Supplementary Material (ESI) for Chemical Communications

Phase-Transfer Catalytic Aza-Michael Addition of *tert*-Butyl benzyloxycarbamate to Electron-Deficient Olefins

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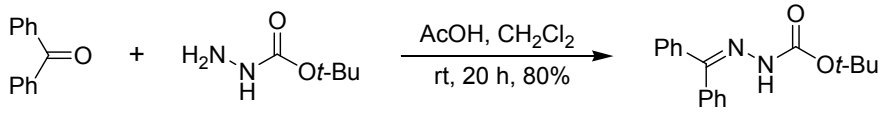
I. General methods

Infrared (IR) spectra were recorded on a JASCO FT/IR-300E and Perkin-Elmer 1710 FT spectrometer. Nuclear magnetic resonance (^{1}H-NMR and ^{13}C-NMR) spectra were measured on a JEOL JNM-LA 300 [300 MHz (^{1}H), 75 MHz (^{13}C)] spectrometer, JEOL JNM-GSX 400 [400 MHz (^{1}H), 100 MHz (^{13}C)] spectrometer, and Bruker AMX 500 [500 MHz (^{1}H), 125 MHz (^{13}C)] spectrometer, using DMSO-d_6 or $CHCl_3$-*d* as a solvent, and were reported in ppm relative to DMSO (δ 2.50) or $CHCl_3$ (δ 7.24) for ^{1}H-NMR and relative to the central DMSO-d_6 (δ 39.51) or $CHCl_3$-*d* (δ 77.23) resonance for ^{13}C-NMR. Coupling constants (*J*) in ^{1}H-NMR are in Hz. Low-resolution mass spectra (MS) were recorded on a VG Trio-2 GC-MS spectrometer, and high-resolution mass spectra (HRMS) were measured on a JEOL JMS-AX 505wA, JEOL JMS-HX 110A spectrometer. For thin-layer chromatography (TLC) analysis, Merck precoated TLC plate (silica gel 60 GF_{254}, 0.25 mm) were used. For flash column chromatography, E. Merck Kieselgel 60 (70~230 mesh) was used. All solvents and commercially available chemicals were used without additional purification.

II. Synthetic procedure and Characterization of new compounds

II-1. Preparation of New Substrates

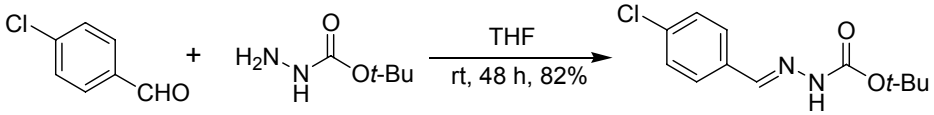
***tert*-Butyl 2-(diphenylmethylene)hydrazinecarboxylate (2).**



2

To a solution of *tert*-butyl carbazate (1.00 g, 7.57 mmol) in CH_2Cl_2 were added benzopheone (1.65 g, 9.08 mmol) and acetic acid (0.52 mL, 9.08 mmol) under argon atmosphere, and the resulting solution was stirred at room temperature. After 20 h, the mixture was neutralized by the addition of a solution of $NaHCO_3$. The organic later was separated, and the aqueous layer was extracted with CH_2Cl_2. The combined organic layer and extracts were dried over $MgSO_4$ and concentrated. The residue was purified by silica gel column chromatography (hexanes:EtOAc = 10:1) to afford **2** (1.80 g, 80% yield) as a white solid. m.p. 148 °C; ^{1}H-NMR (300 MHz, DMSO-d_6): δ 8.60 (s, 1H), 7.60–7.51 (m, 3H), 7.42–7.34 (m, 5H), 7.27–7.24 (m, 2H), 1.41 (s, 9H) ppm; ^{13}C-NMR (100 MHz, DMSO-d_6): δ 152.1, 150.0, 129.5, 128.4, 128.3, 126.8, 80.0, 27.9 ppm; IR (neat): ν 3357, 2977, 1746, 1480, 1367, 1321, 1229, 1156, 1065, 869, 766, 699 cm^{-1}; MS (FAB$^+$): *m/z* 297 $[M+H]^+$; HRMS (FAB$^+$): calcd for $C_{18}H_{21}N_2O_2$ $[M+H]^+$ 297.1603, found: 297.1607.

***tert*-Butyl 2-(4-chlorobenzyl)hydrazinecarboxylate (3).**



3

To a stirred solution of 4-chlorobenzaldehyde (2.00 g, 14.23 mmol) in THF was added a solution of *tert*-butyl carbazate (2.26 g, 17.07 mmol) in THF at room temperature. After stirring for 48 h, EtOAc and water were added, and the organic later was separated. The aqueous layer was extracted with EtOAc, and the combined organic layer was dried over $MgSO_4$, concentrated. The residue was purified by silica gel column chromatography (hexanes:EtOAc = 10:1) to give **3** (2.99 g, 82% yield) as a white solid. m.p. 171 °C; ^{1}H-NMR (300 MHz, DMSO-d_6): δ 10.97 (s, 1H), 7.97 (s, 1H), 7.62–7.59 (m, 2H), 7.46–7.44 (m, 2H), 1.45 (s, 9H) ppm; ^{13}C-NMR (100 MHz, DMSO-d_6): δ

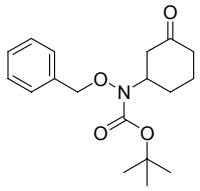
152.3, 141.8, 133.7, 133.5, 128.8, 128.1, 79.5, 28.0 ppm; IR (neat,): ν 3222, 2979, 1705, 1538, 1489, 1367, 1252, 1163, 1090, 1057, 869, 825, 759 cm^{-1}; MS (FAB^+): *m/z* 255 $[M+H]^+$; HRMS (FAB^+): calcd for $C_{12}H_{16}ClN_2O_2$ $[M+H]^+$ 255.0900, found 255.0896.

II-2. Aza-Michael Reaction

General Procedure

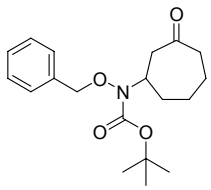
To a solution of *tert*-butyl benzyloxycarbamate (**5**, 2.0 equiv.) and tetra-*n*-butylammonium bromide (0.1 equiv.) was added 50% KOH (1.2 equiv.) and enone (**6**, 1.0 equiv.) successively. The resulting mixture was stirred at room temperature until the starting enone disappeared on TLC. After the reaction was completed, EtOAc and brine were added to the reaction mixture. The organic layer was separated, and the aqueous layer was extracted with EtOAc. The combined organic solution was dried over $MgSO_4$ and concentrated. The residue was purified by silica gel column chromatography to afford the aza-Michael adduct **7**.

***tert*-Butyl benzyloxy(3-oxocyclohexyl)carbamate (7a)**



Colorless oil. ^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.39–7.29 (m, 5H), 4.80 (s, 2H), 4.22–4.11 (m, 1H), 2.69–2.60 (m, 1H), 2.49–2.43 (m, 1H), 2.32–2.12 (m, 2H), 2.06–1.82 (m, 3H), 1.60–1.52 (m, 1H), 1.49 (s, 9H) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$): δ 208.9, 156.3, 135.0, 129.2, 128.5, 128.3, 81.8, 78.5, 58.1, 44.6, 40.3, 28.1, 27.8, 21.8 ppm; IR (neat): ν 2970, 1710, 1455, 1370, 1322, 1257, 1165, 1113, 856, 750, 700 cm^{-1}; MS (FAB^+): *m/z* 320 $[M+H]^+$; HRMS (FAB^+): calcd for $C_{18}H_{26}NO_4$ $[M+H]^+$ 320.1862, found 320.1865.

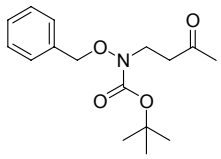
***tert*-Butyl benzyloxy(3-oxocycloheptyl)carbamate (7b)**



Colorless oil. ^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.39–7.31 (m, 5H), 4.81 (dd, *J*=9.54, 12.27 Hz, 2H), 4.12 (s, 1H), 3.00 (dd, *J*=11.37, 14.46 Hz, 1H), 2.61–2.34 (m, 3H),

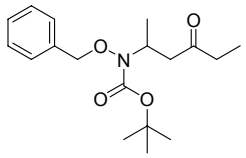
2.01–1.79 (m, 4H), 1.62–1.35 (m, 2H), 1.50 (s, 9H) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$): δ 211.6, 156.8, 135.3, 129.2, 128.5, 128.4, 81.9, 78.5, 57.4, 47.6, 43.8, 34.4, 28.2, 27.1, 23.8 ppm; IR (neat): ν 2932, 1702, 1454, 1369, 1256, 1163, 1070, 856, 748, 700 cm^{-1}; MS (FAB^+): *m/z* 334 $[M+H]^+$; HRMS (FAB^+): calcd for $C_{19}H_{27}NO_4$ $[M+H]^+$ 334.2018, found: 334.2028.

tert-Butyl benzyloxy(3-oxobutyl)carbamate (7c)



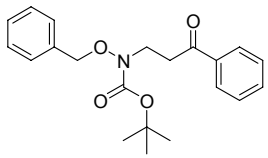
Colorless oil. ^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.37–7.28 (m, 5H), 4.77 (s, 2H), 3.68 (d, *J*=6.9 Hz, 2H), 2.65 (t, *J*=6.9 Hz, 2H), 2.09 (s, 3H), 1.47 (s, 9H) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$): δ 207.0, 156.3, 135.2, 129.4, 128.5, 128.3, 81.5, 76.7, 44.5, 40.7, 30.1, 28.2 ppm; IR (neat): ν 2926, 1712, 1367, 1252, 1162, 1104, 1028, 857, 750, 700 cm^{-1}; MS (FAB^+): *m/z* 294 $[M+H]^+$; HRMS (FAB^+): calcd for $C_{16}H_{24}NO_4$ $[M+H]^+$ 294.1750, found: 294.1703.

tert-Butyl benzyloxy(4-oxohexan-2-yl)carbamate (7d)



Colorless oil. ^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.40–7.28 (m, 5H), 4.80 (s, 2H), 4.55–4.43 (m, 1H), 2.62 (m, 2H), 2.38 (q, *J*=7.32 Hz, 2H), 1.49 (s, 9H), 1.19 (d, *J*=6.78 Hz, 3H), 1.00 (t, *J*=7.32 Hz, 3H) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$): δ 209.2, 156.8, 135.5, 129.3, 128.4, 128.3, 81.6, 78.2, 52.3, 46.2, 36.1, 28.2, 17.5, 7.6 ppm; IR (neat): ν 2977, 2936, 1712, 1455, 1368, 1318, 1167, 1104, 1018, 856, 750, 700 cm^{-1}; MS (FAB^+): *m/z* 322 $[M+H]^+$; HRMS (FAB^+): calcd for $C_{18}H_{28}NO_4$ $[M+H]^+$ 322.2018, found: 322.2028.

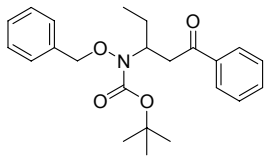
tert-Butyl benzyloxy(3-oxo-3-phenylpropyl)carbamate (7e)



Colorless oil. ^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.89–7.86 (m, 2H), 7.55–7.27 (m, 8H), 4.83 (s, 2H), 3.86 (t, *J*=7.20 Hz, 2H), 3.20 (t, *J*=7.20 Hz, 2H), 1.48 (s, 9H) ppm; ^{13}C-

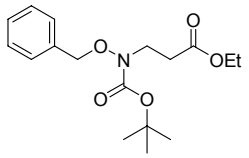
NMR (100 MHz, $CDCl_3$): δ 198.4, 156.4, 136.6, 135.4, 133.1, 129.5, 128.5, 128.4, 128.0, 81.6, 76.8, 45.2, 35.8, 28.2 ppm; IR (neat): ν 2928, 1689, 1451, 1367, 1252, 1212, 1161, 1095, 856, 745, 696 cm^{-1}; MS (FAB^+): *m/z* 356 $[M+H]^+$; HRMS (FAB^+): calcd for $C_{21}H_{26}NO_4$ $[M+H]^+$356.1862, found: 356.1869.

***tert*-Butyl benzyloxy(1-oxo-1-phenylpentan-3-yl)carbamate (7f)**



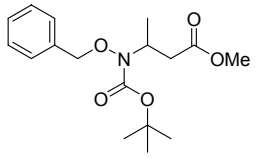
Colorless oil. ^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.90–7.87 (m, 2H), 7.56–7.27 (m, 8H), 4.85 (s, 2H), 4.52–4.47 (m, 1H), 3.26–3.03 (m, 2H), 1.83–1.53 (m, 2H), 1.47 (s, 9H), 0.97 (t, *J*=7.32 Hz, 3H) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$): δ 198.0, 157.0, 136.8, 135.8, 133.0, 129.2, 128.5, 128.3, 128.1, 81.4, 78.4, 58.6, 41.5, 28.2, 25.3, 11.1 ppm; IR (neat): ν 2967, 1702, 1454, 1367, 1322, 1256, 167, 1115, 856, 752, 696 cm^{-1}; MS (FAB^+): *m/z* 384 $[M+H]^+$; HRMS (FAB^+): calcd for $C_{23}H_{30}NO_4$ $[M+H]^+$ 384.2175, found: 384.2165.

Ethyl 3-[benzyloxy(*tert*-butoxycarbonyl)amino]propanoate (7g)



Colorless oil. ^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.38–7.31 (m, 5H), 4.80 (s, 2H), 4.07 (q, *J*=7.14 Hz, 2H), 3.71 (t, *J*=7.14 Hz, 2H), 2.55 (t, *J*=7.14 Hz, 2H), 1.47 (s, 9H), 1.19 (t, *J*=7.14 Hz, 3H) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$): δ 171.6, 156.3, 135.2, 129.4, 128.5, 128.3, 81.6, 76.9, 60.4, 45.4, 32.1, 28.2, 14.0 ppm; IR (neat): ν 2978, 1733, 1453, 1369, 1253, 1161, 1096, 1024, 856, 749, 700 cm^{-1}; MS (FAB^+): *m/z* 324 $[M+H]^+$; HRMS (FAB^+): calcd for $C_{17}H_{26}NO_5$ $[M+H]^+$324.1811, found: 324.1805.

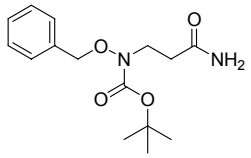
Methyl 3-[benzyloxy(*tert*-butoxycarbonyl)amino]butanoate (7h)



Colorless oil. ^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.38–7.26 (m, 5H), 4.77 (s, 2H), 3.81–3.50 (m, 2H), 3.57 (s, 3H), 2.86–2.75 (m, 1H), 1.47 (s, 9H), 1.12 (d, *J*=7.14 Hz, 3H)

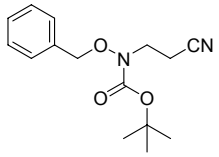
ppm; ^{13}C-NMR (100 MHz, $CDCl_3$): δ 175.3, 156.1, 135.2, 129.3, 128.4, 128.2, 81.4, 76.5, 51.8, 51.6, 37.9, 28.1, 14.6 ppm; IR (neat): ν 2976, 1736, 1456, 1368, 1249, 1167, 857, 751, 700, 616 cm^{-1}; MS (FAB^+): *m/z* 324 $[M+H]^+$; HRMS (FAB^+): calcd for $C_{17}H_{26}NO_5$ $[M+H]^+$324.1811, found: 324.1813.

tert-Butyl 3-amino-3-oxopropyl(benzyloxy)carbamate (7i)



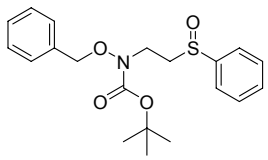
White solid. m.p. 189 °C; ^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.39–7.29 (m, 5H), 5.82 (s, 1H), 5.64 (s, 1H), 4.81 (s, 2H), 3.73 (t, *J*=6.77 Hz, 2H), 2.46 (t, *J*=6.77 Hz, 2H), 1.47 (s, 9H) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$): δ 173.4, 156.4, 135.1, 129.5, 128.6, 128.4, 81.9, 76.9, 45.6, 33.4, 28.2 ppm; IR (neat): ν 3346, 2976, 2673, 1450, 1369, 1292, 1252, 1162, 1024, 855, 750, 700 cm^{-1}; MS (FAB^+): *m/z* 295 $[M+H]^+$; HRMS (FAB^+): calcd for $C_{15}H_{23}N_2O_4$ $[M+H]^+$ 295.1658, found: 295.1654.

tert-Butyl benzyloxy(2-cyanoethyl)carbamate (7j)



Colorless oil. ^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.40–7.30 (m, 5H), 4.85 (s, 2H), 3.62 (t, *J*=6.87 Hz, 2H), 2.50 (t, *J*=6.87 Hz, 2H), 1.49 (s, 9H) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$): δ 156.0, 135.0, 129.4, 128.7, 128.5, 117.6, 82.5, 77.4, 45.9, 28.0, 15.6 ppm; IR (neat): ν 2977, 1705, 1454, 1369, 1253, 1216, 1162, 1104, 1022, 854, 750, 700 cm^{-1}; MS (FAB^+): *m/z* 277 $[M+H]^+$; HRMS (FAB^+): calcd for $C_{15}H_{21}N_2O_3$ $[M+H]^+$ 277.1552, found: 277.1559.

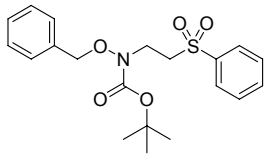
tert-Butyl benzyloxy[2-(phenylsulfinyl)ethyl]carbamate (7k)



Colorless oil. ^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.59–7.47 (m, 5H), 7.36–7.30 (m, 5H), 4.82 (d, *J*=2.91 Hz, 2H), 3.93–3.84 (m, 1H), 3.62–3.53 (m, 1H), 3.07–2.98 (m, 1H), 2.94–2.85 (m, 1H), 1.46 (s, 9H) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$): δ 156.1, 143.3,

135.0, 131.0, 129.4, 129.2, 128.6, 128.4, 123.9, 82.1, 77.0, 53.9, 43.3, 28.1 ppm; IR (neat): ν 3741, 2926, 1704, 1251, 1369, 1285, 1157, 1044, 749, 685 cm^{-1}; MS (FAB^+): *m/z* 376 $[M+H]^+$; HRMS (FAB^+): calcd for $C_{20}H_{26}NO_4S$ $[M+H]^+$ 376.1583, found: 376.1578.

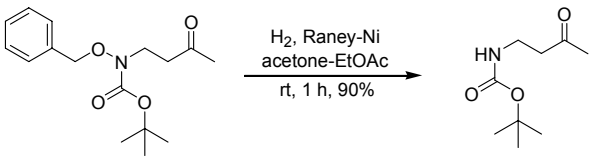
***tert*-Butyl benzyloxy[2-(phenylsulfonyl)ethyl]carbamate (7l)**



Colorless oil. ^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.86–7.84 (m, 2H), 7.66–7.51 (m, 3H), 7.32–7.30 (m, 5H), 4.73 (s, 2H), 3.70–4.07 (m, 2H), 3.30–3.25 (m, 2H), 1.42 (s, 9H) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$): δ 155.9, 138.8, 135.0, 133.8, 129.4, 129.3, 128.6, 128.4, 127.9, 82.2, 77.1, 52.3, 43.9, 28.0 ppm; IR (neat): ν 2927, 1705, 1449, 1368, 1315, 1152, 1085, 1000, 749, 694, 616 cm^{-1}; MS (FAB^+): *m/z* 391 [M]; HRMS (FAB^+): calcd for $C_{20}H_{25}NO_5S$ [M] 391.1435, found: 391.1451.

II-3. Deprotection of the Aza-Michael Adduct

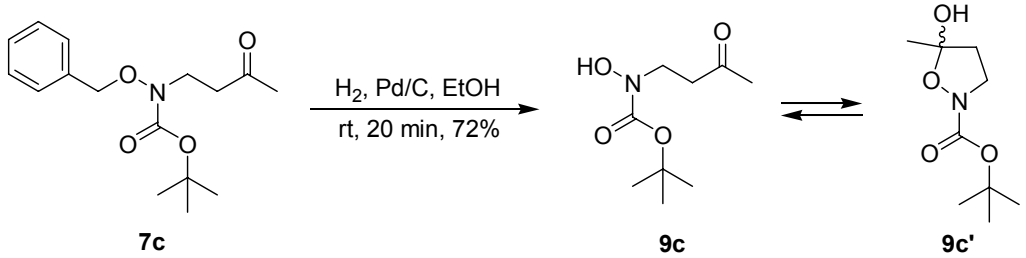
***tert*-Butyl 3-oxobutylcarbamate (8c)**



7c **8c**

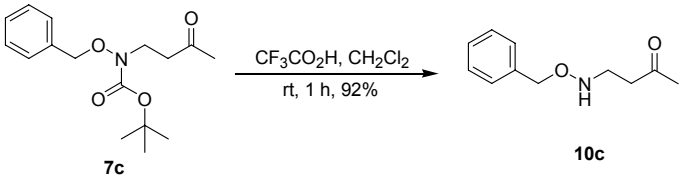
To a solution of **7c** (64 mg, 0.218 mmol) in EtOAc (1 mL) was added a catalytic amount of acetone-washed (5×1 mL) Raney-Ni (4200; slurry in water; active catalyst; Aldrich). The reaction mixture was stirred under hydrogen atmosphere at room temperature. After stirring for 1 h, the mixture was filtered through Celite pad, and washed with EtOAc. The filtrate and washings were combined and concentrated. The residue was purified by silica gel column chromatography (hexanes:EtOAc = 3:1) to afford **8c** (37 mg, 90% yield) as colorless oil. ^{1}H-NMR (300 MHz, $CDCl_3$): δ 4.95 (br s, 1H), 3.31 (m, 2H), 2.63 (t, *J*=5.85 Hz, 2H), 2.12 (s, 3H), 1.39 (s, 9H) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$): δ 208.13, 155.84, 79.27, 43.49, 35.17, 30.14, 28.35 ppm; IR (neat): ν 3365, 2976, 2931, 1712, 1518, 1455, 1366, 1275, 1251, 1168, 866 cm^{-1}; MS (FAB^+): *m/z* 210 $[M+Na]^+$; HRMS (FAB^+): calcd for $C_9H_{17}NO_3Na$ $[M+Na]^+$ 210.1106, found: 210.1109.

tert-Butyl hydroxyl(3-oxobutyl)carbamate (9c)



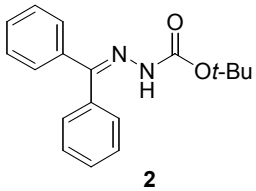
To a solution of **7c** (52 mg, 0.177 mmol) in EtOH was added a catalytic amount of Pd/C (10% on activated carbon) The reaction mixture was stirred under hydrogen atmosphere at room temperature. After stirring for 20 min, the reaction mixture was filtered through Celite pad, and washed with EtOH. The filtrate and washings were combined and concentrated. The residue was purified by silica gel column chromatography (hexanes:EtOAc = 3:1) to afford **9c** (25 mg, 72% yield) as colorless oil. ^{1}H-NMR (300 MHz, C_6D_6): δ 8.06 (br s, 1H), 3.83 (t, *J*=6.78 Hz, 2H), 2.43 (t, *J*=6.78 Hz, 2H), 1.63 (s, 3H), 1.42 (s, 9H) ppm, [*Minor Lactol (**9c'**) δ 5.0 (br, 1H), 3.92–4.02 (m, 1H), 3.25–3.31 (m, 1H), 1.97–2.03 (m, 1H), 1.58 (s, 3H), 1.53–1.58 (m, 1H), 1.42 (s, 9H) ppm*]; ^{13}C-NMR (75 MHz, C_6D_6): δ 205.6, 157.2, 81.3, 47.7, 40.6, 29.5, 28.2 ppm, [*Minor Lactol (**9c'**) δ 159.9, 105.1, 81.3, 45.9, 39.3, 27.9, 23.1 ppm*]; MS (FAB$^+$): *m/z* 226 $[M+Na]^+$; HRMS (FAB$^+$): calcd for $C_9H_{18}NO_4$ $[M+H]^+$ 204.1236, found: 204.1243.

4-(Benzyloxyamino)butan-2-one (10c)



To a solution of **7c** (64 mg, 0.218 mmol) in CH_2Cl_2 (1 mL) was added trifluoroacetic acid (0.5 mL) in water bath. After stirring for 1 h at room temperature, the reaction mixture was diluted with CH_2Cl_2 (10 mL), washed with saturated aqueous $NaHCO_3$ solution (2×1 mL) and then back-extracted with CH_2Cl_2 (2×5 mL). The combined organic layer was dried and concentrated to afford **9c** (39 mg, 92% yield) as colorless oil. ^{1}H-NMR (300 MHz, C_6D_6): δ 7.36–7.11(m, 5H), 4.65 (s, 2H), 2.99 (t, *J*=6.15, 2H), 2.11 (t, *J*=6.15, 2 H), 1.58 (s, 3H) ^{13}C-NMR (75 MHz, C_6D_6): δ 206.0, 138.9, 130.0, 128.7, 128.5, 128.4, 76.2, 47.0, 40.7, 29.4 ppm; MS (FAB$^+$): *m/z* 194 $[M+H]^+$; HRMS (CI$^+$): calcd for $C_{11}H_{16}NO_2$ $[M+H]^+$ 194.1181, found: 194.1183.

III. ^{1}H and ^{13}C-NMR spectra of the new compounds



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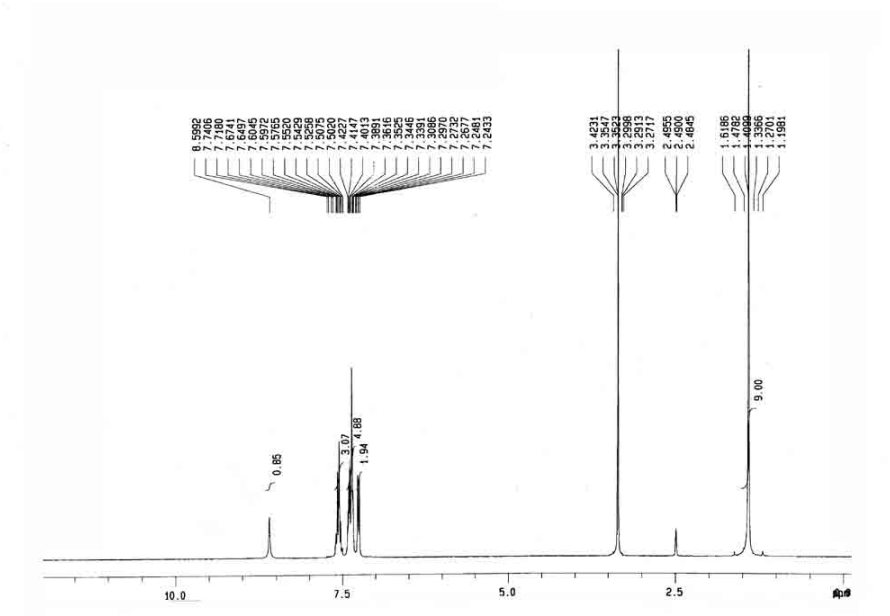
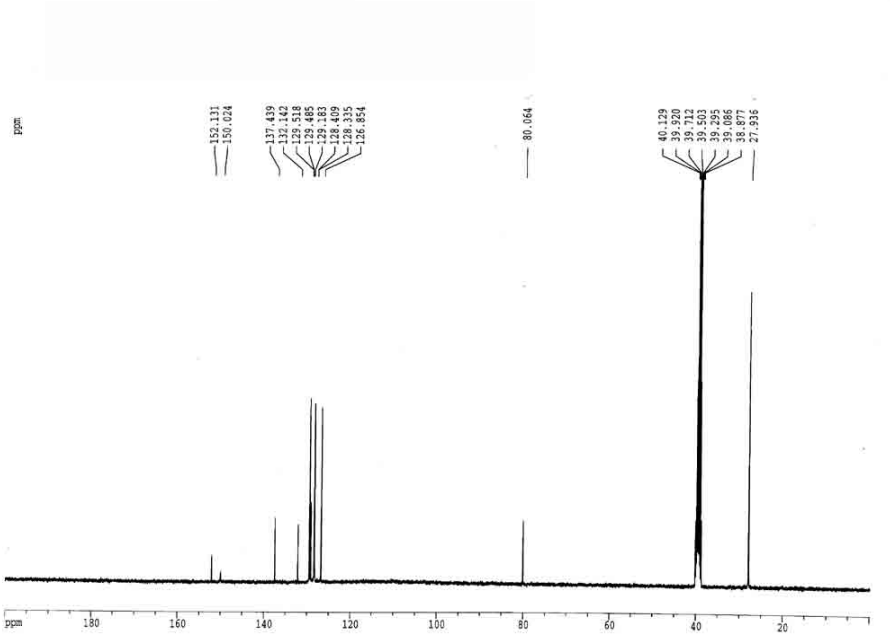
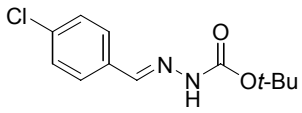
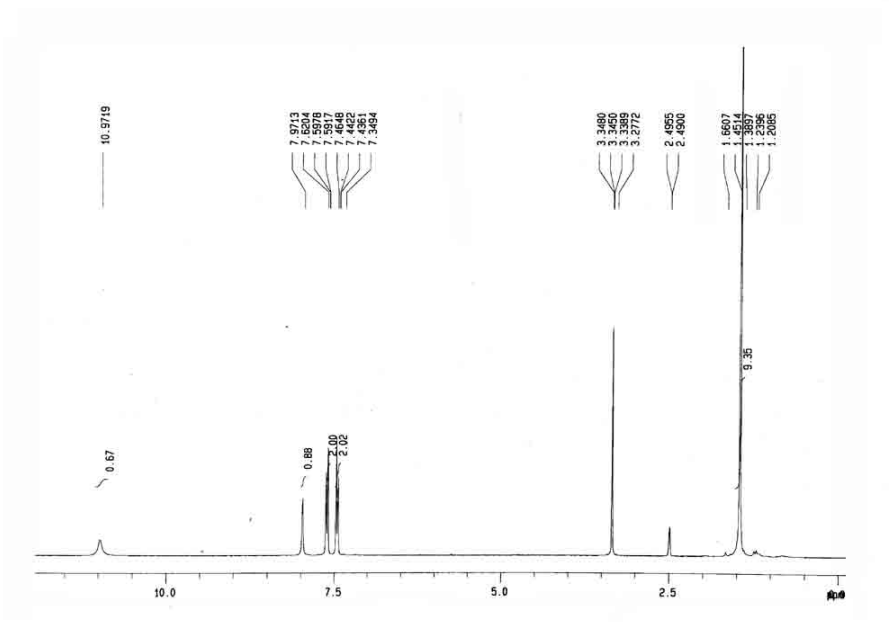





Cl
N
N
H
O
Ot-Bu

3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3.3365
3.2772
2.4855
2.4500
1.6607
1.4514
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9.35
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2.02
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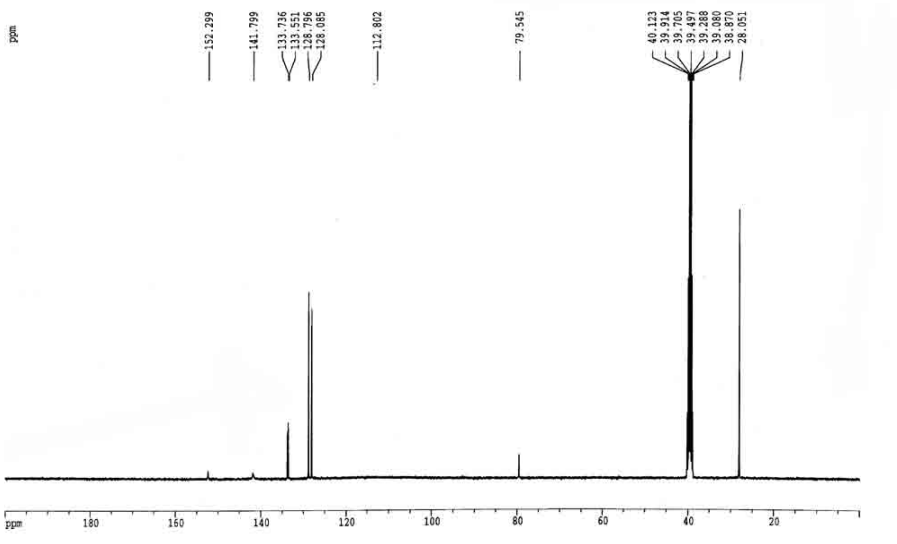
7.5
5.0
2.5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39.080
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ppm
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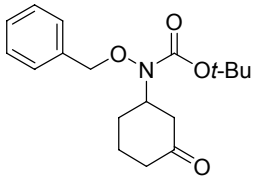
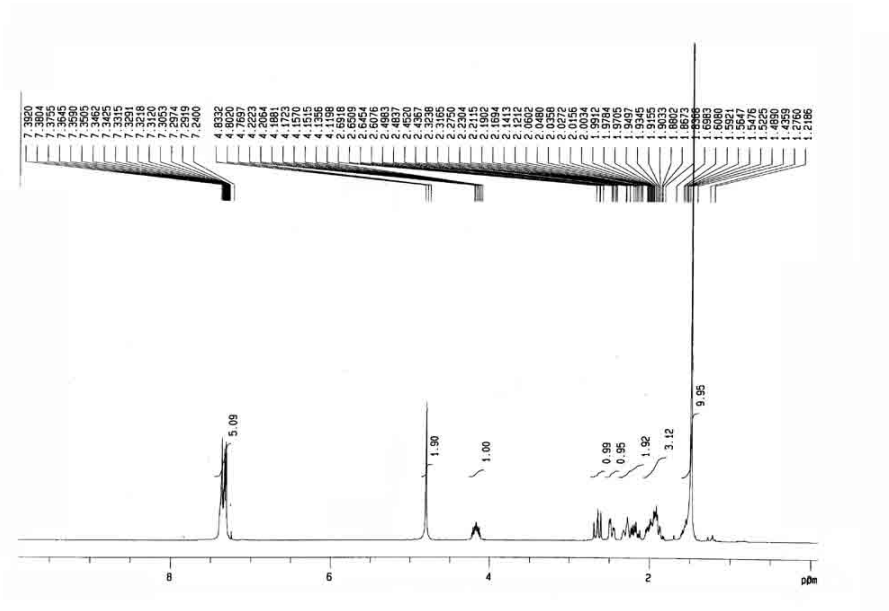
160
140
120
100
80
60
40
20


O
O
N
O
Ot-Bu
O

7a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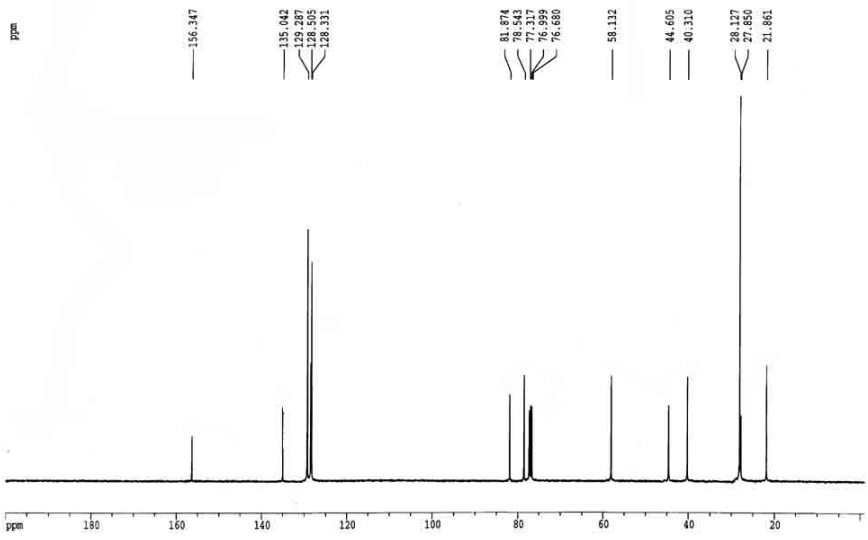
8
6
4
2
ppm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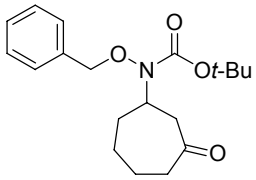
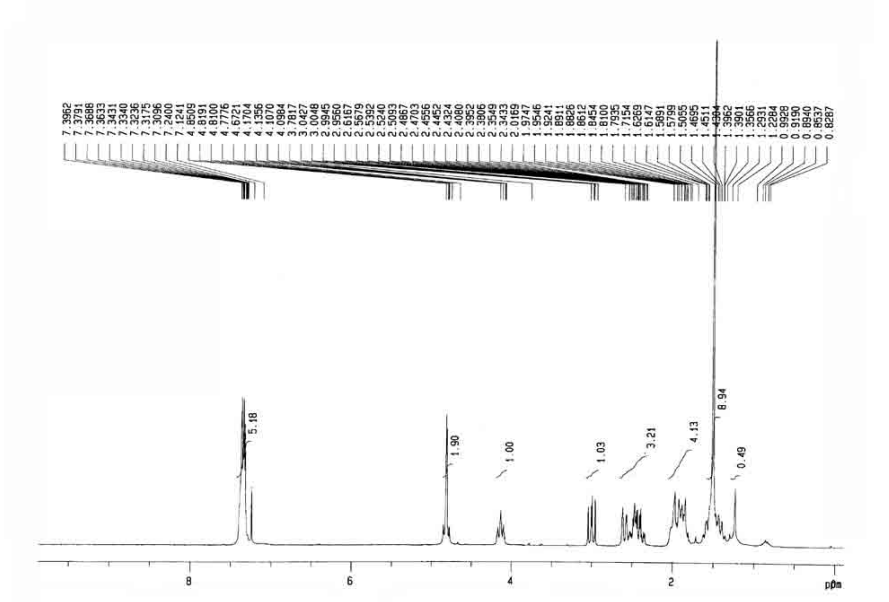
160
140
120
100
80
60
40
20


O
O
N
O
Ot-Bu
O

7b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4
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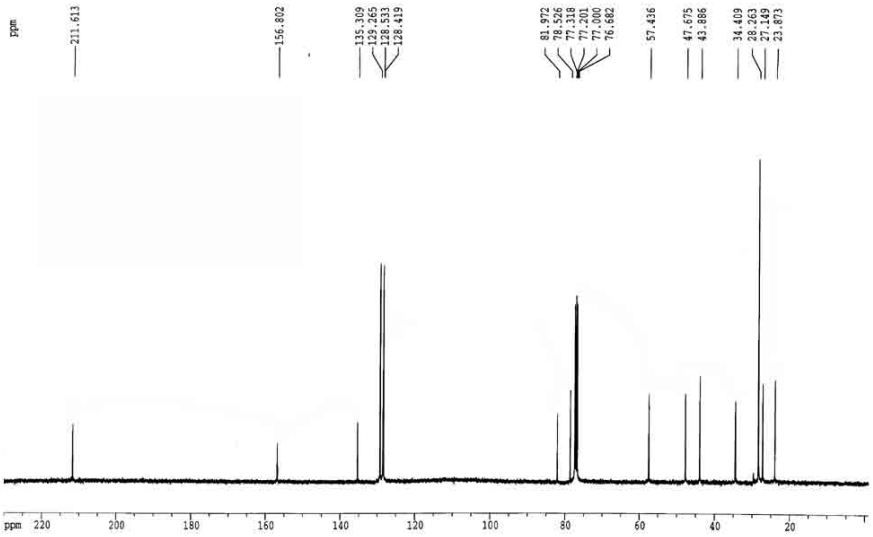
ppm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47.675
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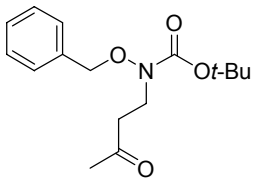
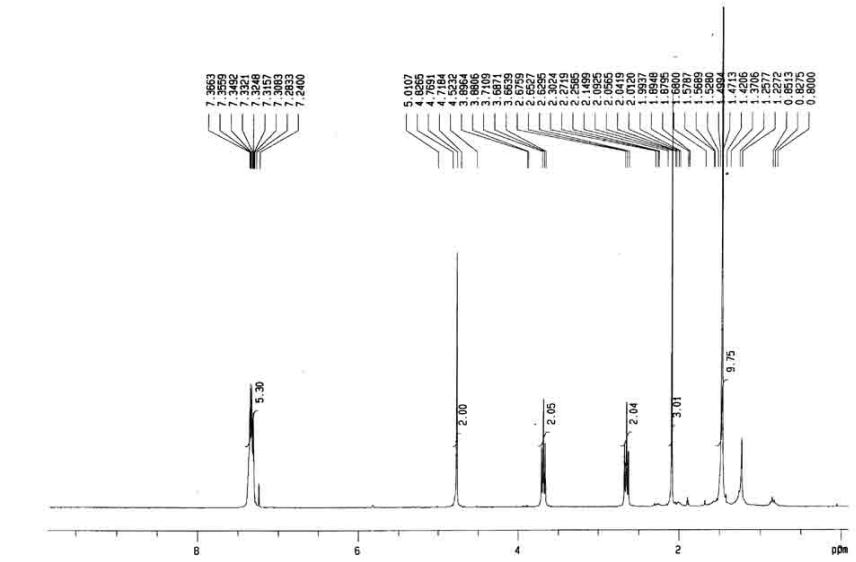
100
80
60
40
20


O
O
N
O
Ot-Bu
O

7c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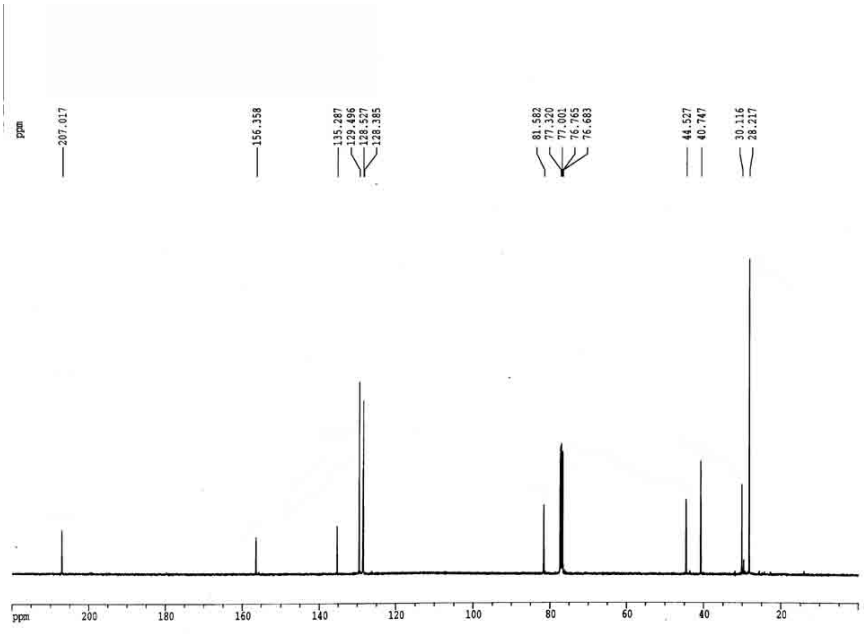
8
6
4
2
ppm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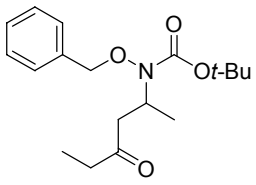
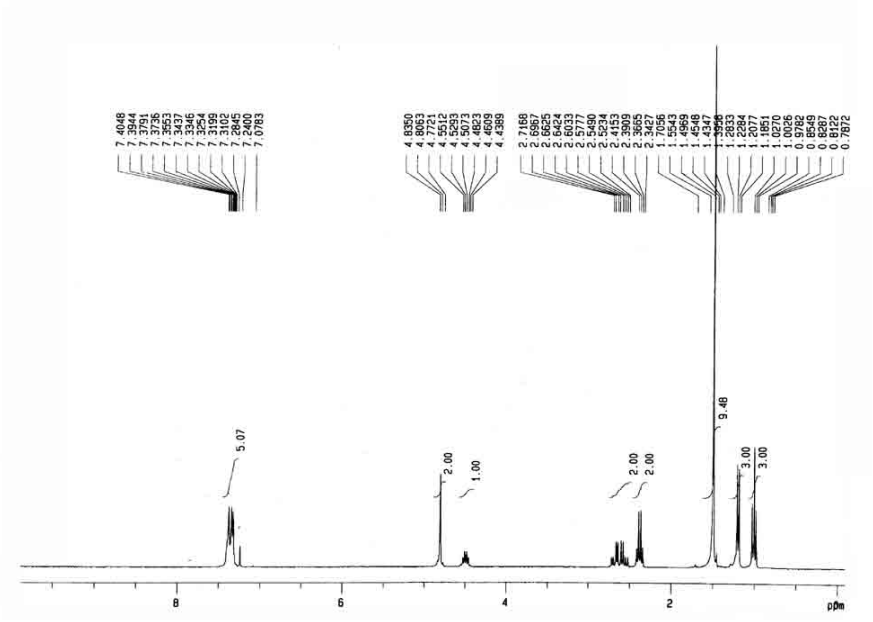
80
60
40
20


O
O
N
Ot-Bu
O

7d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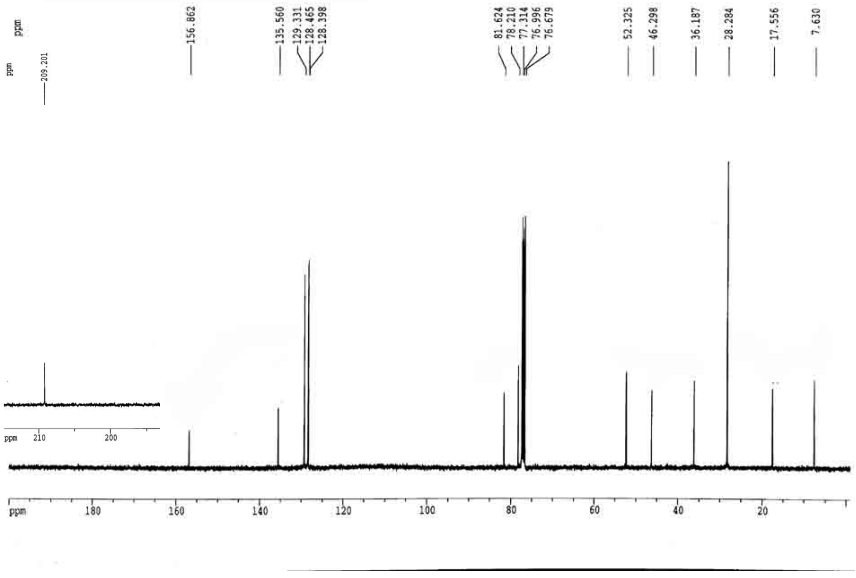
3.00
8
6
4
2
ppm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80
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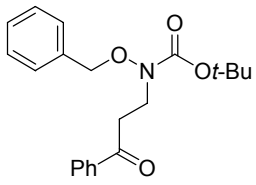
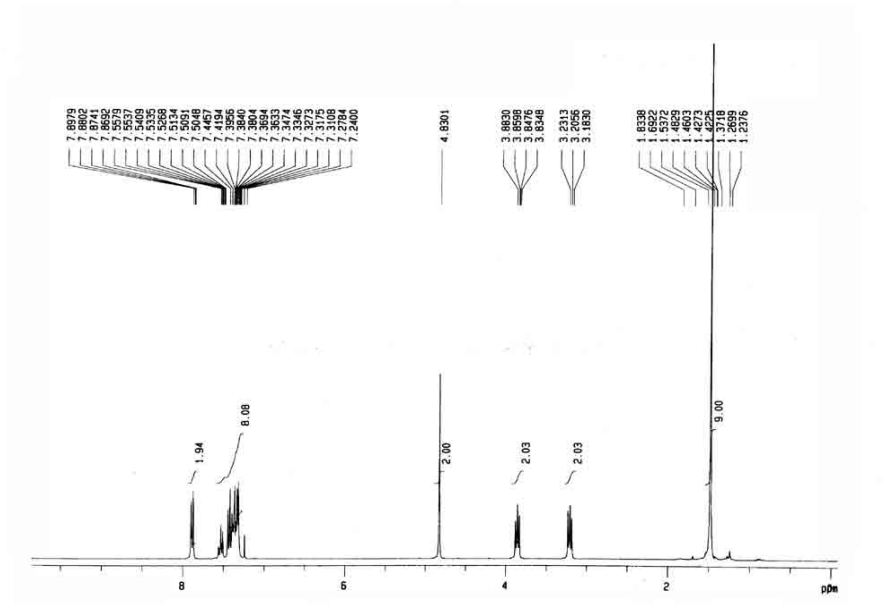

O
O
N
Ot-Bu
Ph
O

7e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7.5048
7.4467
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7.3846
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7.3633
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7.3346
7.3273
7.3175
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7.2784
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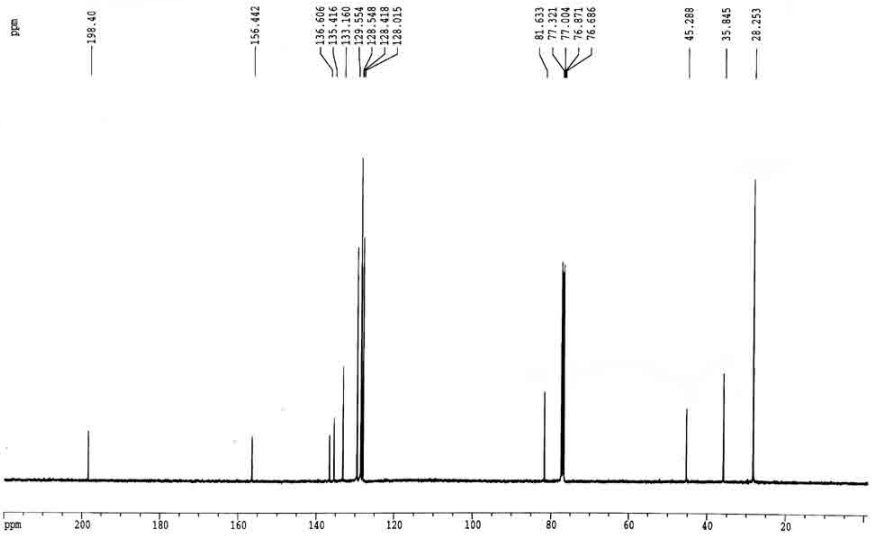
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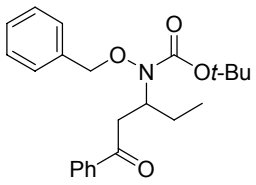
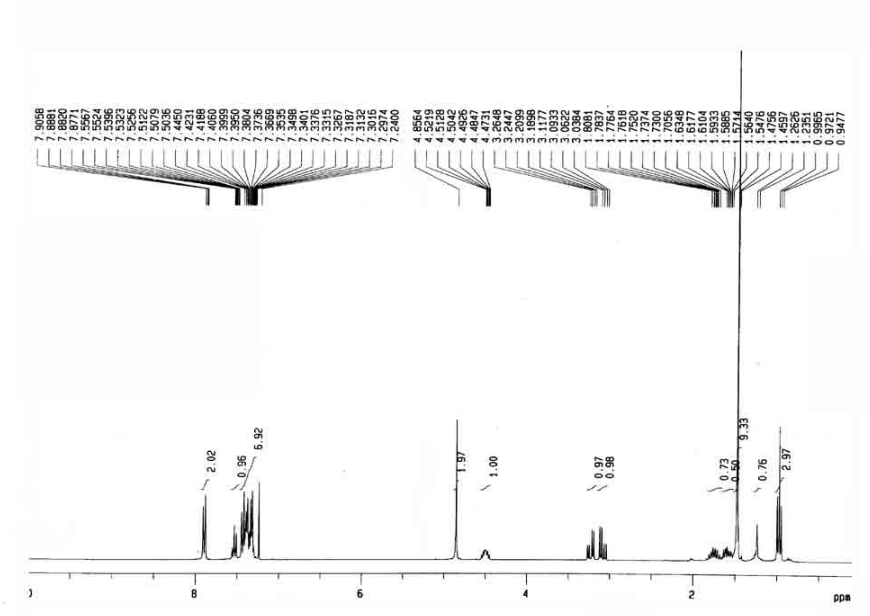
40
20


O
O
N
Ot-Bu
Ph
O

7f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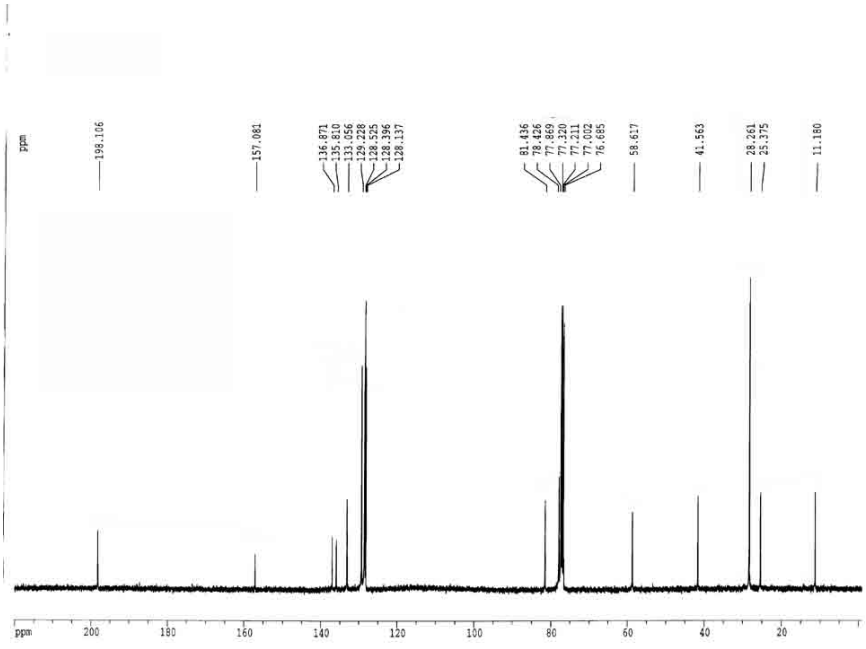
ppm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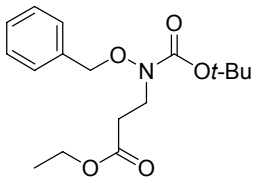
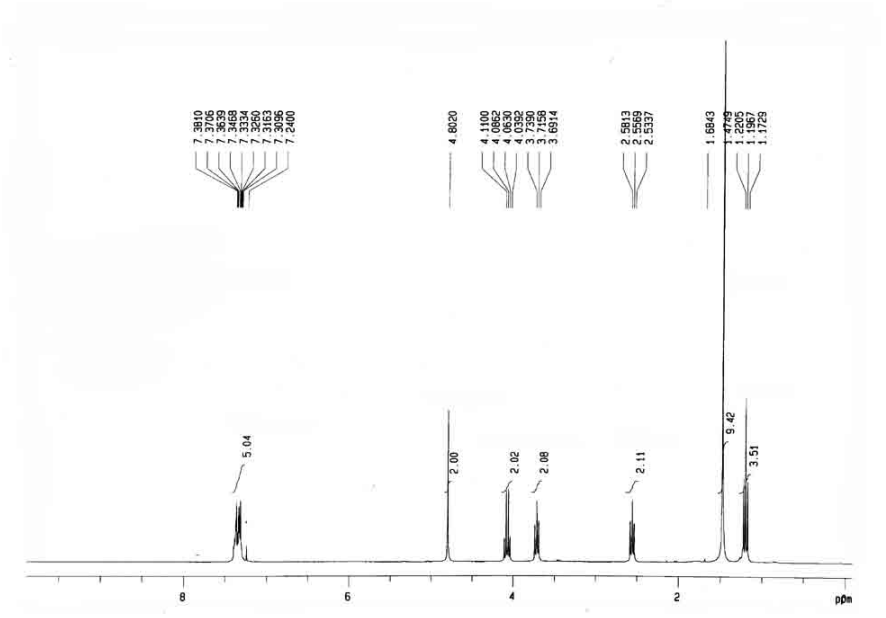

O
O
N
Ot-Bu
O
O

7g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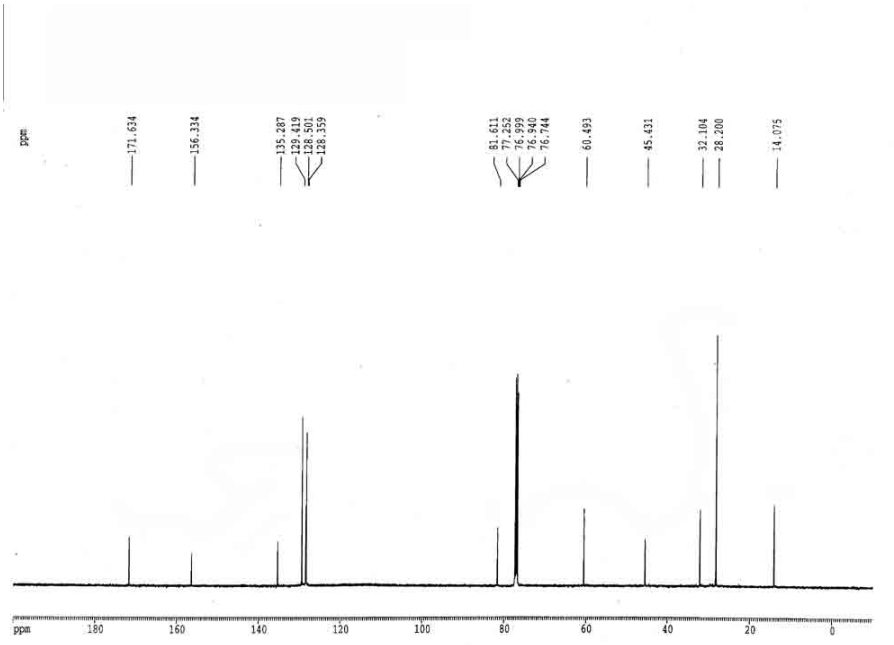
6
4
2
ppm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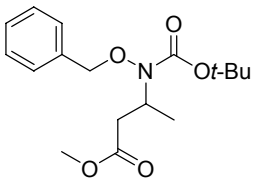
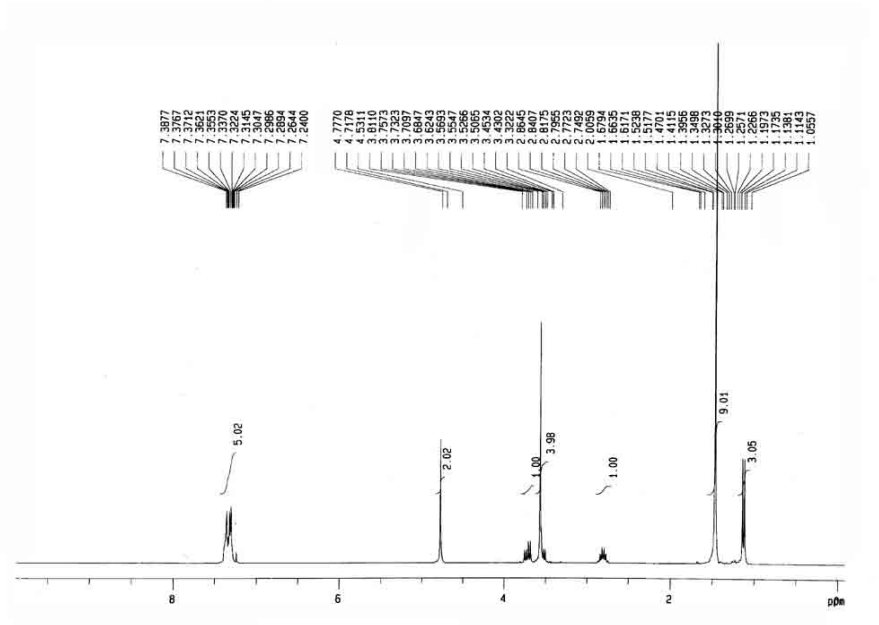

O
O
N
O
Ot-Bu
O
O

7h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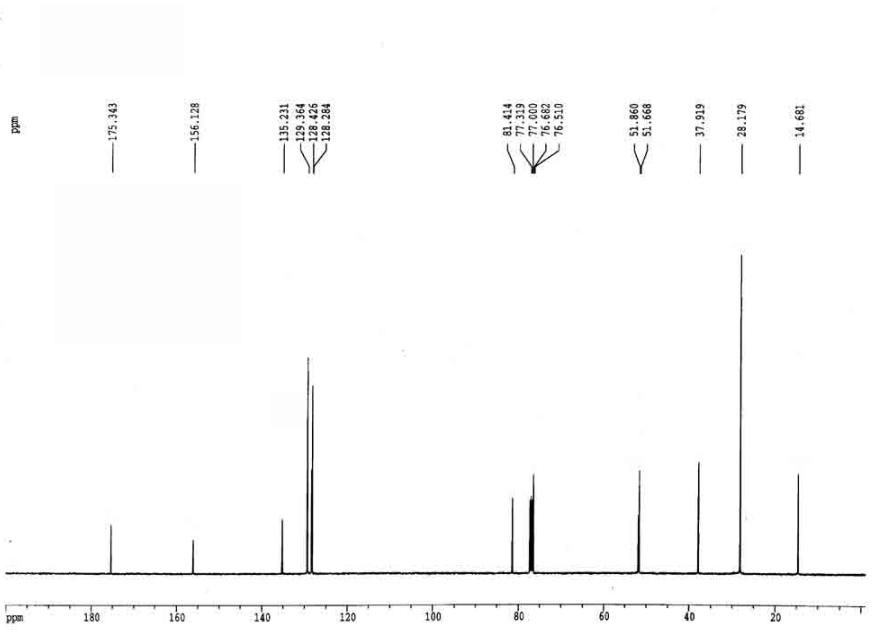
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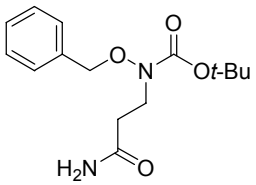
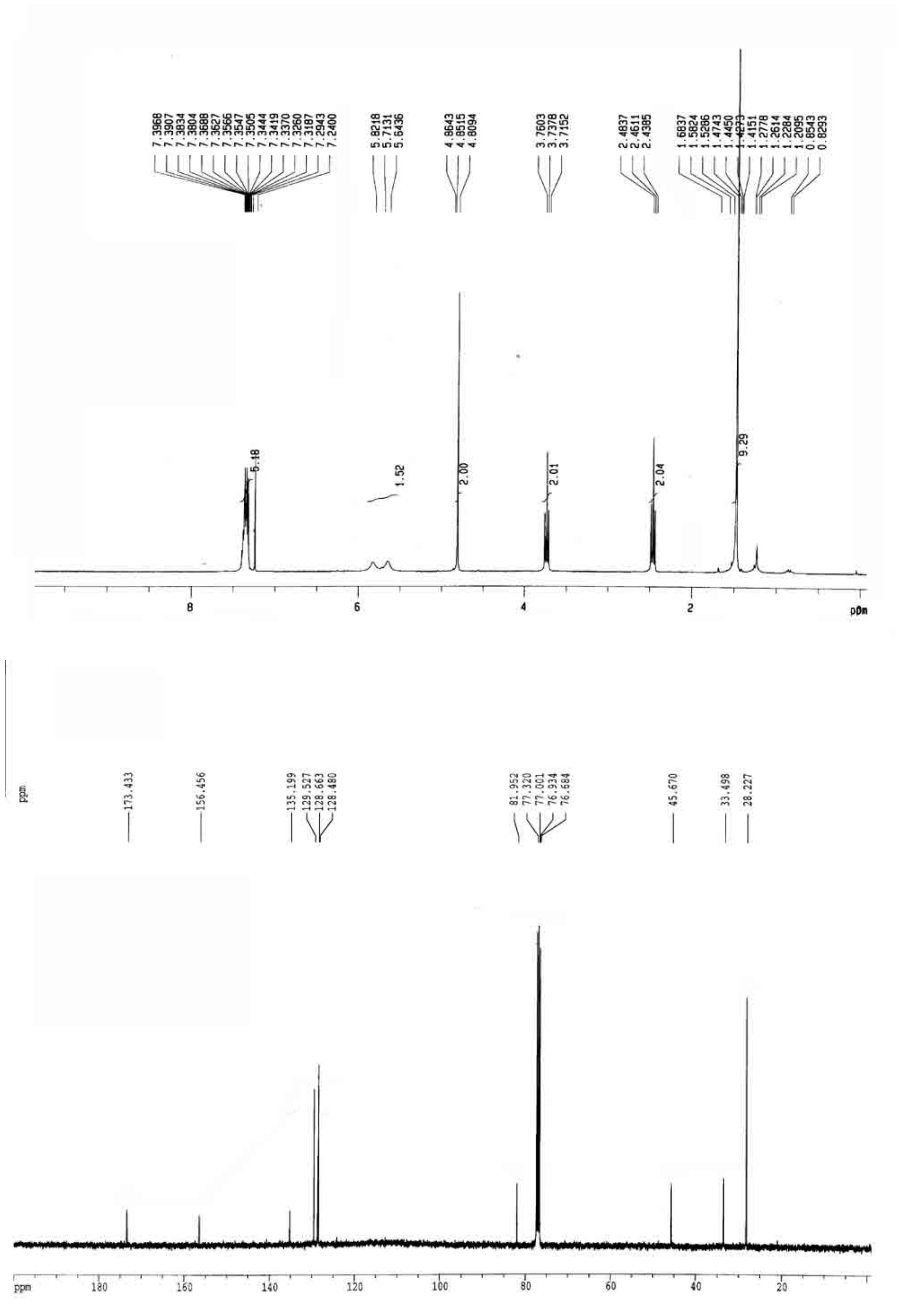

O
O
N
Ot-Bu
H2N
O

7i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7.3370
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7.3187
7.2943
7.2400
5.8218
5.7731
5.6436
4.8643
4.8515
4.8094
3.7603
3.7378
3.7152
2.4837
2.4611
2.4385
1.6837
1.5824
1.5286
1.4743
1.4450
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1.2778
1.2614
1.2284
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2.04
9.29
8
6
4
2
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140
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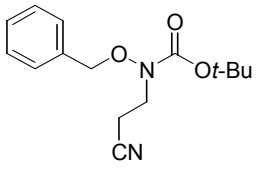
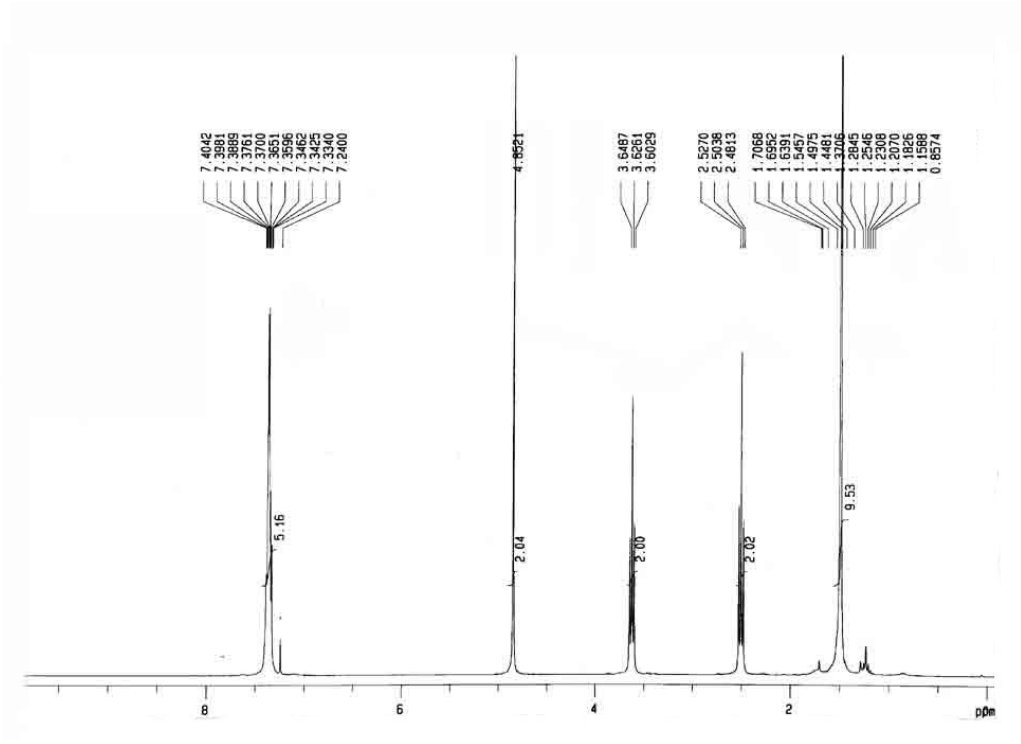
60
40
20


O
O
N
Ot-Bu
CN

7j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7.3981
7.3889
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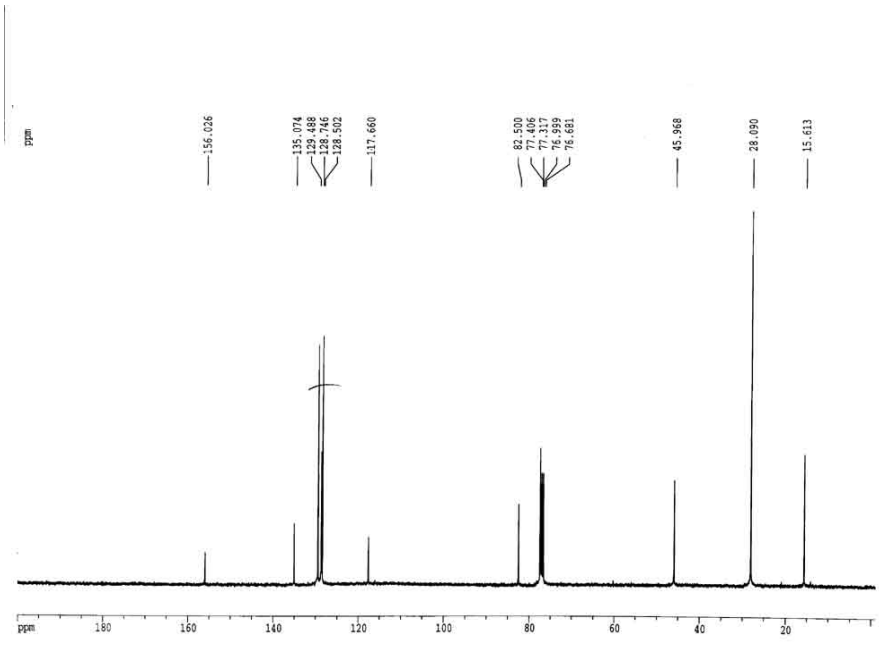
7.3700
7.3651
7.3596
7.3462
7.3425
7.3340
7.2400
4.8521
3.6487
3.6261
3.6029
2.5270
2.5038
2.4813
1.7068
1.6852
1.6591
1.5457
1.4975
1.4681
1.3306
1.2845
1.2546
1.2308
1.2070
1.1826
1.1588
0.8574
5.16
2.04
2.00
2.02
9.53
8
6
4
2
ppm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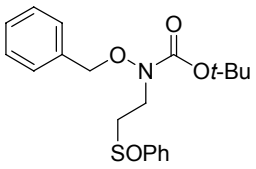
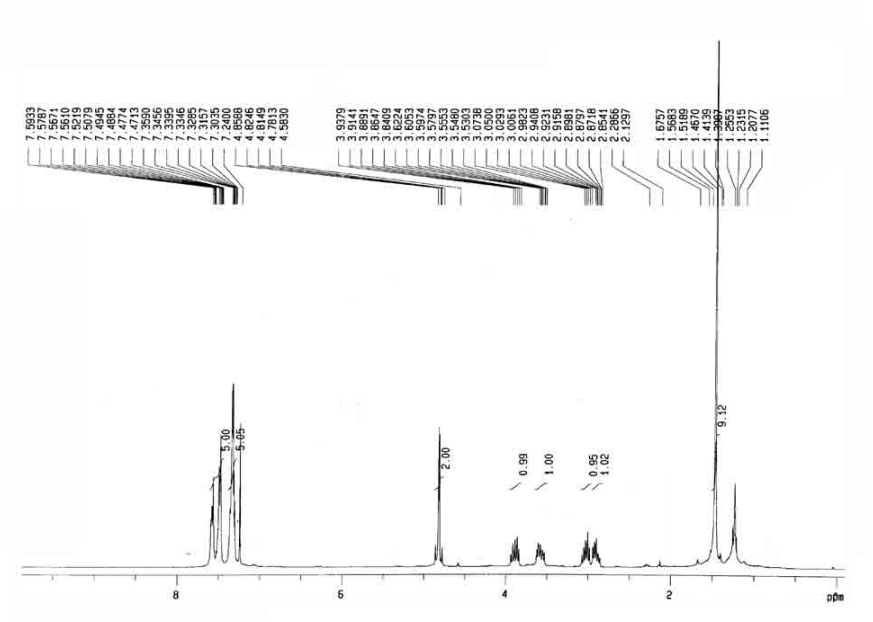
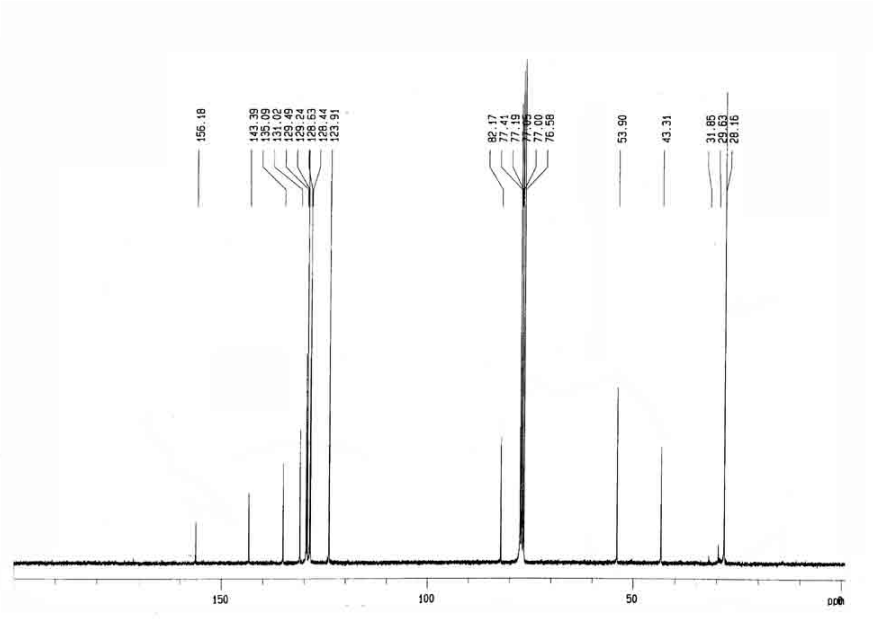
135.074
129.488
128.746
128.502
117.660
82.500
77.406
77.317
76.999
76.681
45.968
28.090
15.613
ppm
180
160
140
120
100
80
60
40
20


O
O
N
Ot-Bu
SOPh

7k


5.00
5.05
2.00
0.99
1.00
0.95
1.02
9.12
8
6
4
2
ppm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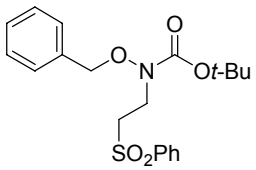
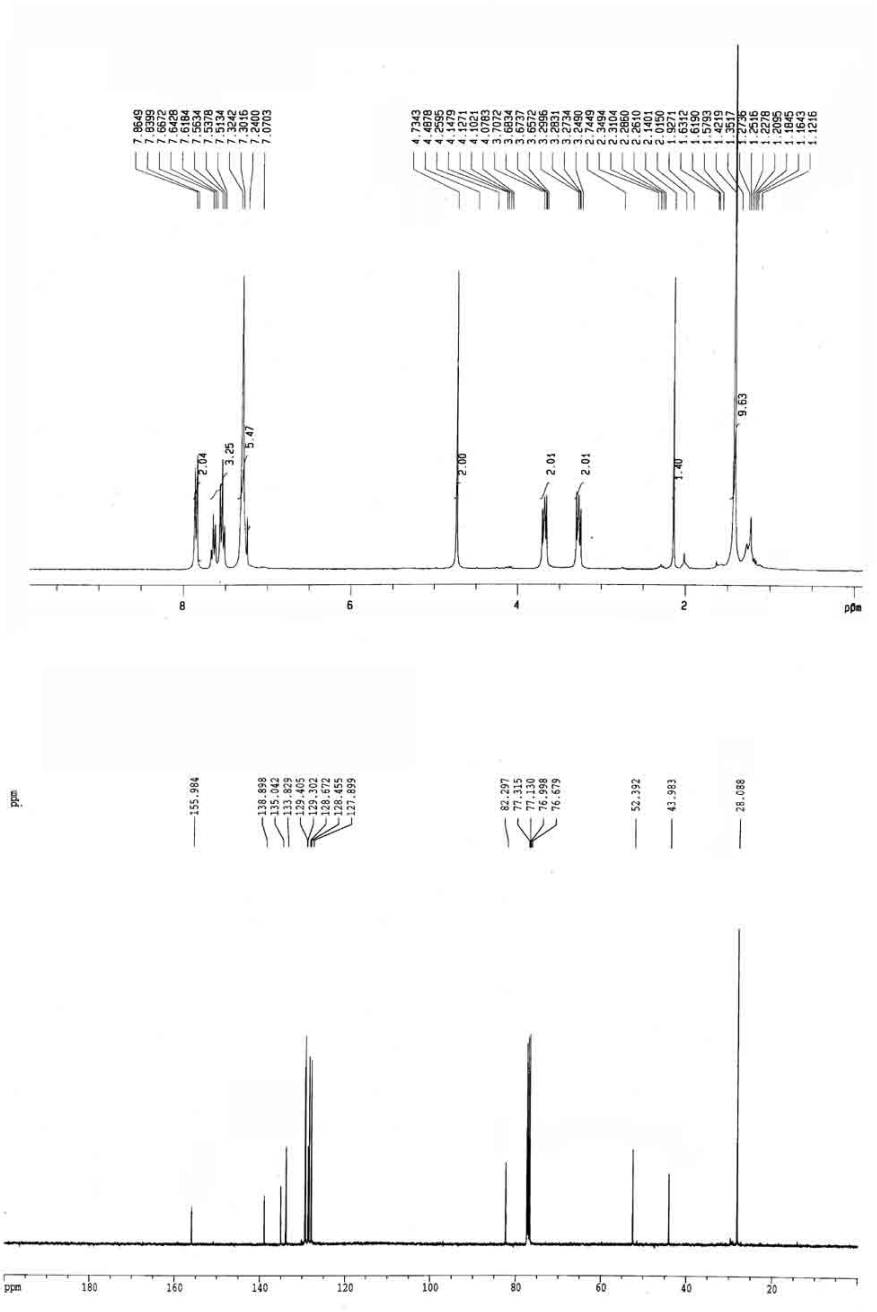
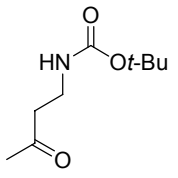
143.39
135.09
131.02
129.49
129.24
128.53
128.44
123.91
82.17
77.41
77.19
77.00
76.58
53.90
43.31
31.85
28.53
28.16
150
100
50
ppm

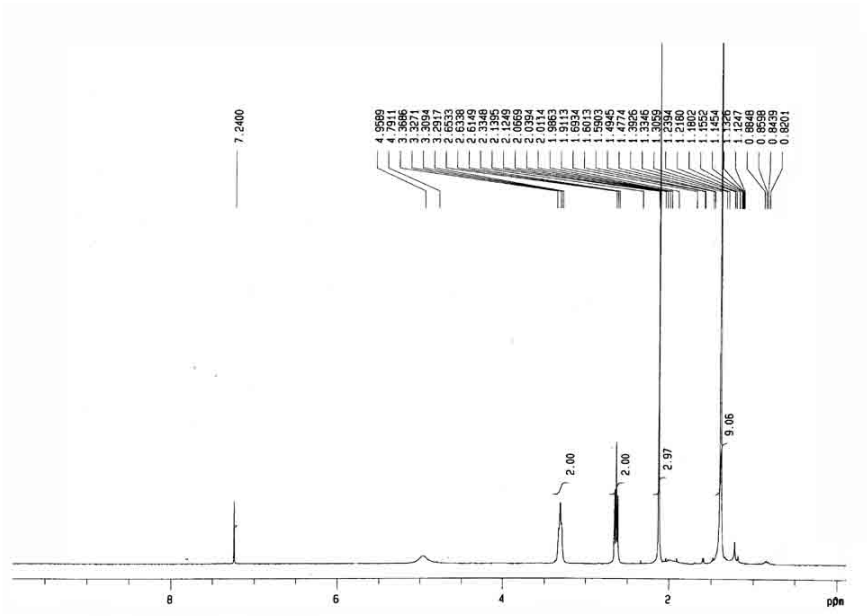

O
O
N
O*t*-Bu
SO_2Ph

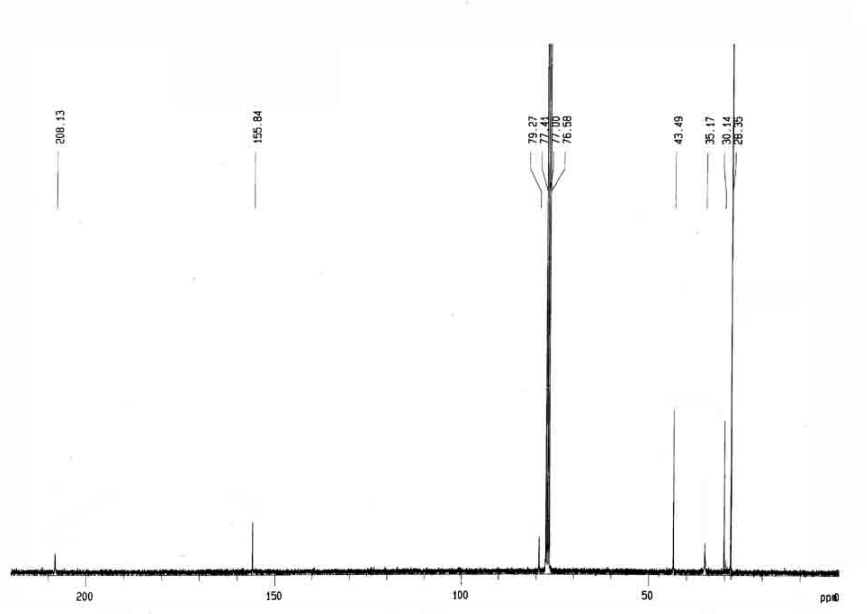
7l

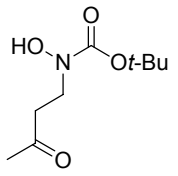

2.04
3.25
5.47
2.00
2.01
2.01
1.40
9.63
8
6
4
2
ppm
ppm
155.984
138.898
135.042
133.829
129.405
129.302
128.672
128.455
127.899
82.297
77.315
77.130
76.958
76.679
52.392
43.983
28.088
ppm
180
160
140
120
100
80
60
40
20


O
HN
Ot-Bu
O

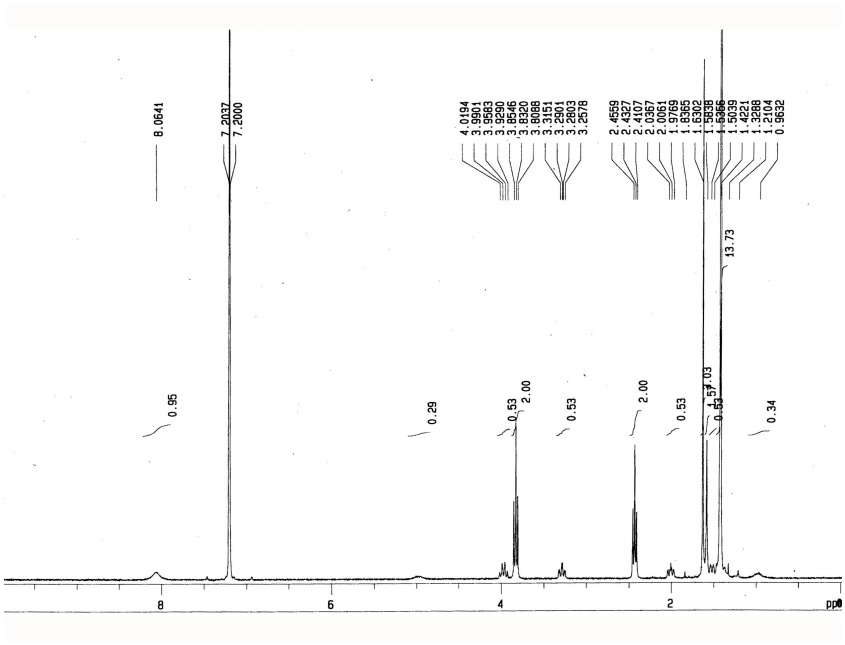
8c


7.2400
2.00
2.00
2.97
9.06
8
6
4
2
ppm


208.13
155.84
79.27
77.41
77.00
76.58
43.49
35.17
30.14
28.35
200
150
100
50
ppm


HO
N
O
Ot-Bu
O

9c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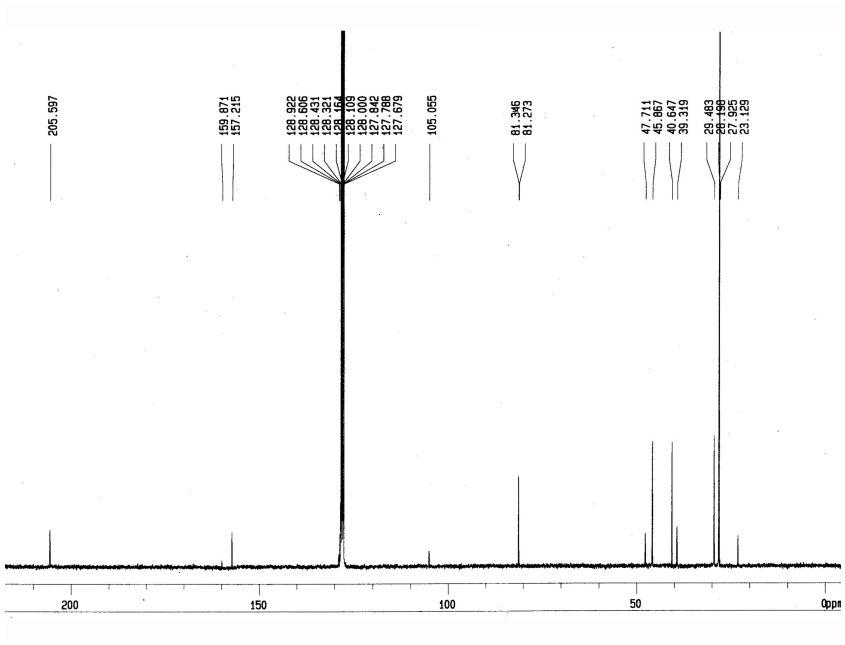
7.2037
7.2000
4.0194
3.9901
3.9583
3.9290
3.8546
3.8330
3.8088
3.3151
3.2901
3.2603
3.2578
2.4659
2.4327
2.4107
2.3957
2.0061
1.9769
1.8365
1.6302
1.5838
1.5636
1.5038
1.4221
1.3288
1.2104
0.9632
13.73
0.95
0.29
0.53
2.00
0.53
2.00
0.53
4.57
3.03
0.34
8
6
4
2
ppm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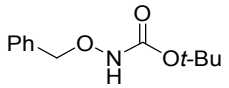
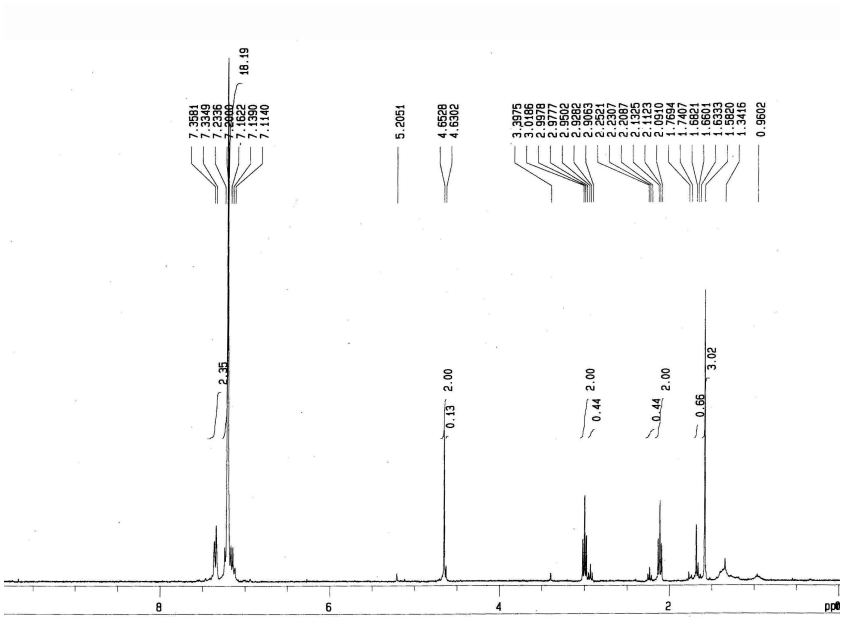
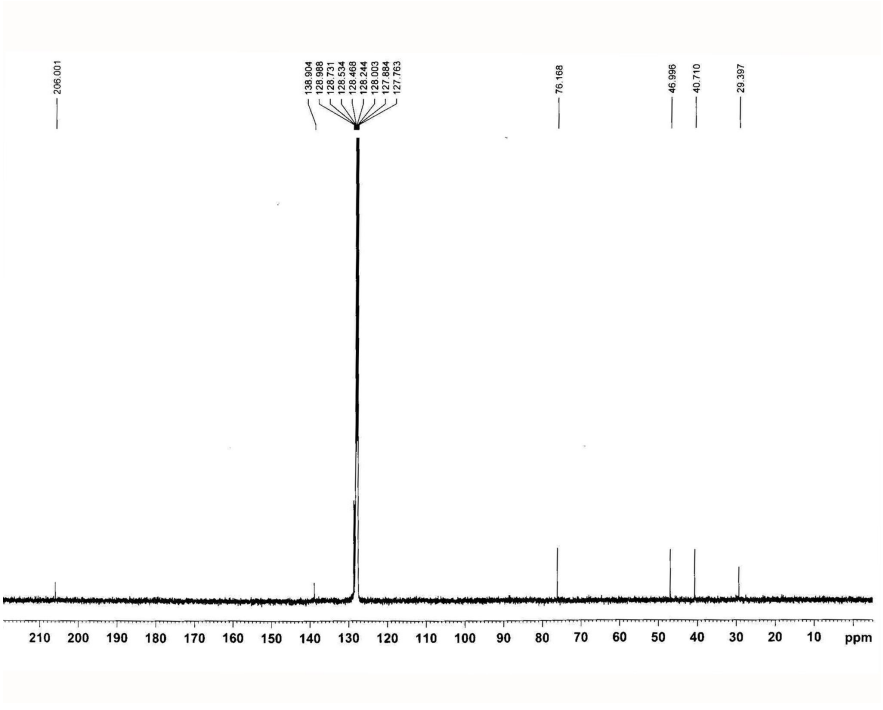
159.871
157.215
128.922
128.606
128.471
128.321
128.164
128.109
128.000
127.842
127.788
127.679
105.055
81.346
81.273
47.711
45.867
40.647
39.319
28.483
28.196
27.925
23.129
200
150
100
50
0ppm


Ph
O
N
H
O
O
Ot-Bu

10c


7.3581
7.3349
7.2336
7.1622
7.1390
7.1140
18.19
5.2051
4.6528
4.6302
3.3975
3.0186
2.9978
2.9777
2.9572
2.9282
2.9063
2.2521
2.2307
2.2097
2.1735
2.1123
2.0910
1.7694
1.7407
1.6821
1.6621
1.6333
1.5820
1.3416
0.9602
2.35
2.00
0.13
2.00
0.44
2.00
0.44
0.66
3.02
8
6
4
2
ppm


206.001
138.904
129.066
128.731
128.534
128.468
128.244
127.015
127.884
127.763
76.168
46.996
40.710
29.397
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm