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Functionalisation of Isoindolinones via a Calcium Catalysed Hosomi-Sakurai Allylation

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Contents

1 - General Experimental

Solvents and reagents

All solvents were purchased from commercial sources and used without purification (HPLC or analytical grade). Anhydrous solvent was obtained from a Pure Solv™ Solvent Purification System. Standard vacuum line techniques were used and glassware was oven dried prior to use. Organic solvents were dried during workup using anhydrous Na_2SO_4. All calcium catalysed reactions where done without the need for anhydrous or air free conditions.

Purification and chromatography

Thin Layer Chromatography (TLC) was carried out using aluminium plates coated with 60 F254 silica gel. Plates were visualised using UV light (254 or 365 nm) or staining with 1% aq. KMnO4. Normal-phase silica gel chromatography was carried out using either a Biotage Isolera One flash column chromatography system (LPLC) or traditional flash column chromatography using Geduran® Silica gel 60, 40–63 microns RE.

Characterisation

Infrared spectroscopy was carried out with a Nicolet® 380 FT/IR – Fourier Transform Infrared Spectrometer. Only the most significant frequencies have been considered during the characterization and selected absorption maxima (νmax) recorded in wavenumbers (cm^{-1}). NMR spectra were recorded using a JEOL® ECS-400 MHz spectrometer using the deuterated solvent stated. Chemical shifts (δ) quoted in parts per million (ppm) and referenced to the residual solvent peak. Multiplicities are denoted as s- singlet, d- doublet, t- triplet, q- quartet and quin- quintet and derivatives thereof (br denotes a broad resonance peak). Coupling constants recorded as Hz and round to the nearest 0.1 Hz. High Resolution Mass Spectrometry (HRMS) was recorded using an Agilent Technologies® 6540 Ultra-High-Definition (UHD) AccurateMass equipped with a time of flight (Q-TOF) analyzer and the samples were ionized by ESI techniques and introduced through a high pressure liquid chromatography (HPLC) model Agilent Technologies® 1260 Infinity Quaternary LC system. Compound names were generated using ChemBioDraw Ultra v14 systematic naming

2 - General Procedures

General procedure **A –** Synthesis of hydroxyisoindolinones via Grignard addition

To a flame dried round bottomed flask purged with argon was added the corresponding phthalimide (1 equiv.) and anhydrous DCM (0.25 M). The suspension was cooled to 0 ^{o}C and the required Grignard reagent (3 equiv) was added dropwise over 5 minutes. The resulting solution was stirred at 0 ^{o}C for 10 minutes before being allowed to warm to room temperature over 2 hours. Once TLC analysis indicated conversion to the product, the reaction was quenched with sat. aq. NH_4Cl, extracted with Et_2O (3 x 25 mL), the combined organic layers washed with water, brine, dried with Na_2SO_4, filtered and concentrated. Flask column chromatography afforded the desired compounds

General procedure **B** - Synthesis of hydroxyisoindolinones via organolithium addition

To a flame dried round bottomed flask purged with argon was added the corresponding (hetero)aryl bromide (3 equiv.) and anhydrous THF (0.25 M). The solution was cooled to -78 ^{o}C and nBuLi (2 equiv rel. to bromide, 2.5 M in hexanes) was added dropwise. The resulting solution was stirred at -78 ^{o}C for 2 hours. Phthalimide was then added in one portion and the reaction was stirred at the stated temperature and time. The reaction was quenched with sat. aq. NH_4Cl, extracted with EtOAc (3 x 25 mL), the combined organic layers washed with water, brine, dried with Na_2SO_4, filtered and concentrated. Flask column chromatography afforded the desired compounds

General procedure **C** – Calcium catalysed allylation

To a 22mL vial equipped with a magnetic stirrer bar was added hydroxyisoindolinone, 1 mol% $Ca(NTf_2)_2$, 1 mol% $n$$Bu_4NPF_6$, followed by 1,2-DCE (1.5 mL), and the corresponding allyl silane (1.5 equiv.). The vial was sealed with a Teflon cap and the mixture stirred at 80 ^{o}C until TLC analysis indicated complete conversion to the product (approx. 30 mins). The mixture was concentrated and purified by flash column chromatography affording the desired compound.

Stock solutions were used for difficult to measure quantities. A 1M solution of both $Ca(NTf_2)_2$ and $n$$Bu_4NPF_6$ in THF was prepared and the desired quantities taken via syringe and placed in the reaction vessel. The solvent was then removed under vacuum before the addition of the other reagents.

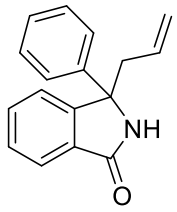
NB It was noted during the course of this study that the catalytic efficiency of the calcium salt slowly degrades over time. To ensure optimum activity, the commercial calcium salt should be thoroughly dried at 100^{o}C under vacuum for 12 hours before use and stored in a dessicator.

3 - Synthesis of hydroxyindolinones

All hydroxyindolinones **(1a,[1] 1b,[2] 1c,[3] 1d,[1] 1e,[1] 1f,[1] 1g,[1] 1h,[4] 1i,[5]1j,[6]1l,[7]1m,[8]1n,[9]1o,[10]1p,[11])** were synthesised via known methods (GP1 and GP2). All spectral data were in accordance to previously described.

4 - Calcium catalyzed allylation reactions

3-allyl-3-phenylisoindolin-1-one (2a)



The title compound was prepared according to general procedure C, from **1a** (250 mg, 1.11 mmol) using $Ca(NTf_2)_2$ (6.7 mg, 0.011 mmol), nBu_4PF_6 (4.3 mg, 0.011 mmol) and allyltrimethylsilane (190 mg, 1.67 mmol) in DCE (2.5 mL). Following conversion to the product and column chromatography (1:1 EtOAc:cyclohexane) afforded **2a** as a colourless oil (270 mg, 98%)

RF (1:1 EtOAc/CycHex) = 0.41

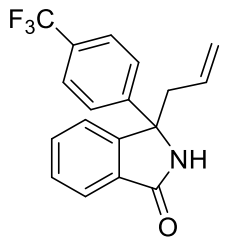
^{1}H NMR (400 MHz, DMSO-d_6) δ 9.16 (1 H, s), 7.59 (2 H, d, *J* = 7.3 Hz), 7.48 - 7.54 (3 H, m), 7.36 - 7.41 (1 H, m), 7.28 - 7.33 (2 H, m), 7.18 - 7.24 (1 H, m), 5.33 (1 H, ddt, *J*= 17.2, 10.1, 7.0, 7.0 Hz), 4.96 - 5.05 (1 H, m), 4.89 (1 H, dd, *J* = 10.3, 2.1 Hz), 2.94 - 3.09 (2 H, m)

^{13}C NMR (101 MHz, DMSO-d_6) δ 169.0, 150.5, 142.6, 132.5, 131.9, 131.3, 128.5, 128.1, 127.3, 125.5, 123.0, 122.9, 119.4, 66.1, 42.7

IR υ_{max} (cm^{-1}) 3238, 3051, 1681. 1228, 1015, 768

HR-ESI-MS: $C_{17}H_{16}NO$ $[M+H]^+$ *m/z* found 250.1234. Cald 250.1232

3-allyl-3-(4-(trifluoromethyl)phenyl)isoindolin-1-one (2b)



The title compound was prepared according to general procedure C, from **1b** (150 mg, 0.512 mmol) using $Ca(NTf_2)_2$ (3 mg, 0.00512 mmol), nBu_4PF_6 (2 mg, 0.00512 mmol) and allyltrimethylsilane (89 mg, 0.768 mmol) in DCE (2 mL). Following conversion to the product and column chromatography (1:1 EtOAc:cyclohexane) afforded **2a** as a white solid (143 mg, 88%)

RF (1:1 EtOAc/CycHex) = 0.71

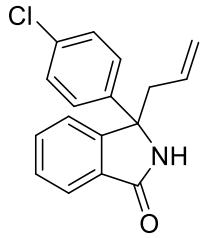
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (1H, s), 7.81 (2H, d, *J* = 7.3 Hz), 7.64 (2H, d, *J* = 8.7 Hz), 7.58 (2H, d, *J* = 8.7 Hz), 7.53 (1H, t, *J* = 7.3 Hz), 7.43 (1H, t, *J* = 7.3 Hz), 7.37 (1H, t, *J* = 7.3 Hz), 5.58-5.45 (1H, m), 5.14 (1H, d, *J* = 16.9 Hz), 5.07 (1H, d, *J* = 10.1 Hz), 3.27 (1H, dd, *J* = 13.9, 6.5 Hz), 3.27 (1H, dd, *J* = 13.9, 6.5 Hz)

^{13}C NMR (101 MHz, $CDCl_3$) δ 170.9, 150.1, 142.2, 132.5, 131.2, 130.5, 130.0 (q, $J_{C\text{-}F}$ = 32.8 Hz), 128.6, 126.0, 125.9 (quin, $J_{C\text{-}F}$ = 3.9 Hz), 124.1, 123.7 (d, $J_{C\text{-}F}$ = 273.5 Hz), 120.7, 66.5, 43.4

IR υ_{max} (cm^{-1}) 3231, 3079, 1678, 1469, 1350. 228, 1015, 768

HR-ESI-MS: $C_{18}H_{15}F_3NO$ $[M+H]^+$ *m/z* found 318.1110 Cald 318.1106

3-allyl-3-(4-chlorophenyl)isoindolin-1-one (2c)



The title compound was prepared according to general procedure C, from **1c** (132 mg, 0.51 mmol) using $Ca(NTf_2)_2$ (3 mg, 0.005 mmol), nBu_4PF_6 (2 mg, 0.005 mmol) and allyltrimethylsilane (87 mg, 0.765 mmol) in DCE (2 mL). Following conversion to the product and column chromatography (1:1 EtOAc:cyclohexane) afforded **2c** as a colourless oil (131 mg, 91%)

RF (1:1 EtOAc/CycHex) = 0.66

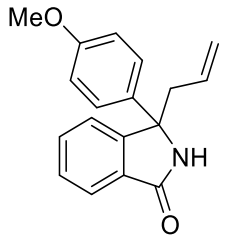
IR υ_{max} (cm^{-1}) 3069, 2906, 1699, 1399, 1064, 749

HR-ESI-MS: $C_{17}H_{15}ClNO$ $[M+H]^+$ *m/z* found 284.0842 Cald 284.0839

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (1H, s), 7.80 (1H, dd, *J* = 6.9, 0.9 Hz), 7.52 (1H, td, *J* = 7.4, 1,4 Hz), 7.47-7.42 (3H, m), 7.36 (1H, dd, *J* = 7.8, 0.9 Hz), 7.32-7.29 (2H, m), 5.50 (1H, ddt, *J* = 17.0, 10.0, 7.1 Hz), 5.09 (1H, dd, *J* = 17.0, 1.8 Hz), 5.02 (1H, dd, *J* = 10.0, 1.8 Hz), 3.22 (1H, dd, *J* = 14.2, 7.1 Hz), 2.88 (1H, dd, *J* = 14.2, 7.1 Hz)

^{13}C NMR (101 MHz, $CDCl_3$) δ 170.8, 150.5, 139.7, 133.6, 132.4, 131.4, 130.5, 128.9, 128.4, 127.0, 124.0, 122.2, 120.4, 66.2, 43.3.

3-allyl-3-(4-methoxyphenyl)isoindolin-1-one (2d)



The title compound was prepared according to general procedure C, from **1d** (102 mg, 0.4 mmol) using $Ca(NTf_2)_2$ (2 mg, 0.004 mmol), $n$$Bu_4PF_6$ (1.5 mg, 0.004 mmol) and allyltrimethylsilane (68 mg, 0.6 mmol) in DCE (2 mL). Following conversion to the product and column chromatography (1:1 EtOAc:cyclohexane) afforded **2d** as a pale yellow oil (104 mg, 93%)

RF (1:1 EtOAc/CycHex) = 0.53

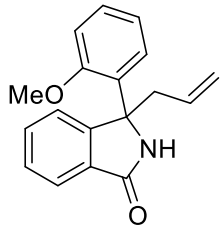
IR υ_{max} (cm^{-1}) 3230, 2956, 1686, 1511, 1276, 1058, 910

HR-ESI-MS: $C_{18}H_{18}NO_2$ $[M+H]^+$ *m/z* found 280.1334 Cald 280.1338

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (1H, s), 7.74 (1H, m), 7.50 (1H, td, *J* = 7.3, 0.9 Hz), 7.43-7.3 (4H, m), 6.86-6.1 (2H, m), 5.57-5.45 (1H, m), 5.02 (1H, dd, *J* = 17.0, 1.4 Hz), 4.98 (1H, dd, *J* = 10.1, 1.8 Hz), 3.75 (3H, s), 3.21 (1H, dd, *J* = 14.0, 6.6 Hz), 2.85 (1H, dd, *J* = 14.0, 7.6 Hz)

^{13}C NMR (101 MHz, $CDCl_3$) δ 170.8, 1.58.9, 151.3, 132.8, 132.2, 131.8, 130.4, 128.0, 126.7, 123.8, 122.2, 120.0, 114.1, 66.2, 55.2, 43.3

3-allyl-3-(2-methoxyphenyl)isoindolin-1-one (2e)



The title compound was prepared according to general procedure C, from **1e** (211 mg, 0.87 mmol) using $Ca(NTf_2)_2$ (5 mg, 0.009 mmol), $n$$Bu_4PF_6$ (3 mg, 0.009 mmol) and allyltrimethylsilane (149 mg, 1.3 mmol) in DCE (2 mL). Following conversion to the product and column chromatography (1:1 EtOAc:cyclohexane) afforded **2e** as a pale yellow oil which solidified upon standing (220 mg, 91%)

RF (1:1 EtOAc/CycHex) = 0.36

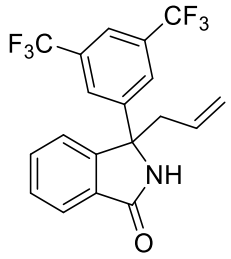
IR υ_{max} (cm^{-1}) 3231, 2967, 1687, 1503, 1277, 1066,

HR-ESI-MS: $C_{18}H_{18}NO_2$ $[M+H]^+$ *m/z* found 280.1340 Cald 280.1338

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86-7.82 (1H, m), 7.68-7.61 (2H, m), 7.53-7.48 (1H, m), 7.42 (1H, br s), 7.31-7.26 (1H, m), 7.24-7.21 (1H, m), 7.00-6.97 (1H, m), 6.86-6.81 (1H, m), 5.39-5.26 (1H, m), 4.98-4.87 (2H, m), 3.69 (3H, s), 3.25-3.18 (1H, m), 2.96-2.89 (1H, m)

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.1, 157.1, 147.9, 132.3, 132.2, 131.2, 129.3, 128.3, 128.3, 127.1, 124.8, 124.1, 120.5, 119.1, 111.5, 66.4, 55.3, 42.5

3-allyl-3-(3,5-bis(trifluoromethyl)phenyl)isoindolin-1-one (2f)



The title compound was prepared according to general procedure C, from **1e** (117 mg, 0.324 mmol) using $Ca(NTf_2)_2$ (2 mg, 0.003 mmol), $n$$Bu_4PF_6$ (1 mg, 0.003 mmol) and allyltrimethylsilane (55 mg, 0.49 mmol) in DCE (2 mL). Following conversion to the product and column chromatography (1:1 EtOAc:cyclohexane) afforded **2f** as an off white solid (108 mg, 87%)

RF (1:1 EtOAc/CycHex) = 0.59

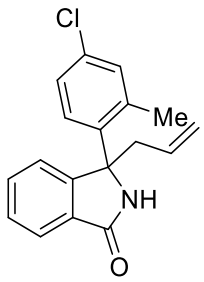
IR υ_{max} (cm^{-1}) 3211, 2986, 1698, 1408, 1317, 1128, 926, 752

HR-ESI-MS: $C_{19}H_{14}F_6NO$ $[M+H]^+$ *m/z* found 386.0983 Cald 386.0980

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (1 H, s), 7.99 (2 H, s), 7.86 (1 H, d, *J*= 7.3 Hz), 7.81 (1 H, s), 7.60 (1 H, td, *J*= 7.3, 0.9 Hz), 7.51 (1 H, t, *J* = 7.3 Hz), 7.39 (1 H, d, *J* = 7.8 Hz), 5.49 (1 H, ddt, *J* = 17.0, 10.0, 7.1, 7.1 Hz), 5.05 - 5.21 (2 H, m), 3.30 (1 H, dd, *J* = 14.1, 7.0 Hz), 2.98 (1 H, dd, *J* = 14.1, 7.0 Hz)

^{13}C NMR (101 MHz, $CDCl_3$) δ 171.0, 149.3, 144.4, 132.9, 132.4 (q, $J_{C\text{-}F}$ = 33.7 Hz), 130.5, 130.5, 129.0, 125.9, 124.4, 122.0, 121.9 (quin, $J_{C\text{-}F}$ = 3.9 Hz), 121.7, 121.3, 66.2, 43.6.

3-allyl-3-(4-chloro-2-methylphenyl)isoindolin-1-one (2g)



The title compound was prepared according to general procedure C, from **1G** (120 mg, 0.432 mmol) using $Ca(NTf_2)_2$ (2.5 mg, 0.004 mmol), $n$$Bu_4PF_6$ (1.7 mg, 0.004 mmol) and allyltrimethylsilane (74 mg, 0.65 mmol) in DCE (2 mL). Following conversion to the product and column chromatography (1:1 EtOAc:cyclohexane) afforded **2g** as a pale yellow solid (114 mg, 88%)

RF (1:1 EtOAc/CycHex) = 0.71

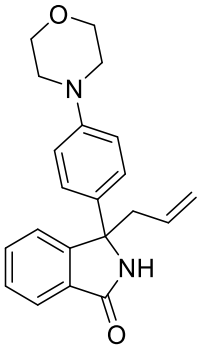
IR υ_{max} (cm^{-1}) 3203, 2980, 2879, 1689, 1491, 1316, 920, 750

HR-ESI-MS: $C_{18}H_{17}ClNO$ $[M+H]^+$ *m/z* found 298.1002 Cald 298.0999

^1H NMR (400 MHz, $CDCl_3$) δ 7.84 (1 H, d, *J* = 7.8 Hz), 7.61 (1 H, d, *J* = 8.7 Hz), 7.44 - 7.55 (2 H, m), 7.23 - 7.26 (1 H, m), 7.11 (1 H, d, *J* = 7.3 Hz), 7.07 (1 H, d, *J* = 2.3 Hz), 6.75 (1 H, s), 5.46 (1 H, ddt, *J* = 17.2, 10.2, 6.9, 6.9 Hz), 5.01 - 5.11 (2 H, m), 3.29 (1 H, dd, *J* = 14.0, 6.6 Hz), 2.89 (1 H, dd, *J* = 13.7, 7.3 Hz)

^{13}C NMR (101 MHz, $CDCl_3$) δ 170.6, 149.8, 140.0, 135.8, 133.8, 133.0, 132.6, 131.9, 131.3, 128.8, 128.4, 126.0, 123.7, 122.1, 120.9, 65.7, 44.6, 20.7

3-allyl-3-(4-morpholinophenyl)isoindolin-1-one (2h)



The title compound was prepared according to general procedure C, from **1h** (120 mg, 0.387 mmol) using $Ca(NTf_2)_2$ (2.3 mg, 0.004 mmol), $n$$Bu_4PF_6$ (1.5 mg, 0.004 mmol) and allyltrimethylsilane (66 mg, 0.58 mmol) in DCE (2 mL). Following conversion to the product and column chromatography (1:1 EtOAc:cyclohexane) afforded **2h** as a pale yellow solid (97 mg, 75%)

RF (1:1 EtOAc/CycHex) = 0.29

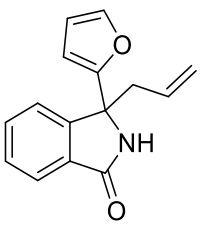
IR υ_{max} (cm^{-1}) 3215, 2969, 2901, 2885, 2824, 2361, 1686, 1514, 1277, 1066

HR-ESI-MS: $C_{21}H_{23}N_2O_2$ $[M+H]^+$ *m/z* found 335.1766 Cald 335.1760

^1H NMR (400 MHz, $CDCl_3$) δ 7.81 (1 H, d, *J*= 7.8 Hz), 7.48 - 7.53 (1 H, m), 7.31 - 7.45 (4 H, m), 6.84 - 6.89 (2 H, m), 6.60 (1 H, s), 5.38 - 5.72 (1 H, m), 5.03 - 5.17 (2 H, m), 3.80 - 3.88 (4 H, m), 3.25 (1 H, dd, *J* = 14.0, 6.2 Hz), 3.10 - 3.18 (4 H, m), 2.80 (1 H, dd, *J*= 14.0, 8.0 Hz).

^{13}C NMR (101 MHz, $CDCl_3$) δ 170.2, 151.5, 150.6, 132.3, 132.1, 131.7, 128.1, 126.4, 123.9, 123.9, 122.1, 120.1, 115.5, 66.8, 65.9, 48.8, 43.3

3-allyl-3-(furan-2-yl)isoindolin-1-one (2i)



The title compound was prepared according to general procedure C, from **1i** (150 mg, 0.698 mmol) using $Ca(NTf_2)_2$ (4 mg, 0.007 mmol), $n Bu_4PF_6$ (2.7 mg, 0.007 mmol) and allyltrimethylsilane (119 mg, 1.05 mmol) in DCE (2 mL). Following conversion to the product and column chromatography (1:1 EtOAc:cyclohexane) afforded **2i** as a yellow oil (135 mg, 81%)

RF (1:1 EtOAc/CycHex) = 0.42

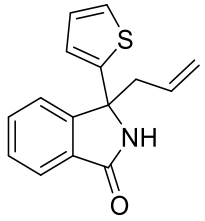
IR υ_{max} (cm^{-1}) 2971, 2901, 1668, 1380, 1034, 788

HR-ESI-MS: $C_{15}H_{14}NO_2$ $[M+H]^+$ *m/z* found 240.1032 Cald 240.1025

^1H NMR (400 MHz, $CDCl_3$) δ 7.83 - 7.86 (1 H, m), 7.55 - 7.59 (2 H, m), 7.45 - 7.51 (1 H, m), 7.41 (1 H, dd, *J* = 1.8, 0.9 Hz), 6.95 (1 H, br. s.), 6.27 - 6.31 (1 H, m), 6.21 (1 H, dd, *J* = 3.2, 0.9 Hz), 5.45 - 5.56 (1 H, m), 5.06 - 5.13 (2 H, m), 3.13 - 3.21 (1 H, m), 2.72 - 2.80 (1 H, m).

^{13}C NMR (101 MHz, $CDCl_3$) δ 170.0, 153.5, 148.1, 142.8, 132.3, 131.4, 130.5, 128.7, 124.0, 122.5, 120.4, 110.3, 106.1, 60.1, 42.7

3-allyl-3-(thiophen-2-yl)isoindolin-1-one (2j)



The title compound was prepared according to general procedure C, from **1j** (50 mg, 0.217 mmol) using $Ca(NTf_2)_2$ (1.3 mg, 0.002 mmol), $n Bu_4PF_6$ (0.8 mg, 0.002 mmol) and allyltrimethylsilane (37 mg, 0.325 mmol) in DCE (1 mL). Following conversion to the product and column chromatography (1:1 EtOAc:cyclohexane) afforded **2j** as a yellow oil (48 mg, 88%)

RF (1:1 EtOAc/CycHex) = 0.49

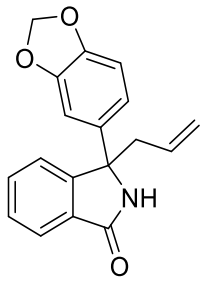
IR υ_{max} (cm^{-1}) 3671, 3182, 3069, 2901, 2361, 1690, 1394, 1067

HR-ESI-MS: $C_{15}H_{14}NOS$ $[M+H]^+$ *m/z* found 256.0790 Cald 256.0796

^1H NMR (400 MHz, $CDCl_3$) δ 7.80 - 7.85 (1 H, m), 7.53 - 7.58 (1 H, m), 7.43 - 7.48 (2 H, m), 7.32 (1 H, s), 7.21 (1 H, dd, *J* = 5.0, 0.9 Hz), 7.10 (1 H, dd, *J*=3.7, 1.4 Hz), 6.96 (1 H, dd, *J* = 5.5, 3.7 Hz), 5.46 - 5.65 (1 H, m), 5.10 - 5.16 (1 H, m), 5.08 (1 H, dd, *J* = 10.3, 1.6 Hz), 3.21 (1 H, dd, *J* = 14.0, 6.6 Hz), 2.88 (1 H, dd, *J* = 14.2, 7.8 Hz)

^{13}C NMR (101 MHz, $CDCl_3$) δ 170.0, 150.2, 146.0, 132.4, 131.3, 130.1, 128.5, 127.2, 124.9, 124.0, 123.9, 122.2, 120.7, 67.7, 45.0

3-allyl-3-(benzo[d][1,3]dioxol-5-yl)isoindolin-1-one (2k)



The title compound was prepared according to general procedure C, from **1k** (71 mg, 0.264 mmol) using $Ca(NTf_2)_2$ (1.6 mg, 0.003 mmol), nBu_4PF_6 (1 mg, 0.003 mmol) and allyltrimethylsilane (45 mg, 0.396 mmol) in DCE (1 mL). Following conversion to the product and column chromatography (1:1 EtOAc:cyclohexane) afforded **2g** as a pale yellow oil (48 mg, 87%)

RF (1:1 EtOAc/CycHex) = 0.21

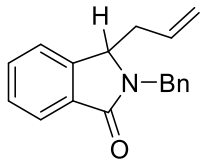
IR υ_{max} (cm^{-1}) 3215, 2971, 1666, 1469, 1278, 852, 631

HR-ESI-MS: $C_{18}H_{16}NO_3$ $[M+H]^+$ *m/z* found 294.1119 Cald 294.1130

^1H NMR (400 MHz, $CDCl_3$) δ 7.73 - 7.88 (2 H, m), 7.50 (1 H, dd, *J* = 7.8, 0.9 Hz), 7.41 (1 H, t, *J*= 6.9 Hz), 7.32 (1 H, d, *J*= 7.3 Hz), 6.95 - 6.98 (1 H, m), 6.90 (1 H, d, *J*= 1.8 Hz), 6.76 (1 H, d, *J* = 8.2 Hz), 6.64 (1 H, br. s.), 5.44 - 5.61 (1 H, m), 3.22 (1 H, dd, *J* = 14.2, 6.4 Hz), 2.75 (1 H, dd, *J* = 14.2, 8.2 Hz)

^{13}C NMR (101 MHz, $CDCl_3$) δ 170.3, 151.2, 148.2, 147.2, 134.7, 134.2, 131.8, 128.3, 124.0, 122.1, 120.3, 118.7, 108.3106.3, 101.3, 66.2, 43.4.

3-allyl-2-benzylisoindolin-1-one (2l)



The title compound was prepared according to general procedure C, from **1l** (71 mg, 0.264 mmol) using $Ca(NTf_2)_2$ (1.6 mg, 0.003 mmol), nBu_4PF_6 (1 mg, 0.003 mmol) and allyltrimethylsilane (45 mg, 0.396 mmol) in DCE (1 mL). Following conversion to the product and column chromatography (1:1 EtOAc:cyclohexane) afforded **2l** as a pale yellow oil (48 mg, 87%)

RF (10:1 EtOAc/CycHex) = 0.34

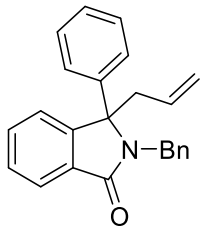
IR υ_{max} (cm^{-1}) 3063, 3974, 2921, 1680, 1455, 1076

HR-ESI-MS: $C_{11}H_{12}NO$ $[M+H]^+$ *m/z* found 174.0922 Cald 174.0919

^1H NMR (400 MHz, $CDCl_3$) δ7.88 (1 H, d, *J* = 7.3 Hz), 7.41 - 7.53 (2 H, m), 7.37 (1 H, d, *J* = 7.3 Hz), 7.22 - 7.33 (5 H, m), 5.41 (1 H, d, *J* = 15.1 Hz), 5.26 - 5.38 (1 H, m), 5.02 (1 H, dd, *J* = 16.9, 1.4 Hz), 4.97 (1 H, d, *J* = 10.1 Hz), 4.41 (1 H, dd, *J* = 5.7, 3.9 Hz), 4.16 (1 H, d, *J* = 15.1 Hz), 2.58 - 2.76 (2 H, m)

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.4, 144.7, 137.0, 132.2, 131.3, 131.1, 128.7, 128.5, 128.1, 128.0, 127.5, 123.7, 122.3, 119.2, 57.8, 43.7, 35.0

3-allyl-2-benzyl-3-phenylisoindolin-1-one (2m)



The title compound was prepared according to general procedure C, from **1m** (100 mg, 0.317 mmol) using $Ca(NTf_2)_2$ (1.9 mg, 0.003 mmol), nBu_4PF_6 (1.2 mg, 0.003 mmol) and allyltrimethylsilane (54 mg, 0.476 mmol) in DCE (2 mL). Following conversion to the product and column chromatography (10% EtOAc:cyclohexane) afforded **2m** as a colourless oil which solidified upon standing (102 mg, 95%)

RF (10% EtOAc/CycHex) = 0.38

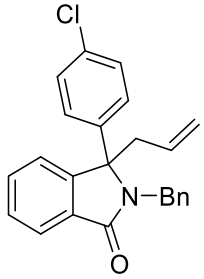
IR υ_{max} (cm^{-1}) 3012, 2043, 2001, 1690, 1439, 897, 432

HR-ESI-MS: $C_{24}H_{22}NO$ $[M+H]^+$ *m/z* found 340.1710 Cald 340.1701

^1H NMR (400 MHz, $CDCl_3$) δ 7.87 - 7.95 (1 H, m), 7.39 - 7.48 (2 H, m), 7.20 - 7.25 (5 H, m), 7.14 - 7.18 (3 H, m), 7.03 - 7.11 (3 H, m), 4.90 (1 H, ddt, *J* = 16.9, 10.1, 6.9, 6.9 Hz), 4.78 (1 H, d, *J* = 15.1 Hz), 4.63 - 4.70 (1 H, m), 4.55 (1 H, dd, *J* = 17.2, 1.6 Hz), 4.05 (1 H, d, *J* = 14.7 Hz), 2.97 - 3.15 (2 H, m)

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.3, 150.0, 139.6, 137.7, 132.0, 130.8, 129.2, 128.6, 128.1, 128.1, 128.0, 127.2, 127.0, 123.6, 122.2, 119.4, 70.9, 44.0, 38.5

3-allyl-2-benzyl-3-(4-chlorophenyl)isoindolin-1-one (2n)



The title compound was prepared according to general procedure C, from **1n** (100 mg, 0.287 mmol) using $Ca(NTf_2)_2$ (1.7 mg, 0.003 mmol), nBu_4PF_6 (1.1 mg, 0.003 mmol) and allyltrimethylsilane (49 mg, 0.429 mmol) in DCE (2 mL). Following conversion to the product and column chromatography (10% EtOAc:cyclohexane) afforded **2n** as a colourless oil which solidified upon standing (102 mg, 95%)

RF (10% EtOAc/CycHex) = 0.49

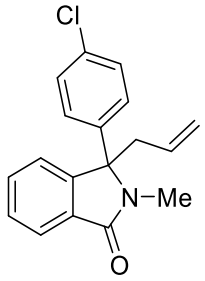
IR υ_{max} (cm^{-1}) 2888, 2649, 1687, 1034, 945, 710, 649

HR-ESI-MS: $C_{24}H_{21}ClNO_3$ $[M+H]^+$ *m/z* found 374.1321 Cald 374.1312

^1H NMR (400 MHz, $CDCl_3$) δ 7.91 - 7.95 (1 H, m), 7.43 - 7.50 (2 H, m), 7.15 - 7.24 (7 H, m), 6.98 - 7.07 (3 H, m), 4.85 - 4.98 (1 H, m), 4.60 - 4.74 (3 H, m), 4.21 (1 H, d, *J*=15.1 Hz), 3.01 - 3.14 (2 H, m)

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.1, 149.5, 138.2, 137.5, 133.9, 132.2, 131.3, 130.3, 129.1, 128.7, 128.4, 128.3, 128.1, 127.2, 123.7, 122.0, 119.7, 77.3, 76.7, 70.2, 43.9, 38.4

3-allyl-3-(4-chlorophenyl)-2-methylisoindolin-1-one (2o)



The title compound was prepared according to general procedure C, from **1o** (100 mg, 0.366 mmol) using $Ca(NTf_2)_2$ (2.2 mg, 0.004 mmol), $n$$Bu_4PF_6$ (1.4 mg, 0.004 mmol) and allyltrimethylsilane (63 mg, 0.549 mmol) in DCE (2 mL). Following conversion to the product and column chromatography (10% EtOAc:cyclohexane) afforded **2o** as a colourless oil (99 mg, 95%)

RF (10% EtOAc/CycHex) = 0.53

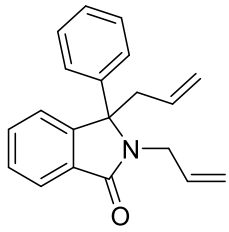
IR υ_{max} (cm^{-1}) 2978, 2004, 1659, 1432, 1054, 930, 888, 721

HR-ESI-MS: $C_{18}H_{17}ClNO$ $[M+H]^+$ *m/z* found 298.0991 Cald 298.0999

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 - 7.87 (1 H, m), 7.37 - 7.47 (2 H, m), 7.22 - 7.30 (2 H, m), 7.08 - 7.13 (3 H, m), 4.98 - 5.07 (2 H, m), 4.88 - 4.93 (1 H, m), 3.21 - 3.29 (1 H, m), 3.00 - 3.14 (1 H, m), 2.81 (3 H, s)

^{13}C NMR (101 MHz, $CDCl_3$) δ168.6, 149.0, 138.3, 133.9, 131.9, 131.4, 130.4, 129.1, 128.3, 127.6, 123.5, 121.7, 119.8, 77.3, 76.7, 69.2, 38.2, 24.9

2,3-diallyl-3-phenylisoindolin-1-one (2p)



The title compound was prepared according to general procedure C, from **1p** (207 mg, 0.781 mmol) using $Ca(NTf_2)_2$ (5 mg, 0.008 mmol), $n$$Bu_4PF_6$ (3 mg, 0.008 mmol) and allyltrimethylsilane (134 mg, 1.172 mmol) in DCE (2 mL). Following conversion to the product and column chromatography (10% EtOAc:cyclohexane) afforded **2n** as a colourless oil which solidified upon standing (207 mg, 92%)

RF (5% EtOAc/CycHex) = 0.39

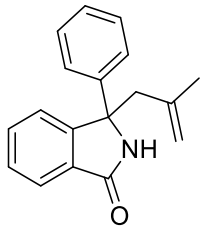
IR υ_{max} (cm^{-1}) 3659, 2658, 1689, 1067, 1000, 931, 654

HR-ESI-MS: $C_{20}H_{20}NO$ $[M+H]^+$ *m/z* found 290.1553 Cald 290.1545

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (1 H, d, *J* = 6.4 Hz), 7.38 - 7.47 (2 H, m), 7.24 - 7.32 (3 H, m), 7.13 - 7.18 (2 H, m), 7.08 (1 H, d, *J* = 6.9 Hz), 5.66 - 5.84 (1 H, m), 5.12 - 5.24 (1 H, m), 5.04 (1 H, dd, *J* = 17.4, 1.4 Hz), 4.93 - 5.00 (2 H, m), 4.89 (1 H, dd, *J* = 10.1, 1.4 Hz), 4.03 - 4.10 (1 H, m), 3.69 (1 H, dd, *J* = 15.6, 7.3 Hz), 3.23 (2 H, d, *J* = 6.9 Hz)

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.8, 149.8, 139.8, 133.6, 131.9, 131.5, 131.1, 128.6, 128.0, 126.7, 123.4, 122.1, 119.7, 117.3, 70.4, 43.3, 38.4

3-(2-methylallyl)-3-phenylisoindolin-1-one (3a)



The title compound was prepared according to general procedure C, from **1a** (110 mg, 0.489 mmol) using $Ca(NTf_2)_2$ (2.9 mg, 0.005 mmol), $n$$Bu_4PF_6$ (1.9 mg, 0.005 mmol) and methylallyltrimethylsilane (94 mg, 0.735 mmol) in DCE (2 mL). Following conversion to the product and column chromatography (1:1 EtOAc:cyclohexane) afforded **3a** as a colourless oil which solidified upon standing (118 mg, 92%)

RF (1:1 EtOAc/CycHex) = 0.51

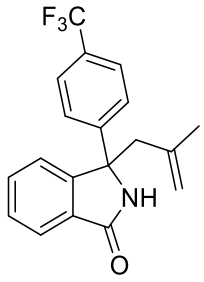
IR υ_{max} (cm^{-1}) 3255, 3076, 2922, 2359, 1378, 1680, 1409, 1227, 953

HR-ESI-MS: $C_{18}H_{18}NO$ $[M+H]^+$ *m/z* found 264.1398 Cald 264.1388

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (1 H, d, *J* = 7.8 Hz), 7.50 - 7.55 (2 H, m), 7.46 - 7.50 (1 H, m), 7.37 - 7.44 (2 H, m), 7.30 - 7.35 (2 H, m), 7.22 - 7.28 (1 H, m), 7.14 (1 H, s), 4.80 (1 H, t, *J* = 1.6 Hz), 4.60 (1 H, d, *J* = 0.9 Hz), 3.24 (1 H, d, *J* = 14.2 Hz), 2.85 (1 H, d, *J* = 14.2 Hz), 1.40 (3 H, s)

^{13}C NMR (101 MHz, $CDCl_3$) δ 170.4, 151.5, 141.7, 140.3, 132.2, 130.1, 128.8, 128.2, 127.7, 125.2, 124.0, 122.5, 116.6, 66.2, 46.8, 24.3

3-(2-methylallyl)-3-(4-(trifluoromethyl)phenyl)isoindolin-1-one (3b)



The title compound was prepared according to general procedure C, from **1b** (163 mg, 0.556 mmol) using $Ca(NTf_2)_2$ (3.3 mg, 0.006 mmol), $n$$Bu_4PF_6$ (2.2 mg, 0.006 mmol) and methylallyltrimethylsilane (107 mg, 0.834 mmol) in DCE (2 mL). Following conversion to the product and column chromatography (1:1 EtOAc:cyclohexane) afforded **3b** as a colourless oil which solidified upon standing (155 mg, 84%)

RF (1:1EtOAc/CycHex) = 0.61

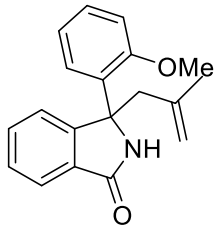
IR υ_{max} (cm^{-1}) 3600, 2967, 2459, 1764, 1683, 1057, 946, 719, 630

HR-ESI-MS: $C_{19}H_{17}F_3NO$ $[M+H]^+$ *m/z* found 332.1266 Cald 332.1262

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 - 7.84 (1 H, m), 7.69 (2 H, d, *J* = 8.2 Hz), 7.60 - 7.63 (3 H, m), 7.54 (1 H, td, *J*=7.4, 1.1 Hz), 7.41 - 7.47 (2 H, m), 4.84 (1 H, t, *J* = 1.8 Hz), 4.62 (1 H, s), 3.28 (1 H, d, *J* = 14.2 Hz), 2.91 (1 H, d, *J* = 14.2 Hz), 1.44 (3 H, s)

^{13}C NMR (101 MHz, $CDCl_3$) δ 170.6, 150.7, 145.9, 139.7, 132.1, 130.1, 128.6, 128.8 (q, J_{C-F} = 3.9 Hz), 125.8, 124.2, 122.4, 116.9, 66.2, 46.7, 24.3

3-(2-methoxyphenyl)-3-(2-methylallyl)isoindolin-1-one (3c)



The title compound was prepared according to general procedure C, from **1e** (164 mg, 0.675 mmol) using $Ca(NTf_2)_2$ (4 mg, 0.007 mmol), $n$$Bu_4PF_6$ (2.5 mg, 0.006 mmol) and methylallyltrimethylsilane (129 mg, 1.02 mmol) in DCE (2 mL). Following conversion to the product and column chromatography (1:1 EtOAc:cyclohexane) afforded **3c** as a pale yellow oil which solidified upon standing (172 mg, 87%)

RF (1:1 EtOAc/CycHex) = 0.27

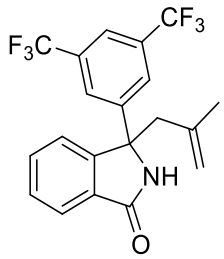
IR υ_{max} (cm^{-1}) 2977, 2901, 1700, 1668, 1393, 1116, 923

HR-ESI-MS: $C_{19}H_{20}NO_2$ $[M+H]^+$ *m/z* found 294.1499 Cald 294.1494

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (1 H, d, *J* = 7.8 Hz), 7.70 - 7.74 (1 H, m), 7.63 (1 H, td, *J* = 7.6, 1.4 Hz), 7.49 (1 H, td, *J* = 7.6, 0.9 Hz), 7.43 (1 H, s), 7.21 - 7.31 (2 H, m), 6.97 (1 H, dd, *J* = 8.2, 0.9 Hz), 6.82 (1 H, td, *J* = 7.6, 0.9 Hz), 4.64 - 4.72 (1 H, m), 4.44 (1 H, d, *J* = 0.9 Hz), 3.97 (3 H, s), 3.25 (1 H, d, *J* = 13.7 Hz), 2.84 (1 H, d, *J* = 13.3 Hz), 1.34 (3 H, s)

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.1, 157.1, 148.1, 140.4, 132.4, 130.9, 129.2, 129.0, 128.4, 126.9, 125.3, 124.1, 120.6, 116.1, 111.5, 66.6, 55.3, 46.1, 24.0

3-(3,5-bis(trifluoromethyl)phenyl)-3-(2-methylallyl)isoindolin-1-one (3d)



The title compound was prepared according to general procedure C, from **1f** (71 mg, 0.197 mmol) using $Ca(NTf_2)_2$ (1 mg, 0.002 mmol), $n$$Bu_4PF_6$ (0.7 mg, 0.002 mmol) and methylallyltrimethylsilane (37 mg, 0.296 mmol) in DCE (1 mL). Following conversion to the product and column chromatography (1:1 EtOAc:cyclohexane) afforded **3d** as a yellow oil (70 mg, 90%)

RF (1:1 EtOAc/CycHex) = 0.60

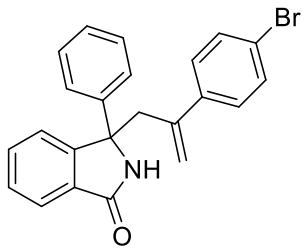
IR υ_{max} (cm^{-1}) 2946, 2001, 1663, 1459. 1236, 907

HR-ESI-MS: $C_{20}H_{16}F_6NO$ $[M+H]^+$ *m/z* found 400.1133 Cald 400.1136

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (2 H, s), 7.81 - 7.86 (2 H, m), 7.56 - 7.61 (1 H, m), 7.47 - 7.52 (2 H, m), 7.39 (1 H, d, *J* = 7.8 Hz), 7.32 (1 H, s), 4.89 (1 H, t, *J* = 1.4 Hz), 4.63 (1 H, s), 3.29 (1 H, d, *J* = 14.2 Hz), 2.93 (1 H, d, *J* = 14.2 Hz), 1.44 (3 H, s)

^{13}C NMR (101 MHz, $CDCl_3$) δ 170.4, 149.9, 144.8, 139.1, 132.9, 132.6 (q, $J_{C\text{-}F}$ = 33.4 Hz), 129.1, 126.1, 125.6, 124.4, 122.1, 122.0 (m), 117.6, 65.8, 46.9, 24.2

3-(2-(4-bromophenyl)allyl)-3-phenylisoindolin-1-one (3e)



The title compound was prepared according to general procedure C, from **1a** (43 mg, 0.191 mmol) using $Ca(NTf_2)_2$ (1 mg, 0.002 mmol), $n$$Bu_4PF_6$ (0.7 mg, 0.002 mmol) and (2-(4-bromophenyl)allyl)trimethylsilane[12] (77 mg, 0.287 mmol) in DCE (1 mL). Following conversion to the product and column chromatography (25% EtOAc:cyclohexane) afforded **3e** as a colourless oil which solidified upon standing (70 mg, 91%)

RF (1:1 EtOAc/CycHex) = 0.61

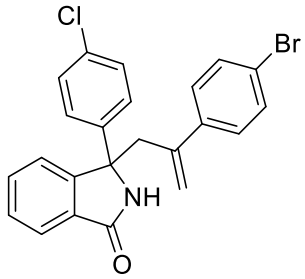
IR υ_{max} (cm^{-1}) 3675, 3180, 2988, 2901, 2361, 1694, 1486, 1229, 802

HR-ESI-MS: $C_{23}H_{19}BrNO$ $[M+H]^+$ *m/z* found 404.0655 Cald 404.0650. *Bromine isotope pattern*

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 - 7.72 (1 H, m), 7.45 - 7.49 (2 H, m), 7.28 - 7.34 (4 H, m), 7.19 - 7.27 (4 H, m), 7.08 (1 H, s), 6.83 - 6.88 (2 H, m), 5.09 (1 H, d, *J* = 0.9 Hz), 4.91 (1 H, s), 3.48 - 3.60 (2 H, m)

^{13}C NMR (101 MHz, $CDCl_3$) δ 170.3, 150.1, 142.6, 141.5, 141.1, 131.8, 131.1, 130.6, 128.8, 128.1, 127.8, 127.8, 125.3, 123.6, 122.8, 121.1, 119.4, 66.7, 44.7

3-(2-(4-bromophenyl)allyl)-3-(4-chlorophenyl)isoindolin-1-one (3f)



The title compound was prepared according to general procedure C, from **1a** (50 mg, 0.191 mmol) using $Ca(NTf_2)_2$ (1 mg, 0.002 mmol), $n$$Bu_4PF_6$ (0.7 mg, 0.002 mmol) and (2-(4-bromophenyl)allyl)trimethylsilane[12] (77 mg, 0.287 mmol) in DCE (1 mL). Following conversion to the product and column chromatography (25% EtOAc:cyclohexane) afforded **3f** as a colourless oil which solidified upon standing (74 mg, 89%)

RF (1:1 EtOAc/CycHex) = 0.67

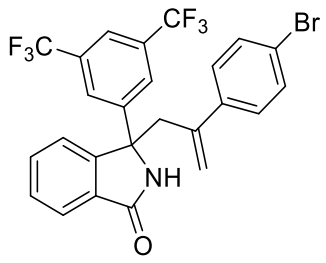
IR υ_{max} (cm^{-1}) 3076, 2922, 2342, 1680, 1456, 1227, 953

HR-ESI-MS: $C_{23}H_{18}BrClNO$ $[M+H]^+$ *m/z* found 438.0261 Cald 438.0260. *Bromine isotope pattern*

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (1 H, d, *J* = 7.3 Hz), 7.28 - 7.35 (3 H, m), 7.17 - 7.26 (3 H, m), 7.12 - 7.16 (2 H, m), 6.87 (1 H, d, *J* = 7.3 Hz), 6.74 - 6.78 (2 H, m), 4.98 (1 H, d, *J* = 0.9 Hz), 4.79 (1 H, s), 3.71 (1 H, d, *J* = 13.3 Hz), 3.54 (1 H, d, *J* = 13.3 Hz)

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.7, 148.5, 142.2, 141.1, 138.4, 134.1, 131.6, 131.2, 130.9, 129.2, 128.1, 127.8, 127.7, 123.1, 122.6, 120.9, 119.4, 69.8, 39.5.

3-(3,5-bis(trifluoromethyl)phenyl)-3-(2-(4-bromophenyl)allyl)isoindolin-1-one (3g)



The title compound was prepared according to general procedure C, from **1a** (69 mg, 0.191 mmol) using $Ca(NTf_2)_2$ (1 mg, 0.002 mmol), $n$$Bu_4PF_6$ (0.7 mg, 0.002 mmol) and (2-(4-bromophenyl)allyl)trimethylsilane[12] (77 mg, 0.287 mmol) in DCE (1 mL). Following conversion to the product and column chromatography (25% EtOAc:cyclohexane) afforded **3g** as a pale yellow oil which solidified upon standing (83 mg, 80%)

RF (1:1 EtOAc/CycHex) = 0.71

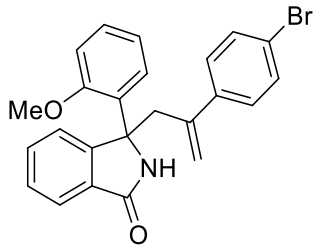
IR υ_{max} (cm^{-1}) 3255, 2970, 2901, 2342, 1663, 1445, 1034

HR-ESI-MS: $C_{25}H_{17}BrF_6NO$ $[M+H]^+$ *m/z* found 540.0389 Cald 540.0398. *Bromine isotope pattern*

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (1 H, s), 7.94 (2 H, s), 7.74 - 7.80 (2 H, m), 7.41 - 7.47 (2 H, m), 7.28 - 7.33 (1 H, m), 7.18 - 7.23 (2 H, m), 6.82 - 6.89 (2 H, m), 5.18 (1 H, s), 5.04 (1 H, s), 3.63 (1 H, d, *J* = 13.7 Hz), 3.48 (1 H, d, *J* = 14.2 Hz)

^{13}C NMR (101 MHz, $CDCl_3$) δ 170.4, 148.7, 144.4, 142.1, 140.5, 132.5, 132.0 (q, J_{C-F} = 33.4 Hz), 131.3, 130.6, 129.0, 127.8, 125.9, 124.1, 122.9 (d, J_{C-F} = 260 Hz), 122.5, 121.8 (quin, J_{C-F} = 3.8 Hz), 121.4, 120.3, 66.4, 45.5

3-(2-(4-bromophenyl)allyl)-3-(2-methoxyphenyl)isoindolin-1-one (3h)



The title compound was prepared according to general procedure C, from **1a** (49 mg, 0.191 mmol) using $Ca(NTf_2)_2$ (1 mg, 0.002 mmol), $n$$Bu_4PF_6$ (0.7 mg, 0.002 mmol) and (2-(4-bromophenyl)allyl)trimethylsilane[12] (77 mg, 0.287 mmol) in DCE (1 mL). Following conversion to the product and column chromatography (25% EtOAc:cyclohexane) afforded **3g** as a pale yellow oil which solidified upon standing (71 mg, 86%)

RF (1:1 EtOAc/CycHex) = 0.31

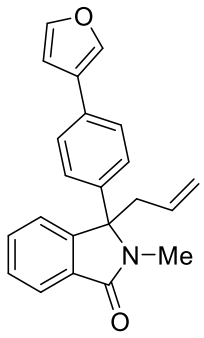
IR υ_{max} (cm^{-1}) 3180, 2971, 1699, 1315, 1034, 908

HR-ESI-MS: $C_{24}H_{21}BrNO_2$ $[M+H]^+$ *m/z* found 434.0751 Cald 434.0756. *Bromine isotope pattern*

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (1 H, d, *J* = 6.4 Hz), 7.49 - 7.53 (1 H, m), 7.43 (2 H, dtd, *J* = 19.2, 7.2, 7.2, 1.1 Hz), 7.22 - 7.28 (3 H, m), 7.20 (1 H, dd, *J* = 7.8, 1.8 Hz), 7.12 (1 H, s), 6.92 (1 H, dd, *J* = 8.2, 0.9 Hz), 6.84 - 6.88 (2 H, m), 6.80 (1 H, td, *J* = 7.6, 0.9 Hz), 4.99 (1 H, d, *J* = 0.9 Hz), 4.73 (1 H, s), 3.93 (3 H, s), 3.78 (1 H, d, *J* = 13.7 Hz), 3.23 (1 H, d, *J* = 13.3 Hz)

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.7, 156.9, 147.0, 142.9, 141.5, 132.6, 130.9, 130.7, 129.4, 128.3, 127.8, 126.9, 125.3, 123.9, 120.9, 120.6, 118.8, 111.4, 66.8, 55.2, 43.7

3-allyl-3-(4-(furan-3-yl)phenyl)-2-methylisoindolin-1-one (5)



To a 22 mL vial equipped with a magnetic stirrer was added **2o** (50mg, 0.168 mmol) and SPhos Pd G3 (7mg, 0.008 mmol) and 3-Furanylboronic acid (28mg, 0.252 mmol) and flask purged with argon. Anhydrous and degassed 1,4-dioxane (1mL) and aq. K_3P0_4 (1.6mL, 0.797 mmol, 0.5M, degassed) was added. The vial was capped with a Teflon cap and stirred at 100 °C for 16 hours. The mixture was passed through a plug of celite, washed with Et2o, and the organic washing concentrated in vacuo. The residue was purified by flash column chromatography (10% EtOAc in hex) to afford the desired compound as a pale yellow solid (48mg, 87%)

RF (20% EtOAc/CycHex) = 0.29

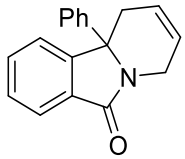
IR υ_{max} (cm^{-1}) 3683, 3659, 2987, 2921, 2360, 1739, 1668, 1410

HR-ESI-MS: $C_{22}H_{19}NO_2$ $[M+H]^+$ *m/z* found 329.1411 Cald 329.1416

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (1 H, dd, *J* = 6.6, 1.6 Hz), 7.72 (1 H, s), 7.40 - 7.49 (5 H, m), 7.15 - 7.23 (3 H, m), 6.66 - 6.69 (1 H, m), 5.00 - 5.18 (2 H, m), 4.91 - 4.96 (1 H, m), 3.33 (1 H, dd, *J* = 14.0, 4.6 Hz), 3.13 (1 H, dd, *J* = 14.0, 6.9 Hz), 2.87 (3 H, s)

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.7, 149.4, 143.8, 138.7, 138.3, 132.2, 131.9, 131.6, 130.7, 128.1, 126.6, 126.3, 125.6, 123.4, 121.8, 119.6, 108.6, 69.6, 38.2, 25.0

10b-phenyl-1,10b-dihydropyrido[2,1-a]isoindol-6(4H)-one (6)



To a 22mL vial was added **2p** (92 mg, 0.346 mmol), $Ca(NTf_2)_2$ (2 mg, 0.003 mmol), $n$$Bu_4PF_6$ (1.3 mg, 0.003 mmol) followed by the addition of allyltrimethylsilane (43 mg, 0.381 mmol) in DCE (2 mL). The mixture was stirred at 80 °C for 1 hour, and TLC analysis indicated complete conversion to the desired product. The mixture was concentrated in vacuo to afford a pale yellow residue which was redissolved in anhydrous DCM (11.5 mL, 0.03M) followed by the addition of Grubb's 1st generation (3 mg, 0.003 mmol) and the resulting mixture stirred at 50 °C for 12 hours. The mixture was then concentrated to afford a dark brown residue which was purified by flash column chromatography (0-5% EtOAc in hexane) to afford the title compound as a colourless oil (70mg, 78%).

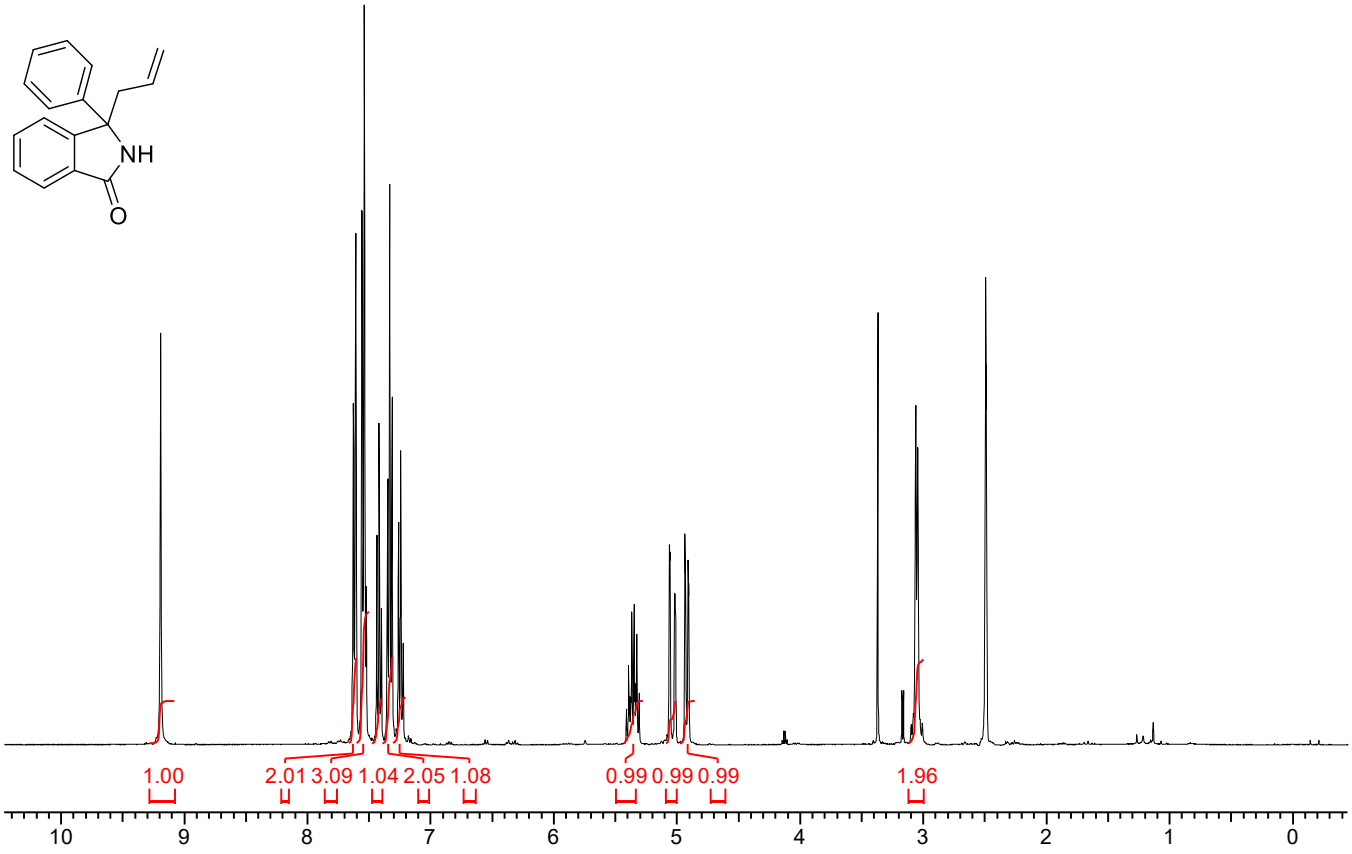
RF (5% EtOAc/CycHex) = 0.26

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 - 7.87 (1 H, m), 7.32 - 7.41 (2 H, m), 7.11 - 7.26 (6 H, m), 5.83 (1 H, ddq, *J* = 10.1, 6.2, 2.2, 2.2, 2.2 Hz), 5.57 - 5.69 (1 H, m), 4.60 - 4.72 (1 H, m), 3.29 - 3.49 (2 H, m), 2.21 - 2.34 (1 H, m)

^{13}C NMR (101 MHz, $CDCl_3$) δ 167.1, 151.9, 138.6, 131.8, 130.5, 128.8, 128.0, 127.8, 125.9, 124.4, 123.9, 122.4, 121.6, 64.5, 38.1, 33.0

Spectral data in accordance to previously published data[13]

5 – Copies of Spectra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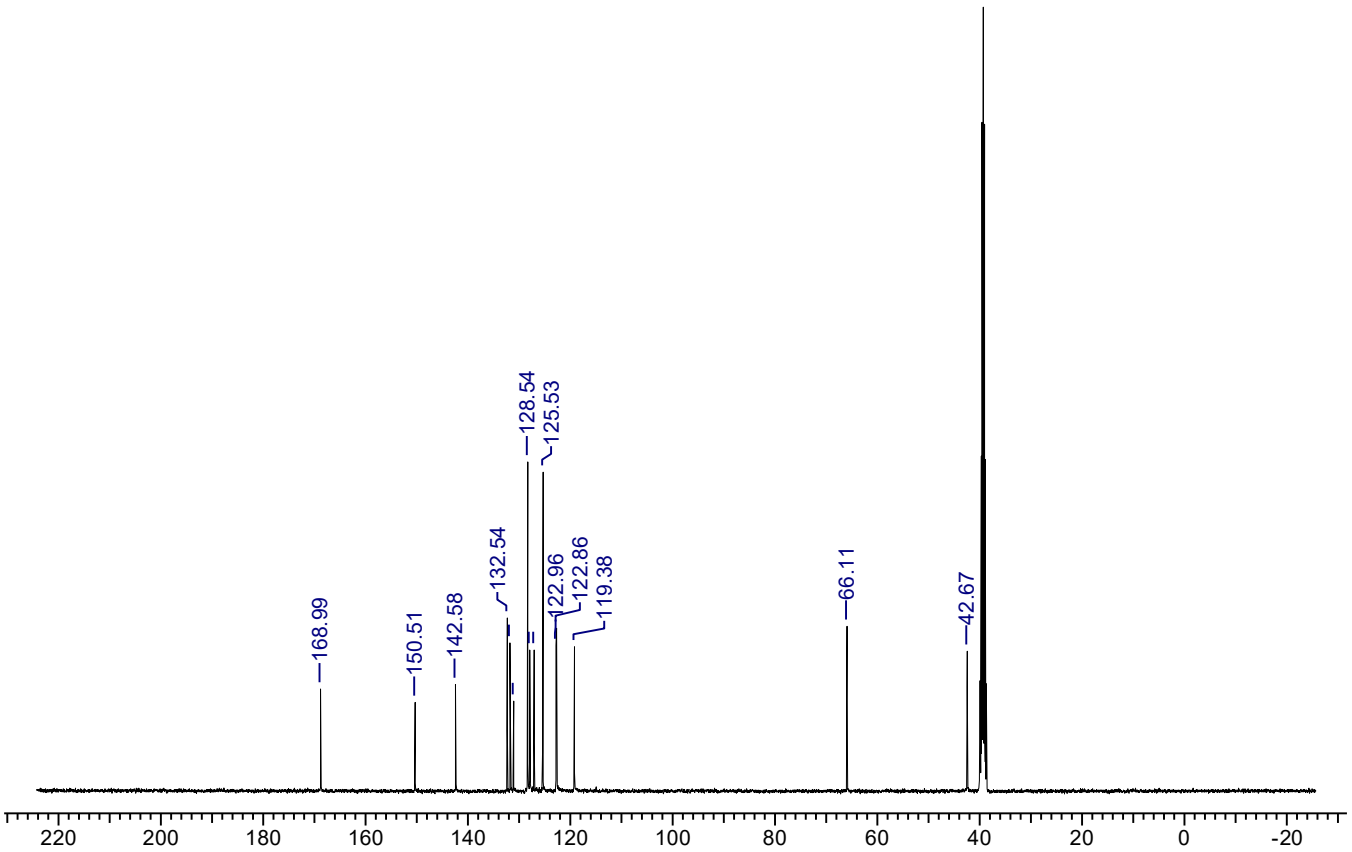
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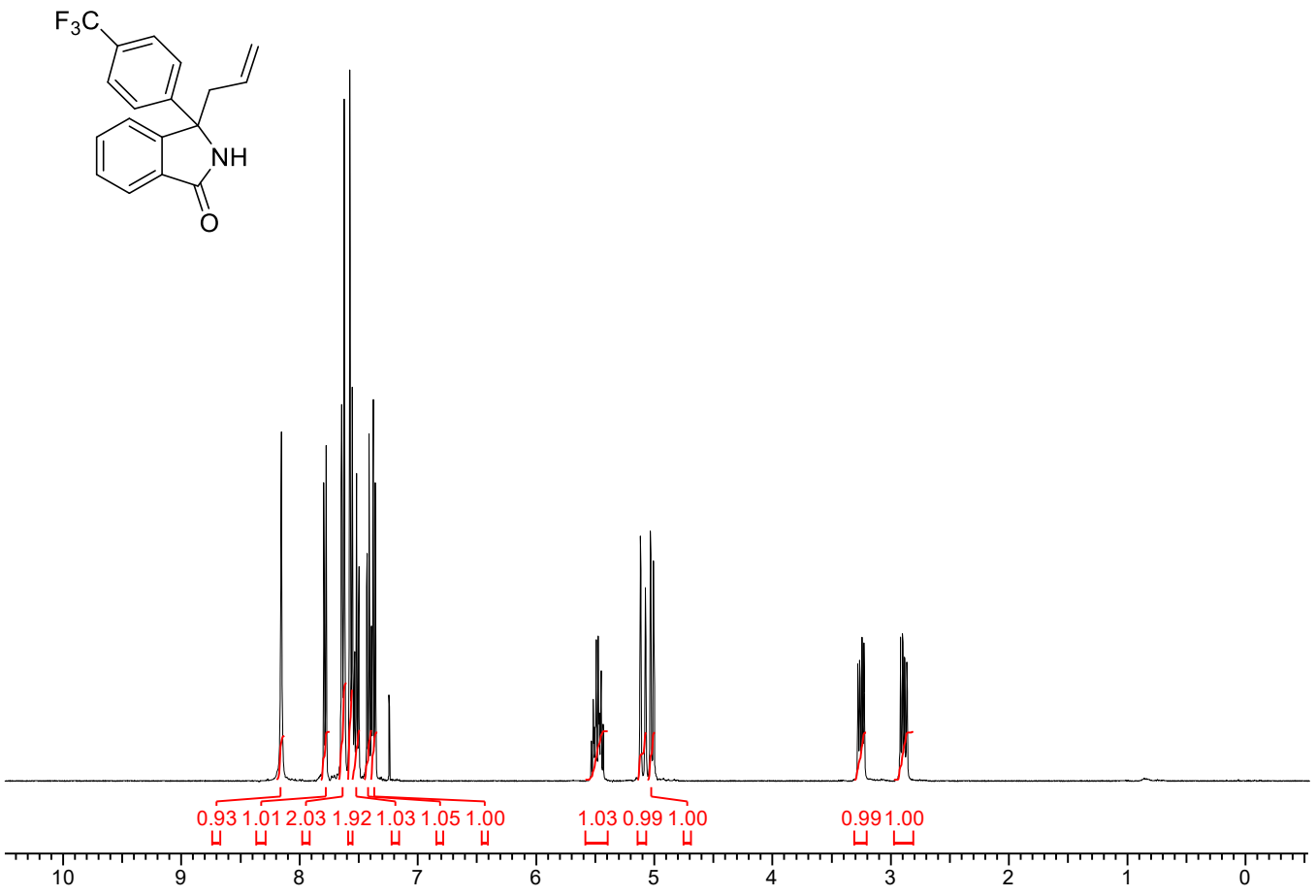
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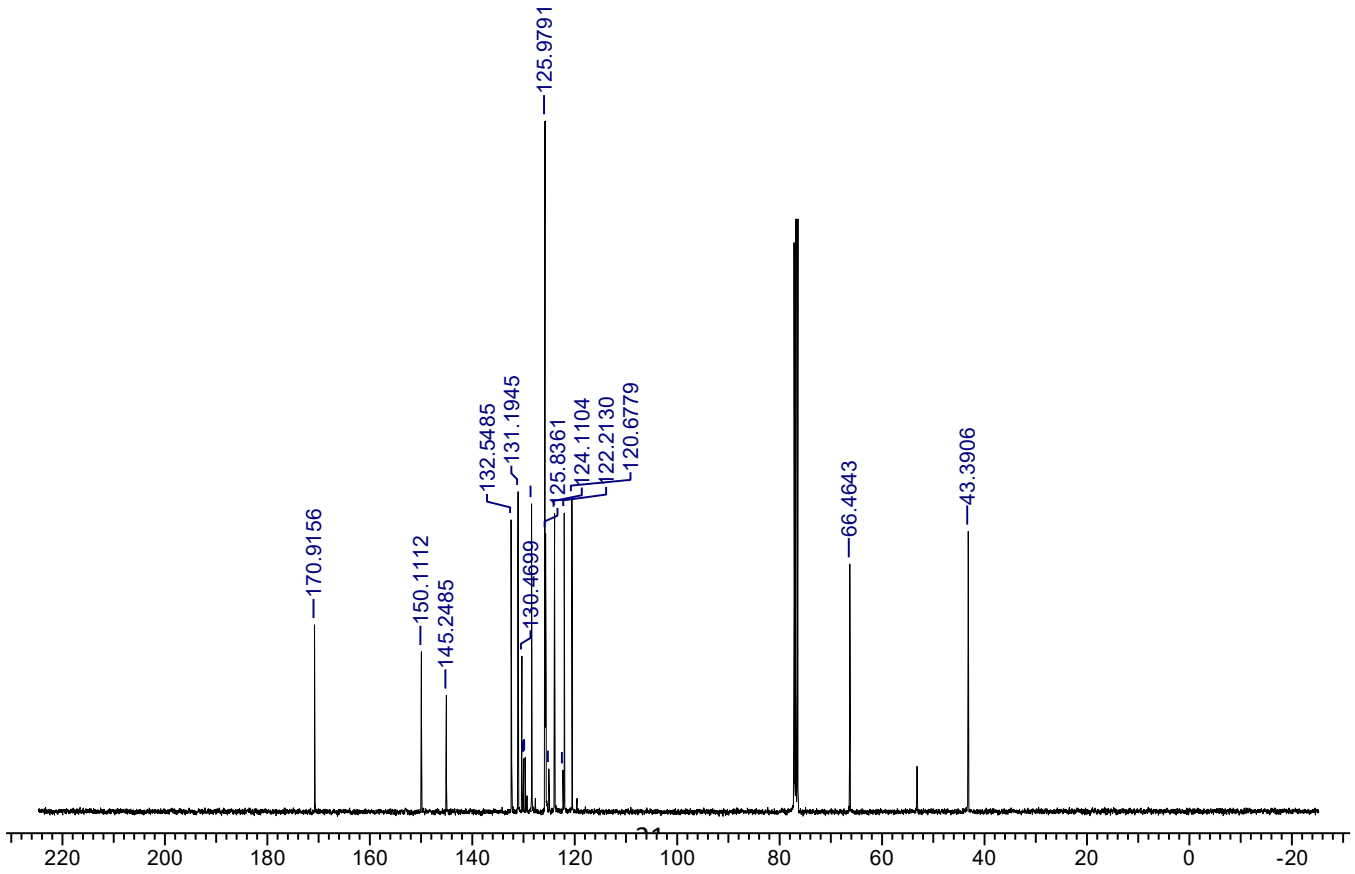
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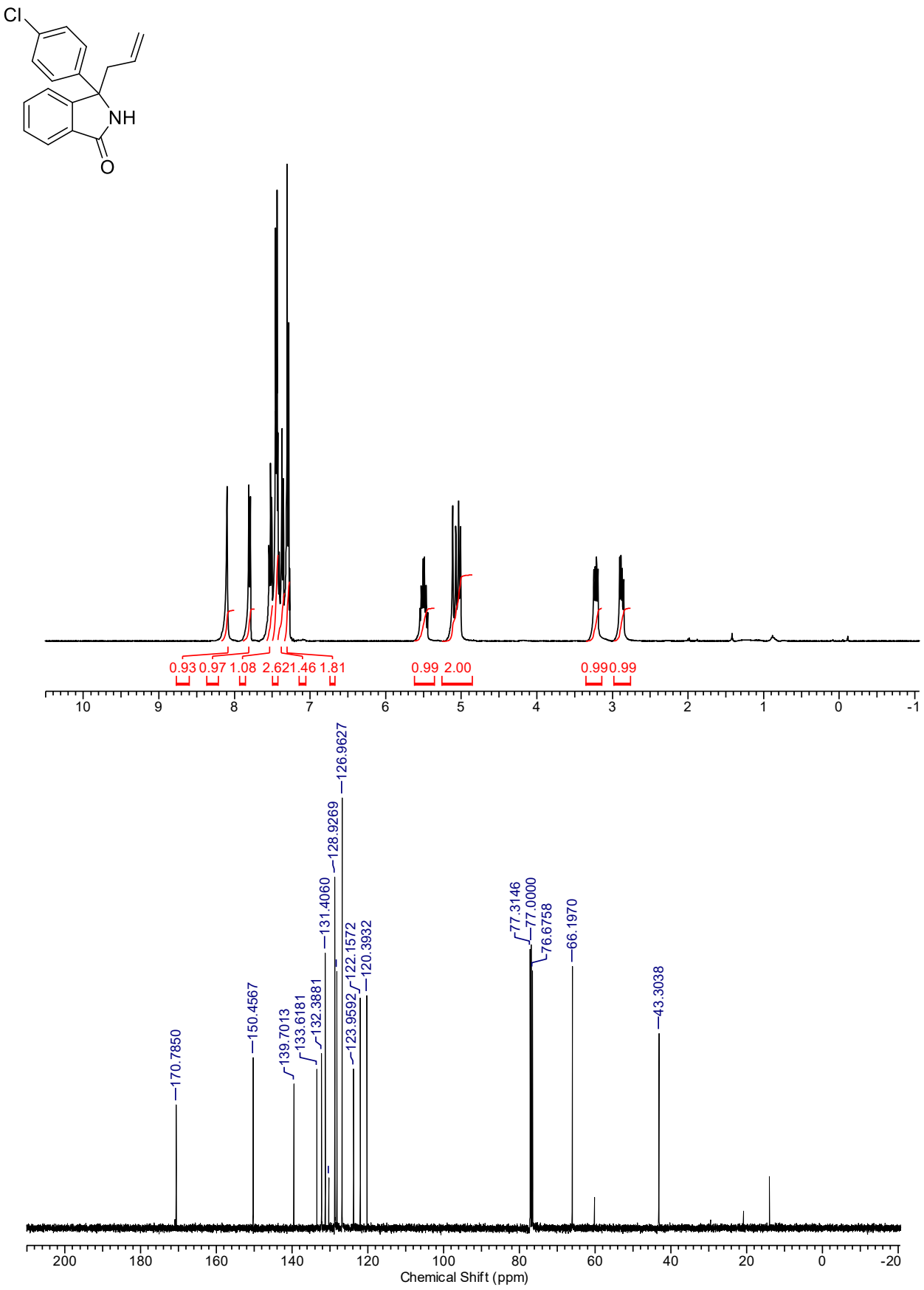
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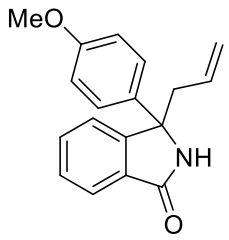
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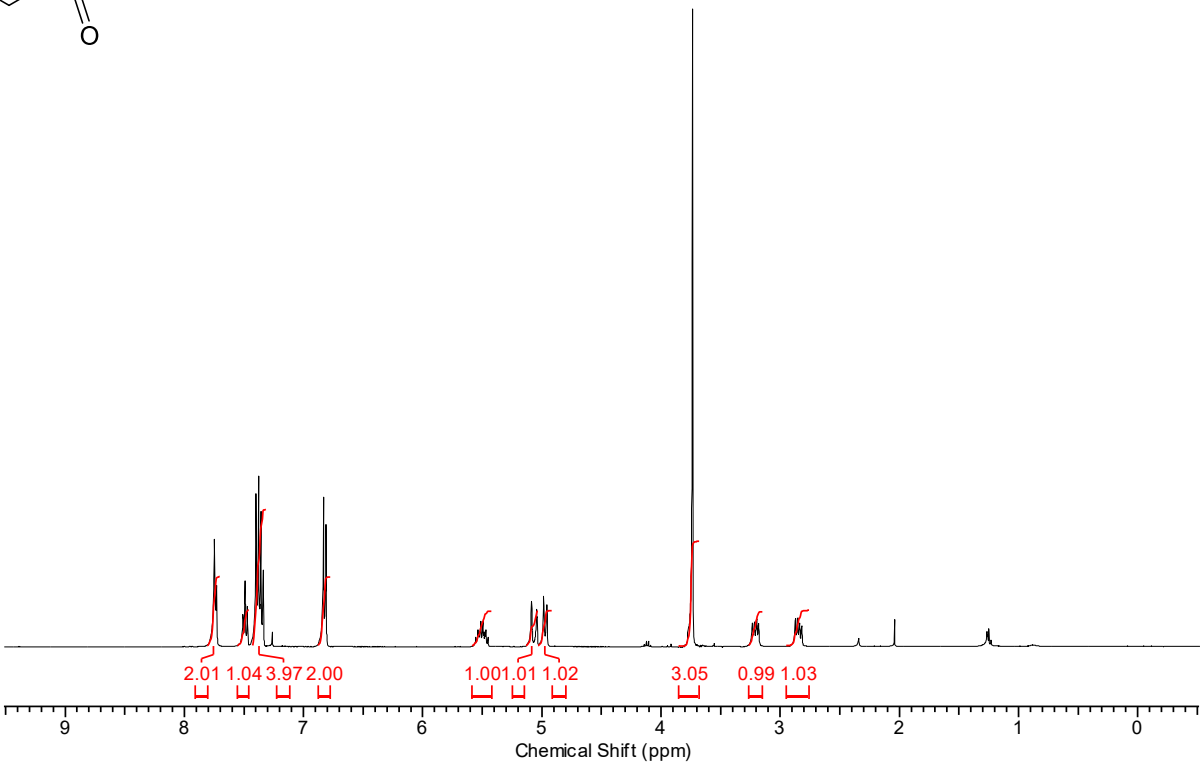
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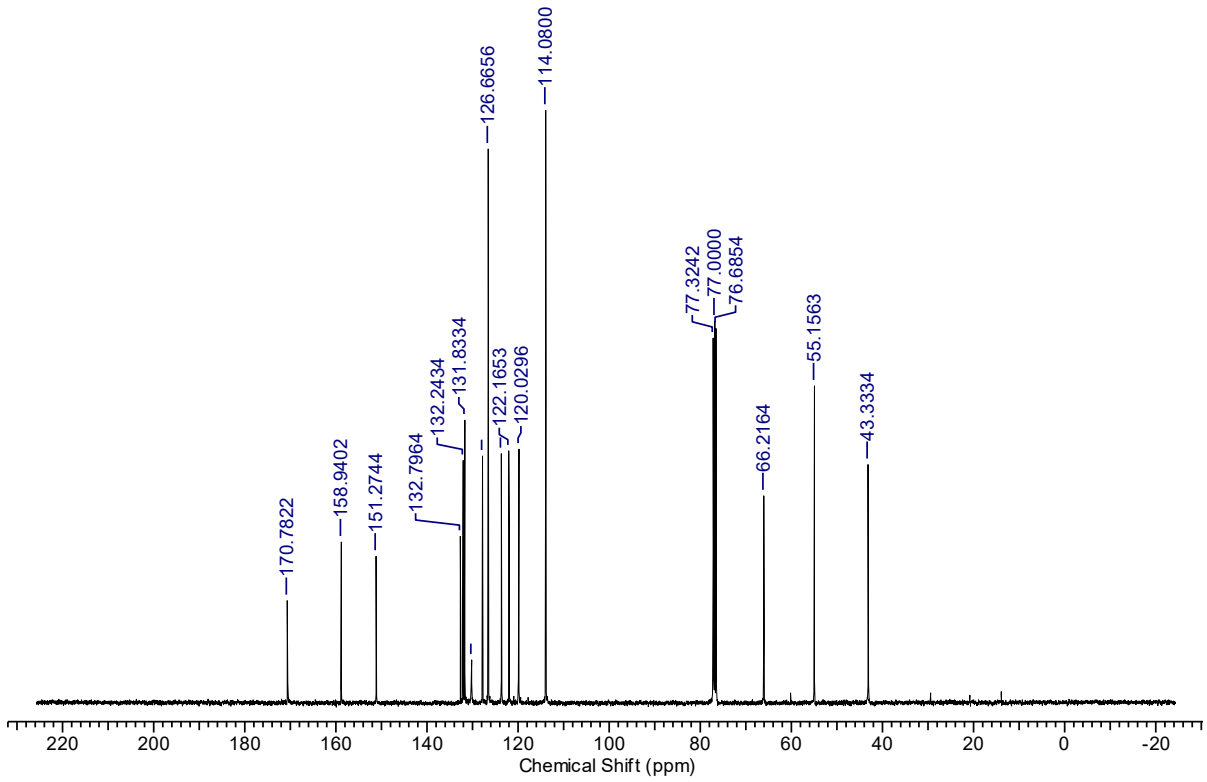
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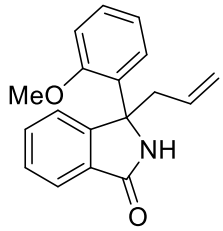
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NH
O

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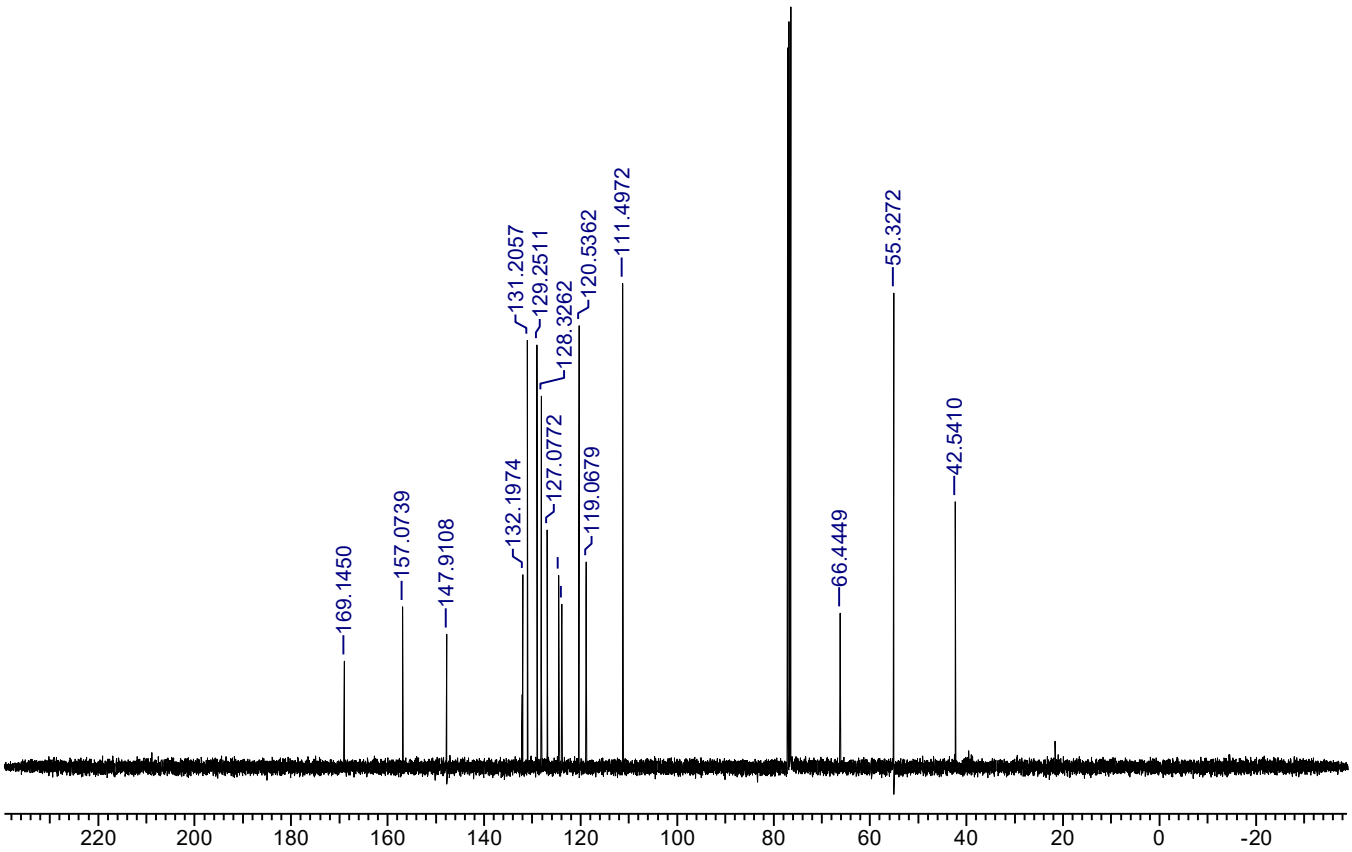
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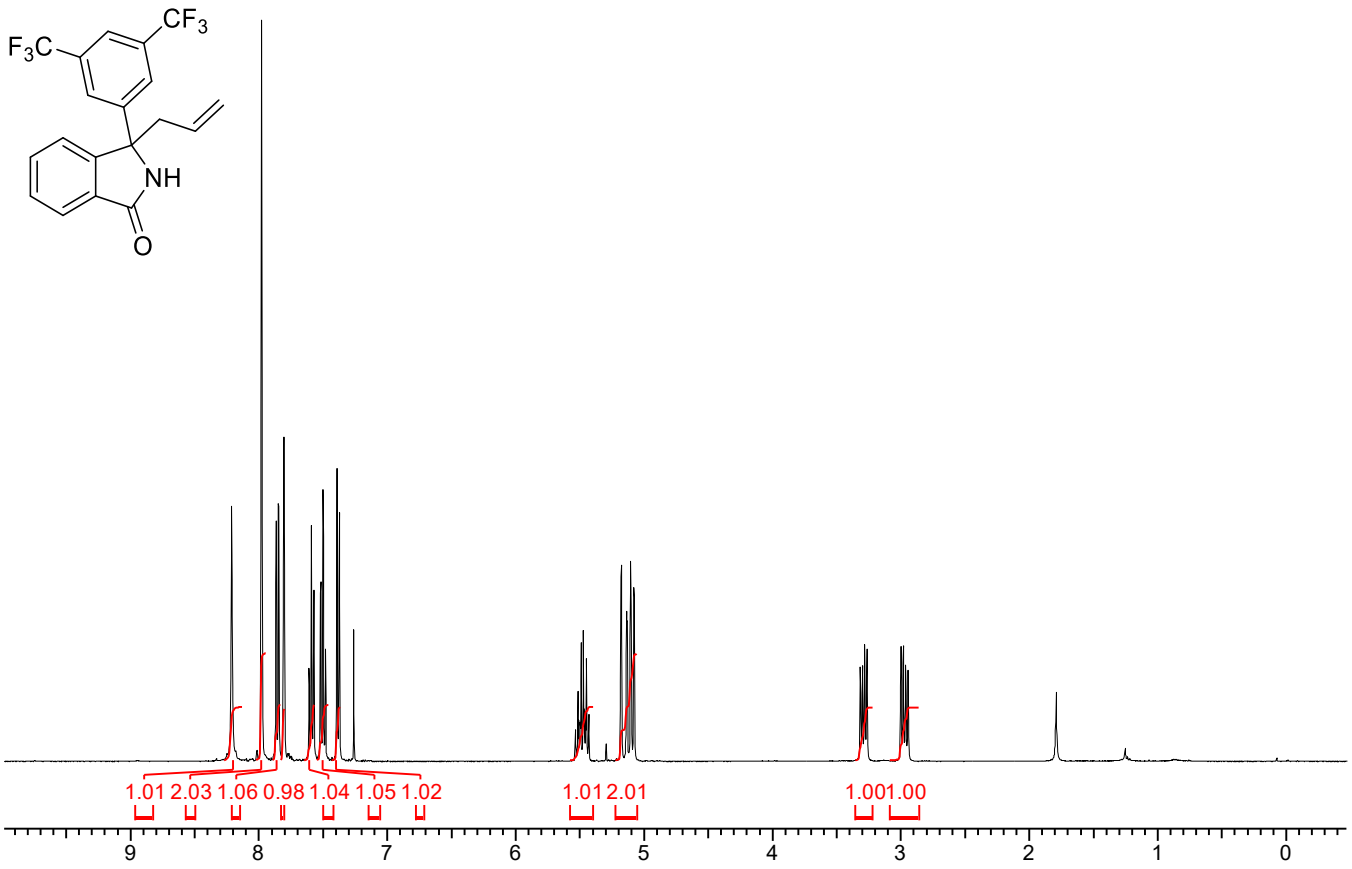
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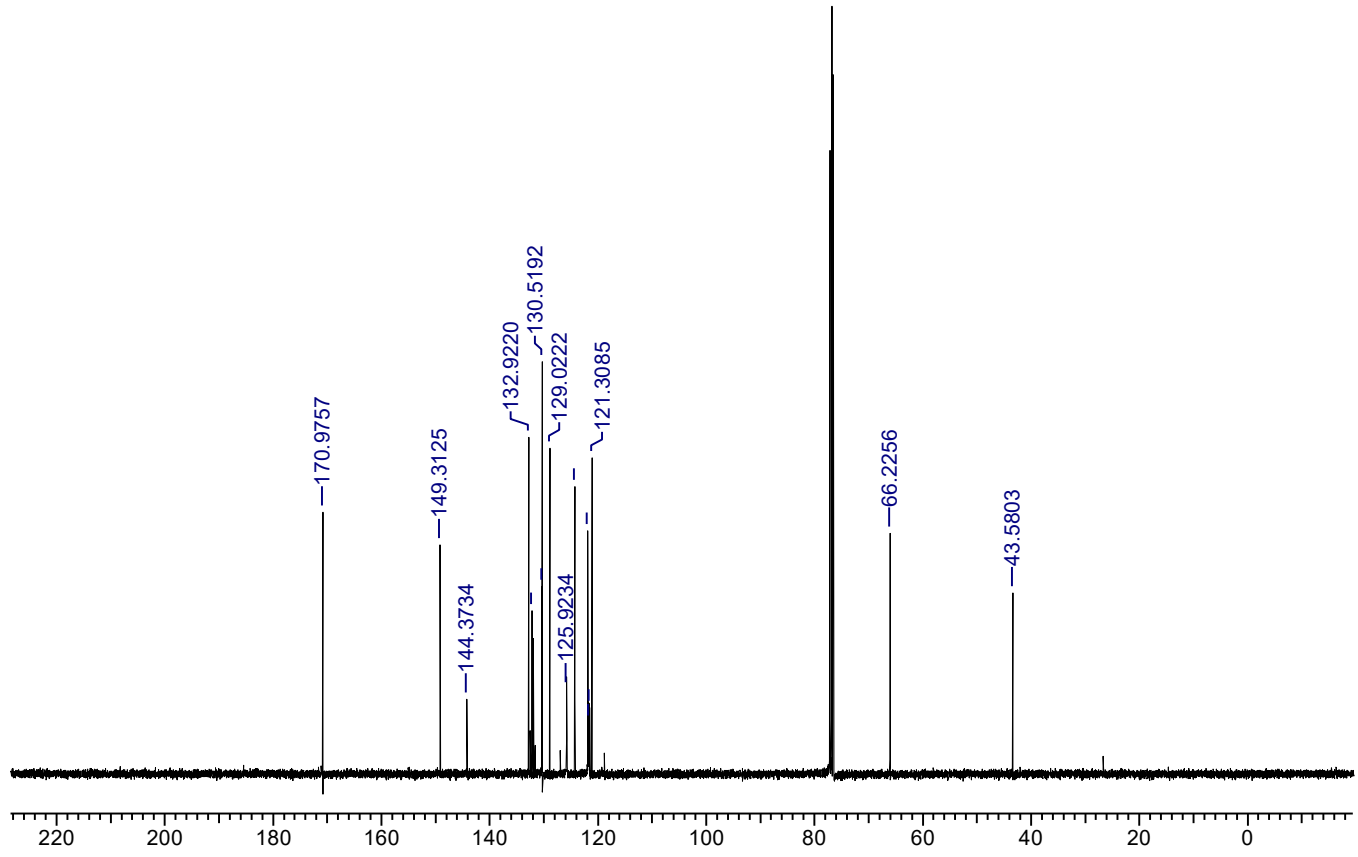
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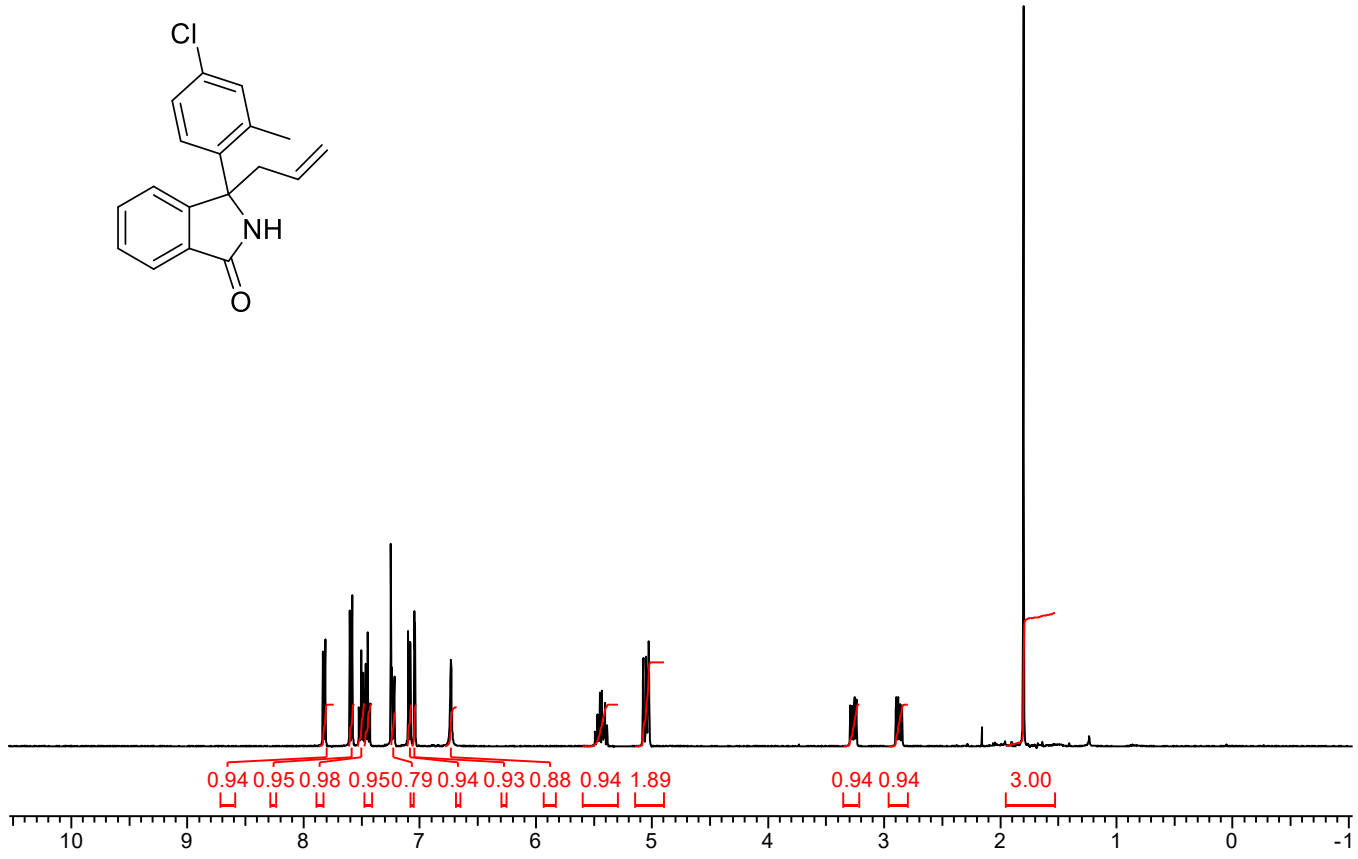
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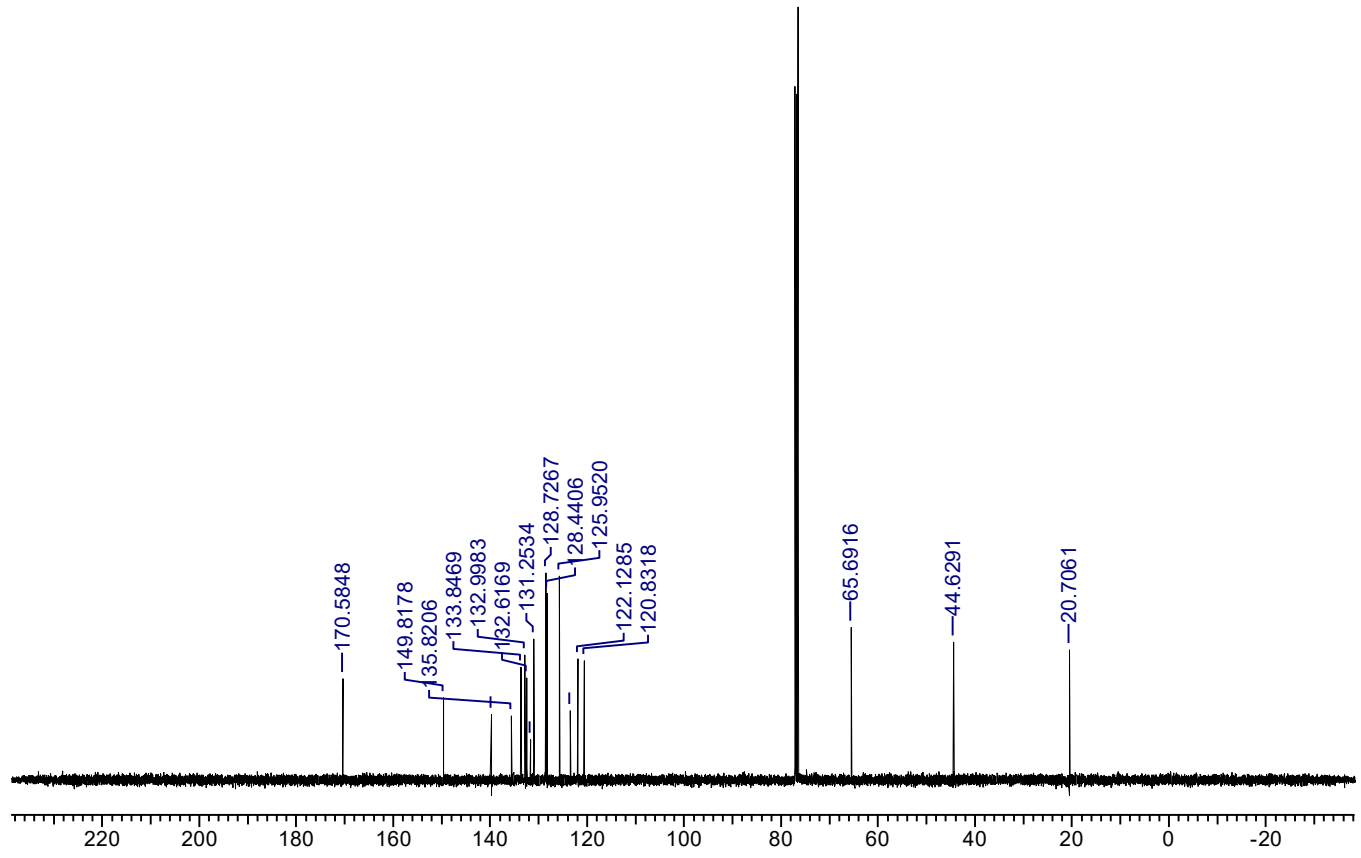
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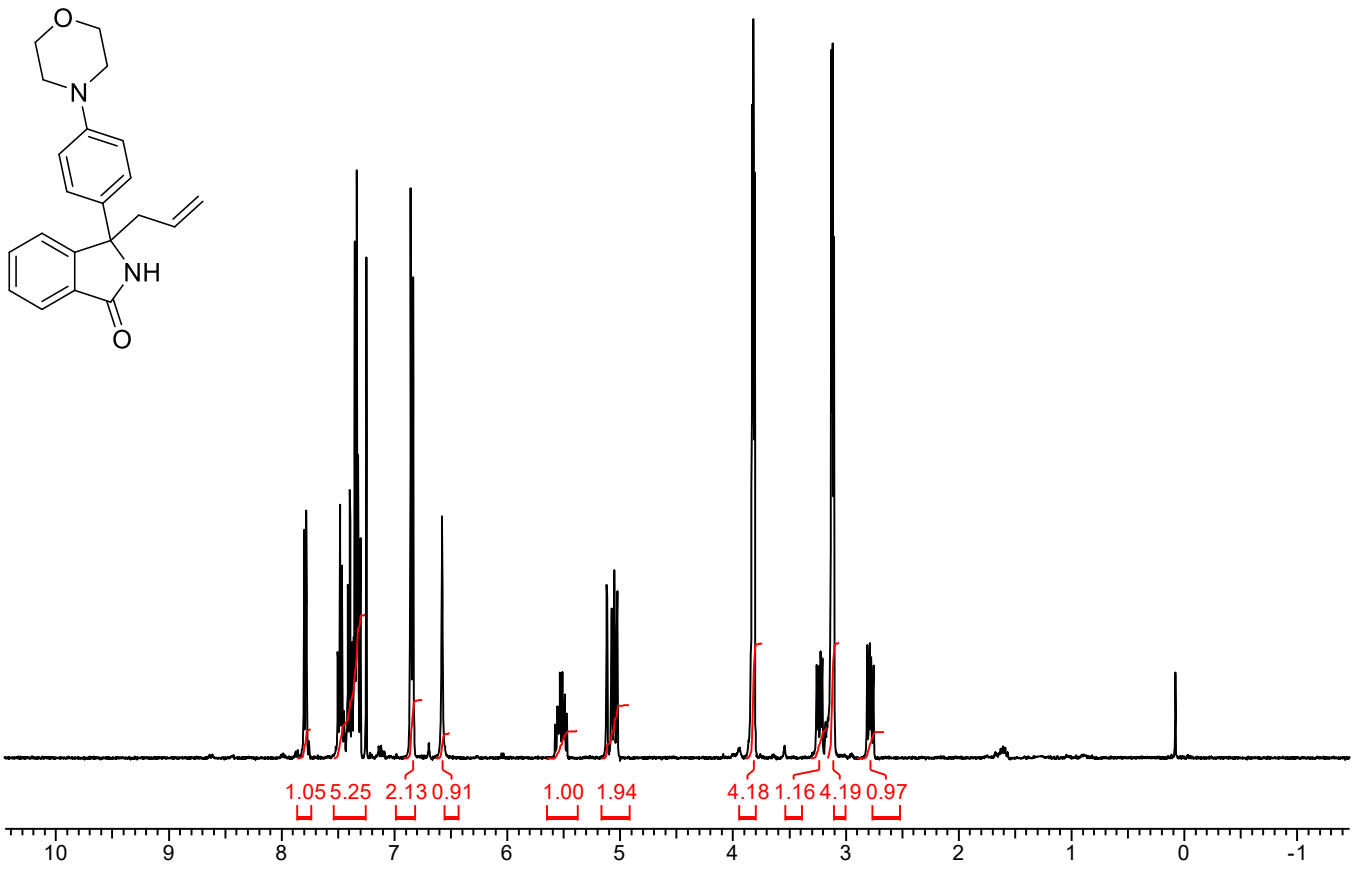
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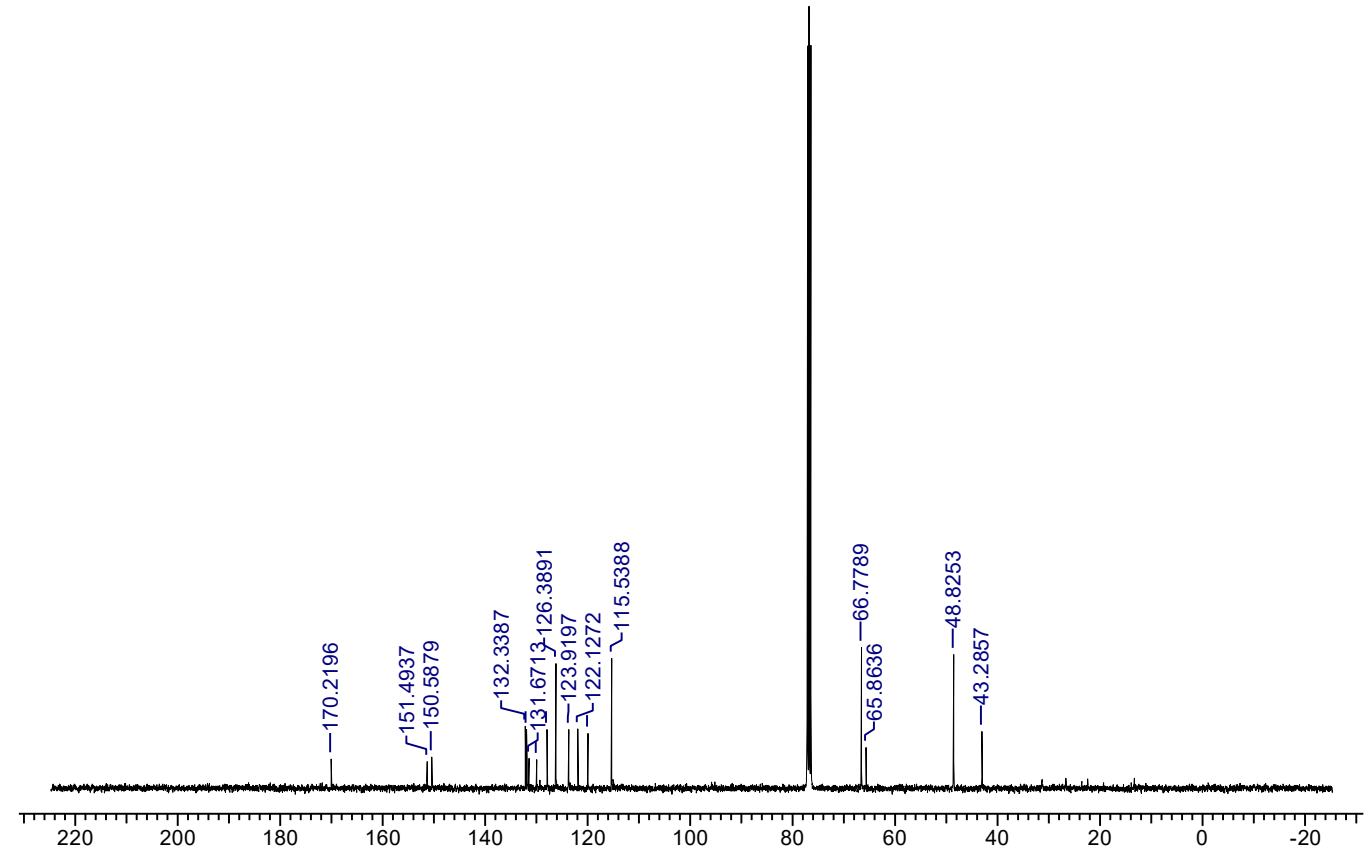
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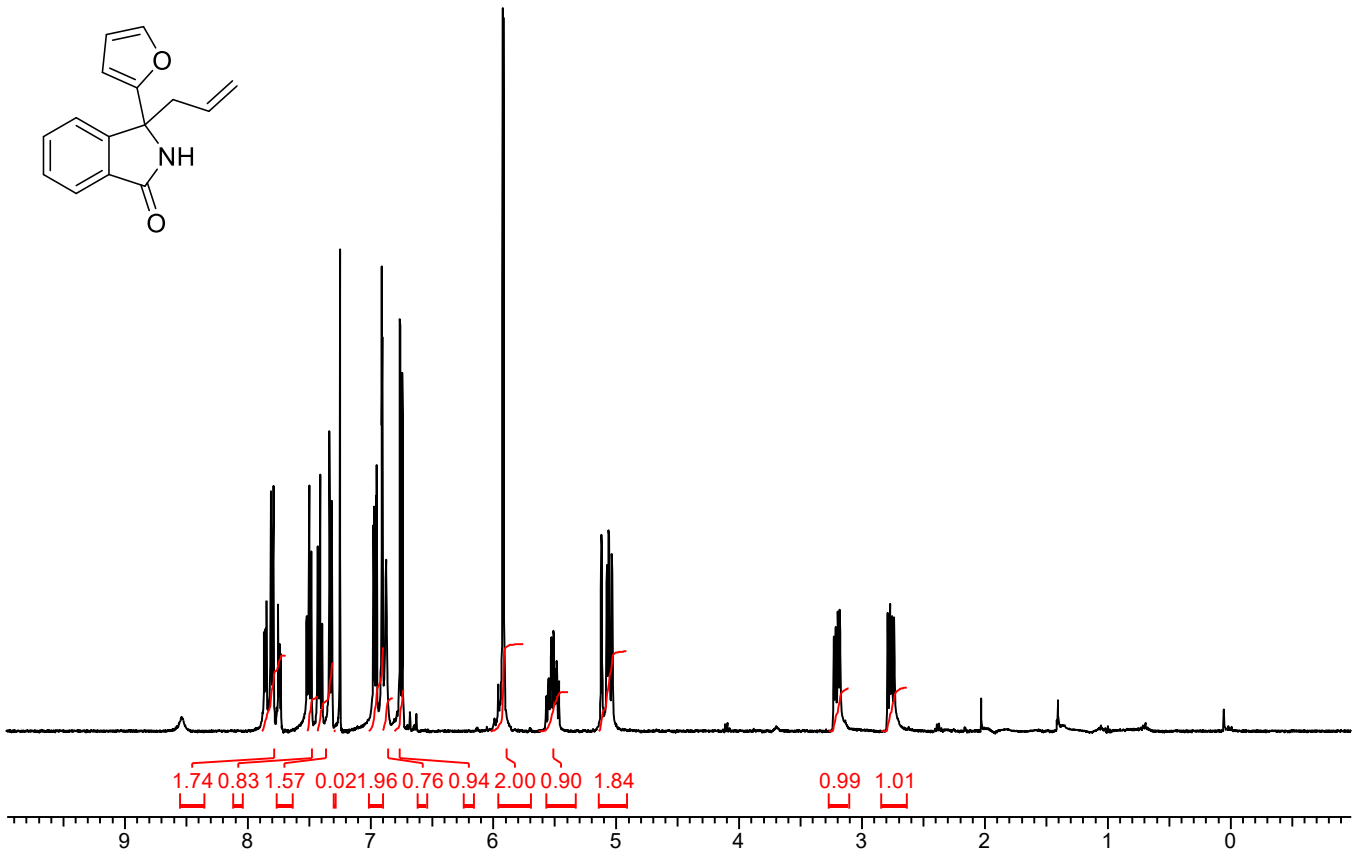
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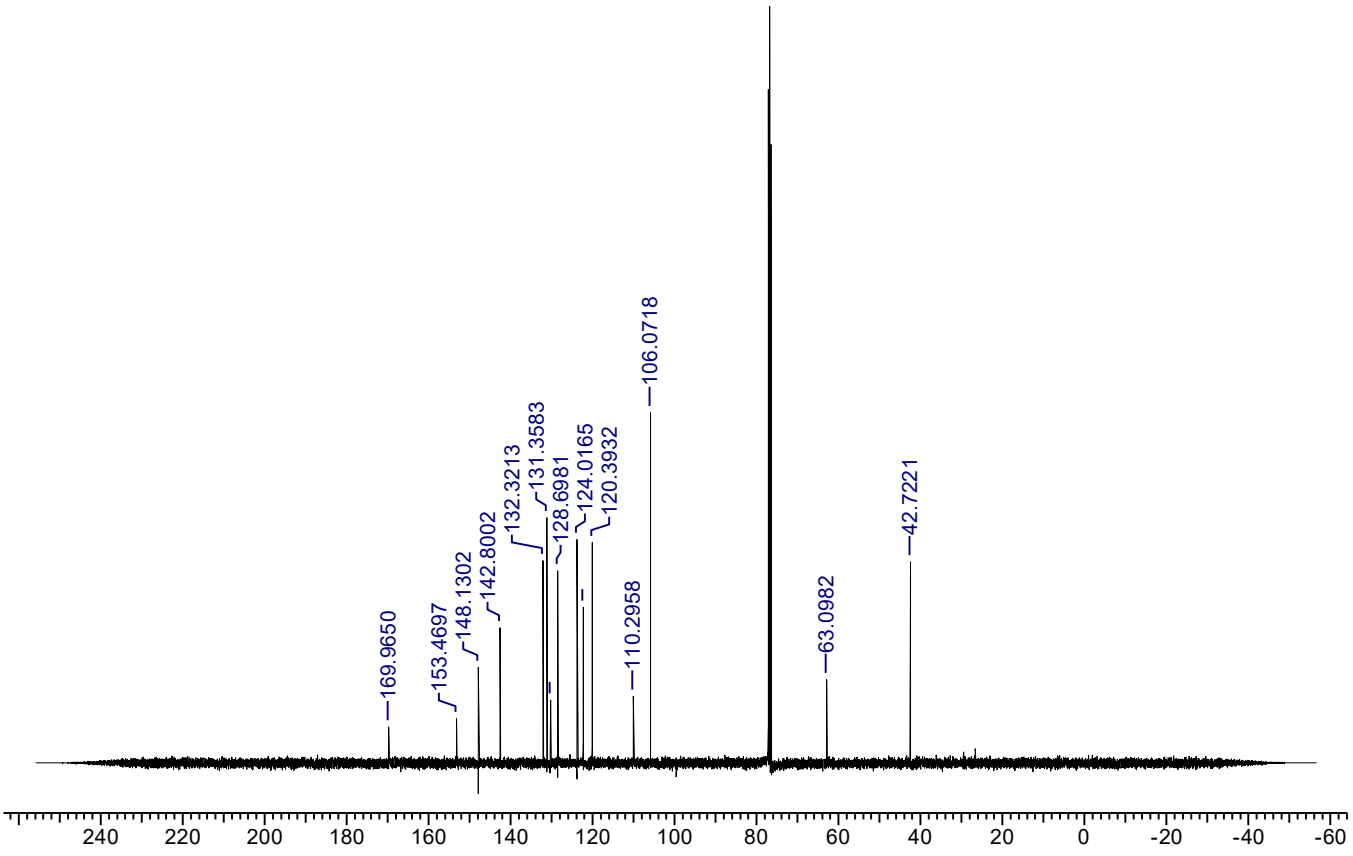
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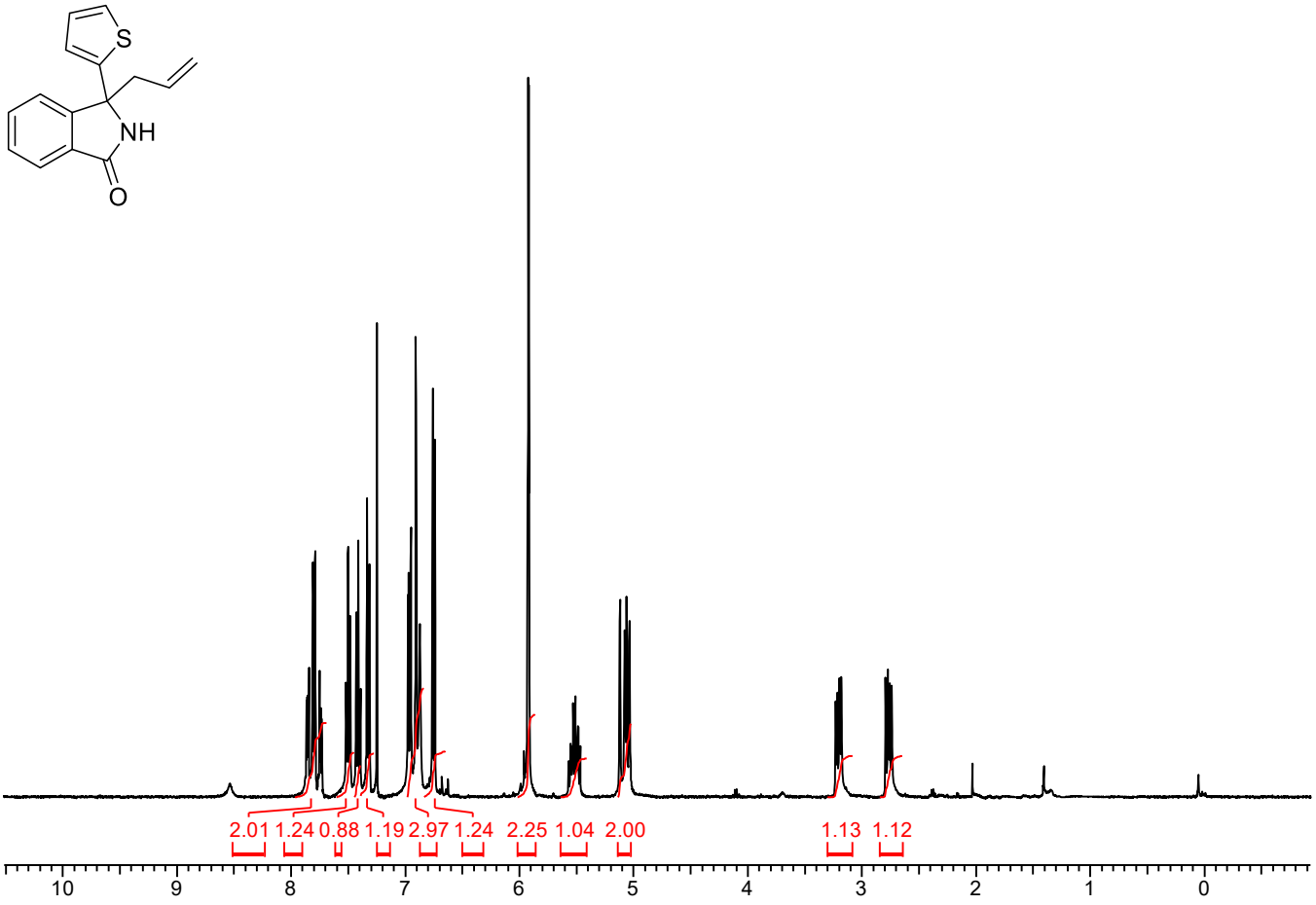
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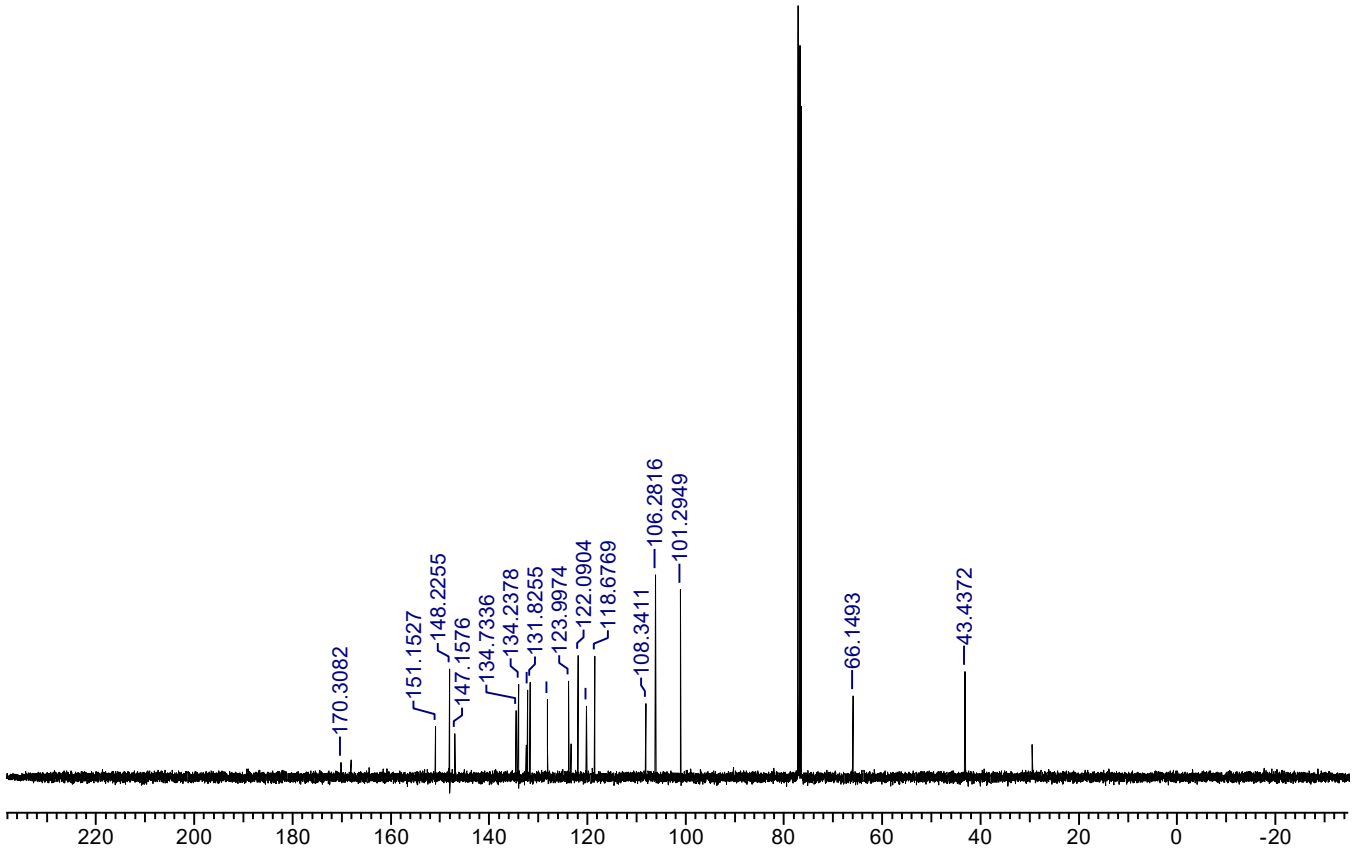
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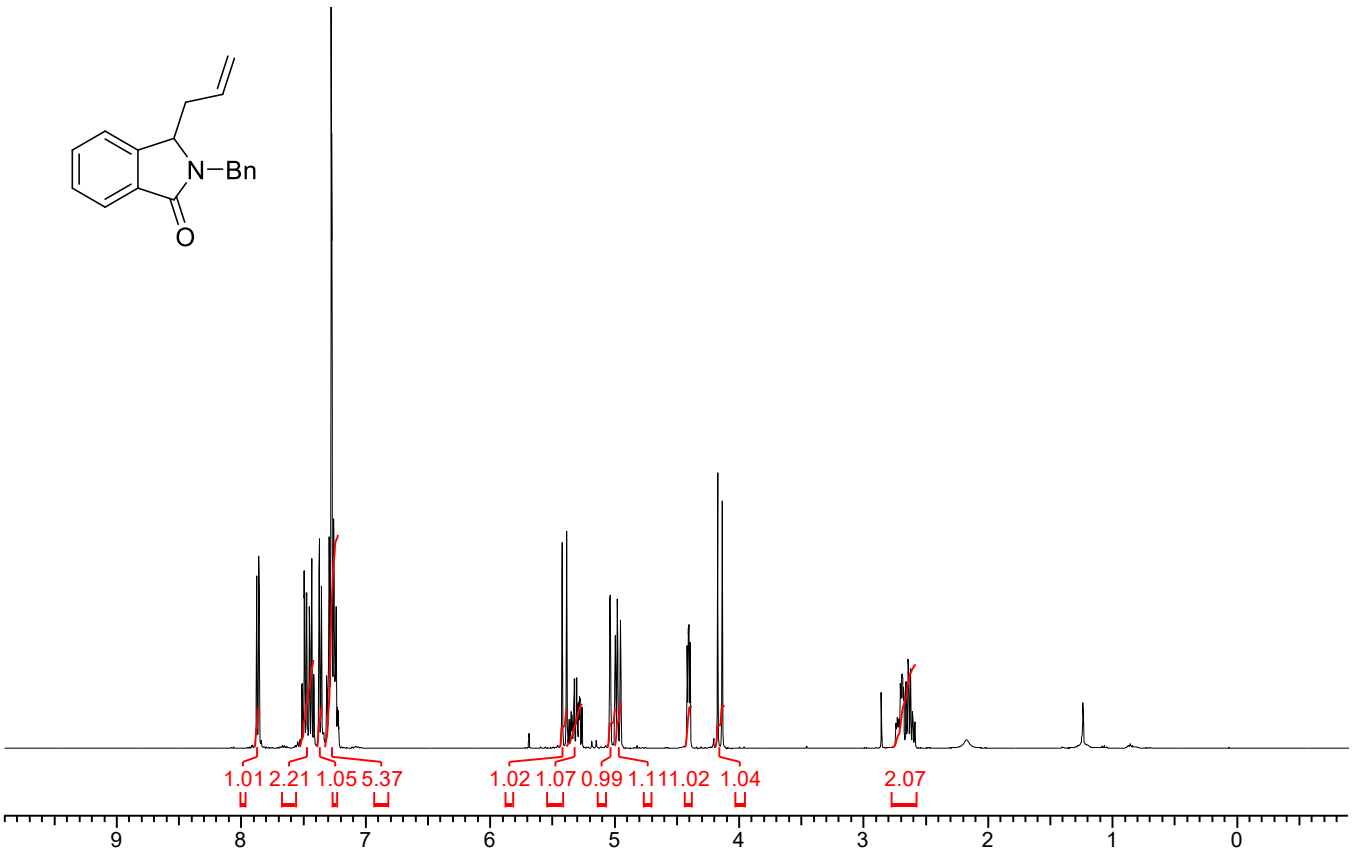
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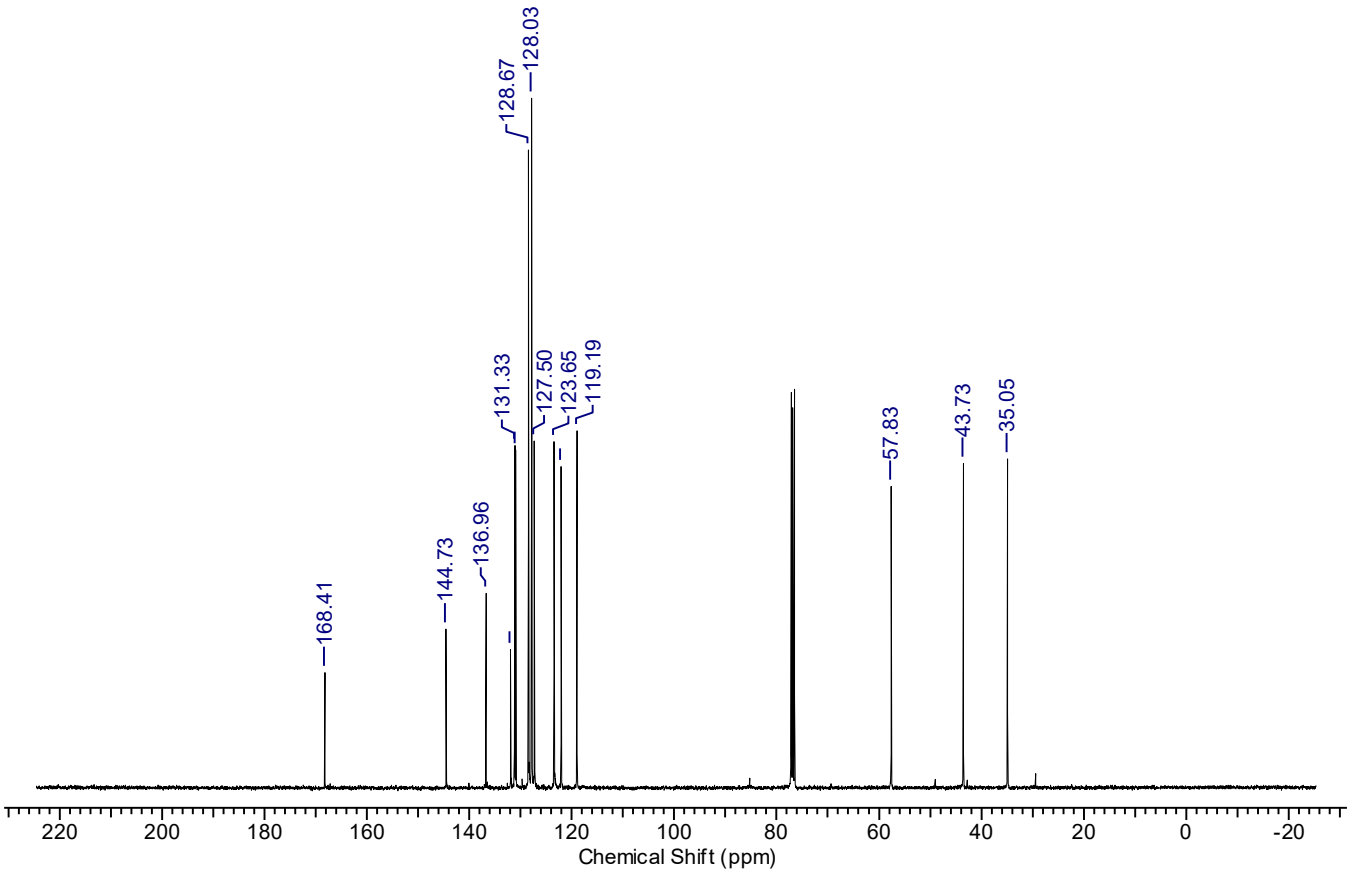
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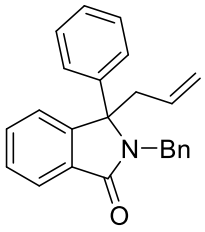
Chemical Shift (ppm)


N–Bn
O

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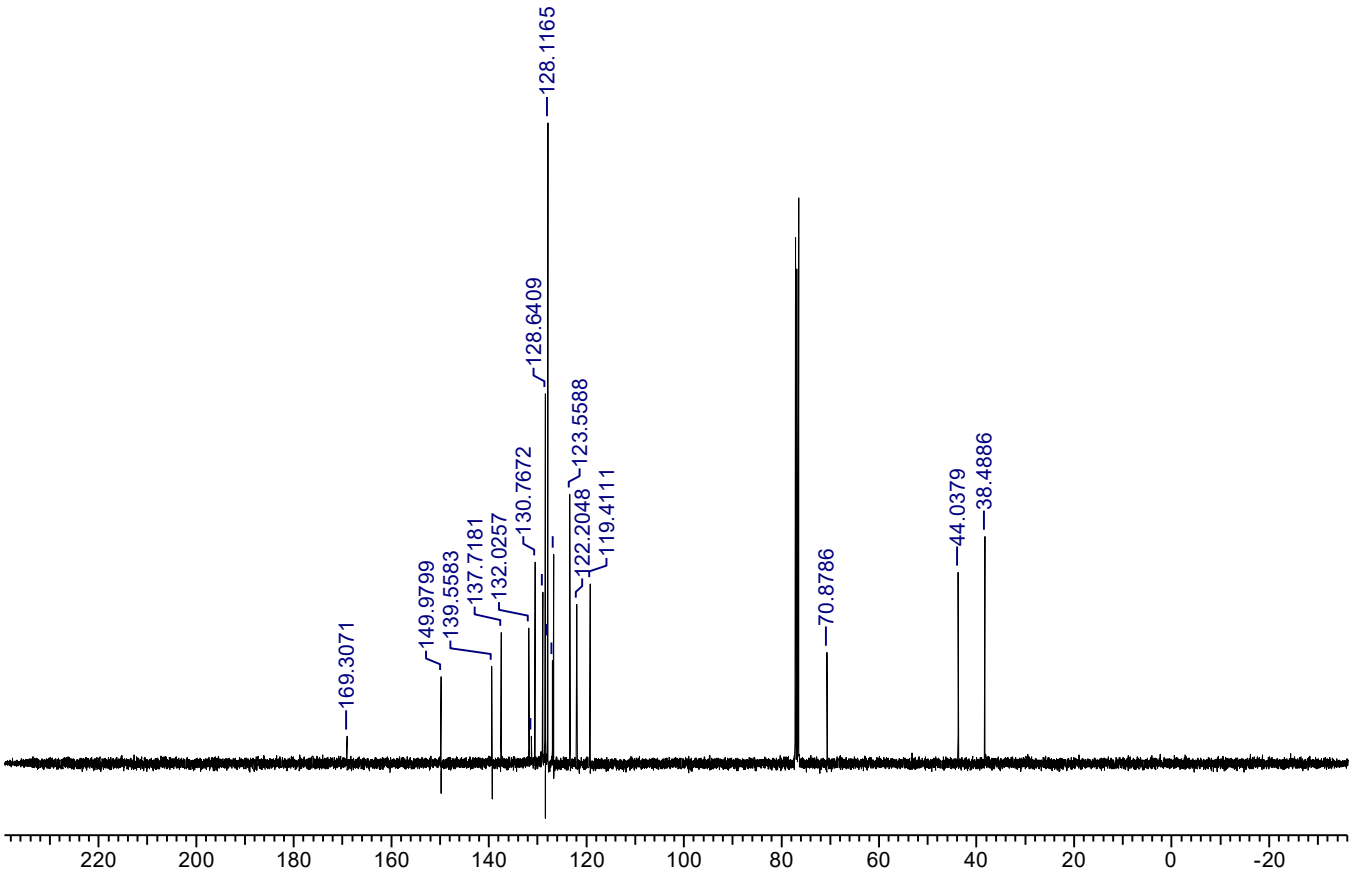
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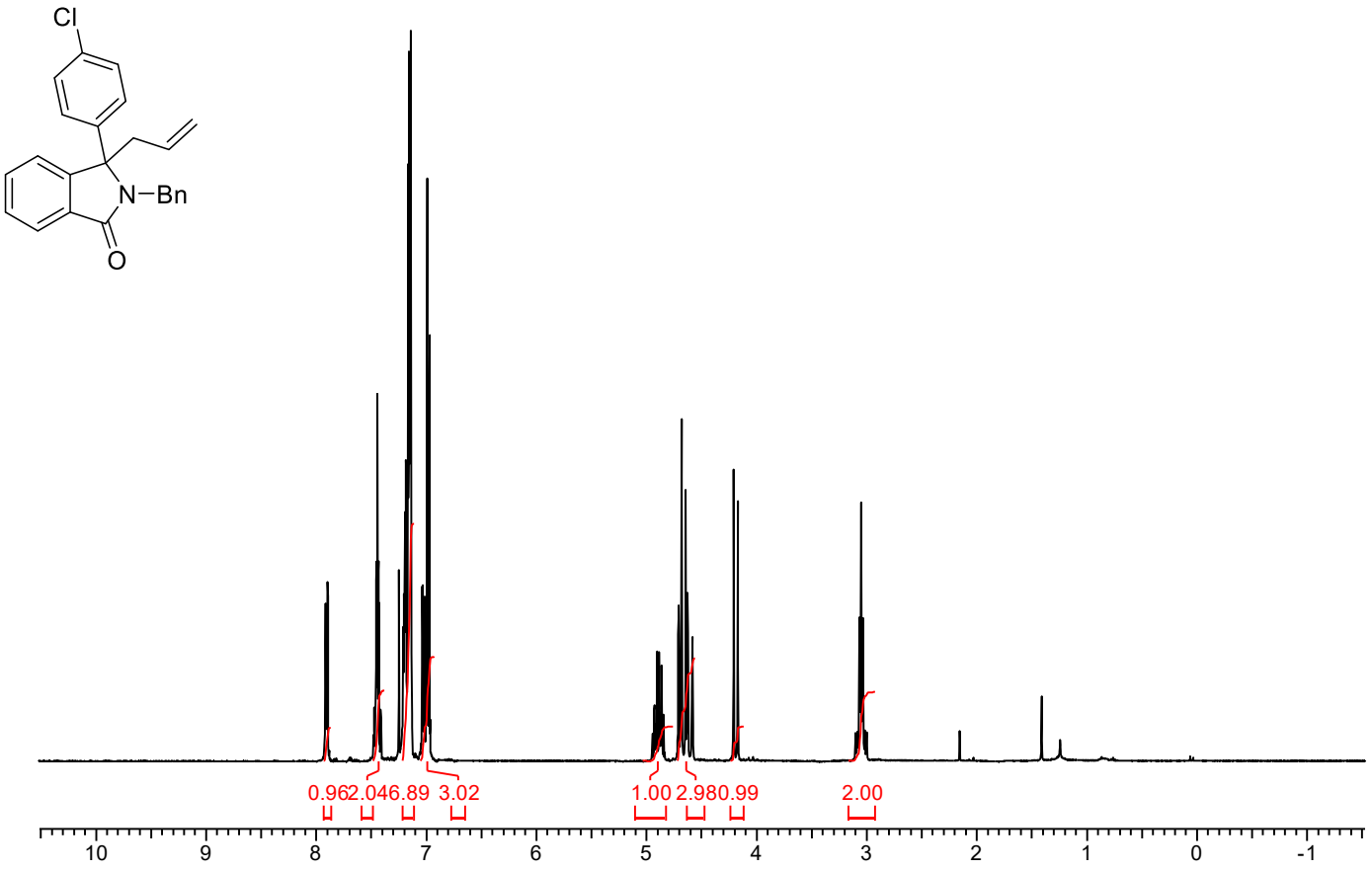
Cl
N—Bn
O
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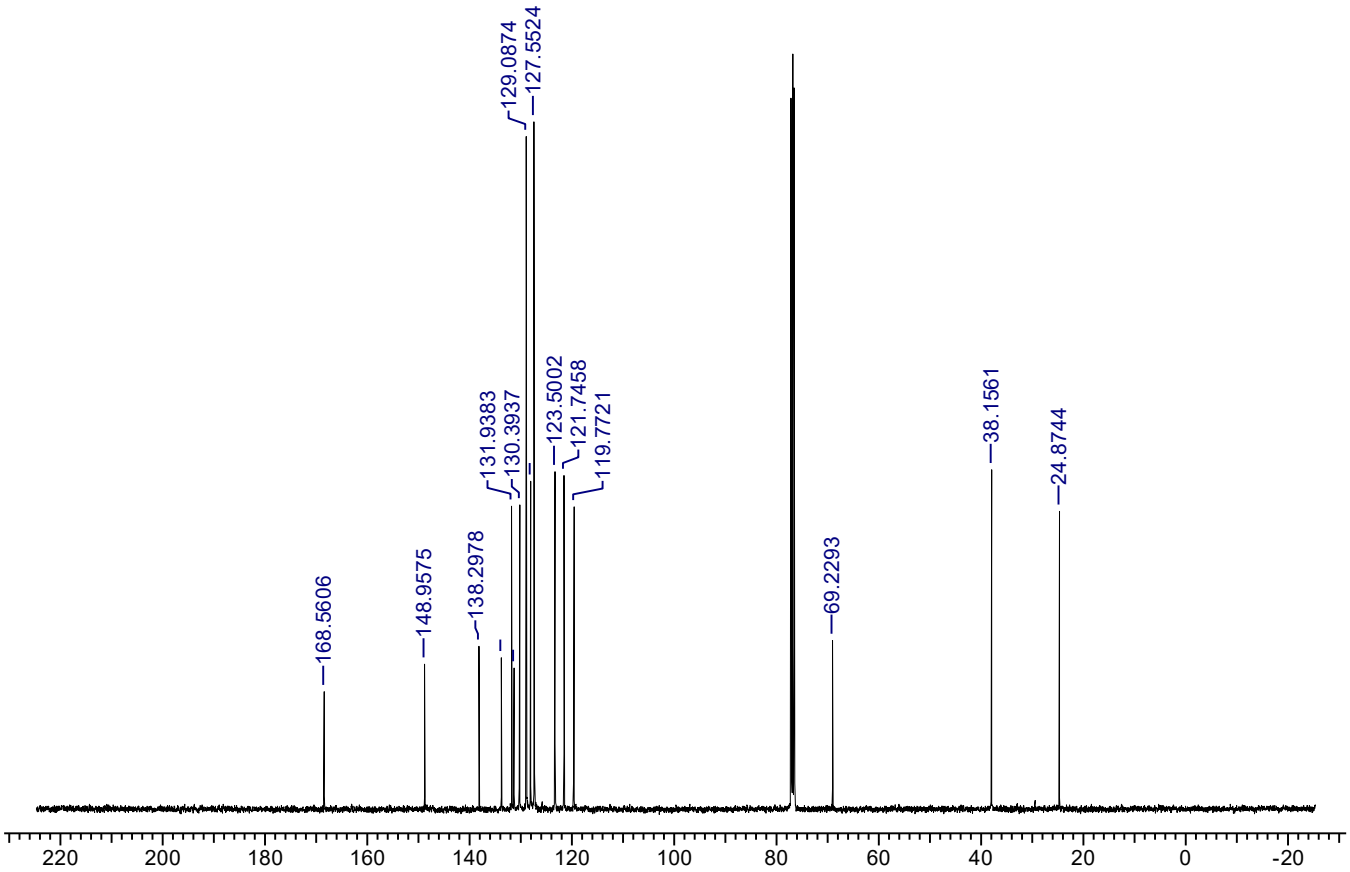
2.04
6.89
3.02
1.00
2.98
0.99
2.00
10
9
8
7
6
5
4
3
2
1
0
-1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130.3937
129.0874
127.5524
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121.7458
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24.8744
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200
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160
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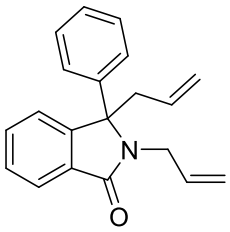
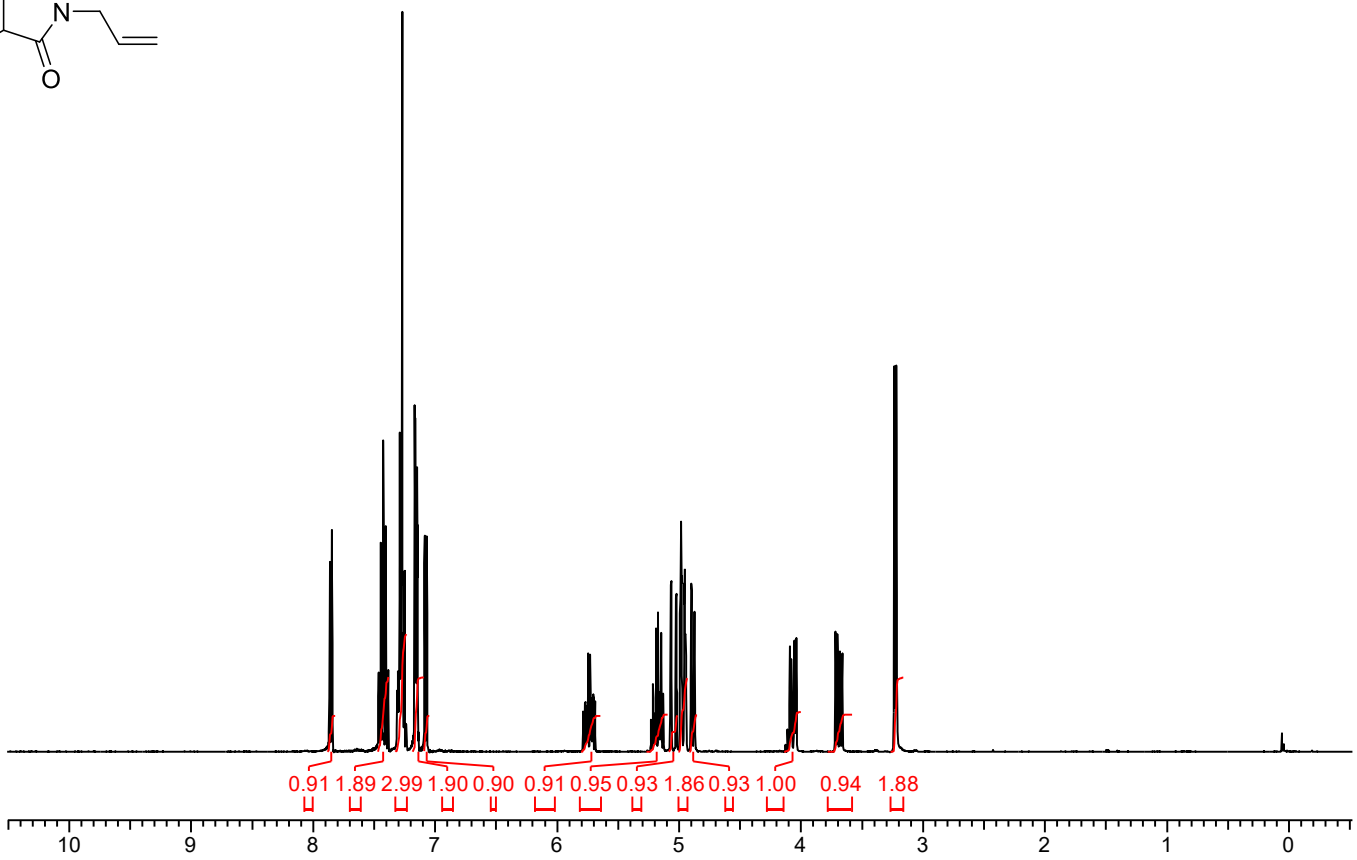
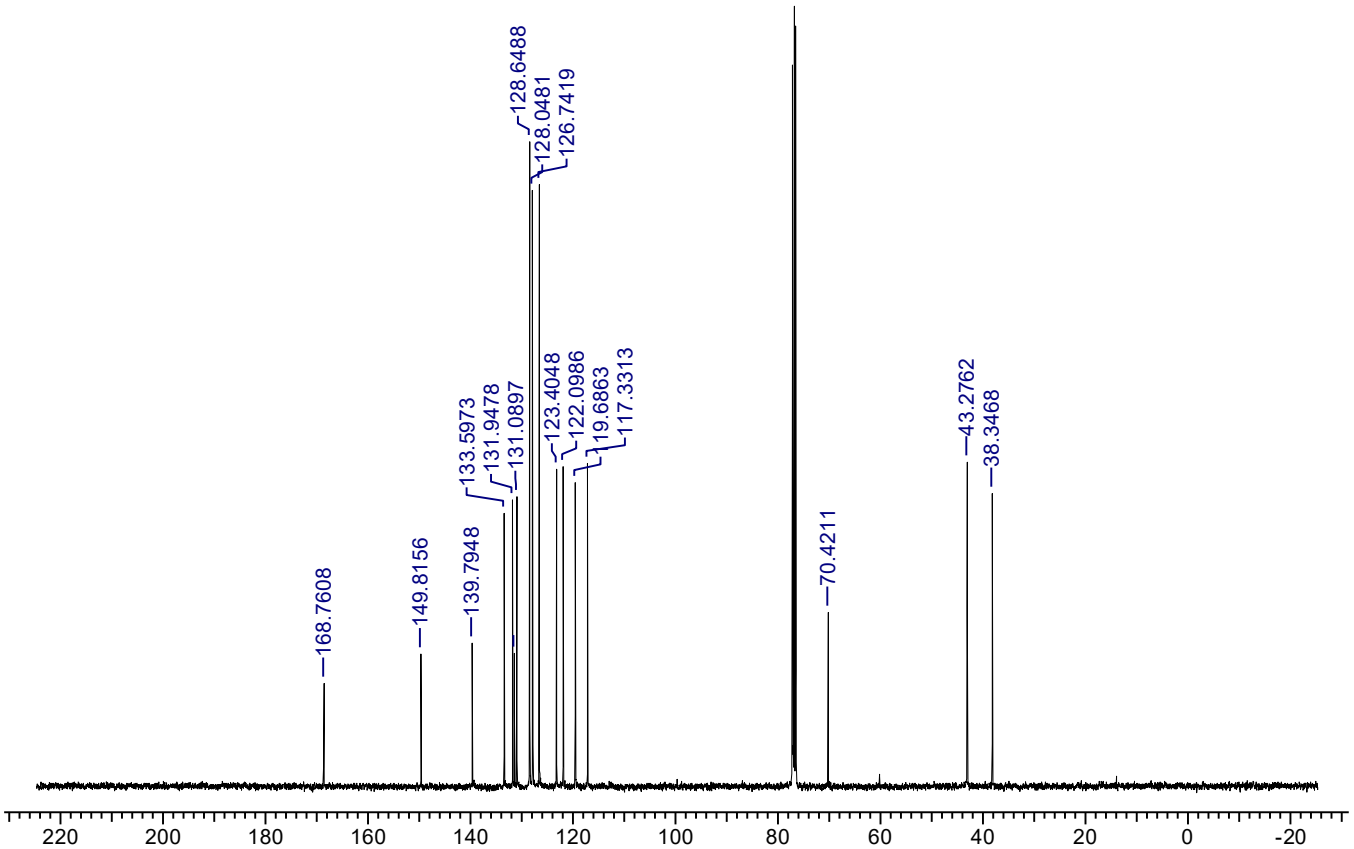
120
100
80
60
40
20
0
-20


N
O


0.91 1.89 2.99 1.90 0.90 0.91 0.95 0.93 1.86 0.93 1.00 0.94 1.88
10 9 8 7 6 5 4 3 2 1 0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128.6488
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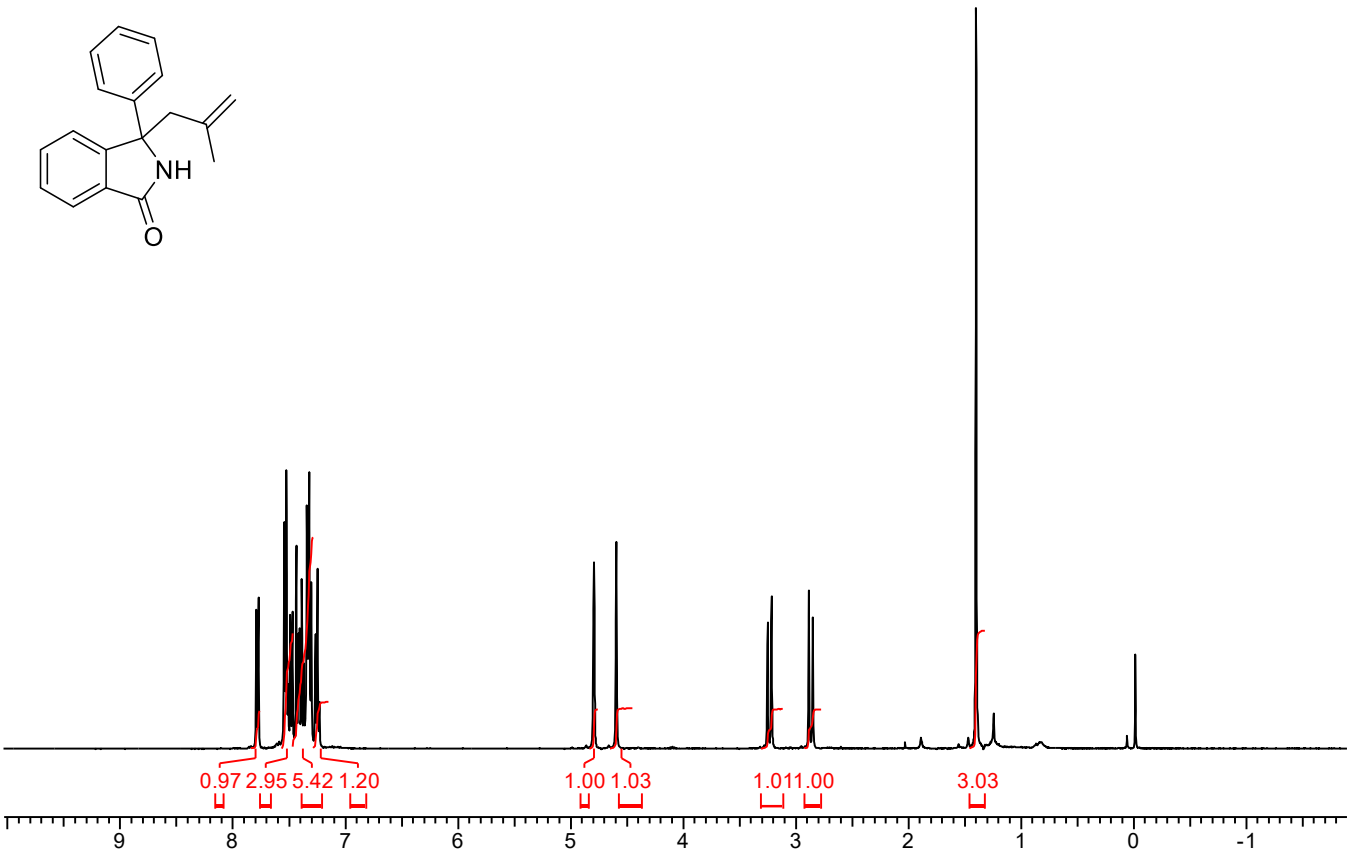
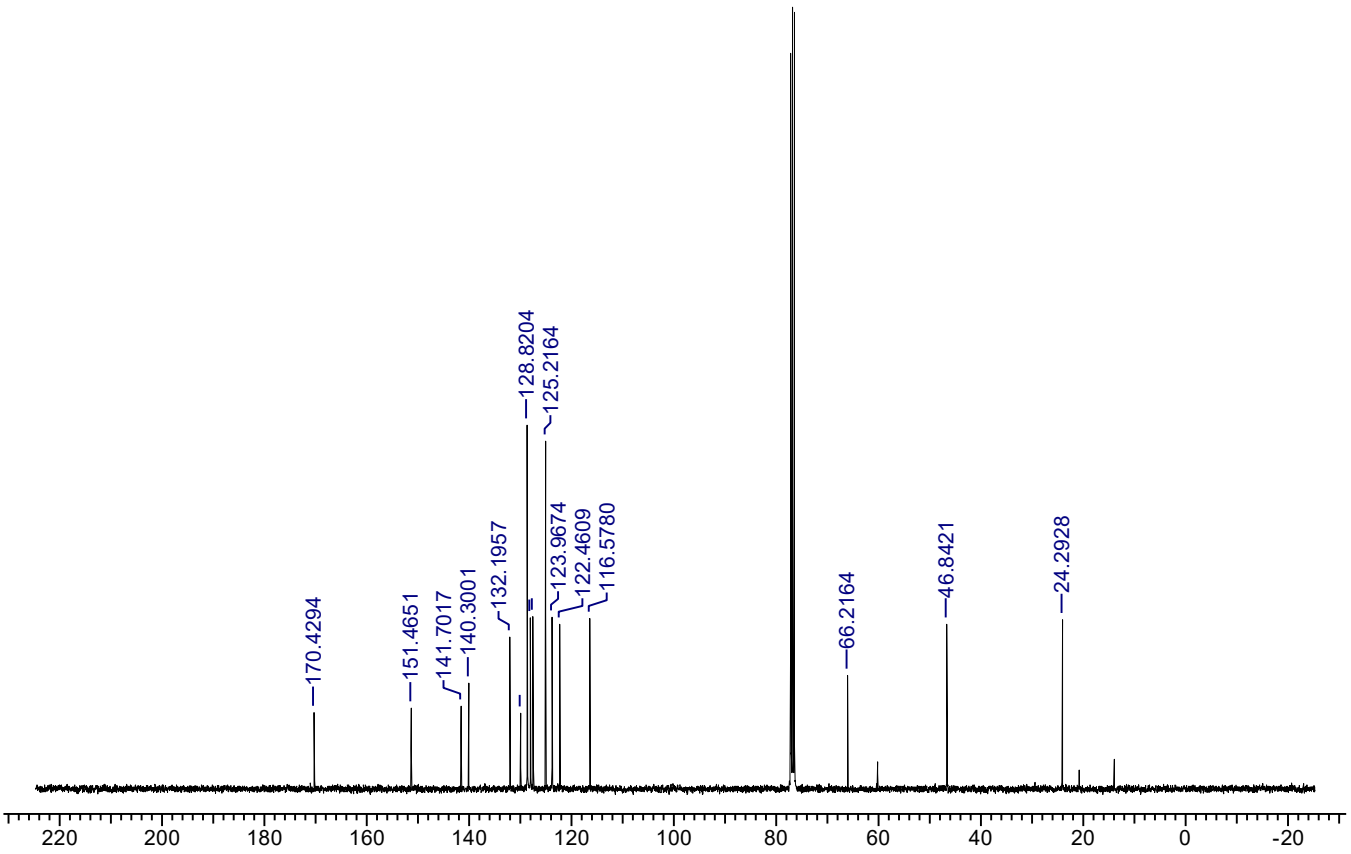
119.6863
117.3313
70.4211
43.2762
38.3468
220 200 180 160 140 120 100 80 60 40 20 0 -20


NH
O
0.97 2.95 5.42 1.20
1.00 1.03
1.01 1.00
3.03
9
8
7
6
5
4
3
2
1
0
-1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151.4651
141.7017
140.3001
132.1957
128.8204
125.2164
123.9674
122.4609
116.5780
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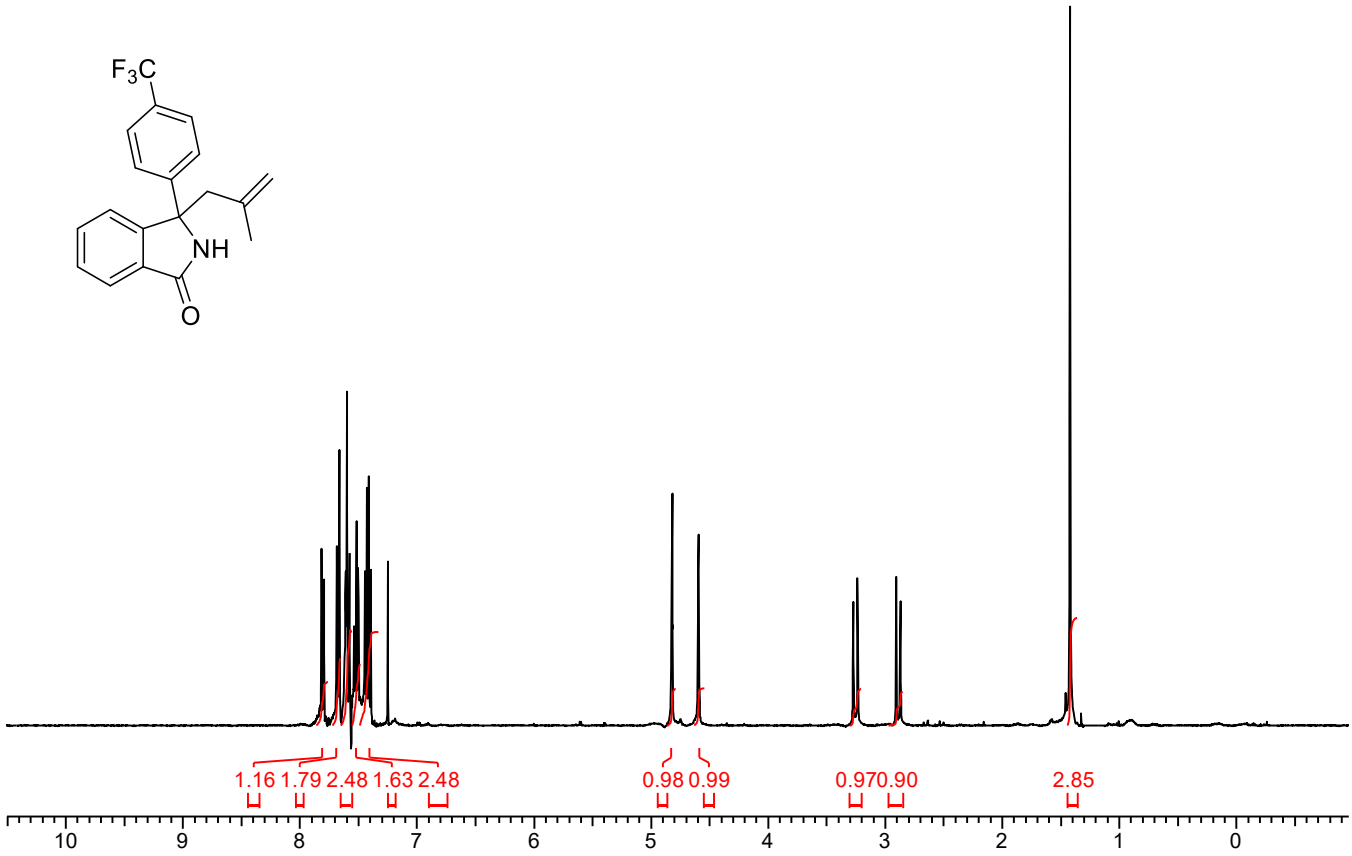
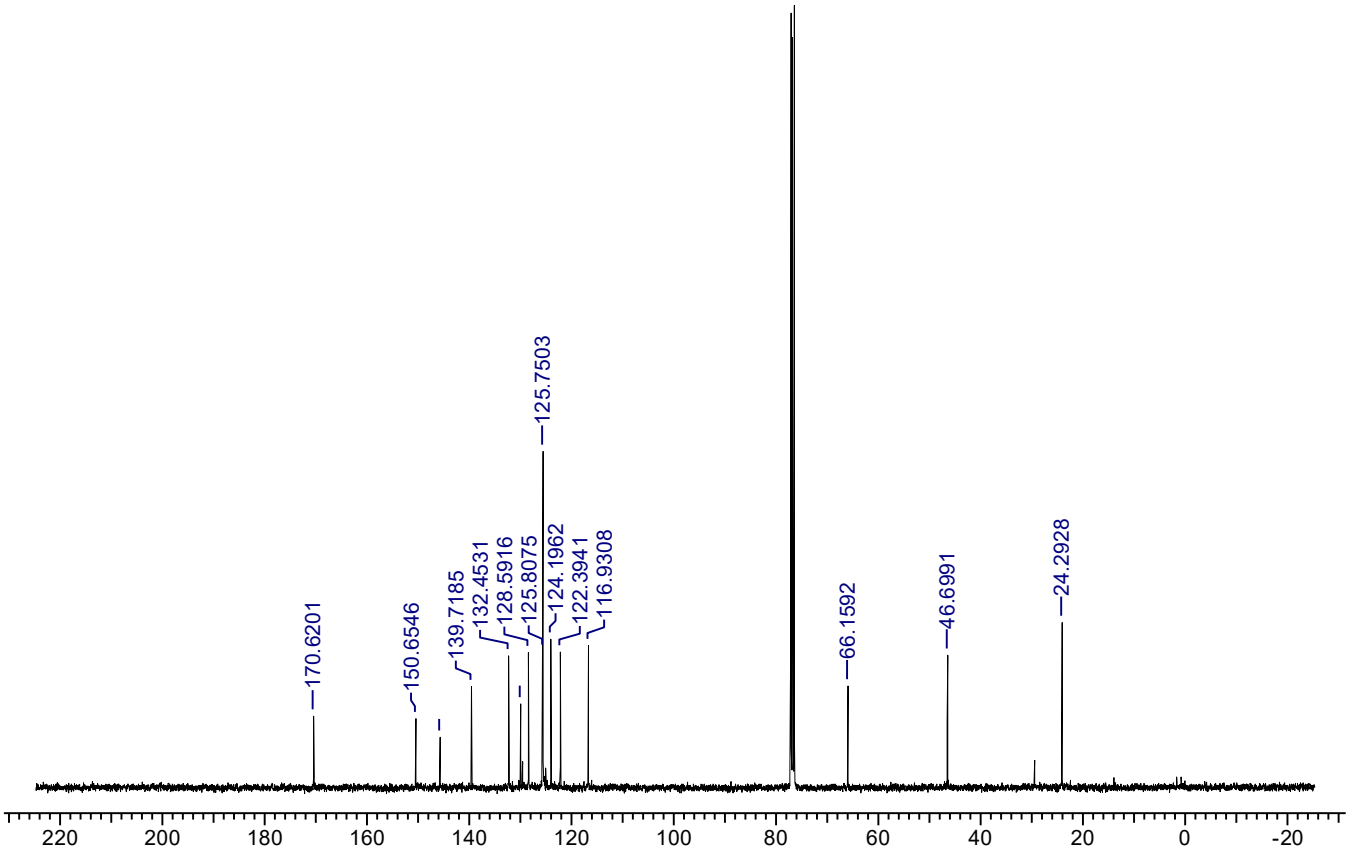
46.8421
24.2928
220
200
180
160
140
120
100
80
60
40
20
0
-20


F3C
NH
O
1.16 1.79 2.48 1.63 2.48
0.98 0.99
0.97 0.90
2.85
10 9 8 7 6 5 4 3 2 1 0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150.6546
139.7185
132.4531
128.5916
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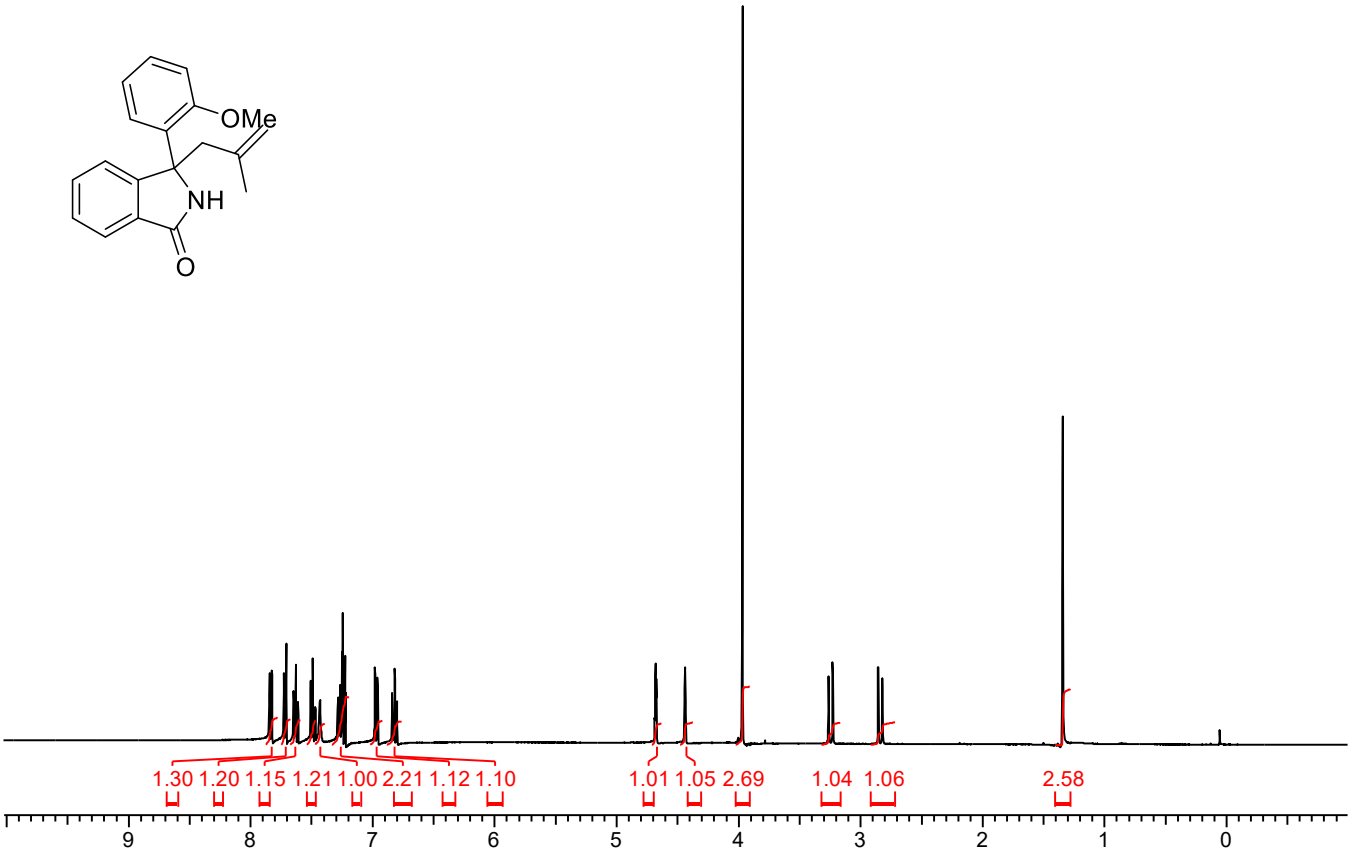
66.1592
46.6991
24.2928
220 200 180 160 140 120 100 80 60 40 20 0 -20


OMe
NH
O
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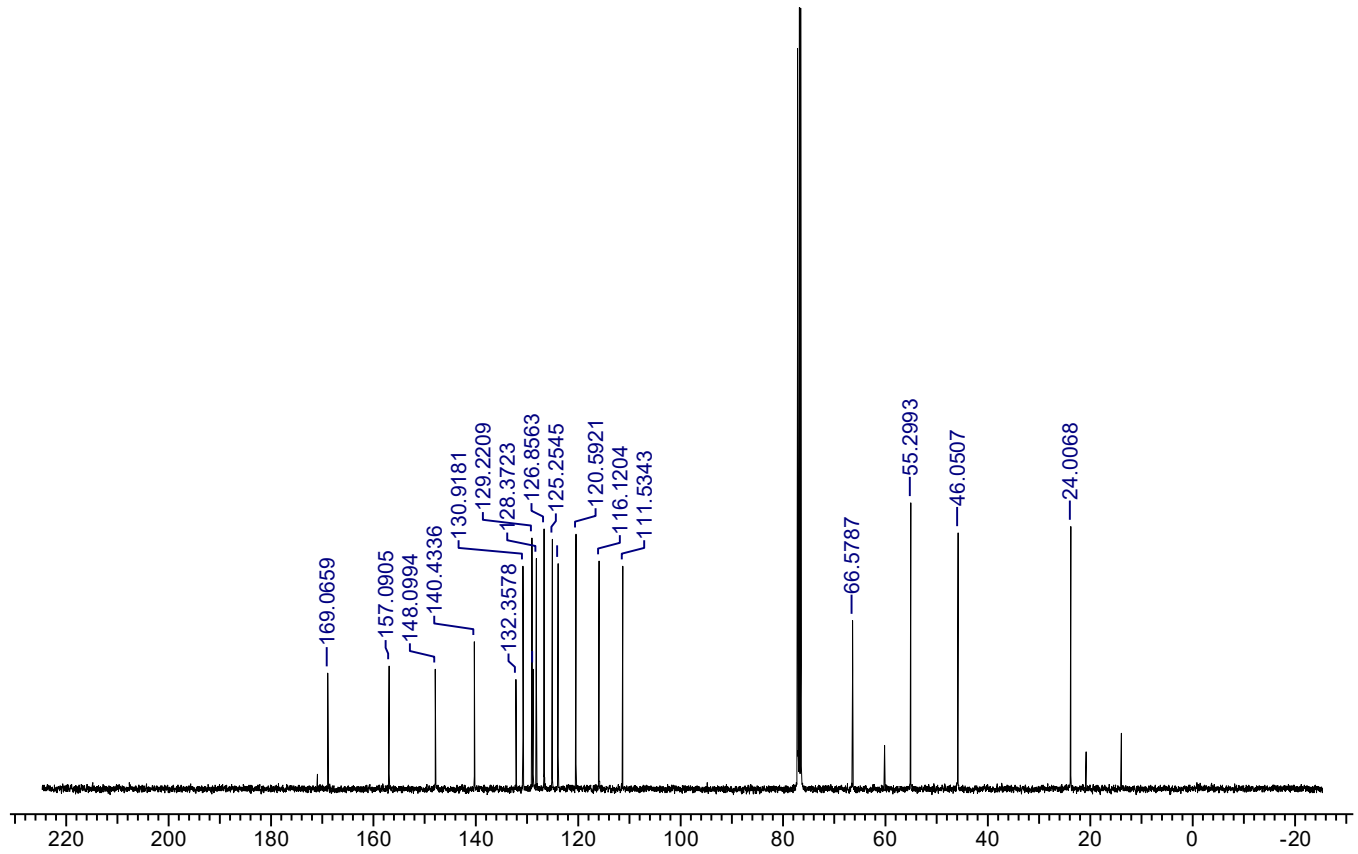
1.01 1.05 2.69
1.04 1.06
2.58
9 8 7 6 5 4 3 2 1 0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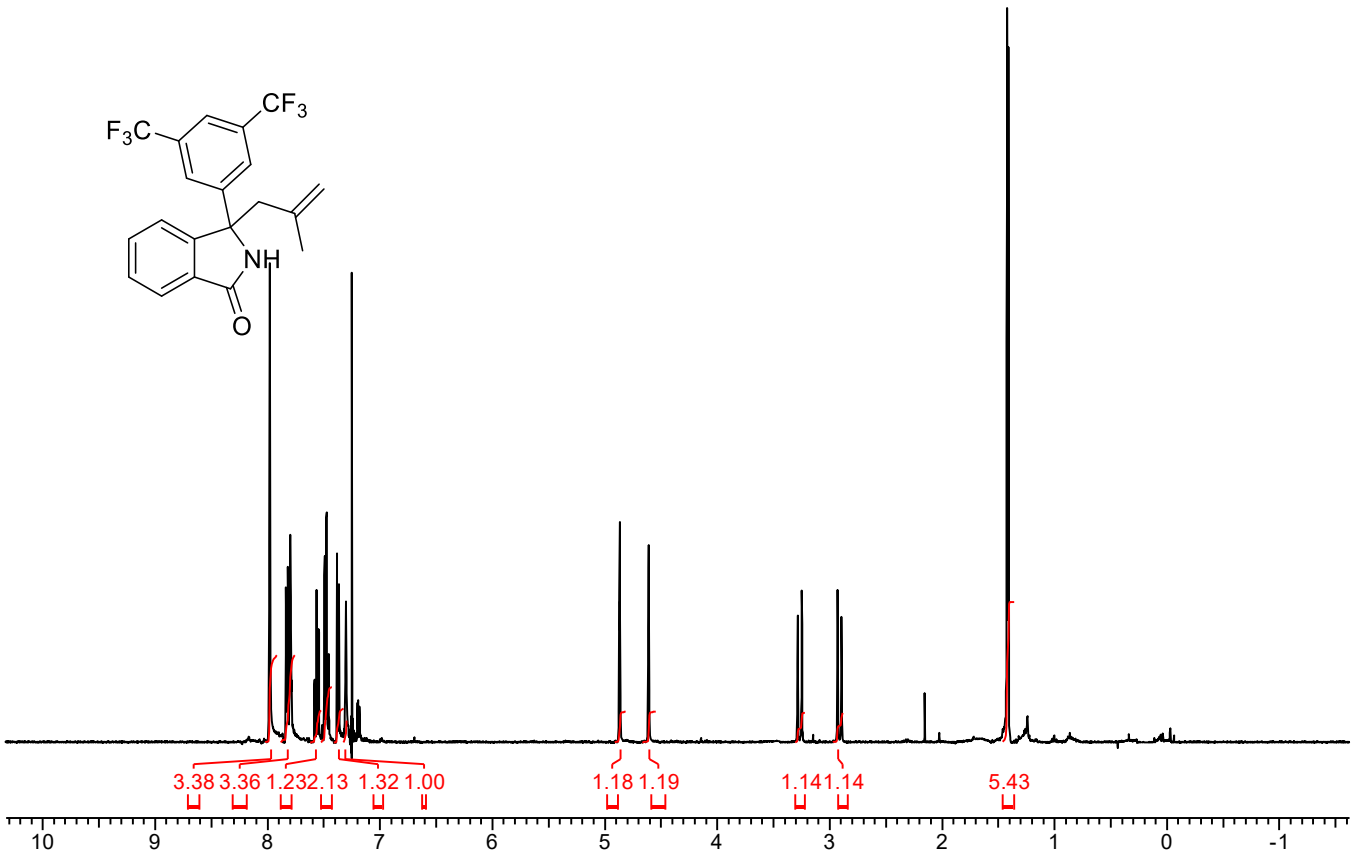
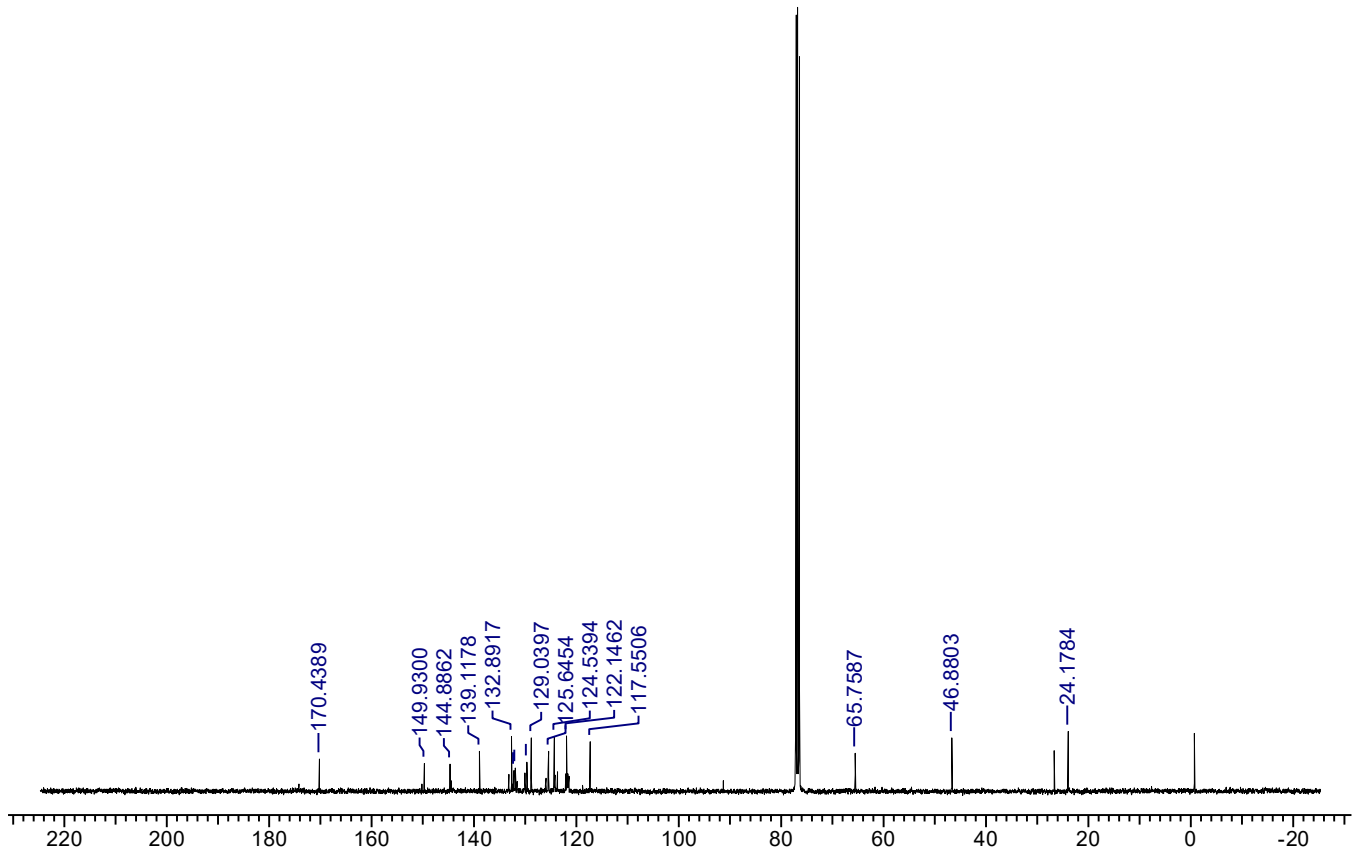
157.0905
148.0994
140.4336
132.3578
130.9181
129.2209
128.3723
126.8563
125.2545
120.5921
116.1204
111.5343
66.5787
55.2993
46.0507
24.0068
220 200 180 160 140 120 100 80 60 40 20 0 -20


F3C
CF3
NH
O
3.38 3.36 1.23 2.13 1.32 1.00
1.18 1.19
1.14 1.14
5.43
10 9 8 7 6 5 4 3 2 1 0 -1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139.1178
132.8917
129.0397
125.6454
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122.1462
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65.7587
46.8803
24.1784
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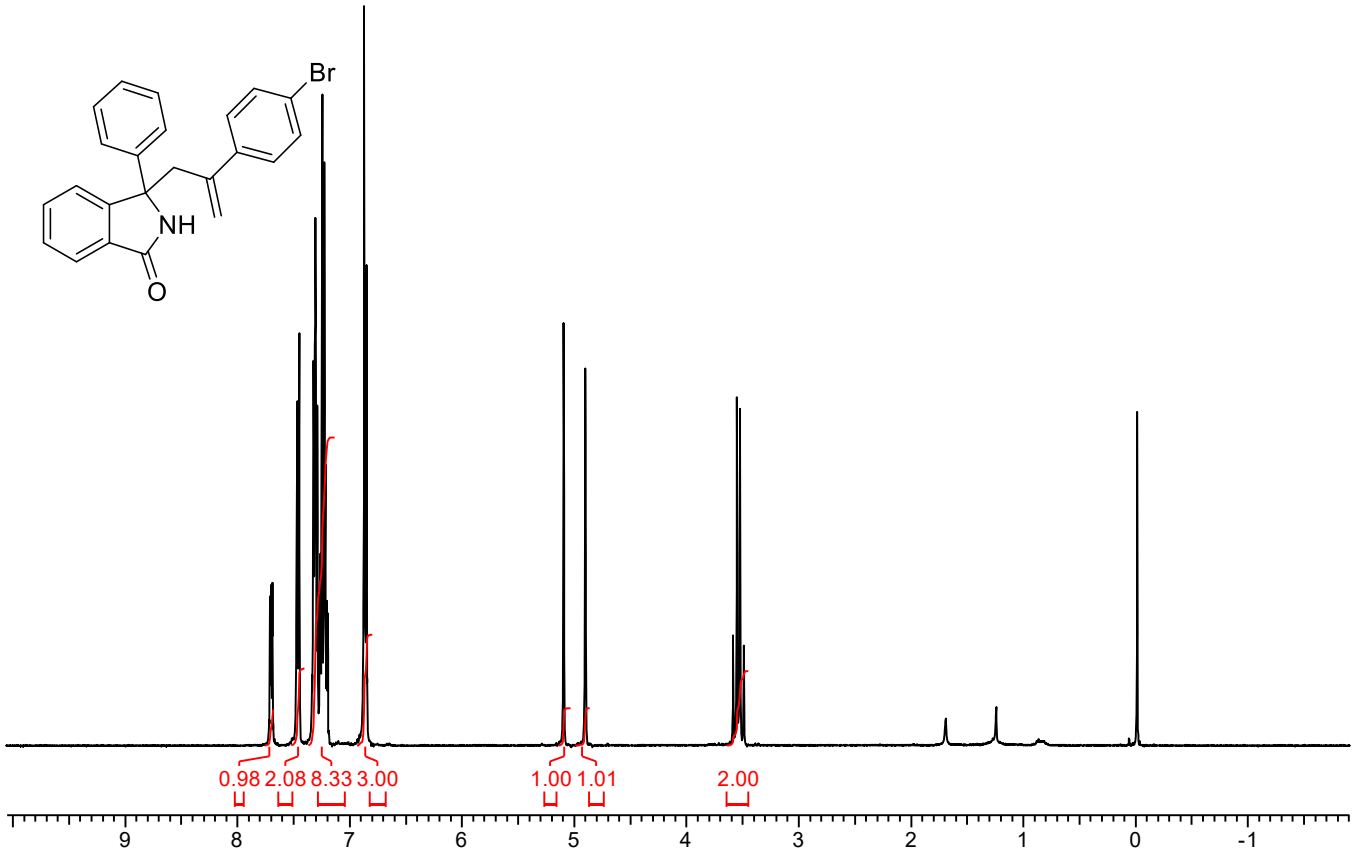
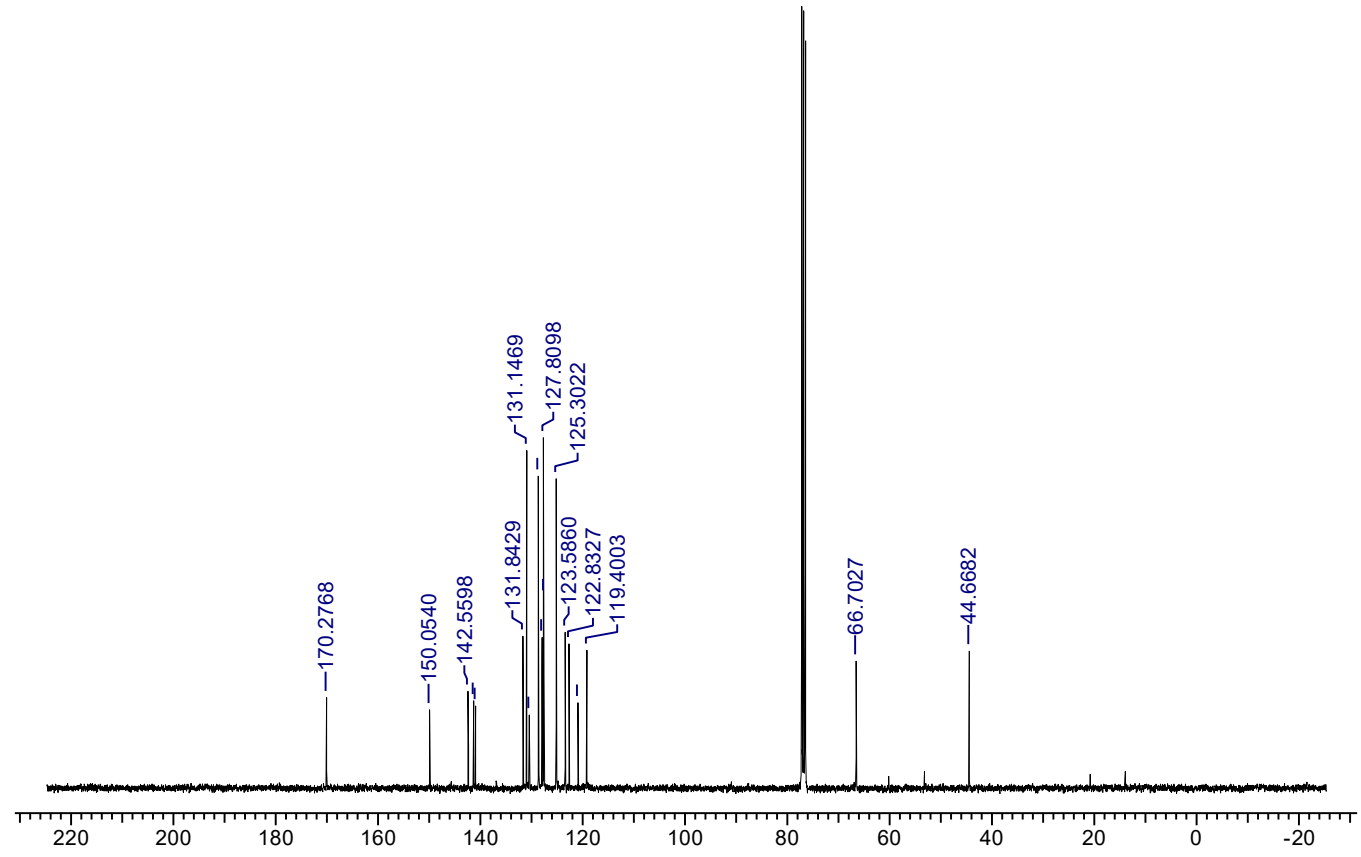

Br
NH
O
0.98
2.08
8.33
3.00
1.00
1.01
2.00
9
8
7
6
5
4
3
2
1
0
-1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150.0540
142.5598
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131.1469
127.8098
125.3022
123.5860
122.8327
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66.7027
44.6682
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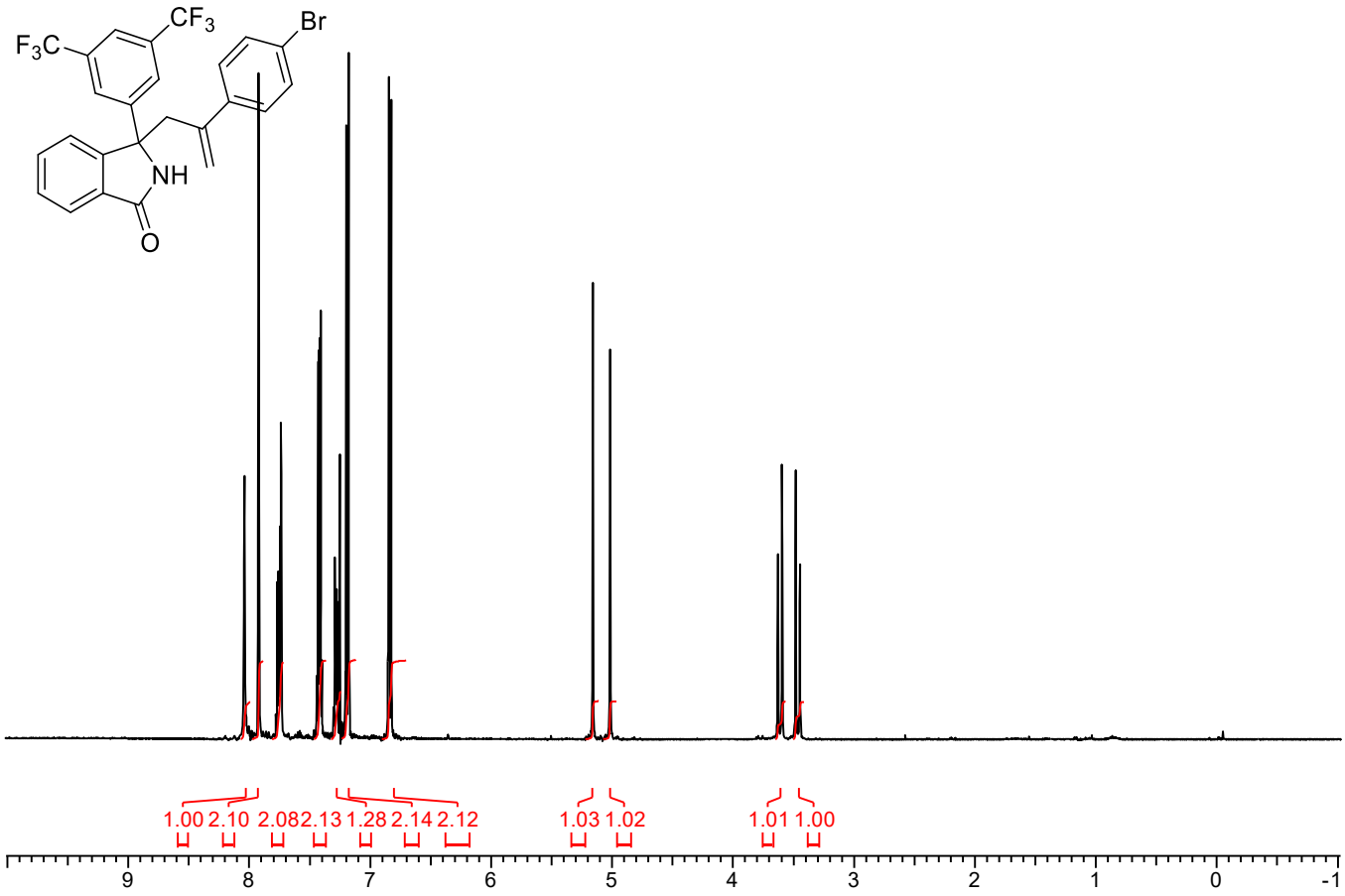
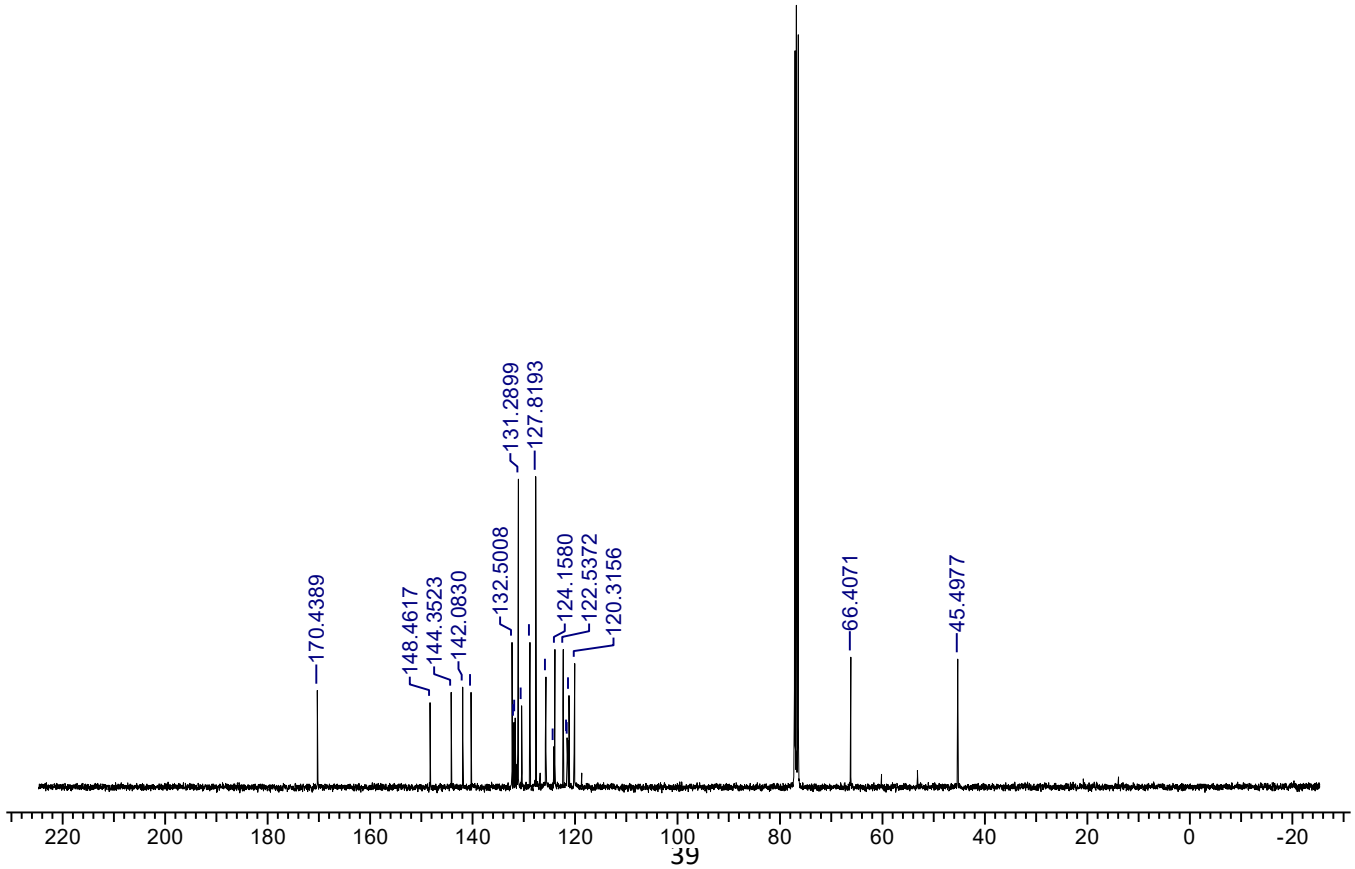
200
180
160
140
120
100
80
60
40
20
0
-20


CF3
F3C
Br
NH
O
1.00 2.10 2.08 2.13 1.28 2.14 2.12
1.03 1.02
1.01 1.00
9
8
7
6
5
4
3
2
1
0
-1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142.0830
132.5008
131.2899
127.8193
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122.5372
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45.4977
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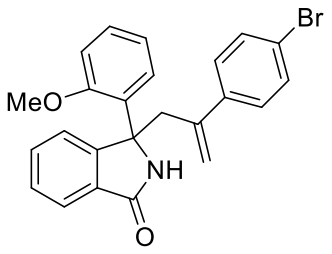
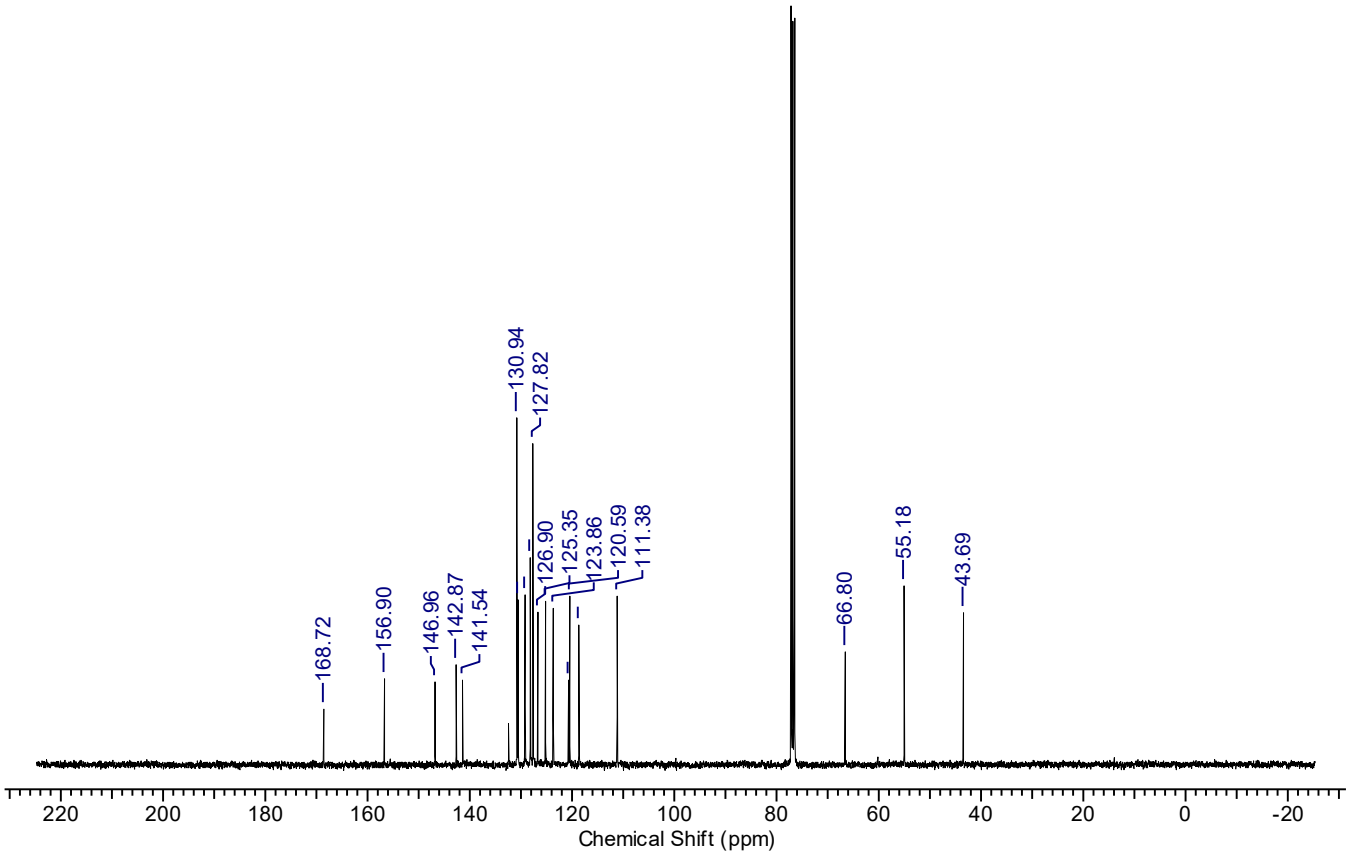
200
180
160
140
120
100
80
60
40
20
0
-20


Br
MeO
NH
O

1.01 1.02 2.10 3.22 1.02 0.86 1.03 2.02 1.02
0.99 1.00
2.98 1.02 1.00
9 8 7 6 5 4 3 2 1 0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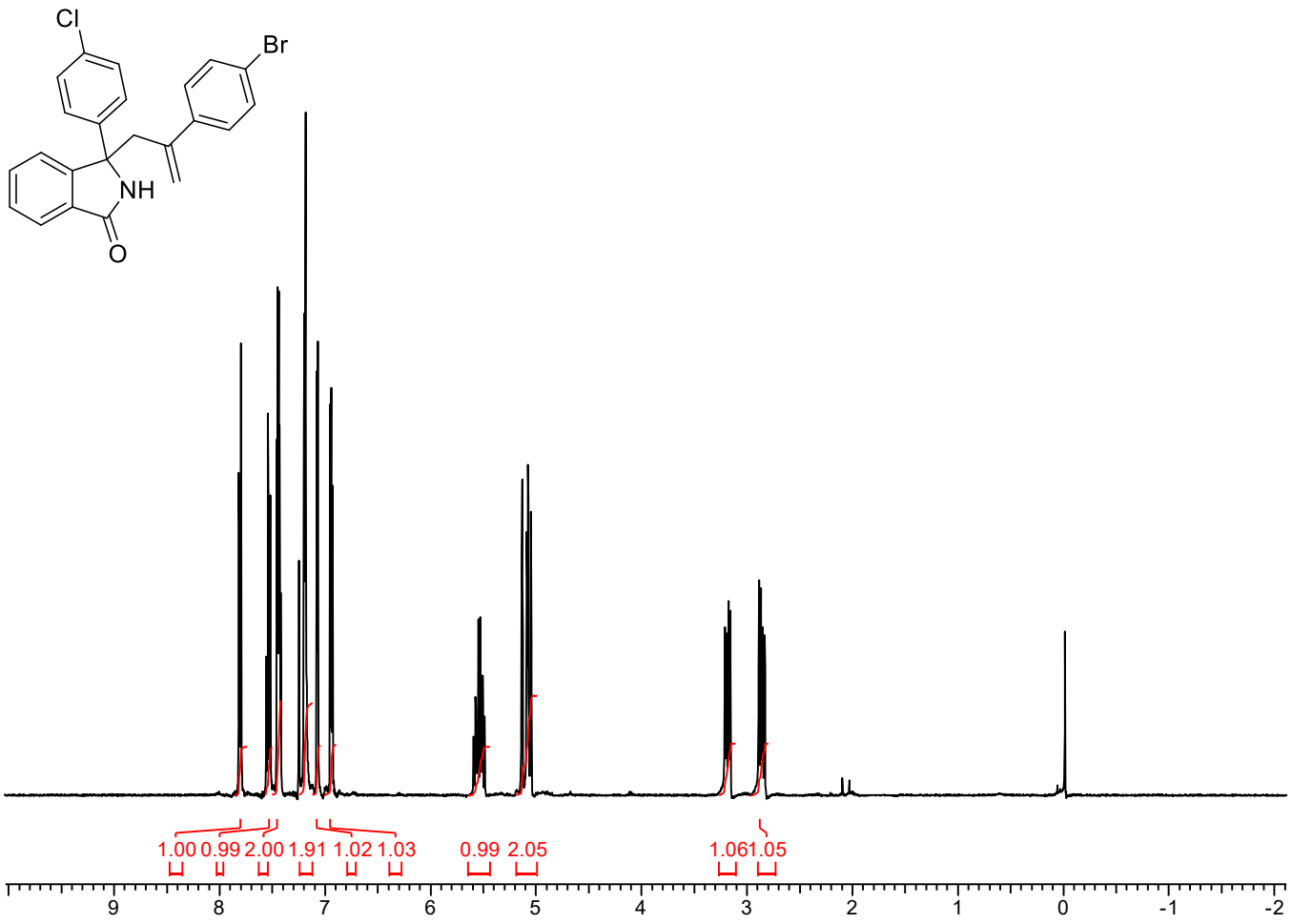
142.87
141.54
130.94
127.82
126.90
125.35
123.86
120.59
111.38
66.80
55.18
43.69
220 200 180 160 140 120 100 80 60 40 20 0 -20
Chemical Shift (ppm)


Cl
Br
NH
O
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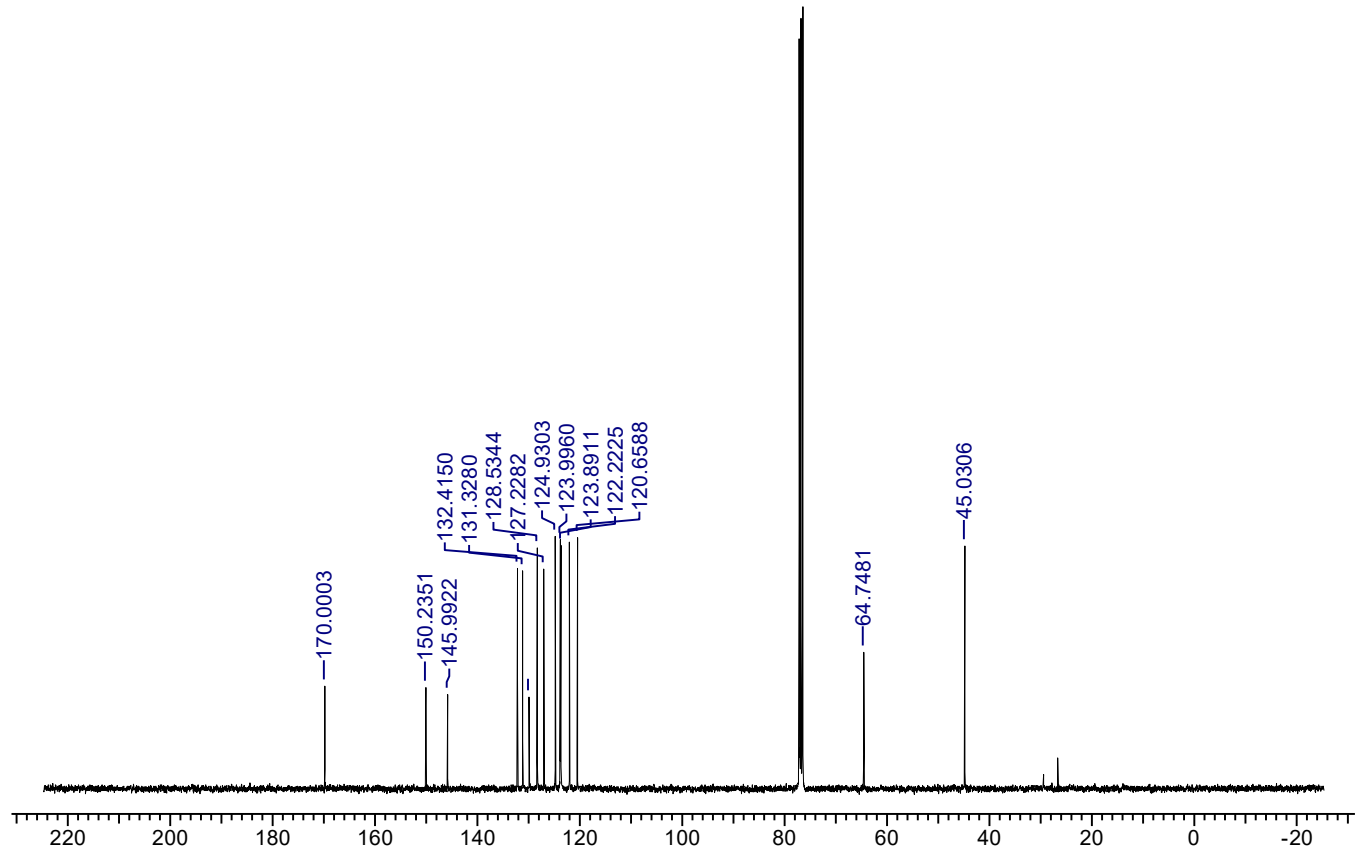
9 8 7 6 5 4 3 2 1 0 -1 -2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145.9922
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131.3280
128.5344
127.2282
124.9303
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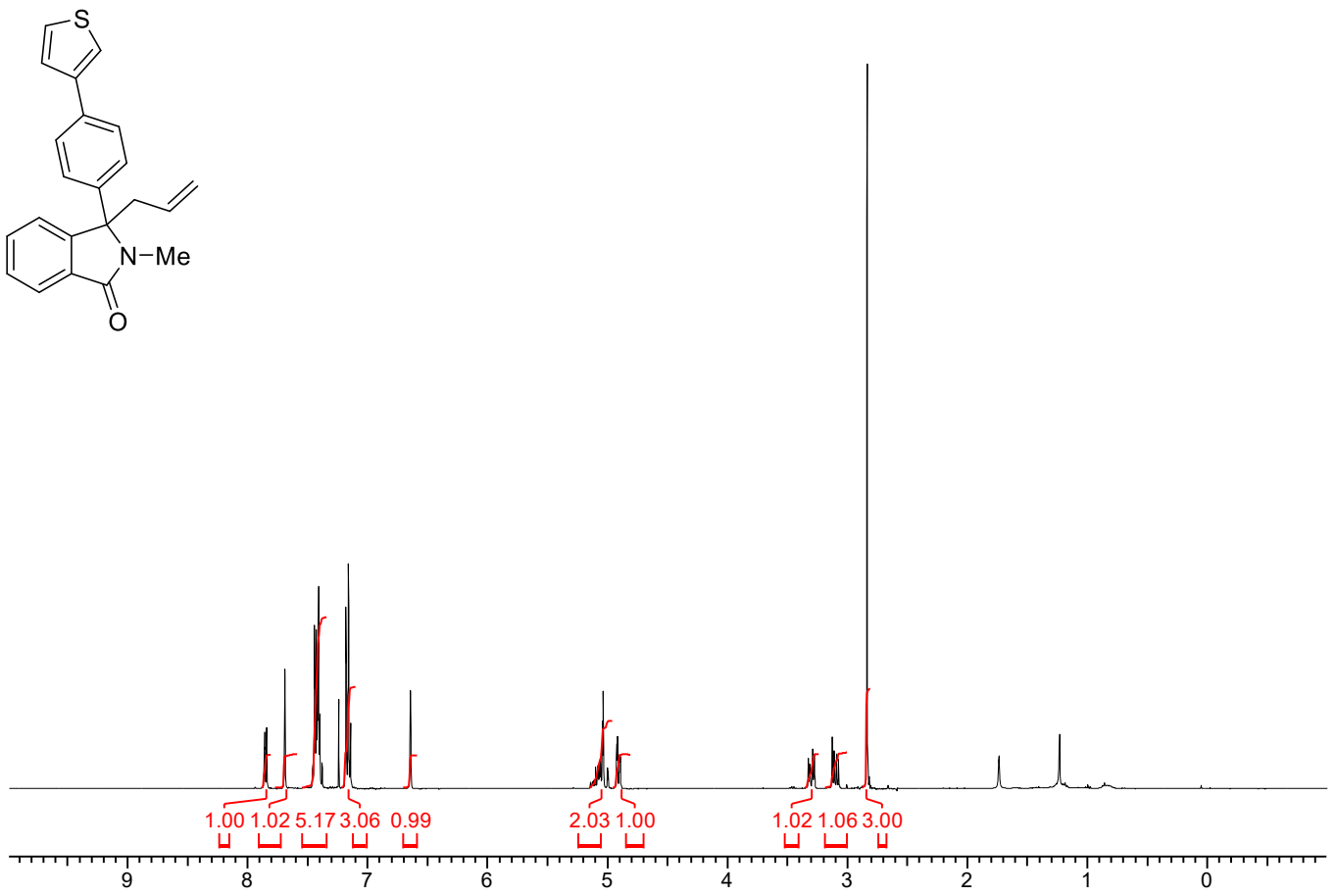
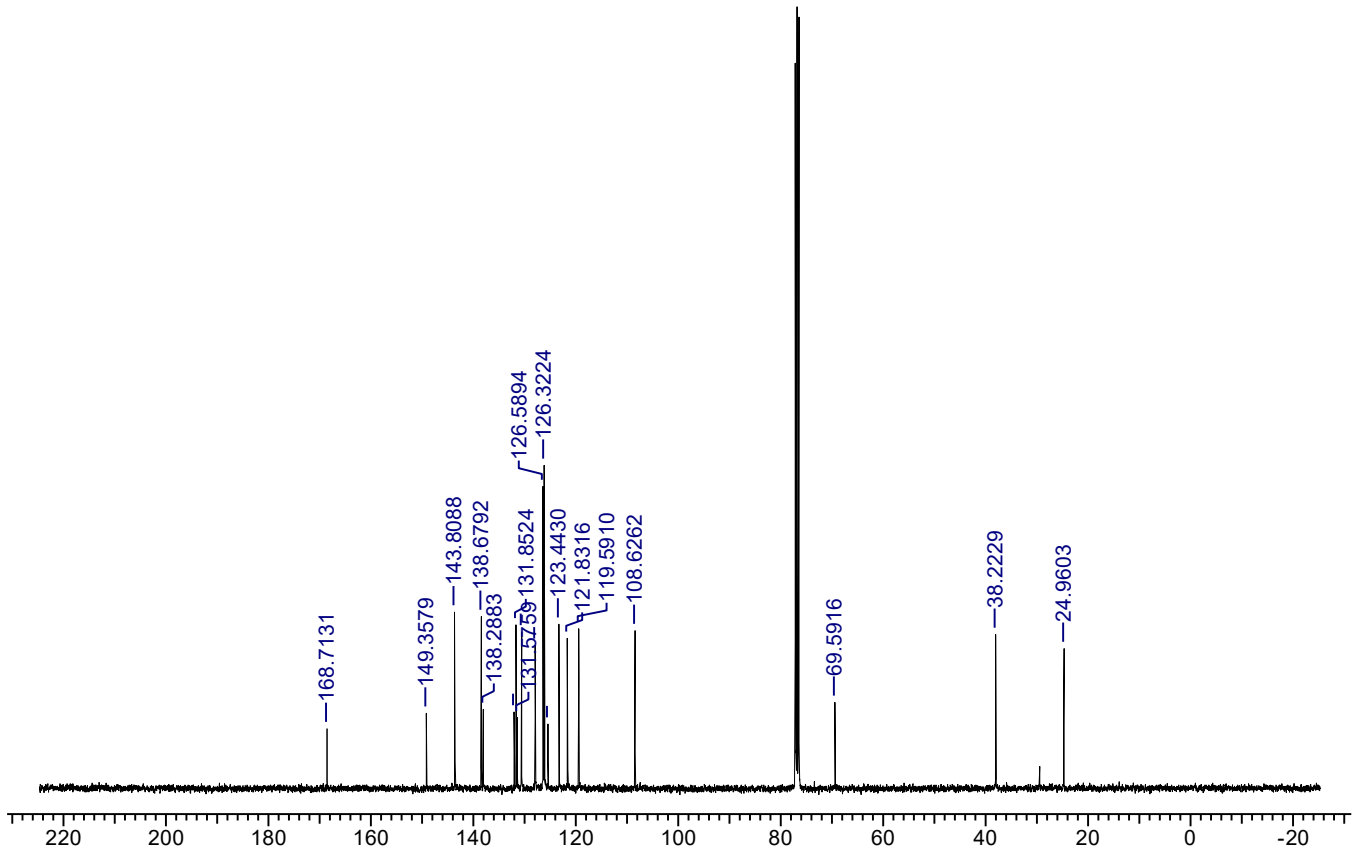
123.8911
122.2225
120.6588
64.7481
45.0306
220 200 180 160 140 120 100 80 60 40 20 0 -20


S
N−Me
O
1.00 1.02 5.17 3.06 0.99
2.03 1.00
1.02 1.06 3.00
9
8
7
6
5
4
3
2
1
0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126.3224
123.4430
121.8316
119.5910
108.6262
69.5916
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24.9603
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200
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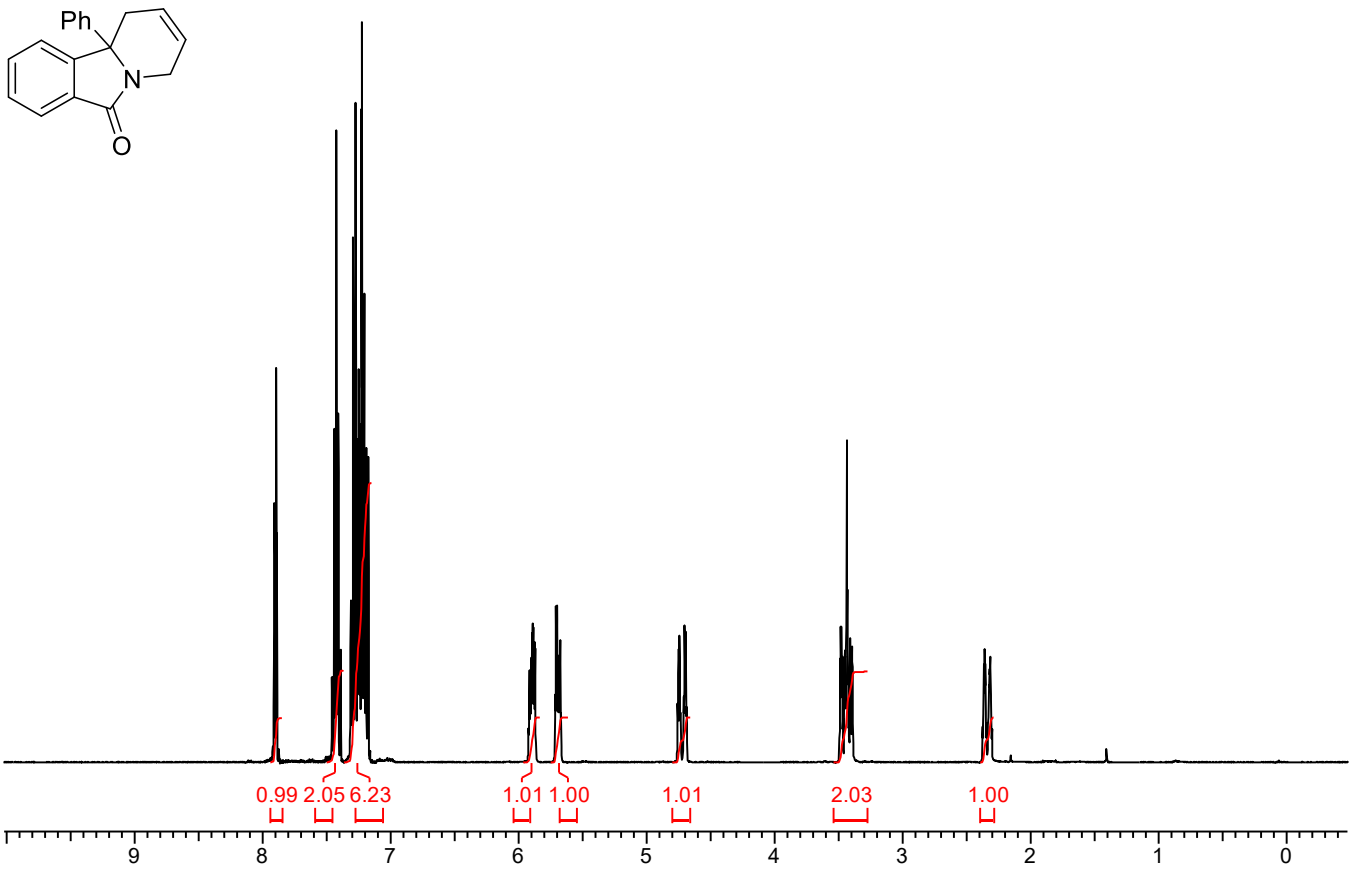
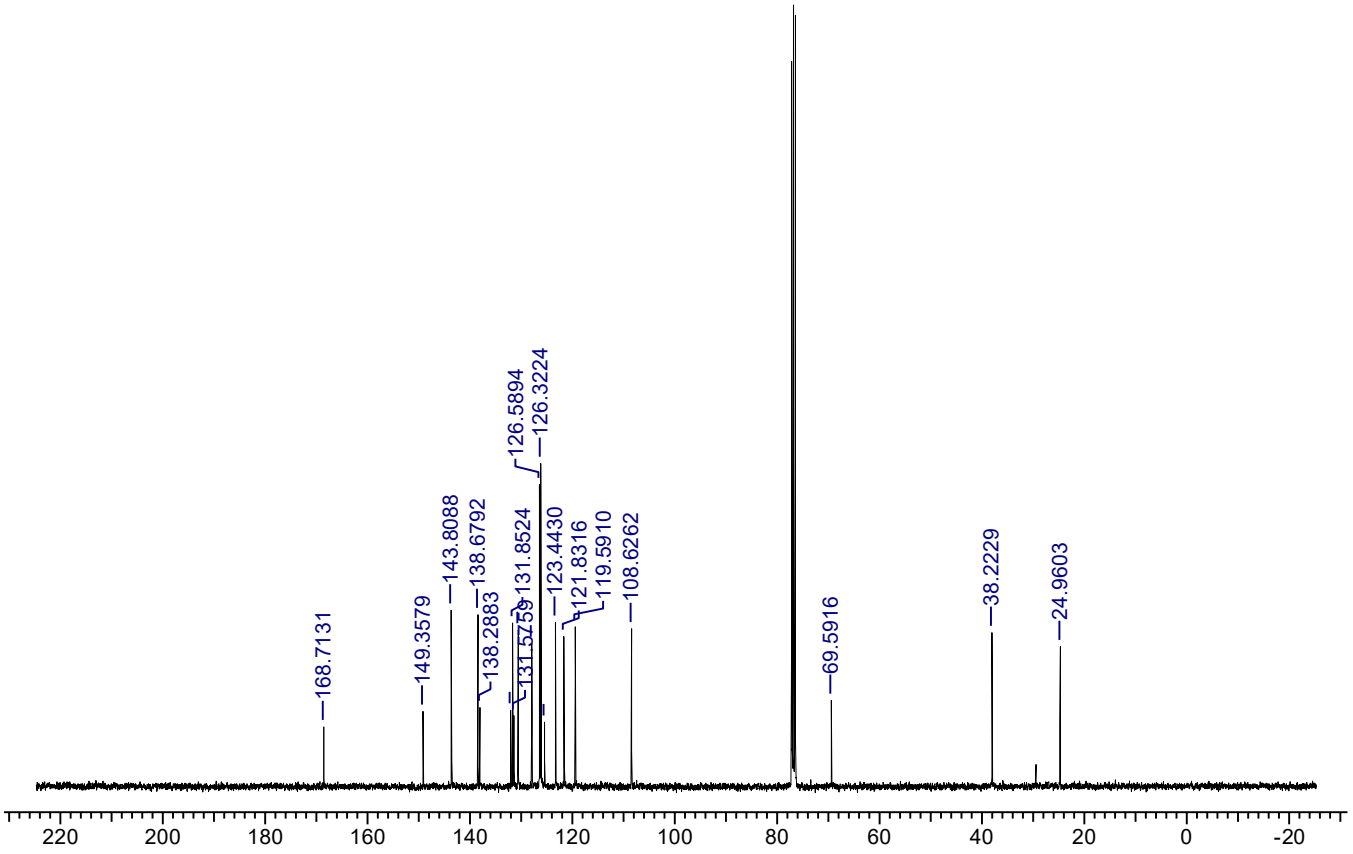
160
140
120
100
80
60
40
20
0
-20


Ph
N
O
0.99
2.05
6.23
1.01
1.00
1.01
2.03
1.00
9
8
7
6
5
4
3
2
1
0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149.3579
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108.6262
69.5916
38.2229
24.9603
220
200
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140
120
100
80
60
40
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0
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