Electronic Supplementary Material (ESI) for Green Chemistry.

Fe-catalyzed decarbonylative alkylation-peroxidation of alkenes with aliphatic aldehydes and hydroperoxide under mild conditions

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Table of Contents

I. General information

Unless otherwise noted, all reactions were run in thick-wall pressure-proof glass tube (Chemglass brand microwave tube, CG-4920-01, claimed to withstand pressures up to 300 PSI (20 bars)) to avoiding the possible crack of the reactor, though it never happened in our lab. All commercially available compounds (Energy-Chemical Company in Shanghai, China) were used as purchased without further purification.

Gas chromatograph (GC) was measured on Shimadzu GC-2014 instrument with a FID detector and *n*-dodecane as the internal standard; Gas Chromatograph-Mass Spectrometer (GC-MS) was measured on Agilent 7890-5975C instrument under the EI ionization model, instead. High-resolution mass spectra (HRMS) were obtained from a JEOL JMS-700 instrument (ESI). Thin-layer chromatography (TLC) was performed using E. Merck silica gel 60 F254 precoated plates (0.25 mm); the developed chromatography was analyzed by UV lamp (254 nm). Melting points are uncorrected.

Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker Avance 400 spectrometer at ambient temperature. Chemical shifts for ^{1}H NMR spectra are reported in parts per million (ppm) from tetramethylsilane with the solvent resonance as the internal standard (chloroform: δ 7.26 ppm, dimethylsulfoxide: δ 2.50 ppm). Chemical shifts for ^{13}C NMR spectra are reported in parts per million (ppm) from tetramethylsilane with the solvent as the internal standard (chloroform: δ 77.16 ppm, dimethylsulfoxide: δ 39.51 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, dd = doublet of doublets, t = triplet, q = quartet, m = multiplet), coupling constant (Hz), and integration.

II. General experimental procedures

(1) Alkylation-peroxidation cascade reaction:

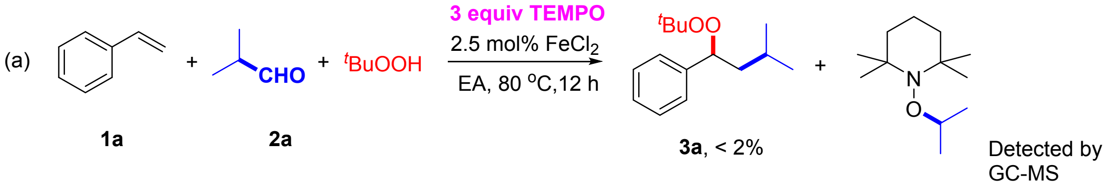
An oven-dried microwave reaction vessel was charged with $FeCl_2$ (2.5 mol%) in EA (1 mL, pre-prepared solution), styrene (**1a**, 0.2 mmol, 1.0 equiv), *tert*-Butyl hydroperoxide (TBHP, 5.5 M in decane, 0.6 mmol, 3.0 equiv) and isobutyraldehyde (**2a**, 0.5 mmol, 2.5 equiv) under argon atmosphere. The vessel was sealed and stirred at 80 ^{o}C (oil bath temperature) for 12 h. Afterwards the resulting mixture was cooled to room temperature, and concentrated in vacuo and purified by column chromatography on silica gel with a mixture of dichloromethane/petroleum ether as eluent to give the product **3a**.

(2) One-pot alkylation-peroxidation cascade reaction:

An oven-dried microwave reaction vessel was charged with $FeCl_2$ (2.5 mol%) in EA (1 mL, pre-prepared solution), styrene (**1a**, 0.2 mmol, 1.0 equiv), *tert*-Butyl hydroperoxide (TBHP, 5.5 M in decane, 0.6 mmol, 3.0 equiv) and isobutyraldehyde (**2a**, 0.5 mmol, 2.5 equiv) under argon atmosphere. The vessel was sealed and heated at 80 °C (oil bath temperature) for 12 h. Afterwards the resulting mixture was cooled to room temperature, and DBU(0.3 mmol, 1.5 equiv) was added and the mixture was heated for another 12 hours at 80 °C. The resulting mixture was cooled to room temperature, concentrated in vacuo and purified by column chromatography on silica gel with a mixture of ethyl acetate/petroleum ether as eluent to give the product **5a**.

III. Radical inhibition experiment

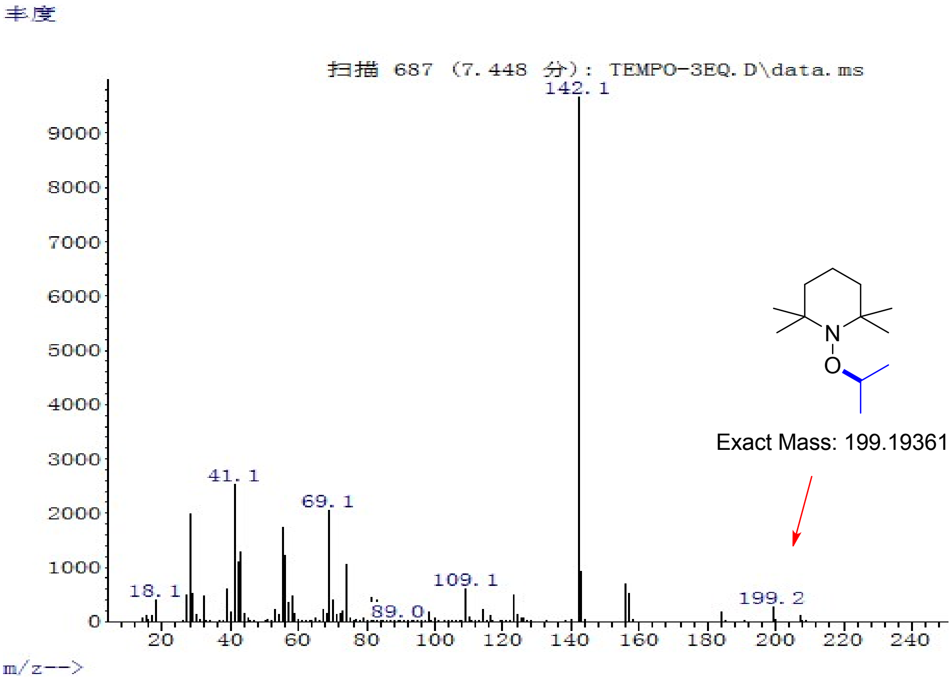
The cascade reaction of **1a** and **2a** was completely inhibited in the presence of 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO), instead, the decarbonylated alkyl radical was captured as 1-isopropoxy-2,2,6,6-tetramethylpiperidine (Scheme 3a). The 1-isopropoxy-2,2,6,6-tetramethylpiperidine was detected by GC-MS.



1a **2a** **3a**, < 2%

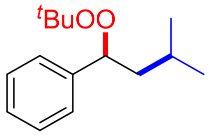
Detected by GC-MS

GC-MS spectrum of the captured alkyl radical: (retention time 7.448 min)



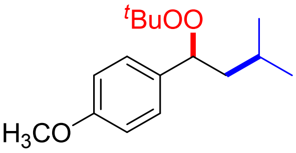
IV. Spectra data of products 3a-3q, 4b-4k, 5a-5k, 6, 7a, 8a

(3a) (1-(*tert*-butylperoxy)-3-methylbutyl)benzene



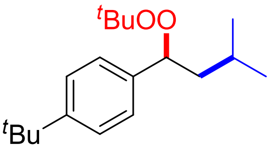
The title compound was prepared according to the general procedure described above by the reaction between styrene (**1a**) with TBHP and isobutyraldehyde (**2a**), and purified by flash column chromatography as colorless oil (32.6 mg, 69%).
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.37 – 7.24 (m, 5H), 4.90 (dd, J = 7.6, 6.4 Hz, 1H), 1.81 – 1.72 (m, 1H), 1.70 – 1.58 (m, 1H), 1.54 – 1.44 (m, 1H), 1.19 (s, 9H), 0.93 (dd, J = 6.4, 4.4 Hz, 6H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 142.40, 128.23, 127.55, 127.04, 84.35, 80.05, 44.54, 26.66, 25.01, 22.99, 22.97. IR (cm^{-1}): 2957, 2929, 2870, 1454, 1362, 1198, 698. HRMS: calcd. for $C_{11}H_{15}O^+$ [M - ^{t}BuO]$^+$: 163.1117; Found: 163.1120.

(3b) 1-(1-(*tert*-butylperoxy)-3-methylbutyl)-4-methoxybenzene



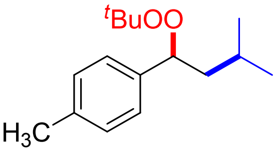
The title compound was prepared according to the general procedure described above by the reaction between 4-methoxylstyrene (**1b**) with TBHP and isobutyraldehyde (**2a**), and purified by flash column chromatography as colorless oil (29.3mg, 55%).
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.25 (d, J = 8.4 Hz, 2H), 6.87 (d, J = 8.4 Hz, 2H), 4.84 (t, J = 7.2 Hz, 1H), 3.80 (s, 3H), 1.82 – 1.72 (m, 1H), 1.65 – 1.56 (m, 1H), 1.55 – 1.45 (m, 1H), 1.18 (s, 9H), 0.91 (dd, J = 6.4, 2.8 Hz, 6H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 159.14, 134.19, 128.42, 113.63, 84.02, 80.01, 55.34, 44.05, 26.66, 25.00, 23.14, 22.79. IR (cm^{-1}): 2956, 2930, 2869, 1513, 1247, 1197, 829. HRMS: calcd. for $C_{12}H_{17}O_2^+$ [M-^{t}BuO]$^+$: 193.1223; Found: 193.1227.

(3c) 1-(*tert*-butyl)-4-(1-(*tert*-butylperoxy)-3-methylbutyl)benzene



The title compound was prepared according to the general procedure described above by the reaction between 4-*tert*-butylstyrene (**1c**) with TBHP and isobutyraldehyde (**2a**), and purified by flash column chromatography as colorless oil (35.0mg, 60%).
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.36 – 7.32 (m, 2H), 7.26 – 7.22 (m, 2H), 4.87 (t, J = 6.8 Hz, 1H), 1.83 – 1.73 (m, 1H), 1.72 – 1.60 (m, 1H), 1.55 – 1.44 (m, 1H), 1.31 (s, 9H), 1.20 (s, 9H), 0.92 (t, J = 6.4 Hz, 6H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 150.30, 139.10, 126.68, 125.15, 84.24, 80.10, 44.50, 34.63, 31.54, 26.68, 24.99, 23.02, 22.97. IR (cm^{-1}): 2959, 2931, 2869, 1467, 1362, 1198, 832. HRMS: calcd. for $C_{15}H_{23}O^+$ [M-^{t}BuO]$^+$: 219.1743; Found: 219.1747.

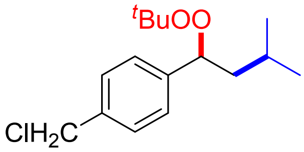
(3d) 1-(1-(*tert*-butylperoxy)-3-methylbutyl)-4-methylbenzene



The title compound was prepared according to the general procedure described above by the reaction between 4-methylstyrene (**1d**) with TBHP and isobutyraldehyde (**2a**), and purified by flash column chromatography as colorless oil (28.5mg, 57%).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.21 (d, J = 8.4 Hz, 2H), 7.14 (d, J = 8.0 Hz, 2H), 4.87 (t, J = 7.2 Hz, 1H), 2.34 (s, 3H), 1.82 – 1.71 (m, 1H), 1.68 – 1.58 (m, 1H), 1.53 – 1.43 (m, 1H), 1.19 (s, 9H), 0.92 (dd, J = 6.4, 3.6Hz, 6H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 139.24, 137.19, 128.96, 127.06, 84.26, 80.02, 44.39, 26.67, 25.01, 23.07, 22.89, 21.33. IR (cm^{-1}): 2957, 2928, 2869, 1467, 1362, 1198, 812. HRMS: calcd. for $C_{12}H_{17}O^+$ [M-^{t}BuO]$^+$: 177.1274; Found:177.1277.

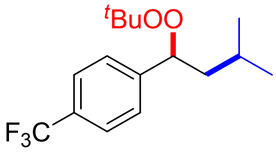
(3e) 1-(1-(*tert*-butylperoxy)-3-methylbutyl)-4-(chloromethyl)benzene



The title compound was prepared according to the general procedure described above by the reaction between 4-chloromethylstyrene (**1e**) with TBHP and isobutyraldehyde (**2a**), and purified by flash column chromatography as colorless oil (33.5mg, 59%).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.39 – 7.29 (m, 4H), 4.90 (dd, J = 8.0, 6.4 Hz, 1H), 4.59 (s, 2H), 1.79 – 1.58 (m, 2H), 1.50 – 1.39 (m, 1H), 1.19 (s, 9H), 0.93 (t, J = 6.4 Hz, 6H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 142.96, 136.59, 128.57, 127.32, 83.93, 80.16, 46.34, 44.65, 26.65, 24.99, 23.00, 22.91. IR (cm^{-1}): 2957, 2929, 2869, 1468, 1385, 1197, 680. HRMS: calcd. for $C_{12}H_{16}OCl^+$ [M- ^{t}BuO]$^+$: 211.0884; Found: 211.0889.

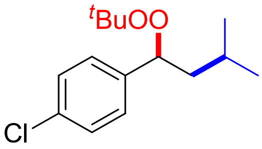
(3f) (1-(*tert*-butylperoxy)-3-methylbutyl)-4-(trifluoromethyl)benzene



The title compound was prepared according to the general procedure described above by the reaction between 4-trifluoromethylstyrene (**1f**) with TBHP and isobutyraldehyde (**2a**), and purified by flash column chromatography as colorless oil (31.6mg, 52%).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.59 (d, J = 8.4 Hz, 2H), 7.44 (d, J = 8.0 Hz, 2H), 4.96 (dd, J = 8.0, 5.6 Hz, 1H), 1.87 – 1.63 (m, 2H), 1.47 – 1.35 (m, 1H), 1.20 (s, 9H), 0.94 (dd, J = 9.6, 6.8 Hz, 6H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 146.94, 129.63 (q, J = 32.2 Hz), 127.06, 125.26 (q, J = 3.7 Hz), 124.43 (d, J = 270.3 Hz), 83.59, 80.33, 44.86, 26.61, 24.99, 23.08, 22.75. IR (cm^{-1}): 2960, 2932, 2872, 1469, 1364, 1197, 607. HRMS: calcd. for $C_{16}H_{23}F_3O_2Na^+$ [M+Na]$^+$: 327.1542; Found: 327.1541.

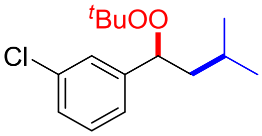
(3g) 1-(1-(*tert*-butylperoxy)-3-methylbutyl)-4-chlorobenzene



The title compound was prepared according to the general procedure described above by the reaction between 4-chlorostyrene (**1g**) with TBHP and isobutyraldehyde (**2a**), and purified by flash column chromatography as colorless oil (36.2mg, 67%).

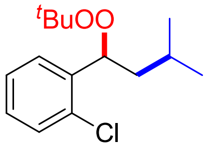
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.33 – 7.23 (m, 4H), 4.87 (dd, J = 7.6, 6.4 Hz, 1H), 1.77 – 1.59 (m, 2H), 1.47 – 1.38 (m, 1H), 1.18 (s, 9H), 0.92 (t, J = 6.4 Hz, 6H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 141.11, 133.15, 128.43, 128.37, 83.58, 80.18, 44.50, 26.63, 24.98, 22.96, 22.90. IR (cm^{-1}): 2958, 2929, 2870, 1490, 1362, 1197, 819. HRMS: calcd. for $C_{11}H_{14}OCl^+$ [M-^{t}BuO]$^+$: 197.0728; Found: 197.0730.

(3h) 1-(1-(*tert*-butylperoxy)-3-methylbutyl)-3-chlorobenzene



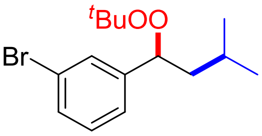
The title compound was prepared according to the general procedure described above by the reaction between 3-chlorostyrene (**1h**) with TBHP and isobutyraldehyde (**2a**), and purified by flash column chromatography as colorless oil (32.9mg, 61%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.19 (m, 4H), 4.86 (dd, J = 7.6, 6.0 Hz, 1H), 1.78 – 1.61 (m, 2H), 1.48 – 1.37 (m, 1H), 1.19 (s, 9H), 0.93 (dd, J = 7.2, 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.90, 134.12, 129.55, 127.63, 127.07, 125.08, 83.62, 80.25, 44.67, 26.64, 24.99, 23.05, 22.82. IR (cm^{-1}): 2958, 2930, 2870, 1470, 1362, 1196, 782. HRMS: calcd. for $C_{11}H_{14}OCl^+$ [M- ^{t}BuO]$^+$: 197.0728; Found: 197.0731.

(3i) 1-(1-(*tert*-butylperoxy)-3-methylbutyl)-2-chlorobenzene



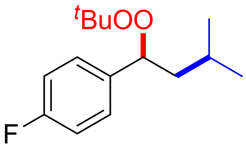
The title compound was prepared according to the general procedure described above by the reaction between 2-chlorostyrene (**1i**) with TBHP and isobutyraldehyde (**2a**), and purified by flash column chromatography as colorless oil (32.9mg, 64%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (dd, J = 8.0, 2.0 Hz, 1H), 7.18 (td, J = 8.0, 1.2 Hz, 2H), 7.18 (td, J = 8.0, 2.0 Hz, 1H), 5.44 (dd, J = 9.2, 4.4 Hz, 1H), 1.92 – 1.76 (m, 1H), 1.65 – 1.55 (m, 1H), 1.53 – 1.42(m, 1H), 1.23 (s, 9H), 1.02 (d, J = 6.8 Hz, 3H), 0.94 (d, J = 6.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.56, 132.39, 129.33, 128.25, 127.83, 126.93, 80.41, 80.32, 44.16, 26.65, 25.25, 23.61, 22.24. IR (cm^{-1}): 2958, 2931, 2870, 1469, 1362, 1197, 752. HRMS: calcd. for $C_{11}H_{14}OCl^+$ [M-^{t}BuO]$^+$: 197.0728; Found: 197.0729.

(3j) 1-bromo-3-(1-(*tert*-butylperoxy)-3-methylbutyl)benzene



The title compound was prepared according to the general procedure described above by the reaction between 3-boromostyrene (**1j**) with TBHP and isobutyraldehyde (**2a**), and purified by flash column chromatography as colorless oil (43.3mg, 69%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 – 7.44 (m, 1H), 7.41 – 7.37 (m, 1H), 7.26 – 7.15(m, 2H), 4.85 (dd, J = 8.0, 5.6 Hz, 1H), 1.76 – 1.61 (m, 2H), 1.45 – 1.37 (m, 1H), 1.19 (s, 9H), 0.93 (dd, J = 8.0, 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.20, 130.56, 129.99, 129.88, 125.54, 122.39, 83.56, 80.27, 44.70, 26.64, 24.99, 23.06, 22.81. IR (cm^{-1}): 2957, 2929, 2869, 1469, 1362, 1195, 780. HRMS: calcd. for $C_{11}H_{14}OBr^+$ [M-^{t}BuO]$^+$: 241.0223; Found: 241.0224.

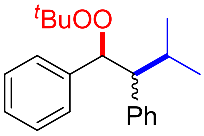
(3k) 1-(1-(*tert*-butylperoxy)-3-methylbutyl)-4-fluorobenzene



The title compound was prepared according to the general procedure described above by the reaction between 4-fluorostyrene (**1k**) with TBHP and isobutyraldehyde (**2a**), and purified by flash column chromatography as colorless oil (30.5mg, 60%).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.34 – 7.26 (m, 2H), 7.06 – 6.98 (m, 2H), 4.87 (t, J = 7.2 Hz, 1H), 1.80 – 1.69 (m, 1H), 1.68 – 1.58 (m, 1H), 1.50 – 1.40 (m, 1H), 1.18 (s, 9H), 0.92 (dd, J = 6.4, 4.8 Hz, 6H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 162.34 (d, J = 243.3 Hz), 138.17 (d, J = 3.1 Hz), 128.64 (d, J = 7.9 Hz), 115.07 (d, J = 21.1 Hz), 83.63, 80.12, 44.43, 26.64, 24.99, 22.97, 22.91. IR (cm^{-1}): 2958, 2930, 2871, 1510, 1363, 1223, 834. HRMS: calcd. for $C_{11}H_{14}OF^+$ [M-^{t}BuO]$^+$: 181.1023; Found: 181.1023.

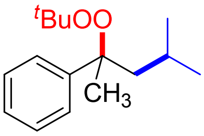
(3l) (1-(*tert*-butylperoxy)-3-methylbutane-1,2-diyl)dibenzene



The title compound was prepared according to the general procedure described above by the reaction between (E)-1,2-diphenylethene (**1l**) with TBHP and isobutyraldehyde (2a), and purified by flash column chromatography as colorless oil (24.9mg, 40%, dr ≈1:1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.25 – 6.85 (m, 10H), 5.37 (d, J = 6.0 Hz, 0.5×1H), 5.26 (d, J = 8.8 Hz, 0.5×1H), 2.96 (dd, J = 8.8, 6.0 Hz, 0.5×1H), 2.50 (dd, J = 8.4, 6.4 Hz, 0.5×1H), 2.42 – 2.28 (m, 0.5×1H), 2.18 – 2.04 (m, 0.5×1H), 1.19 (s, 4.5×1H), 1.14 (s, 4.5×1H), 1.06 (d, J = 6.4 Hz, 1.5×1H), 0.96 (d, J = 6.8 Hz, 1.5×1H), 0.91 (d, J = 6.8 Hz, 1.5×1H), 0.72 (d, J = 6.8 Hz, 1.5×1H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 141.72, 140.77, 139.44, 138.68, 130.42, 130.16, 127.94, 127.65, 127.40, 127.37, 127.03, 126.96, 126.17, 126.13, 85.96, 84.95, 79.91, 79.86, 59.94, 57.05, 29.40, 28.32, 26.80, 26.74, 22.14, 21.85, 20.61, 19.12. IR (cm^{-1}): 2976, 2928, 2872, 1452, 1362, 1197, 700. HRMS: calcd. for $C_{21}H_{28}O_2Na^+$ [M+Na]$^+$: 335.1982; Found: 335.1976.

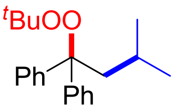
(3m) (2-(*tert*-butylperoxy)-4-methylpentan-2-yl)benzene



The title compound was prepared according to the general procedure described above by the reaction between α-methylstyrene (**1m**) with TBHP and isobutyraldehyde (**2a**), and purified by flash column chromatography as colorless oil (30.0mg, 60%).

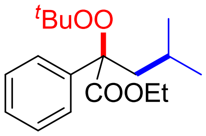
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.43 – 7.38 (m, 2H), 7.33 – 7.27 (m, 2H), 7.24 – 7.17 (m, 1H), 1.74 – 1.65 (m, 2H), 1.64 (s, 3H), 1.62 – 1.55(m, 1H), 1.24 (s, 9H), 0.81 (dd, J = 10.4, 6.4 Hz, 6H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 146.23, 127.79, 126.42, 126.01, 84.31, 78.73, 50.75, 26.94, 24.75, 24.60, 24.44, 24.30. IR (cm^{-1}): 2978, 2955, 1446, 1362, 1199, 698. HRMS: calcd. for $C_{16}H_{26}O_2Na^+$ [M+Na]$^+$: 273.1825; Found: 273.1818.

(3n) (1-(*tert*-butylperoxy)-3-methylbutane-1,1-diyl)dibenzene



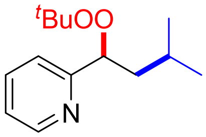
The title compound was prepared according to the general procedure described above by the reaction between 1,1-diphenylethene (**1n**) with TBHP and isobutyraldehyde (**2a**), and purified by flash column chromatography as colorless oil (39.9mg, 64%).
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.37 – 7.17 (m, 10H), 2.33 (d, J = 5.2 Hz, 2H), 1.66 – 1.56 (m, 1H), 1.08 (s, 9H), 0.77 (d, J = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 145.22, 127.68, 127.46, 126.66, 87.15, 79.05, 45.48, 26.72, 24.49, 23.69. IR (cm^{-1}): 2954, 2928, 2868, 1446, 1362, 1197, 642. HRMS: calcd. for $C_{21}H_{28}O_2Na^+$ [M+Na]$^+$: 335.1982; Found: 335.1974.

(3o) ethyl 2-(*tert*-butylperoxy)-4-methyl-2-phenylpentanoate



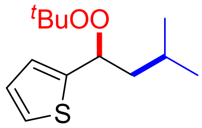
The title compound was prepared according to the general procedure described above by the reaction between ethyl 2-phenylacrylate (**1o**) with TBHP and isobutyraldehyde (**2a**), and purified by flash column chromatography as colorless oil (27.7mg, 45%).
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.40 – 7.23 (m, 5H), 4.23 – 4.07 (m, 2H), 2.34 (dd, J = 14.8, 6.8 Hz, 1H), 2.09 (dd, J = 14.8, 5.2 Hz, 1H), 1.58 – 1.44 (m, 1H), 1.30 (s, 9H), 1.20 (t, J = 7.2 Hz, 3H), 0.91 (d, J = 6.8 Hz, 3H), 0.64 (d, J = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 172.49, 139.25, 128.01, 127.43, 125.63, 87.61, 80.18, 61.08, 42.94, 26.79, 24.42, 23.76, 23.48, 14.12. IR (cm^{-1}): 2956, 2932, 2870, 1736, 1449, 1364, 1243, 1197, 705. HRMS: calcd. for $C_{18}H_{28}O_4Na^+$ [M+Na]$^+$: 331.1880; Found: 331.1882.

(3p) 2-(1-(tert-butylperoxy)-3-methylbutyl)pyridine



The title compound was prepared according to the general procedure described above but prolong time to 24 hours by the reaction between 2-vinylpyridine (**1p**) with TBHP and isobutyraldehyde (**2a**), and purified by flash column chromatography as colorless oil (17.5mg, 37%).
^{1}H NMR (400 MHz, CDCl$_3$) δ 8.54 (d, J = 4.4 Hz, 1H), 7.68 (td, J = 8.0, 2.0 Hz, 1H), 7.47 (d, J = 8.0 Hz, 1H), 7.16 (ddd, J = 7.2, 4.8, 0.8 Hz, 1H), 5.06 (dd, J = 9.2, 5.2 Hz, 1H), 1.87 – 1.72 (m, 1H), 1.71 – 1.62(m, 1H), 1.62 – 1.52 (m, 1H), 1.22 (s, 9H), 0.99 (d, J = 6.4Hz, 3H), 0.95 (d, J = 6.4 Hz, 3H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 162.58, 148.80, 136.53, 122.30, 121.09, 85.27, 80.41, 43.97, 26.60, 25.02, 23.46, 22.48. IR (cm^{-1}): 2957, 2932, 2870, 1591, 1470, 1385, 1362, 1197. HRMS: calcd. for $C_{14}H_{23}NO_2Na^+$ [M+Na]$^+$: 260.1621; Found: 260.1624.

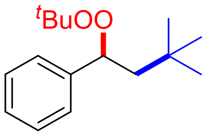
(3q) 2-(1-(tert-butylperoxy)-3-methylbutyl)thiophene



The title compound was prepared according to the general procedure described above but prolong time to 24 hours by the reaction between 2-vinylthiophene (**1q**) with TBHP and isobutyraldehyde (**2a**), and purified by flash column chromatography as colorless oil (15.5mg, 32%).
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.27 – 7.24 (m, 1H), 7.02 (dd, J = 3.2, 0.8 Hz, 1H), 6.96 (dd, J = 4.8, 3.2 Hz, 1H), 5.12 (t, J = 7.2 Hz, 1H), 1.94 – 1.84 (m, 1H), 1.74 – 1.59 (m, 2H), 1.18 (s, 9H), 0.94 (dd, J = 6.4, 4.0 Hz, 6H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 145.19, 126.37, 125.70, 124.95, 80.26,

79.51, 44.08, 26.56, 25.12, 23.01, 22.67. IR (cm^{-1}): 2958, 2930, 2870, 1467, 1385, 1363, 1197, 697. HRMS: calcd. for $C_{13}H_{22}SO_2Na^+$ [M+Na]$^+$: 265.1233; Found: 265.1236.

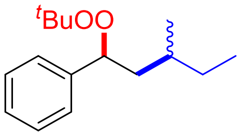
(4b) (1-(*tert*-butylperoxy)-3,3-dimethylbutyl)benzene [1]



The title compound was prepared according to the general procedure described above by the reaction between styrene (**1a**) with TBHP and pivaldehyde (**2b**), and purified by flash column chromatography as colorless oil (39.5mg, 79%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.22 (m, 5H), 4.94 (dd, J = 8.0, 4.4 Hz, 1H), 1.77 (dd, J = 14.8, 8.0 Hz, 1H), 1.52 (dd, J = 14.8, 4.4 Hz, 1H), 1.17 (s, 9H), 0.96 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.88, 128.15, 127.30, 126.99, 83.40, 79.50, 49.47, 30.49, 30.32, 26.83. IR (cm^{-1}): 3030, 2955, 2868, 1455, 1362, 1198, 698.

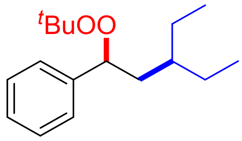
(4c) (1-(*tert*-butylperoxy)-3-methylpentyl)benzene



The title compound was prepared according to the general procedure described above by the reaction between styrene (**1a**) with TBHP and 2-methylbutanal (**2c**), and purified by flash column chromatography as colorless oil (36.5 mg, 73%, dr ≈1:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37– 7.24 (m, 5H), 4.95 – 4.90 (m, 1H), 1.90 – 1.81 (m, 0.55×1H), 1.75 – 1.59 (m, 1H), 1.54 – 1.47 (m, 0.49×1H), 1.45 – 1.28 (m, 2H), 1.25 – 1.20 (m, 0.45×1H), 1.19 (s, 4.76×1H), 1.18 (s, 4.30×H), 1.17 – 1.11 (m, 0.52×1H), 0.95 – 0.78 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.83, 142.07, 128.23, 128.22, 127.62, 127.45, 127.22, 126.88, 84.57, 83.99, 80.06, 80.05, 42.86, 41.83, 31.24, 31.23, 29.77, 26.67, 26.65, 19.54, 19.51, 11.22·. IR (cm^{-1}): 2964, 2930, 2875, 1455,1362, 1197, 699. HRMS: calcd. for $C_{12}H_{17}O^+$ [M-^{t}BuO]$^+$: 177.1274; Found: 177.1275.

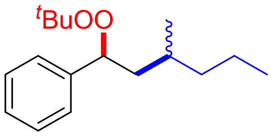
(4d) (1-(*tert*-butylperoxy)-3-ethylpentyl)benzene



The title compound was prepared according to the general procedure described above by the reaction between styrene (**1a**) with TBHP and 2-ethylbutanal (**2d**), and purified by flash column chromatography as colorless oil (32.7 mg, 62%).

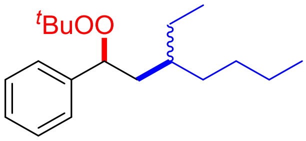
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.22 (m, 5H), 4.90 (t, J = 7.2 Hz, 1H), 1.84 – 1.74 (m, 1H), 1.57 – 1.49 (m, 1H), 1.43 – 1.26 (m, 5H), 1.19 (s, 9H), 0.89 – 0.76 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.53, 128.22, 127.53, 127.05, 84.40, 80.05, 38.88, 37.06, 26.66, 25.64, 25.60, 10.70, 10.62. IR (cm^{-1}): 2963, 2932, 2875, 1455, 1362, 1198, 698. HRMS: calcd. for $C_{13}H_{19}O^+$ [M-^{t}BuO]$^+$: 191.1430; Found:191.1434.

(4e) (1-(*tert*-butylperoxy)-3-methylhexyl)benzene



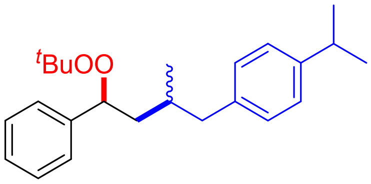
The title compound was prepared according to the general procedure described above by the reaction between styrene (**1a**) with TBHP and 2-methylpentanal (**2e**), and purified by flash column chromatography as colorless oil (36.9 mg, 70%, dr ≈1:1).
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.38 – 7.22 (m, 5H), 4.92 (m, 1H), 1.89 – 1.79 (m, 0.47×1H), 1.75 – 1.59 (m, 1.54×1H), 1.44 – 1.21(m, 4H), 1.19 (s, 4.27×1H), 1.18 (s, 4.73×1H), 1.16 – 1.01 (m, 1H), 0.97 – 0.77 (m, 6H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 142.86, 142.07, 128.23, 128.22, 127.62, 127.43, 127.22, 126.86, 84.54, 83.97, 80.05, 80.03, 43.29, 42.20, 39.60, 29.47, 29.43, 26.67, 26.65, 20.07, 20.03, 19.99, 14.43, 14.41. IR (cm^{-1}): 2958, 2929, 2872, 1454, 1362, 1198, 698. HRMS: calcd. for $C_{13}H_{19}O^+$ [M-^{t}BuO]$^+$: 191.1430; Found: 191.1433.

(4f) (1-(*tert*-butylperoxy)-3-ethylheptyl)benzene



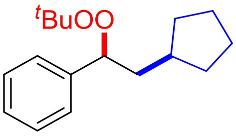
The title compound was prepared according to the general procedure described above by the reaction between styrene (**1a**) with TBHP and 2-ethylhexanal (**2f**), and purified by flash column chromatography as colorless oil (37.4 mg, 64%, dr ≈1:1).
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.38 – 7.22 (m, 5H), 4.90 (t, J = 7.2 Hz, 1H), 1.86 – 1.73 (m, 1H), 1.57 – 1.49 (m, 1H), 1.42 – 1.20 (m, 9H), 1.19 (s, 9H), 0.94 – 0.77 (m, 6H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 142.59, 142.49, 128.21, 127.52, 127.51, 127.07, 127.03, 84.44, 84.38, 80.02, 39.39, 39.25, 35.65, 33.06, 33.01, 28.75, 28.67, 26.66, 26.18, 26.12, 23.21, 23.17, 14.28, 10.67, 10.56. IR (cm^{-1}): 2960, 2929, 2873, 1455, 1362, 1198, 698. HRMS: calcd. for $C_{19}H_{32}O_2Na^+$ [M+Na]$^+$: 315.2295, Found: 315.2275.

(4g) 1-(4-(*tert*-butylperoxy)-2-methyl-4-phenylbutyl)-4-isopropylbenzene



The title compound was prepared according to the general procedure described above by the reaction between styrene (**1a**) with TBHP and 3-(4-isopropylphenyl)-2-methylpropanal (**2g**), and purified by flash column chromatography as colorless oil (51.7mg, 73%, dr ≈1:1).
^{1}H NMR (400 MHz, CDCl3) δ 7.36 – 7.24 (m, 5H), 7.14 – 6.97 (m, 4H), 4.99 – 4.88 (m, 1H), 2.94 – 2.79 (m, 1H), 2.771– 2.61(m, 1H), 2.40 – 2.27 (m, 1H), 2.00 – 1.84 (m, 1H), 1.80 – 1.64 (m, 1.5×1H), 1.50 – 1.42 (m, 0.5×1H), 1.23 (d, J = 6.8 Hz, 6H), 1.19 (s, 9H), 0.90 (dd, J = 6.4, 4.8 Hz, 3H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 146.39, 146.37, 142.49, 141.64, 138.30, 138.25, 129.24, 128.26, 128.23, 127.70, 127.56, 127.35, 127.00, 126.29, 126.26, 84.56, 84.03, 80.07, 43.39, 43.35, 42.89, 41.61, 33.83, 31.81, 31.79, 26.69, 24.22, 19.99, 19.96. IR (cm^{-1}): 2960, 2928, 2870, 1454, 1362, 1197, 698. HRMS: calcd. for $C_{24}H_{34}O_2Na^+$ [M+Na]$^+$: 377.2451; Found: 377.2445.

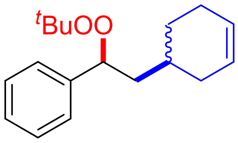
(4h) (1-(*tert*-butylperoxy)-2-cyclopentylethyl)benzene



The title compound was prepared according to the general procedure described above by the reaction between styrene (**1a**) with TBHP and cyclopentanecarbaldehyde (**2h**), and purified by flash column chromatography as colorless oil (29.3 mg, 56%).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.38 – 7.22 (m, 5H), 4.85 (t, J = 7.2 Hz, 1H), 1.94 – 1.84 (m, 1H), 1.84 – 1.70 (m, 3H), 1.70 – 1.40 (m, 5H), 1.19 (s, 9H), 1.16 – 1.08 (m, 2H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 142.27, 128.21, 127.56, 127.10, 85.63, 80.08, 41.71, 36.75, 33.19, 32.92, 26.65, 25.30, 25.17. IR (cm^{-1}): 3030, 2950, 2868, 1454, 1362, 1197, 698. HRMS: calcd. for $C_{13}H_{17}O^+$ [M-^{t}BuO]$^+$: 189.1274; Found: 189.1278.

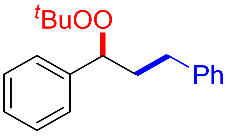
(4i) (1-(*tert*-butylperoxy)-2-(cyclohex-3-en-1-yl)ethyl)benzene



The title compound was prepared according to the general procedure described above by the reaction between styrene (**1a**) with TBHP and cyclohex-3-enecarbaldehyde (**2i**), and purified by flash column chromatography as colorless oil (24.7mg, 45%, dr ≈1:1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.38 – 7.22 (m, 5H), 5.63 (t, J = 12.8 Hz , 2H), 4.95 (dd, J = 7.6, 6.4 Hz 1H), 2.14 (d, J = 15.2 Hz, 1H), 2.07 – 1.93 (m, 2H), 1.90 – 1.50 (m, 5H), 1.36 – 1.24 (m, 1H), 1.19 (s, 9H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 142.42, 142.22, 128.28, 128.26, 127.62, 127.58, 127.11, 127.07, 126.98, 126.44, 126.37, 83.91, 83.81, 80.12, 80.08, 42.29, 42.23, 32.19, 32.09, 30.38, 30.32, 29.17, 29.02, 26.67, 26.64, 25.09. IR (cm^{-1}): 2977, 2924, 2837, 1453, 1362, 1197, 698. HRMS: calcd. for $C_{14}H_{17}O^+$ [M-^{t}BuO]$^+$: 201.1274; Found: 201.1277.

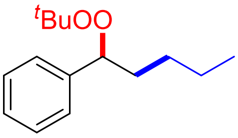
(4j) (1-(*tert*-butylperoxy)propane-1,3-diyl)dibenzene



The title compound was prepared according to the general procedure described above by the reaction between styrene (**1a**) with TBHP and 2-phenylacetaldehyde (**2j**), and purified by flash column chromatography as colorless oil (19.3mg, 34%).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.37 – 7.22 (m, 7H), 7.19 – 7.13 (m, 3H), 4.86 (t, J = 6.8 Hz, 1H), 2.77 – 2.57 (m, 2H), 2.30 – 2.19 (m, 1H), 2.06 – 1.94 (m, 1H), 1.20 (s, 9H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 141.89, 141.47, 128.48, 128.33, 127.79, 127.17, 125.98, 85.37, 80.28, 36.65, 32.18, 26.66. IR (cm^{-1}): 3027, 2977, 2929, 1454, 1362, 1198, 698. HRMS: calcd. for $C_{19}H_{24}O_2Na^+$ [M+ Na]$^+$: 307.1669; Found: 307.1681.

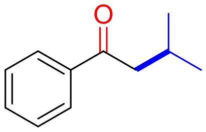
(4k) (1-(tert-butylperoxy)pentyl)benzene



The title compound was prepared according to the general procedure described above by the reaction

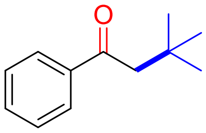
between styrene (**1a**) with TBHP and *n*-butanal (**2k**), and purified by flash column chromatography as colorless oil (7.1mg, 15%).
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.36 – 7.26 (m, 5H), 4.81 (t, J = 6.8 Hz, 1H), 1.95 – 1.82 (m, 1H), 1.72 – 1.57 (m, 1H), 1.37 – 1.22 (m, 4H), 1.20 (s, 9H), 0.87 (t, J = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 142.05, 128.23, 127.58, 127.08, 86.20, 80.21, 34.98, 28.21, 26.65, 22.85, 14.08. IR (cm^{-1}): 2931, 1454, 1362, 1197, 698.

(5a) 3-methyl-1-phenylbutan-1-one



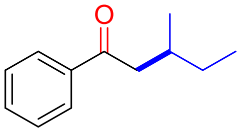
The title compound was prepared according to the general procedure described above by the reaction between styrene (**1a**) with TBHP and isobutyraldehyde (**2a**) and DBU, and purified by flash column chromatography as colorless oil (23.0mg, 71%).
^{1}H NMR (400 MHz, CDCl$_3$) δ 8.00– 7.91(m, 2H), 7.59 – 7.52 (m, 1H), 7.50 – 7.43 (m, 2H), 2.84 (d, J = 6.8 Hz, 2H), 2.38 – 2.22 (m, 1H), 1.00 (d, J = 6.4 Hz, 6H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 200.46, 137.55, 132.99, 128.68, 128.25, 47.66, 25.31, 22.91. IR (cm^{-1}): 2957, 2929, 2871, 1687, 1213, 751, 691.

(5b) 3,3-dimethyl-1-phenylbutan-1-one



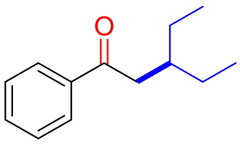
The title compound was prepared according to the general procedure described above by the reaction between styrene (**1a**) with TBHP and pivaldehyde (**2b**) and DBU, and purified by flash column chromatography as colorless oil (30.6mg, 87%).
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.97 – 7.90 (m, 2H), 7.59 – 7.50 (m, 1H), 7.49 – 7.39 (m, 2H), 2.87 (s, 2H), 1.07 (s, 9H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 200.66, 138.75, 132.84, 128.62, 128.36, 50.22, 31.56, 30.23. IR (cm^{-1}): 2955, 2868, 1675, 1448, 749, 690.

(5c) 3-methyl-1-phenylpentan-1-one



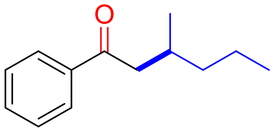
The title compound was prepared according to the general procedure described above by the reaction between styrene (**1a**) with TBHP and 2-methylbutanal (**2c**) and DBU, and purified by flash column chromatography as colorless oil (25.0mg, 71%).
^{1}H NMR (400 MHz, CDCl$_3$) δ 8.01 – 7.91 (m, 2H), 7.60 – 7.52 (m, 1H), 7.50 –7.40(m, 2H), 2.96 (dd, J = 15.6, 5.6 Hz, 1H), 2.75 (dd, J = 15.6, 8.0 Hz, 1H), 2.18 –2.00 (m, 1H), 1.53 – 1.35 (m, 1H), 1.35 – 1.19 (m, 1H), 0.94 (dd, J = 14.4, 6.8 Hz, 6H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 200.70, 137.63, 132.97, 128.68, 128.25, 45.76, 31.56, 29.87, 19.68, 11.56. IR (cm^{-1}): 2961, 2928, 2875, 1686, 1206, 750, 690.

(5d) 3-ethyl-1-phenylpentan-1-one



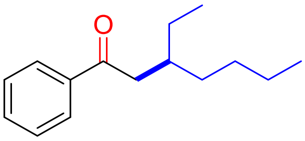
The title compound was prepared according to the general procedure described above by the reaction between styrene (**1a**) with TBHP and 2-ethylbutanal (**2d**) and DBU, and purified by flash column chromatography as colorless oil(30.8mg, 81%) .
^{1}H NMR (400 MHz, CDCl$_3$) δ 8.00 – 7.90 (m, 2H), 7.60 – 7.52 (m, 1H), 7.50 – 7.42 (m, 2H), 2.87 (d, J = 6.8 Hz, 2H), 2.09 – 1.88 (m, 1H), 1.45 – 1.31 (m, 4H), 0.89 (t, J = 7.2 Hz, 6H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 200.98, 137.61, 132.93, 128.66, 128.21, 42.76, 37.30, 26.11, 11.06. IR (cm^{-1}): 2961, 2931, 2876, 1686, 1459, 1272, 1202, 690.

(5e) 3-methyl-1-phenylhexan-1-one



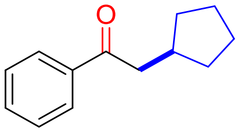
The title compound was prepared according to the general procedure described above by the reaction between styrene (**1a**) with TBHP and 2-methylpentanal (**2e**) and DBU, and purified by flash column chromatography as colorless oil (28.1 mg, 74%).
^{1}H NMR (400 MHz, CDCl$_3$) δ 8.00 – 7.91 (m, 2H), 7.59 – 7.52 (m, 1H), 7.51 – 7.40 (m, 2H), 2.95 (dd, J = 16.0, 5.6 Hz, 1H), 2.75 (dd, J = 16.0, 8.0 Hz, 1H), 2.28 – 2.09 (m, 1H), 1.42 – 1.21 (m, 4H), 0.95 (d, J = 6.8 Hz, 3H), 0.90 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 200.69, 137.60, 132.97, 128.68, 128.24, 46.15, 39.57, 29.71, 20.29, 20.13, 14.37. IR (cm^{-1}): 2957, 2928, 2872, 1685, 1448, 751, 690.

(5f) 3-ethyl-1-phenylheptan-1-one



The title compound was prepared according to the general procedure described above by the reaction between styrene (**1a**) with TBHP and 2-ethylhexanal (**2f**) and DBU, and purified by flash column chromatography as colorless oil (31.4 mg, 72%).
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.98 – 7.93 (m, 2H), 7.60 – 7.51 (m, 1H), 7.51 – 7.43 (m, 2H), 2.92 – 2.81 (m, 2H), 2.15 – 1.97 (m, 1H), 1.51 – 1.19 (m, 8H), 0.92 – 0.85 (m, 6H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 201.00, 137.67, 132.94, 128.68, 128.23, 43.23, 35.84, 33.42, 29.04, 26.63, 23.11, 14.23, 11.05. IR (cm^{-1}): 2958, 2928, 2872, 1684, 1448, 749, 690.

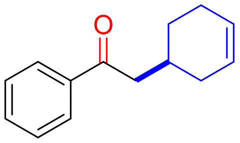
(5h) 2-cyclopentyl-1-phenylethanone



The title compound was prepared according to the general procedure described above by the reaction between styrene (**1a**) with TBHP and cyclopentanecarbaldehyde (**2h**) and DBU, and purified by flash column chromatography as colorless oil (29.7 mg, 79%).
^{1}H NMR (400 MHz, CDCl$_3$) δ 8.00 – 7.92 (m, 2H), 7.59 – 7.51 (m, 1H), 7.50 – 7.41 (m, 2H), 2.99 (d, J = 7.2 Hz, 2H), 2.48 – 2.30 (m, 1H), 1.98 – 1.79 (m, 2H), 1.72 – 1.49 (m, 4H), 1.32 – 1.08 (m,

2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.60, 137.40, 132.97, 128.67, 128.25, 44.97, 36.22, 32.87, 25.13. IR (cm^{-1}): 2950, 2867, 1685, 1448, 750, 690.

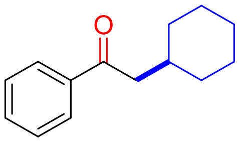
(5i) 2-(cyclohex-3-en-1-yl)-1-phenylethanone



The title compound was prepared according to the general procedure described above by the reaction between styrene (**1a**) with TBHP and 1-cyclohexene-3-carboxaldehyde (**2i**) and DBU, and purified by flash column chromatography as colorless oil (22.8 mg, 57%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 – 7.92 (m, 2H), 7.61 – 7.53 (m, 1H), 7.50 – 7.43 (m, 2H), 5.75 – 5.59 (m, 2H), 2.93 (d, J = 7.2 Hz, 2H), 2.37 – 2.25 (m, 1H), 2.24 – 2.13 (m, 1H), 2.12 – 2.02 (m, 2H), 1.86 – 1.73 (m, 2H), 1.42 – 1.30 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.15, 137.51, 133.06, 128.70, 128.26, 127.06, 126.10, 45.20, 31.73, 30.29, 28.88, 24.90. IR (cm^{-1}): 3022, 2914, 2836, 1686, 1448, 749, 689.

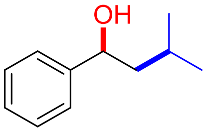
(5k) 2-cyclohexyl-1-phenylethanone



The title compound was prepared according to the general procedure described above by the reaction between styrene (**1a**) with TBHP and cyclohexanecarboxaldehyde (**2k**) and DBU, and purified by flash column chromatography as colorless oil (27.1mg, 67%) .

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 – 7.88 (m, 2H), 7.59 – 7.52 (m, 1H), 7.51 – 7.43 (m, 2H), 2.83 (d, J = 6.8 Hz, 2H), 2.07 – 1.87 (m, 1H), 1.80 – 1.62 (m, 5H), 1.38 – 0.90 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.48, 137.60, 132.98, 128.67, 128.28, 46.37, 34.69, 33.58, 26.39, 26.29. IR (cm^{-1}): 2922, 2851, 1685, 1448, 1286, 1193, 690.

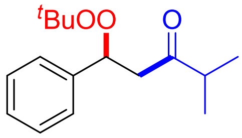
(6) 3-methyl-1-phenylbutan-1-ol



In a 2 necked-flask, Pd/C (10% dry on carbon, 20 mg) was introduced and the atmosphere was replaced with argon, **3a** (118 mg, 0.5 mmol) and acetonitrile (5 mL) was added. The atmosphere was then replaced with hydrogen and the reaction was reacted at room temperature under vigorous stirring for 1.5 hours. The mixture was filtered on Celite (washed by dichloromethane), and the resulting concentrated solution was purified by column chromatography using a mixture of hexanes and ethyl acetate to afford **6** (70 mg, 85% yield) as a clear oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.24 (m, 5H), 4.74 (dd, J = 8.4, 6.0 Hz, 1H), 1.90 – 1.60 (m, 4H), 1.55 – 1.46 (m, 1H), 0.95 (dd, J = 6.4, 2.0 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.34, 128.57, 127.59, 125.99, 72.87, 48.45, 24.90, 23.23, 22.38. IR (cm^{-1}): 3373, 2955, 2925, 2868, 1467, 1056, 754, 699.

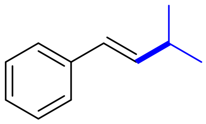
(7a) 1-(tert-butylperoxy)-4-methyl-1-phenylpentan-3-one



The title compound was prepared according to the general procedure described above by the reaction between styrene (**1a**) with TBHP and isobutyraldehyde (**2a**), and purified by flash column chromatography as colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.50 – 7.27 (m, 5H), 5.43 (dd, J = 7.6, 5.2 Hz, 1H), 3.16 (dd, J = 16.0, 7.6 Hz, 1H), 2.70 (dd, J = 16.0, 5.6 Hz, 1H), 2.66 – 2.53 (m, 1H), 1.20 (s, 9H), 1.08 (d, J = 6.8 Hz, 3H), 1.02 (d, J = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 211.66, 140.27, 128.50, 128.16, 127.09, 82.17, 80.87, 46.00, 41.78, 26.52, 17.88. IR (cm^{-1}): 2974, 1714, 1466, 1363, 1196, 913, 747, 698. HRMS: calcd. for $C_{16}H_{24}O_3Na^+$ [M+Na]$^+$: 287.1618; Found: 287.1624.

(8a) (3-methylbut-1-en-1-yl)benzene [2]



The title compound was prepared according to the general procedure described above by the reaction between styrene (**1a**) with TBHP and isobutyraldehyde (**2a**), and purified by flash column chromatography as colorless oil.

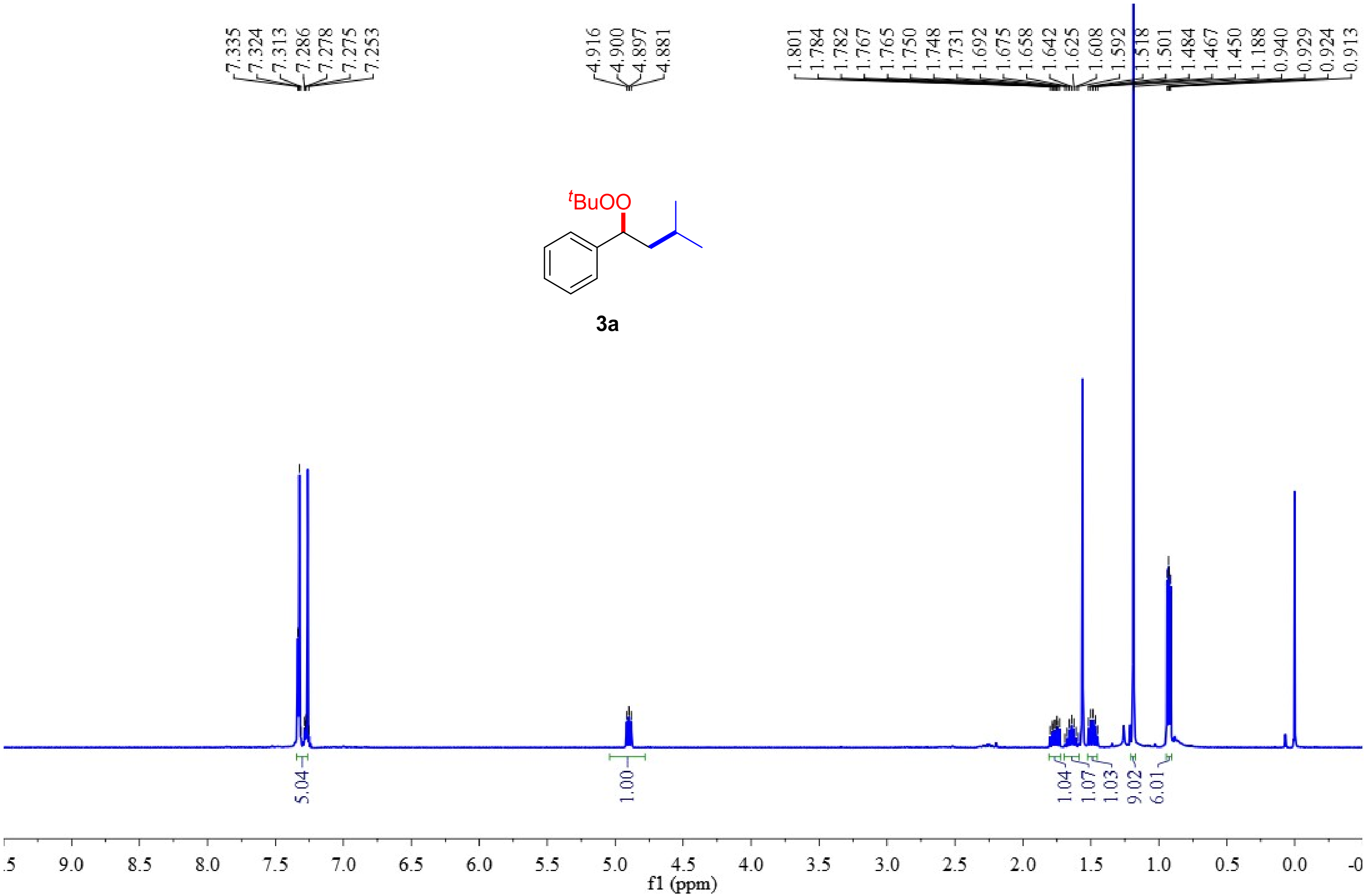
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.40 – 7.26 (m, 4H), 7.17 (t, J = 8.0 Hz, 1H), 6.34 (d, J = 16.0 Hz, 1H), 6.18 (dd, J =16.0, 8.0 Hz, 1H), 2.48 – 2.43 (m, 1H), 1.10 (d, J = 8.0 Hz, 6H). ^{13}C NMR (100 MHz, CDCl$_3$) δ 138.16, 138.09, 128.60, 126.95, 126.89, 126.10, 31.67, 22.59. IR (cm^{-1}): 2959, 913, 744.

V. References

[1] Weiping Liu, Yuanming Li, Kaisheng Liu, and Zhiping Li, *J. Am. Chem. Soc.* **2011,** *133,* 10756-10759.

[2] G. Cahiez, O. Gager, J. Buendia and C .Patinote, *Chem. Eur. J.* **2012,** *18,* 5860-5863.

VI. Copies of ^{1}H and ^{13}C NMR spectra of products 3a-3q, 4b-4k, 5a-5k, 6, 7a, 8a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1.784
1.782
1.767
1.765
1.750
1.748
1.731
1.692
1.675
1.658
1.642
1.625
1.608
1.592
1.518
1.501
1.484
1.467
1.450
1.188
0.940
0.929
0.924
0.913
^{t}BuOO
3a
5.04
1.00
1.04
1.07
1.03
9.02
6.01
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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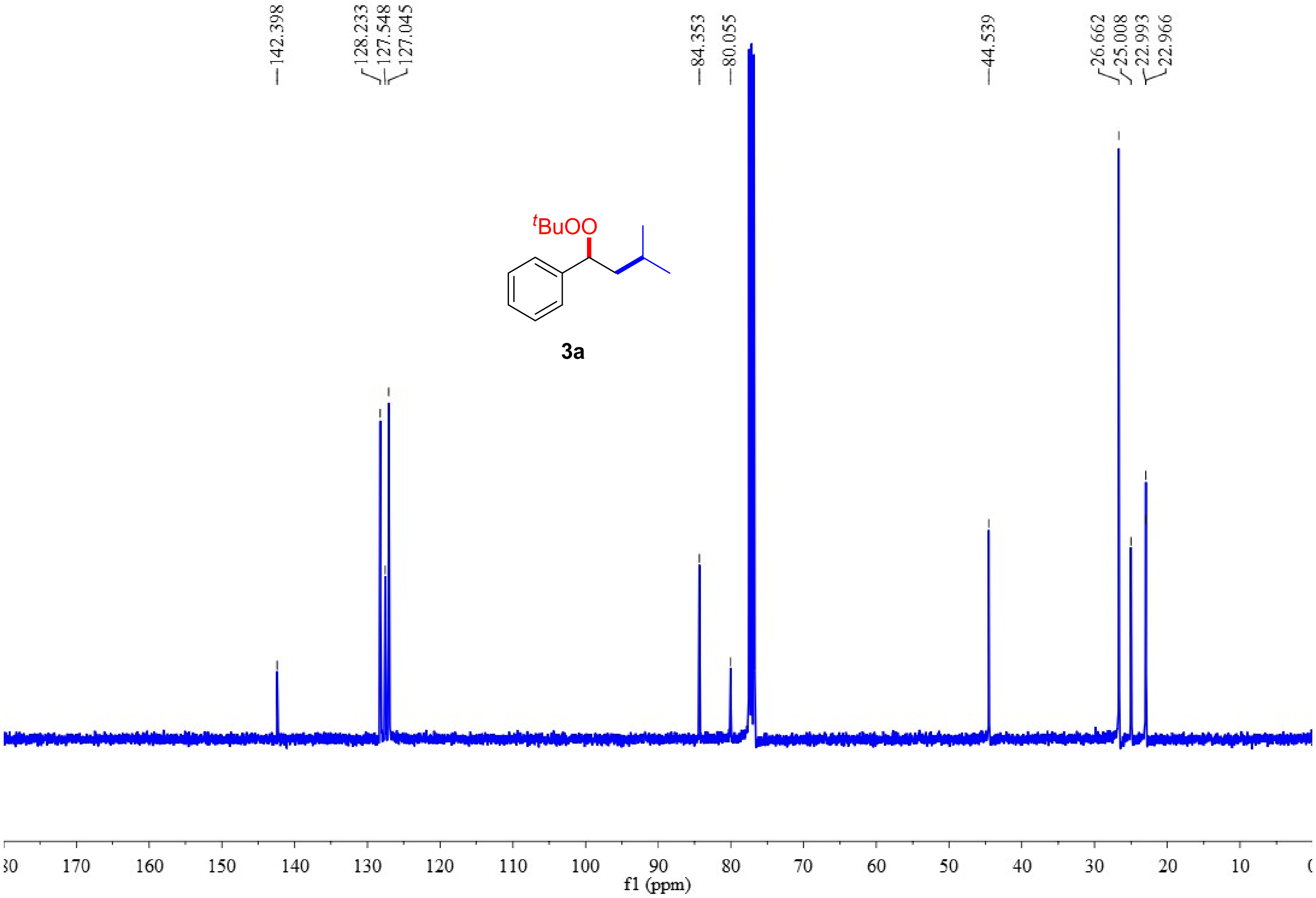
f1 (ppm)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26.662
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160
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140
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120
110
100
90
80
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60
50
40
30
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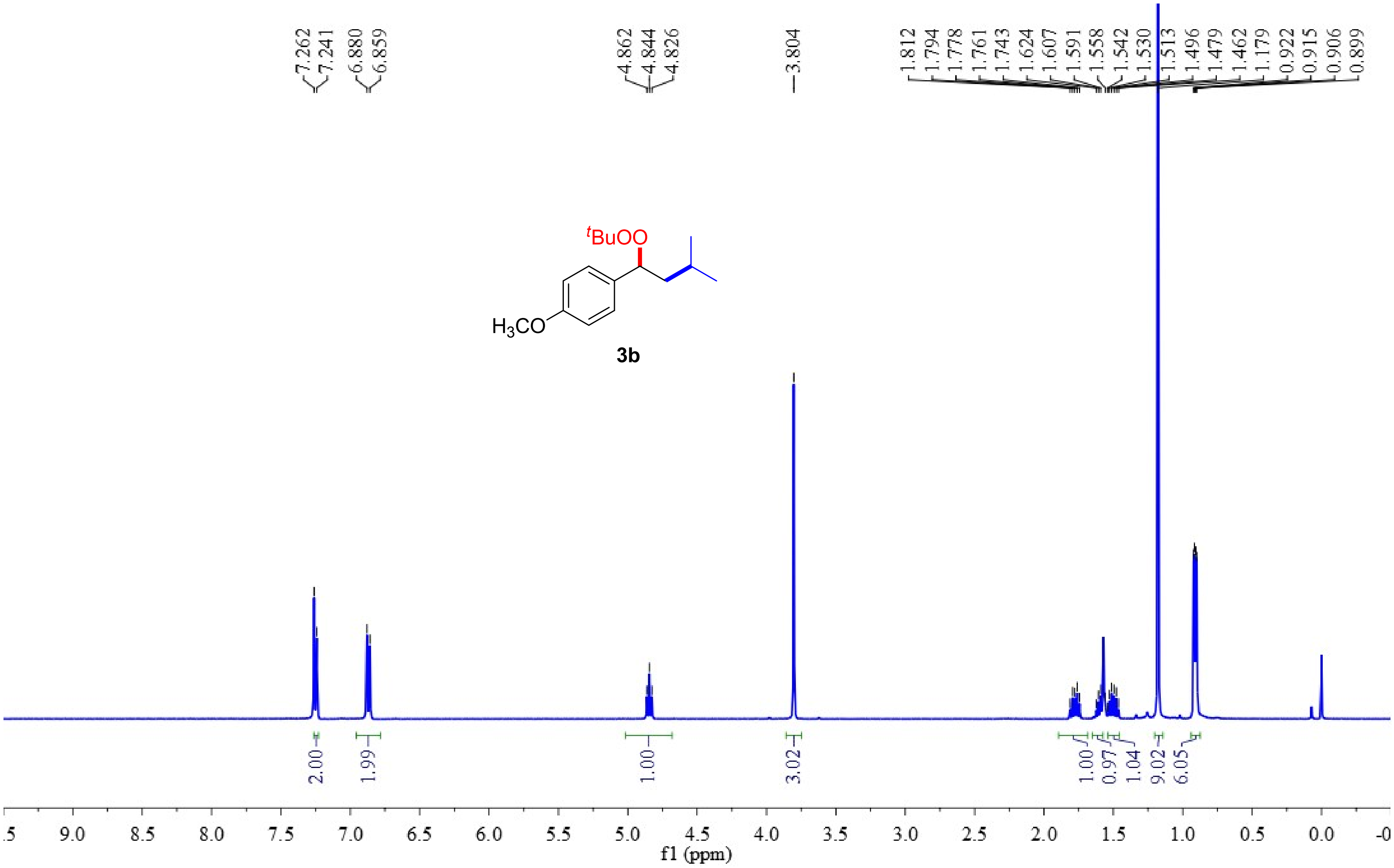
10
f1 (ppm)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1.761
1.743
1.624
1.607
1.591
1.558
1.542
1.530
1.513
1.496
1.479
1.462
1.179
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0.915
0.906
0.899
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H_3CO
3b
2.00
1.99
1.00
3.02
1.00
0.97
1.04
9.02
6.05
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9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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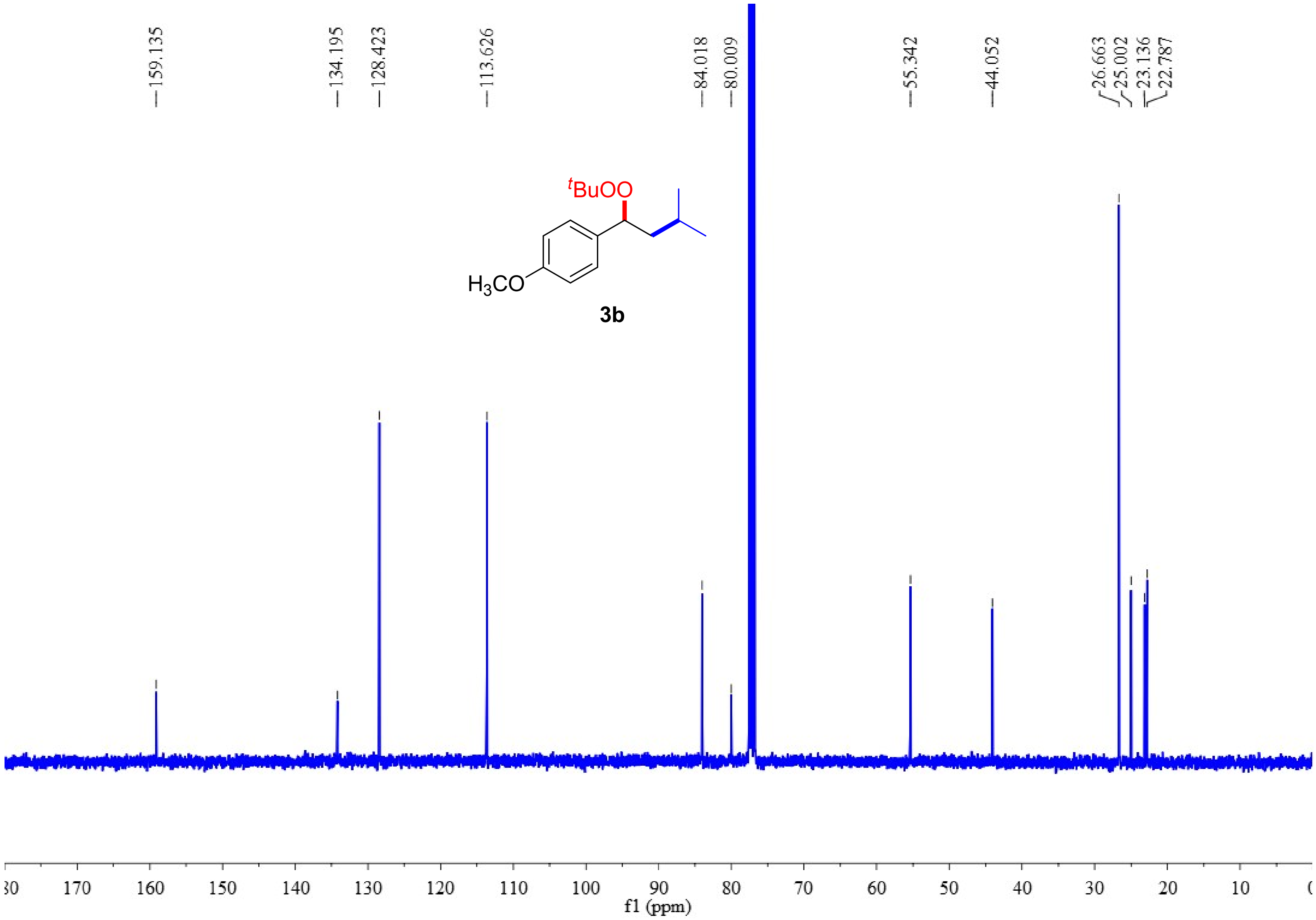
f1 (ppm)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84.018
80.009
55.342
44.052
26.663
25.002
23.136
22.787
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H$_3$CO
3b
30
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150
140
130
120
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90
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70
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50
40
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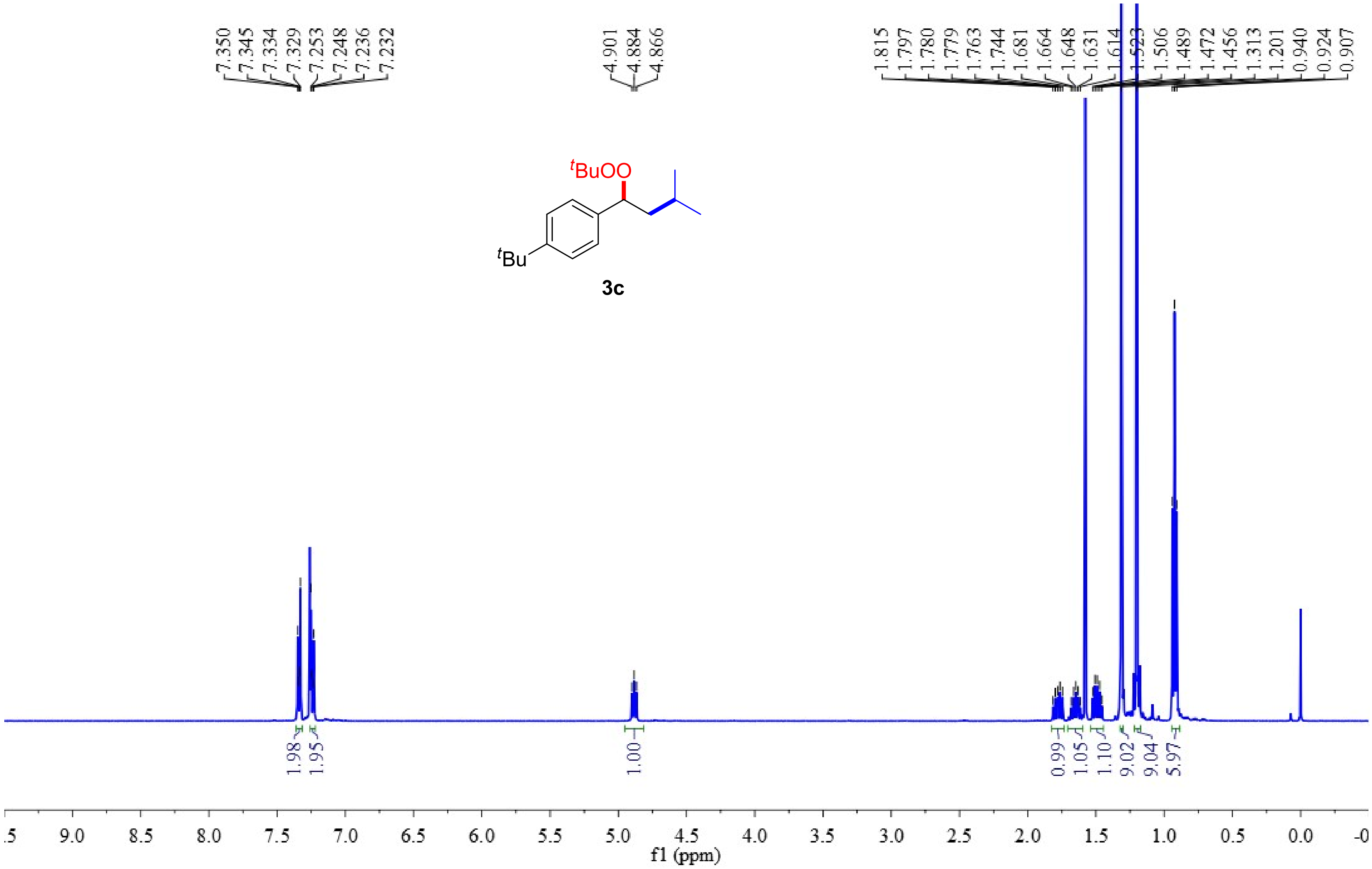
10
(
f1 (ppm)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1.780
1.779
1.763
1.744
1.681
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1.523
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1.313
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0.99
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1.10
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9.04
5.97
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7.0
6.5
6.0
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5.0
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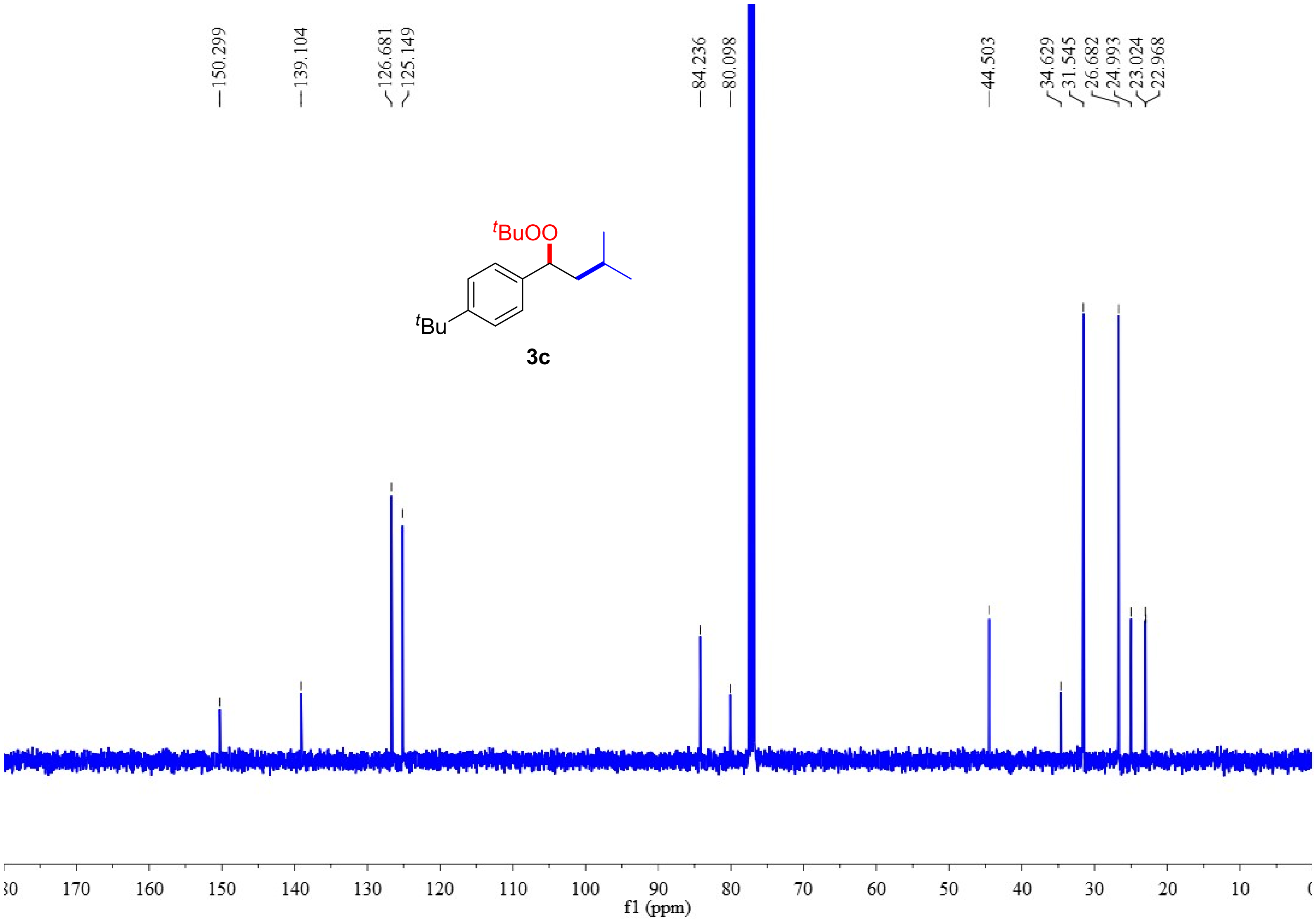
f1 (ppm)

3c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^{t}Bu
3c
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100
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60
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20
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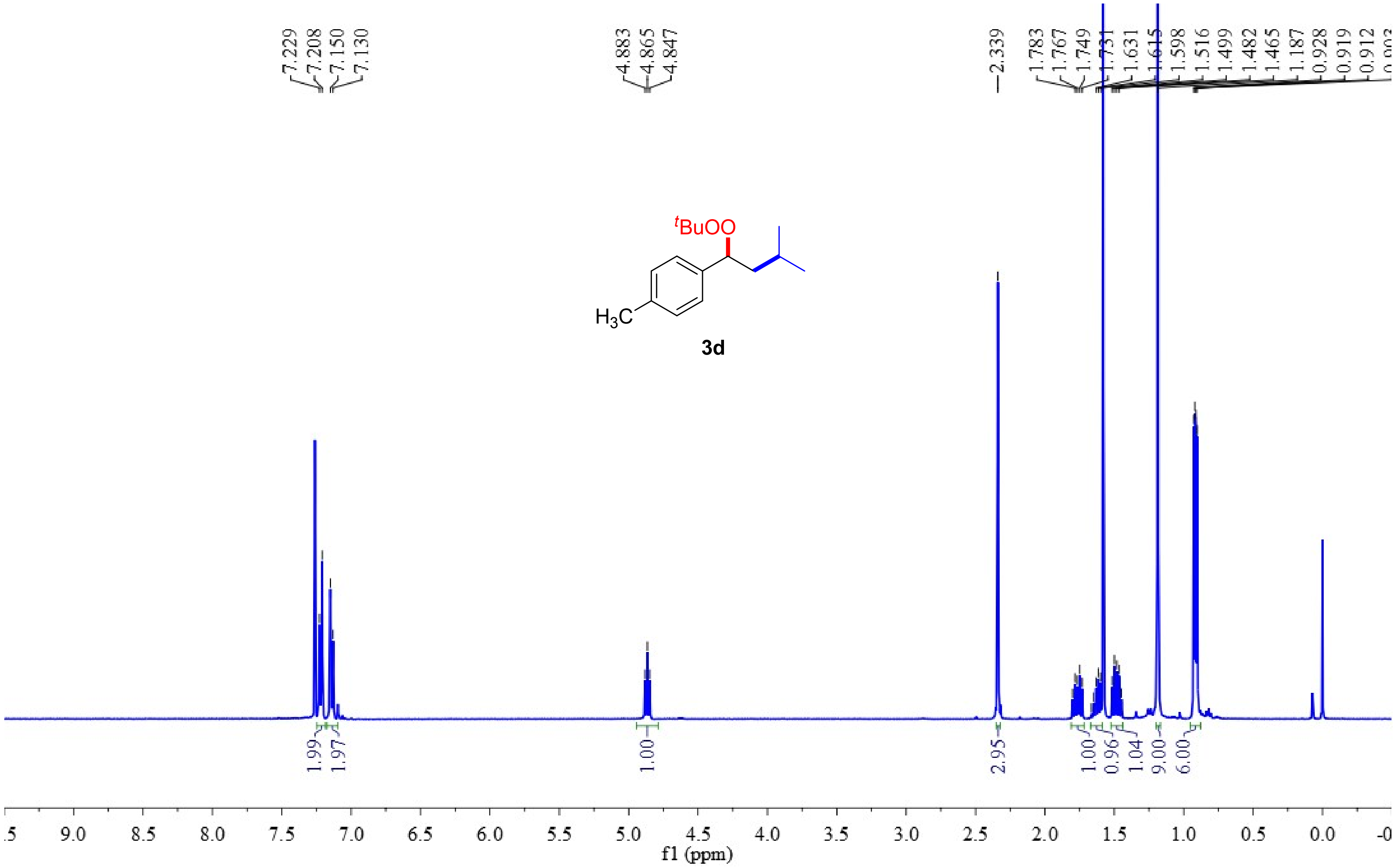
f1 (ppm)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1.631
1.615
1.598
1.516
1.499
1.482
1.465
1.187
0.928
0.919
0.912
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1.00
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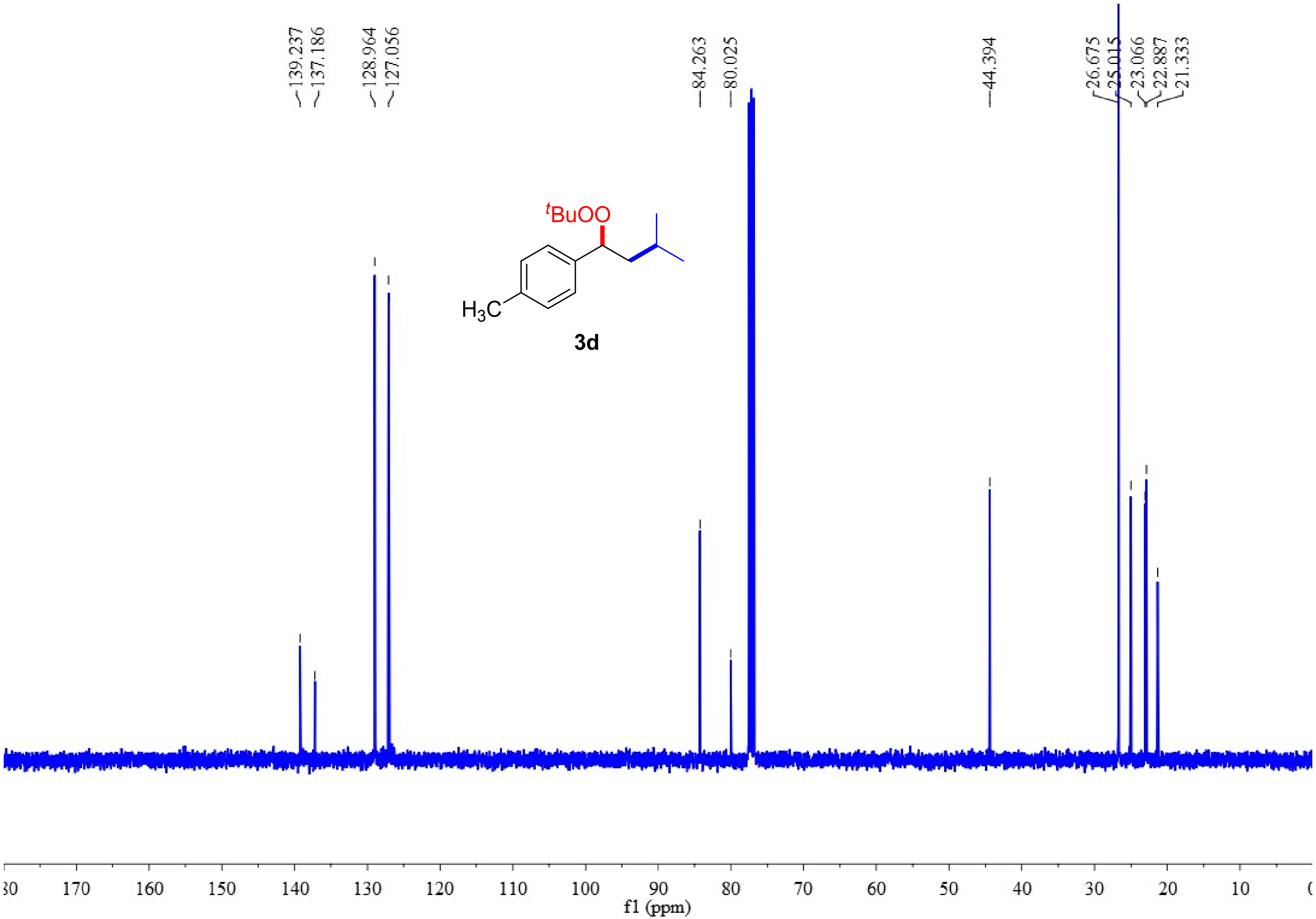
f1 (ppm)

3d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100
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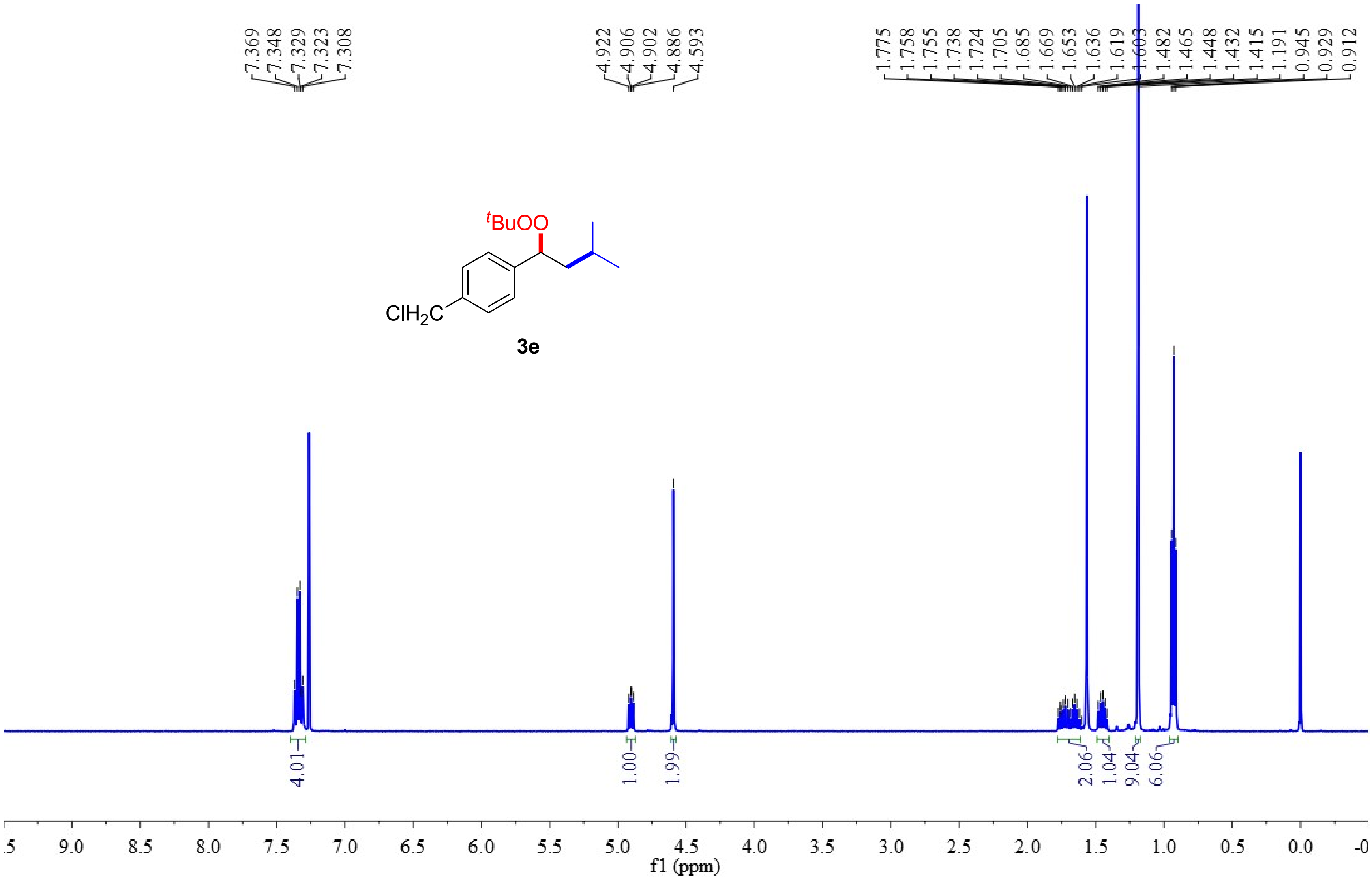
f1 (ppm)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6.06
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6.0
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5.0
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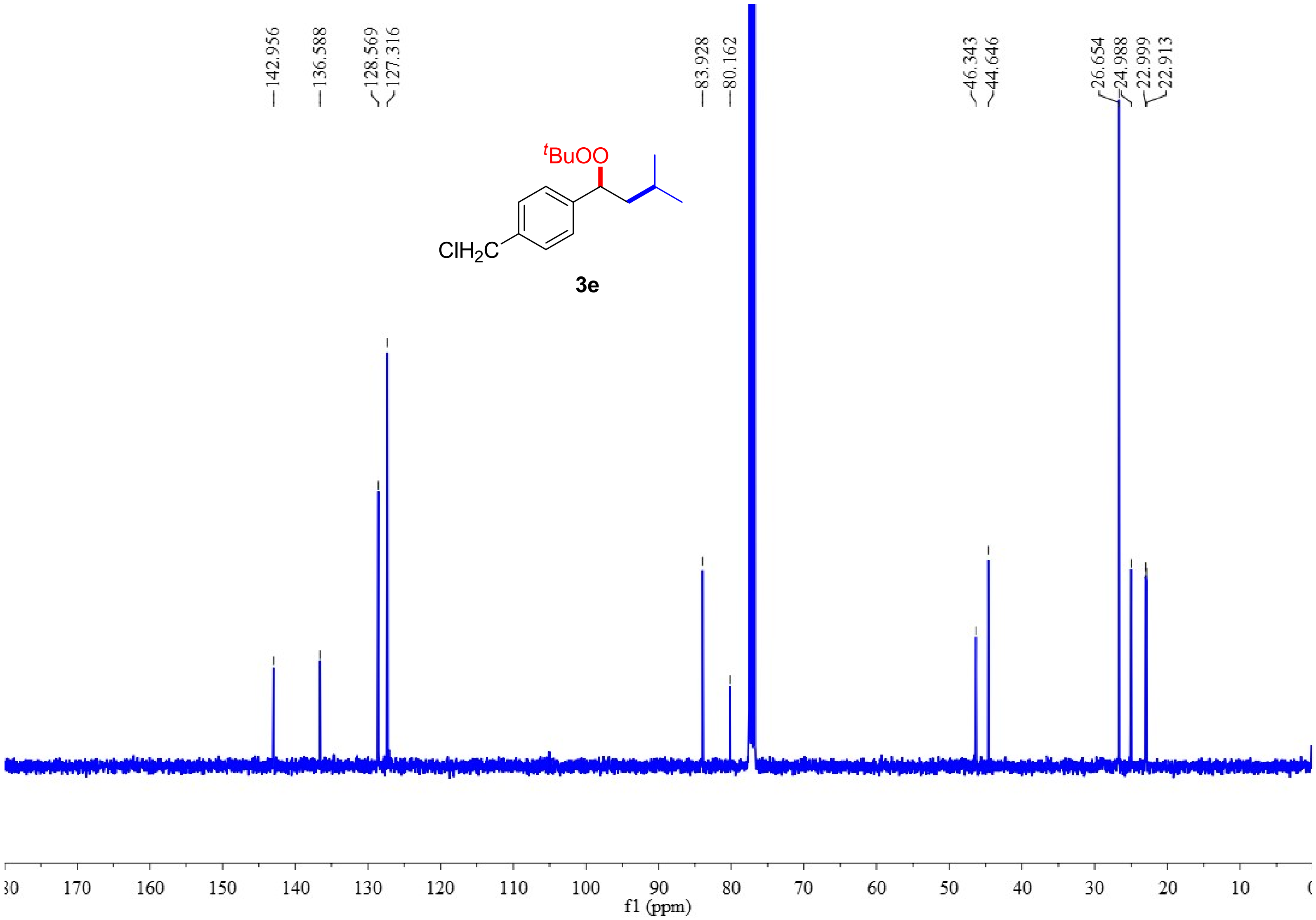
f1 (ppm)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30
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30
20
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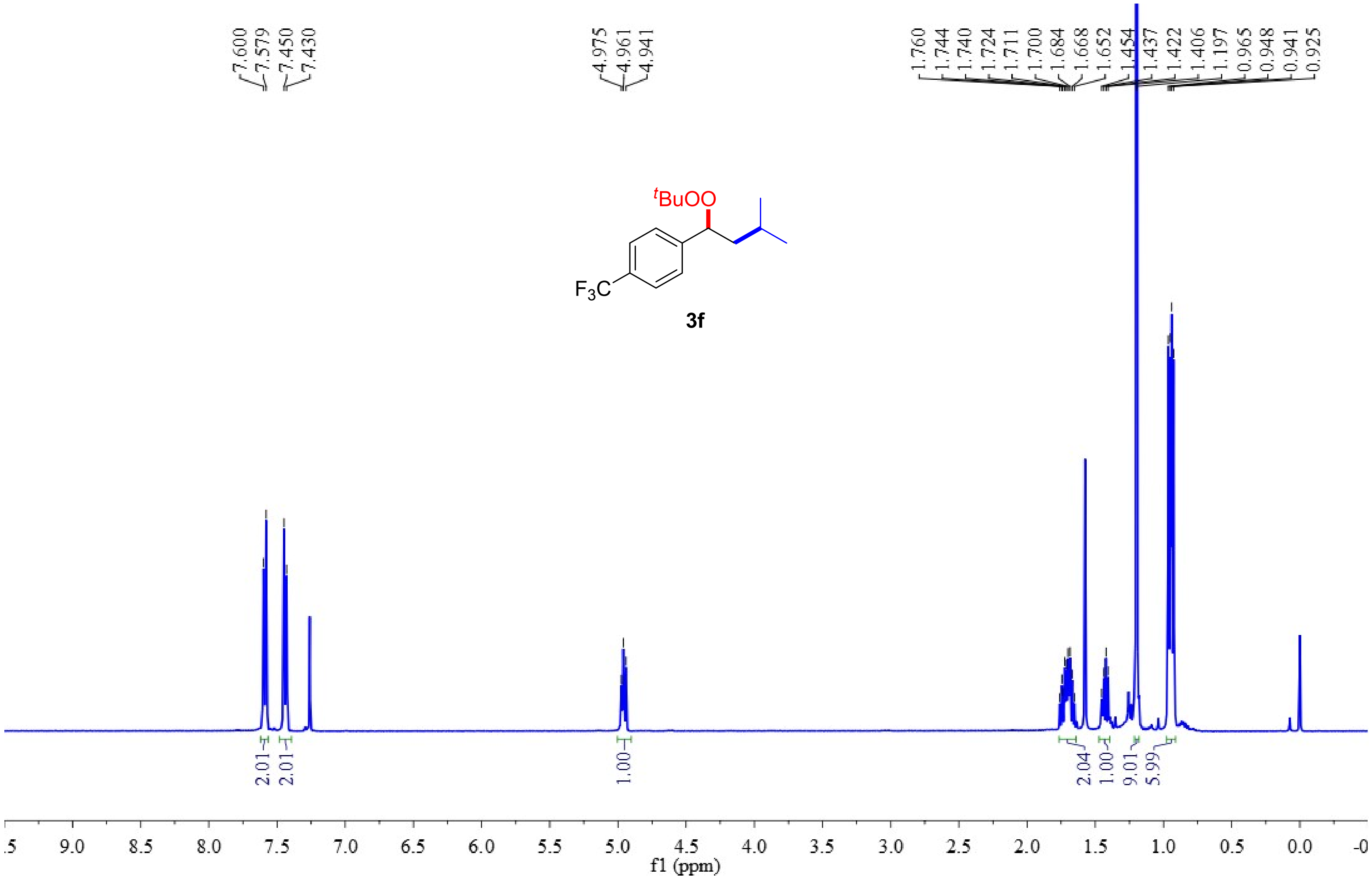
f1 (ppm)

3e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1.406
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F_3C
3f
2.01
2.01
1.00
2.04
1.00
9.01
5.99
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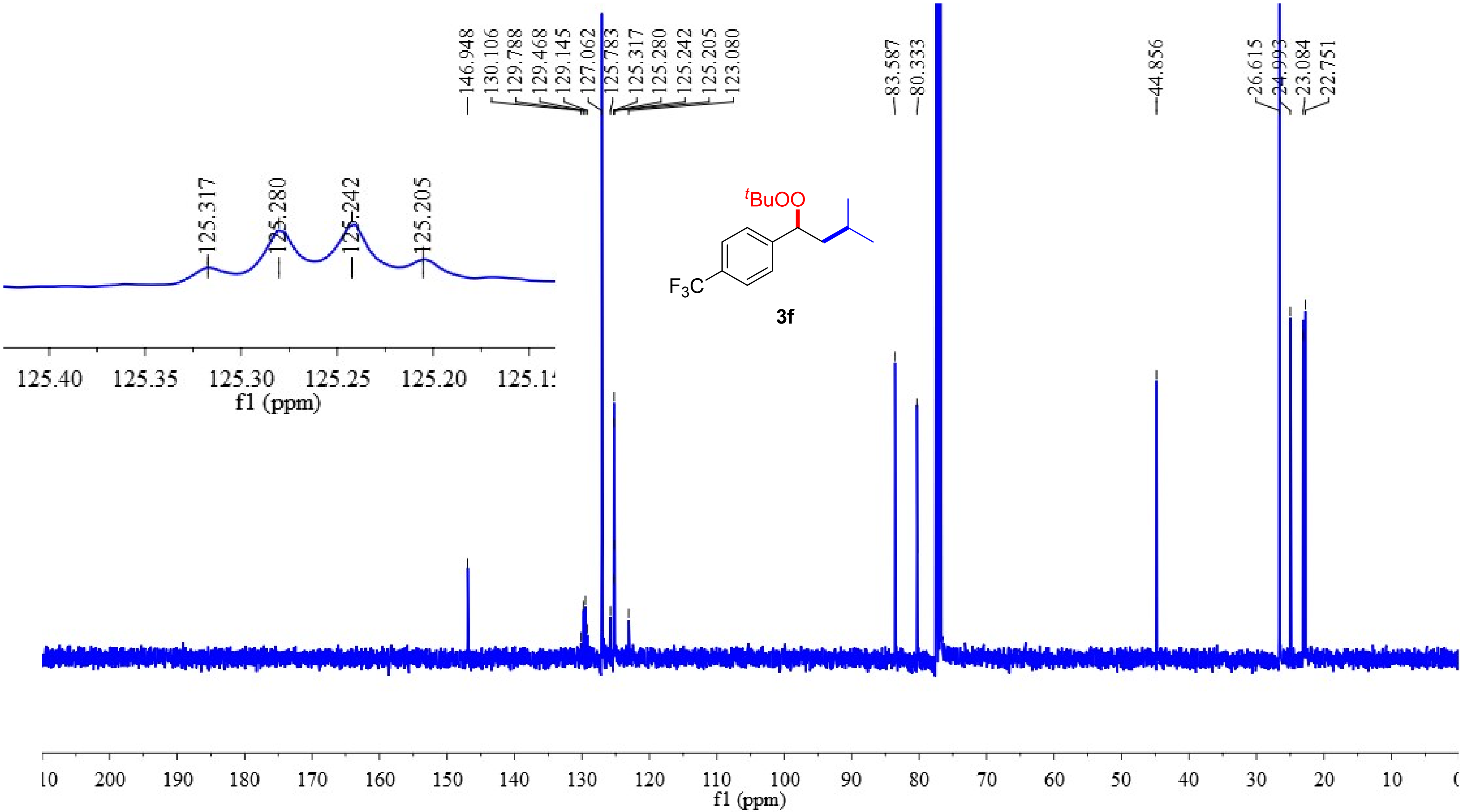
f1 (ppm)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125.280
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125.205
125.40
125.35
125.30
125.25
125.20
125.1
f1 (ppm)
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F_3C
3f
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200
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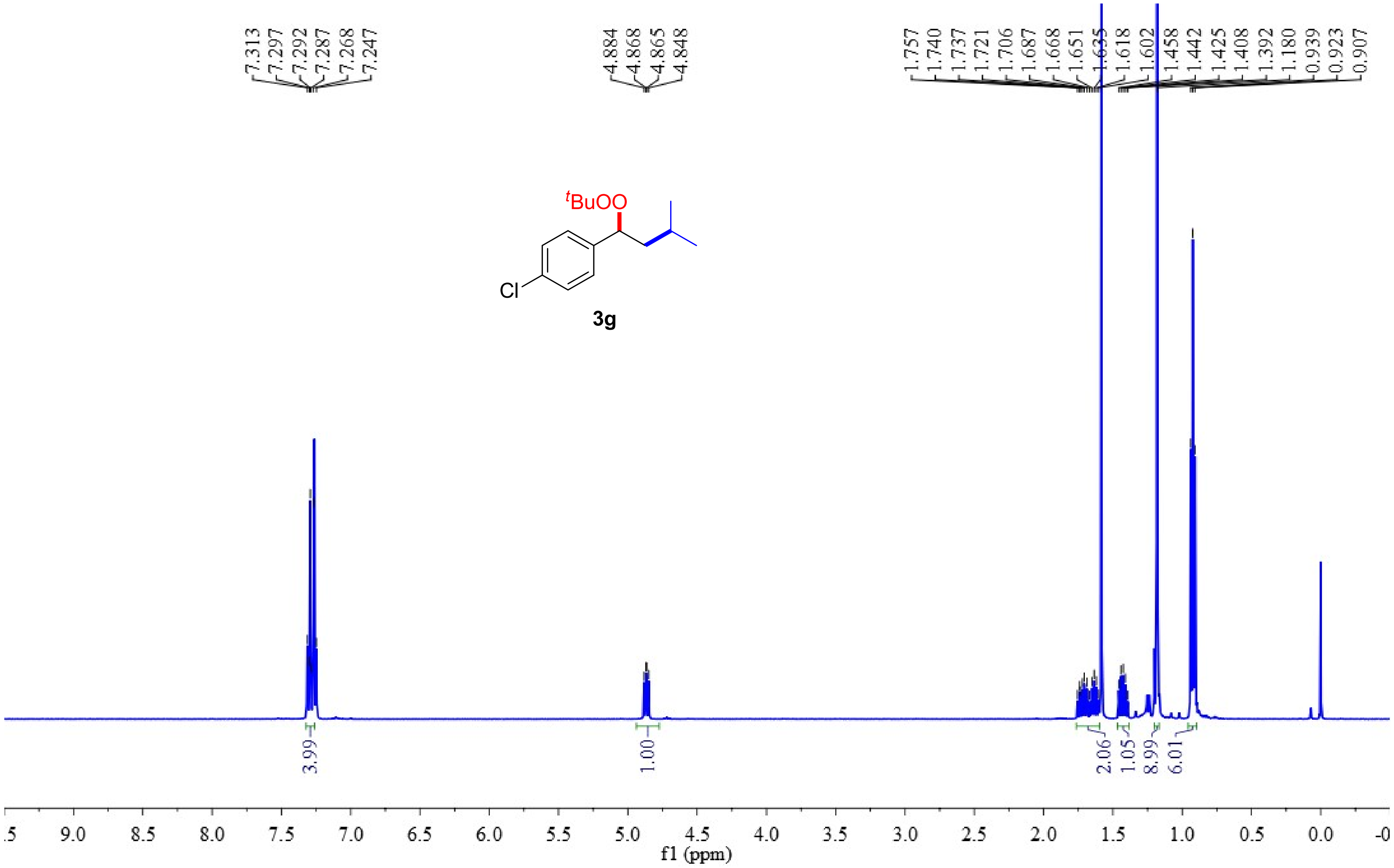
f1 (ppm)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0.907
^{t}BuOO
Cl
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1.00
2.06
1.05
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2.0
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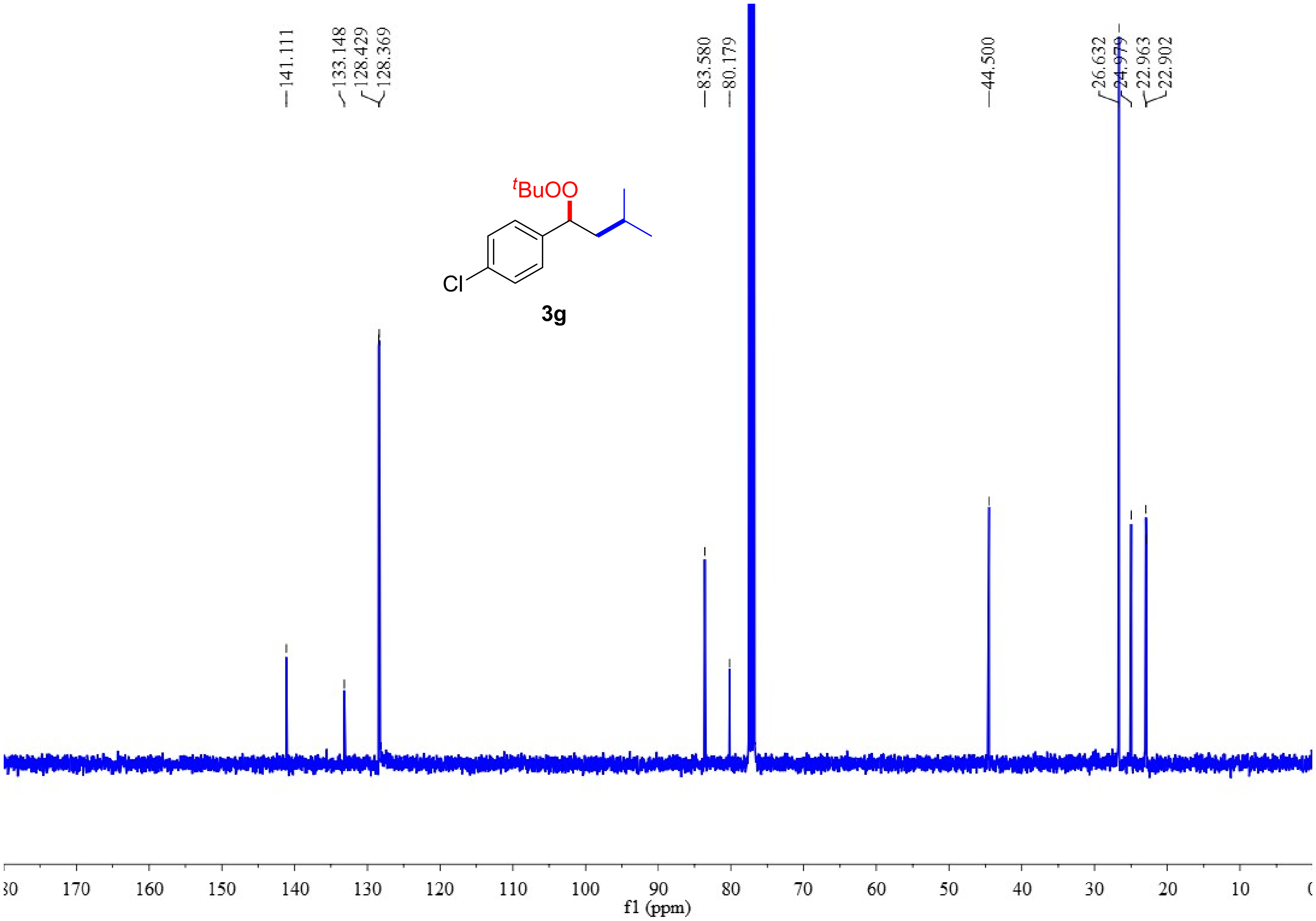
-0
f1 (ppm)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Cl
30
170
160
150
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120
110
100
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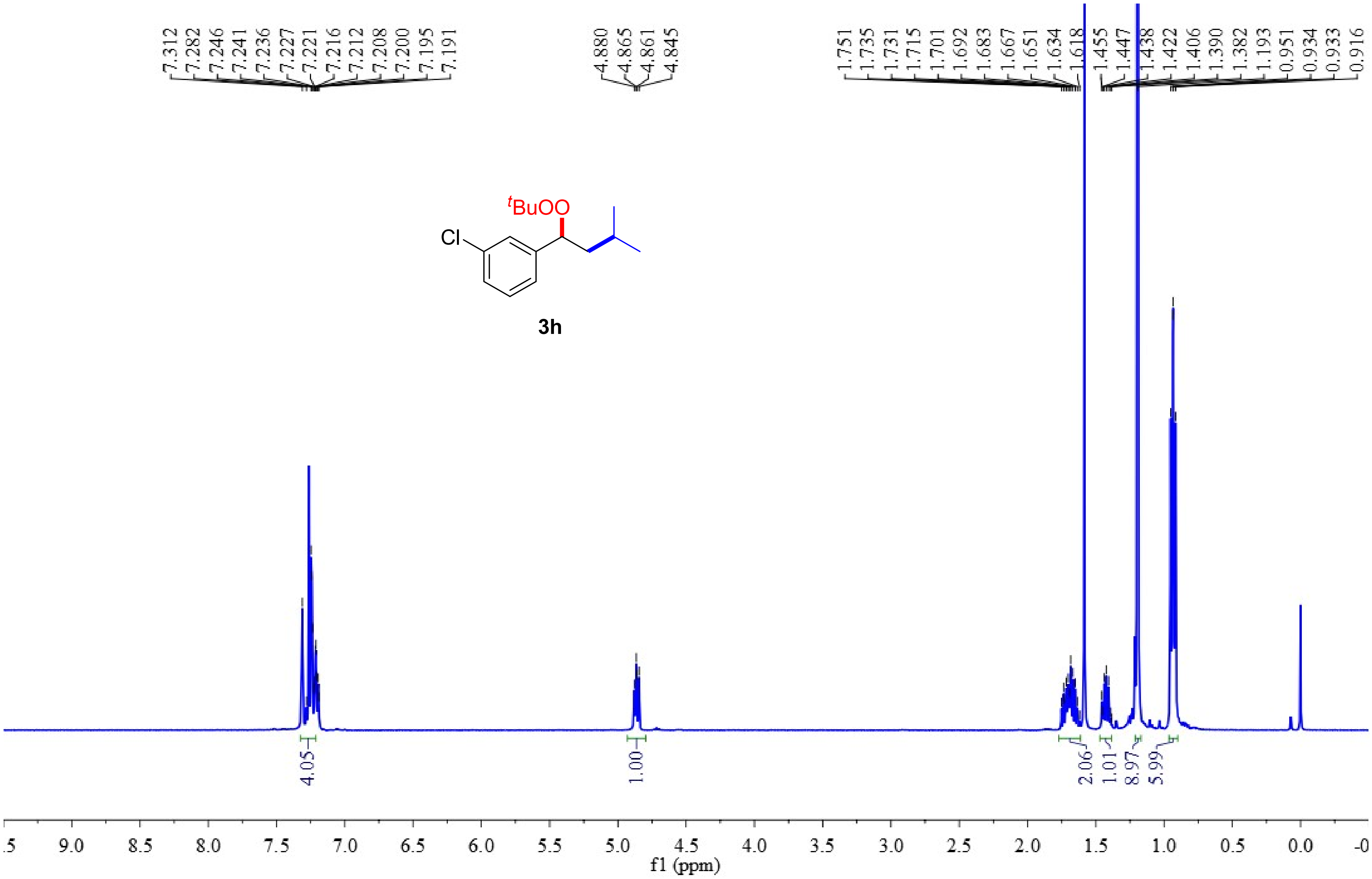
70
60
50
40
30
20
10
0
f1 (ppm)

3g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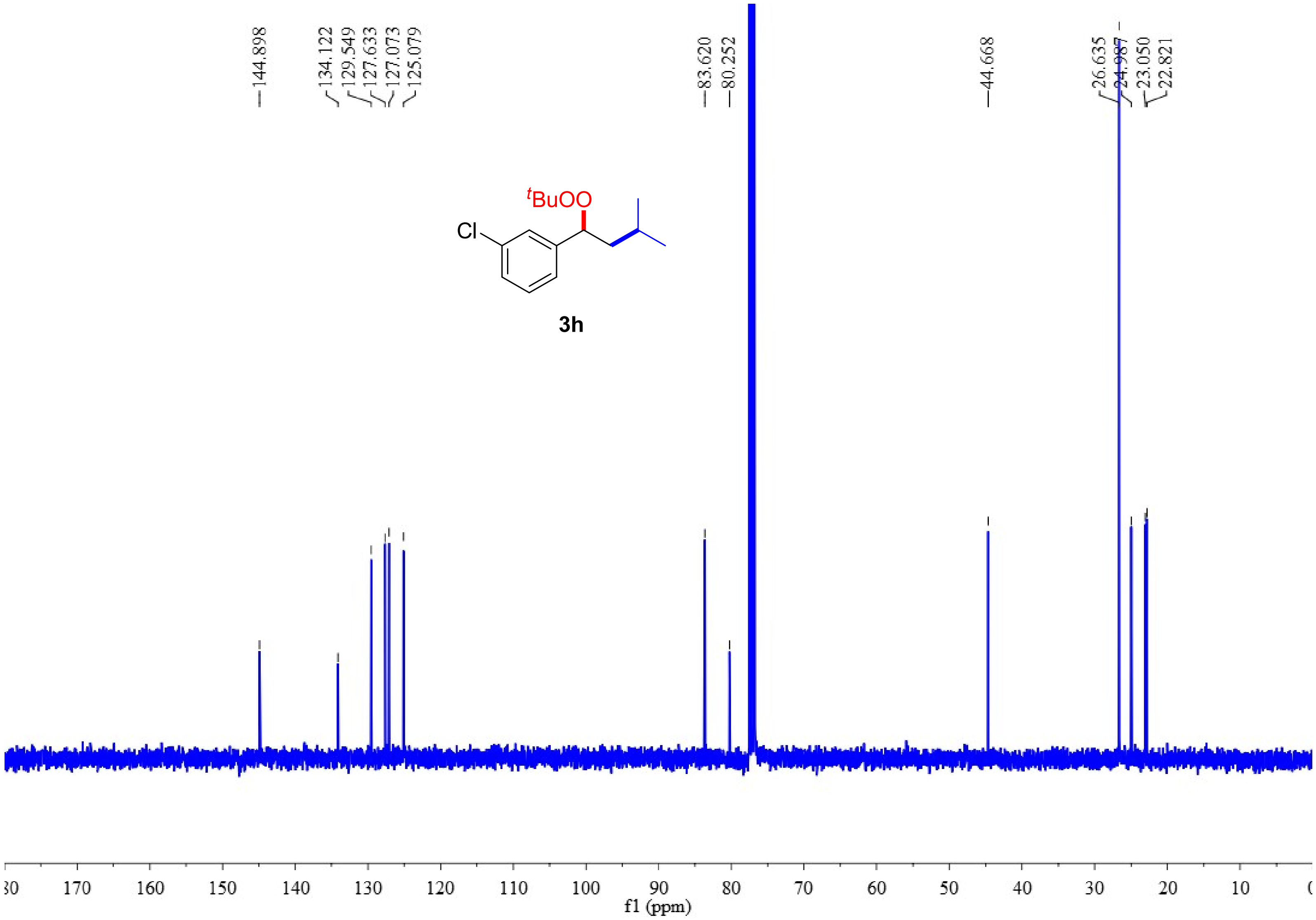
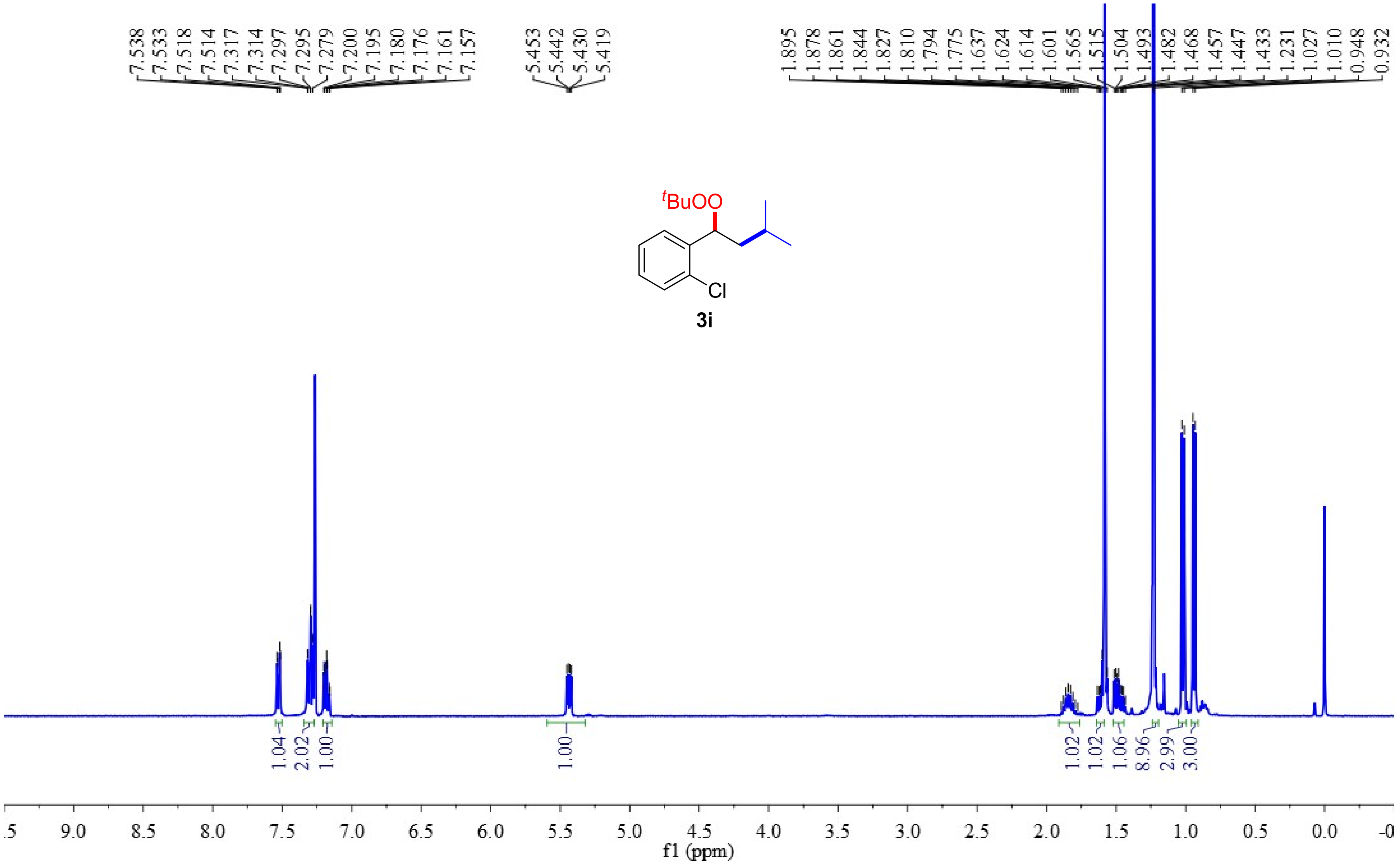
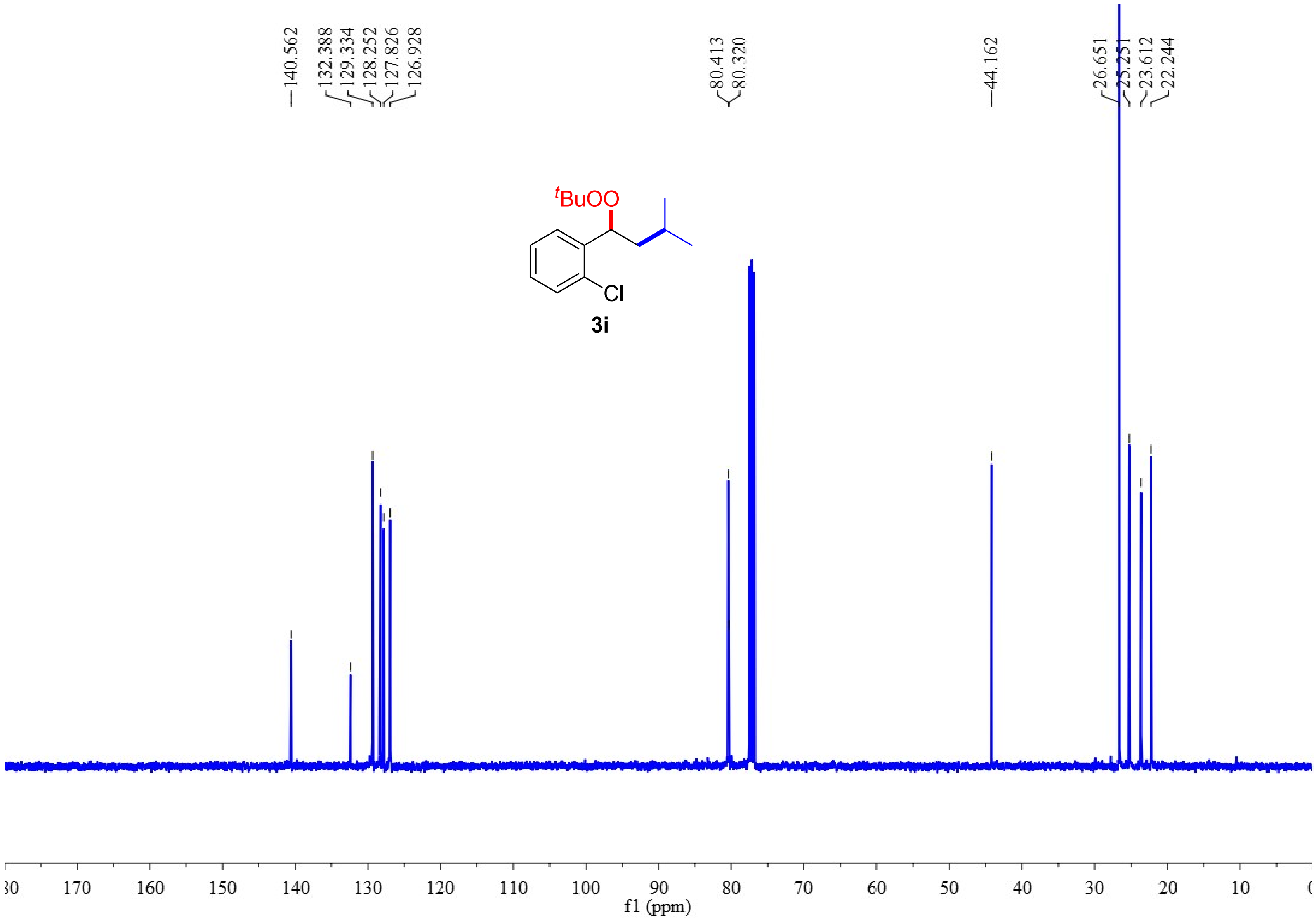
7.191
4.880
4.865
4.861
4.845
1.751
1.735
1.731
1.715
1.701
1.692
1.683
1.667
1.651
1.634
1.618
1.455
1.447
1.438
1.422
1.406
1.390
1.382
1.193
0.951
0.934
0.933
0.916
Cl
^{t}BuOO
3h
4.05
1.00
2.06
1.01
8.97
5.99
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

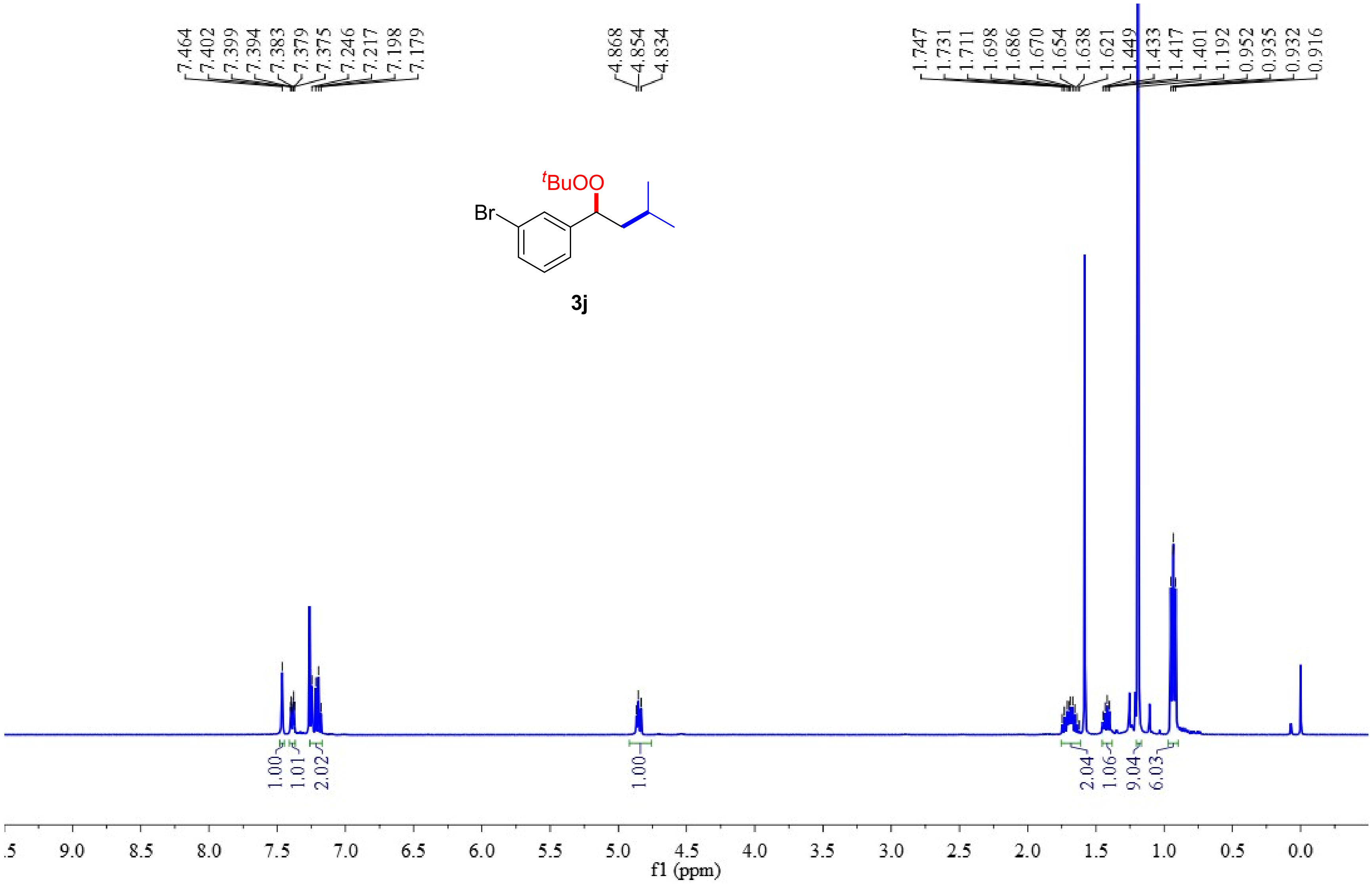

144.898
134.122
129.549
127.633
127.073
125.079
83.620
80.252
44.668
26.635
24.987
23.050
22.821
^{t}BuOO
Cl
f1 (ppm)
30
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

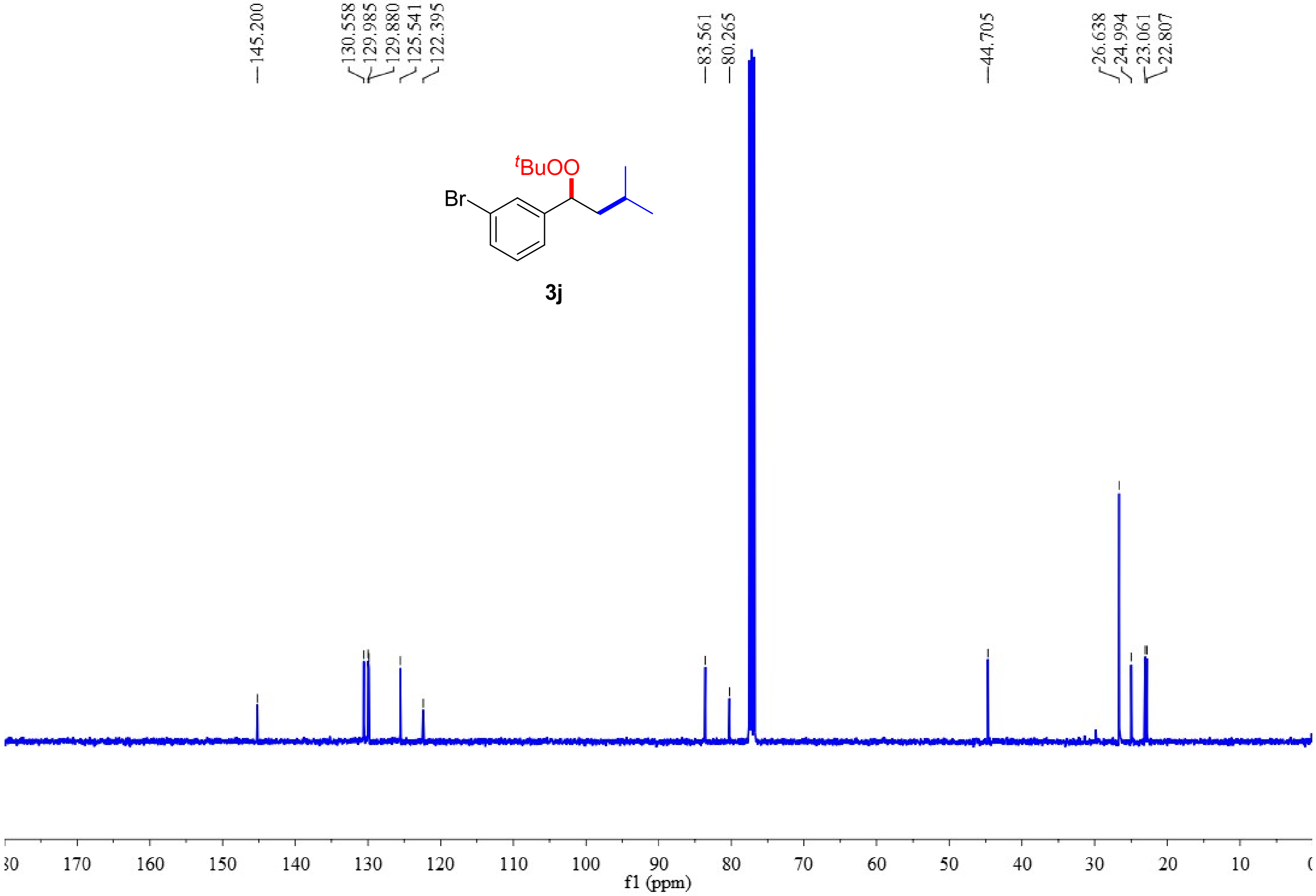
3h

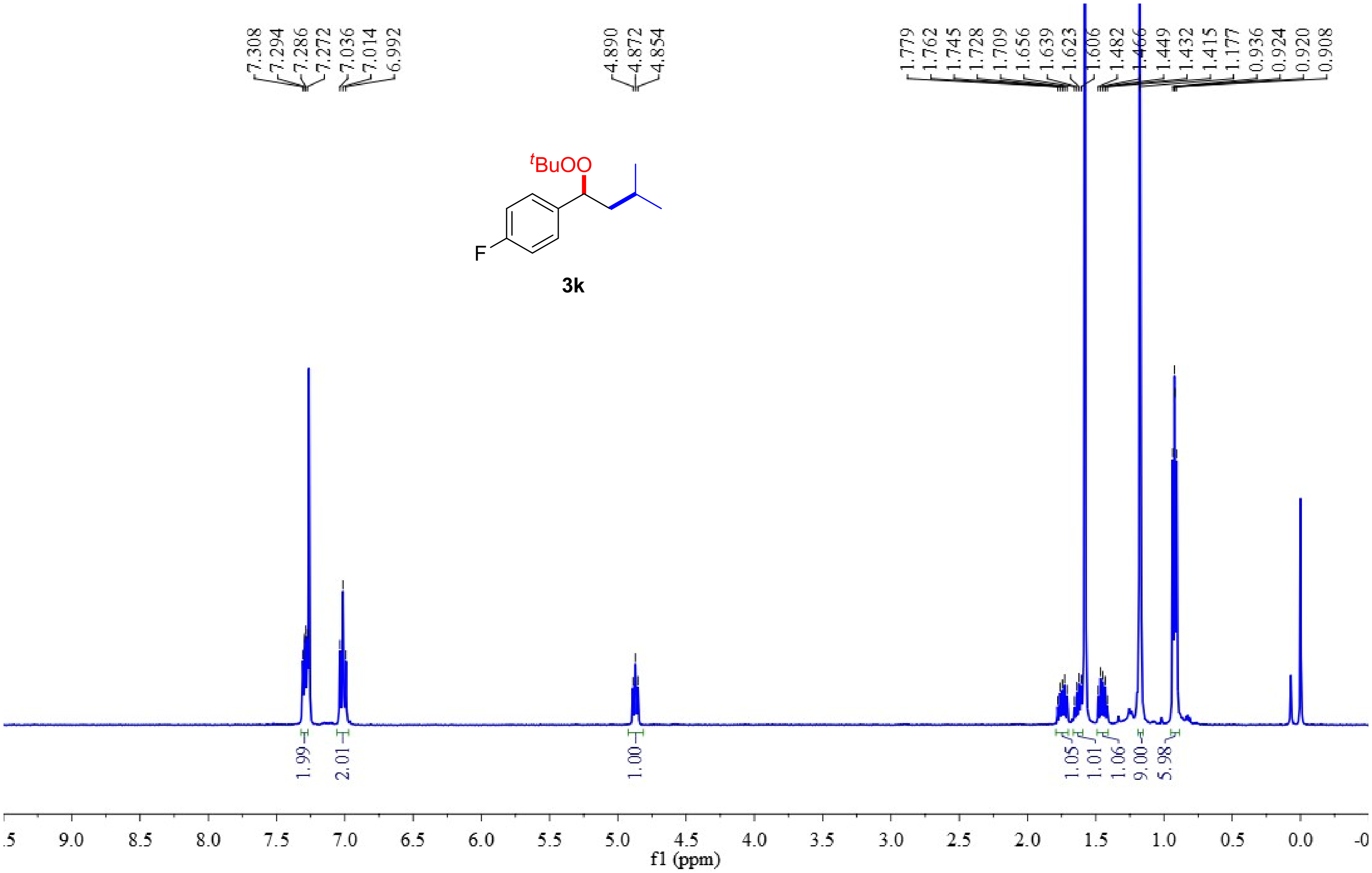

7.538
7.533
7.518
7.514
7.317
7.314
7.297
7.295
7.279
7.200
7.195
7.180
7.176
7.161
7.157
5.453
5.442
5.430
5.419
1.895
1.878
1.861
1.844
1.827
1.810
1.794
1.775
1.637
1.624
1.614
1.601
1.565
1.515
1.504
1.493
1.482
1.468
1.457
1.447
1.433
1.231
1.027
1.010
0.948
0.932
^{t}BuOO
Cl
3i
1.04
2.02
1.00
1.00
1.02
1.02
1.06
8.96
2.99
3.00
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

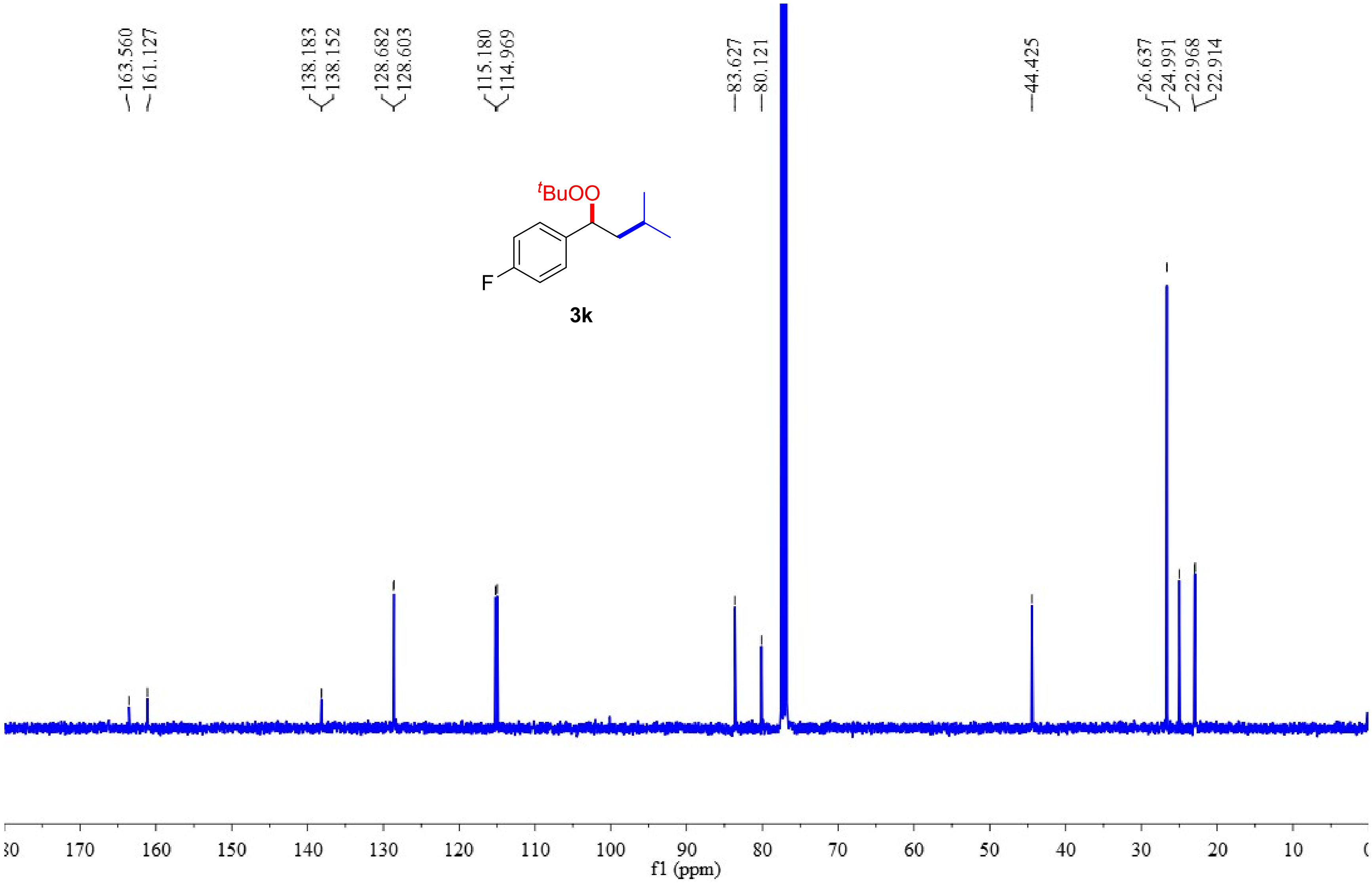

140.562
132.388
129.334
128.252
127.826
126.928
80.413
80.320
44.162
26.651
25.251
23.612
22.244
ᵗBuOO
Cl
30
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

3i

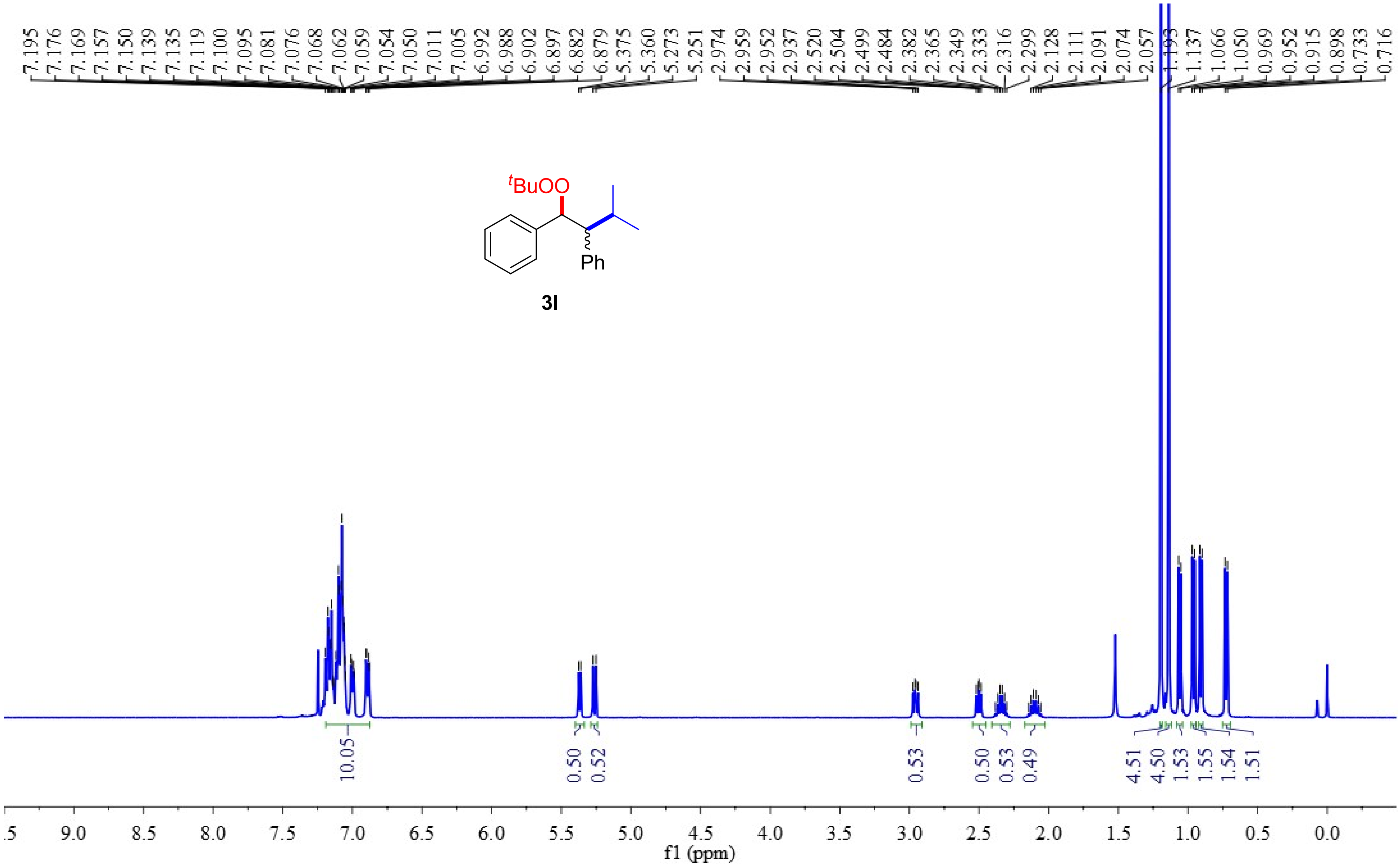

7.464
7.402
7.399
7.394
7.383
7.379
7.375
7.246
7.217
7.198
7.179
4.868
4.854
4.834
1.747
1.731
1.711
1.698
1.686
1.670
1.654
1.638
1.621
1.449
1.433
1.417
1.401
1.192
0.952
0.935
0.932
0.916
Br
^{t}BuOO
3j
1.00
1.01
2.02
1.00
2.04
1.06
9.04
6.03
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


145.200
130.558
129.985
129.880
125.541
122.395
83.561
80.265
44.705
26.638
24.994
23.061
22.807
^{t}BuOO
Br
3j
30
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

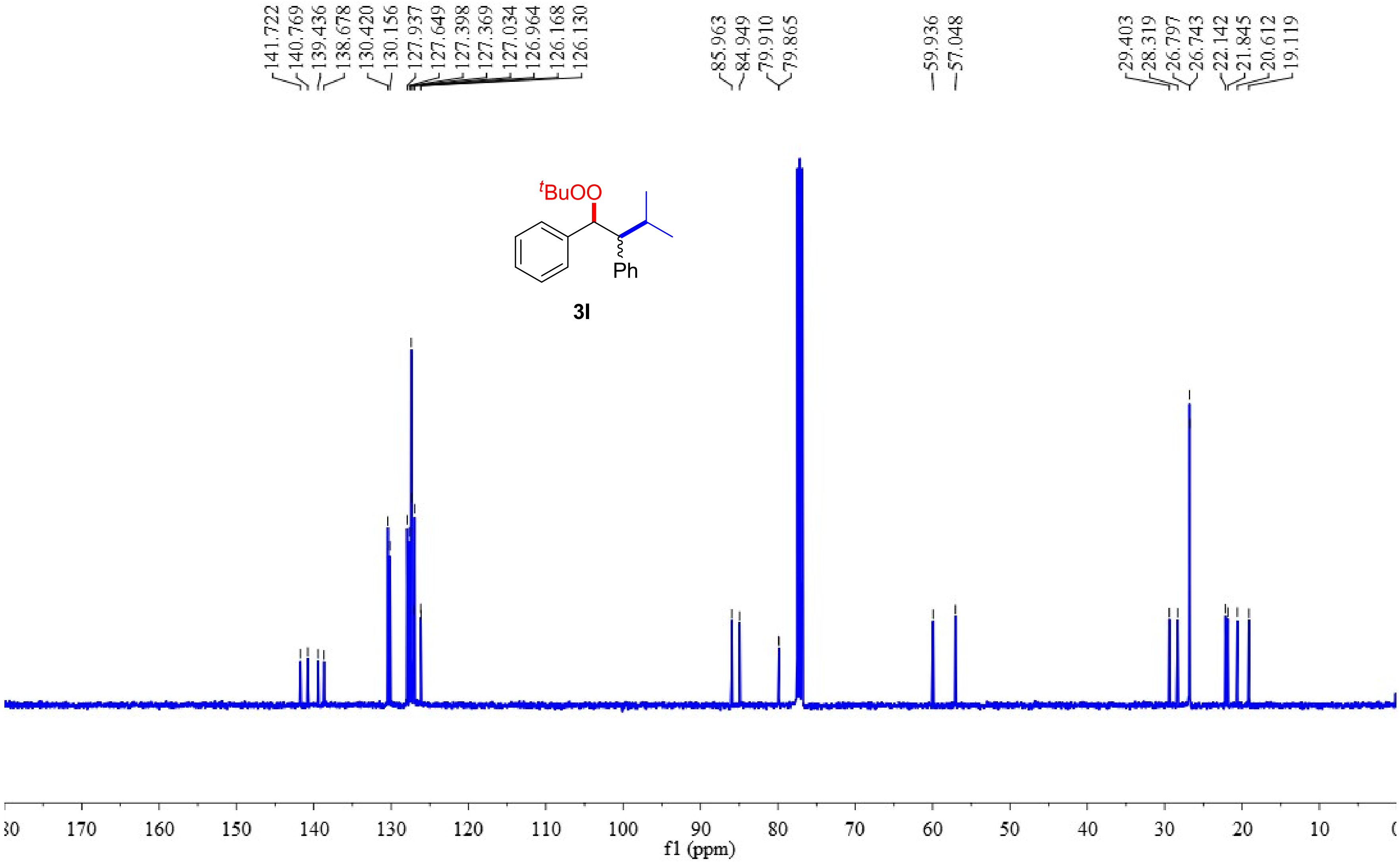

7.308
7.294
7.286
7.272
7.036
7.014
6.992
4.890
4.872
4.854
1.779
1.762
1.745
1.728
1.709
1.656
1.639
1.623
1.606
1.482
1.466
1.449
1.432
1.415
1.177
0.936
0.924
0.920
0.908
^{t}BuOO
F
3k
1.99
2.01
1.00
1.05
1.01
1.06
9.00
5.98
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

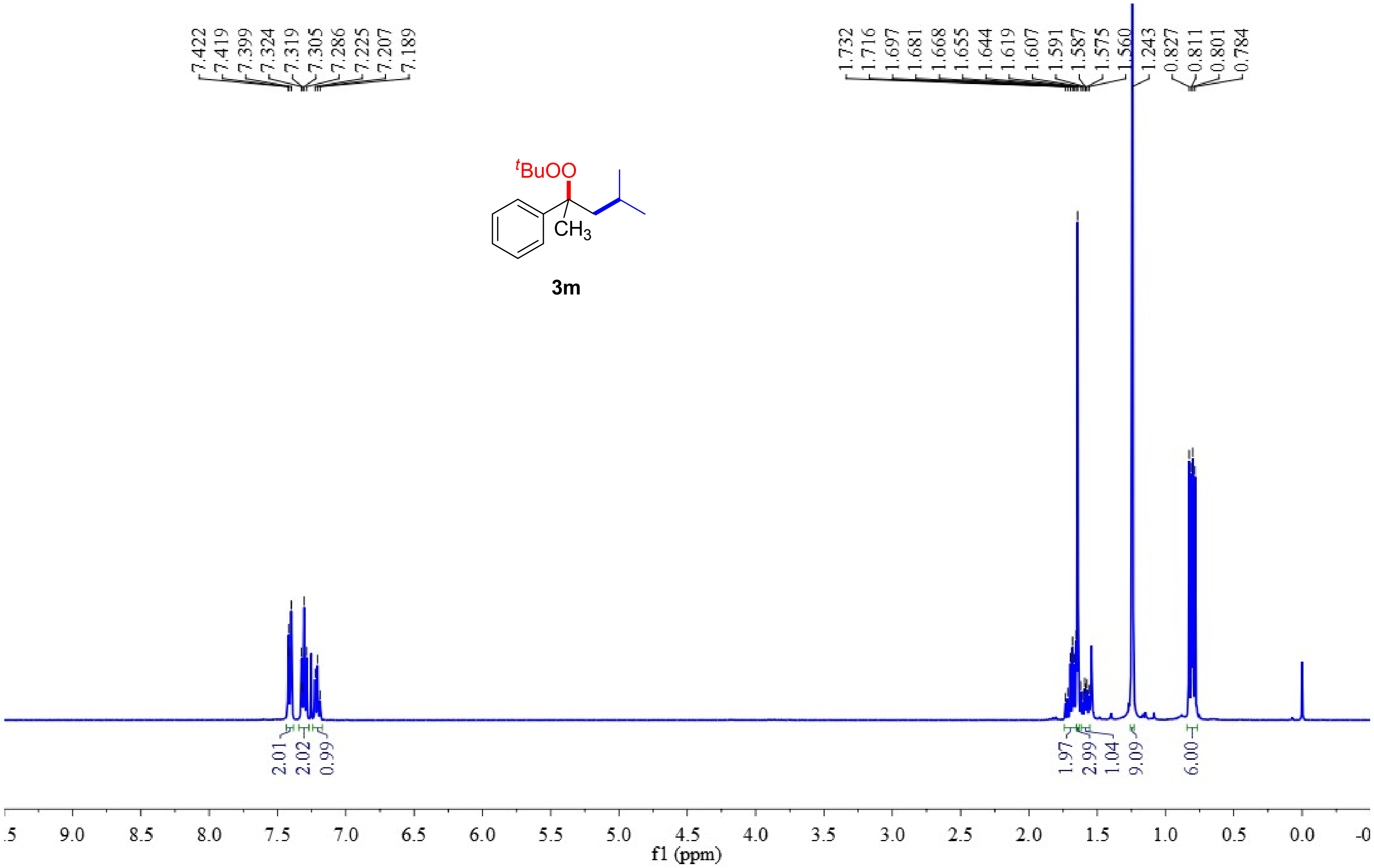

163.560
161.127
138.183
138.152
128.682
128.603
115.180
114.969
83.627
80.121
44.425
26.637
24.991
22.968
22.914
^tBuOO
F
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

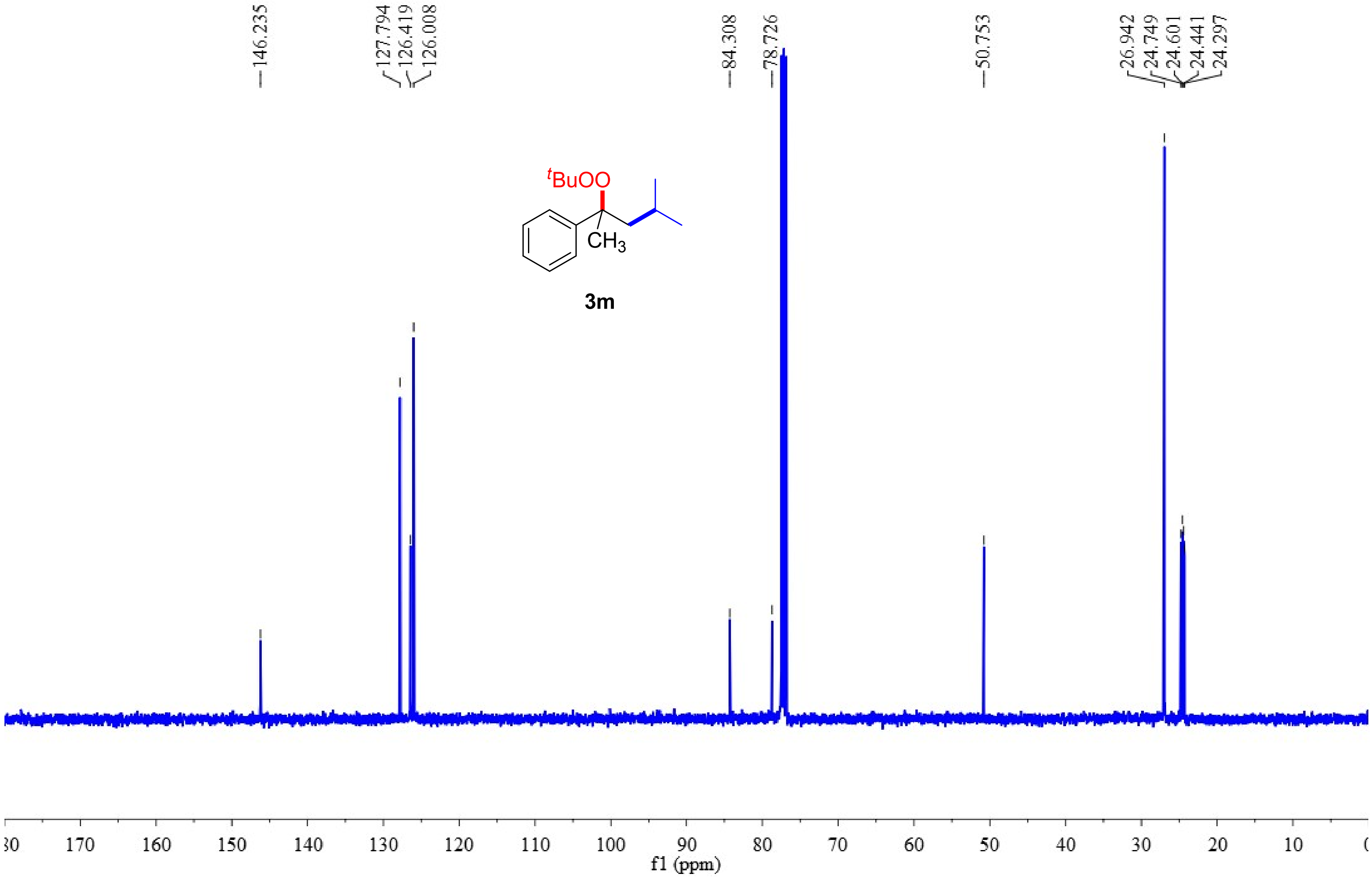
3k

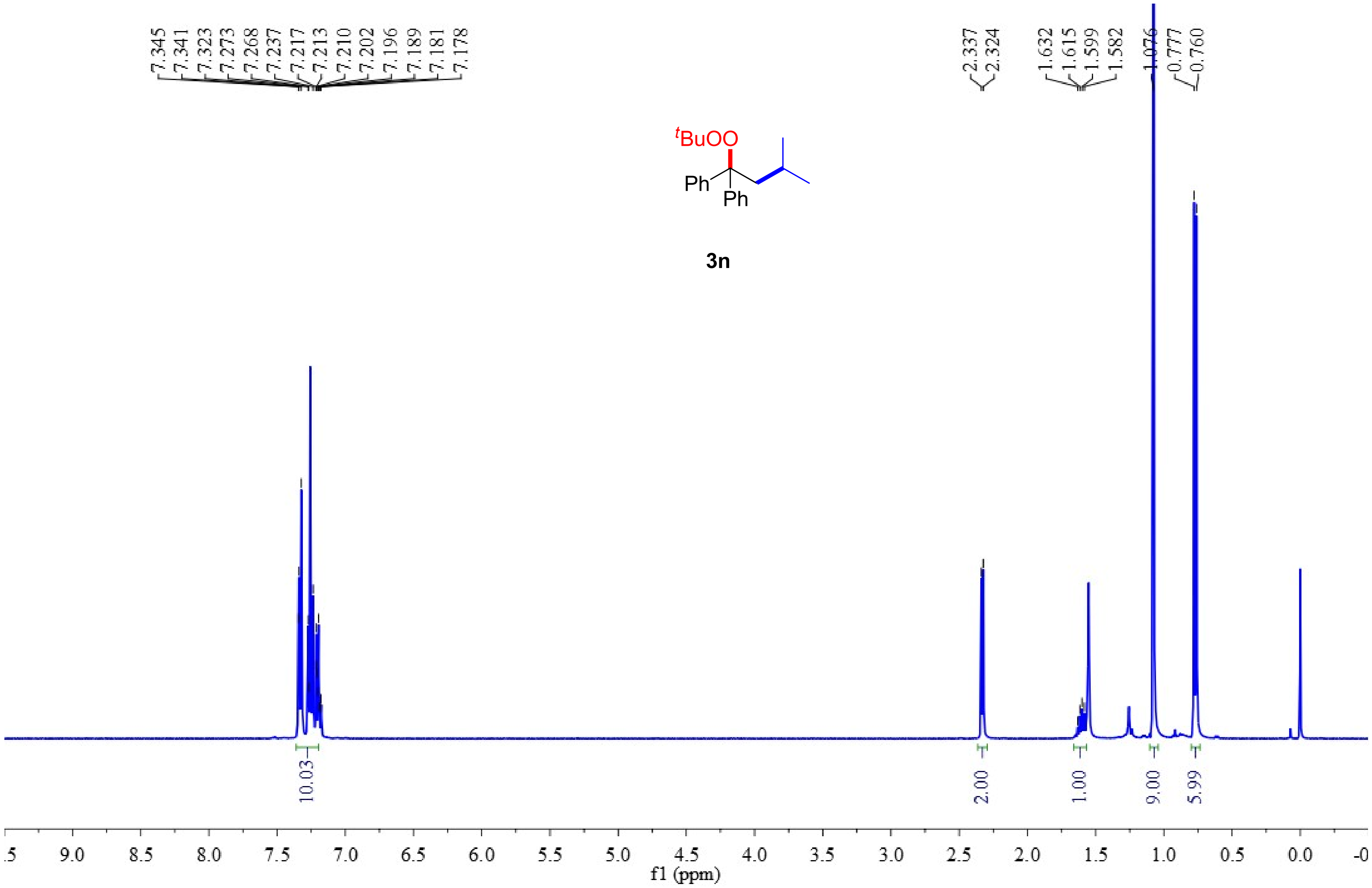

7.195
7.176
7.169
7.157
7.150
7.139
7.135
7.119
7.100
7.095
7.081
7.076
7.068
7.062
7.059
7.054
7.050
7.011
7.005
6.992
6.988
6.902
6.897
6.882
6.879
5.375
5.360
5.273
5.251
2.974
2.959
2.952
2.937
2.520
2.504
2.499
2.484
2.382
2.365
2.349
2.333
2.316
2.299
2.128
2.111
2.091
2.074
2.057
1.193
1.137
1.066
1.050
0.969
0.952
0.915
0.898
0.733
0.716
tBuOO
Ph
10.05
0.50
0.52
0.53
0.50
0.53
0.49
4.51
4.50
1.53
1.55
1.54
1.51
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

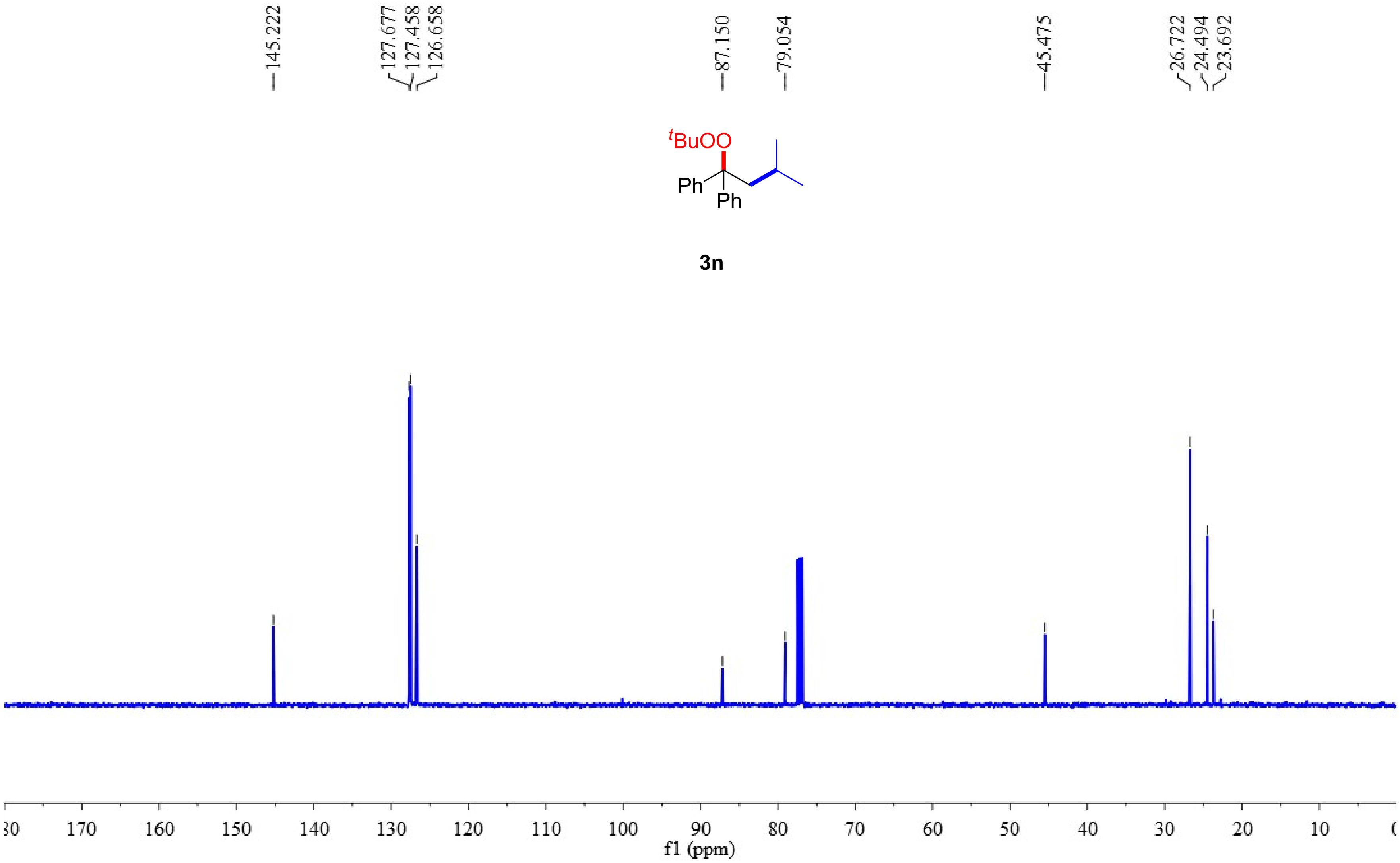
3l


141.722
140.769
139.436
138.678
130.420
130.156
127.937
127.649
127.398
127.369
127.034
126.964
126.168
126.130
85.963
84.949
79.910
79.865
59.936
57.048
29.403
28.319
26.797
26.743
22.142
21.845
20.612
19.119
^{t}BuOO
Ph
3l
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.422
7.419
7.399
7.324
7.319
7.305
7.286
7.225
7.207
7.189
1.732
1.716
1.697
1.681
1.668
1.655
1.644
1.619
1.607
1.591
1.587
1.575
1.560
1.243
0.827
0.811
0.801
0.784
^{t}BuOO
CH$_3$
3m
2.01
2.02
0.99
1.97
2.99
1.04
9.09
6.00
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

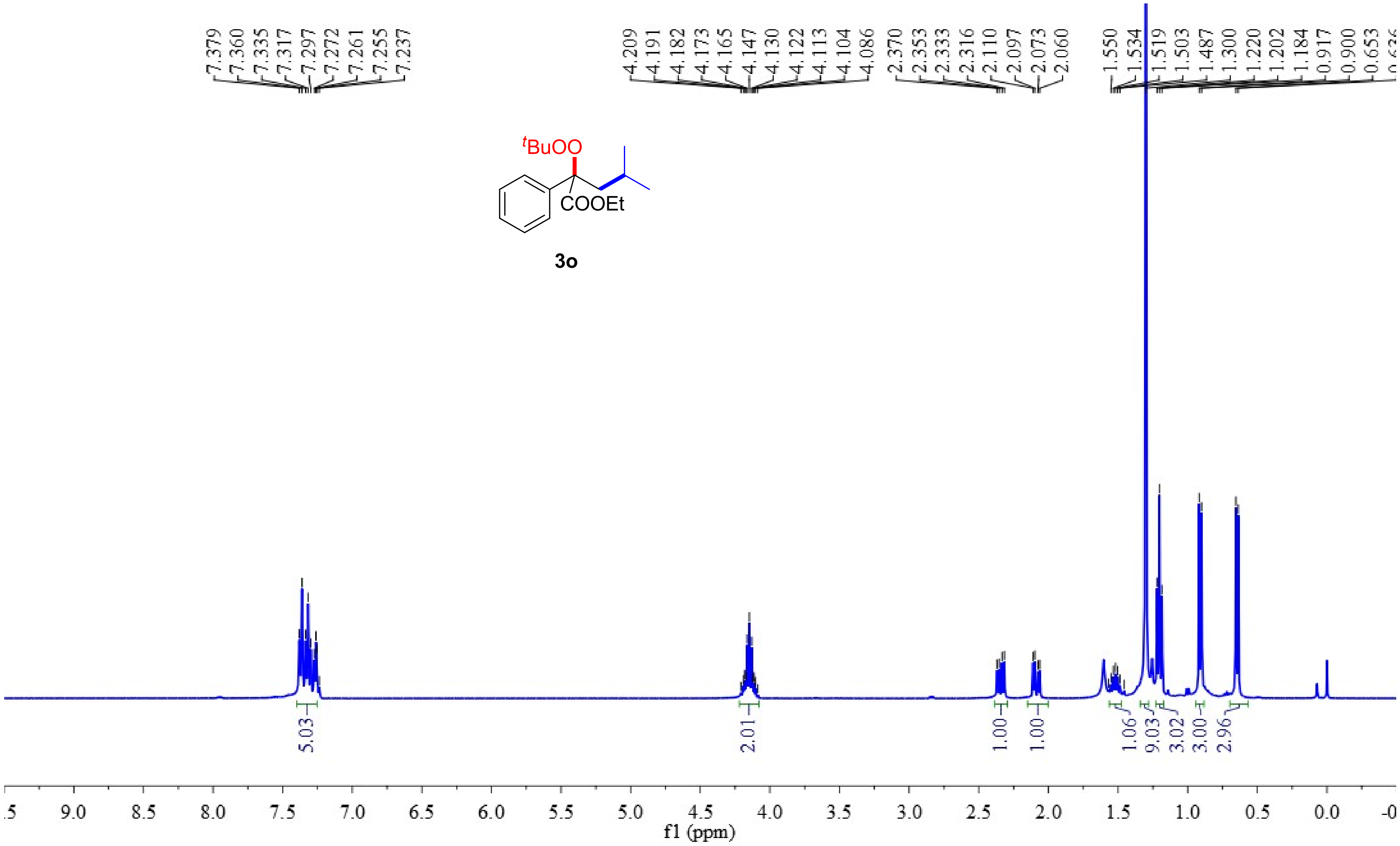

146.235
127.794
126.419
126.008
84.308
78.726
50.753
26.942
24.749
24.601
24.441
24.297
^{t}BuOO
CH$_3$
3m
30
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.345
7.341
7.323
7.273
7.268
7.237
7.217
7.213
7.210
7.202
7.196
7.189
7.181
7.178
2.337
2.324
1.632
1.615
1.599
1.582
1.076
0.777
0.760
tBuOO
Ph
Ph
3n
10.03
2.00
1.00
9.00
5.99
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)



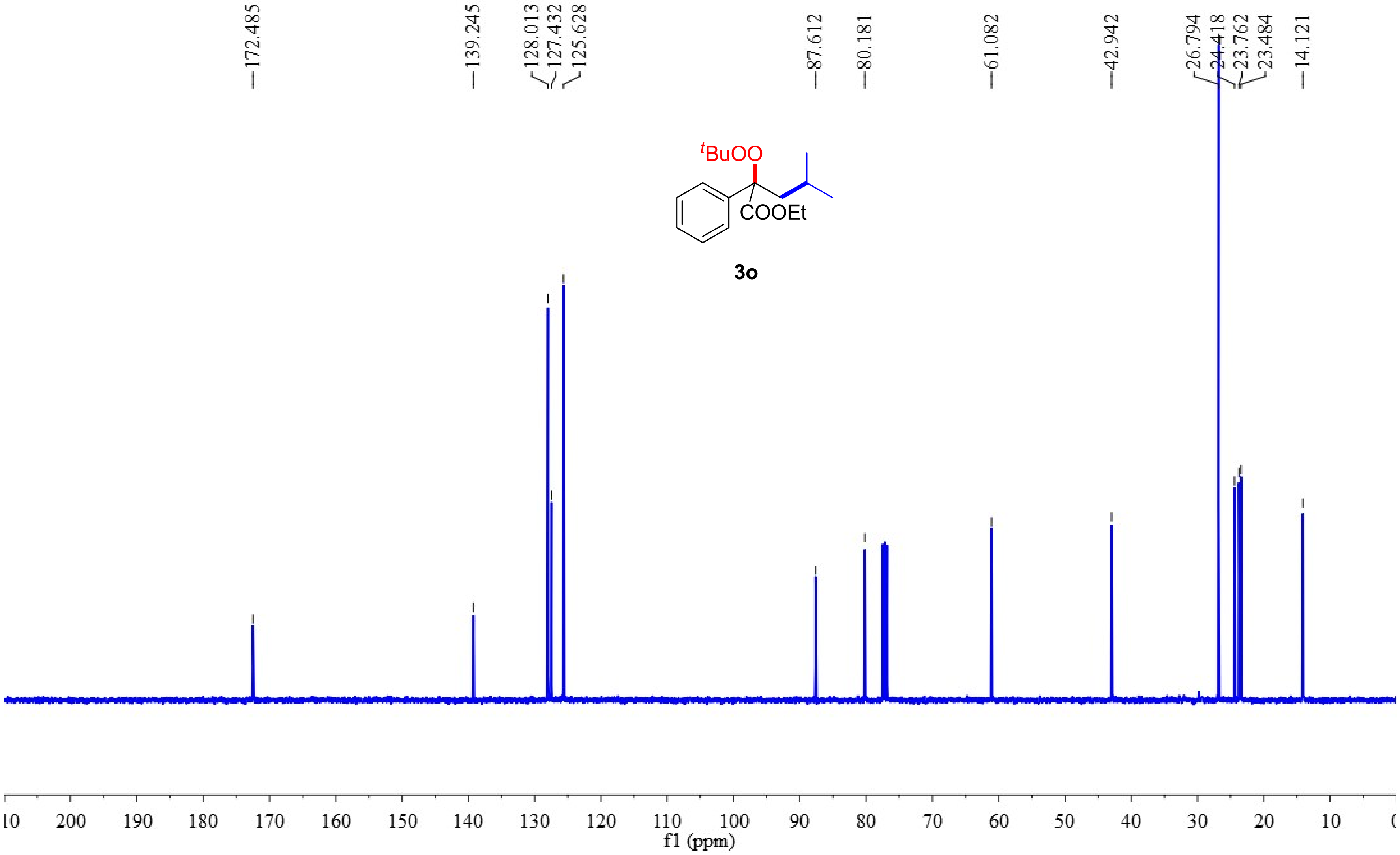
3n

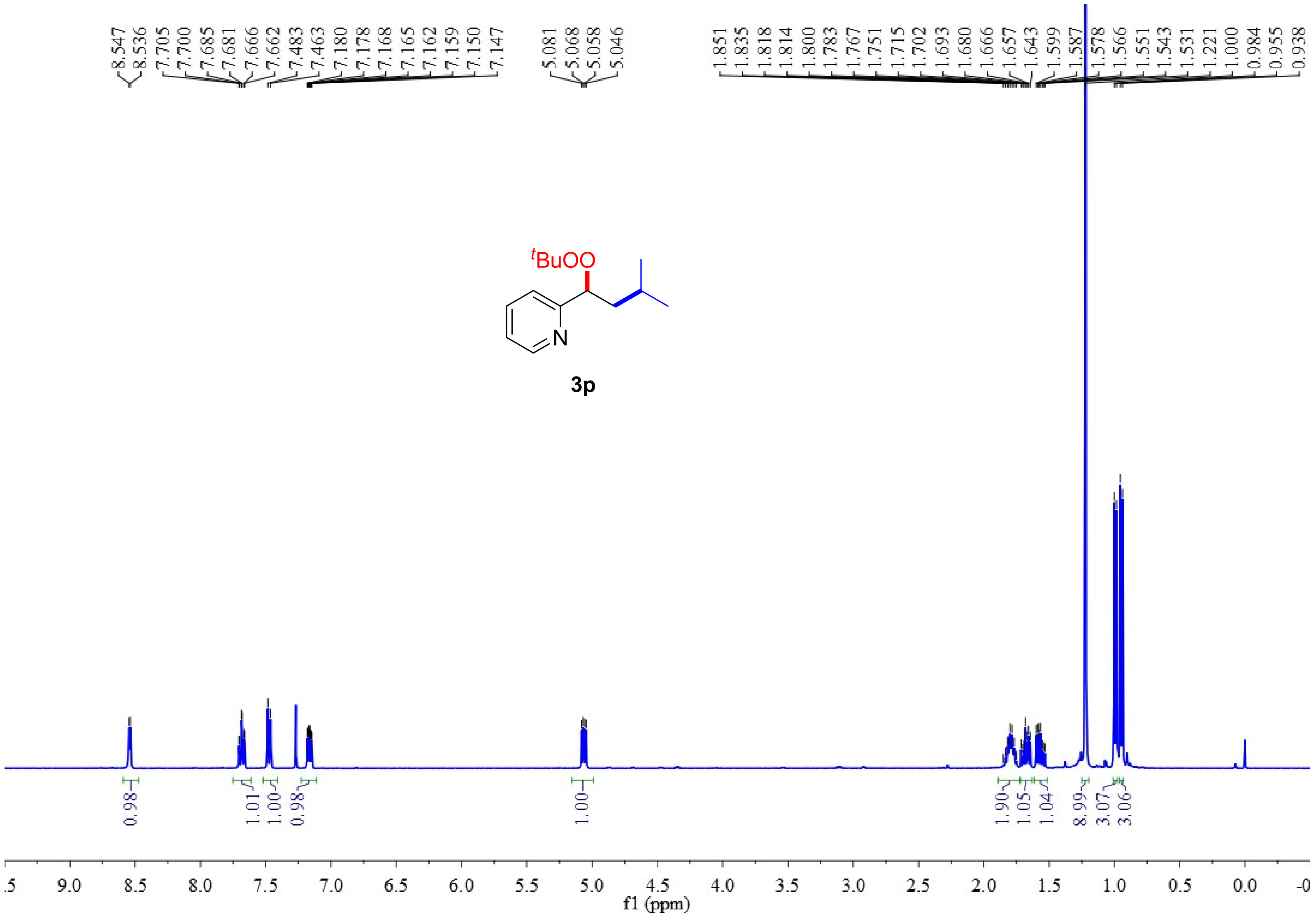
145.222
127.677
127.458
126.658
87.150
79.054
45.475
26.722
24.494
23.692
^{t}BuOO
Ph
Ph
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)



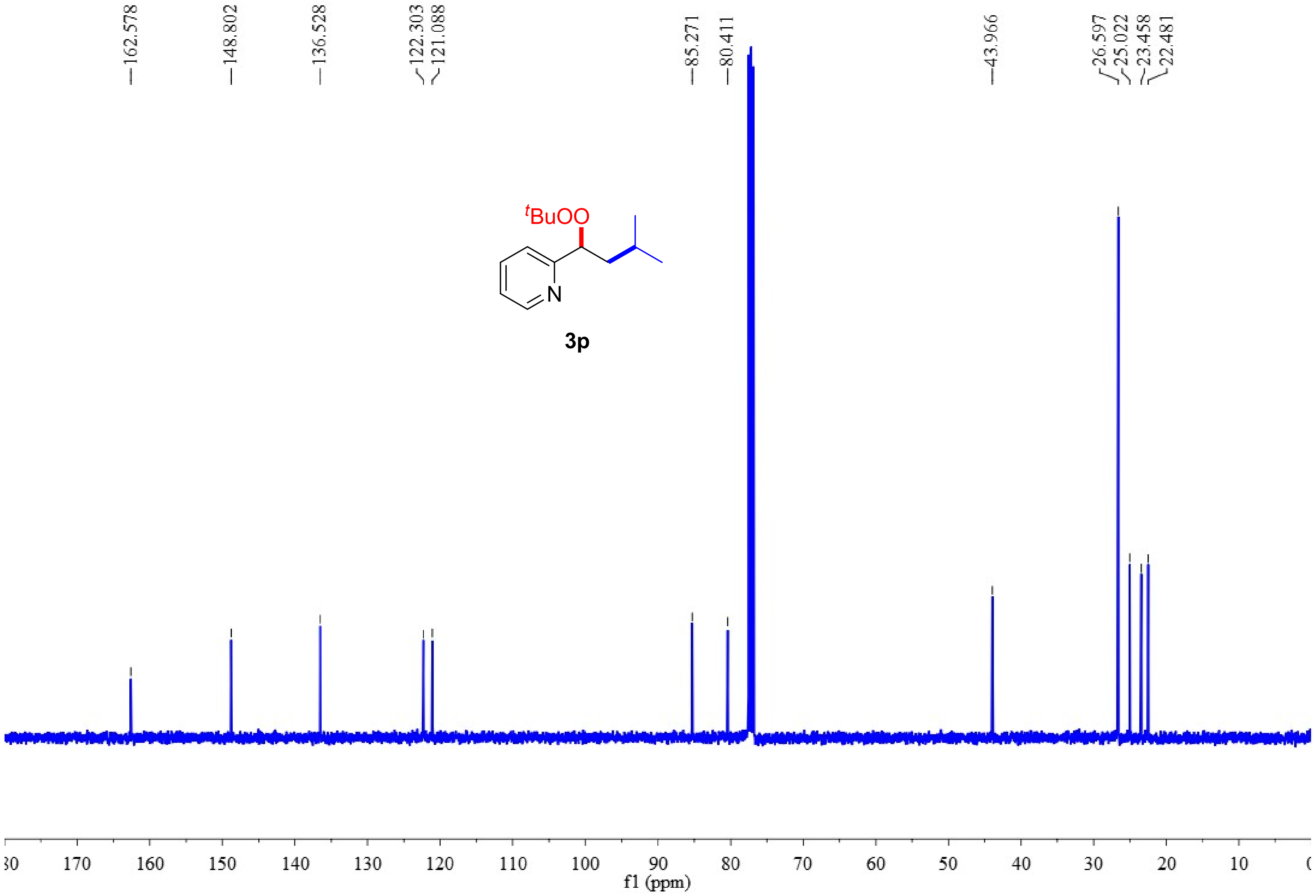
3o

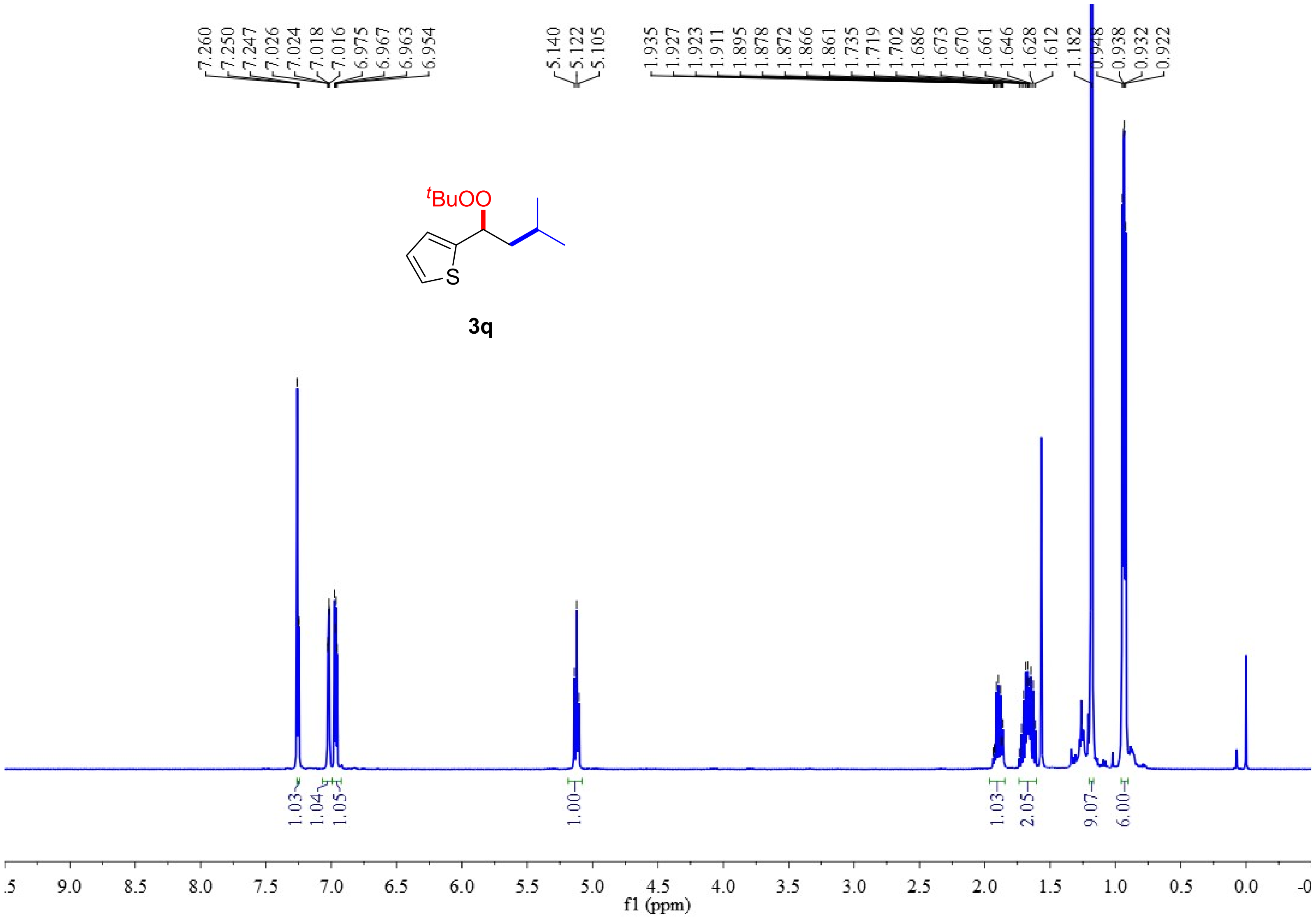
7.379
7.360
7.335
7.317
7.297
7.272
7.261
7.255
7.237
4.209
4.191
4.182
4.173
4.165
4.147
4.130
4.122
4.113
4.104
4.086
2.370
2.353
2.333
2.316
2.110
2.097
2.073
2.060
1.550
1.534
1.519
1.503
1.487
1.300
1.220
1.202
1.184
0.917
0.900
0.653
^{t}BuOO
COOEt
5.03
2.01
1.00
1.00
1.06
9.03
3.02
3.00
2.96
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


172.485
139.245
128.013
127.432
125.628
87.612
80.181
61.082
42.942
26.794
24.418
23.762
23.484
14.121
^{t}BuOO
COOEt
3o
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

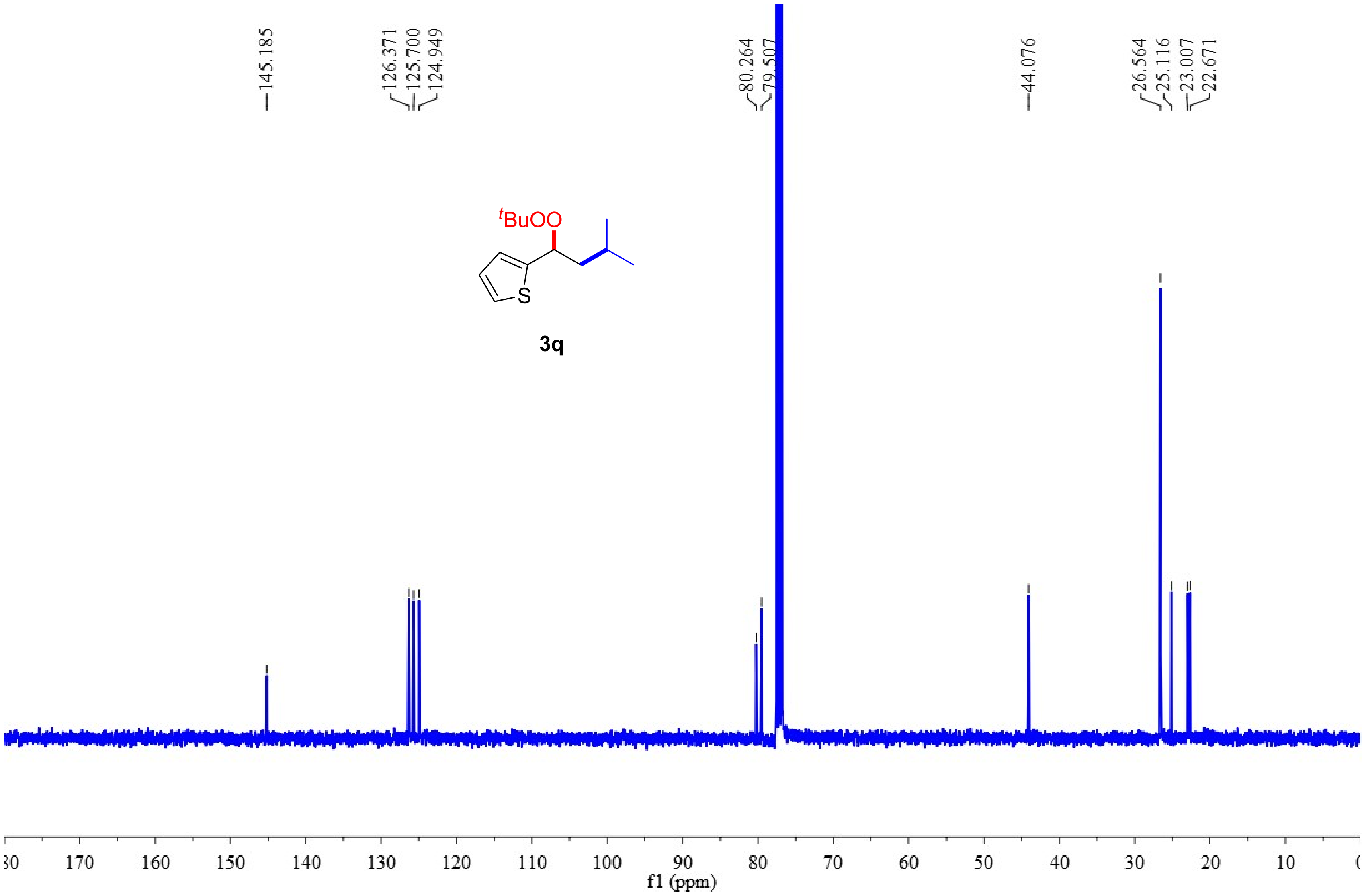

8.547
8.536
7.705
7.700
7.685
7.681
7.666
7.662
7.483
7.463
7.180
7.178
7.168
7.165
7.162
7.159
7.150
7.147
5.081
5.068
5.058
5.046
1.851
1.835
1.818
1.814
1.800
1.783
1.767
1.751
1.715
1.702
1.693
1.680
1.666
1.657
1.643
1.599
1.587
1.578
1.566
1.551
1.543
1.531
1.221
1.000
0.984
0.955
0.938
^{t}BuOO
N
0.98
1.01
1.00
0.98
1.00
1.90
1.05
1.04
8.99
3.07
3.06
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

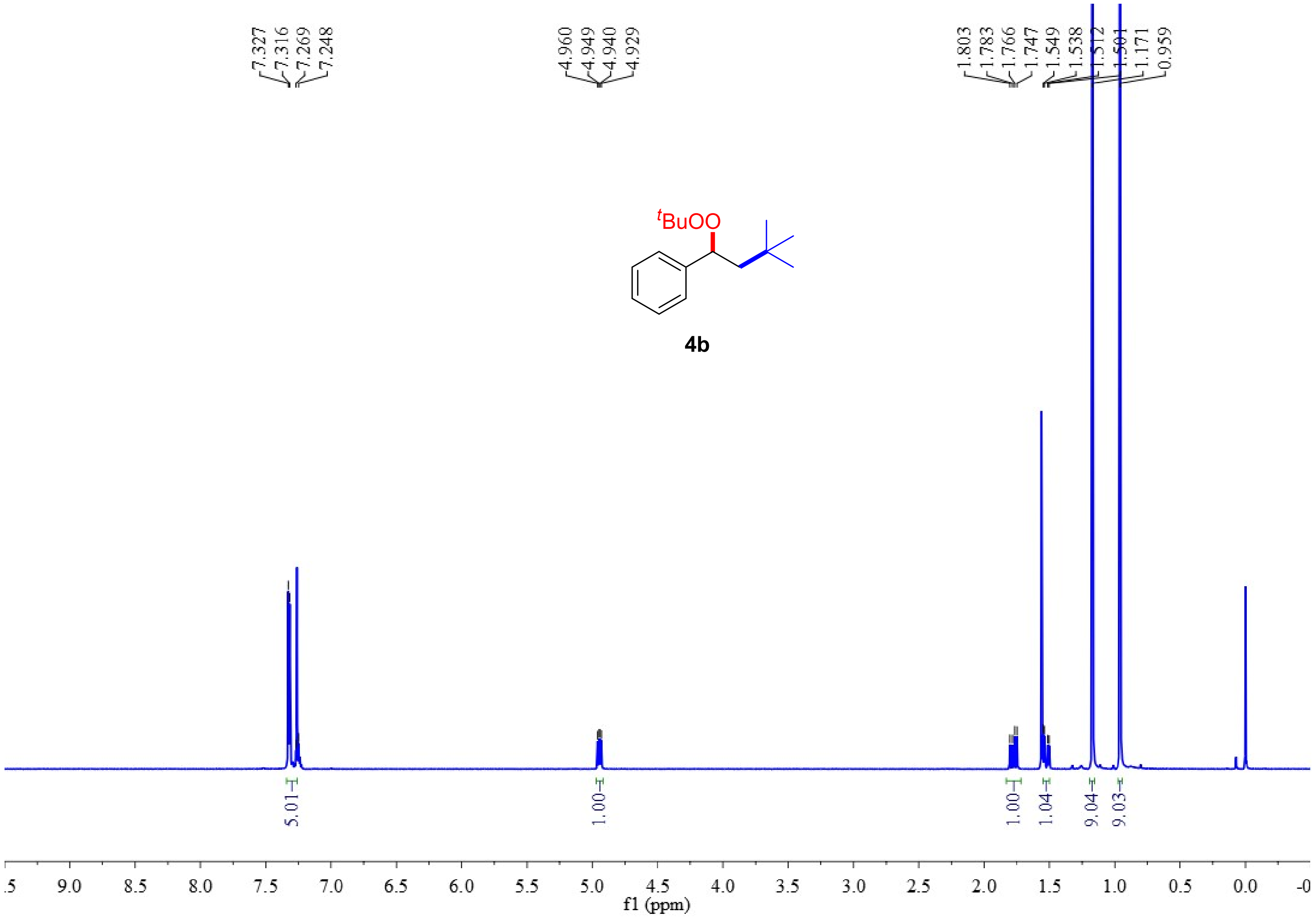
3p

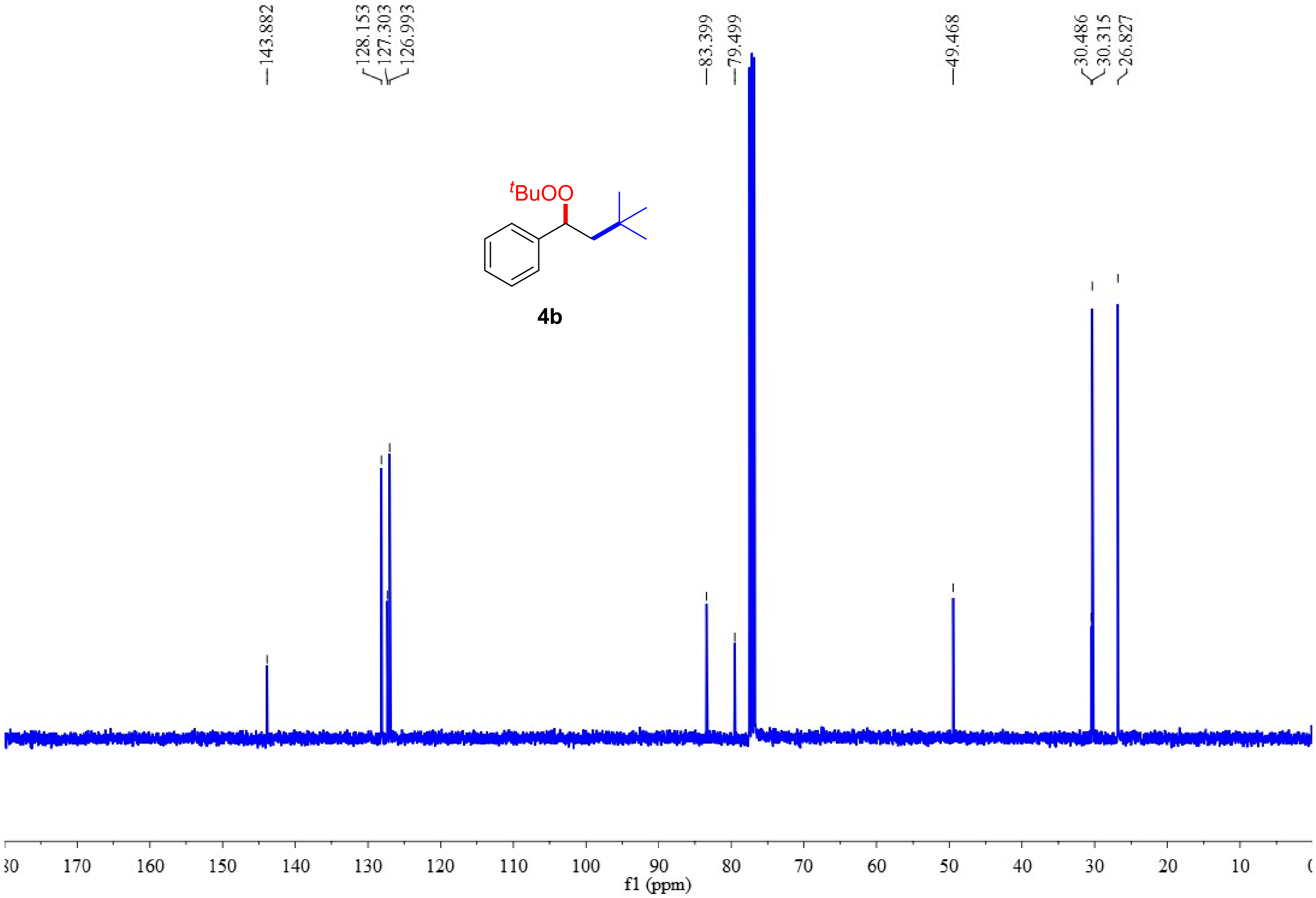

162.578
148.802
136.528
122.303
121.088
85.271
80.411
43.966
26.597
25.022
23.458
22.481
^{t}BuOO
N
3p
30
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

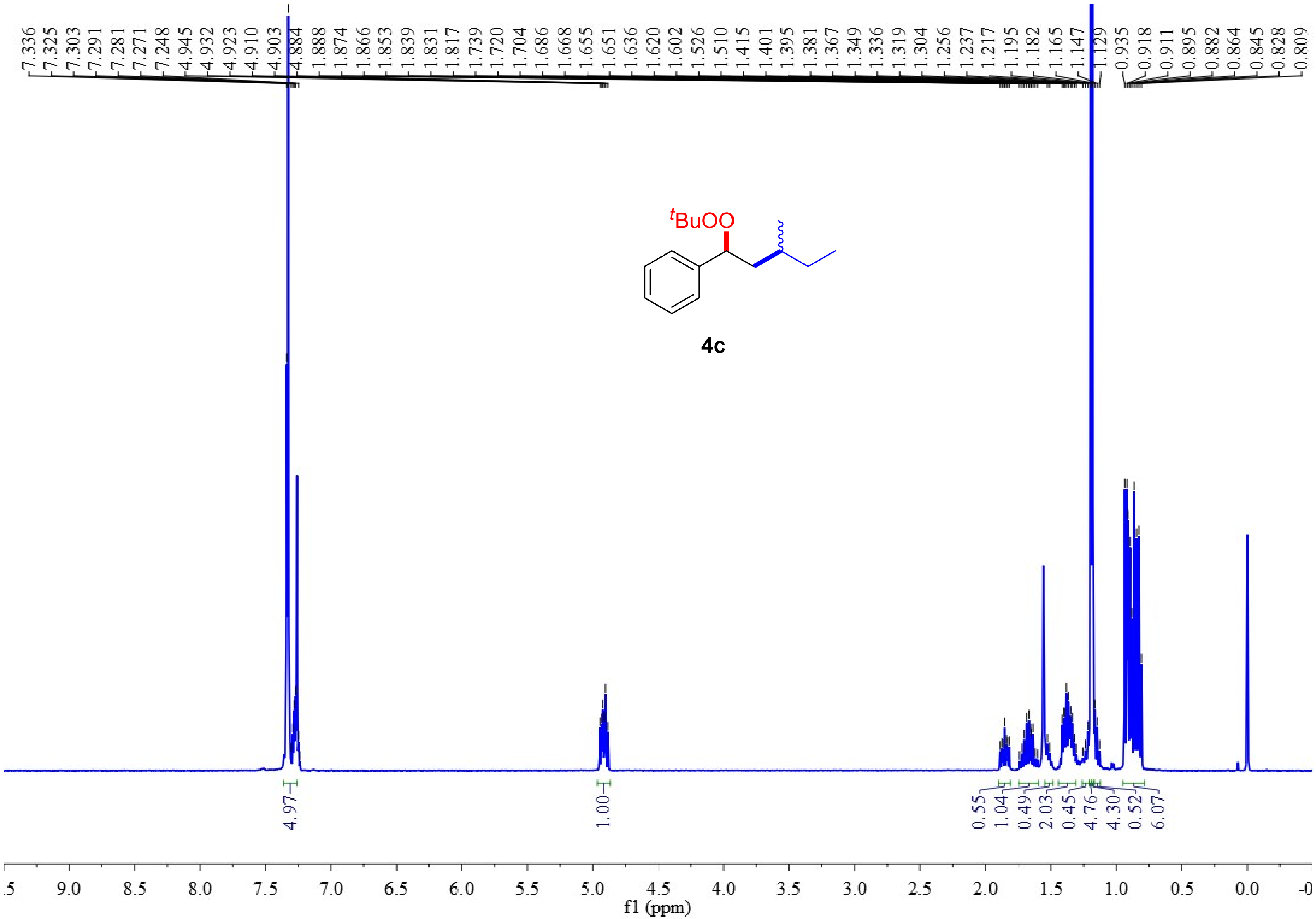

7.260
7.250
7.247
7.026
7.024
7.018
7.016
6.975
6.967
6.963
6.954
5.140
5.122
5.105
1.935
1.927
1.923
1.911
1.895
1.878
1.872
1.866
1.861
1.735
1.719
1.702
1.686
1.673
1.670
1.661
1.646
1.628
1.612
1.182
0.948
0.938
0.932
0.922
tBuOO
S
1.03
1.04
1.05
1.00
1.03
2.05
9.07
6.00
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

3q

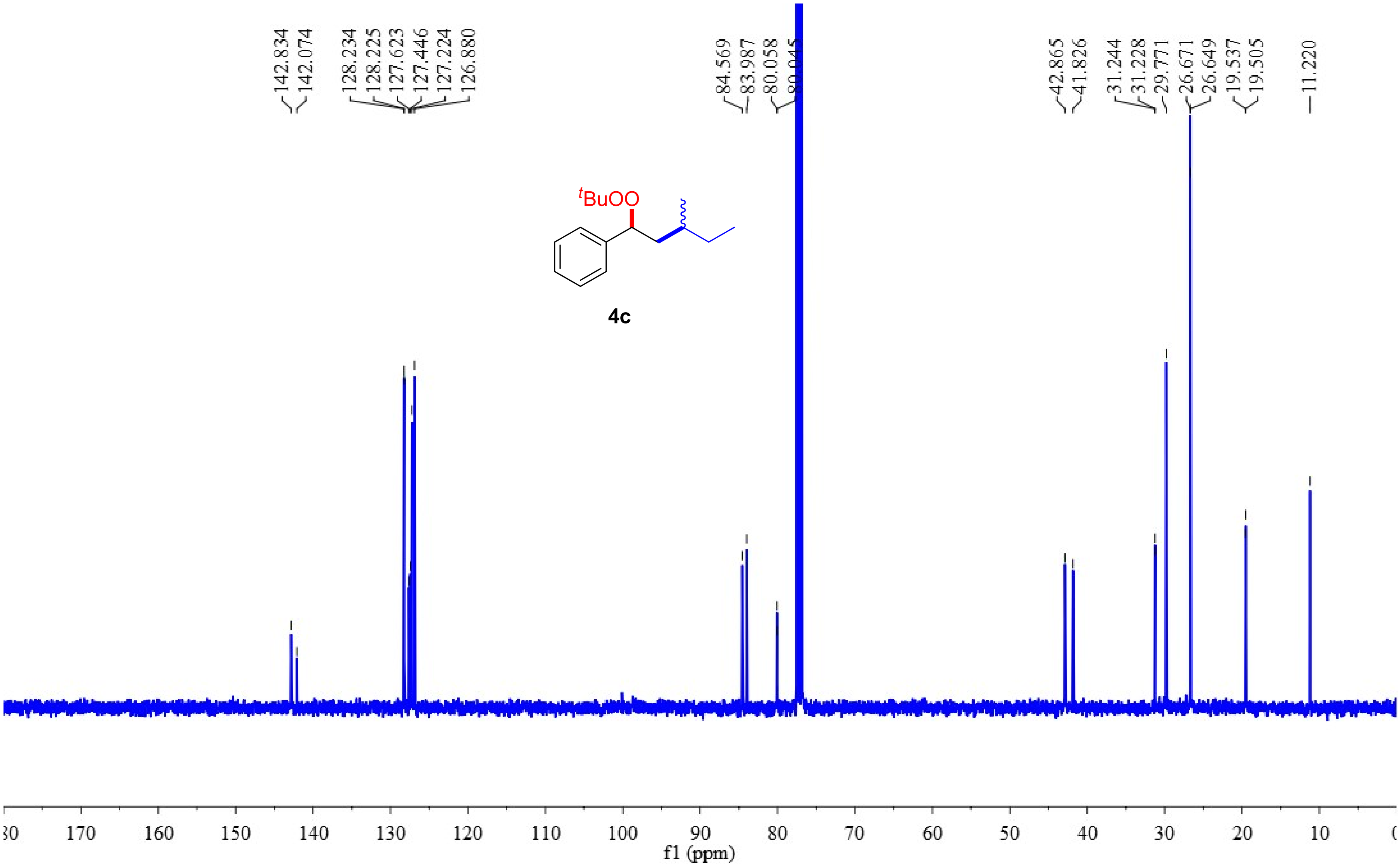

145.185
126.371
125.700
124.949
80.264
79.507
44.076
26.564
25.116
23.007
22.671
^{t}BuOO
S
3q
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

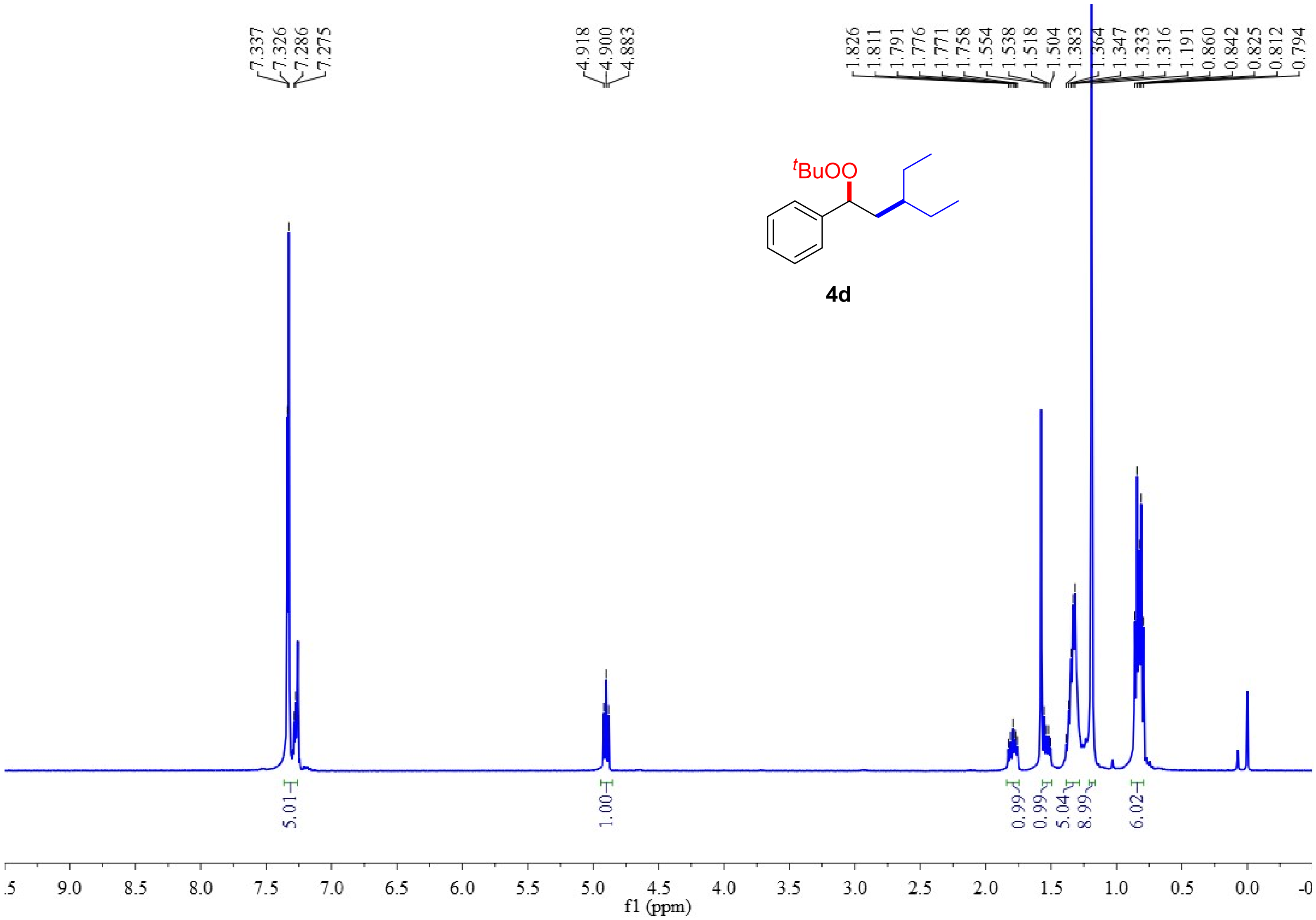

7.327
7.316
7.269
7.248
4.960
4.949
4.940
4.929
1.803
1.783
1.766
1.747
1.549
1.538
1.512
1.501
1.171
0.959
^{t}BuOO
4b
5.01
1.00
1.00
1.04
9.04
9.03
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)


143.882
128.153
127.303
126.993
83.399
79.499
49.468
30.486
30.315
26.827
^tBuOO
4b
30
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

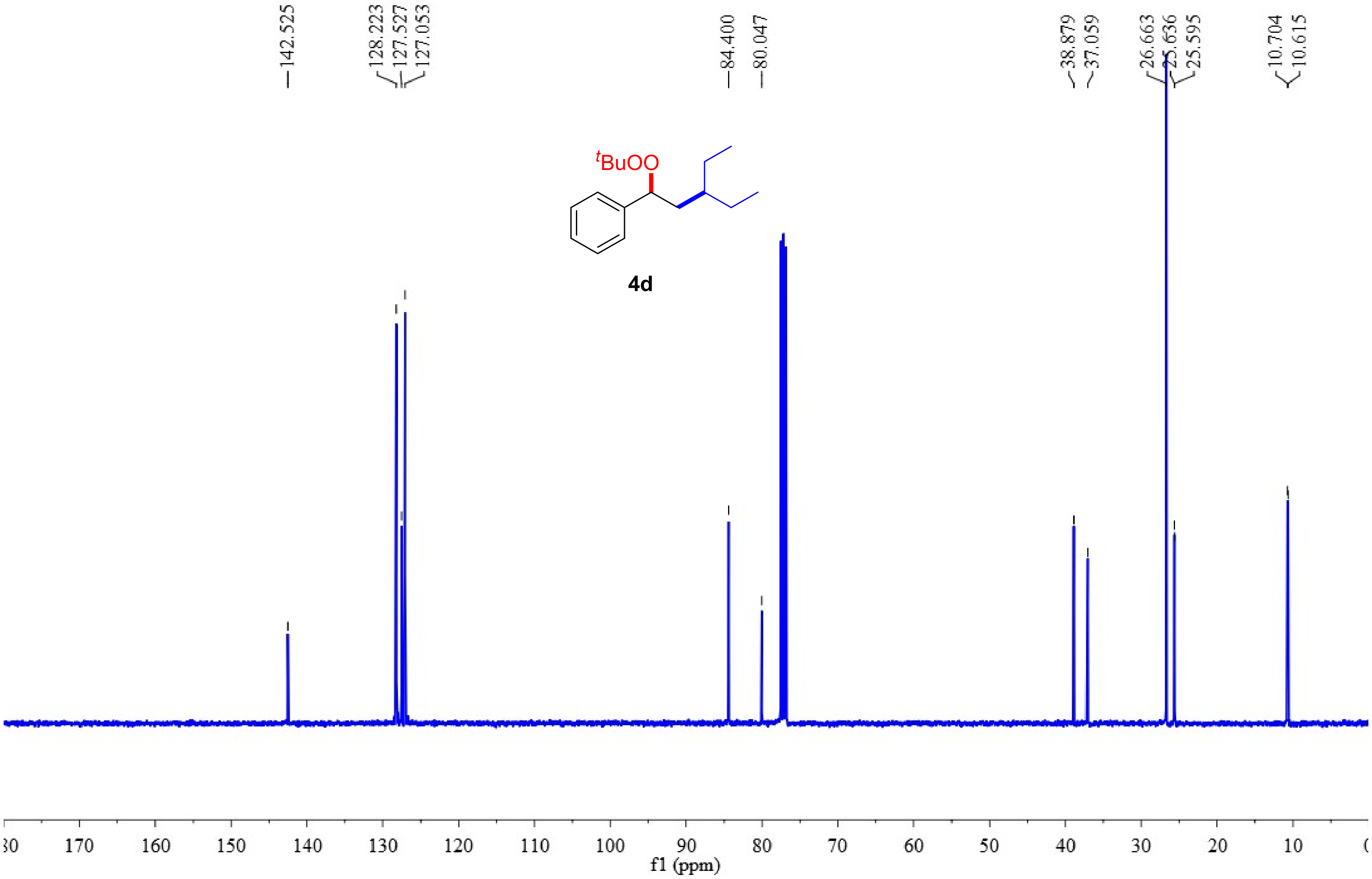

7.336
7.325
7.303
7.291
7.281
7.271
7.248
4.945
4.932
4.923
4.910
4.903
4.884
1.888
1.874
1.866
1.853
1.839
1.831
1.817
1.739
1.720
1.704
1.686
1.668
1.655
1.651
1.636
1.620
1.602
1.526
1.510
1.415
1.401
1.395
1.381
1.367
1.349
1.336
1.319
1.304
1.256
1.237
1.217
1.195
1.182
1.165
1.147
1.129
0.935
0.918
0.911
0.895
0.882
0.864
0.845
0.828
0.809
^{t}BuOO
4.97
1.00
0.55
1.04
0.49
2.03
0.45
4.76
4.30
0.52
6.07
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

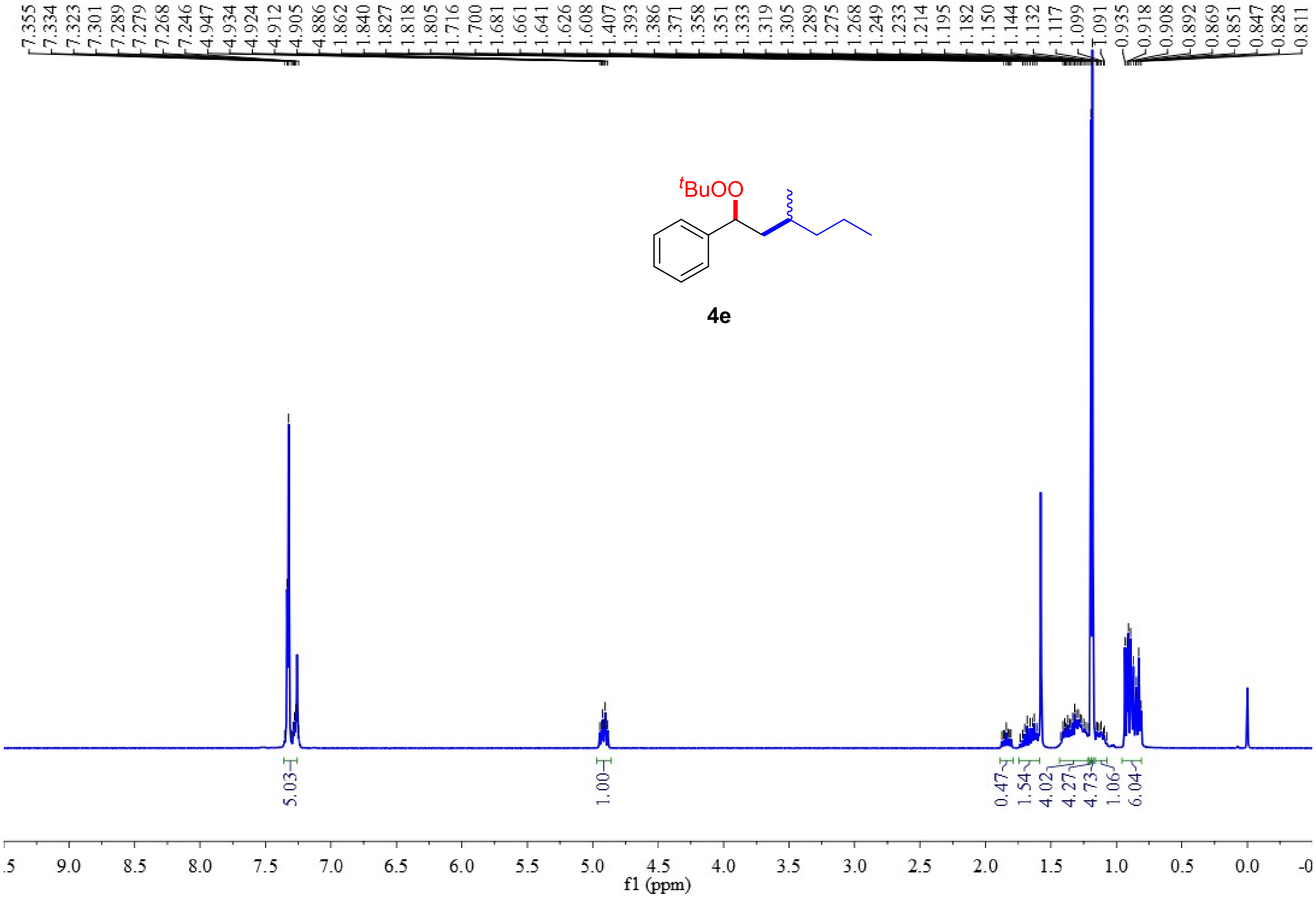
4c


142.834
142.074
128.234
128.225
127.623
127.446
127.224
126.880
84.569
83.987
80.058
80.045
42.865
41.826
31.244
31.228
29.771
26.671
26.649
19.537
19.505
11.220
^{t}BuOO
4c
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.337
7.326
7.286
7.275
4.918
4.900
4.883
1.826
1.811
1.791
1.776
1.771
1.758
1.554
1.538
1.518
1.504
1.383
1.364
1.347
1.333
1.316
1.191
0.860
0.842
0.825
0.812
0.794
^{t}BuOO
5.01
1.00
0.99
0.99
5.04
8.99
6.02
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

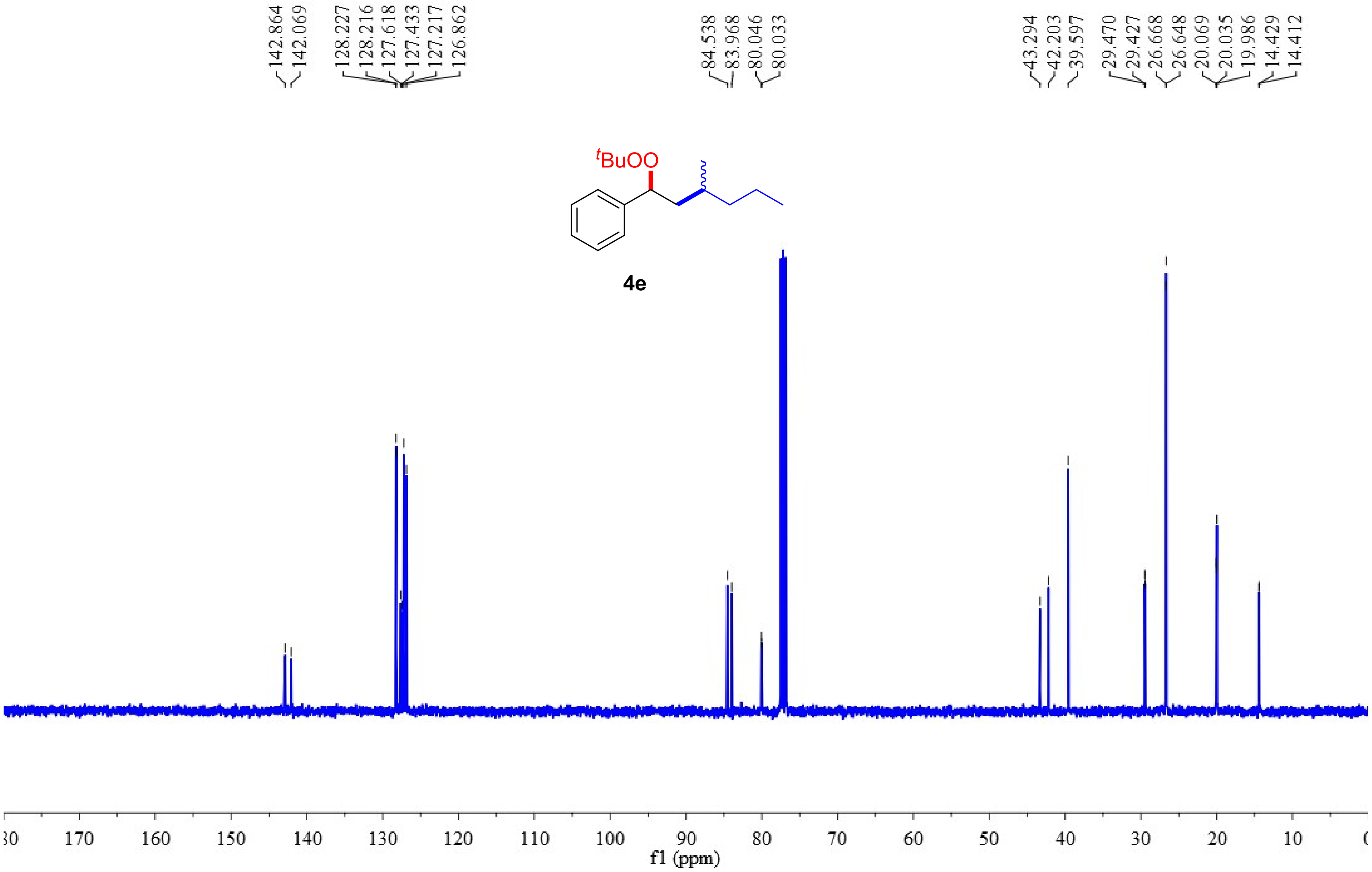
4d


142.525
128.223
127.527
127.053
84.400
80.047
38.879
37.059
26.663
25.636
25.595
10.704
10.615
^{t}BuOO
4d
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)



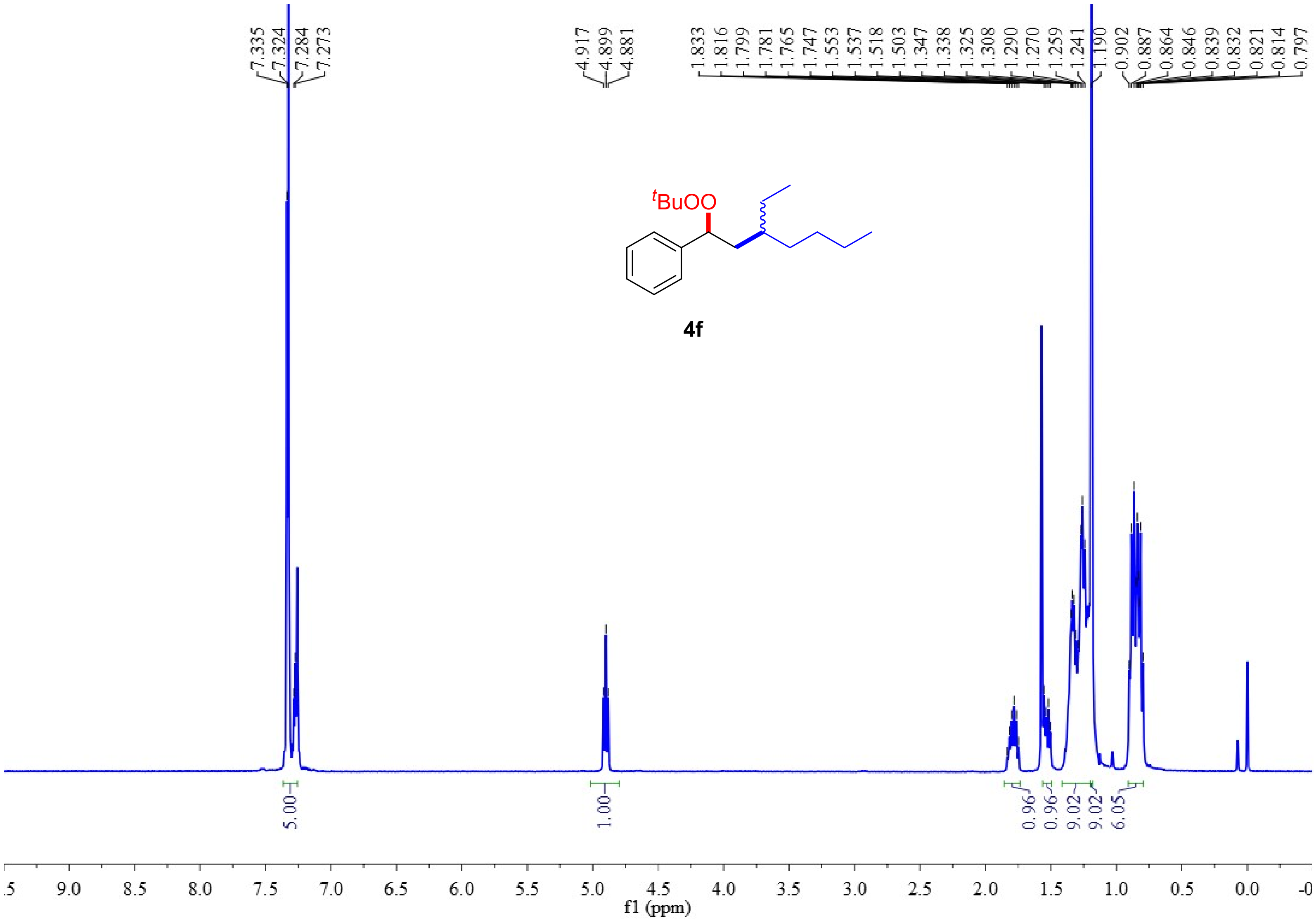
4e

7.355
7.334
7.323
7.301
7.289
7.279
7.268
7.246
4.947
4.934
4.924
4.912
4.905
4.886
1.862
1.840
1.827
1.818
1.805
1.716
1.700
1.681
1.661
1.641
1.626
1.608
1.407
1.393
1.386
1.371
1.358
1.351
1.333
1.319
1.305
1.289
1.275
1.268
1.249
1.233
1.214
1.195
1.182
1.150
1.144
1.132
1.117
1.099
1.091
0.935
0.918
0.908
0.892
0.869
0.851
0.847
0.828
0.811
^{t}BuOO
5.03
1.00
0.47
1.54
4.02
4.27
4.73
1.06
6.04
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

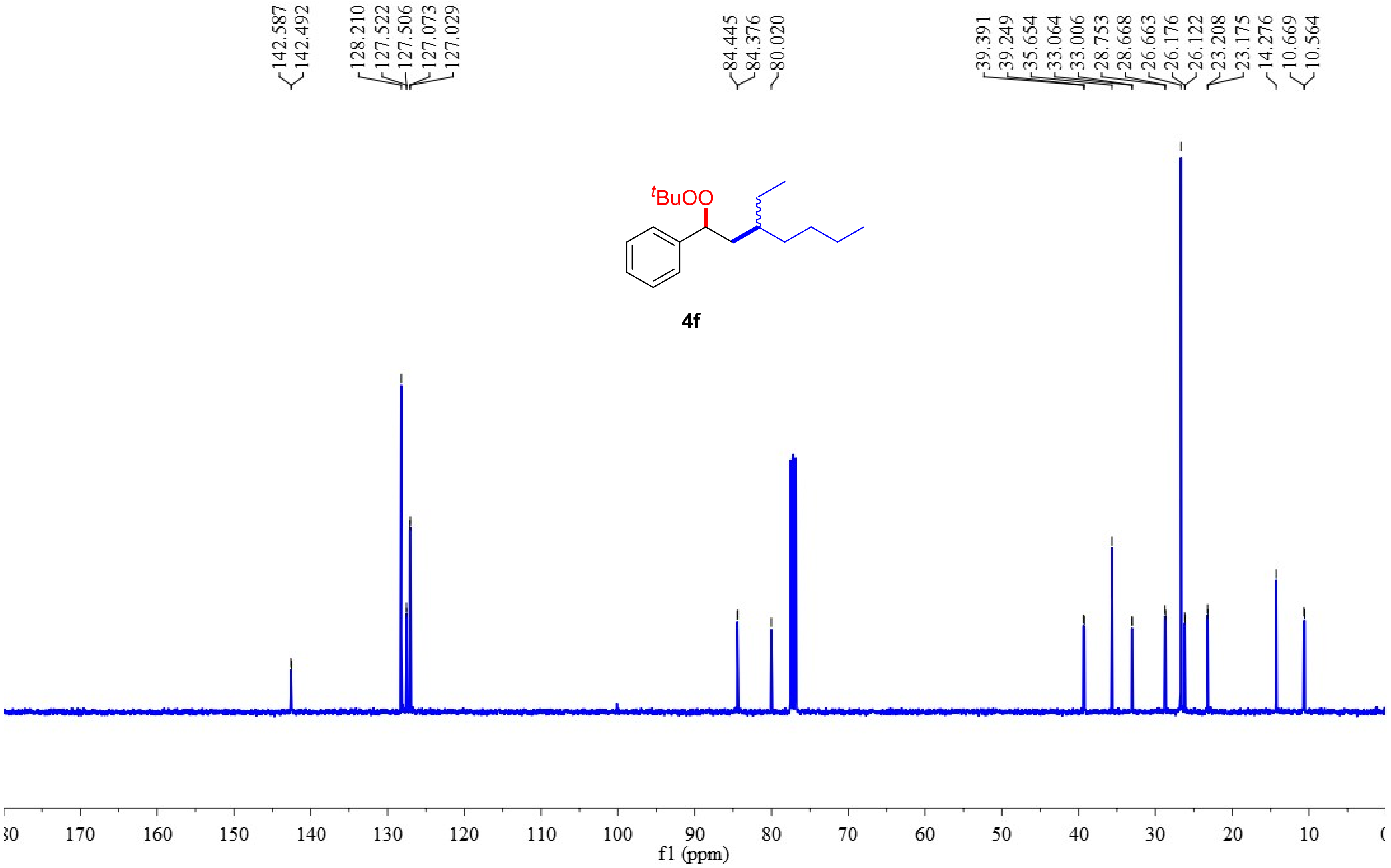


4e

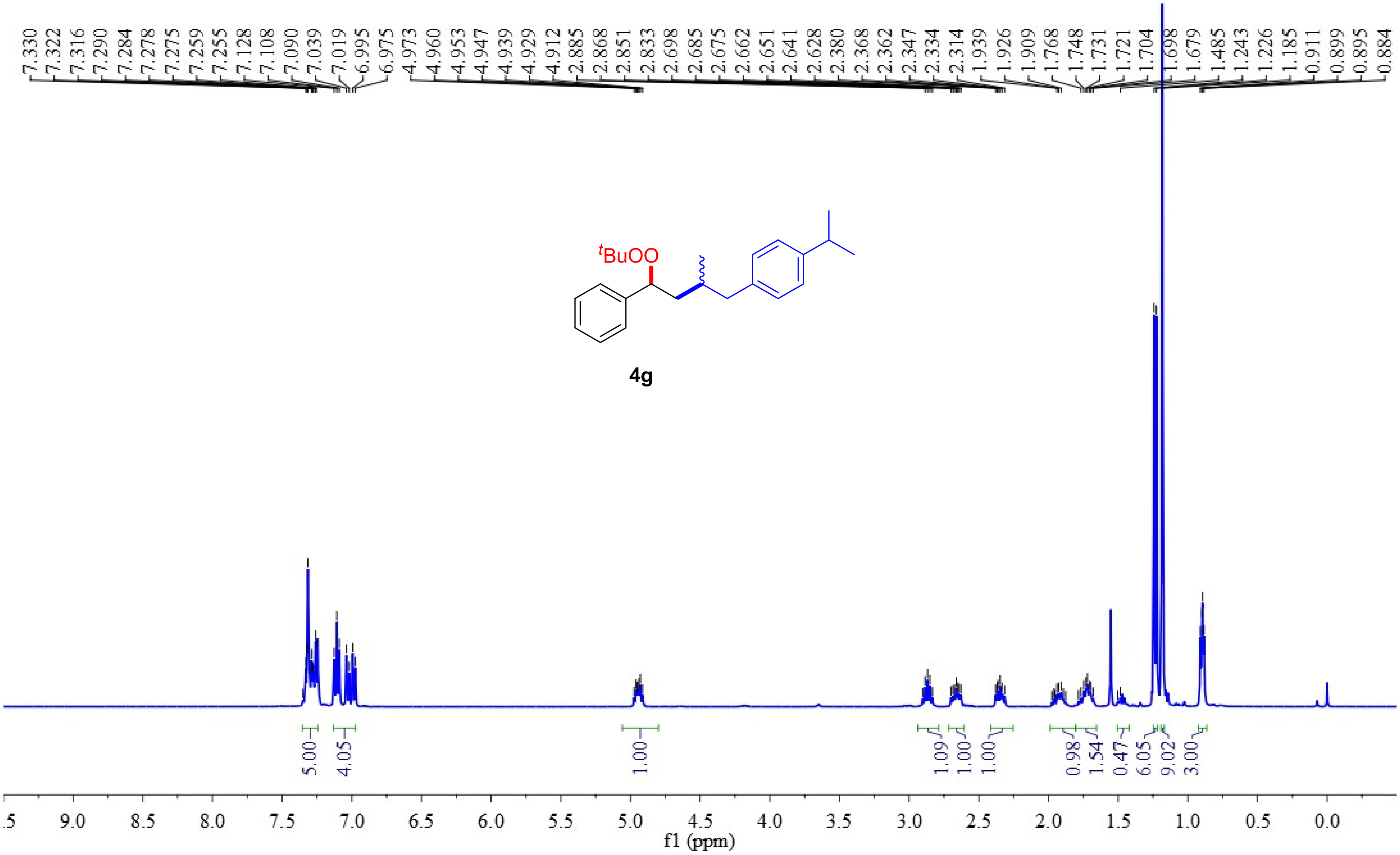
142.864
142.069
128.227
128.216
127.618
127.433
127.217
126.862
84.538
83.968
80.046
80.033
43.294
42.203
39.597
29.470
29.427
26.668
26.648
20.069
20.035
19.986
14.429
14.412
^{t}BuOO
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.335
7.324
7.284
7.273
4.917
4.899
4.881
1.833
1.816
1.799
1.781
1.765
1.747
1.553
1.537
1.518
1.503
1.347
1.338
1.325
1.308
1.290
1.270
1.259
1.241
1.190
0.902
0.887
0.864
0.846
0.839
0.832
0.821
0.814
0.797
^{t}BuOO
5.00
1.00
0.96
0.96
9.02
9.02
6.05
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

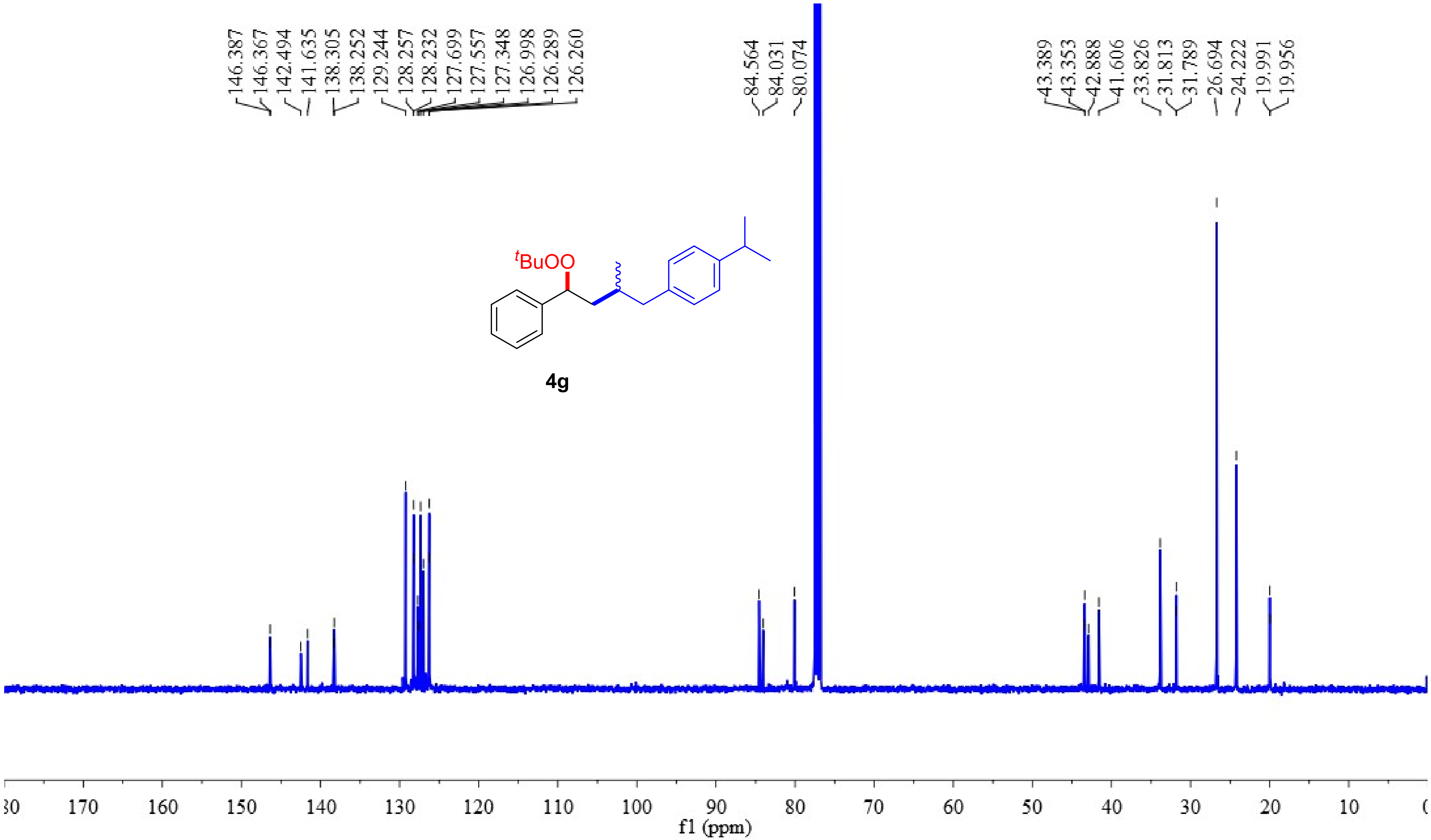
4f



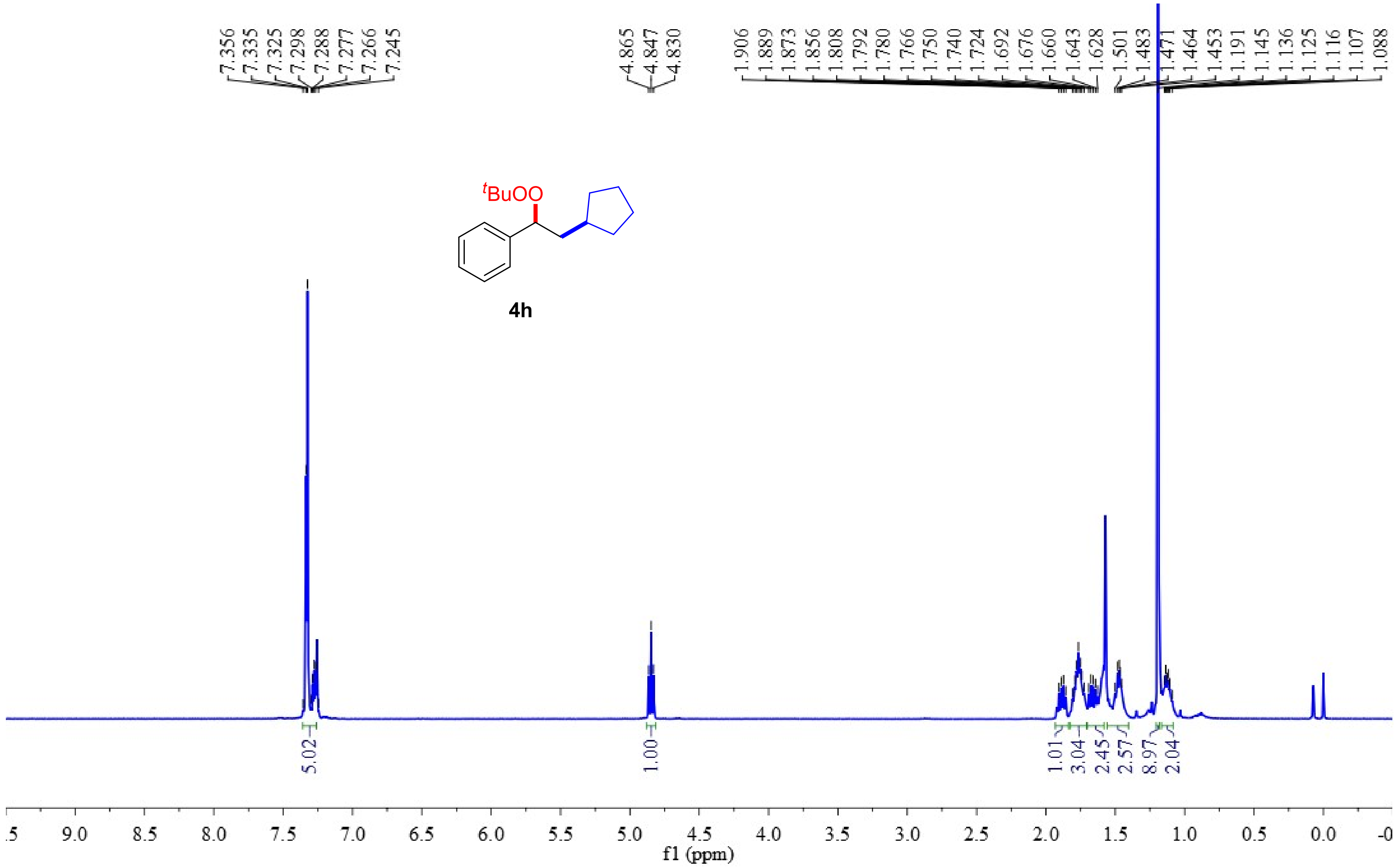
142.587
142.492
128.210
127.522
127.506
127.073
127.029
84.445
84.376
80.020
39.391
39.249
35.654
33.064
33.006
28.753
28.668
26.663
26.176
26.122
23.208
23.175
14.276
10.669
10.564
^{t}BuOO
4f
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


4g

7.330
7.322
7.316
7.290
7.284
7.278
7.275
7.259
7.255
7.128
7.108
7.090
7.039
7.019
6.995
6.975
4.973
4.960
4.953
4.947
4.939
4.929
4.912
2.885
2.868
2.851
2.833
2.698
2.685
2.675
2.662
2.651
2.641
2.628
2.380
2.368
2.362
2.347
2.334
2.314
1.939
1.926
1.909
1.768
1.748
1.731
1.721
1.704
1.698
1.679
1.485
1.243
1.226
1.185
0.911
0.899
0.895
0.884
tBuOO
5.00
4.05
1.00
1.09
1.00
1.00
0.98
1.54
0.47
6.05
9.02
3.00
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

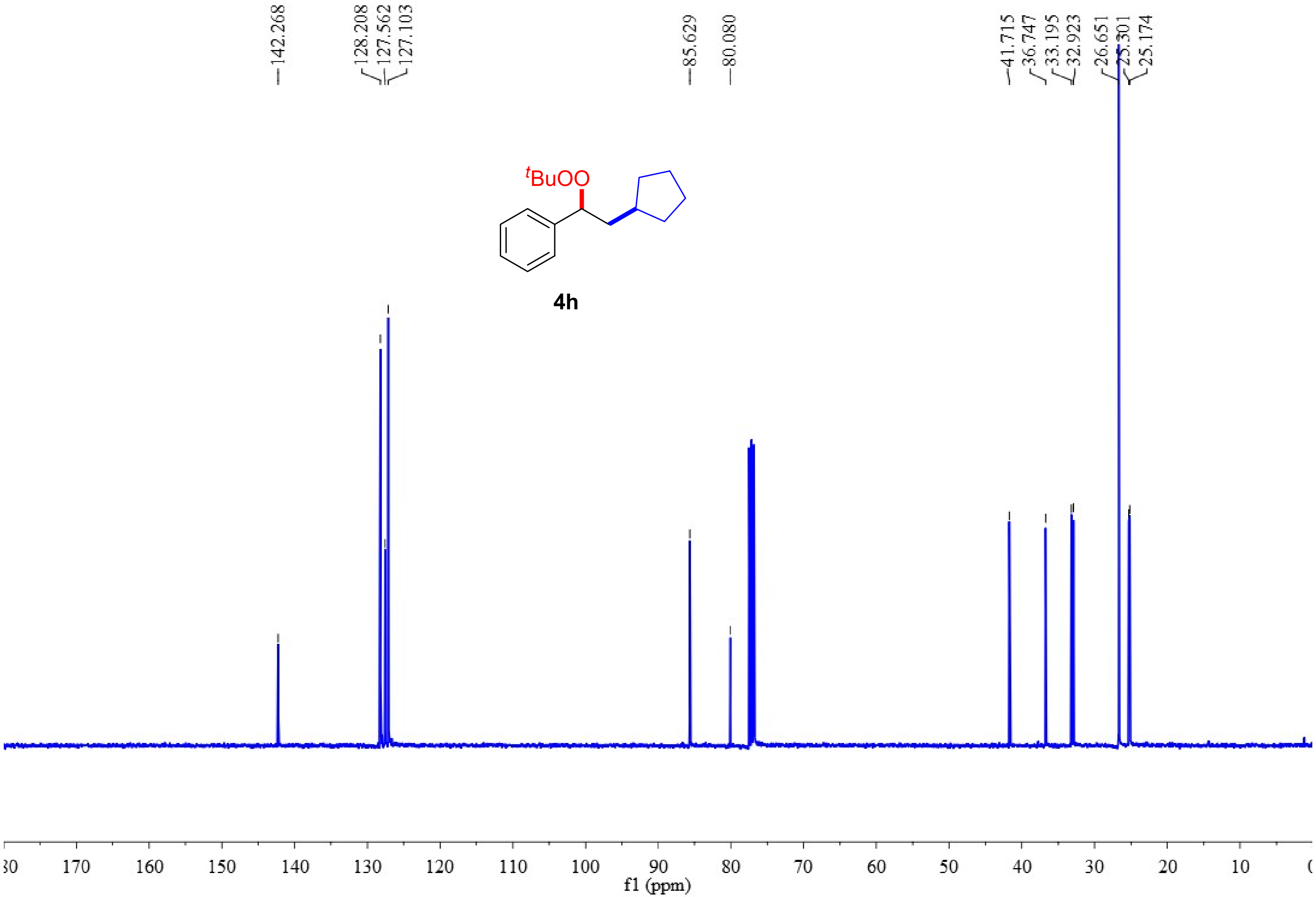

146.387
146.367
142.494
141.635
138.305
138.252
129.244
128.257
128.232
127.699
127.557
127.348
126.998
126.289
126.260
84.564
84.031
80.074
43.389
43.353
42.888
41.606
33.826
31.813
31.789
26.694
24.222
19.991
19.956
^{t}BuOO
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

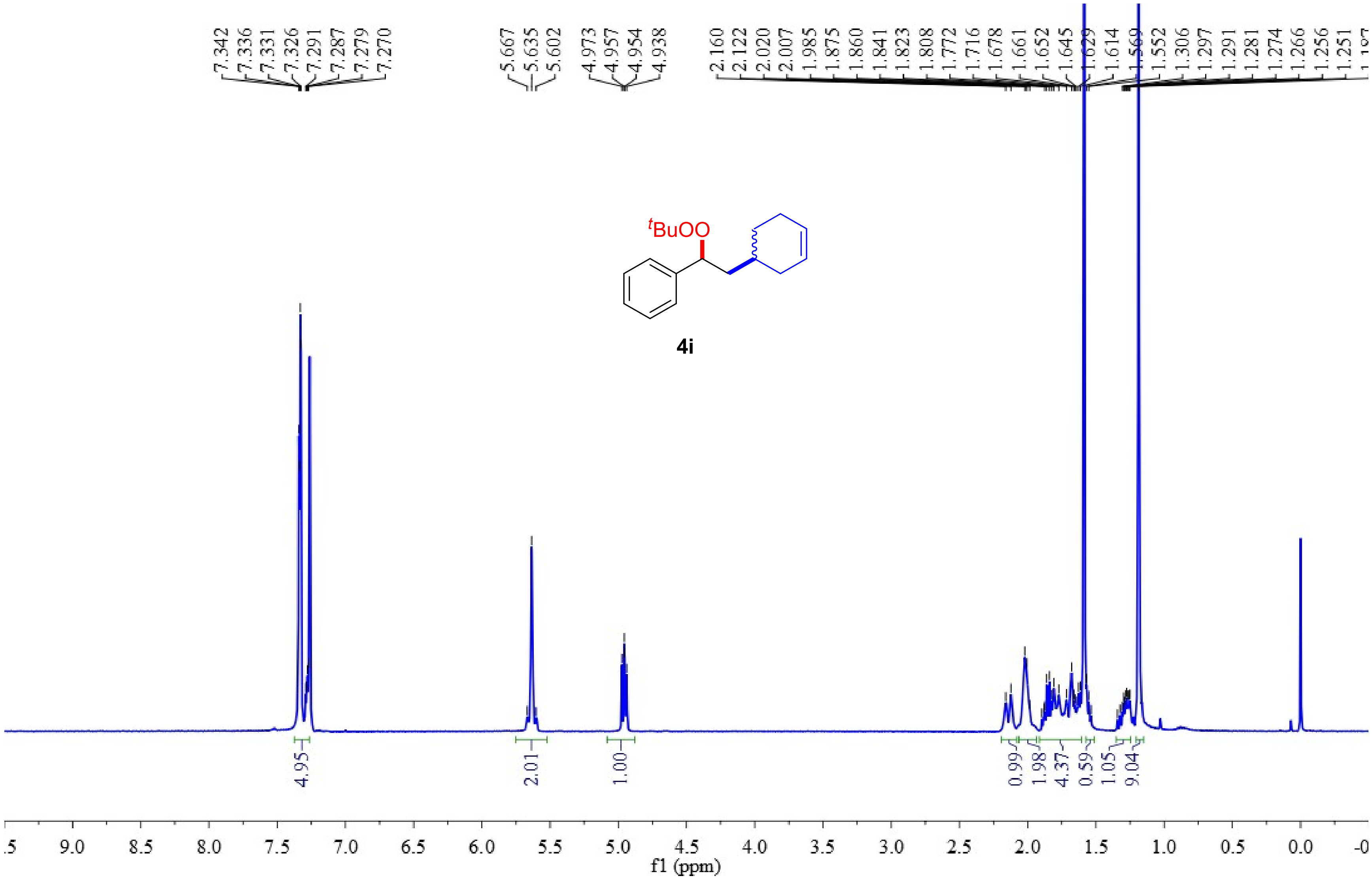
4g



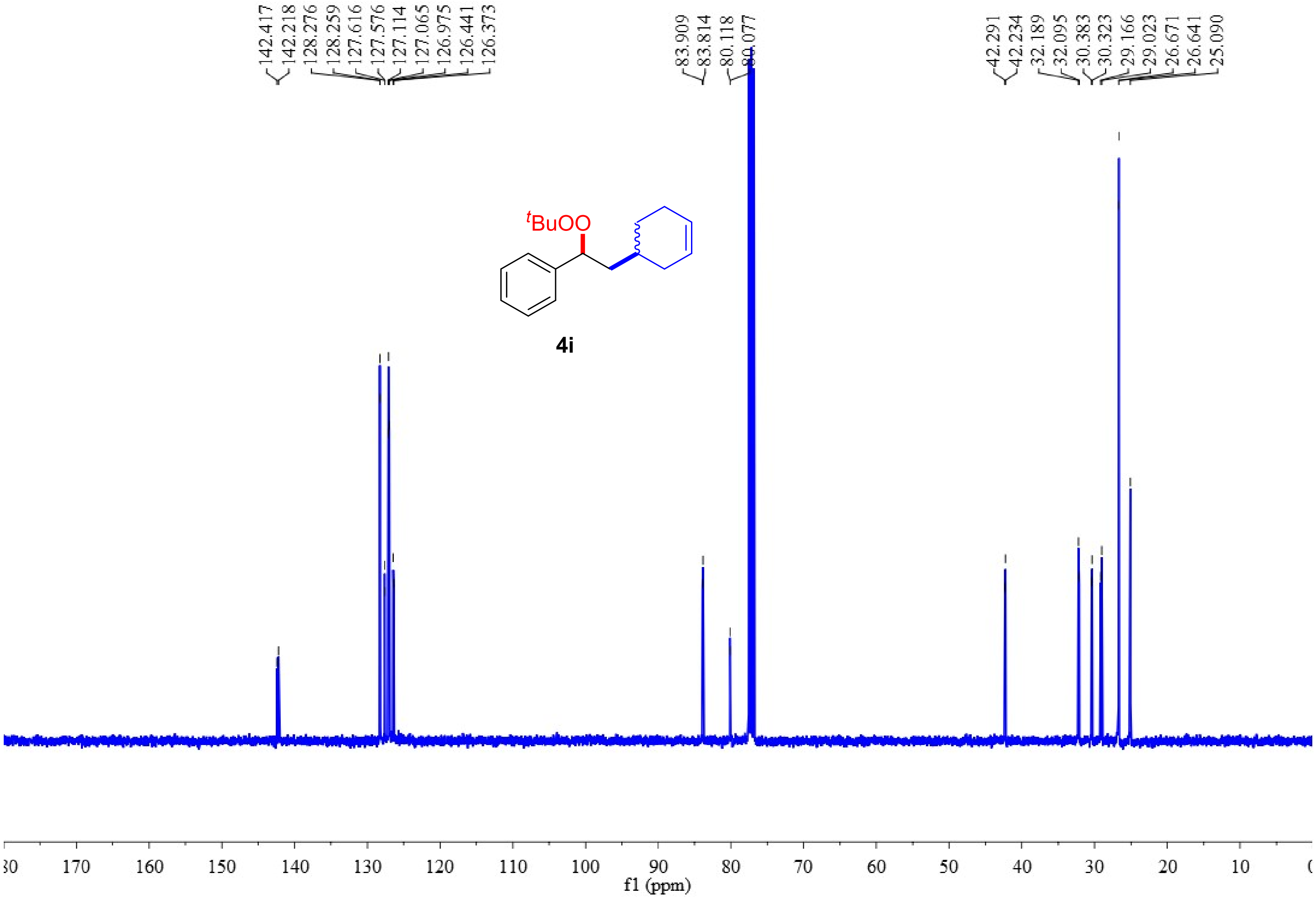
7.356
7.335
7.325
7.298
7.288
7.277
7.266
7.245
4.865
4.847
4.830
1.906
1.889
1.873
1.856
1.808
1.792
1.780
1.766
1.750
1.740
1.724
1.692
1.676
1.660
1.643
1.628
1.501
1.483
1.471
1.464
1.453
1.191
1.145
1.136
1.125
1.116
1.107
1.088
^{t}BuOO
5.02
1.00
1.01
3.04
2.45
2.57
8.97
2.04
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

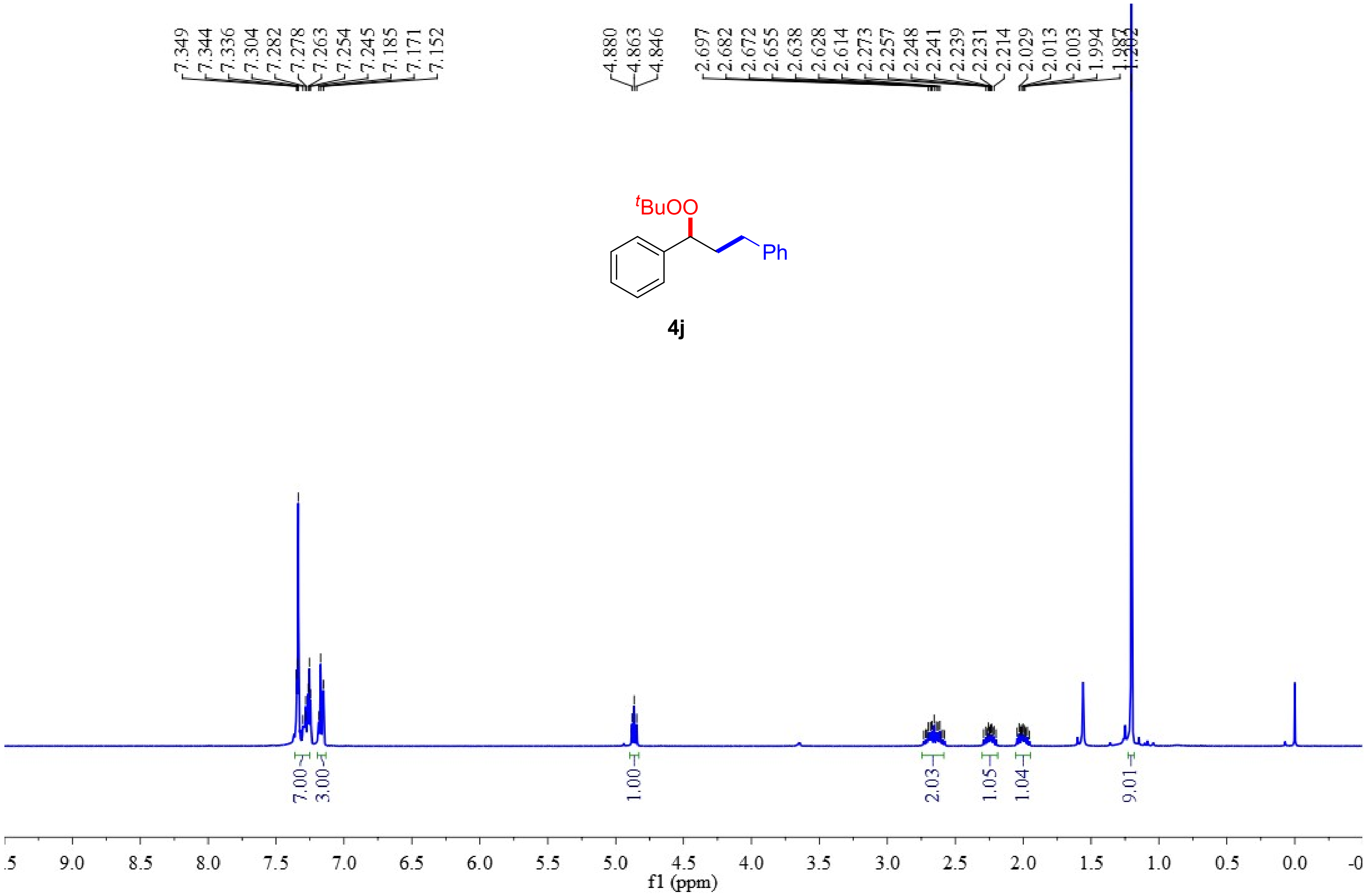
4h

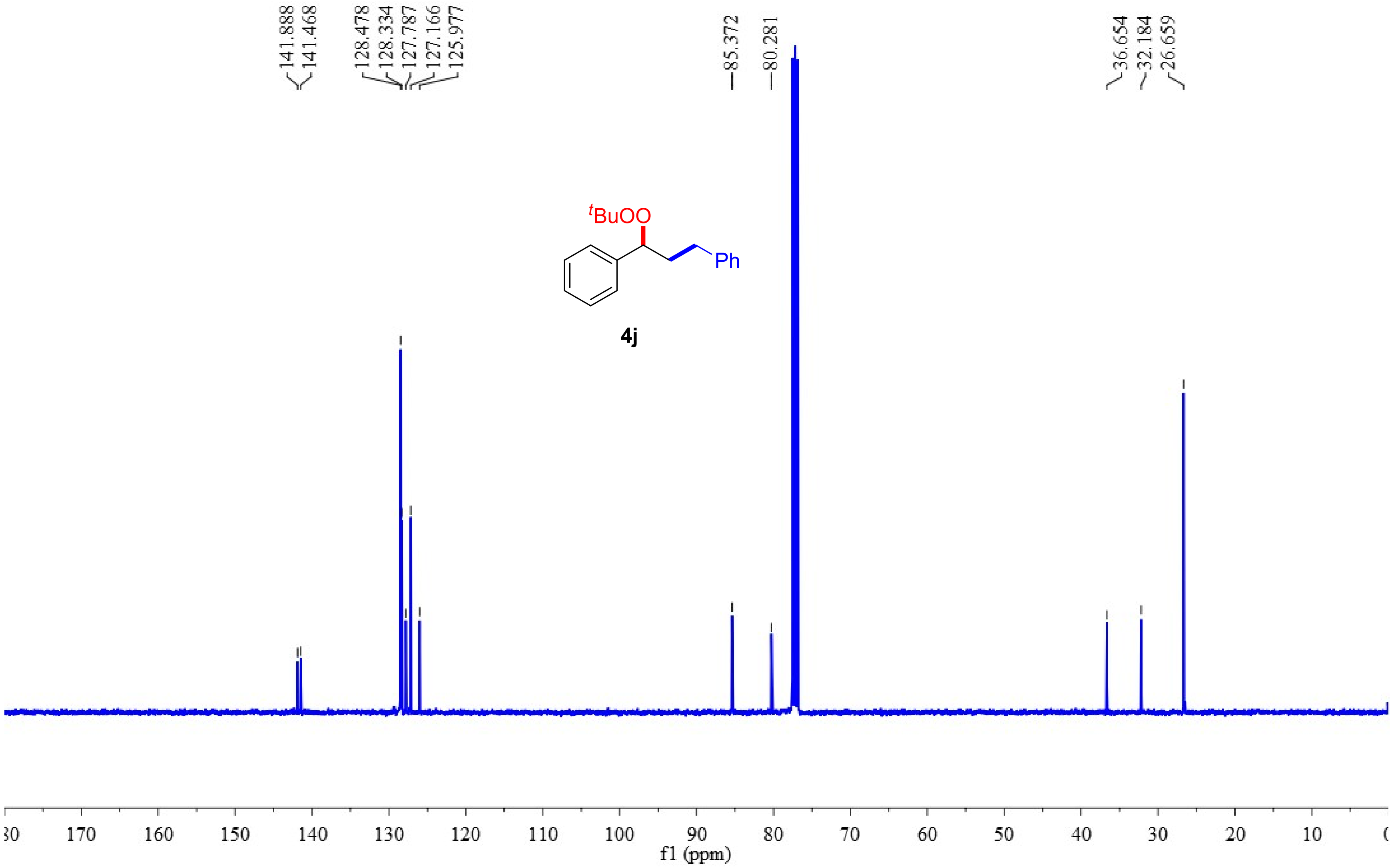

142.268
128.208
127.562
127.103
85.629
80.080
41.715
36.747
33.195
32.923
26.651
25.301
25.174
^{t}BuOO
4h
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

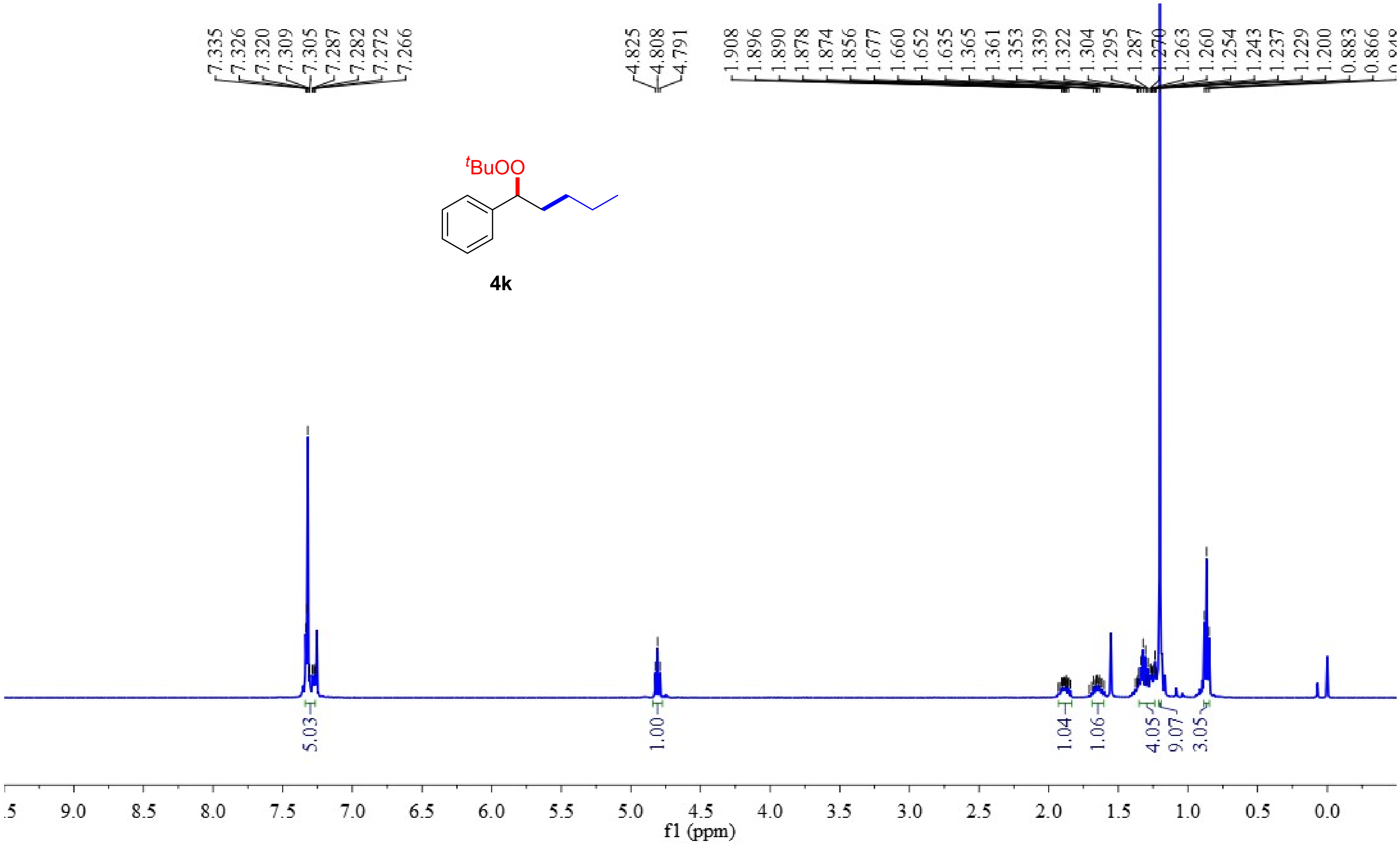

7.342
7.336
7.331
7.326
7.291
7.287
7.279
7.270
5.667
5.635
5.602
4.973
4.957
4.954
4.938
2.160
2.122
2.020
2.007
1.985
1.875
1.860
1.841
1.823
1.808
1.772
1.716
1.678
1.661
1.652
1.645
1.629
1.614
1.569
1.552
1.306
1.297
1.291
1.281
1.274
1.266
1.256
1.251
^{t}BuOO
4.95
2.01
1.00
0.99
1.98
4.37
0.59
1.05
9.04
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

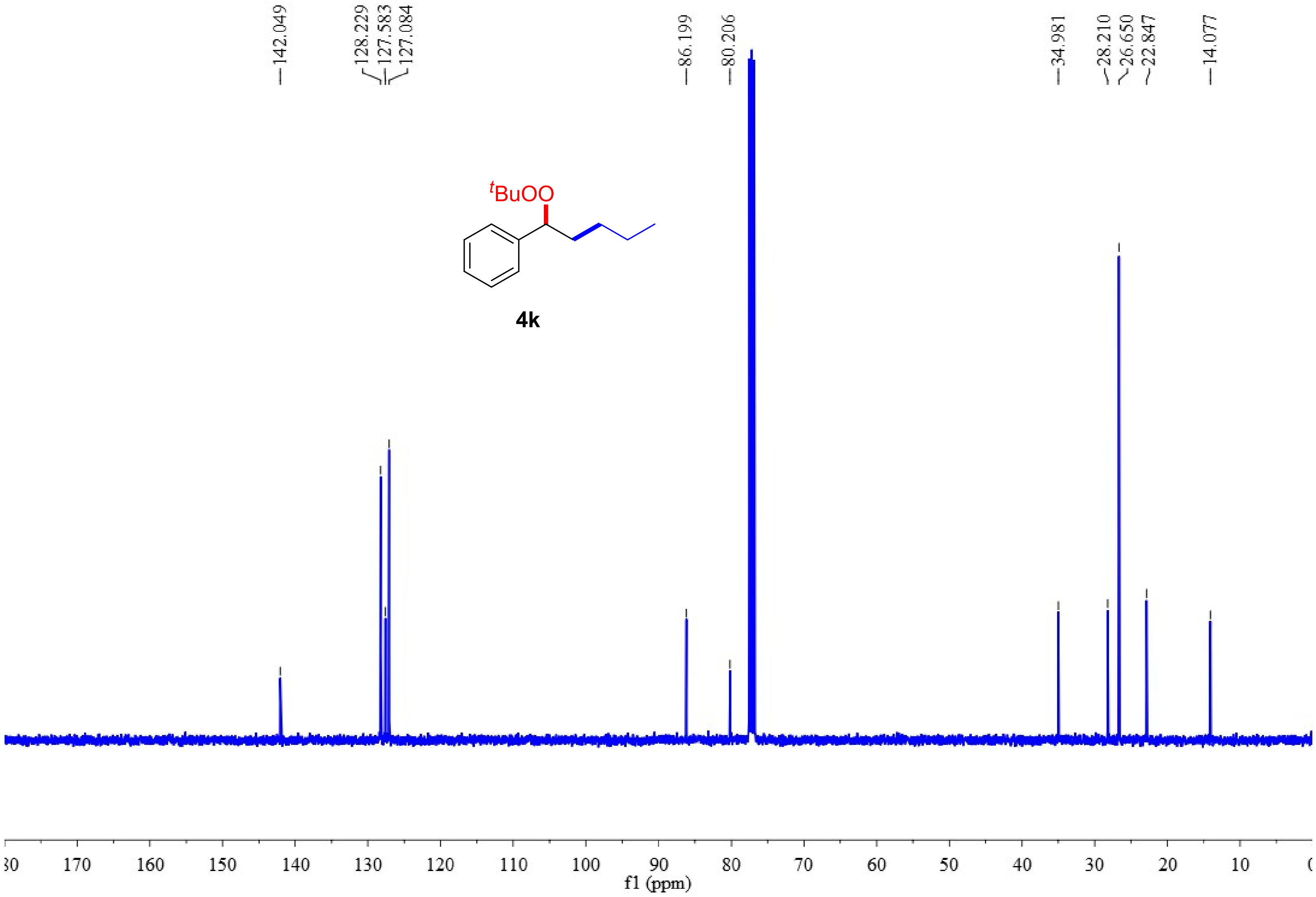
4i

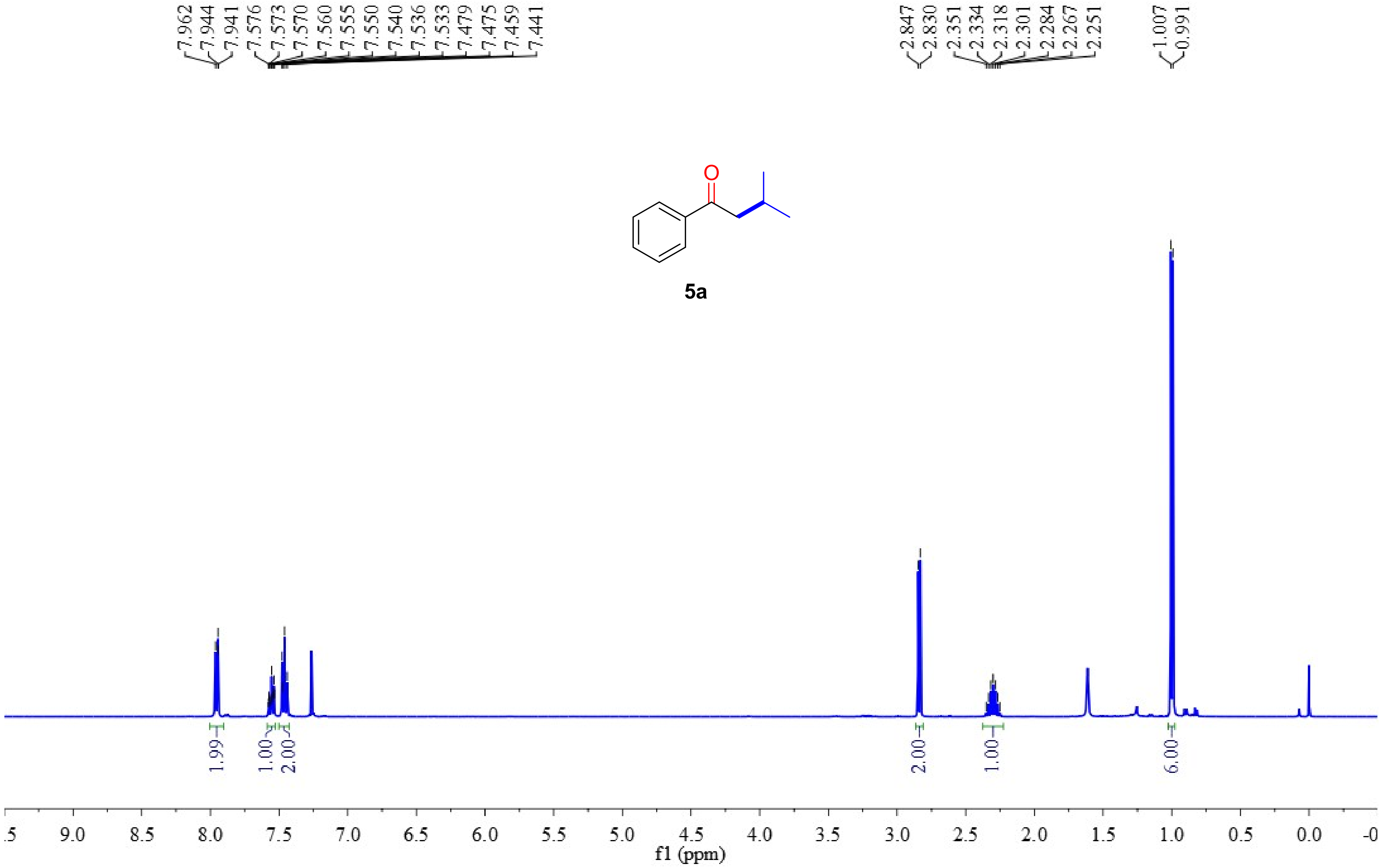

142.417
142.218
128.276
128.259
127.616
127.576
127.114
127.065
126.975
126.441
126.373
83.909
83.814
80.118
80.077
42.291
42.234
32.189
32.095
30.383
30.323
29.166
29.023
26.671
26.641
25.090
^{t}BuOO
4i
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.349
7.344
7.336
7.304
7.282
7.278
7.263
7.254
7.245
7.185
7.171
7.152
4.880
4.863
4.846
2.697
2.682
2.672
2.655
2.638
2.628
2.614
2.273
2.257
2.248
2.241
2.239
2.231
2.214
2.029
2.013
2.003
1.994
1.987
1.202
tBuOO
Ph
4j
7.00
3.00
1.00
2.03
1.05
1.04
9.01
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)


141.888
141.468
128.478
128.334
127.787
127.166
125.977
85.372
80.281
36.654
32.184
26.659
^{t}BuOO
Ph
4j
30
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.335
7.326
7.320
7.309
7.305
7.287
7.282
7.272
7.266
4.825
4.808
4.791
1.908
1.896
1.890
1.878
1.874
1.856
1.677
1.660
1.652
1.635
1.365
1.361
1.353
1.339
1.322
1.304
1.295
1.287
1.270
1.263
1.260
1.254
1.243
1.237
1.229
1.200
0.883
0.866
^{t}BuOO
4k
5.03
1.00
1.04
1.06
4.05
9.07
3.05
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


142.049
128.229
127.583
127.084
86.199
80.206
34.981
28.210
26.650
22.847
14.077
^{t}BuOO
4k
30
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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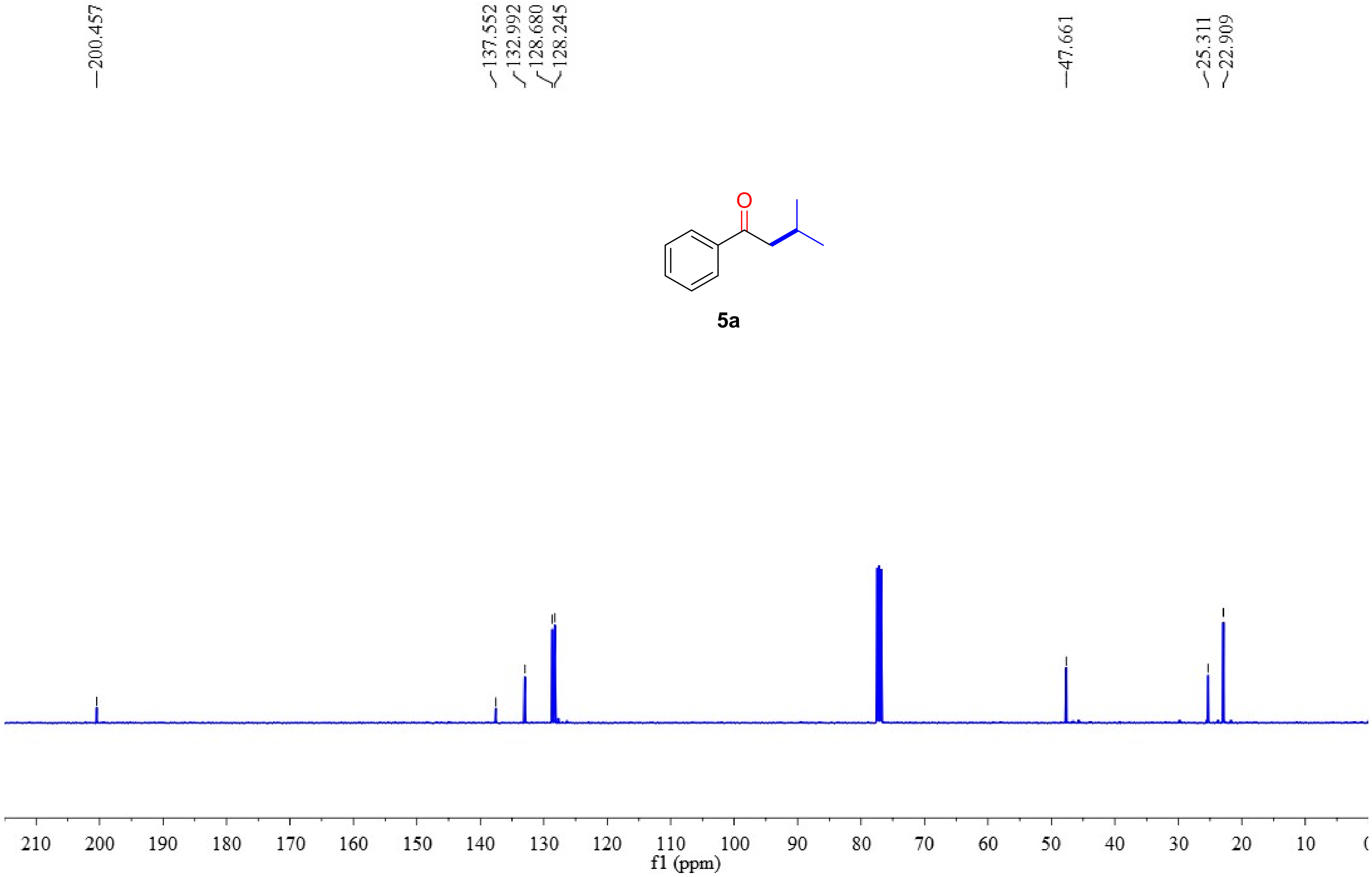
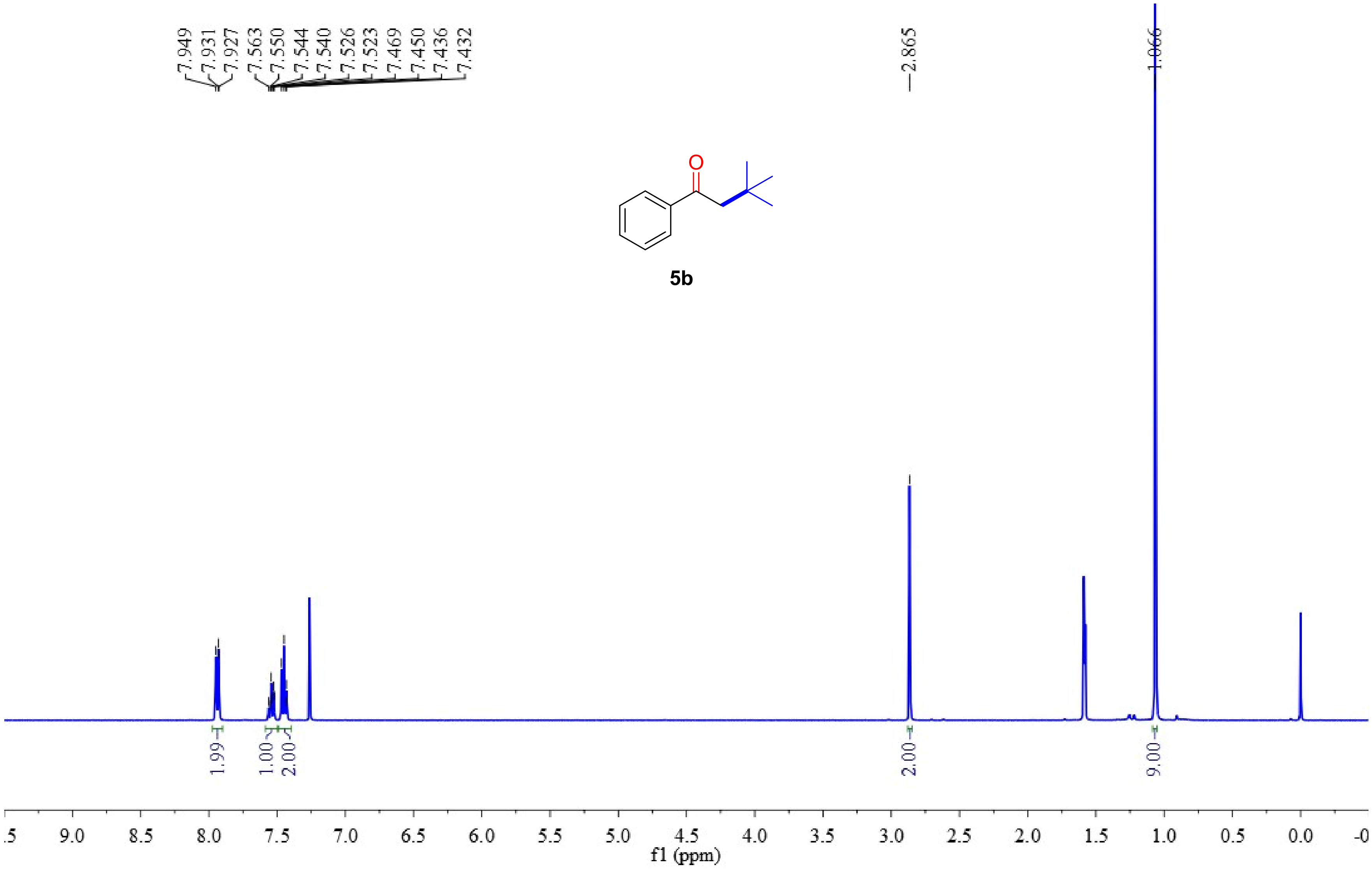
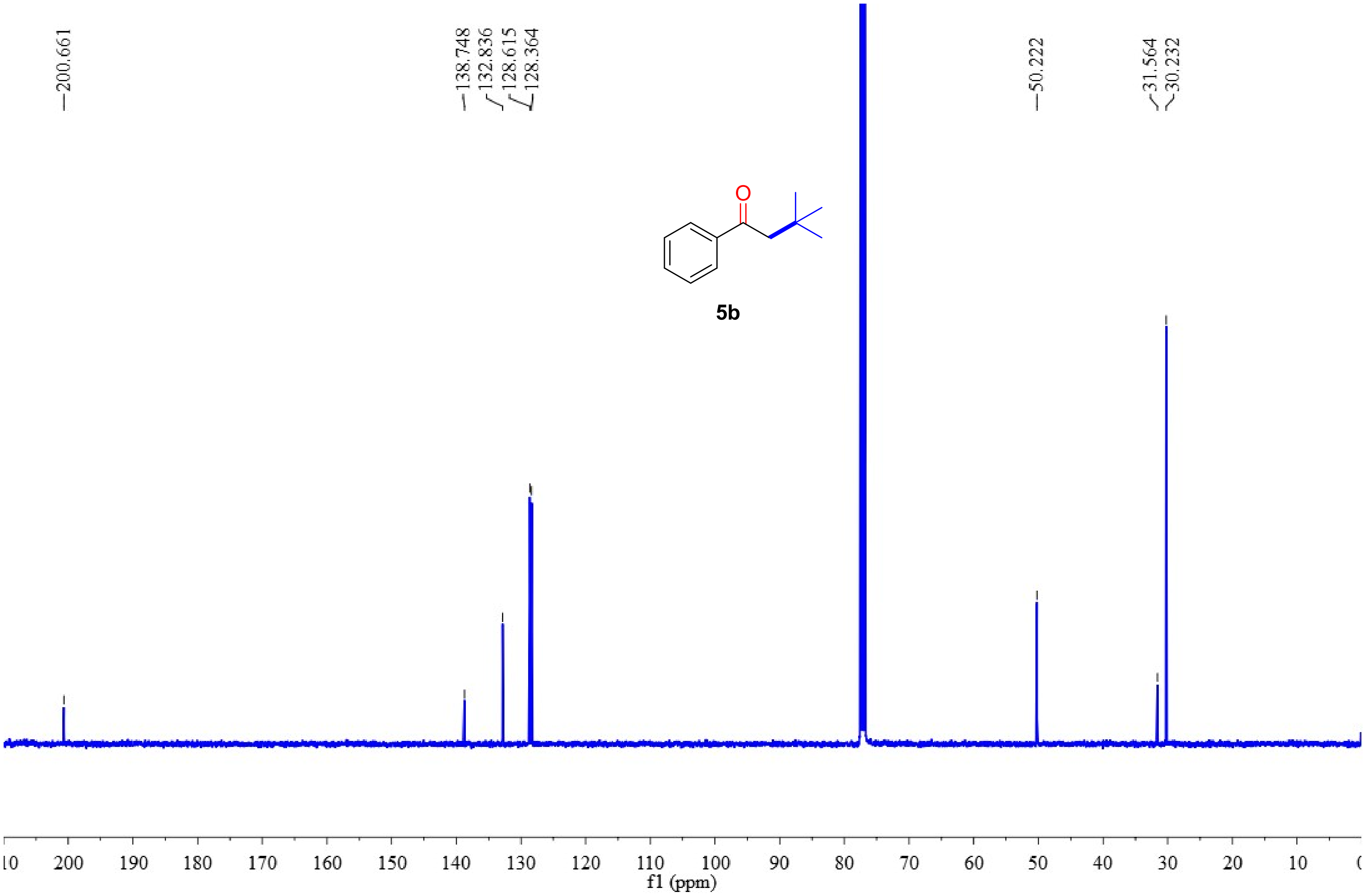
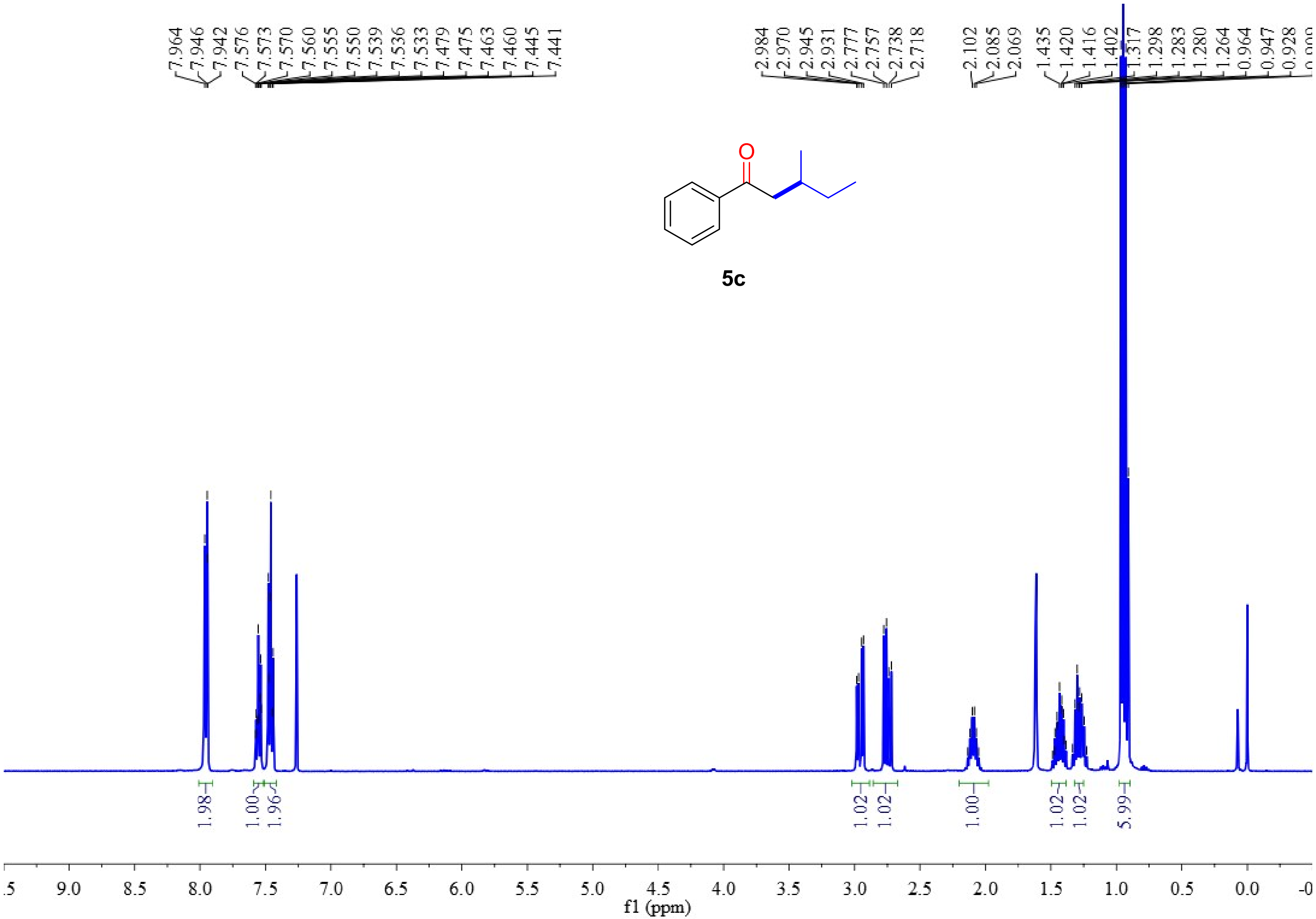
7.941
7.576
7.573
7.570
7.560
7.555
7.550
7.540
7.536
7.533
7.479
7.475
7.459
7.441
2.847
2.830
2.351
2.334
2.318
2.301
2.284
2.267
2.251
1.007
0.991
O
5a
1.99
1.00
2.00
2.00
1.00
6.00
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)


200.457
137.552
132.992
128.680
128.245
47.661
25.311
22.909
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

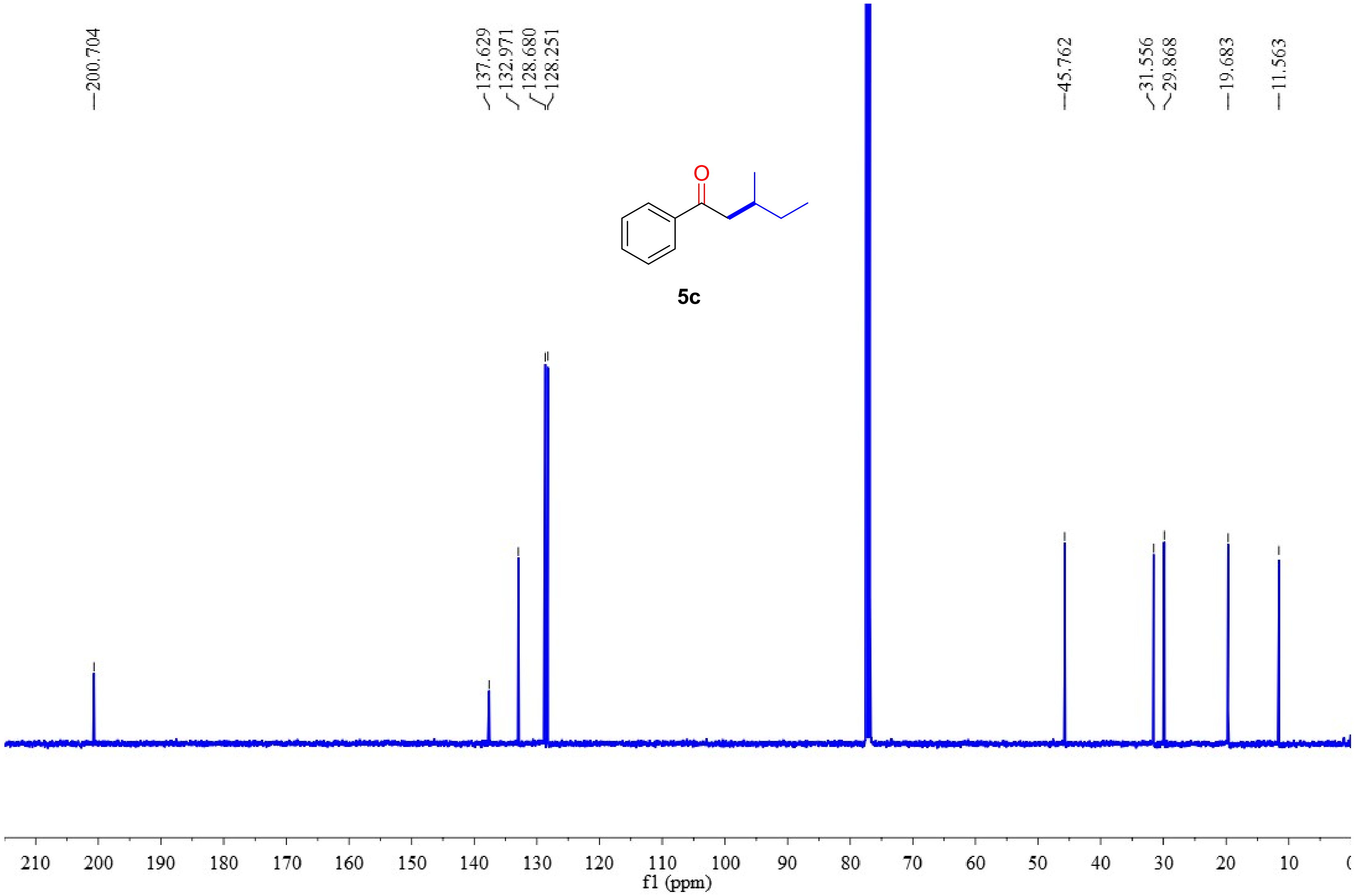
5a

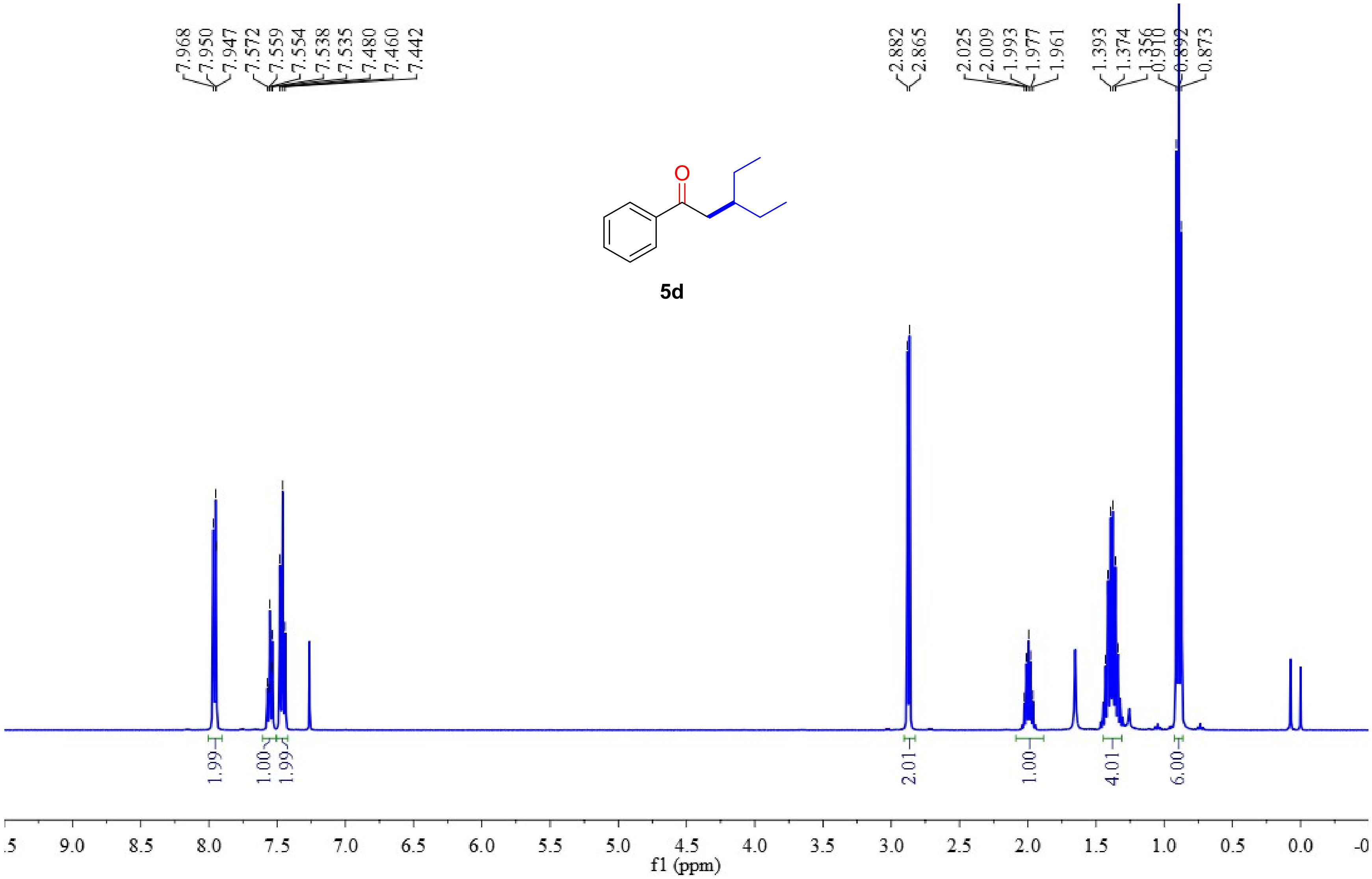

7.949
7.931
7.927
7.563
7.550
7.544
7.540
7.526
7.523
7.469
7.450
7.436
7.432
2.865
1.066
O
5b
1.99
1.00
2.00
2.00
9.00
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)


200.661
138.748
132.836
128.615
128.364
50.222
31.564
30.232
O
5b
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.964
7.946
7.942
7.576
7.573
7.570
7.560
7.555
7.550
7.539
7.536
7.533
7.479
7.475
7.463
7.460
7.445
7.441
2.984
2.970
2.945
2.931
2.777
2.757
2.738
2.718
2.102
2.085
2.069
1.435
1.420
1.416
1.402
1.317
1.298
1.283
1.280
1.264
0.964
0.947
0.928
O
1.98
1.00
1.96
1.02
1.02
1.00
1.02
1.02
5.99
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

5c


200.704
137.629
132.971
128.680
128.251
45.762
31.556
29.868
19.683
11.563
O
5c
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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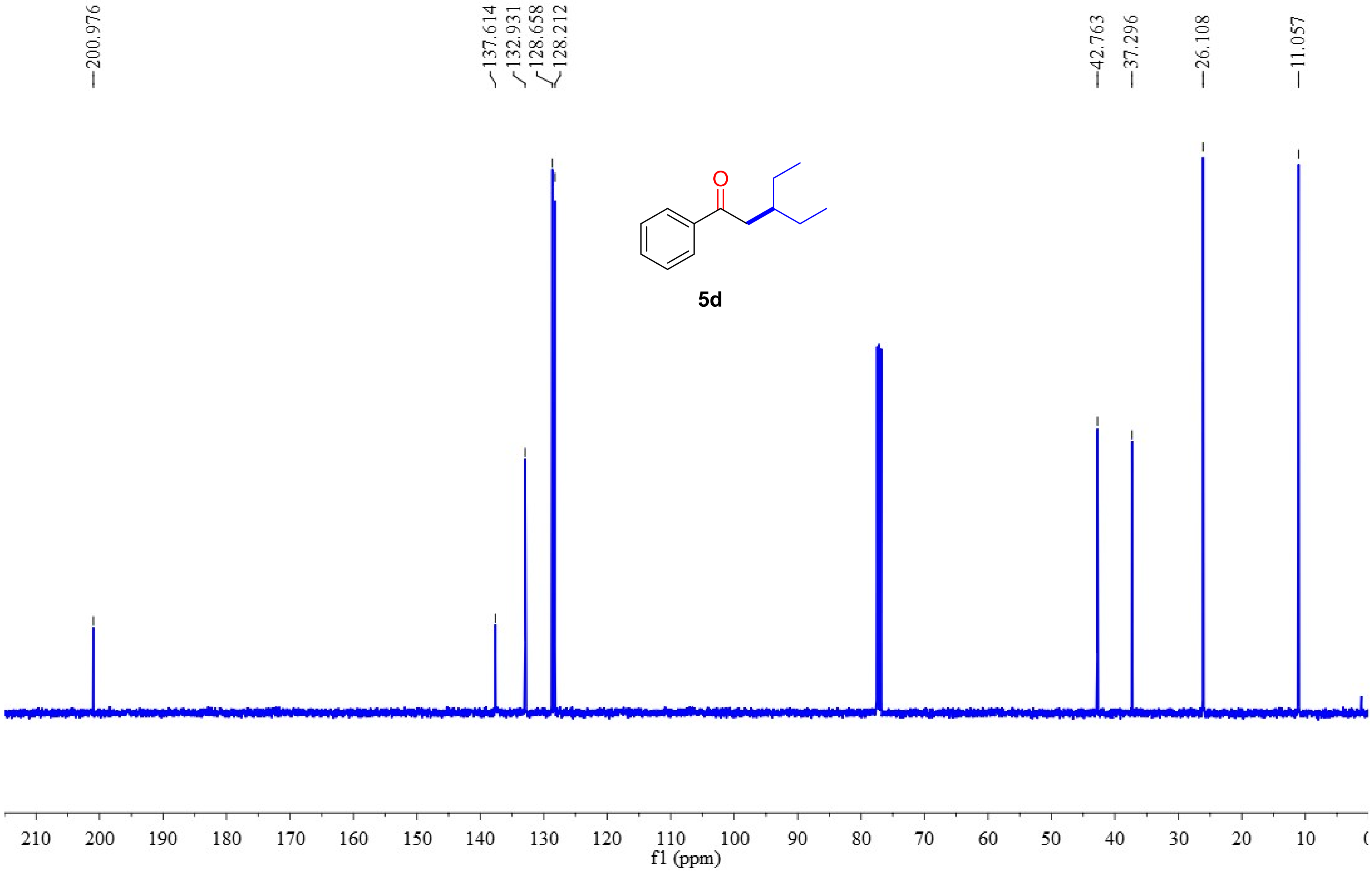
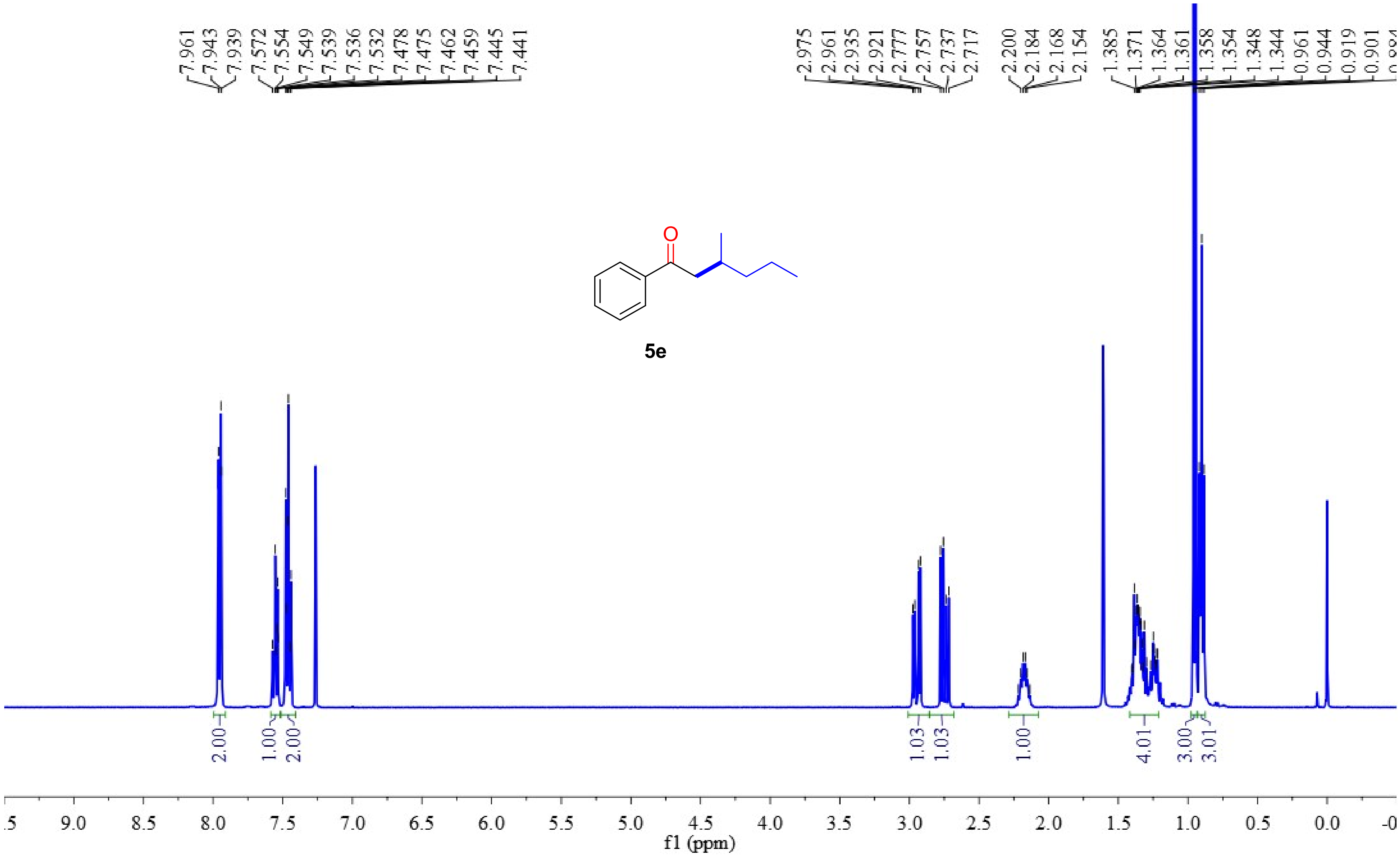
7.950
7.947
7.572
7.559
7.554
7.538
7.535
7.480
7.460
7.442
2.882
2.865
2.025
2.009
1.993
1.977
1.961
1.393
1.374
1.356
0.910
0.892
0.873
O
5d
1.99
1.00
1.99
2.01
1.00
4.01
6.00
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)


200.976
137.614
132.931
128.658
128.212
42.763
37.296
26.108
11.057
O
5d
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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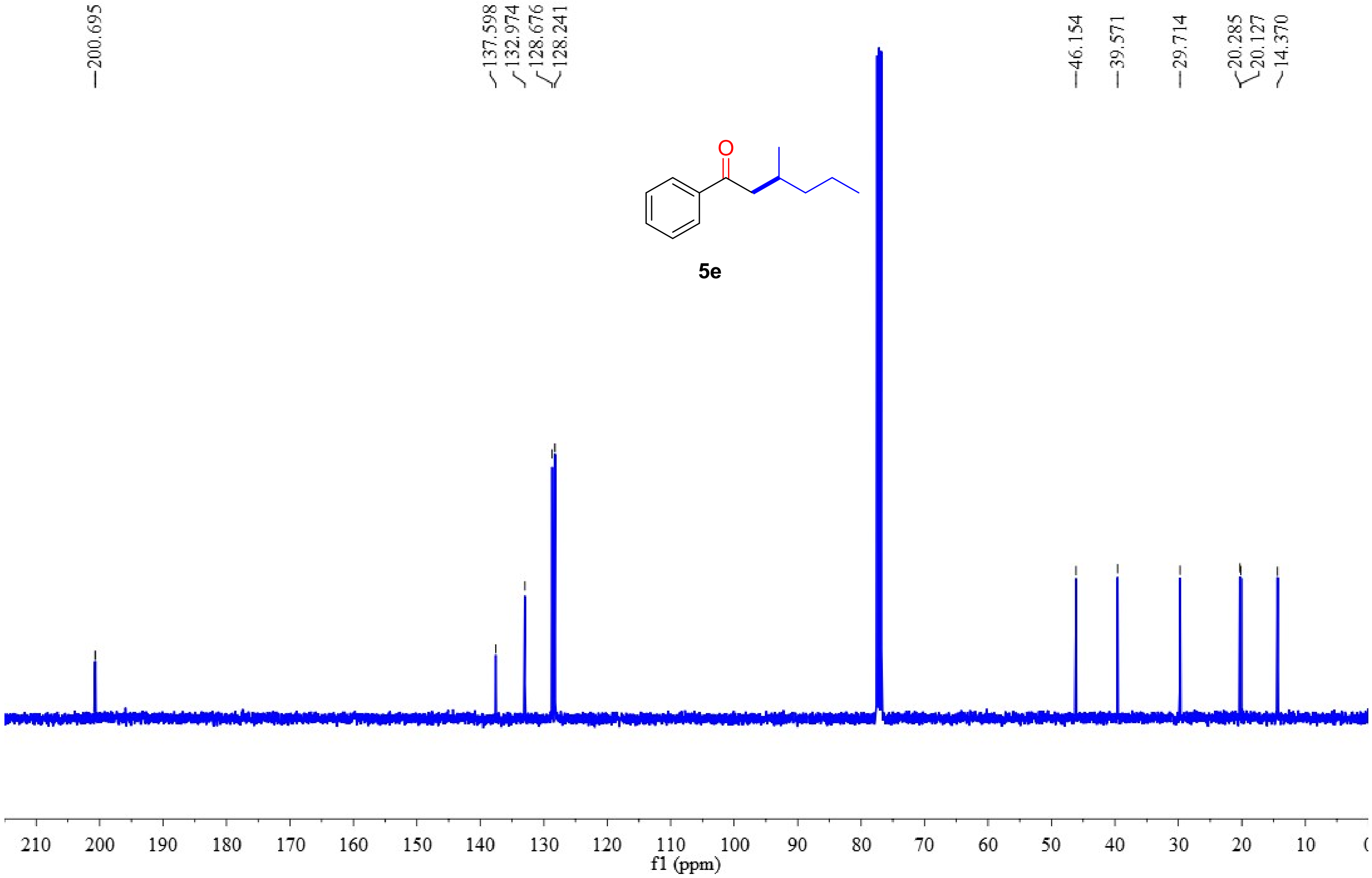
7.943
7.939
7.572
7.554
7.549
7.539
7.536
7.532
7.478
7.475
7.462
7.459
7.445
7.441
2.975
2.961
2.935
2.921
2.777
2.757
2.737
2.717
2.200
2.184
2.168
2.154
1.385
1.371
1.364
1.361
1.358
1.354
1.348
1.344
0.961
0.944
0.919
0.901
O
5e
2.00
1.00
2.00
1.03
1.03
1.00
4.01
3.00
3.01
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)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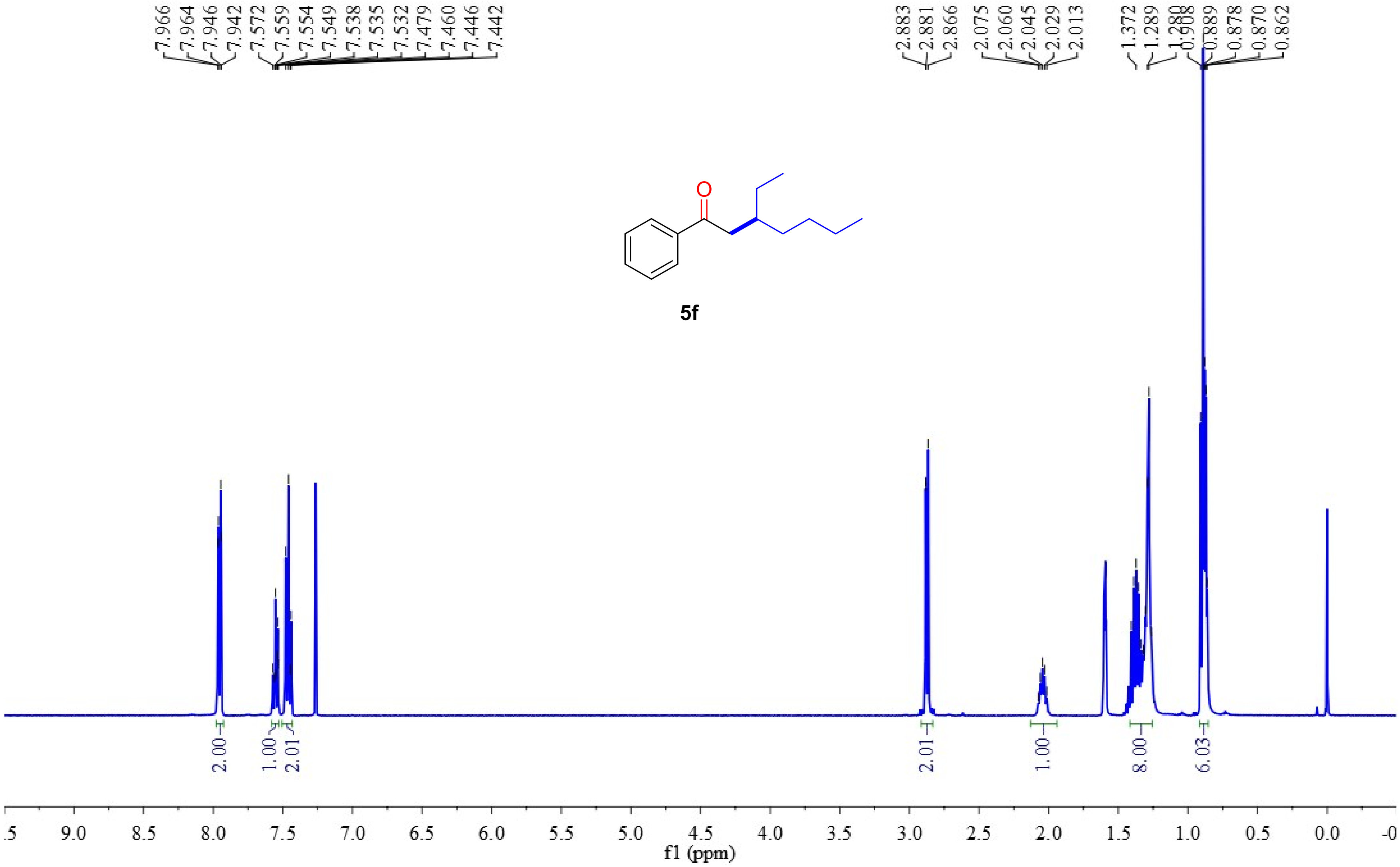
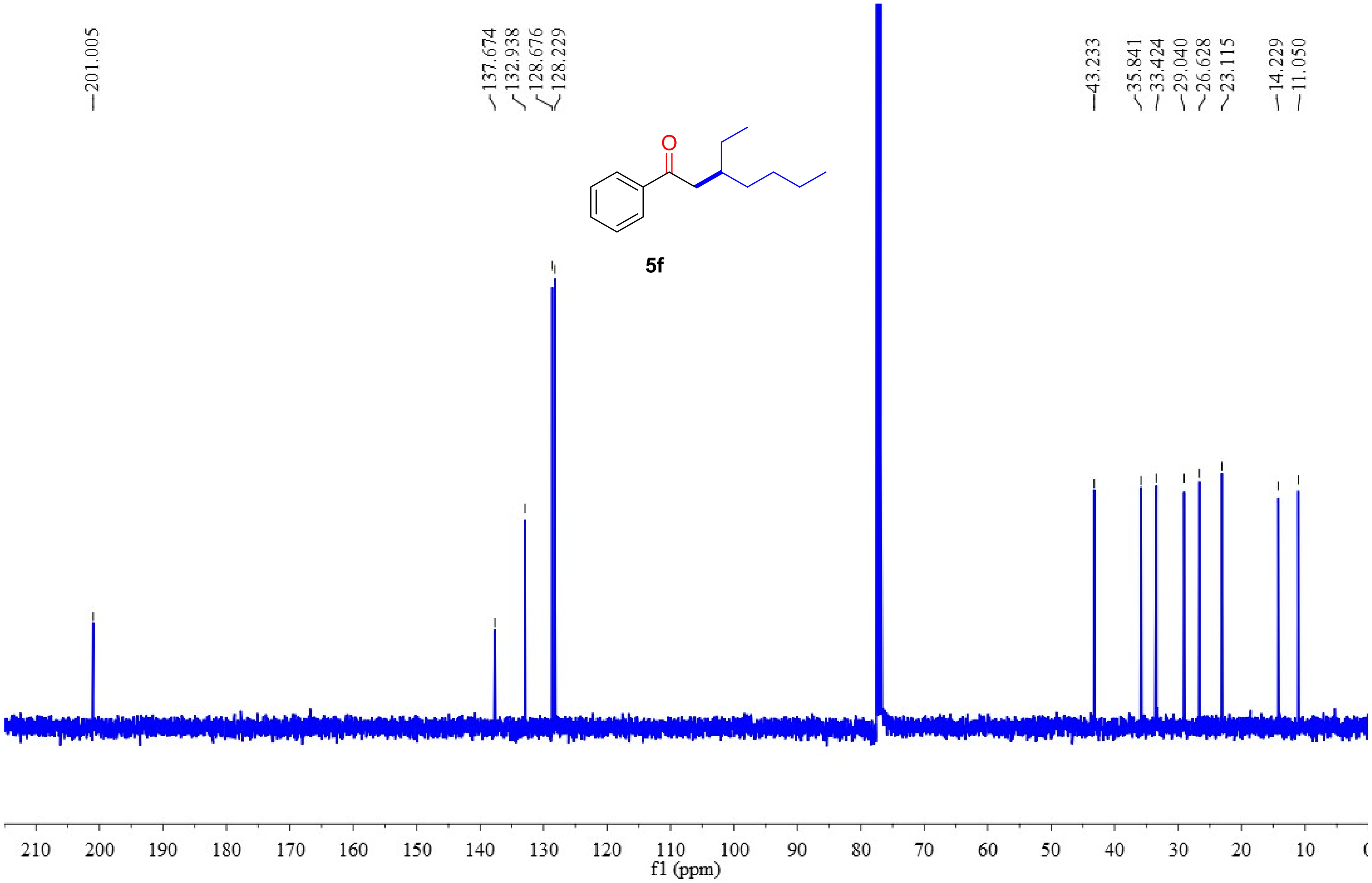
137.598
132.974
128.676
128.241
46.154
39.571
29.714
20.285
20.127
14.370
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

5e


7.966
7.964
7.946
7.942
7.572
7.559
7.554
7.549
7.538
7.535
7.532
7.479
7.460
7.446
7.442
2.883
2.881
2.866
2.075
2.060
2.045
2.029
2.013
1.372
1.289
1.280
0.908
0.889
0.878
0.870
0.862
O
5f
2.00
1.00
2.01
2.01
1.00
8.00
6.03
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)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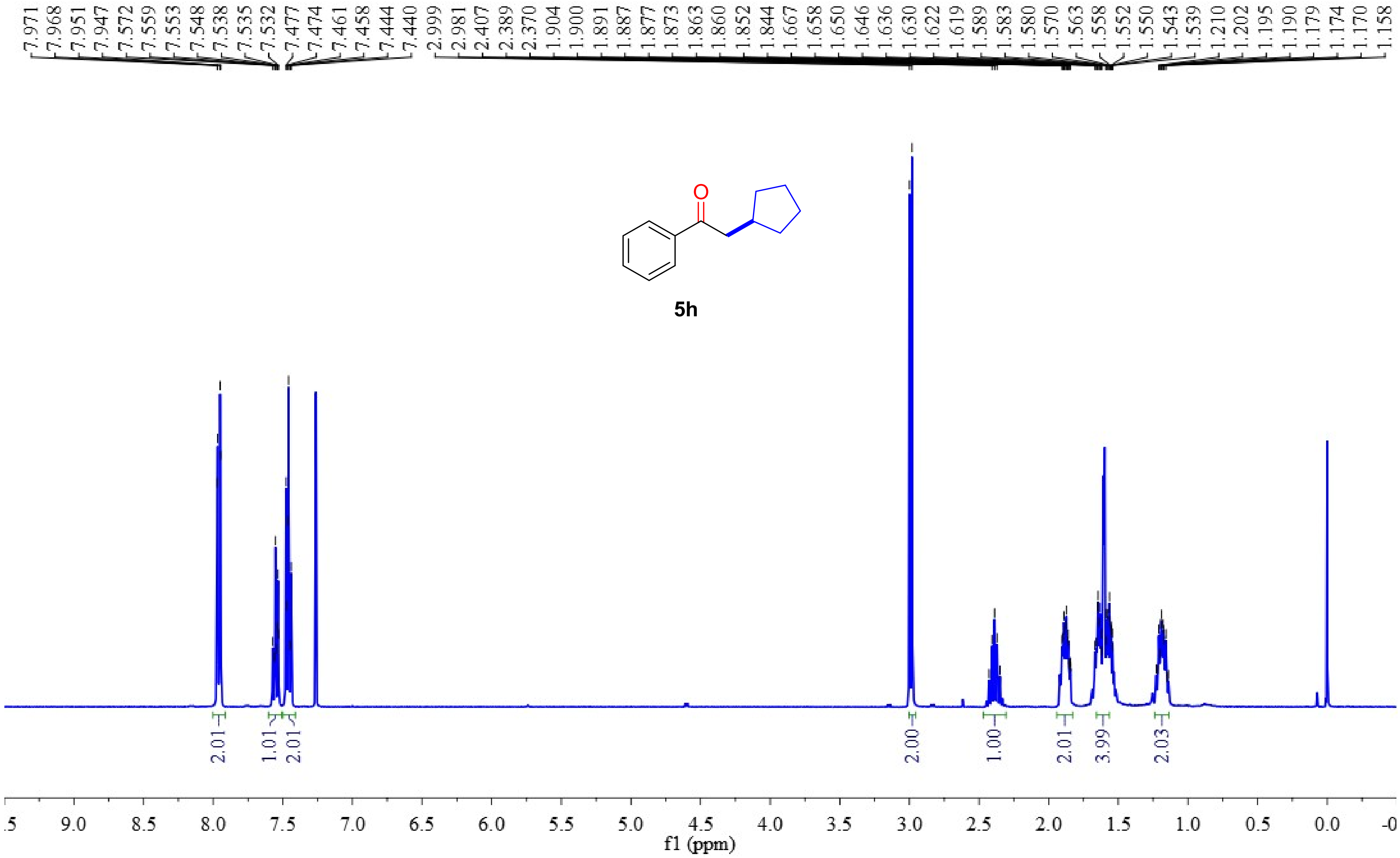
137.674
132.938
128.676
128.229
43.233
35.841
33.424
29.040
26.628
23.115
14.229
11.050
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

5f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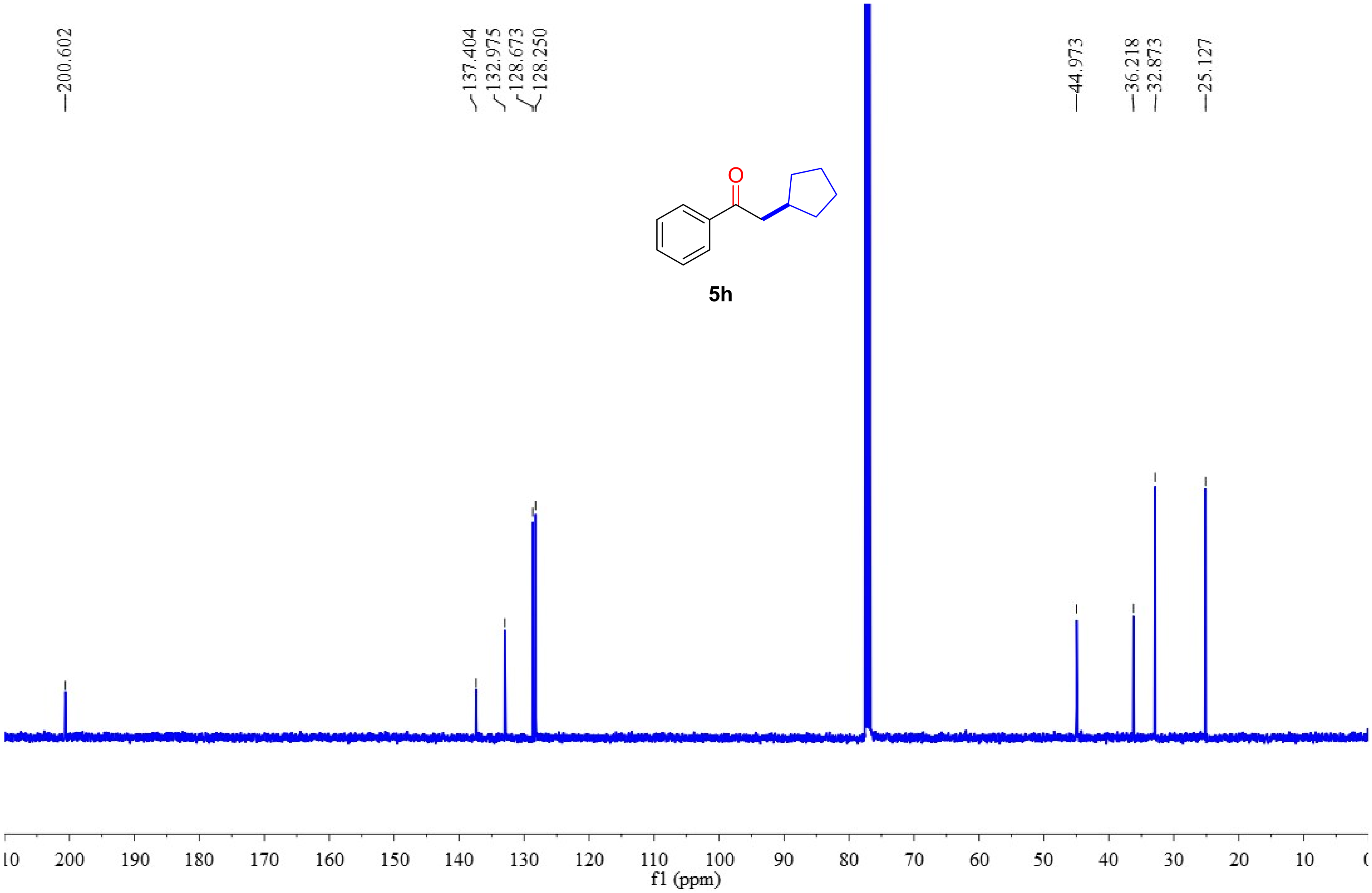
7.968
7.951
7.947
7.572
7.559
7.553
7.548
7.538
7.535
7.532
7.477
7.474
7.461
7.458
7.444
7.440
2.999
2.981
2.407
2.389
2.370
1.904
1.900
1.891
1.887
1.877
1.873
1.863
1.860
1.852
1.844
1.667
1.658
1.650
1.646
1.636
1.630
1.622
1.619
1.589
1.583
1.580
1.570
1.563
1.558
1.552
1.550
1.543
1.539
1.210
1.202
1.195
1.190
1.179
1.174
1.170
1.158
O
5h
2.01
1.01
2.01
2.00
1.00
2.01
3.99
2.03
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)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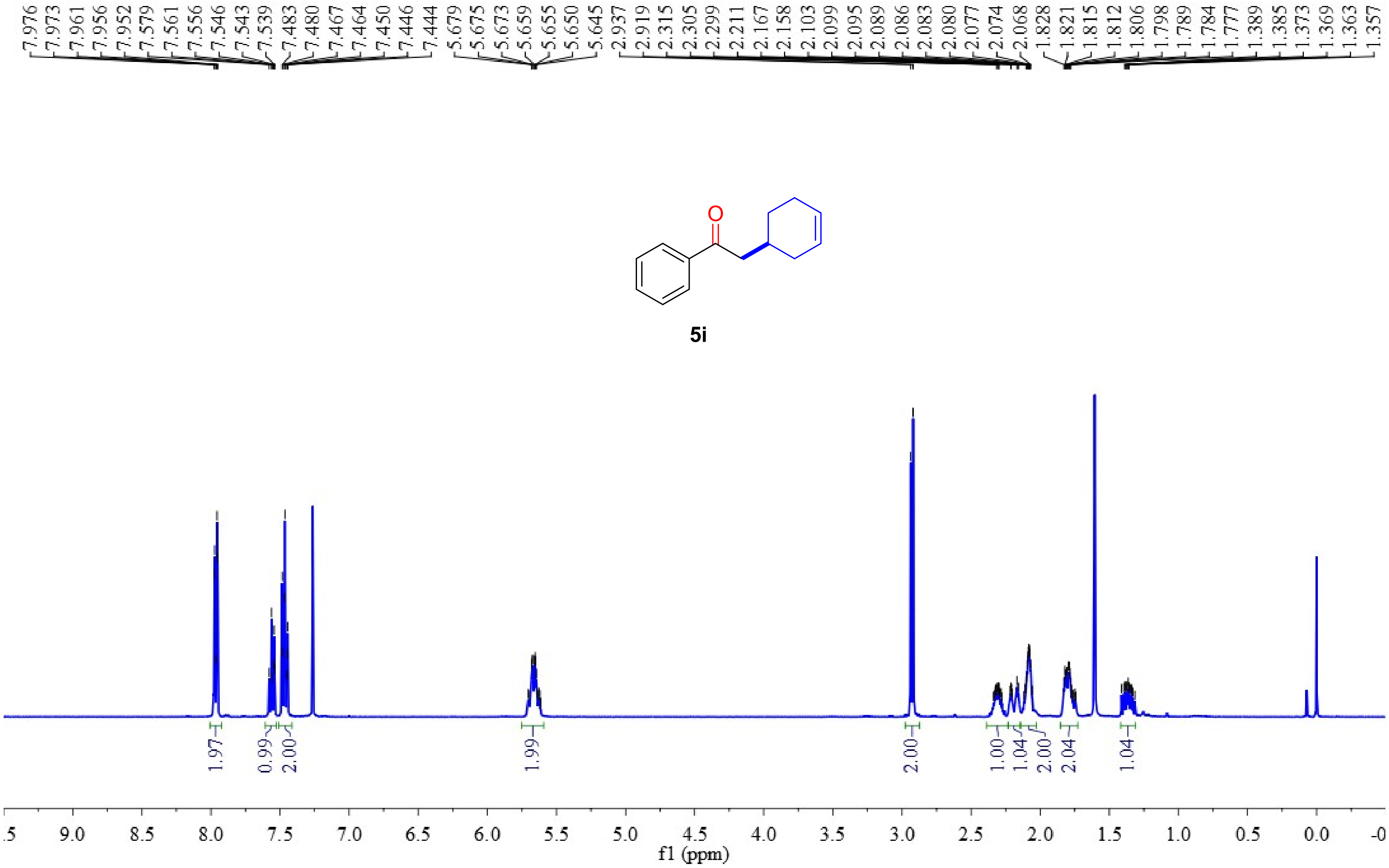
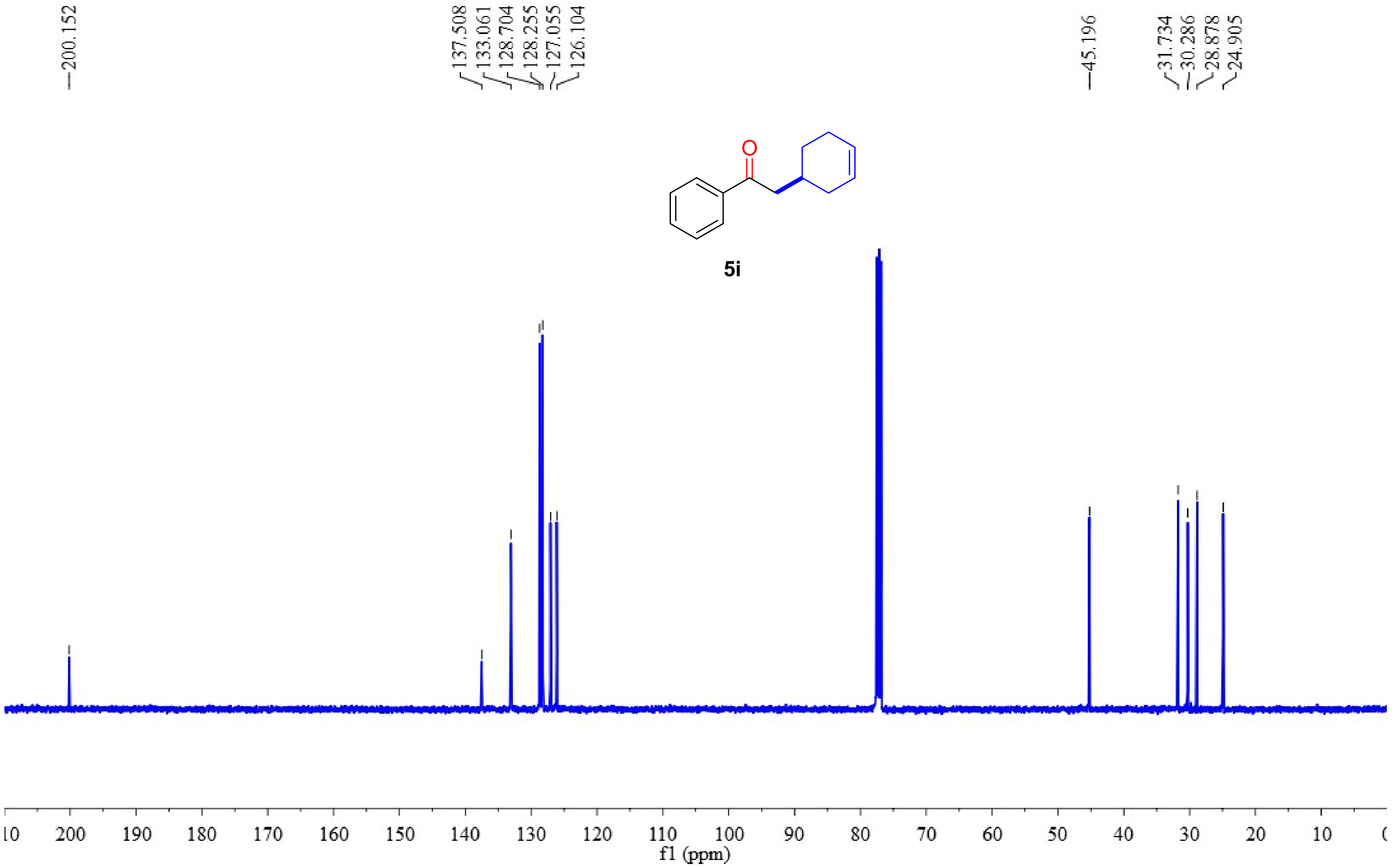
137.404
132.975
128.673
128.250
44.973
36.218
32.873
25.127
O
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

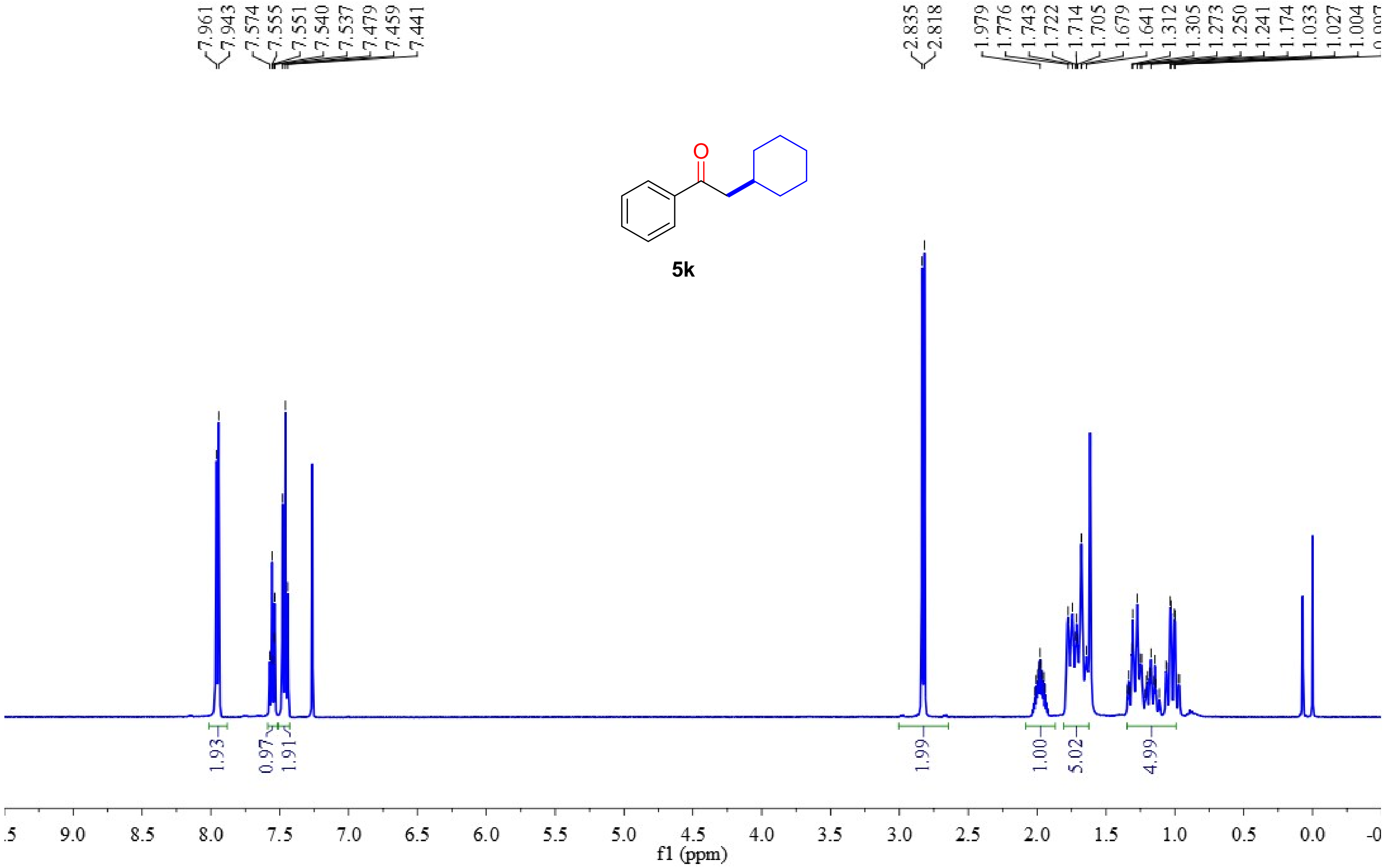
5h


7.976
7.973
7.961
7.956
7.952
7.579
7.561
7.556
7.546
7.543
7.539
7.483
7.480
7.467
7.464
7.450
7.446
7.444
5.679
5.675
5.673
5.659
5.655
5.650
5.645
2.937
2.919
2.315
2.305
2.299
2.211
2.167
2.158
2.103
2.099
2.095
2.089
2.086
2.083
2.080
2.077
2.074
2.068
1.828
1.821
1.815
1.812
1.806
1.798
1.789
1.784
1.777
1.389
1.385
1.373
1.369
1.363
1.357
O
5i
1.97
0.99
2.00
1.99
2.00
1.00
1.04
2.00
2.04
1.04
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)



5i

200.152
137.508
133.061
128.704
128.255
127.055
126.104
45.196
31.734
30.286
28.878
24.905
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)
O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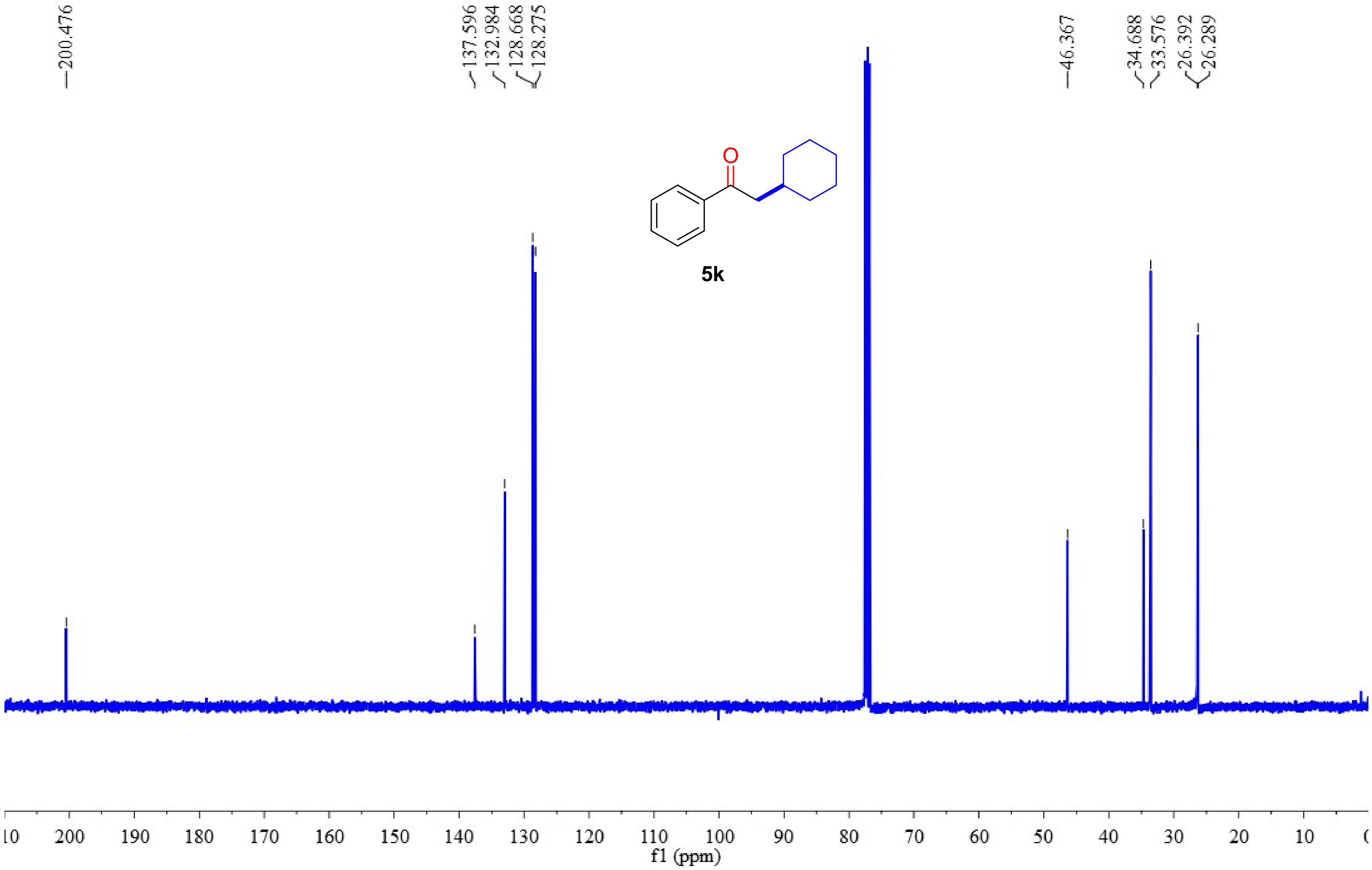
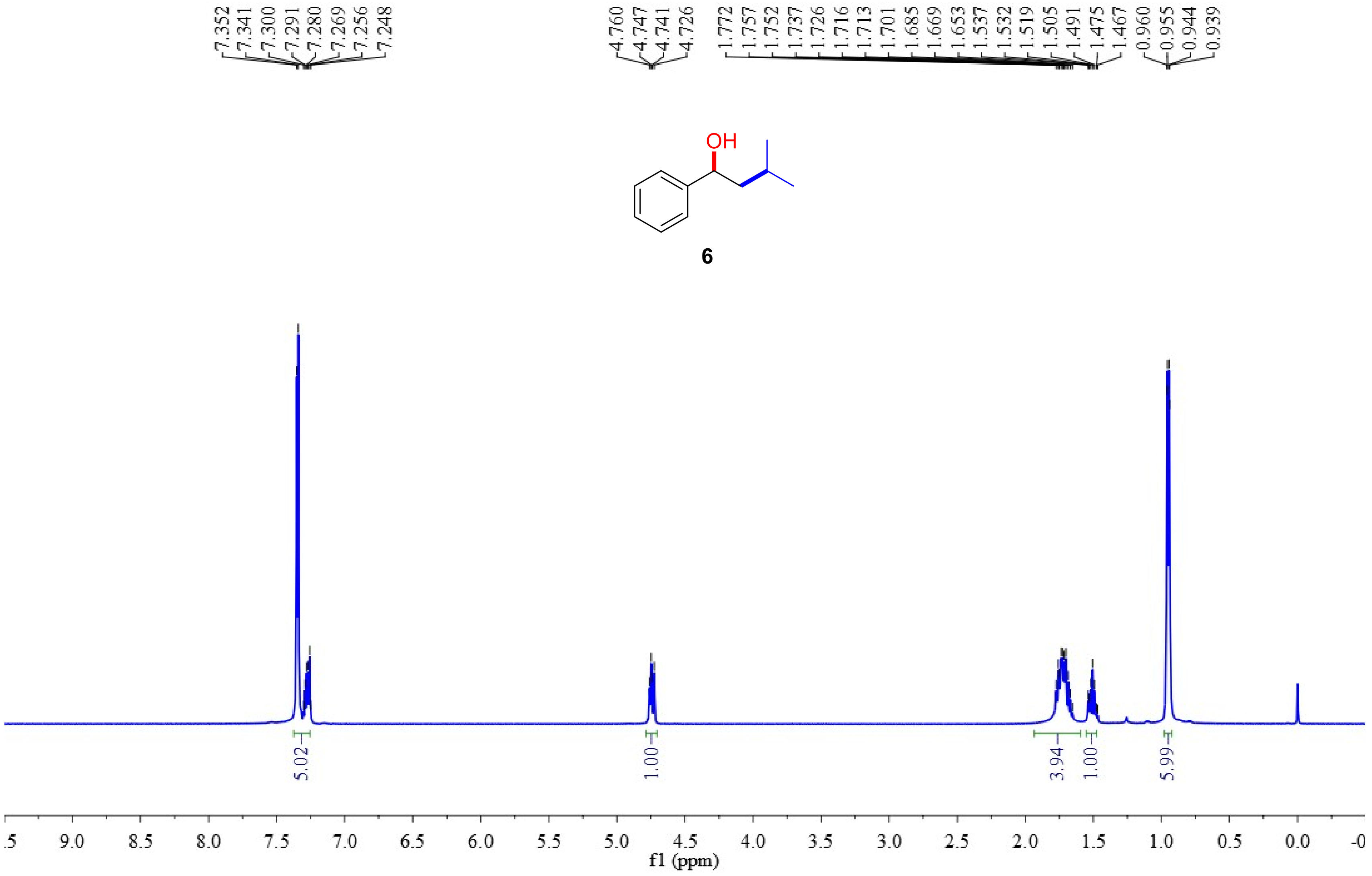
7.943
7.574
7.555
7.551
7.540
7.537
7.479
7.459
7.441
2.835
2.818
1.979
1.776
1.743
1.722
1.714
1.705
1.679
1.641
1.312
1.305
1.273
1.250
1.241
1.174
1.033
1.027
1.004
O
5k
1.93
0.97
1.91
1.99
1.00
5.02
4.99
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)


200.476
137.596
132.984
128.668
128.275
46.367
34.688
33.576
26.392
26.289
O
5k
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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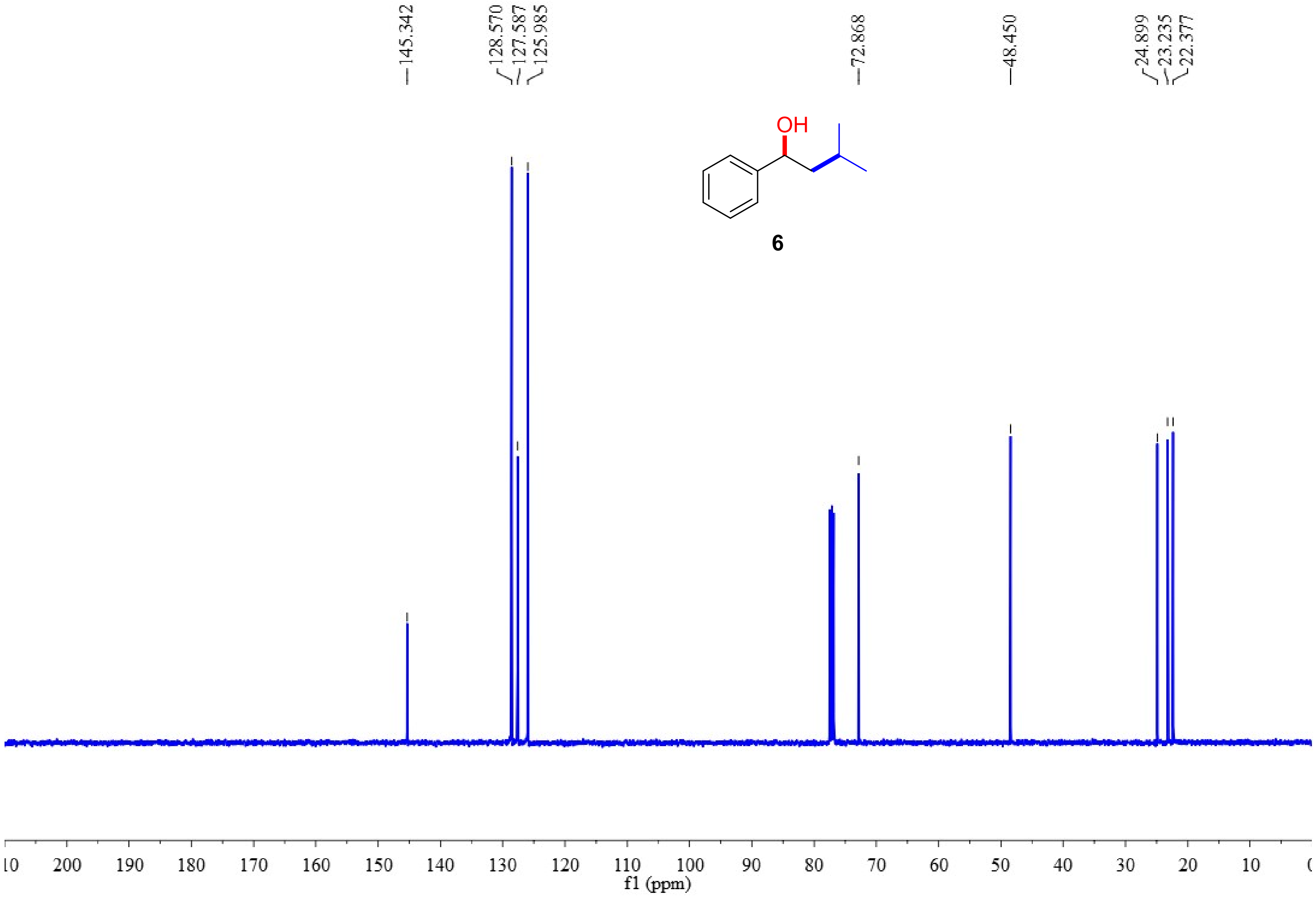
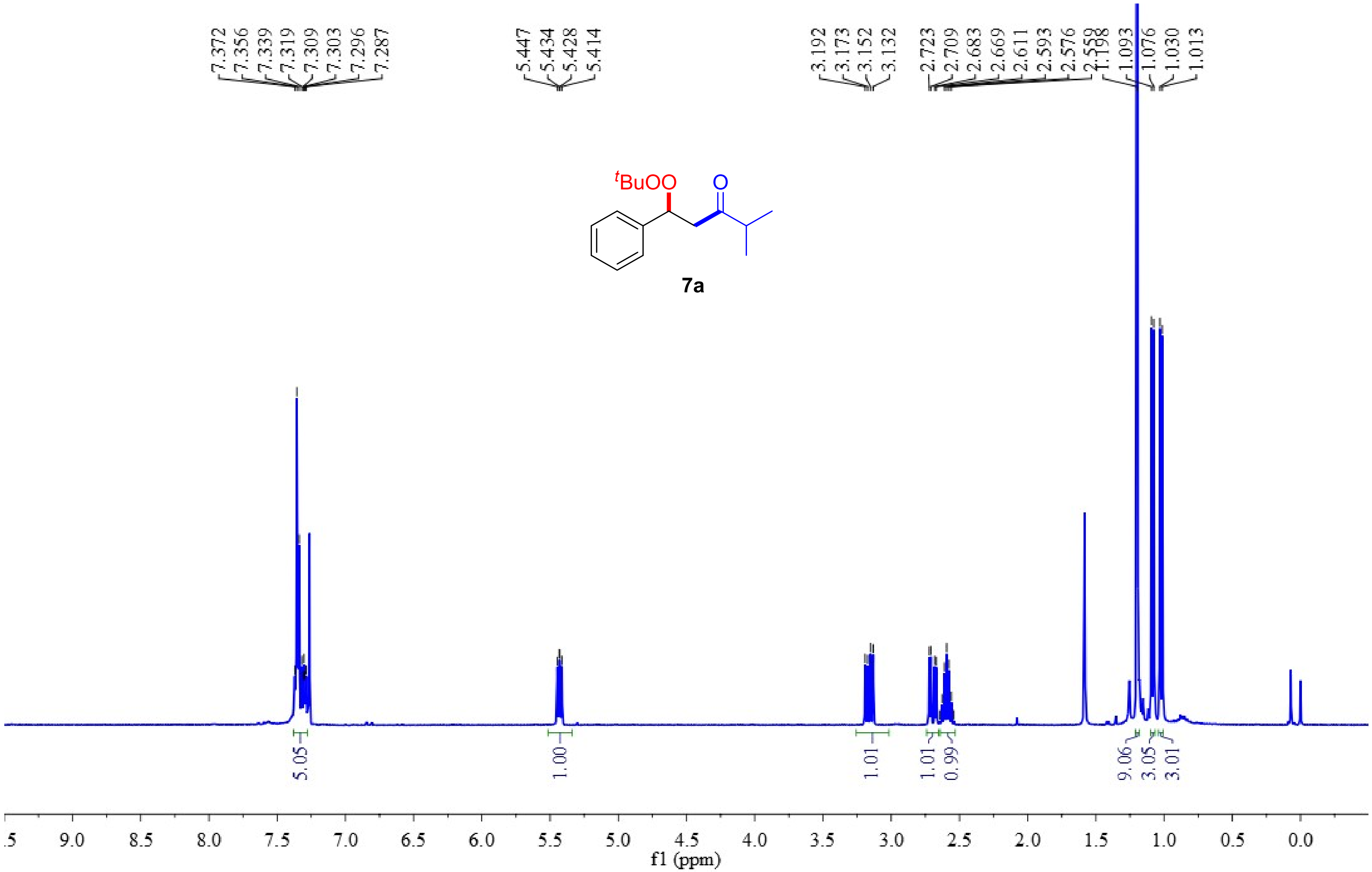
7.280
7.269
7.256
7.248
4.760
4.747
4.741
4.726
1.772
1.757
1.752
1.737
1.726
1.716
1.713
1.701
1.685
1.669
1.653
1.537
1.532
1.519
1.505
1.491
1.475
1.467
0.960
0.955
0.944
0.939
OH
6
5.02
1.00
3.94
1.00
5.99
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)


145.342
128.570
127.587
125.985
72.868
48.450
24.899
23.235
22.377
OH
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

6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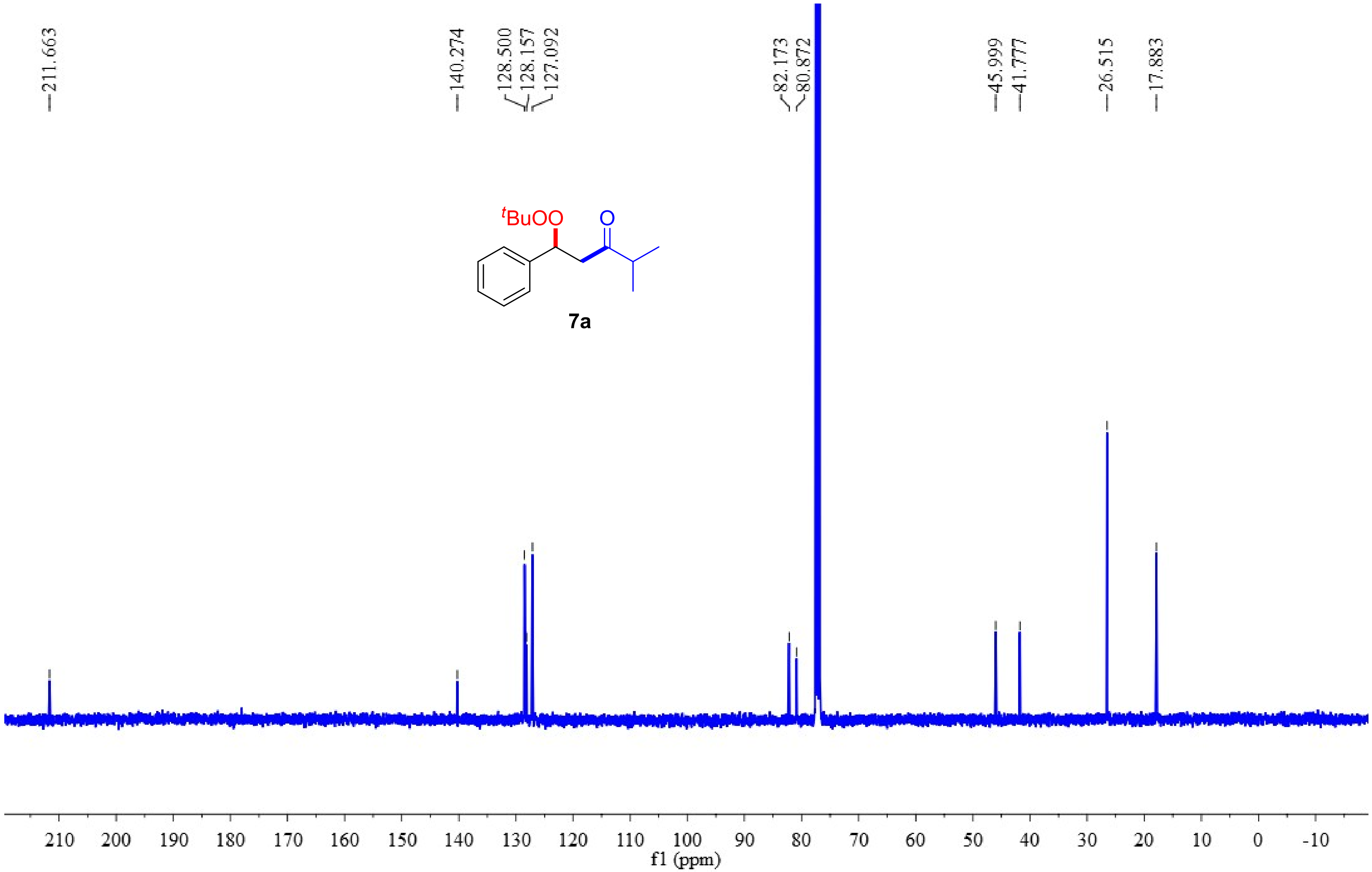
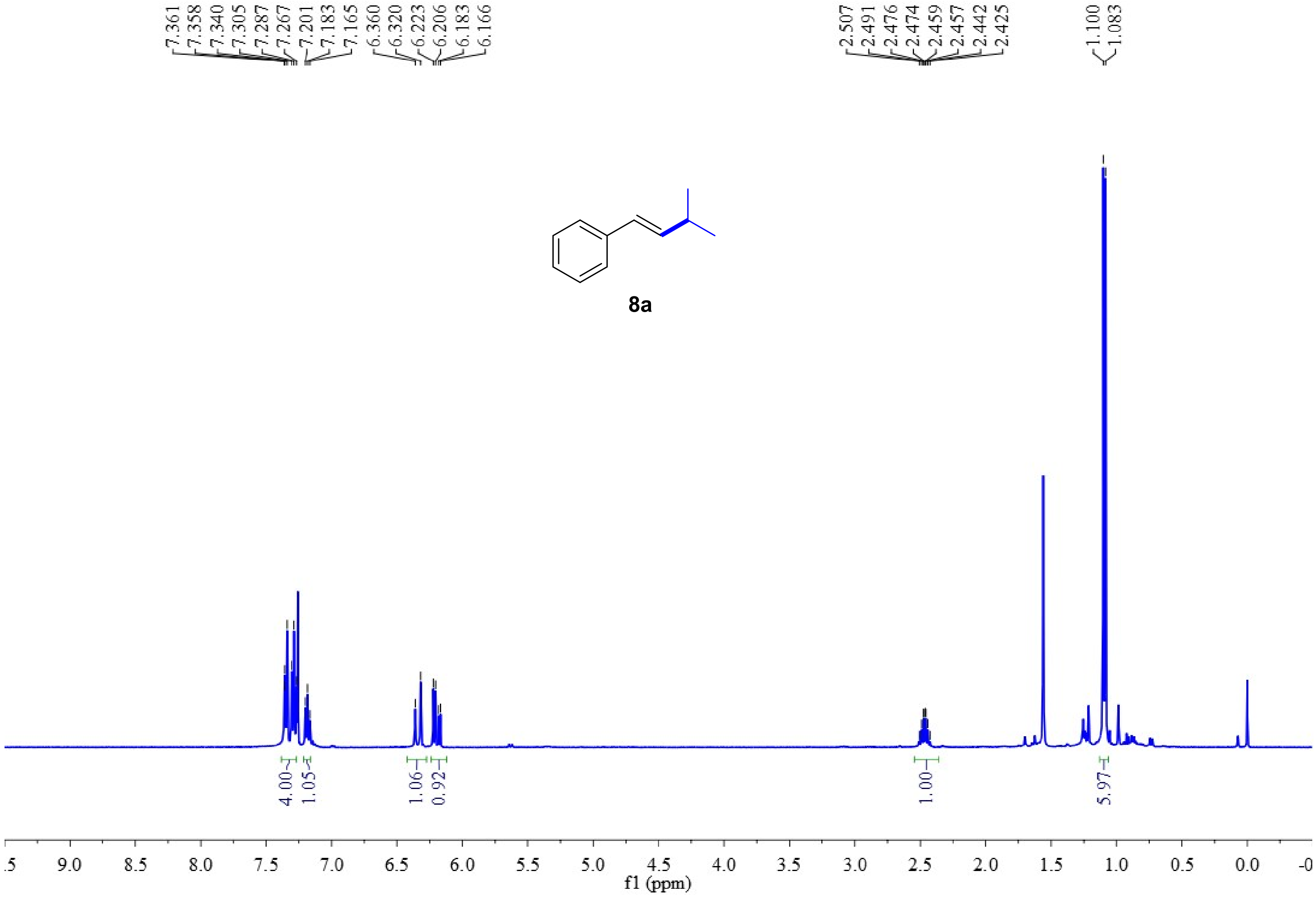
7.356
7.339
7.319
7.309
7.303
7.296
7.287
5.447
5.434
5.428
5.414
3.192
3.173
3.152
3.132
2.723
2.709
2.683
2.669
2.611
2.593
2.576
2.559
1.198
1.093
1.076
1.030
1.013
^{t}BuOO
O
5.05
1.00
1.01
1.01
0.99
9.06
3.05
3.01
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

7a


211.663
140.274
128.500
128.157
127.092
82.173
80.872
45.999
41.777
26.515
17.883
^{t}BuOO
O
7a
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.361
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7.305
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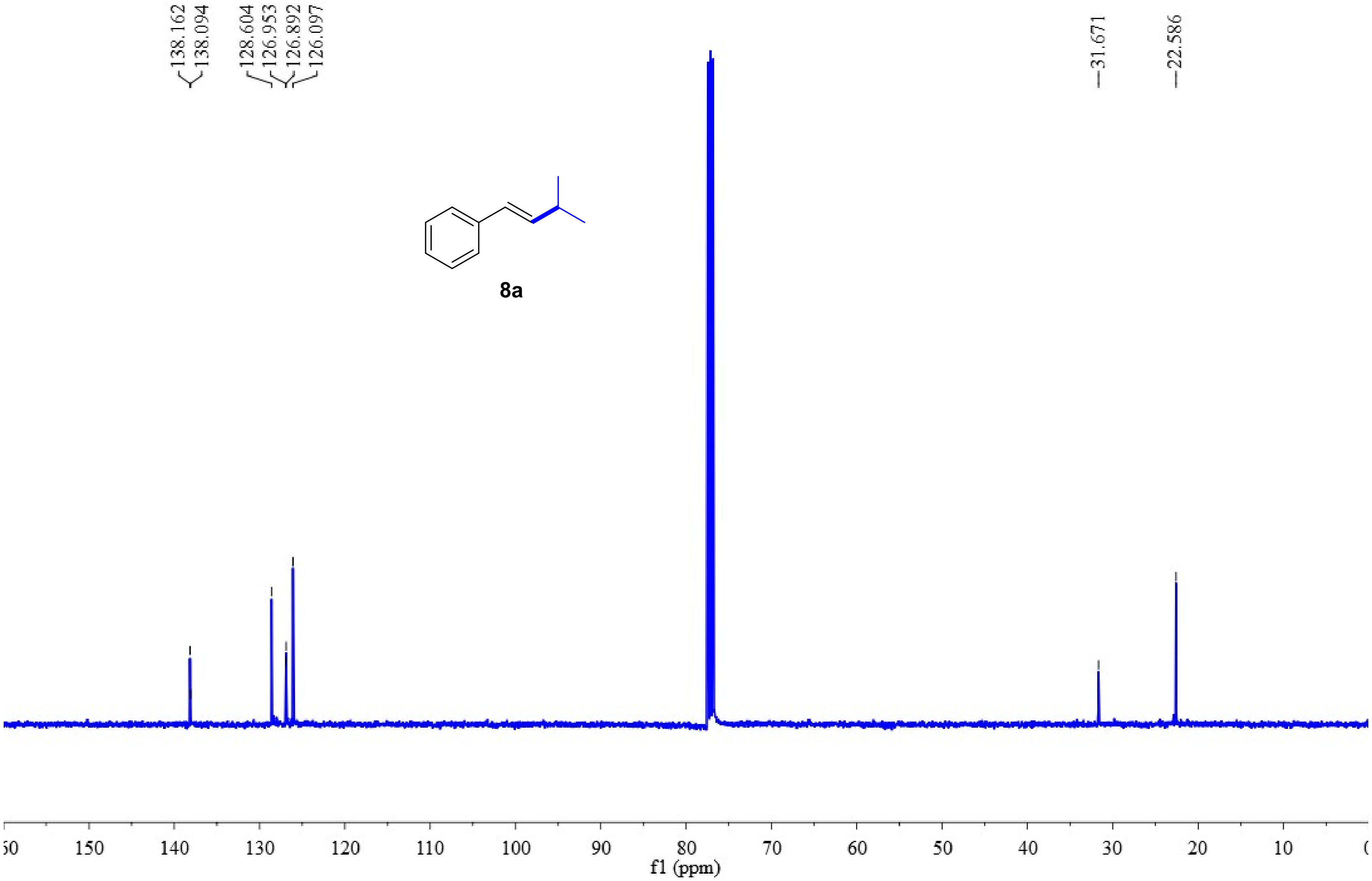
7.267
7.201
7.183
7.165
6.360
6.320
6.223
6.206
6.183
6.166
2.507
2.491
2.476
2.474
2.459
2.457
2.442
2.425
1.100
1.083
8a
4.00
1.05
1.06
0.92
1.00
5.97
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)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126.953
126.892
126.097
31.671
22.586
8a
50
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)