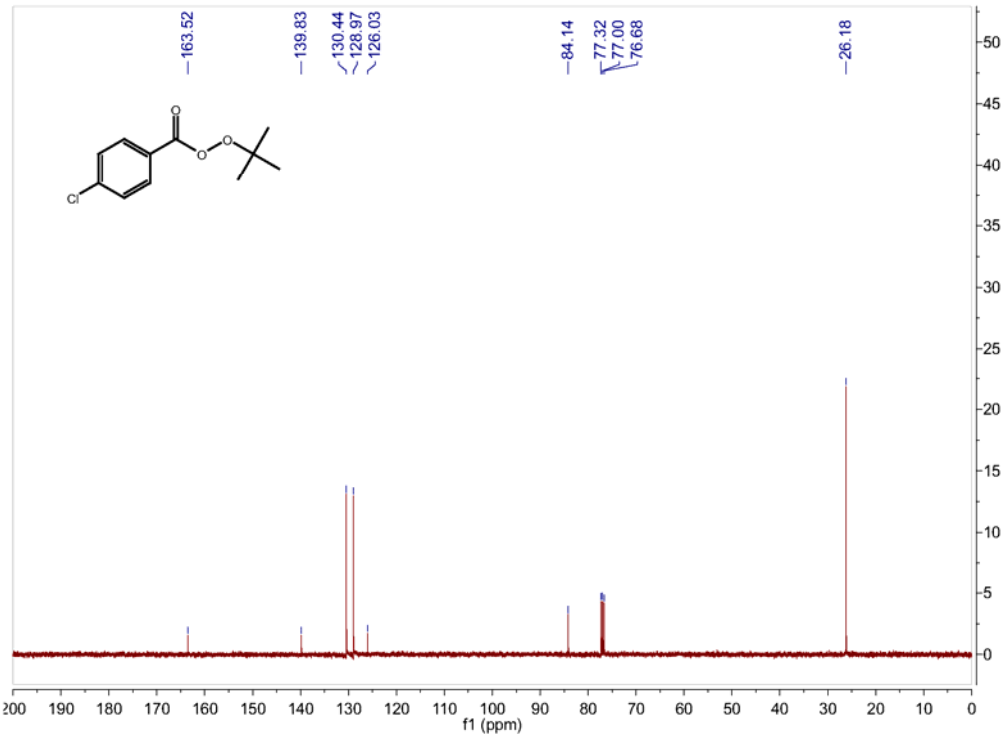
3i


7.83
7.81
7.62
7.60
1.41
0.00
2.00
2.01
9.05
f1 (ppm)

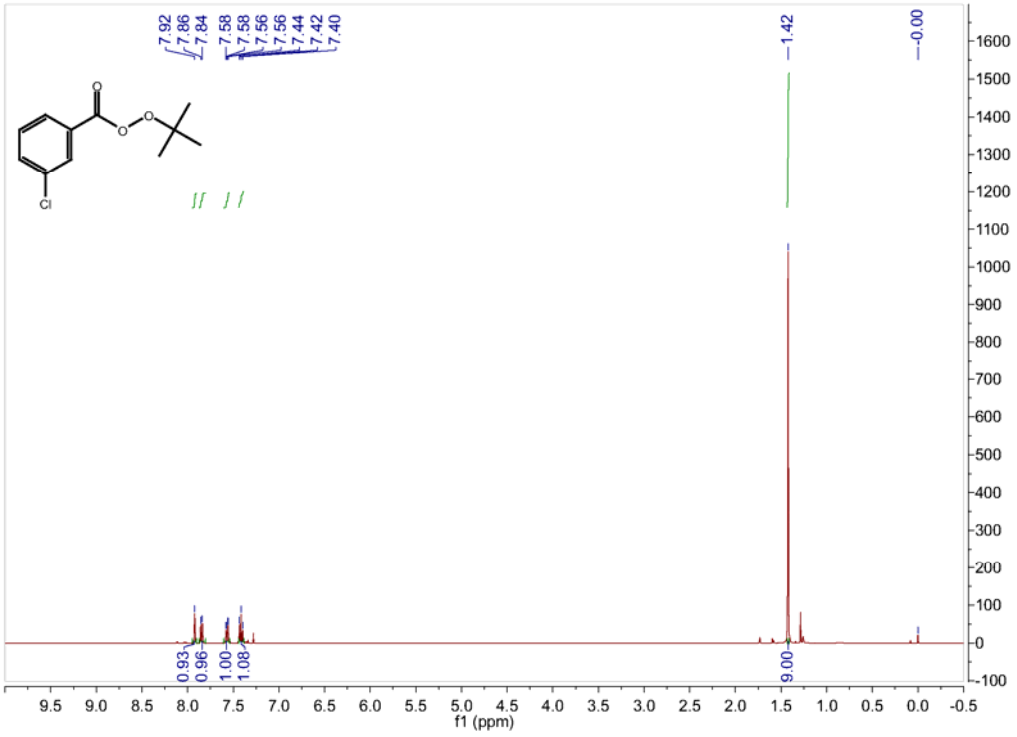

163.67
131.97
130.55
128.49
126.50
84.17
77.32
77.00
76.68
26.19
f1 (ppm)

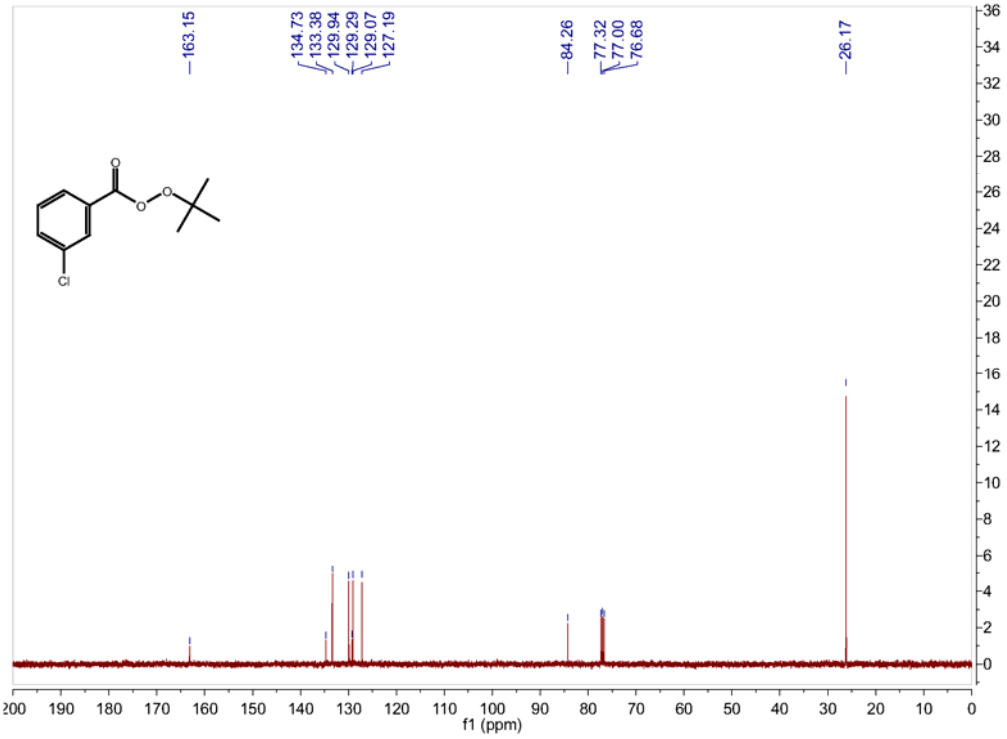
3j


7.91
7.89
7.45
7.43
1.42
0.00
2.00
2.02
9.08
f1 (ppm)

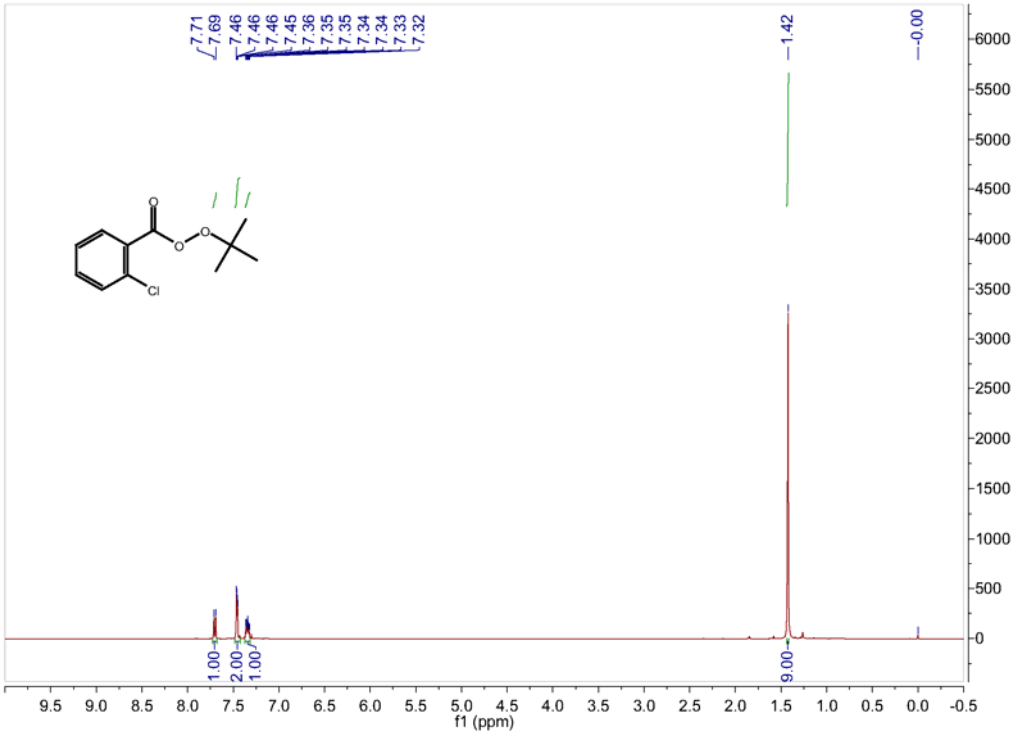

163.52
139.83
130.44
128.97
126.03
84.14
77.32
77.00
76.68
26.18
f1 (ppm)

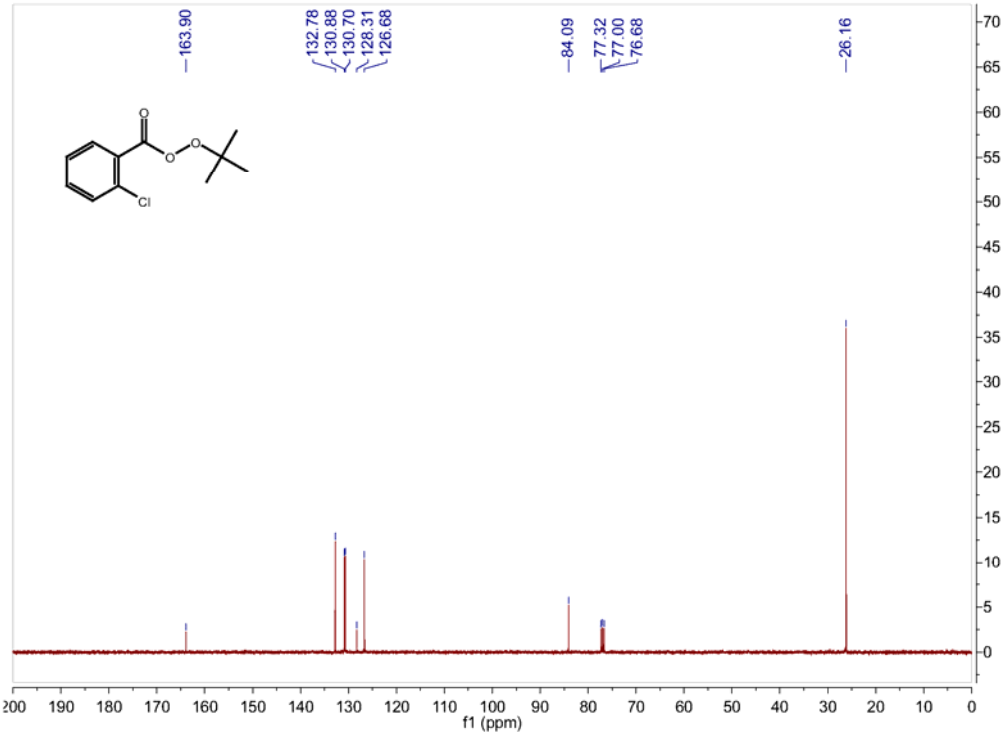
3k


7.92
7.86
7.84
7.58
7.58
7.56
7.56
7.44
7.42
7.40
1.42
0.00
0.93
0.96
1.00
1.08
9.00
Cl
f1 (ppm)

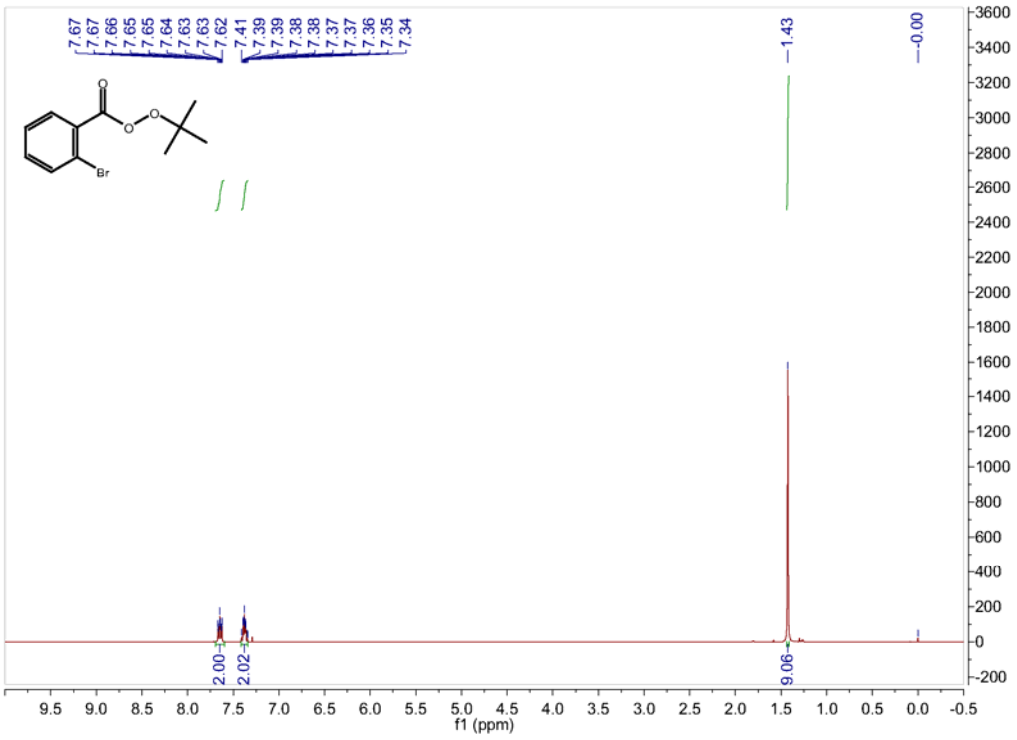

163.15
134.73
133.38
129.94
129.29
129.07
127.19
84.26
77.32
77.00
76.68
26.17
Cl
f1 (ppm)

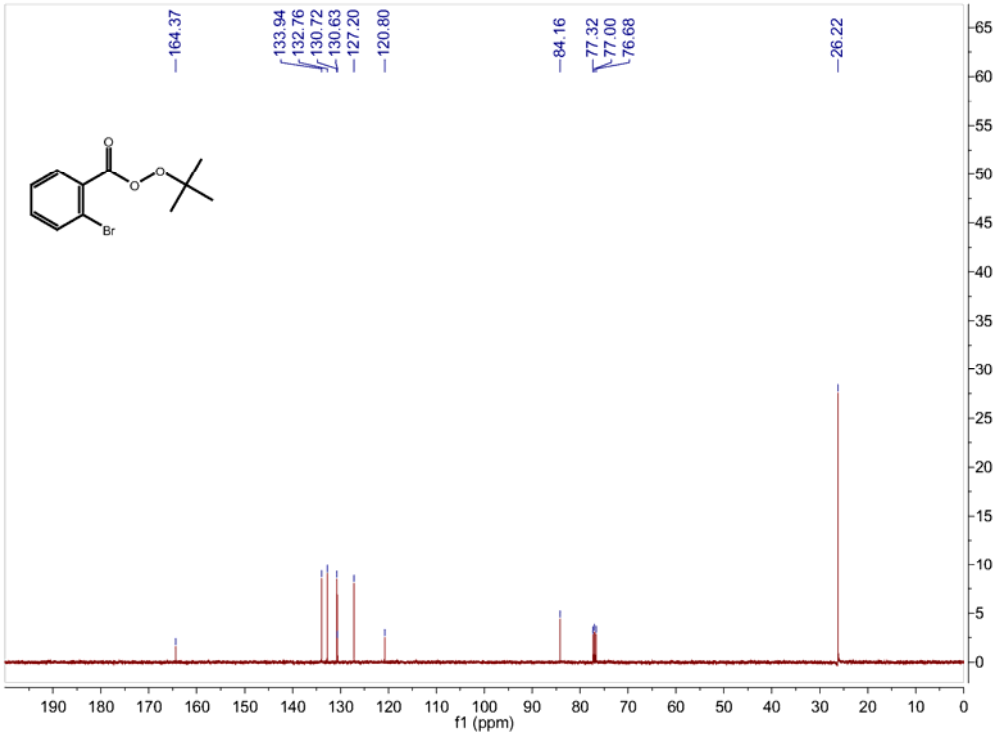
3l


7.71
7.69
7.46
7.46
7.46
7.45
7.36
7.35
7.35
7.34
7.34
7.33
7.32
1.42
0.00
1.00
2.00
1.00
9.00
f1 (ppm)

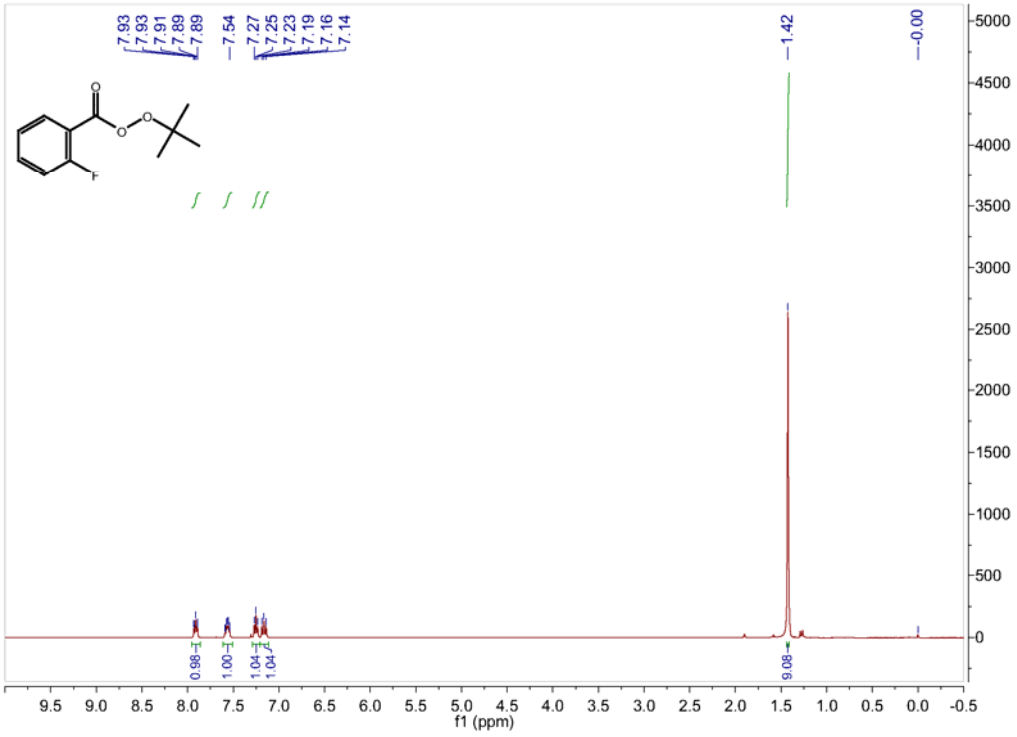

163.90
132.78
130.88
130.70
128.31
126.68
84.09
77.32
77.00
76.68
26.16
f1 (ppm)

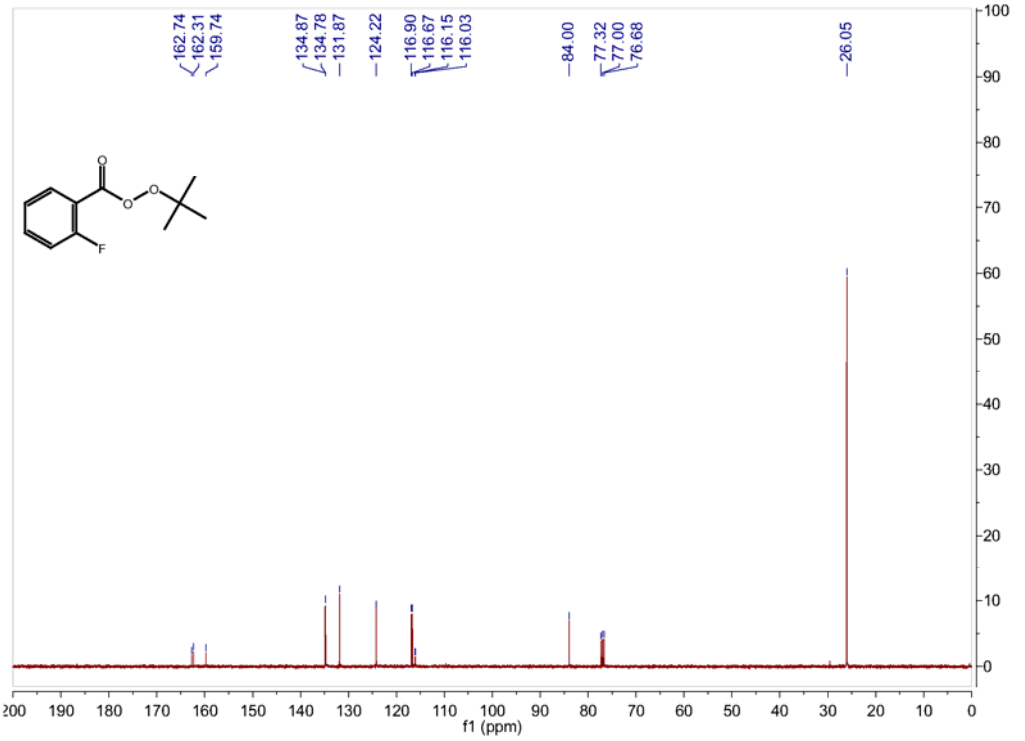
3m


7.67
7.67
7.66
7.65
7.65
7.64
7.63
7.63
7.62
7.41
7.39
7.39
7.38
7.38
7.37
7.37
7.36
7.35
7.34
1.43
0.00
Br
2.00
2.02
9.06
f1 (ppm)

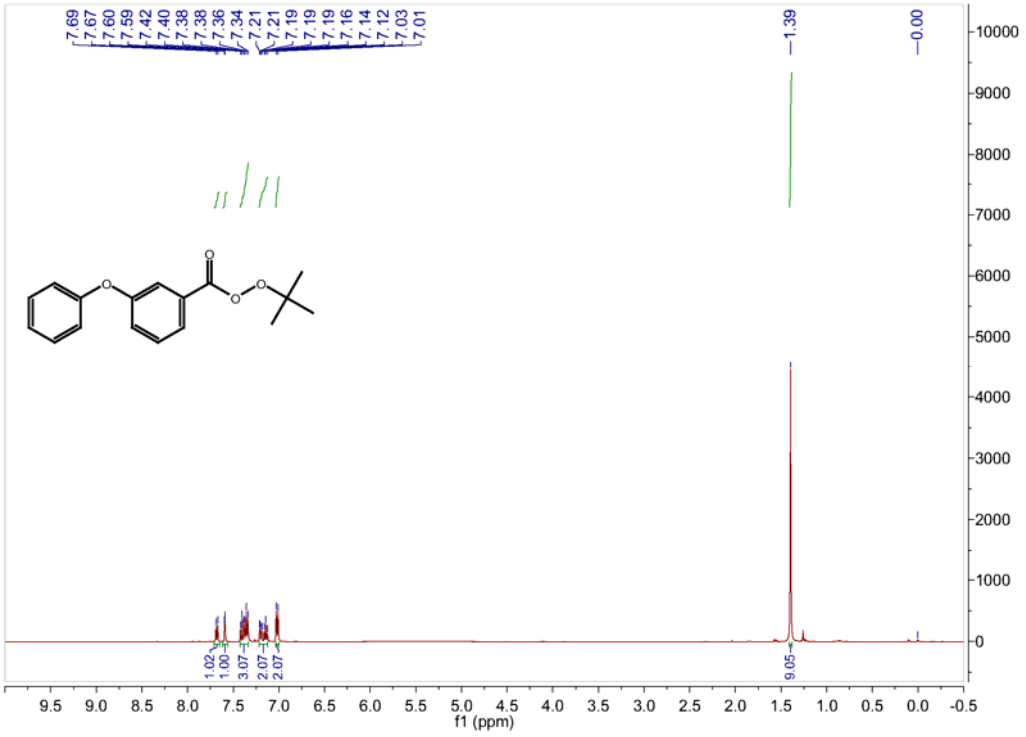

164.37
133.94
132.76
130.72
130.63
127.20
120.80
84.16
77.32
77.00
76.68
26.22
Br
f1 (ppm)

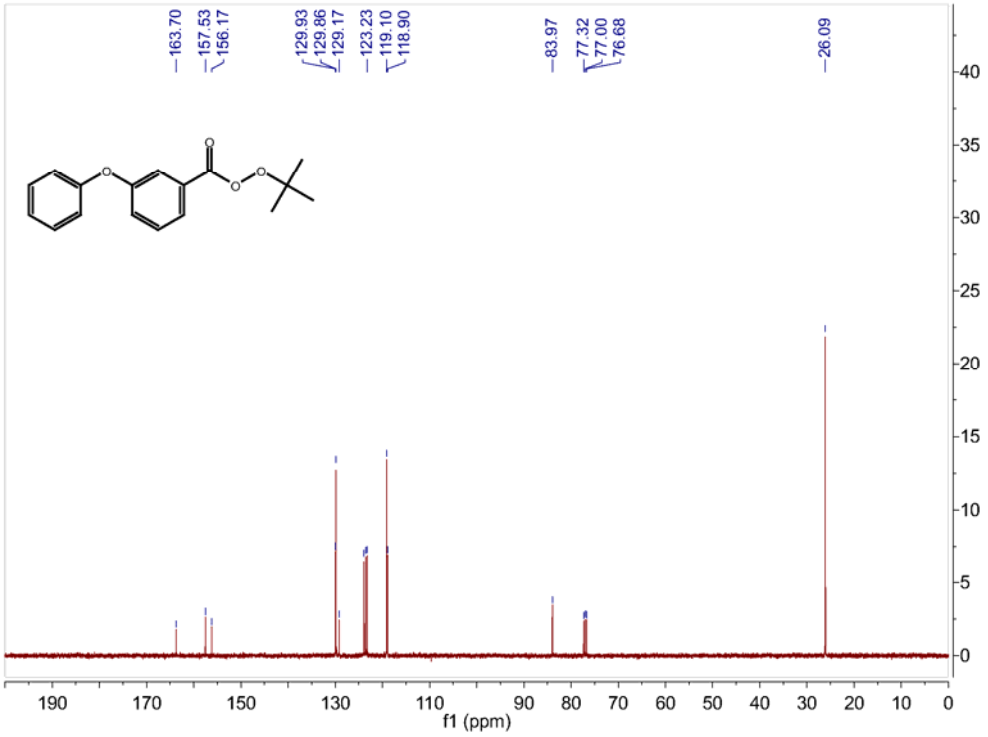
3n


7.93
7.93
7.91
7.89
7.89
7.54
7.27
7.25
7.23
7.19
7.16
7.14
1.42
0.00
0.98
1.00
1.04
1.04
9.08
f1 (ppm)

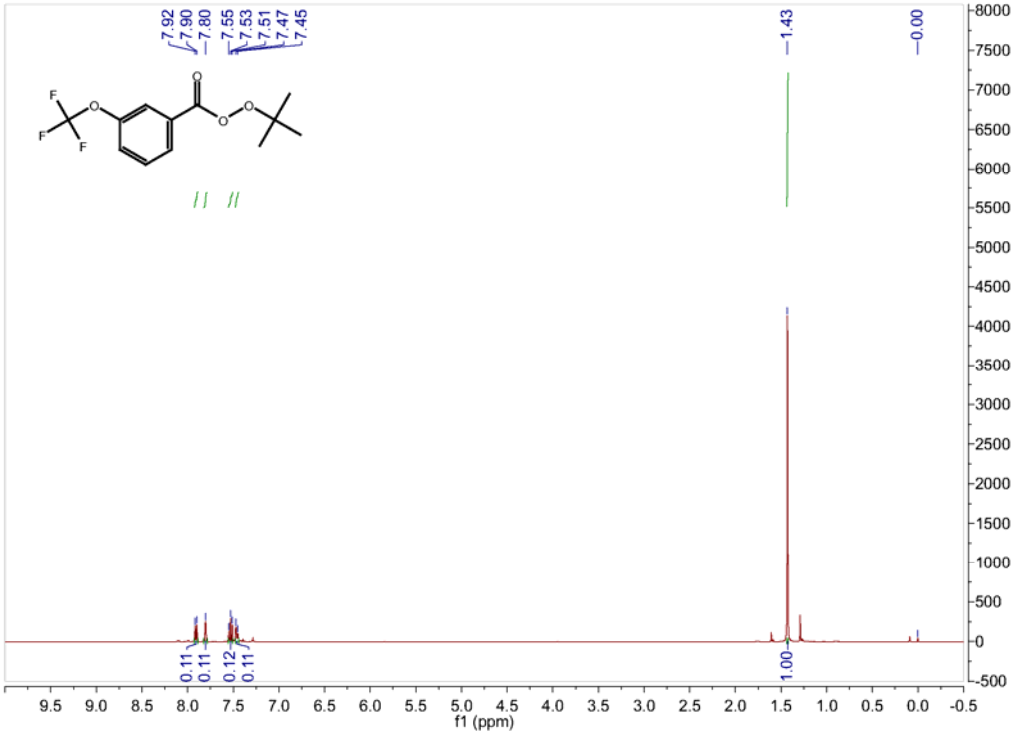

162.74
162.31
159.74
134.87
134.78
131.87
124.22
116.90
116.67
116.15
116.03
84.00
77.32
77.00
76.68
26.05
f1 (ppm)

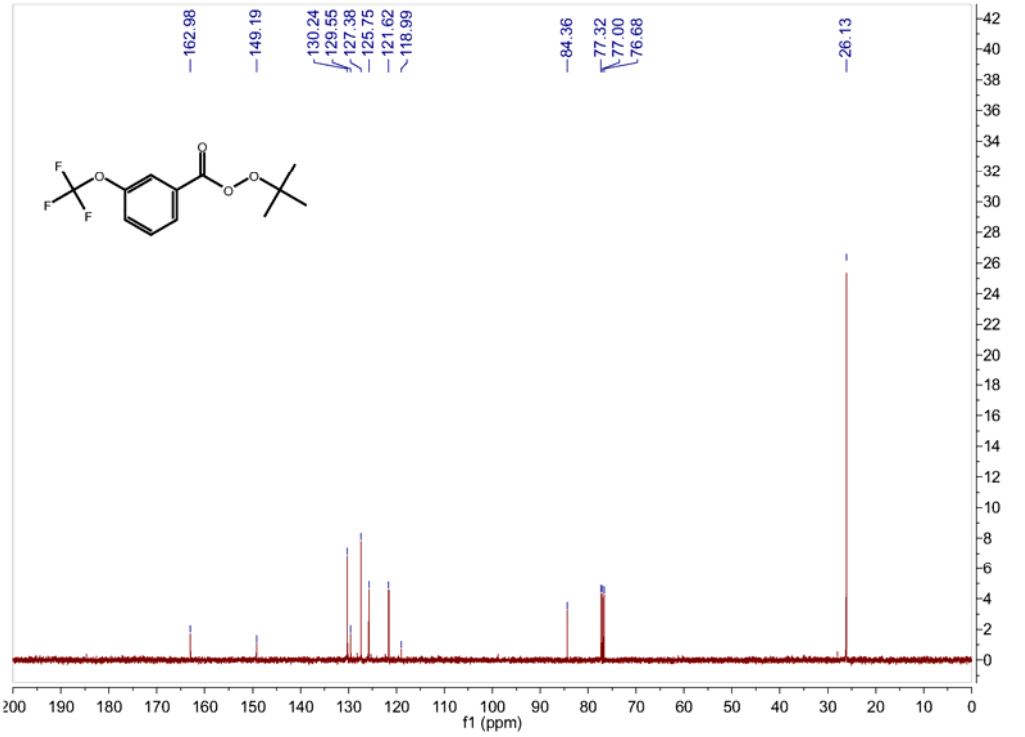
3o


7.69
7.67
7.60
7.59
7.42
7.40
7.38
7.38
7.36
7.34
7.21
7.21
7.19
7.19
7.19
7.16
7.14
7.12
7.03
7.01
1.39
0.00
1.02
1.00
3.07
2.07
2.07
9.05
f1 (ppm)

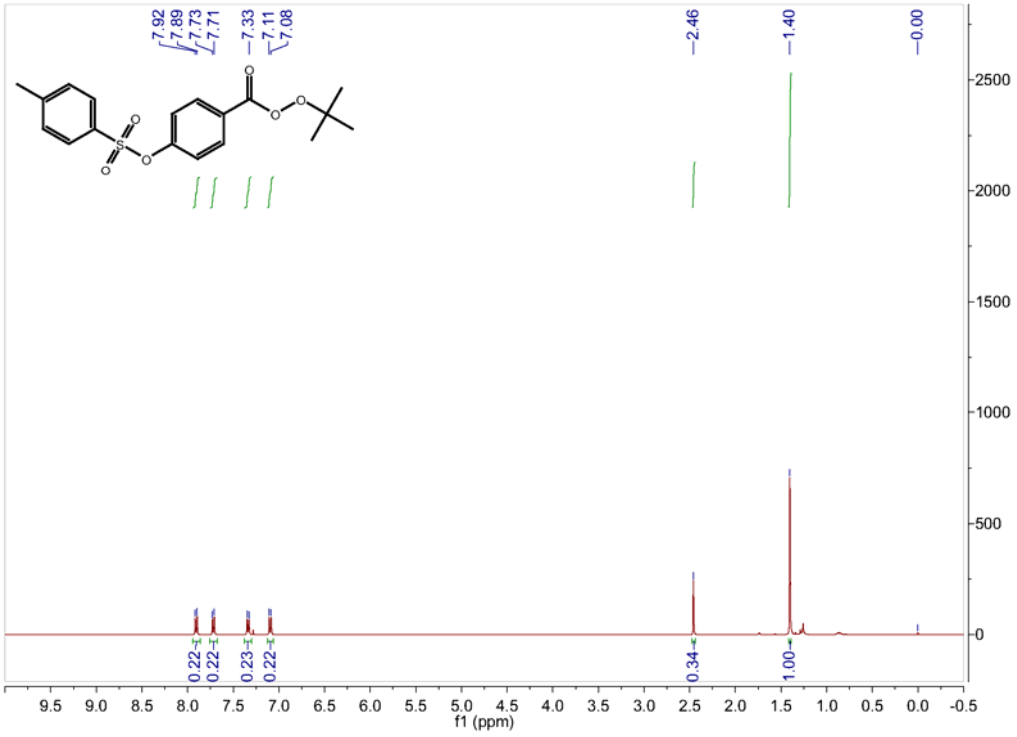

163.70
157.53
156.17
129.93
129.86
129.17
123.23
119.10
118.90
83.97
77.32
77.00
76.68
26.09
f1 (ppm)

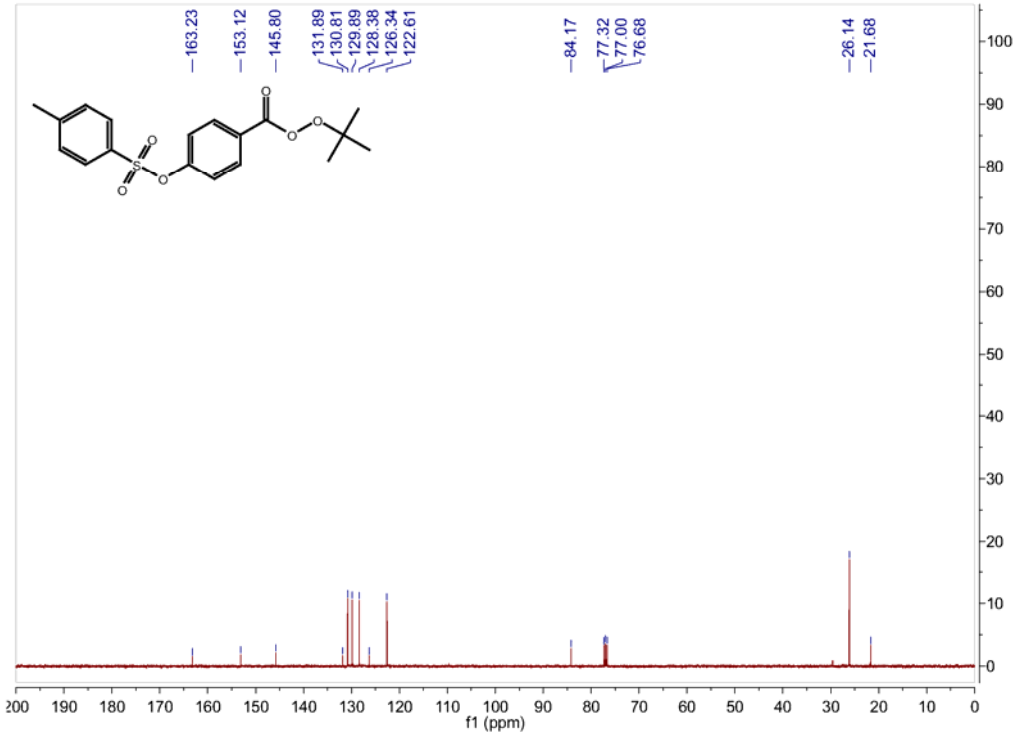
3p


7.92
7.90
7.80
7.55
7.53
7.51
7.47
7.45
1.43
0.00
0.11
0.11
0.12
0.11
1.00
f1 (ppm)

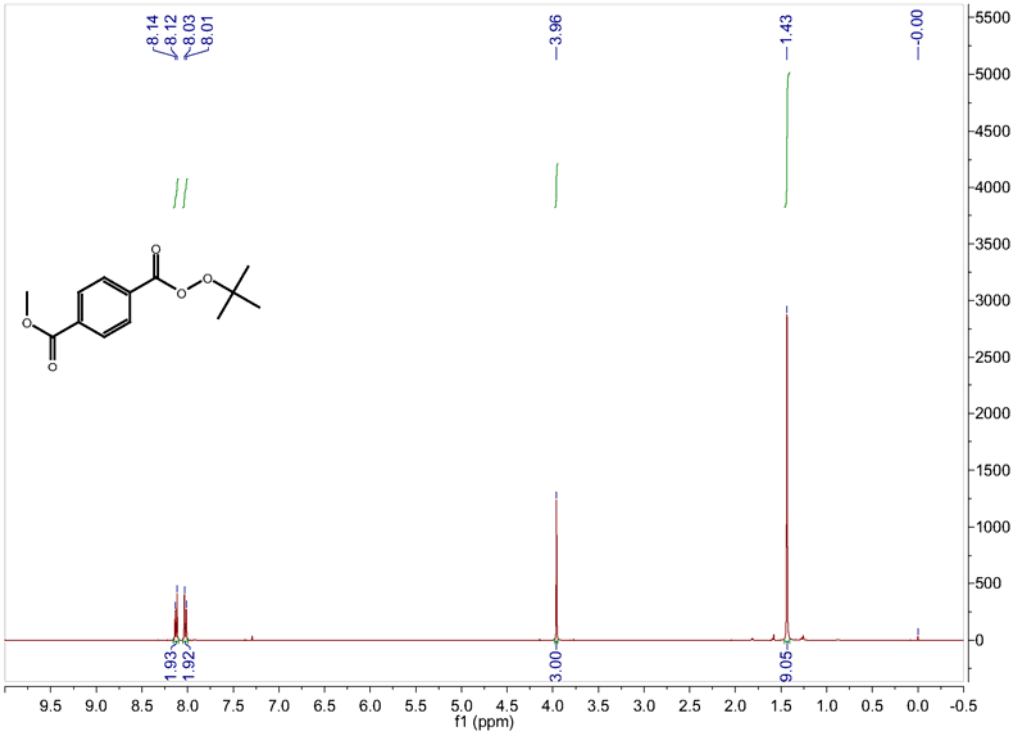

162.98
149.19
130.24
129.55
127.38
125.75
121.62
118.99
84.36
77.32
77.00
76.68
26.13
f1 (ppm)

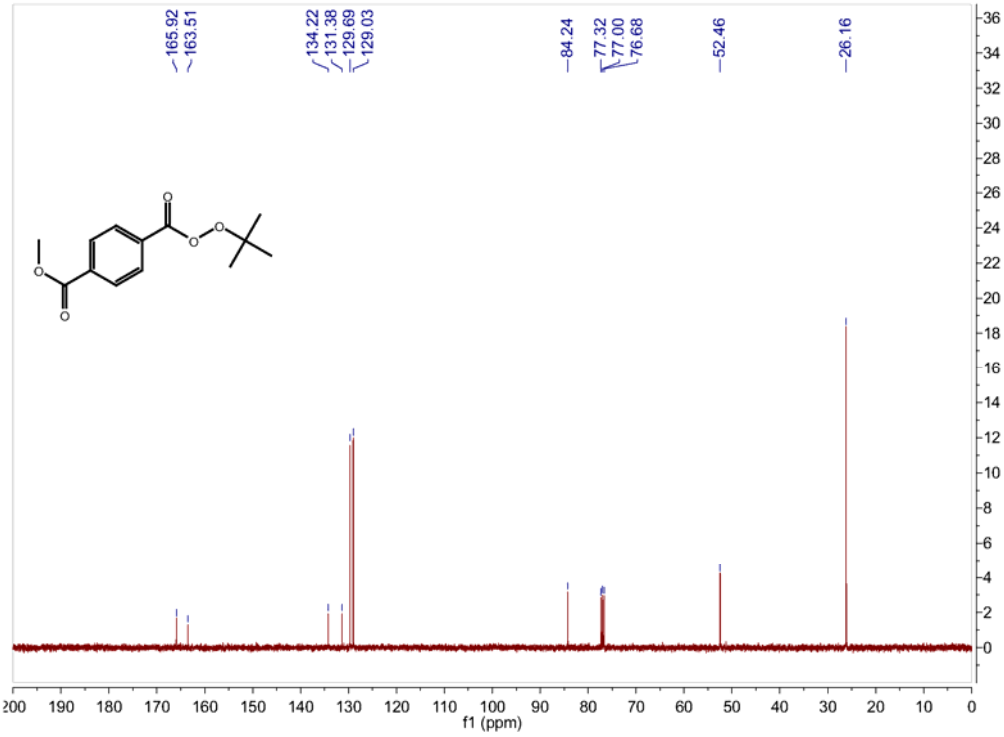
3q


7.92
7.89
7.73
7.71
7.33
7.11
7.08
2.46
1.40
0.00
0.22
0.22
0.23
0.22
0.34
1.00
f1 (ppm)

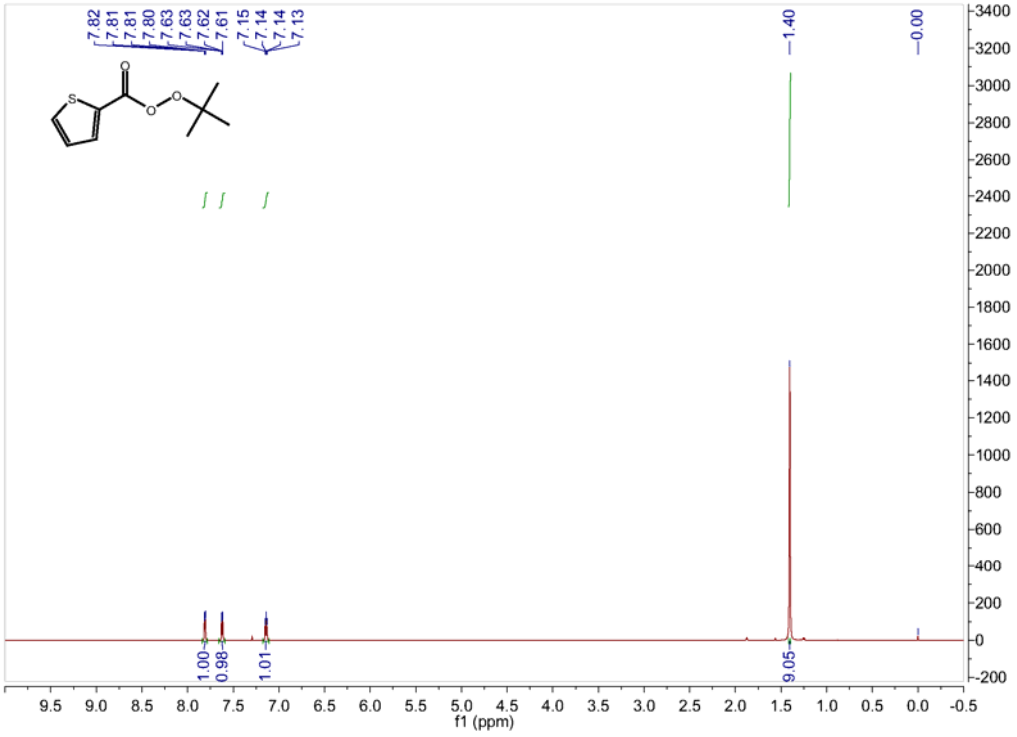

163.23
153.12
145.80
131.89
130.81
129.89
128.38
126.34
122.61
84.17
77.32
77.00
76.68
26.14
21.68
f1 (ppm)

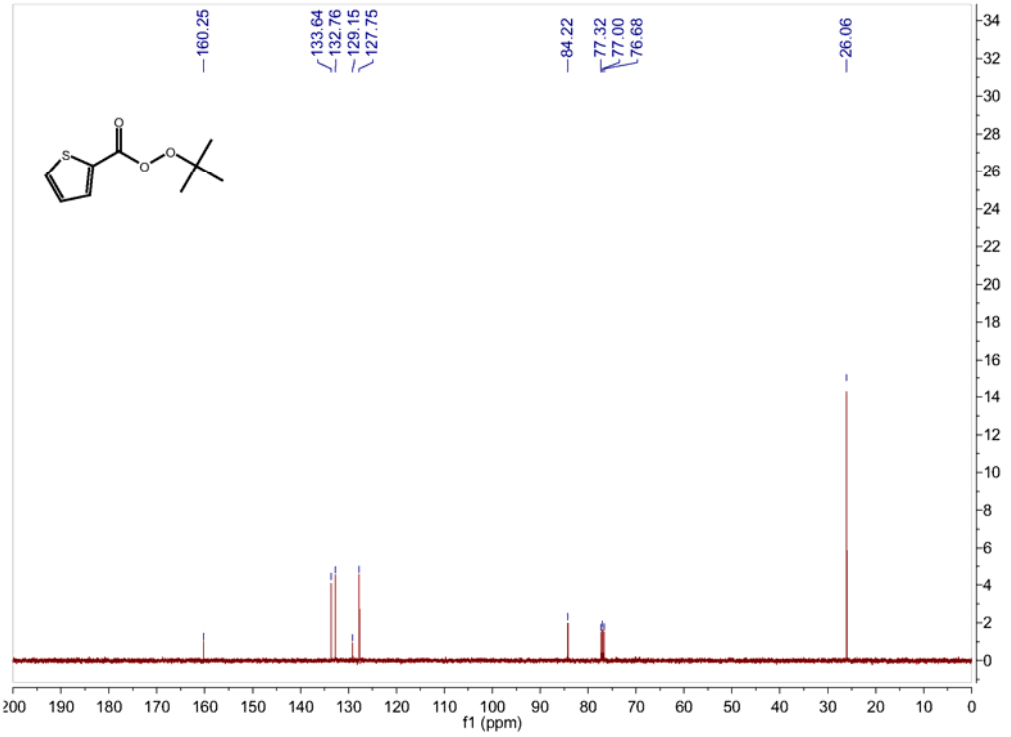
3r


8.14
8.12
8.03
8.01
3.96
1.43
0.00
1.93
1.92
3.00
9.05
f1 (ppm)

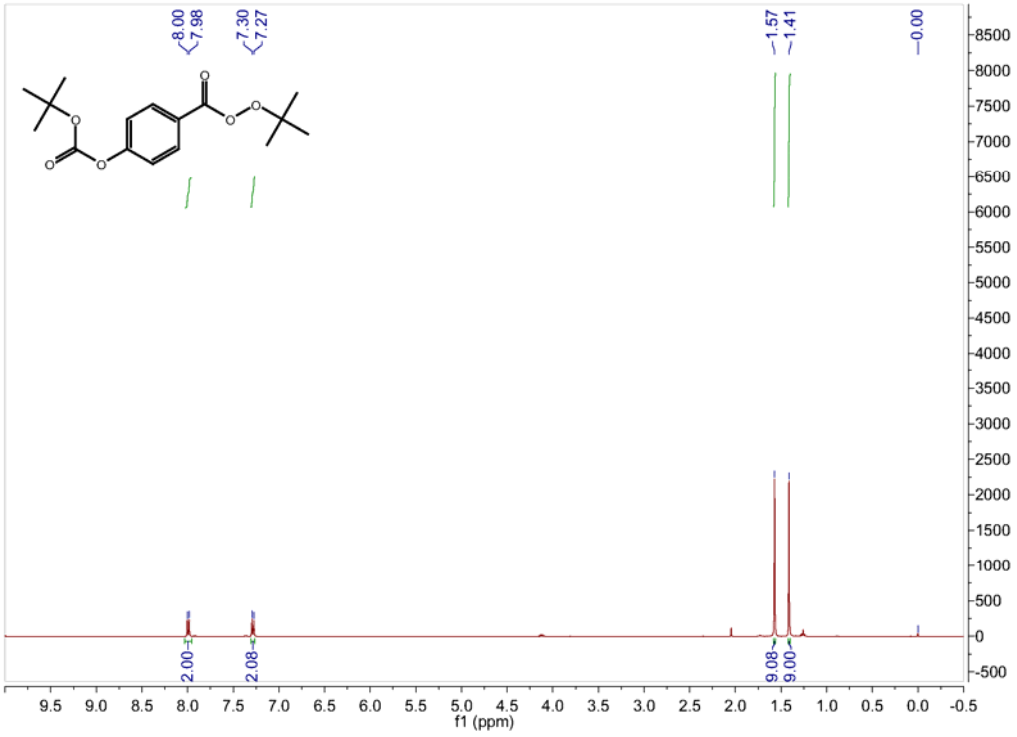

165.92
163.51
134.22
131.38
129.69
129.03
84.24
77.32
77.00
76.68
52.46
26.16
f1 (ppm)

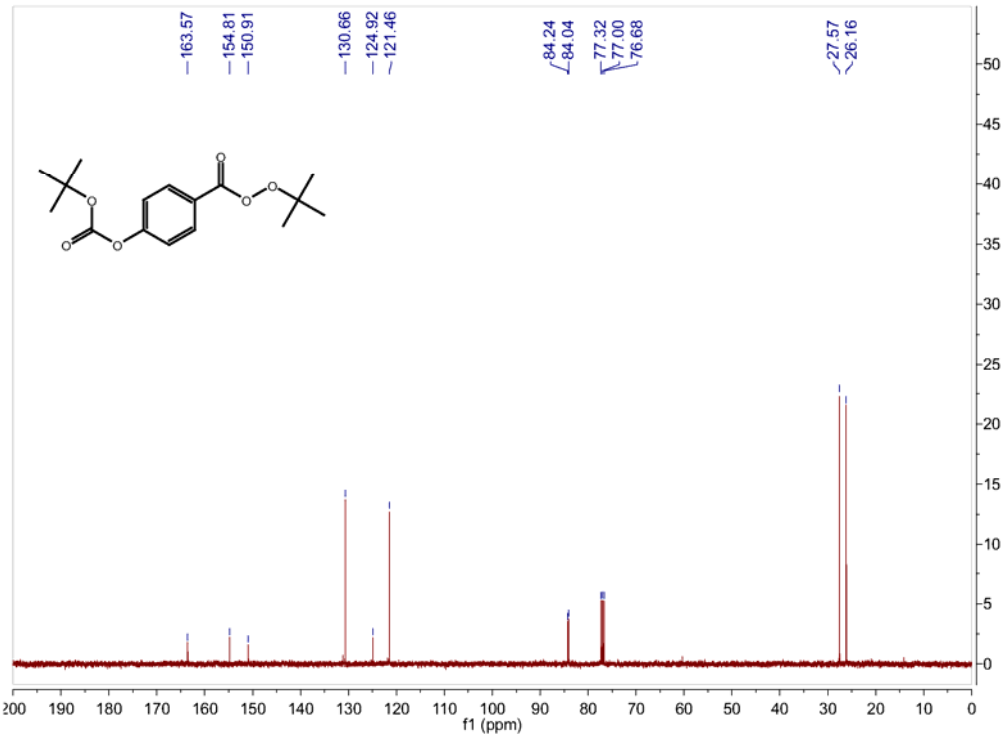
3s


7.82
7.81
7.81
7.80
7.63
7.63
7.62
7.61
7.15
7.14
7.14
7.13
1.40
0.00
1.00
0.98
1.01
9.05
f1 (ppm)

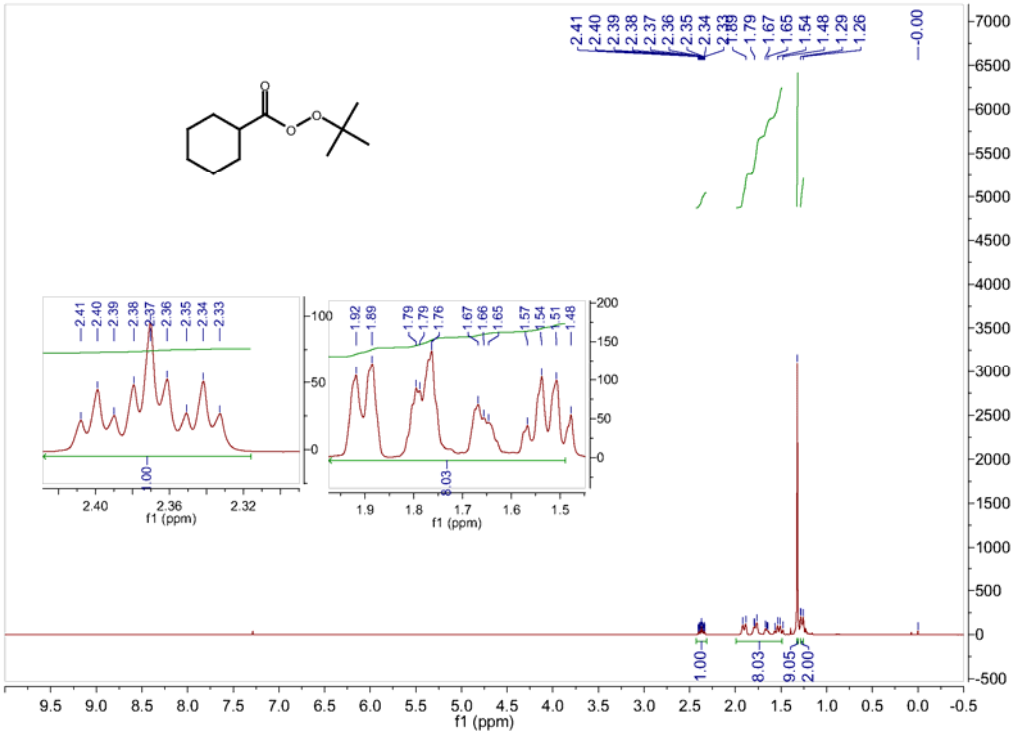

160.25
133.64
132.76
129.15
127.75
84.22
77.32
77.00
76.68
26.06
f1 (ppm)

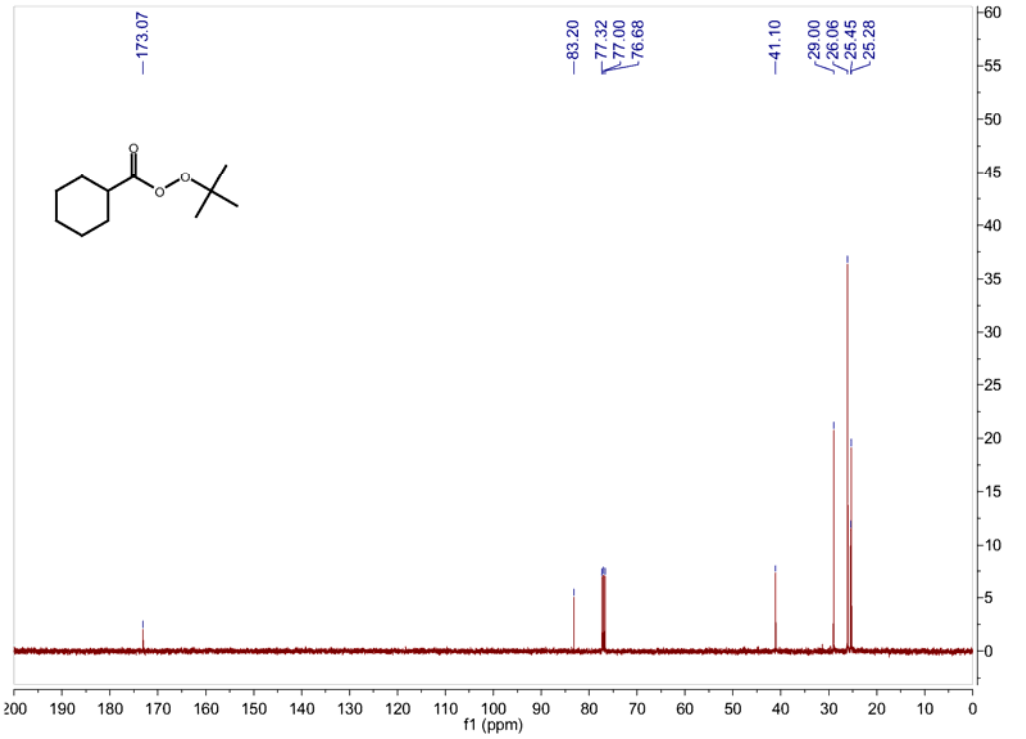
3t


8.00
7.98
7.30
7.27
1.57
1.41
0.00
2.00
2.08
9.08
9.00
f1 (ppm)

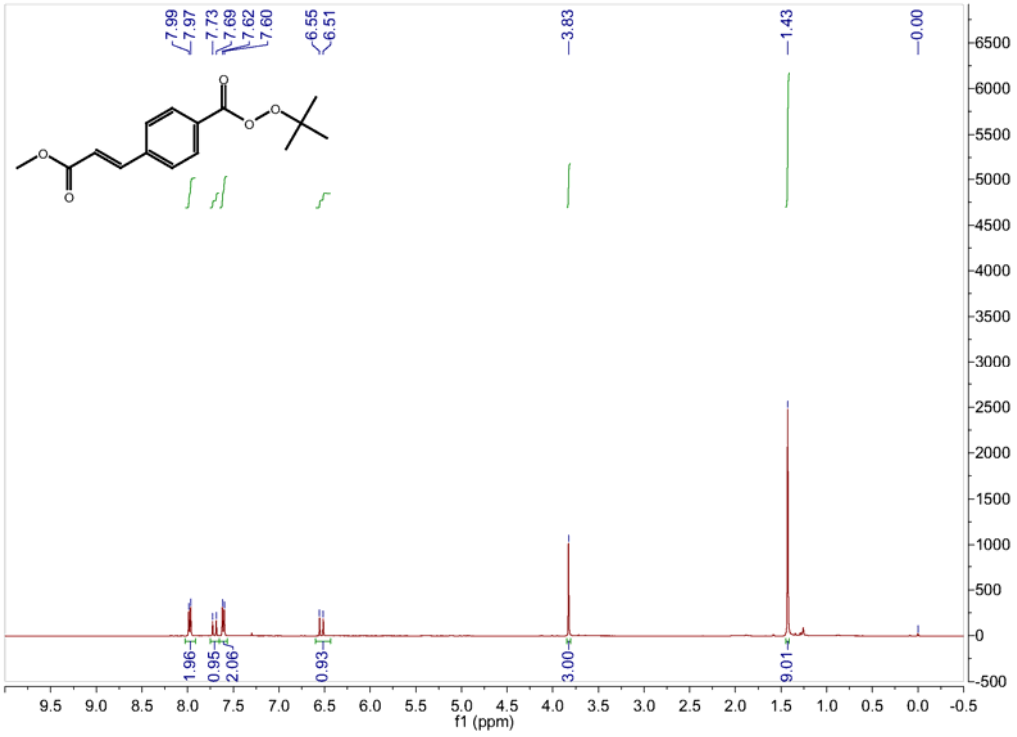

163.57
154.81
150.91
130.66
124.92
121.46
84.24
84.04
77.32
77.00
76.68
27.57
26.16
f1 (ppm)

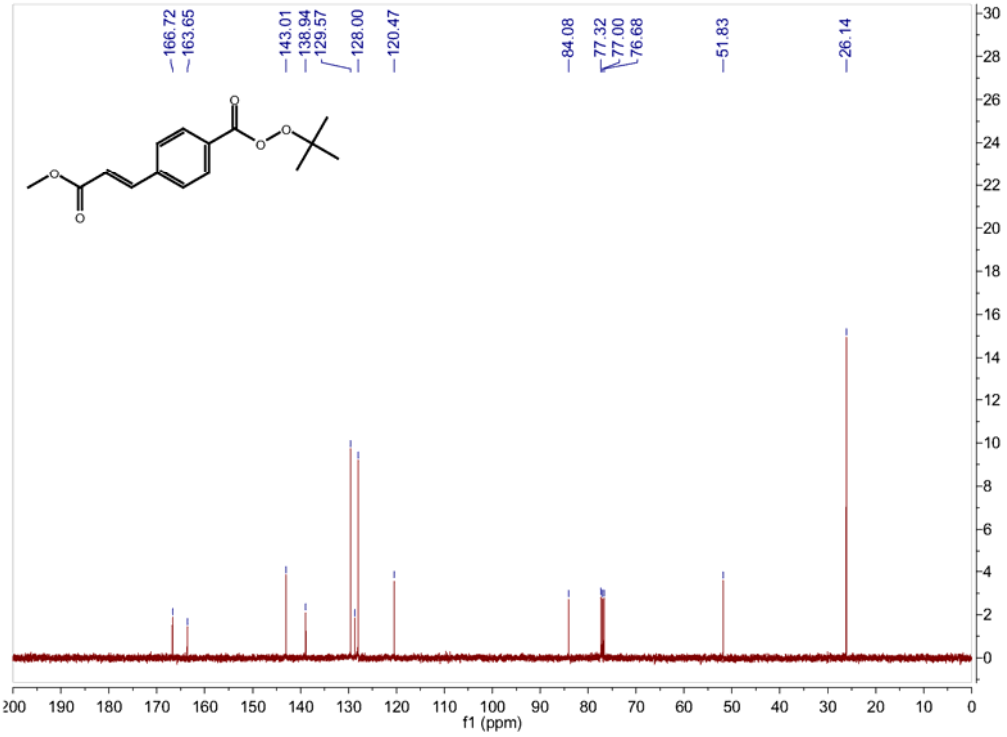
3u


2.41
2.40
2.39
2.38
2.37
2.36
2.35
2.34
1.89
1.79
1.67
1.65
1.54
1.48
1.29
1.26
0.00
1.00
8.03
9.05
2.00
f1 (ppm)

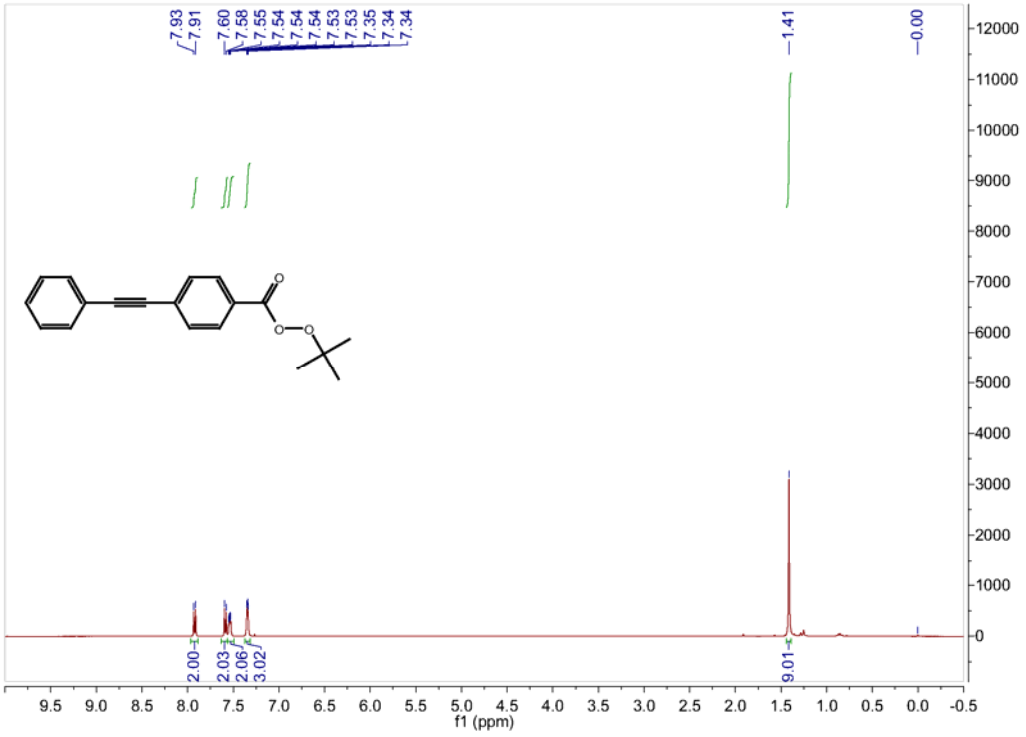

173.07
83.20
77.32
77.00
76.68
41.10
29.00
26.06
25.45
25.28
f1 (ppm)

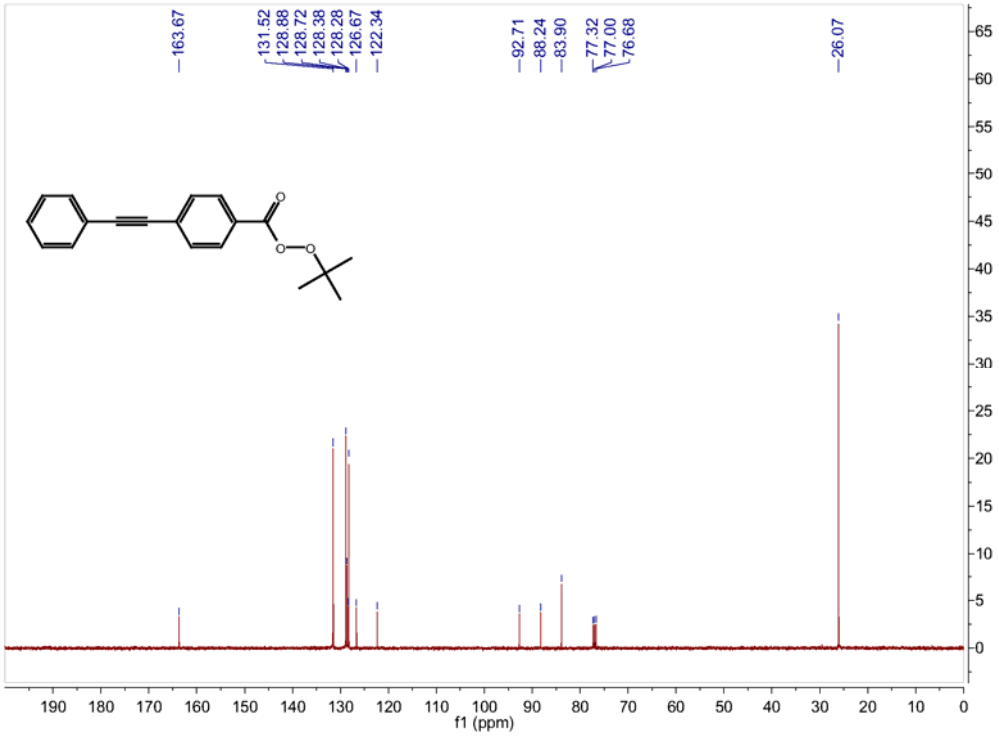
3v


7.99
7.97
7.73
7.69
7.62
7.60
6.55
6.51
3.83
1.43
0.00
1.96
0.95
2.06
0.93
3.00
9.01
f1 (ppm)

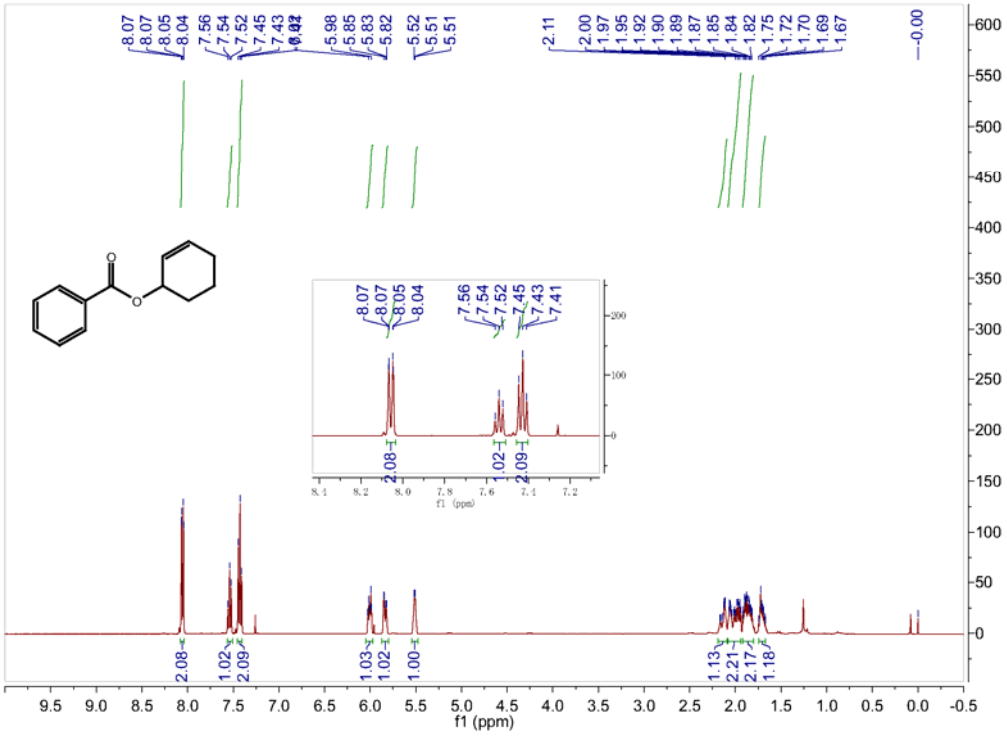

166.72
163.65
143.01
138.94
129.57
128.00
120.47
84.08
77.32
77.00
76.68
51.83
26.14
f1 (ppm)

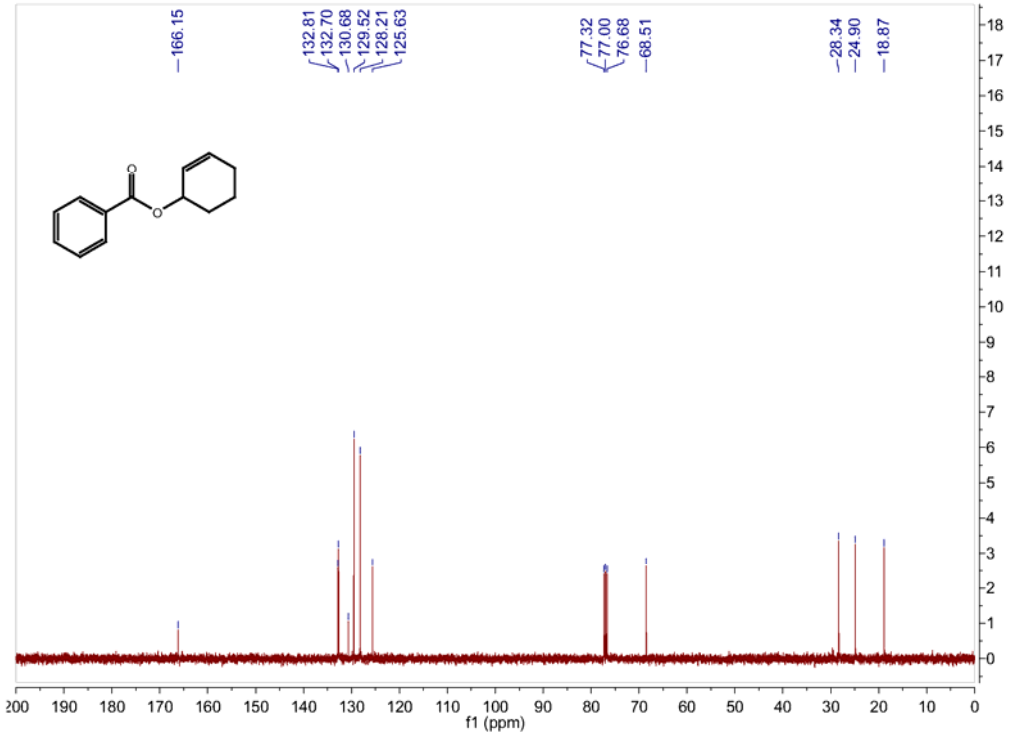
3w


7.93
7.91
7.60
7.58
7.55
7.54
7.54
7.54
7.53
7.53
7.35
7.34
7.34
1.41
0.00
2.00
2.03
2.06
3.02
9.01
f1 (ppm)

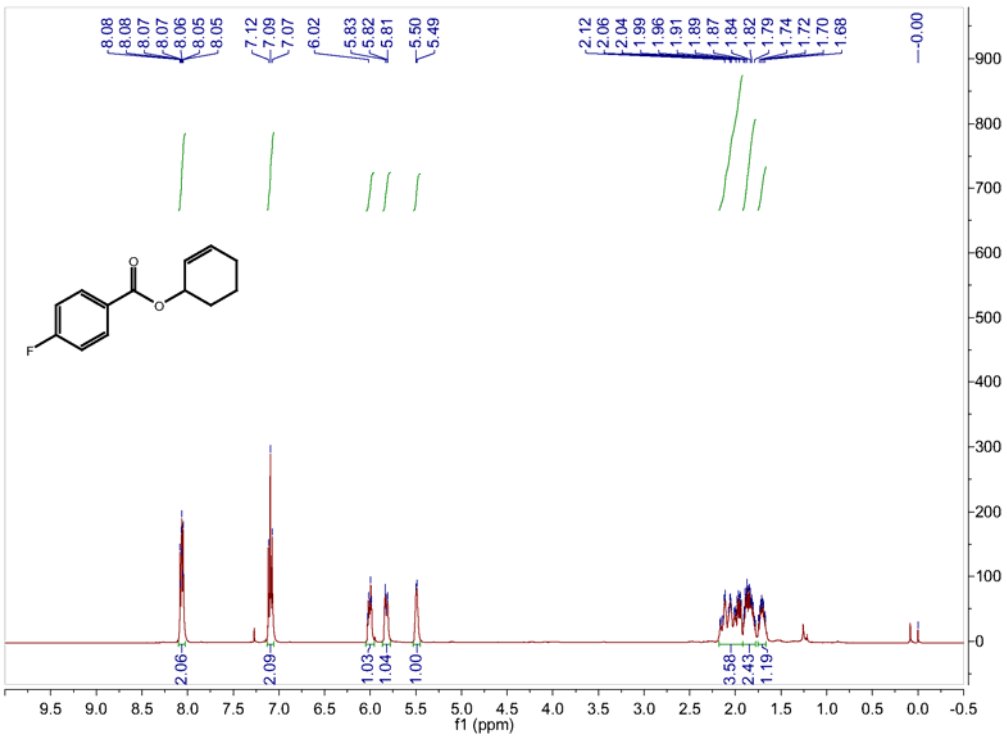

163.67
131.52
128.88
128.72
128.38
128.28
126.67
122.34
92.71
88.24
83.90
77.32
77.00
76.68
26.07
f1 (ppm)

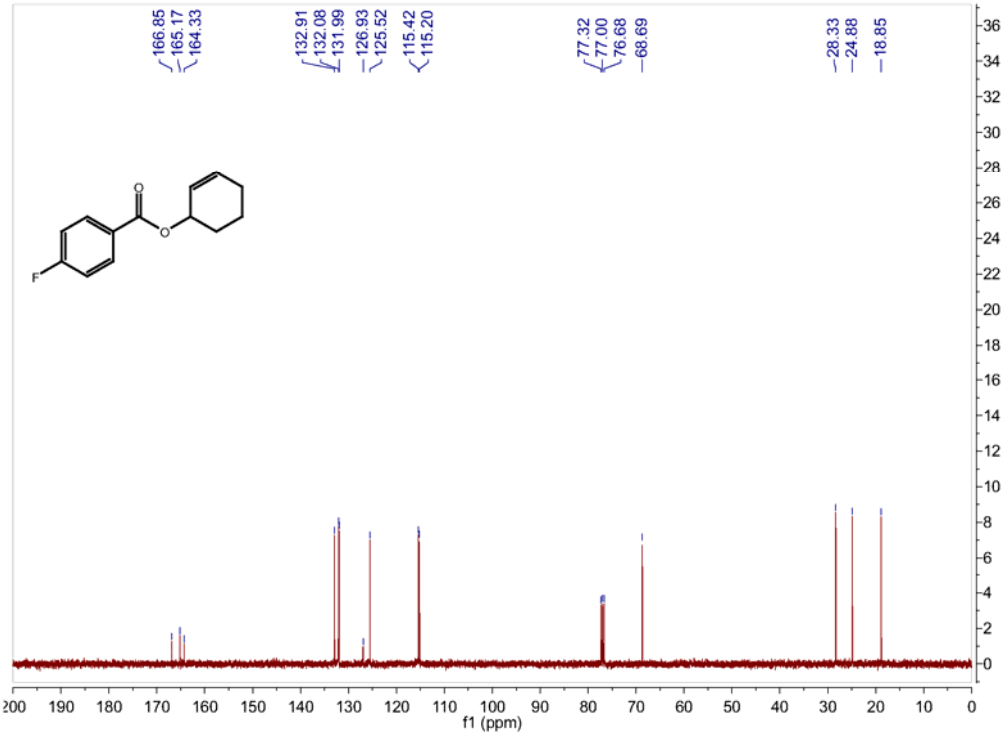
4a


8.07
8.07
8.05
8.04
7.56
7.54
7.52
7.45
7.43
5.98
5.85
5.83
5.82
5.52
5.51
5.51
2.11
2.00
1.97
1.95
1.92
1.90
1.89
1.87
1.85
1.84
1.82
1.75
1.72
1.70
1.69
1.67
0.00
2.08
1.02
2.09
1.03
1.02
1.00
1.13
2.21
2.17
1.18
f1 (ppm)

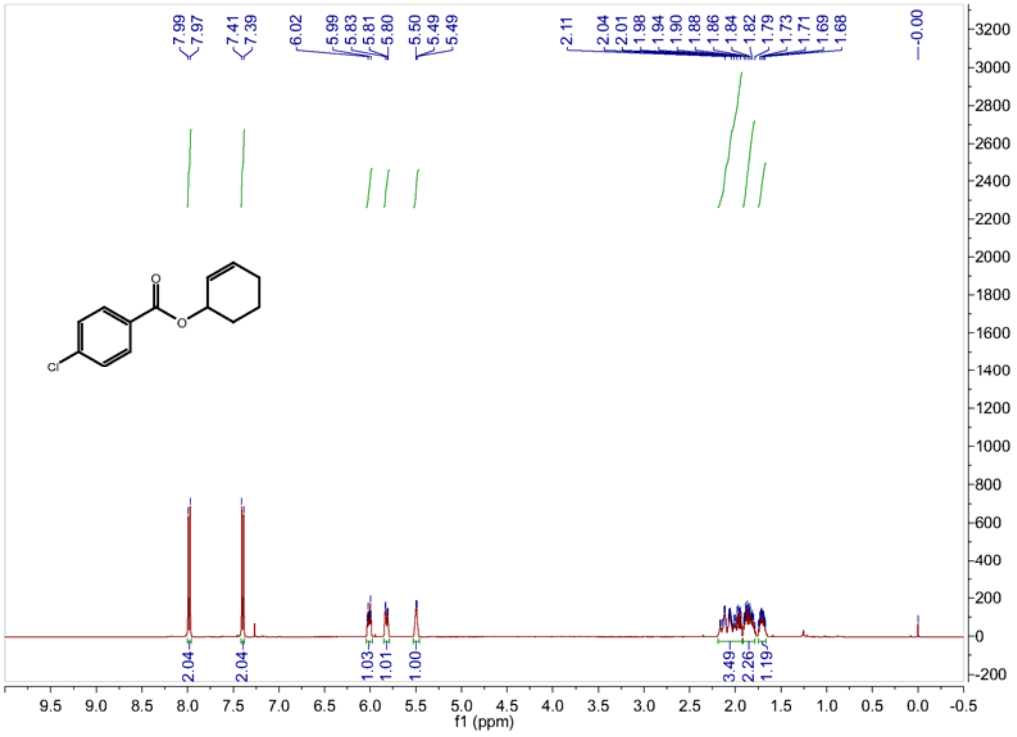

166.15
132.81
132.70
130.68
129.52
128.21
125.63
77.32
77.00
76.68
68.51
28.34
24.90
18.87
f1 (ppm)

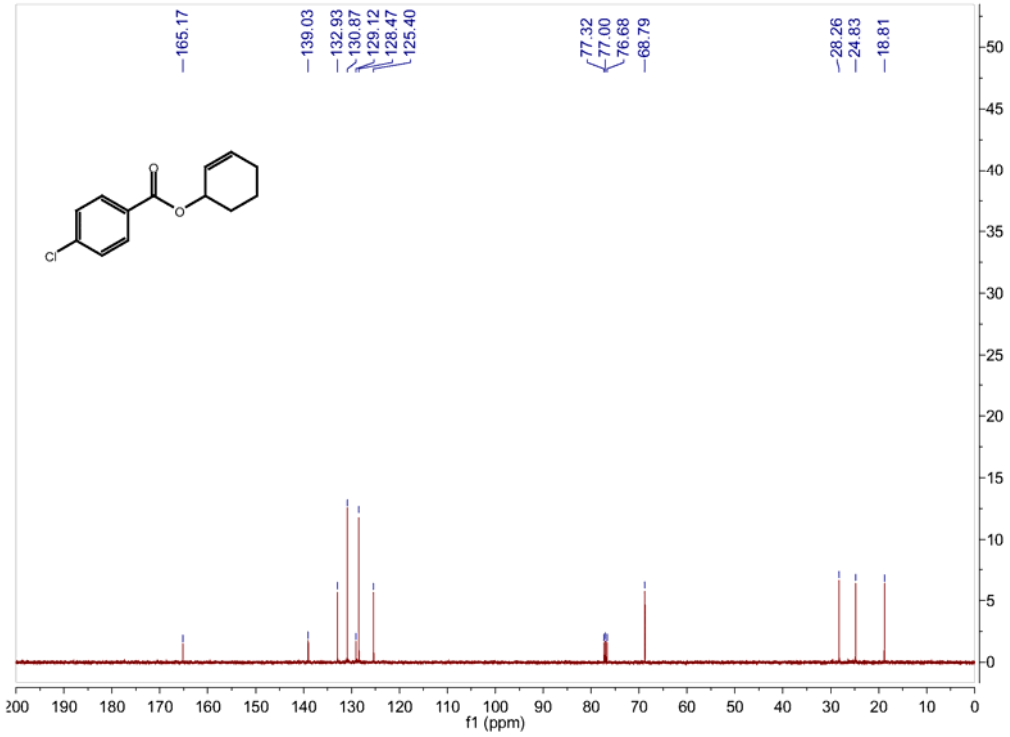
4b


8.08
8.08
8.07
8.07
8.06
8.05
8.05
7.12
7.09
7.07
6.02
5.83
5.82
5.81
5.50
5.49
2.12
2.06
2.04
1.99
1.96
1.91
1.89
1.87
1.84
1.82
1.79
1.74
1.72
1.70
1.68
0.00
2.06
2.09
1.03
1.04
1.00
3.58
2.43
1.19
f1 (ppm)

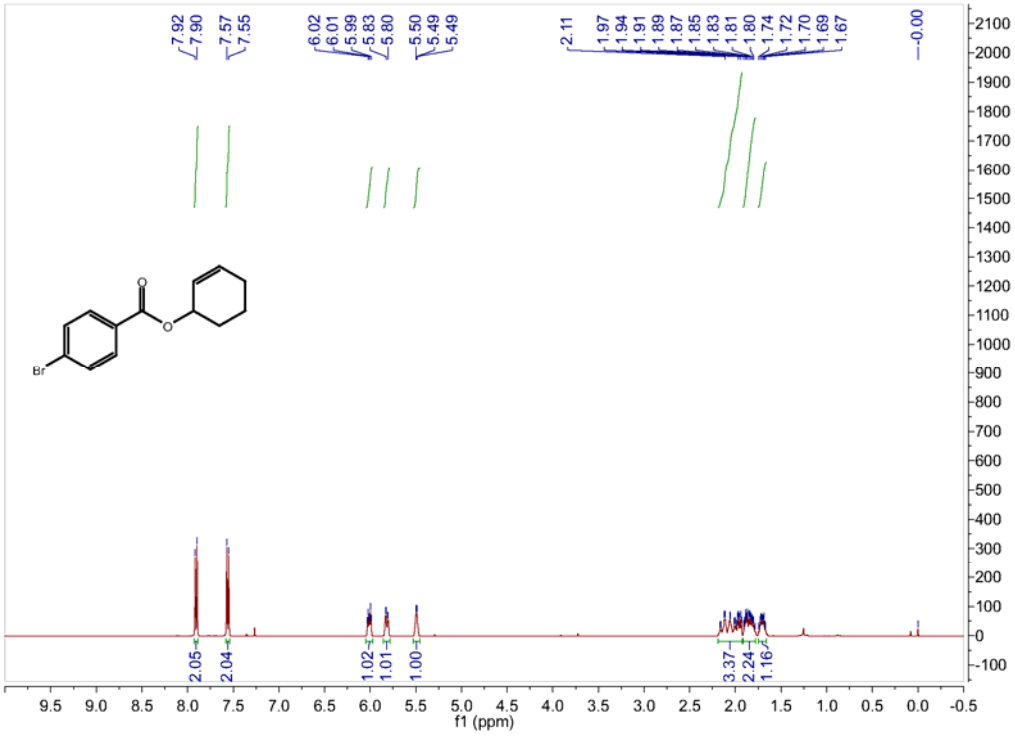

166.85
165.17
164.33
132.91
132.08
131.99
126.93
125.52
115.42
115.20
77.32
77.00
76.68
68.69
28.33
24.88
18.85
f1 (ppm)

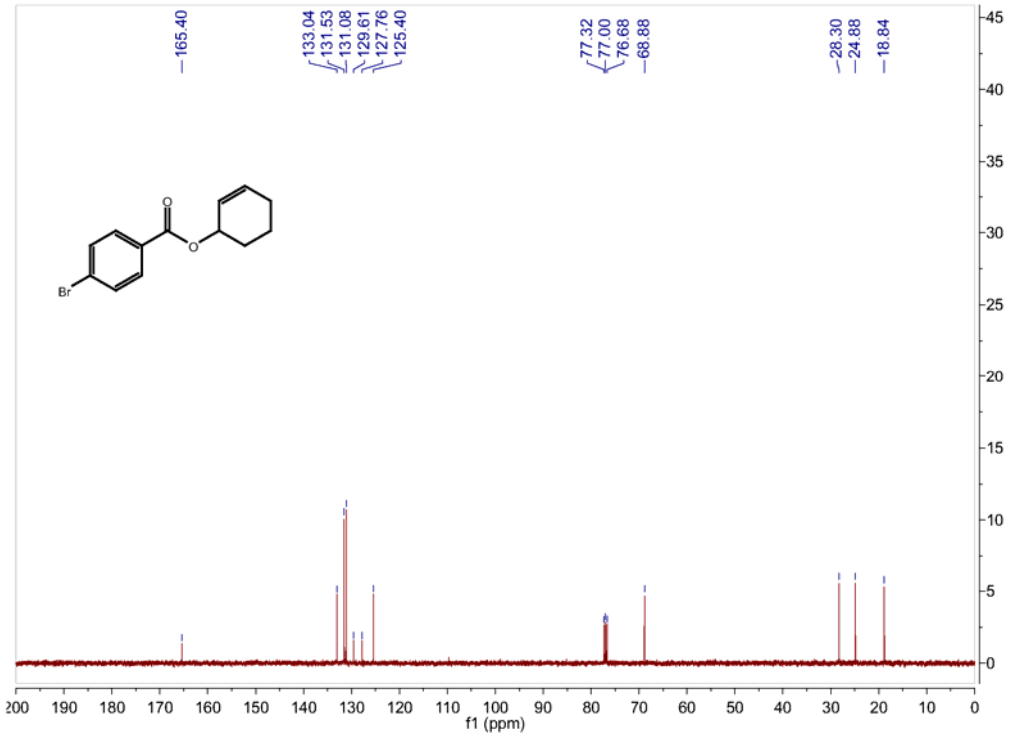
4c


7.99
7.97
7.41
7.39
6.02
5.99
5.83
5.81
5.80
5.50
5.49
5.49
2.11
2.04
2.01
1.98
1.94
1.90
1.88
1.86
1.84
1.82
1.79
1.73
1.71
1.69
1.68
0.00
2.04
2.04
1.03
1.01
1.00
3.49
2.26
1.19
f1 (ppm)

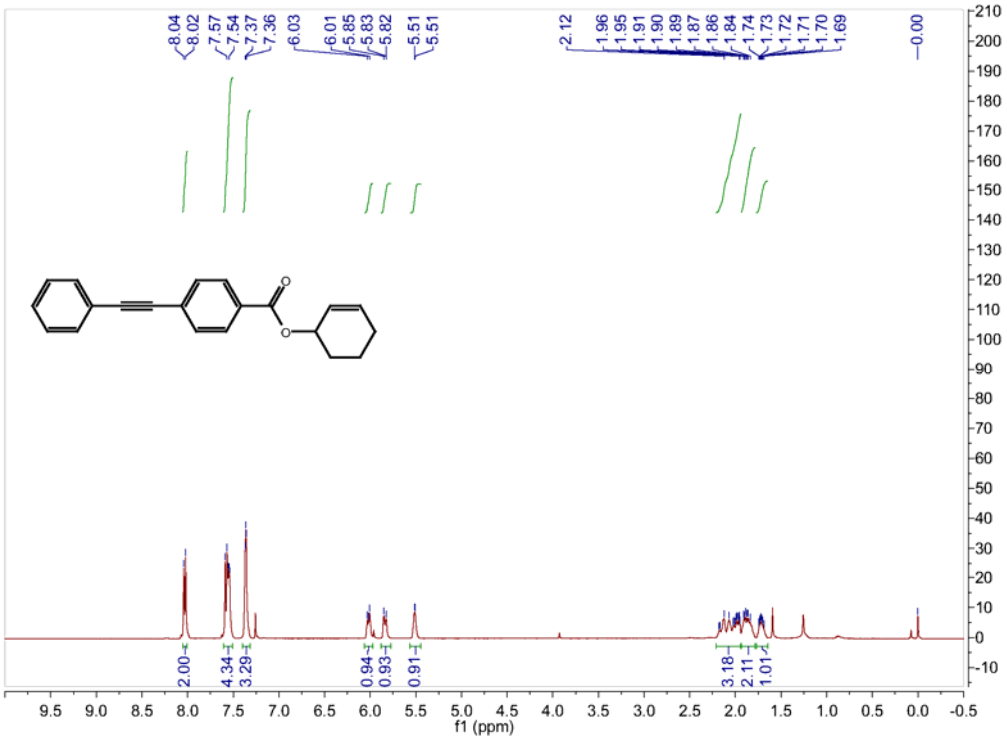

165.17
139.03
132.93
130.87
129.12
128.47
125.40
77.32
77.00
76.68
68.79
28.26
24.83
18.81
f1 (ppm)

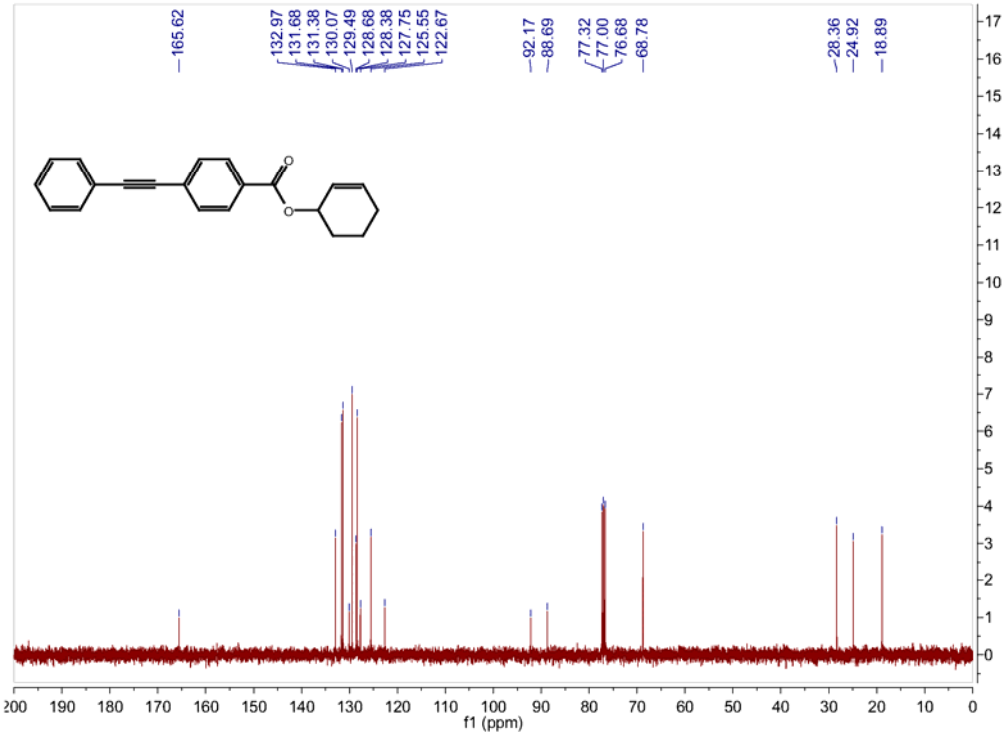
4d


7.92
7.90
7.57
7.55
6.02
6.01
5.99
5.83
5.80
5.50
5.49
5.49
2.11
1.97
1.94
1.91
1.89
1.87
1.85
1.83
1.81
1.80
1.74
1.72
1.70
1.69
1.67
0.00
2.05
2.04
1.02
1.01
1.00
3.37
2.24
1.16
Br
f1 (ppm)

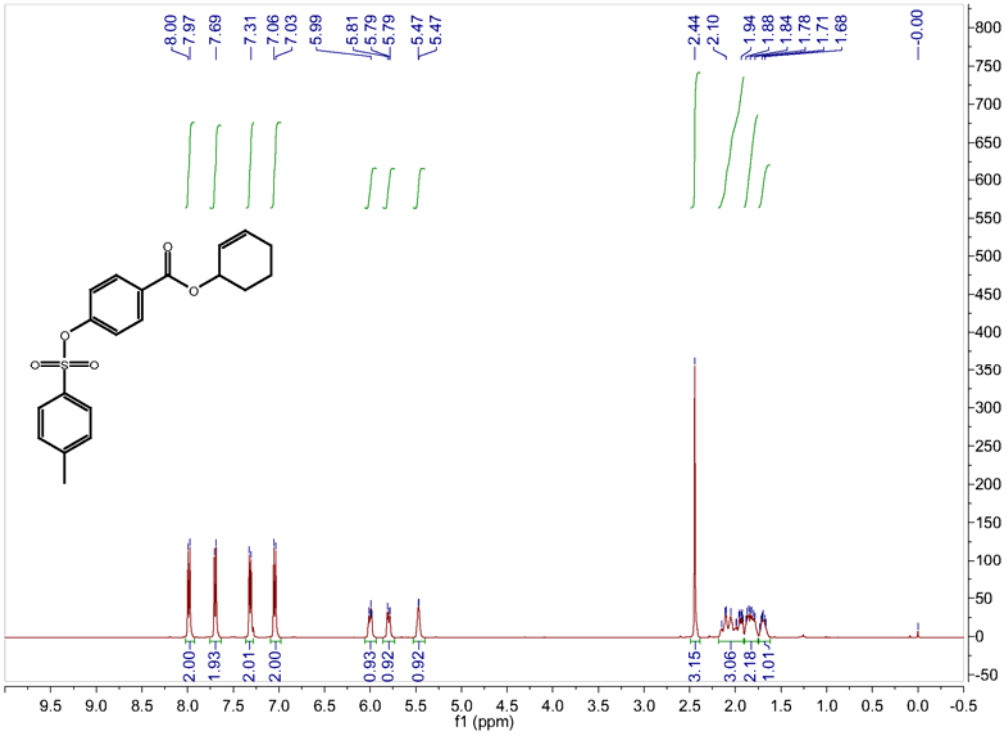

165.40
133.04
131.53
131.08
129.61
127.76
125.40
77.32
77.00
76.68
68.88
28.30
24.88
18.84
Br
f1 (ppm)

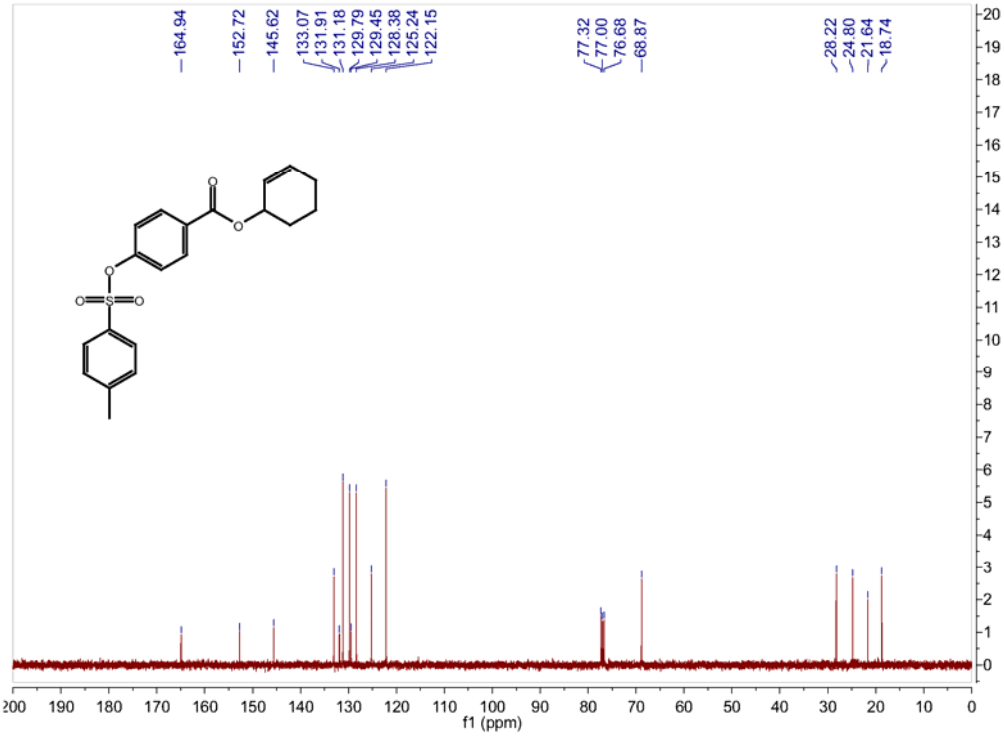
4e


8.04
8.02
7.57
7.54
7.37
7.36
6.03
6.01
5.85
5.83
5.82
5.51
5.51
2.12
1.96
1.95
1.91
1.90
1.89
1.87
1.86
1.84
1.74
1.73
1.72
1.71
1.70
1.69
0.00
2.00
4.34
3.29
0.94
0.93
0.91
3.18
2.11
1.01
f1 (ppm)

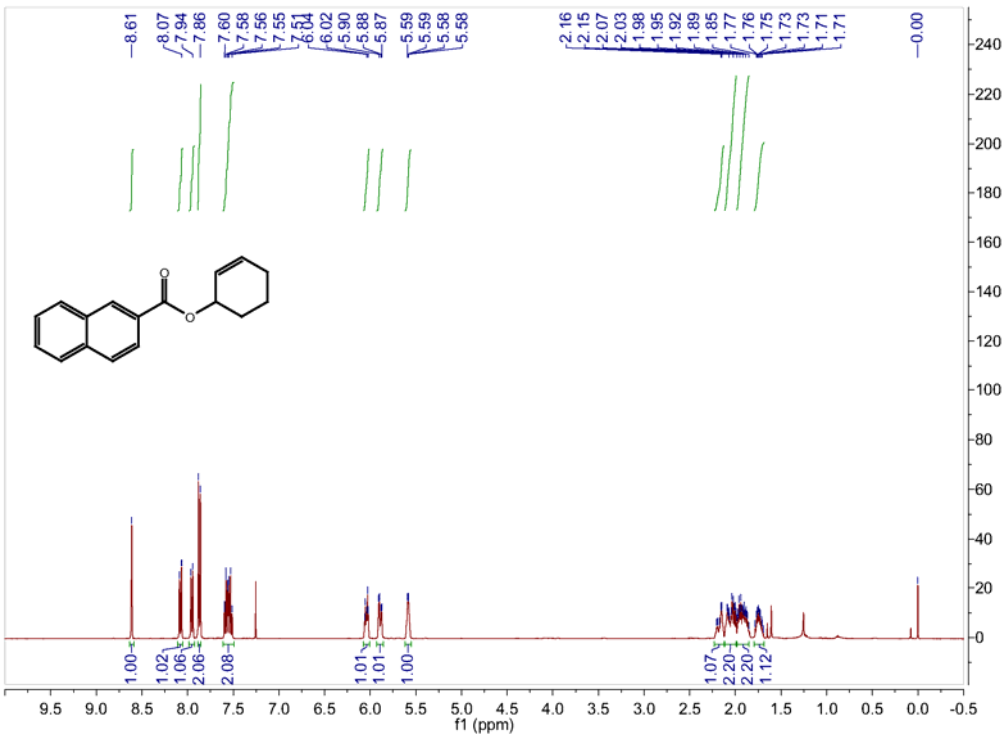

165.62
132.97
131.68
131.38
130.07
129.49
128.68
128.38
127.75
125.55
122.67
92.17
88.69
77.32
77.00
76.68
68.78
28.36
24.92
18.89
f1 (ppm)

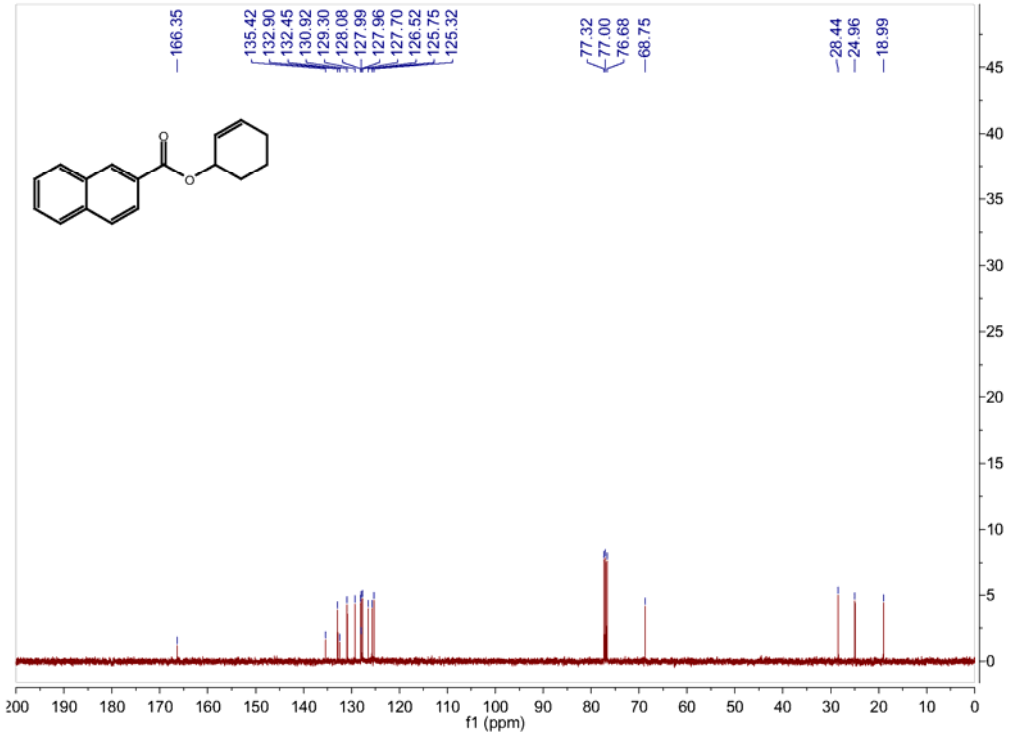
4f


8.00
7.97
7.69
7.31
7.06
7.03
5.99
5.81
5.79
5.79
5.47
5.47
2.44
2.10
1.94
1.88
1.84
1.78
1.71
1.68
0.00
2.00
1.93
2.01
2.00
0.93
0.92
0.92
3.15
3.06
2.18
1.01
f1 (ppm)

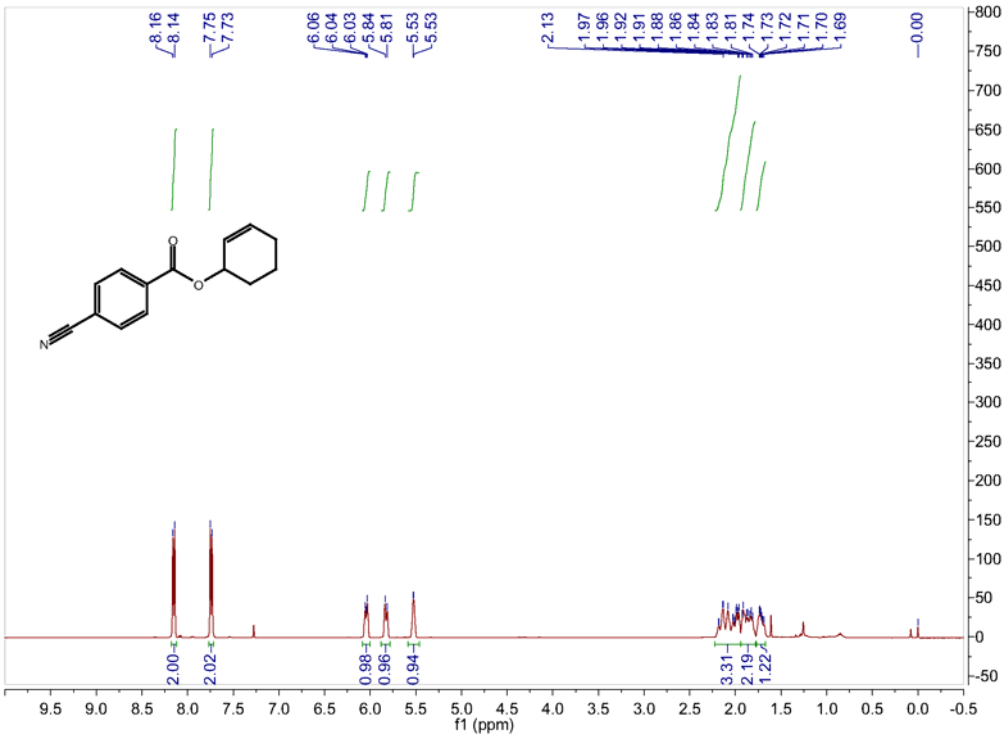

164.94
152.72
145.62
133.07
131.91
131.18
129.79
129.45
128.38
125.24
122.15
77.32
77.00
76.68
68.87
28.22
24.80
21.64
18.74
f1 (ppm)

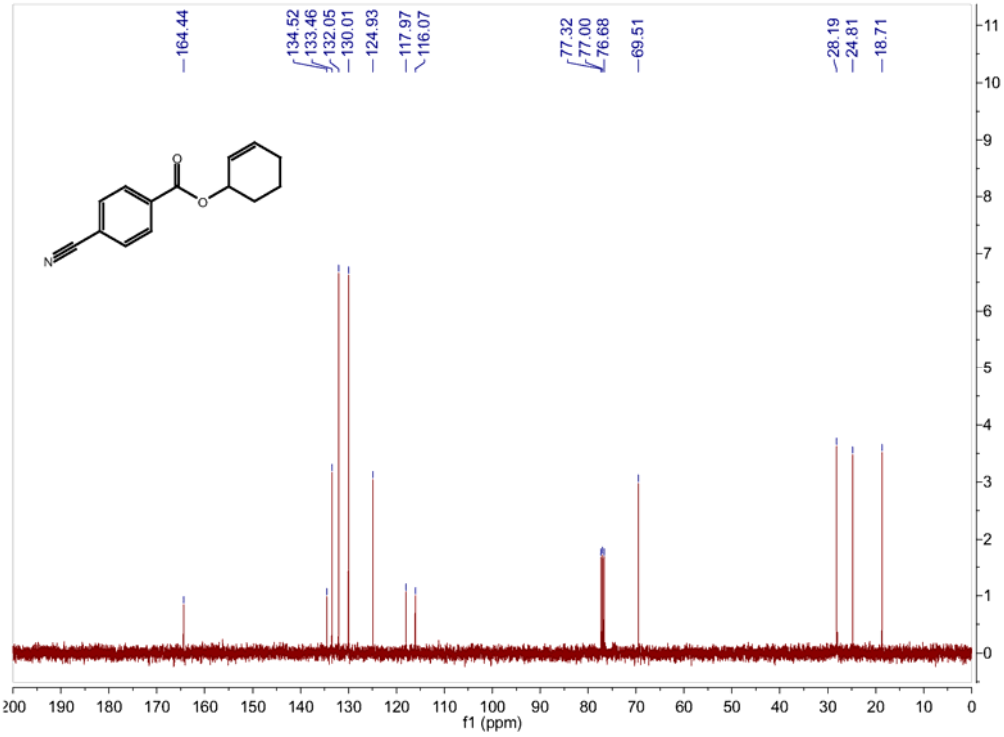
4g


8.61
8.07
7.94
7.86
7.60
7.58
7.56
7.55
7.51
6.04
6.02
5.90
5.88
5.87
5.59
5.59
5.58
5.58
2.16
2.15
2.07
2.03
1.98
1.95
1.92
1.89
1.85
1.77
1.76
1.75
1.73
1.73
1.71
1.71
0.00
1.00
1.02
1.06
2.06
2.08
1.01
1.01
1.00
1.07
2.20
2.20
1.12
f1 (ppm)

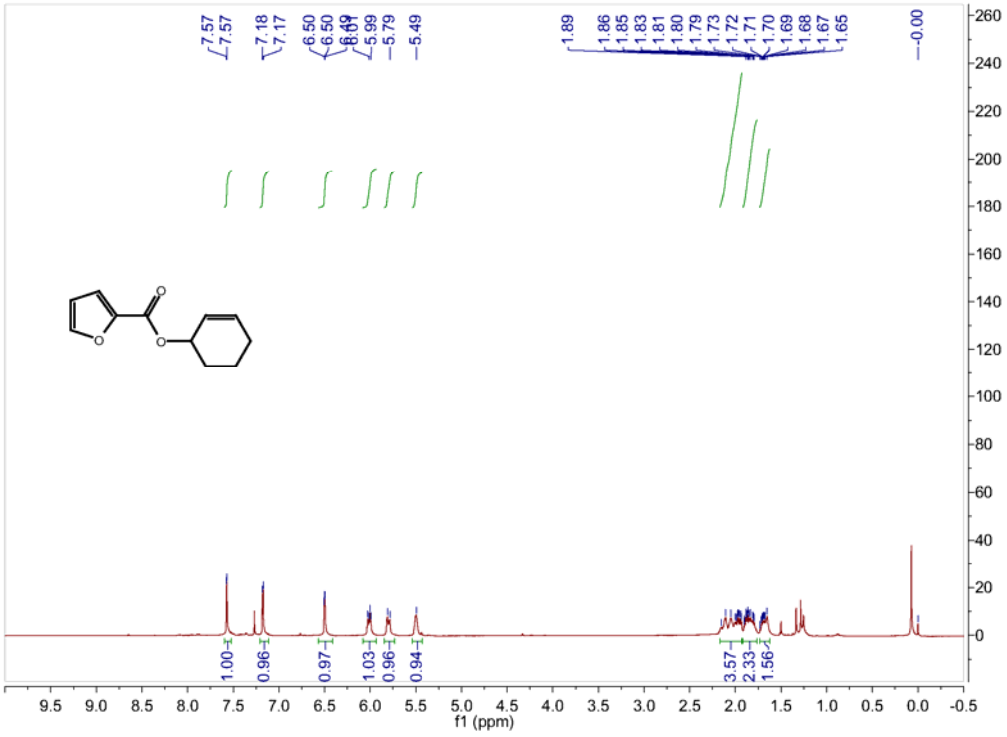

166.35
135.42
132.90
132.45
130.92
129.30
128.08
127.99
127.96
127.70
126.52
125.75
125.32
77.32
77.00
76.68
68.75
28.44
24.96
18.99
f1 (ppm)

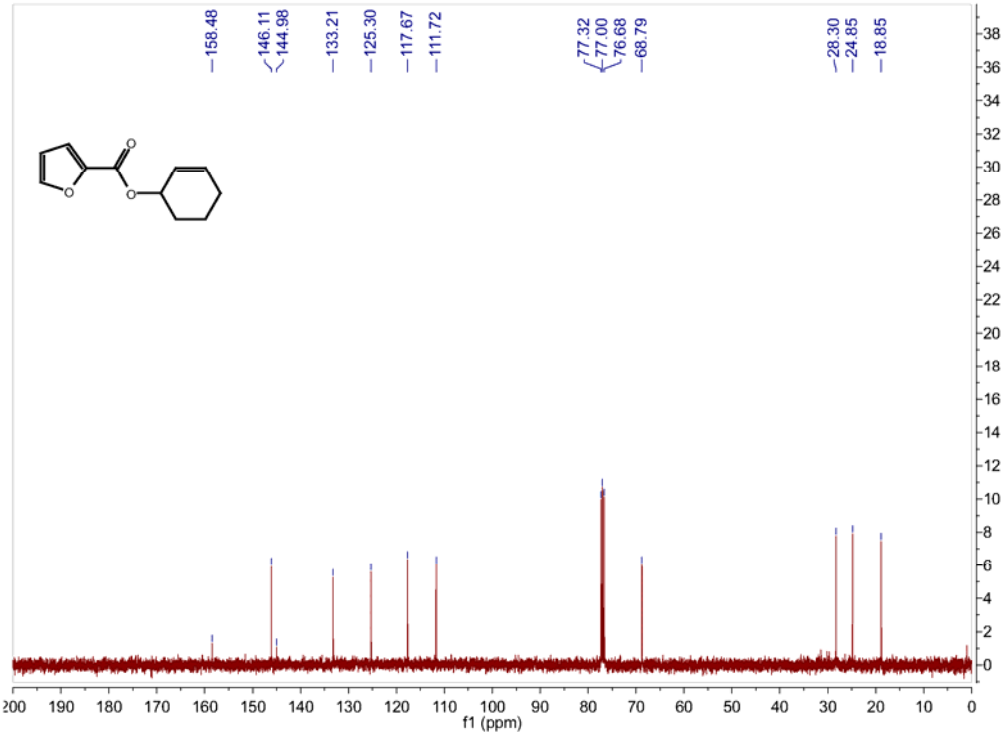
4h


8.16
8.14
7.75
7.73
6.06
6.04
6.03
5.84
5.81
5.53
5.53
2.13
1.97
1.96
1.92
1.91
1.88
1.86
1.84
1.83
1.81
1.74
1.73
1.72
1.71
1.70
1.69
0.00
2.00
2.02
0.98
0.96
0.94
3.31
2.19
1.22
f1 (ppm)

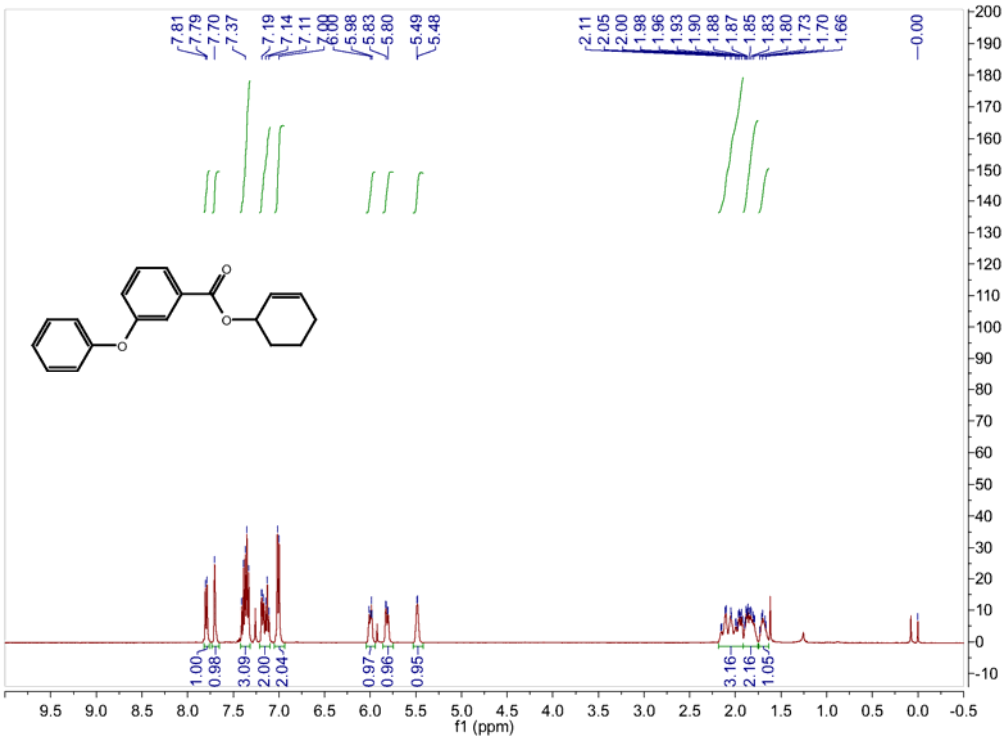

164.44
134.52
133.46
132.05
130.01
124.93
117.97
116.07
77.32
77.00
76.68
69.51
28.19
24.81
18.71
f1 (ppm)

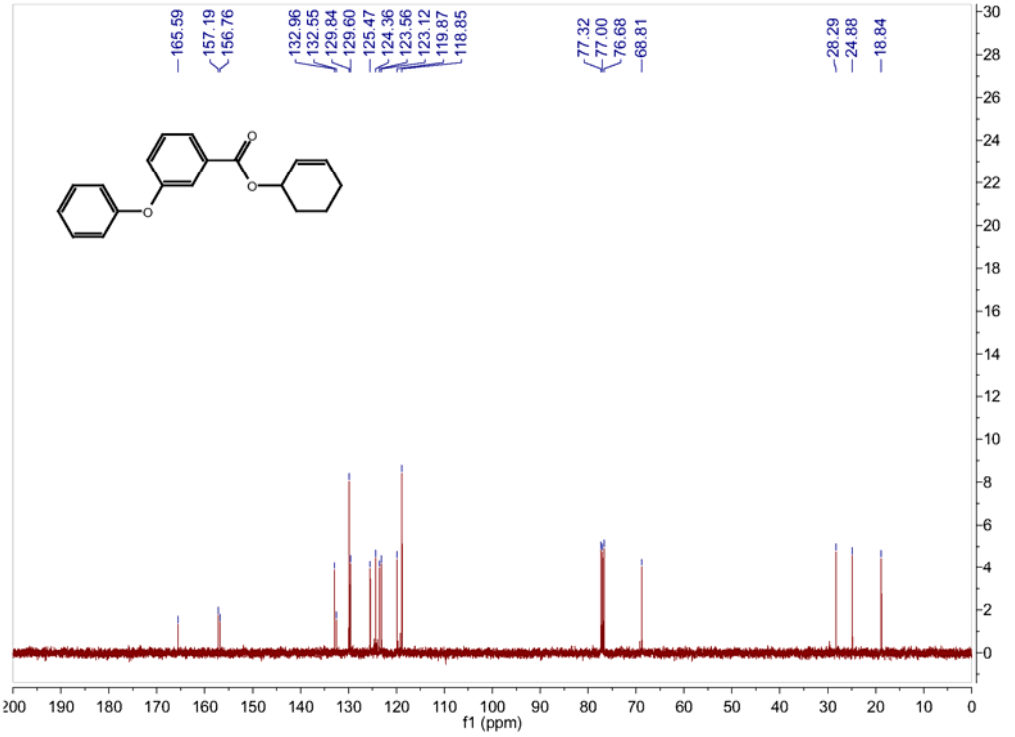
4i


7.57
7.57
7.18
7.17
6.50
6.50
5.99
5.79
5.49
1.89
1.86
1.85
1.83
1.81
1.80
1.79
1.73
1.72
1.71
1.70
1.69
1.68
1.67
1.65
0.00
1.00
0.96
0.97
1.03
0.96
0.94
3.57
2.33
1.56
f1 (ppm)

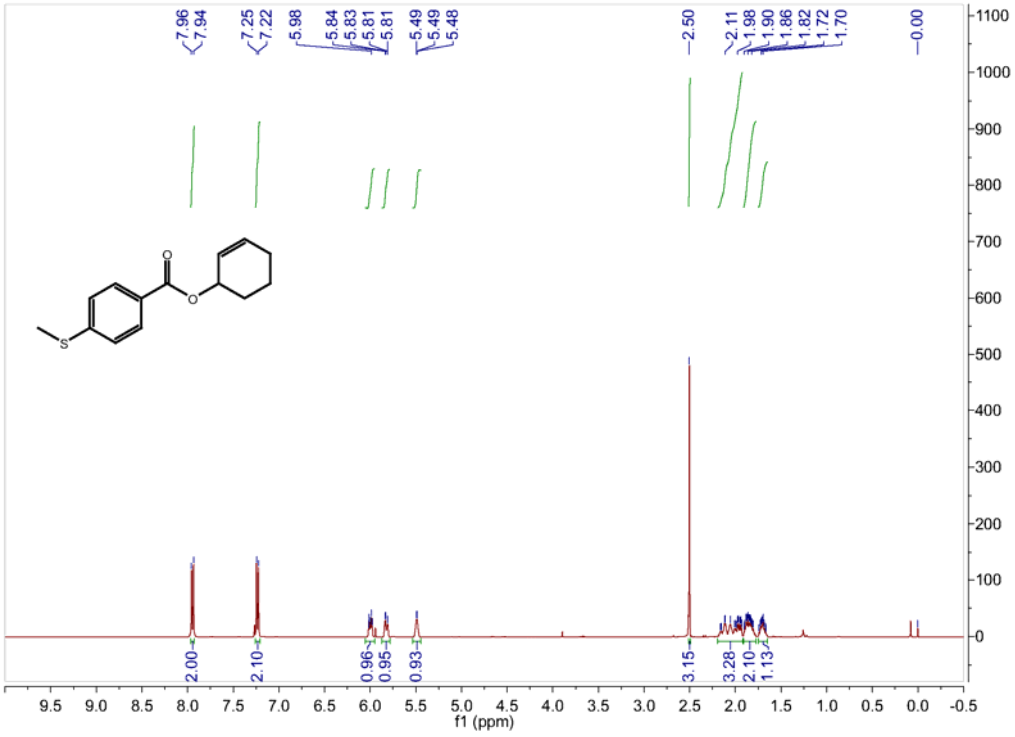

158.48
146.11
144.98
133.21
125.30
117.67
111.72
77.32
77.00
76.68
68.79
28.30
24.85
18.85
f1 (ppm)

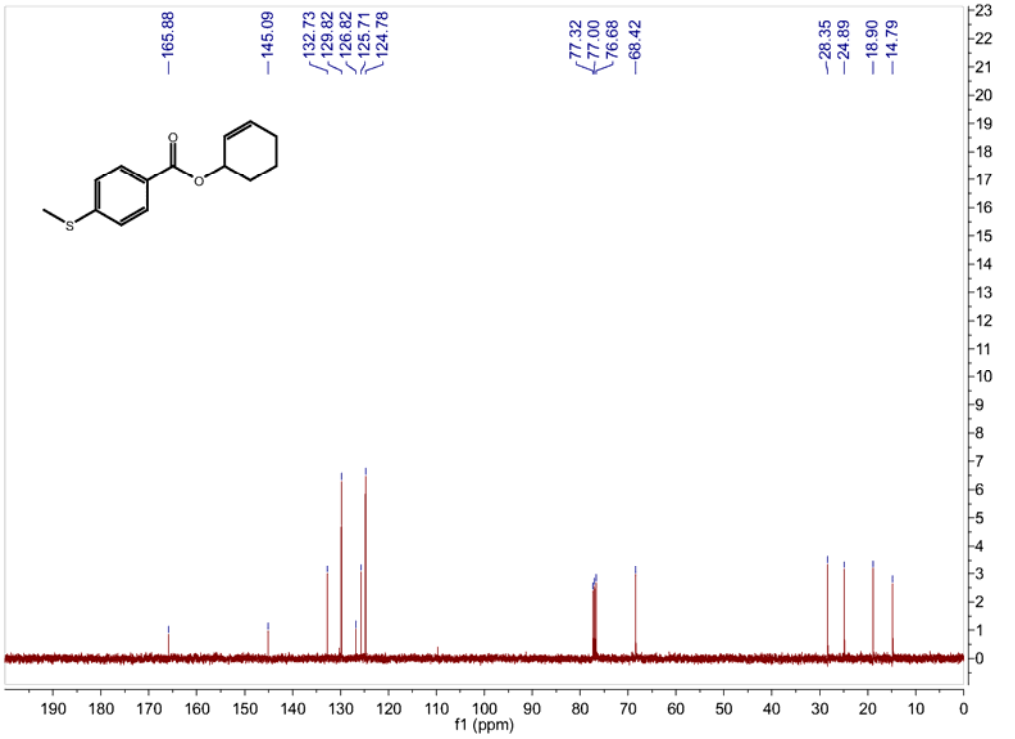
4j


7.81
7.79
7.70
7.37
7.19
7.14
7.11
7.00
6.98
5.83
5.80
5.49
5.48
2.11
2.05
2.00
1.98
1.96
1.93
1.90
1.88
1.87
1.85
1.83
1.80
1.73
1.70
1.66
0.00
1.00
0.98
3.09
2.00
2.04
0.97
0.96
0.95
3.16
2.16
1.05
f1 (ppm)

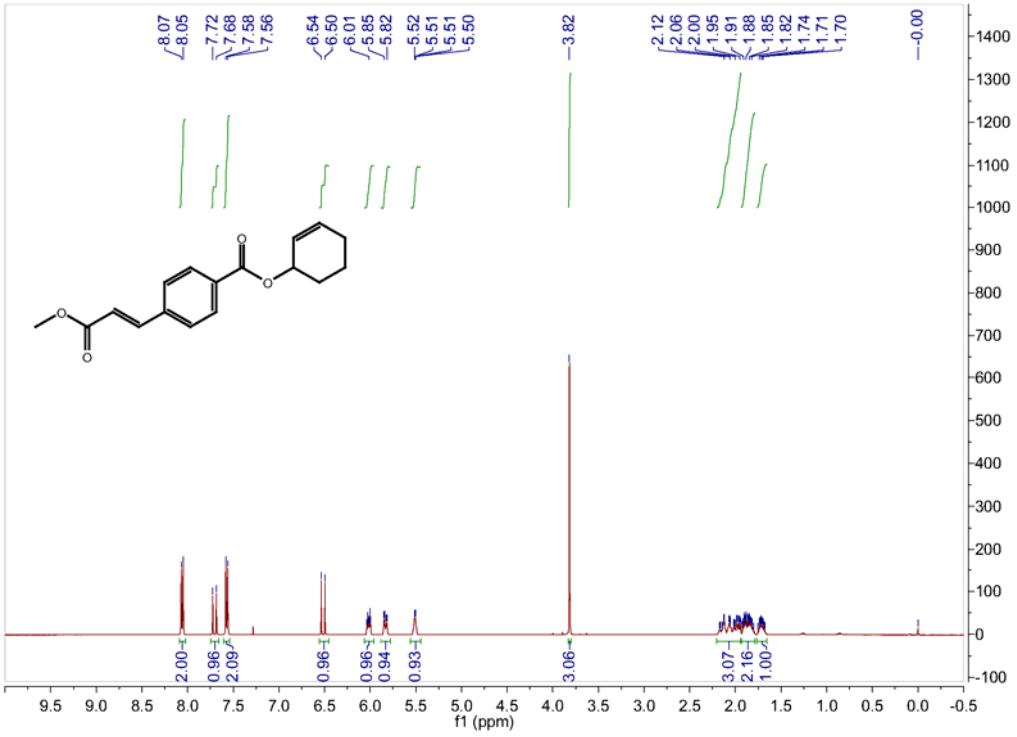

165.59
157.19
156.76
132.96
132.55
129.84
129.60
125.47
124.36
123.56
123.12
119.87
118.85
77.32
77.00
76.68
68.81
28.29
24.88
18.84
f1 (ppm)

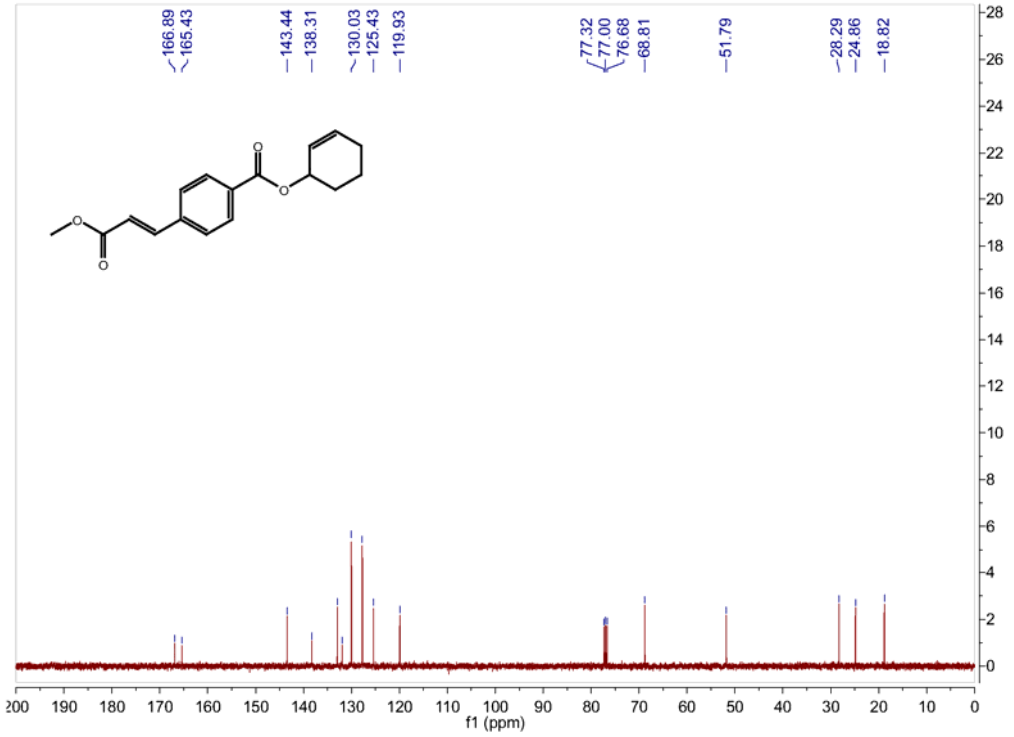
4k


7.96
7.94
7.25
7.22
5.98
5.84
5.83
5.81
5.81
5.49
5.49
5.48
2.50
2.11
1.98
1.90
1.86
1.82
1.72
1.70
0.00
2.00
2.10
0.96
0.95
0.93
3.15
3.28
2.10
1.13
f1 (ppm)

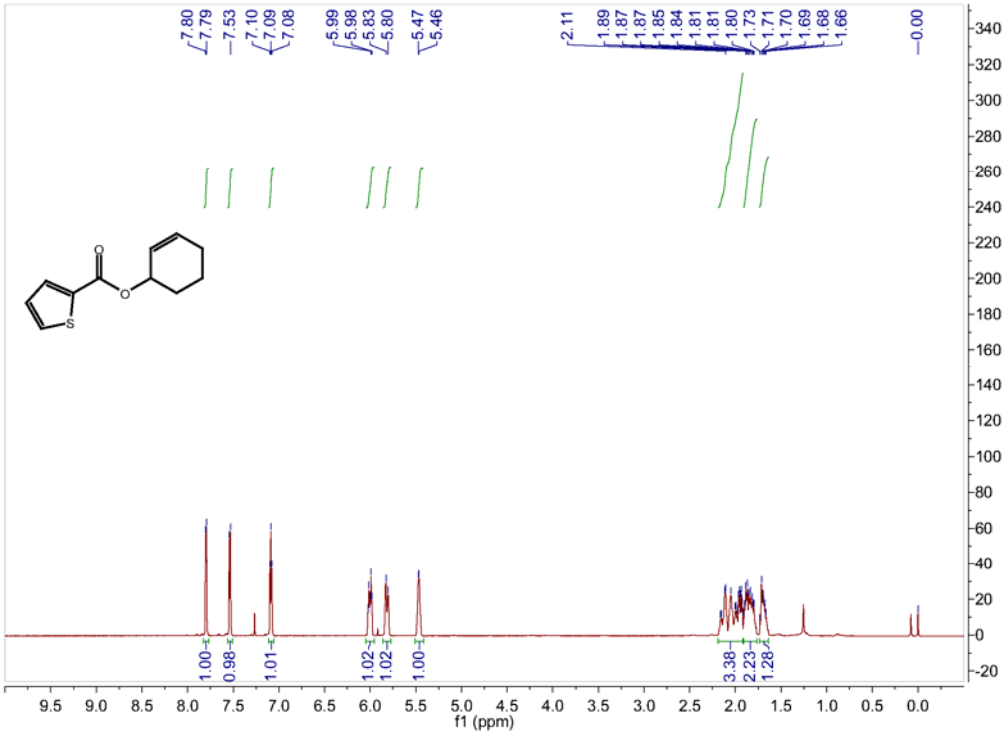

165.88
145.09
132.73
129.82
126.82
125.71
124.78
77.32
77.00
76.68
68.42
28.35
24.89
18.90
14.79
f1 (ppm)

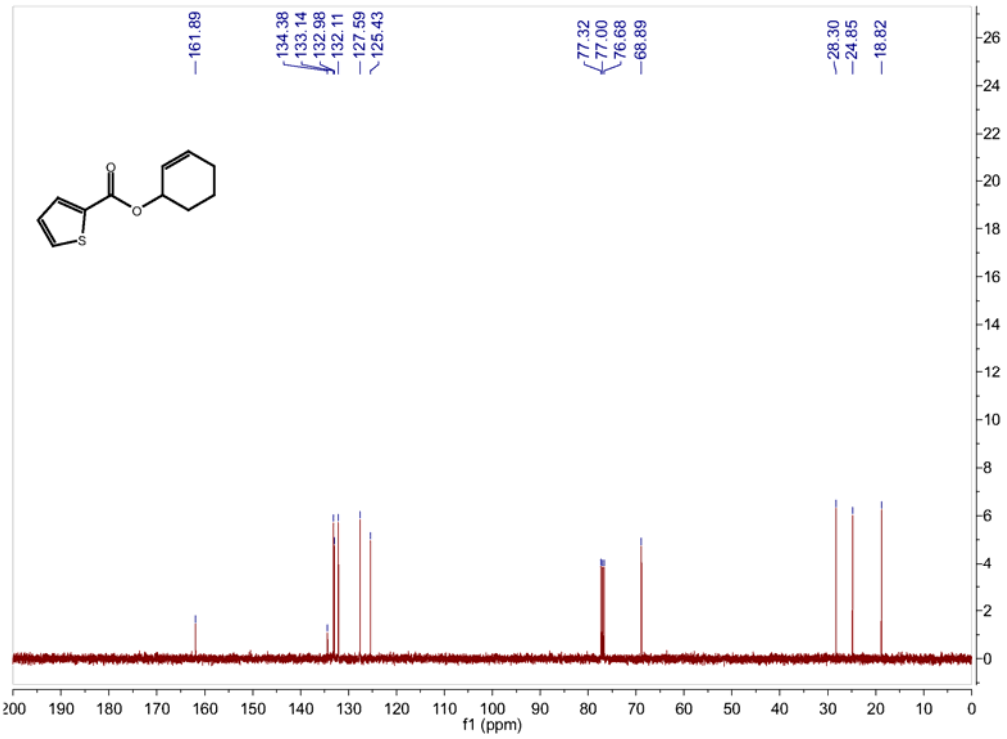
4l


8.07
8.05
7.72
7.68
7.58
7.56
6.54
6.50
6.01
5.85
5.82
5.52
5.51
5.51
5.50
3.82
2.12
2.06
2.00
1.95
1.91
1.88
1.85
1.82
1.74
1.71
1.70
0.00
2.00
0.96
2.09
0.96
0.96
0.94
0.93
3.06
3.07
2.16
1.00
f1 (ppm)

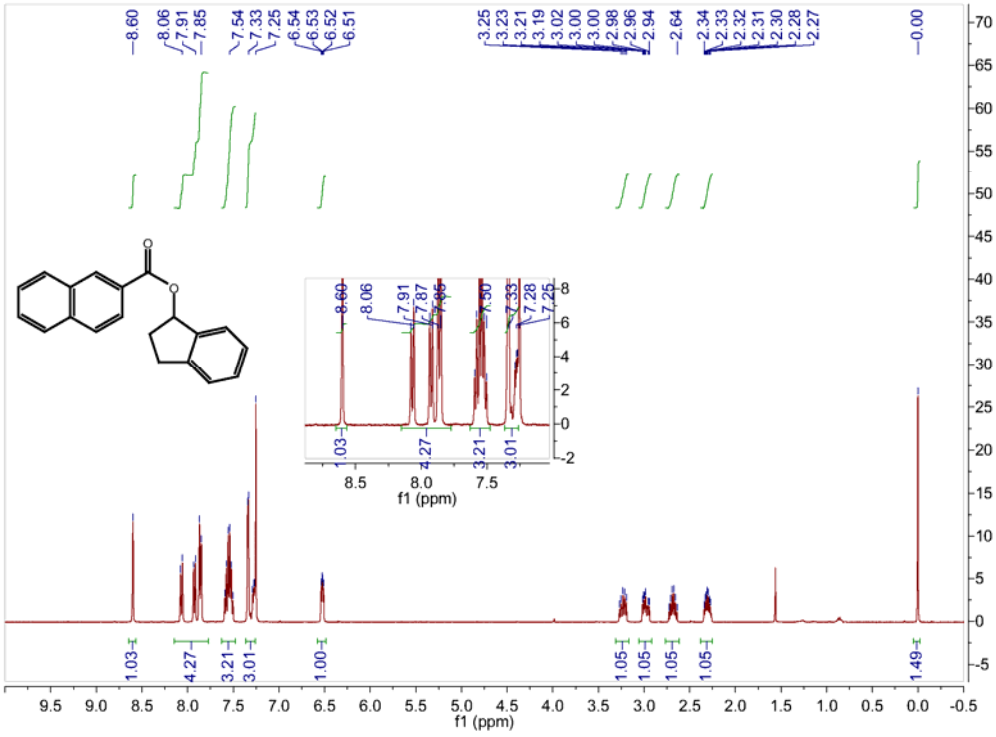

166.89
165.43
143.44
138.31
130.03
125.43
119.93
77.32
77.00
76.68
68.81
51.79
28.29
24.86
18.82
f1 (ppm)

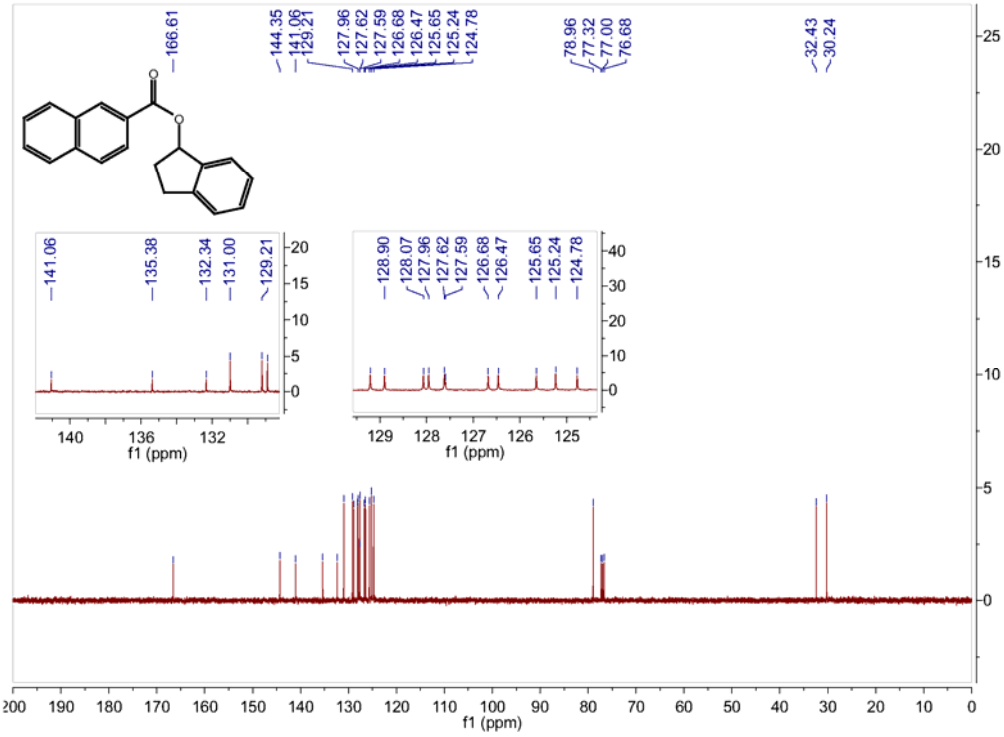
4m


7.80
7.79
7.53
7.10
7.09
7.08
5.99
5.98
5.83
5.80
5.47
5.46
2.11
1.89
1.87
1.87
1.85
1.84
1.81
1.81
1.80
1.73
1.71
1.70
1.69
1.68
1.66
0.00
1.00
0.98
1.01
1.02
1.02
1.00
3.38
2.23
1.28
f1 (ppm)

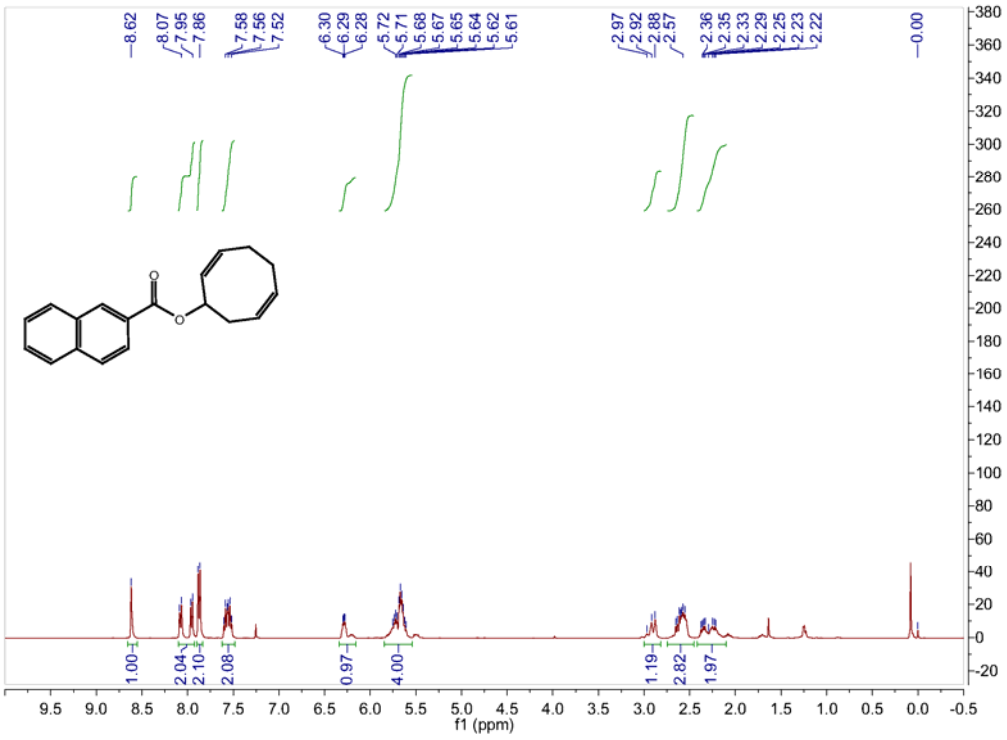

161.89
134.38
133.14
132.98
132.11
127.59
125.43
77.32
77.00
76.68
68.89
28.30
24.85
18.82
f1 (ppm)

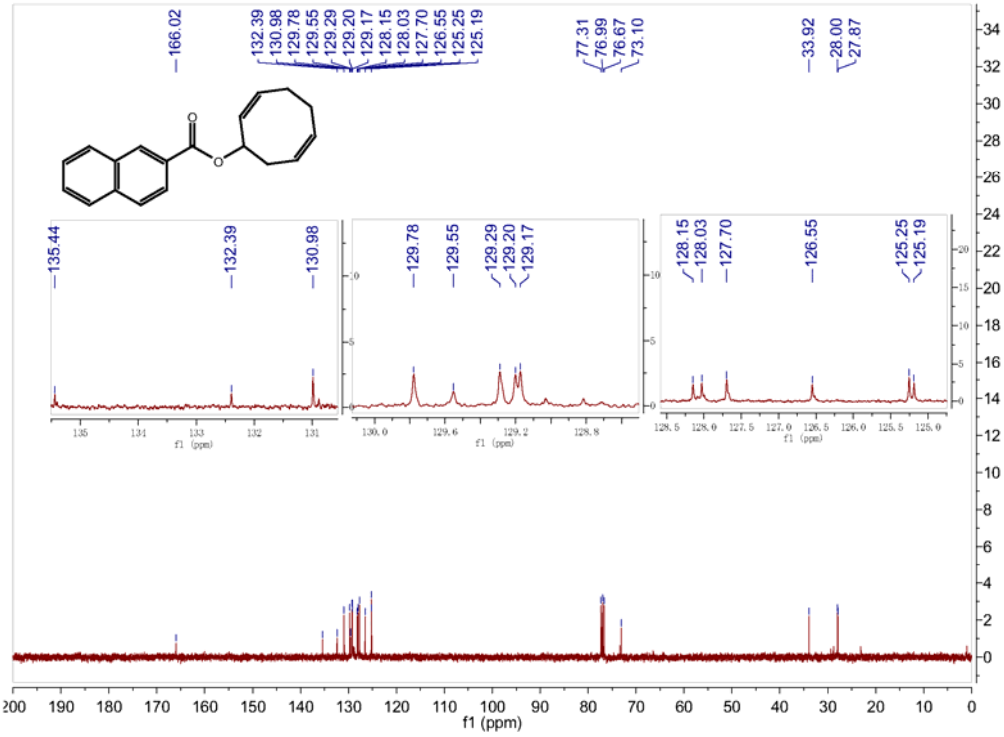
4n


8.60
8.06
7.91
7.85
7.54
7.33
7.25
6.54
6.53
6.52
6.51
3.25
3.23
3.21
3.19
3.02
3.00
3.00
2.98
2.96
2.94
2.64
2.34
2.33
2.32
2.31
2.30
2.28
2.27
0.00
1.03
4.27
3.21
3.01
1.00
1.05
1.05
1.05
1.05
1.49
f1 (ppm)

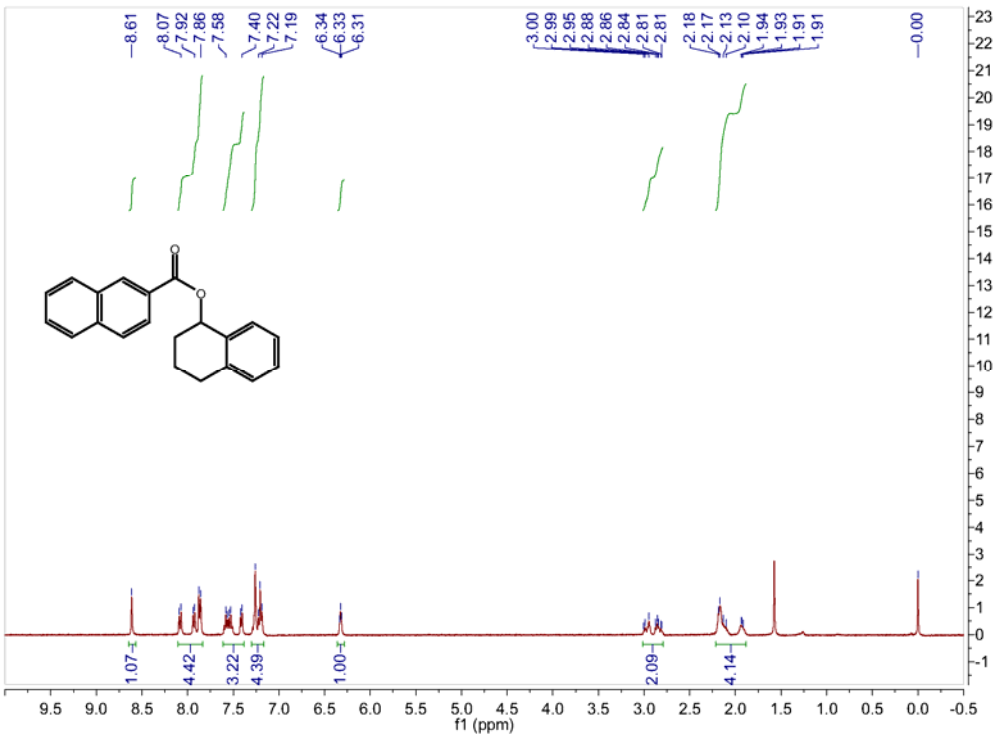

166.61
144.35
141.06
129.21
127.96
127.62
127.59
126.68
126.47
125.65
125.24
124.78
78.96
77.32
77.00
76.68
32.43
30.24
f1 (ppm)

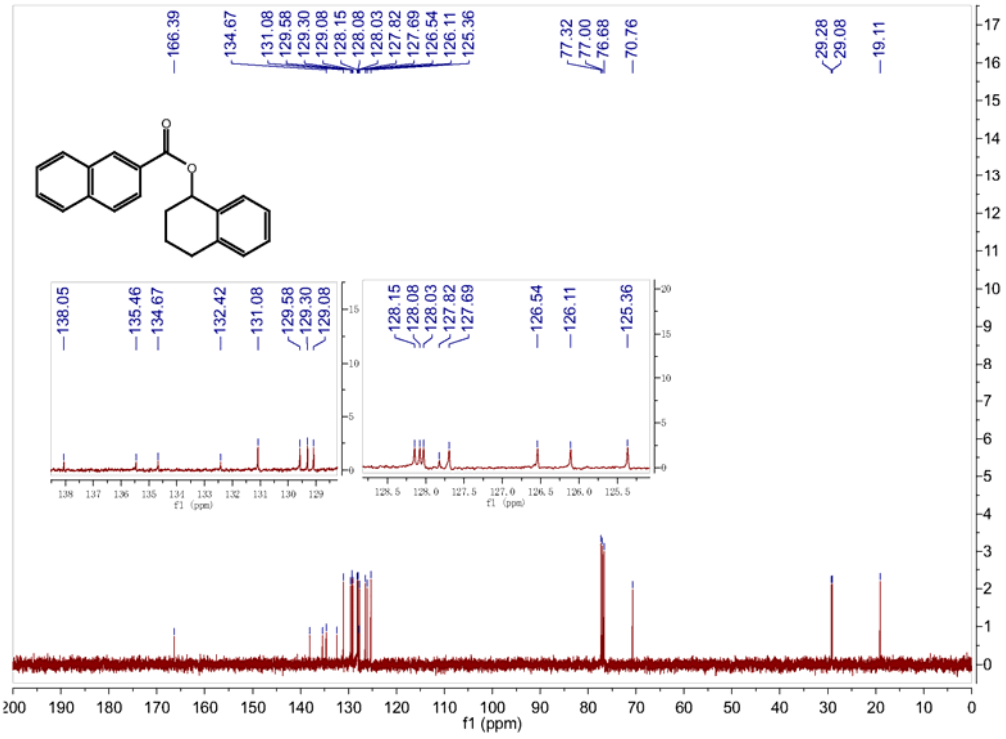
4o


8.62
8.07
7.95
7.86
7.58
7.56
7.52
6.30
6.29
6.28
5.72
5.71
5.68
5.67
5.65
5.64
5.62
5.61
2.97
2.92
2.88
2.57
2.36
2.35
2.33
2.29
2.25
2.23
2.22
0.00
1.00
2.04
2.10
2.08
0.97
4.00
1.19
2.82
1.97
f1 (ppm)

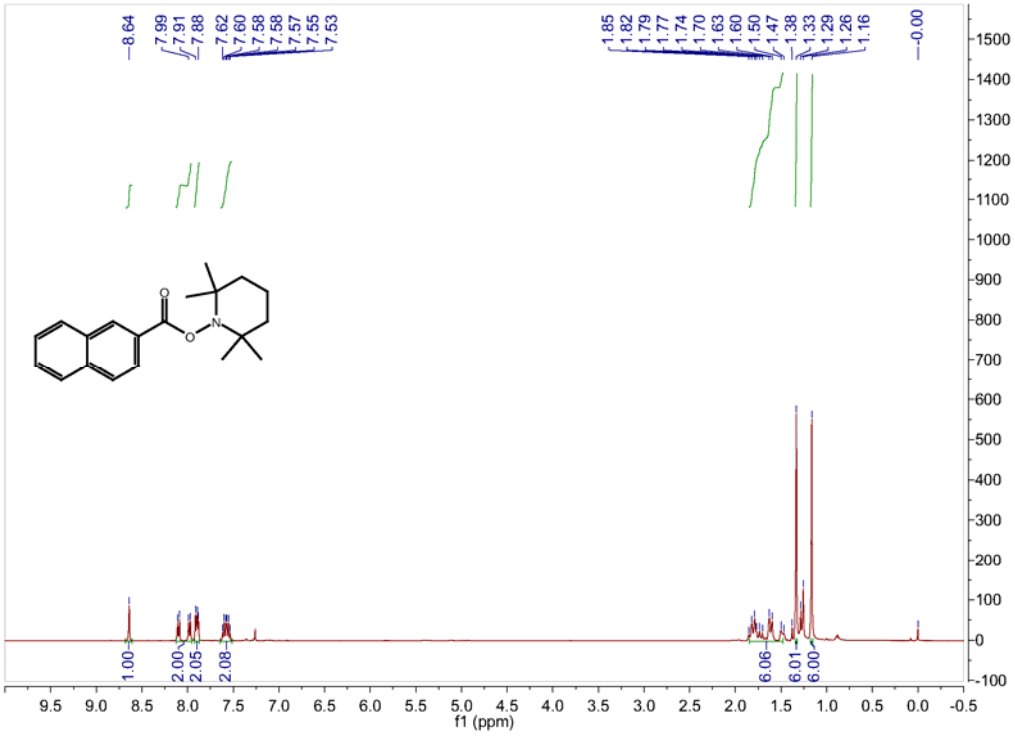

166.02
132.39
130.98
129.78
129.55
129.29
129.20
129.17
128.15
128.03
127.70
126.55
125.25
125.19
77.31
76.99
76.67
73.10
33.92
28.00
27.87
f1 (ppm)

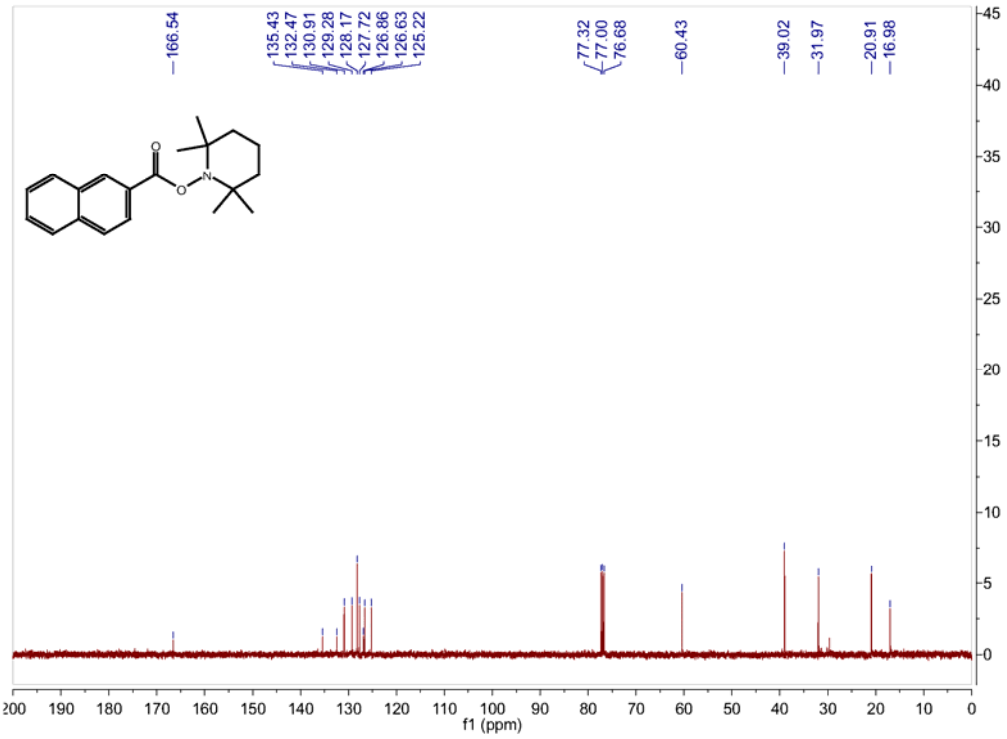
4p


8.61
8.07
7.92
7.86
7.58
7.40
7.22
7.19
6.34
6.33
6.31
3.00
2.99
2.95
2.88
2.86
2.84
2.81
2.81
2.18
2.17
2.13
2.10
1.94
1.93
1.91
1.91
0.00
1.07
4.42
3.22
4.39
1.00
2.09
4.14
f1 (ppm)


166.39
134.67
131.08
129.58
129.30
129.08
128.15
128.08
128.03
127.82
127.69
126.54
126.11
125.36
77.32
77.00
76.68
70.76
29.28
29.08
19.11
138.05
135.46
134.67
132.42
131.08
129.58
129.30
129.08
128.15
128.08
128.03
127.82
127.69
126.54
126.11
125.36
f1 (ppm)

5


8.64
7.99
7.91
7.88
7.62
7.60
7.58
7.58
7.57
7.55
7.53
1.85
1.82
1.79
1.77
1.74
1.70
1.63
1.60
1.50
1.47
1.38
1.33
1.29
1.26
1.16
0.00
1.00
2.00
2.05
2.08
6.06
6.01
6.00
f1 (ppm)


166.54
135.43
132.47
130.91
129.28
128.17
127.72
126.86
126.63
125.22
77.32
77.00
76.68
60.43
39.02
31.97
20.91
16.98
f1 (ppm)