Synthesis of unsymmetrical benzils via palladium-catalysed α-arylation–oxidation of 2-hydroxyacetophenones with aryl bromides

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Supporting Information

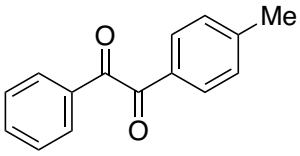
General. All reactions were carried out with standard Schlenk techniques under a nitrogen or an argon atmosphere. Column chromatography was carried out on Wakogel® C-200 (75–150 μm). Preparative thin-layer chromatography (TLC) was performed on Wakogel® B-5F. Proton chemical shifts (δ) were referenced to tetramethylsilane (at 0.00 ppm) or residual $CHCl_3$ (at 7.26 ppm). Carbon chemical shifts (δ) were referenced to $CDCl_3$ (at 77.0 ppm). Fluorine chemical shifts (δ) were referenced to C_6F_6 (at −164.9 ppm).

Materials. α-Hydroxy ketones[1] and **2k**[2] were prepared by the literature methods. All other reagents and solvents were obtained from commercial sources and used without further purification.

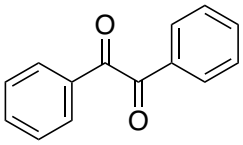
General Procedure for the Palladium-Catalysed α-Arylation–Oxidation of α-Hydroxy Ketones 1. To a sealed tube with a rubber septum under an argon atmosphere charged with $[PdCl(allyl)]_2$ (3.7 mg, 0.010 mmol), XPhos (19.1 mg, 0.040 mmol), K_3PO_4 (106.1 mg, 0.500 mmol) and α-hydroxy ketone **1** (0.200 mmol) was added *t*-BuOH (0.50 mL). The mixture was allowed to stir at room temperature for 30 min. Aryl bromide **2** (0.500 mmol) was added via a syringe through the septum, and the septum was replaced with a screw cap, and the reaction mixture was stirred at 100 °C for 18 h. After cooling to room temperature, the reaction mixture

1 (*a*) Y. Yang and W. Yang, *Chem. Commun.*, 2018, **54**, 12182–12185; (*b*) Y. Zhang, L. He and L. Shi, *Adv. Synth. Catal.*, 2018, **360**, 1926–1931.
2 H. Noguchi, K. Hojo and M. Suginome, *J. Am. Chem. Soc.*, 2007, **129**, 758–759.

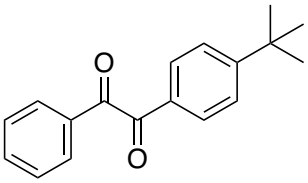
was filtered through a plug of Florisil® eluting with hexane–AcOEt (3:1), and the filtrate was concentrated. The residue was purified by preparative TLC (hexane–AcOEt) to afford benzil **3**.



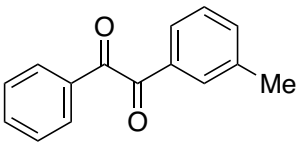
1-(4-Methylphenyl)-2-phenylethane-1,2-dione (3aa). The general procedure was followed using **1a** (27.2 mg, 0.200 mmol) and **2a** (85.6 mg, 0.500 mmol). Purification by preparative TLC (hexane:AcOEt = 10:1) yielded **3aa** (40.3 mg, 0.180 mmol, 90%) as a yellow solid. Mp 90–92 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98–7.95 (m, 2H), 7.87 (d, *J* = 8.0 Hz, 2H), 7.64 (dt, *J* = 1.1, 7.4 Hz, 1H), 7.50 (t, *J* = 7.4 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 2.43 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 194.8, 194.3, 146.2, 134.8, 133.0, 130.5, 130.0, 129.8, 129.7, 128.9, 21.9. The spectral data matched those reported in the literature.[3]



1,2-Diphenylethane-1,2-dione (3ab). The general procedure was followed using **1a** (27.1 mg, 0.199 mmol) and **2b** (78.3 mg, 0.499 mmol). Purification by preparative TLC (hexane:AcOEt = 10:1) yielded **3ab** (37.1 mg, 0.176 mmol, 88%) as a yellow solid. Mp 92–93 °C; ^{1}H NMR (301 MHz, $CDCl_3$) δ 8.01–7.95 (m, 4H), 7.70–7.62 (m, 2H), 7.51 (t, *J* = 7.9 Hz, 4H); ^{13}C NMR (75.6 MHz, $CDCl_3$) δ 194.6, 134.9, 132.9, 129.9, 129.0. The spectral data matched those reported in the literature.[3]



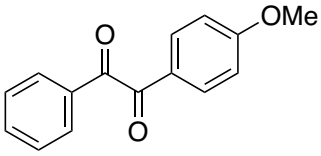
1-(4-*tert*-Butylphenyl)-2-phenylethane-1,2-dione (3ac). The general procedure was followed using **1a** (27.1 mg, 0.199 mmol) and **2c** (106.9 mg, 0.502 mmol). Purification by preparative TLC (hexane:AcOEt = 10:1) yielded **3ac** (45.7 mg, 0.172 mmol, 86%) as a yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99–7.96 (m, 2H), 7.93–7.90 (m, 2H), 7.65 (t, *J* = 7.4 Hz, 1H), 7.54–7.48 (m, 4H), 1.34 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 194.8, 194.3, 159.0, 134.8, 133.1, 130.4, 129.9 [overlapping], 128.9, 126.0, 35.4, 30.9. The spectral data matched those reported in the literature.[3]



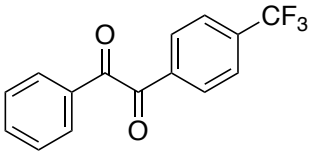
1-(3-Methylphenyl)-2-phenylethane-1,2-dione (3ad). The general procedure was followed using **1a** (27.2 mg, 0.200 mmol) and **2d** (85.6 mg, 0.500 mmol). Purification by preparative TLC (hexane:AcOEt = 10:1) yielded **3ad** (43.9 mg, 0.196 mmol, 98%) as a yellow solid. Mp 88–

3 Y. Kumar, Y. Jaiswal and A. Kumar, *Eur. J. Org. Chem.,* 2018, 494–505.

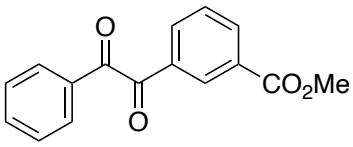
89 °C; ^{1}H NMR (301 MHz, $CDCl_3$) δ 8.00–7.95 (m, 2H), 7.80–7.74 (m, 2H), 7.69–7.62 (m, 1H), 7.55–7.45 (m, 3H), 7.43–7.36 (m, 1H), 2.41 (s, 3H); ^{13}C NMR (75.6 MHz, $CDCl_3$) δ 194.8, 194.7, 139.0, 135.7, 134.8, 133.0, 132.9, 130.2, 129.9, 129.0, 128.9, 127.2, 21.2. The spectral data matched those reported in the literature.[3]



1-(4-Methoxyphenyl)-2-phenylethane-1,2-dione (3ae). The general procedure was followed using **1a** (27.2 mg, 0.200 mmol) and **2e** (93.3 mg, 0.499 mmol). Purification by preparative TLC (hexane:AcOEt = 7:1) yielded **3ae** (37.2 mg, 0.155 mmol, 78%) as a yellow oil. ^{1}H NMR (301 MHz, $CDCl_3$) δ 7.96 (t, J = 7.7 Hz, 4H), 7.64 (t, J = 7.4 Hz, 1H), 7.50 (t, J = 7.6 Hz, 2H), 6.97 (d, J = 8.6 Hz, 2H), 3.88 (s, 3H); ^{13}C NMR (75.6 MHz, $CDCl_3$) δ 194.8, 193.2, 165.0, 134.7, 133.1, 132.3, 129.8, 128.9, 126.0, 114.3, 55.6. The spectral data matched those reported in the literature.[3]

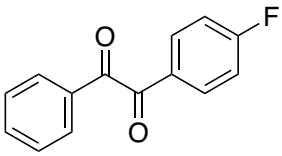


1-Phenyl-2-[4-(trifluoromethyl)phenyl]ethane-1,2-dione (3af). The general procedure was followed using **1a** (27.1 mg, 0.199 mmol) and **2f** (112.8 mg, 0.501 mmol). Purification by preparative TLC (hexane:AcOEt = 10:1) yielded **3af** (39.0 mg, 0.140 mmol, 70%) as a yellow solid. Mp 57–58 °C; ^{1}H NMR (301 MHz, $CDCl_3$) δ 8.14–8.08 (m, 2H), 8.01–7.96 (m, 2H), 7.81–7.76 (m, 2H), 7.69 (tt, J = 7.4, 1.5 Hz, 1H), 7.58–7.50 (m, 2H); ^{13}C NMR (75.6 MHz, $CDCl_3$) δ 193.5, 193.0, 135.8 (q, $^2J_{C–F}$ = 33.0 Hz), 135.5 (q, $^4J_{C–F}$ = 1.4 Hz), 135.3, 132.5, 130.2, 130.0, 129.1, 126.0 (q, $^3J_{C–F}$ = 3.6 Hz), 123.3 (q, $^1J_{C–F}$ = 273.1 Hz). The spectral data matched those reported in the literature.[3]

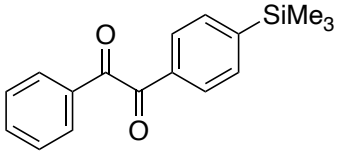


1-[3-(Methoxycarbonyl)phenyl]-2-phenylethane-1,2-dione (3ag). The general procedure was followed using **1a** (27.2 mg, 0.200 mmol) and **2g** (107.5 mg, 0.500 mmol). Purification by preparative TLC (hexane:AcOEt = 5:1) yielded **3ag** (32.4 mg, 0.121 mmol, 61%) as a yellow oil. ^{1}H NMR (301 MHz, $CDCl_3$) δ 8.63–8.60 (m, 1H), 8.33 (dt, J = 7.8, 1.5 Hz, 1H), 8.22–8.16 (m, 1H), 7.99 (dd, J = 8.6, 1.4 Hz, 2H), 7.72–7.59 (m, 2H), 7.57–7.50 (m, 2H), 3.94 (s, 3H); ^{13}C NMR (75.6 MHz, $CDCl_3$) δ 193.7, 193.4, 165.7, 135.5, 135.1, 133.7, 133.2, 132.7, 131.2, 130.9, 130.0, 129.3, 129.1, 52.5. The spectral data matched those reported in the literature.[4]

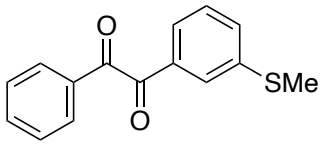
4 A. R. Katritzky, D. Zhang and K. Kirichenko, *J. Org. Chem.*, 2005, **70**, 3271–3274.



1-(4-Fluorophenyl)-2-phenylethane-1,2-dione (3ah). The general procedure was followed using **1a** (27.2 mg, 0.200 mmol) and **2h** (87.4 mg, 0.499 mmol). Purification by preparative TLC (hexane:AcOEt = 10:1) yielded **3ah** (40.7 mg, 0.178 mmol, 89%) as a yellow solid. Mp 61–62 °C; ^{1}H NMR (301 MHz, $CDCl_3$) δ 8.06–7.95 (m, 4H), 7.67 (tt, J = 7.4, 1.4 Hz, 1H), 7.56–7.48 (m, 2H), 7.23–7.15 (m, 2H); ^{13}C NMR (75.6 MHz, $CDCl_3$) δ 194.1, 192.7, 166.7 (d, $^{1}J_{C–F}$ = 258.6 Hz), 135.0, 132.8, 132.7 (d, $^{3}J_{C–F}$ = 10.1 Hz), 129.9, 129.4 (d, $^{4}J_{C–F}$ = 2.9 Hz), 129.0, 116.4 (d, $^{2}J_{C–F}$ = 22.4 Hz). The spectral data matched those reported in the literature.[3]

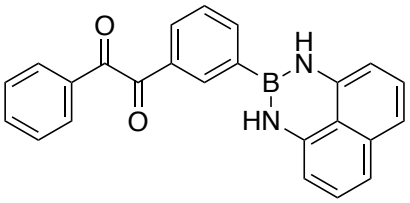


1-Phenyl-2-[4-(trimethylsilyl)phenyl]ethane-1,2-dione (3ai). The general procedure was followed using **1a** (27.1 mg, 0.199 mmol) and **2i** (114.7 mg, 0.500 mmol). Purification by preparative TLC (hexane:AcOEt = 10:1) yielded **3ai** (55.2 mg, 0.195 mmol, 98%) as a yellow solid. Mp 60–61 °C; ^{1}H NMR (301 MHz, $CDCl_3$) δ 8.00–7.95 (m, 2H), 7.93 (d, J = 8.3 Hz, 2H), 7.70–7.62 (m, 3H), 7.55–7.47 (m, 2H), 0.30 (s, 9H); ^{13}C NMR (75.6 MHz, $CDCl_3$) δ 194.9, 194.6, 149.9, 134.8, 133.8, 133.0, 132.9, 129.9, 129.0, 128.6, −1.5. The spectral data matched those reported in the literature.[5]

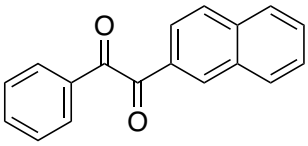


1-[3-(Methylthio)phenyl]-2-phenylethane-1,2-dione (3aj). The general procedure was followed using **1a** (27.2 mg, 0.200 mmol) and **2j** (101.6 mg, 0.500 mmol). Purification by preparative TLC (hexane:AcOEt = 7:1) yielded **3aj** (38.8 mg, 0.141 mmol, 76%) as a yellow oil. ^{1}H NMR (301 MHz, $CDCl_3$) δ 7.96 (dd, J = 8.3, 1.0 Hz, 2H), 7.88–7.85 (m, 1H), 7.71–7.63 (m, 2H), 7.55–7.48 (m, 3H), 7.40 (t, J = 7.7 Hz, 1H), 2.52 (s, 3H); ^{13}C NMR (75.6 MHz, $CDCl_3$) δ 194.2, 194.1, 140.6, 135.0, 133.4, 132.8, 132.5, 129.9, 129.2, 129.0, 126.7, 126.3, 15.4; IR (ATR, ν/cm^{-1}): 1683, 1596, 1581, 1211; HRMS (ESI) calcd for $C_{15}H_{13}O_2S$ $[M + H]^+$ 257.0631, found 257.0636;

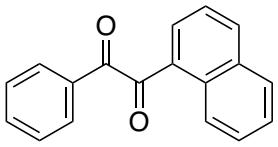
5 A. Gao, F. Yang, J. Li and Y. Wu, *Tetrahedron*, 2012, **68**, 4950–4954.



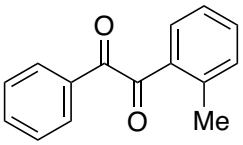
1-[3-(Naphtho[1,8-*de*][1,3,2]diazaborinin-2-yl)-phenyl]-2-phenylethane-1,2-dione (3ak). The general procedure was followed using **1a** (27.2 mg, 0.200 mmol) and **2k** (161.5 mg, 0.500 mmol). Purification by preparative TLC (hexane:AcOEt = 5:1) yielded **3ak** (37.1 mg, 0.0987 mmol, 49%) as an orange solid. Mp 165 °C (decomp); ^{1}H NMR (301 MHz, $CDCl_3$) δ 8.27 (s, 1H), 8.04–7.98 (m, 3H), 7.91 (d, *J* = 7.6 Hz, 1H), 7.72–7.64 (m, 1H), 7.59–7.49 (m, 3H), 7.17–7.03 (m, 4H), 6.42 (d, *J* = 6.9 Hz, 2H), 6.06 (s, 2H); ^{13}C NMR (75.6 MHz, $CDCl_3$) δ 194.6, 194.4, 140.6, 137.8, 136.2, 135.1, 132.9, 132.6, 132.5, 131.7, 130.0, 129.1, 128.9, 127.6, 119.9, 118.2, 106.3 [The carbon atom attached to the boron atom was not observed due to quadrupolar broadening]; IR (ATR, ν/cm^{-1}): 3400, 1669, 1602, 1408, 1198, 766; HRMS (ESI) calcd for $C_{24}H_{18}BN_2O_2$ $[M + H]^+$ 377.1460, found 377.1456.



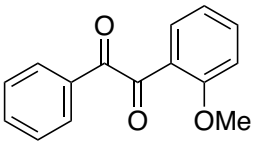
1-(2-Naphthyl)-2-phenylethane-1,2-dione (3al). The general procedure was followed using **1a** (27.3 mg, 0.201 mmol) and **2l** (103.5 mg, 0.500 mmol). Purification by column chromatography on silica gel (hexane:AcOEt = 15:1) yielded **3al** (41.0 mg, 0.158 mmol, 79%) as a yellow solid. Mp 86–87 °C; ^{1}H NMR (301 MHz, $CDCl_3$) δ 8.42 (s, 1H), 8.10 (dd, *J* = 8.8, 1.5 Hz, 1H), 8.06–8.01 (m, 2H), 7.95 (d, *J* = 8.6 Hz, 1H), 7.92–7.85 (m, 2H), 7.69–7.59 (m, 2H), 7.57–7.48 (m, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 194.6 [overlapping], 136.4, 134.9, 133.5, 133.1, 132.3, 130.3, 130.0, 129.9, 129.5, 129.2, 129.0, 127.9, 127.2, 123.6. The spectral data matched those reported in the literature.[3]



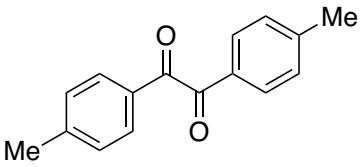
1-(1-Naphthyl)-2-phenylethane-1,2-dione (3am). The general procedure was followed using **1a** (27.1 mg, 0.199 mmol) and **2m** (103.7 mg, 0.501 mmol). Purification by preparative TLC (hexane:AcOEt = 10:1) yielded **3am** (30.0 mg, 0.115 mmol, 58%) as a yellow solid. Mp 99–100 °C; ^{1}H NMR (301 MHz, $CDCl_3$) δ 9.31 (d, *J* = 8.6 Hz, 1H), 8.11 (d, *J* = 8.3 Hz, 1H), 8.03 (dd, *J* = 8.3, 1.0 Hz, 2H), 7.96–7.89 (m, 2H), 7.78–7.71 (m, 1H), 7.66–7.59 (m, 2H), 7.55–7.45 (m, 3H); ^{13}C NMR (75.6 MHz, $CDCl_3$) δ 197.1, 194.6, 136.0, 135.1, 134.7, 134.0, 133.3, 130.9, 130.0, 129.4, 129.0, 128.8, 128.5, 127.1, 125.9, 124.4. The spectral data matched those reported in the literature.[3]



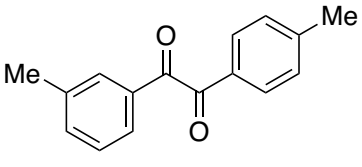
1-(2-Methylphenyl)-2-phenylethane-1,2-dione (3an). The general procedure was followed using **1a** (27.2 mg, 0.200 mmol) and **2n** (85.7 mg, 0.501 mmol). Purification by preparative TLC (hexane:AcOEt = 10:1) yielded **3an** (20.2 mg, 0.090 mmol, 45%) as a yellow solid. Mp 56–57 °C; ^{1}H NMR (301 MHz, $CDCl_3$) δ 8.00–7.95 (m, 2H), 7.69–7.62 (m, 2H), 7.55–7.46 (m , 3H), 7.34 (d, *J* = 7.9 Hz, 1H), 7.30–7.23 (m, 1H), 2.71 (s, 3H); 13C NMR (126 MHz. $CDCl_3$) δ 196.8, 194.8, 141.4, 134.7 [overlapping], 133.8, 133.0, 132.5, 131.7, 129.9, 129.0, 126.0, 21.9. The spectral data matched those reported in the literature.[6]



1-(2-Methoxyphenyl)-2-phenylethane-1,2-dione (3ao). The general procedure was followed using **1a** (27.2 mg, 0.200 mmol) and **2o** (93.6 mg, 0.500 mmol). Purification by preparative TLC (hexane:AcOEt = 7:1) yielded **3ao** (21.2 mg, 0.088 mmol, 44%) as a yellow solid. Mp 70–71 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.03 (dd, *J* = 8.0, 1.7 Hz, 1H), 7.94–7.91 (m, 2H), 7.64–7.57 (m, 2H), 7.49 (t, *J* = 8.0 Hz, 2H), 7.13 (t, *J* = 7.4 Hz, 1H), 6.94 (d, *J* = 8.0 Hz, 1H), 3.56 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 194.6, 193.5, 160.4, 136.5, 133.8, 132.9, 130.5, 129.3, 128.7, 123.8, 121.5, 112.3, 55.6. The spectral data matched those reported in the literature.[3]



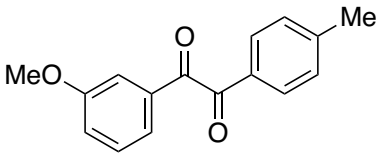
1,2-Bis(4-methylphenyl)ethane-1,2-dione (3ba). The general procedure was followed using **1b** (29.9 mg, 0.199 mmol) and **2a** (85.5 mg, 0.500 mmol). Purification by preparative TLC (hexane:AcOEt = 10:1) yielded **3ba** (43.5 mg, 0.183 mmol, 92%) as a yellow solid. Mp 103–105 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.2 Hz, 4H), 7.29 (d, *J* = 8.2 Hz, 4H), 2.42 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.5, 146.1, 130.6, 130.0, 129.7, 21.9. The spectral data matched those reported in the literature.[3]



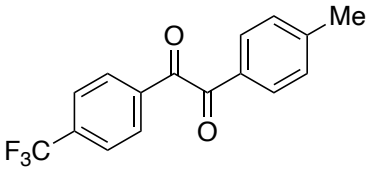
1-(3-Methylphenyl)-2-(4-methylphenyl)ethane-1,2-dione (3ca). The general procedure was followed using **1c** (30.1 mg, 0.200 mmol) and **2a** (85.5 mg, 0.500 mmol). Purification by preparative TLC (hexane:AcOEt = 10:1) yielded **3ca** (45.3 mg, 0.190 mmol, 95%) as a yellow solid. Mp 61–62 °C; ^{1}H NMR (301 MHz, $CDCl_3$) δ 7.89–7.84 (m, 2H), 7.79–7.73 (m, 2H), 7.49–7.44 (m, 1H), 7.42–7.35 (m, 1H), 7.31 (d, *J* = 8.6 Hz, 2H), 2.44 (s, 3H), 2.40 (s, 3H); ^{13}C

6 X. Zhu, P. Li, Q. Shi and L. Wang, *Green Chem.*, 2016, **18**, 6373–6379.

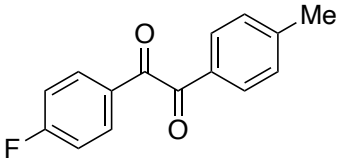
NMR (126 MHz, $CDCl_3$) δ 195.0, 194.4, 146.1, 138.9, 135.6, 133.0, 130.6, 130.2, 130.0, 129.7, 128.8, 127.2, 21.9, 21.2. The spectral data matched those reported in the literature.[6]



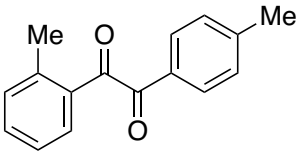
1-(3-Methoxyphenyl)-2-(4-methylphenyl)ethane-1,2-dione (3da). The general procedure was followed using **1d** (33.2 mg, 0.200 mmol) and **2a** (85.6 mg, 0.500 mmol). Purification by preparative TLC (hexane:AcOEt = 5:1) yielded **3da** (40.5 mg, 0.159 mmol, 80%) as a yellow oil. ^{1}H NMR (301 MHz, $CDCl_3$) δ 7.86 (d, J = 8.3 Hz, 2H), 7.56–7.52 (m, 1H), 7.50–7.45 (m, 1H), 7.38 (t, J = 7.9 Hz, 1H), 7.31 (d, J = 7.9 Hz, 2H), 7.22–7.17 (m, 1H), 3.86 (s, 3H), 2.44 (s, 3H); ^{13}C NMR (75.6 MHz, $CDCl_3$) δ 194.7, 194.2, 160.0, 146.2, 134.3, 130.5, 130.0 [overlapping], 129.7, 123.2, 121.8, 112.7, 55.5, 21.9; IR (ATR, ν/cm^{-1}): 1682, 1605, 1256; HRMS (ESI) calcd for $C_{16}H_{14}NaO_3$ $[M + Na]^+$ 277.0835, found 277.0834.



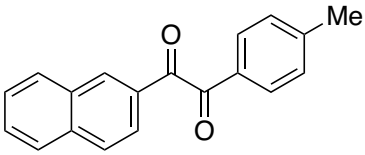
1-(4-Methylphenyl)-2-[4-(trifluoromethyl)phenyl]-ethane-1,2-dione (3ea). The general procedure was followed using **1e** (40.9 mg, 0.200 mmol) and **2a** (85.5 mg, 0.500 mmol). Purification by preparative TLC (hexane:AcOEt = 10:1) yielded **3ea** (37.3 mg, 0.128 mmol, 64%) as a yellow solid. Mp 101–102 °C; ^{1}H NMR (301 MHz, $CDCl_3$) δ 8.10 (d, J = 7.9 Hz, 2H), 7.87 (d, J = 8.3 Hz, 2H), 7.78 (d, J = 8.6 Hz, 2H), 7.33 (d, J = 8.6 Hz, 2H), 2.45 (s, 3H); ^{13}C NMR (75.6 MHz, $CDCl_3$) δ 193.19, 193.15, 146.7, 135.66 (q, $^2J_{C–F}$ = 33.0 Hz), 135.65, 130.2, 130.11, 130.05, 129.8, 125.9 (q, $^3J_{C–F}$ = 3.6 Hz),123.3 (q, $^1J_{C–F}$ = 273.1 Hz), 21.9. The spectral data matched those reported in the literature.[3]



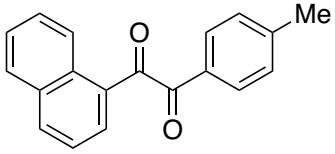
1-(4-Fluorophenyl)-2-(4-methylphenyl)ethane-1,2-dione (3fa). The general procedure was followed using **1f** (30.8 mg, 0.200 mmol) and **2a** (85.5 mg, 0.500 mmol). Purification by preparative TLC (hexane:AcOEt = 10:1) yielded **3fa** (38.0 mg, 0.157 mmol, 79%) as a yellow solid. Mp 78–79 °C; ^{1}H NMR (301 MHz, $CDCl_3$) δ 8.04–7.98 (m, 2H), 7.86 (d, J = 8.3 Hz, 2H), 7.31 (d, J = 8.3 Hz, 2H), 7.22–7.13 (m, 2H), 2.44 (s, 3H); ^{13}C NMR (75.6 MHz, $CDCl_3$) δ 193.8, 192.9, 166.7 (d, $^1J_{C–F}$ = 257.9 Hz), 146.4, 132.7 (d, $^3J_{C–F}$ = 10.1 Hz), 130.3, 130.0, 129.7, 129.5 (d, $^4J_{C–F}$ = 2.9 Hz), 116.3 (d, $^2J_{C–F}$ = 22.4 Hz), 21.9. The spectral data matched those reported in the literature.[6]



1-(2-Methylphenyl)-2-(4-methylphenyl)ethane-1,2-dione (3ga). The general procedure was followed using **1g** (30.0 mg, 0.200 mmol) and **2a** (85.6 mg, 0.500 mmol). Purification by preparative TLC (hexane:AcOEt = 10:1) yielded **3ga** (33.6 mg, 0.141 mmol, 71%) as a yellow solid. Mp 101–102 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81–7.77 (m, 2H), 7.55 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.40 (dt, *J* = 1,4, 7.5 Hz, 1H), 7.27–7.21 (m, 3H), 7.20–7.15 (m, 1H), 2.62 (s, 3H), 2.36 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 197.0, 194.6, 146.0, 141.3, 133.7, 133.0, 132.5, 131.9, 130.7, 130.0, 129.7, 126.0, 21.9 [overlapping]. The spectral data matched those reported in the literature.[7]



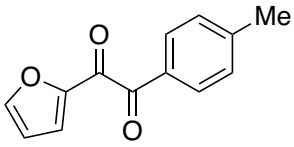
1-(4-Methylphenyl)-2-(2-naphthyl)ethane-1,2-dione (3ha). The general procedure was followed using **1h** (37.3 mg, 0.200 mmol) and **2a** (85.7 mg, 0.501 mmol). Purification by preparative TLC (hexane:AcOEt = 10:1) yielded **3ha** (40.7 mg, 0.148 mmol, 74%) as a yellow solid. Mp 88–89 °C; ^{1}H NMR (301 MHz, $CDCl_3$) δ 8.40 (s, 1H), 8.09 (dd, *J* = 8.8, 1.5 Hz, 1H), 7.97–7.85 (m, 5H), 7.62 (dt, *J* = 10.4, 3.8 Hz, 1H), 7.57–7.50 (m, 1H), 7.31 (d, *J* = 8.3 Hz, 2H), 2.43 (s, 3H); ^{13}C NMR (75.6 MHz, $CDCl_3$) δ 194.8, 194.4, 146.2, 136.3, 133.5, 132.3, 130.6, 130.3, 130.0, 129.9, 129.7, 129.4, 129.1, 127.9, 127.1, 123.6, 21.9. The spectral data matched those reported in the literature.[8]



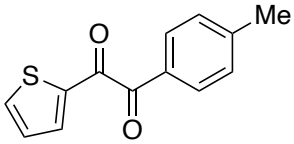
1-(4-Methoxyphenyl)-2-(1-naphthyl)ethane-1,2-dione (3ia). The general procedure was followed using **1i** (37.3 mg, 0.200 mmol) and **2a** (85.5 mg, 0.500 mmol). Purification by preparative TLC (hexane:AcOEt = 10:1) yielded **3ia** (39.1 mg, 0.143 mmol, 72%) as a yellow solid. Mp 93–94 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.30 (d, *J* = 8.7 Hz, 1H), 8.09 (d, *J* = 8.2 Hz, 1H), 7.94–7.88 (m, 4H), 7.76–7.70 (m, 1H), 7.64–7.58 (m, 1H), 7.46 (dd, *J* = 8.2, 7.3 Hz, 1H), 7.30 (d, *J* = 8.2 Hz, 2H), 2.42 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.3, 194.3, 146.0, 135.8, 135.0, 134.0, 130.9 [overlapping], 130.1, 129.7, 129.3, 128.73, 128.68, 127.0, 125.9, 124.4, 21.9; IR (ATR, ν/cm^{-1}): 1669, 1605, 1510, 1225, 1176, 890, 769; HRMS (ESI) calcd for $C_{19}H_{15}O_2$ [M + H]$^{+}$ 275.1067, found 275.1063.

7 D. Ragno, O. Bortolini, P. P. Giovannini, A. Massi, S. Pacifico and A. Zaghi, *Org. Biomol. Chem.*, 2014, **12**, 5733–5744.

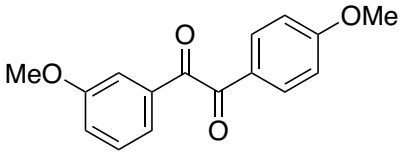
8 M. R. Rohman, I. Kharkongor, M. Rajbangshi, H. Mecadon, B. M. Laloo, P. R. Sahu, I. Kharbangar and B. Myrboh, *Eur. J. Org. Chem.*, 2012, 320–328.



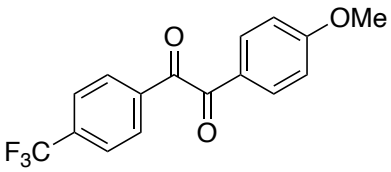
1-(2-Furyl)-2-(4-methylphenyl)ethane-1,2-dione (3ja). The general procedure was followed using **1j** (25.3 mg, 0.201 mmol) and **2a** (85.8 mg, 0.502 mmol). Purification by preparative TLC (hexane:AcOEt = 5:1) yielded **3ja** (23.5 mg, 0.110 mmol, 55%) as a yellow solid. Mp 75–76 °C; ^{1}H NMR (301 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.3 Hz, 2H), 7.77–7.75 (m, 1H), 7.38 (d, *J* = 3.4 Hz, 1H), 7.32 (d, *J* = 8.6 Hz, 2H), 6.62 (dd, *J* = 3.4, 1.7 Hz, 1H), 2.44 (s, 3H); ^{13}C NMR (75.6 MHz, $CDCl_3$) δ 191.3, 180.7, 149.9, 149.1, 146.2, 130.3, 130.0, 129.6, 123.2, 112.9, 21.9; IR (ATR, *ν*/cm^{-1}): 1652, 1605, 1461; HRMS (ESI) calcd for $C_{13}H_{10}NaO_3$ $[M + Na]^+$ 237.0522, found 237.0516.



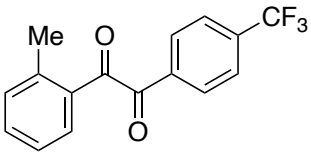
1-(4-Methylphenyl)-2-(2-thienyl)ethane-1,2-dione (3ka). The general procedure was followed using **1k** (28.3 mg, 0.199 mmol) and **2a** (85.5 mg, 0.500 mmol). Purification by preparative TLC (hexane:AcOEt = 5:1) yielded **3ka** (34.9 mg, 0.152 mmol, 76%) as a yellow solid. Mp 63–64 °C; ^{1}H NMR (301 MHz, $CDCl_3$) δ 7.97–7.91 (m, 2H), 7.83 (dd, *J* = 4.8, 1.0 Hz, 1H), 7.79 (dd, *J* = 3.8, 1.0 Hz, 1H), 7.34–7.29 (m, 2H), 7.20–7.16 (m, 1H), 2.44 (s, 3H); ^{13}C NMR (75.6 MHz, $CDCl_3$) δ 191.8, 185.9, 146.2, 139.9, 136.71, 136.65, 130.3, 130.1, 129.6, 128.8, 21.9; IR (ATR, *ν*/cm^{-1}): 1668, 1655, 1603, 1410, 1226; HRMS (ESI) calcd for $C_{13}H_{11}O_2S$ $[M + H]^+$ 231.0474, found 231.0470.



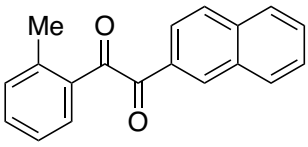
1-(3-Methoxyphenyl)-2-(4-methoxyphenyl)ethane-1,2-dione (3de). The general procedure was followed using **1d** (33.2 mg, 0.200 mmol) and **2e** (93.6 mg, 0.500 mmol). Purification by preparative TLC (hexane:AcOEt = 4:1) yielded **3de** (38.2 mg, 0.141 mmol, 71%) as a yellow solid. Mp 81–82 °C; ^{1}H NMR (301 MHz, $CDCl_3$) δ 7.97–7.91 (m, 2H), 7.56–7.52 (m, 1H), 7.50–7.46 (m, 1H), 7.38 (t, *J* = 7.9 Hz, 1H), 7.19 (ddd, *J* = 8.3, 2.8, 1.0 Hz, 1H), 7.00–6.94 (m, 2H), 3.88 (s, 3H), 3.86 (s, 3H); ^{13}C NMR (75.6 MHz, $CDCl_3$) δ 194.8, 193.1, 164.9, 160.0, 134.4, 132.3, 130.0, 126.0, 123.2, 121.7, 114.3, 112.8, 55.6, 55.5; IR (ATR, *ν*/cm^{-1}): 1666, 1654, 1600, 1574, 1261, 1171; HRMS (ESI) calcd for $C_{16}H_{14}NaO_4$ $[M + Na]^+$ 293.0784, found 293.0783.



1-(4-Methoxyphenyl)-2-[4-(trifluoromethyl)phenyl]-ethane-1,2-dione (3ee). The general procedure was followed using **1e** (40.6 mg, 0.199 mmol) and **2e** (93.8 mg, 0.501 mmol). Purification by preparative TLC (hexane:AcOEt = 7:1) yielded **3ee** (36.4 mg, 0.118 mmol, 59%) as a yellow solid. Mp 95–97 °C, ^{1}H NMR (301 MHz, $CDCl_3$) δ 8.10 (d, J = 8.3 Hz, 2H), 7.99–7.93 (m, 2H), 7.77 (d, J = 8.3 Hz, 2H), 7.03–6.96 (m, 2H), 3.90 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 193.3, 192.0, 165.3, 135.8, 135.7 (q, $^{2}J_{C–F}$ = 32.8 Hz), 132.5, 130.2, 125.9 (q, $^{3}J_{C–F}$ = 3.6 Hz), 125.6, 123.3 (q, $^{1}J_{C–F}$ = 272.7 Hz), 114.5, 55.7. The spectral data matched those reported in the literature.[9]

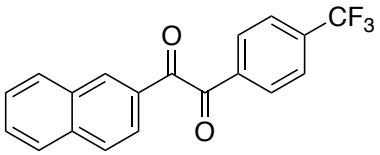


1-(2-Methylphenyl)-2-[4-(trifluoromethyl)phenyl]ethane-1,2-dione (3gf). The general procedure was followed using **1g** (29.7 mg, 0.196 mmol) and **2f** (112.5 mg, 0.500 mmol). Purification by preparative TLC (hexane:AcOEt = 10:1) yielded **3gf** (37.5 mg, 0.128 mmol, 65%) as a yellow solid. Mp 85–86 °C; ^{1}H NMR (301 MHz, $CDCl_3$) δ 8.13–8.08 (m, 2H), 7.79 (d, J = 8.3 Hz, 2H), 7.64–7.59 (m, 1H), 7.53 (dt, J = 1,4, 7.6 Hz, 1H), 7.37 (d, J = 7.9 Hz, 1H), 7.31 (d, J = 7.6 Hz, 1H), 2.71 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 195.8, 193.3, 141.6, 135.8, 135.7 (q, $^{2}J_{C–F}$ = 32.8 Hz), 134.1, 133.0, 132.7, 131.4, 130.2, 126.1, 126.0 (q, $^{3}J_{C–F}$ = 3.6 Hz), 123.3 (q, $^{1}J_{C–F}$ = 273.1 Hz), 21.9; ^{19}F NMR (375 MHz, $CDCl_3$) δ −66.5; IR (ATR, ν/cm^{-1}): 1683, 1667, 1331, 1182, 1123; HRMS (ESI) calcd for $C_{16}H_{12}F_3O_2$ $[M + H]^+$ 293.0784, found 293.0783.

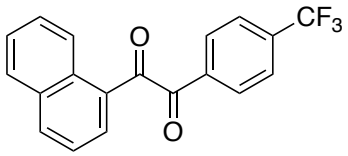


1-(2-Methylphenyl)-2-(2-naphthyl)ethane-1,2-dione (3gl). The general procedure was followed using **1g** (30.0 mg, 0.200 mmol) and **2l** (103.5 mg, 0.500 mmol). Purification by column chromatography on silica gel (hexane:AcOEt = 15:1) yielded **3gl** (38.5 mg, 0.140 mmol, 70%) as a yellow solid. Mp 76–77 °C; ^{1}H NMR (301 MHz, $CDCl_3$) δ 8.43 (s, 1H), 8.12–8.06 (m, 1H), 7.98–7.86 (m, 3H), 7.71–7.60 (m, 2H), 7.59–7.45 (m, 2H), 7.36 (d, J = 7.6 Hz, 1H), 7.26 (t, J = 7.4 Hz, 1H), 2.75 (s, 3H); ^{13}C NMR (75.6 MHz, $CDCl_3$) δ 196. 8, 195.0, 141.4, 136.3, 133.8, 133.3, 133.1, 132.6, 132.3, 131.9, 130.4, 129.9, 129.4, 129.1, 127.9, 127.1, 126.0, 123.8, 22.0; IR (ATR, ν/cm^{-1}): 1669, 1627, 1184, 733; HRMS (ESI) calcd for $C_{19}H_{14}NaO_2$ $[M + Na]^+$ 297.0886, found 297.0891.

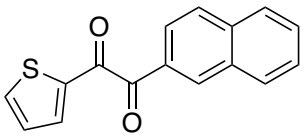
9 J. B. Bharate, S. Abbat, R. Sharma, P. V. Bharatam, R. A. Vishwakarma and S. B. Bharate, *Org. Biomol. Chem.*, 2015, **13**, 5235–5242.



1-(2-Naphthyl)-2-[4-(trifluoromethyl)phenyl]ethane-1,2-dione (3hf). The general procedure was followed using **1h** (37.3 mg, 0.200 mmol) and **2f** (112.4 mg, 0.500 mmol). Purification by preparative TLC (hexane:AcOEt = 10:1) yielded **3hf** (40.3 mg, 0.123 mmol, 62%) as a yellow solid. Mp 104–105 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.41 (s, 1H), 8.15 (d, J = 8.0 Hz, 2H), 8.09 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.98 (d, *J* = 8.6 Hz, 1H), 7.94–7.88 (m, 2H), 7.79 (d, *J* = 8.6 Hz, 2H), 7.68–7.64 (m, 1H), 7.59–7.55 (m, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 193.5, 193.1, 136.5, 135.8 (q, $^{2}J_{C–F}$ = 32.8 Hz), 135.7, 133.7, 132.3, 130.3, 130.0, 129.9, 129. 8, 129.3, 128.0, 127.3, 126.0 (q, $^{3}J_{C–F}$ = 3.6 Hz), 123.6, 123.3 (q, $^{1}J_{C–F}$ = 272.7 Hz); ^{19}F NMR (375 MHz, $CDCl_3$) δ −66.5; IR (ATR, *ν*/cm^{-1}): 1675, 1663, 1328, 1119; HRMS (ESI) calcd for $C_{19}H_{12}F_3O_2$ $[M + H]^+$ 329.0784, found 329.0788.

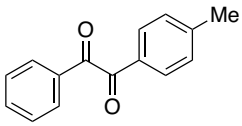


1-(1-Naphthyl)-2-[4-(trifluoromethyl)phenyl]ethane-1,2-dione (3if). The general procedure was followed using **1i** (37.2 mg, 0.200 mmol) and **2f** (112.6 mg, 0.500 mmol). Purification by preparative TLC (hexane:AcOEt = 10:1) yielded **3if** (40.0 mg, 0.122 mmol, 61%) as a yellow solid. Mp 125–126 °C; ^{1}H NMR (301 MHz, $CDCl_3$) δ 9.29 (d, *J* = 8.6 Hz, 1H), 8.19–8.13 (m, 3H), 7.97 (d, *J* = 8.3 Hz, 1H), 7.91–7.86 (m, 1H), 7.82–7.74 (m, 3H), 7.69–7.62 (m, 1H), 7.52 (t, *J* = 7.7 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 196.0, 193.1, 136.4, 136.0, 135.7 (q, $^{2}J_{C–F}$ = 32.8 Hz), 135.2, 134.1, 130.9, 130.3, 129.7, 128.9, 128.2, 127.3, 126.1 (q, $^{3}J_{C–F}$ = 3.6 Hz), 125.8, 124.4, 123.3 (q, $^{1}J_{C–F}$ = 273.1 Hz); ^{19}F NMR (375 MHz, $CDCl_3$) δ −66.5; IR (ATR, *ν*/cm^{-1}): 1682, 1659, 1329, 1317, 1167, 1121; HRMS (ESI) calcd for $C_{19}H_{11}F_3NaO_2$ $[M + Na]^+$ 351.0603, found 351.0611.



1-(2-Naphthyl)-2-(2-thienyl)ethane-1,2-dione (3kl). The general procedure was followed using **1k** (28.4 mg, 0.200 mmol) and **2l** (103.5 mg, 0.500 mmol). Purification by column chromatography on silica gel (hexane:AcOEt = 7:1) yielded **3kl** (43.6 mg, 0.164 mmol, 82%) as a yellow solid. Mp 51–53 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.54 (s, 1H), 8.11 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.98–7.92 (m, 2H), 7.89 (d, *J* = 8.0 Hz, 1H), 7.86–7.83 (m, 2H), 7.66–7.62 (m, 1H), 7.58–7.53 (m, 1H), 7.21–7.17 (m, 1H); ^{13}C NMR (75.6 MHz, $CDCl_3$) δ 192.1, 185.7, 140.0, 136.9, 136.8, 136.3, 133.9, 132.3, 130.0, 129.8, 129.5, 129.0, 128.8, 127.9, 127.1, 124.0; IR (ATR, *ν*/cm^{-1}): 1666, 1648, 1413, 740; HRMS (ESI) calcd for $C_{16}H_{10}NaO_2S$ $[M + Na]^+$ 289.0294, found 289.0291.

3aa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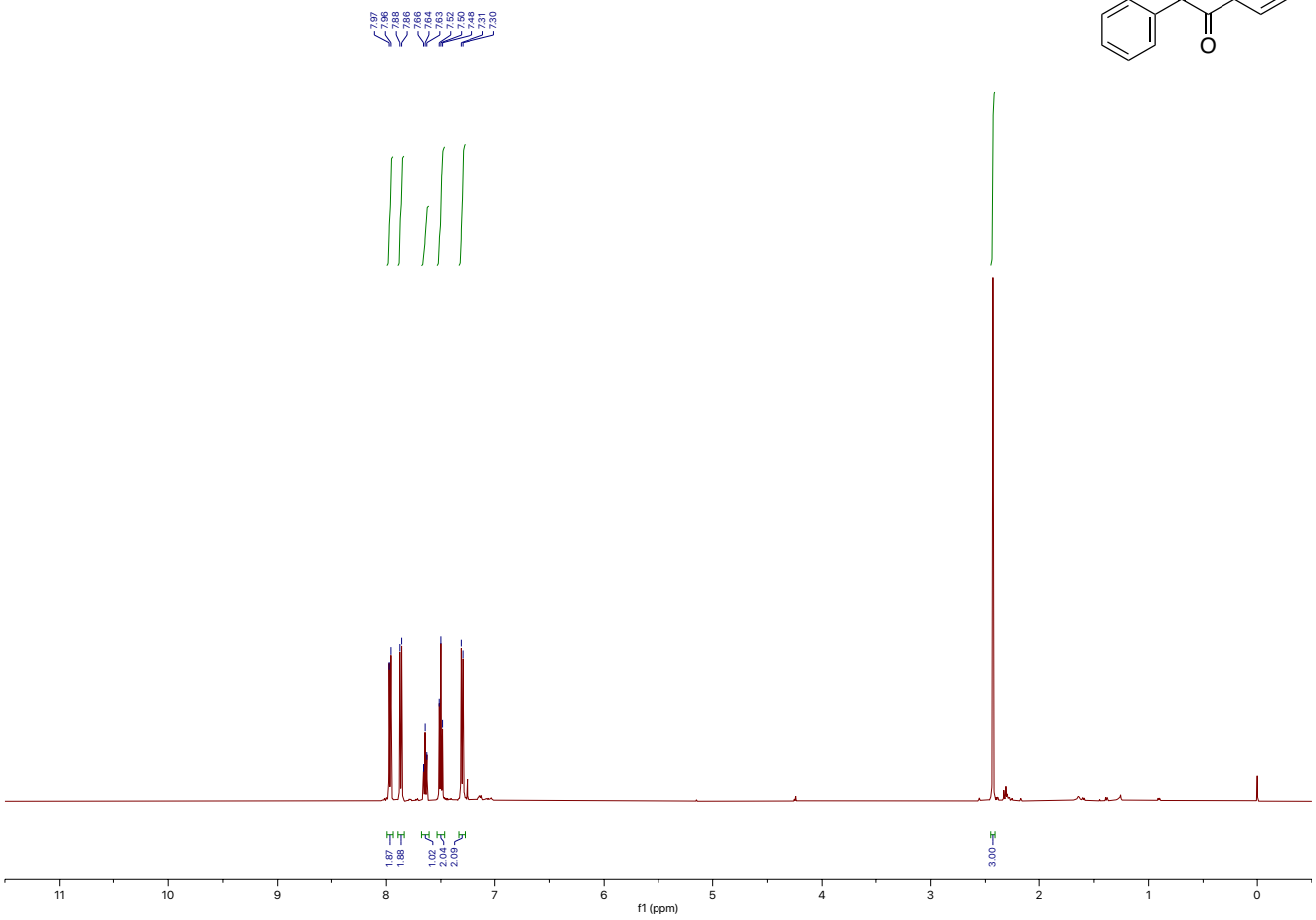
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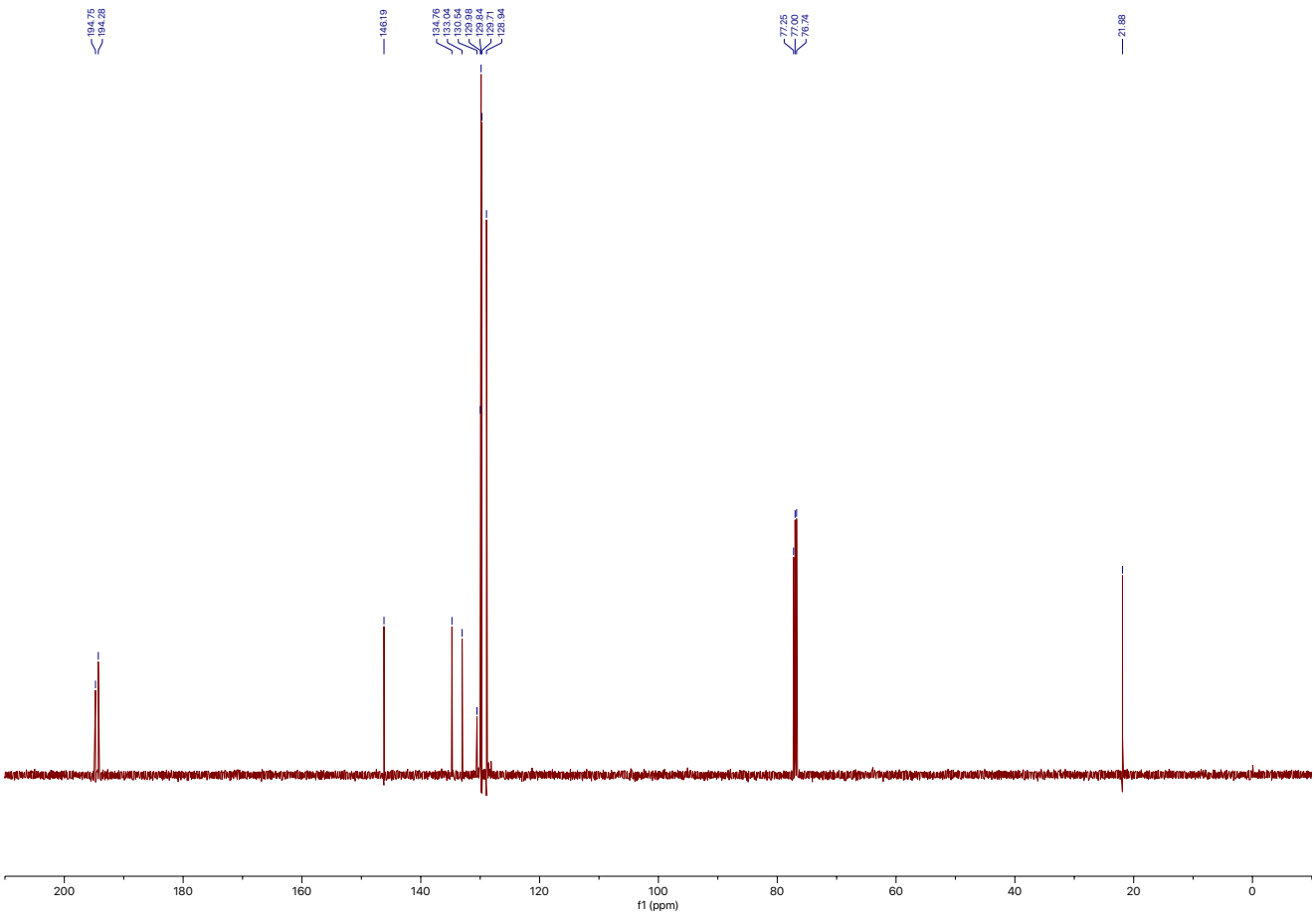
f1 (ppm)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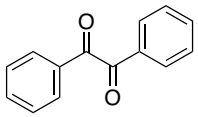
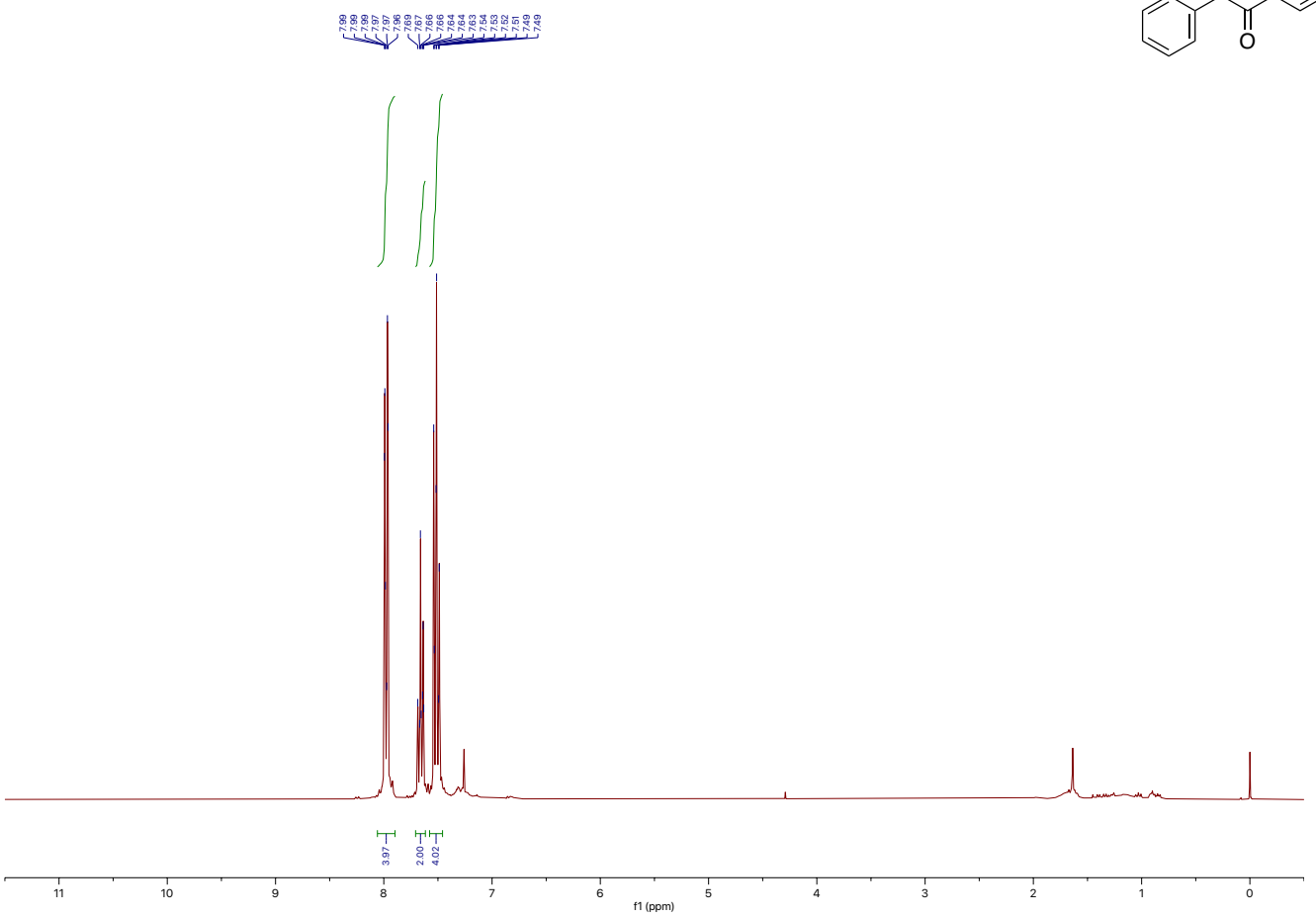
3ab

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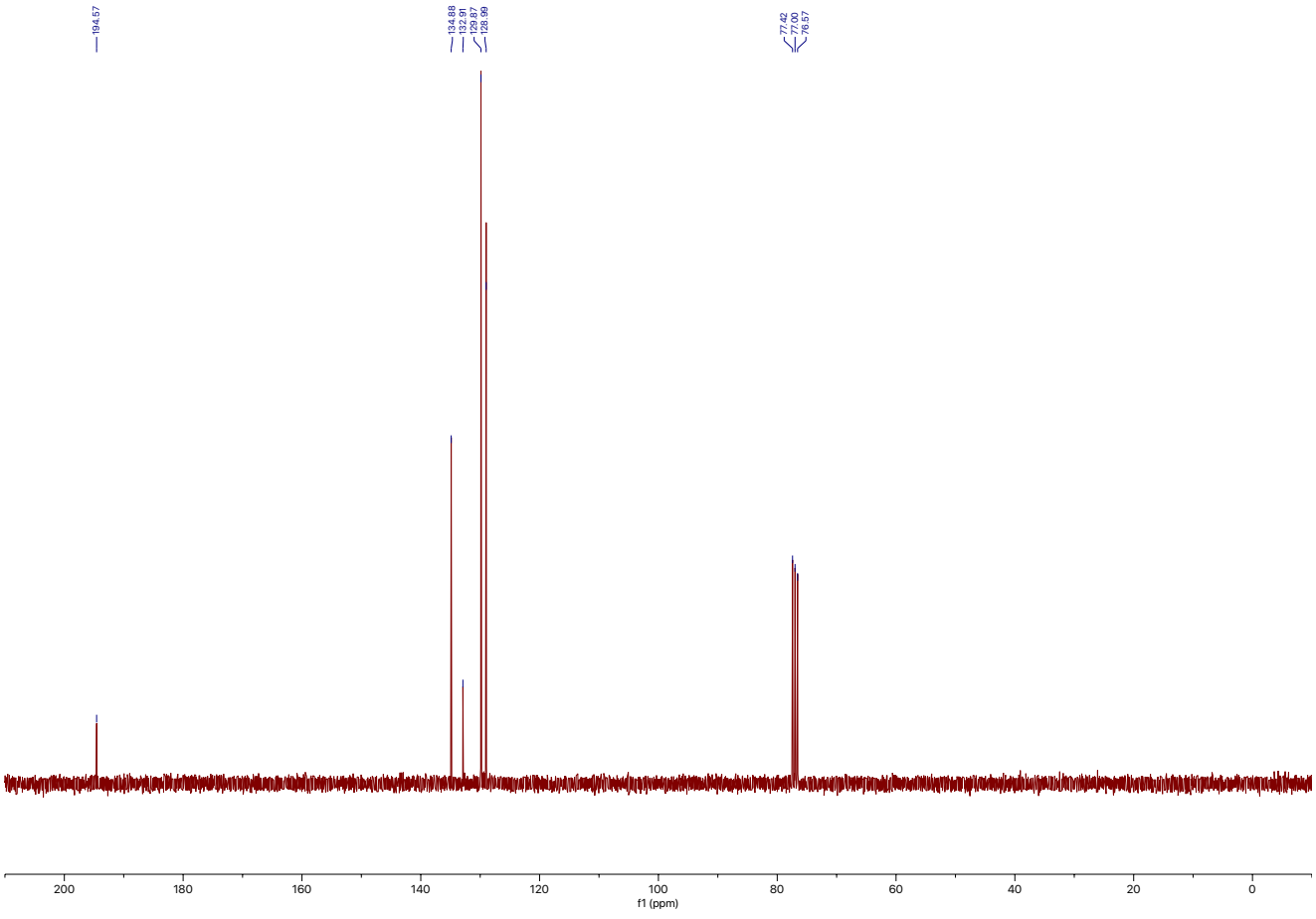
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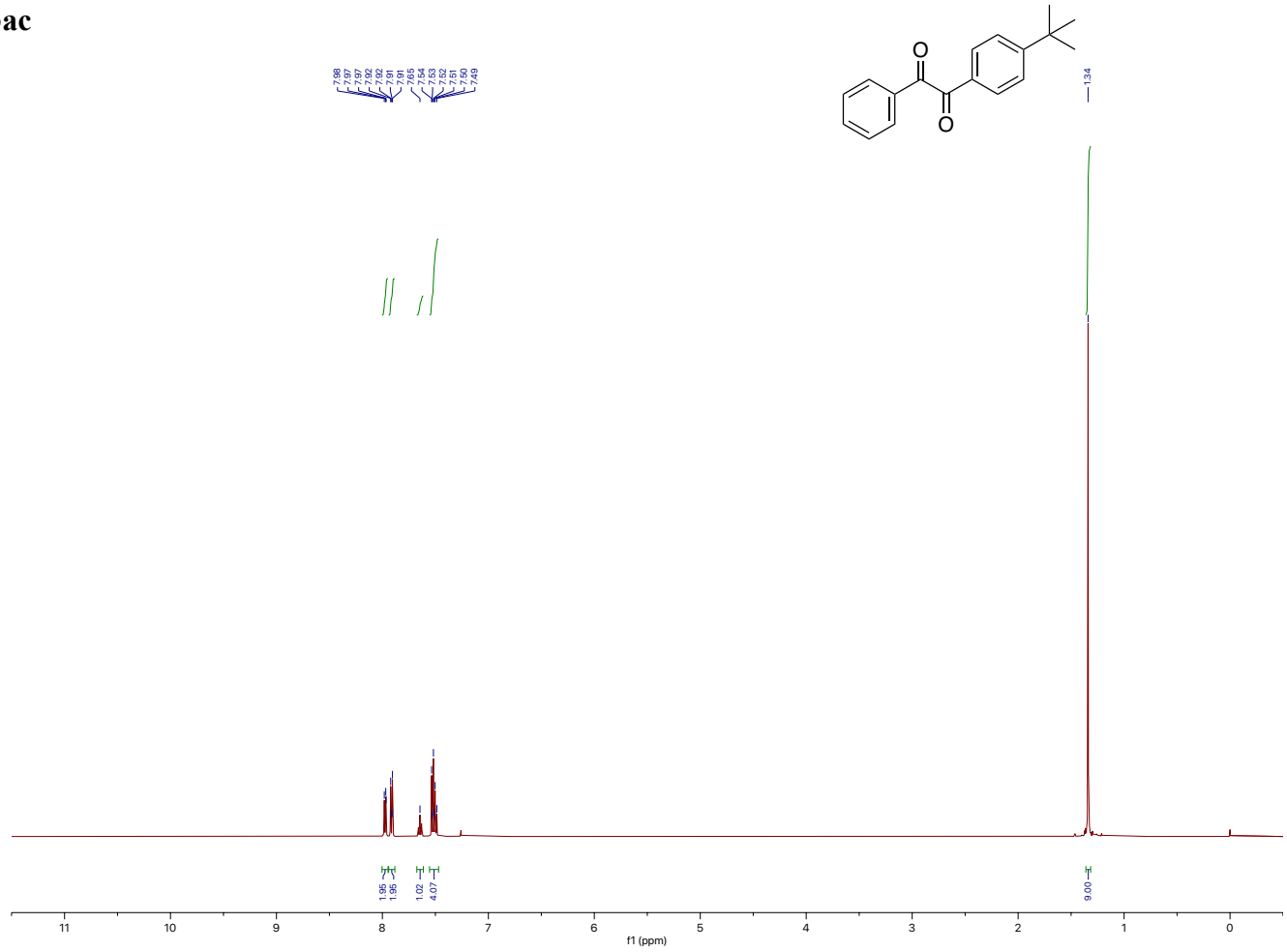
3ac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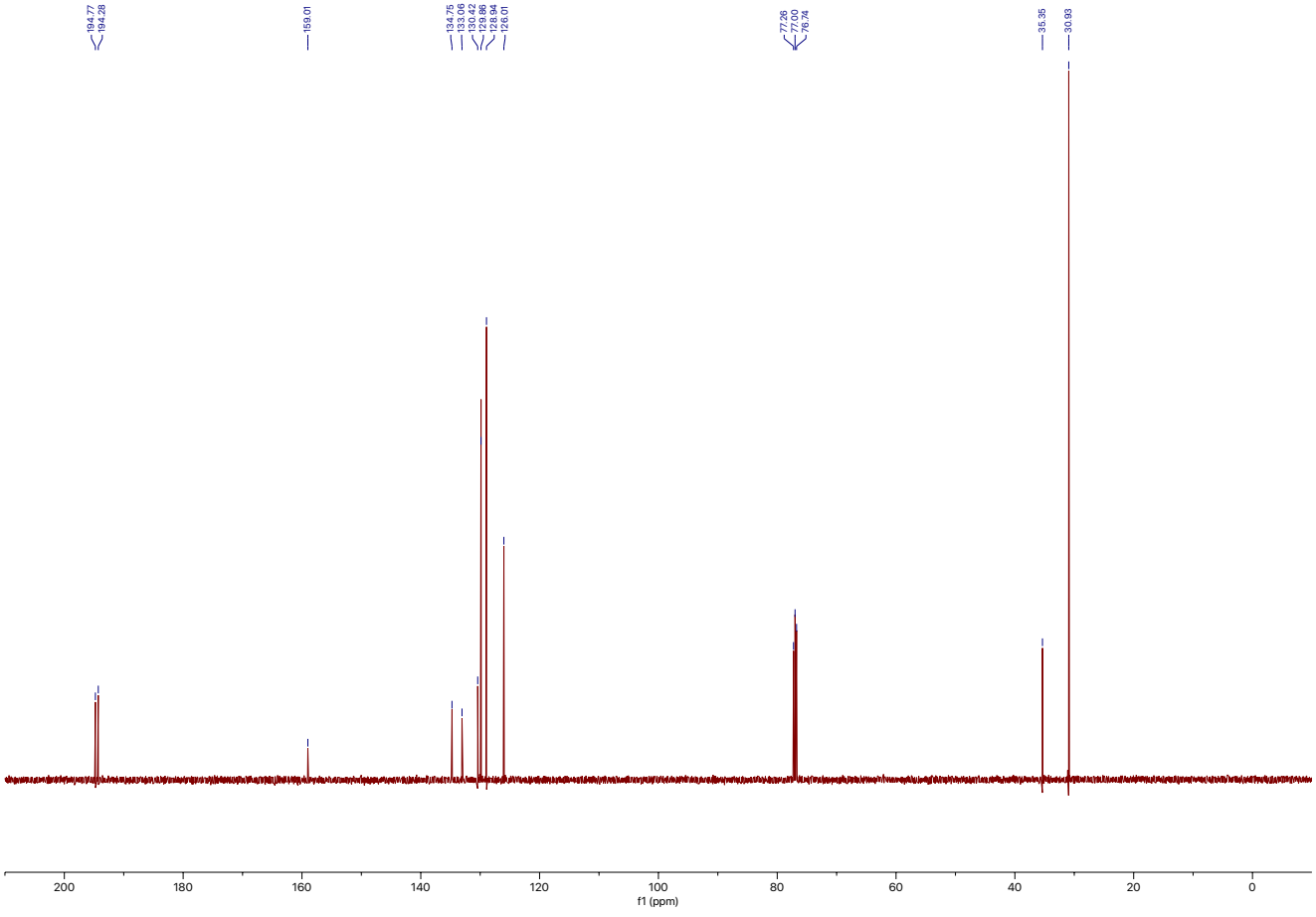
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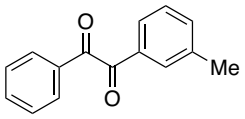
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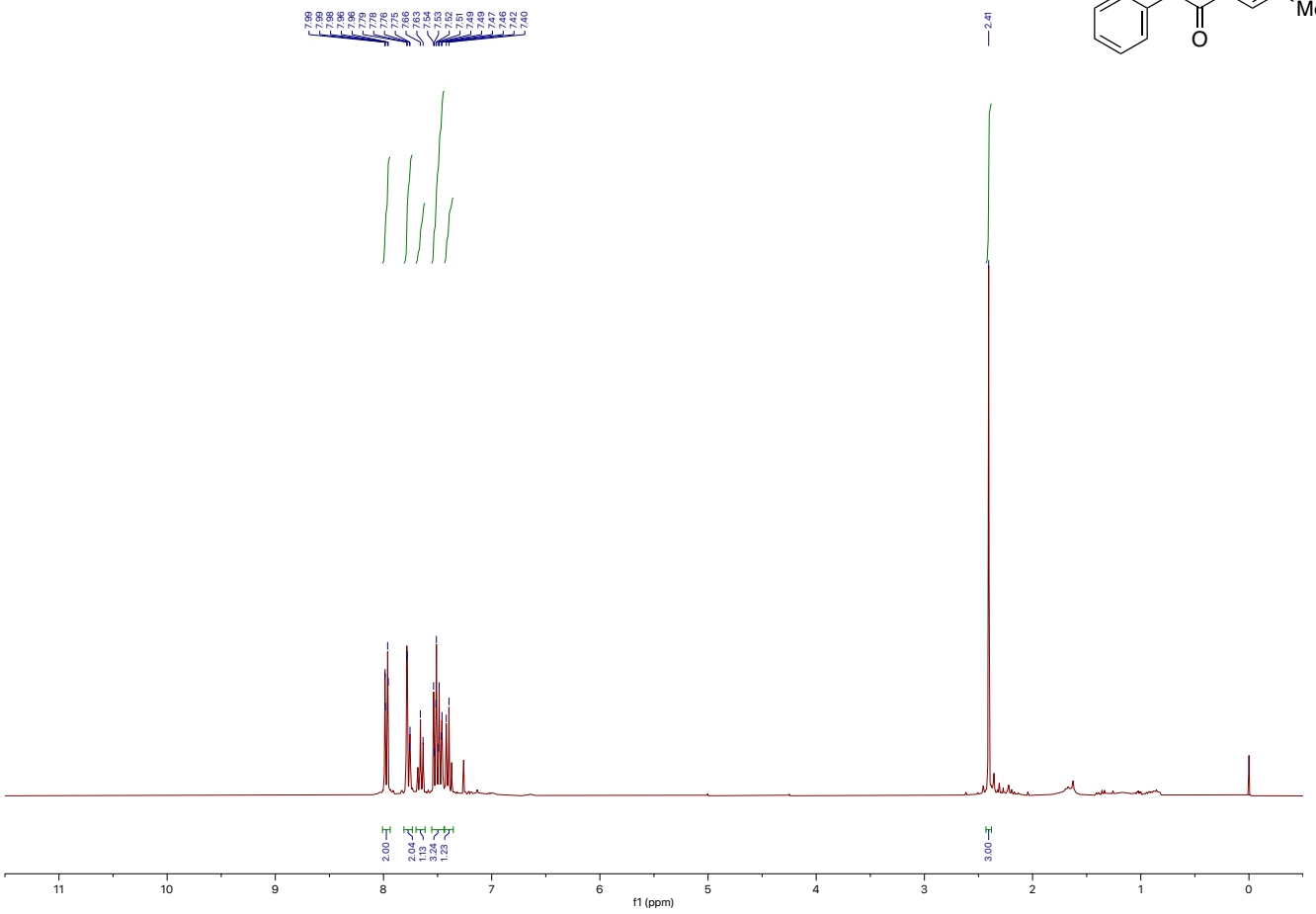
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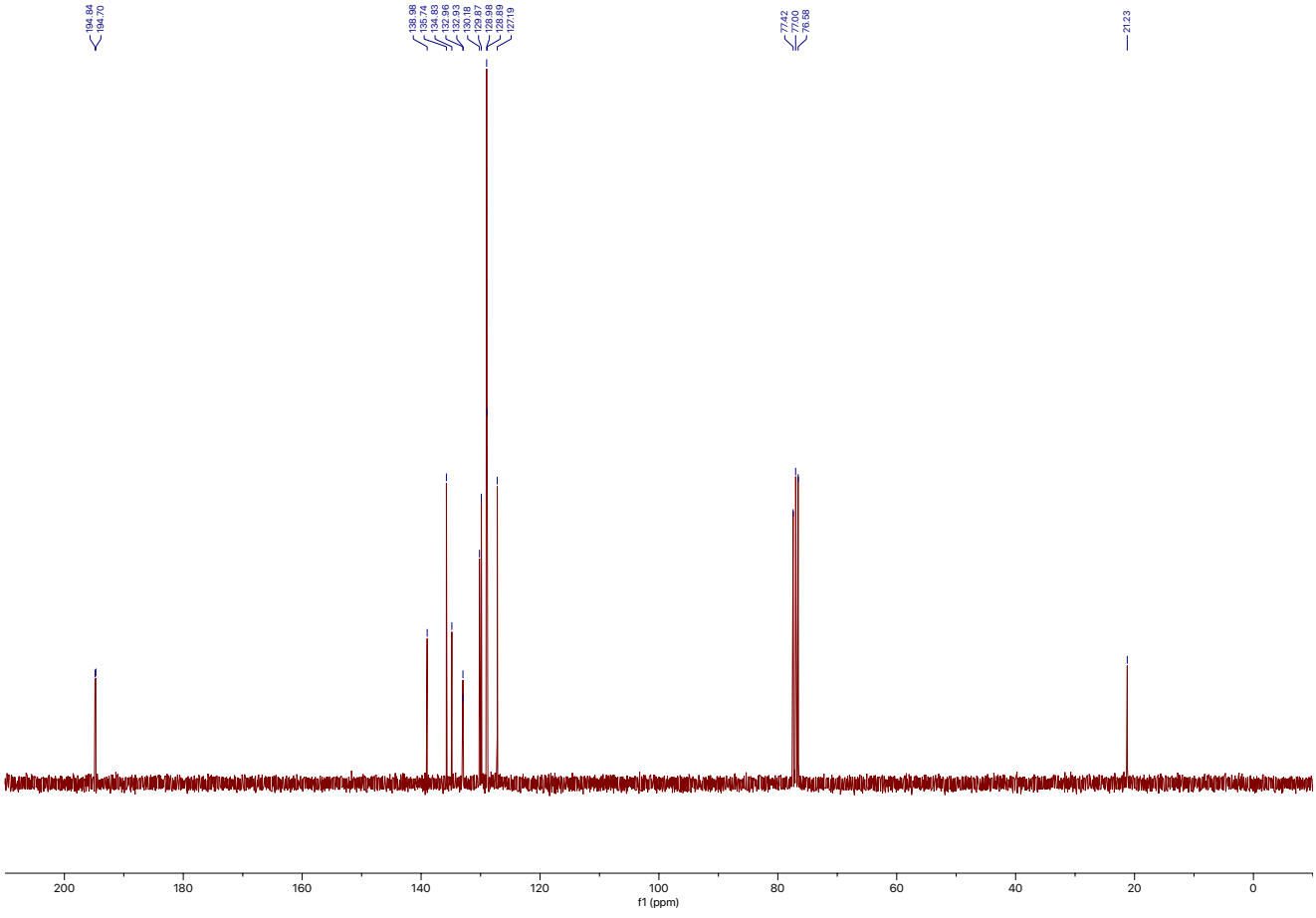
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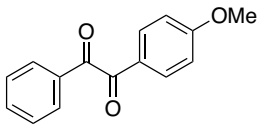
3ae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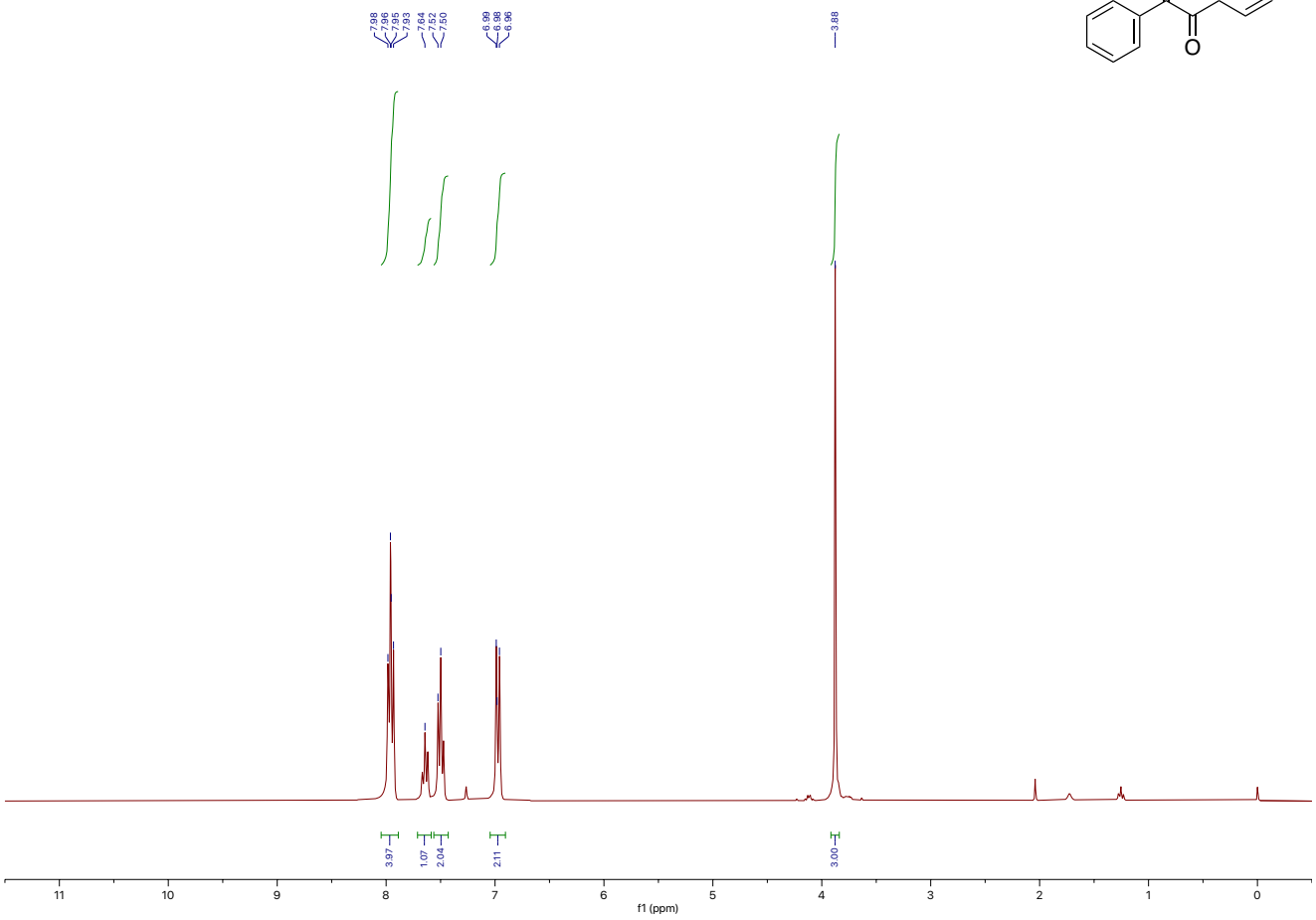
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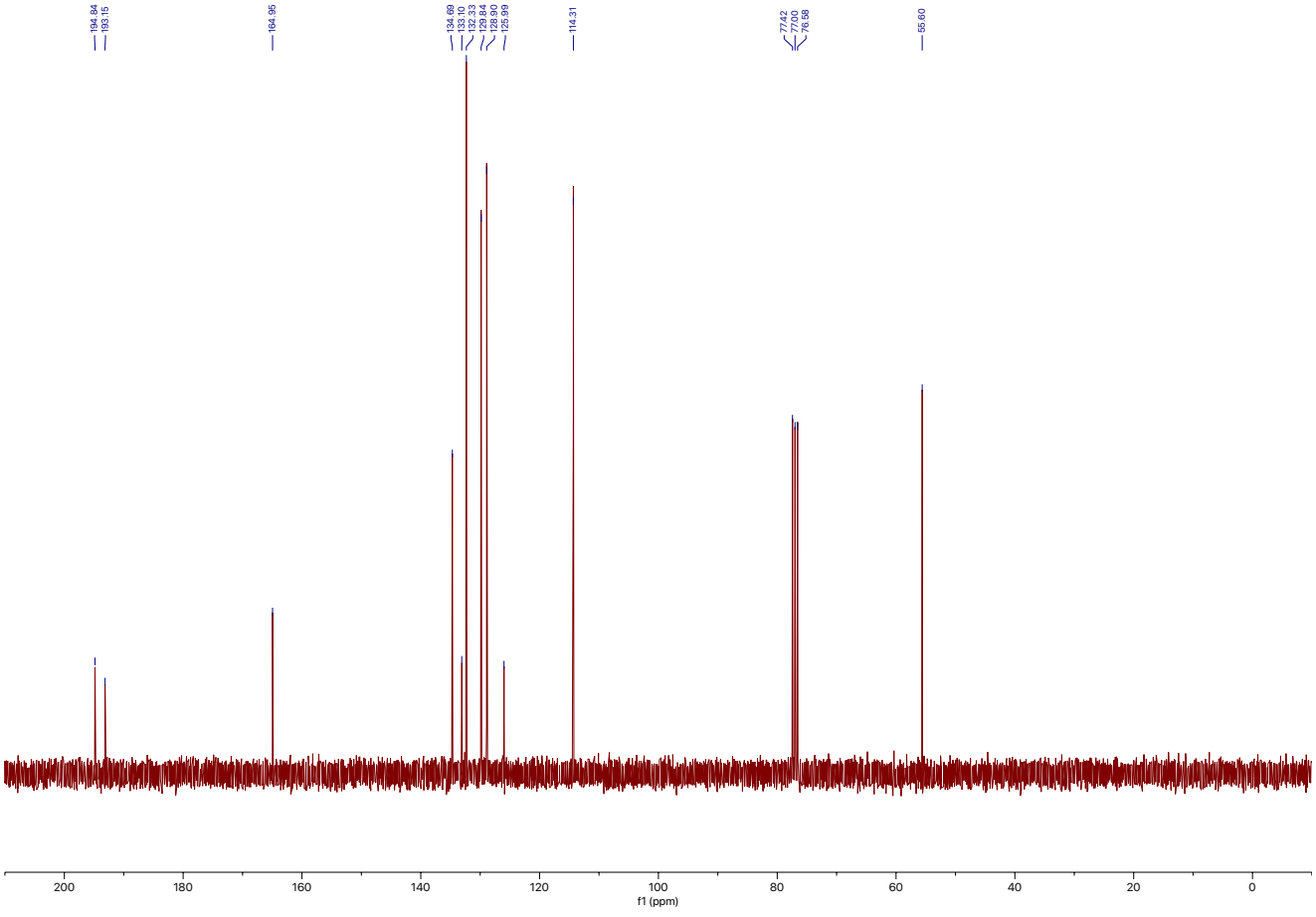
f1 (ppm)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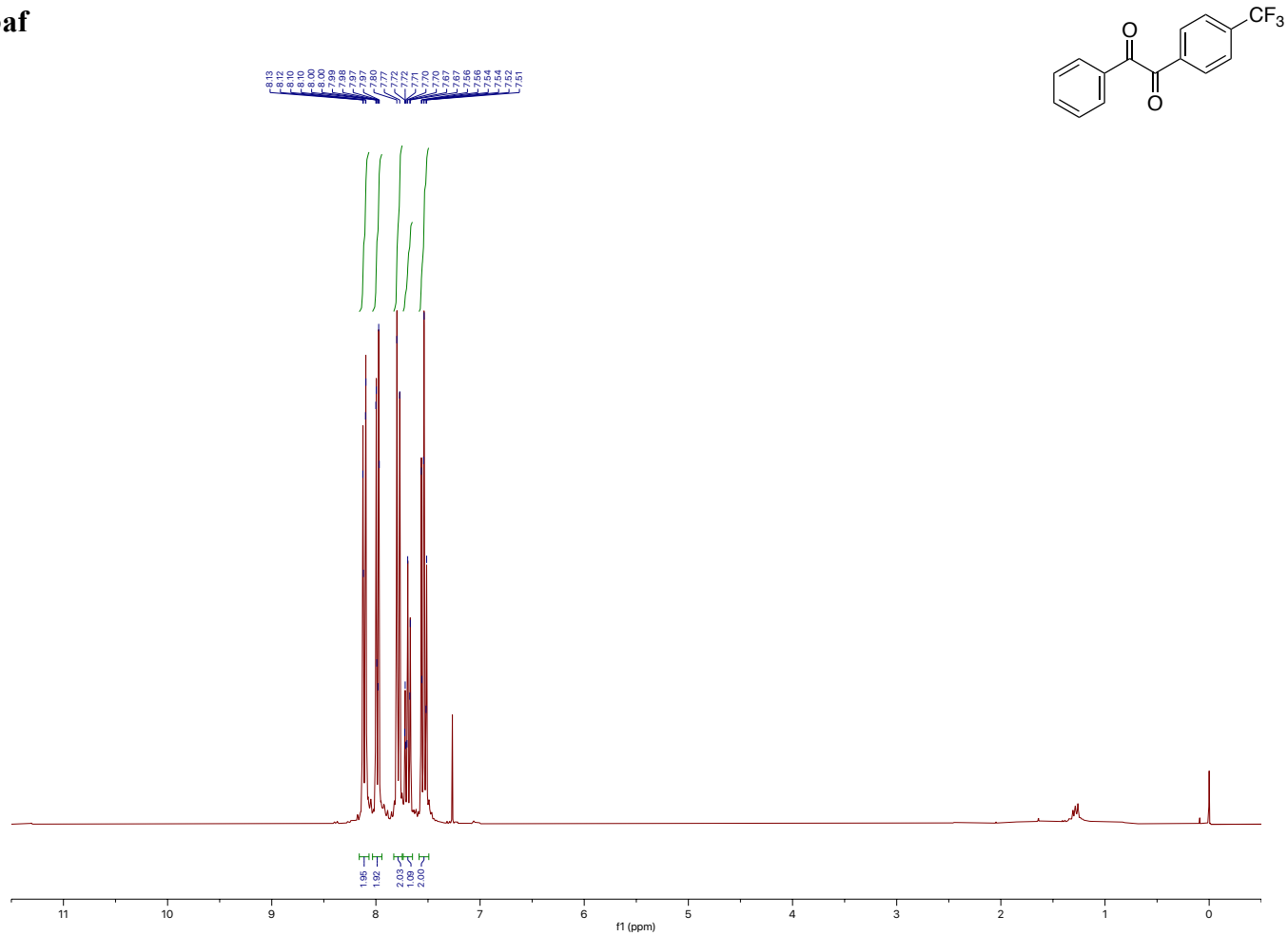
3af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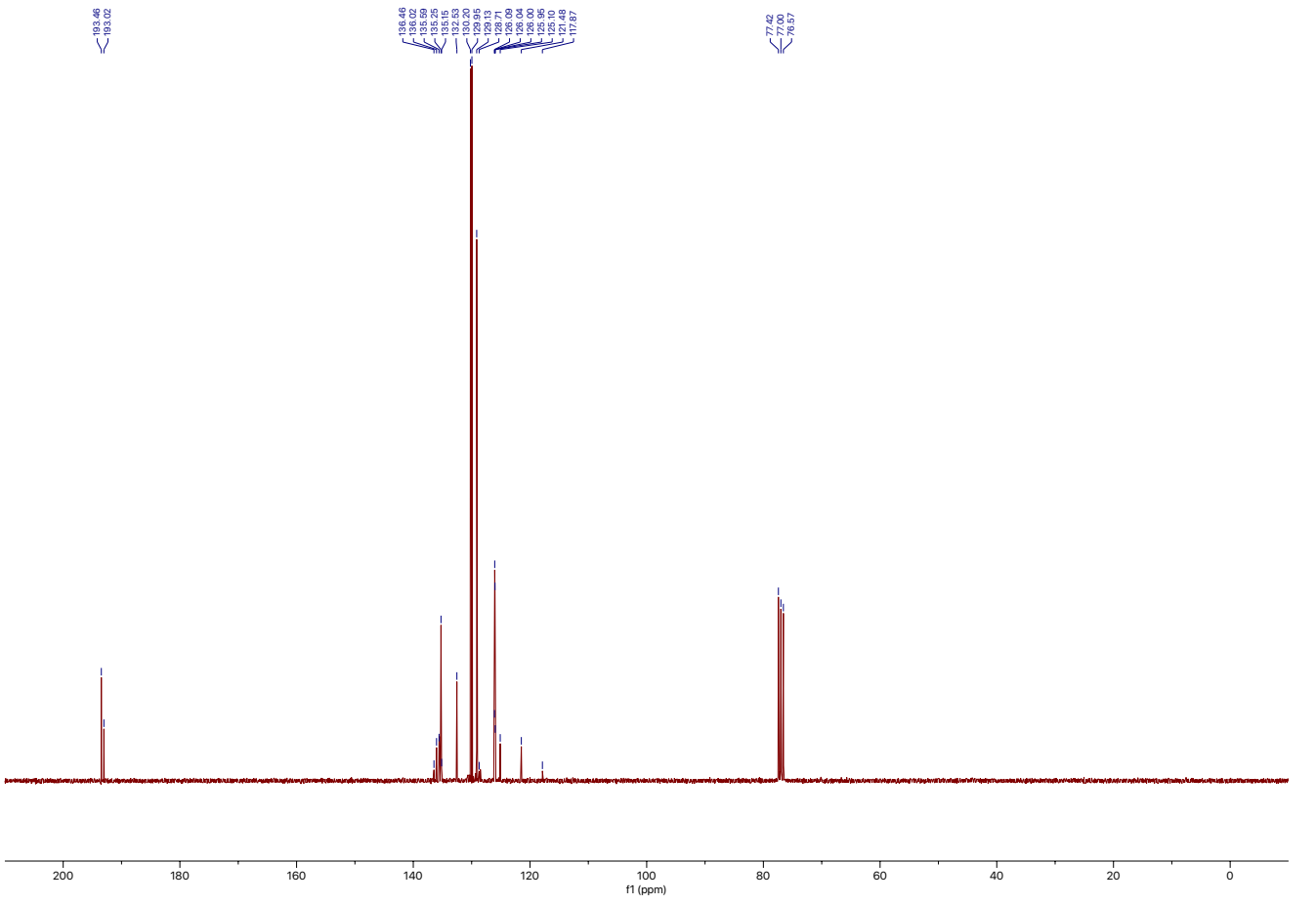
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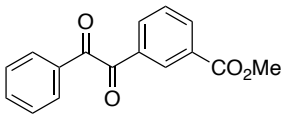
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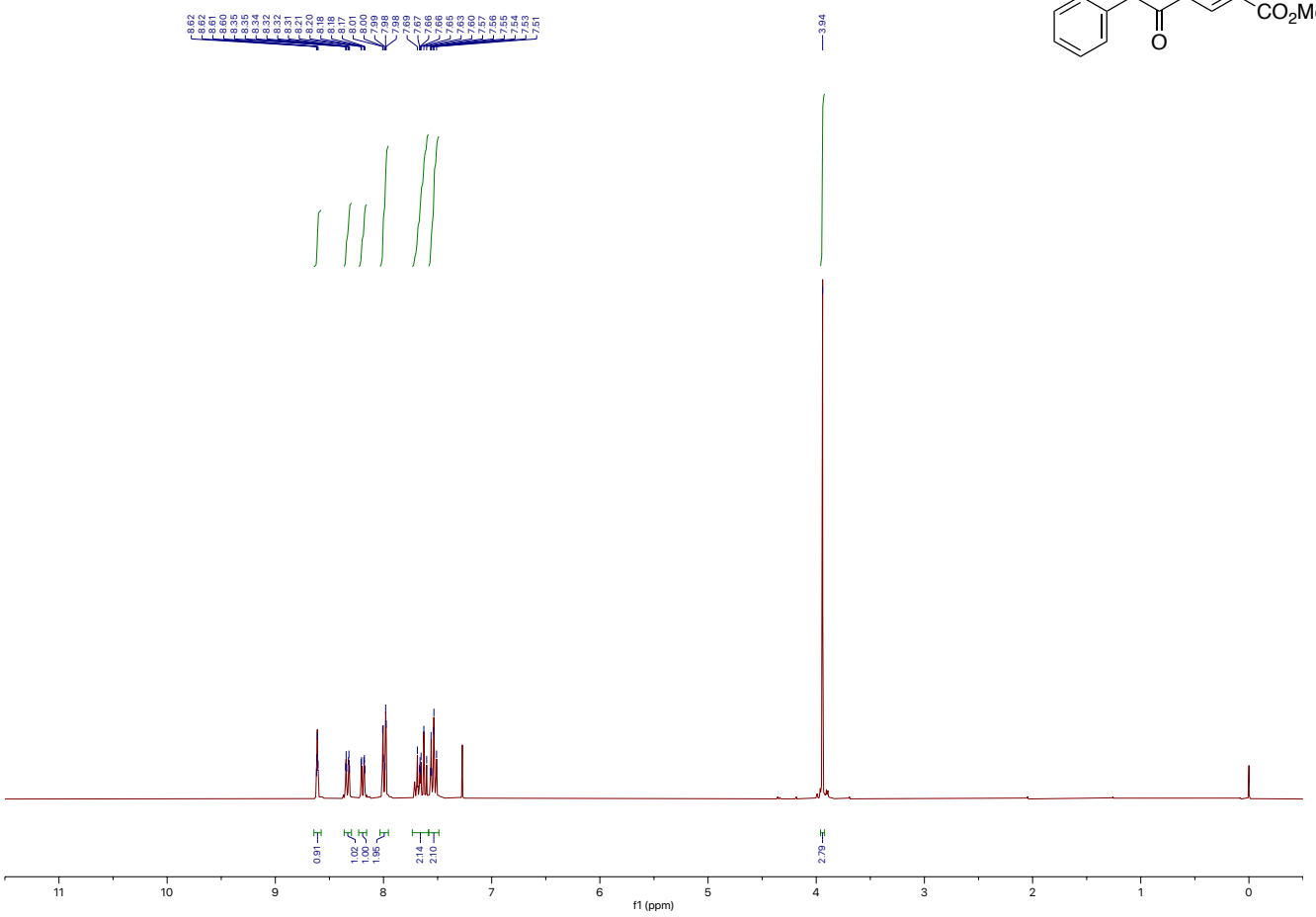
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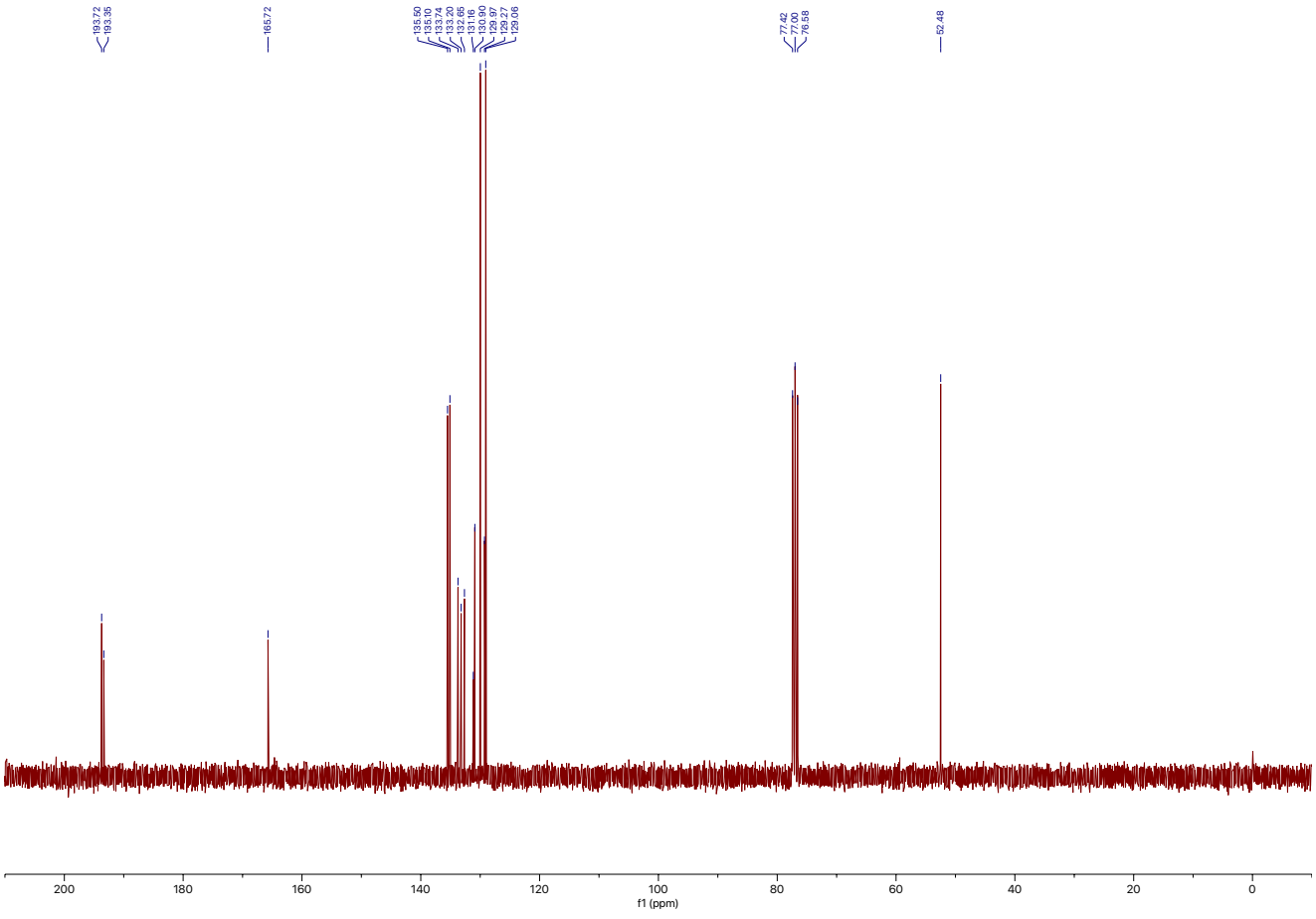
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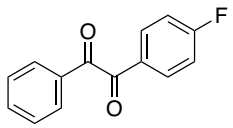
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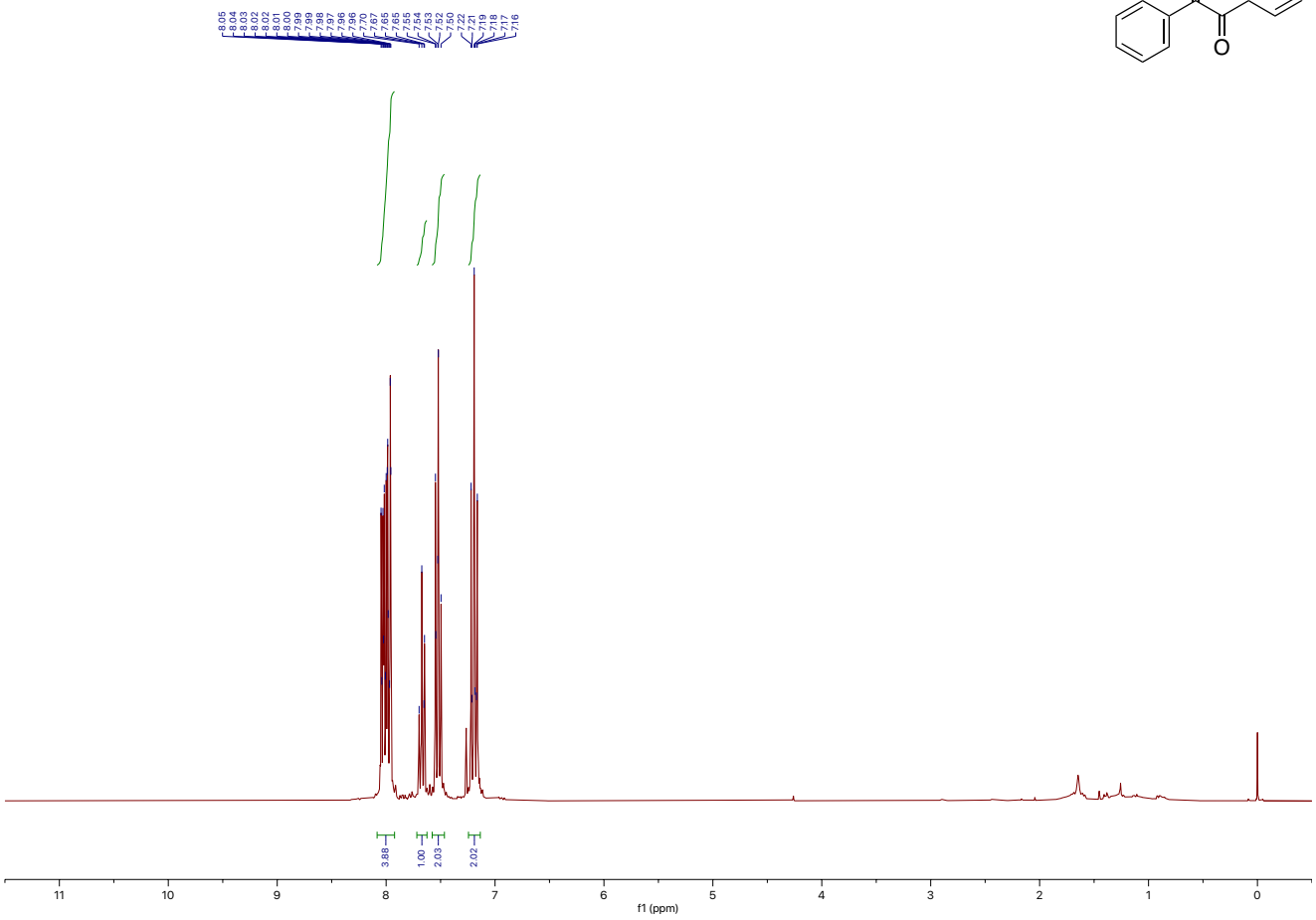
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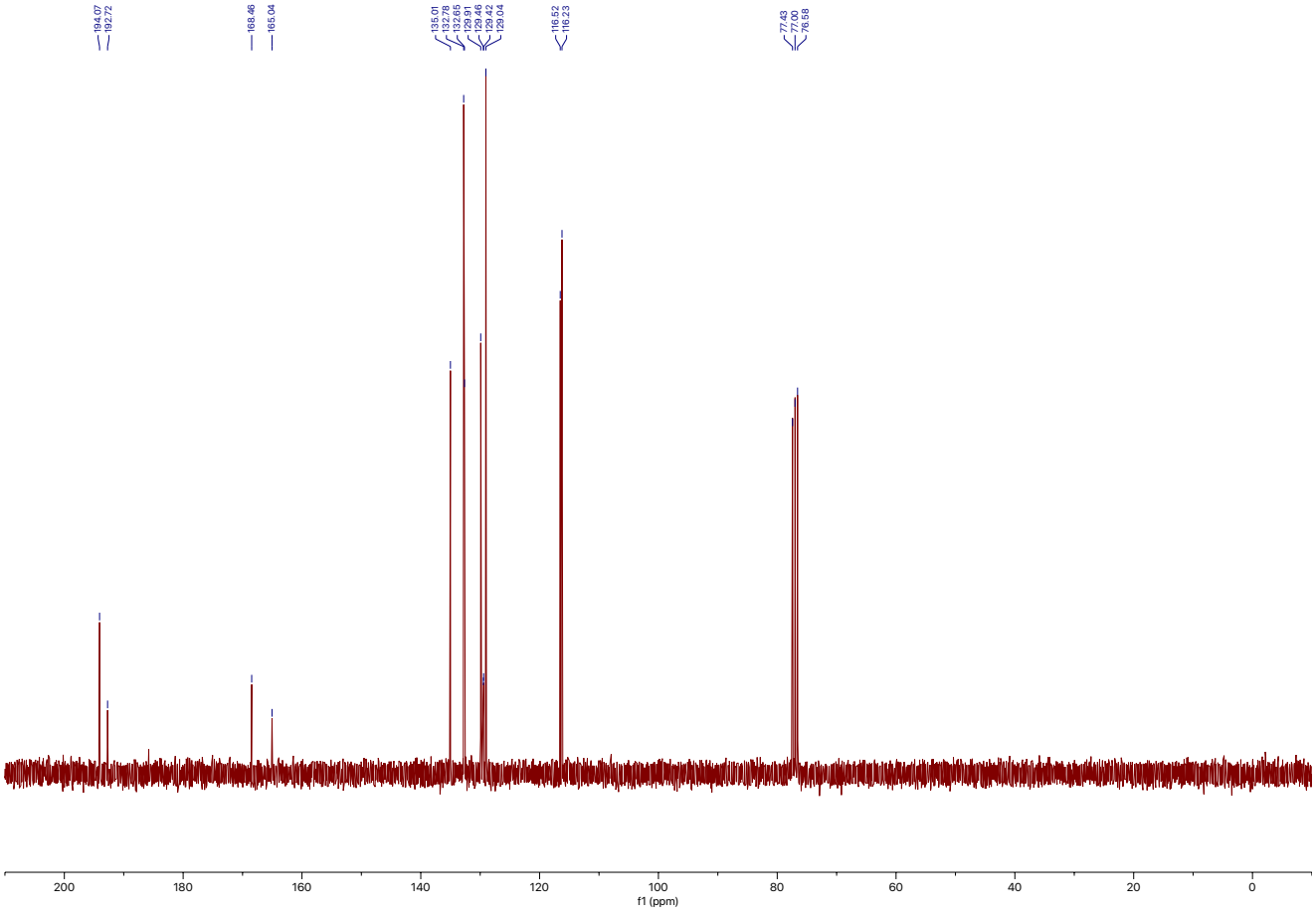
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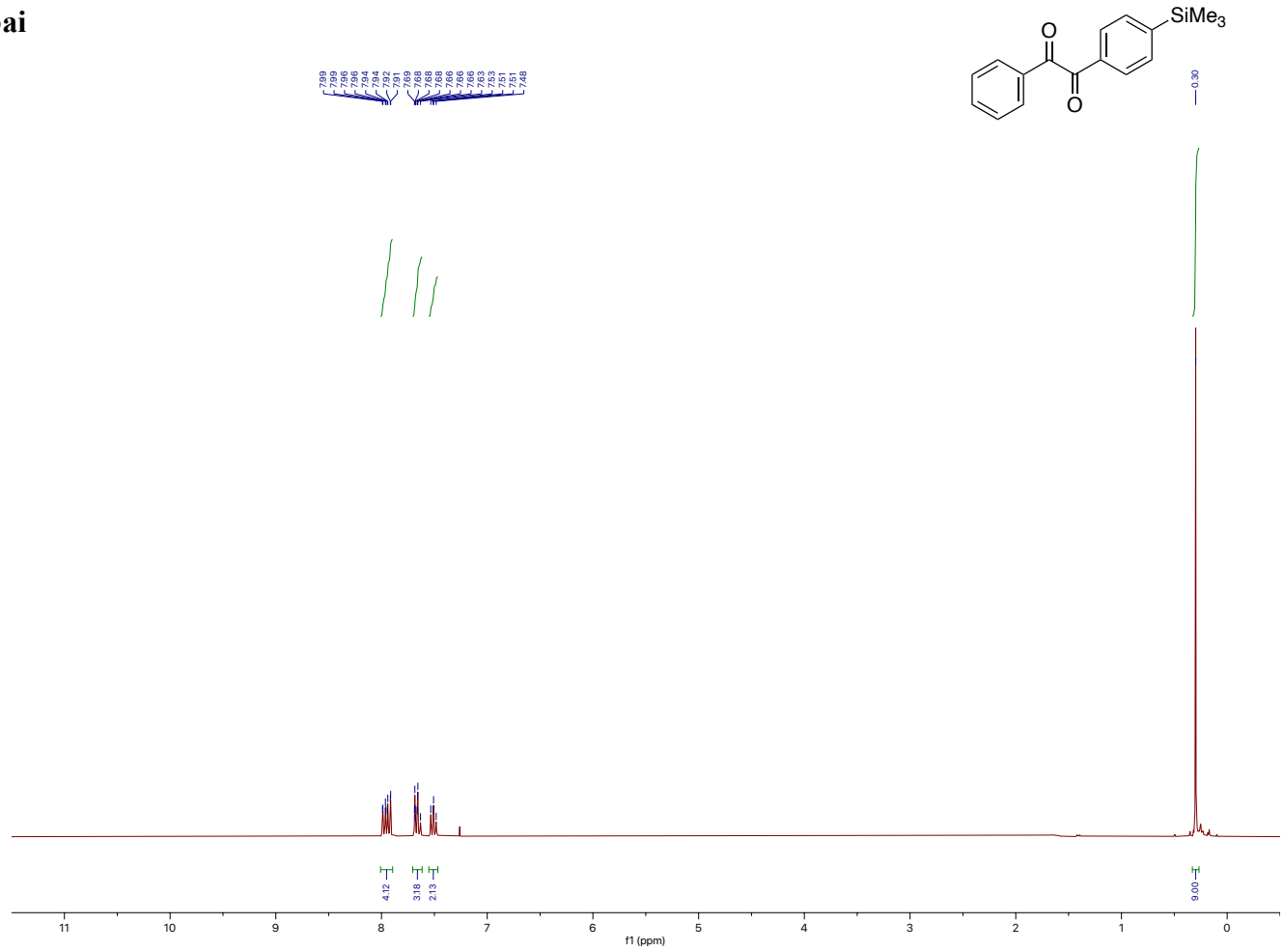
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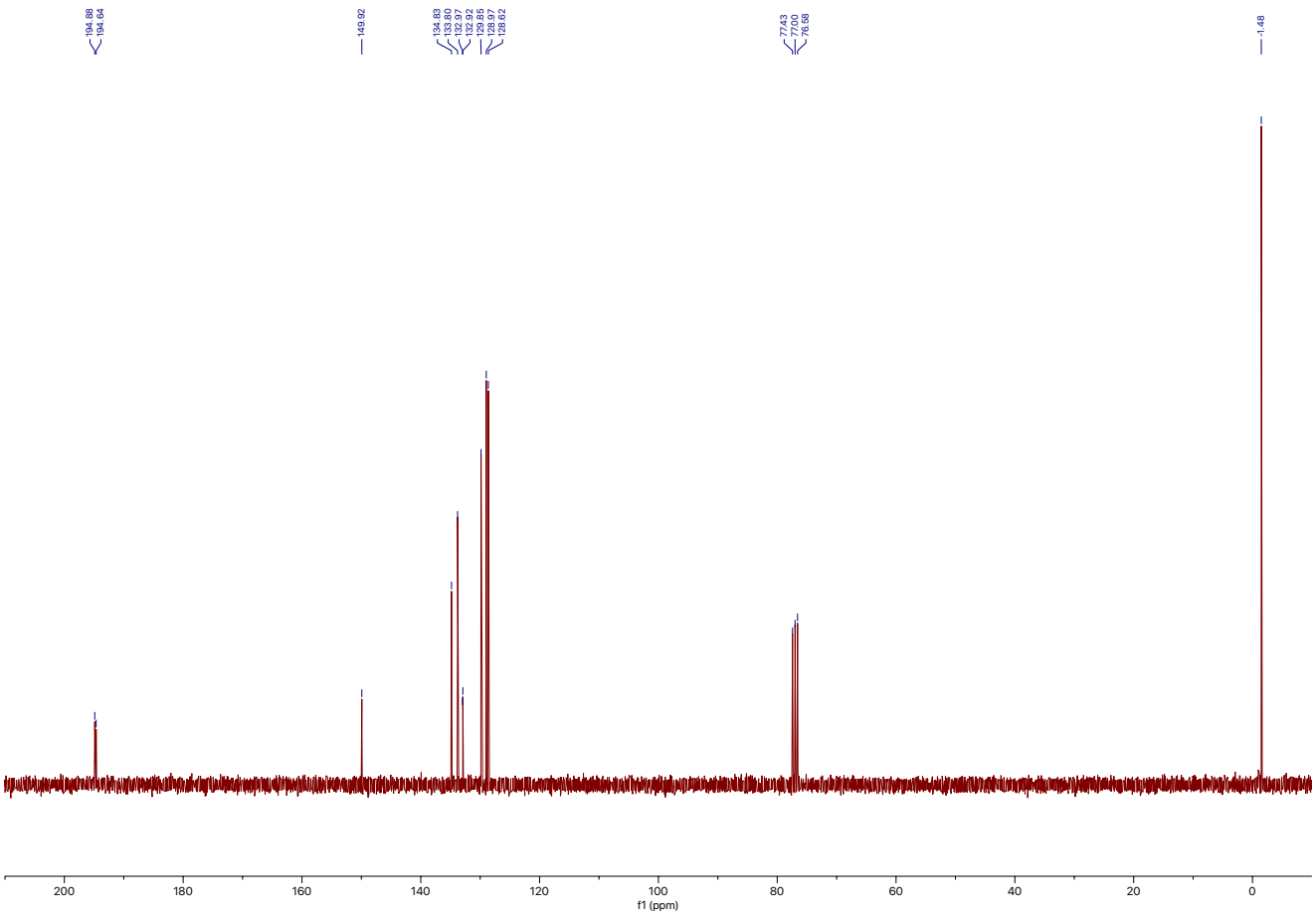
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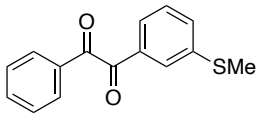
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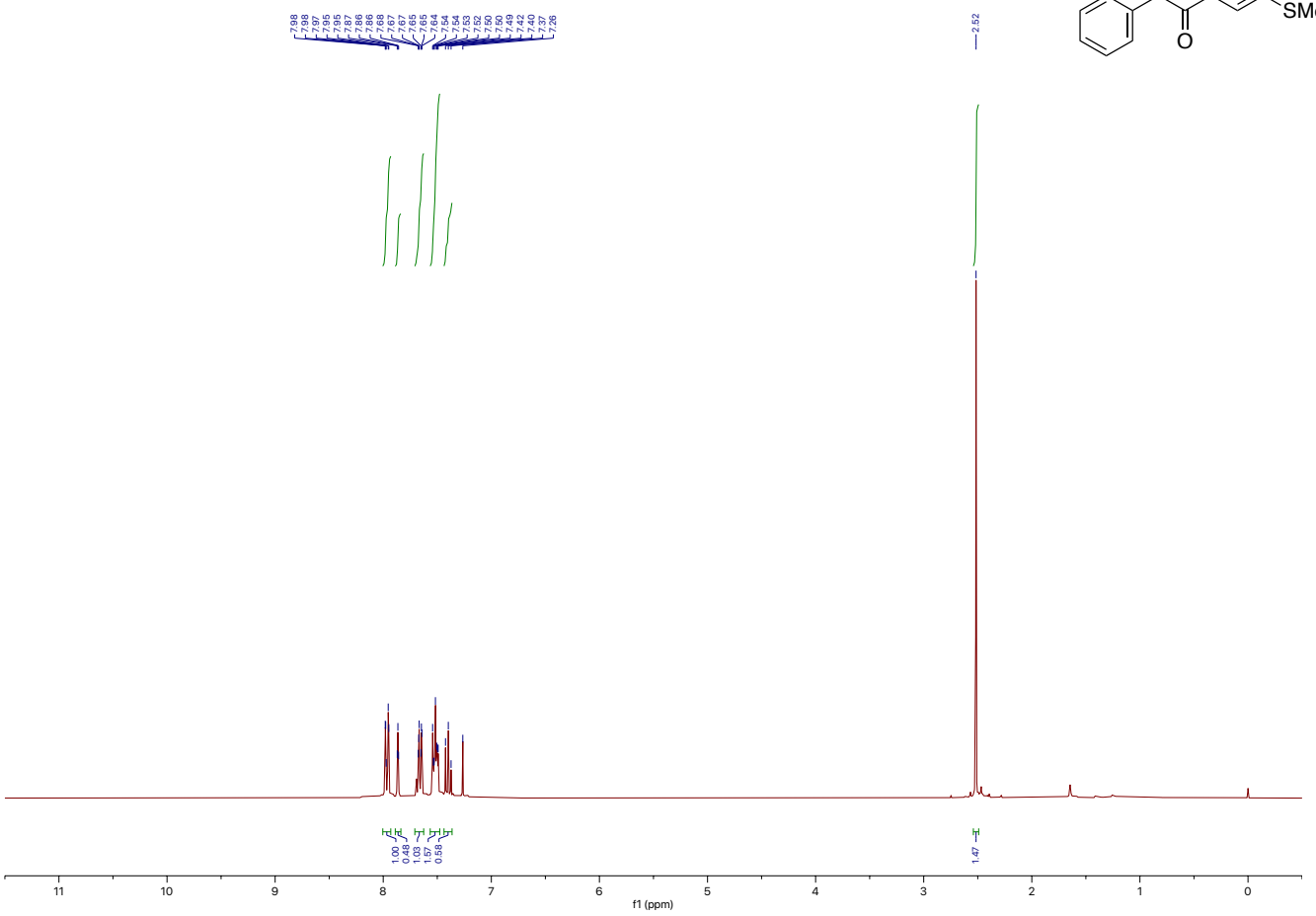
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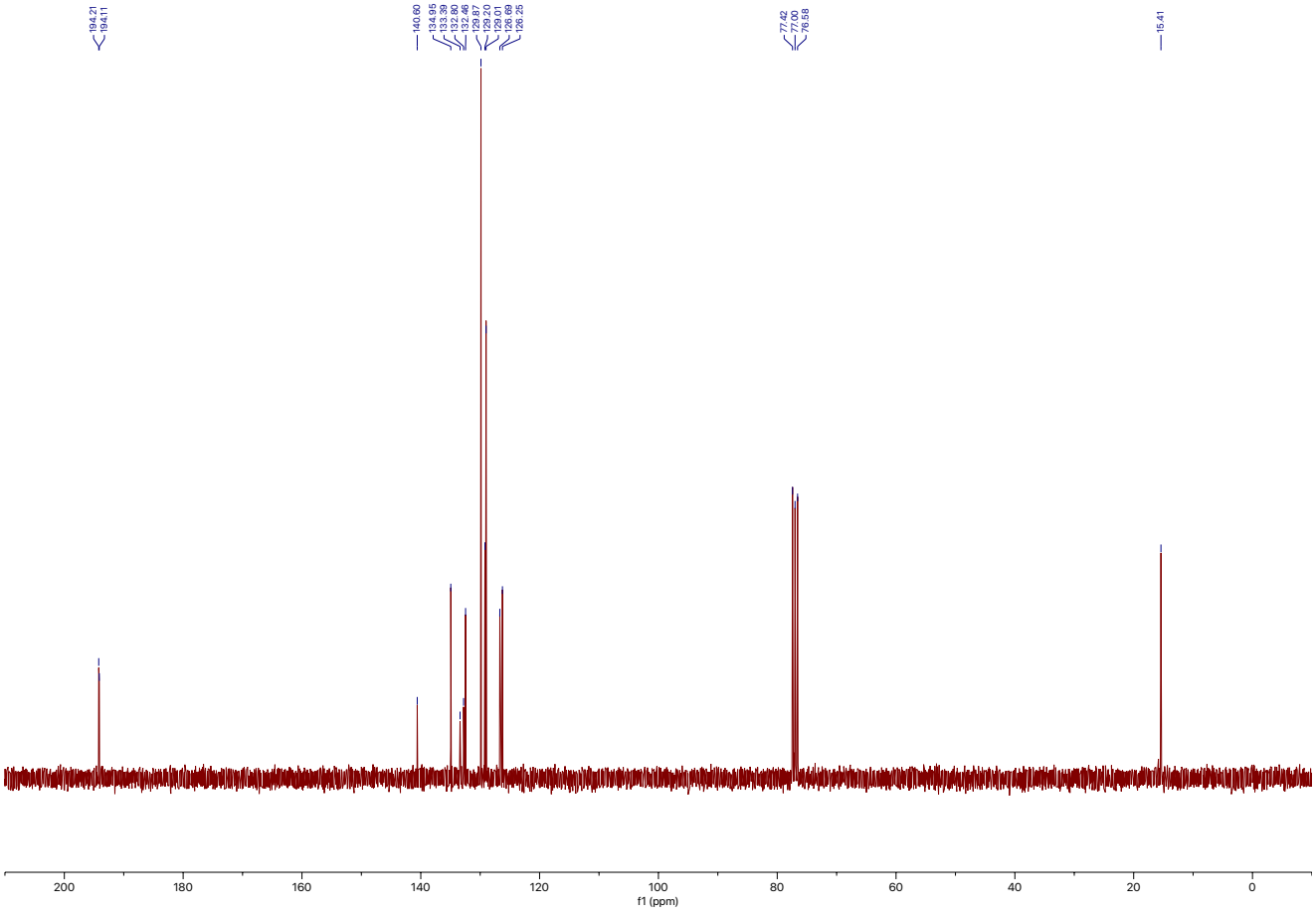
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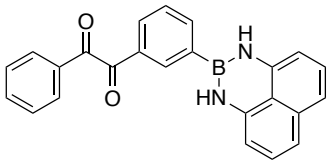
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HN

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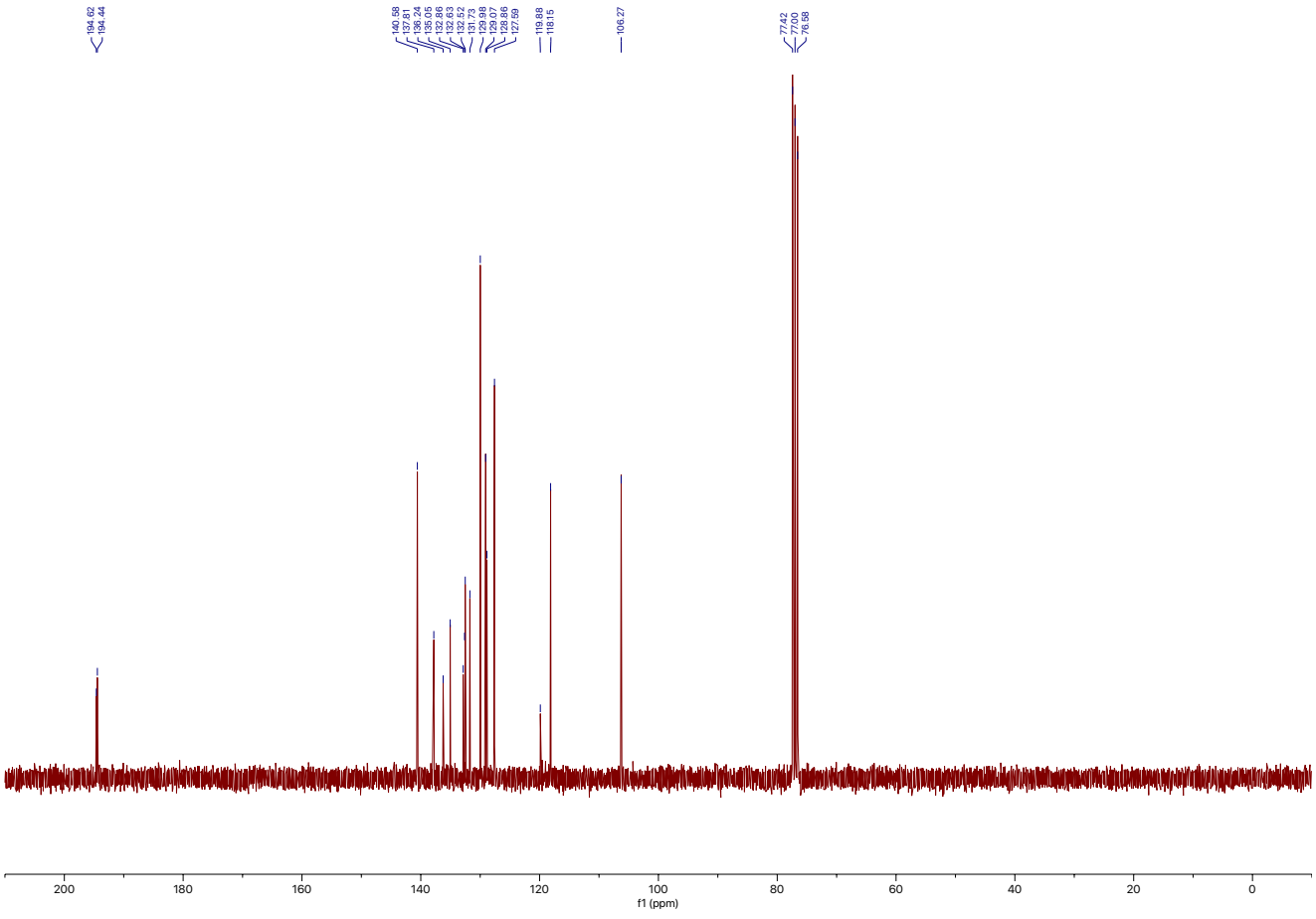
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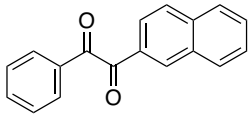

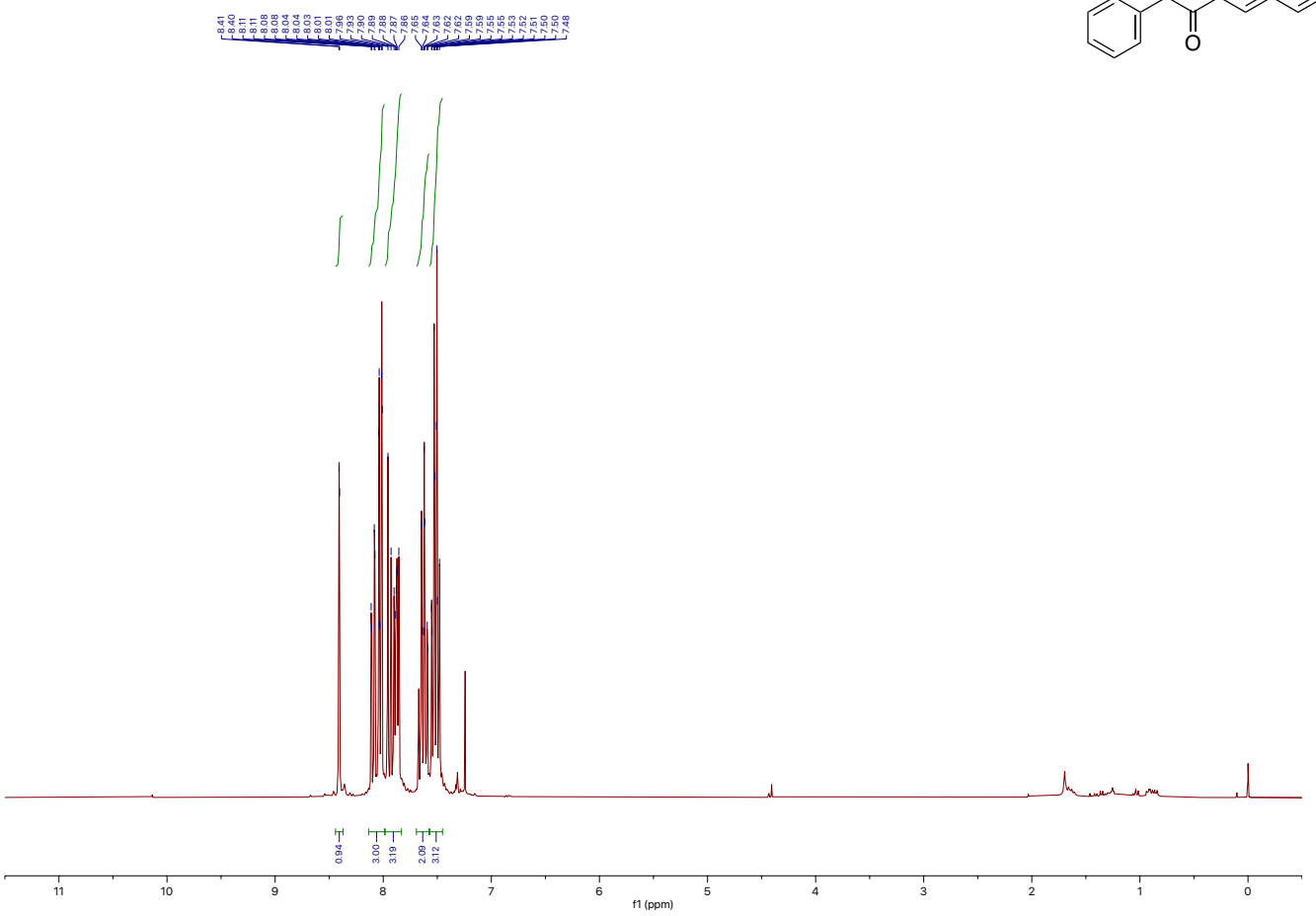
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8.01
7.96
7.95
7.90
7.89
7.88
7.87
7.86
7.65
7.64
7.63
7.62
7.62
7.59
7.59
7.55
7.55
7.53
7.52
7.51
7.50
7.50
7.48
0.94
3.00
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3.12
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7
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5
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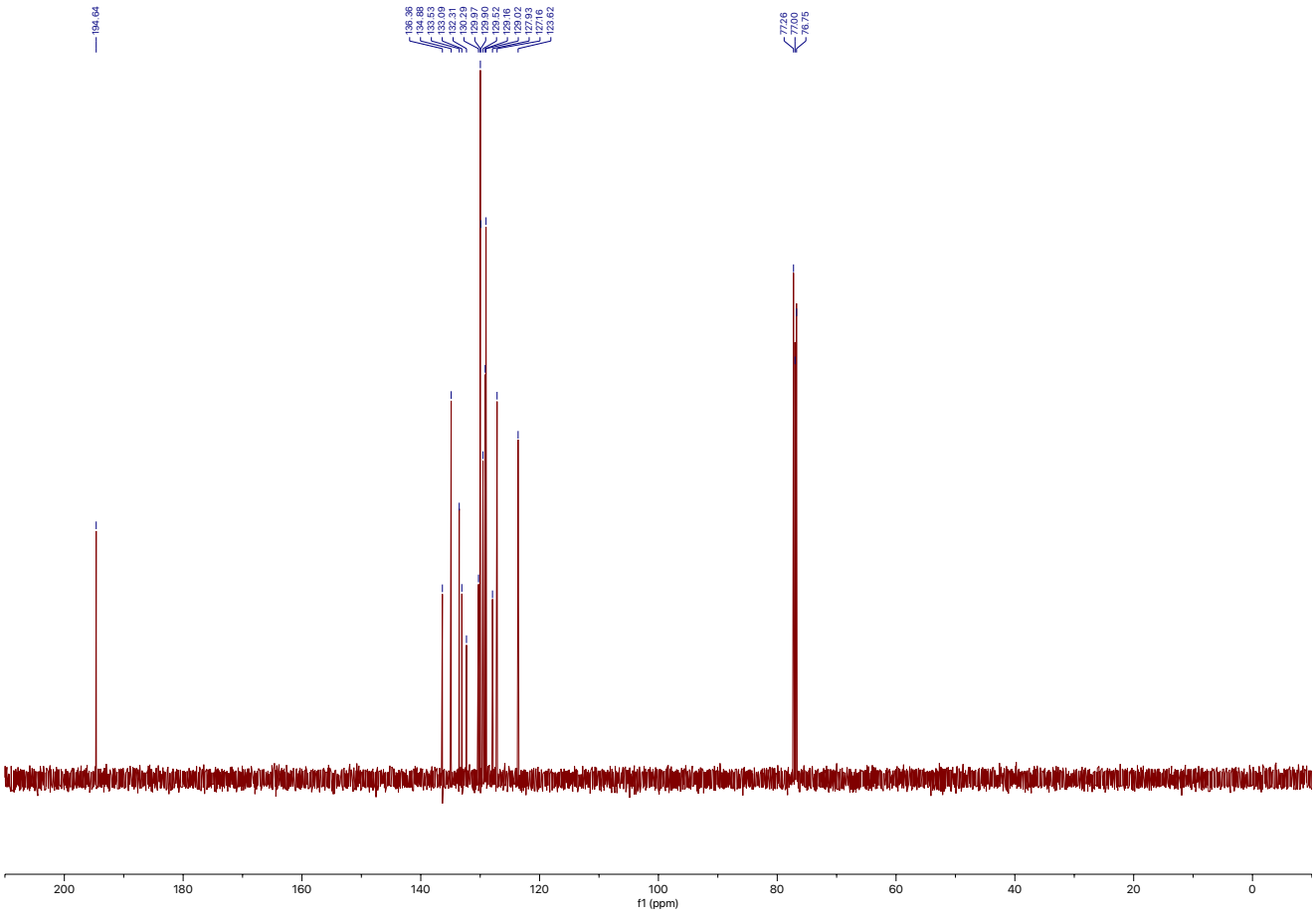
1
0
f1 (ppm)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134.88
133.53
133.09
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132.29
129.97
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129.52
129.16
129.05
129.02
127.93
127.16
123.62
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76.75
200
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160
140
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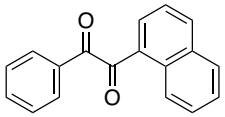
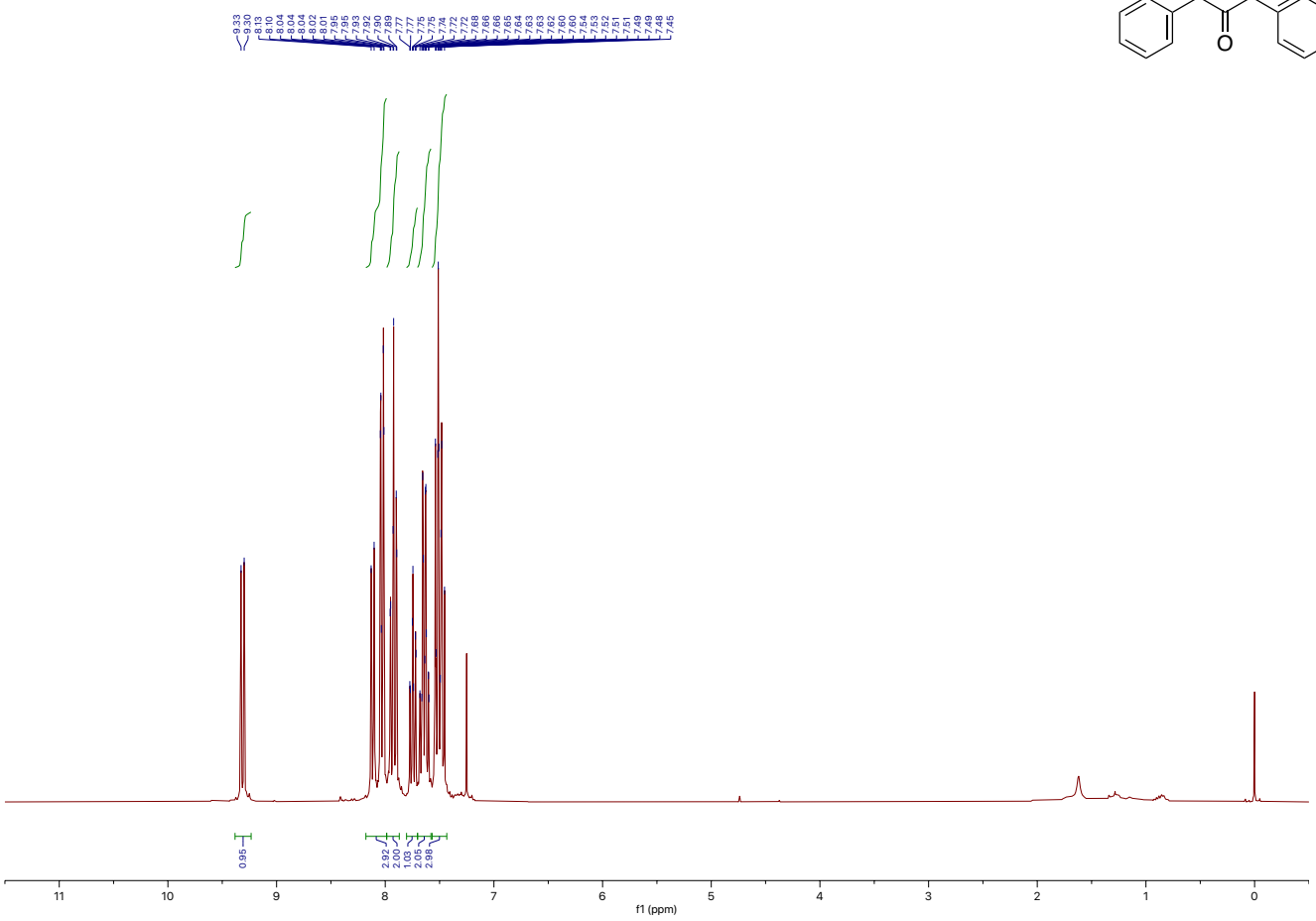
60
40
20
0
f1 (ppm)

3am

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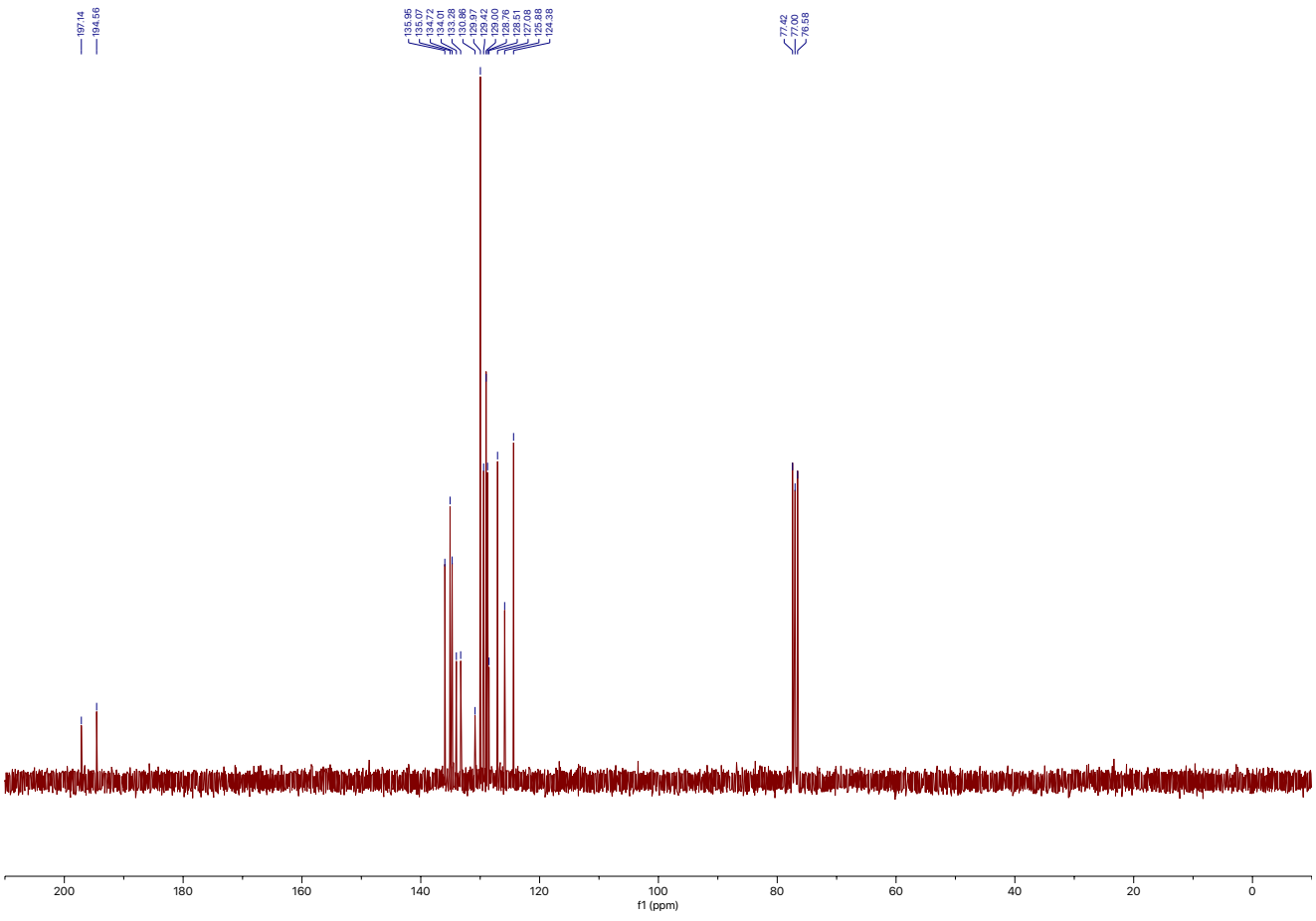
8.04
8.02
8.01
7.95
7.93
7.92
7.90
7.89
7.77
7.77
7.75
7.74
7.72
7.72
7.68
7.66
7.65
7.64
7.63
7.62
7.60
7.60
7.54
7.53
7.52
7.51
7.51
7.49
7.49
7.46
7.45
0.95
2.92
2.00
1.03
2.05
2.98
f1 (ppm)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134.01
133.38
133.28
130.88
129.97
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128.51
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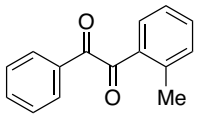
125.88
124.38
77.42
77.00
76.58
f1 (ppm)

3an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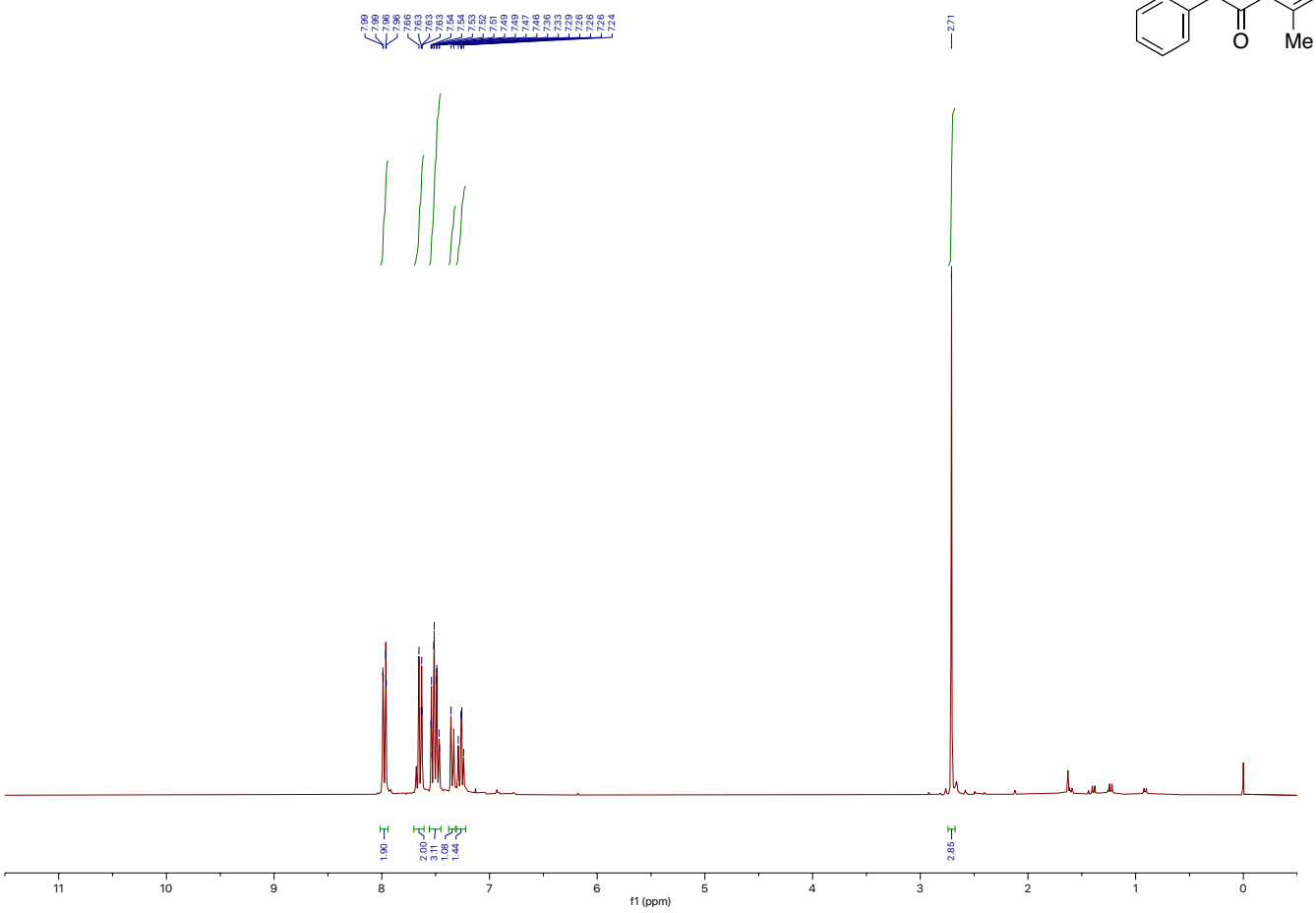
O
Me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7.52
7.51
7.49
7.49
7.47
7.46
7.36
7.33
7.29
7.26
7.26
7.26
7.24
2.71
1.90
2.00
3.01
1.08
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2.85
11
10
9
8
7
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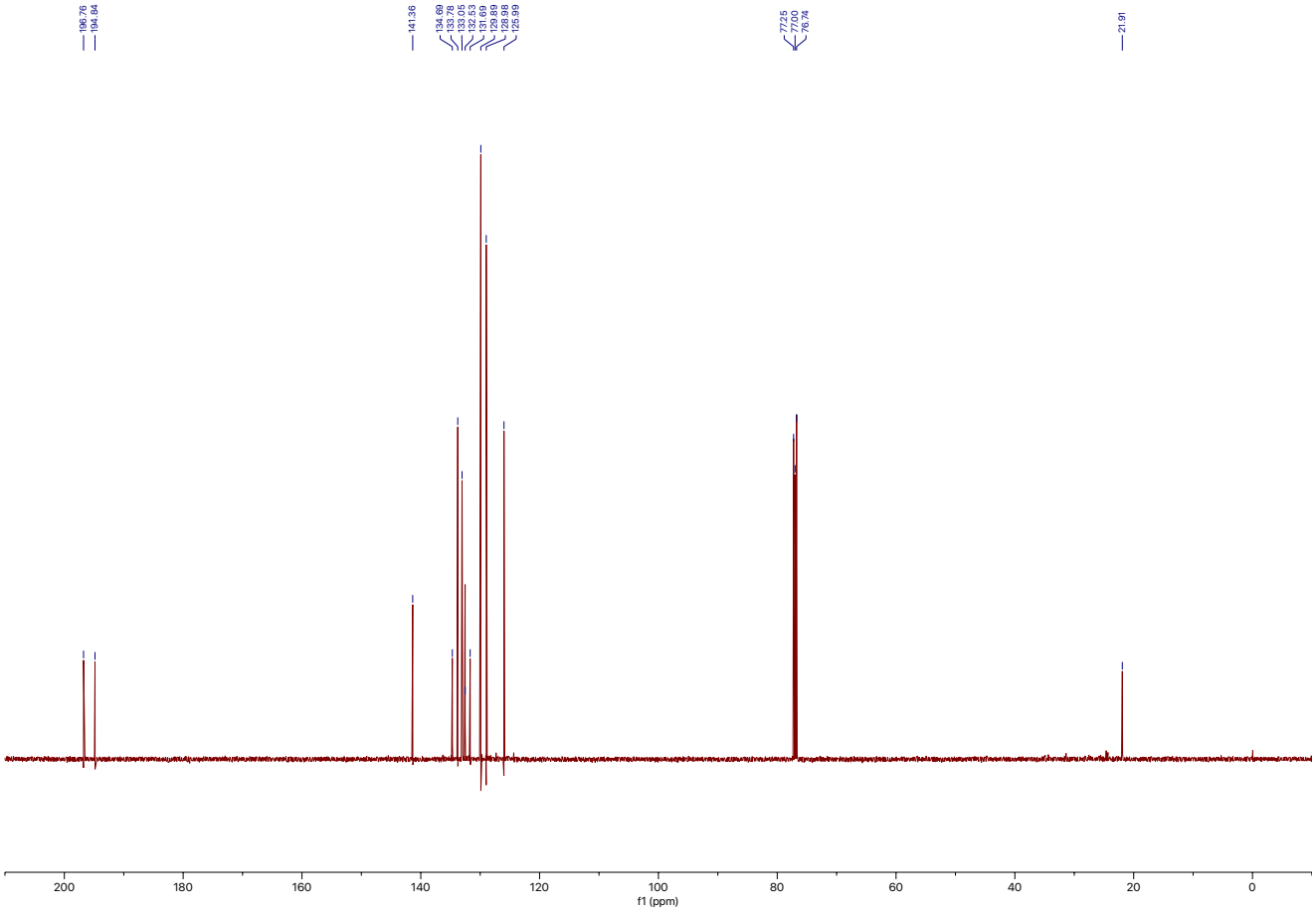
5
4
3
2
1
0
f1 (ppm)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133.05
132.53
131.69
129.69
128.98
125.99
77.25
77.00
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21.91
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160
140
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60
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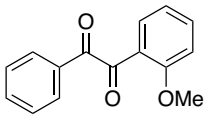
20
0
f1 (ppm)

3ao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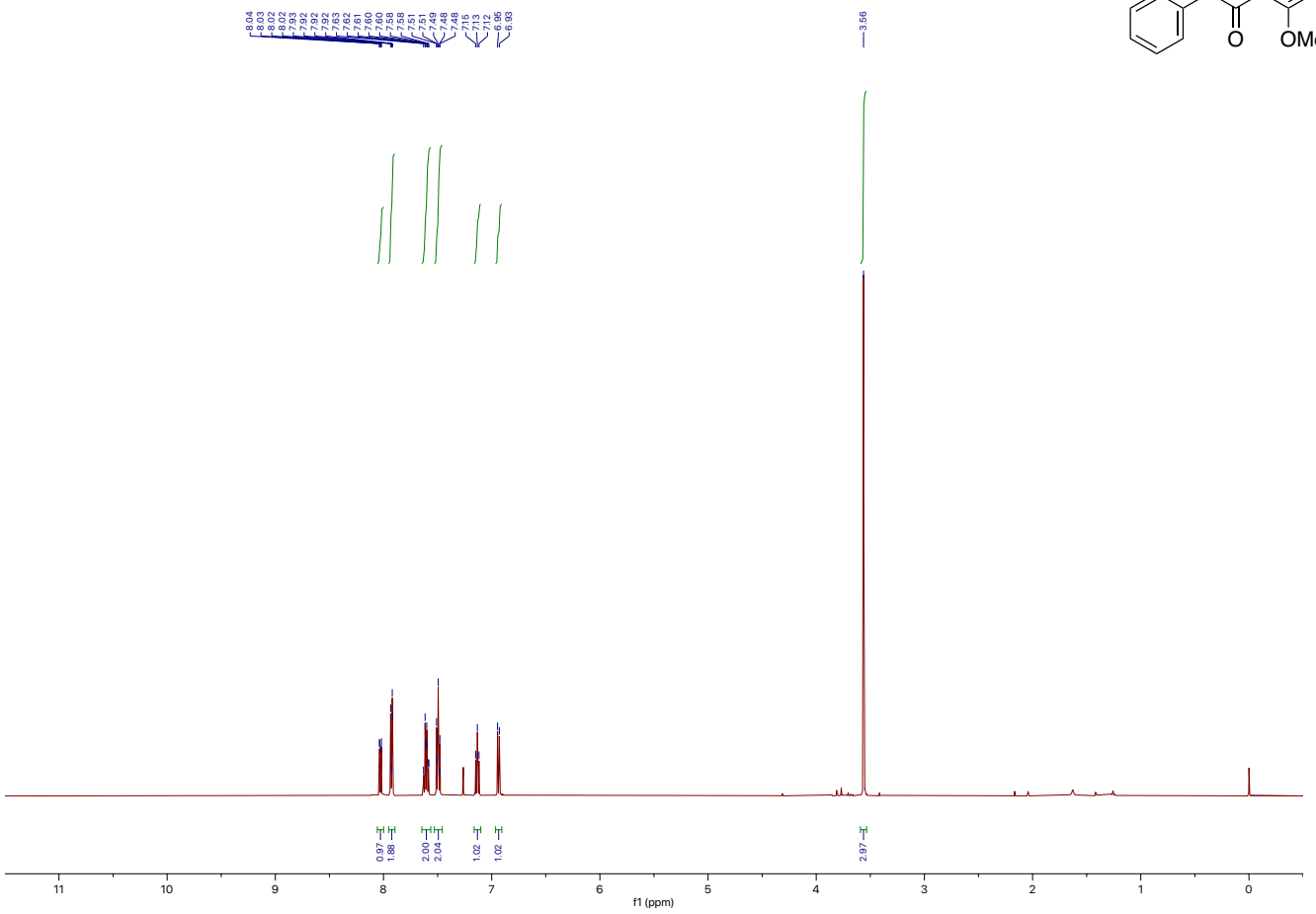
O
OMe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8.03
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8.02
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7.92
7.92
7.92
7.63
7.62
7.61
7.60
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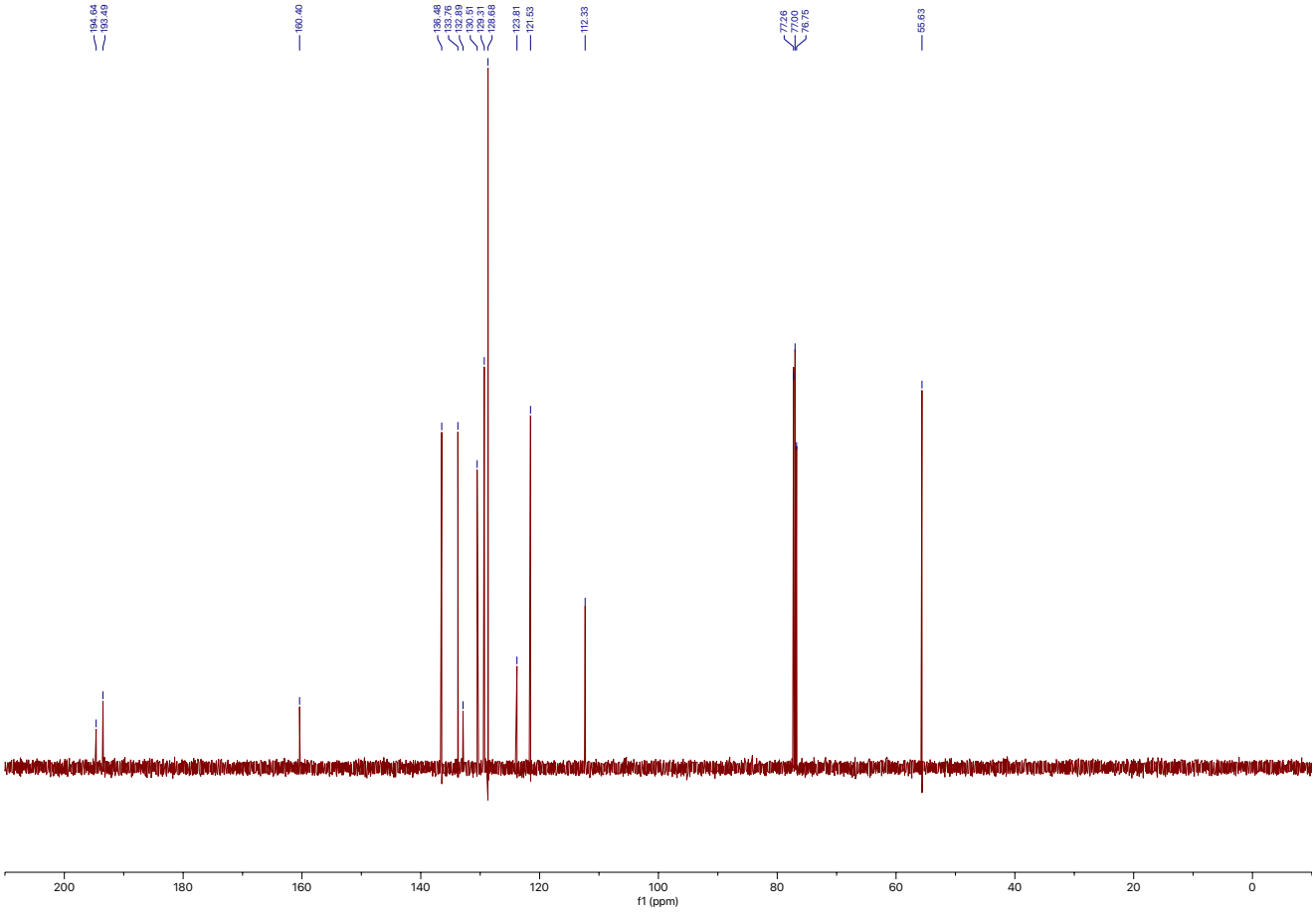
7.59
7.58
7.51
7.51
7.49
7.48
7.15
7.13
7.12
6.95
6.93
3.56
0.97
1.88
2.00
2.04
1.02
1.02
2.97
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)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193.49
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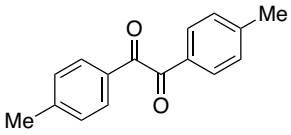
136.49
133.76
132.89
130.51
129.31
128.68
123.81
121.53
112.33
77.26
77.00
76.75
55.63
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

3ba


Me
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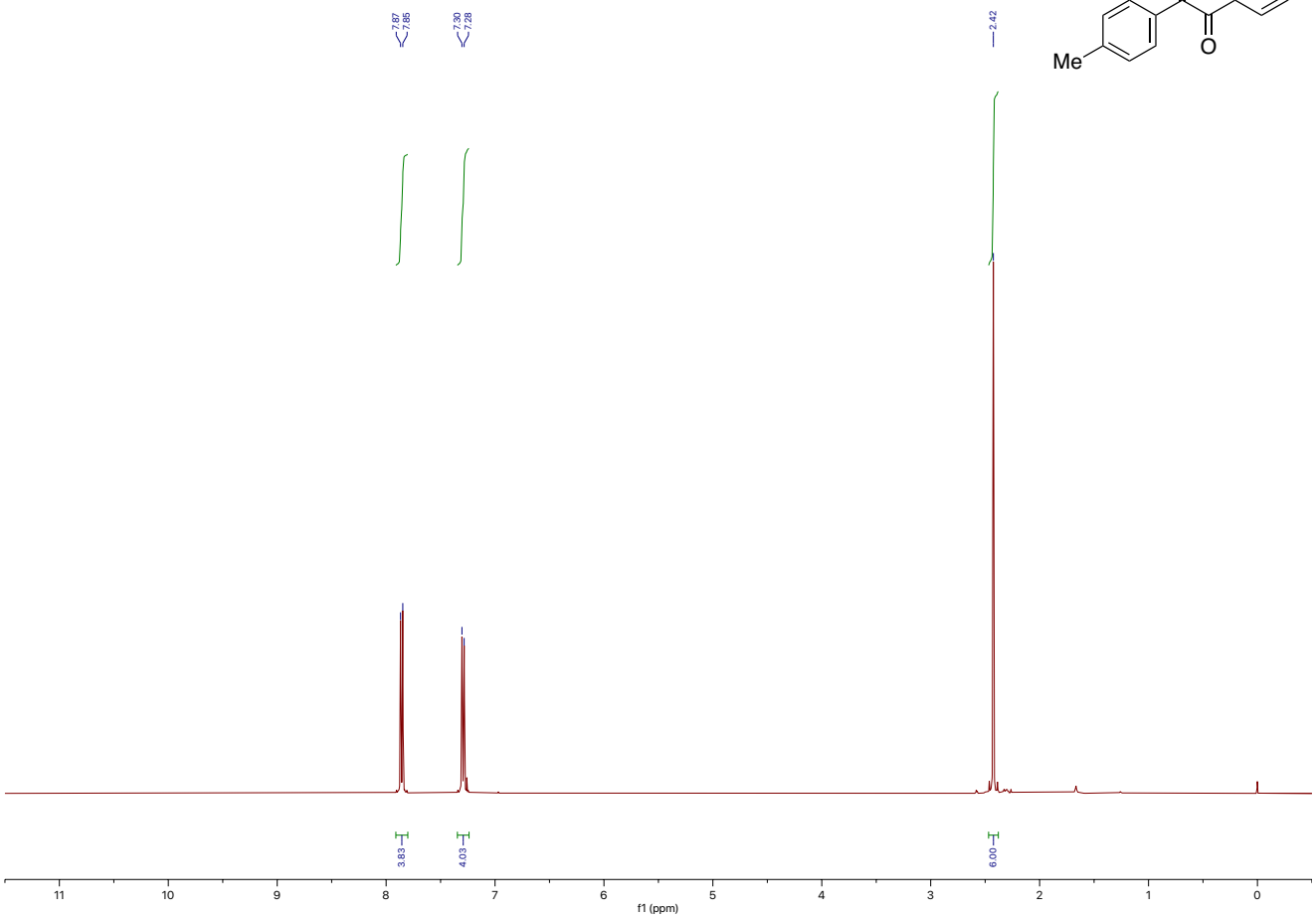
O
Me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7.28
2.42
3.83
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6.00
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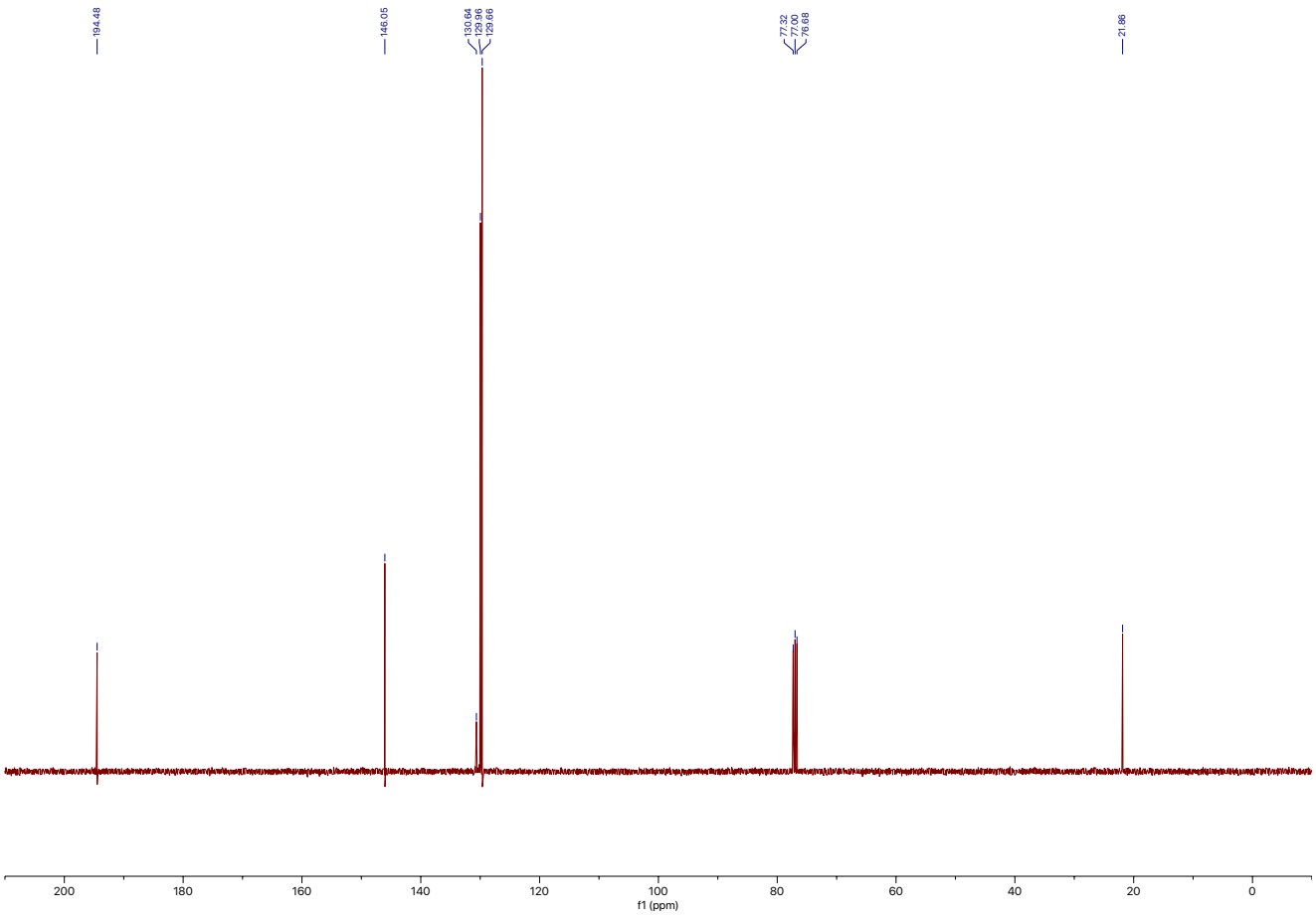
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)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146.05
130.64
129.66
129.65
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77.00
76.68
21.86
200
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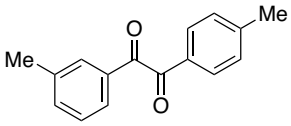
160
140
120
100
80
60
40
20
0
f1 (ppm)

3ca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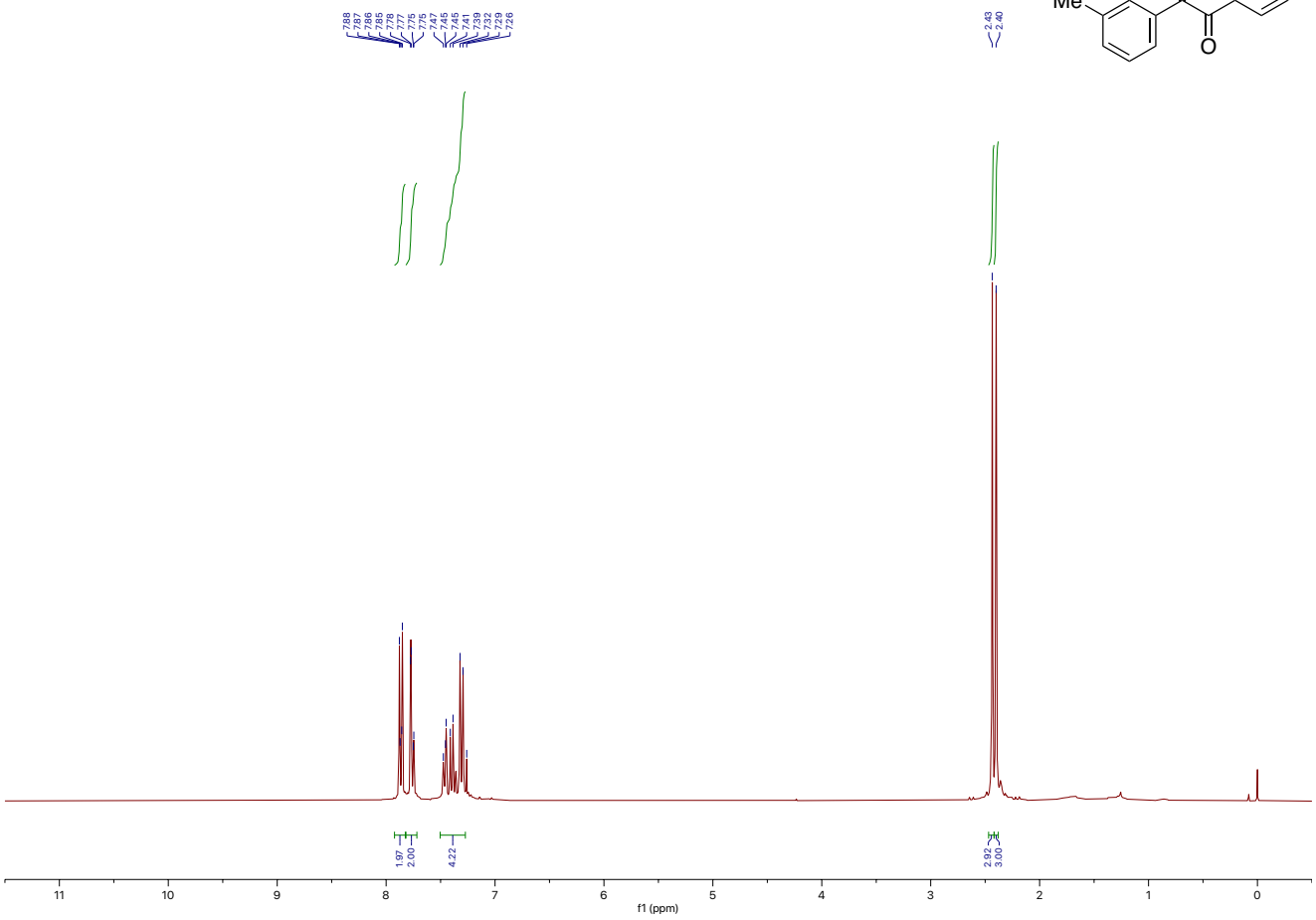
O
Me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7.75
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7.45
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7.26
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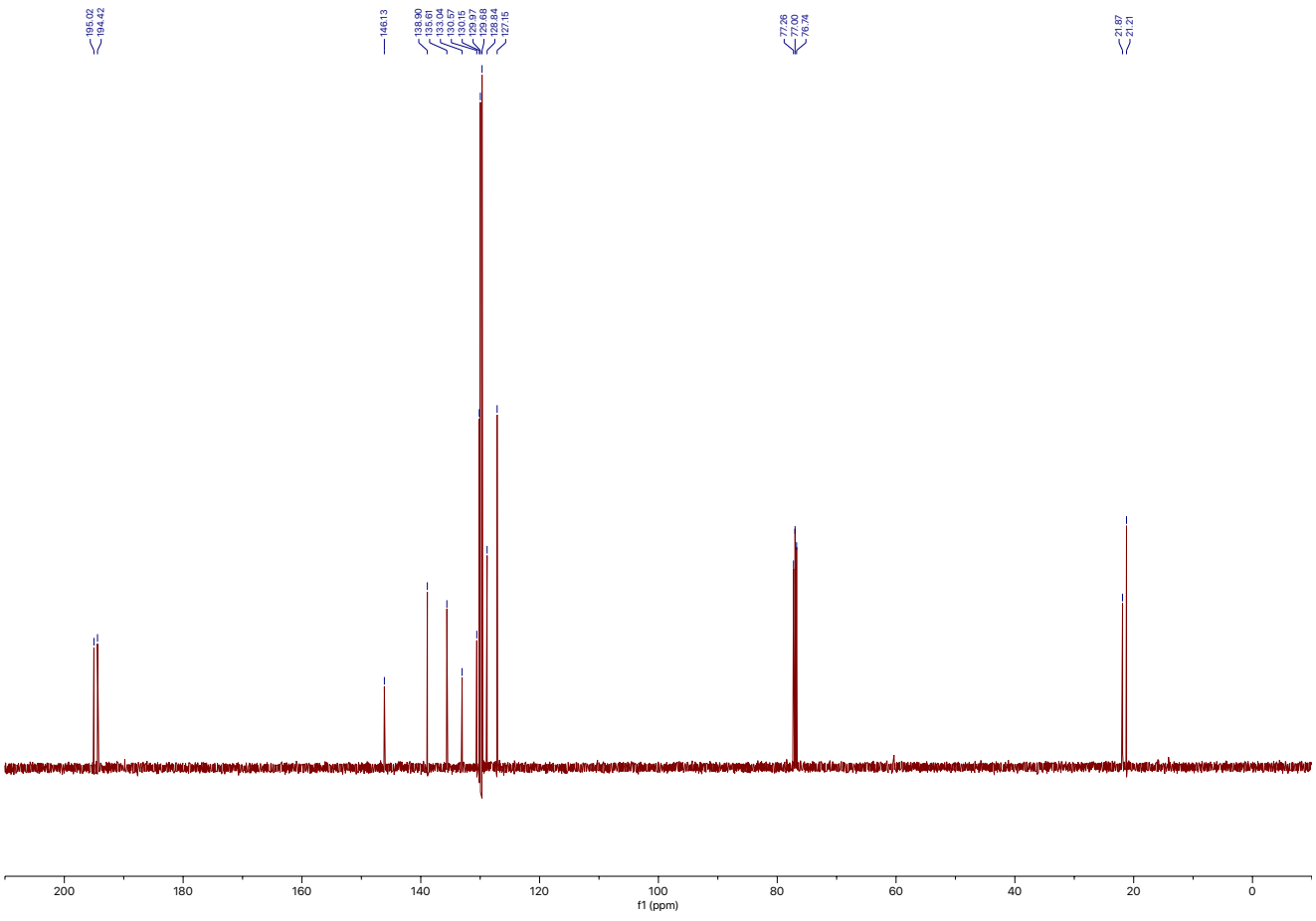
2.40
1.97
2.00
4.22
2.92
3.00
f1 (ppm)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194.42
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138.90
135.61
133.04
130.57
130.15
129.97
129.68
129.64
127.15
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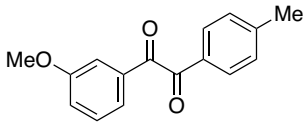
76.74
21.87
21.21
f1 (ppm)

3da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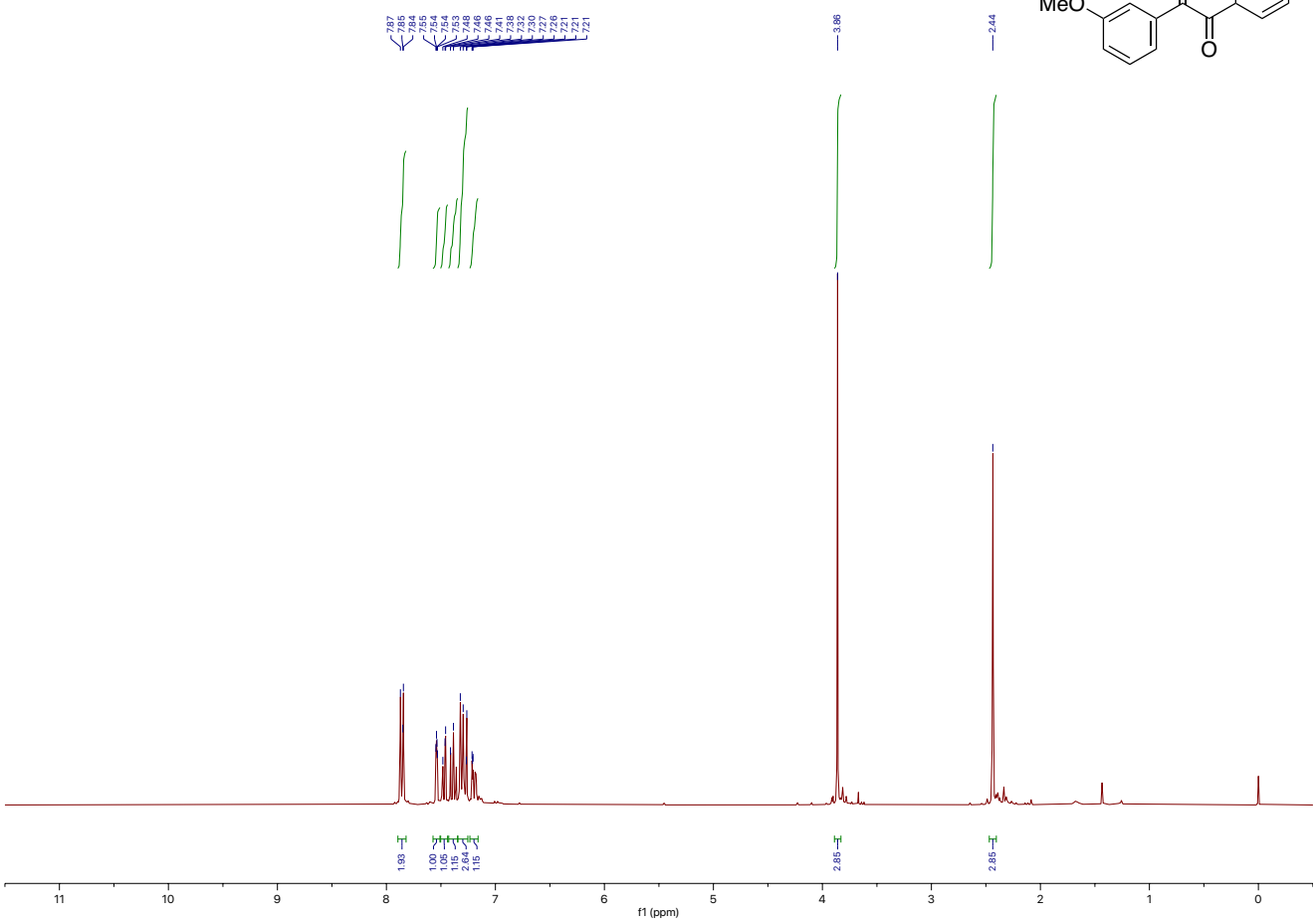
O
O
Me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7.48
7.46
7.41
7.38
7.32
7.30
7.27
7.26
7.21
7.21
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1.93
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1.05
1.16
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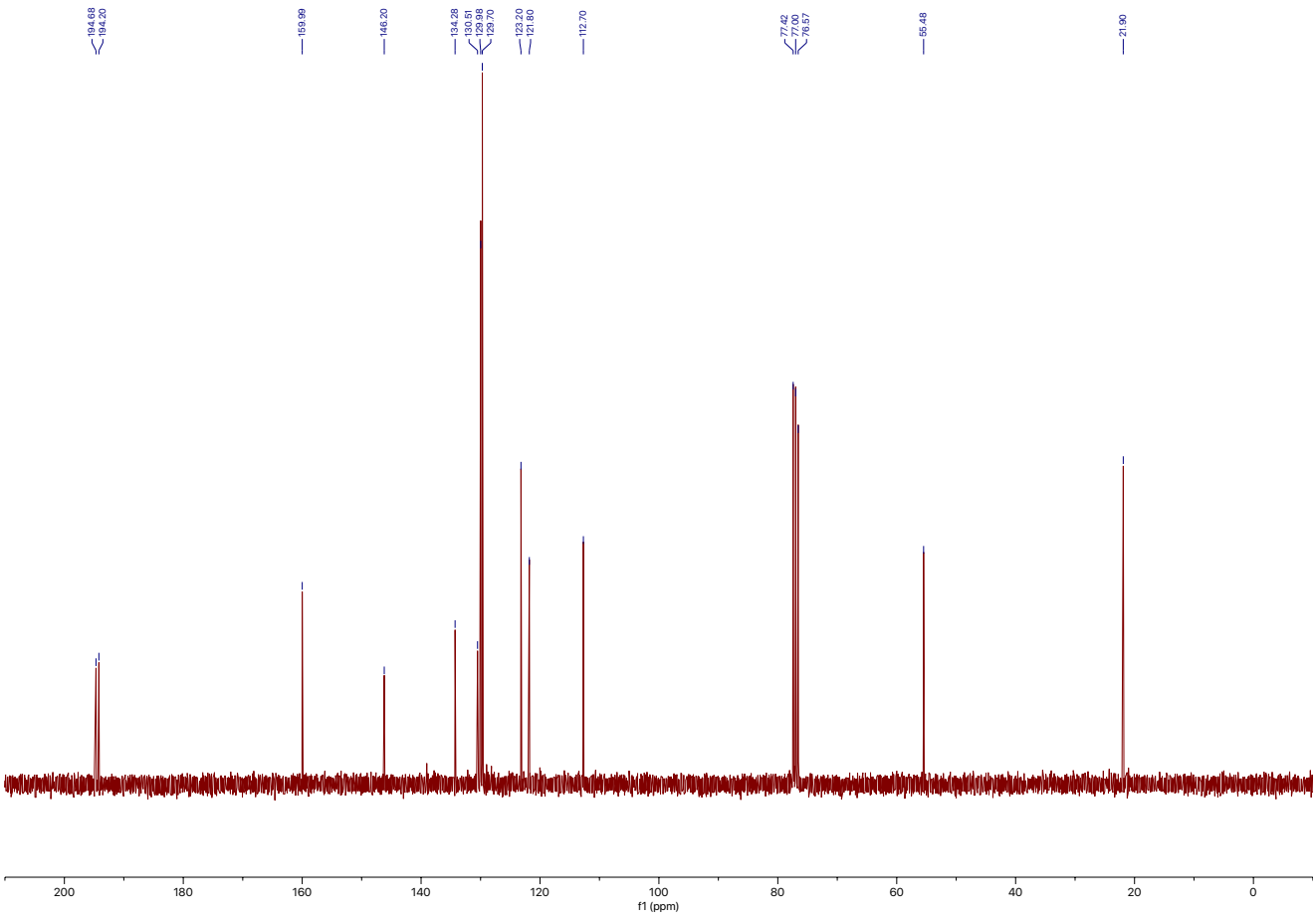
1.15
2.85
2.85
f1 (ppm)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77.42
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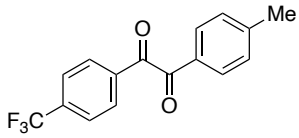
76.57
55.48
21.90
f1 (ppm)

3ea


Me
O
O
F3C

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7.89
7.86
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7.32
7.26
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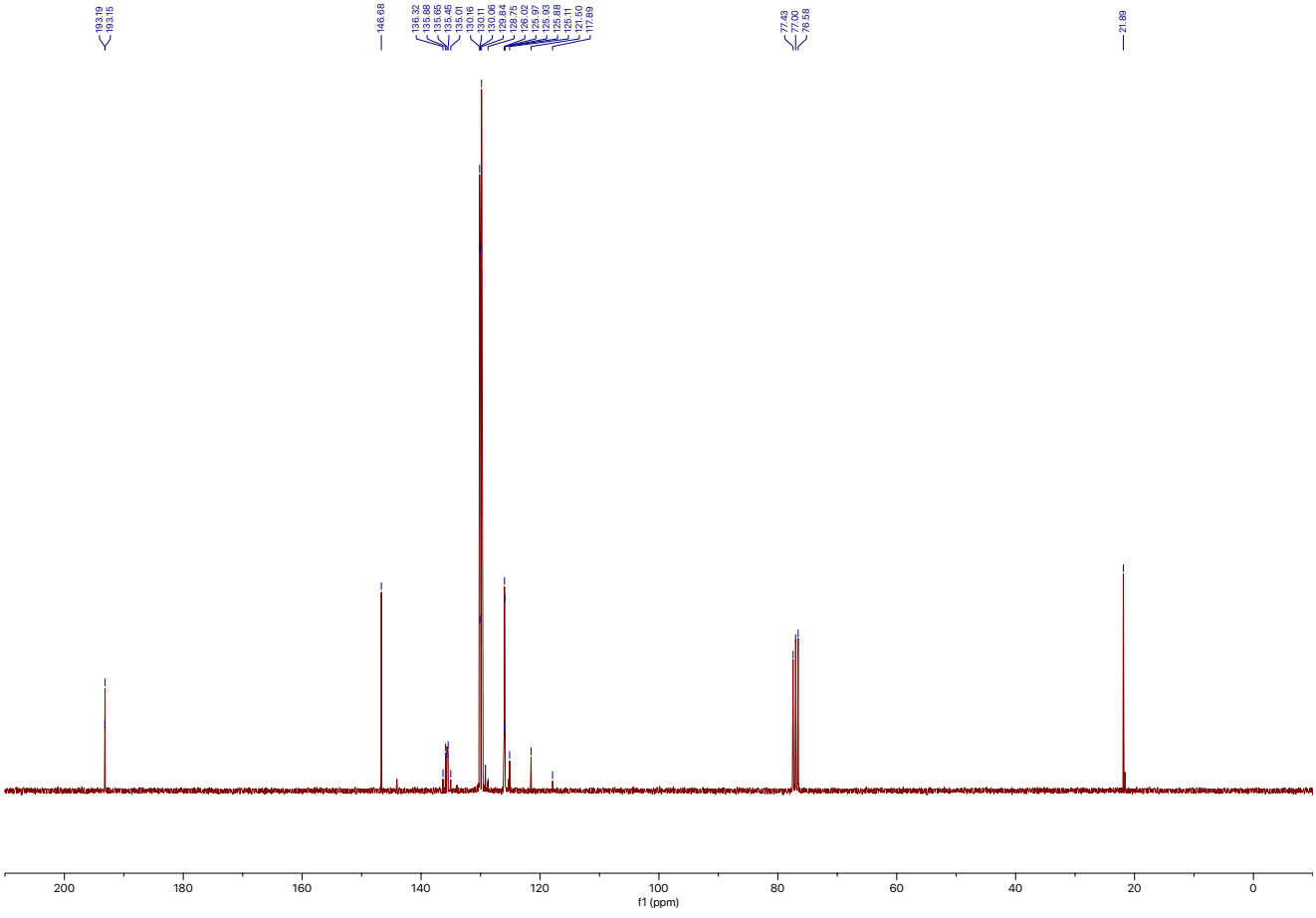
1.94
2.04
2.01
2.00
2.92
f1 (ppm)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136.32
135.88
135.65
135.45
135.01
130.16
130.11
130.06
129.84
128.75
126.02
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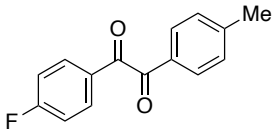
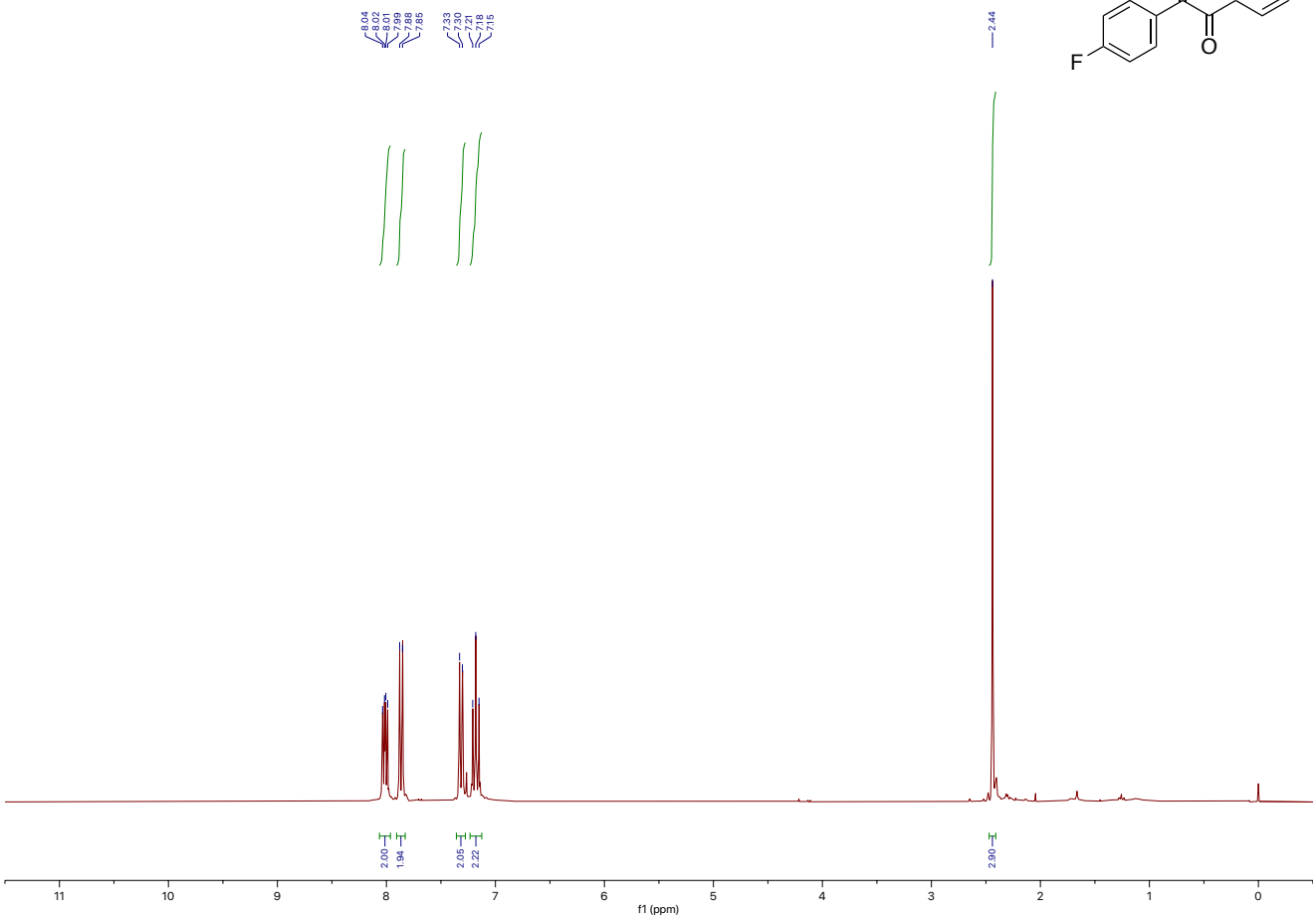
125.88
125.11
121.80
117.89
77.43
77.00
76.58
21.89
f1 (ppm)

3fa


O
O
F
Me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8.02
8.01
7.89
7.88
7.85
7.33
7.30
7.21
7.19
7.15
2.44
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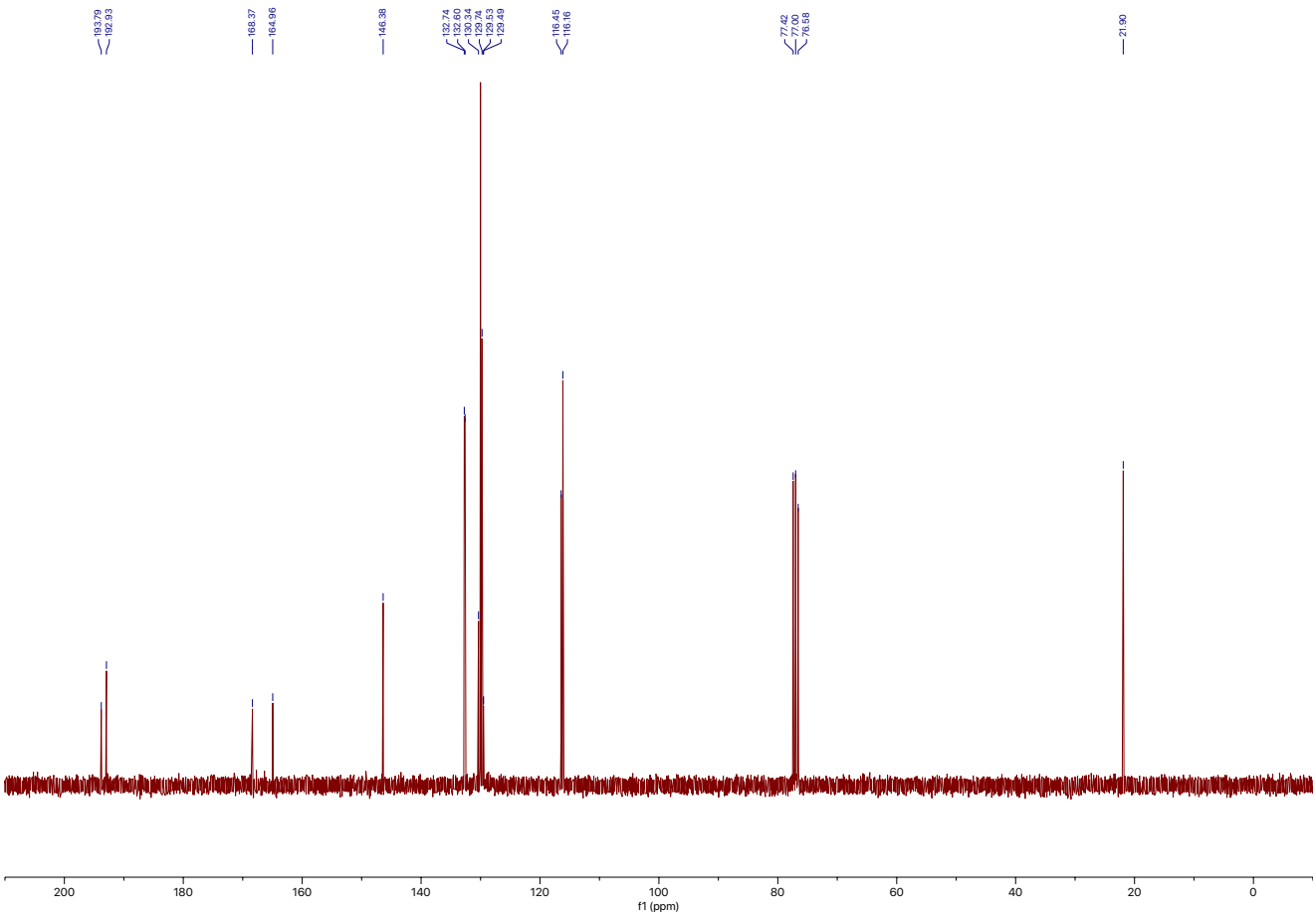
1.94
2.05
2.22
2.90
f1 (ppm)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168.37
164.96
146.38
132.74
132.60
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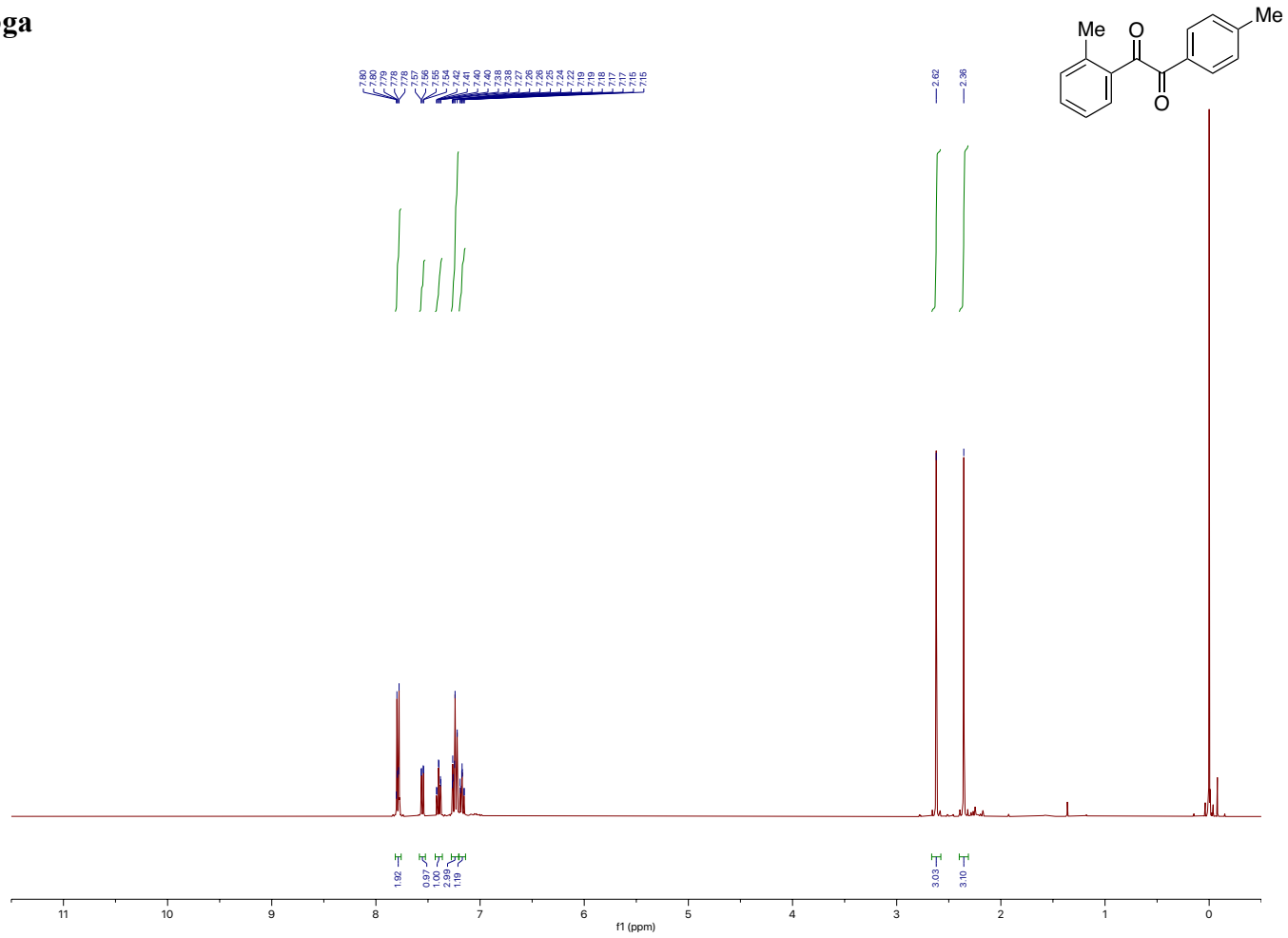
130.24
129.53
129.49
116.45
116.16
77.42
77.00
76.58
21.90
f1 (ppm)

3ga


Me
O
O
Me
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7.80
7.79
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7.78
7.57
7.56
7.55
7.54
7.42
7.41
7.40
7.40
7.38
7.38
7.27
7.26
7.25
7.24
7.22
7.19
7.19
7.18
7.17
7.17
7.15
7.15
2.62
2.36
1.92
0.97
1.00
2.99
1.19
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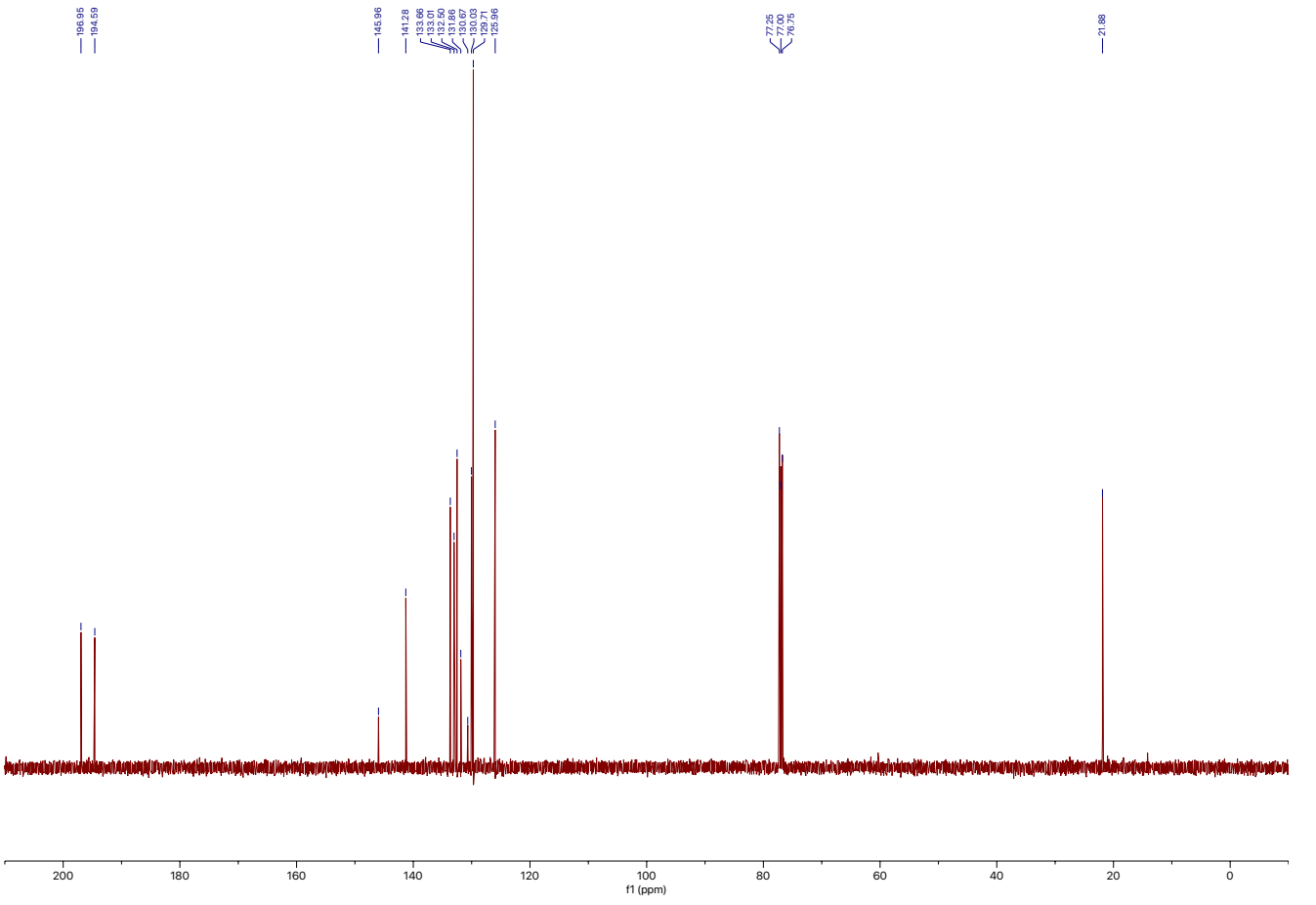
3.10
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)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133.01
132.50
131.86
130.67
130.03
129.71
125.96
77.25
77.00
76.75
21.88
200
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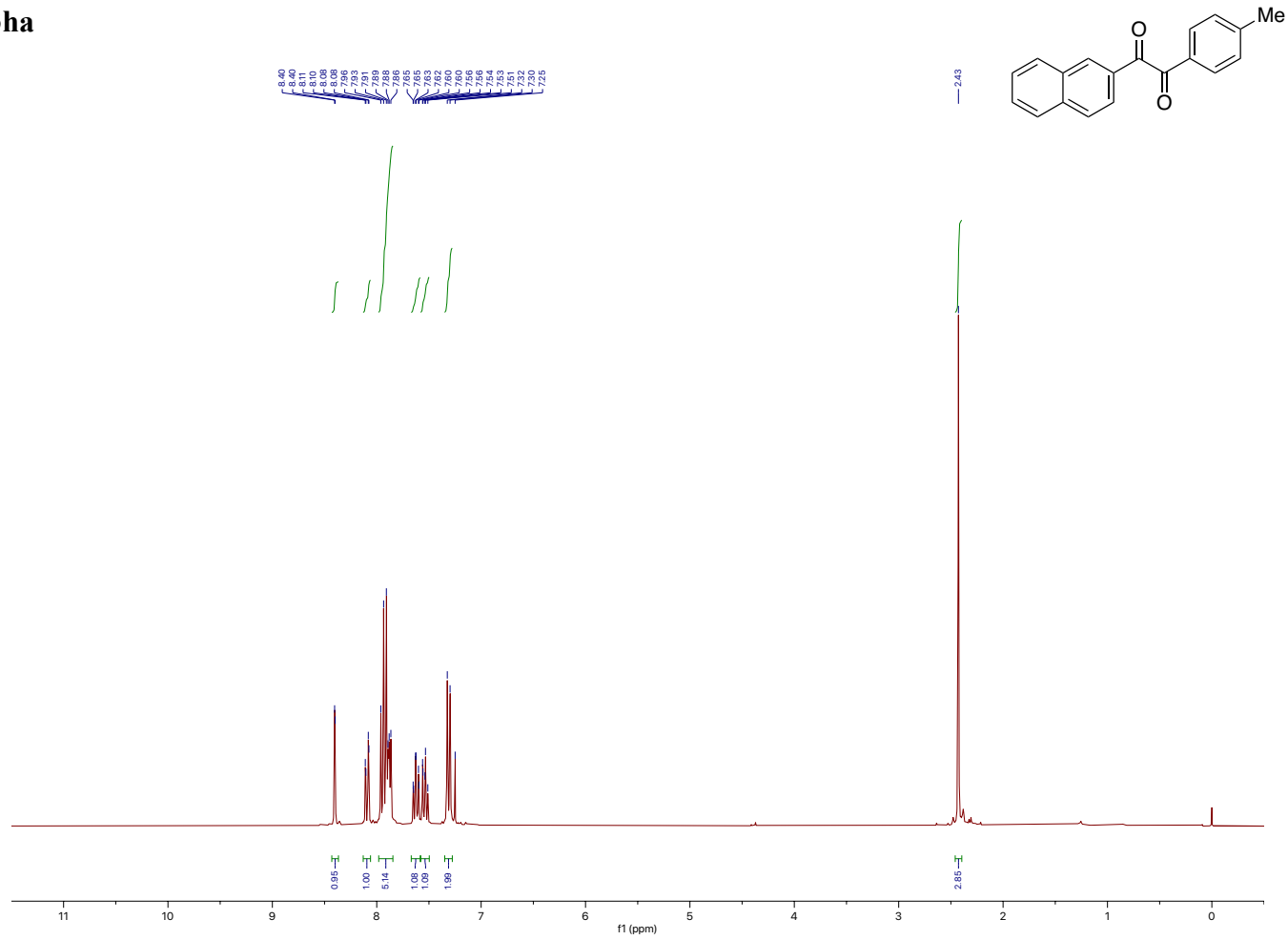
160
140
120
100
80
60
40
20
0
f1 (ppm)

3ha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7.89
7.88
7.86
7.65
7.65
7.63
7.62
7.62
7.60
7.60
7.56
7.56
7.56
7.54
7.53
7.51
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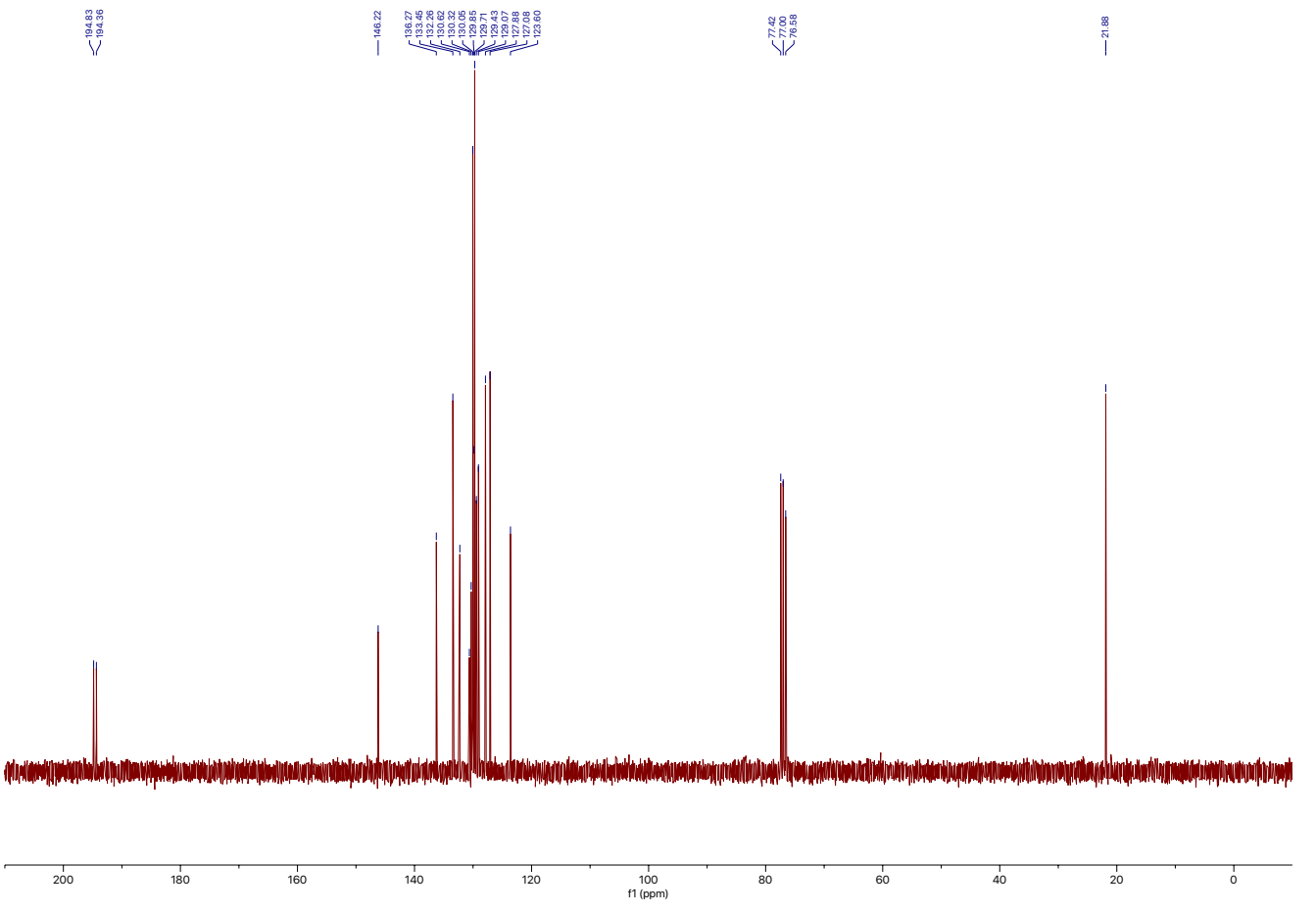
5.14
1.08
1.09
1.99
2.85
f1 (ppm)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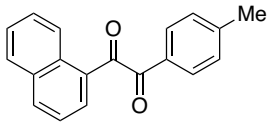
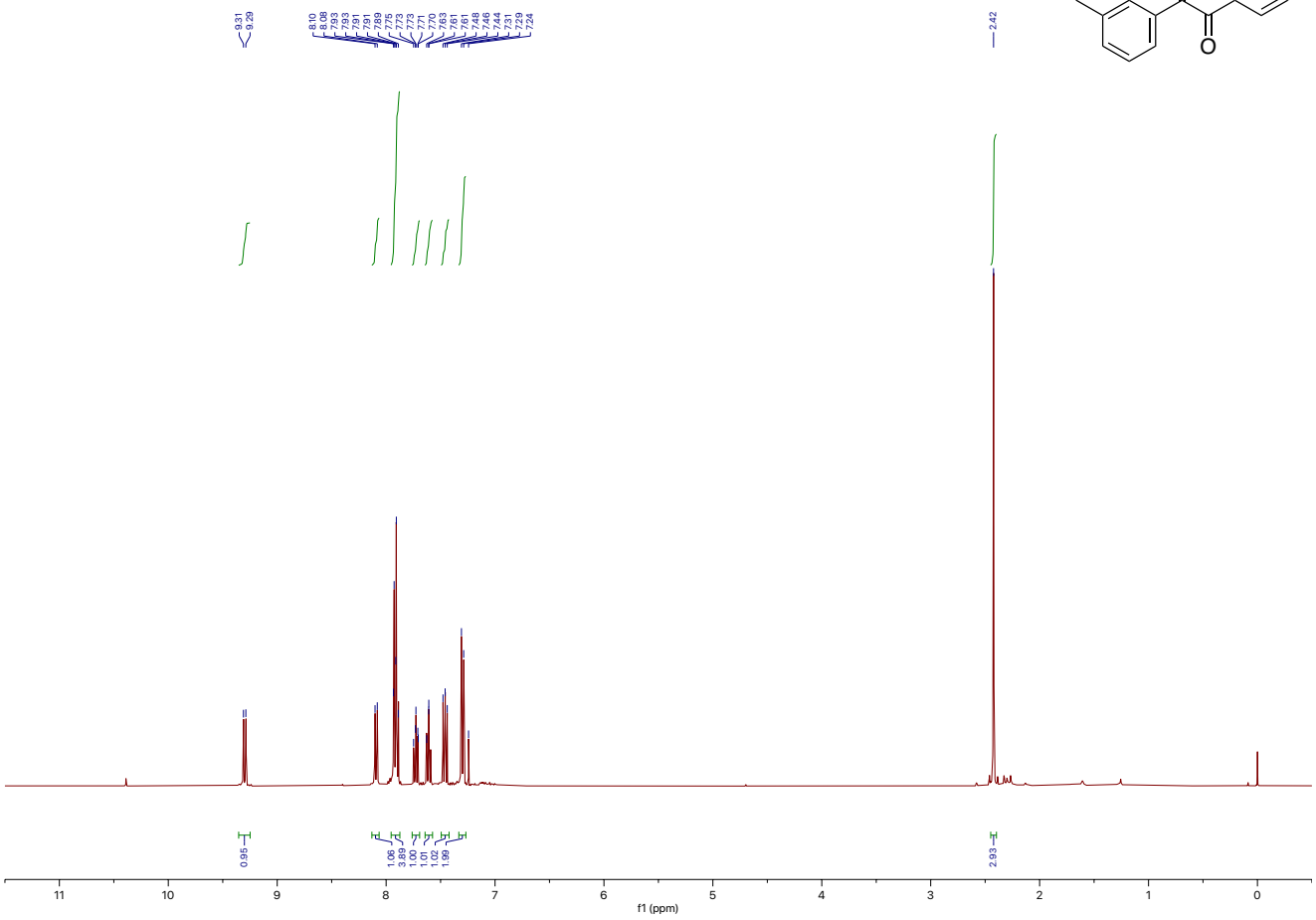
77.00
76.58
21.88
f1 (ppm)

3ia


Me
O
O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8.08
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7.91
7.91
7.89
7.75
7.73
7.73
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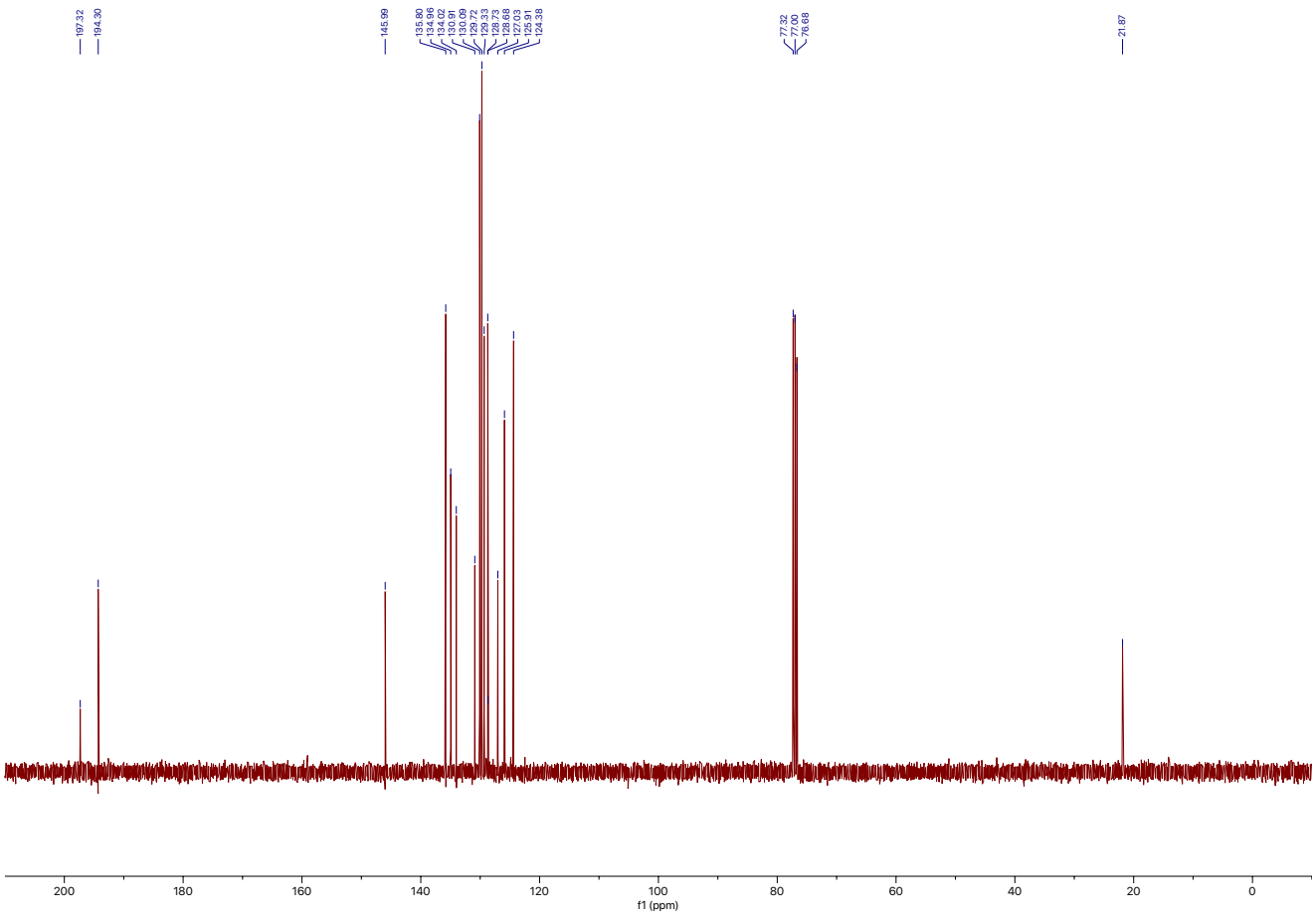
7.70
7.63
7.61
7.61
7.48
7.46
7.44
7.31
7.29
7.24
2.42
0.95
1.06
3.89
1.00
1.02
1.02
1.99
2.93
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)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134.02
130.91
130.09
129.72
129.33
128.73
128.68
127.03
125.91
124.38
77.32
77.00
76.68
21.67
200
180
160
140
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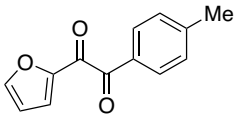
100
80
60
40
20
0
f1 (ppm)

3ja


Me
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O
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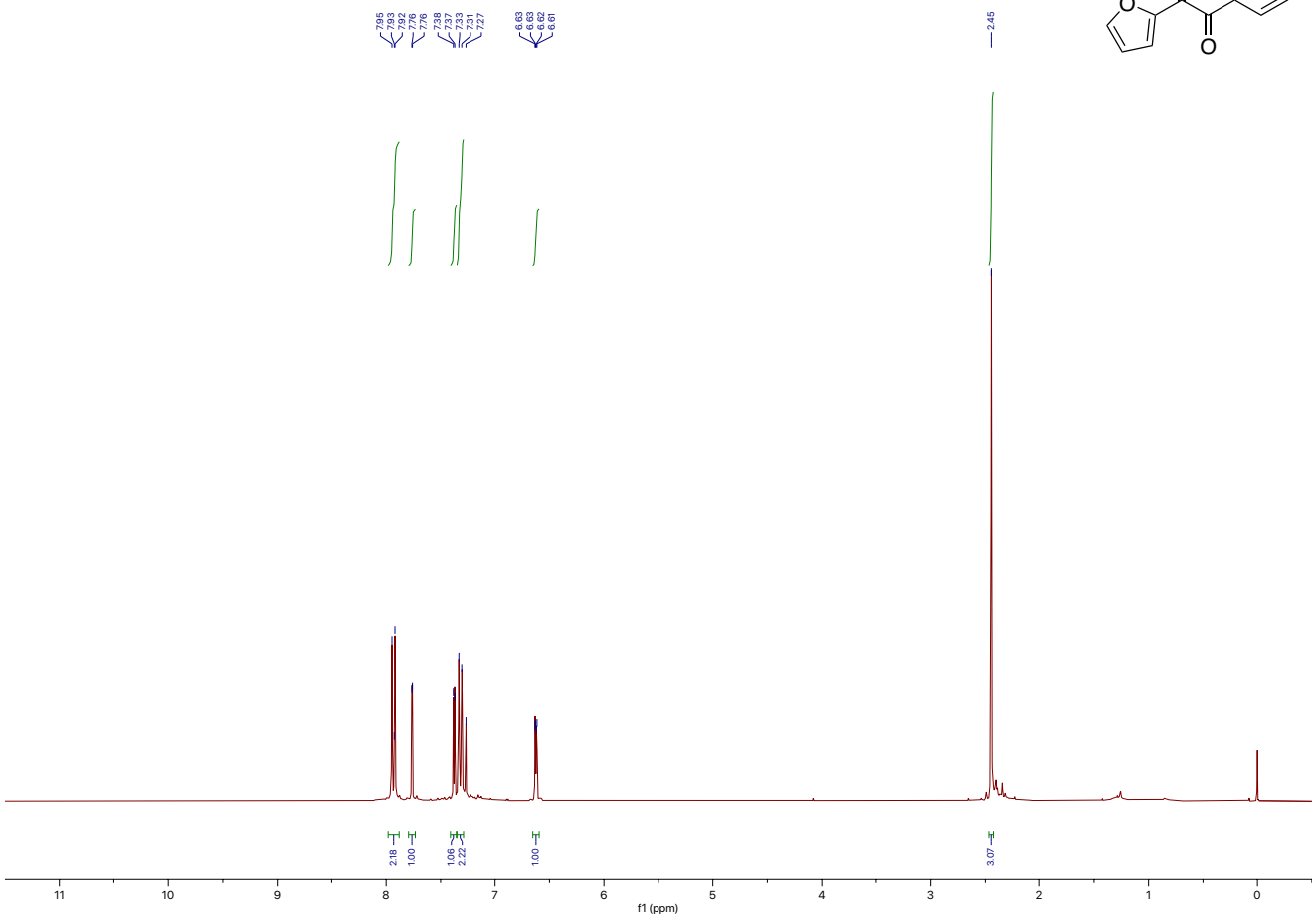
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7.93
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7.76
7.38
7.37
7.33
7.31
7.27
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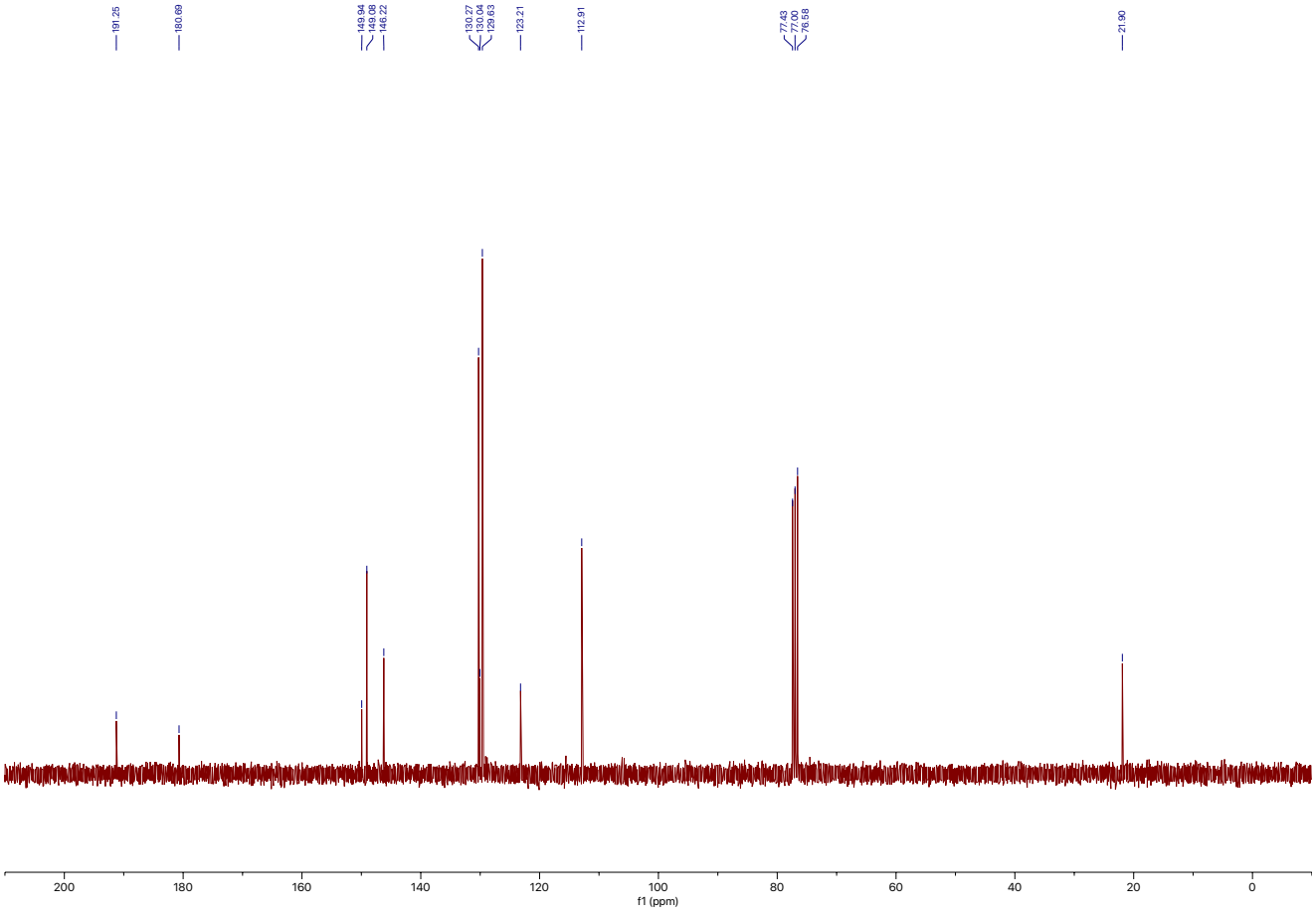
6.63
6.62
6.61
2.45
2.18
1.00
1.06
2.22
1.00
3.07
f1 (ppm)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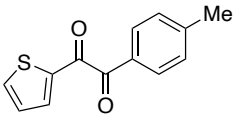
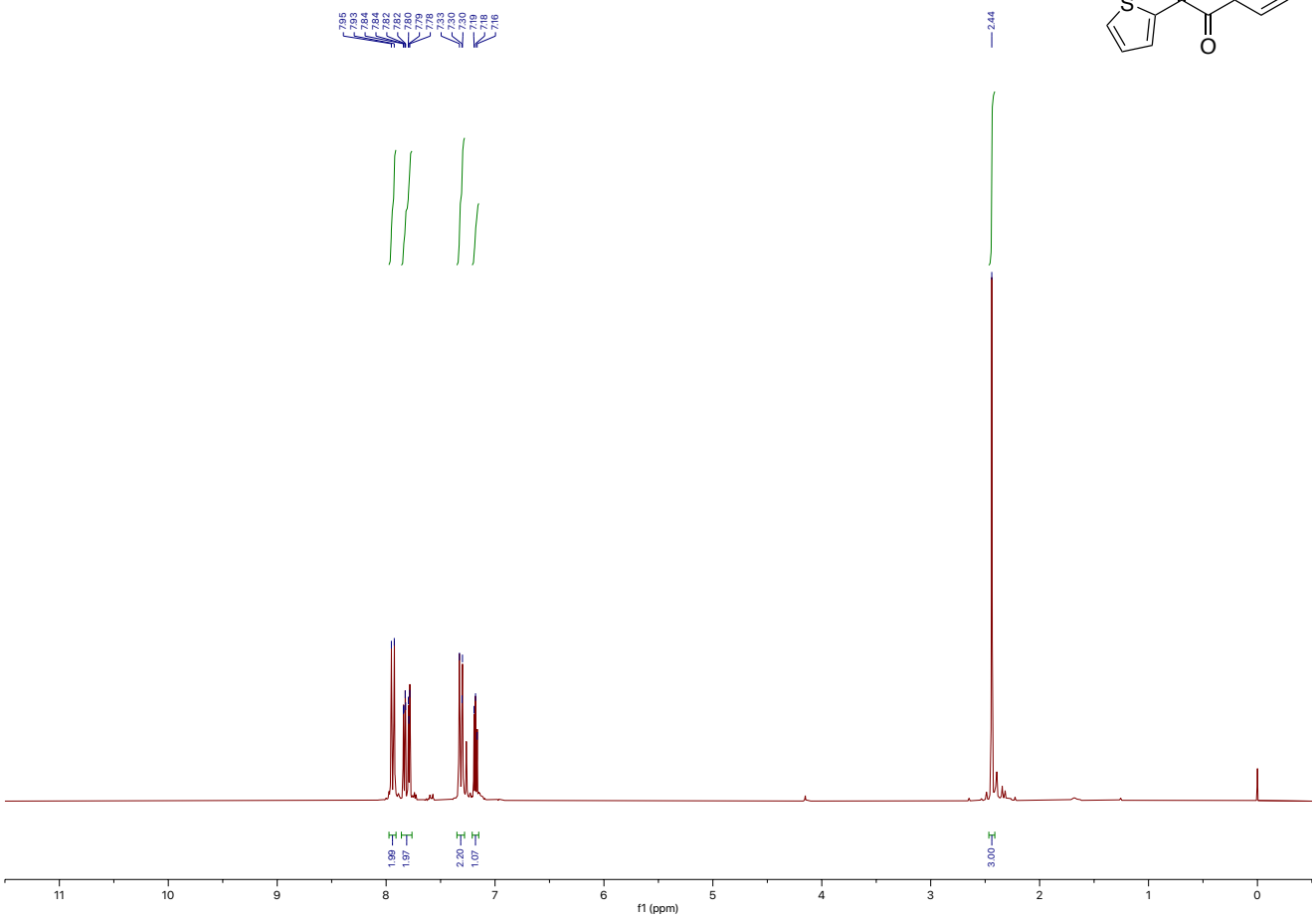
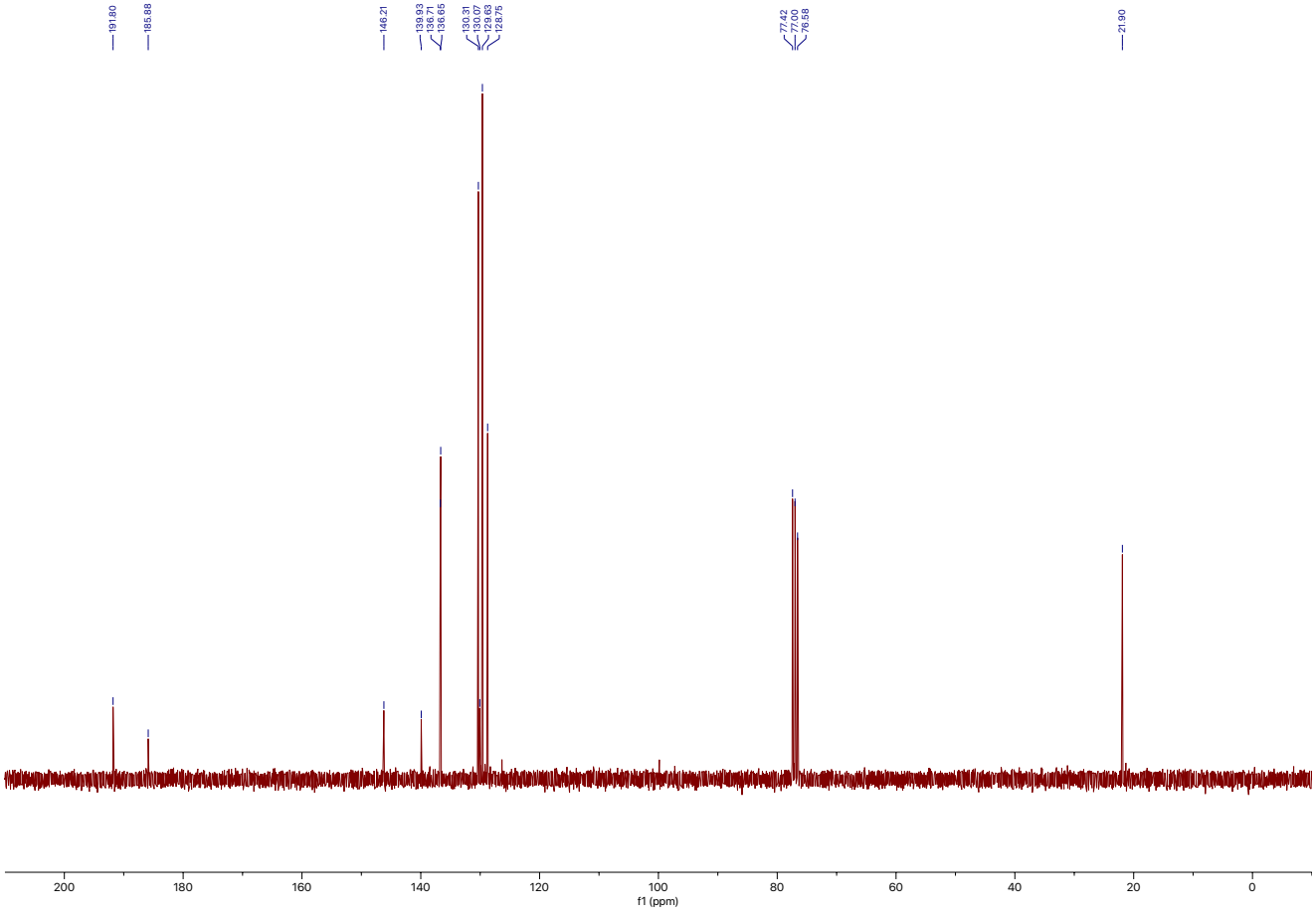
129.63
123.21
112.91
77.43
77.00
76.58
21.90
f1 (ppm)

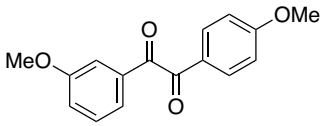
3ka

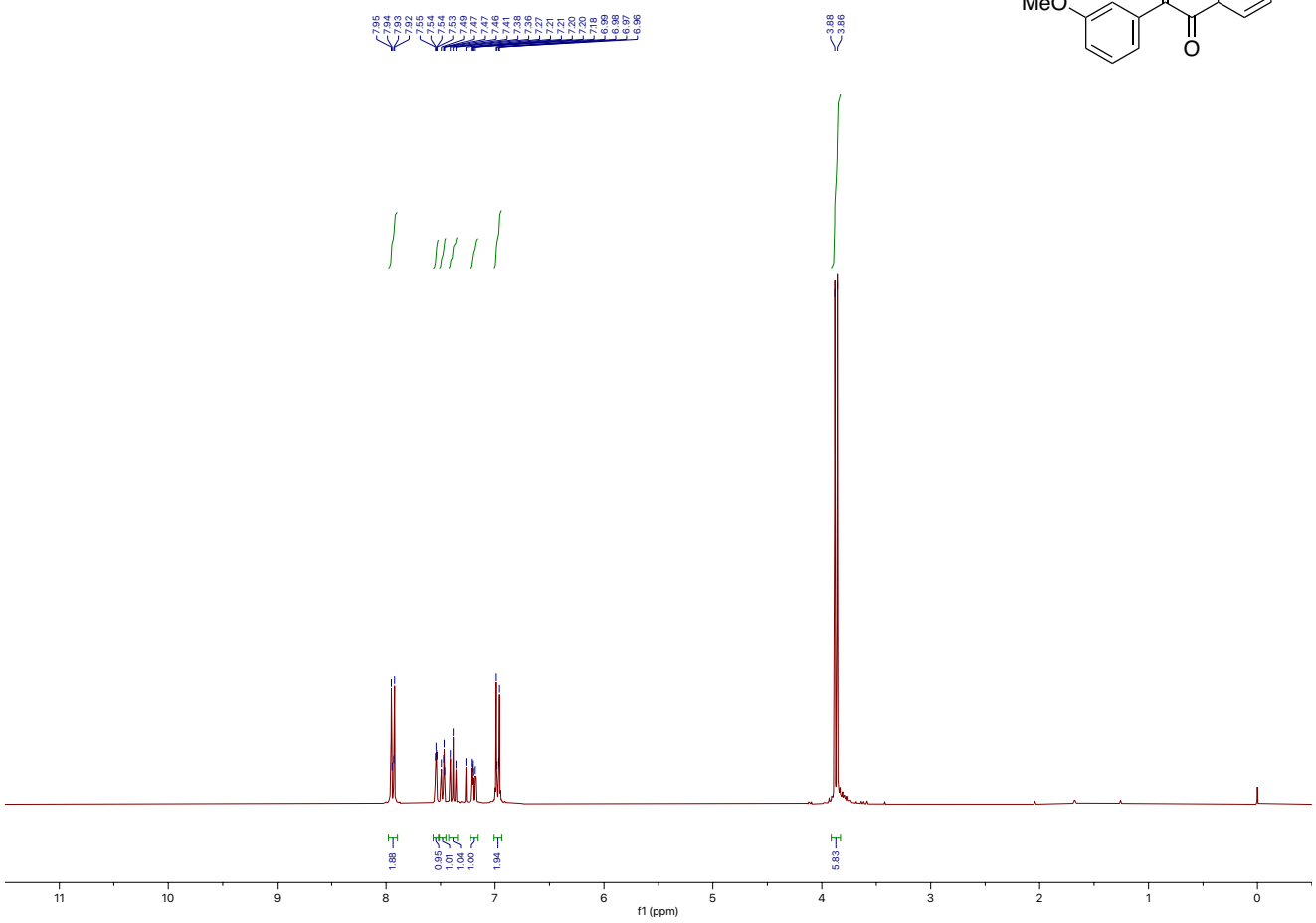

O
S
O
Me

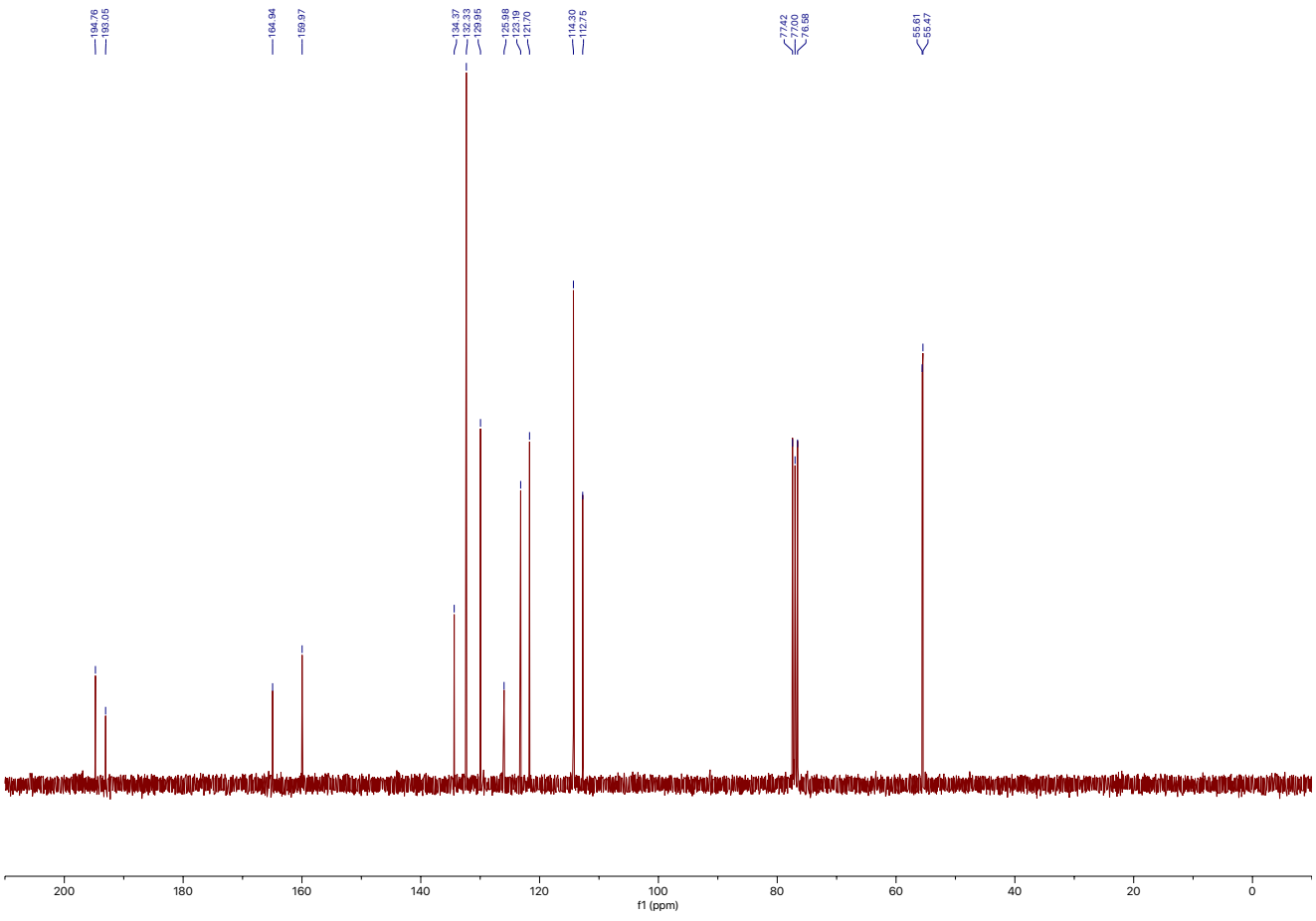

7.95
7.93
7.84
7.84
7.82
7.82
7.80
7.79
7.78
7.33
7.30
7.30
7.19
7.18
7.16
2.44
1.99
1.97
2.20
1.07
3.00
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)


191.80
185.88
146.21
139.93
136.71
136.65
130.31
130.07
129.63
128.75
77.42
77.00
76.58
21.90
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

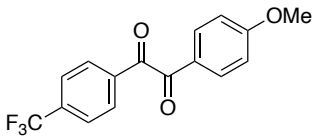
3de

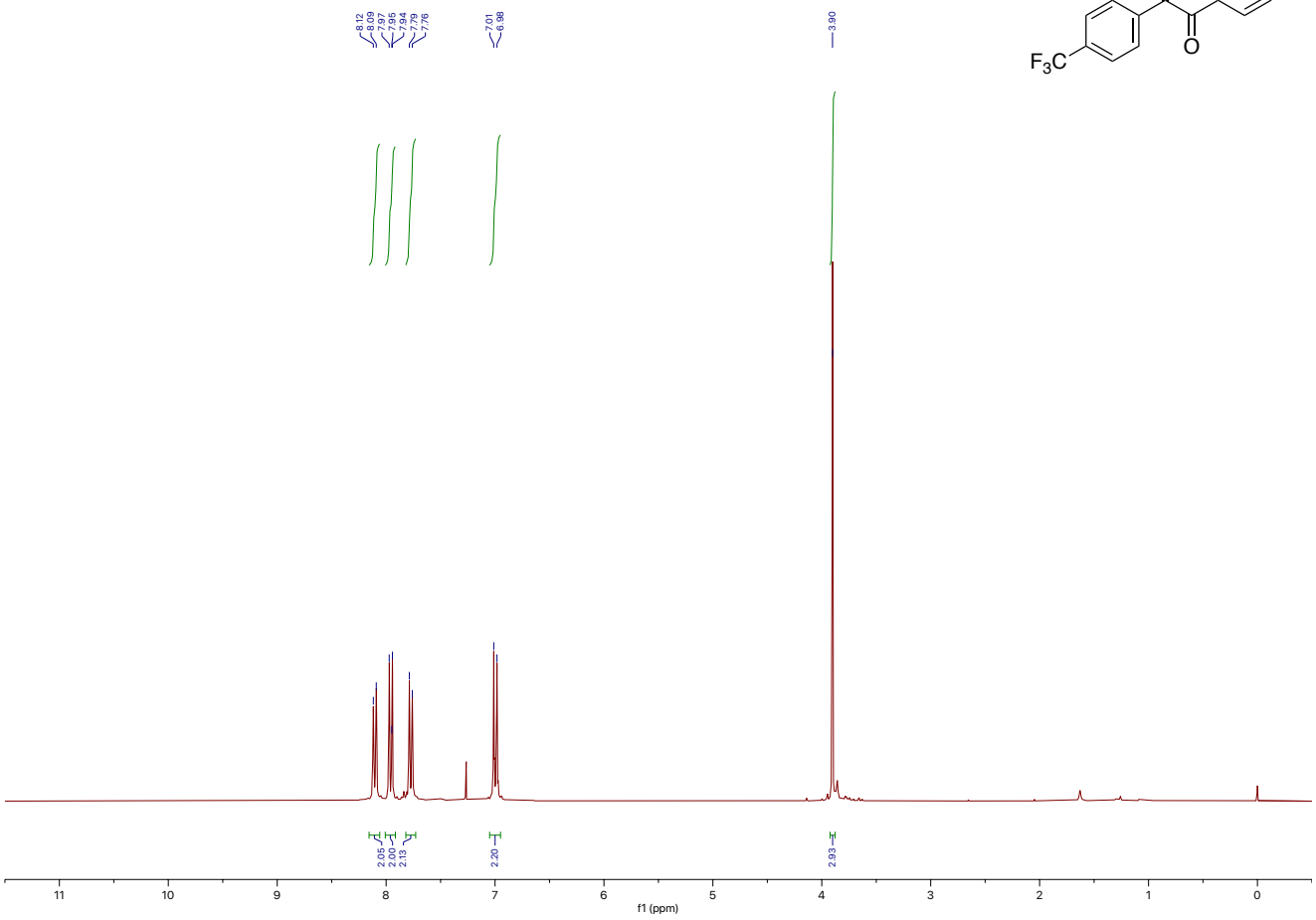

MeO
OMe
O
O

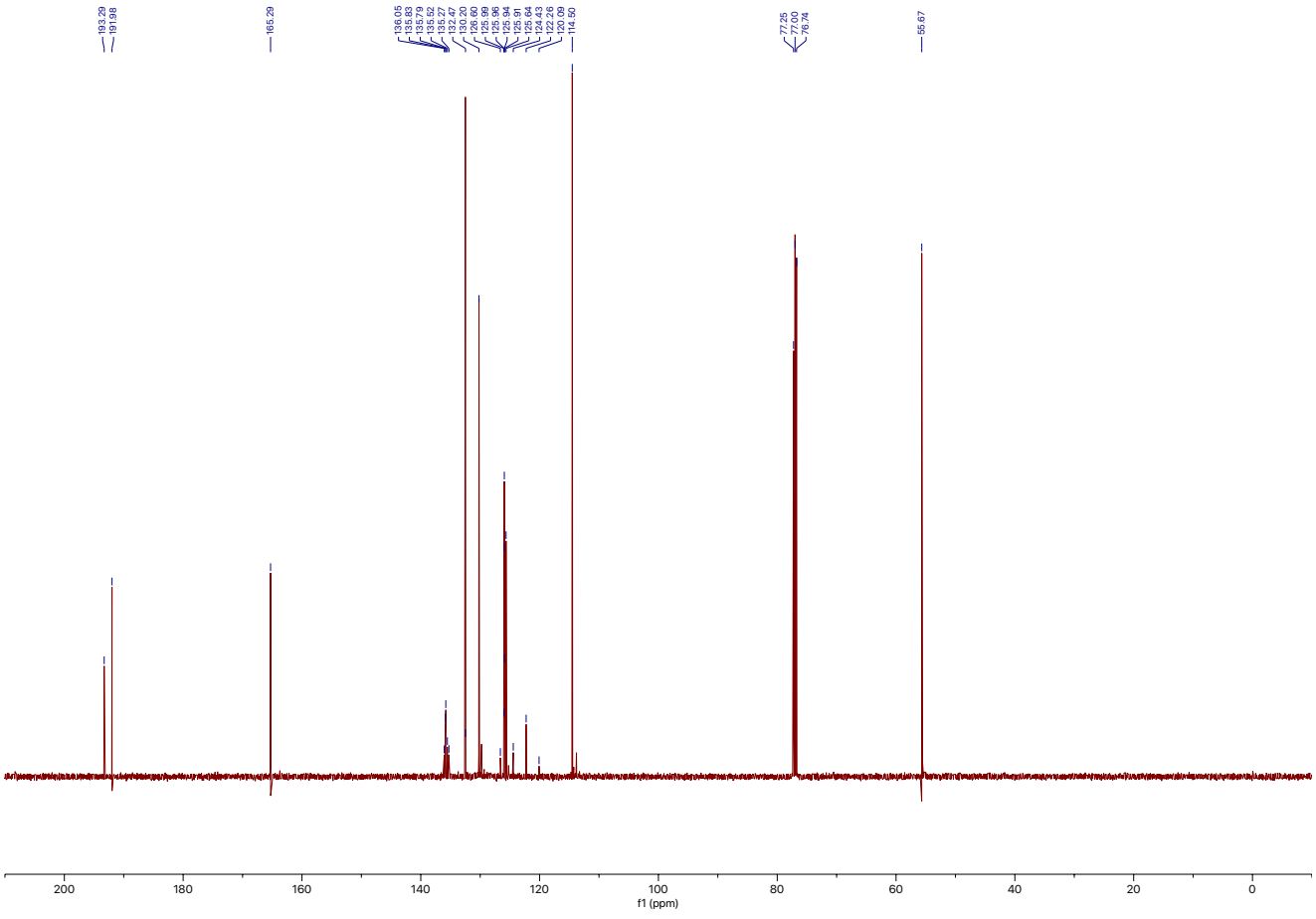

7.95
7.94
7.93
7.92
7.55
7.54
7.54
7.53
7.49
7.47
7.46
7.41
7.38
7.36
7.27
7.21
7.21
7.20
7.20
7.18
6.98
6.98
6.97
6.96
3.88
3.86
1.88
0.95
1.01
1.04
1.00
1.94
5.83
f1 (ppm)


194.76
193.05
164.94
159.97
134.37
132.33
129.95
125.98
123.19
121.70
114.30
112.75
77.42
77.00
76.58
55.61
55.47
f1 (ppm)

3ee


OMe
O
O
F3C


8.12
8.09
7.97
7.95
7.94
7.79
7.76
7.01
6.98
3.90
2.05
2.00
2.13
2.20
2.93
f1 (ppm)


193.29
191.98
165.29
136.05
135.83
135.79
135.52
135.27
133.47
132.20
126.60
125.99
125.96
125.94
125.91
125.64
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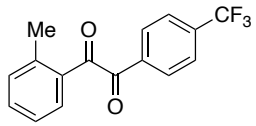
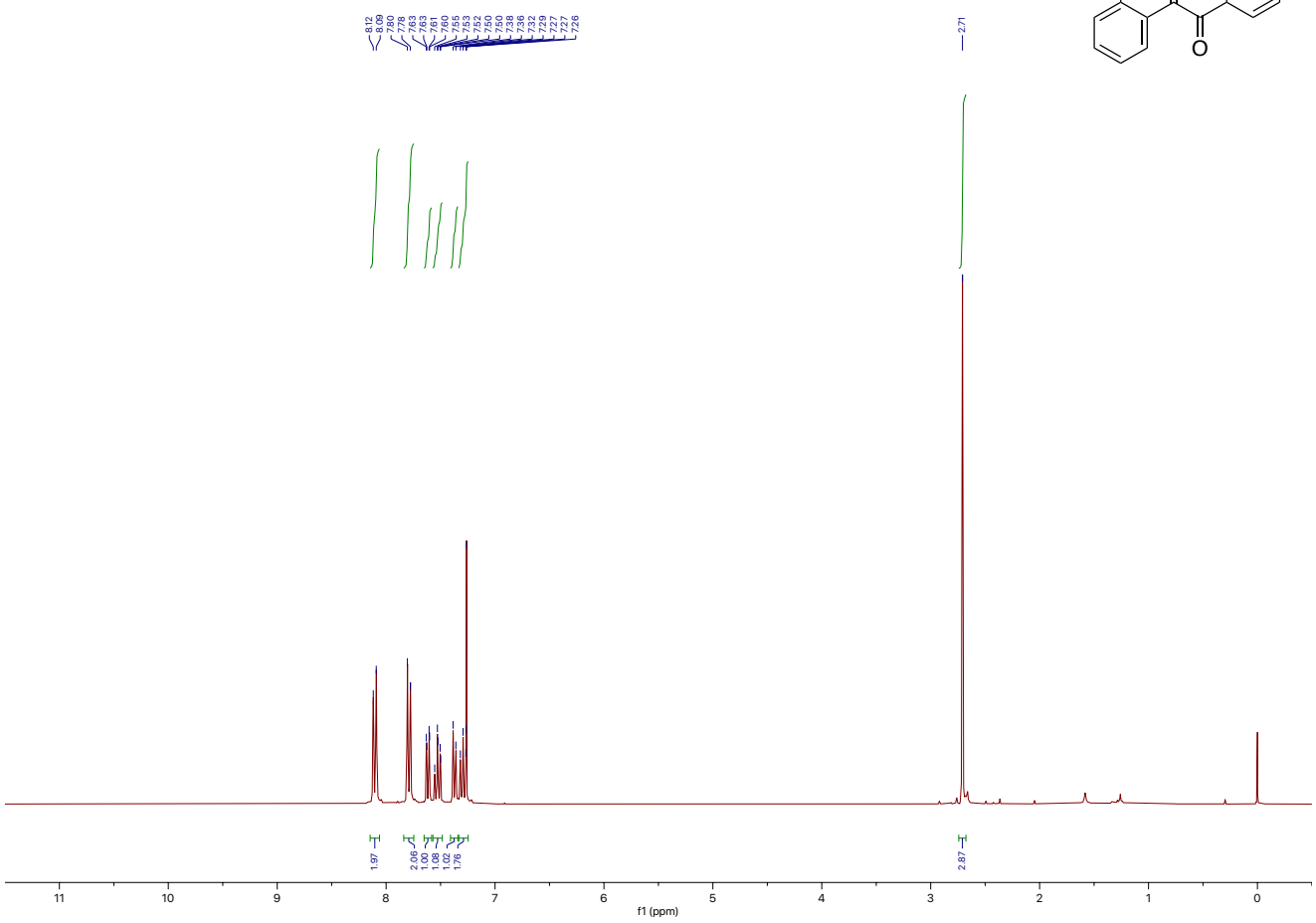
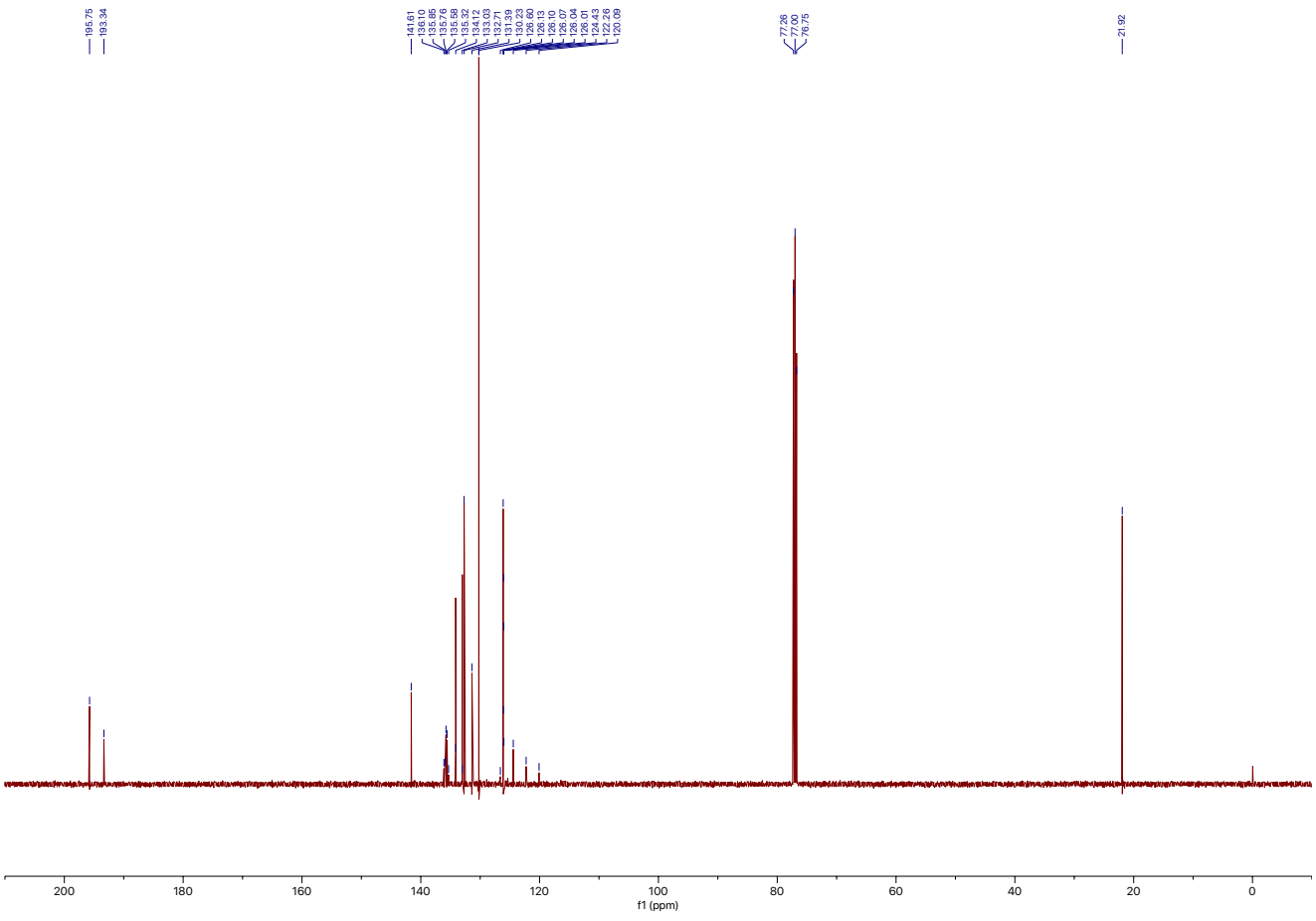
122.26
120.89
114.50
77.25
77.00
76.74
55.67
f1 (ppm)

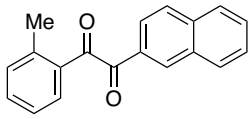
3gf

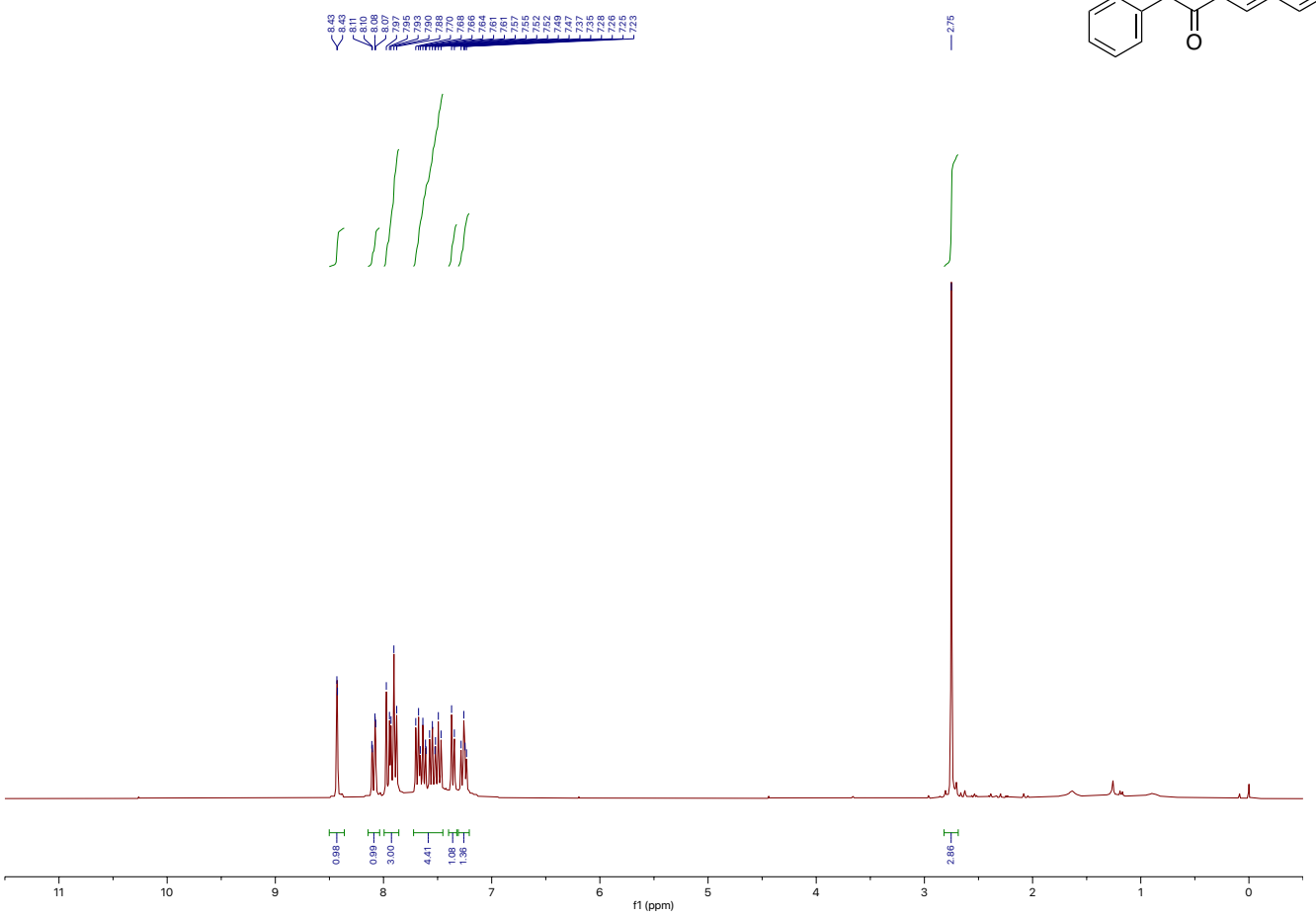

Me
O
O
CF3

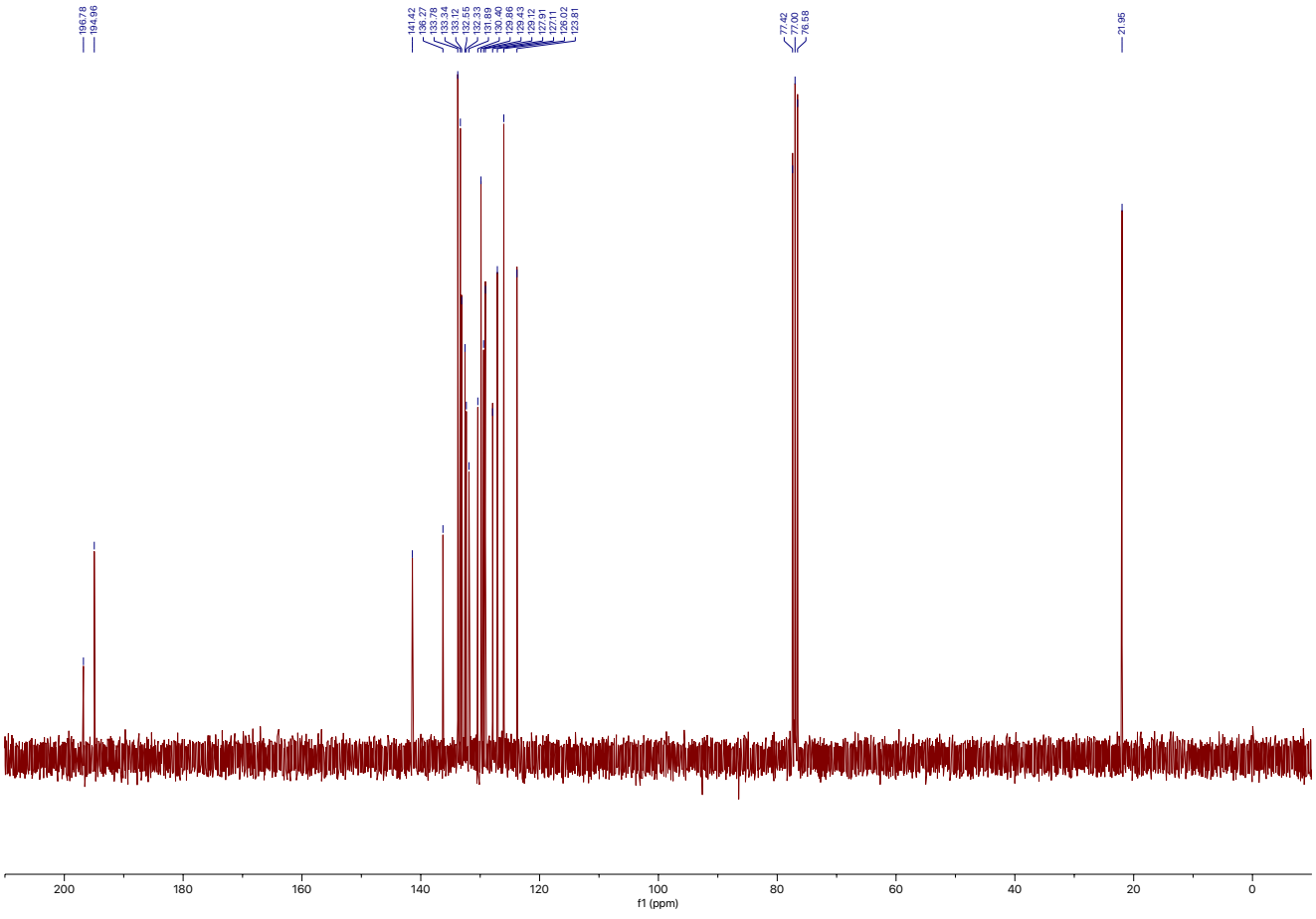

8.12
8.09
7.80
7.78
7.63
7.63
7.61
7.60
7.55
7.53
7.52
7.52
7.50
7.50
7.38
7.36
7.32
7.29
7.27
7.27
7.26
2.71
1.97
2.05
2.00
1.08
1.02
1.76
2.87
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)


195.75
193.34
141.61
136.10
135.85
135.76
135.58
135.32
134.12
133.03
132.71
131.39
130.93
126.60
126.13
126.10
126.07
126.04
126.01
124.43
122.26
120.09
77.26
77.00
76.75
21.92
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

3gl


Me
O
O


8.43
8.43
8.11
8.10
8.08
8.07
7.97
7.95
7.93
7.90
7.88
7.79
7.78
7.66
7.64
7.61
7.61
7.57
7.55
7.52
7.52
7.49
7.47
7.37
7.35
7.28
7.26
7.25
7.23
2.75
0.98
0.99
3.00
4.41
1.08
1.36
2.86
f1 (ppm)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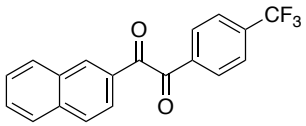
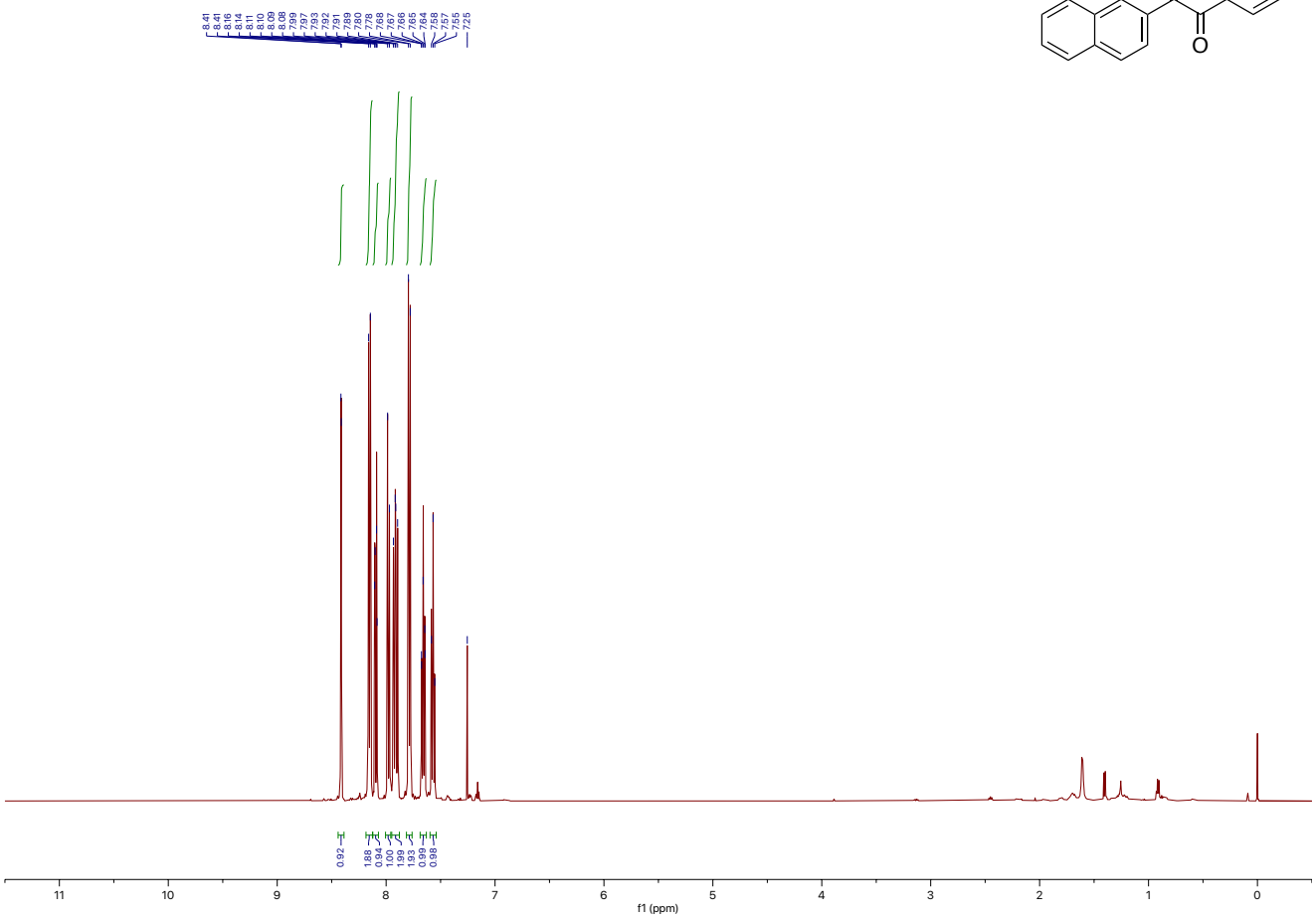
194.96
141.42
136.27
133.78
133.34
133.12
132.55
132.33
131.89
130.40
129.86
129.43
129.12
127.91
127.11
126.02
123.81
77.42
77.00
76.58
21.95
f1 (ppm)

3hf


O
O
CF3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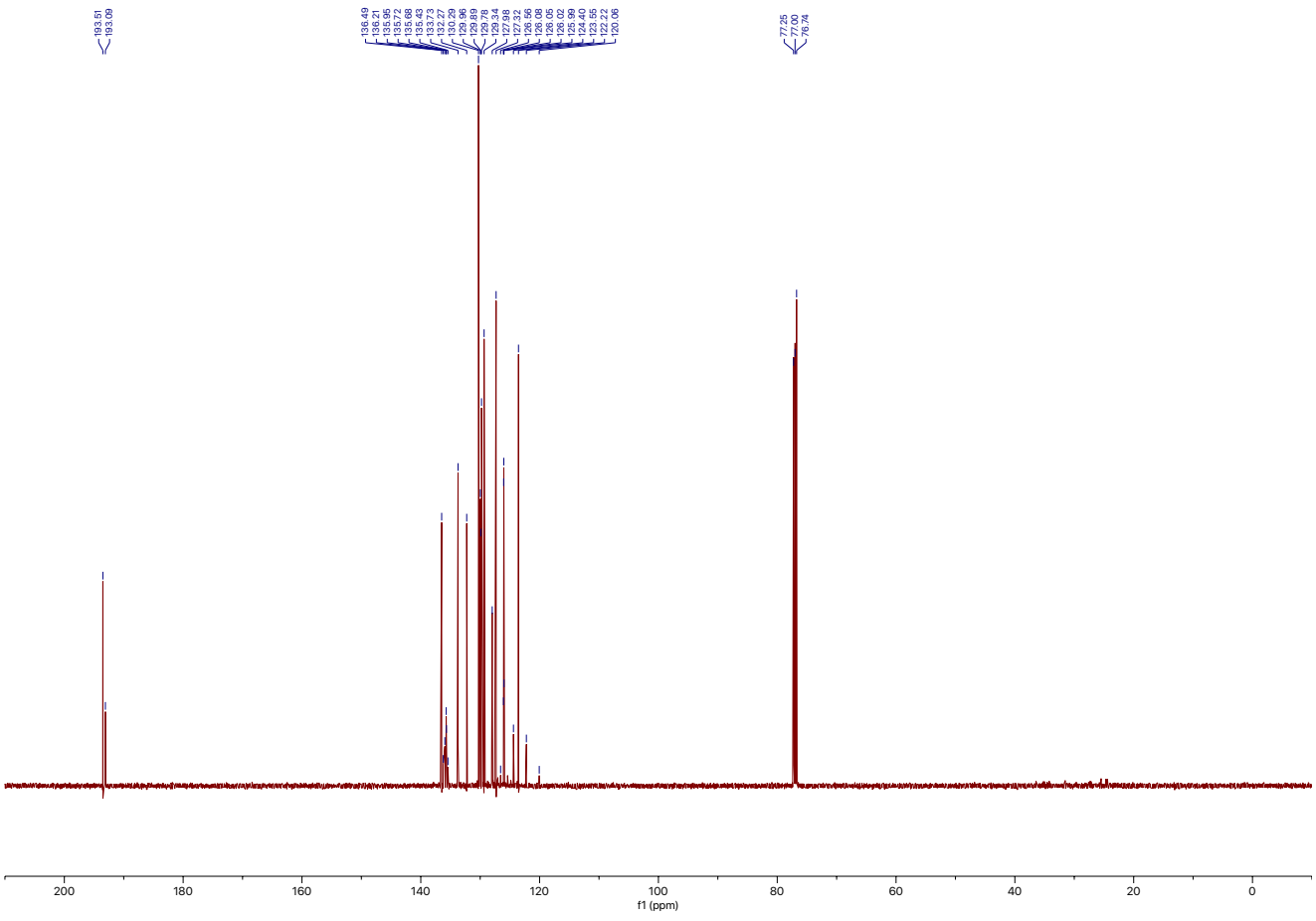
8.41
8.15
8.14
8.11
8.10
8.09
8.08
7.99
7.97
7.93
7.92
7.91
7.89
7.80
7.78
7.78
7.67
7.66
7.65
7.64
7.58
7.57
7.55
7.25
0.92
1.88
0.94
1.00
1.99
1.93
0.94
0.98
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)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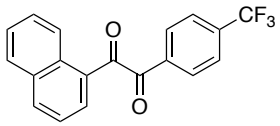
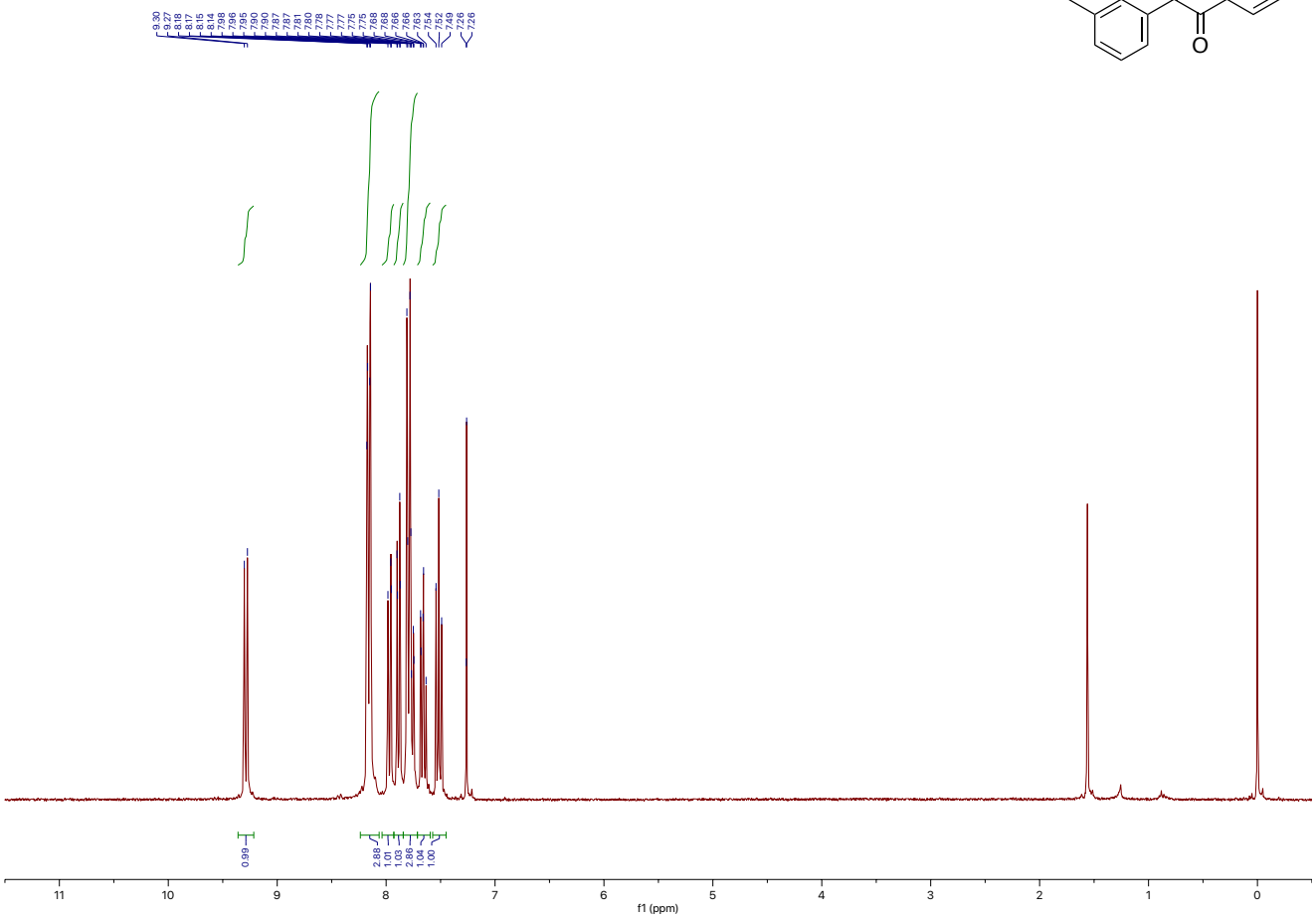
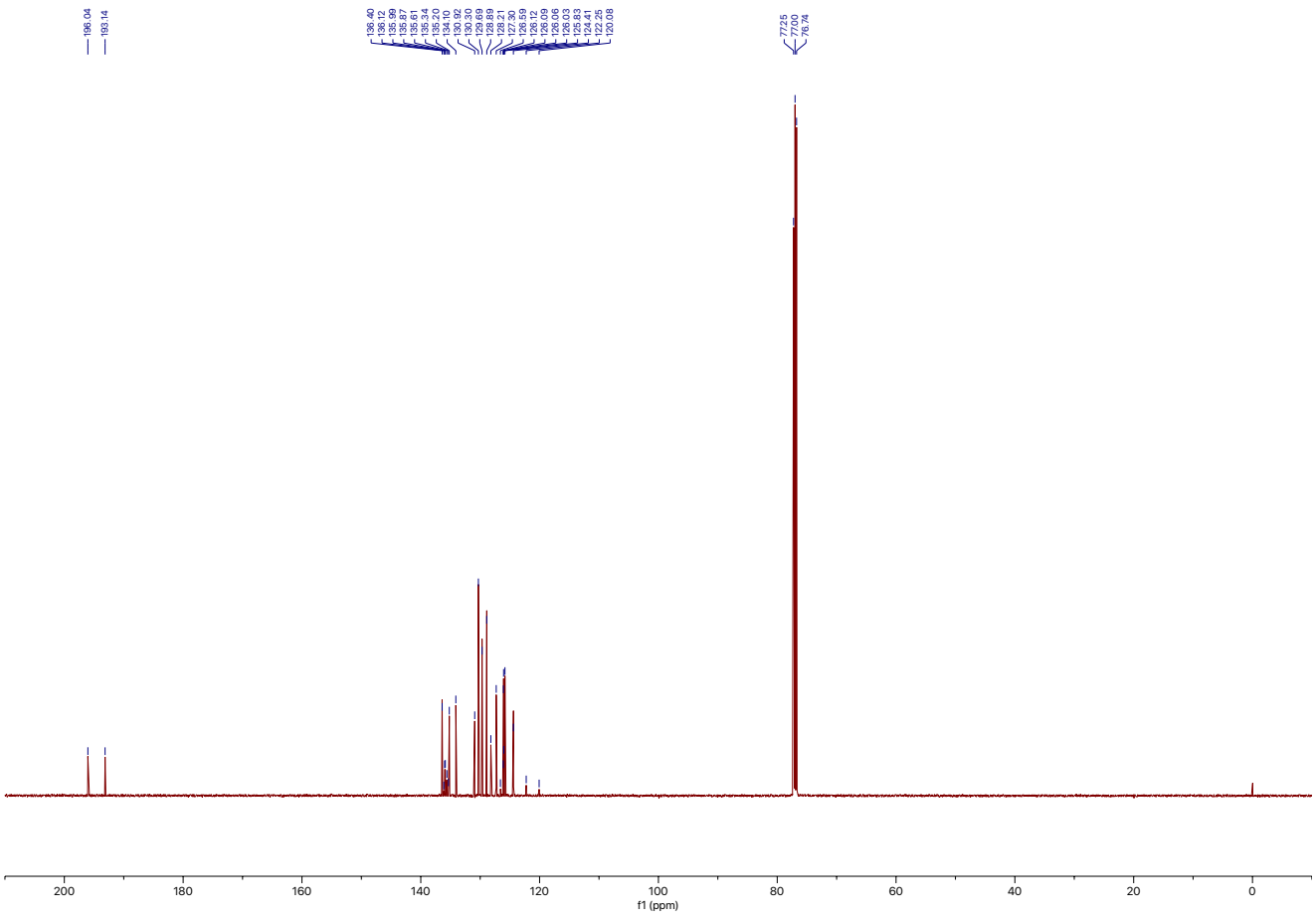
135.72
135.68
135.43
133.73
132.27
130.79
129.96
129.89
129.76
129.34
127.98
127.32
126.56
126.08
126.05
126.02
125.99
124.40
123.55
122.22
120.06
77.25
77.00
76.74
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

3if


O
O
CF3


9.30
9.27
8.18
8.17
8.15
8.14
7.798
7.796
7.795
7.790
7.787
7.787
7.781
7.780
7.778
7.777
7.777
7.775
7.775
7.768
7.768
7.766
7.766
7.763
7.754
7.752
7.749
7.726
7.726
0.99
2.88
1.01
1.03
2.00
1.04
1.00
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)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193.14
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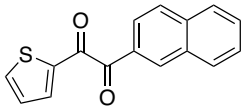
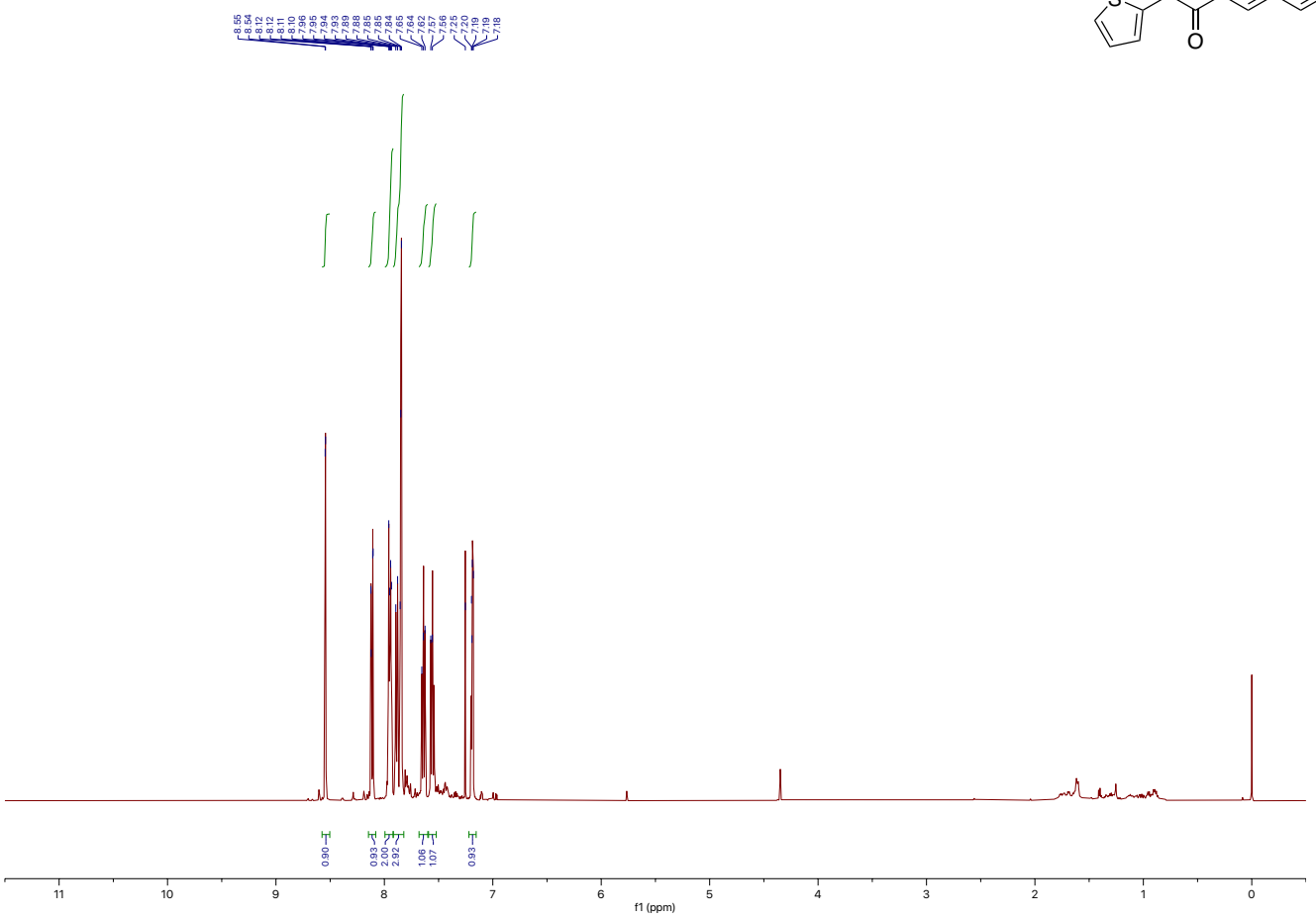
135.87
135.61
135.34
135.20
134.10
130.92
130.30
129.68
128.89
128.21
127.30
126.59
126.12
126.09
126.06
126.03
125.83
124.41
122.25
120.08
77.25
77.00
76.74
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

3kl


S
O
O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8.12
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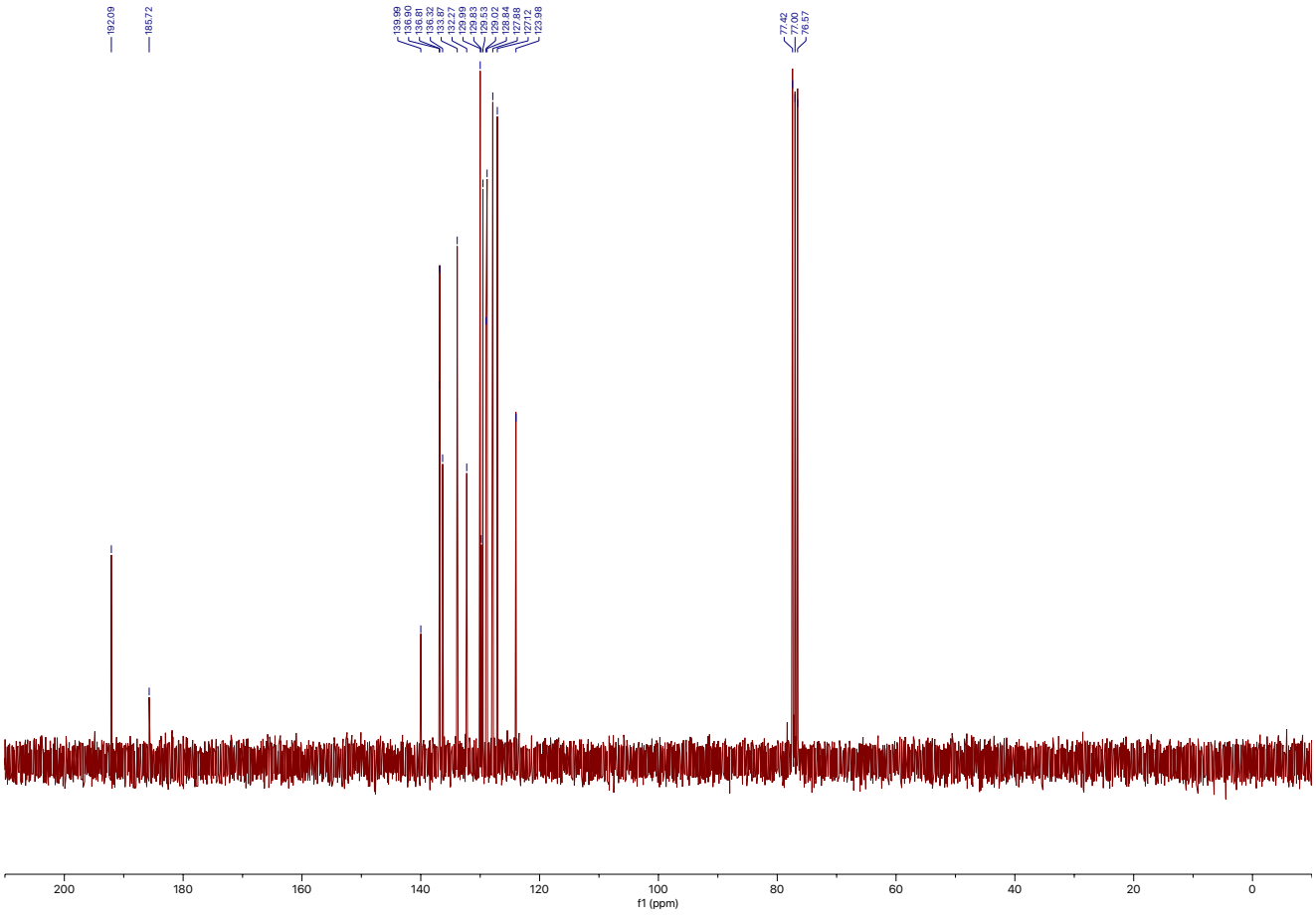
8.11
8.10
7.96
7.95
7.94
7.93
7.89
7.88
7.85
7.85
7.84
7.65
7.64
7.62
7.57
7.56
7.25
7.20
7.19
7.19
7.18
0.90
0.93
2.00
2.92
1.06
1.07
0.93
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)


192.09
185.72
139.99
138.90
136.81
136.32
133.87
133.27
129.99
129.83
129.53
129.02
128.84
127.88
127.12
123.98
77.42
77.00
76.57
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)