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for DOI: 10.1055/s-0037-1609082

Thieme

Cycloaddition of Benzyne with Alkoxy-Substituted Pyrroline-*N*-oxides: Unexpected Rearrangement to an *N*-Phenylpyrrole

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Supporting Information

General Information. Reactions requiring anhydrous conditions were carried out under nitrogen, and the solvents were appropriately dried before use. R_f values refer to TLC on 0.25 mm silica gel plates with the same eluant indicated for column chromatography unless otherwise stated. Melting points (Mp) were determined on a Thiele Electrothermal apparatus. Polarimetric measurements were performed on a JASCO DIP-370 polarimeter. NMR spectra were measured on Varian Gemini (^{1}H, 200 MHz, ^{13}C, 50 MHz), Varian Mercury (^{1}H, 400 MHz, ^{13}C, 100 MHz) and Varian INOVA (^{1}H, 400 MHz, ^{13}C, 100 MHz), nuclear magnetic resonance spectrometers. ^{1}H and ^{13}C NMR data are reported in δ (ppm) relative to $CDCl_3$ (7.26 and 77.0 ppm), and peak assignments were made on the basis of ^{1}H–^{1}H COSY, HSQC and HMBC experiments. IR spectra were recorded with a Perkin-Elmer Spectrum BX FT-IR System spectrophotometer. Elemental analyses were performed with a Perkin-Elmer 2400 analyzer. MS (ESI): were recorded on a LCQ Fleet Ion Trap Mass Spectrometer with Surveyor Plus LC System (Thermo Scientific) operating in positive ($^+$ESI): and negative ($^-$ESI): ion mode by direct infusion of a sample solution in methanol or acetonitrile. Accurate mass spectra were recorded on a LTQ-Orbitrap high-resolution mass spectrometer (Thermo, San Jose, CA, USA), equipped with a conventional ESI source. Chromatographic purifications were performed on silica gel 60 (0.040–0.063 mm, 230–400 mesh ASTM, Merk) using flash-column technique.

(1*R*,2*R*,9b*R*)- and (1*R*,2*R*,9b*S*)-1,2-Di-*tert*-butoxy-1,2,3,9b-tetrahydrobenzo[*d*]pyrrolo[1,2-*b*]isoxazole [(3a) and (4a)], and 2-(3-(*tert*-butoxy)-1-phenyl-1*H*-pyrrol-2-yl)phenol (5a). A mixture of **1a**[1] (300 mg, 1.3 mmol), **2** (0.476 mL, 1.96 mmol) and Bu_4NF (1 M in THF, 1.55 mL) in anhydrous DMF (12 mL) was stirred at room temperature for 2.5 h. DMF was evaporated under a flow of nitrogen. Purification by chromatography on silica gel (eluent: EtOAc/petroleum ether from 1:99 to 5:95) of the crude mixture afforded the two diastereomeric cycloadducts **3a** (154 mg, 39% yield) and **4a** (116 mg, 29% yield) as pale yellow oils and pyrrole **5a** (51 mg, 13% yield) as a pale yellow solid.

3a: R_f = 0.33 (EtOAc/petroleum ether 1:16). $[\alpha]_D^{24}$ = –90.6 (c = 0.25, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.25 (dm, *J* = 7.4 Hz, 1H, 9-H), 7.17-7.12 (m, 1H, 7-H), 6.90 (pseudo dt, *J* = 0.9, 7.4 Hz, 1H, 8-H), 6.73 (br d, *J* = 8.0 Hz, 1H, 6-H), 4.77 (br s, 1H, 9b-H), 4.13-4.10 (m, 1H, 1-H), 3.96 (ddd, *J* = 6.0, 4.9, 3.6 Hz, 1H, 2-H), 3.58 (dd, *J* = 11.6, 4.9 Hz, 1H, 3-Ha), 3.16 (ddm, *J* = 11.6, 6.0

Hz, 1H, 3-Hb), 1.29 (s, 9H, CH_3x3), 1.06 (s, 9H, CH_3x3) ppm. ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 156.3 (s; C-5a), 128.5 (d; C-7), 127.1 (s; C-9a), 123.4 (d; C-9), 120.9 (d; C-8), 107.1 (d; C-6), 82.0 (d; C-1), 76.6 (d; C-2), 75.6 (d; C-9b), 74.5 (s, *C*Me_3), 73.6 (s, *C*Me_3), 62.1 (t; C-3), 28.7 (q; 3C, CH_3x3), 28.3 (q; 3C, CH_3x3) ppm. IR ($CDCl_3$): *v* = 2977, 2871, 1597, 1480, 1456, 1390, 1365, 1253, 1190, 1099, 1079 cm^{-1}. MS ($^+$ESI): *m/z* = 306 $[M+H]^+$; 250 $[M+H–(isobutene)]^+$; 194 $[M+H–2(isobutene)]^+$. $C_{18}H_{27}NO_3$ (305.41): calcd. C, 70.79; H, 8.91; N, 4.59; found C, 70.56; H, 8.69; N, 4.98.

4a: R_f = 0.23 (EtOAc/petroleum ether 1:16). $[\alpha]_D^{21}$ = –12.7 (c = 0.22, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.32 (br d, *J* = 7.5 Hz, 1H, 9-H), 7.17 (pseudo tm, *J* = 8.0 Hz, 1H, 7-H), 6.91 (pseudo dt, *J* = 0.8, 7.4 Hz, 1H, 8-H), 6.76 (br d, *J* = 8.1 Hz, 1H, 6-H), 4.87 (d, *J* = 6.8 Hz, 1H, 9b-H), 4.24 (pseudo t, *J* = 7.2 Hz, 1H, 1-H), 3.82 (pseudo q, *J* = 7.9 Hz, 1H, 2-H), 3.49 (dd, *J* = 14.0, 7.6 Hz, 1H, 3-Ha), 3.23 (dd, *J* = 14.0, 8.6 Hz, 1H, 3-Hb), 1.28 (s, 9H, CH_3x3), 1.11 (s, 9H, CH_3x3) ppm. ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 157.2 (s; C-5a), 128.4 (d; C-7), 125.9 (d; C-9), 125.2 (s; C-9a), 120.8 (d; C-8), 107.2 (d; C-6), 77.6 (d; C-1), 74.3 (s, *C*Me_3), 73.7 (s, *C*Me_3), 73.0 (d; C-2), 68.4 (d; C-9b), 62.5 (t; C-3), 28.5(q; 6C, CH_3x6) ppm. IR ($CDCl_3$): *v* = 2977, 2935, 1593, 1474, 1458, 1390, 1365, 1236, 1192, 1119 cm^{-1}. MS (ESI): *m/z* = 306 $[M+H]^+$; 250 $[M+H–(isobutene)]^+$; 194 $[M+H–2(isobutene)]^+$. $C_{18}H_{27}NO_3$ (305.41): calcd. C, 70.79; H, 8.91; N, 4.59; found C, 70.51; H, 9.12; N, 4.56.

5a: R_f = 0.35 (EtOAc/petroleum ether 1:32), one orange spot with *p*-anisaldehyde stain. Mp = 110-112 °C. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.18 (s, 1H, OH, disappears on addition of D_2O), 7.30-7.25 (m, 2H, H_{Ar}), 7.24-7.18 (m, 1H, H_{Ar}), 7.11-7.01 (m, 4H, H_{Ar}), 6.84 (d, *J* = 3.2 Hz, 1H, 5-H), 6.60-6.51 (m, 2H, H_{Ar}), 6.14 (d, *J* = 3.2 Hz, 1H, 4-H), 1.23 (s, 9H, CH_3x3) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 153.7 (s; C_{Ar}), 140.7 (s; C_{Ar}), 139.5 (s; C_{Ar}), 130.8 (d; CH_{Ar}), 128.9 (d; 2C, CH_{Ar}), 128.0 (d; CH_{Ar}), 126.3 (d; CH_{Ar}), 125.3 (d; 2C, CH_{Ar}), 122.4 (d; C-5), 120.9 (s; C_{Ar}), 119.6 (d; CH_{Ar}), 119.0 (s; C_{Ar}), 118.3 (d; CH_{Ar}), 105.1 (d; C-4), 81.6 (s; *C*Me_3), 28.0 (q; 3C, CH_3x3) ppm. C/H coupled ^{13}C NMR ($CDCl_3$, 100 MHz): δ: (selection of signals) 122.4 (dd, *J* = 187.6, 7.0 Hz; C-5), 105.1 (dd, *J* = 173.3, 7.2 Hz; C-4) ppm. IR ($CDCl_3$): *v* = 3255 (broad), 3075, 2981, 2934, 1599, 1556, 1502, 1352, 1235, 1164 cm^{-1}. MS ($^+$ESI): *m/z* = 330 $[M+Na]^+$. MS ($^-$ESI): *m/z* = 307 $[M]^-$. HRMS ($^+$ESI): *m/z* $[MH]^+$ calcd for $C_{20}H_{22}NO_2^+$: 308.16451; found: 308.16444.

2-((2*R*,3*R*,4*R*)-3,4-Di-*tert*-butoxypyrrolidin-2-yl)phenol (6a). A mixture of isoxazolidine **3a** (167 mg, 0.55 mmol) and zinc powder (1.43 g) in AcOH:H_2O [1:1 (v/v), 16.4 mL) was heated in an oil bath at 70 °C for 2 h. The reaction mixture was diluted with MeOH, filtered through cotton wool and then concentrated under reduced pressure. The residue was dissolved in CH_2Cl_2 and the solution was basified to pH = 9 with a saturated aq. $NaHCO_3$ solution and solid Na_2CO_3 at 0 °C. The two phases were separated. The aqueous phase was sequentially extracted with CH_2Cl_2 (3x30 mL) and EtOAc (2x30 mL). The combined organic phases were washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. Purification of the crude product by chromatography on silica gel [eluent: CH_2Cl_2/MeOH (1% NH_4OH) 98:2] afforded **6a** (127 mg, 0.41 mmol) in 75% yield as a white solid.

6a: R_f = 0.31. Mp = 124-125 °C. $[\alpha]_D^{21}$ = –43.4 (c = 0.43, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.13 (pseudo dt, *J* = 1.7, 7.7 Hz, 1H, H_{Ar}), 7.00 (dd, *J* = 7.5, 1.7 Hz, 1H, H_{Ar}), 6.80 (dd, *J* = 8.1, 1.1 Hz, 1H, H_{Ar}), 6.74 (pseudo dt, *J* = 1.1, 7.4 Hz, 1H, H_{Ar}), 4.08 (dd, *J* = 7.9, 5.3 Hz, 1H, 3-H), 3.99 (dd, *J* = 7.4, 5.3 Hz, 1H, 4-H), 3.94 (d, *J* = 7.9 Hz, 1H, 2-H), 3.30 (dd, *J* = 10.6, 7.4 Hz, 1H, 5-Ha), 3.01 (dd, *J* = 10.6, 4.4 Hz, 1H, 5-Hb), 1.19 (s, 9H, CH_3x3), 0.93 (s, 9H, CH_3x3) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 158.0 (s; C_{Ar}), 129.6 (d; CH_{Ar}), 128.7 (d; CH_{Ar}), 123.3 (s; C_{Ar}), 118.4 (d; CH_{Ar}), 116.7 (d; CH_{Ar}), 80.7 (d; C-3), 76.3 (d; C-4), 74.6 (s, *C*Me_3), 73.8 (s, *C*Me_3), 66.6 (d; C-2), 50.9 (t; C-5), 28.7 (q; 3C, CH_3x3), 28.6 (q; 3C, CH_3x3) ppm.IR ($CDCl_3$): *v* = 2977, 2935, 1589, 1489, 1392, 1367, 1257, 1190, 1106, 1068 cm^{-1}. MS ($^+$ESI): *m/z* = 308 $[M+H]^+$. MS ($^-$ESI): *m/z* = 306 $[M–H]^-$. $C_{18}H_{29}NO_3$ (307.43): calcd. C, 70.32; H, 9.51; N, 4.56; found C, 70.53; H, 9.66; N, 4.17.

2-(3-(Benzoyloxy)-1-phenyl-1*H*-pyrrol-2-yl)phenol (5b), 2-((2*R*,3*R*,4*R*)- and 2-((2*S*,3*R*,4*R*)-3,4-bis(benzoyloxy)pyrrolidin-2-yl)phenol [(6b) and (7b)]. A mixture of **1b**[6c] (300 mg, 0.92 mmol), **2** (0.336 mL, 1.38 mmol) and Bu_4NF (1 M in THF, 1.1 mL) in anhydrous DMF (9 mL) was stirred at room temperature for 2.5 h. DMF was evaporated under a flow of nitrogen. The residue was filtered through a short pad of silica gel [eluent: petroleum ether/EtOAc (1% Et_3N), 1:9] to afford a mixture of **3b**, **4b** and **5b** in ca 1.1:1:0.4 ratio (305 mg) that was used in the next step without further purification.

(1*R*,2*R*,9b*R*)-1,2-Bis(benzoyloxy)-1,2,3,9b-tetrahydrobenzo[*d*]pyrrolo[1,2-*b*]isoxazole (**3b**): ^{1}H NMR ($CDCl_3$, 400 MHz): δ = (discernible signals), 5.67 (pseudo dt, *J* = 3.8, 1.1 Hz, 1H, 2-H), 5.61-5.60 (m, 1H, 1-H), 5.27 (br s, 1H, 9b-H), 3.94 (dd, *J* = 15.8, 3.8 Hz, 1H, 3-Hb) ppm. **(1*R*,2*R*,9b*S*)-1,2-bis(benzoyloxy)-1,2,3,9b-tetrahydrobenzo[*d*]pyrrolo[1,2-*b*]isoxazole** (**4b**): ^{1}H NMR ($CDCl_3$, 400 MHz): δ = (discernible signals), 5.89 (dd, *J* = 6.3, 4.5 Hz, 1H, 1-H), 5.77 (pseudo dt, *J* = 4.6, 5.8 Hz, 1H, 2-H), 5.51 (br d, *J* = 6.3 Hz, 1H, 9b-H), 4.08 (dd, *J* = 14.1, 6.0 Hz, 1H, 3-Ha), 3.73 (dd, *J* = 14.1, 5.7 Hz, 1H, 3-Hb) ppm.

AcOH (0.83 mL) and Pd/C (10% in weight, 15 mg) were added to a solution of the cycloaddition reaction mixture (305 mg) in MeOH (10 mL). The reaction mixture was stirred in a H_2 atmosphere (1 atm) at room temperature for 4 h, then filtered through a short pad of Celite® and washing with MeOH. Purification by chromatography on silica gel [eluent: EtOAc (1% Et_3N)/petroleum ether 1:3] afforded the two diastereomeric pyrrolidines **6b** (109 mg, 29% yield) and **7b** (100 mg, 27% yield) as white solids and pyrrole **5b** (25 mg, 8% yield) as a pale yellow solid.

5b: R_f = 0.52 (EtOAc/petroleum ether 1:4), one red spot with *p*-anisaldehyde stain.. Mp = 135-137 °C (dec). ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.15-8.09 (m, 2H, H_{Bz}), 7.62-7.56 (m, 1H, H_{Bz}), 7.49-7.42 (m, 2H, H_{Bz}), 7.31-7.11 (m, 6H, H_{Ar}), 7.03 (d, *J* = 3.2 Hz, 1H, 5-H), 6.93 (dm, *J* = 8.2 Hz, 1H, H_{Ar}), 6.88 (dd, *J* = 7.6, 1.6 Hz, 1H, H_{Ar}), 6.76-6.69 (m, 1H, H_{Ar}), 6.44 (d, *J* = 3.2 Hz, 1H, 4-H), 6.06 (br s, 1H, OH) ppm. ^{13}C-NMR ($CDCl_3$, 100 MHz): δ = 165.8 (s; CO), 154.8 (s; C_{Ar}), 139.6 (s; C_{Ar}), 136.8 (s; C_{Ar}), 133.6 (d; CH_{Bz}), 132.1 (d; CH_{Ar}), 130.3 (d; 2C, CH_{Bz}), 130.1 (d; CH_{Ar}), 129.0 (d; 2C, CH_{Ar}), 128.9 (s; CH_{Bz}), 128.5 (d; 2C, CH_{Bz}), 126.8 (d; CH_{Ar}), 124.9 (d; 2C, CH_{Ar}), 121.6 (d; C-5), 120.1 (d; CH_{Ar}), 117.3 (s; C_{Ar}), 116.4 (s; C_{Ar}), 116.1 (d; CH_{Ar}), 103.3 (d; C-4) ppm. IR ($CDCl_3$): *v* = 3072, 2927, 1726, 1600, 1502, 1356, 1267, 1228, 1068, 1025 cm^{-1}. MS ($^+$ESI): *m/z* = 356 $[M+1]^+$. MS ($^-$ESI): *m/z* = 354 $[M-1]^-$. HRMS ($^+$ESI): *m/z* $[MH]^+$ calcd for $C_{23}H_{18}NO_3^+$: 356.12812; found: 356.12788.

6b: R_f = 0.15 (EtOAc/petroleum ether 1:4). Mp = 162-164 °C. $[\alpha]_D^{27}$ = –57.4 (c = 1.0, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.08-8.03 (m, 2H, H_{Bz}), 8.96-7.91 (m, 2H, H_{Bz}), 7.63-7.58 (m, 1H, H_{Bz}), 7.57-7.52 (m, 1H, H_{Bz}), 7.50-7.44 (m, 2H, H_{Bz}), 7.43-7.37 (m, 2H, H_{Bz}), 7.23 (dd, *J* = 7.6, 1.6 Hz, 1H, H_{Ar}), 7.18 (pseudo dt, *J* = 1.6, 7.7 Hz, 1H, H_{Ar}), 6.88 (dd, *J* = 8.2, 1.2 Hz, 1H, H_{Ar}), 6.80 (pseudo dt, *J* = 1.2, 7.4 Hz, 1H, H_{Ar}), 5.71 (dd, *J* = 4.3, 1.6 Hz, 1H, 3-H), 5.62 (pseudo dt, *J* = 5.5, 1.6 Hz, 1H, 4-H), 4.68 (d, *J* = 4.3 Hz, 1H, 2-H), 3.70 (dd, *J* = 12.1, 5.5 Hz, 1H, 5-Ha), 3.44 (dm, *J* = 12.1 Hz, 1H, 5-Hb) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 165.7 (s; CO), 165.3 (s; CO), 157.8 (s; C_{Ar}), 133.5 (d; CH_{Bz}), 133.3 (d; CH_{Bz}), 129.9 (d; 2C, CH_{Bz}), 129.7 (d; 2C, CH_{Bz}), 129.3 (s; C_{Bz}), 129.2 (s; C_{Bz}), 129.1 (d; CH_{Ar}), 128.5 (d; 2C, CH_{Bz}), 128.4 (d; CH_{Ar}), 128.3 (d; 2C, CH_{Bz}), 121.1 (s; C_{Ar}), 119.2 (d; CH_{Ar}), 117.4 (d; CH_{Ar}), 83.0 (d; C-3), 77.3 (d; C-4), 67.0 (d; C-2), 51.2 (t; C-5) ppm. IR ($CDCl_3$): *v* = 3348, 3065, 2959, 2858, 1719, 1602, 1585, 1491, 1451, 1278, 1258, 1110 cm^{-1}. MS ($^+$ESI): *m/z* = 404 $[M+H]^+$. MS ($^-$ESI): *m/z* = 402 $[M-H]^-$. $C_{24}H_{21}NO_5$ (403.43): calcd. C, 71.45; H, 5.25; N, 3.47; found C, 71.08; H, 5.07; N, 3.44.

7b: R_f = 0.34 (EtOAc/petroleum ether 1:4). Mp = 60-62 °C. $[\alpha]_D^{26}$ = –45.3 (c = 0.5, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.11-8.07 (m, 2H, H_{Bz}), 7.97-7.93 (m, 2H, H_{Bz}), 7.66-7.60 (m, 1H, H_{Bz}), 7.54-7.47 (m, 3H, H_{Bz}), 7.40-7.35 (m, 2H, H_{Bz}), 7.08-7.00 (m, 2H, H_{Ar}), 6.76 (dd, *J* = 8.2, 1.2 Hz, 1H, H_{Ar}), 6.72 (pseudo dt, *J* = 1.2, 7.4 Hz, 1H, H_{Ar}), 5.77 (dd, *J* = 4.7, 1.1 Hz, 1H, 3-H), 5.55 (dm, *J* = 5.1 Hz, 1H, 4-H), 4.97 (d, *J* = 4.7 Hz, 1H, 2-H), 3.85 (dd, *J* = 12.6, 5.1 Hz, 1H, 5-Ha), 3.35 (dd, *J* = 12.6, 2.3 Hz, 1H, 5-Hb) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 165.3 (s; CO), 165.2 (s; CO), 159.2 (s; C_{Ar}), 133.6 (d; CH_{Bz}), 133.2 (d; CH_{Bz}), 129.9 (d; 2C, CH_{Bz}), 129.8 (d; 2C, CH_{Bz}), 129.3 (s; C_{Bz}), 129.1 (s; C_{Bz} + d; CH_{Ar}), 128.6 (d; 2C, CH_{Bz}), 128.5 (d; CH_{Ar}), 128.3 (d; 2C, CH_{Bz}), 118.8 (d; CH_{Ar}), 118.4 (s; C_{Ar}), 117.1 (d; CH_{Ar}), 78.8 (d; C-3), 77.1 (d; C-4), 65.0 (d; C-2), 50.3 (t; C-5) ppm. IR ($CDCl_3$): *v* = 3366, 3065, 2958, 2871, 1721, 1601, 1586, 1492, 1452, 1316, 1260, 1109 cm^{-1}. MS ($^+$ESI): *m/z* = 404 $[M+H]^+$. MS ($^-$ESI): *m/z* = 402 $[M-H]^-$. $C_{24}H_{21}NO_5$ (403.43): calcd. C, 71.45; H, 5.25; N, 3.47; found C, 71.44; H, 5.13; N, 3.37.

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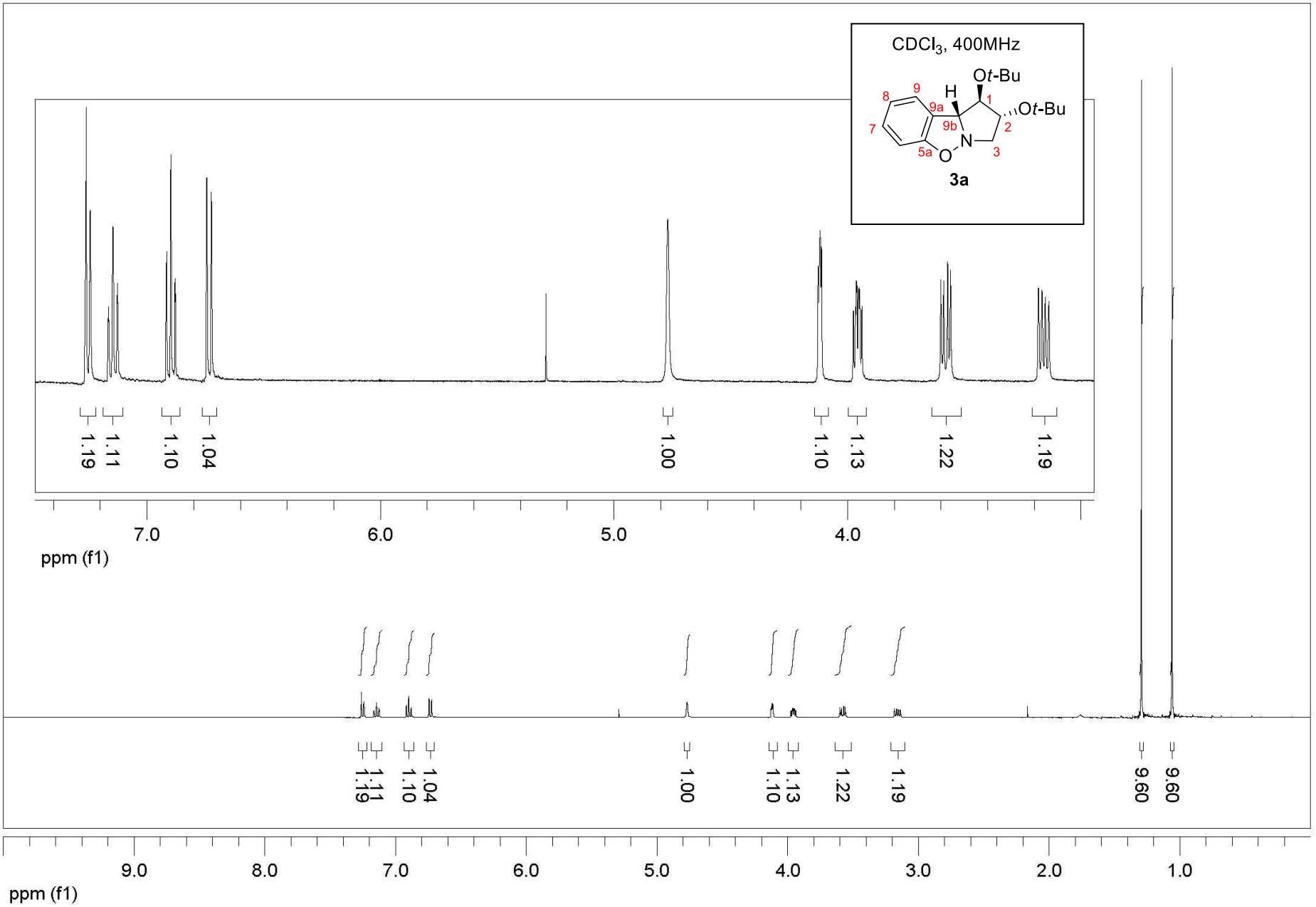
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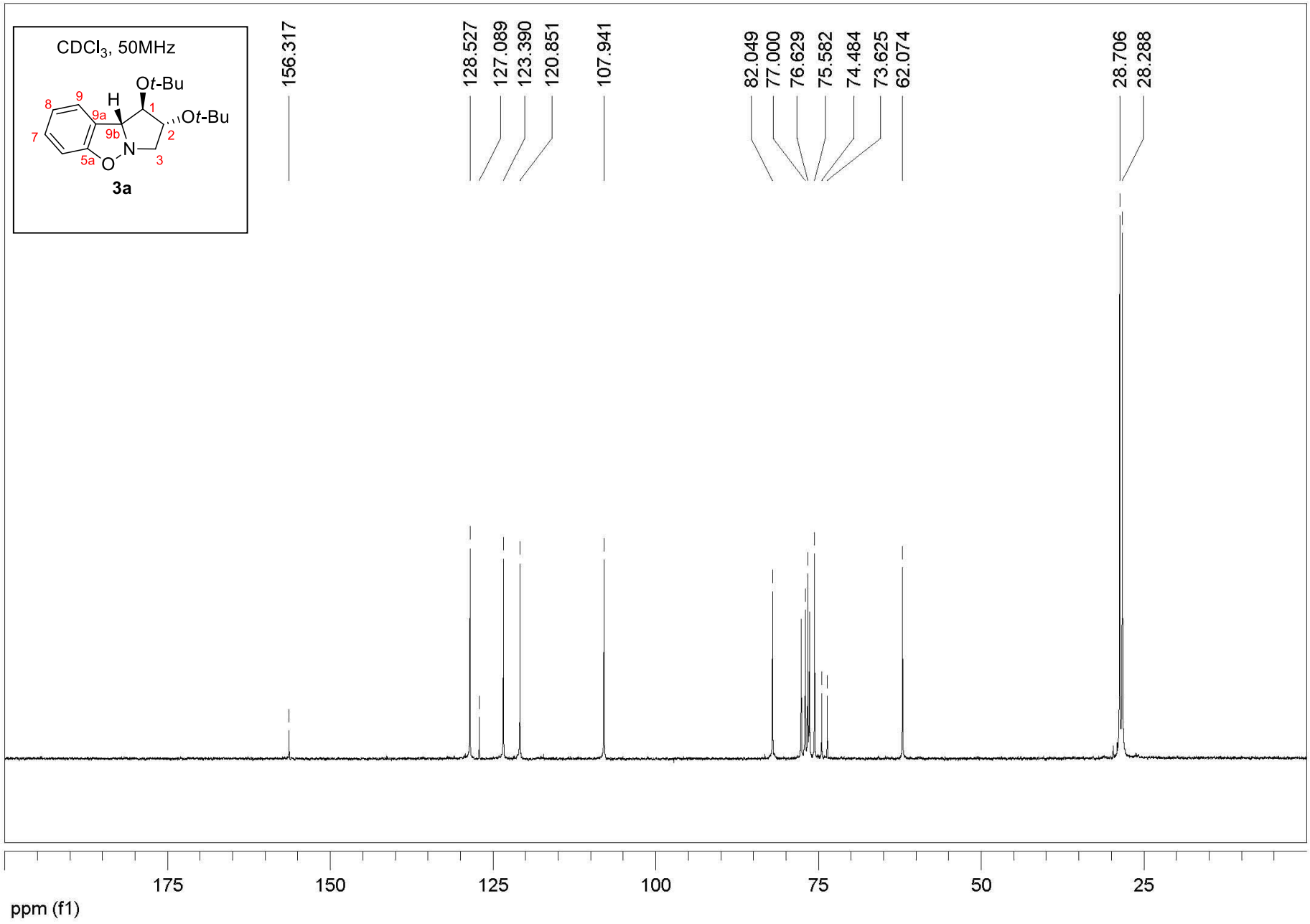
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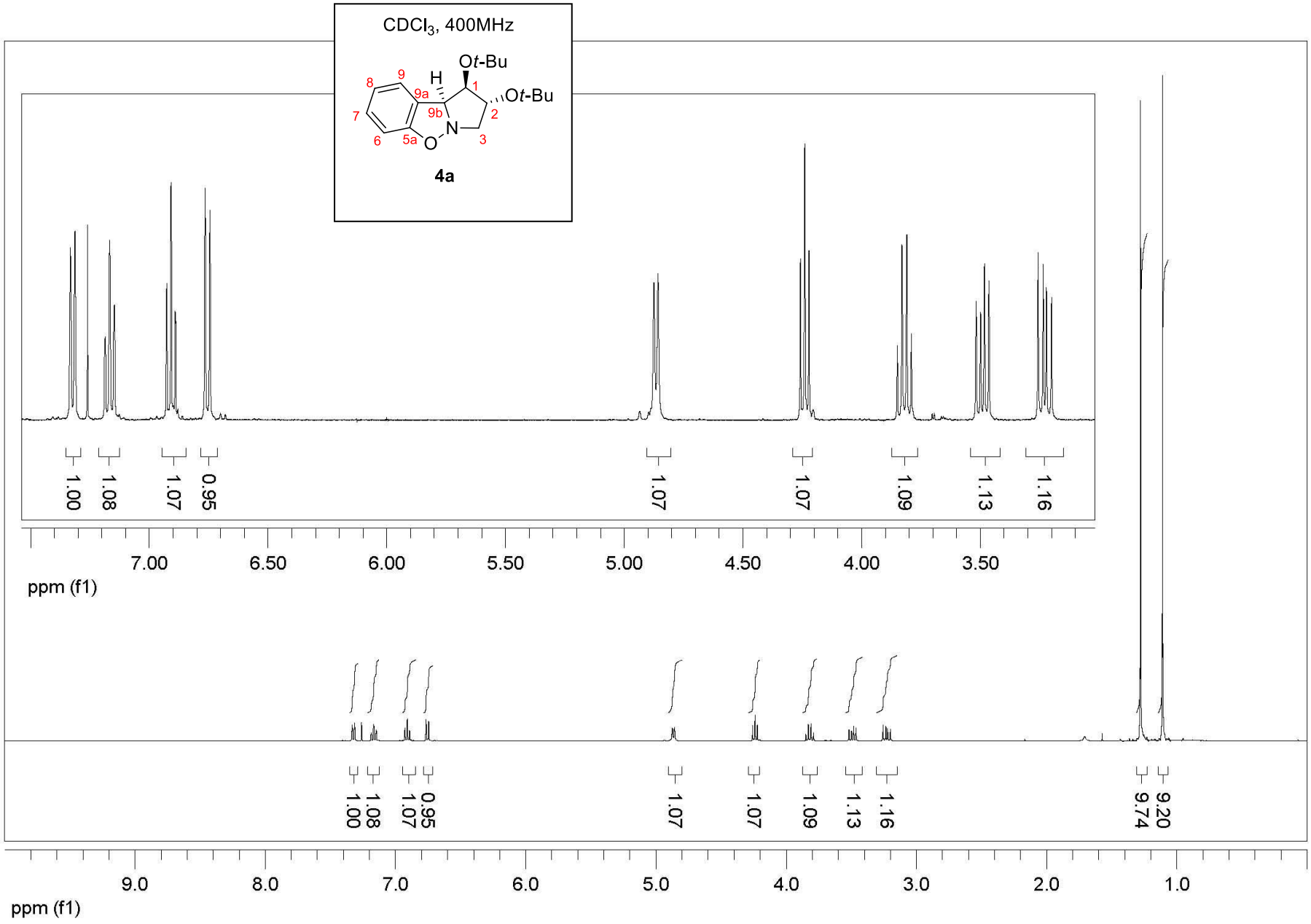
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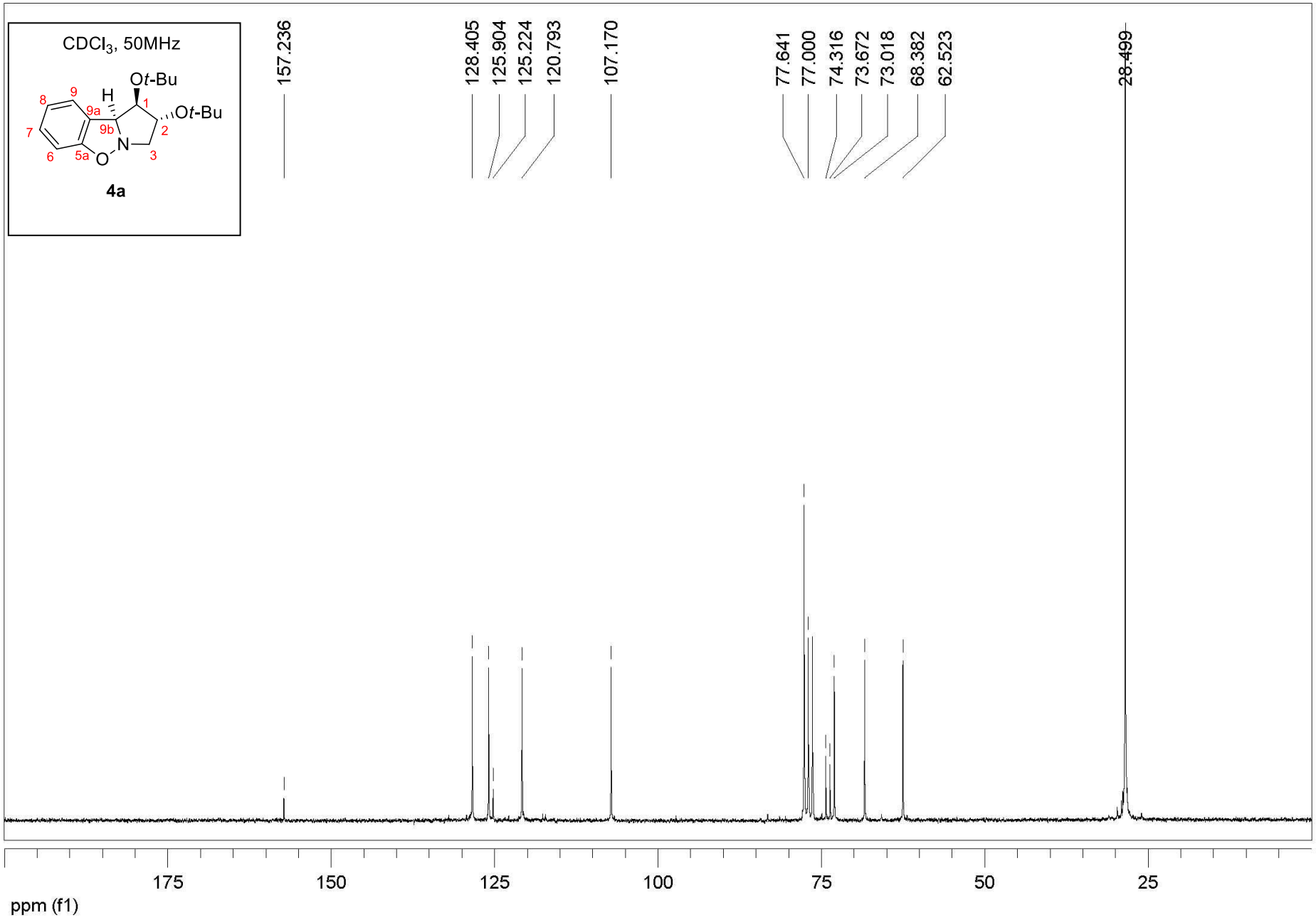
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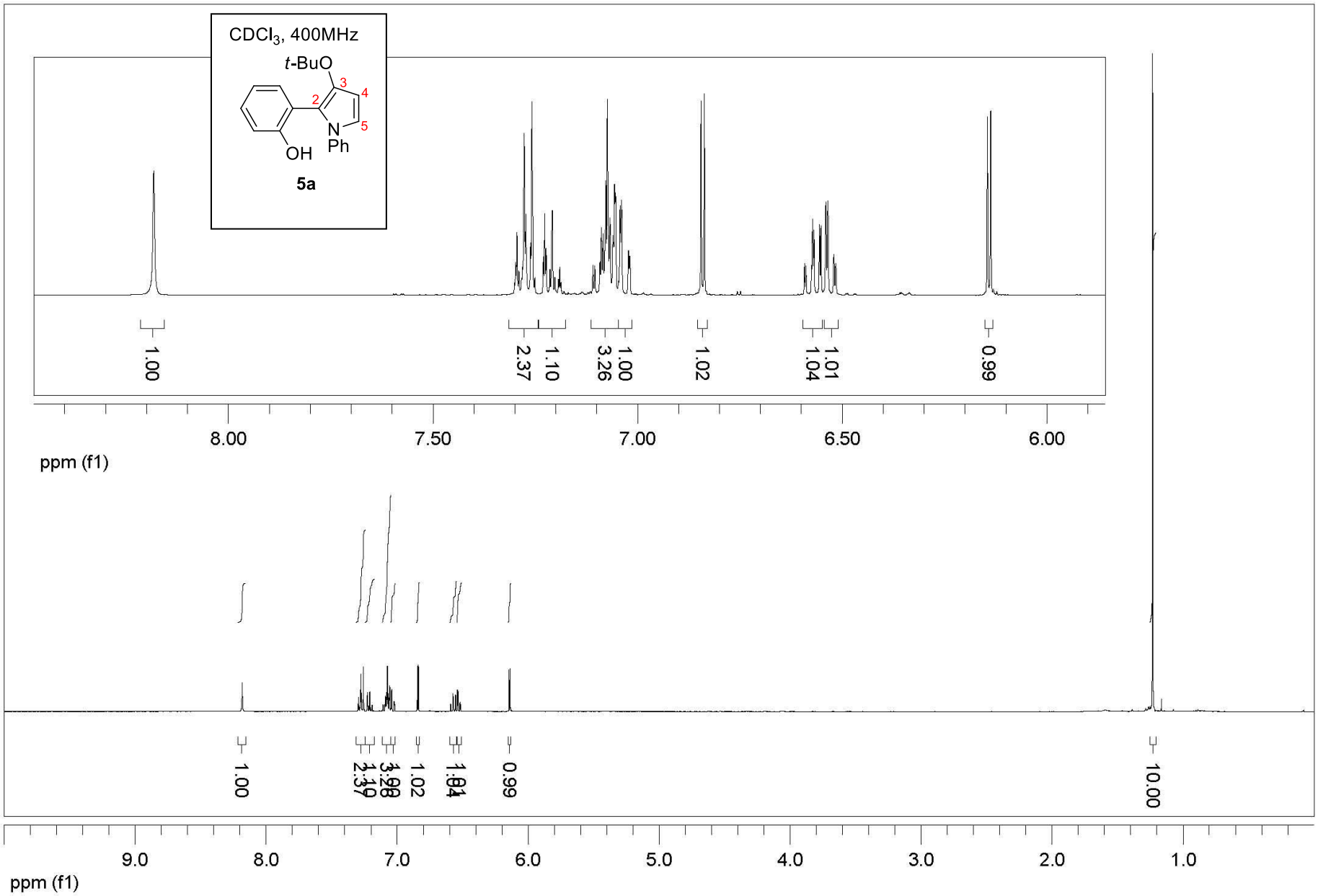
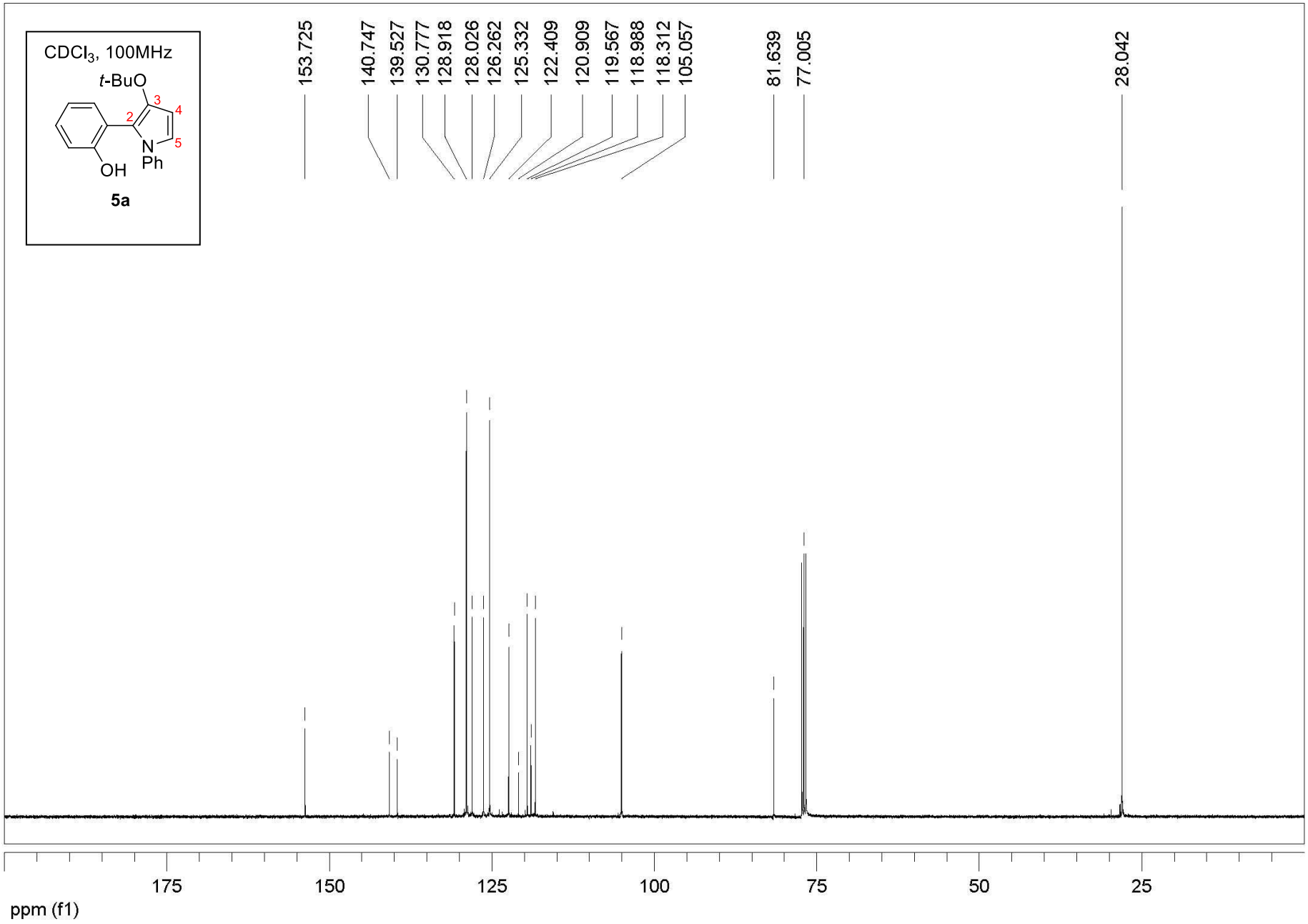
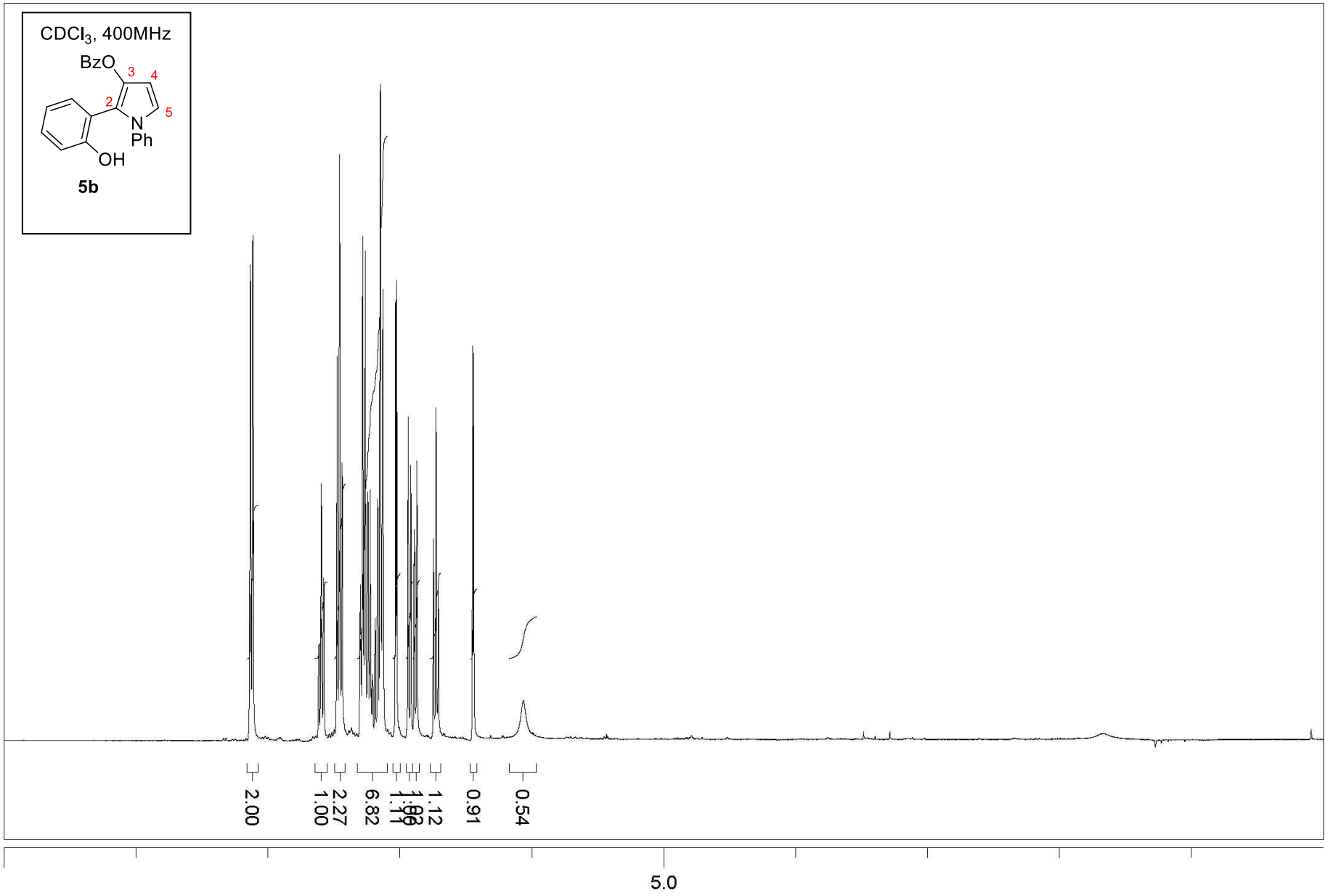
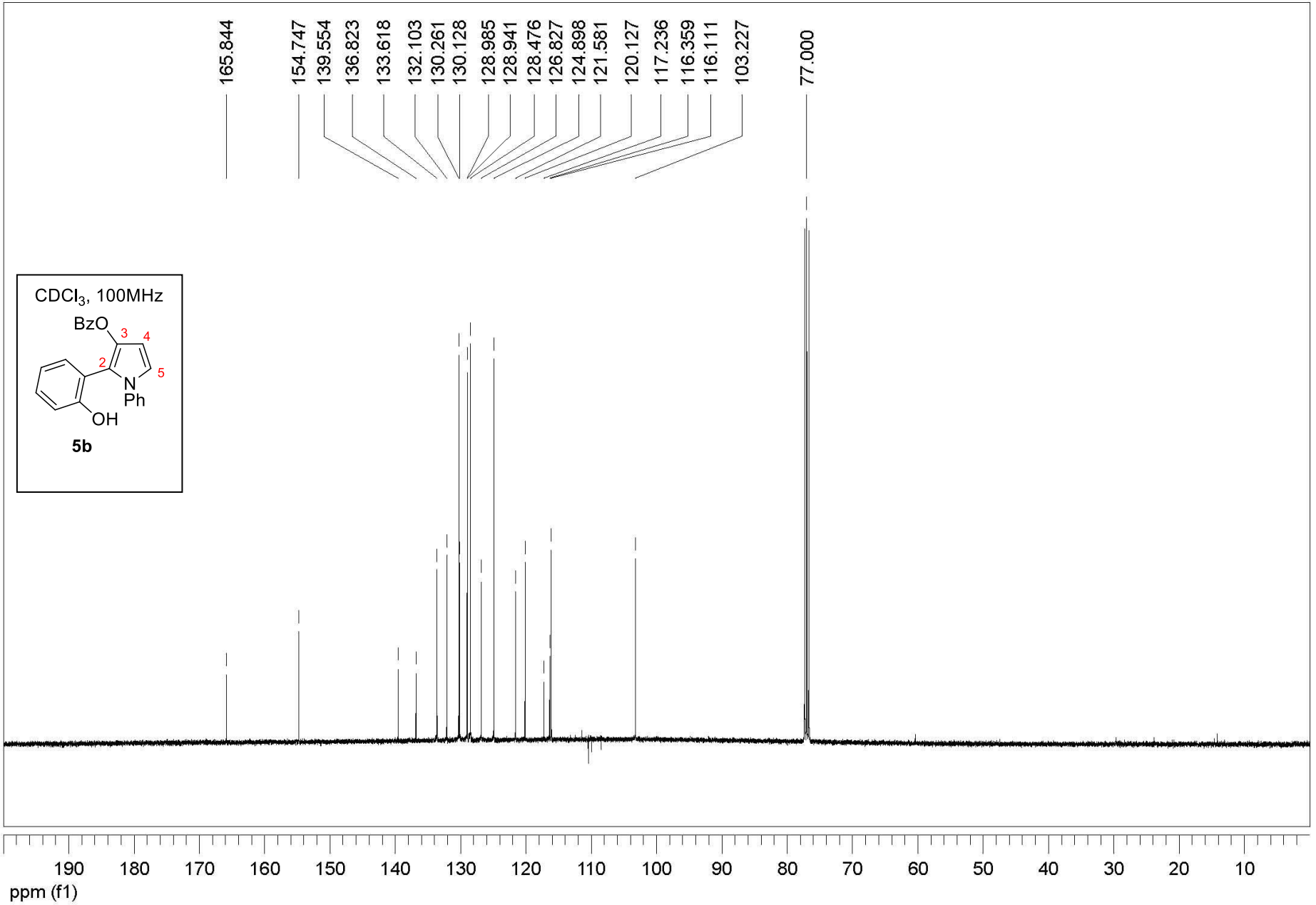
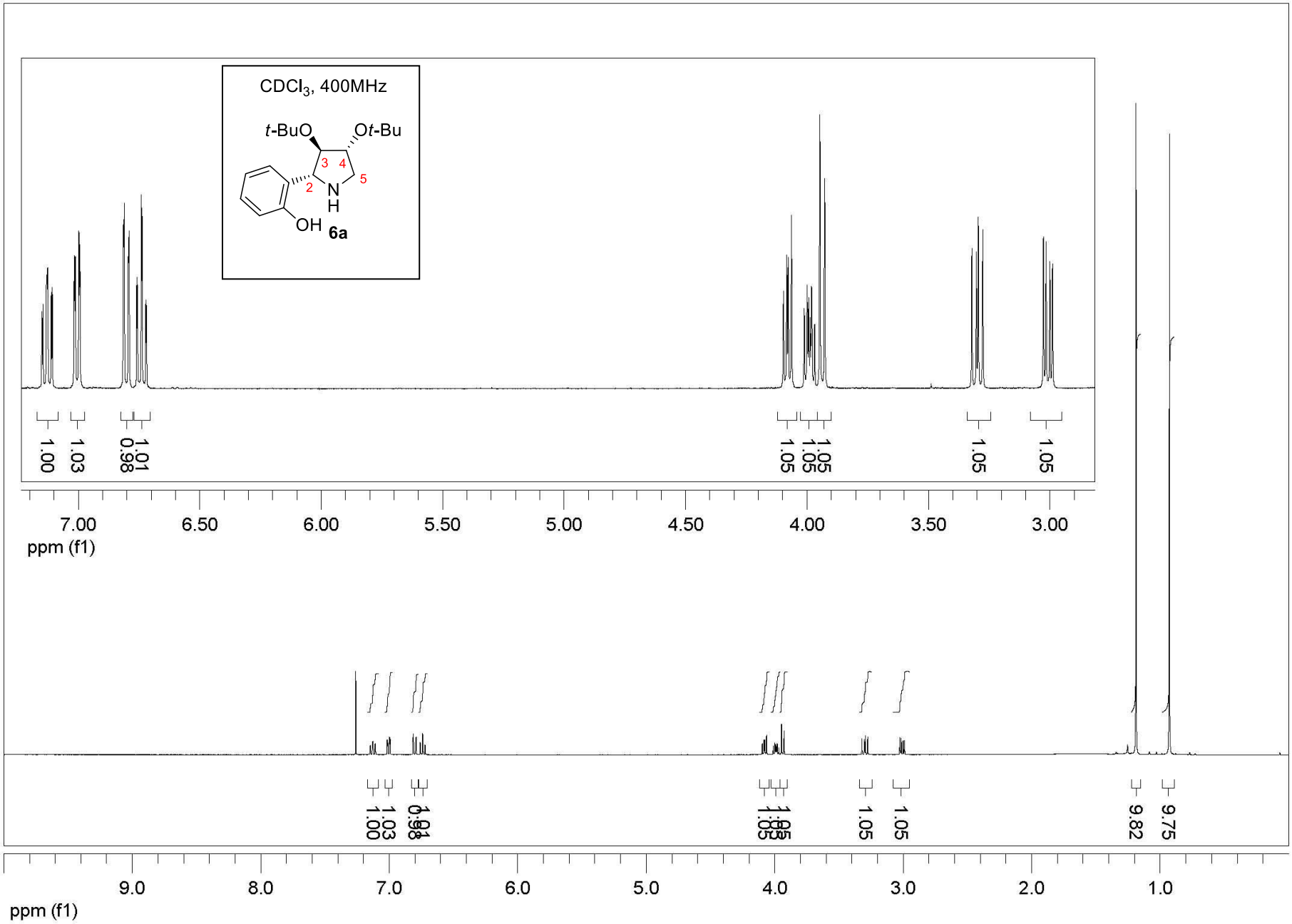
25
ppm (f1)

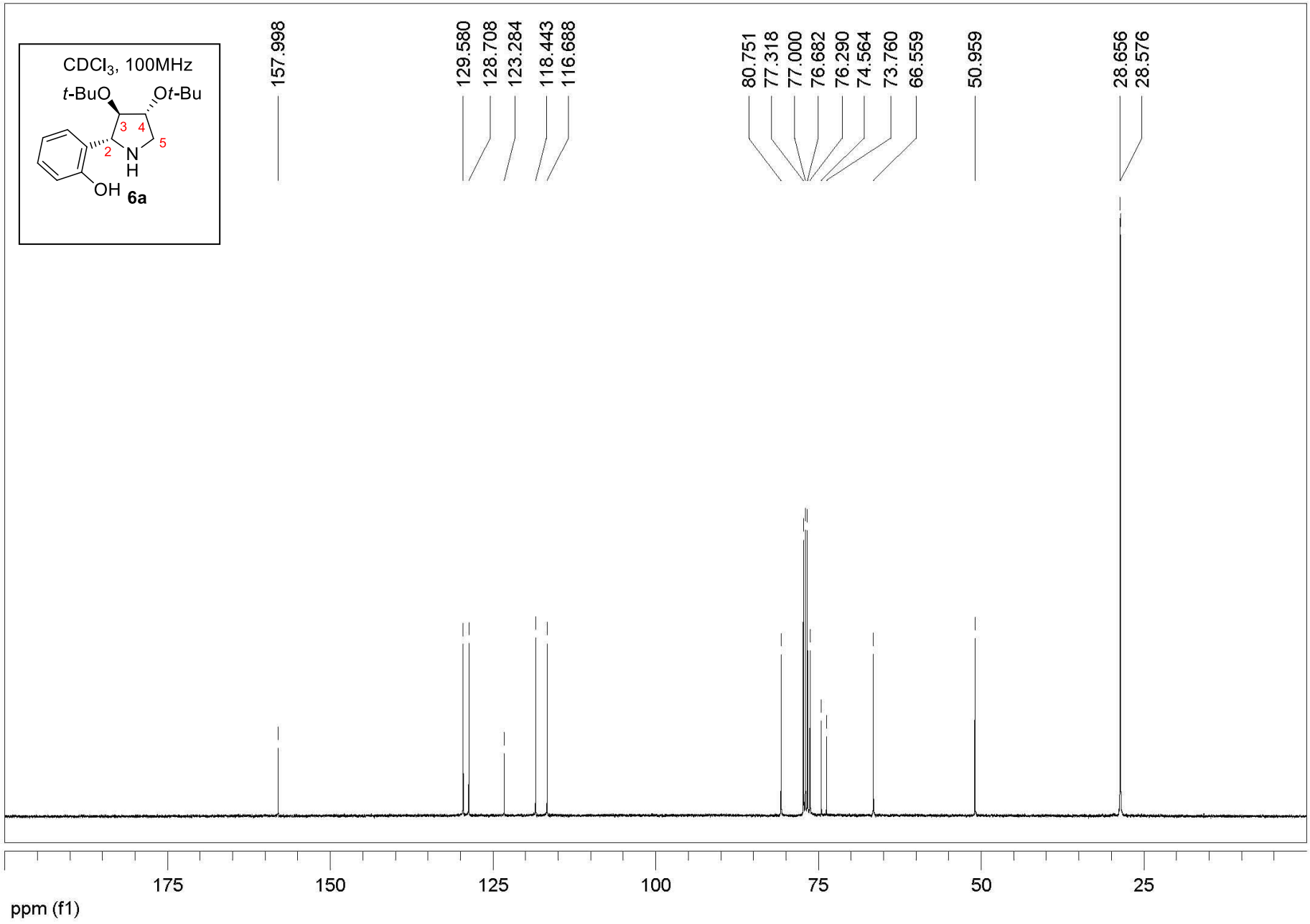

CDCl3, 400MHz
t-BuO
OH
Ph
5a
1.00
2.37
1.10
3.26
1.00
1.02
1.04
1.01
0.99
8.00
7.50
7.00
6.50
6.00
ppm (f1)
1.00
1.02
0.99
10.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)

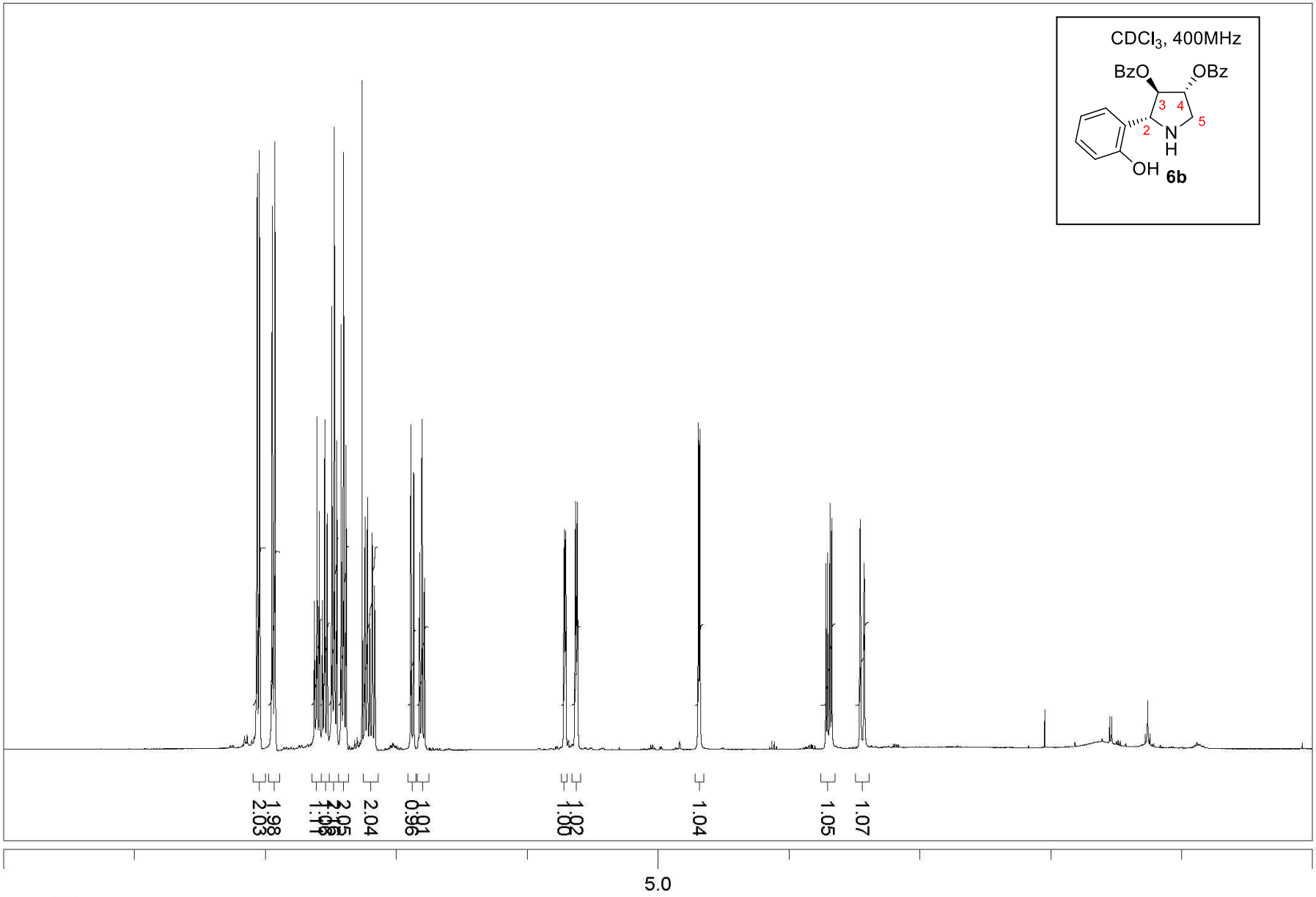

CDCl3, 100MHz
t-BuO
OH
Ph
5a
153.725
140.747
139.527
130.777
128.918
128.026
126.262
125.332
122.409
120.909
119.567
118.988
118.312
105.057
81.639
77.005
28.042
175
150
125
100
75
50
25
ppm (f1)

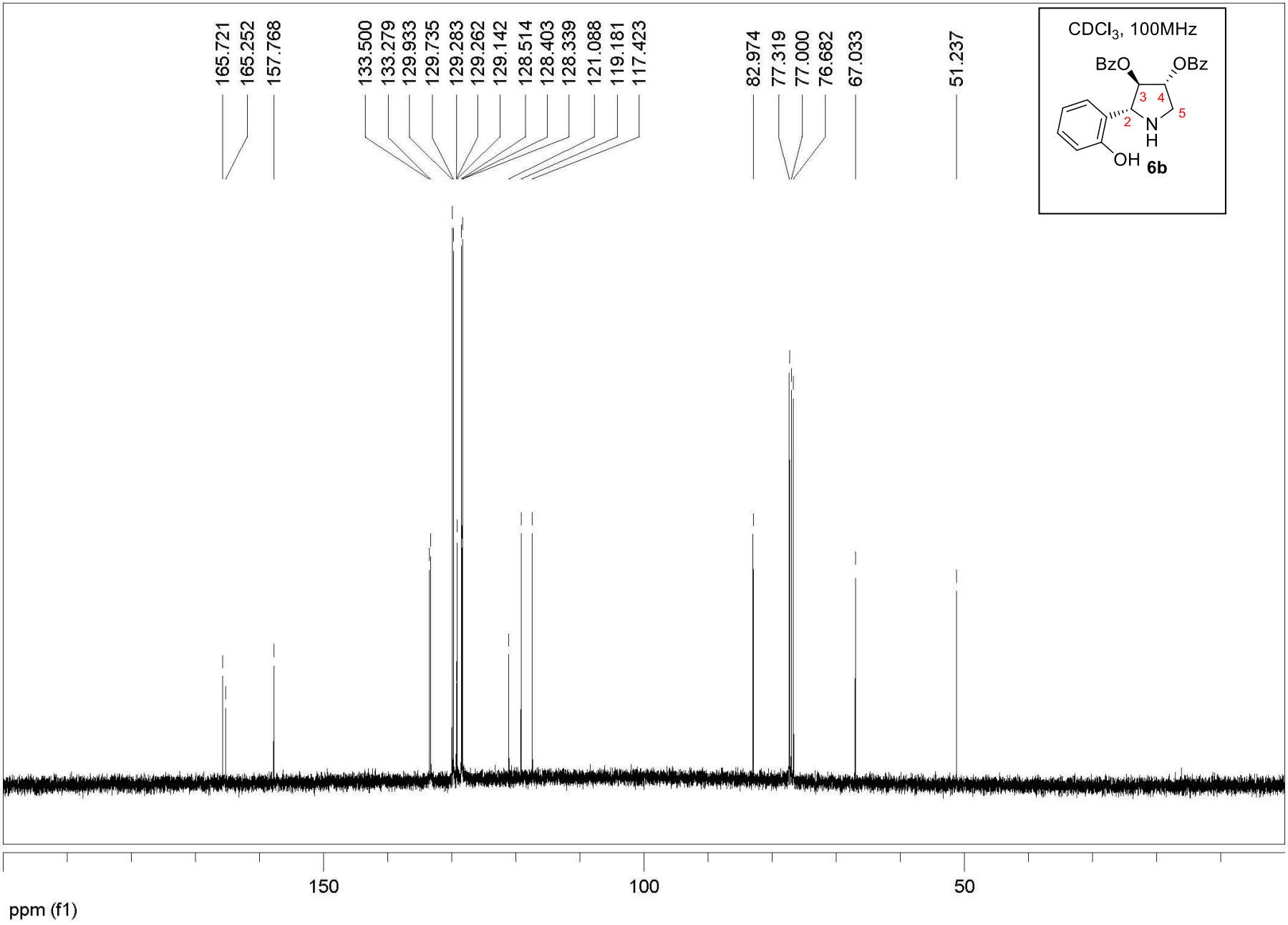

CDCl3, 400MHz
BzO
3
4
2
5
N
Ph
OH
5b
2.00
1.00
2.27
6.82
1.12
0.91
0.54
5.0
ppm (f1)

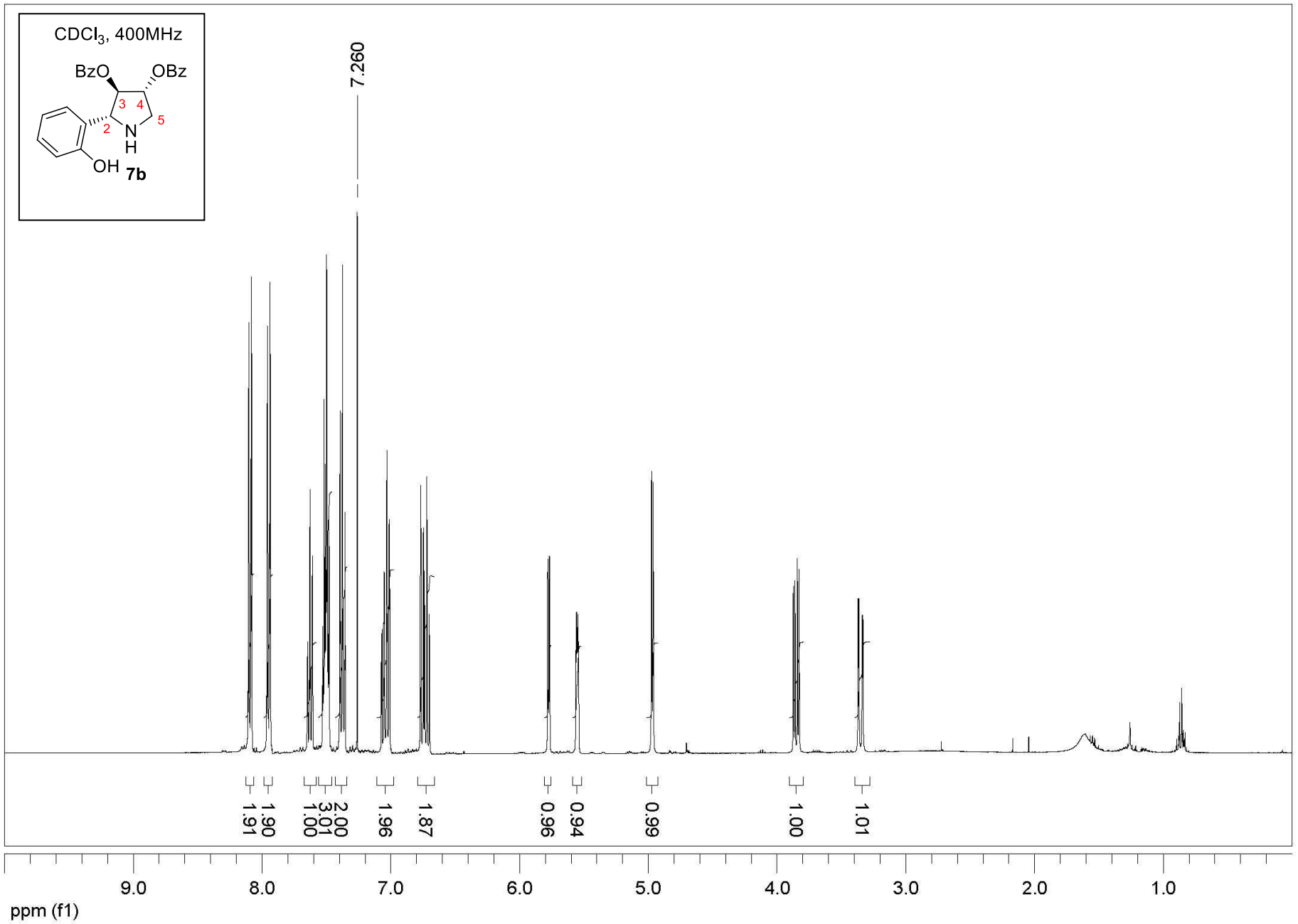

165.844
154.747
139.554
136.823
133.618
132.103
130.261
130.128
128.985
128.941
128.476
126.827
124.898
121.581
120.127
117.236
116.359
116.111
103.227
77.000
CDCl3, 100MHz
BzO
3
4
2
5
N
Ph
OH
5b
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm (f1)

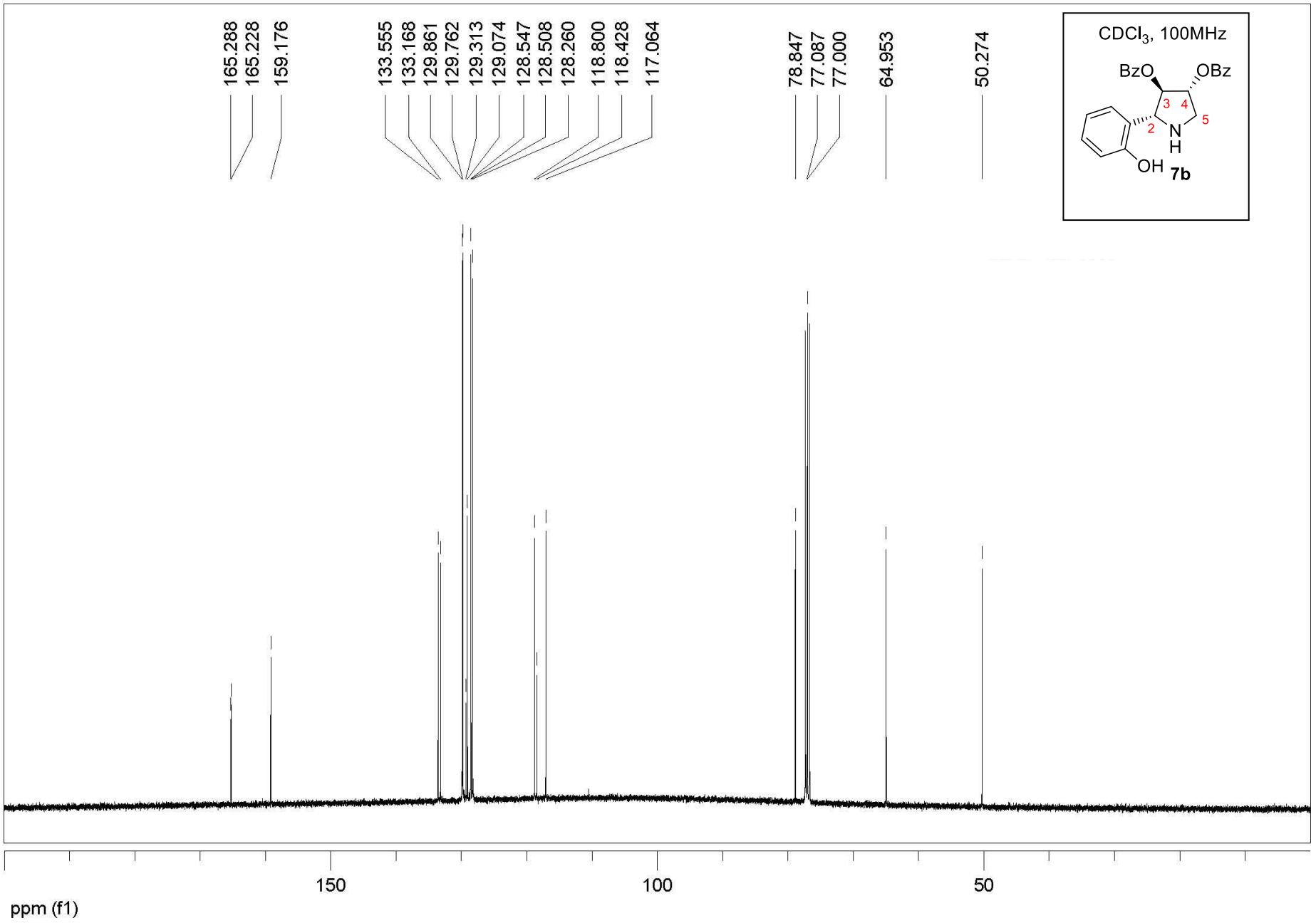

CDCl3, 400MHz
t-BuO
Ot-Bu
3 4
2
N
H
5
OH
6a
1.00
1.03
1.01
0.98
1.05
1.05
1.05
1.05
1.05
7.00
6.50
6.00
5.50
5.00
4.50
4.00
3.50
3.00
ppm (f1)
1.00
1.03
0.98
1.01
1.05
1.05
1.05
1.05
1.05
9.82
9.75
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)


CDCl3, 100MHz
t-BuO
Ot-Bu
3 4
2
5
N
H
OH
6a
157.998
129.580
128.708
123.284
118.443
116.688
80.751
77.318
77.000
76.682
76.290
74.564
73.760
66.559
50.959
28.656
28.576
175
150
125
100
75
50
25
ppm (f1)


CDCl3, 400MHz
BzO
OBz
3
4
5
2
N
H
OH
6b
2.03
1.98
2.04
0.96
1.86
1.04
1.05
1.07
5.0
ppm (f1)


165.721
165.252
157.768
133.500
133.279
129.933
129.735
129.283
129.262
129.142
128.514
128.403
128.339
121.088
119.181
117.423
82.974
77.319
77.000
76.682
67.033
51.237
CDCl3, 100MHz
BzO
OBz
3
4
5
2
N
H
OH
6b
150
100
50
ppm (f1)


CDCl3, 400MHz
BzO
OBz
OH
7b
7.260
1.91
1.90
1.00
3.01
2.00
1.96
1.87
0.96
0.94
0.99
1.00
1.01
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (f1)


165.288
165.228
159.176
133.555
133.168
129.861
129.762
129.313
129.074
128.547
128.508
128.260
118.800
118.428
117.064
78.847
77.087
77.000
64.953
50.274
CDCl3, 100MHz
BzO
OBz
3
4
5
2
N
H
OH
7b
150
100
50
ppm (f1)