Electronic Supplementary Material (ESI) for ChemComm.

Cobalt-catalyzed Regioselective Annulation of Silyl Alkynes with *o*-Methoxycarbonylphenylboronic acid

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Supporting Information

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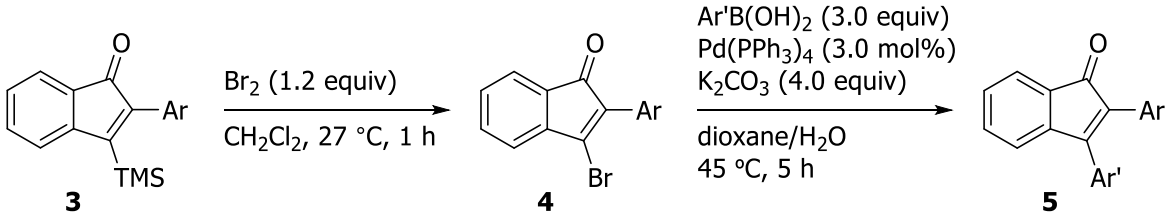
General

The products were purified by column chromatography on silica gel [Kanto Chem. Co. Silica Gel 60N (spherical, neutral, 40-50 μm)]. If necessary, there were further purified by preparative HPLC (Japan Analytical Industry Co., Ltd.,LC-908, LC-918) equipped with GPC columns (JAIGEL-1H + JAIGEL-2H columns) using $CHCl_3$ as an eluent. ^1H NMR spectra were recorded on JEOL ECS-400 (400 MHz) and referenced to the solvent peak at 7.26 ppm for $CDCl_3$ or TMS peak at 0.00 ppm. ^{13}C NMR spectra were recorded on JEOL ECS-400 (100 MHz) and referenced to the solvent peak at 77.16 ppm for $CDCl_3$. Splitting patterns are indicated as follows; s: singlet, d: doublet, dd: double doublet, t: triplet, br: broad, m: multiplet. Infrared spectra were obtained on a JASCO FT/IR-4100 spectrometer; absorptions are reported in reciprocal centimeters. High-resolution mass spectra were recorded with a JEOL MS-700 spectrometer. Melting point were recorded with a BÜCHI Melting Point B-540. MeCN was distilled from CaH_2 before use. All commercially available reagents were used without further purification.

General Procedure

A 25 mL sealed tube equipped with a magnetic stirring bar was charged with $Co(acac)_2$ (0.05 mmol, 10 mol %), DPPE (0.05 mmol, 10 mol %), an alkyne **1** (0.50 mmol, 1.0 equiv), *o*-methoxyphenylboronic acid **2** (0.75 mmol, 1.5 equiv), and MeCN (2.0 mL). The reaction mixture was stirred at 80 °C for 12 h. The mixture was then filtered through silica gel pad. The filtrate was concentrated, and the residue was purified by column chromatography on silica gel to yield the desired product **3**. If necessary, there were further purified by preparative HPLC to afford the desired product **3**.

General Procedure 2



Bromination:

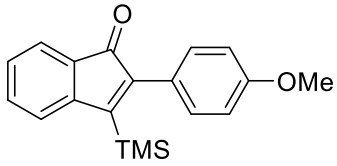
To a solution of a 2-aryl-3-silylindenone **3** in anhydrous CH_2Cl_2 at 27 °C under Ar, Br_2 (1.2 equiv) was added. The reaction mixture was stirred at 27 °C for 1 h. The reaction mixture was then quenched with saturated Na_2SO_3 solution, and diluted with H_2O. The resulting solution was extracted with CH_2Cl_2, and the separated aqueous layer was re-extracted with CH_2Cl_2. The combined organic layer was washed with brine and dried over Na_2SO_4, and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel to give the desired product **4**.

Suzuki-Miyaura cross coupling reaction:

The reaction was carried out in a two-necked flask. To a suspension of 3-bromoindenone **4**, $Pd(PPh_3)_4$ (3.0 mol %), and arylboronic acid (3.0 equiv) in dioxane, a 2M solution of K_2CO_3 was added. The reaction mixture was stirred at 45 °C for 5 h. The reaction mixture was diluted with H_2O and extracted 3 times with CH_2Cl_2. The combined organic layer was dried Na_2SO_4, and filtered. The filtrate was concentrated under reduced pressure. The residue was purified by column chromatography on silica gel to give the desired product **5**.

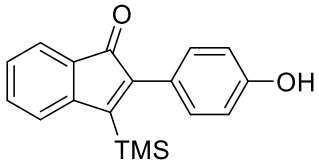
Spectrum Data

2-(4-Methoxyphenyl)-3-(trimethylsilyl)-1*H*-inden-1-one (3a)



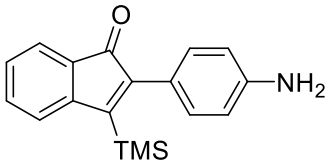
Orange solid (135.6 mg, 88% yield); m.p. = 144.4-144.9 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.18 (s, 9H), 3.84 (s, 3H), 6.92 (d, *J* = 8.8 Hz, 2H), 7.16-7.24 (m, 4H), 7.37 (t, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.48 (d, *J* = 6.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 0.33, 55.3, 113.5, 123.1, 123.2, 125.6, 127.9, 130.1, 131.1, 134.1, 147.6, 149.0, 155.9, 159.9, 198.2; IR (neat) 1439, 1609, 1709, 2340, 2358, 2834, 2897, 3065, 3390 cm^{-1}; EI-MS *m/z* (relative intensity) 308 ([M$^+$], 100), 293 (62), 275 (44), 235 (11), 165 (13); HRMS (EI) *m/z* calcd for $C_{19}H_{20}O_2Si$ ([M]$^+$) 308.1233, found: 308.1233.

2-(4-Hydroxyphenyl)-3-(trimethylsilyl)-1*H*-inden-1-one (3b)



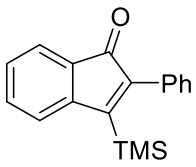
Red solid (82.2 mg, 56% yield); m.p. = 158.1-158.4 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.18 (s, 9H), 4.83 (brs, 1H), 6.84 (d, *J* = 8.4 Hz, 2H), 7.13 (d, *J* = 8.0 Hz, 2H), 7.17-7.26 (m, 2H), 7.37 (t, *J* = 7.2 Hz, 1H), 7.48 (d, *J* = 6.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 0.23, 115.2, 123.2, 123.4, 125.1, 128.0, 130.1, 131.2, 134.4, 147.8, 148.9, 156.4, 156.8, 199.2; IR (neat) 1251, 1455, 1608, 1696, 2359 cm^{-1}; EI-MS *m/z* (relative intensity) 294([M$^+$], 100), 279 (85), 261 (86), 151 (9), 221 (16) 189 (13), 165 (25), 89 (13), 85 (40), 73 (25); HRMS (EI) *m/z* calcd for $C_{18}H_{18}O_2Si$ ([M]$^+$) 294.1076, found: 294.1063.

2-(4-Aminophenyl)-3-(trimethylsilyl)-1*H*-inden-1-one (3c)



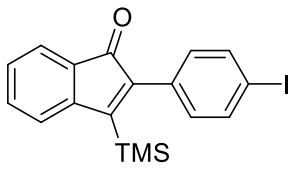
Red oil (70.0 mg, 48% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.19 (s, 9H), 3.76 (brs, 2H), 6.69 (d, *J* = 8.8 Hz, 2H), 7.06 (d, *J* = 8.4 Hz, 2H), 7.15-7.26 (m, 2H), 7.35 (t, *J* = 7.4 Hz, 1H), 7.52 (d, *J* = 6.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 0.35, 114.5, 122.97, 123.0, 123.1, 127.7, 130.2, 131.0, 134.1, 146.9, 148.0, 149.2, 154.9, 198.5; IR (neat) 1455, 1606, 1704, 2955 cm^{-1}; EI-MS *m/z* (relative intensity) 293 ([M$^+$], 100), 278 (54), 260 (32), 150 (19), 83 (29); HRMS (EI) *m/z* calcd for $C_{18}H_{19}NOSi$ ([M]$^+$) 293.1236, found: 293.1243.

2-Phenyl-3-(trimethylsilyl)-1*H*-inden-1-one (3d)[1,2]



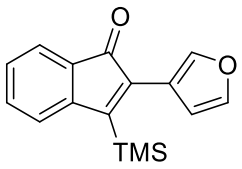
Yellow oil (54.5 mg, 39% yield); the structure was determined by ^{1}H NMR and ^{13}C NMR of previous reports.[1,2] ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.16 (s, 9H), 7.21-7.26 (m, 4H), 7.36-7.41 (m, 4H), 7.50 (d, *J* = 7.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 0.19, 123.3, 128.0 (two peaks overlap), 128.2, 128.4, 129.9, 130.2, 133.4, 134.2, 148.1, 148.8, 156.9, 197.9; IR (neat) 1252, 1455, 1708, 2955 cm^{-1}; EI-MS *m/z* (relative intensity) 278 ([M^{+}], 100), 263 (83), 245 (81), 212 (12), 203 (11), 176 (10), 129 (9), 115 (11), 75 (8), 73 (20); HRMS (EI) *m/z* calcd for $C_{18}H_{18}OSi$ ([M]$^{+}$) 278.1127, found: 278.1123.

2-(4-Iodophenyl)-3-(trimethylsilyl)-1*H*-inden-1-one (3e)



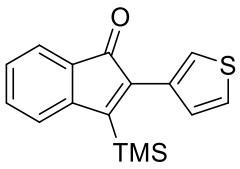
Orange solid (70.2 mg, 35% yield); m.p. = 84.1-84.9 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.18 (s, 9H), 6.98 (d, *J* = 8.4 Hz, 2H), 7.20-7.26 (m, 2H), 7.39 (dt, *J* = 7.2 Hz, 1.6 Hz, 1H), 7.49 (d, *J* = 7.6 Hz, 1H), 7.72 (d, *J* = 6.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 0.30, 94.5, 123.4, 123.5, 128.4, 130.0, 131.7, 132.9, 134.3, 137.1, 146.8, 148.6, 157.6, 197.4; IR (neat) 1604, 1708, 2953 cm^{-1}; EI-MS *m/z* (relative intensity) 404 ([M^{+}], 100), 389 (16), 262 (64), 247 (23), 213 (34); HRMS (EI) *m/z* calcd for $C_{18}H_{17}IOSi$ ([M]$^{+}$) 404.0093, found: 404.0083.

2-(Furan-3-yl)-3-(trimethylsilyl)-1*H*-inden-1-one (3f)



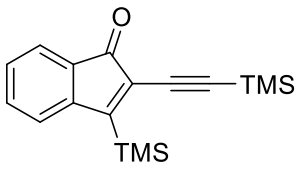
Brown oil (70.2 mg, 52% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.30 (s, 9H), 6.45-6 (s, 1 H), 7.18 (t, *J* = 7.4 Hz, 1H), 7.22-7.26 (m, 1H), 7.36 (t, *J* = 7.4 Hz, 1H), 7.46-7.47 (m, 2H), 7.56 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 0.42, 112.3, 117.1, 123.2, 123.3, 128.0, 130.2, 134.2, 139.4, 142.4, 142.6, 149.2, 156.2, 197.7; IR (neat) 1714, 2900, 2957 cm^{-1}; EI-MS *m/z* (relative intensity) 268 ([M^{+}], 13), 256 (12), 149 (28), 85 (68), 83 (100), 71 (31), 69 (20), 57 (23); HRMS (EI) *m/z* calcd for $C_{16}H_{16}O_2Si$ ([M]$^{+}$) 268.0920, found: 268.0924.

2-(Thiophen-3-yl)-3-(trimethylsilyl)-1*H*-inden-1-one (3g)



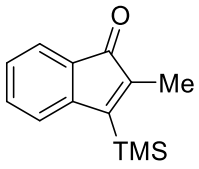
Brown oil (78.8 mg, 55% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.22 (s, 9H), 7.05 (dd, *J* = 5.2 Hz, 1.2 Hz, 1H), 7.20 (t, *J* = 6.0 Hz, 1H), 7.22-7.26 (m, 1H), 7.33-7.39 (m, 2H), 7.48 (d, *J* = 6.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 0.15, 123.2, 123.3, 125.1, 125.5, 128.1, 129.4, 130.1, 133.3, 134.2, 143.1, 148.9, 156.9, 197.7; IR (neat) 1671, 1712, 2359, 2898, 3070, 3107 cm^{-1}; EI-MS *m/z* (relative intensity) 284 ([M$^+$], 100), 269 (88), 151 (76), 211 (16), 209 (12), 165 (18), 139 (28), 83 (31), 73 (27); HRMS (EI) *m/z* calcd for $C_{16}H_{16}OSSi$ ([M]$^+$) 284.0691, found: 284.0686.

3-(Trimethylsilyl)-2-[(trimethylsily)ethynyl]-1*H*-inden-1-one (3h)



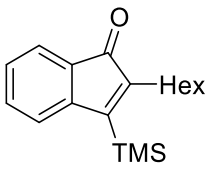
Brown oil (82.1 mg, 55% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.25 (s, 9 H), 0.44 (s, 9 H), 7.20-7.24 (m, 2 H), 7.44 (dt, *J* = 7.2 Hz, 1.2 Hz, 1 H), 7.43-7.45 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ -0.47, -0.16, 98.6, 108.0, 123.6, 123.7, 129.1, 129.2, 130.5, 134.5, 148.2, 165.4, 194.5; IR (neat) 1604, 1715, 1724, 2142, 2898, 2960, 3072, 3421 cm^{-1}; EI-MS *m/z* (relative intensity) 298 ([M$^+$], 100), 283 (55), 224 (29), 209 (19), 165 (10), 85 (13), 73 (42); HRMS (EI) *m/z* calcd for $C_{17}H_{22}OSi_2$ ([M]$^+$) 298.1209, found: 298.1208.

2-Methyl-3-(trimethylsilyl)-1*H*-inden-1-one (3i)[1,2]



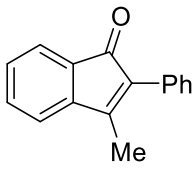
Yellow solid, a mixture of two regio isomers, *d.r.* = 94:6 (87.4 mg, 76% yield); the structures of both regio isomers were determined by ^{1}H NMR and ^{13}C NMR of previous reports.[1,2] Spectrum data of major regio isomer was shown; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.38 (s, 9H), 1.94 (s, 3H), 7.08-7.12 (m, 2H), 7.26-7.31 (m, 1H), 7.38 (d, *J* = 6.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 0.03, 10.5, 122.3, 122.7, 127.3, 130.3, 134.0, 144.1, 149.4, 153.9, 199.6; IR (neat) 1168, 1251, 1457, 1590, 1607, 1710, 2956 cm^{-1}; EI-MS *m/z* (relative intensity) 216([M]$^+$, 32), 201 (42), 176 (82), 149 (90), 133 (100), 105 (84), 104 (92), 77 (67), 76 (64), 57 (30); HRMS (EI) *m/z* calcd for $C_{13}H_{16}OSi$ ([M]$^+$) 216.0970, found: 216.0957.

2-Hexyl-3-(trimethylsilyl)-1*H*-inden-1-one (3j)



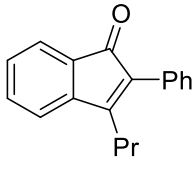
Yellow oil, a mixture of two regio isomers, *d.r.* = 81:19 (44.5 mg, 32% yield); ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.28 (s, 2.06H; Si(CH_3)$_3$ of minor), 0.38 (s, 9H; Si(CH_3)$_3$ of major), 0.86-0.92 (m, 3H; -CH_3 of major + 0.7H; -CH_3 of minor), 1.28-1.45 (m, 6H; -(CH_2)$_3$- of major + 1.4H; -(CH_2)$_3$- of minor), 2.32-2.36 (m, 2H; C-CH_2- of major), 2.64-2.68 (m, 0.5H; C-CH_2- of minor), 7.09-7.13 (m, 2H; Ar-*H* of major + 0.3H; Ar-*H* of minor), 7.27-7.31 (m, 1H; Ar-*H* of major + 0.3H; Ar-*H* of minor), 7.34-7.41 (m, 1H; Ar-*H* of major + 0.3H; Ar-*H* of minor); ^{13}C NMR (100 MHz, $CDCl_3$): major δ 0.21, 14.2, 22.7, 25.2, 29.7, 30.9, 31.8, 122.4, 122.7, 127.3, 130.4, 134.0, 149.1, 149.4, 153.5, 199.6; minor δ 0.14, 14.2, 22.7, 29.22, 29.24, 29.9, 31.8, 119.3, 121.8, 129.0, 132.5, 133.0, 146.7, 149.1, 173.5, 202.5; IR (neat) 1601, 1714, 2928 cm^{-1}; EI-MS *m/z* (relative intensity) 286 ([M]$^+$, 32), 271 (30), 257 (20), 216 (87), 201 (71), 185 (26), 146 (56), 141 (71), 115 (33), 83 (49) 73 (100); HRMS (EI) *m/z* calcd for $C_{18}H_{26}OSi$ 286.1753, found: 286.1737.

3-Methyl-2-phenyl-1*H*-inden-1-one (3k)[1]



Orange solid, a mixture of two regio isomers, *d.r.* = 83:17 (89.6 mg, 81 % yield); m.p. = 62.8-63.9 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.93 (s, 0.6H; -CH_3 of minor), 2.33 (s, 3H; -CH_3 of major), 7.07 (d, *J* = 3.6 Hz, 0.2H; Ar-*H* of minor), 7.17-7.21 (m, 1H; Ar-*H* of major + 0.2H; Ar-*H* of minor), 7.25-7.30 (m, 1H; Ar-*H* of major + 0.2H; Ar-*H* of minor), 7.32-7.37 (m, 1H; Ar-*H* of major), 7.40-7.45 (m, 4H; Ar-*H* of major + 0.8H; Ar-*H* of minor), 7.46-7.54 (m, 2H; Ar-*H* of major + 0.4H; Ar-*H* of minor); ^{13}C NMR (100 MHz, $CDCl_3$): major δ 12.7, 119.6, 122.2, 127.8, 128.4, 129.0, 129.7, 130.5, 131.3, 133.5, 133.7, 146.0, 154.8, 196.5; minor δ 8.7, 120.5, 122.6, 128.1, 128.2, 128.8, 129.3, 131.1, 131.2, 132.8, 133.3, 145.8, 154.8, 198.3; IR (neat) 1708, 2359, 2934, 2962 cm^{-1}; EI-MS *m/z* (relative intensity) 220 ([M$^+$], 100), 219 (37), 191 (43), 165 (18), 83 (29); HRMS (EI) *m/z* calcd for $C_{16}H_{12}O$ 220.0888, found:220.0888. **3k** was known in literature.[1]

2-Phenyl-3-propyl-1*H*-inden-1-one(3l)

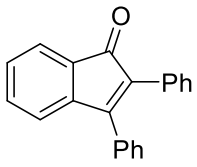


Orange oil; a mixture of two regio isomers, *d.r.* = 87:13 (89.4 mg, 72 % yield); Spectrum data of major regio isomer (**3l**) was shown; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.03 (t, *J* = 7.2 Hz, 3H), 1.70-1.76 (m, 2 H), 2.66-2.71 (m, 2 H), 7.19 (d, *J* = 7.2 Hz, 1 H), 7.25-7.28 (m, 1 H), 7.33-7.48 (m, 6 H), 7.50 (d, *J* = 7.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.6, 21.8, 28.9, 120.0, 122.4, 127.8, 128.4, 128.8, 129.5, 130.8, 131.4, 133.5, 133.6, 145.3, 158.8, 196.9; IR (neat) 1666, 2250, 3399

cm^{-1}; EI-MS *m/z* (relative intensity) 248 ([M^+], 100), 219 (55), 189 (24), 165 (12), 115 (12), 83 (27), 73 (9), 57 (10); HRMS (EI) *m/z* calcd for $C_{18}H_{16}O$ 248.1201, found: 248.1201.

Spectrum data of minor regio isomer (**3l'**) was shown; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.89 (t, *J* = 7.6 Hz, 3H), 1.51-1.56 (m, 2 H), 2.32 (t, *J* = 7.6 Hz, 2H), 7.00 (d, *J* = 7.2 Hz, 1H), 7.18-7.22 (m, 1H), 7.26-7.31 (m, 1H), 7.43-7.52 (m, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.4, 22.8, 25.5, 120.6, 122.6, 127.9, 128.3, 128.9, 129.2, 131.1, 133.0, 133.3, 135.5, 146.0, 155.3, 198.5; IR (neat) 1456, 1704, 2849, 2917, 2961, 3020 cm^{-1}; EI-MS *m/z* (relative intensity) 248 ([M^+], 70), 219 (100), 189 (44), 165 (27), 149 (13) ; HRMS (EI) *m/z* calcd for $C_{18}H_{16}O$ 248.1201, found: 248.1201.

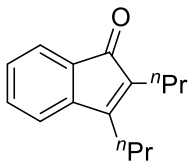
2,3-Diphenyl-1*H*-inden-1-one (3m)[1]



Orange solid (138.3 mg, 98% yield); m.p. = 152.1-152.4 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.15 (d, *J* = 7.2 Hz, 2H), 7.20-7.31 (m, 5H), 7.32-7.42 (m, 6H), 7.59 (d, *J* = 6.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 121.4, 123.1, 127.9, 128.2, 128.6, 128.9, 129.1, 129.4, 130.1, 130.87, 130.89, 132.5, 132.9, 133.6, 145.4, 155.5, 196.7. **3m** was known in literature.[1]

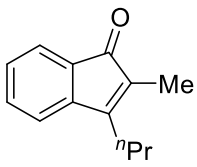
3n is a mixture of **2-(4-methoxyphenyl)-3-phenyl-1*H*-inden-1-one (5a)** and **3-(4-methoxyphenyl)-2-phenyl-1*H*-inden-1-one (5d)**. See the spectrum data of **5a** and **5d**.

2,3-Dipropyl-1*H*-inden-1-one (3o) [1]



Yellow oil (100.6 mg, 94% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.94 (t, *J* = 7.2 Hz, 3H), 1.03 (t, *J* = 7.2 Hz, 3H), 1.49 (sexted, *J* = 7.2 Hz, 2H), 1.65 (sexted, *J* = 7.2 Hz, 2H), 2.24 (t, *J* = 7.0 Hz, 2H), 2.52 (t, *J* = 7.0 Hz, 2H), 7.03 (d, *J* = 6.8 Hz, 1H), 7.12-7.18 (m, 1H), 7.28-7.34 (m, 1H), 7.37 (d, *J* = 6.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.3, 14.5, 21.4, 22.7, 25.0, 28.3, 119.1, 121.7, 128.0, 131.2, 133.2, 135.0, 145.8, 157.8, 198.6. **3o** was known in literature.[1]

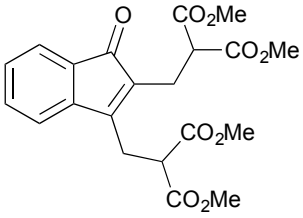
2-Metyl-3-propyl-1*H*-inden-1-one (3p)[2,3]



Yellow oil (81.0 mg, 87% yield), a mixture of two regio isomers, *d.r.* = 59:41; the structures of both regio isomers were determined by ^{1}H NMR and ^{13}C NMR of previous reports. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.92 (t, *J* = 7.2 Hz, 2.3H; -CH_2-C*H*$_3$ of minor), 1.01 (t, *J* = 7.4 Hz, 3H; -CH_2-C*H*$_3$ of major), 1.46-1.55 (m, 1.3H; -C*H*$_2$-CH_3 of minor), 1.62-1.67 (m, 2H; -C*H*$_2$-CH_3 of major), 1.81 (s, 3H; -C*H*$_3$ of major), 2.12 (s, 2.1H; -C*H*$_3$ of minor), 2.26 (t, *J* = 7.6 Hz, 1.4H; -C*H*$_2$-CH_2-CH_3

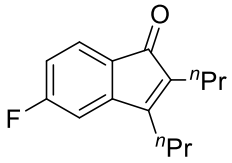
of minor), 2.52 (t, *J* = 7.4 Hz, 2H; -C*H*$_2$-CH$_2$-CH$_3$ of major), 7.00-7.03 (m, 1H; Ar-*H* of major + 0.7H; Ar-*H* of minor), 7.12-7.19 (m, 1H; Ar-*H* of major + 0.7H; Ar-*H* of minor), 7.26- 7.38 (m, 2H; Ar-*H* of major + 1.4H; Ar-*H* of minor); ^{13}C NMR (100 MHz, $CDCl_3$) major δ 7.7, 14.3, 20.9, 28.1, 118.9, 121.7, 127.8, 130.7, 133.2, 135.0, 145.8, 157.6, 198.6; minor δ 11.5, 14.1, 22.3, 24.7, 118.6, 121.5, 128.1, 130.9, 131.2, 135.0, 146.3, 154.1, 198.2; IR (neat) 1456, 1607, 1628, 2871, 2933, 2962, 3071 cm^{-1}; EI-MS *m/z* (relative intensity); 186 ([M]$^+$, 100), 178 (62), 149 (53), 85 (45), 71 (57), 57 (52); HRMS (EI) *m/z* calcd for $C_{13}H_{14}O$ ([M]$^+$) 186.1045, found: 186.1022. **3p** was known in literature.[2]

Tetramethyl 2,2'-[(1-oxo-1*H*-indene-2,3-diyl)bis(methylene)]dimalonate (3q)



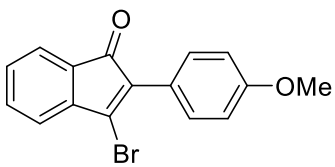
Yellow oil (207.2 mg, 99% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.93 (d, *J* = 8.0 Hz, 2H), 3.23 (d, *J* = 7.6 Hz, 2H), 3.72 (s, 6H), 3.73 (s, 6H), 3.73-3.78 (m, 1H), 3.89 (t, *J* = 7.4 Hz, 1H), 7.08 (d, *J* = 6.8 Hz, 1H), 7.21 (t, *J* = 6.8 Hz, 1H), 7.36 (dt, *J* = 6.8 Hz, 1.2 Hz, 1H), 7.4 (d, *J* = 6.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.9, 25.2, 49.6, 49.9, 52.7, 53.0, 119.6, 122.5, 128.9, 130.4, 132.3, 133.8, 144.1, 155.2, 168.8, 169.2, 197.2; IR (neat) 1456, 1605, 1627, 1748, 2258, 2846, 3401, 3469, 3646 cm^{-1}; EI-MS *m/z* (relative intensity) 418 ([M$^+$], 3), 386 (10), 354 (49), 262 (20), 254 (24), 223 (100), 207 (7), 195 (12), 167 (17), 139 (15), 115 (15), 59 (40).

5-Fluoro-2,3-dipropyl-1*H*-inden-1-one (3r)



Yellow oil (21.0 mg, 18% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.94 (t, *J* = 7.2 Hz, 3H), 1.03 (t, *J* = 7.2 Hz, 3H), 1.49 (sexted, *J* = 7.2 Hz, 2H), 1.63 (sexted, *J* = 7.2 Hz, 2H), 2.24 (t, *J* = 7.6 Hz, 2H), 2.49 (t, *J* = 7.6 Hz, 2H), 6.73-6.81 (m, 2H), 7.34 (dd, *J* = 7.6 Hz, 5.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.3, 14.5, 21.3, 22.6, 25.2, 28.3, 108.2 ($J_{C\text{-}F}$ = 24.8 Hz), 113.2 ($J_{C\text{-}F}$ = 22.9 Hz), 123.5 ($J_{C\text{-}F}$ = 9.6 Hz), 127.0, 136.6, 149.2 ($J_{C\text{-}F}$ = 9.5 Hz), 155.5 ($J_{C\text{-}F}$ = 1.9 Hz), 166.7 ($J_{C\text{-}F}$ = 251.1 Hz), 196.9; IR (neat) 1471, 1597, 1613, 1713, 2872, 2933, 2961 cm^{-1}; EI-MS *m/z* (relative intensity) 232 ([M$^+$], 60), 205 (19), 203 (100), 189 (30), 175 (35), 161 (52), 146 (19), 133 (30).

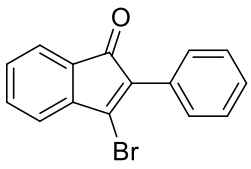
3-Bromo-2-(4-methoxyphenyl)-1*H*-inden-1-one (4a)



Red solid (50.5 mg, 82% yield); m.p. = 147.8-148.2 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.86 (s, 3H), 7.00 (d, *J* = 8.8 Hz, 2H), 7.26-7.32 (m, 2H), 7.45-7.49 (m, 2H), 7.68 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.4, 113.9, 121.3, 122.25,

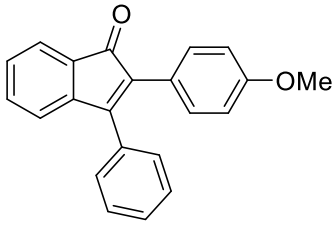
122.31, 129.7, 129.9, 131.0, 134.2, 134.4, 140.5, 143.7, 160.1, 193.0; IR (neat) 1715, 2360 cm^{-1}; EI-MS *m/z* (relative intensity) 314 ([M^+], 56), 235 (100), 207 (21), 163 (17), 127 (17), 85 (25), 71 (28), 69 (18); HRMS (EI) *m/z* calcd for $C_{16}H_{11}BrO_2$ 313.9942, found: 313.9942

3-Bromo-2-phenyl-1*H*-inden-1-one (4d)



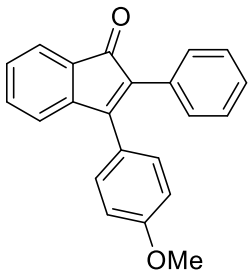
Yellow oil (48.0 mg, 94% yield); ^1H NMR (400 MHz, $CDCl_3$) δ 7.26-7.41 (m, 2H), 7.42-7.52 (m, 5H), 7.68 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 121.6, 122.4, 128.4, 128.9, 129.6, 129.8, 129.9, 130.1, 134.2, 135.0, 142.3, 143.5, 192.6; IR (neat) 1464, 1539, 2852, 2922 cm^{-1}; EI-MS *m/z* (relative intensity) 284 ([M^+], 30), 248 (52), 205 (100), 176 (39), 149 (39), 127 (17), 89 (34), 83 (34), 57 (31); HRMS (EI) *m/z* calcd for $C_{15}H_9BrO$ 283.9837, found: 283.9834.

2-(4-Methoxyphenyl)-3-phenyl-1*H*-inden-1-one (5a)[4]



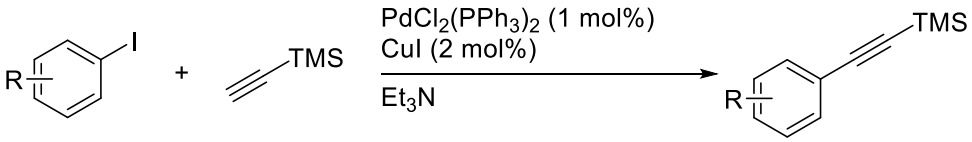
Red solid (46.6 mg, 93% yield); m.p. = 116.3-116.9 ºC; ^1H NMR (400 MHz, $CDCl_3$) δ 3.79 (s, 3H), 6.80 (d, *J* = 9.2 Hz, 2H), 7.11 (d, *J* = 7.2 Hz, 1H), 7.21-7.28 (m, 3H), 7.34-7.45 (m, 6H), 7.56 (d, *J* = 6.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.3, 113.8, 121.1, 123.0, 123.2, 128.6, 128.8, 128.9, 129.2, 130.8, 131.4, 132.0, 133.1, 133.6, 145.7, 153.9, 159.3, 197.1; IR (neat) 1606, 1711 cm^{-1}; EI-MS *m/z* (relative intensity) 312 ([M^+], 53), 295 (22), 281 (20), 239 (17), 221 (39), 207 (12), 167 (37), 149 (100), 113 (29); HRMS (EI) *m/z* calcd for $C_{22}H_{16}O_2$ 312.1150, found:312.1244. **5a** was known in literature.[3]

3-(4-Methoxyphenyl)-2-phenyl-1*H*-inden-1-one (5d)[5]



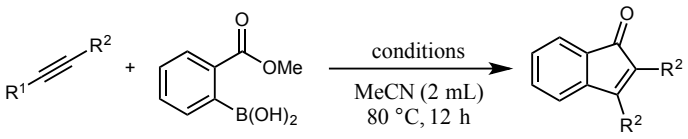
Red solid (46.5 mg, 88% yield); m.p. = 119.7-120.4 ºC; ^1H NMR (400 MHz, $CDCl_3$) δ 3.86 (s, 3H), 6.93 (d, *J* = 8.8 Hz, 2H), 7.20 (d, *J* = 7.6 Hz, 1H), 7.27-7.31 (m, 6H), 7.33-7.40 (m, 3H), 7.58 (d, *J* = 6.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.4, 114.3, 121.4, 122.9, 124.9, 127.7, 128.2, 129.0, 130.1, 130.3, 131.16, 131.22, 131.8, 133.4, 145.3, 155.3, 160.6, 196.8; IR (neat) 1510, 1605, 1704 cm^{-1}; EI-MS *m/z* (relative intensity) 312 ([M^+], 100), 281 (31), 268 (19), 239 (45), 83 (48), 71 (34), 57 (24); HRMS (EI) *m/z* calcd for $C_{22}H_{16}O_2$ 312.1150, found: 312.1164. **5d** was known in literature.[4]

Experimental Procedure and Spectrum Data for Trimethylsilyl Alkynes



To a mixture of aryl iodide (20 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (0.2 mmol, 0.01 equiv), and copper(I) iodide (0.4 mmol, 0.02 equiv) in 60 mL of trimethylamine, a solution of trimethylsilyl acetylene (30 mmol, 1.5 equiv) was added dropwise. The reaction mixture was stirred at room temperature for 3 h under argon atmosphere. The reaction mixture was then concentrated, and purified by column chromatography on silica gel to afford the corresponding silyl alkynes. Silyl alkynes **1a**-**1j** were known in literature.[6-10]

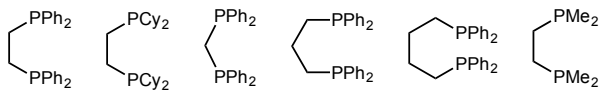
A survey of cobalt catalysts, ligands, and solvents



1 **2** (x equiv) **3**

1o : R^1= *n*-Pr, R^2= *n*-Pr
1i : R^1= TMS, R^2= Me

Entry	**1**	Cobalt (mol %)	Ligand (mol %)	**2** (x equiv)	Yield (%)
1	**1o**	$CoCl_2$ (5)	DPPE (5)[a]	2.0	5
2	**1o**	$Co(OAc)_2•H_2O$ (5)	DPPE (5)	2.0	83
3	**1o**	$Co(acac)_2$ (5)	DPPE (5)	2.0	95
4	**1o**	none	DPPE (5)	2.0	No reaction
5	**1o**	$Co(acac)_2$ (5)	DCyPE (5)[a]	2.0	40
6	**1o**	$Co(acac)_2$ (5)	PPh_3 (10)	2.0	No reaction
7	**1o**	$Co(acac)_2$ (2)	DPPE (2)	2.0	95
8	**1o**	$Co(acac)_2$ (2)	DPPE (2)	1.5	94
9	**1i**	$Co(acac)_2$ (2)	DPPE (2)	1.5	30 (94:6)[b]
10	**1i**	$Co(acac)_2$ (10)	DPPE (10)	1.5	76 (94:6)[b]
11	**1i**	$Co(acac)_2$ (10)	DPPM (10)[a]	1.5	<5 (62:38)[b]
12	**1i**	$Co(acac)_2$ (10)	DPPP(10)[a]	1.5	53 (80:20)[b]
13	**1i**	$Co(acac)_2$ (10)	DPPB (10)[a]	1.5	No reaction
14	**1i**	$Co(acac)_2$ (10)	DMPE (10)[a]	1.5	No reaction
15	**1i**	$Co(acac)_2$ (10)	PPh3 (20)	1.5	No reaction

[a] 

[b] Regioselectivities were determined by NMR analysis.

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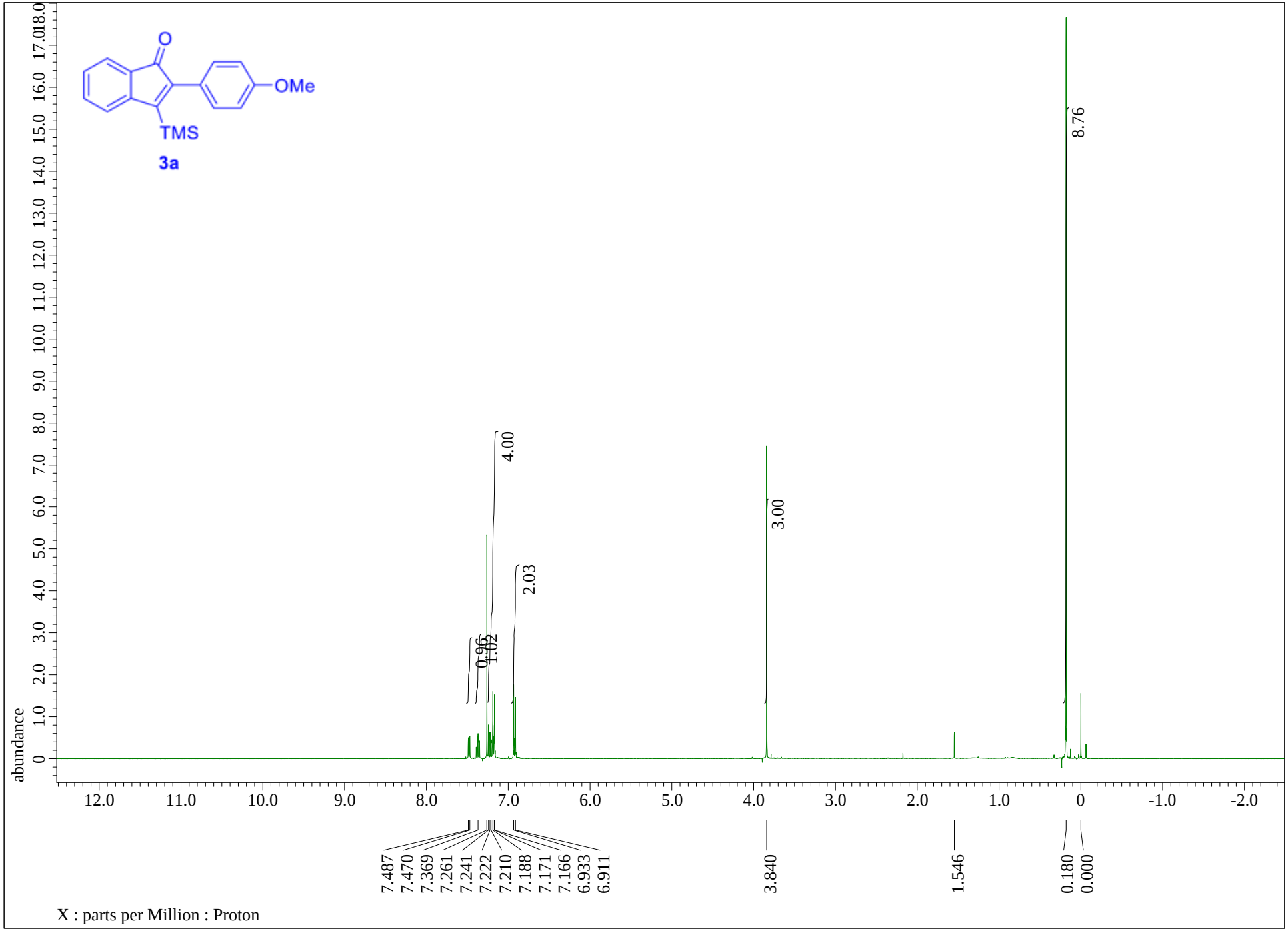
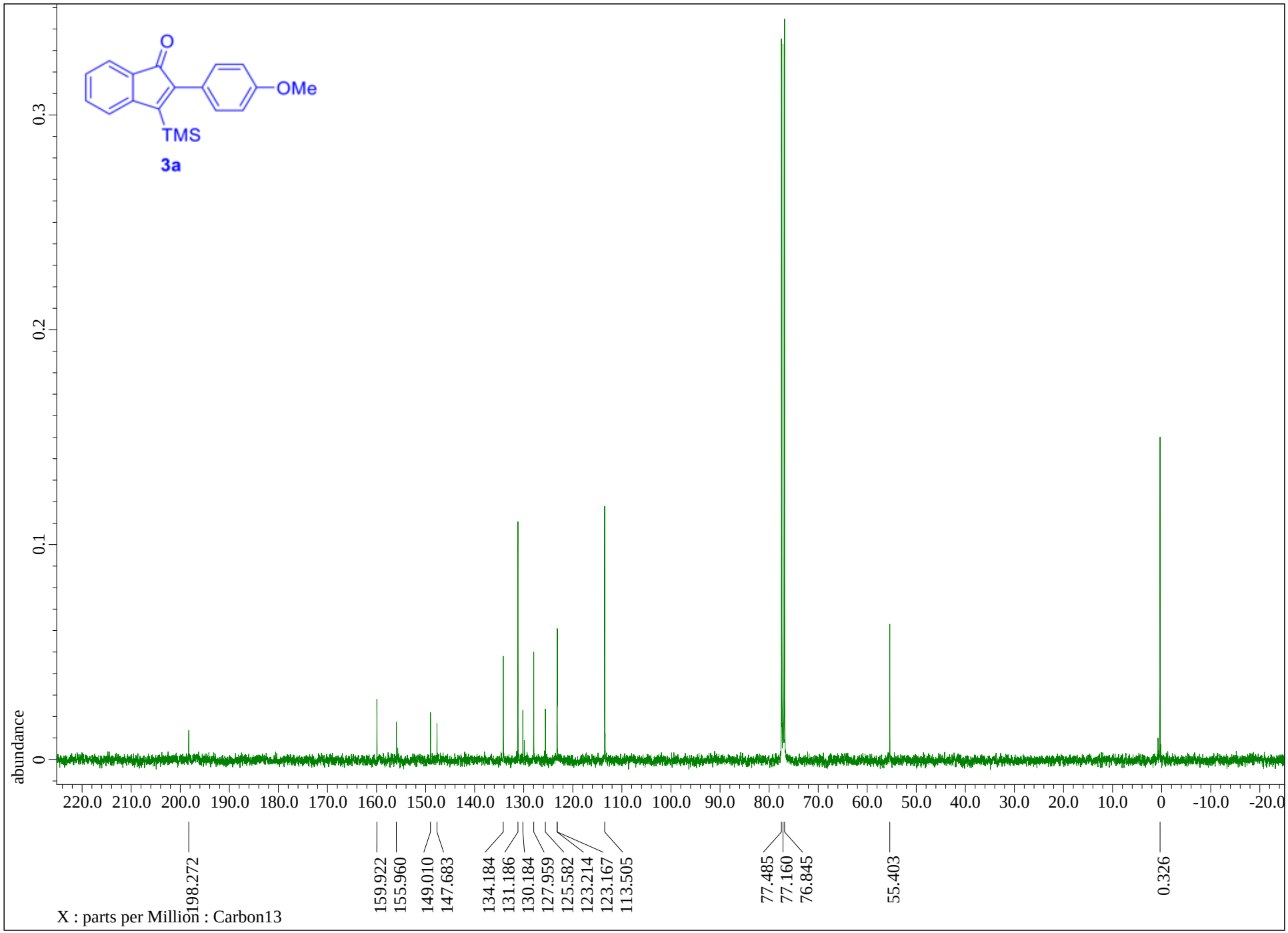
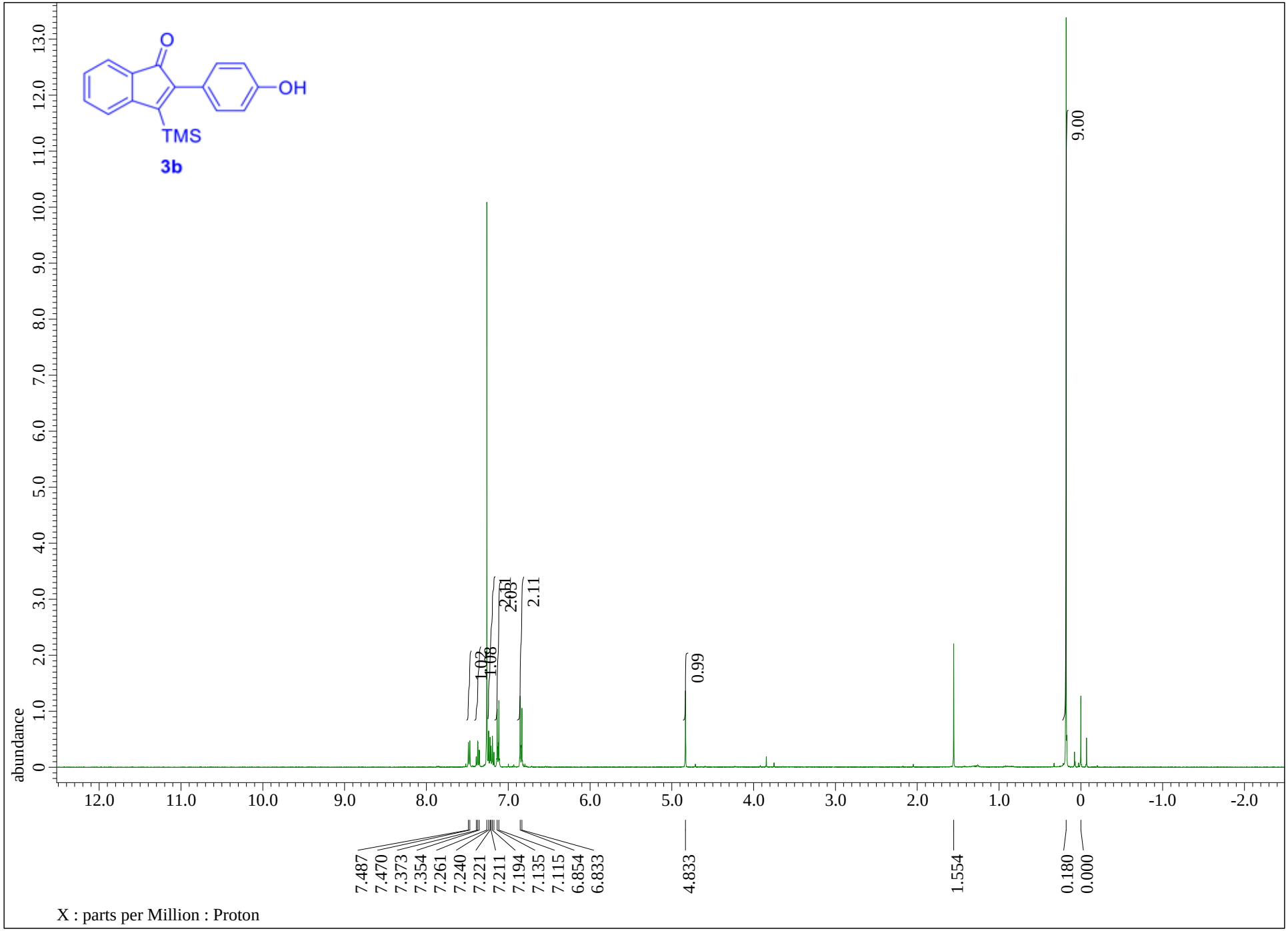
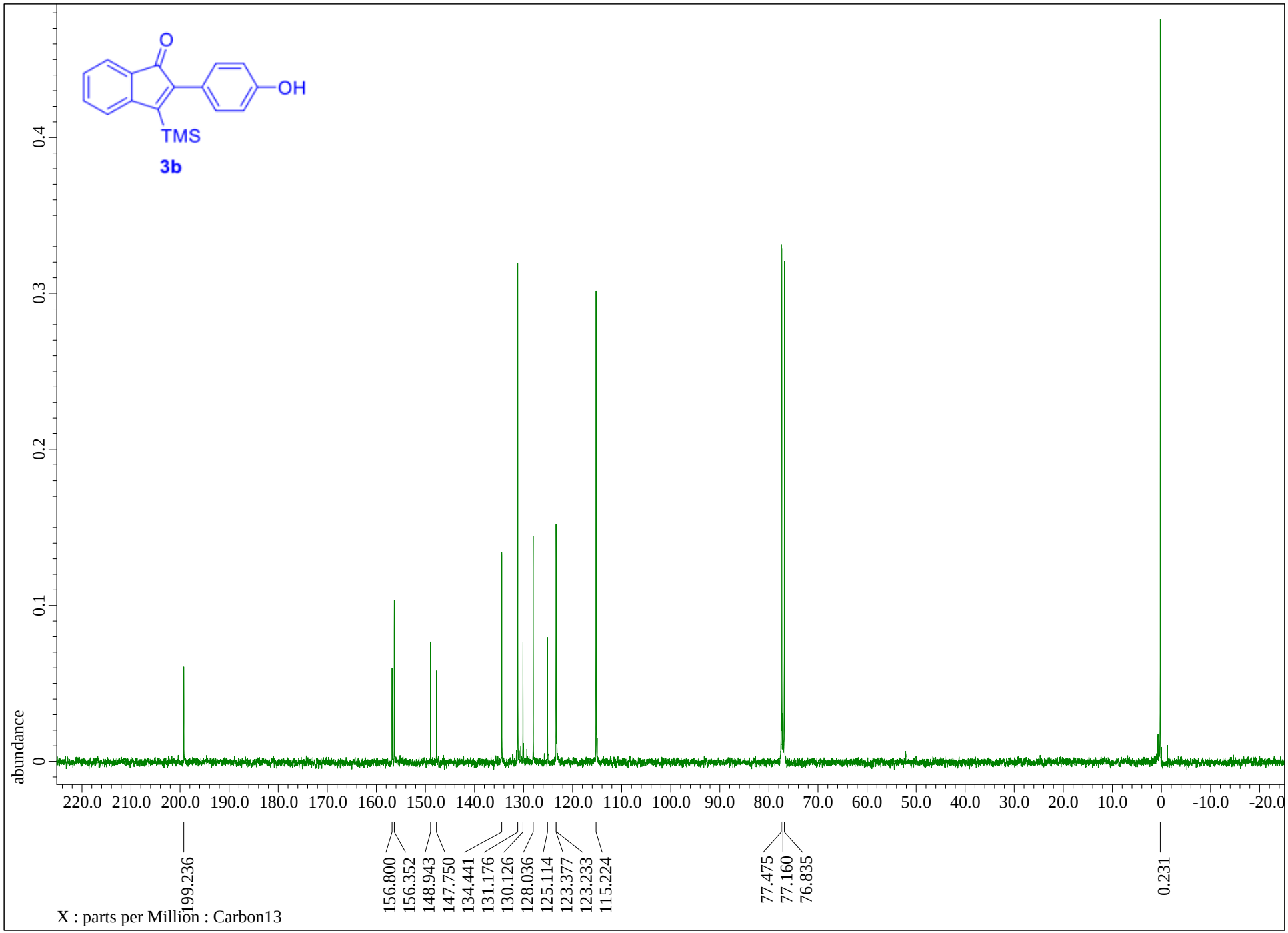

O
OMe
TMS
3a
abundance
8.76
4.00
3.00
2.03
0.96
1.02
7.487
7.470
7.369
7.261
7.241
7.222
7.210
7.188
7.171
7.166
6.933
6.911
3.840
1.546
0.180
0.000
X : parts per Million : Proton


O
OMe
TMS
3a
abundance
0
0.1
0.2
0.3
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
198.272
159.922
155.960
149.010
147.683
134.184
131.186
130.184
127.959
125.582
123.214
123.167
113.505
77.485
77.160
76.845
55.403
0.326
X : parts per Million : Carbon13


O
OH
TMS
3b
9.00
2.11
2.03
2.01
1.02
1.08
0.99
abundance
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
11.0
12.0
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
-2.0
7.487
7.470
7.373
7.354
7.261
7.240
7.221
7.211
7.194
7.135
7.115
6.854
6.833
4.833
1.554
0.180
0.000
X : parts per Million : Proton


O
OH
TMS
3b
abundance
0
0.1
0.2
0.3
0.4
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
199.236
156.800
156.352
148.943
147.750
134.441
131.176
130.126
128.036
125.114
123.377
123.233
115.224
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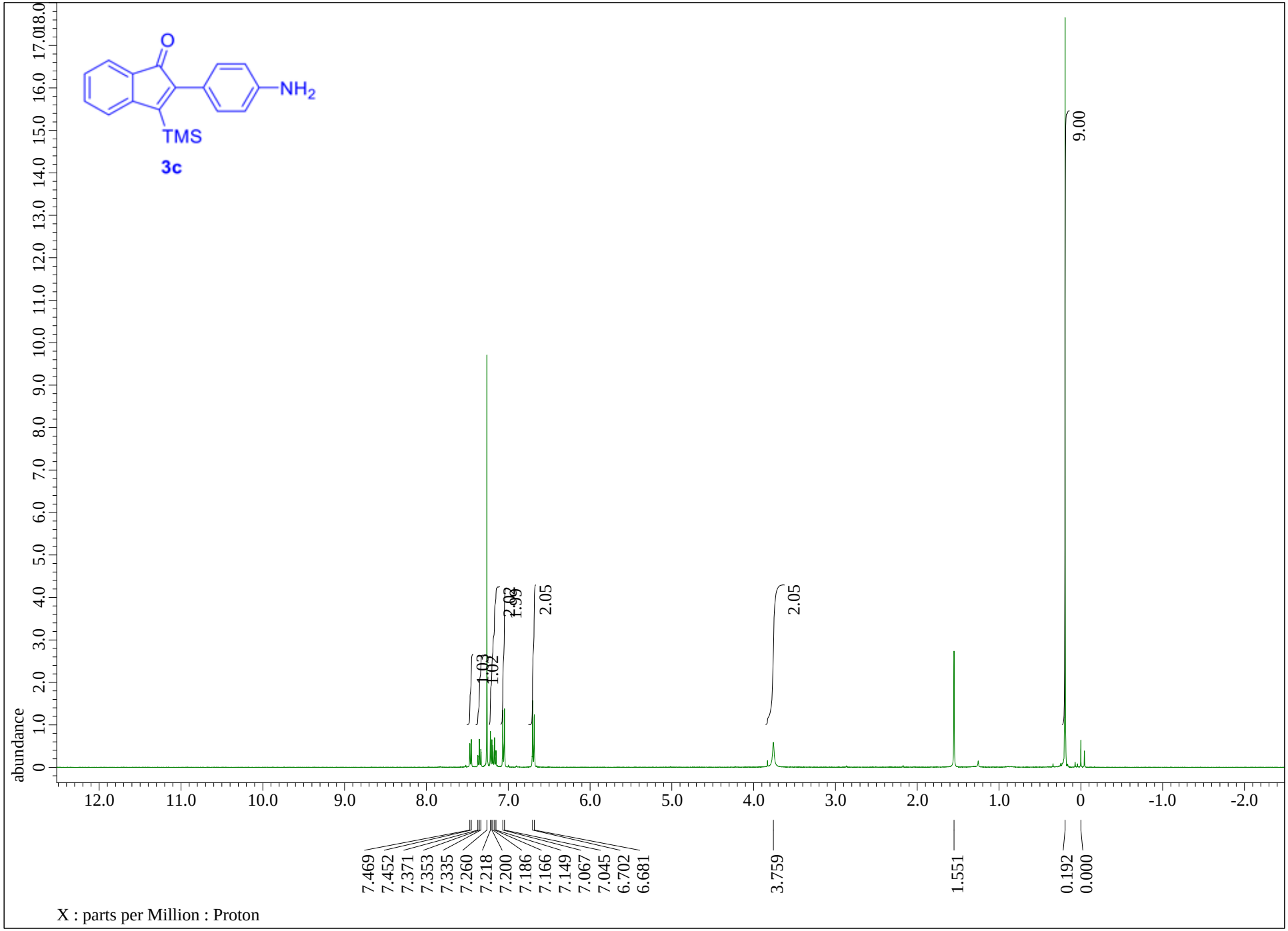
77.160
76.835
0.231
X : parts per Million : Carbon13


O
NH2
TMS
3c
abundance
9.00
2.05
2.05
1.03
1.02
7.469
7.452
7.371
7.353
7.335
7.260
7.218
7.200
7.186
7.166
7.149
7.067
7.045
6.702
6.681
3.759
1.551
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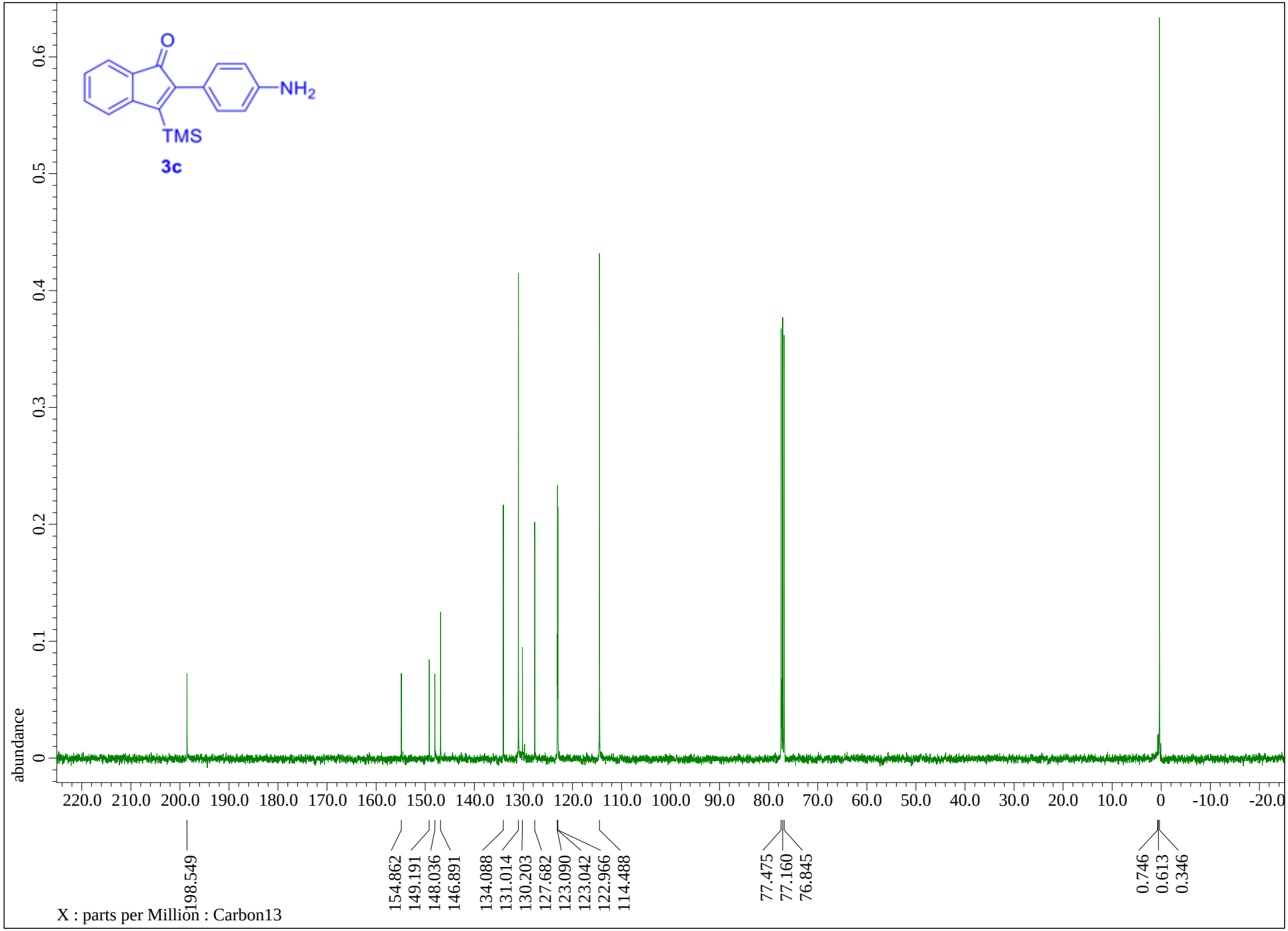
0.000
X : parts per Million : Proton


O
NH2
TMS
3c
abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
198.549
154.862
149.191
148.036
146.891
134.088
131.014
130.203
127.682
123.090
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114.488
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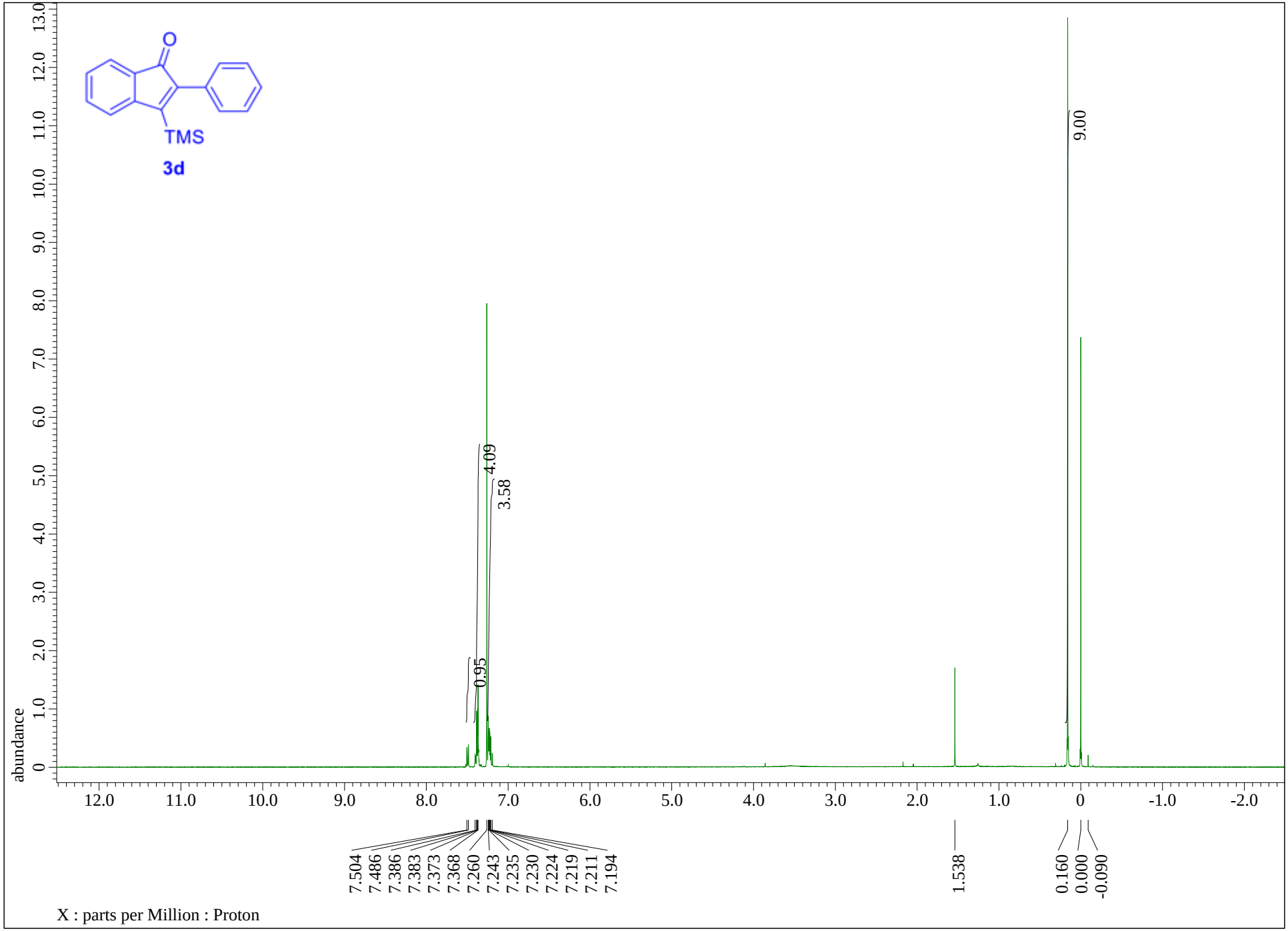
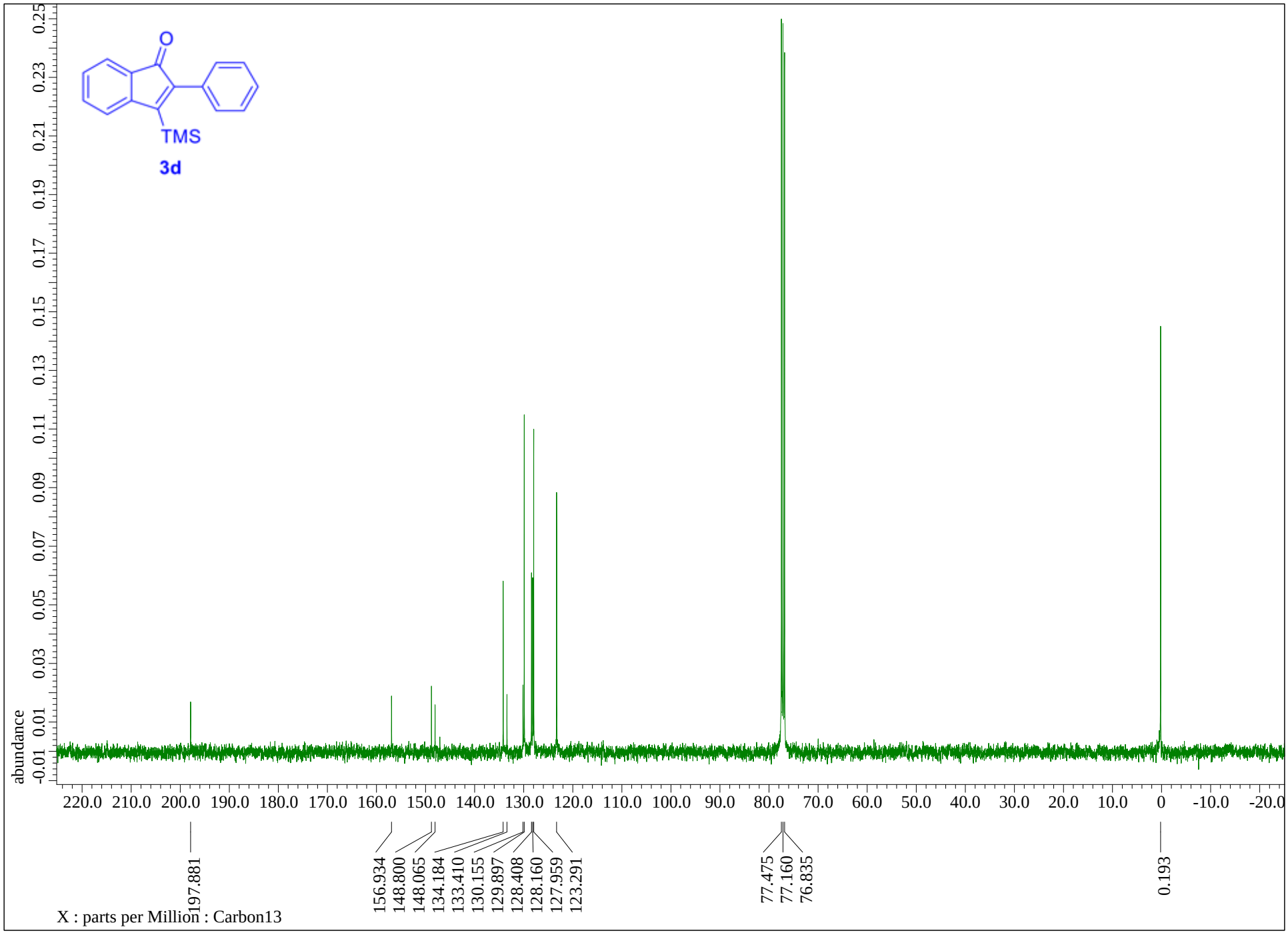
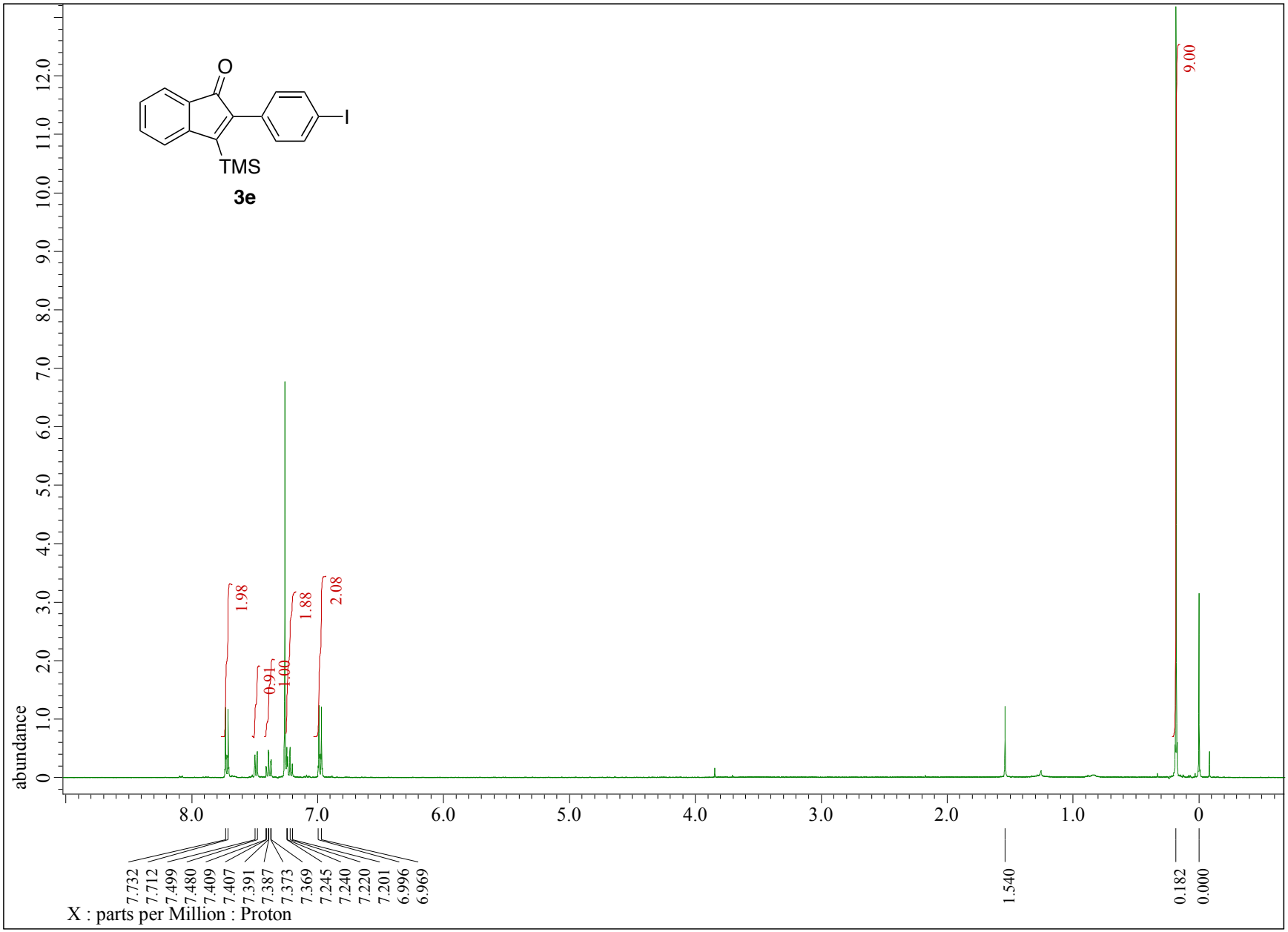
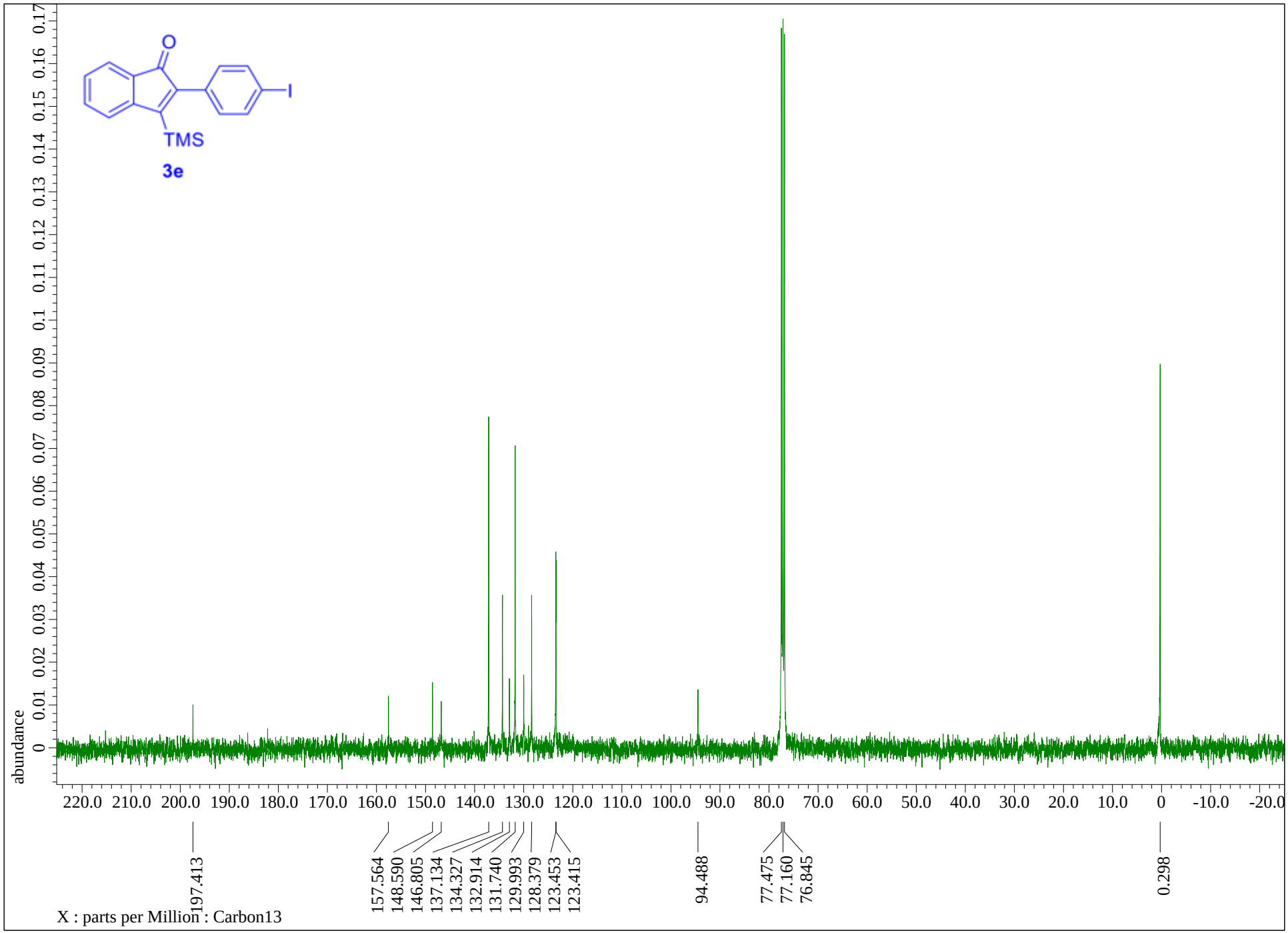
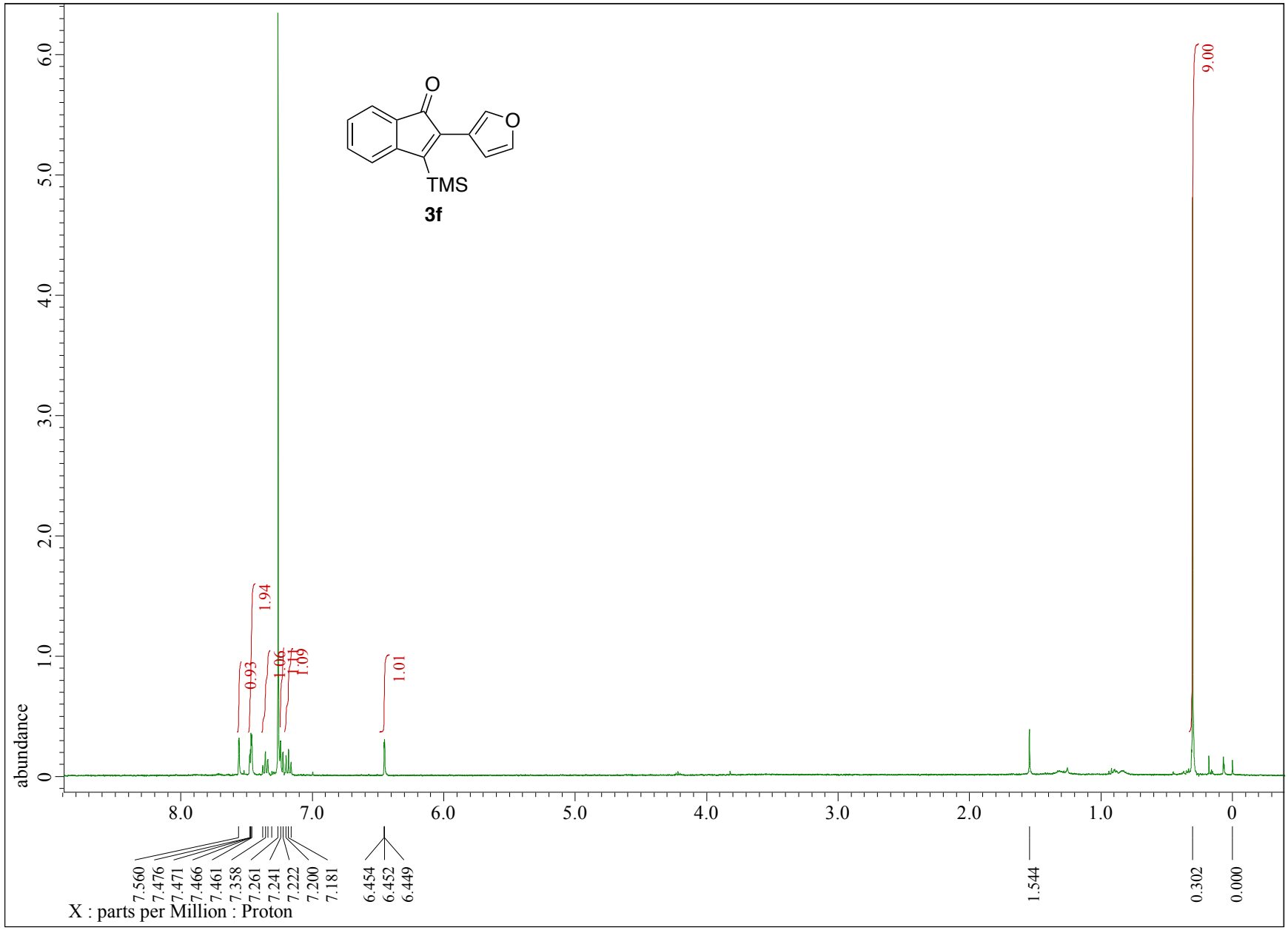
77.160
76.845
0.746
0.613
0.346
X : parts per Million : Carbon13

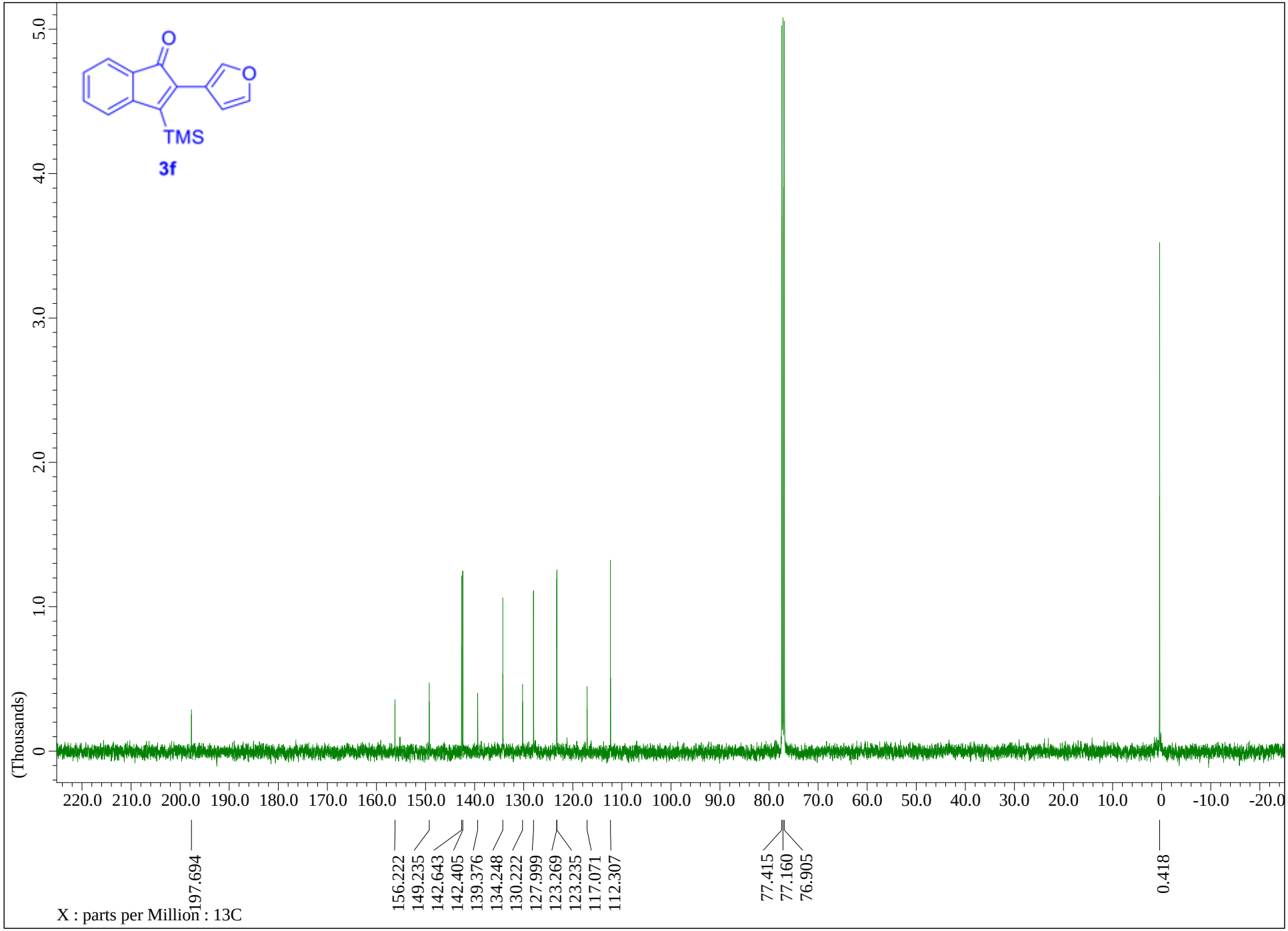

O
TMS
3d
abundance
9.00
4.09
3.58
0.95
7.504
7.486
7.386
7.383
7.373
7.368
7.260
7.243
7.235
7.230
7.224
7.219
7.211
7.194
1.538
0.160
0.000
-0.090
X : parts per Million : Proton

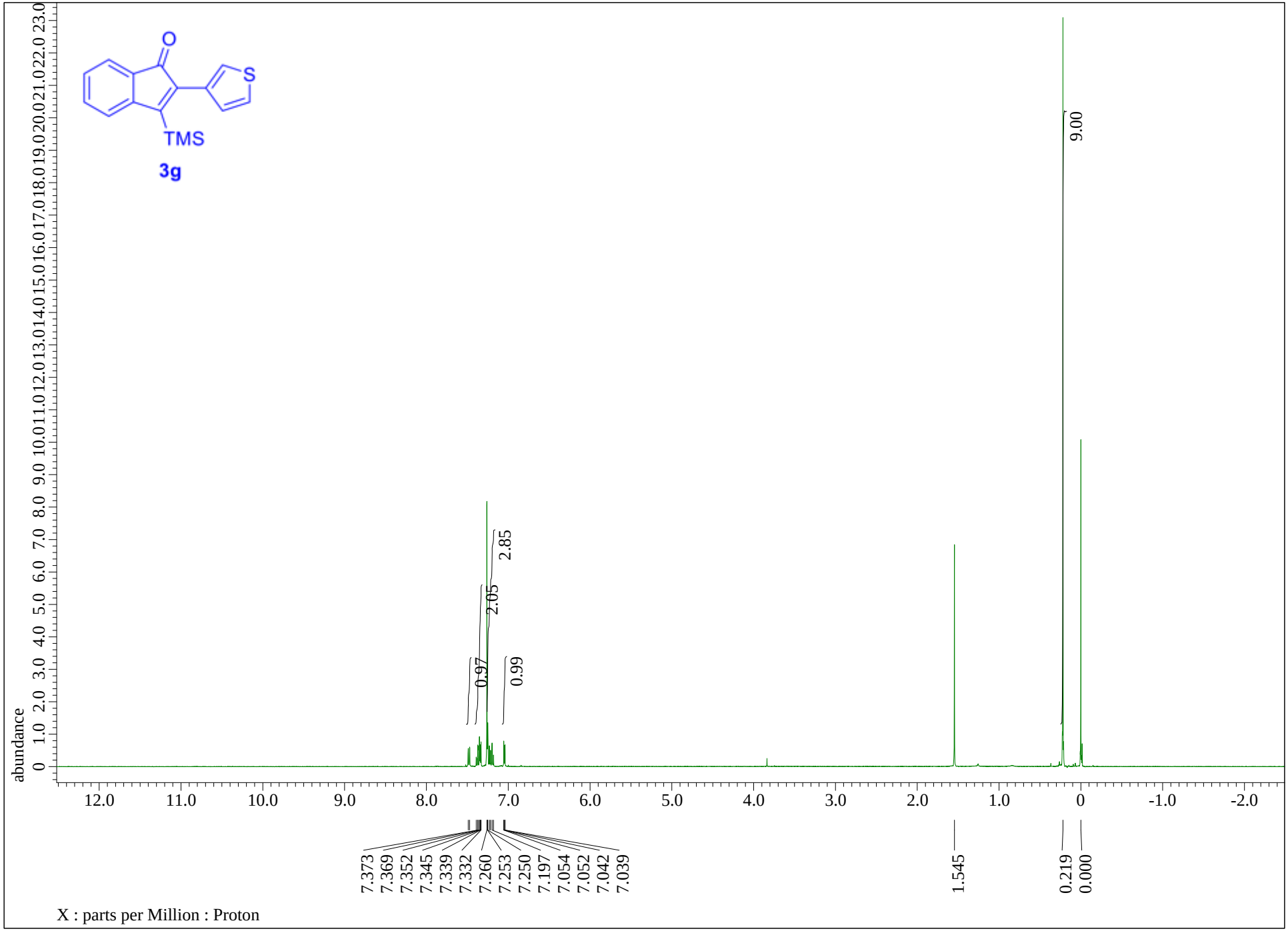

O
TMS
3d
abundance
-0.01
0.01
0.03
0.05
0.07
0.09
0.11
0.13
0.15
0.17
0.19
0.21
0.23
0.25
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
-20.0
197.881
156.934
148.800
148.065
134.184
133.410
130.155
129.897
128.408
128.160
127.959
123.291
77.475
77.160
76.835
0.193
X : parts per Million : Carbon13

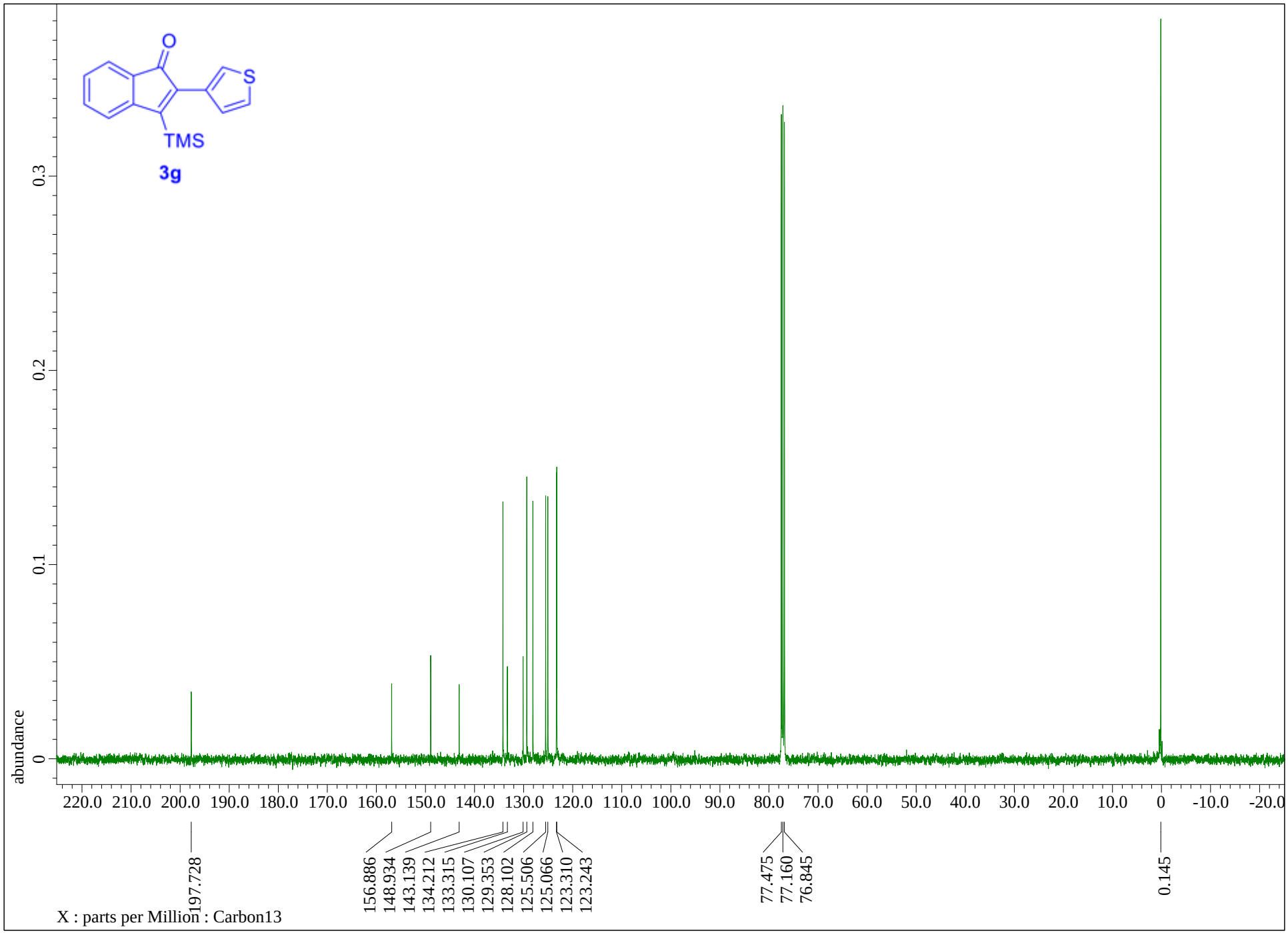

O
I
TMS
3e
abundance
9.00
1.98
0.91
1.00
1.88
2.08
7.732
7.712
7.499
7.480
7.409
7.407
7.391
7.387
7.373
7.369
7.245
7.240
7.220
7.201
6.996
6.969
1.540
0.182
0.000
X : parts per Million : Proton

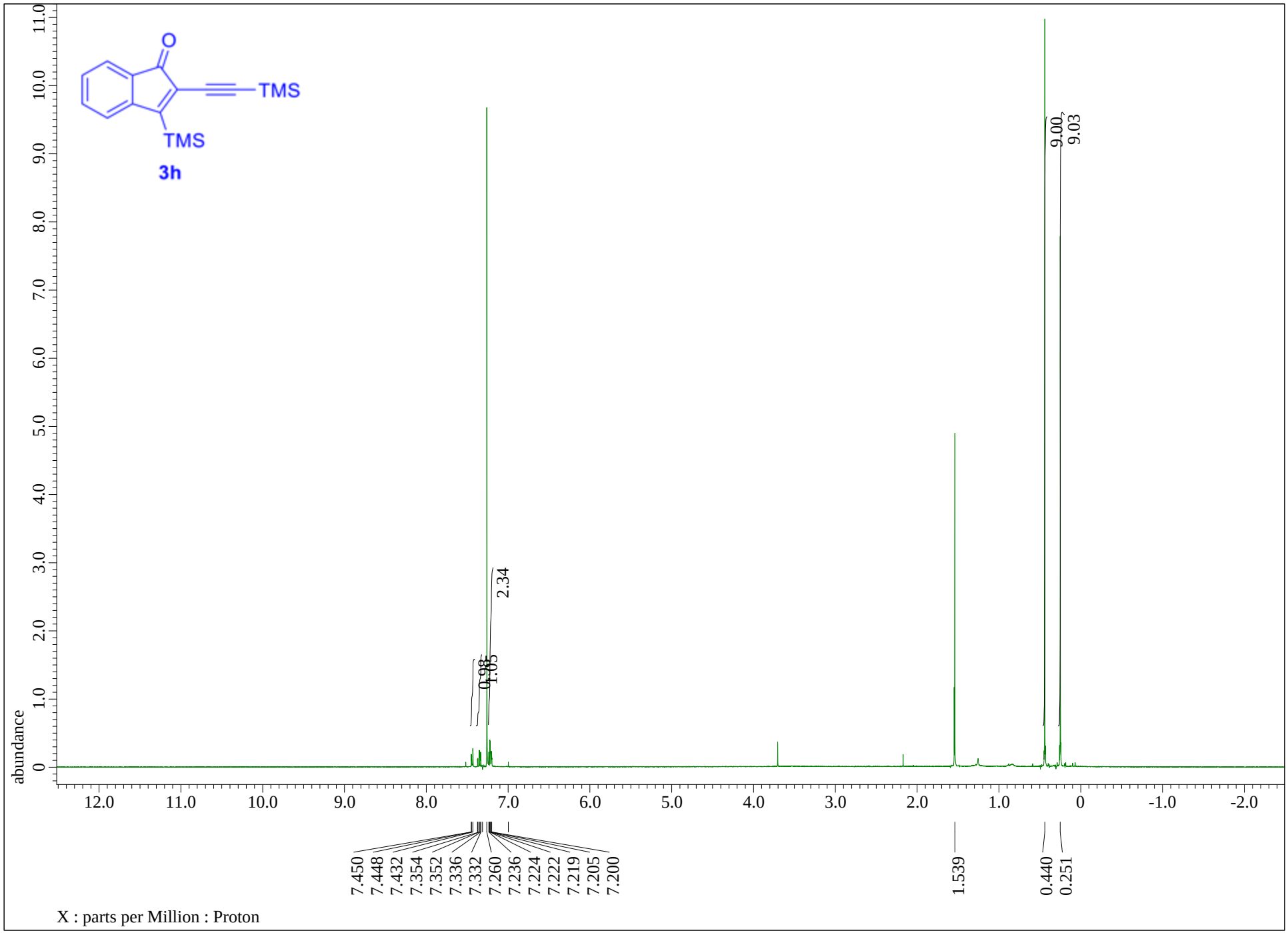

O
I
TMS
3e
abundance
0 0.01 0.02 0.03 0.04 0.05 0.06 0.07 0.08 0.09 0.1 0.11 0.12 0.13 0.14 0.15 0.16 0.17
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
197.413
157.564
148.590
146.805
137.134
134.327
132.914
131.740
129.993
128.379
123.453
123.415
94.488
77.475
77.160
76.845
0.298
X : parts per Million : Carbon13

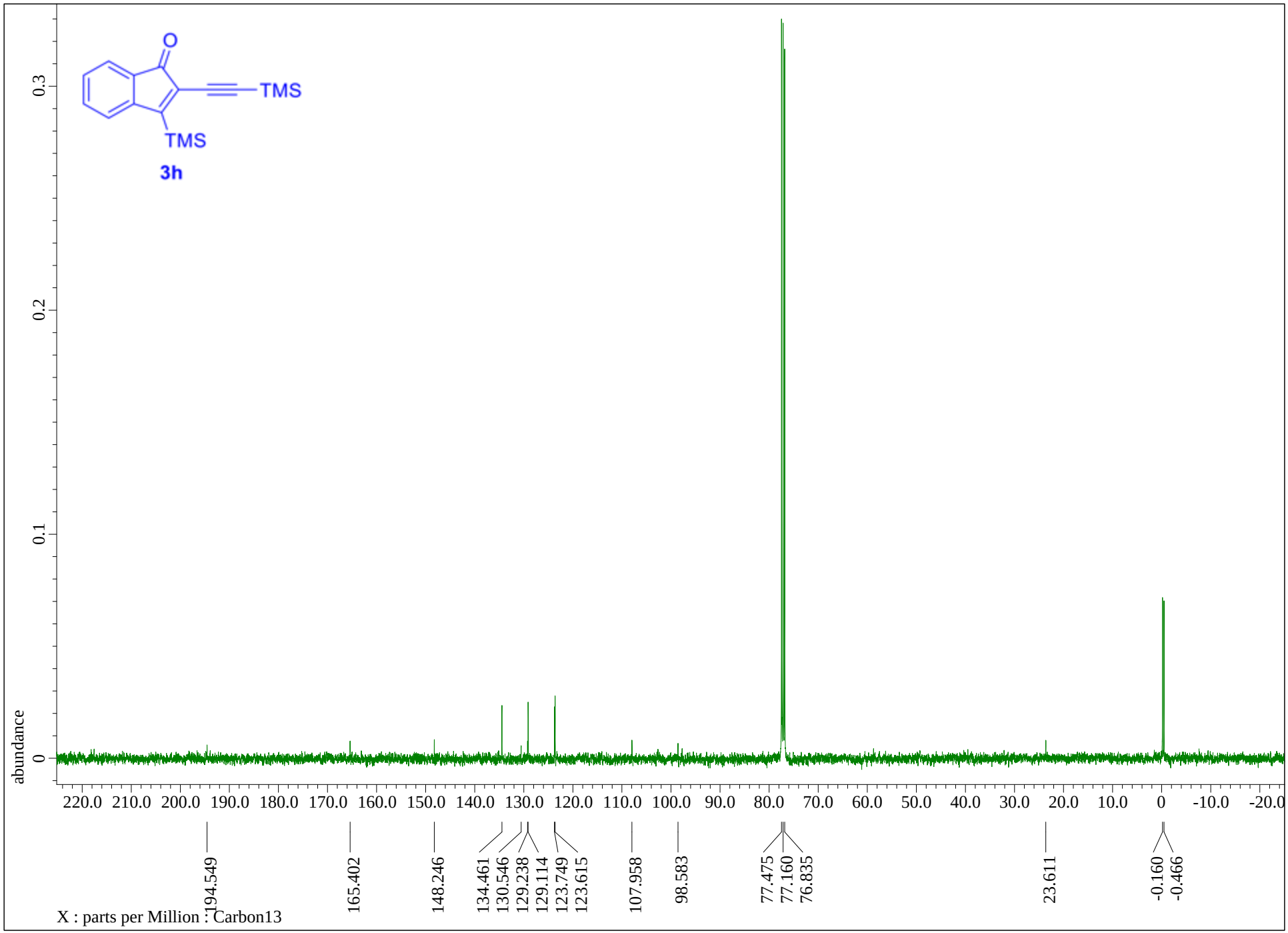

O
O
TMS
3f
9.00
1.94
0.93
1.06
1.04
1.09
1.01
abundance
6.0
5.0
4.0
3.0
2.0
1.0
0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.560
7.476
7.471
7.466
7.461
7.358
7.261
7.241
7.222
7.200
7.181
6.454
6.452
6.449
1.544
0.302
0.000
X : parts per Million : Proton

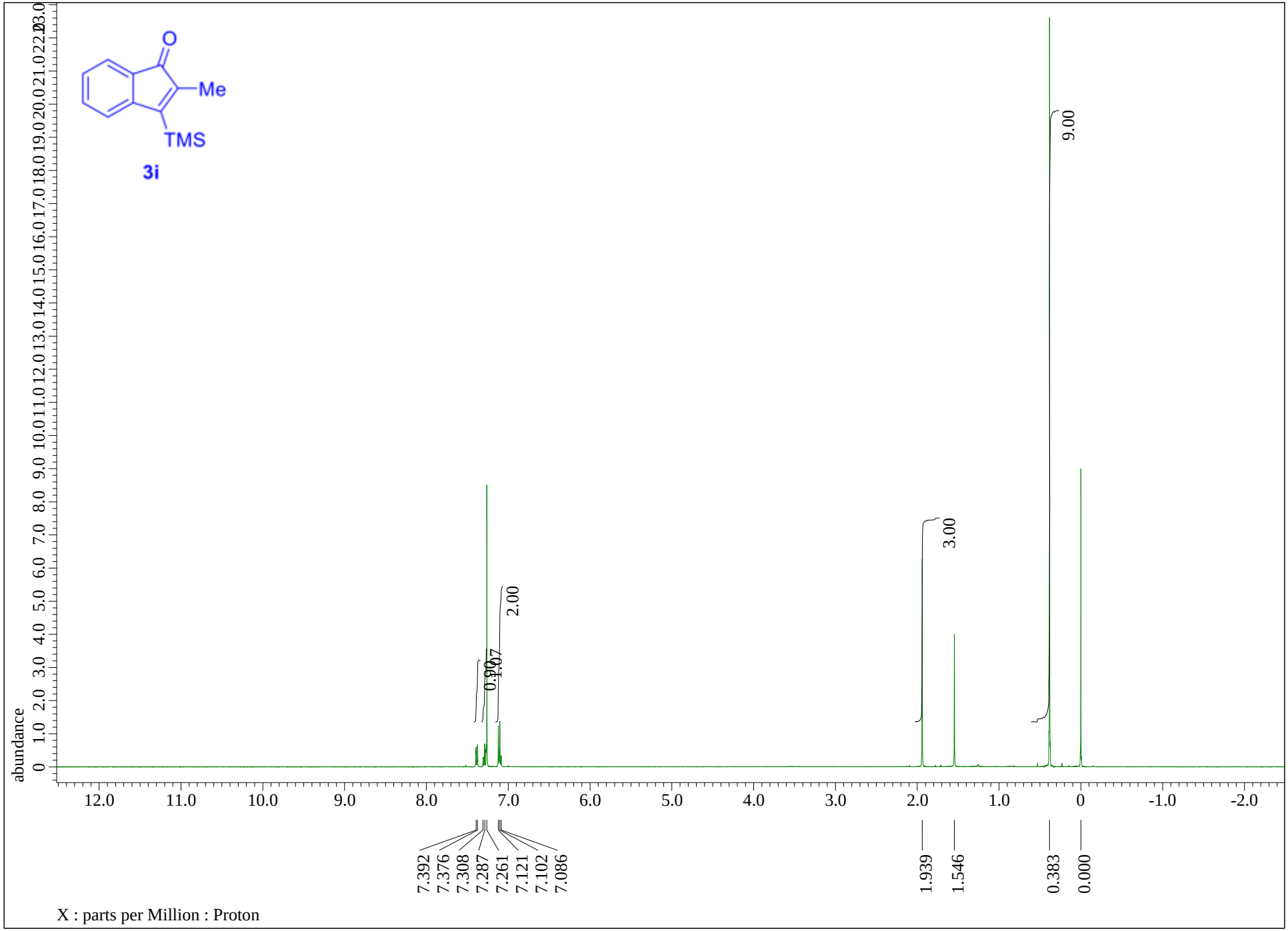

O
O
TMS
3f
(Thousands)
5.0
4.0
3.0
2.0
1.0
0
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
197.694
156.222
149.235
142.643
142.405
139.376
134.248
130.222
127.999
123.269
123.235
117.071
112.307
77.415
77.160
76.905
0.418
X : parts per Million : 13C

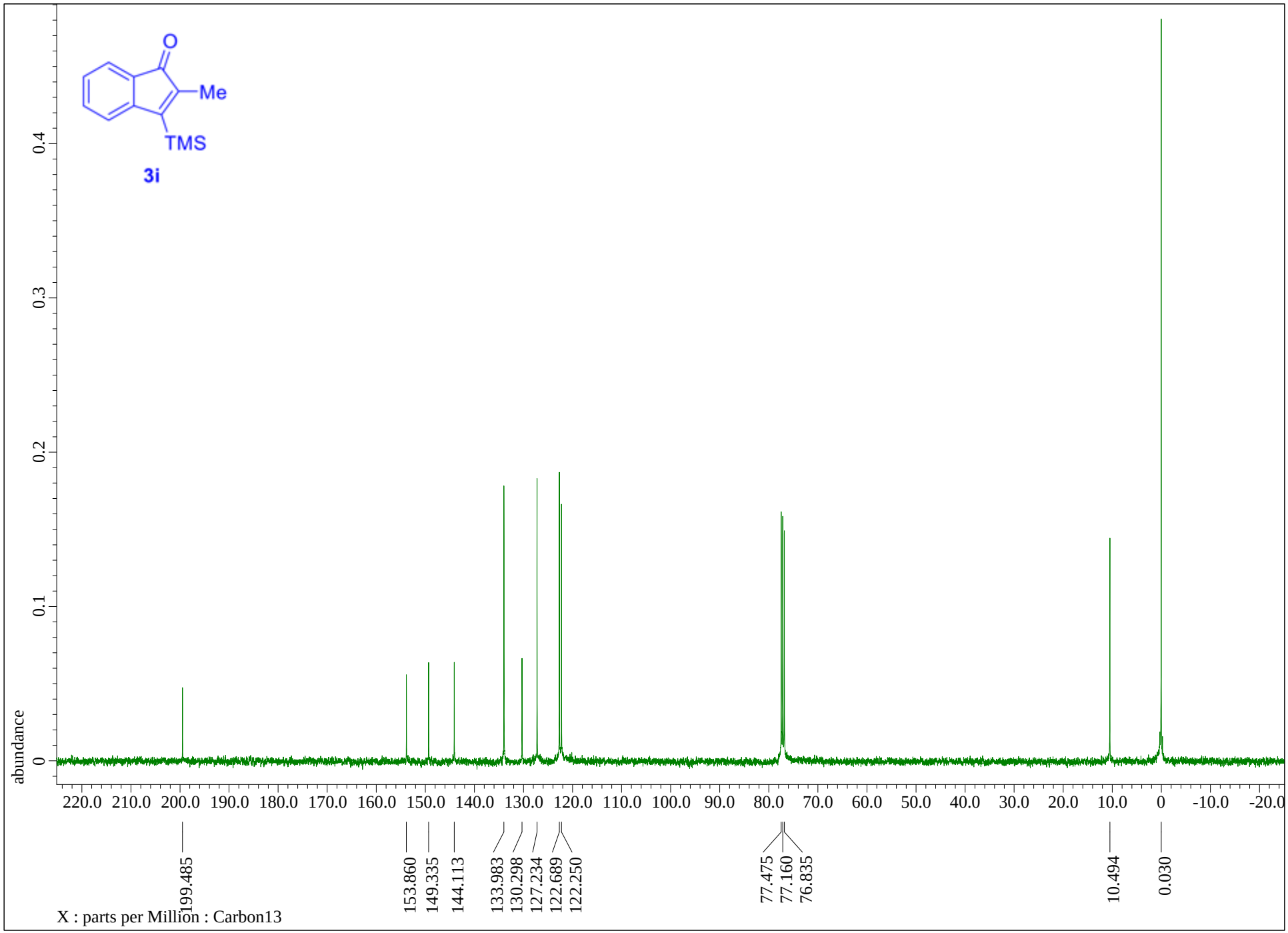

O
S
TMS
3g
9.00
2.85
2.05
0.97
0.99
abundance
0 1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0 9.0 10.0 11.0 12.0 13.0 14.0 15.0 16.0 17.0 18.0 19.0 20.0 21.0 22.0 23.0
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0 -1.0 -2.0
7.373 7.369 7.352 7.345 7.339 7.332 7.260 7.253 7.250 7.197 7.054 7.052 7.042 7.039
1.545
0.219 0.000
X : parts per Million : Proton

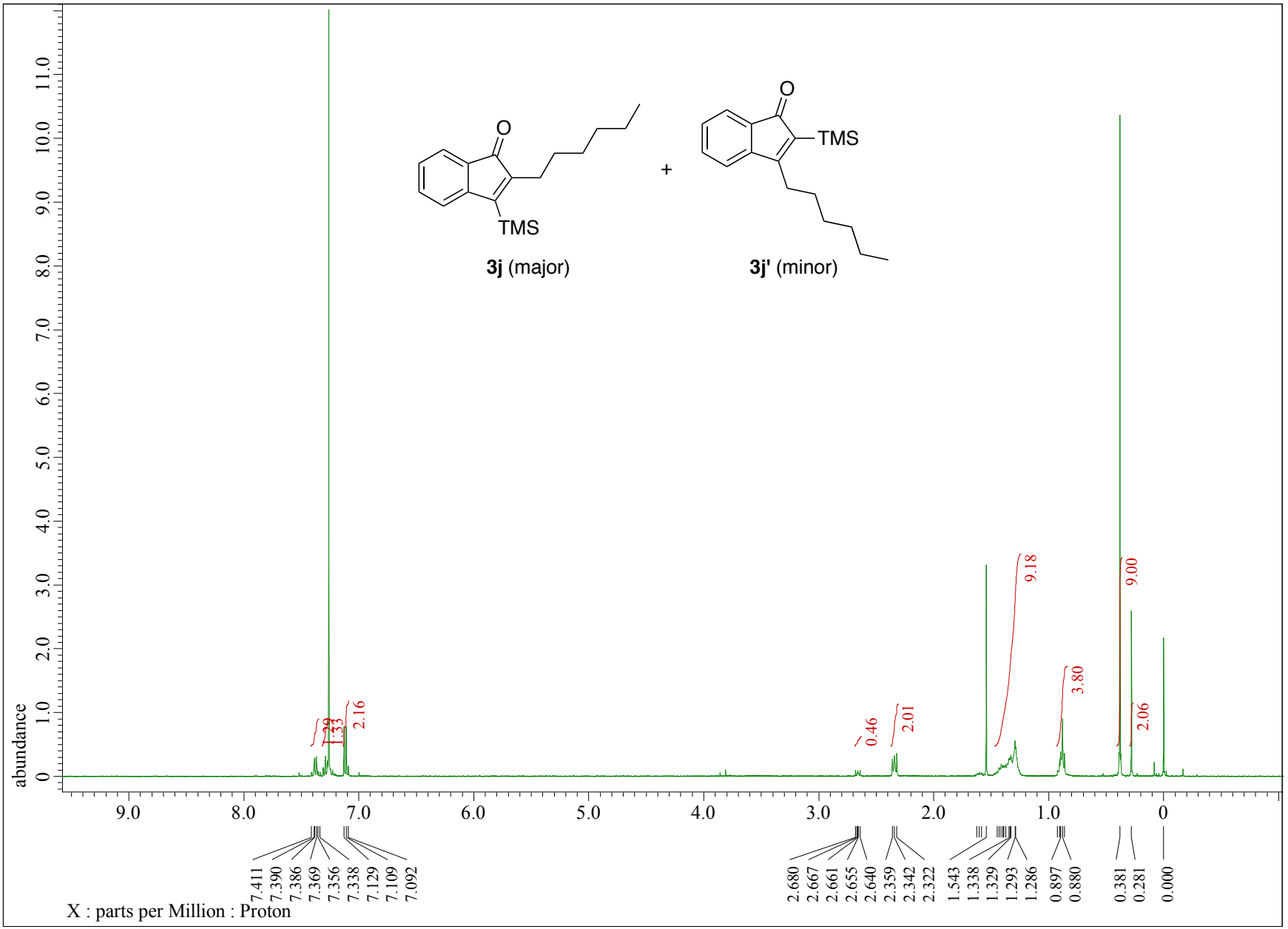

O
S
TMS
3g
abundance
0
0.1
0.2
0.3
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
197.728
156.886
148.934
143.139
134.212
133.315
130.107
129.353
128.102
125.506
125.066
123.310
123.243
77.475
77.160
76.845
0.145
X : parts per Million : Carbon13

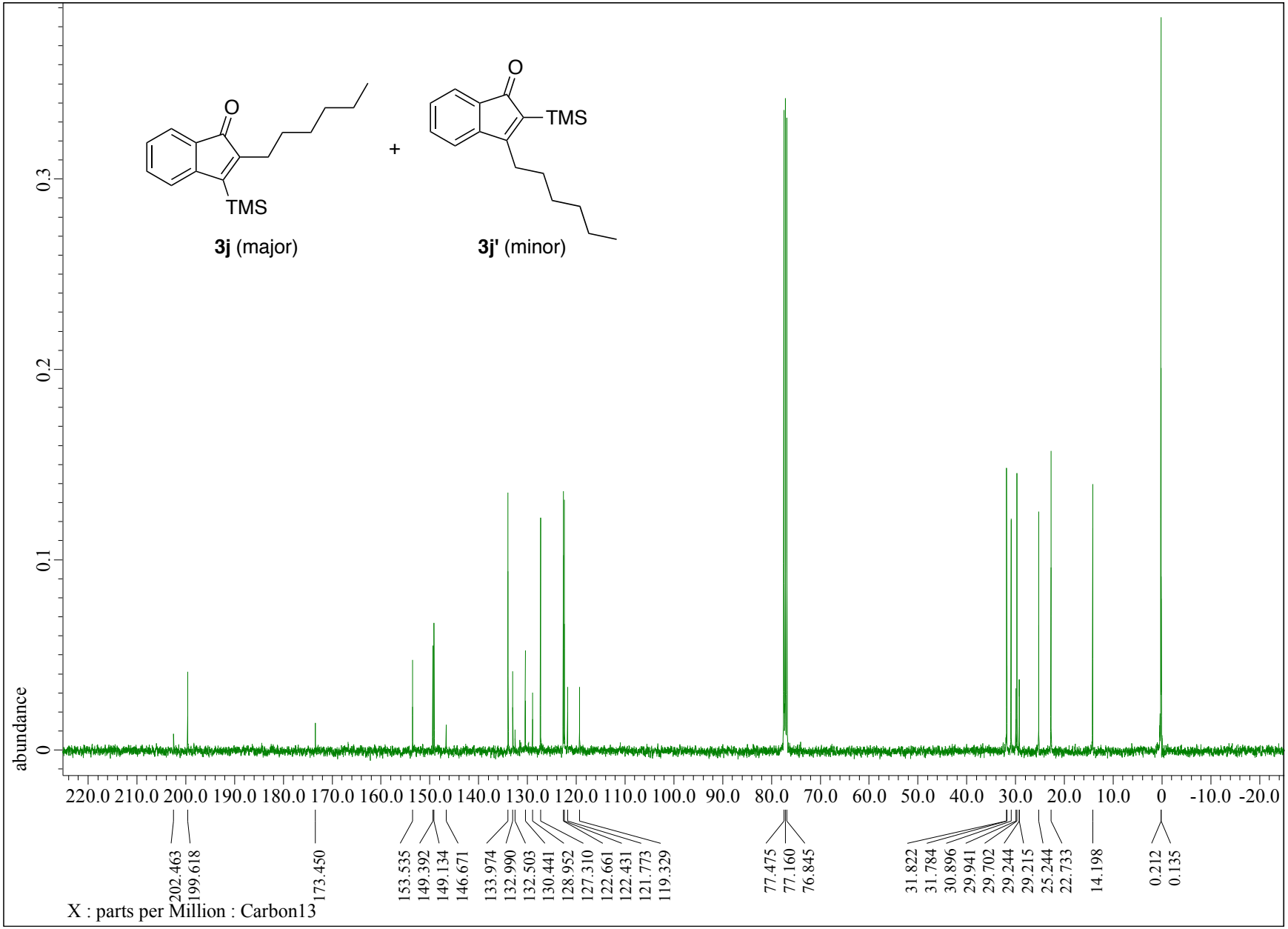

O
TMS
TMS
3h
abundance
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
-2.0
9.00
9.03
2.34
0.98
1.05
7.450
7.448
7.432
7.354
7.352
7.336
7.332
7.260
7.236
7.224
7.222
7.219
7.205
7.200
1.539
0.440
0.251
X : parts per Million : Proton

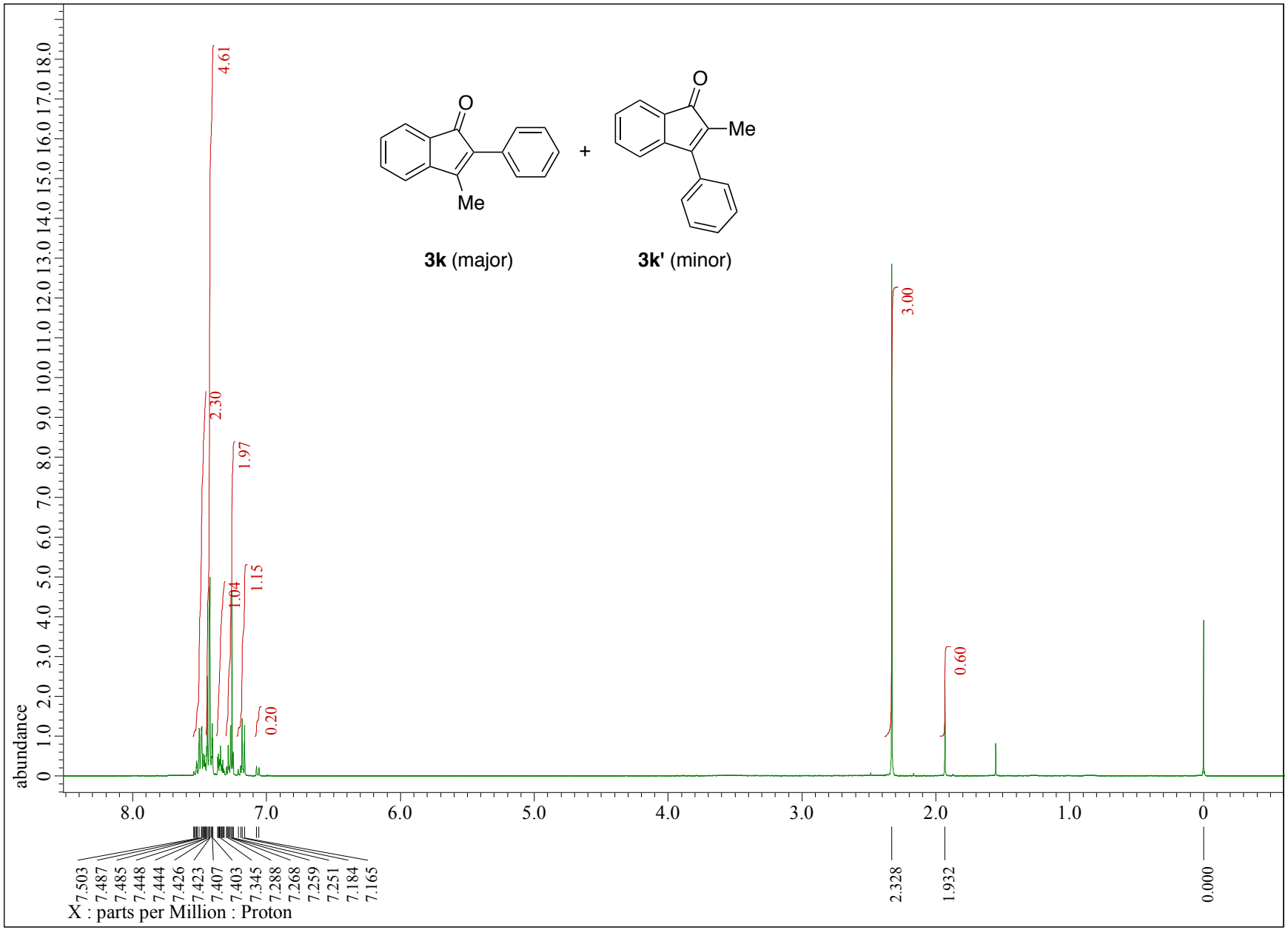

O
TMS
TMS
3h
abundance
0.3
0.2
0.1
0
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
194.549
165.402
148.246
134.461
130.546
129.238
129.114
123.749
123.615
107.958
98.583
77.475
77.160
76.835
23.611
-0.160
-0.466
X : parts per Million : Carbon13

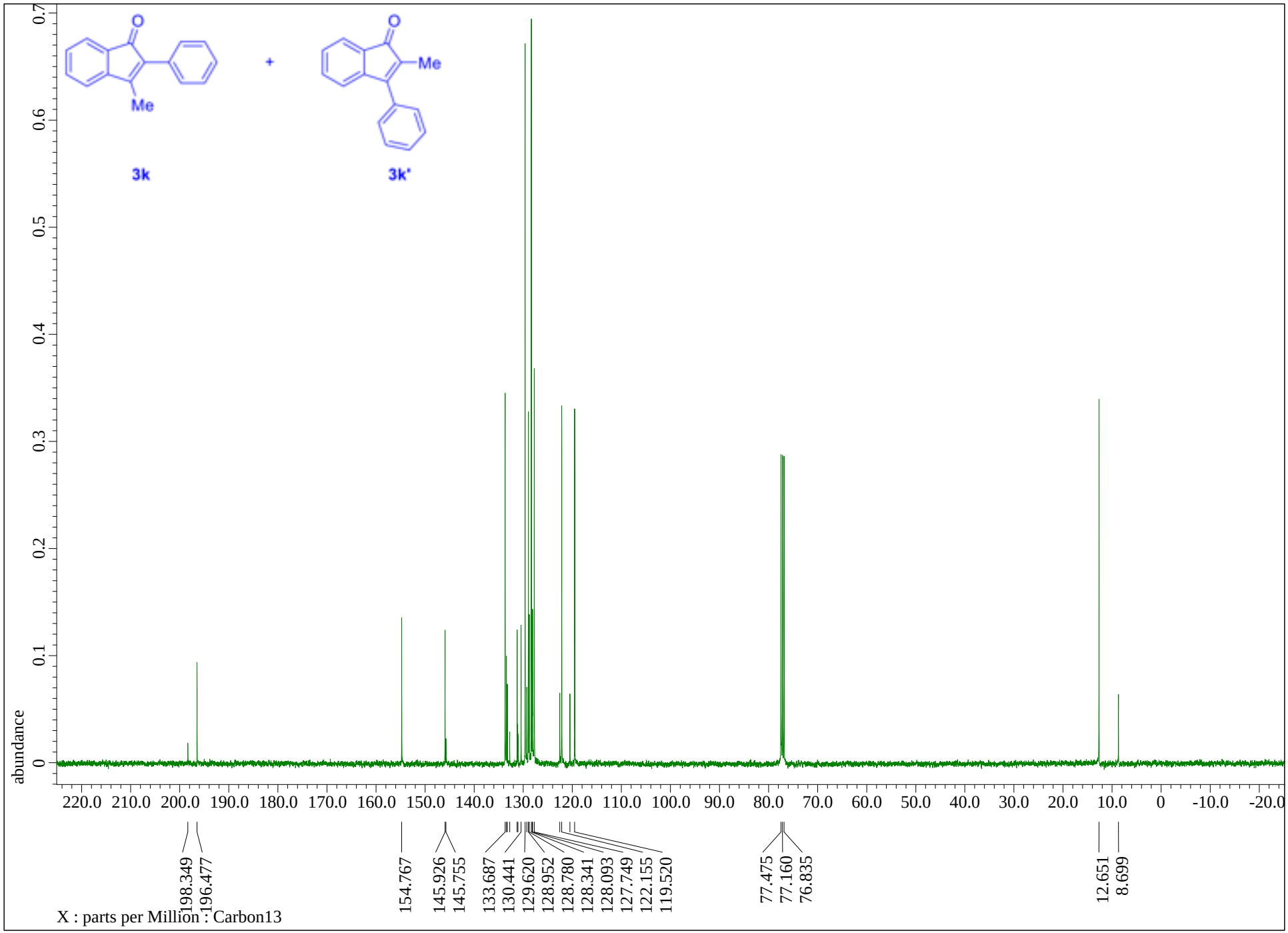

O
Me
TMS
3i
abundance
9.00
3.00
2.00
0.907
0.900
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
-2.0
7.392
7.376
7.308
7.287
7.261
7.121
7.102
7.086
1.939
1.546
0.383
0.000
X : parts per Million : Proton

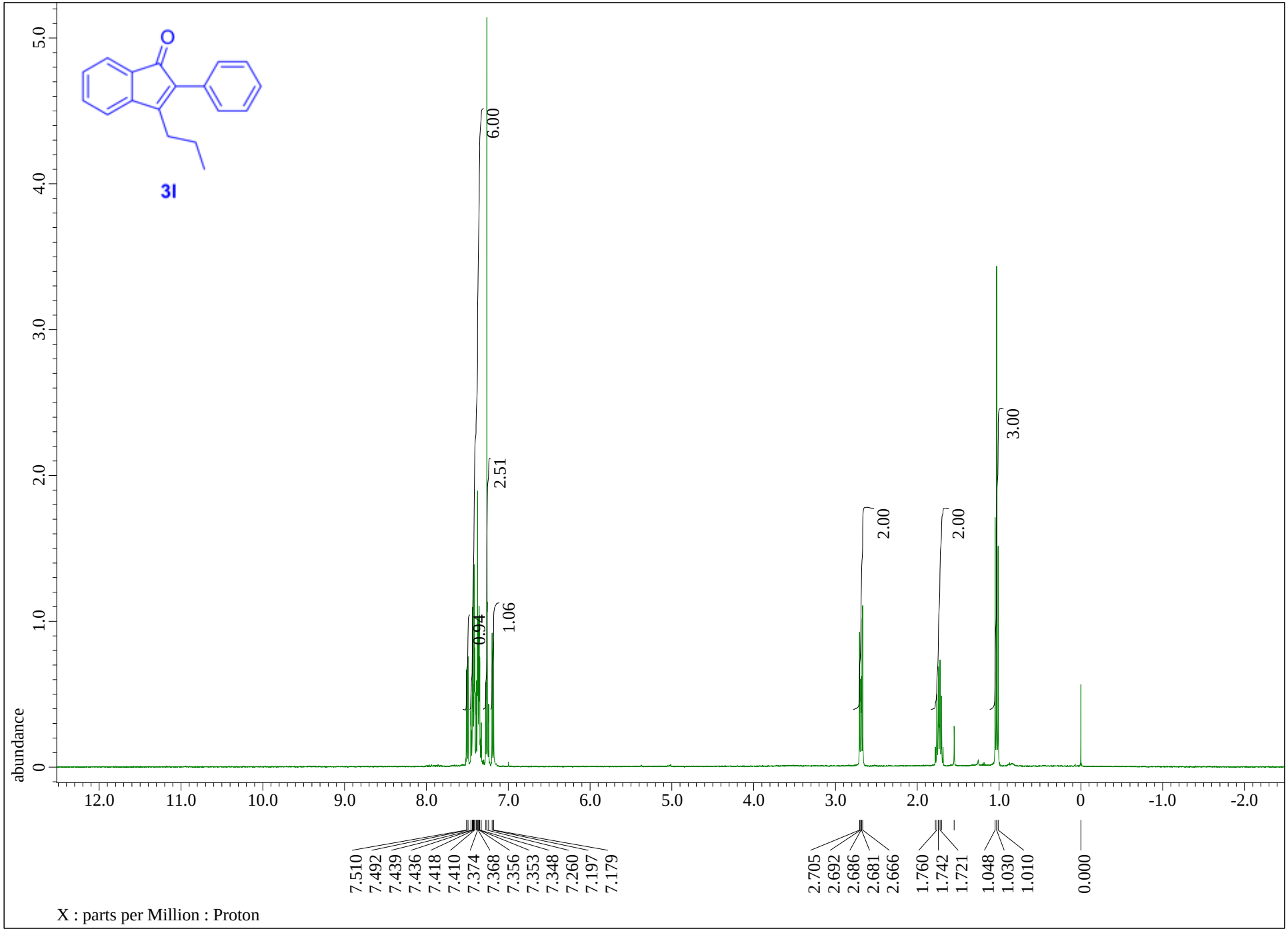

O
Me
TMS
3i
abundance
0
0.1
0.2
0.3
0.4
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
199.485
153.860
149.335
144.113
133.983
130.298
127.234
122.689
122.250
77.475
77.160
76.835
10.494
0.030
X : parts per Million : Carbon13

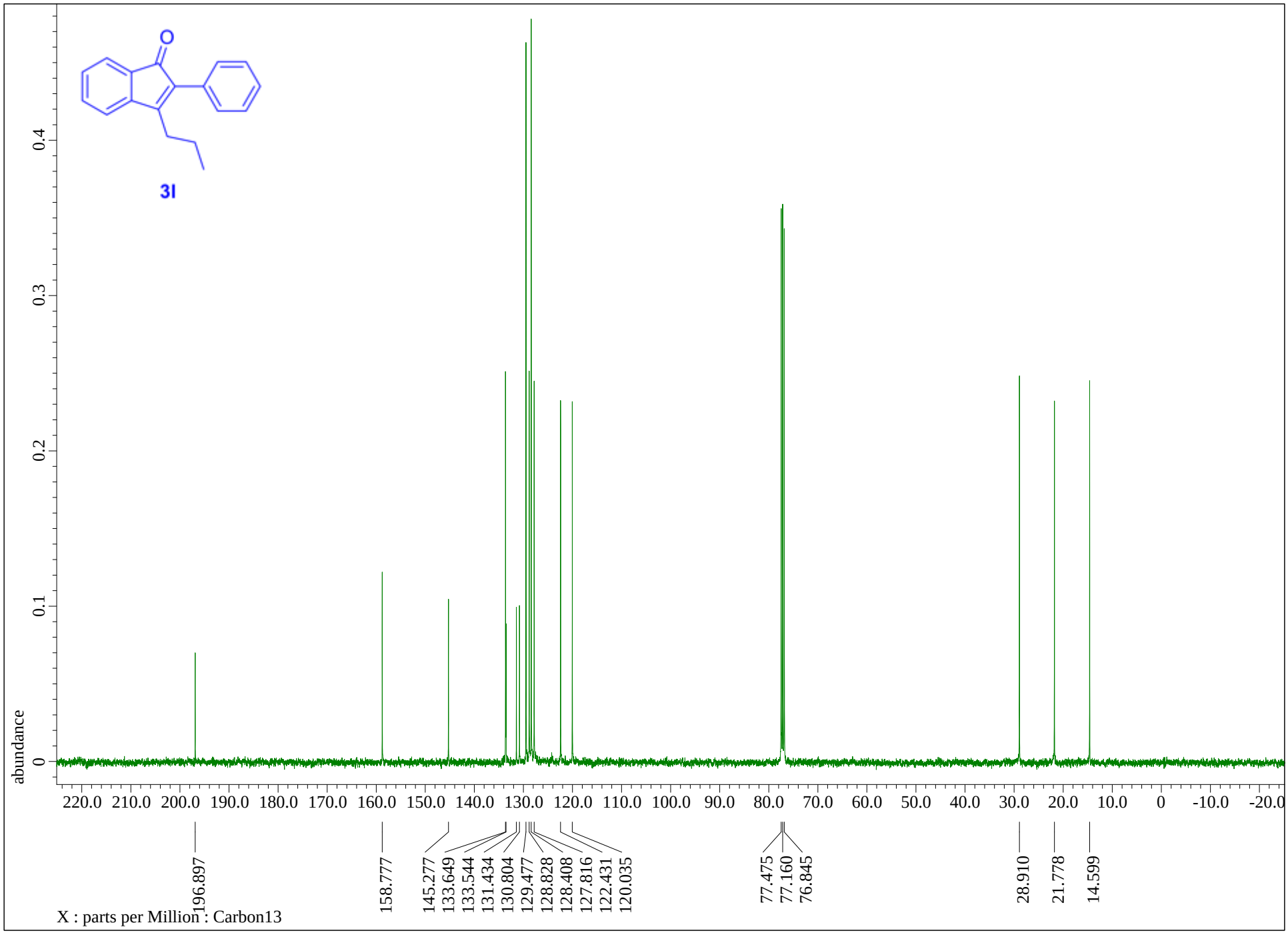

O
TMS
3j (major)
+
O
TMS
3j' (minor)
abundance
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
11.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
1.33
2.16
0.46
2.01
9.18
3.80
9.00
2.06
7.411
7.390
7.386
7.369
7.356
7.338
7.129
7.109
7.092
2.680
2.667
2.661
2.655
2.640
2.359
2.342
2.322
1.543
1.338
1.329
1.293
1.286
0.897
0.880
0.381
0.281
0.000
X : parts per Million : Proton

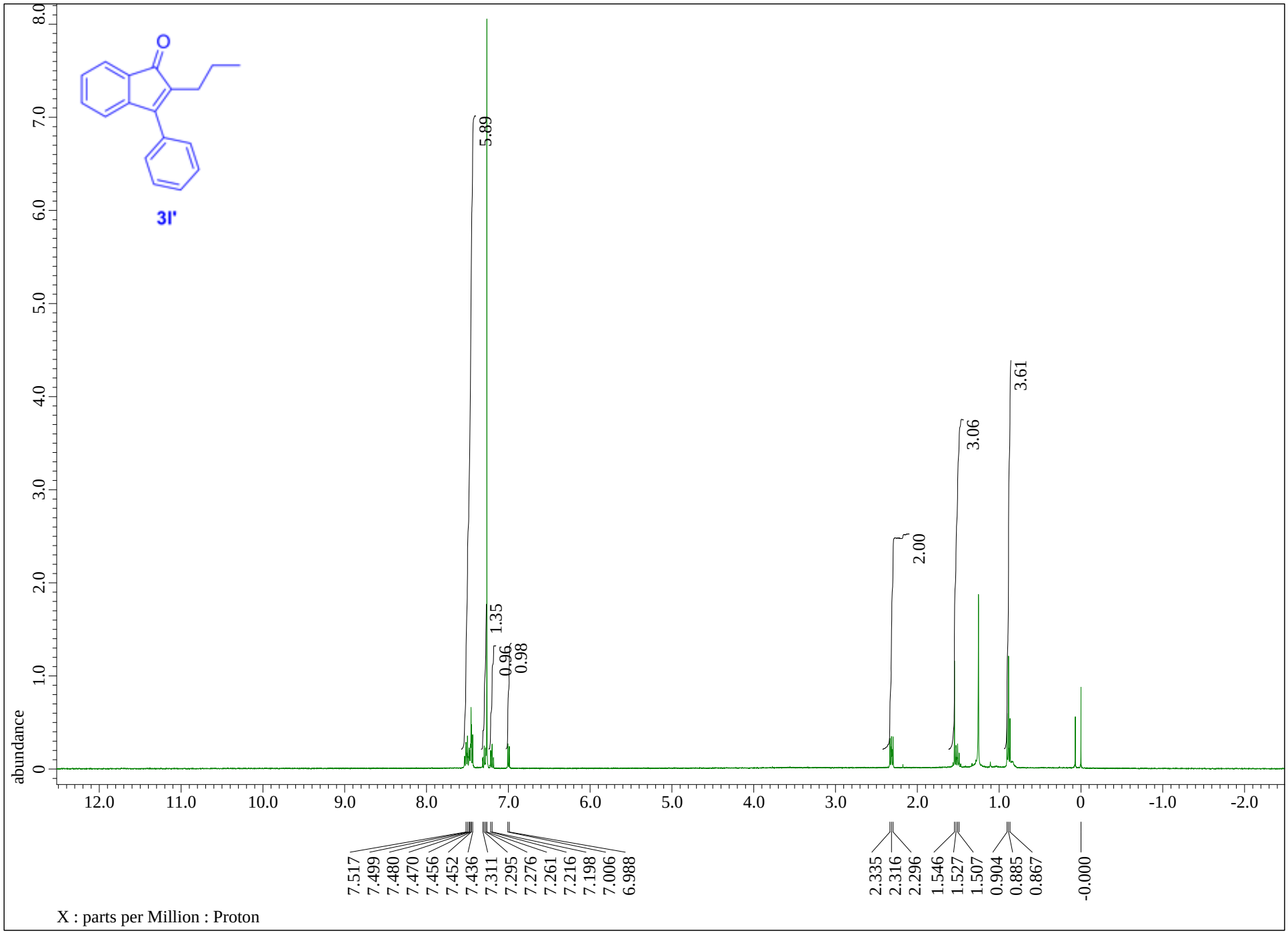

O
TMS
3j (major)
+
O
TMS
3j' (minor)
abundance
0
0.1
0.2
0.3
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
202.463
199.618
173.450
153.535
149.392
149.134
146.671
133.974
132.990
132.503
130.441
128.952
127.310
122.661
122.431
121.773
119.329
77.475
77.160
76.845
31.822
31.784
30.896
29.941
29.702
29.244
29.215
25.244
22.733
14.198
0.212
0.135
X : parts per Million : Carbon13

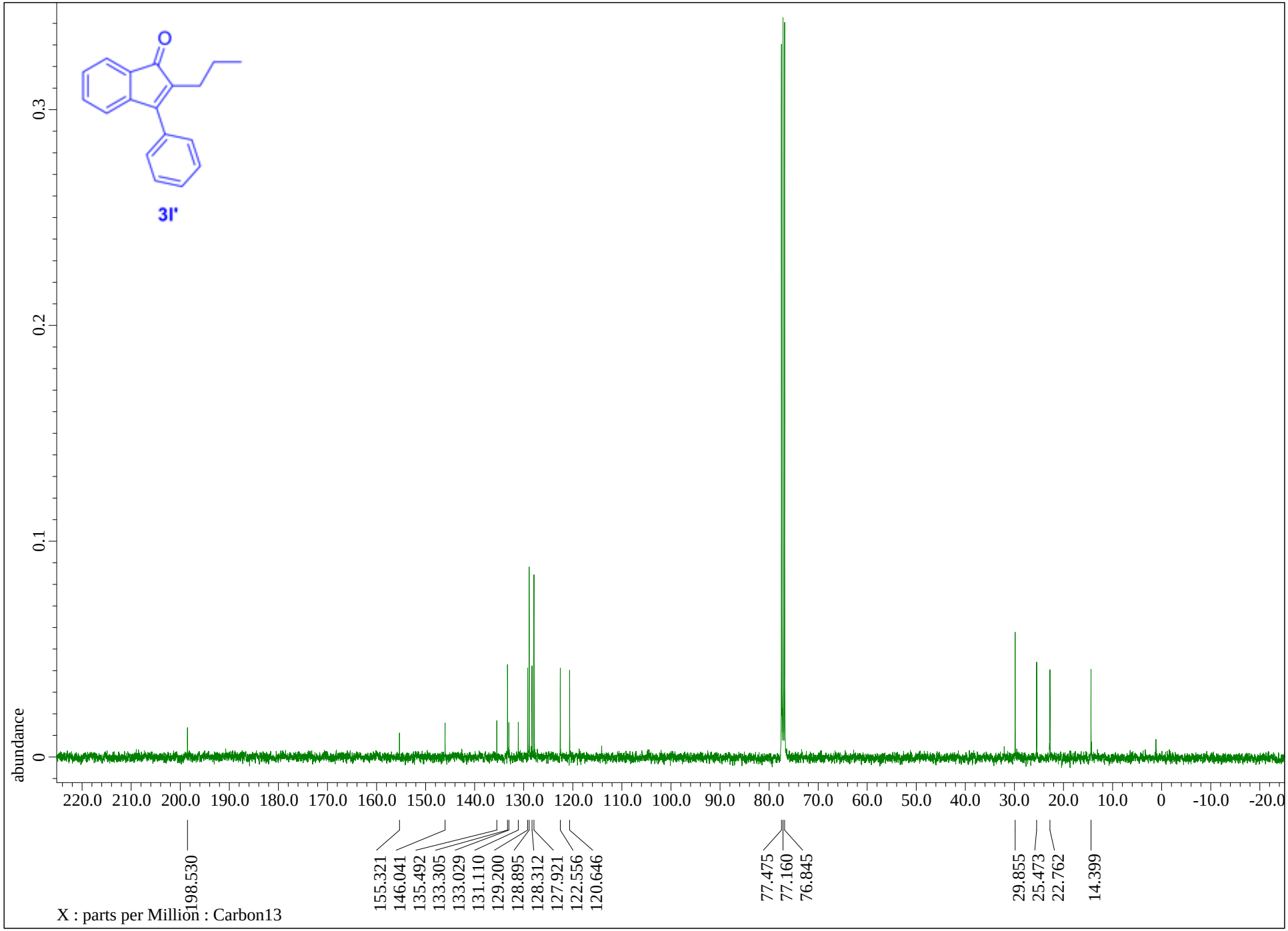

O
Me
+
O
Me
3k (major)
3k' (minor)
abundance
4.61
2.30
1.97
1.04
1.15
0.20
3.00
0.60
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.503
7.487
7.485
7.448
7.444
7.426
7.423
7.407
7.403
7.345
7.288
7.268
7.259
7.251
7.184
7.165
2.328
1.932
0.000
X : parts per Million : Proton

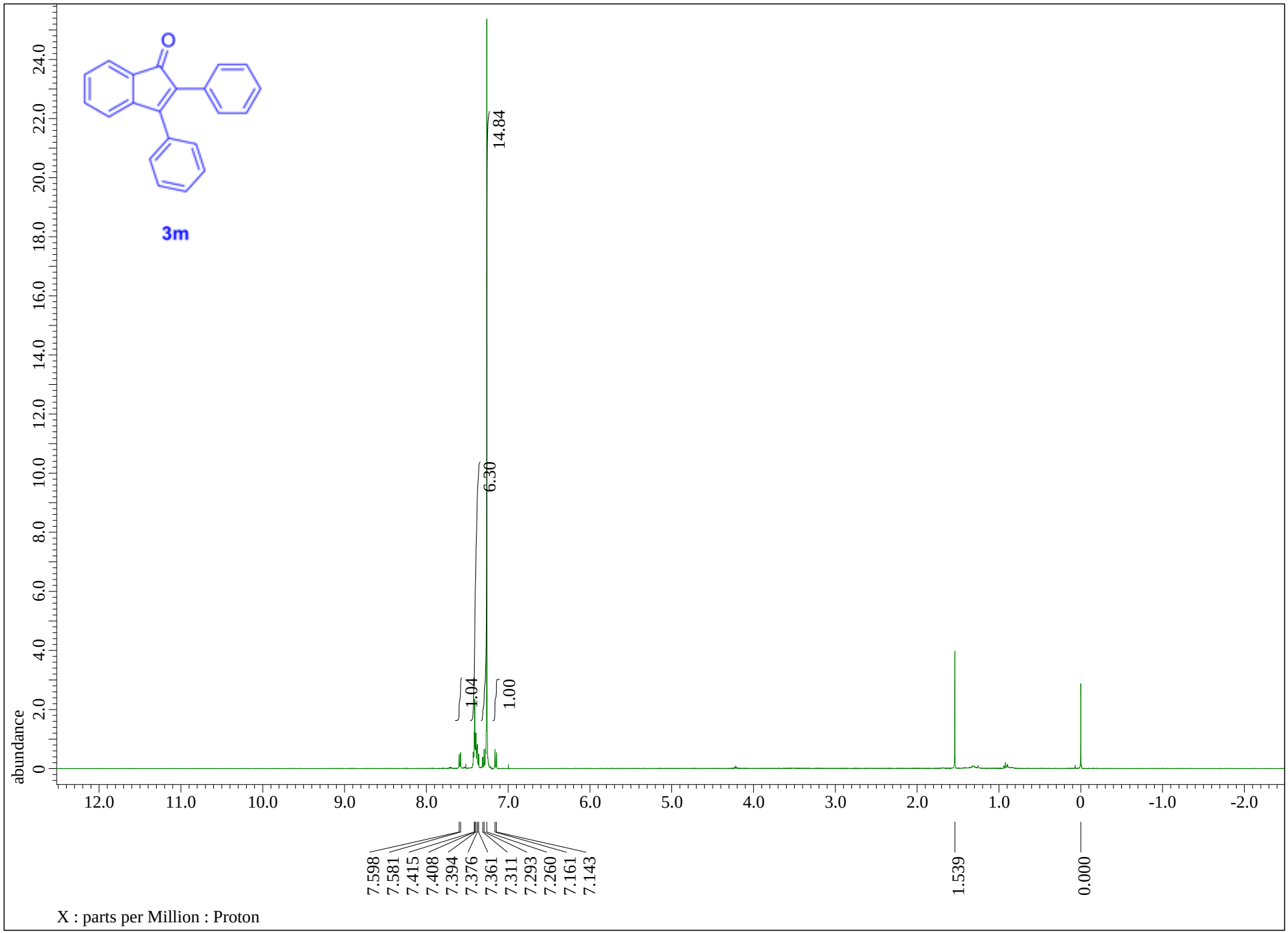

O
Me
3k
+
O
Me
3k'
abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
198.349
196.477
154.767
145.926
145.755
133.687
130.441
129.620
128.952
128.780
128.341
128.093
127.749
122.155
119.520
77.475
77.160
76.835
12.651
8.699
X : parts per Million : Carbon13

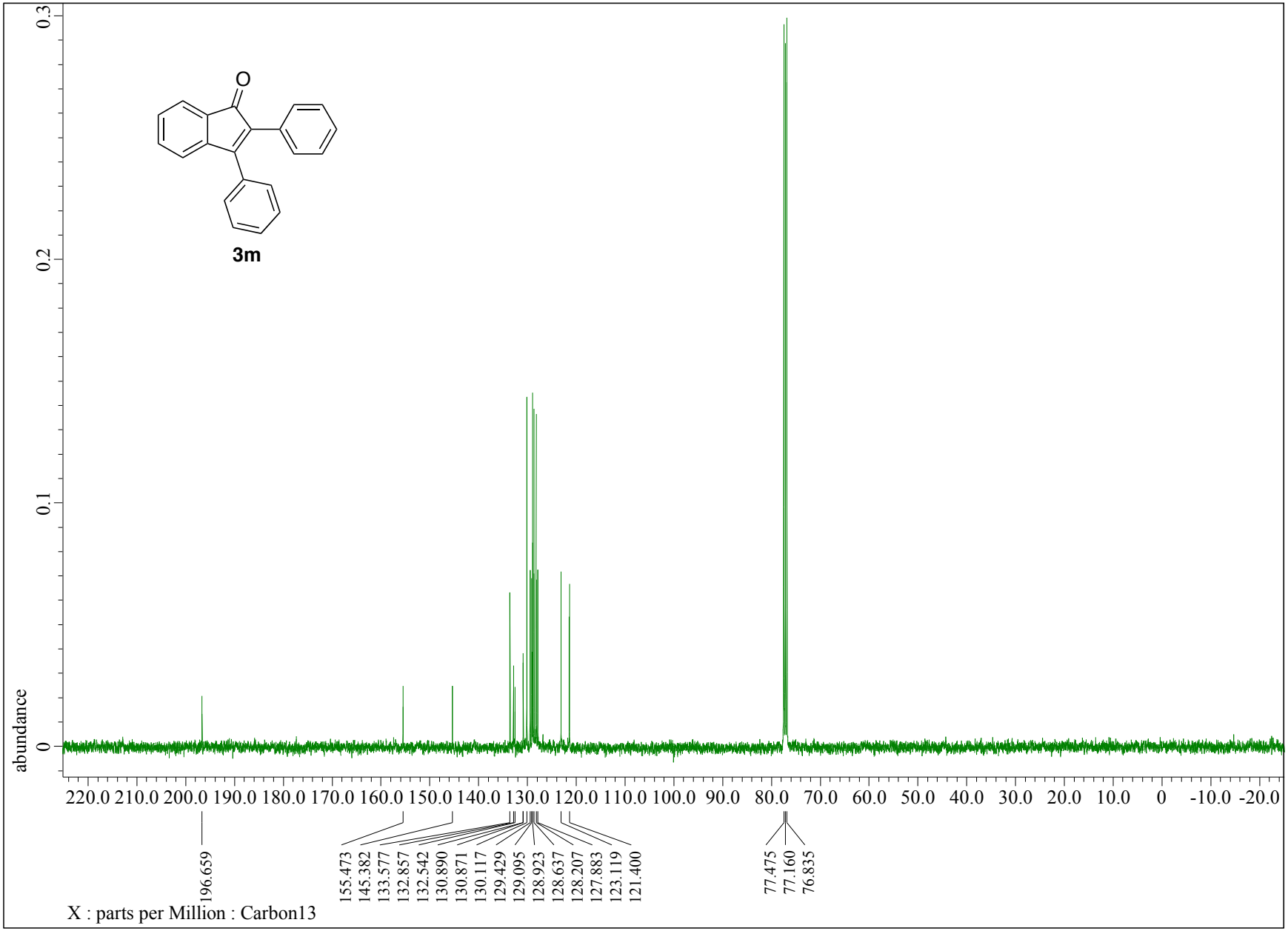

3l
abundance
X : parts per Million : Proton
6.00
2.51
0.94
1.06
2.00
2.00
3.00
7.510
7.492
7.439
7.436
7.418
7.410
7.374
7.368
7.356
7.353
7.348
7.260
7.197
7.179
2.705
2.692
2.686
2.681
2.666
1.760
1.742
1.721
1.048
1.030
1.010
0.000

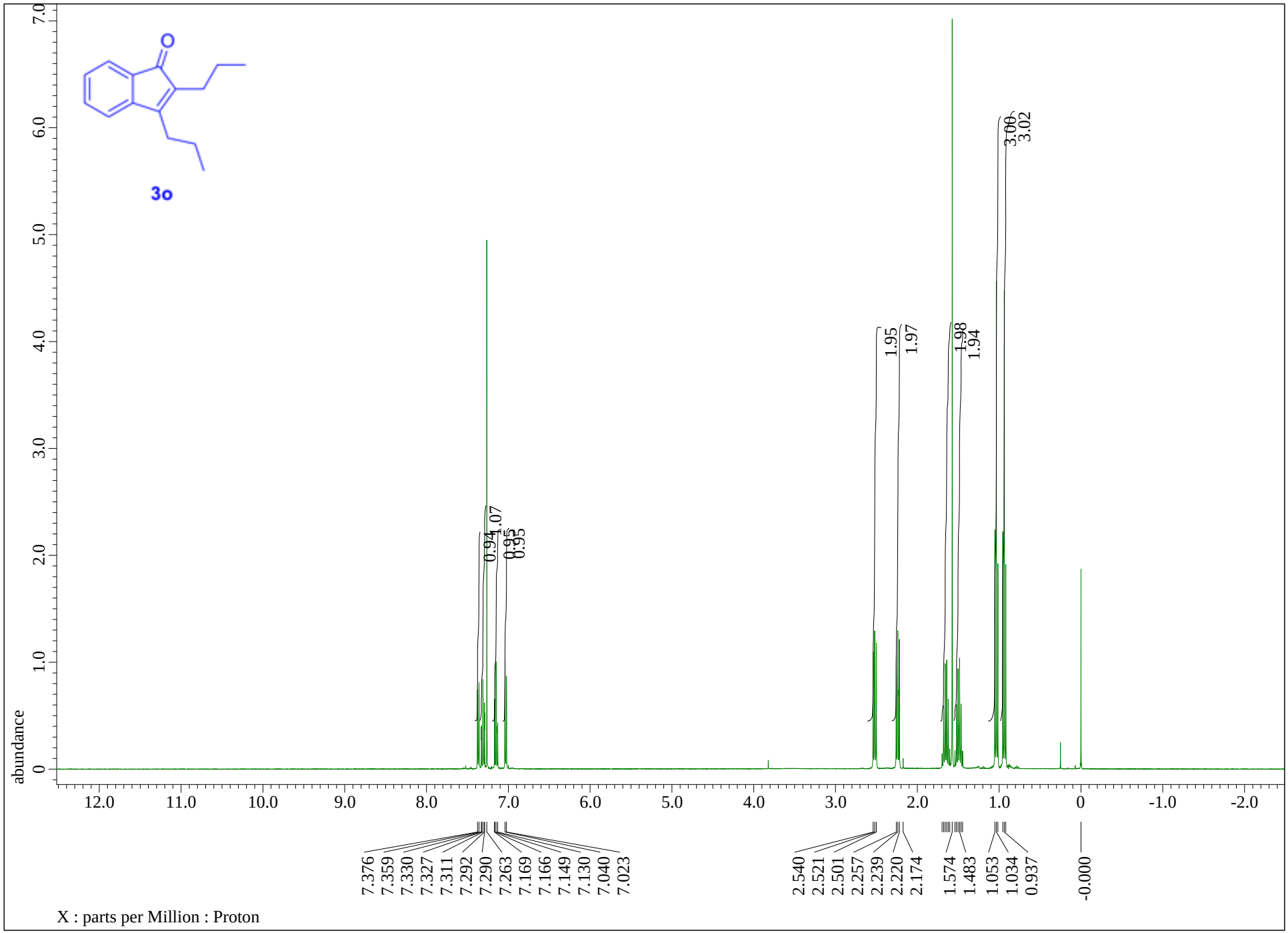

3l
abundance
0
0.1
0.2
0.3
0.4
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
196.897
158.777
145.277
133.649
133.544
131.434
130.804
129.477
128.828
128.408
127.816
122.431
120.035
77.475
77.160
76.845
28.910
21.778
14.599
X : parts per Million : Carbon13

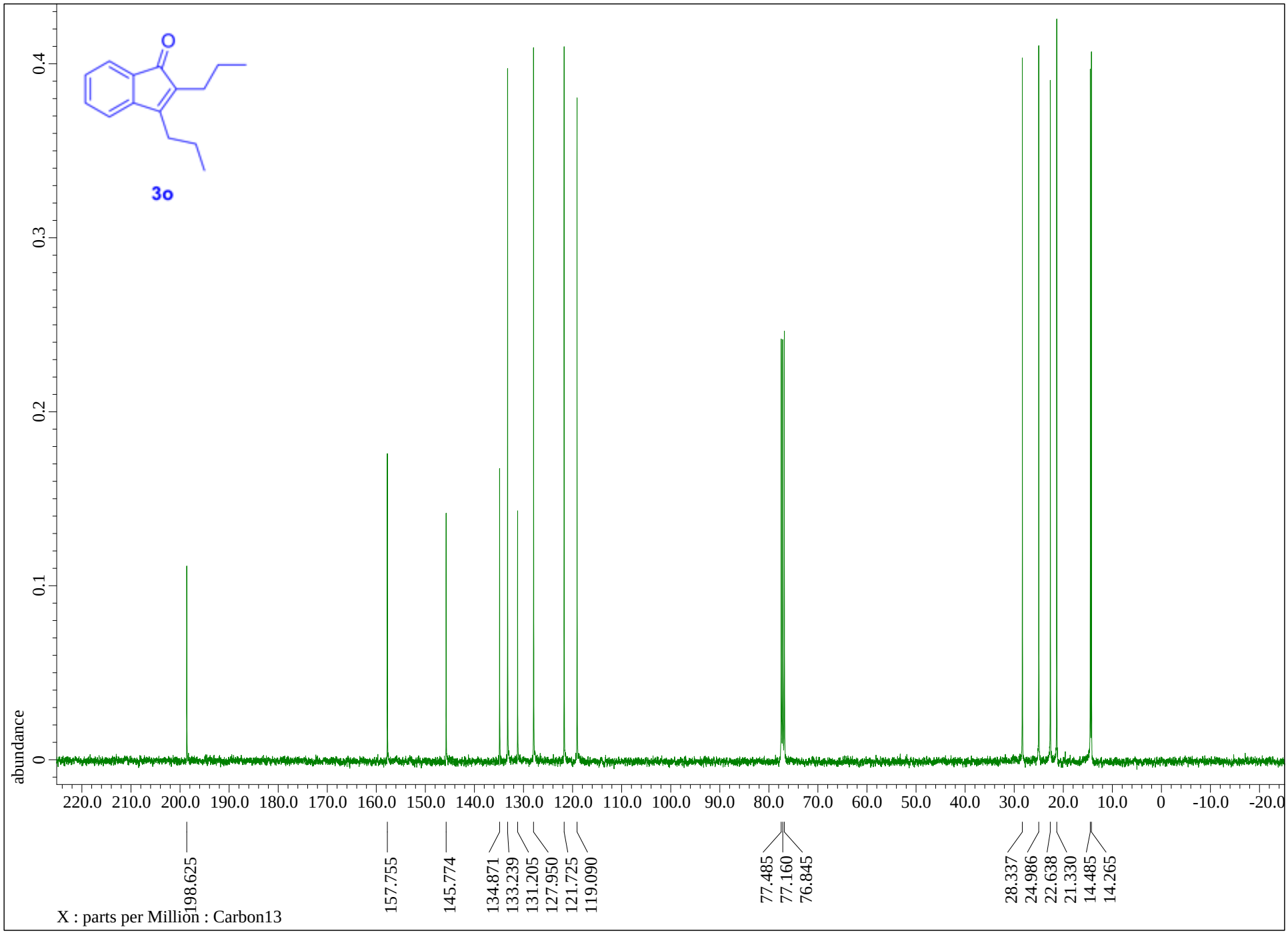

3l'
abundance
X : parts per Million : Proton
5.89
1.35
0.96
0.98
2.00
3.06
3.61
7.517
7.499
7.480
7.470
7.456
7.452
7.436
7.311
7.295
7.276
7.261
7.216
7.198
7.006
6.988
2.335
2.316
2.296
1.546
1.527
1.507
0.904
0.885
0.867
-0.000

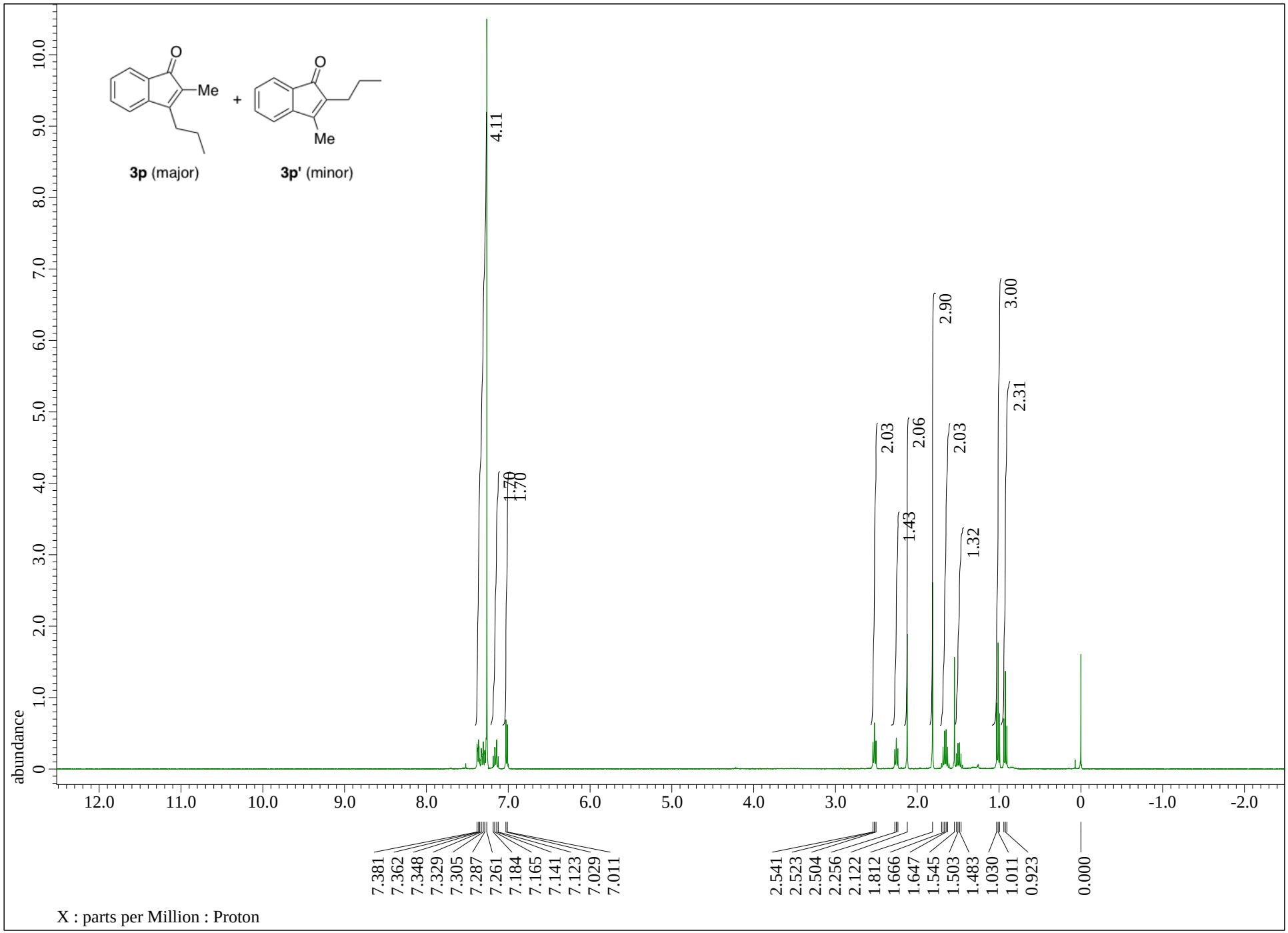

3l'
abundance
0
0.1
0.2
0.3
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
198.530
155.321
146.041
135.492
133.305
133.029
131.110
129.200
128.895
128.312
127.921
122.556
120.646
77.475
77.160
76.845
29.855
25.473
22.762
14.399
X : parts per Million : Carbon13

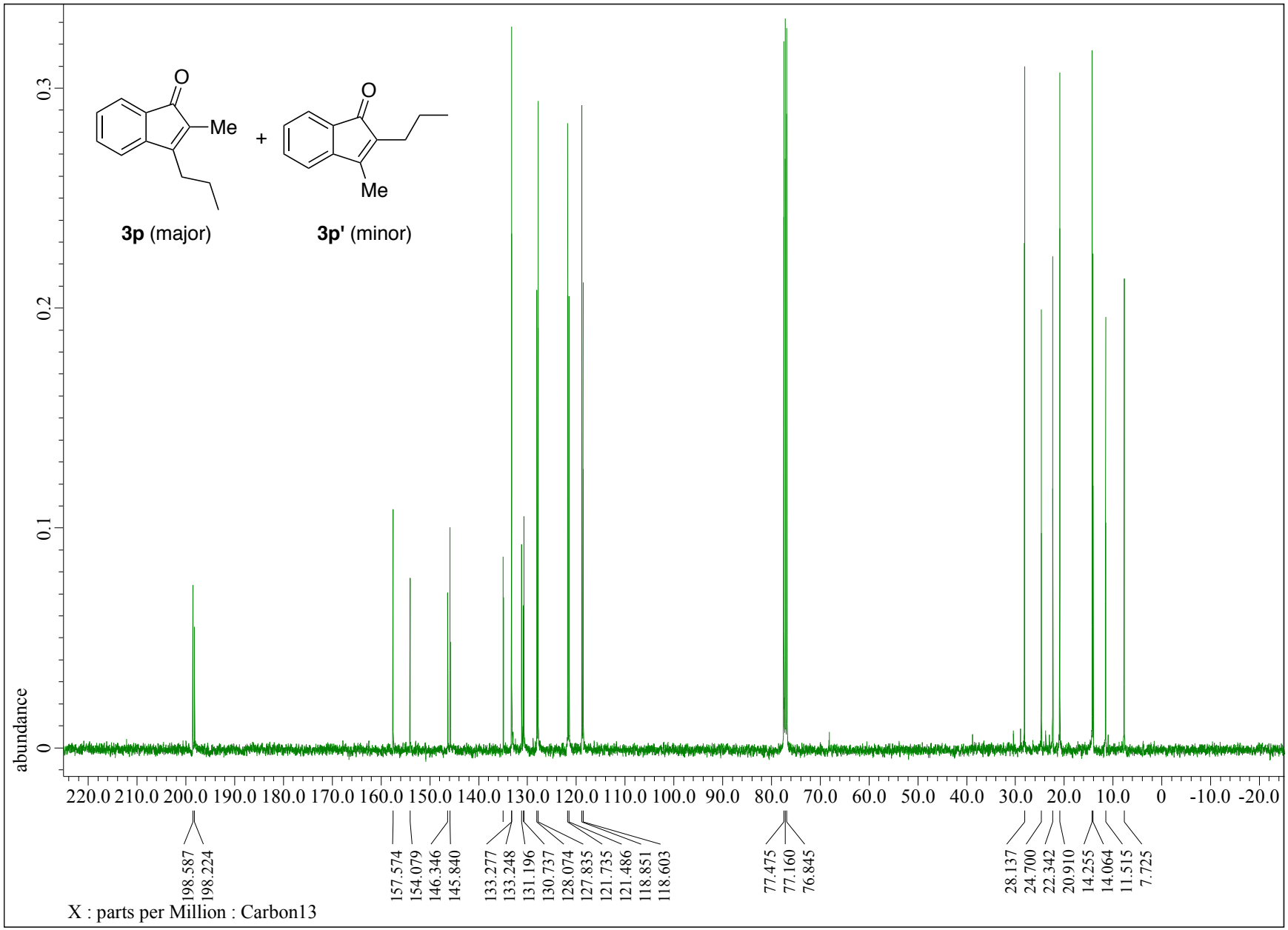

3m
abundance
14.84
6.30
1.04
1.00
7.598
7.581
7.415
7.408
7.394
7.376
7.361
7.311
7.293
7.260
7.161
7.143
1.539
0.000
X : parts per Million : Proton

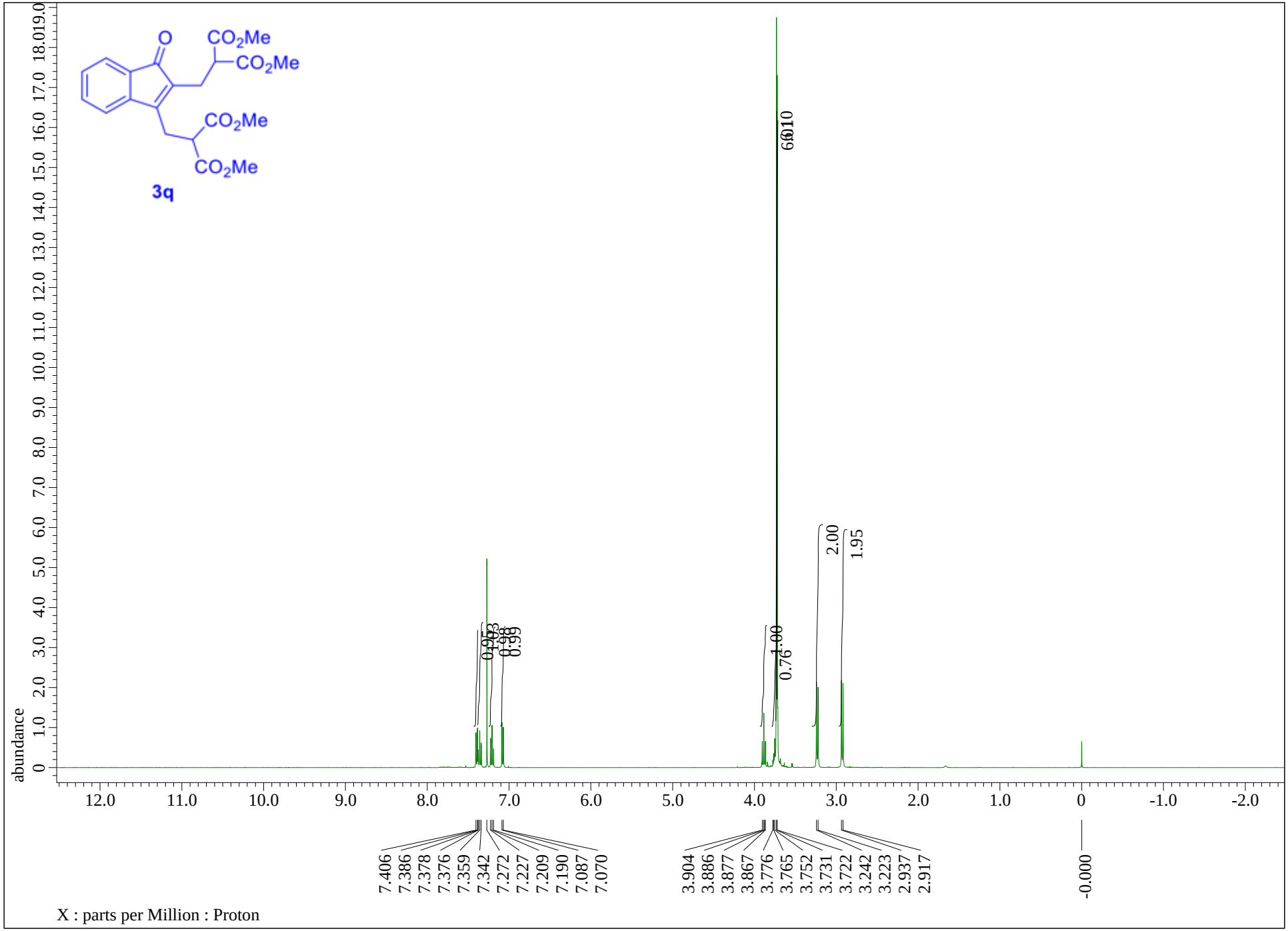

O
3m
abundance
0.3
0.2
0.1
0
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
196.659
155.473
145.382
133.577
132.857
132.542
130.890
130.871
130.117
129.429
129.095
128.923
128.637
128.207
127.883
123.119
121.400
77.475
77.160
76.835
X : parts per Million : Carbon13

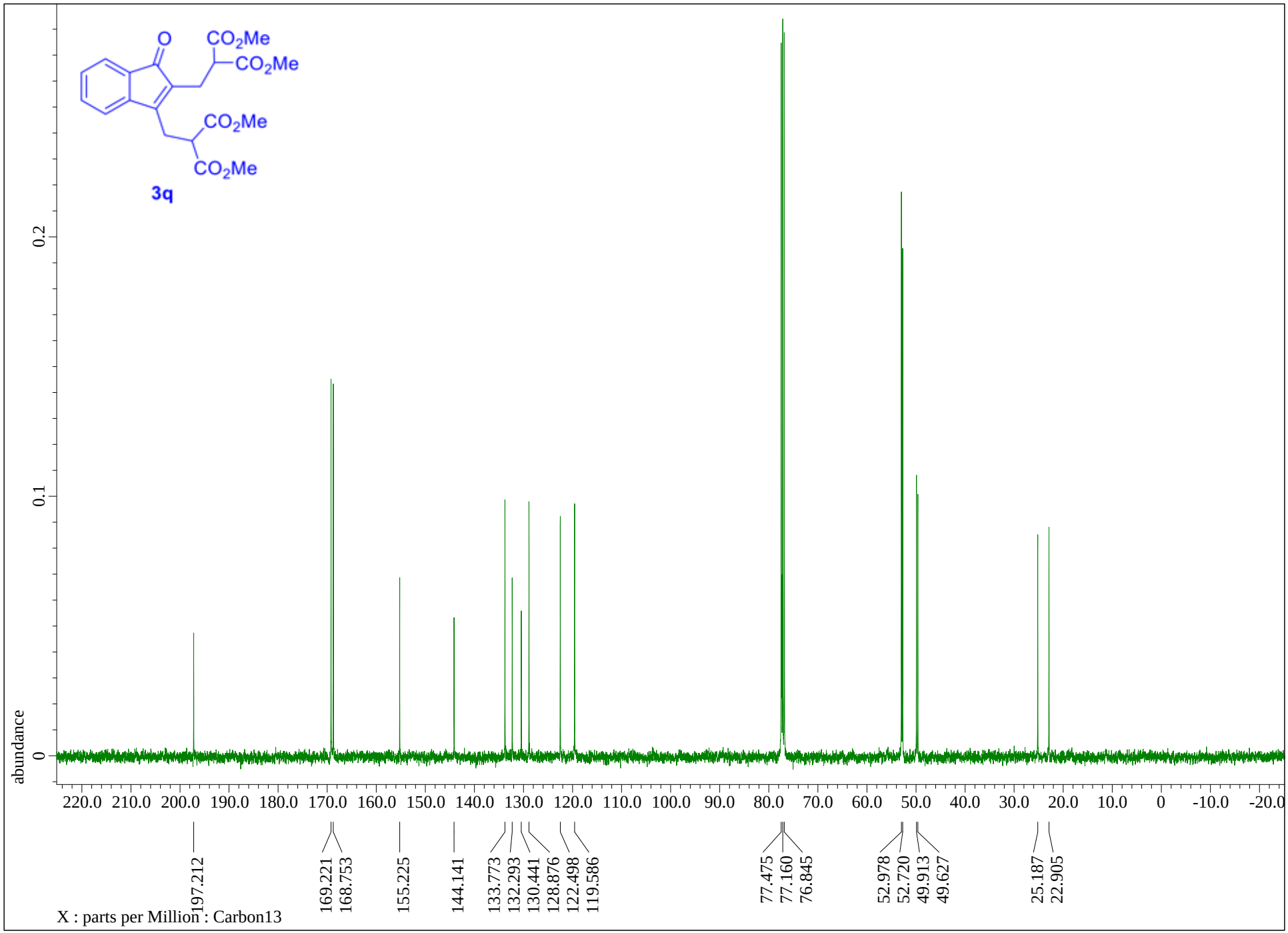

3o
abundance
X : parts per Million : Proton
7.376
7.359
7.330
7.327
7.311
7.292
7.290
7.263
7.169
7.166
7.149
7.130
7.040
7.023
2.540
2.521
2.501
2.257
2.239
2.220
2.174
1.574
1.483
1.053
1.034
0.937
-0.000

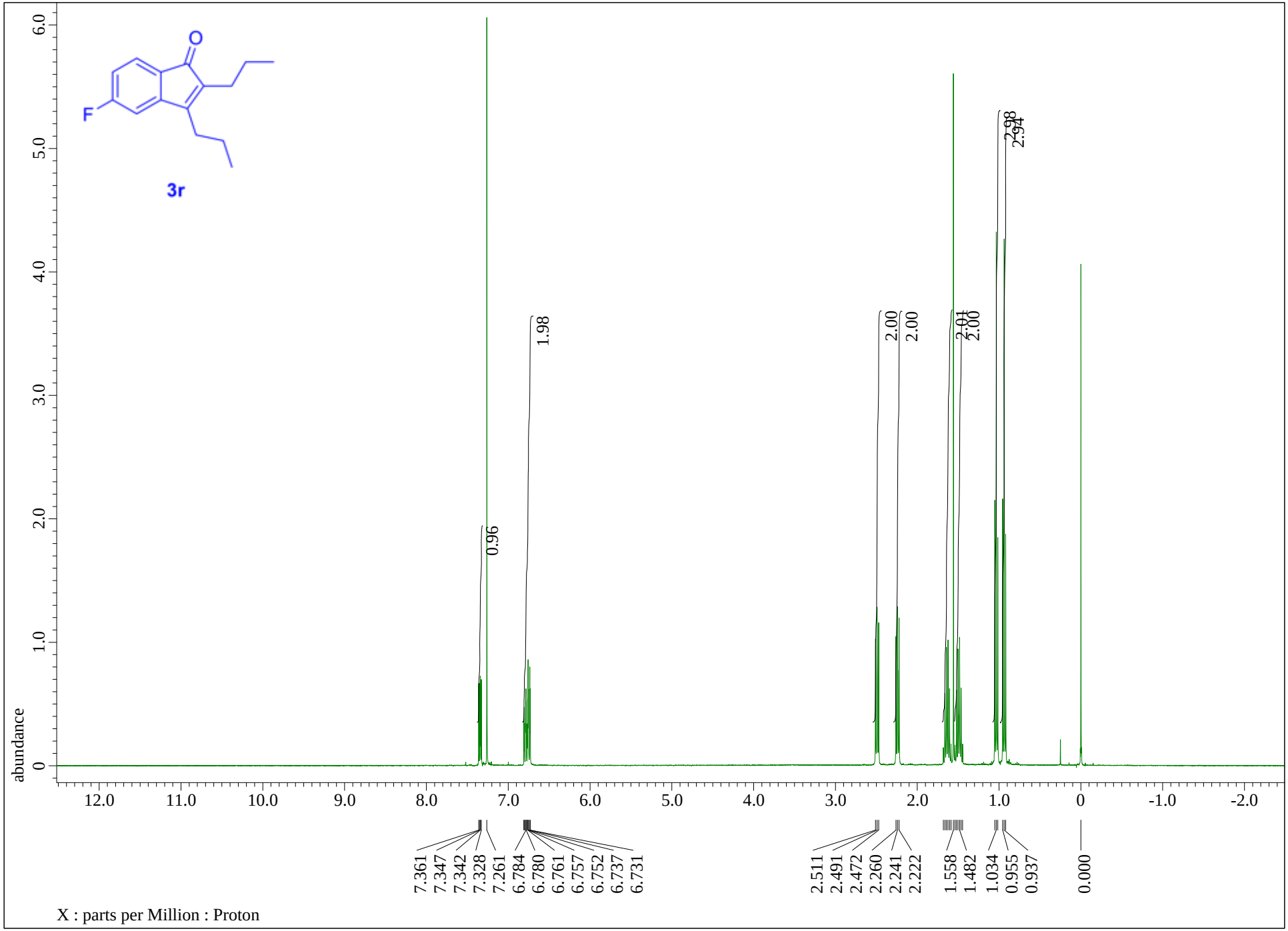

3o
abundance
0
0.1
0.2
0.3
0.4
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
198.625
157.755
145.774
134.871
133.239
131.205
127.950
121.725
119.090
77.485
77.160
76.845
28.337
24.986
22.638
21.330
14.485
14.265
X : parts per Million : Carbon13

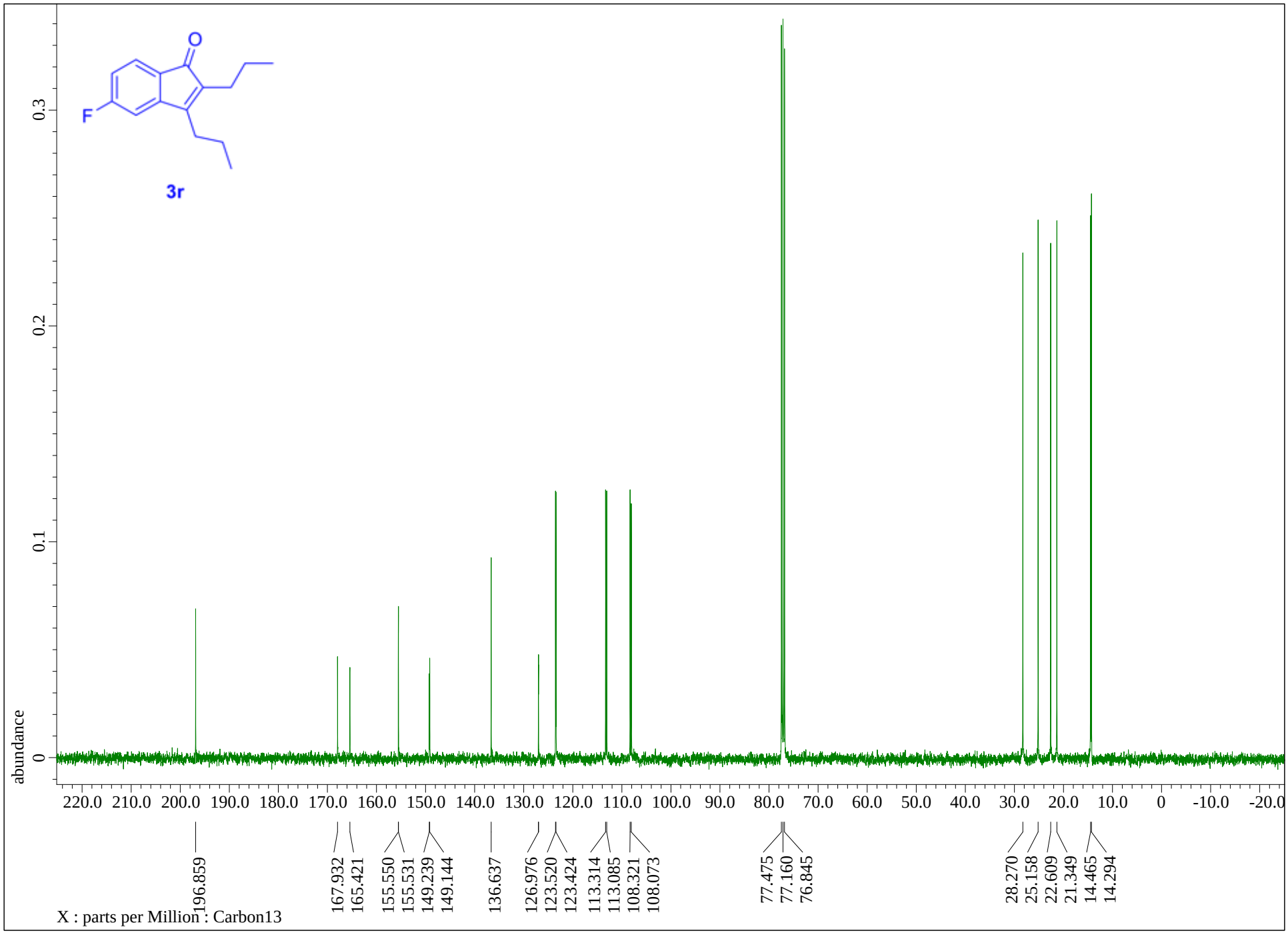

Me
+
Me
3p (major)
3p' (minor)
4.11
1.70
1.70
2.03
1.43
2.06
2.90
2.03
1.32
2.31
3.00
abundance
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
-2.0
7.381
7.362
7.348
7.329
7.305
7.287
7.261
7.184
7.165
7.141
7.123
7.029
7.011
2.541
2.523
2.504
2.256
2.122
1.812
1.666
1.647
1.545
1.503
1.483
1.030
1.011
0.923
0.000
X : parts per Million : Proton

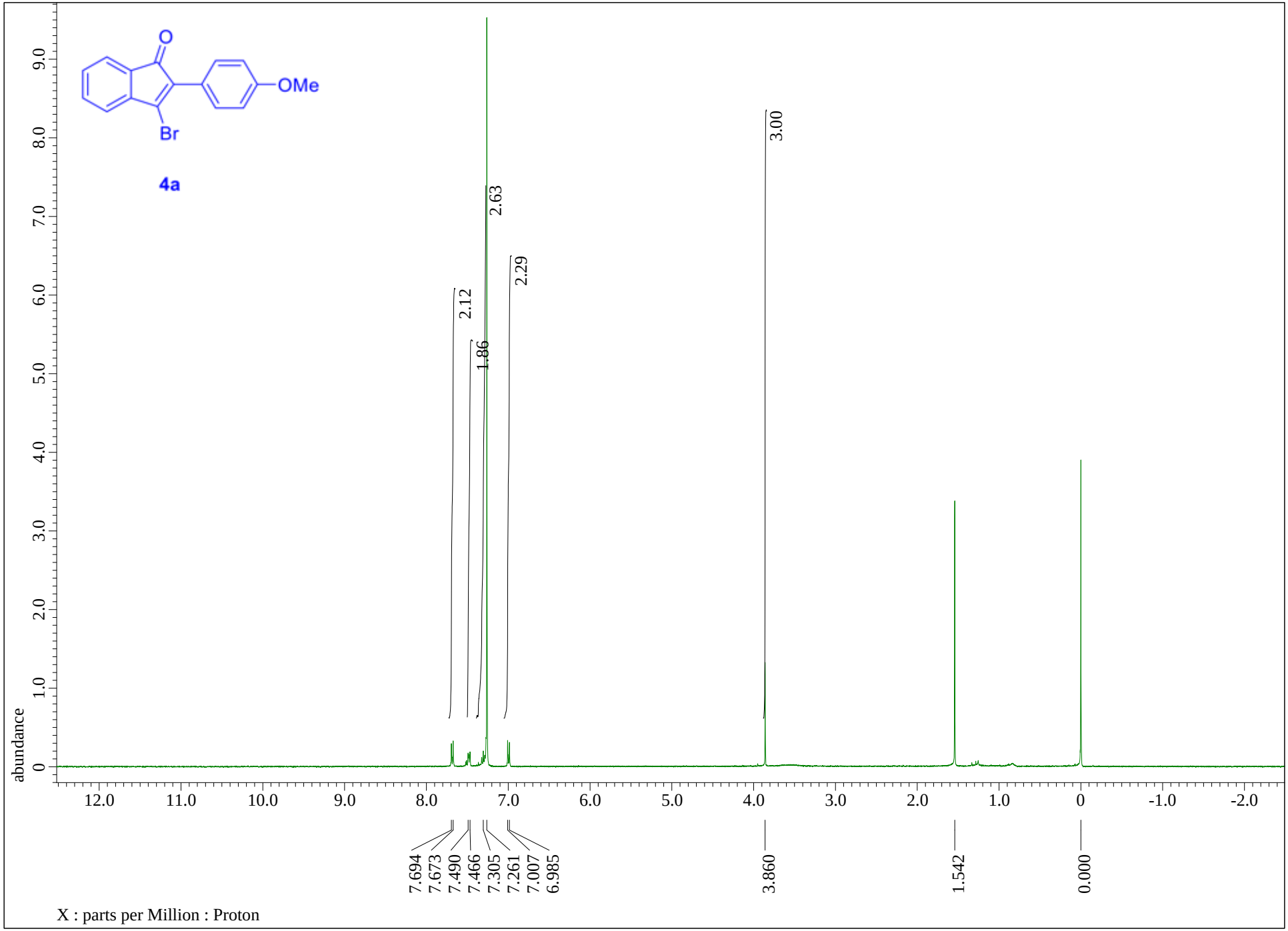

O
Me
+
O
Me
3p (major)
3p' (minor)
abundance
0
0.1
0.2
0.3
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
198.587
198.224
157.574
154.079
146.346
145.840
133.277
133.248
131.196
130.737
128.074
127.835
121.735
121.486
118.851
118.603
77.475
77.160
76.845
28.137
24.700
22.342
20.910
14.255
14.064
11.515
7.725
X : parts per Million : Carbon13

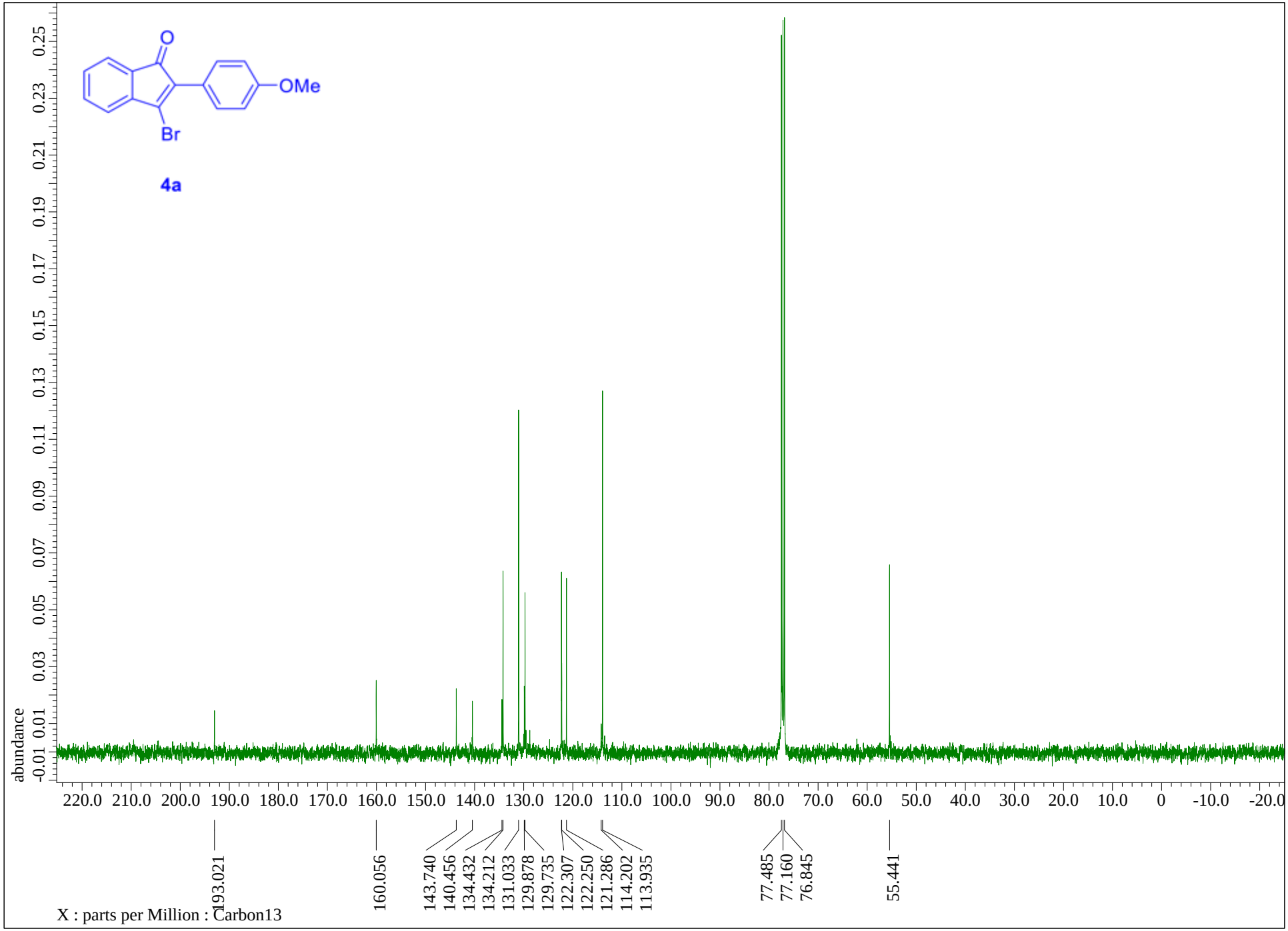

O
CO2Me
CO2Me
CO2Me
CO2Me
3q
abundance
X : parts per Million : Proton
7.406
7.386
7.378
7.376
7.359
7.342
7.272
7.227
7.209
7.190
7.087
7.070
3.904
3.886
3.877
3.867
3.776
3.765
3.752
3.731
3.722
3.242
3.223
2.937
2.917
-0.000
6.10
2.00
1.95
1.00
0.76

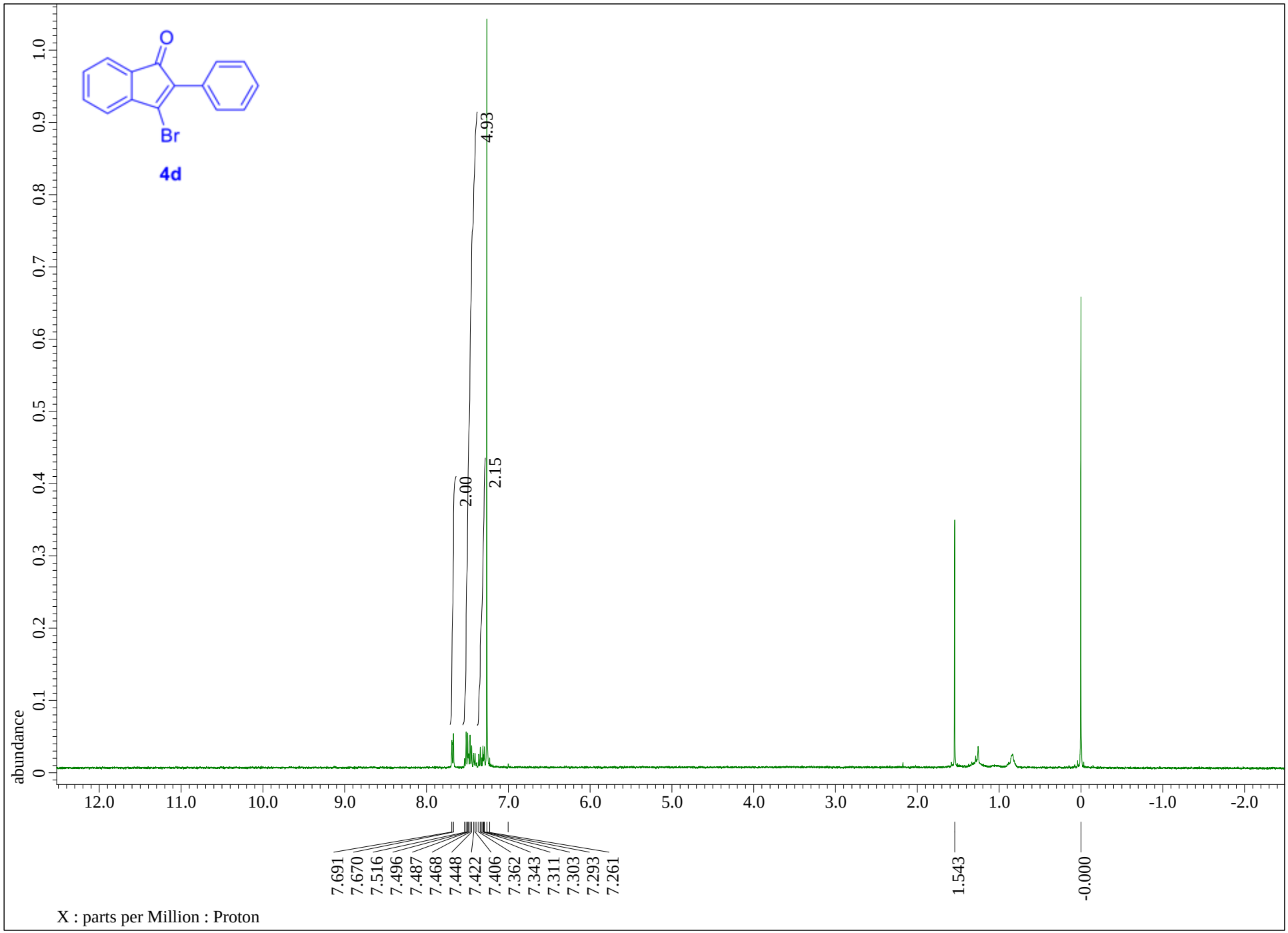

O
CO2Me
CO2Me
CO2Me
CO2Me
3q
abundance
0
0.1
0.2
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
197.212
169.221
168.753
155.225
144.141
133.773
132.293
130.441
128.876
122.498
119.586
77.475
77.160
76.845
52.978
52.720
49.913
49.627
25.187
22.905
X : parts per Million : Carbon13

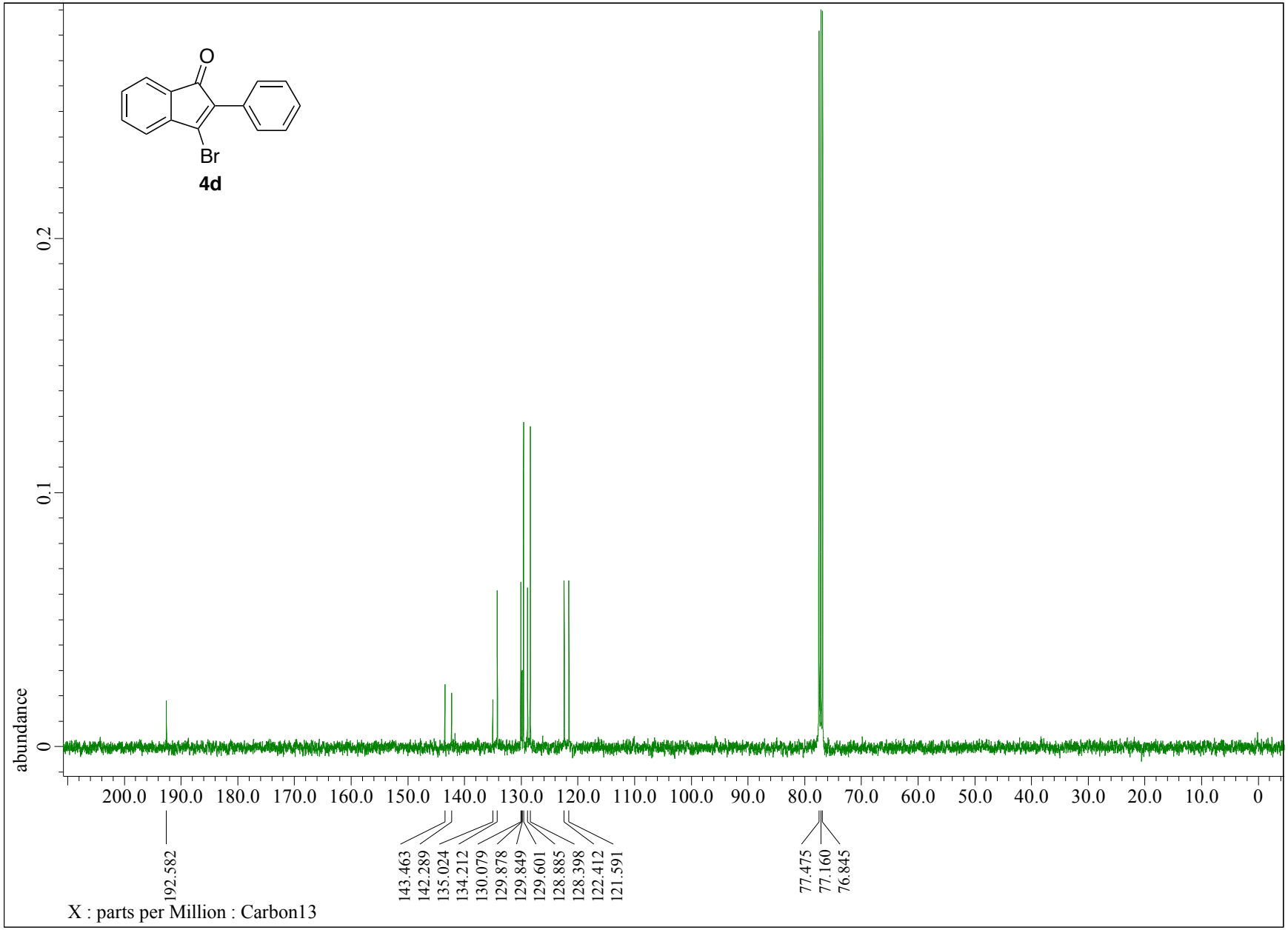

O
F
3r
abundance
0
1.0
2.0
3.0
4.0
5.0
6.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
-2.0
0.96
1.98
2.00
2.00
2.01
2.00
2.98
2.94
7.361
7.347
7.342
7.328
7.261
6.784
6.780
6.761
6.757
6.752
6.737
6.731
2.511
2.491
2.472
2.260
2.241
2.222
1.558
1.482
1.034
0.955
0.937
0.000
X : parts per Million : Proton

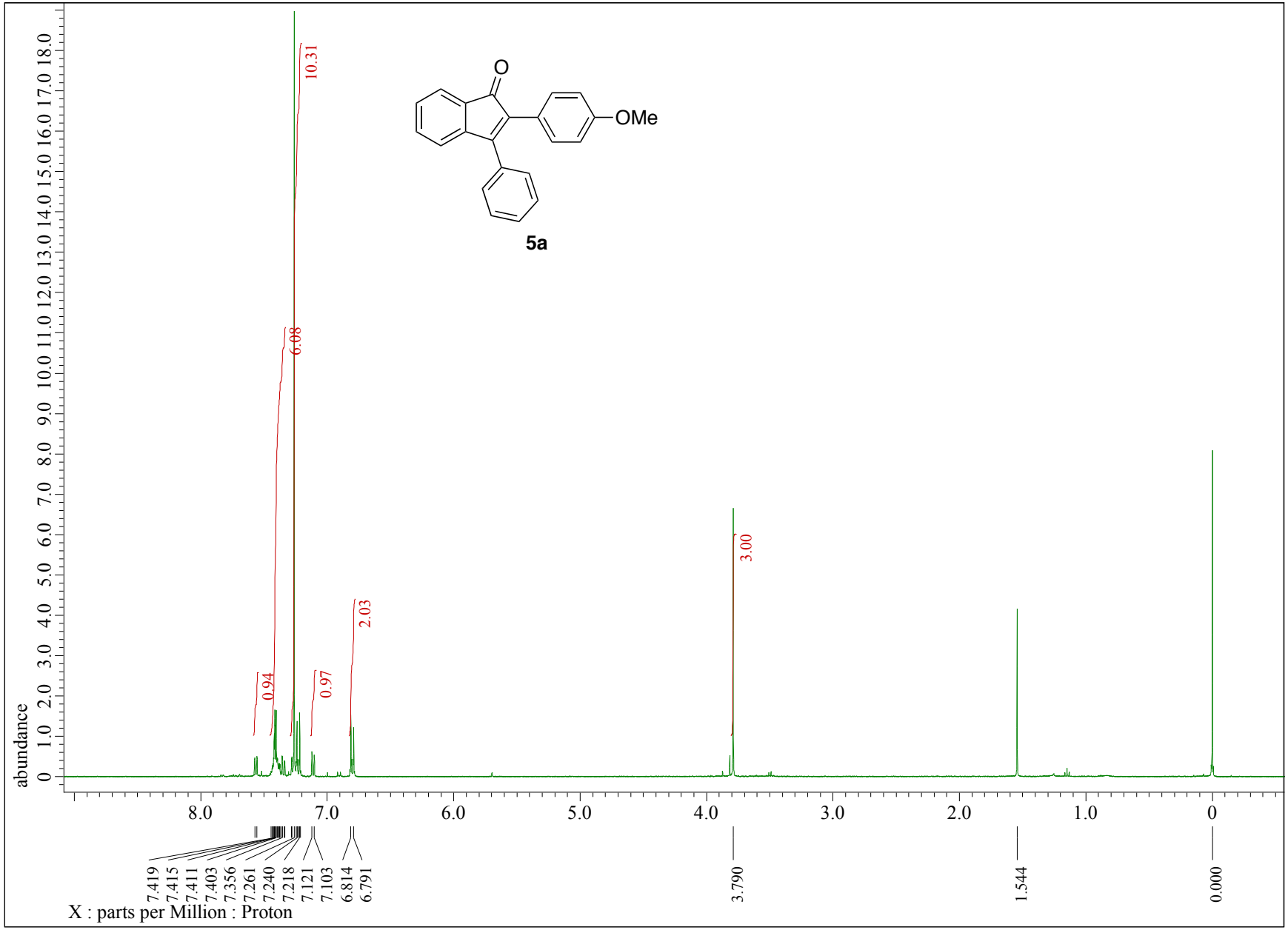

O
F
3r
abundance
0
0.1
0.2
0.3
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
196.859
167.932
165.421
155.550
155.531
149.239
149.144
136.637
126.976
123.520
123.424
113.314
113.085
108.321
108.073
77.475
77.160
76.845
28.270
25.158
22.609
21.349
14.465
14.294
X : parts per Million : Carbon13

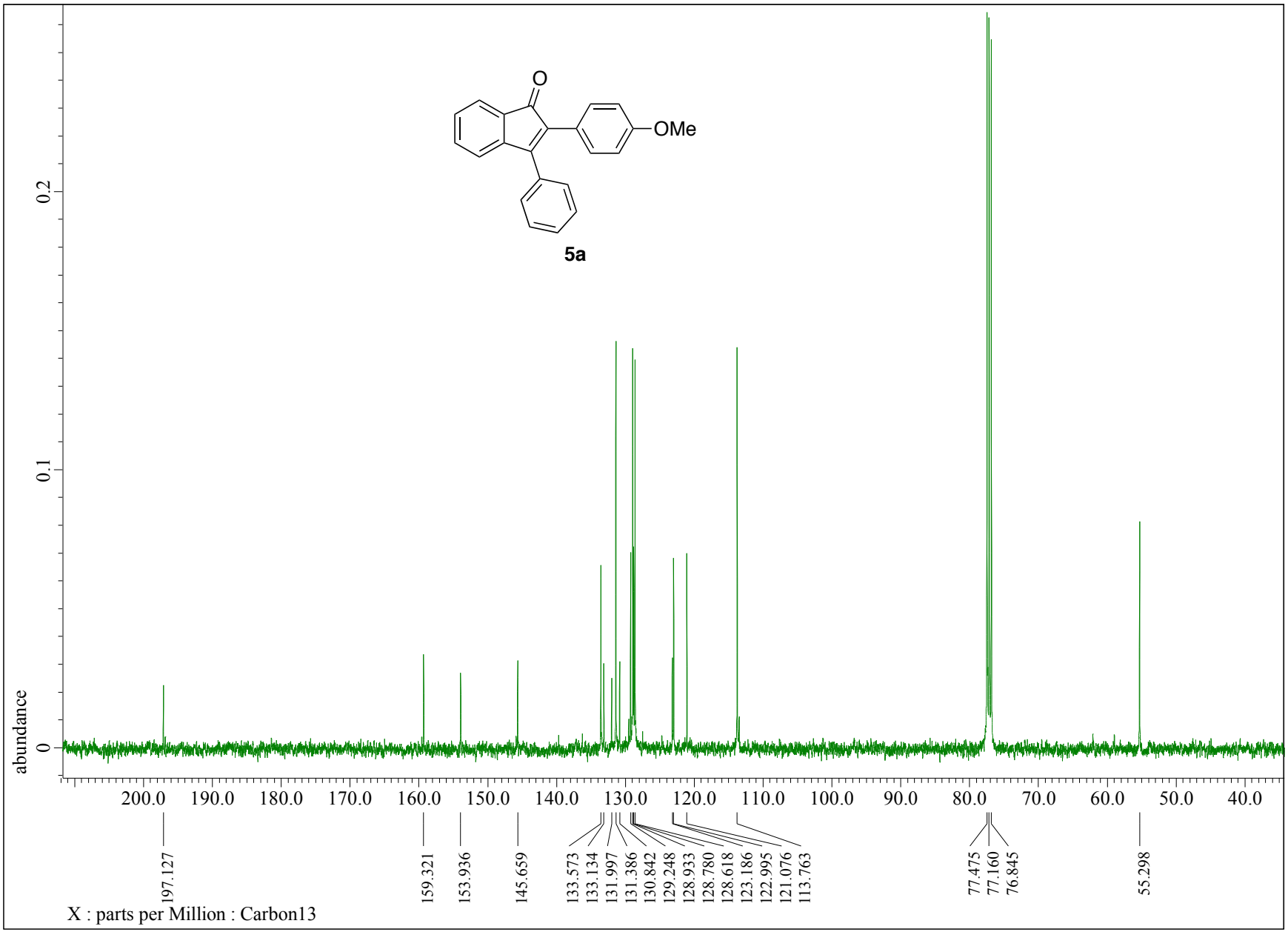

O
OMe
Br
4a
abundance
2.12
1.86
2.63
2.29
3.00
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
-2.0
7.694
7.673
7.490
7.466
7.305
7.261
7.007
6.985
3.860
1.542
0.000
X : parts per Million : Proton

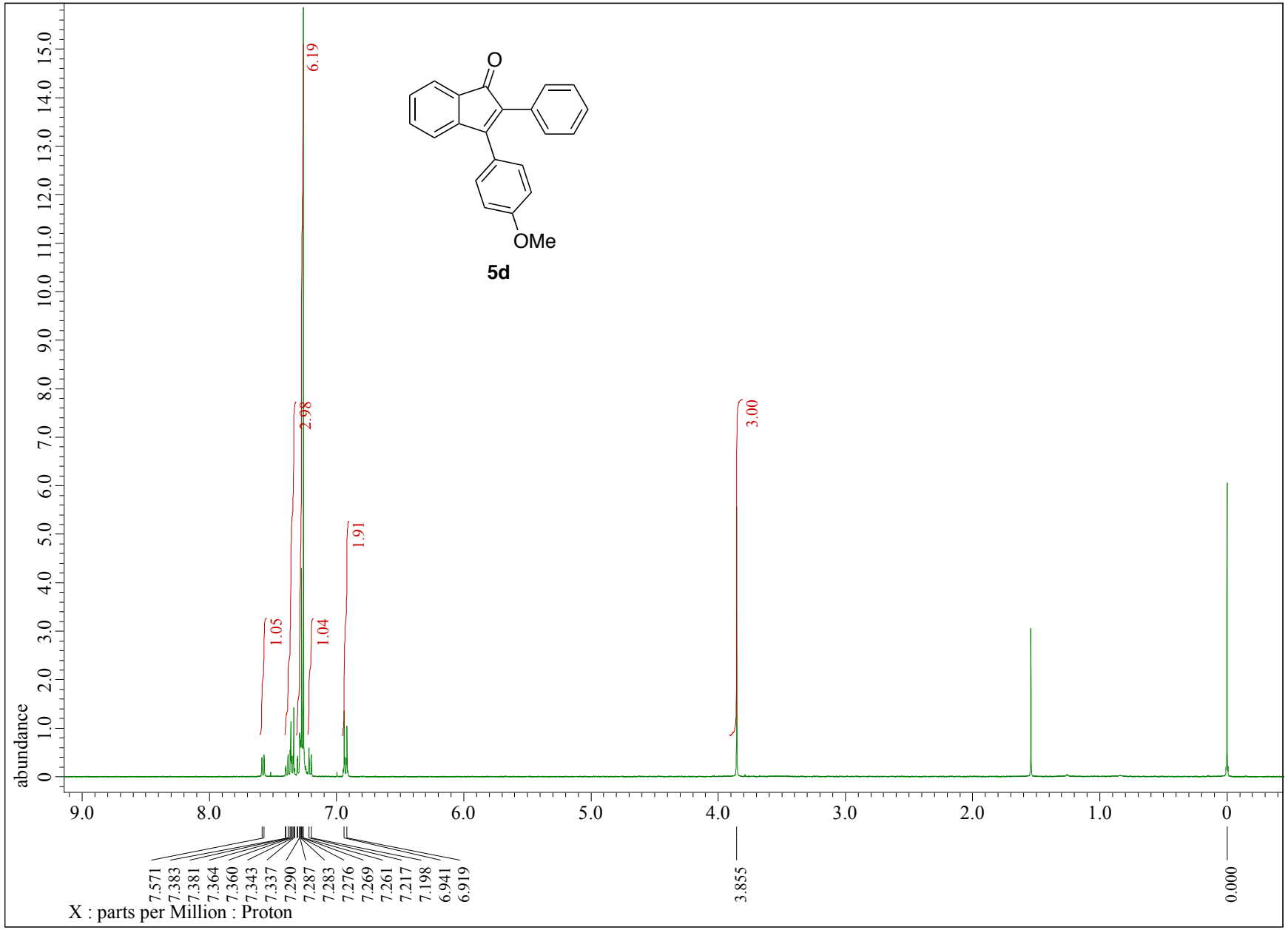

O
OMe
Br
4a
abundance
-0.01 0.01 0.03 0.05 0.07 0.09 0.11 0.13 0.15 0.17 0.19 0.21 0.23 0.25
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
193.021
160.056
143.740
140.456
134.432
134.212
131.033
129.878
129.735
122.307
122.250
121.286
114.202
113.935
77.485
77.160
76.845
55.441
X : parts per Million : Carbon13

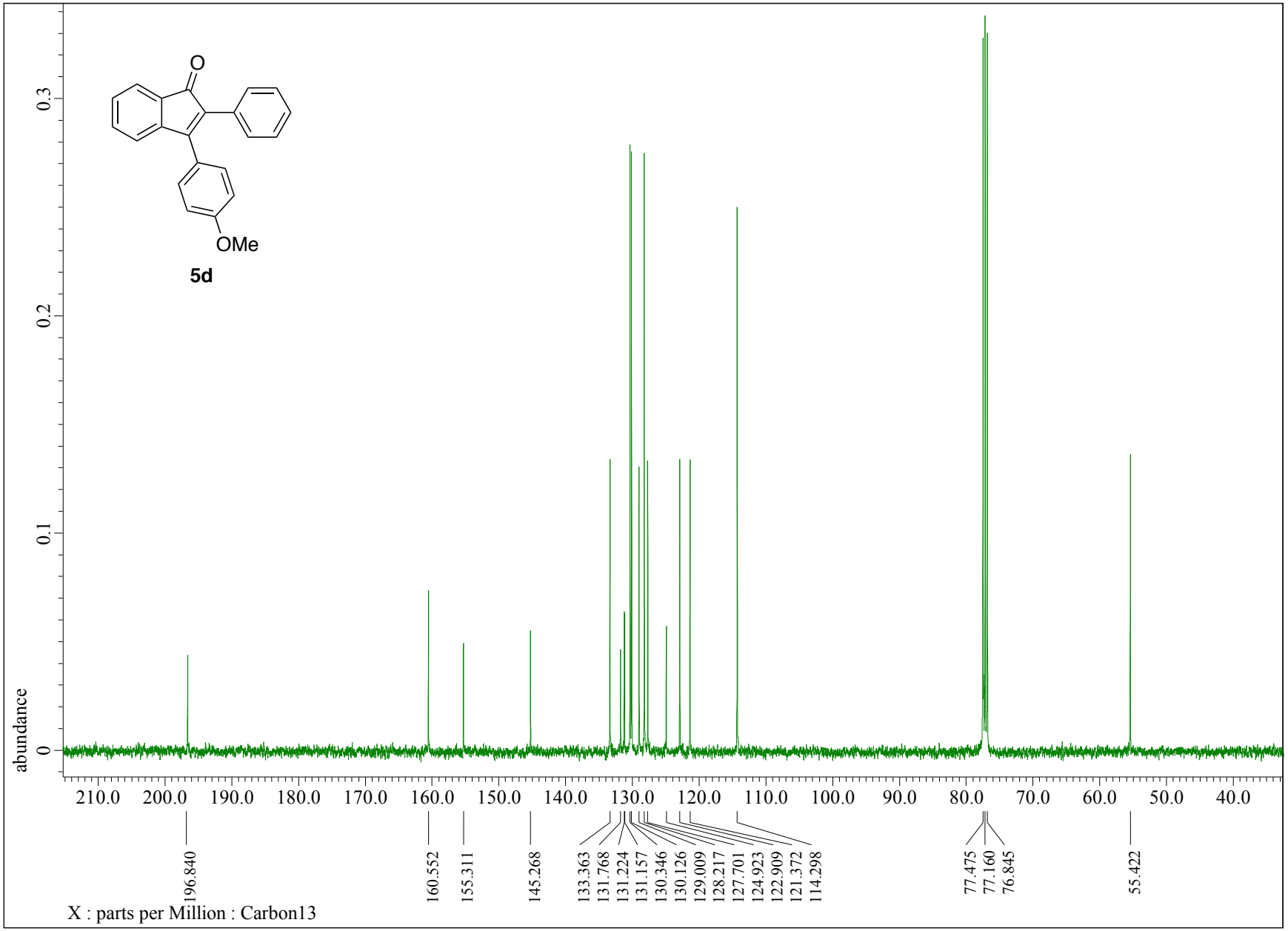

O
Br
4d
4.93
2.00
2.15
abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
-2.0
7.691
7.670
7.516
7.496
7.487
7.468
7.448
7.422
7.406
7.362
7.343
7.311
7.303
7.293
7.261
1.543
-0.000
X : parts per Million : Proton


O
Br
4d
abundance
0
0.1
0.2
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
192.582
143.463
142.289
135.024
134.212
130.079
129.878
129.849
129.601
128.885
128.398
122.412
121.591
77.475
77.160
76.845
X : parts per Million : Carbon13


O
OMe
5a
abundance
10.31
6.98
0.94
0.97
2.03
3.00
7.419
7.415
7.411
7.403
7.356
7.261
7.240
7.218
7.121
7.103
6.814
6.791
3.790
1.544
0.000
X : parts per Million : Proton


O
OMe
5a
abundance
0
0.1
0.2
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
197.127
159.321
153.936
145.659
133.573
133.134
131.997
131.386
130.842
129.248
128.933
128.780
128.618
123.186
122.995
121.076
113.763
77.475
77.160
76.845
55.298
X : parts per Million : Carbon13


O
OMe
5d
6.19
2.98
1.91
1.05
1.04
3.00
abundance
0 1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0 9.0 10.0 11.0 12.0 13.0 14.0 15.0
9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
7.571 7.383 7.381 7.364 7.360 7.343 7.337 7.290 7.287 7.283 7.276 7.269 7.261 7.217 7.198 6.941 6.919
3.855
0.000
X : parts per Million : Proton


O
OMe
5d
abundance
0
0.1
0.2
0.3
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
196.840
160.552
155.311
145.268
133.363
131.768
131.224
131.157
130.346
130.126
129.009
128.217
127.701
124.923
122.909
121.372
114.298
77.475
77.160
76.845
55.422
X : parts per Million : Carbon13