Supporting Information

Enantioselective [2+2] Annulation of Simple Aldehydes with Isatin-Derived Ketimines via Oxidative N-Heterocyclic Carbene Catalysis

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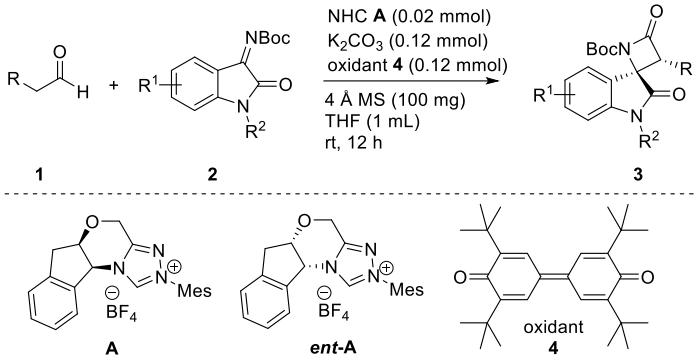
General Information

Commercially available materials purchased from Energy-Chemical were used as received. Proton nuclear magnetic resonance (^{1}H NMR) spectra were recorded on a Bruker BBFO or AV 400 (400 MHz) spectrometer. Chemical shifts were recorded in parts per million (ppm, δ) relative to tetramethylsilane (δ 0.00) or chloroform (δ = 7.26, singlet). ^{1}H NMR splitting patterns are designated as singlet (s), doublet (d), triplet (t), quartet (q), dd (doublet of doublets); m (multiplets), and etc. All first-order splitting patterns were assigned on the basis of the appearance of the multiplet. Splitting patterns that could not be easily interpreted are designated as multiplet (m) or broad (br). Carbon nuclear magnetic resonance (^{13}C NMR) spectra were recorded on a Bruker BBFO or AV 400 (100 MHz) spectrometer. High resolution mass spectral analysis (HRMS) was performed on Waters Xevo G2-S QTof mass spectrometer. The determination of ee was performed via chiral HPLC analysis using Waters Breeze 2 HPLC system. X-ray crystallography analysis was performed on Bruker X8 APEX X-ray diffractionmeter. Optical rotations were measured using a 1 mL cell with a 1 dm path length on a Rudolph Autopol IV automatic polarimeter and are reported as follows: $[\alpha]^{rt}_{D}$ (*c* in g per 100 mL solvent). Analytical thin-layer chromatography (TLC) was carried out on with GF 254 silica gel coated plates. Flash column chromatography was carried out using 200−300 mesh silica gel. Melting points are uncorrected. N-alkyl protected isatin derived ketimines were synthesized according to reported method.[1] NHC precatalyst **A** and ***ent*-A** were prepared by known protocol.[2]

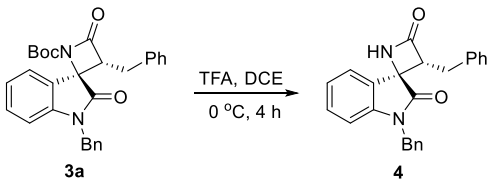
General procedure for the catalytic synthesis of products 3:



To a dry 10 mL Schlenk tube equipped with a magnetic stir bar, were added isatin-derived ketamine **2** (0.1 mmol), K_2CO_3 (0.12 mmol), oxidant **4** (0.12 mmol), 4Å MS (100 mg), and chiral NHC pre-catalyst **A** (0.02 mmol). The tube was sealed with a septum, evacuated and refilled with nitrogen (3 cycles). Freshly distilled anhydrous THF (1 mL) and aldehyde **1** (0.15 mmol) were then added and the reaction mixture was stirred at room temperature for another 12 hours. After completion of the reaction (monitored by TLC), the reaction mixture was concentrated under reduced pressure and the residue was subjected to column chromatography using hexane/EtOAc = 5/1 as eluent to afford the desired product **3**.

*Note:*The racemic catalyst that used for the preparation of the corresponding racemic products for HPLC analysis was synthesized by mixing chiral pre-catalyst **A** and *ent*-**A** in a 1:1 ratio.

Transformation of 3a to 4:



To a stirred solution of **3a** (46.8 mg, 0.1mmol, 96% ee) in DCE (3 mL) was carefully added TFA (0.5 mL) at 0 °C and the reaction mixture was stirred at 0˚C for another 4 hours. After **c**ompletion of the reaction (monitored by TLC), the reaction mixture was quenched by saturated $NaHCO_3$ aq. (4 mL). The aqueous phase was extracted with DCE, and the combined organic phase was washed with brine, dried over Na_2SO_4. Filtration and removal of solvent under reduced pressure gave a residue, which was purified by column chromatography using hexane/EtOAc = 3/1 as eluent to afford the desired product **4** (26.9 mg, 0.073 mmol, 73% yield, >20:1 dr, 98% ee).

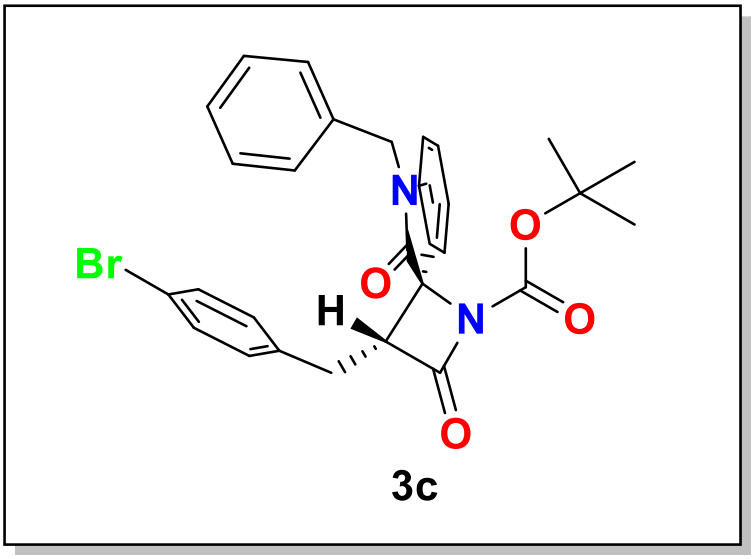
References cited in the SI:

1. Yan, W.; Wang, D.; Feng, J.; Li, P.; Zhao, D.; Wang, R. *Org. Lett.* **2012**, *14*, 2512-2515.

2. He, M.; Struble, J. R.; Bode, J. W. *J. Am. Chem. Soc.* **2006**, *128*, 8418-8420.

X-ray structure of product 3c

Absolute configurations of the products **3** were assigned based on the crystal X-ray structures of **3c**. CCDC 1526711 (**3c**, obtained as colorless needles *via* evaporation of a hexane/EtOAc solution) contains the supplementary X-ray crystallographic data. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.



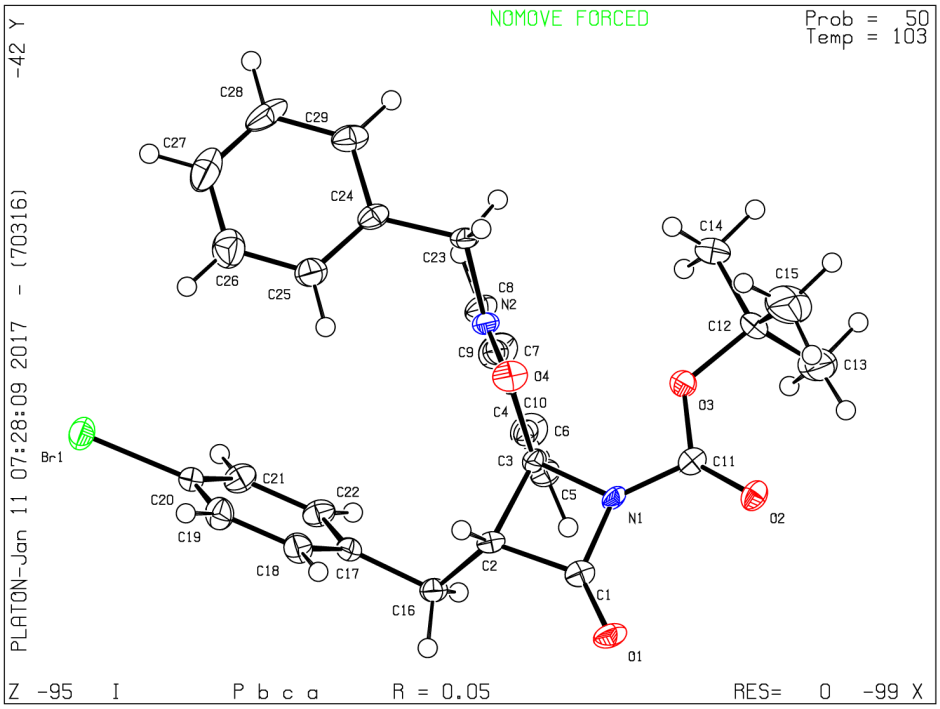
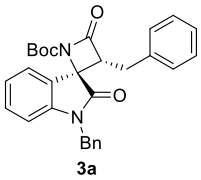


Table S1. Crystal data and structure refinement for 3c.

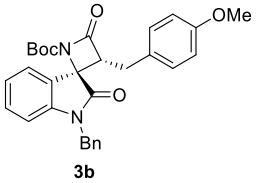
Empirical formula	$C_{29}H_{27}BrN_2O_4$	
Formula weight	547.43	
Temperature	103(2) K	
Wavelength	0.71073 Å	
Crystal system, Space group	orthorhombic,	P b c a
Unit cell dimensions	a= 11.8785(7) Å	α=90°
	b= 16.1043(11) Å	β=90°
	c= 27.3233(16) Å	γ=90°
Volume	5226.8(6) $Å^3$	
Z	8	
Density (calculated)	1.391 Mg/m^3	
Absorption coefficient	1.610 mm^{-1}	
F(000)	2256.0	
Crystal size	0.4*0.3*0.28 mm^3	
Theta range for data collection	2.60 to 27.13 °.	
Index ranges	-13<=h<=15, -20<=k<=20, -34<=l<=26	
Reflections collected	58212	
Independent reflections	5773 [*R*(int)= 0.0932]	
Completeness to theta= 27.130 °	99.8%	
Absorption correction	Multi-scan from equivalents	
Max. and min. transmission	0.660 and 0.540	
Refinement method	Full-matrix squares on F2	
Data/restraints/parameters	5773/0/328	
Goodness-of-fit on F2	1.082	
Final R indices [I>2sigma(I)]	*R1*= 0.0523, *wR2*= 0.1084	
R indices(all data)	*R1*= 0.0841, *wR2*= 0.1187	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.844 and -0.578 e.$Å^{-3}$	

Characterization of products:



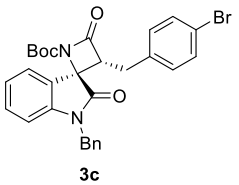
3a

(2*R*,3*R*) -*tert*-Butyl-1',3-dibenzyl-2',4-dioxospiro[azetidine-2,3'-indoline]-1-carboxylate (3a): 34.2 mg, 73% yield, 11:1 dr, yellow solid; mp 53-55 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, J = 6.8 Hz, 1H), 7.29-7.22 (m, 4H), 7.15 (t, J = 7.6 Hz, 1H), 7.09 (t, J = 7.2 Hz, 1H), 7.01 (t, J = 7.6 Hz, 2H), 6.96-6.93 (m, 2H), 6.64-6.60 (m, 3H), 5.02 (d, J = 16.0 Hz, 1H), 4.56 (d, J = 16.0 Hz, 1H), 4.02 (dd, J_1 = 11.6 Hz, J_2 = 4.8 Hz, 1H), 3.28 (dd, J_1 = 14.8 Hz, J_2 = 4.8 Hz, 1H), 2.92 (dd, J_1 = 14.4 Hz, J_2 = 11.2 Hz, 1H), 1.28 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.6, 165.4, 146.0, 143.8, 136.6, 134.8, 130.5, 128.8, 128.5, 128.1, 127.6, 126.8, 126.5, 124.5, 123.1, 122.7, 109.8, 84.0, 62.8, 60.5, 44.1, 30.4, 27.7; HRMS (ESI, m/z): calcd. for $C_{29}H_{28}N_2O_4Na^+$ 491.1947, found 491.1942. $[\alpha]^{20}_D$ = -0.4 (c = 0.25 in $CHCl_3$); HPLC analysis: 96% ee, [CHIRALPAK IA column; 1.0 mL/min; solvent system: *i*-PrOH/hexane = 20:80; retention times: 13.9 min (minor), 21.8 min (major)].



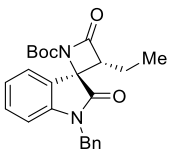
3b

(*2R*,*3R*)-*tert*-Butyl-1'-benzyl-3-(4-methoxybenzyl)-2',4-dioxo spiro[azetidine-2,3'-indoline]-1-carboxylate (3b): 40.3 mg, 81% yield, 10:1 dr, viscous solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (d, J = 7.2 Hz, 1H), 7.30-7.22 (m, 4H), 7.16 (t, J = 7.2 Hz, 1H), 6.93-6.91 (m, 2H), 6.62 (d, J = 7.6 Hz, 1H), 6.56-6.50 (m, 4H), 5.08 (d, J = 16.0 Hz, 1H), 4.53 (d, J = 16.0 Hz, 1H), 3.97 (dd, J_1 = 11.6 Hz, J_2 = 4.4 Hz, 1H), 3.69 (s, 3H), 3.21 (dd, J_1 = 14.8 Hz, J_2 = 4.8 Hz, 1H), 2.87 (dd, J_1 = 14.8 Hz, J_2 = 11.6 Hz, 1H), 1.28 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.6, 165.4, 158.1, 146.0, 143.8, 134.8, 130.4, 129.1, 128.7, 128.5, 127.6, 126.8, 124.5, 123.2, 122.7, 113.9, 109.8, 84.0, 62.7, 60.9, 55.0, 44.0, 29.5, 27.7; HRMS (ESI, m/z): calcd. for $C_{30}H_{30}N_2O_5Na^+$ 521.2052, found 521.2056. $[\alpha]^{20}_D$ = +6.0 (c = 0.25 in $CHCl_3$); HPLC analysis: 98% ee, [CHIRALPAK IA column; 1.0 mL/min; solvent system: *i*-PrOH/hexane =20:80; retention times: 16.5 min (major), 22.1 min (minor)].



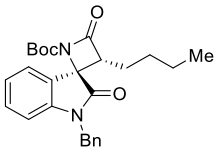
3c

(*2R*,*3R*)-*tert*-Butyl-1'-benzyl-3-(4-bromobenzyl)-2',4-dioxo spiro[azetidine-2,3'-indoline]-1-carboxylate (3c): 41.1 mg, 75% yield, 11:1 dr, yellow solid; mp 51-53 ^{o}C; ^{1}H NMR (400MHz, $CDCl_3$) δ 7.44 (d, J = 7.6 Hz, 1H), 7.31-7.28 (m, 4H), 7.17-7.11 (m, 3H), 6.98-6.96 (m, 2H), 6.66 (d, J = 8.0 Hz, 1H), 6.48 (d, J = 8.4 Hz, 2H), 5.07 (d, J = 15.6 Hz, 1H), 4.55 (d, J = 15.6 Hz, 1H), 3.95 (dd, J_1 = 11.6 Hz, J_2 = 4.4 Hz, 1H), 3.21 (dd, J_1 = 15.2 Hz, J_2 = 4.8 Hz, 1H), 2.88 (dd, J_1 = 14.8 Hz, J_2 = 11.6 Hz, 1H), 1.29 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.4, 164.9, 145.9, 143.8, 135.6, 134.7, 131.6, 130.6, 129.8, 128.9, 127.8, 126.8, 124.4, 122.9, 122.8, 120.5 109.9, 84.2, 62.6, 60.2, 44.1, 29.9, 27.7; HRMS (ESI, m/z): calcd. for $C_{29}H_{27}BrN_2O_4Na^+$ 569.1052, found 569.1049. $[\alpha]^{20}_D$ = +5.2 (c = 0.25 in $CHCl_3$); HPLC analysis: 98% ee, [CHIRALPAK IA column; 1.0 mL/min; solvent system: *i*-PrOH/hexane =20:80; retention times: 16.5 min (minor), 24.1 min (major)].



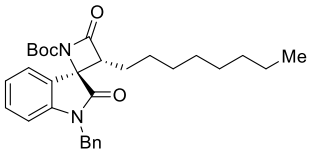
3d

(*2R,3R*)-*tert*-Butyl-1'-benzyl-3-ethyl-2',4-dioxospiro[azetidine-2,3'-indoline]-1-carboxylate (3d): 28.0 mg, 69% yield. 7:1 dr, yellow solid; mp 55-56 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.26 (m, 7H), 7.08 (t, J = 7.6 Hz, 1H), 6.78 (d, J = 8.0 Hz, 1H), 5.05 (d, J = 15.6 Hz, 1H), 4.86 (d, J = 15.6 Hz, 1H), 3.55 (dd, J_1 = 9.6 Hz, J_2 = 7.2 Hz, 1H), 1.99-1.88 (m, 1H), 1.61-1.53 (m, 1H), 1.27 (s, 9H), 0.70 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.1, 166.1, 146.2, 143.6, 135.2, 130.3, 128.9, 127.8, 127.3, 124.4, 123.0, 122.8, 109.7, 83.9, 62.5, 60.8, 44.2, 27.7, 18.9, 11.5; HRMS (ESI, m/z): calcd. for $C_{24}H_{26}N_2O_4Na^+$ 429.1790, found 429.1787. $[\alpha]^{20}_D$ = -33.6 (*c* = 0.25 in $CHCl_3$); HPLC analysis: 98% ee, [CHIRALPAK IA column; 1.0 mL/min; solvent system: *i*-PrOH/hexane =20:80; retention times: 9.2 min (minor), 11.3 min (major)].



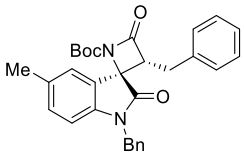
3e

(*2R,3R*)-*tert*-Butyl-1'-benzyl-3-butyl-2',4-dioxospiro[azetidine-2,3'-indoline]-1-carboxylate (3e): 32.1 mg, 74% yield, 5:1 dr, yellow solid; mp 57-59 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.26 (m, 7H), 7.08 (t, J = 7.2 Hz, 1H), 6.78 (d, J = 7.6 Hz, 1H), 5.08 (d, J = 15.6 Hz, 1H), 4.83 (d, J = 15.6 Hz, 1H), 3.62 (dd, J_1 = 9.6 Hz, J_2 = 6.8 Hz, 1H), 1.90-1.83 (m, 1H), 1.56-1.50 (m, 1H), 1.27 (s, 10H), 1.16-1.07 (m, 2H), 0.90-0.84 (m, 1H), 0.71 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.0, 166.2, 146.1, 143.6, 135.2, 130.3, 128.8, 127.8, 127.3, 124.5, 123.1, 122.8, 109.7, 83.9, 62.6, 59.3, 44.2, 29.1, 27.7, 24.7, 21.9, 13.6; HRMS (ESI, m/z): calcd. for $C_{26}H_{30}N_2O_4Na^+$ 457.2103, found 457.2108. $[\alpha]^{20}_D$ = -38.4 (*c* = 0.25 in $CHCl_3$); HPLC analysis: 98% ee, [CHIRALPAK IA column; 1.0 mL/min; solvent system: *i*-PrOH/hexane =20:80; retention times: 9.2 min (minor), 11.1 min (major)].



3f

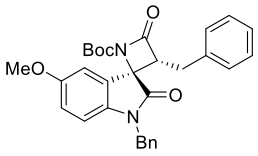
(*2R,3R*)-*tert*-Butyl-1'-benzyl-3-octyl-2',4-dioxospiro [azetidine-2,3'-indoline]-1-carboxylate (3f): 37.7 mg, 77% yield, 5:1 dr, yellow solid; 62-63 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.26 (m, 7H), 7.08 (t, J = 7.2 Hz, 1H), 6.78 (d, J = 8.0 Hz, 1H), 5.07 (d, J = 15.6 Hz,1H), 4.84 (d, J = 16.0 Hz, 1H), 3.61 (dd, J_1 = 10.0 Hz, J_2 = 6.8 Hz, 1H), 1.89-1.82 (m, 1H), 1.59-1.49 (m, 1H), 1.27 (s, 9H), 1.22-1.05 (m, 12H), 0.84 (t, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.0, 166.2, 146.1, 143.5, 135.2, 130.2, 128.8, 127.8, 127.3, 124.4, 123.1, 122.7, 109.7, 83.8, 62.6, 59.3, 44.2, 31.6, 29.0, 28.9, 28.8, 27.7, 26.8, 25.0, 22.5, 14.0; HRMS (ESI, m/z): calcd. for $C_{30}H_{38}N_2O_4Na^+$ 513.2729, found 7513.2728. $[\alpha]^{20}_D$ = -45.6 (*c* = 0.25 in $CHCl_3$); HPLC analysis: 95% ee, [CHIRALPAK IA column; 1.0 mL/min; solvent system: *i*-PrOH/hexane = 20:80; retention times: 10.0 min (minor), 11.6 min (major)].



3g

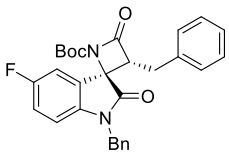
(*2R,3R*)-*tert*-Butyl-1',3-dibenzyl-5'-methyl-2',4-dioxospiro [azetidine-2,3'-indoline]-1-carboxylate (3g): 38.1 mg, 79% yield, >20:1 dr, yellow solid; mp 55-57 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 (d, J = 1.6 Hz, 1H), 7.23 (t, J = 2.4 Hz, 3H), 7.09-7.01 (m, 4H), 6.97-6.94 (m, 2H), 6.64 (d, J = 7.2 Hz, 2H), 6.50

(d, J = 8.0 Hz, 1H), 5.00 (d, J = 16.0 Hz, 1H), 4.57 (d, J = 16.0 Hz, 1H), 4.00 (dd, J_1 = 10.8 Hz, J_2 = 4.8 Hz, 1H), 3.26 (dd, J_1 = 14.8 Hz, J_2 = 4.8 Hz, 1H), 2.92 (dd, J_1 = 14.8 Hz, J_2 = 11.2 Hz, 1H), 2.38 (s, 3H), 1.30 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.5, 165.4, 146.0, 141.4, 136.8, 135.0, 132.4, 130.7, 128.8, 128.5, 128.1, 127.5, 126.8, 126.5, 125.3, 123.1, 109.6, 84.0, 62.9, 60.4, 44.1, 30.4, 27.7, 21.1; HRMS (ESI, m/z): calcd. for $C_{30}H_{30}N_2O_4H^+$ 483.2284, found 483.2273. $[\alpha]^{20}_D$ = +7.6 (c = 0.25 in $CHCl_3$); HPLC analysis: 94% ee, [CHIRALPAK IA column; 1.0 mL/min; solvent system: *i*-PrOH/hexane =20:80; retention times: 13.3 min (minor), 19.3 min (major)].



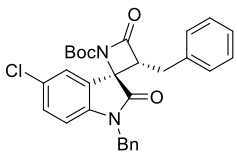
3h

(*2R,3R*)-*tert*-Butyl-1',3-dibenzyl-5'-methoxy-2',4-dioxospiro [azetidine-2,3'-indoline]-1-carboxylate (3h): 36.4 mg, 73% yield, 12:1 dr, yellow solid; mp 50-52 ^oC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24-7.22 (m, 3H), 7.23-7.12 (m, 1H), 7.10-7.04 (m, 3H), 6.97-6.95 (m, 2H), 6.78 (dd, J_1 = 8.4 Hz, J_2 = 2.4 Hz, 1H), 6.68 (d, J = 7.6 Hz, 2H), 6.50 (d, J = 8.4 Hz, 1H), 5.00 (d, J = 16.0 Hz, 1H), 4.56 (d, J = 15.6 Hz, 1H), 4.02 (dd, J_1 = 11.2 Hz, J_2 =4.8 Hz, 1H), 3.81 (s, 3H), 3.28 (dd, J_1 = 14.8 Hz, J_2 = 4.8 Hz, 1H), 2.93 (dd, J_1 = 14.8 Hz, J_2 = 11.6 Hz, 1H), 1.32 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.3, 165.3, 156.0, 146.0, 137.1, 136.6, 134.9, 128.8, 128.5, 128.1, 127.5, 126.8, 126.5, 124.3, 114.8, 111.8, 110.3, 84.1, 63.0, 60.4, 56.0, 44.1, 30.4, 27.8; HRMS (ESI, m/z): calcd. for $C_{30}H_{30}N_2O_5Na^+$ 521.2052, found 521.2060. $[\alpha]^{20}_D$ = +10.8 (c = 0.25 in $CHCl_3$); HPLC analysis: 93% ee, [CHIRALPAK IA column; 1.0 mL/min; solvent system: *i*-PrOH/hexane =20:80; retention times: 16.3 min (minor), 18.9 min (major)].



3i

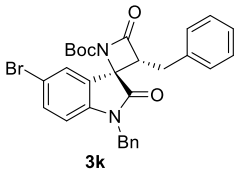
(*2R,3R*)-*tert*-Butyl-1',3-dibenzyl-5'-fluoro-2',4-dioxospiro [azetidine-2,3'-indoline]-1-carboxylate (3i): 29.2 mg, 60% yield, >20:1 dr, yellow solid; mp 81-83 ^oC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26-7.20 (m, 4H), 7.12-7.02 (m, 3H), 6.98-6.94 (m,3H), 6.70 (d, J = 7.2 Hz, 2H), 6.50 (dd, J_1 = 8.8 Hz, J_2 = 4.0 Hz, 1H), 4.99 (d, J = 16.0 Hz, 1H), 4.58 (d, J = 16.0 Hz, 1H), 4.05 (dd, J_1 = 11.2 Hz, J_2 = 4.8 Hz, 1H), 3.31 (dd, J_1 =15.2 Hz, J_2 = 5.2 Hz, 1H), 2.90 (dd, J_1 =14.8 Hz, J_2 = 11.2 Hz, 1H), 1.33 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.4, 164.8, 169.0 (d, $J_{C\text{-}F}$ = 241 Hz), 139.7 (d, $J_{C\text{-}F}$ = 2 Hz), 136.2, 134.5, 128.9, 128.6, 128.0, 127.7, 127.4, 126.8, 126.6, 124.8 (d, $J_{C\text{-}F}$ = 7 Hz), 116.7 (d, $J_{C\text{-}F}$ = 23 Hz), 112.6 (d, $J_{C\text{-}F}$ = 25 Hz), 110.5 (d, $J_{C\text{-}F}$ = 8 Hz), 84.4, 62.7, 60.5, 44.2, 30.4, 27.8; HRMS (ESI, m/z): calcd. for $C_{29}H_{27}FN_2O_4H^+$ 487.2033, found 487.2026. $[\alpha]^{20}_D$ = -9.6 (c = 0.25 in $CHCl_3$); HPLC analysis: 98% ee, [CHIRALPAK IA column; 1.0 mL/min; solvent system: *i*-PrOH/hexane =20:80; retention times: 14.2 min (minor), 20.4 min (major)].



3j

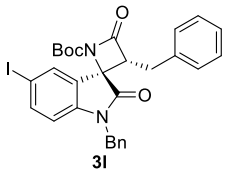
(*2R,3R*)-*tert*-Butyl-1',3-dibenzyl-5'-chloro-2',4-dioxospiro [azetidine-2,3'-indoline]-1-carboxylate (3j): 29.2 mg, 58% yield, >20:1 dr, yellow solid; mp 42-44 ^oC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (s, 1H), 7.43-7.21 (m, 4H), 7.10-7.02 (m, 3H),

6.95-6.93 (m, 2H), 6.69 (d, J = 7.2 Hz, 2H), 6.49 (d, J = 8.4 Hz, 1H), 4.97 (d, J = 16.0 Hz, 1H), 4.59 (d, J = 16.0 Hz, 1H), 4.04 (dd, J_1 = 11.6 Hz, J_2 = 5.2 Hz, 1H), 3.31 (dd, J_1 = 14.8 Hz, J_2 = 4.8 Hz, 1H), 2.90 (dd, J_1 = 14.8 Hz, J_2 = 11.2 Hz, 1H), 1.34 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.2, 164.7, 146.0, 142.2, 136.2, 134.4, 130.3, 128.9, 128.6, 128.3, 128.0, 127.7, 126.8, 126.6, 124.9, 124.8, 110.7, 84.5, 62.4, 60.5, 44.2, 30.4, 27.8; HRMS (ESI, m/z): calcd. for $C_{29}H_{27}ClN_2O_4Na^+$ 525.1557, found 527.1556. $[\alpha]^{20}_D$ = +15.6 (c = 0.25 in $CHCl_3$); HPLC analysis: 98% ee, [CHIRALPAK IA column; 1.0 mL/min; solvent system: *i*-PrOH/hexane =20:80; retention times: 15.7 min (minor), 20.4 min (major)].



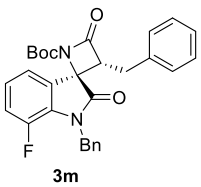
3k

(*2R,3R*)-*tert*-Butyl-1',3-dibenzyl-5'-bromo-2',4-dioxospiro [azetidine-2,3'-indoline]-1-carboxylate (3k): 32.3 mg, 59% yield, >20:1 dr, yellow solid; mp 51-53 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (d, J = 2.0 Hz, 1H), 7.36 (dd, J_1 = 8.4 Hz, J_2 = 2.0 Hz, 1H), 7.25-7.23 (m, 3H), 7.10-7.04 (m, 3H), 6.95-6.93 (m, 2H), 6.69 (d, J = 7.2 Hz, 2H), 6.44 (d, J = 8.4 Hz, 1H), 4.96 (d, J = 16.0 Hz, 1H), 4.58 (d, J = 15.6 Hz, 1H), 4.04 (dd, J_1 = 11.2 Hz, J_2 = 4.8 Hz, 1H), 3.31 (dd, J_1 = 15.2 Hz, J_2 = 5.2 Hz, 1H), 2.90 (dd, J_1 = 15.2 Hz, J_2 = 11.6 Hz, 1H), 1.34 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.1, 164.6, 146.0, 142.7, 136.2, 134.3, 133.2, 128.9, 128.6, 128.1, 127.7, 127.6, 126.8, 126.6, 125.2, 115.4, 111.2, 84.5, 62.3, 60.5, 44.2, 30.4, 27.8; HRMS (ESI, m/z): calcd. for $C_{29}H_{27}BrN_2O_4Na^+$ 569.1052, found 569.1049. $[\alpha]^{20}_D$ = +20.0 (c = 0.25 in $CHCl_3$); HPLC analysis: 94% ee, [CHIRALPAK IA column; 1.0 mL/min; solvent system: *i*-PrOH/hexane =20:80; retention times: 17.0 min (minor), 22.1 min (major)].



3l

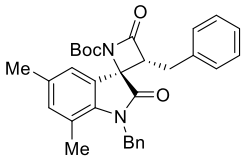
(*2R,3R*)-*tert*-Butyl-1',3-dibenzyl-5'-iodo-2',4-dioxospiro [azetidine-2,3'-indoline]-1-carboxylate (3l): 33.9 mg, 57% yield, >20:1 dr, pale red solid; mp 53-54 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, J = 1.6 Hz, 1H), 7.55 (d, J = 8.4 Hz, 1H), 7.25-7.23 (m, 3H), 7.10-7.03 (m, 3H), 6.94-6.92 (m, 2H), 6.69 (d, J = 6.8 Hz, 2H), 6.34 (d, J = 8.4 Hz, 1H), 4.95 (d, J = 16.0 Hz, 1H), 4.58 (d, J = 16.0 Hz, 1H), 4.03 (dd, J_1 = 11.6 Hz, J_2 = 5.2 Hz, 1H), 3.31 (dd, J_1 = 15.2 Hz, J_2 = 5.2 Hz, 1H), 2.89 (dd, J_1 = 14.8 Hz, J_2 = 11.6 Hz, 1H), 1.34 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.9, 164.7, 146.0, 143.4, 139.1, 136.2, 134.3, 133.1, 128.9, 128.6, 128.1, 127.7, 126.8, 126.6, 125.5, 111.7, 84.9, 84.5, 62.2, 60.5, 44.1, 30.5, 27.8; HRMS (ESI, m/z): calcd. for $C_{29}H_{27}IN_2O_4Na^+$ 617.0913, found 617.0918. $[\alpha]^{20}_D$ = +112.0 (c = 0.25 in $CHCl_3$); HPLC analysis: 98% ee, [CHIRALPAK IA column; 1.0 mL/min; solvent system: *i*-PrOH/hexane =20:80; retention times: 17.2 min (minor), 22.8 min (major)].



3m

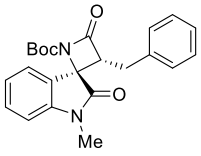
(*2R,3R*)-*tert*-Butyl-1',3-dibenzyl-7'-fluoro-2',4-dioxospiro [azetidine-2,3'-indoline]-1-carboxylate (3m): 24.8 mg, 51% yield, 10:1 dr, yellow solid; mp 53-55 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25-7.22 (m, 4H), 7.11-7.00 (m, 7H), 6.64 (d, J = 7.2 Hz, 2H), 5.02 (d, J = 15.6 Hz, 1H), 4.84 (d, J = 15.6 Hz, 1H), 4.02 (dd, J_1 = 11.2

Hz, J_2 = 5.2 Hz, 1H), 3.27 (dd, J_1 = 14.8 Hz, J_2 = 4.8 Hz, 1H), 2.86 (dd, J_1 = 15.2 Hz, J_2 = 11.6 Hz, 1H), 1.29 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.3, 164.9, 147.6 (d, J_{C-F} = 240 Hz), 136.2, 136.1, 130.4 (d, J_{C-F} = 9 Hz), 128.6, 128.5, 128.3, 127.9, 127.5, 127.0, 126.7, 126.1 (d, J_{C-F} = 3 Hz), 123.5 (d, J_{C-F} = 6 Hz), 120.4 (d, J_{C-F} = 3 Hz), 118.6 (d, J_{C-F} = 19 Hz), 82.9 (d, J_{C-F} = 302 Hz), 62.6, 60.8, 45.6 (d, J_{C-F} = 5 Hz), 30.3, 27.8 (d, J_{C-F} = 27 Hz); HRMS (ESI, m/z): calcd. for $C_{29}H_{27}FN_2O_4H^+$ 487.2033, found 487.2040. $[\alpha]^{20}_D$ = -8.8 (*c* = 0.25 in $CHCl_3$); HPLC analysis: 95% ee, [CHIRALPAK ID column; 1.0 mL/min; solvent system: *i*-PrOH/hexane = 20:80; retention times: 42.3 min (minor), 58.6 min (major)].



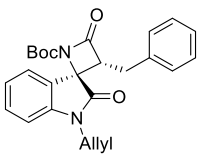
3n

(*2R,3R*)-*tert*-Butyl-1',3-dibenzyl-5',7'-dimethyl-2',4-dioxospiro[azetidine-2,3'-indoline]-1-carboxylate (3n): 20.4 mg, 41% yield, >20:1 dr, yellow solid; mp 49-50 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20-7.18 (m, 3H), 7.15-7.08 (m, 4H), 6.87 (s, 1H), 6.81-6.79 (m, 2H), 6.60 (d, *J* = 7.2 Hz, 2H), 5.13 (d, *J* = 16.8 Hz, 1H), 4.90 (d, *J* = 17.2 Hz, 1H), 3.97 (dd, J_1 = 11.2 Hz, J_2 = 4.8 Hz, 1H), 3.25 (dd, J_1 = 14.8 Hz, J_2 = 5.2 Hz, 1H), 2.91 (dd, J_1 = 14.8 Hz, J_2 = 11.6 Hz, 1H), 2.36 (s, 3H), 2.09 (s, 3H), 1.37 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.5, 165.4, 146.3, 139.5, 136.9, 136.9, 134.9, 132.3, 128.9, 128.7, 128.4, 127.0, 126.6, 125.3, 123.8, 123.1, 120.4, 84.1, 62.3, 60.9, 45.3, 30.3, 27.9, 20.8, 18.3; HRMS (ESI, m/z): calcd. for $C_{31}H_{32}N_2O_4Na^+$ 519.2260, found 519.2253. $[\alpha]^{20}_D$ = -2.8 (*c* = 0.25 in $CHCl_3$); HPLC analysis: 94% ee, [CHIRALPAK OD-H column; 1.0 mL/min; solvent system: *i*-PrOH/hexane = 20:80; retention times: 10.0 min (minor), 11.1 min (major)].



3o

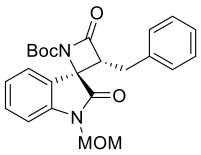
(*2R,3R*)-*tert*-Butyl-3-benzyl-1'-methyl-2',4-dioxospiro[azetidine-2,3'-indoline]-1-carboxylate (3o): 29.0 mg, 74% yield. >20:1 dr, yellow solid; mp 141-142 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42-7.37 (m, 2H), 7.16 (t, *J* = 8.0 Hz, 1H), 7.07-7.05 (m, 3H), 6.76 (d, *J* = 7.6 Hz, 1H), 6.72-6.70 (m, 2H), 3.94 (dd, J_1 = 10.0 Hz, J_2 = 6.0 Hz, 1H), 3.27-3.21 (m, 1H), 3.10 (s, 3H), 2.81 (dd, J_1 = 14.8 Hz, J_2 = 10.0 Hz, 1H), 1.28 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.3, 165.4, 146.0, 144.6, 136.6, 130.5, 128.3, 128.1, 126.5, 124.4, 123.1, 122.7, 108.6, 83.9, 62.7, 60.0, 30.6, 27.7, 26.4; HRMS (ESI, m/z): calcd. for $C_{23}H_{24}N_2O_4Na^+$ 415.1634, found 415.1641. $[\alpha]^{20}_D$ = -40.8 (*c* = 0.25 in $CHCl_3$); HPLC analysis: 98% ee, [CHIRALPAK IA column; 1.0 mL/min; solvent system: *i*-PrOH/hexane = 20:80; retention times: 10.1 min (minor), 11.9 min (major)].



3p

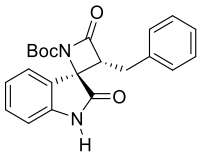
(*2R,3R*)-*tert*-Butyl-1'-allyl-3-benzyl-2',4-dioxospiro[azetidine-2,3'-indoline]-1-carboxylate (3p): 34.3 mg, 82% yield, 14:1 dr, yellow solid; mp 87-89 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (d, *J* = 7.2 Hz, 1H), 7.35 (t, *J* = 8.0 Hz, 1H), 7.17 (t, *J* = 7.6 Hz, 1H), 7.04-7.01 (m, 3H), 6.74 (d, *J* = 8.0 Hz, 1H), 6.64-6.62 (m, 2H), 5.60-5.50 (m, 1H), 5.04 (d, *J* = 10.4 Hz, 1H), 4.82 (d, *J* = 17.2 Hz, 1H), 4.42-4.36 (m, 1H), 4.04-3.94 (m, 2H), 3.25 (dd, J_1 = 14.8 Hz, J_2 = 4.8 Hz, 1H), 2.86 (dd, J_1 = 14.8 Hz, J_2

= 11.2 Hz, 1H), 1.29 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.1, 165.3, 146.0, 143.8, 136.5, 130.4, 130.4, 128.4, 128.1, 126.5, 124.4, 123.1, 122.6, 117.6, 109.6, 84.0, 62.7, 60.5, 42.4, 30.3, 27.7; HRMS (ESI, m/z): calcd. for $C_{25}H_{26}N_2O_4Na^+$ 441.1790, found 441.1785. $[\alpha]^{20}_D$ = -28.8 (*c* = 0.25 in $CHCl_3$); HPLC analysis: 97% ee, [CHIRALPAK IA column; 1.0 mL/min; solvent system: *i*-PrOH/hexane = 20:80; retention times: 9.6 min (minor), 12.1 min (major)].



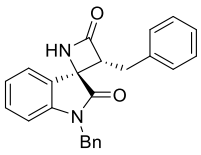
3q

(*2R,3R*)-*tert*-Butyl-3-benzyl-1'-(methoxymethyl)-2',4-dioxospiro [azetidine-2,3'-indoline]-1-carboxylate (3q): 33.8 mg, 80% yield, >20:1 dr, yellow solid; 105-107 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (d, *J* = 7.6 Hz, 1H), 7.41 (t, *J* = 7.6 Hz, 1H), 7.23 (t, *J* = 7.6 Hz, 1H), 7.05-7.03 (m, 3H), 6.99 (d, *J* = 7.6 Hz, 1H), 6.63-6.61 (m, 2H), 5.04 (d, *J* = 10.8 Hz, 1H), 4.93 (d, *J* = 10.8 Hz, 1H), 3.94 (dd, J_1 = 10.8 Hz, J_2 = 4.8 Hz, 1H), 3.25 (dd, J_1 = 14.8 Hz, J_2 = 5.2 Hz, 1H), 2.97 (s, 3H), 2.87 (dd, J_1 = 14.8 Hz, J_2 = 11.2 Hz, 1H), 1.30 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.0, 165.1, 146.0, 142.9, 136.6, 130.7, 128.4, 128.1, 126.6, 124.5, 123.2, 122.6, 110.3, 84.2, 71.4, 62.8, 60.9, 56.0, 30.3, 27.7; HRMS (ESI, m/z): calcd. for $C_{24}H_{26}N_2O_5Na^+$ 445.1739, found 445.1838. $[\alpha]^{20}_D$ = -22.4 (*c* = 0.25 in $CHCl_3$); HPLC analysis: 94% ee, [CHIRALPAK IA column; 1.0 mL/min; solvent system: *i*-PrOH/hexane =20:80; retention times: 10.0 min (minor), 11.9 min (major)]



3r

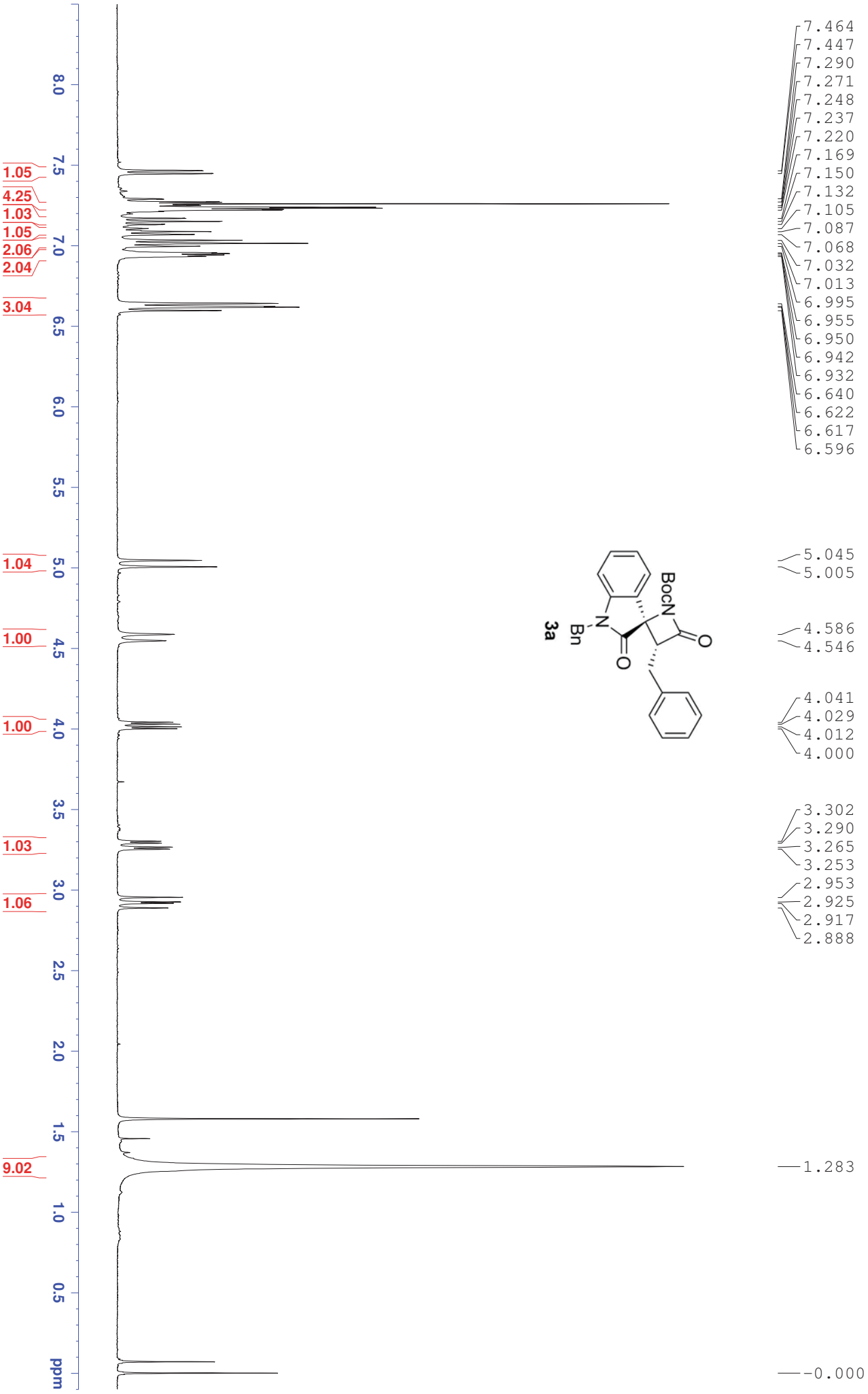
(*2R,3R*)-*tert*-Butyl-3-benzyl-2',4-dioxospiro[azetidine-2,3'-indoline]-1-carboxylate (3r): 12.5 mg, 33% yield, >20:1 dr, yellow solid; mp 53-54 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (br, 1H), 7.42 (d, *J* = 7.2 Hz, 1H), 7.33 (t, *J* = 7.6 Hz, 1H), 7.15 (t, *J* = 7.6 Hz, 1H), 7.09-7.07 (m, 3H), 6.83 (d, *J* = 7.6 Hz, 1H), 6.76-6.74 (m, 2H), 3.92 (dd, J_1 = 10.0 Hz, J_2 = 6.0 Hz, 1H), 3.24 (dd, J_1 = 14.8 Hz, J_2 = 6.0 Hz, 1H), 2.85 (dd, J_1 = 14.8 Hz, J_2 = 10.0 Hz, 1H), 1.31 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.2, 165.4, 146.0, 141.6, 136.6, 130.6, 128.5, 128.1, 126.6, 124.9, 123.3, 122.8, 110.6, 84.2, 63.0, 60.1, 30.7, 27.7; HRMS (ESI, m/z): calcd. for $C_{22}H_{22}N_2O_4Na^+$ 401.1477, found 401.1475. $[\alpha]^{20}_D$ = -6.4 (*c* = 0.25 in $CHCl_3$); HPLC analysis: 94% ee, [CHIRALPAK OD-H column; 1.0 mL/min; solvent system: *i*-PrOH/hexane =20:80; retention times: 7.2 min (minor), 8.7 min (major)]



4

(*2R,3R*)-1',3-Dibenzylspiro[azetidine-2,3'-indoline]-2',4-dione (4): 26.9 mg, 73% yield, >20:1 dr, yellow solid; mp 58-60 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (d, *J* = 7.2 Hz, 1H), 7.28-7.14 (m, 5H), 7.05 (t, *J* = 7.2 Hz, 1H), 6.98 (t, *J* = 7.6 Hz, 2H), 6.86 (t, *J* = 5.2 Hz, 2H), 6.63 (br, 1H), 6.58 (t, *J* = 7.6 Hz, 3H), 4.97 (d, *J* = 16.0 Hz, 1H), 4.47 (d, *J* = 15.6 Hz, 1H), 4.00 (dd, J_1 = 11.6 Hz, J_2 = 4.8 Hz, 1H), 3.22 (dd, J_1 = 14.8 Hz, J_2 = 4.8 Hz, 1H), 2.86 (dd, J_1 = 14.8 Hz, J_2 = 11.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.9, 169.1, 143.2, 137.1, 134.8, 130.3, 128.8, 128.3, 128.0, 127.4, 126.6, 126.2, 125.1, 124.7, 122.7, 109.7, 63.6, 60.6, 44.0, 31.1; HRMS (ESI, m/z): calcd. for $C_{24}H_{20}N_2O_2H^+$ 369.1603, found 369.1599. $[\alpha]^{20}_D$ = -25.6 (*c* = 0.25 in

$CHCl_3$); HPLC analysis: 98% ee, [CHIRALPAK IA column; 1.0 mL/min; solvent system: *i*-PrOH/hexane = 20:80; retention times: 14.5 min (minor), 17.1 min (major)]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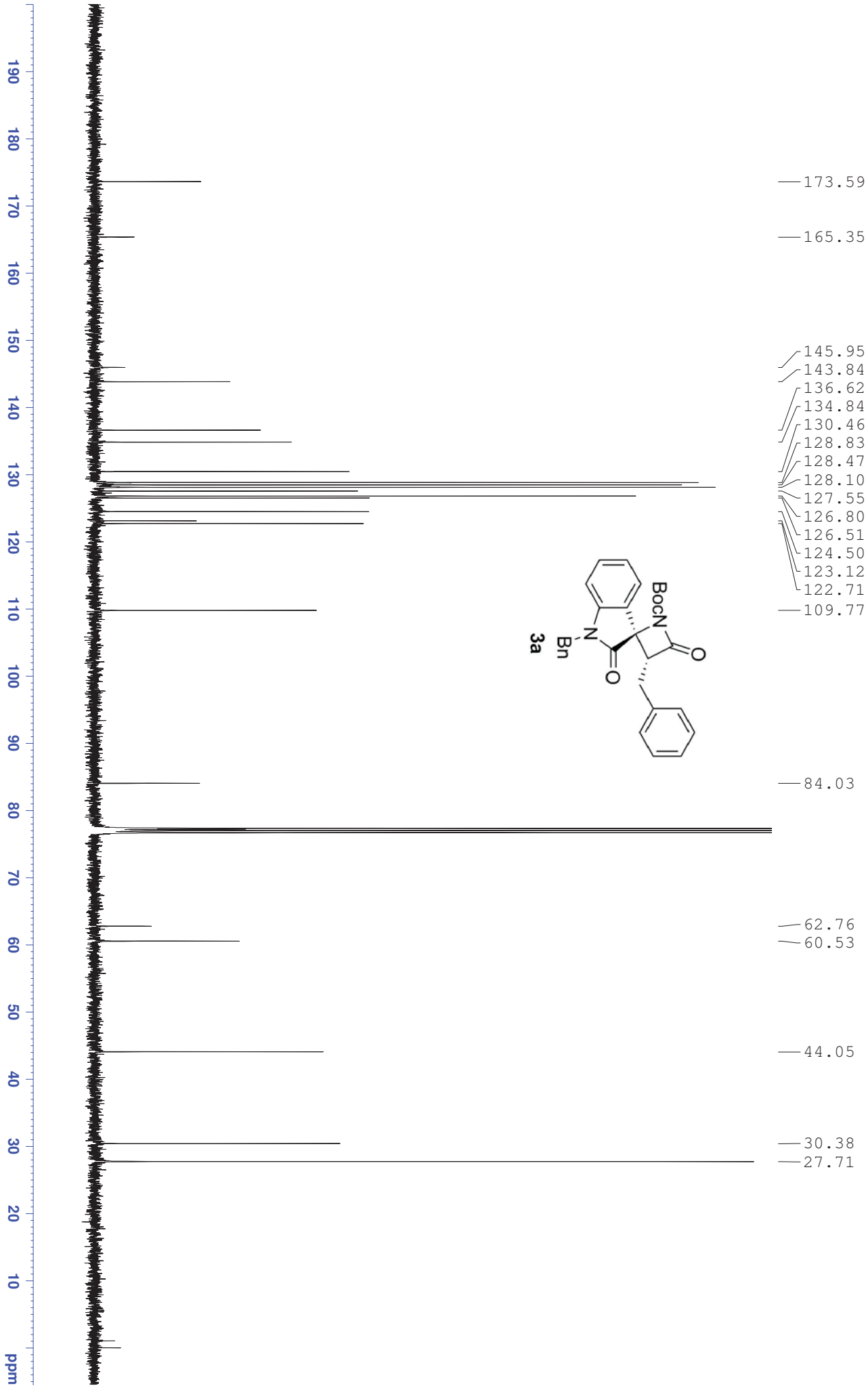
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Bn
O
O
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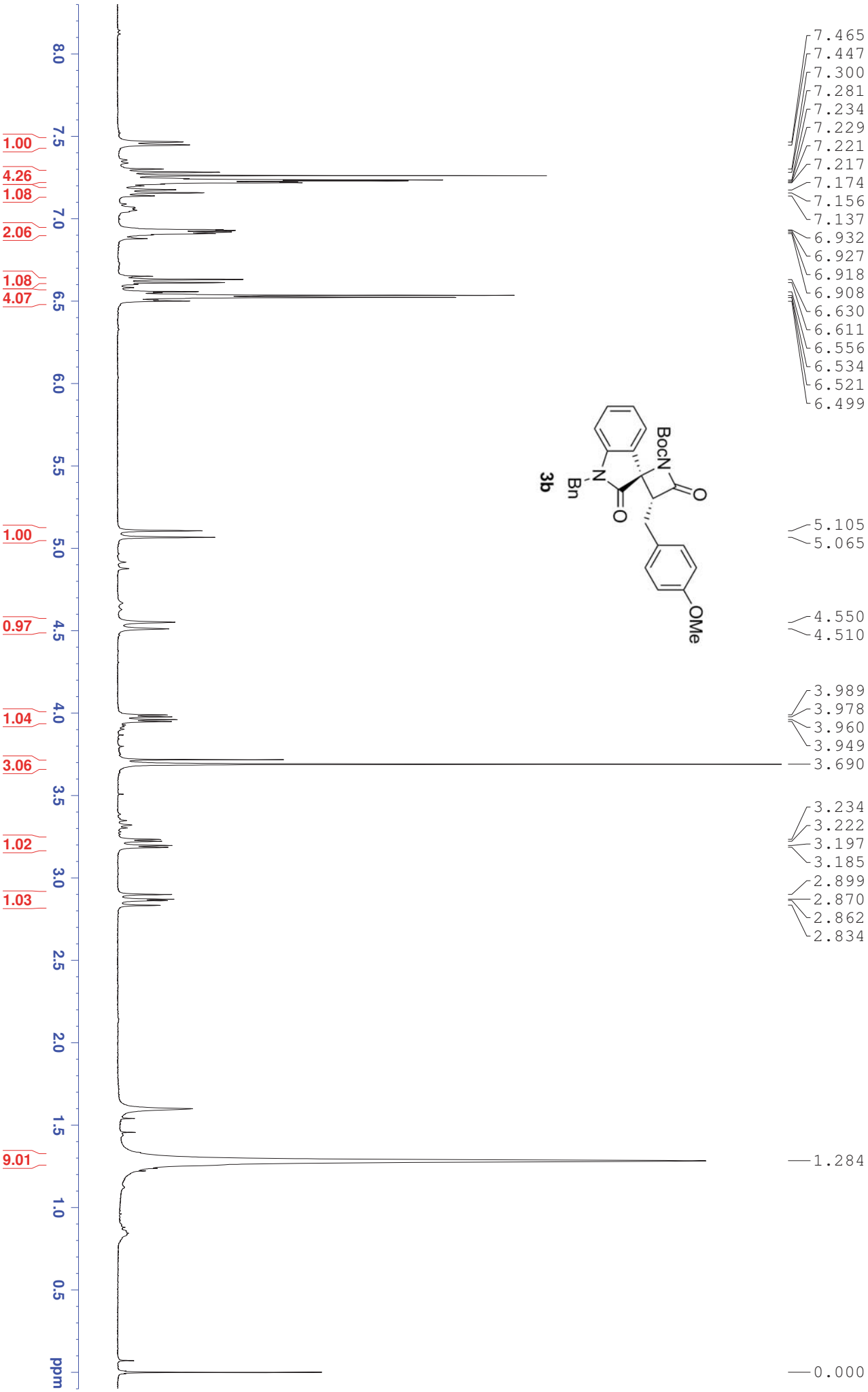
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4.07
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3.06
1.02
1.03
9.01
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4.0
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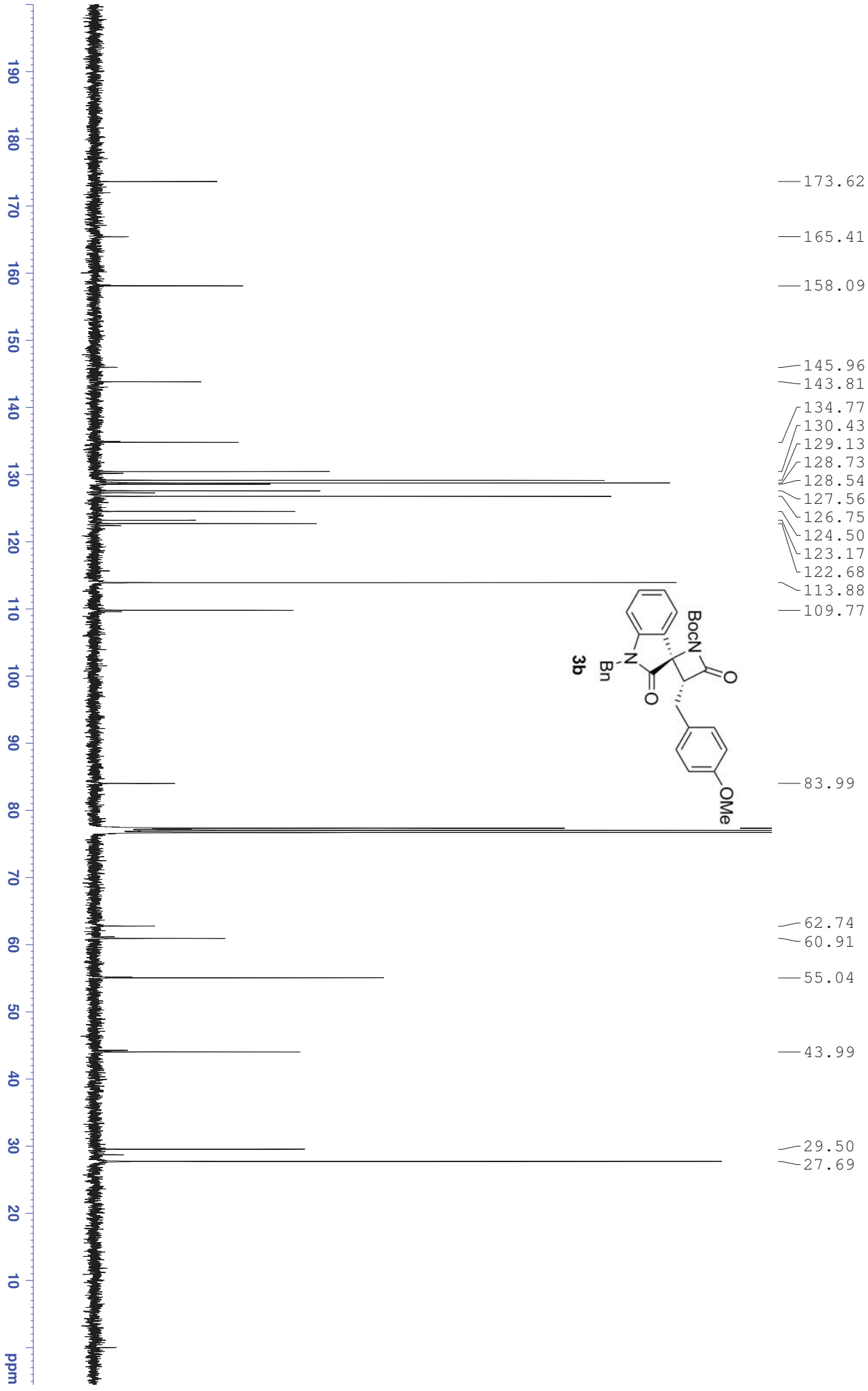
O
OMe
3b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Bn
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O
OMe
3b
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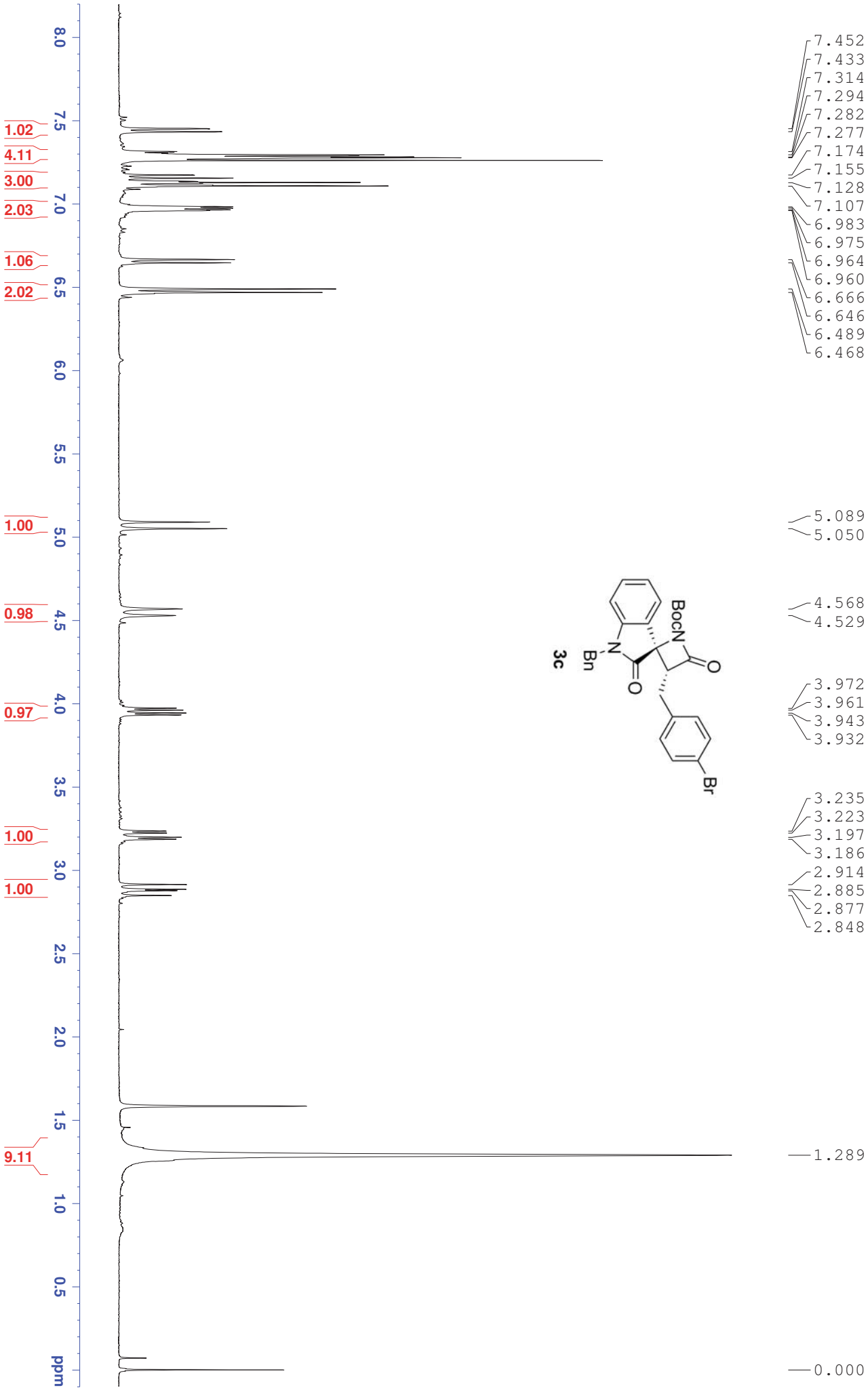
ppm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6.975
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6.468
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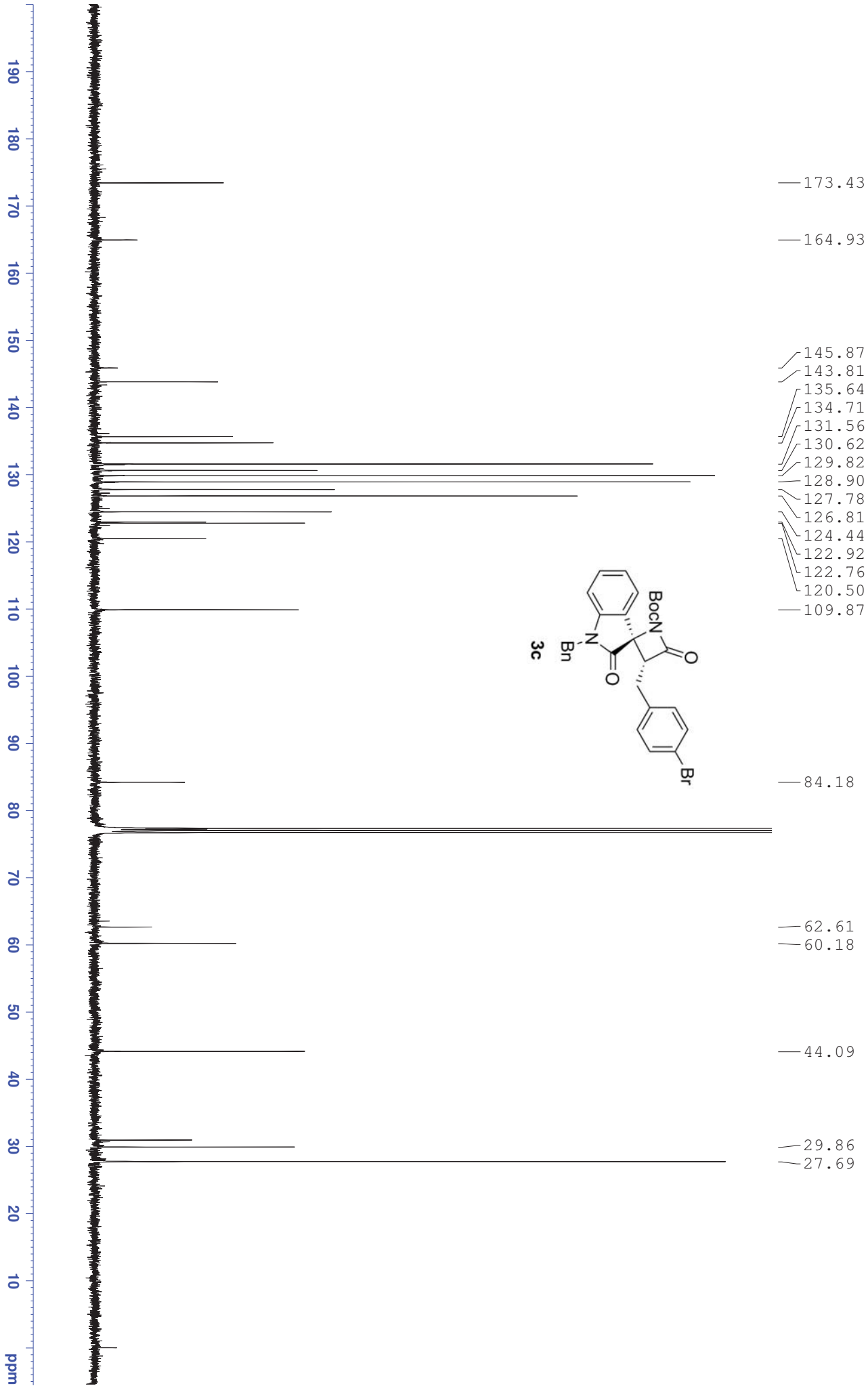
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120.50
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Bn
O
O
Br
3c
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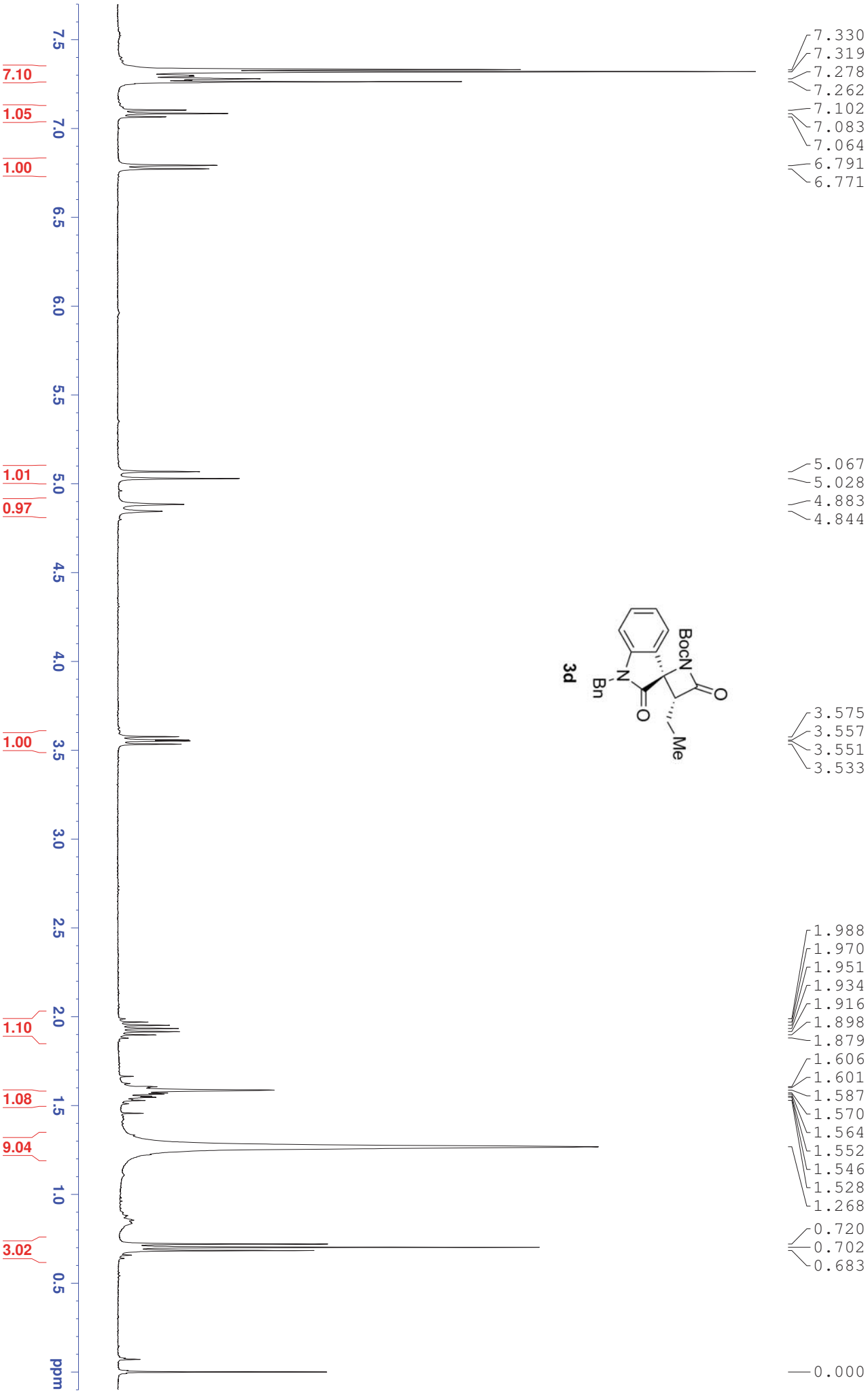
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3.575
3.557
3.551
3.533
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1.951
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1.916
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1.879
1.606
1.601
1.587
1.570
1.564
1.552
1.546
1.528
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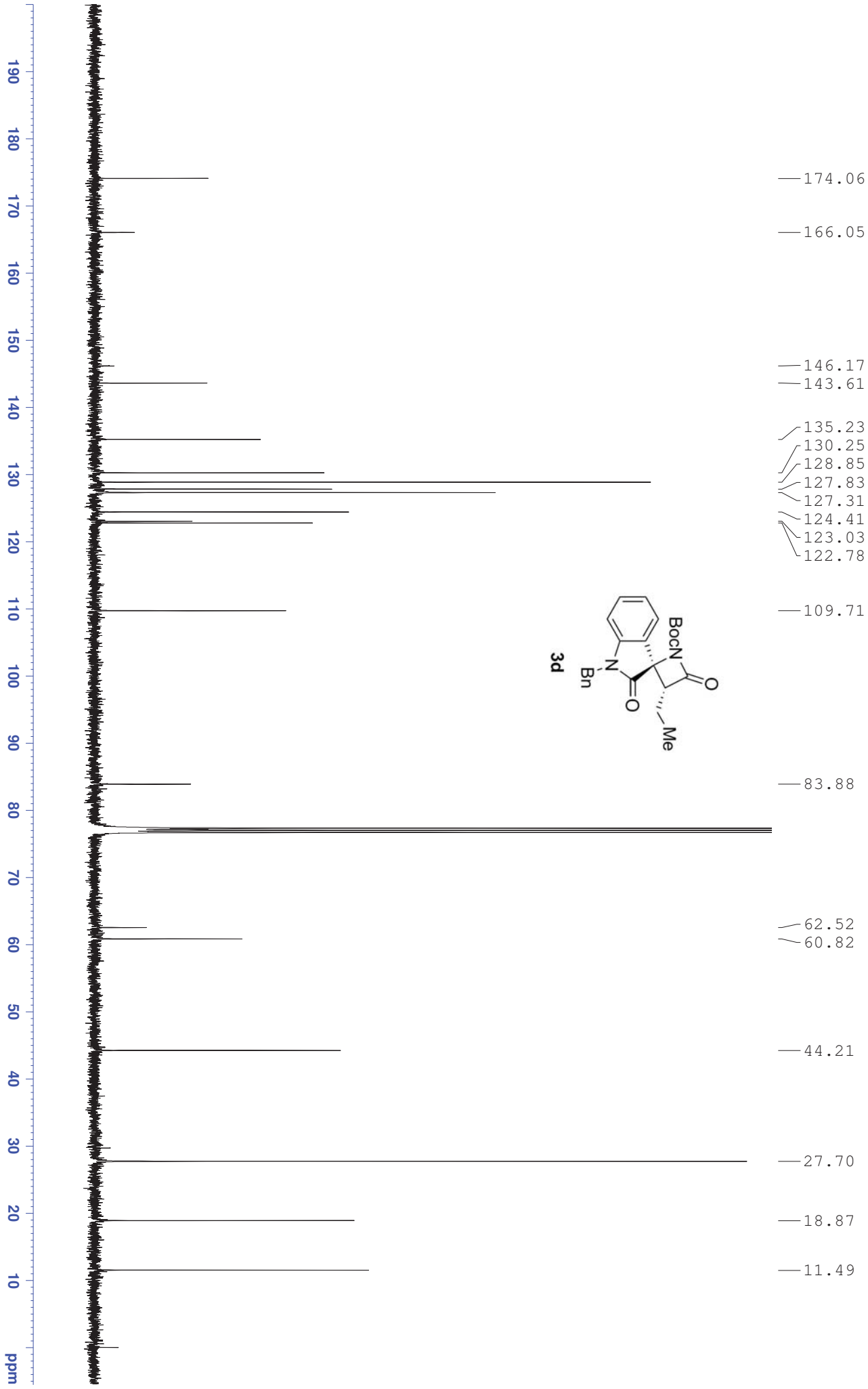
Me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BocN
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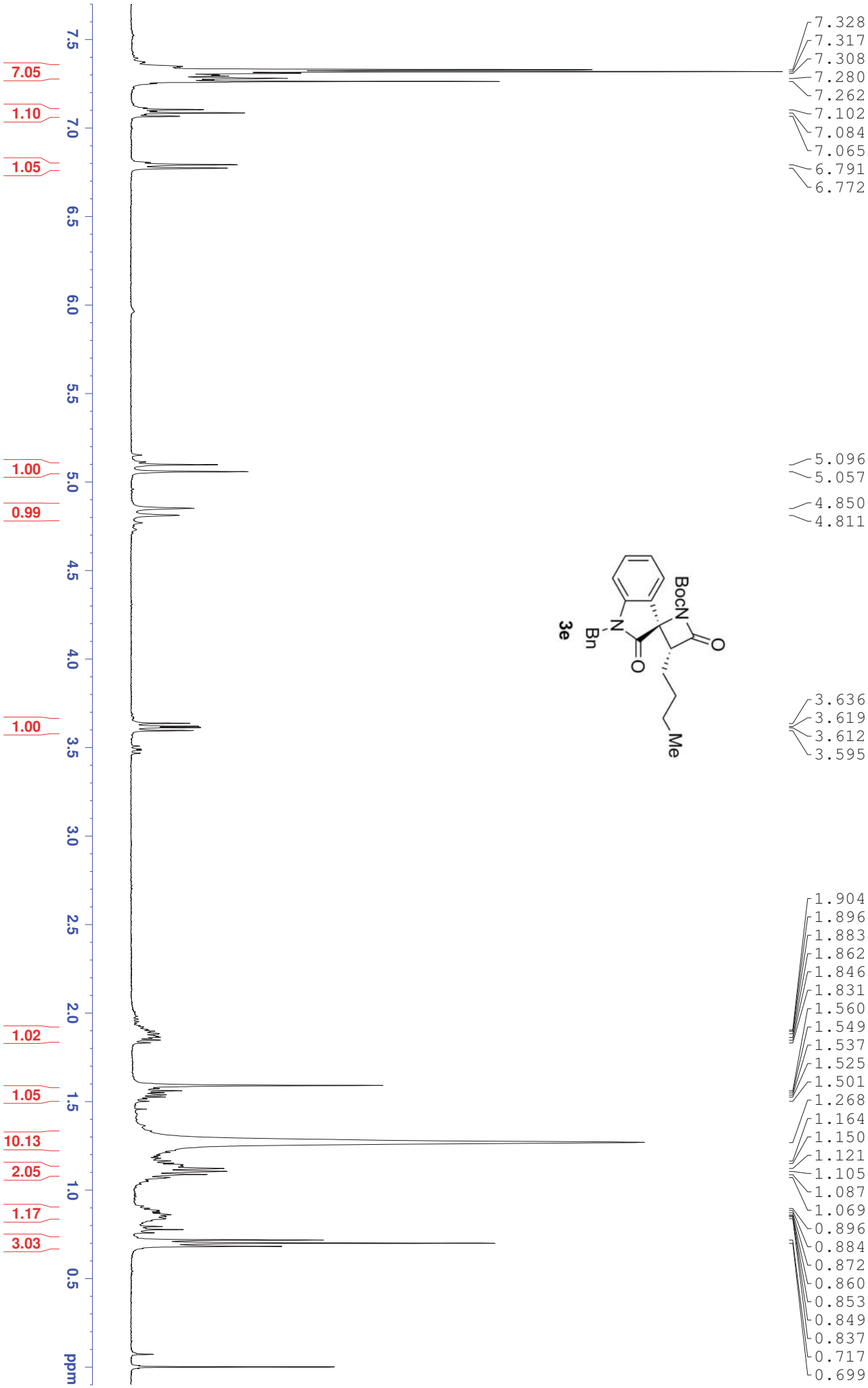
O
Me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3.612
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1.883
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1.831
1.560
1.549
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1.525
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1.087
1.069
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1.05
1.00
0.99
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1.17
3.03
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N
Bn
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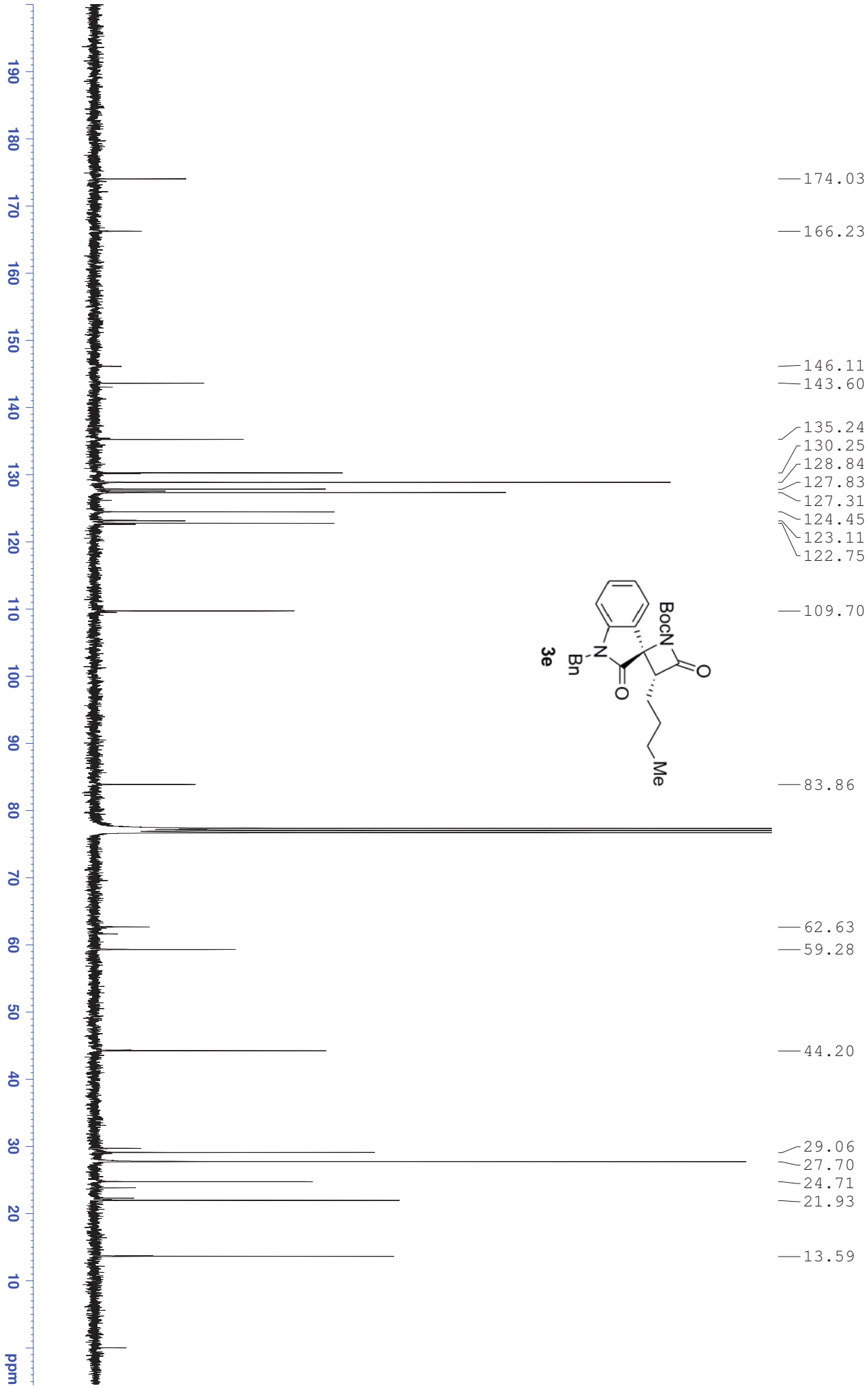
Me
3e
ppm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Me
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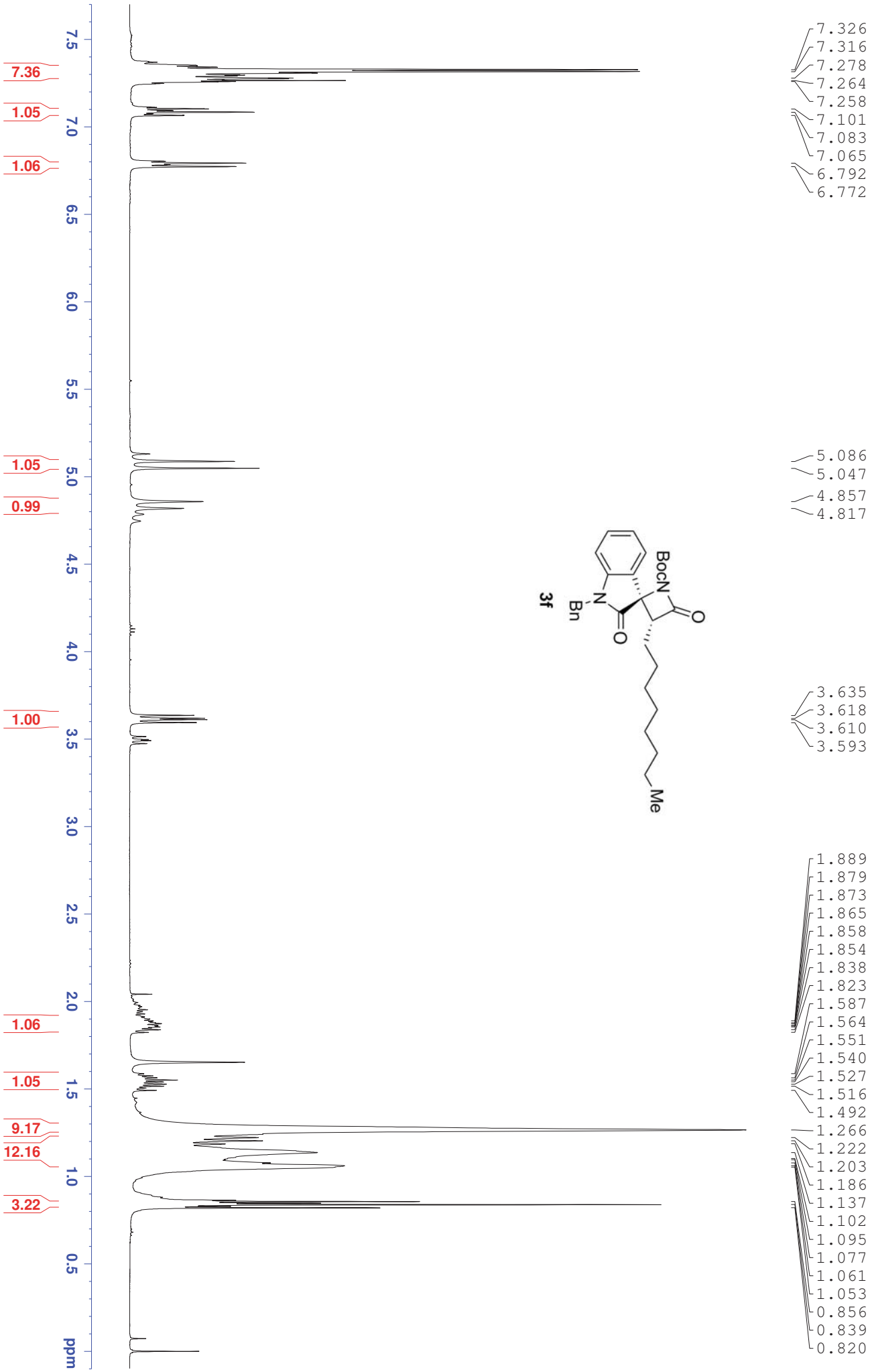
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1.873
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1.266
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1.05
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Bn
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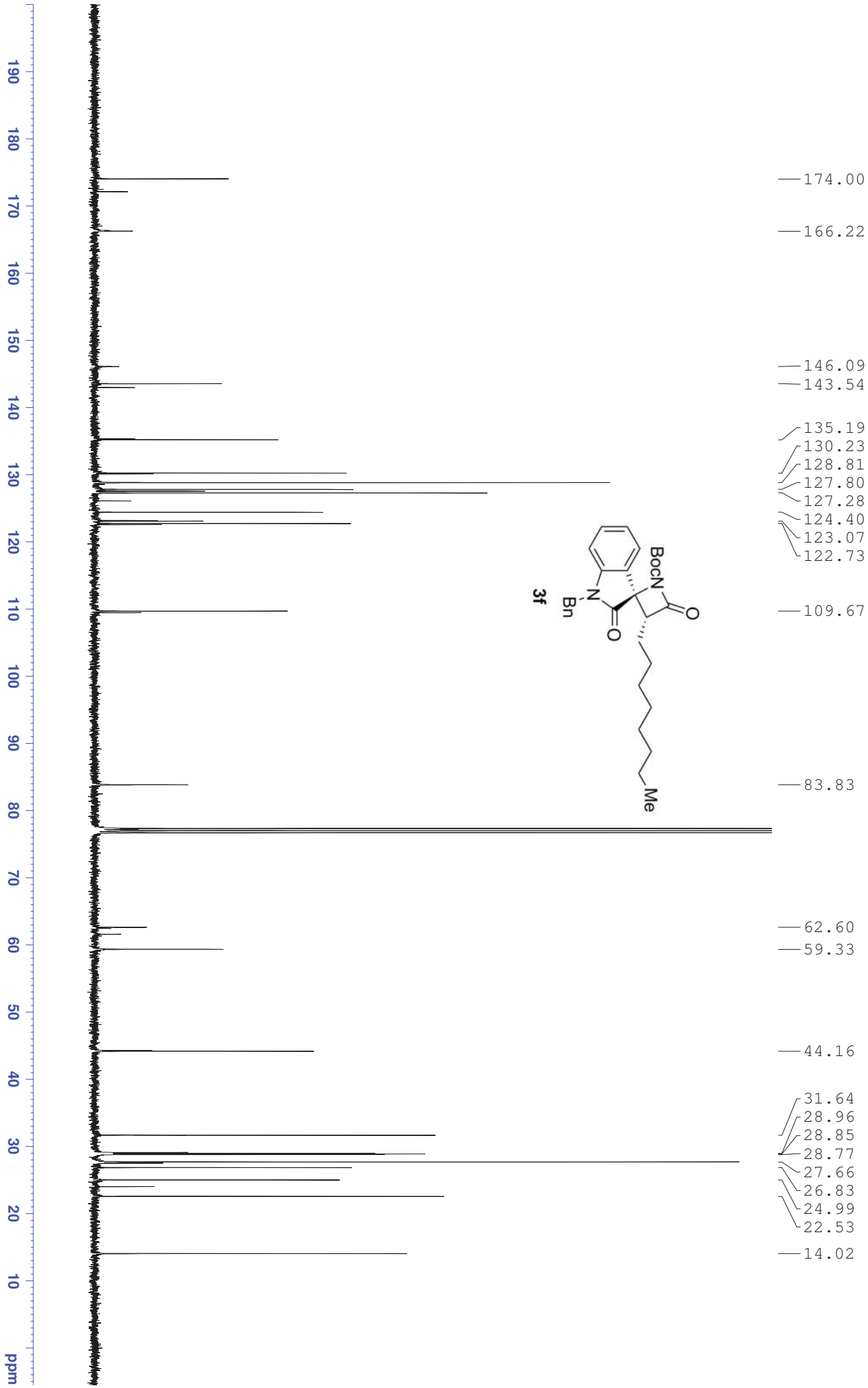
O
Me
3f
ppm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3f
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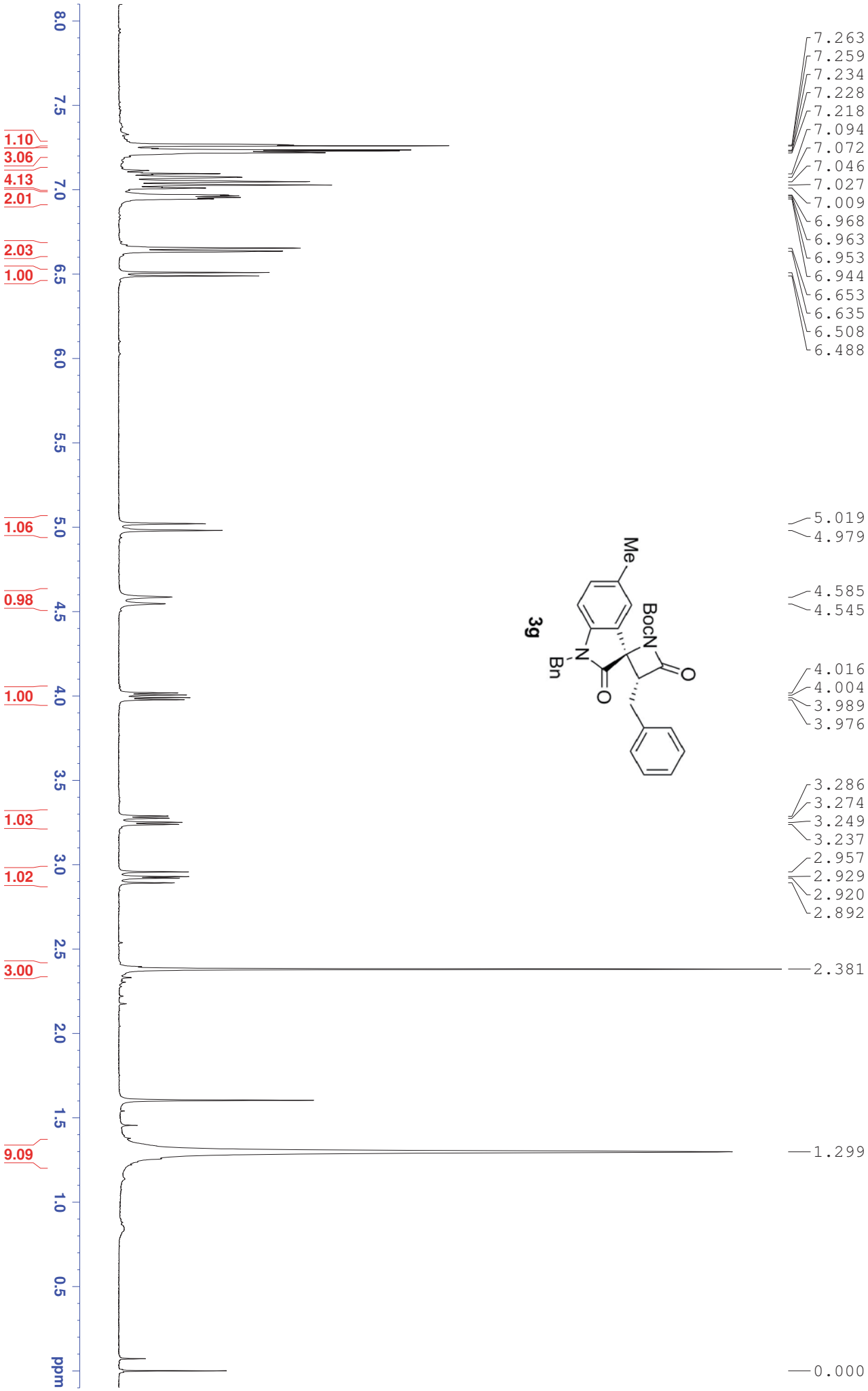
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1.02
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7.027
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6.953
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6.635
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6.488
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4.545
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3.237
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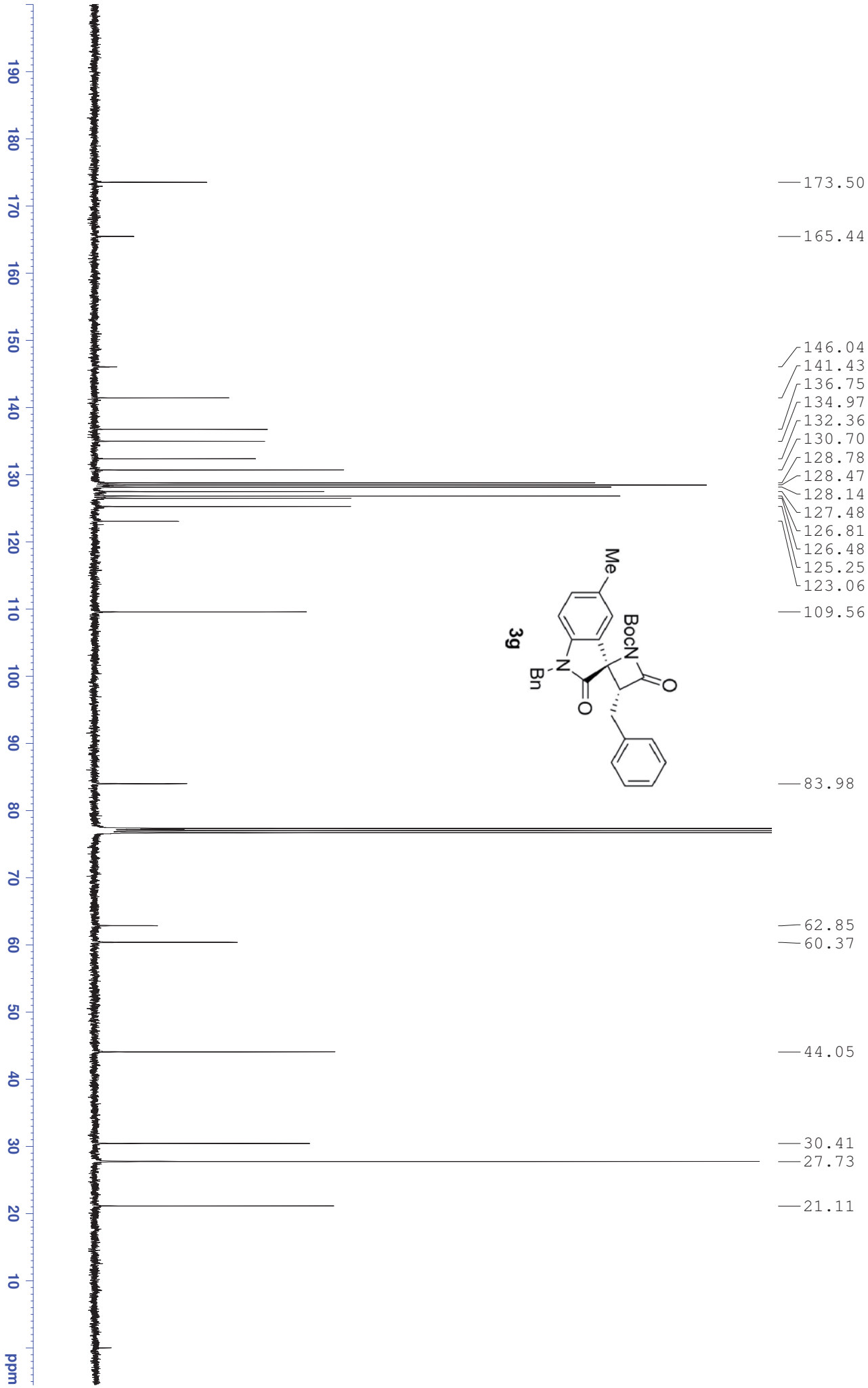
Bn
O
O
3g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30.41
27.73
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Me
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N
Bn
O
O
3g
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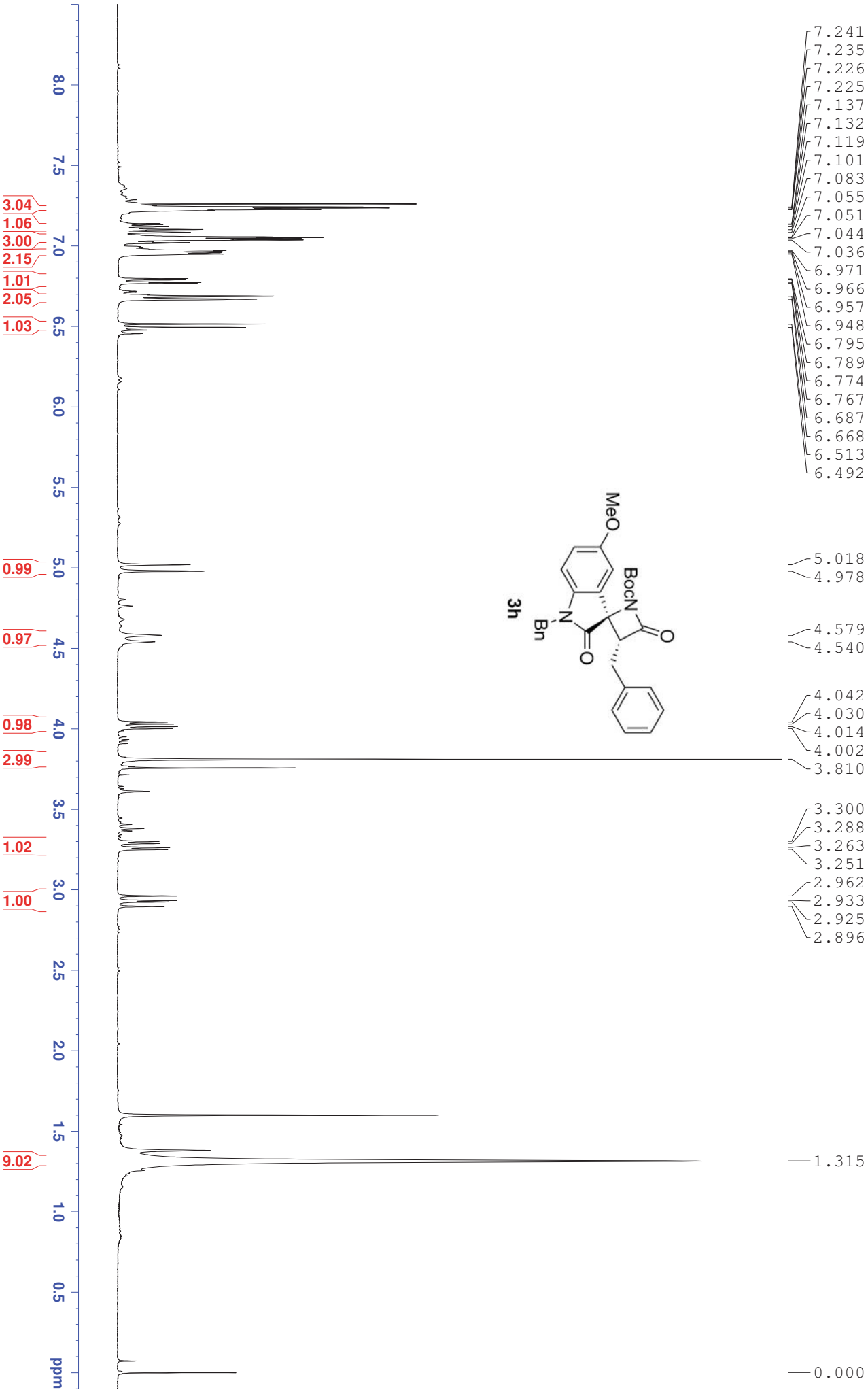
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6.492
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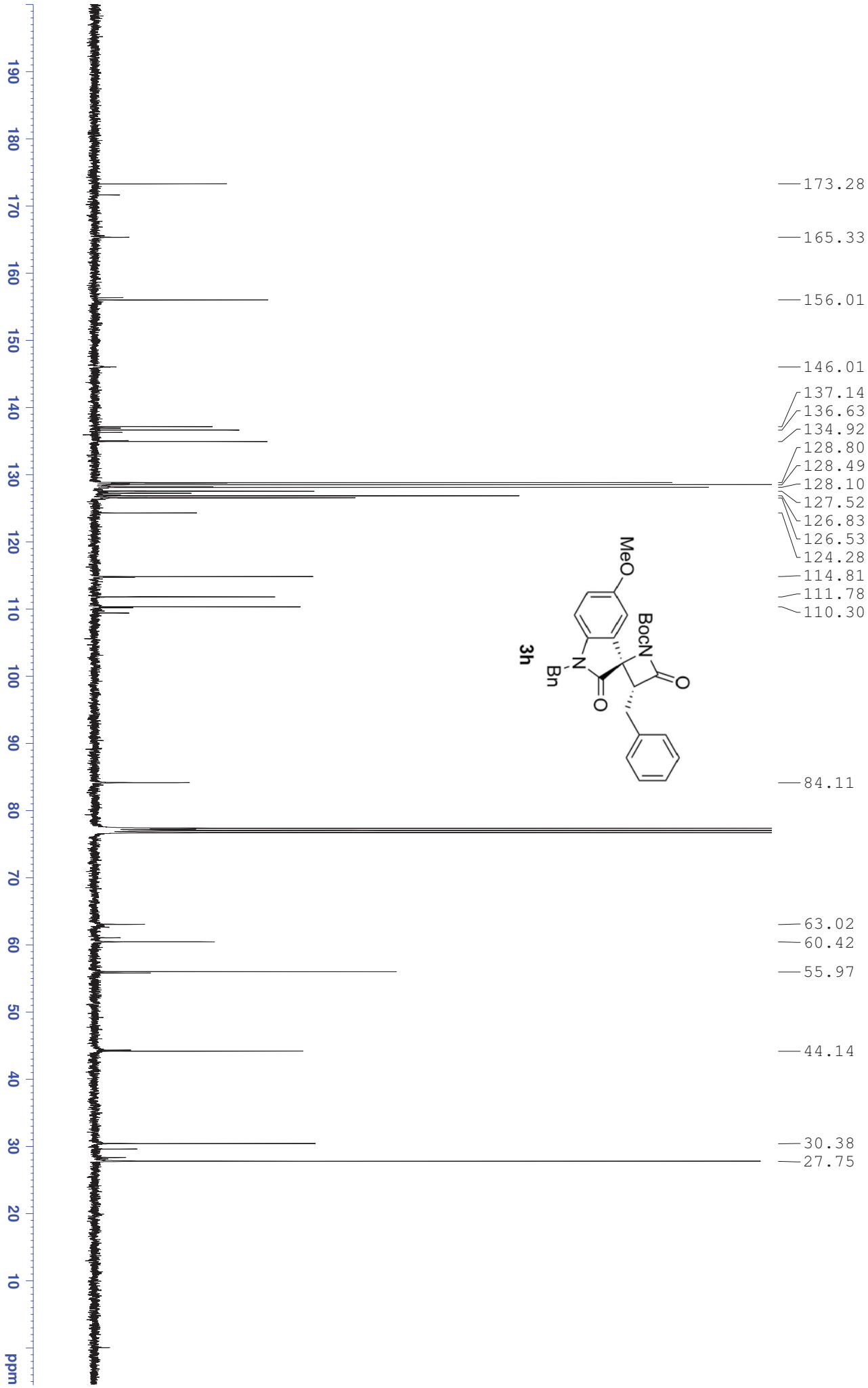
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30.38
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Bn
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O
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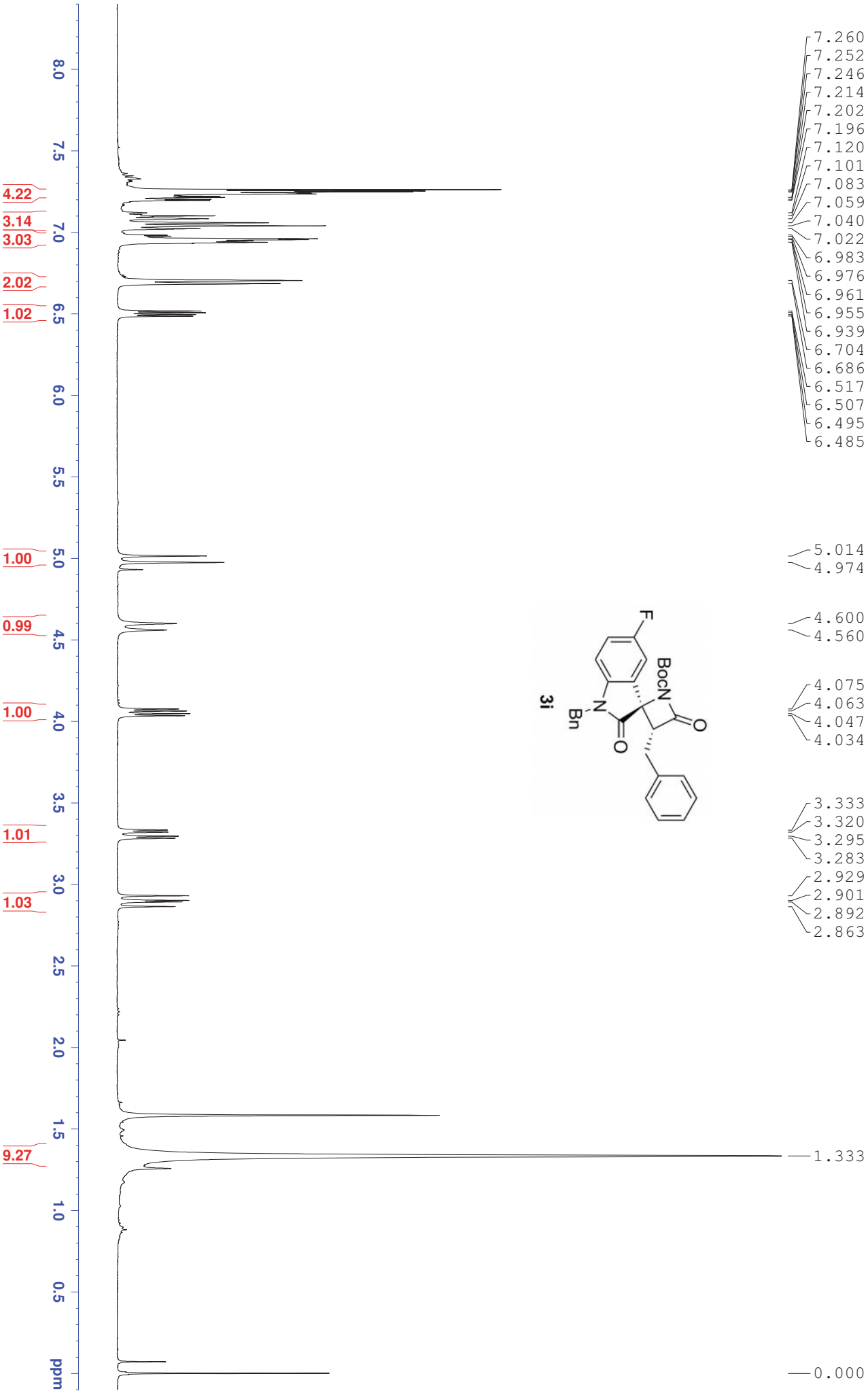
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1.01
1.03
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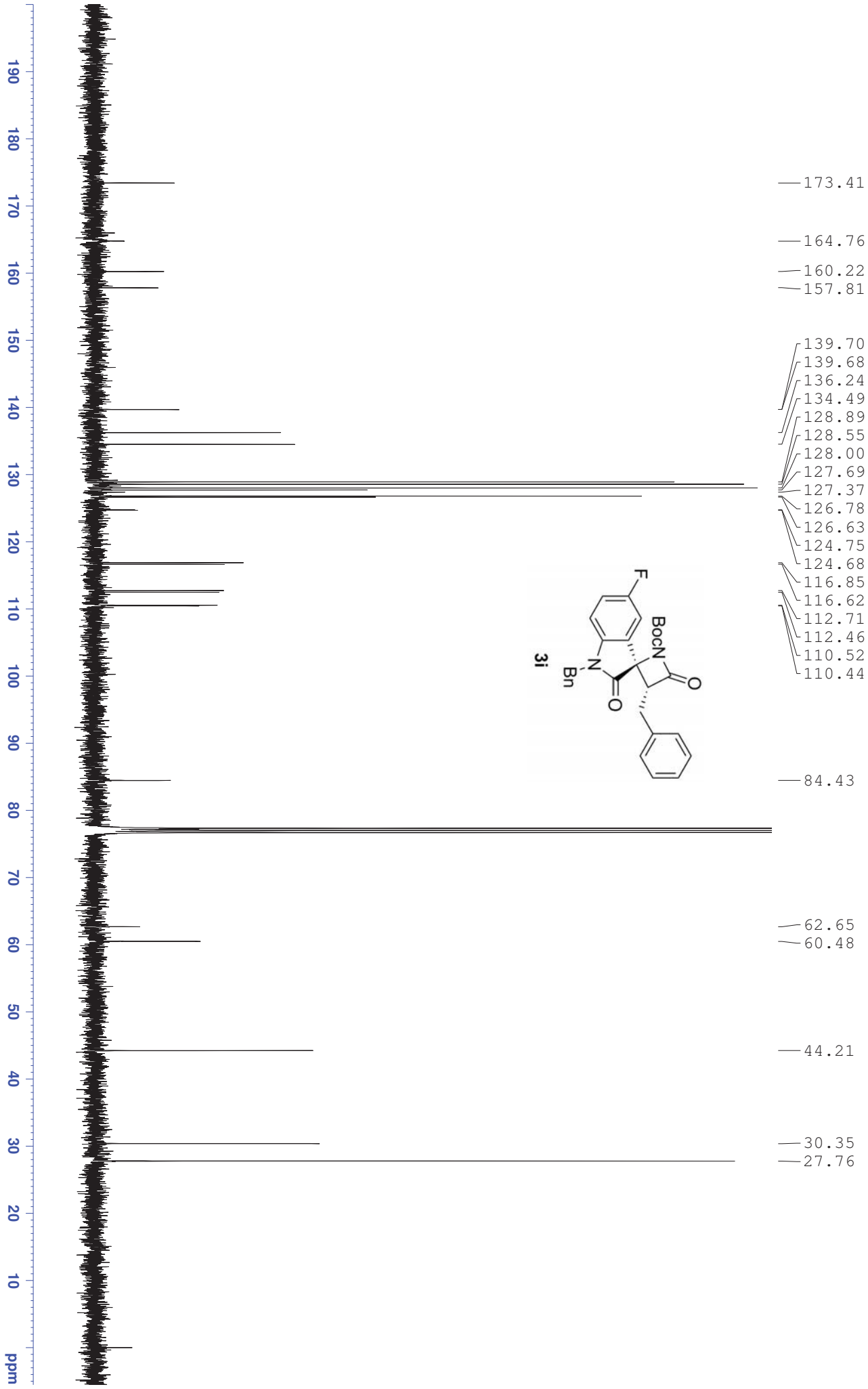
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O
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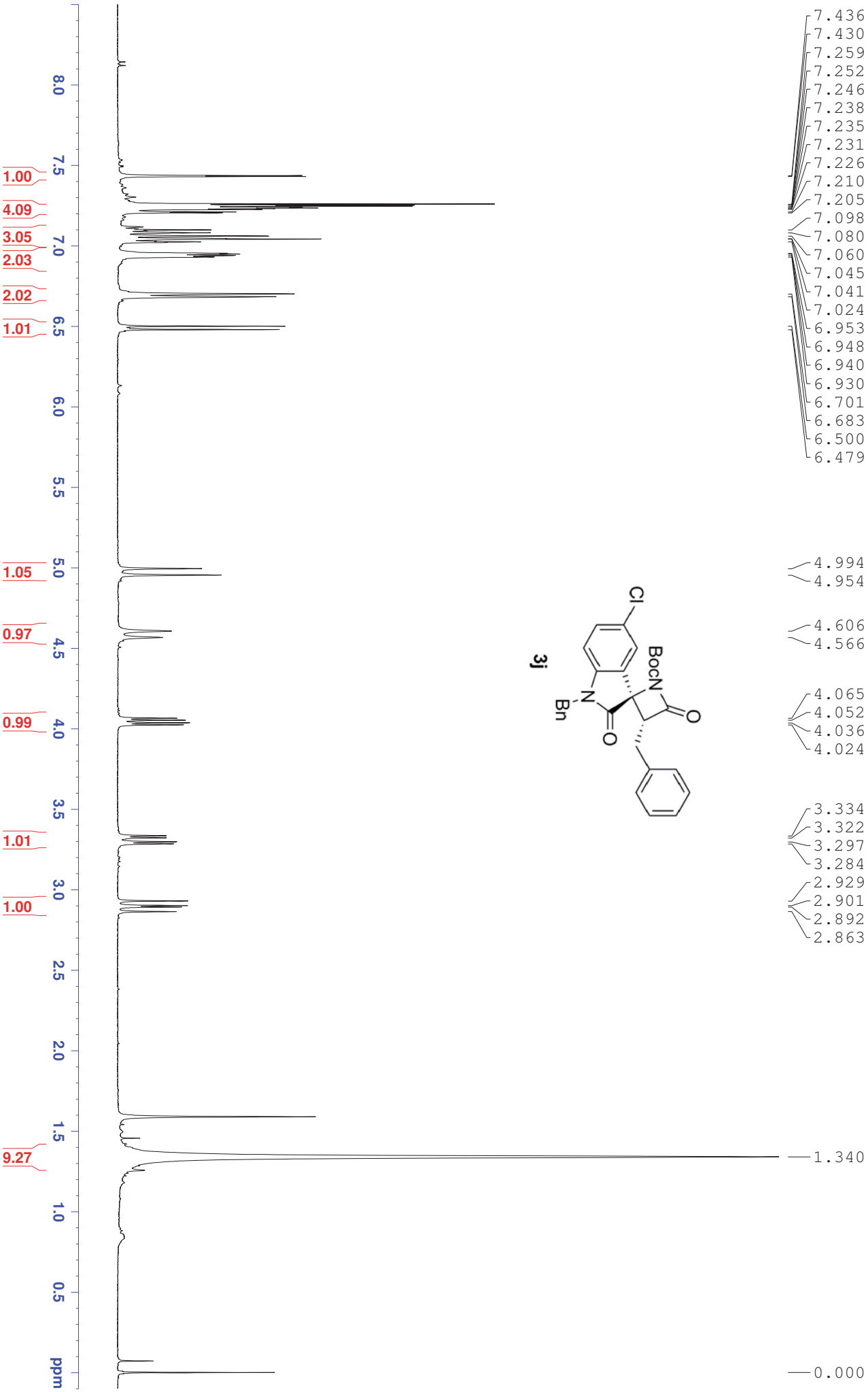
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2.901
2.892
2.863
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2.02
1.01
1.05
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0.99
1.01
1.00
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4.0
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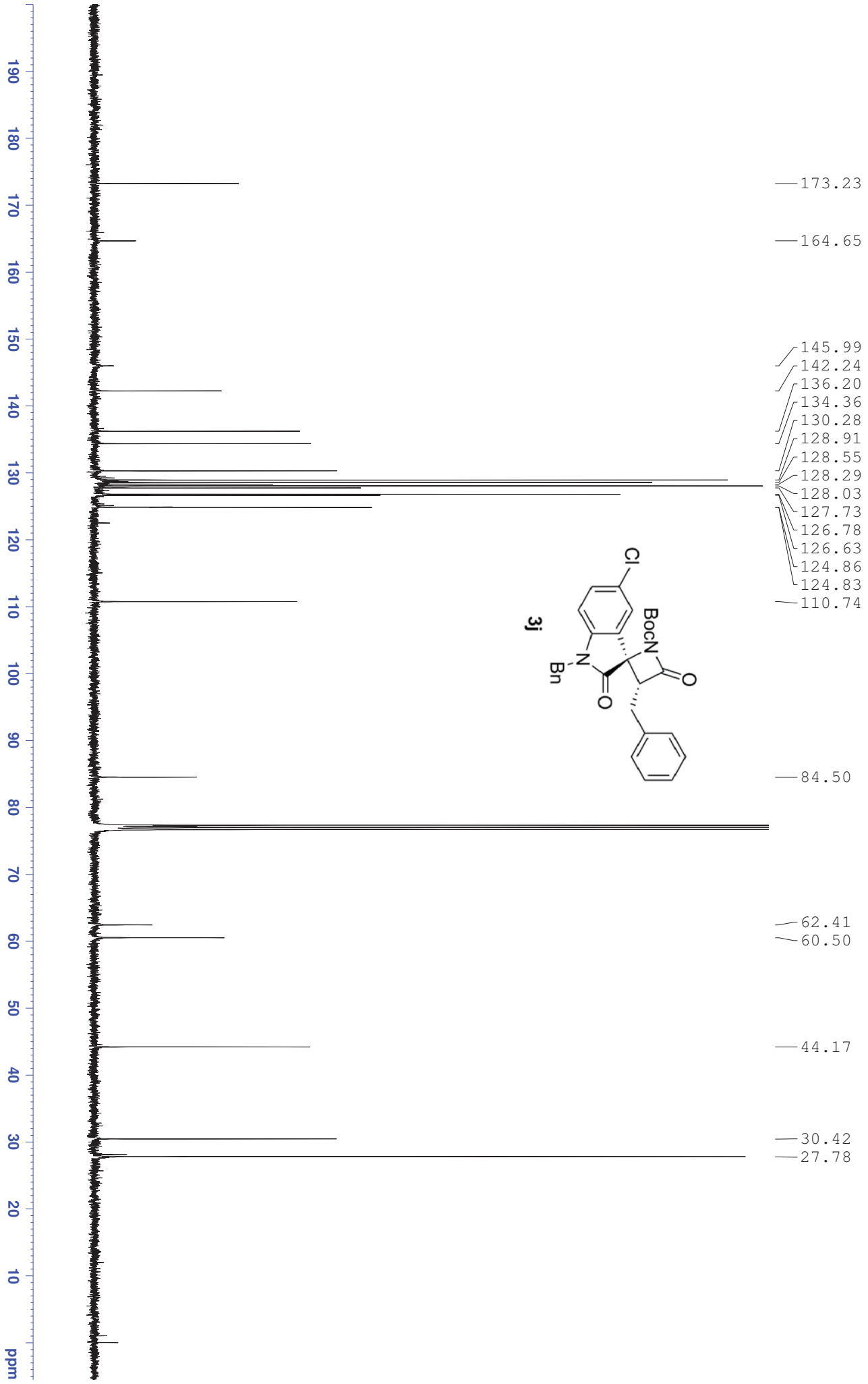
Bn
O
O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60.50
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30.42
27.78
3j
Cl
BocN
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Bn
O
O
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10
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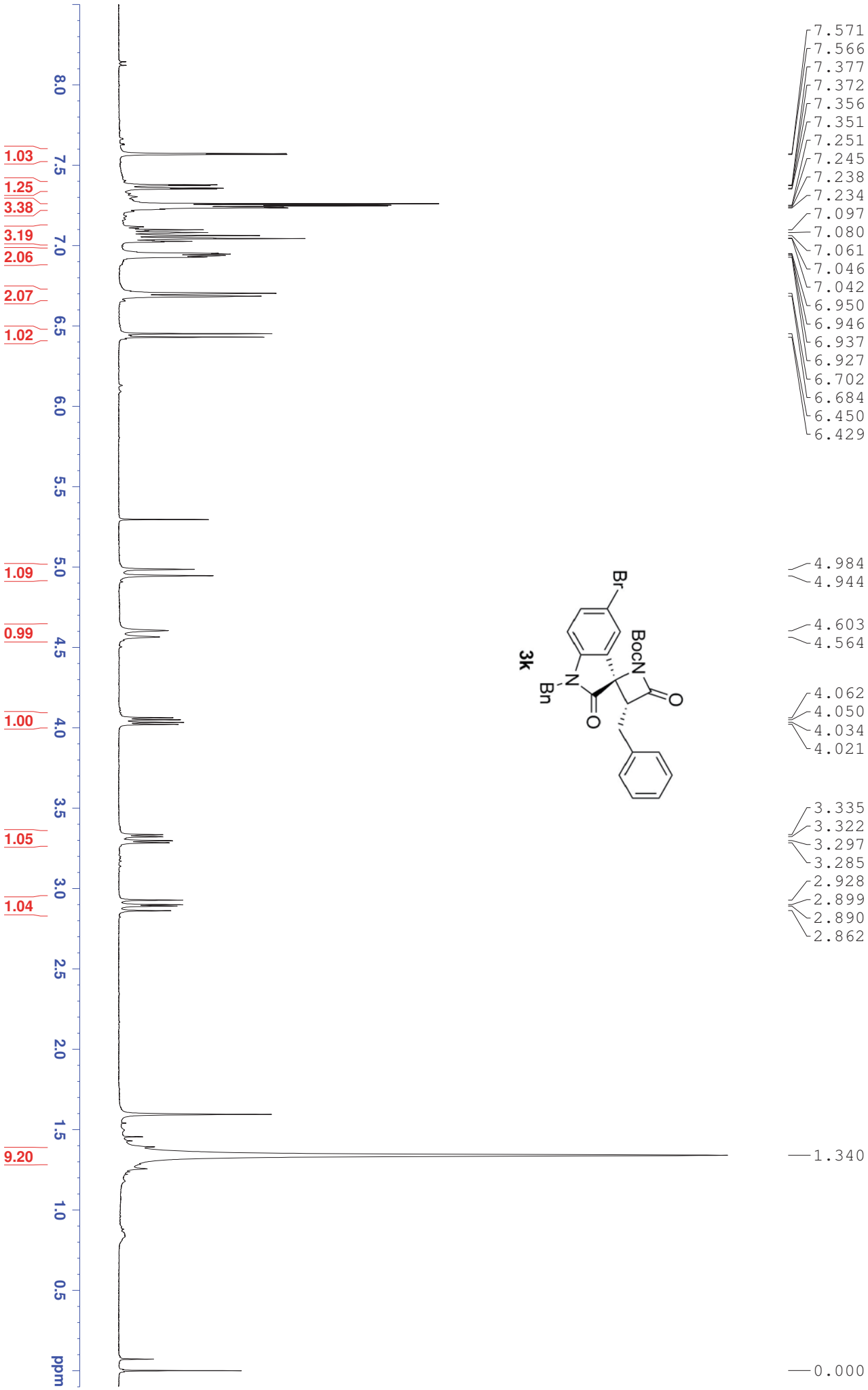
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1.05
1.04
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5.0
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2.5
2.0
1.5
1.0
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Bn
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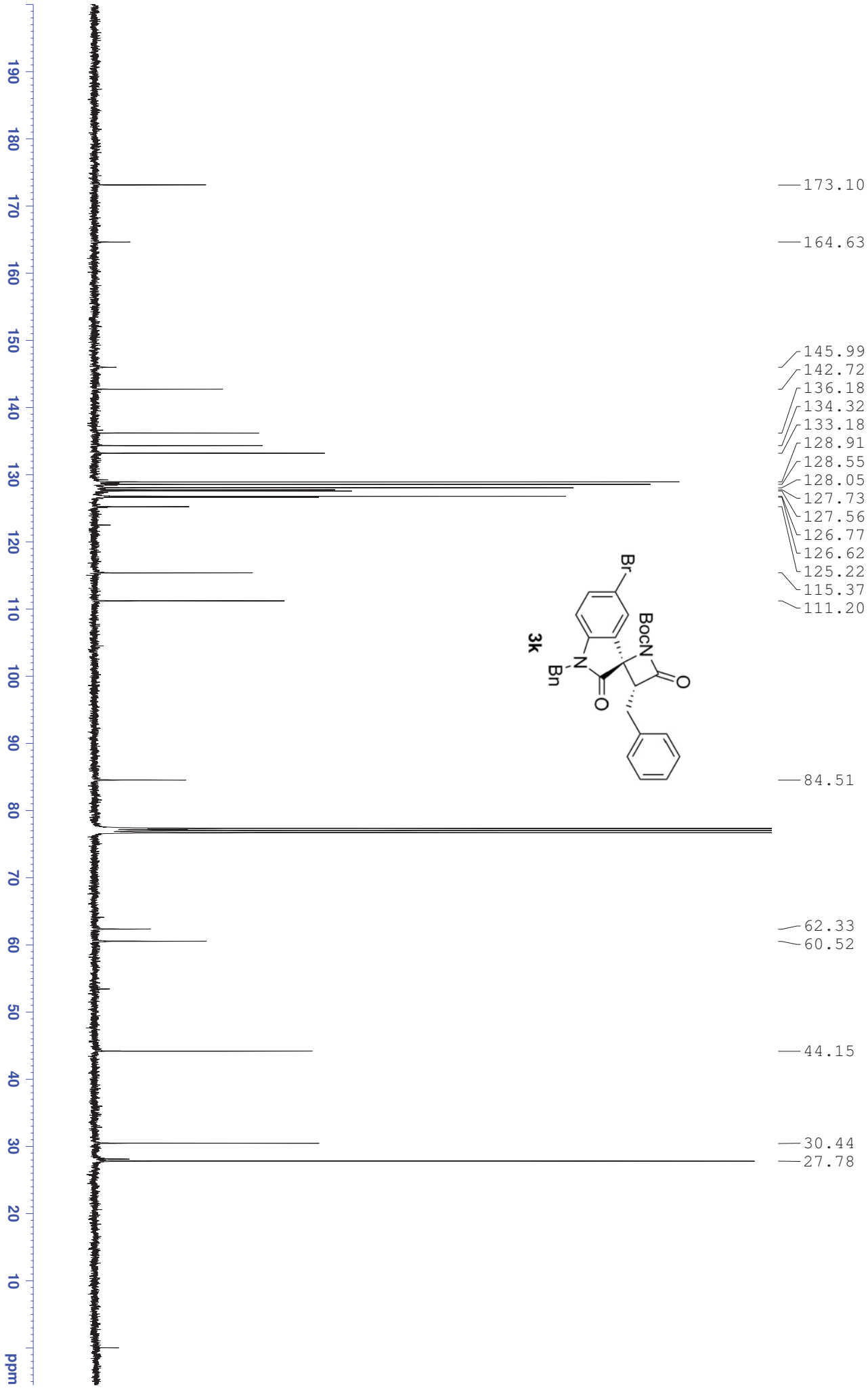
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30.44
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Bn
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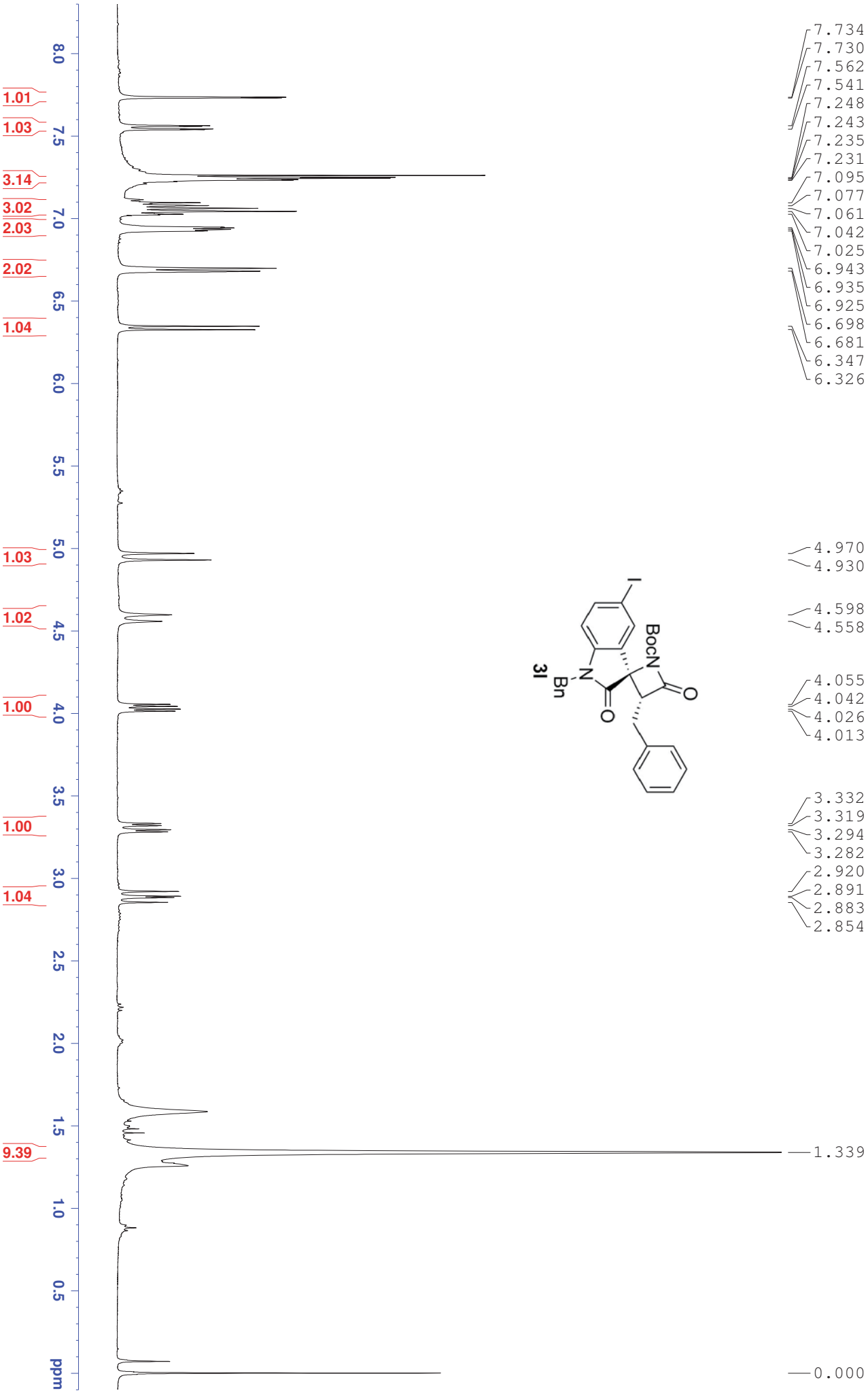
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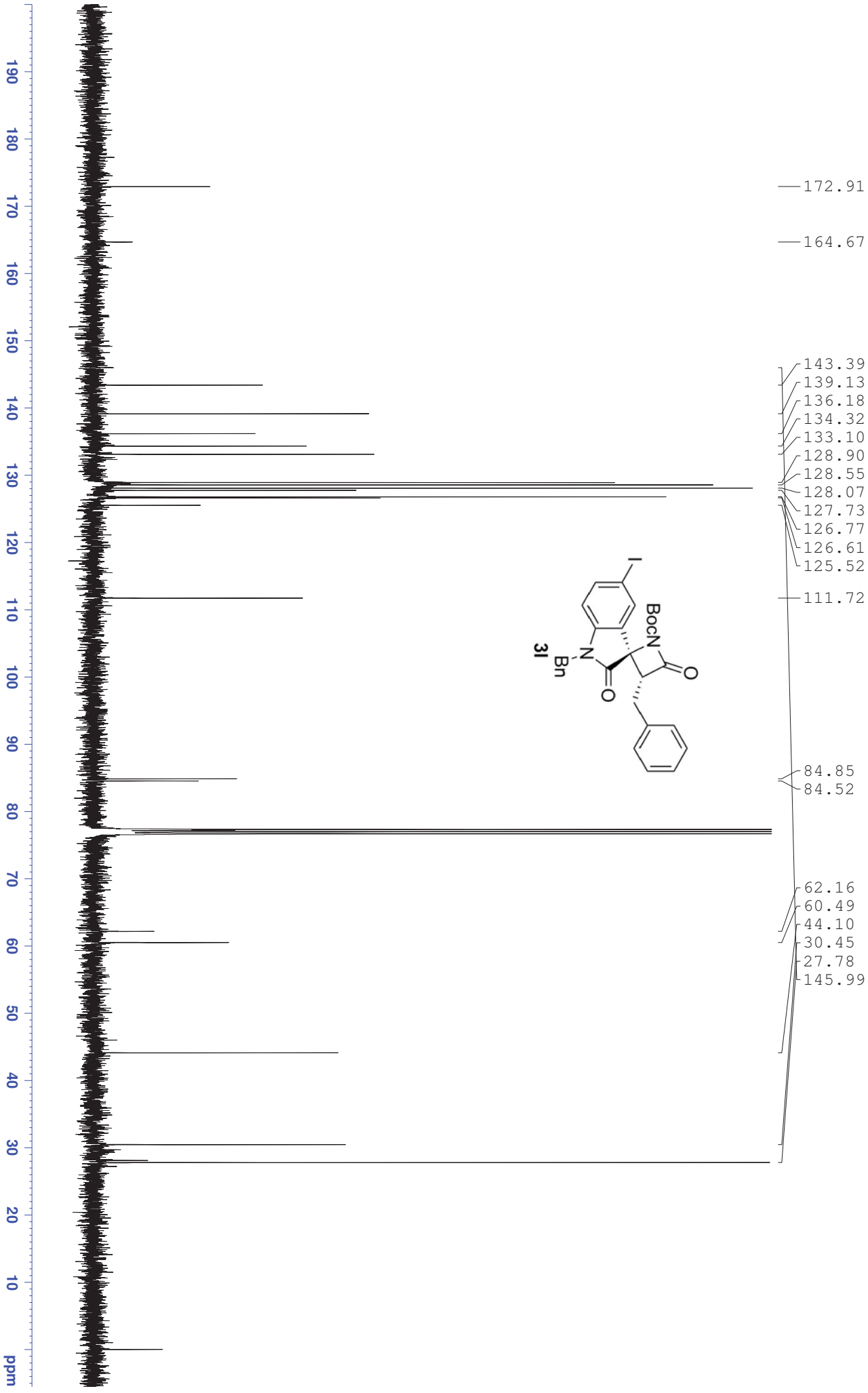
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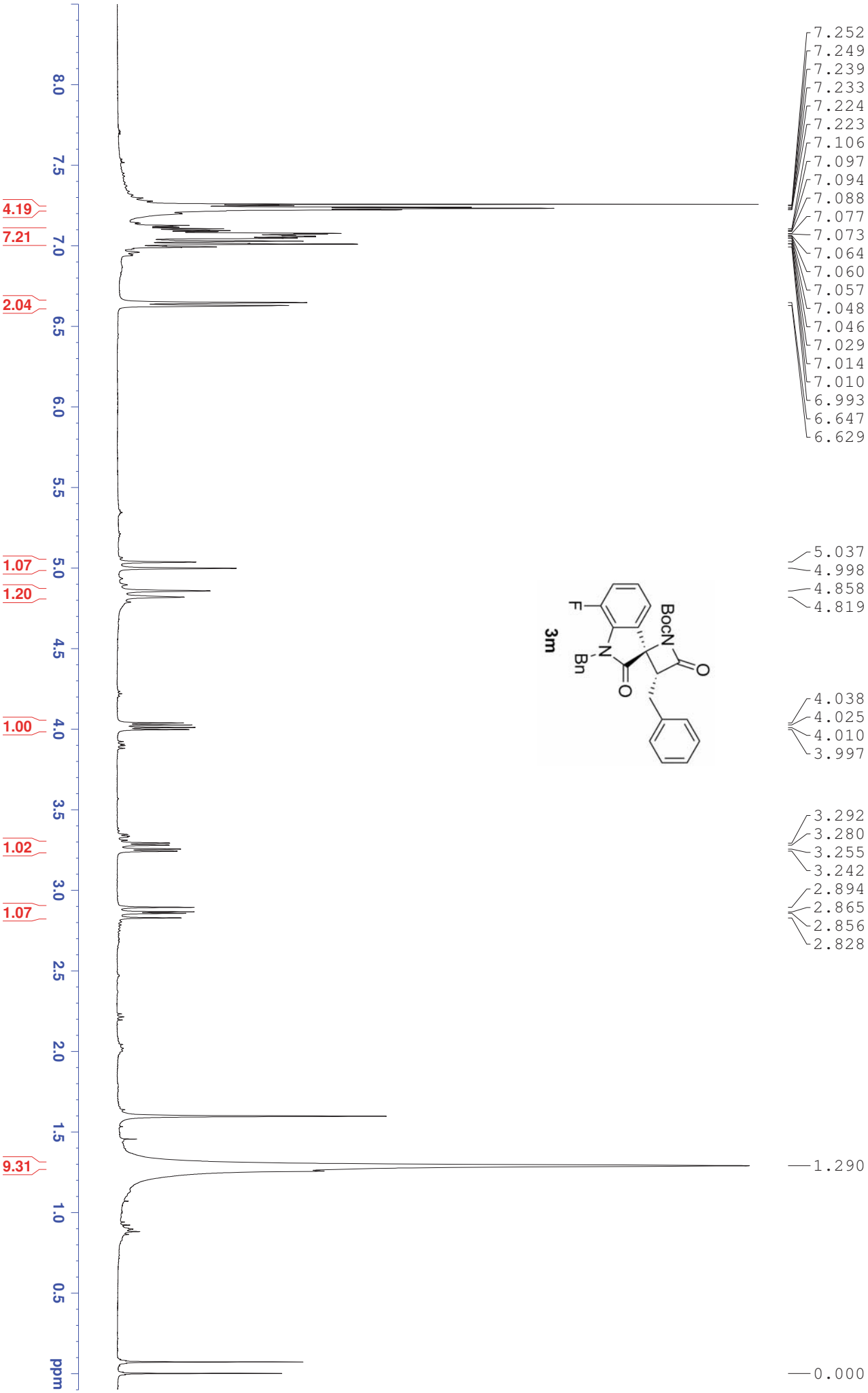
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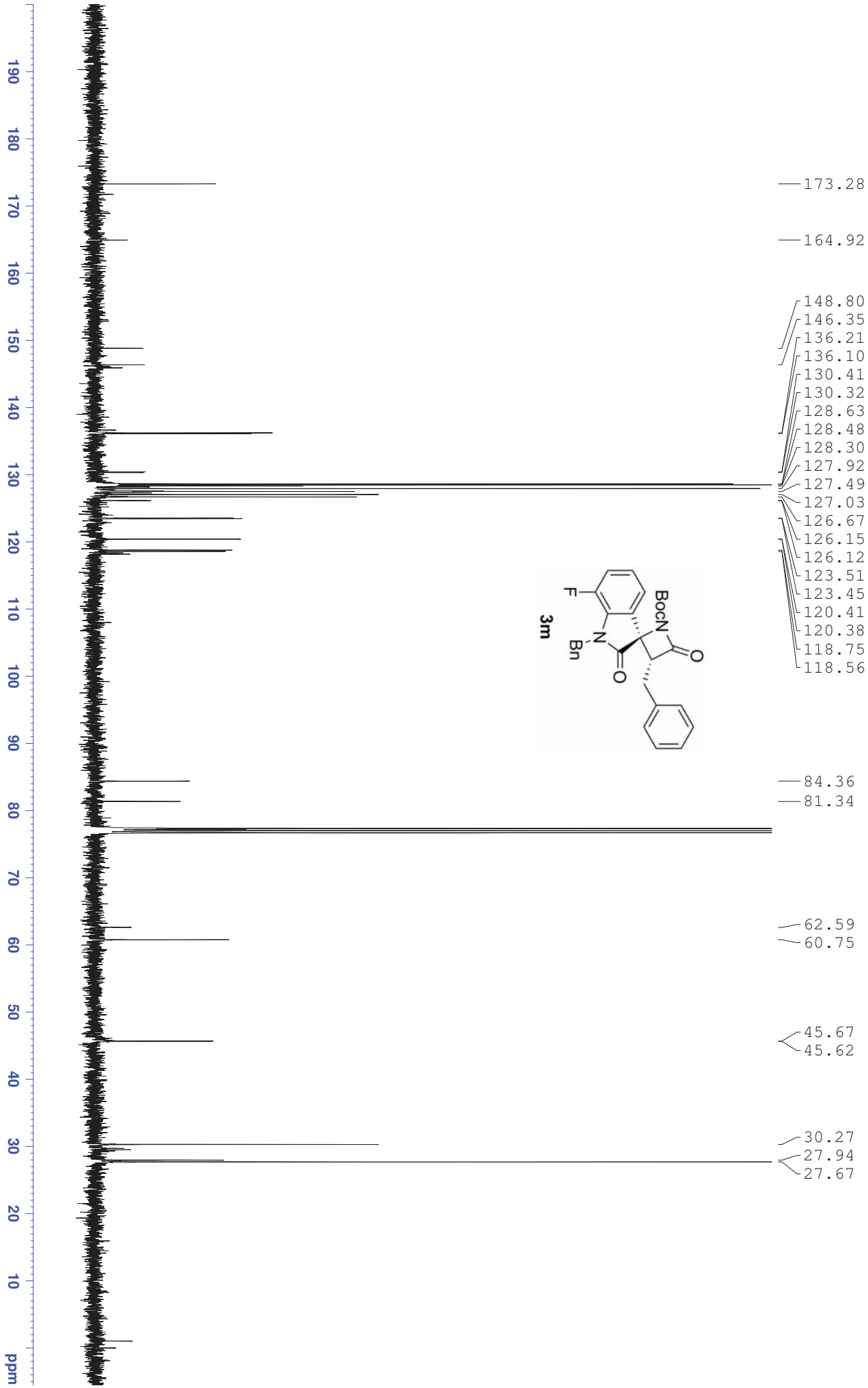
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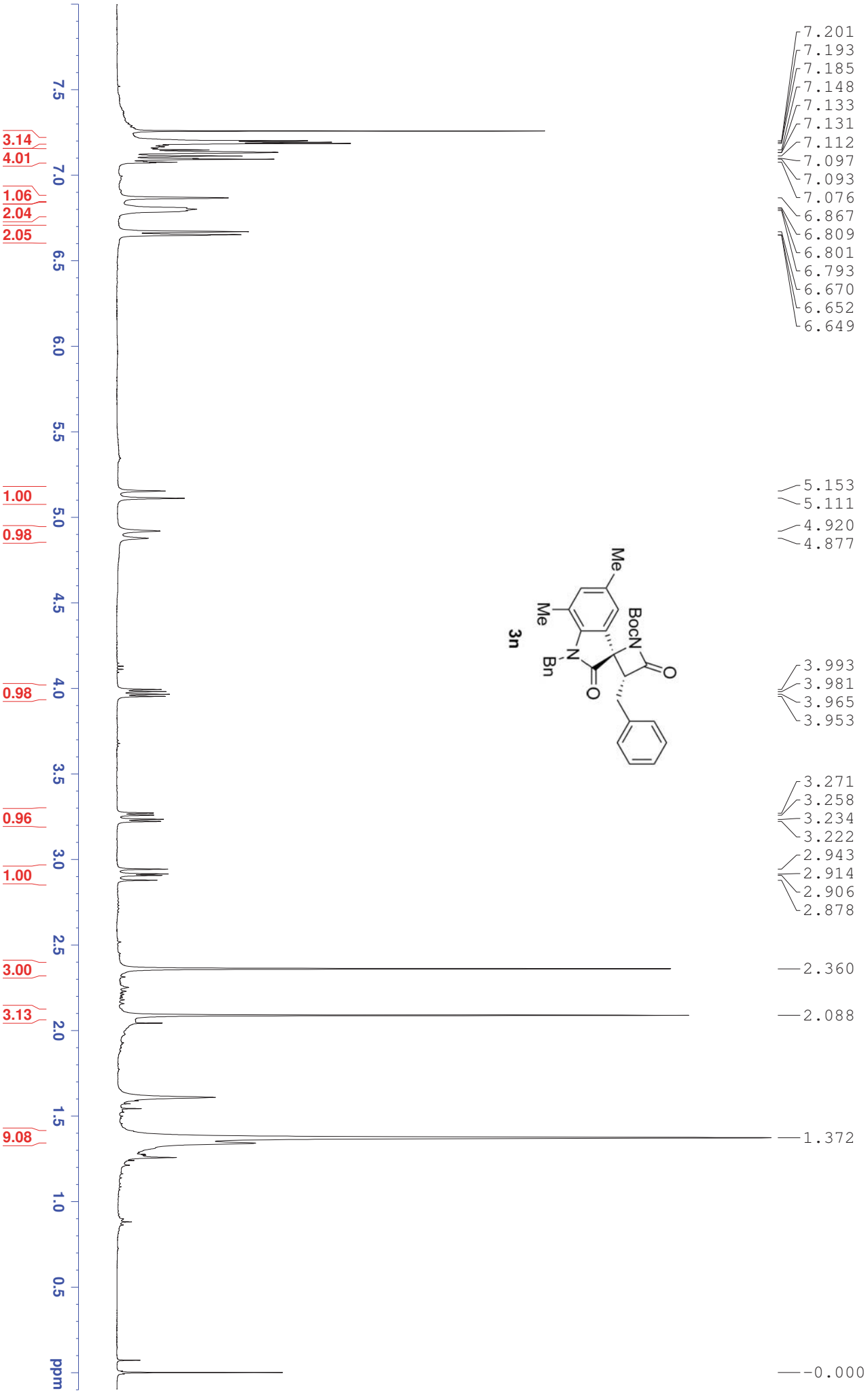
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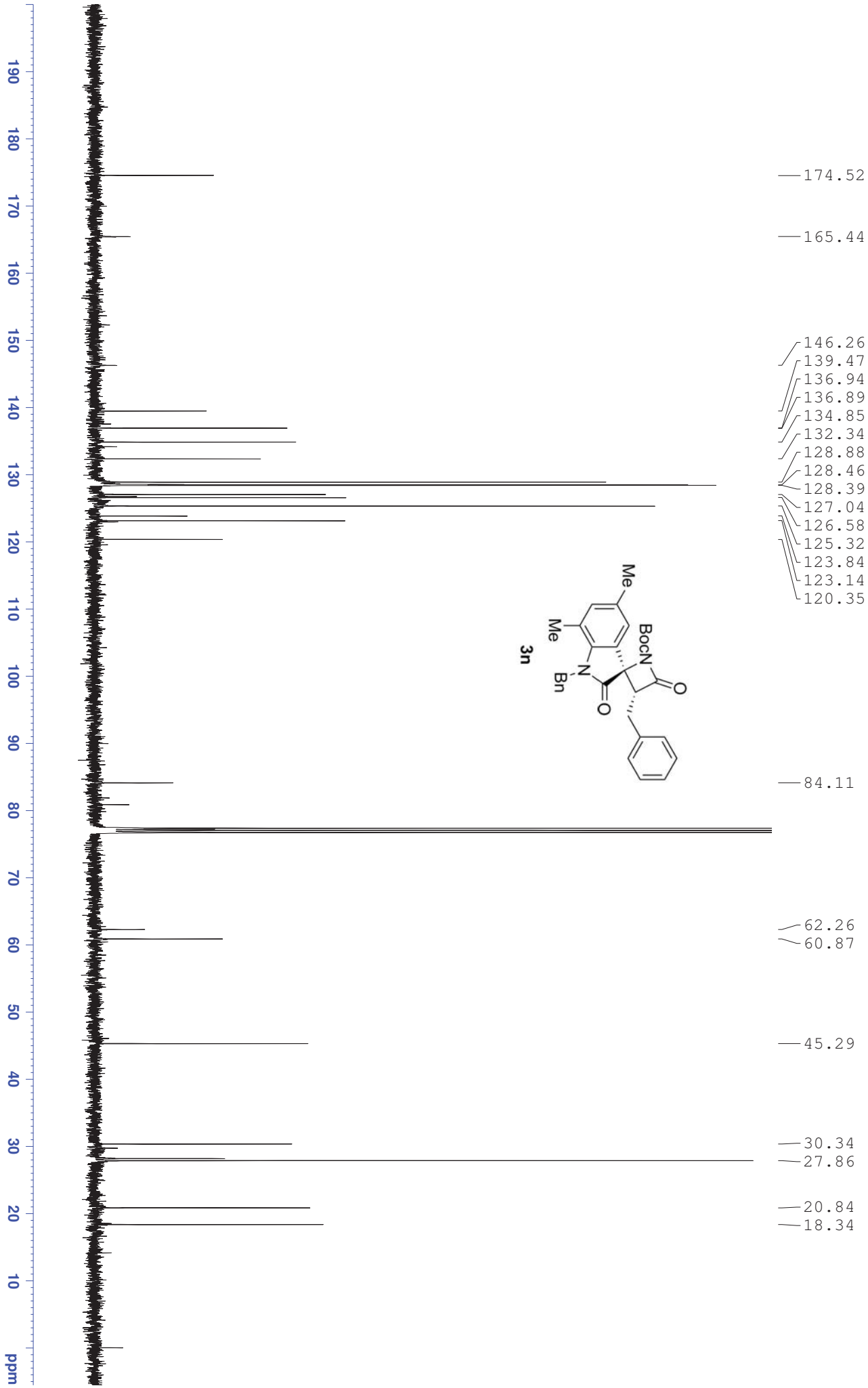
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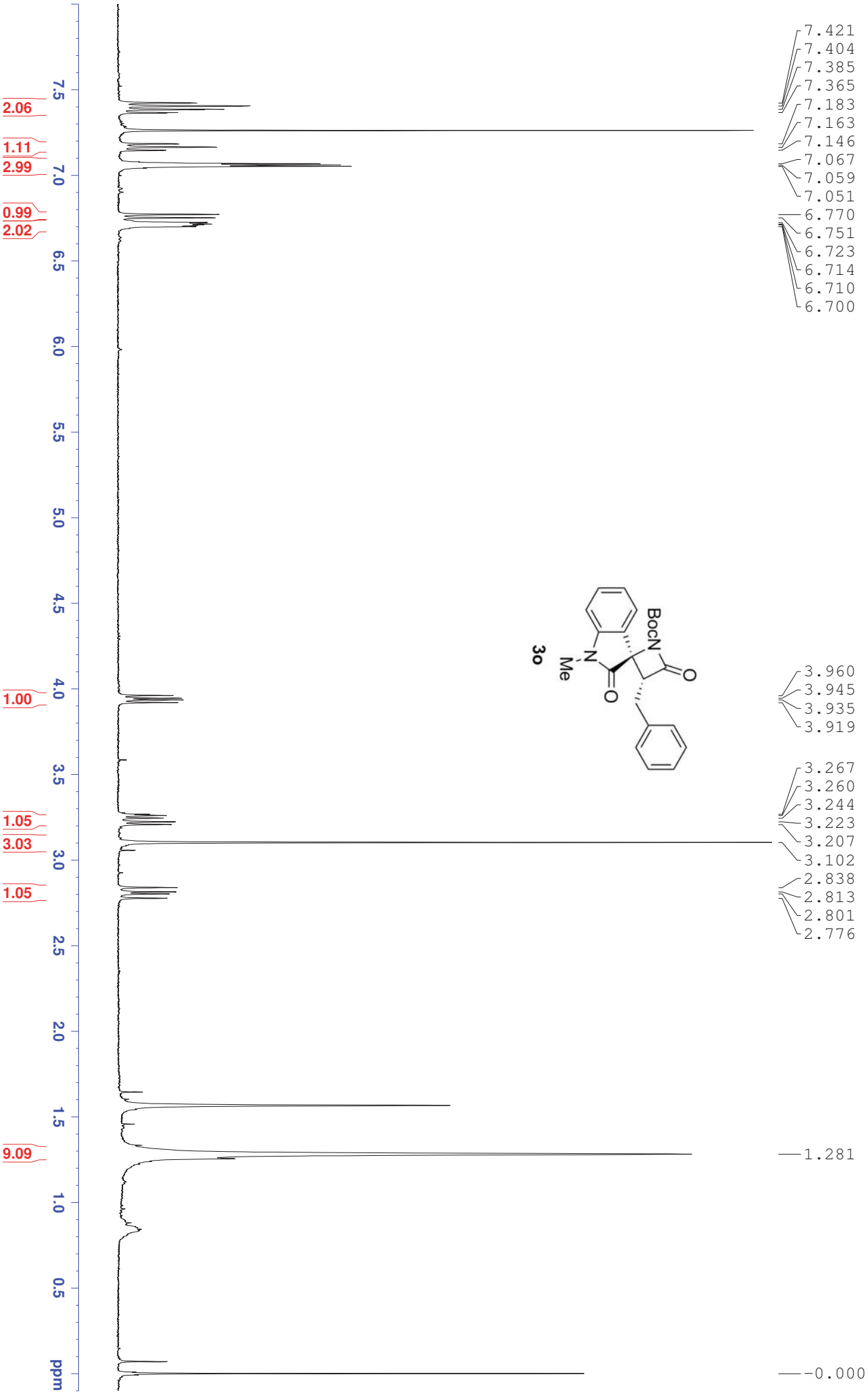
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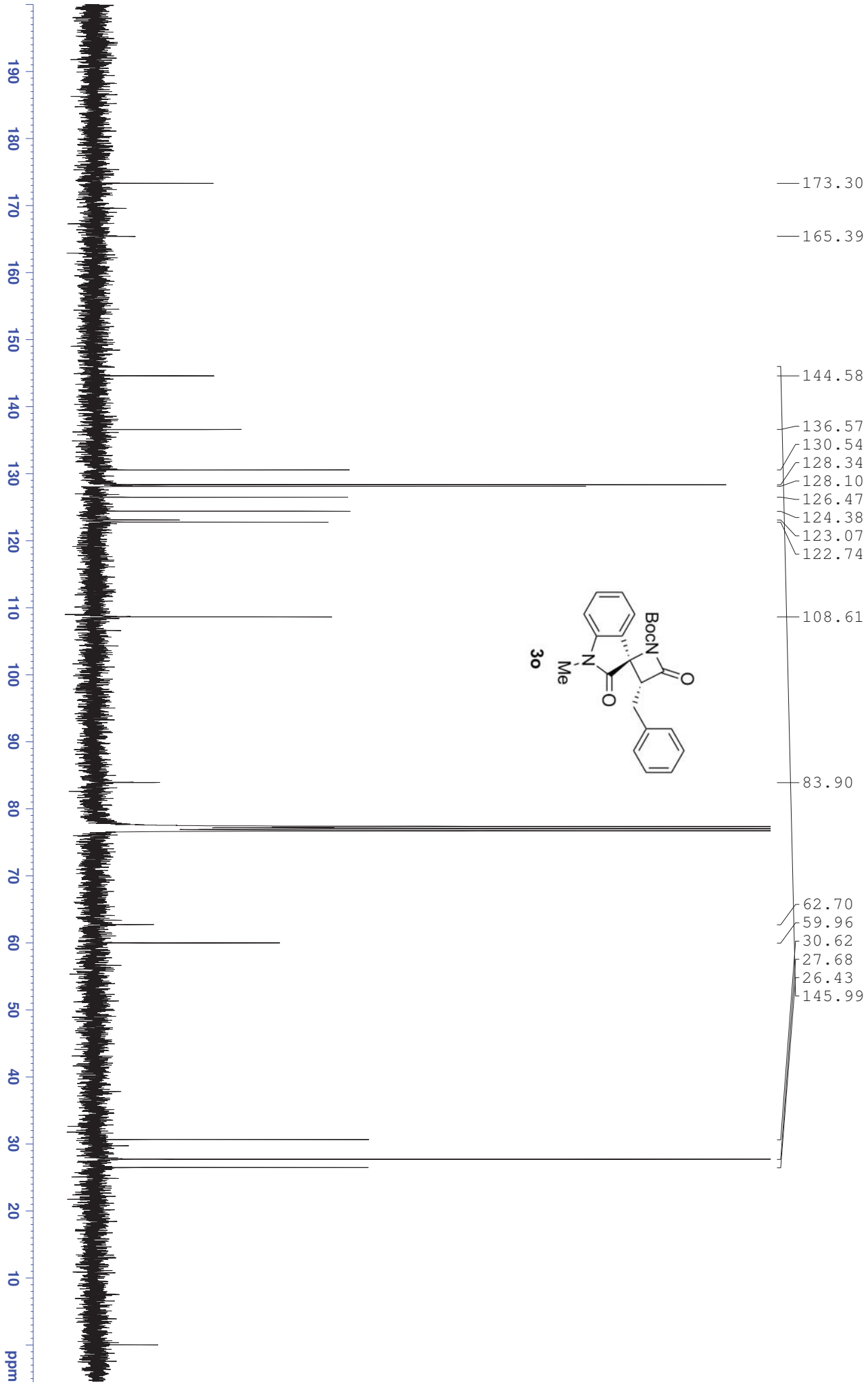
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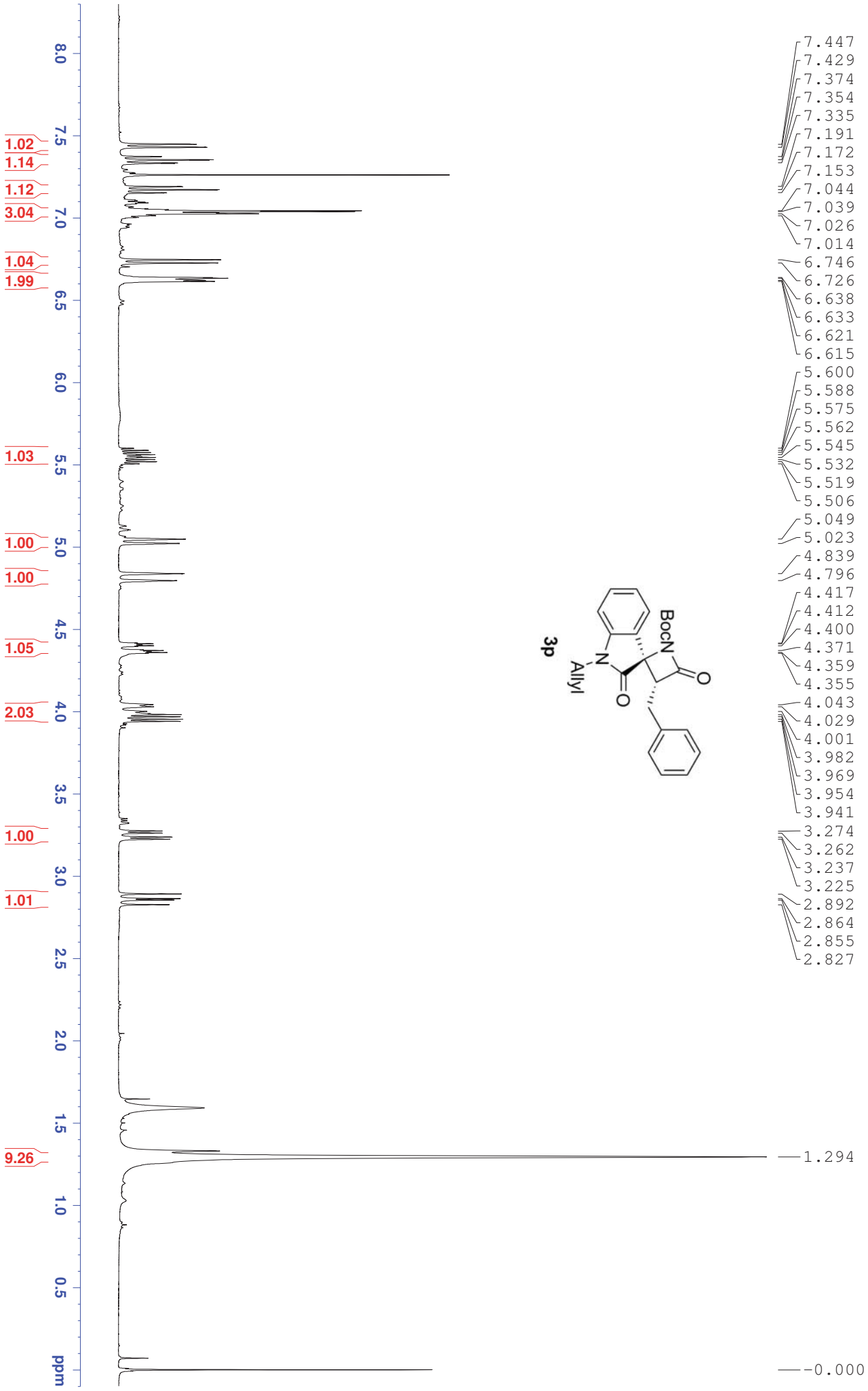
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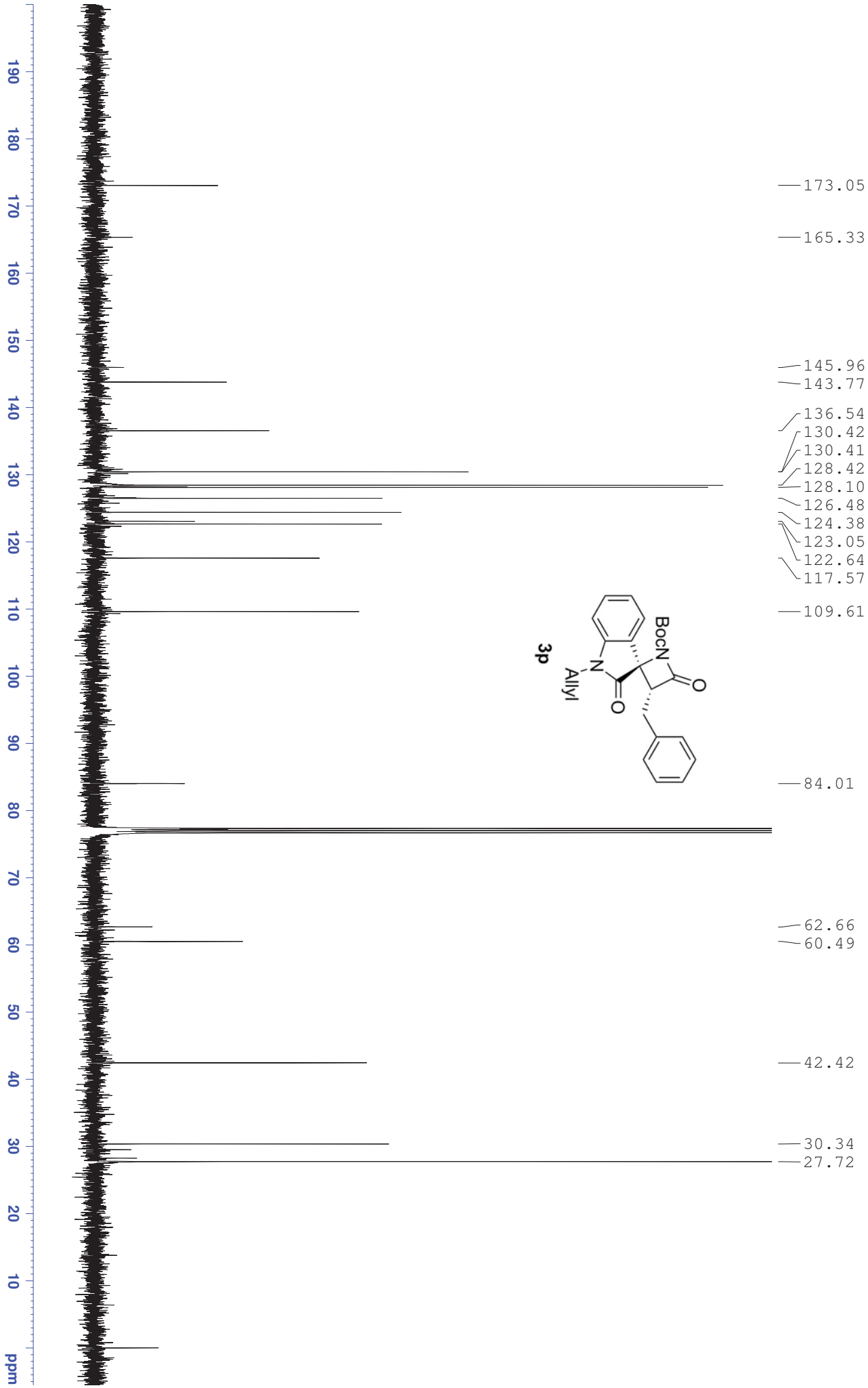
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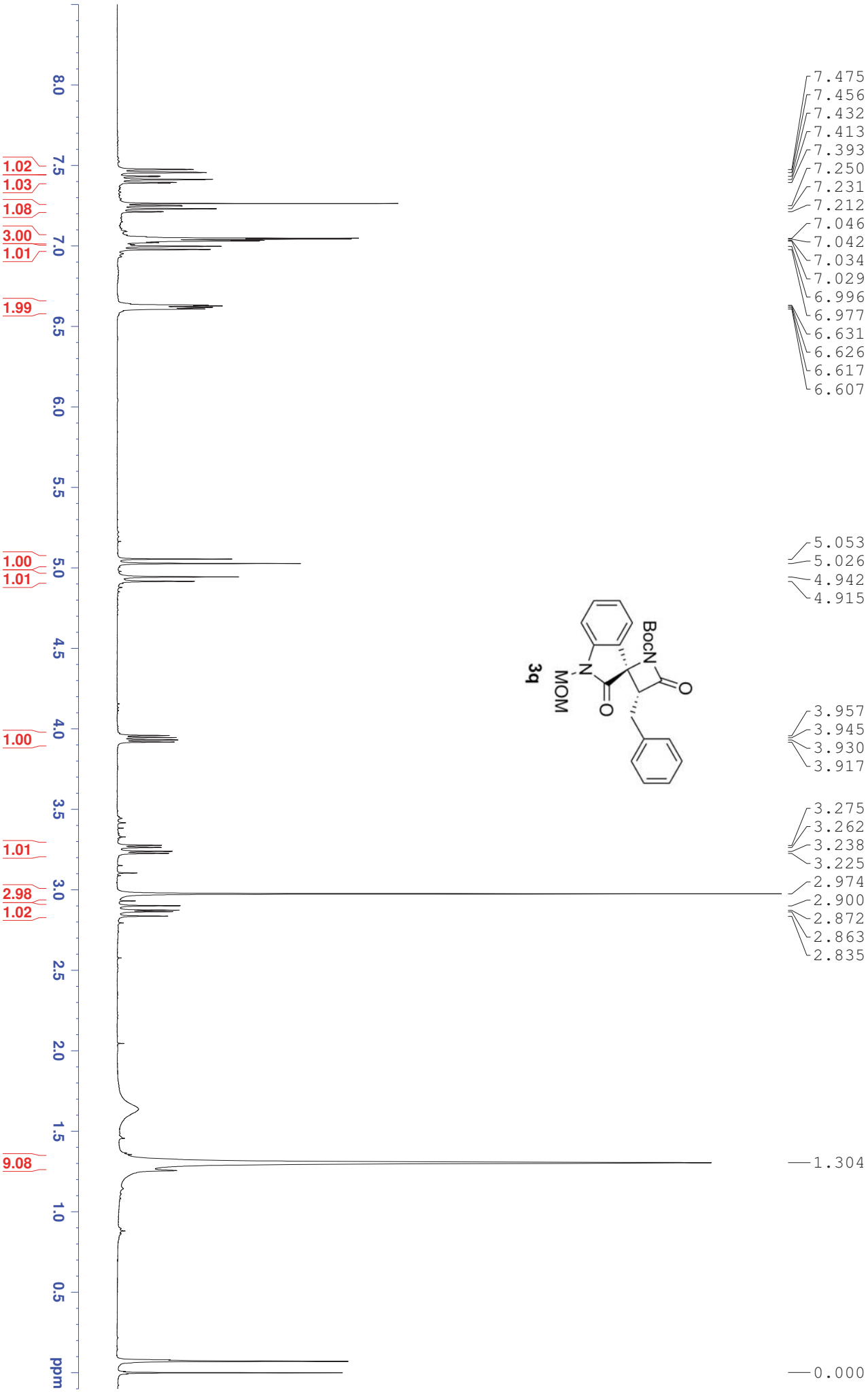
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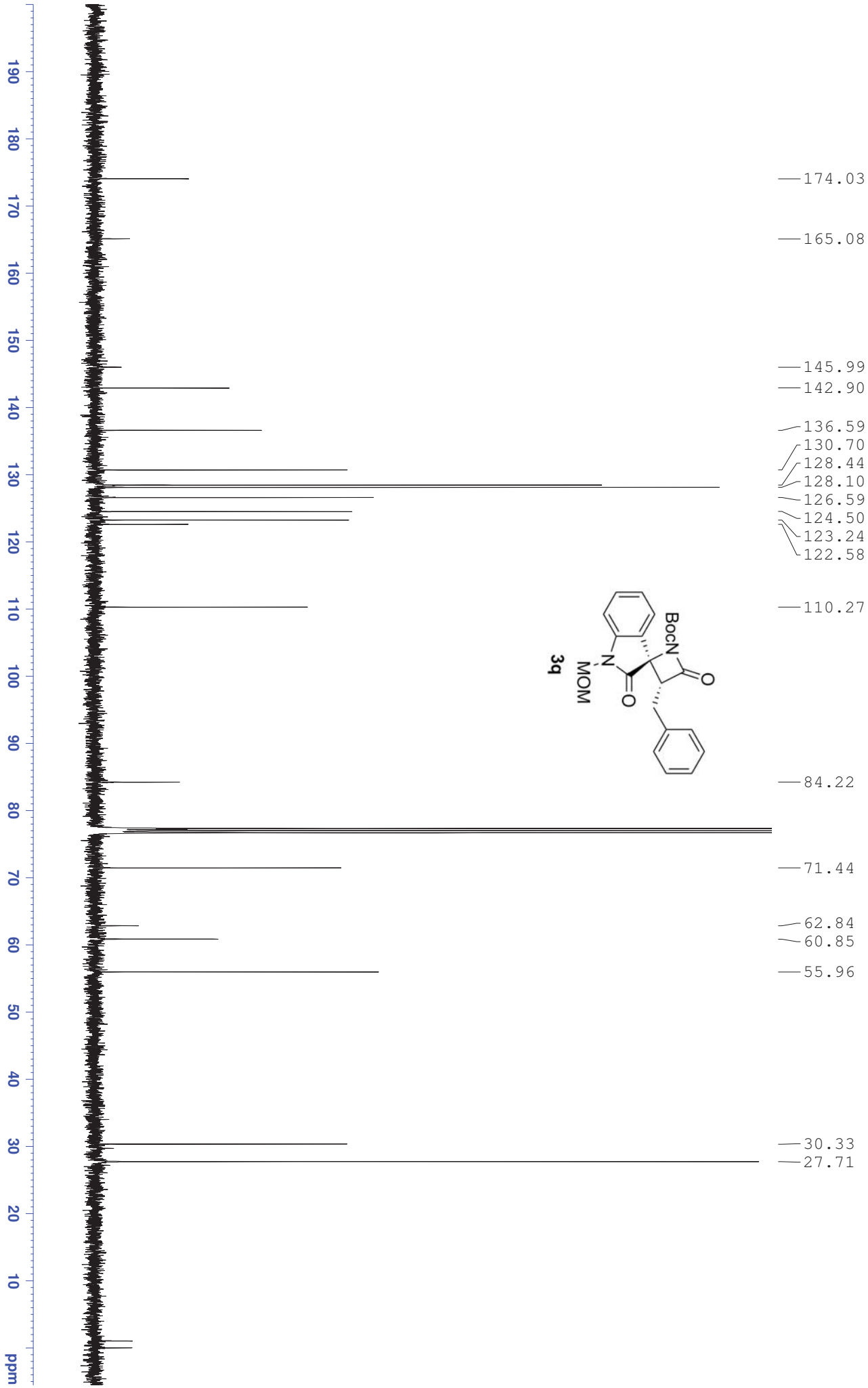
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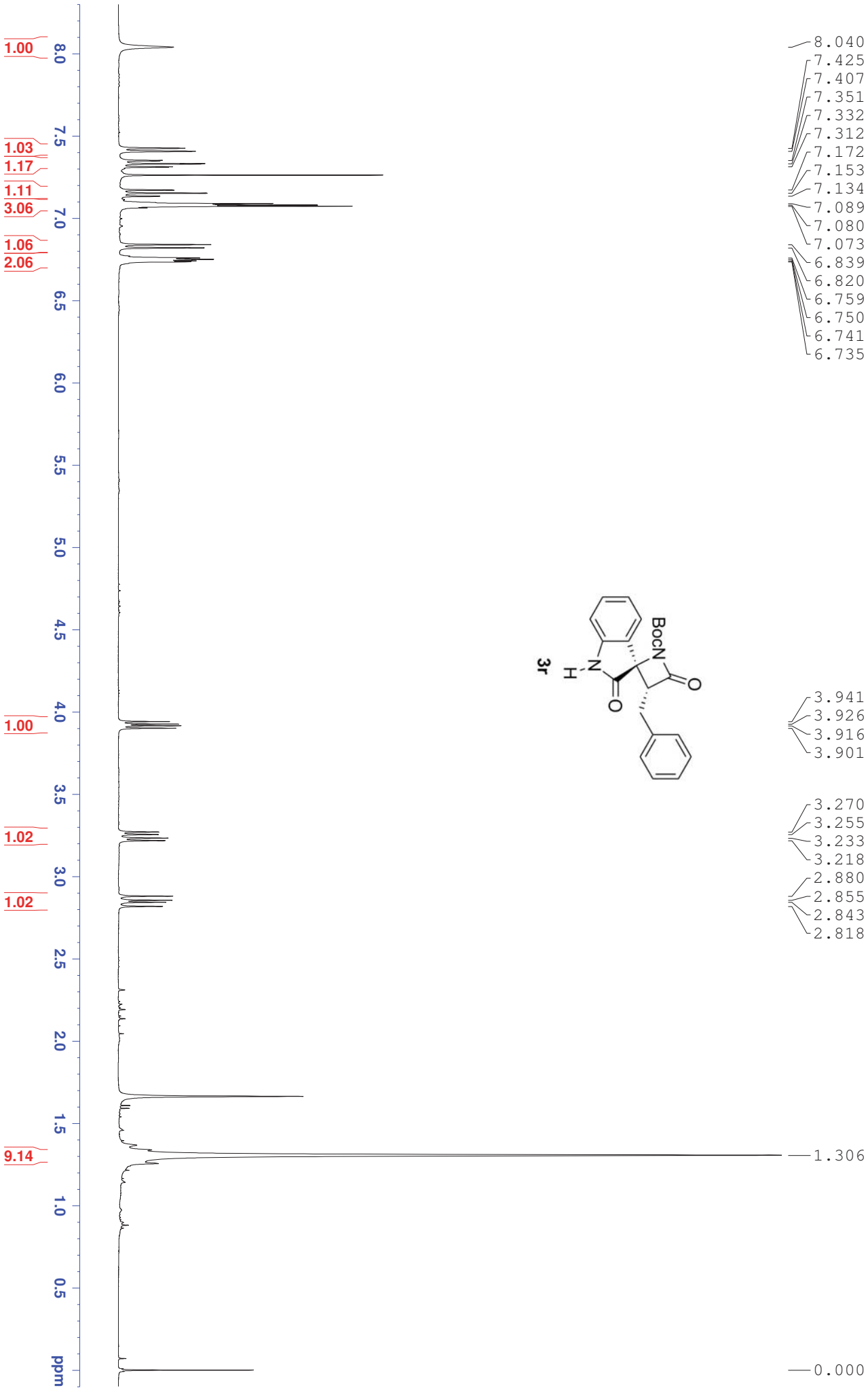
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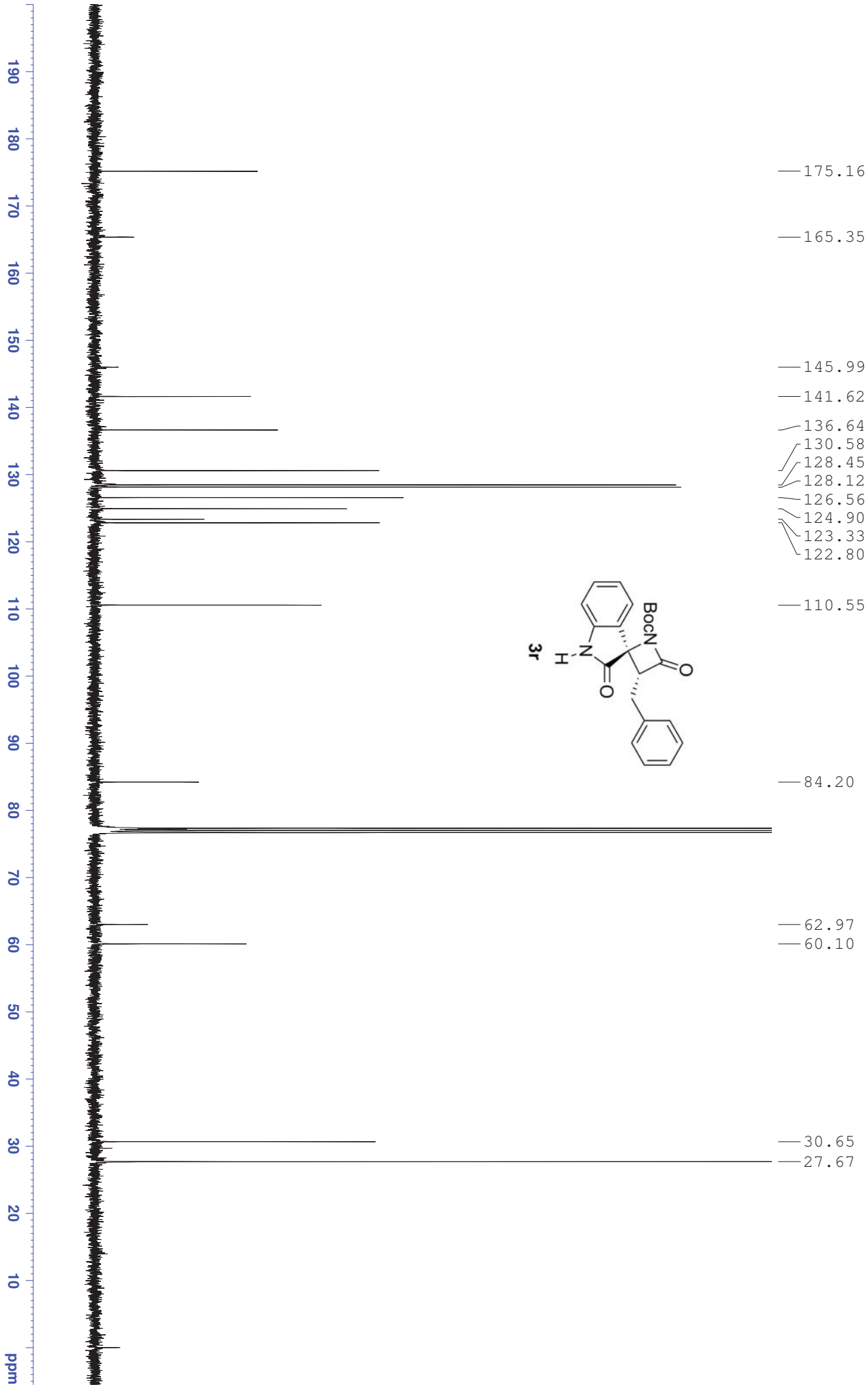
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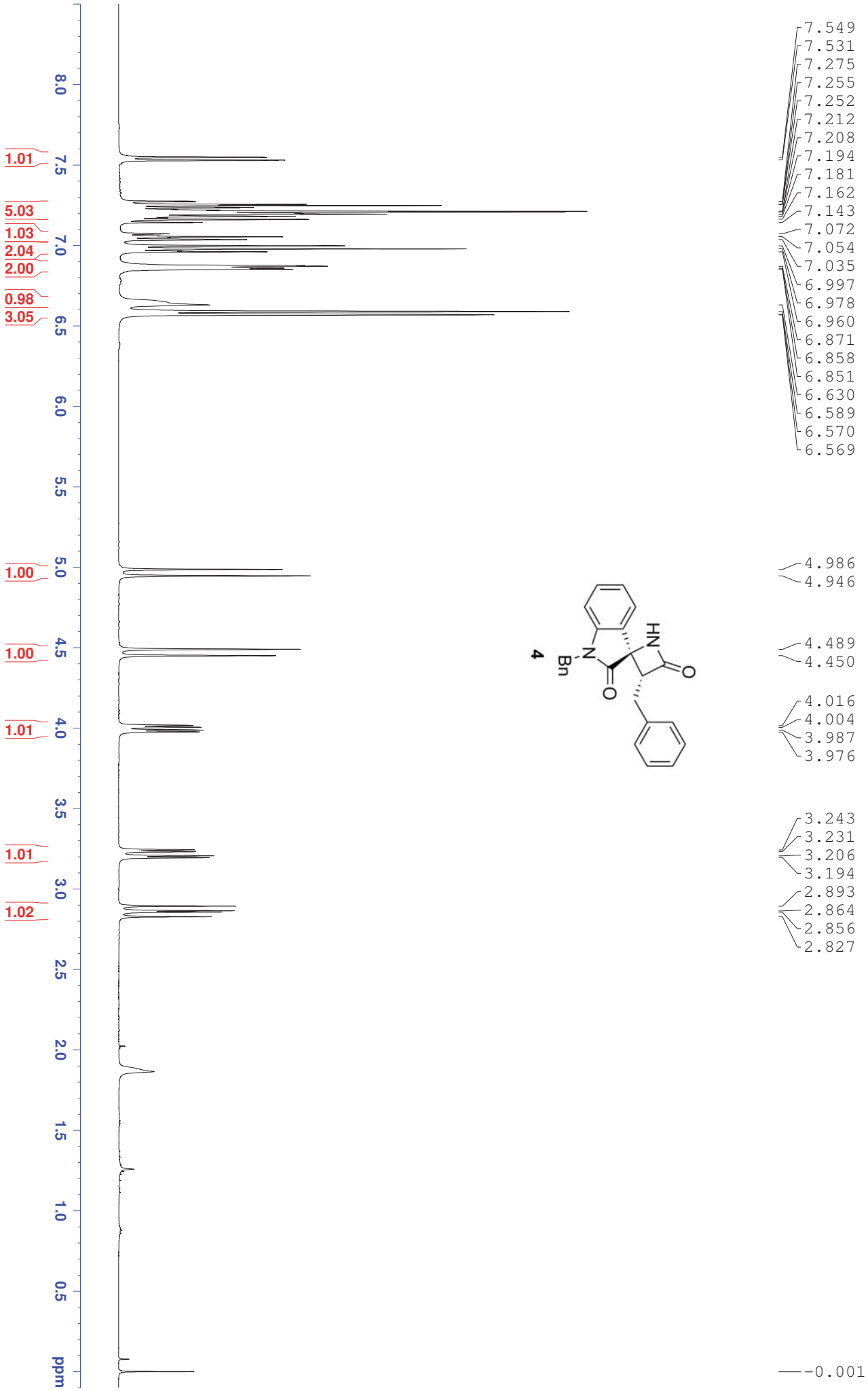
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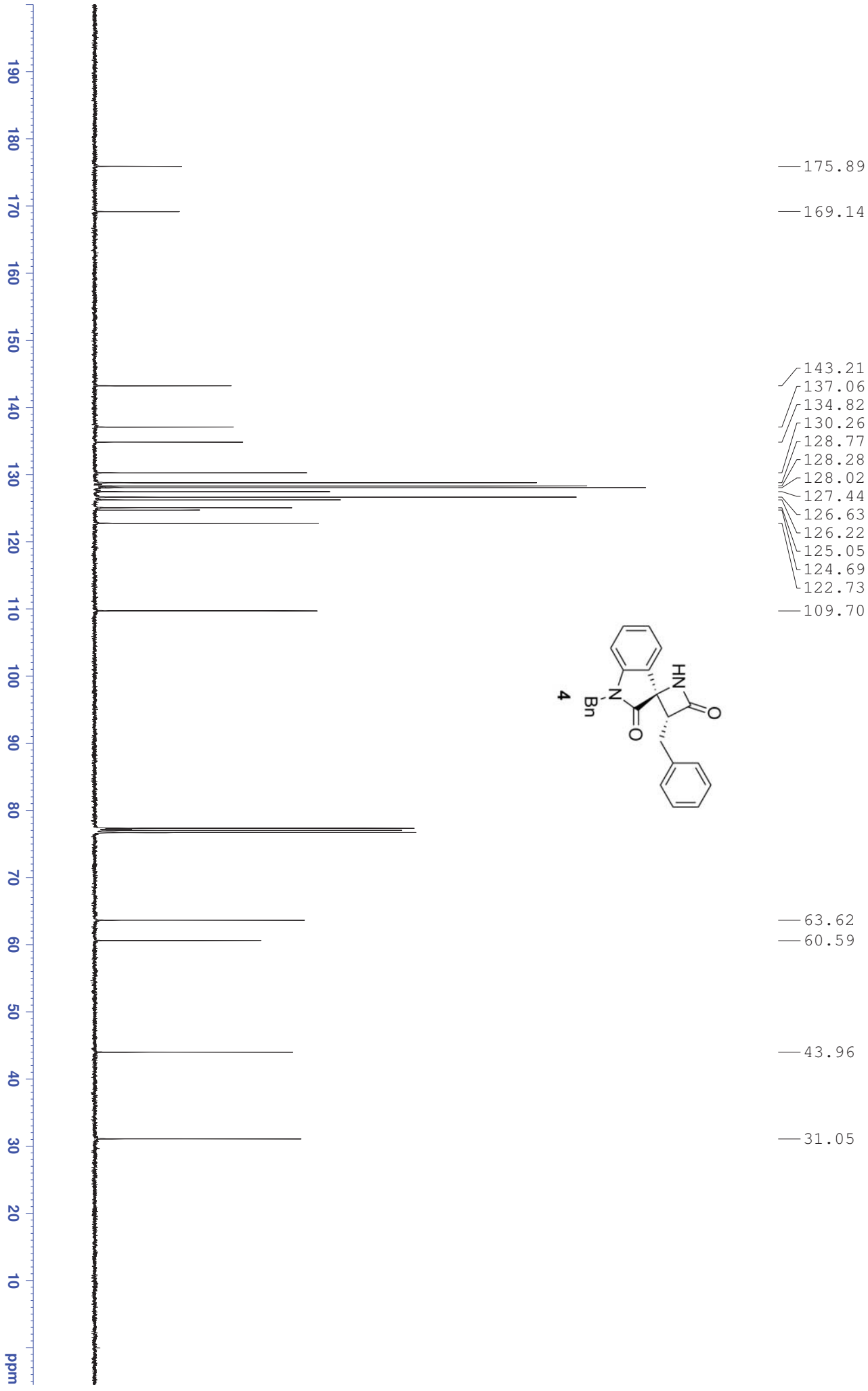
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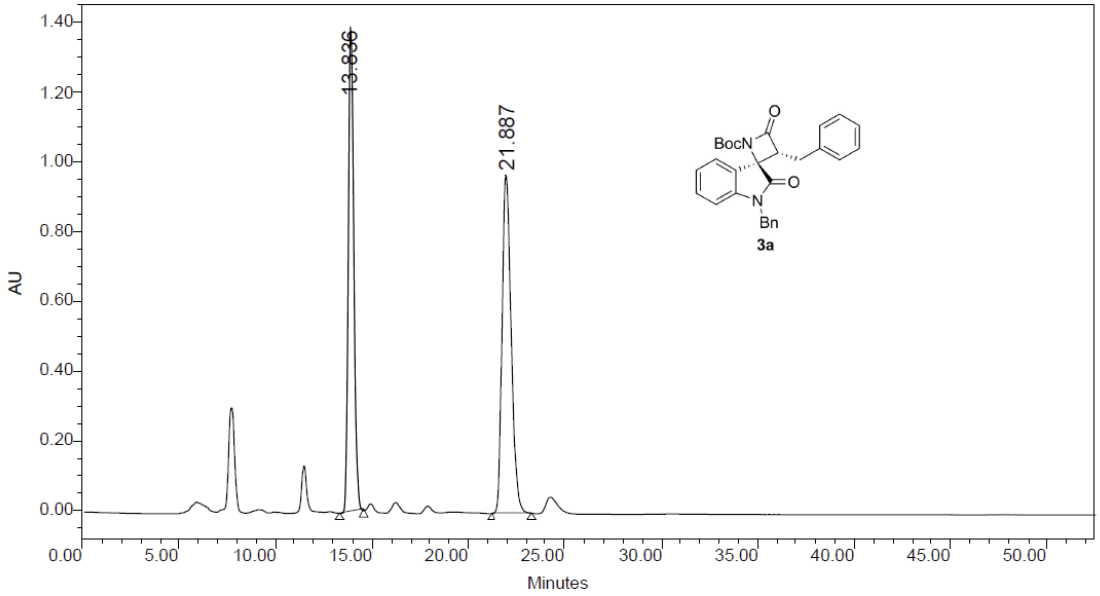
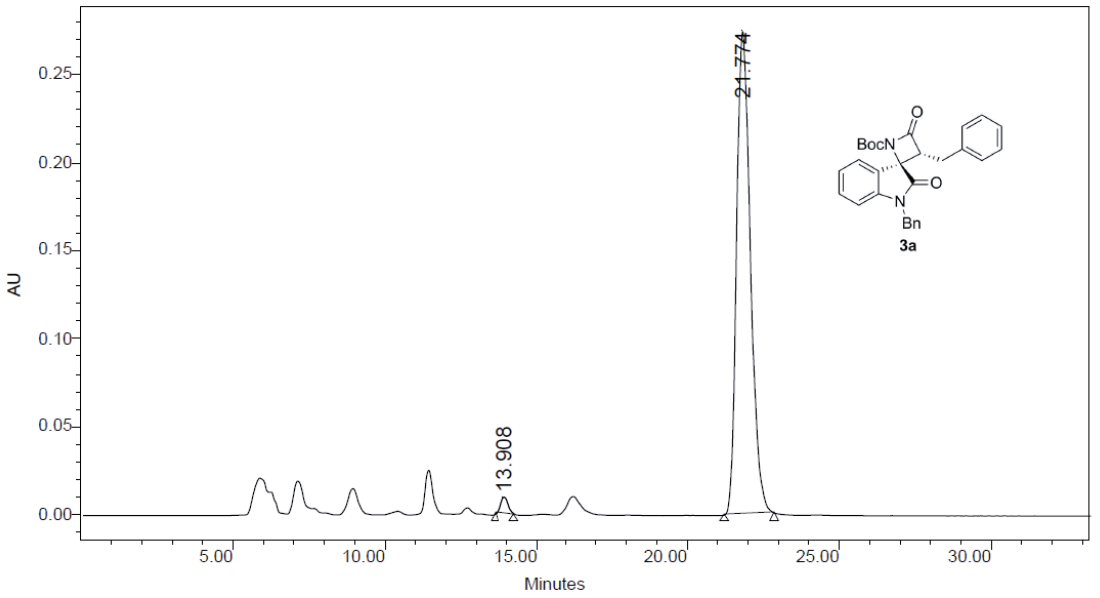
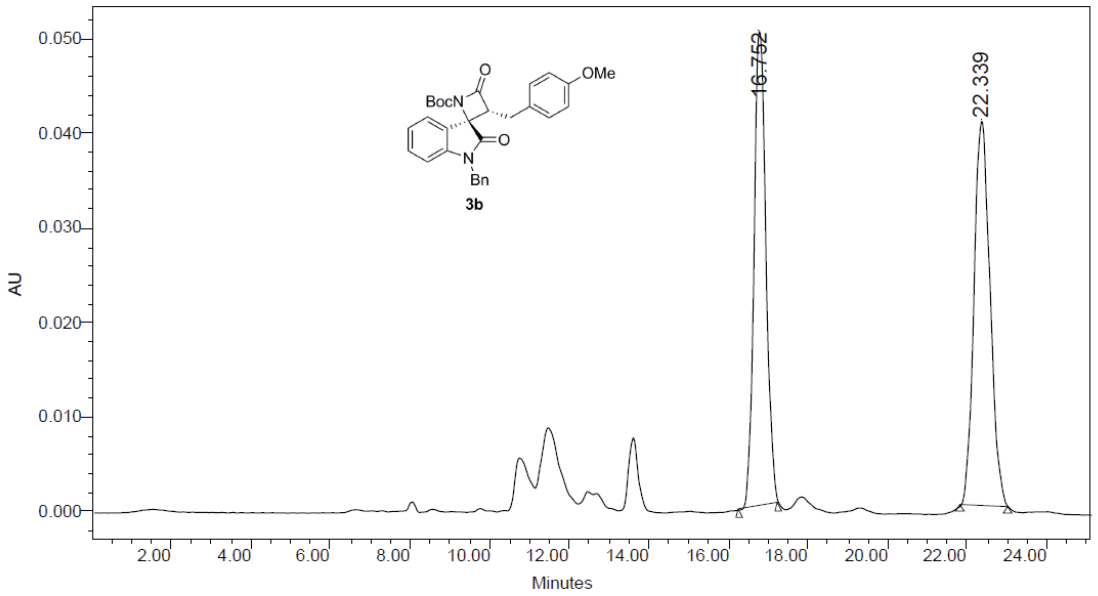


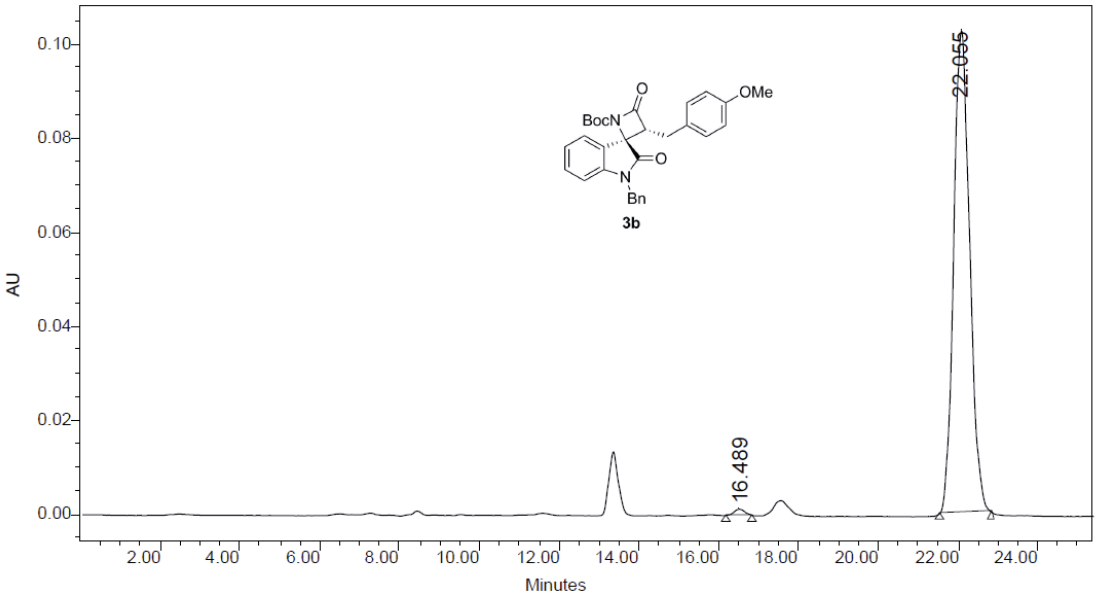
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)
1	13.836	Unknown	27445109	46.31	1382718	58.80	bb	75	13.317	14.567
2	21.887	Unknown	31824273	53.69	968712	41.20	bb	123	21.200	23.250



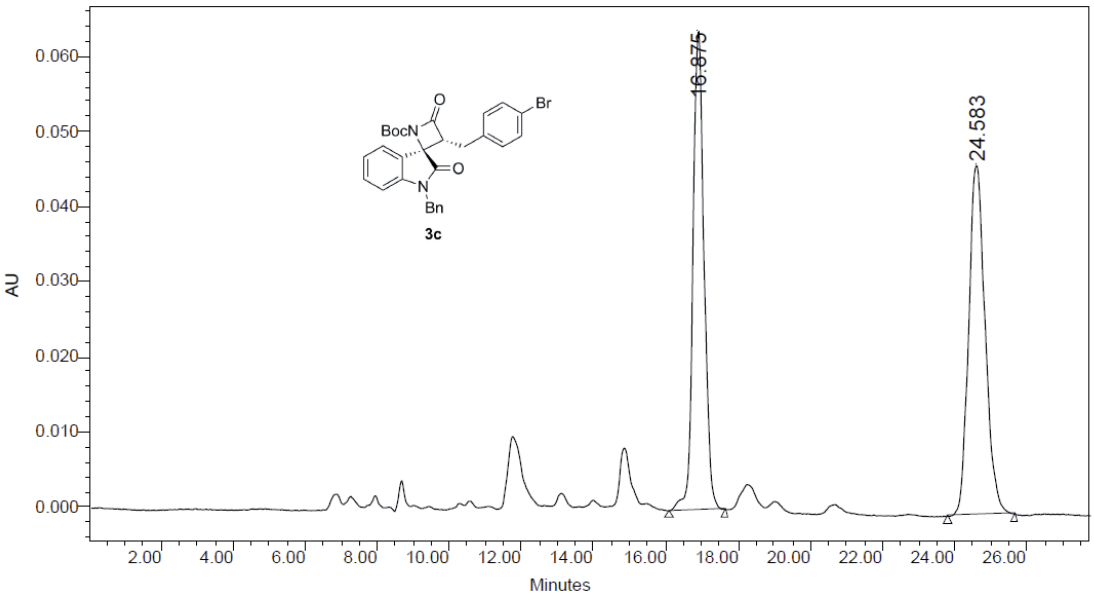
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	13.908	Unknown	164011	1.85	9513	3.35	bb	35	13.650	14.250	13.650
2	21.774	Unknown	8707415	98.15	274291	96.65	bb	98	21.200	22.850	21.200



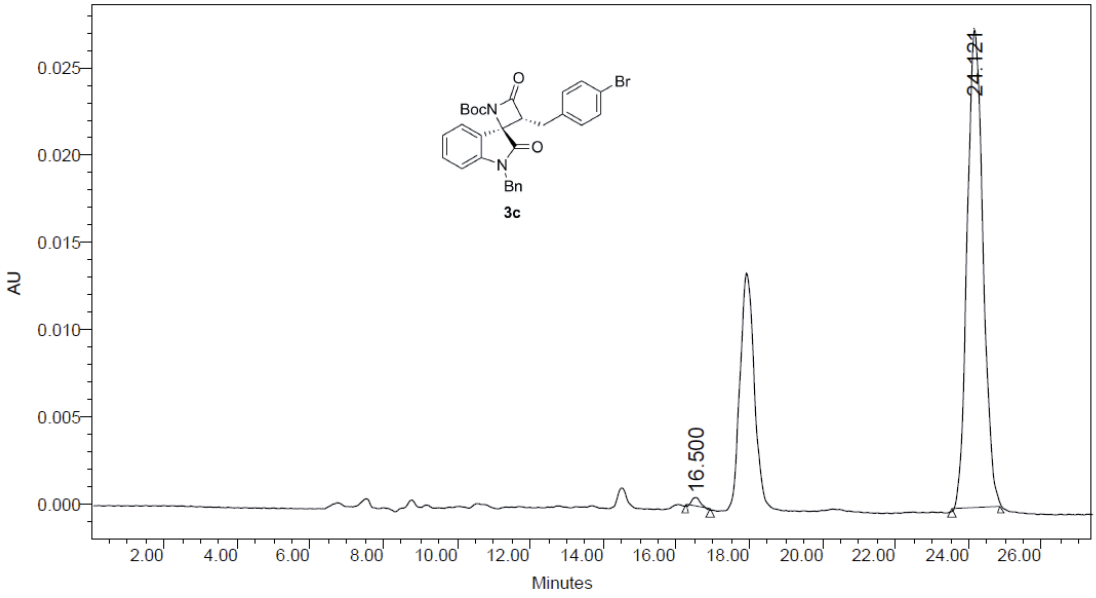
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	16.752	Unknown	1049940	47.85	50261	55.27	bb	59	16.267	17.250	16.267
2	22.339	Unknown	1144166	52.15	40676	44.73	bb	71	21.817	23.017	21.817



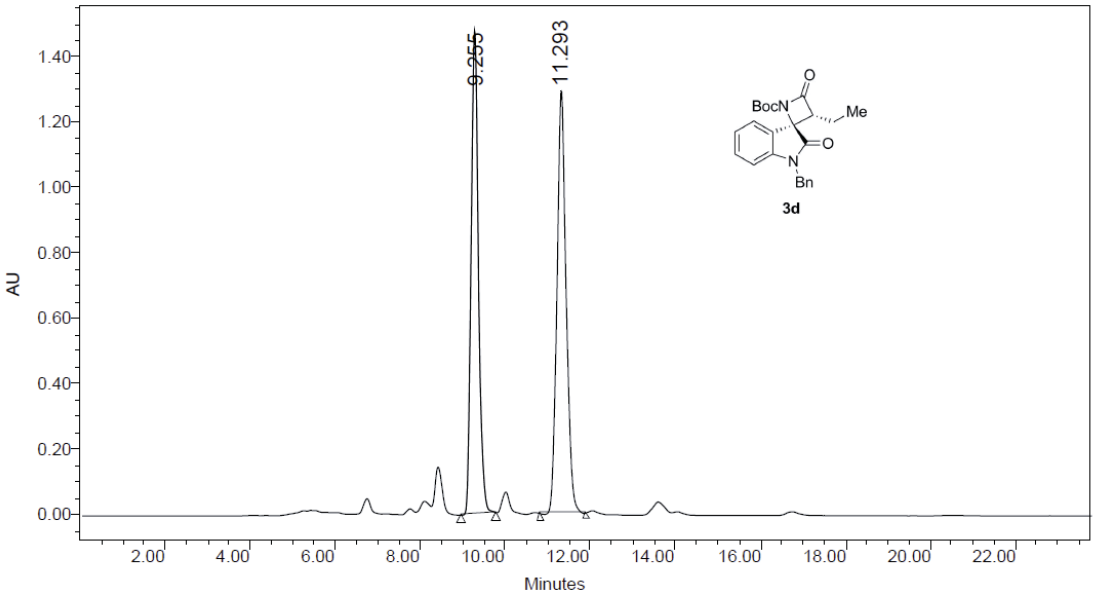
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	16.489	Unknown	23881	0.83	1287	1.24	bb	37	16.183	16.817	16.183
2	22.055	Unknown	2860568	99.17	102727	98.76	bb	78	21.517	22.817	21.517



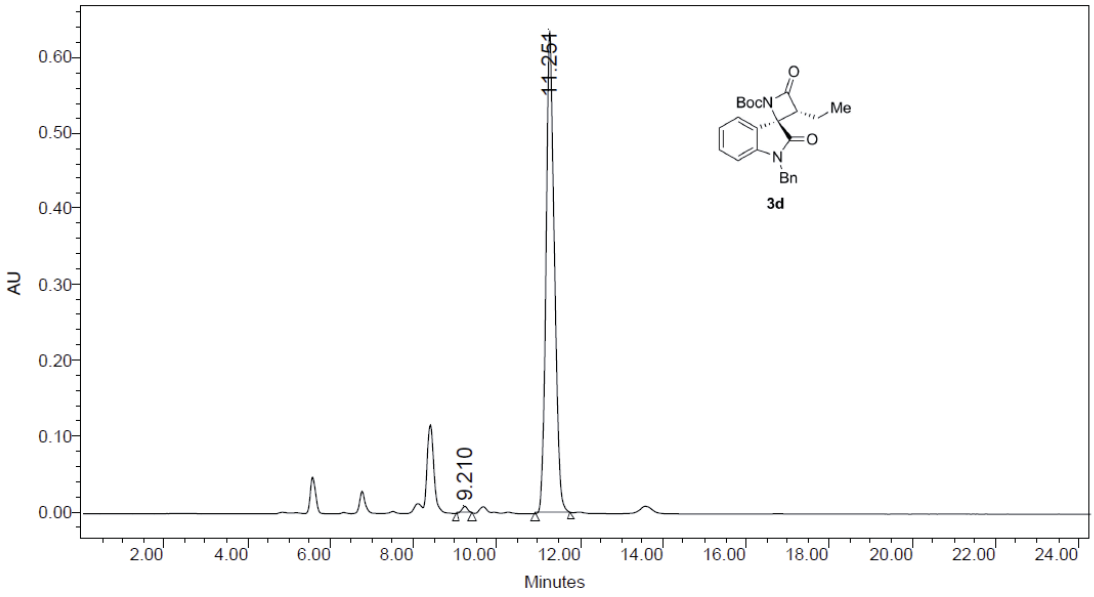
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	16.875	Unknown	1362336	47.83	63765	57.80	bb	92	16.100	17.633	16.100
2	24.583	Unknown	1486173	52.17	46554	42.20	bb	112	23.800	25.667	23.800



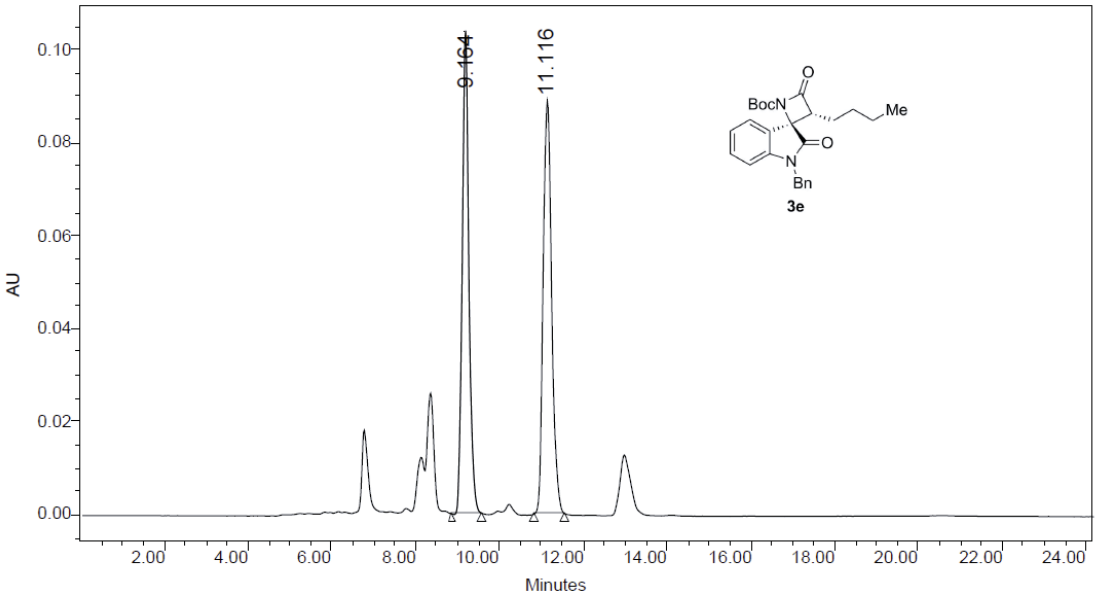
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	16.500	Unknown	10102	1.20	574	2.05	bb	39	16.250	16.917	16.250
2	24.121	Unknown	832574	98.80	27485	97.95	bb	80	23.533	24.867	23.533



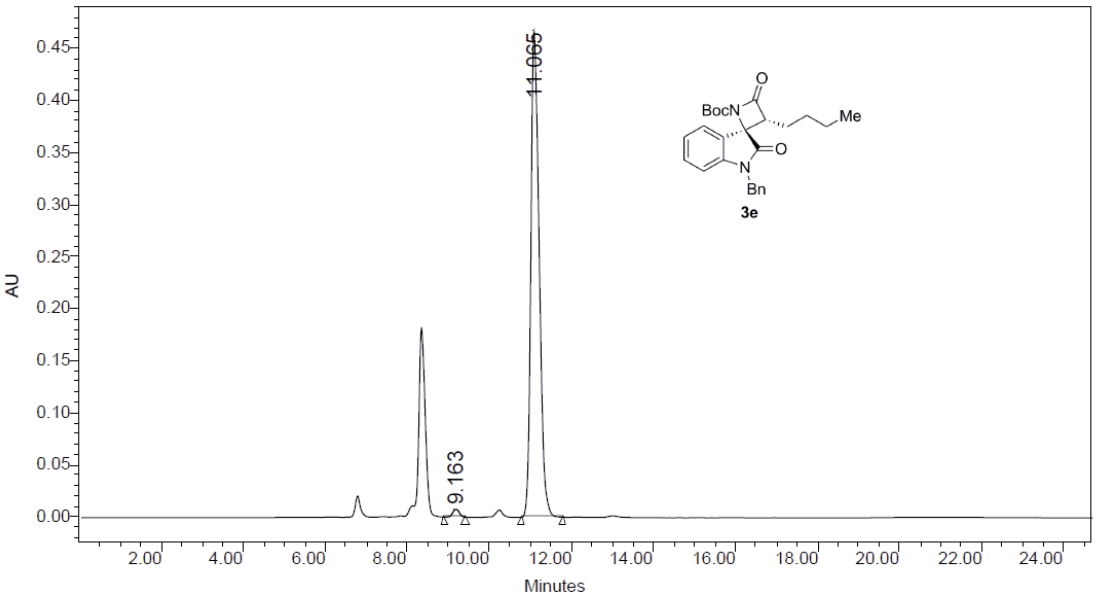
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)
1	9.255	Unknown	17641008	47.74	1480747	53.40	bb	48	8.950	9.767
2	11.293	Unknown	19307571	52.26	1292204	46.60	bb	64	10.817	11.900



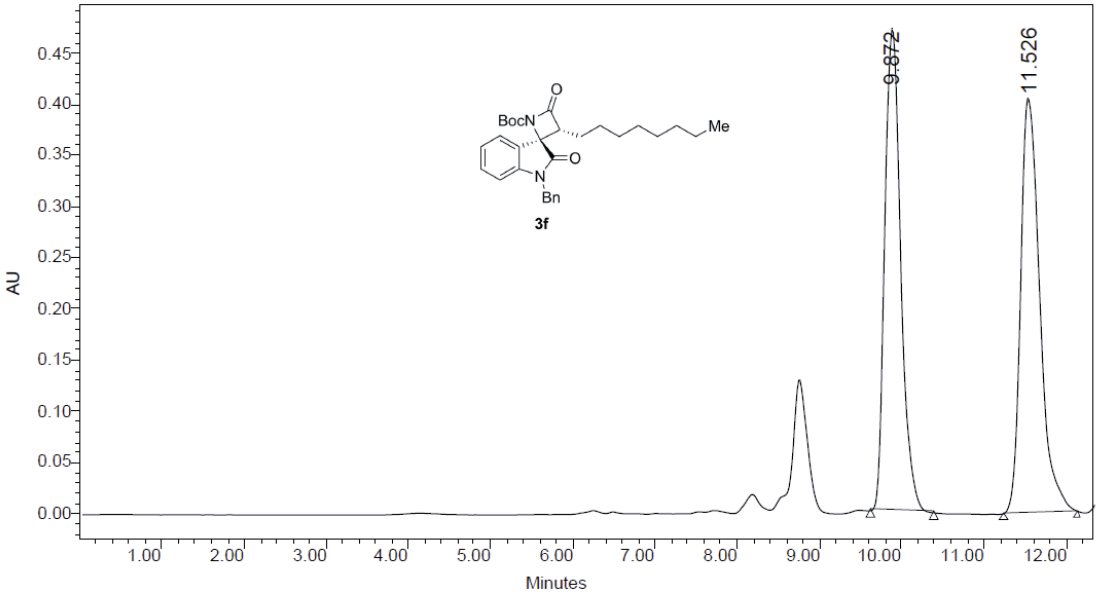
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	9.210	Unknown	94256	1.05	9278	1.44	bb	24	9.017	9.417	9.017
2	11.251	Unknown	8869158	98.95	634553	98.56	bb	50	10.933	11.783	10.933



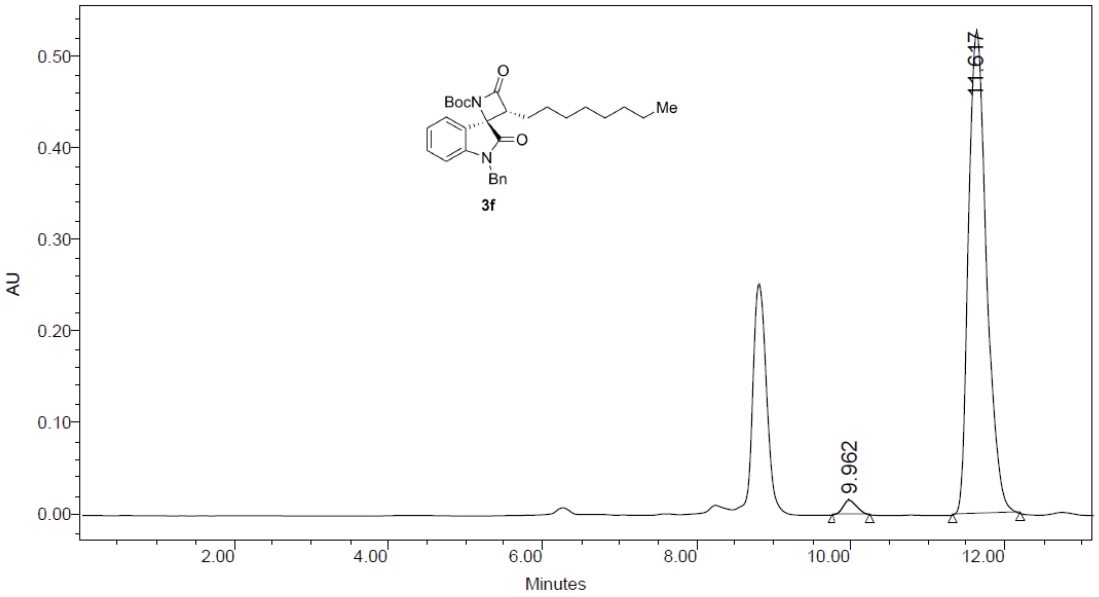
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	9.164	Unknown	1145002	47.73	103992	53.96	bb	42	8.867	9.567	8.867
2	11.116	Unknown	1254001	52.27	88735	46.04	bb	42	10.817	11.533	10.817



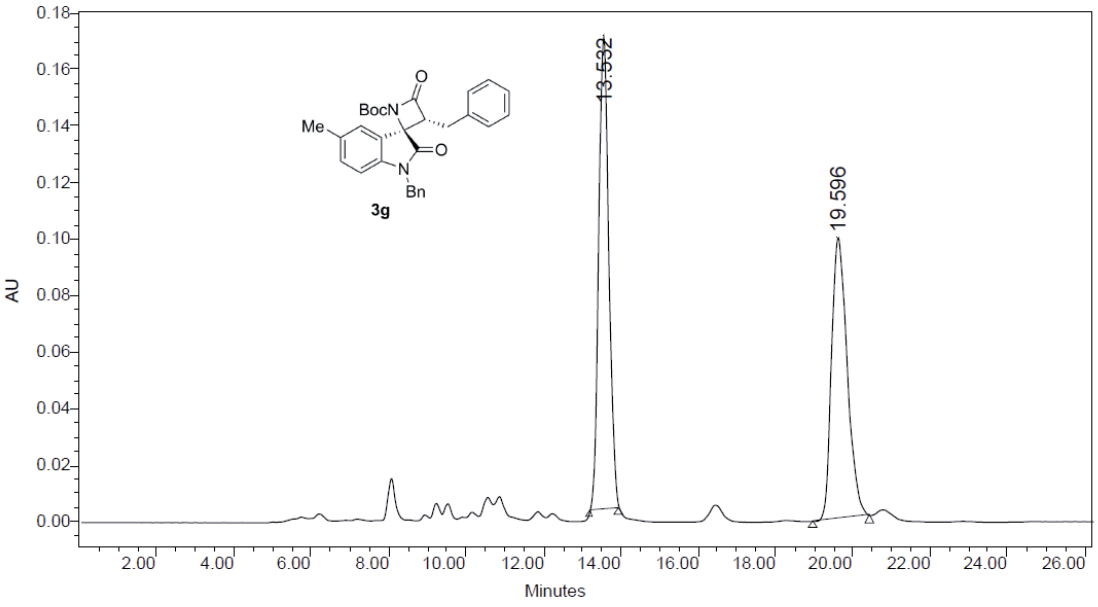
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	9.163	Unknown	82716	1.24	7428	1.57	bb	30	8.900	9.400	8.900
2	11.065	Unknown	6572767	98.76	466556	98.43	bb	60	10.767	11.783	10.767



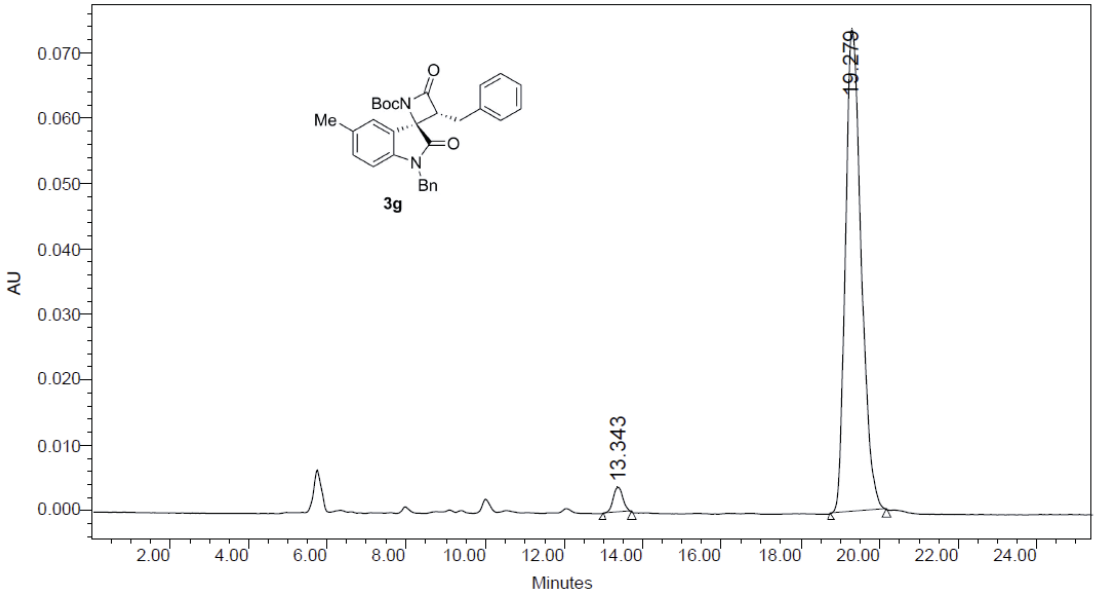
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	9.872	Unknown	6368333	49.31	471098	53.72	bb	46	9.617	10.383	9.617
2	11.526	Unknown	6545314	50.69	405890	46.28	bb	54	11.233	12.133	11.233



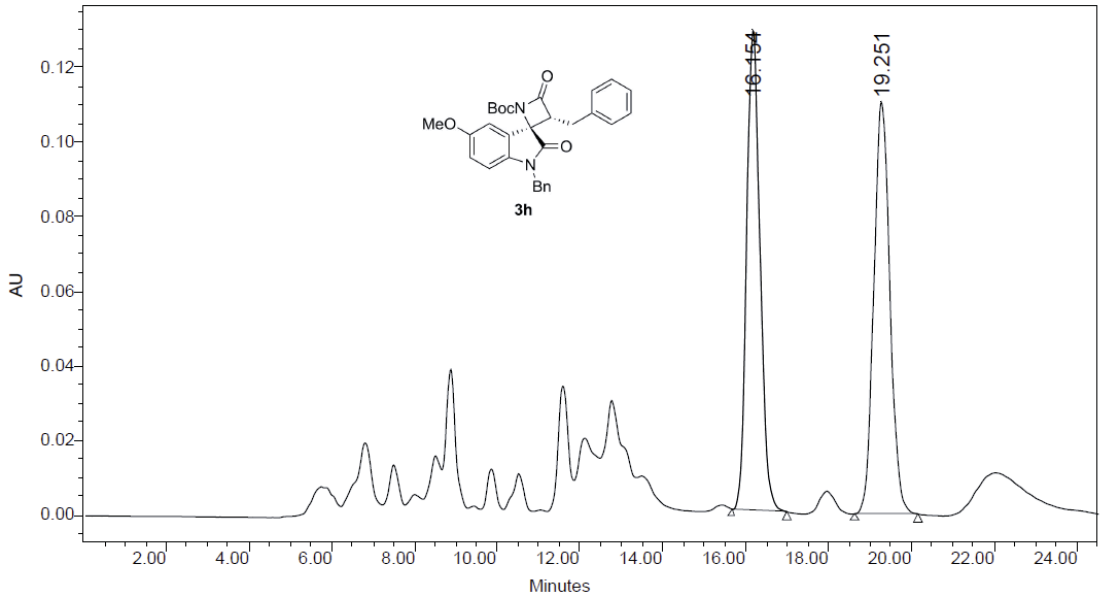
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	9.962	Unknown	206022	2.33	16293	2.99	bb	30	9.750	10.250	9.750
2	11.617	Unknown	8631575	97.67	527968	97.01	bb	52	11.317	12.200	11.317



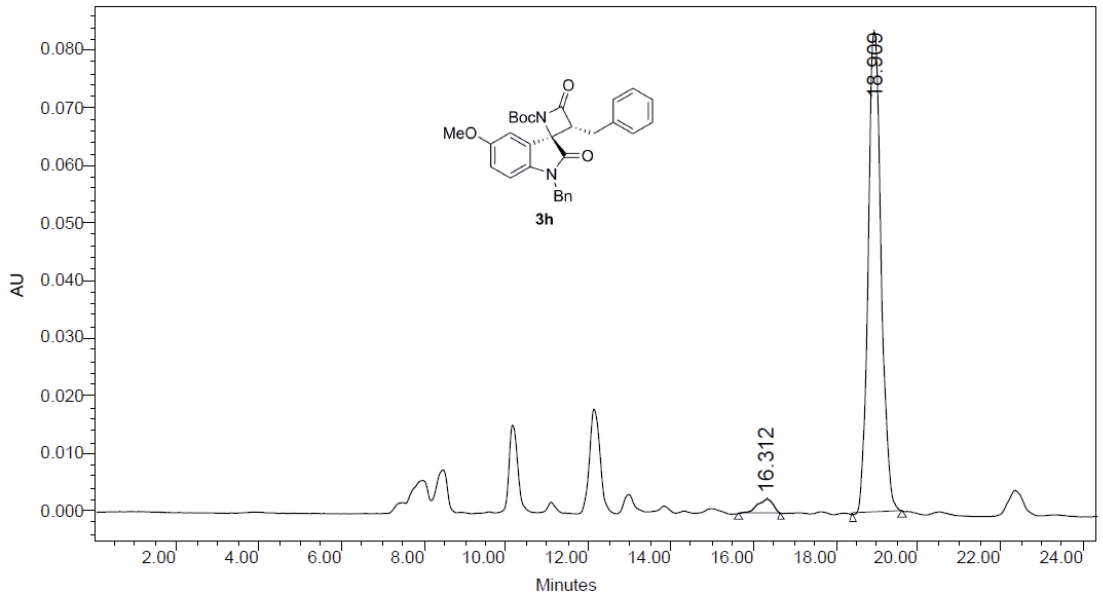
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	13.532	Unknown	3038596	51.97	167112	62.67	bb	45	13.183	13.933	13.183
2	19.596	Unknown	2808221	48.03	99539	37.33	bb	87	18.950	20.417	18.950



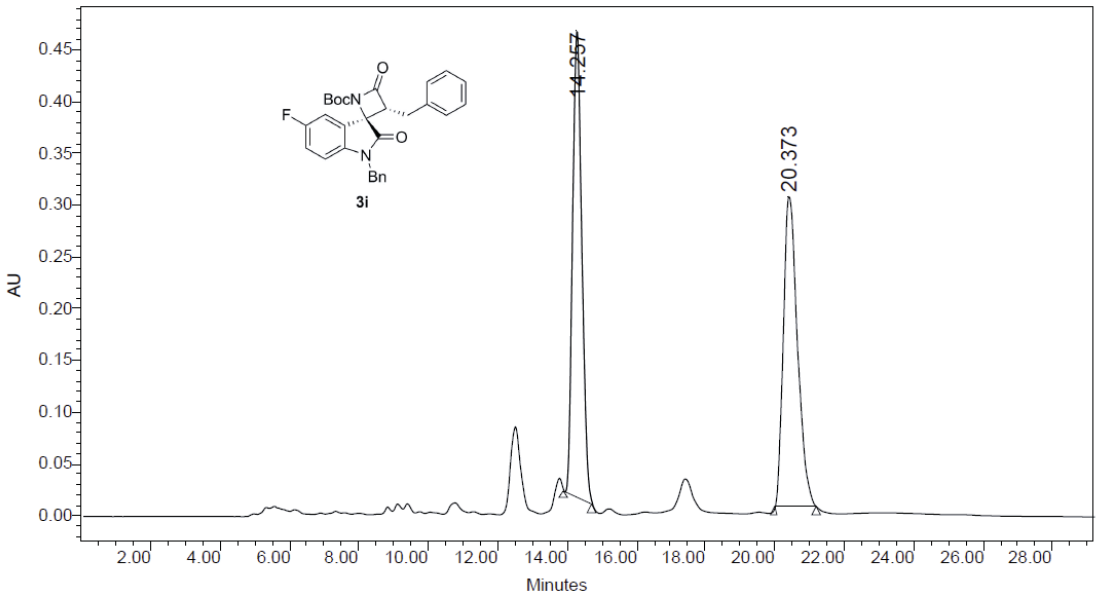
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	13.343	Unknown	70330	3.30	3944	5.08	bb	45	12.983	13.733	12.983
2	19.279	Unknown	2058439	96.70	73764	94.92	bb	83	18.767	20.167	18.767



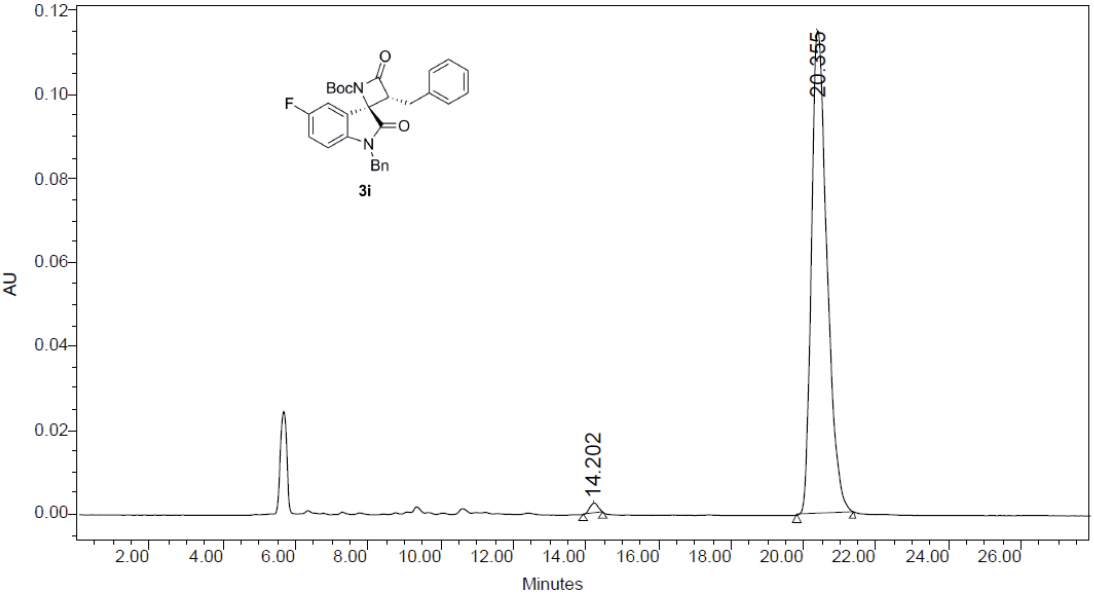
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	16.154	Unknown	2898233	49.14	128764	53.80	bb	79	15.650	16.983	15.650
2	19.251	Unknown	2999184	50.86	110591	46.20	bb	92	18.617	20.167	18.617



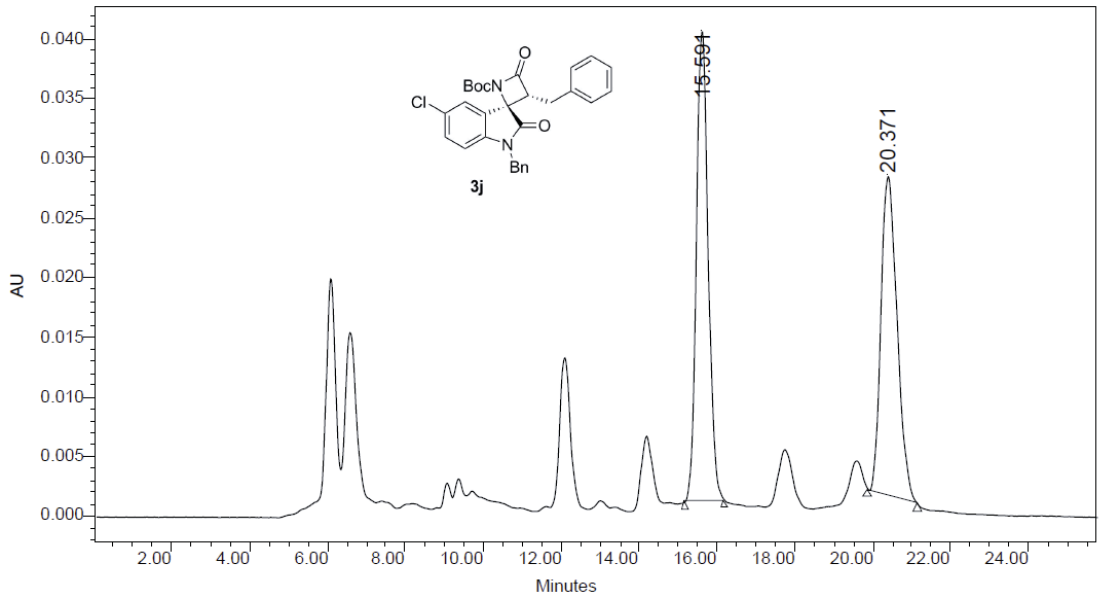
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	16.312	Unknown	65764	3.46	2467	2.86	bb	60	15.650	16.667	15.650
2	18.909	Unknown	1835180	96.54	83682	97.14	bb	71	18.417	19.600	18.417



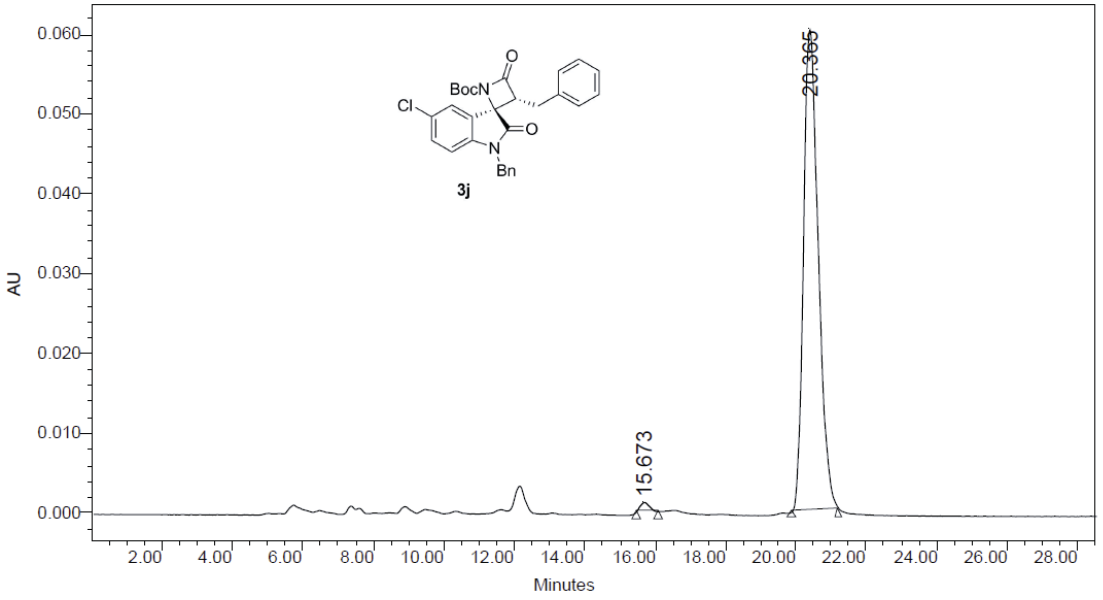
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	14.257	Unknown	8333004	49.39	451129	60.00	bb	48	13.900	14.717	13.900
2	20.373	Unknown	8538165	50.61	300733	40.00	bb	73	19.967	21.183	19.967



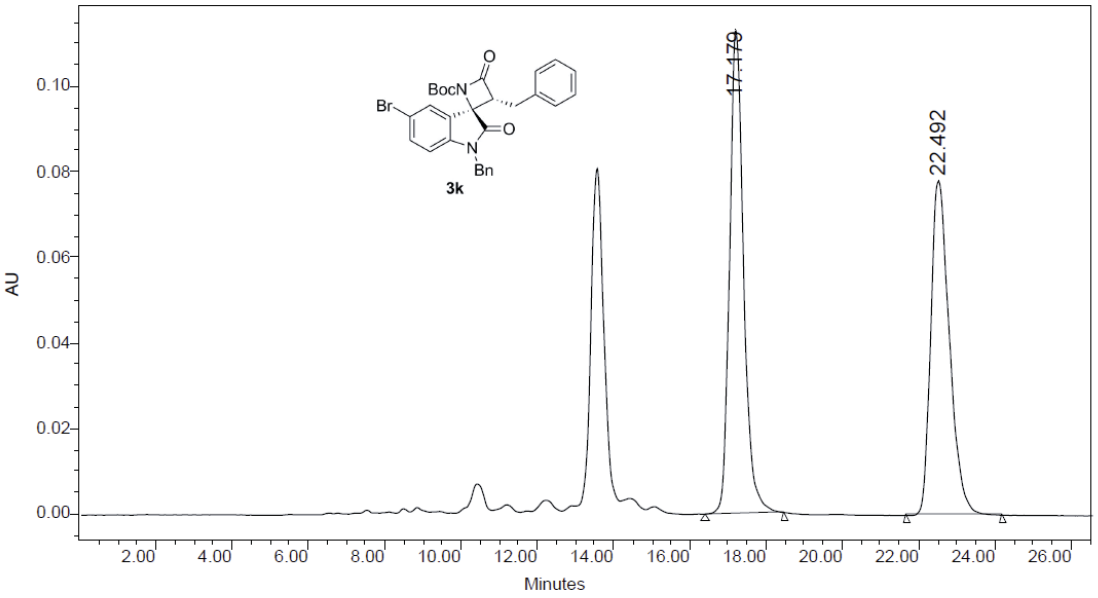
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	14.202	Unknown	40715	1.17	2508	2.13	bb	31	13.917	14.450	13.917
2	20.355	Unknown	3441028	98.83	115351	97.87	bb	93	19.800	21.350	19.800



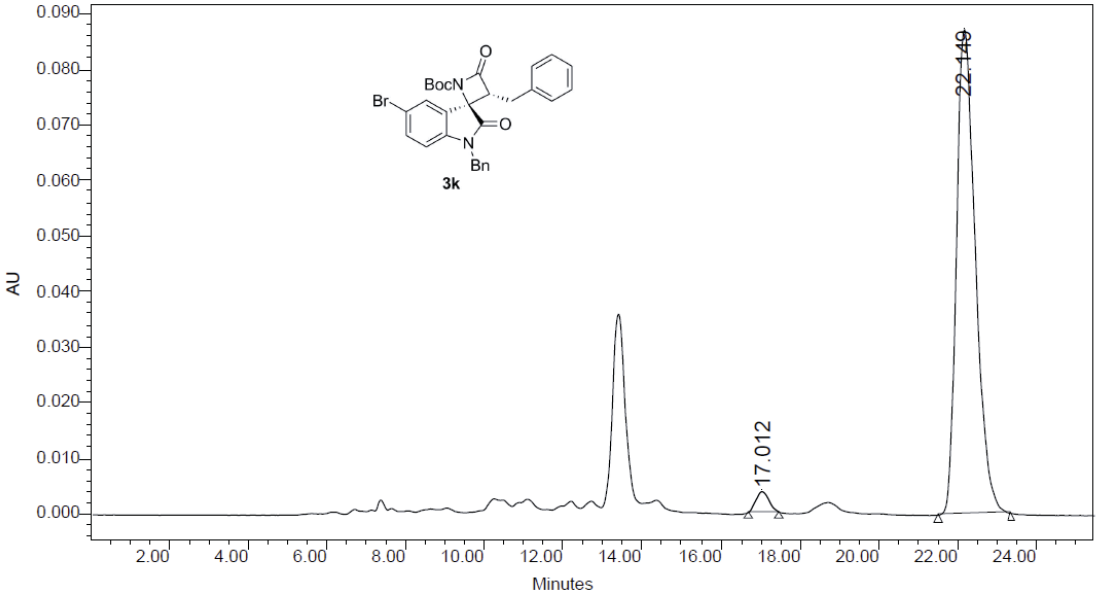
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	15.591	Unknown	828615	51.71	39375	59.54	bb	60	15.167	16.183	15.167
2	20.371	Unknown	773929	48.29	26759	40.46	bb	75	19.867	21.133	19.867



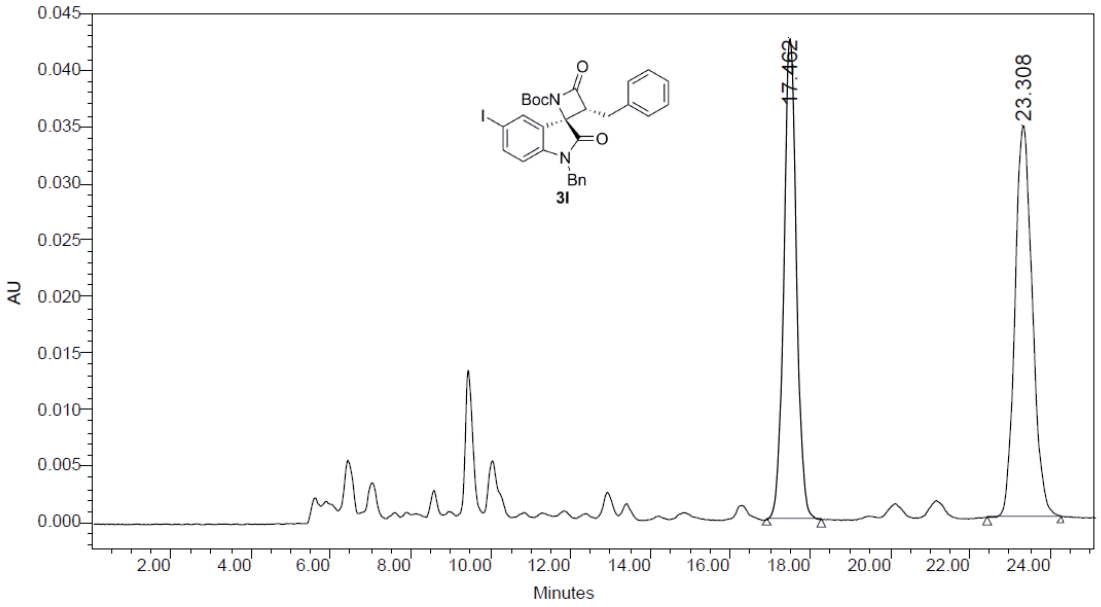
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	15.673	Unknown	20341	1.15	1139	1.86	bb	37	15.450	16.083	15.450
2	20.365	Unknown	1750131	98.85	60176	98.14	bb	78	19.883	21.200	19.883



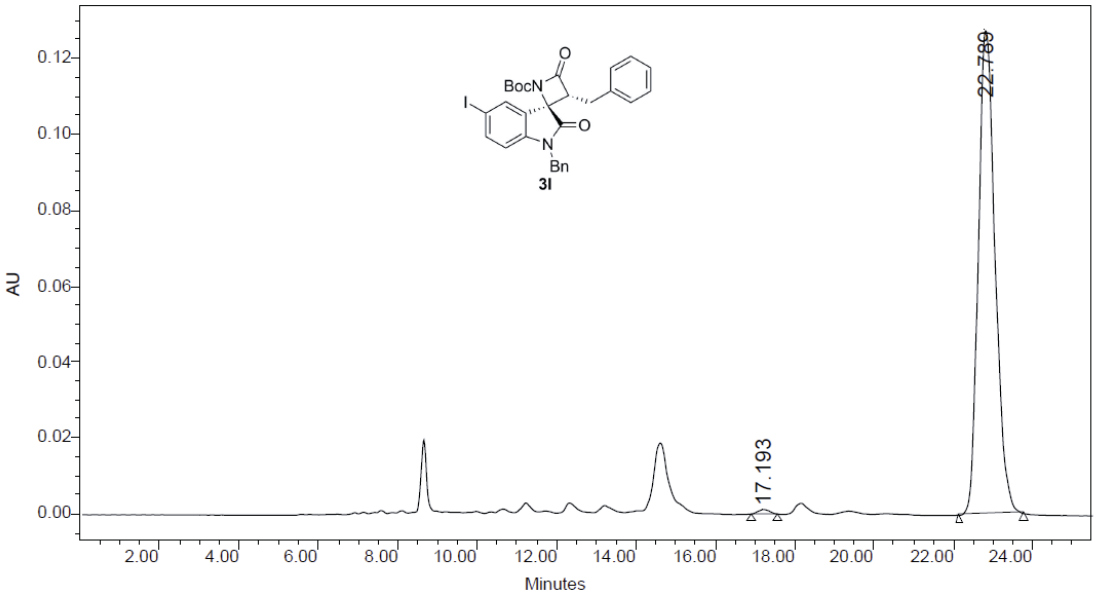
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	17.179	Unknown	2893127	52.05	112915	59.10	bb	124	16.400	18.483	16.400
2	22.492	Unknown	2664887	47.95	78132	40.90	bb	150	21.683	24.200	21.683



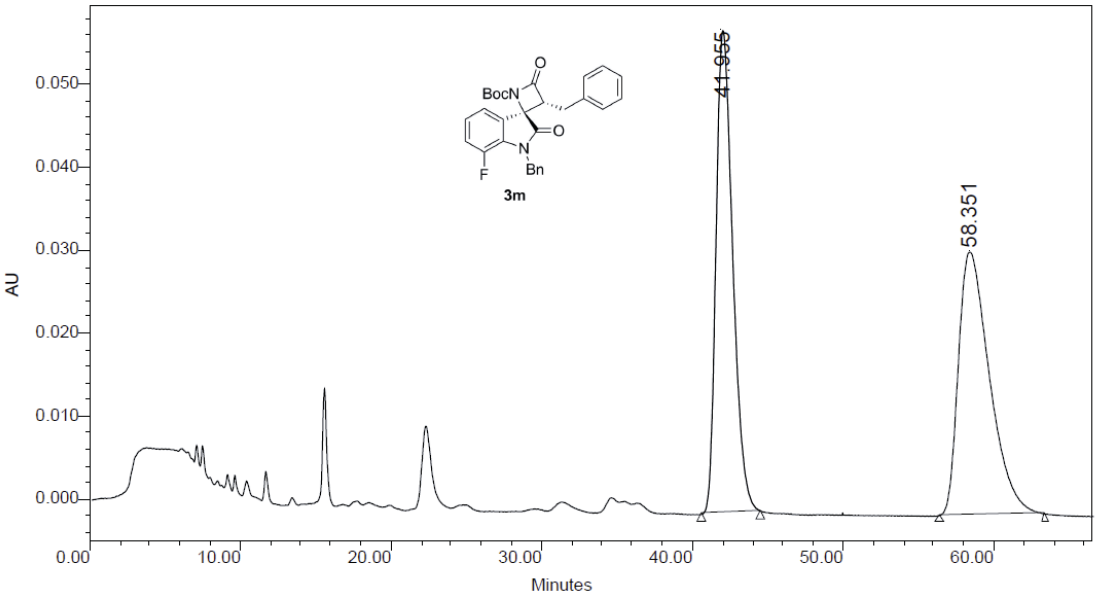
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	17.012	Unknown	84085	2.80	3679	4.05	bb	45	16.683	17.450	16.683
2	22.149	Unknown	2919257	97.20	87239	95.95	bb	110	21.500	23.350	21.500



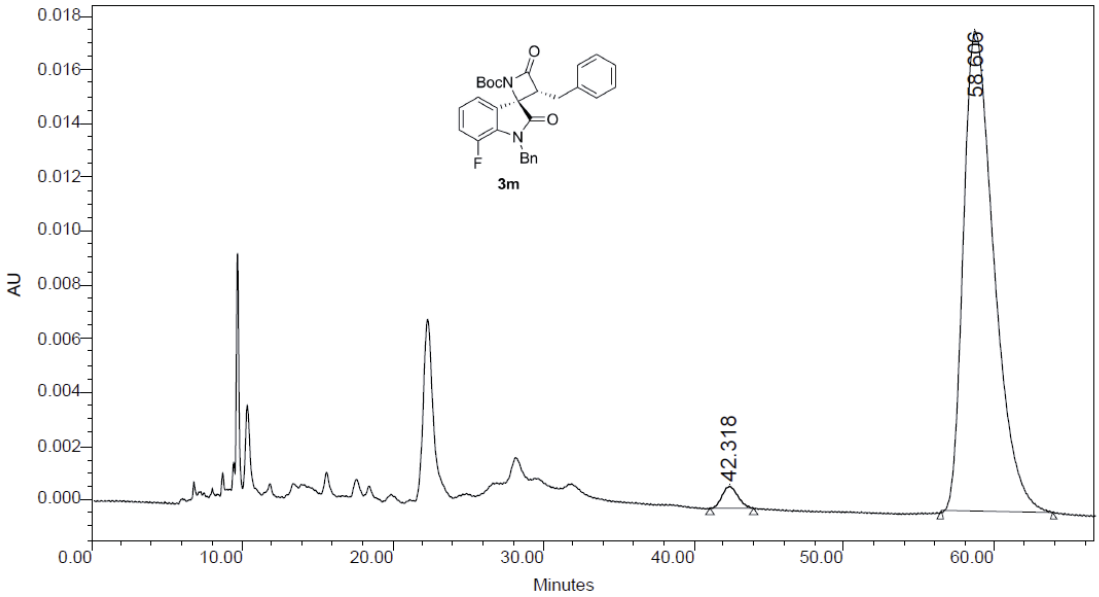
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	17.462	Unknown	934333	47.20	42452	55.13	bb	82	16.900	18.267	16.900
2	23.308	Unknown	1045375	52.80	34557	44.87	bb	109	22.433	24.267	22.433



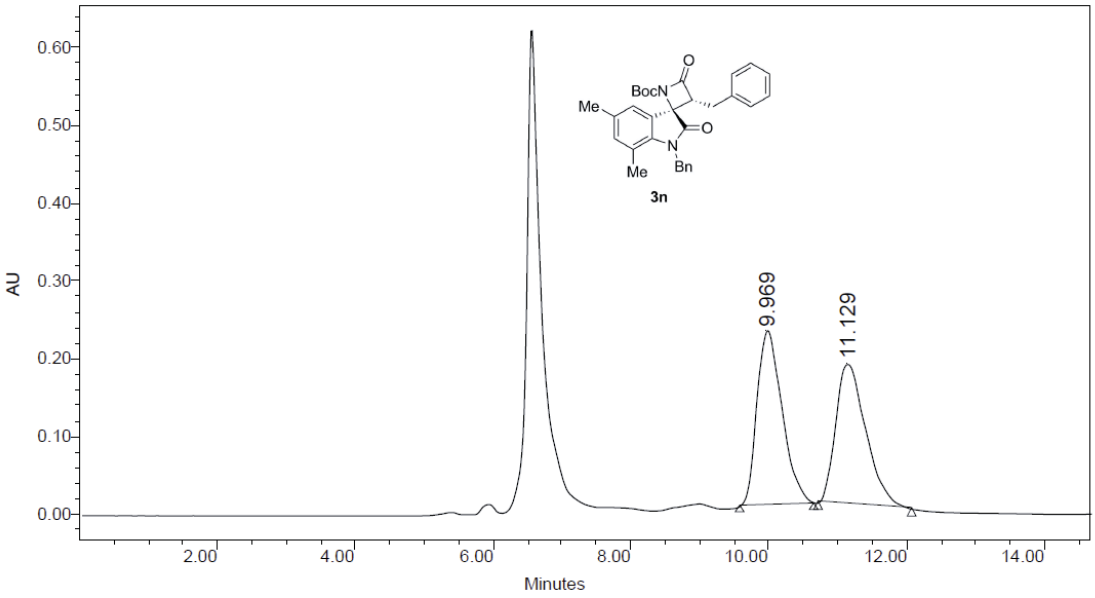
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	17.193	Unknown	23793	0.63	1179	0.92	bb	40	16.900	17.567	16.900
2	22.789	Unknown	3750885	99.37	127552	99.08	bb	97	22.150	23.783	22.150



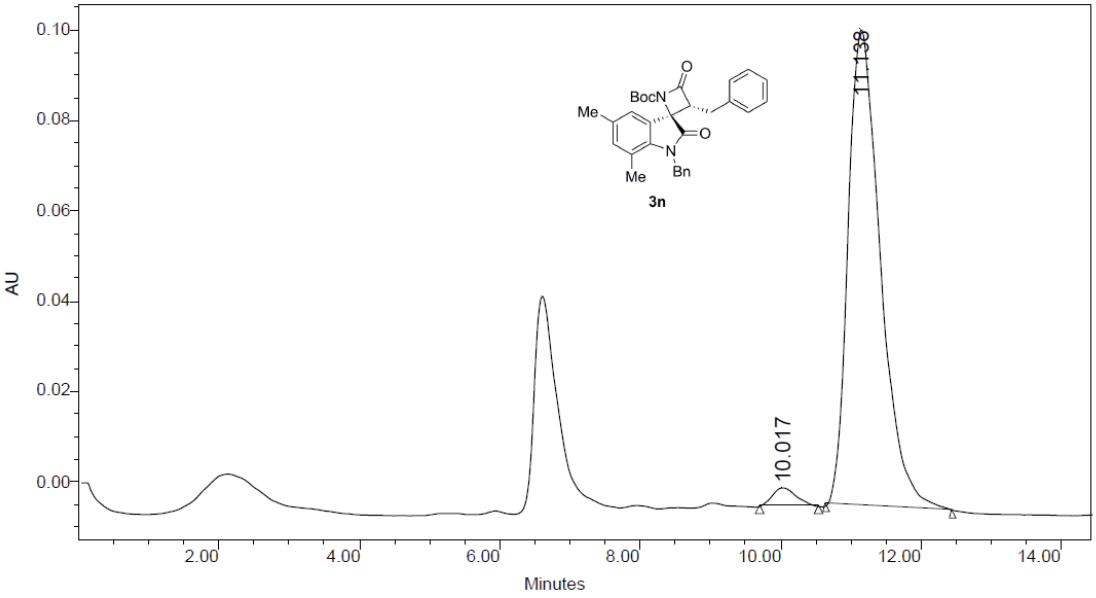
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	41.955	Unknown	4481894	49.59	58193	64.71	bb	232	40.617	44.500	40.617
2	58.351	Unknown	4555174	50.41	31735	35.29	bb	420	56.383	63.400	56.383



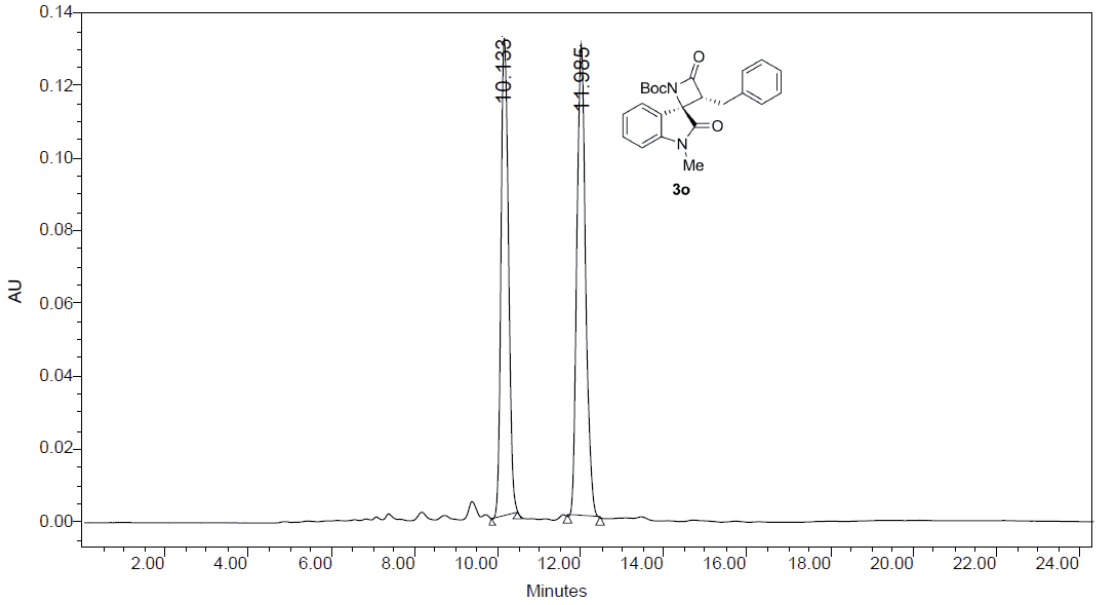
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	42.318	Unknown	61295	2.36	834	4.45	bb	179	41.033	44.017	41.033
2	58.606	Unknown	2535859	97.64	17918	95.55	bb	450	56.400	63.900	56.400



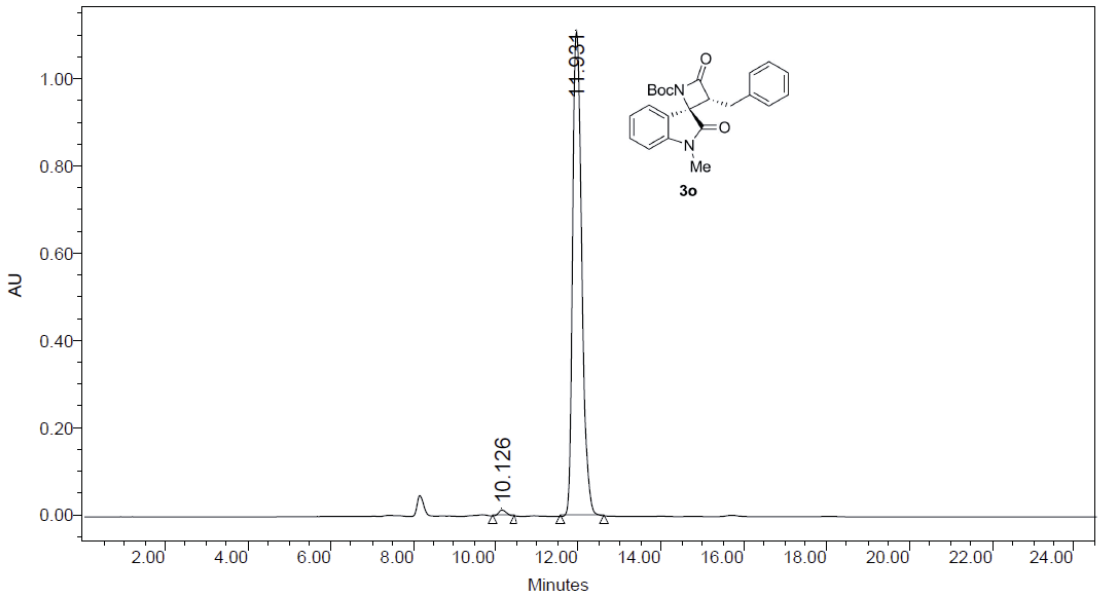
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	9.969	Unknown	5659796	50.60	223316	55.35	bb	63	9.583	10.650	9.583
2	11.129	Unknown	5525569	49.40	180158	44.65	bb	81	10.717	12.067	10.717



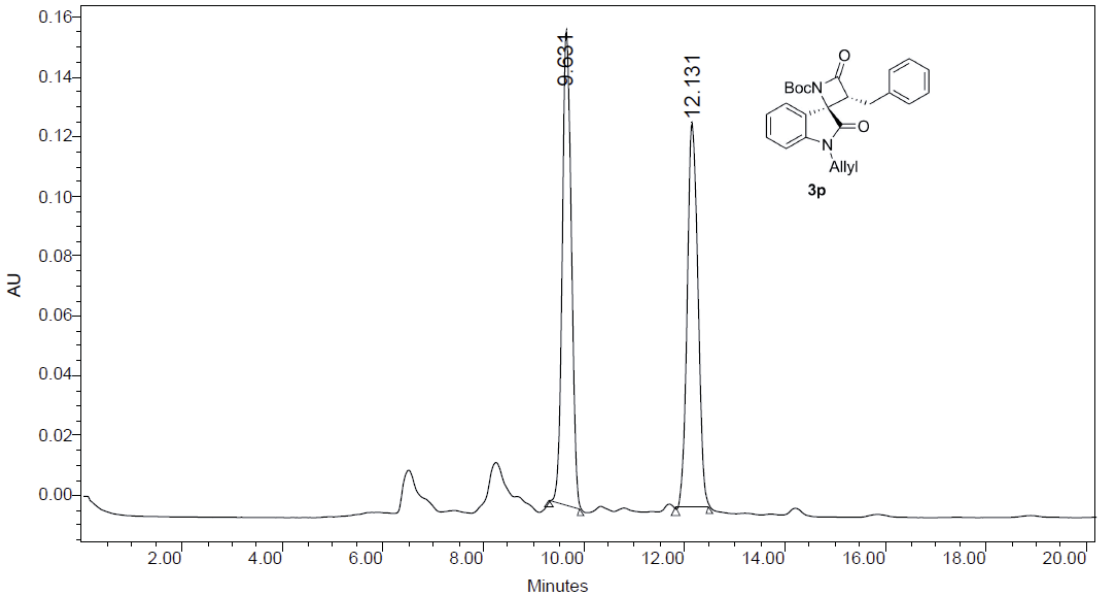
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	10.017	Unknown	97649	2.80	4064	3.71	bb	49	9.700	10.533	9.700
2	11.138	Unknown	3389511	97.20	105595	96.29	bb	108	10.650	12.450	10.650



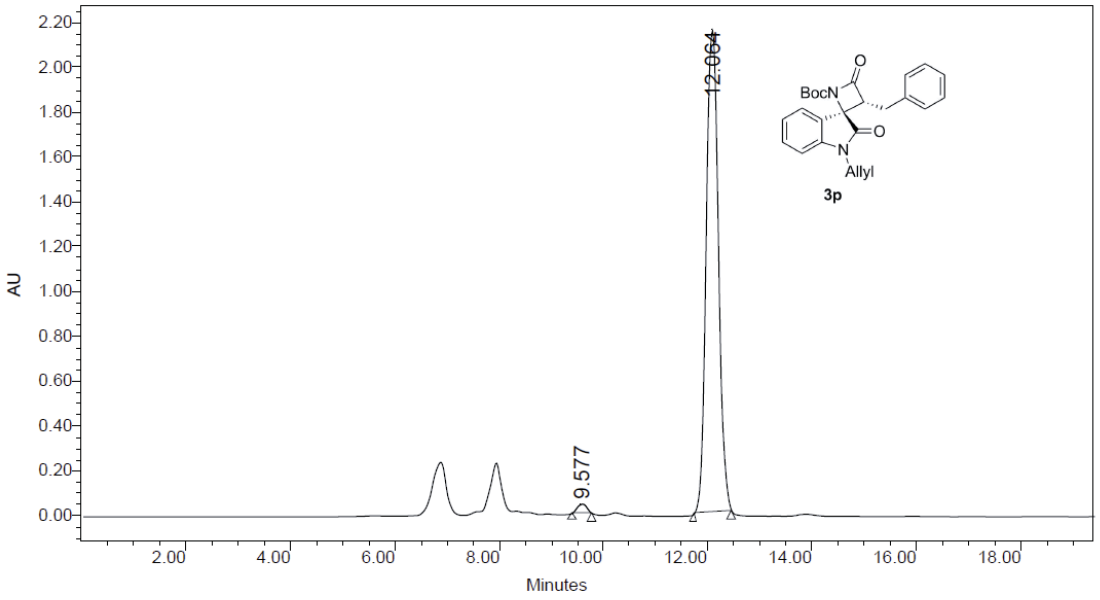
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	10.133	Unknown	1647463	46.27	131714	50.34	bb	35	9.867	10.467	9.867
2	11.985	Unknown	1912741	53.73	129935	49.66	bb	46	11.683	12.450	11.683



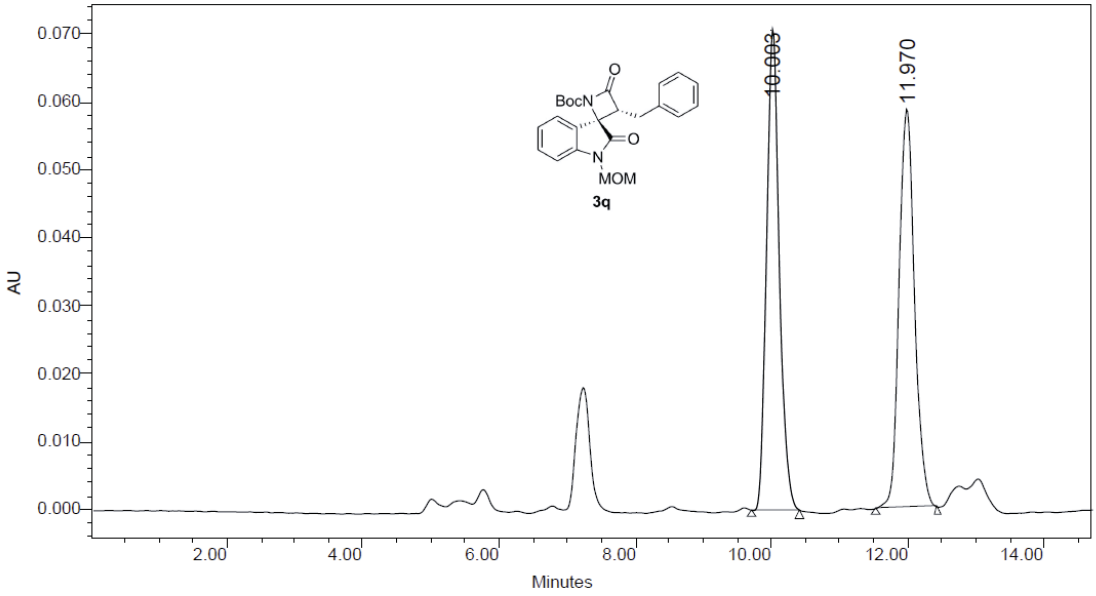
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)
1	10.126	Unknown	167635	0.97	13278	1.18	bb	30	9.933	10.433
2	11.931	Unknown	17144273	99.03	1109736	98.82	bb	63	11.550	12.617



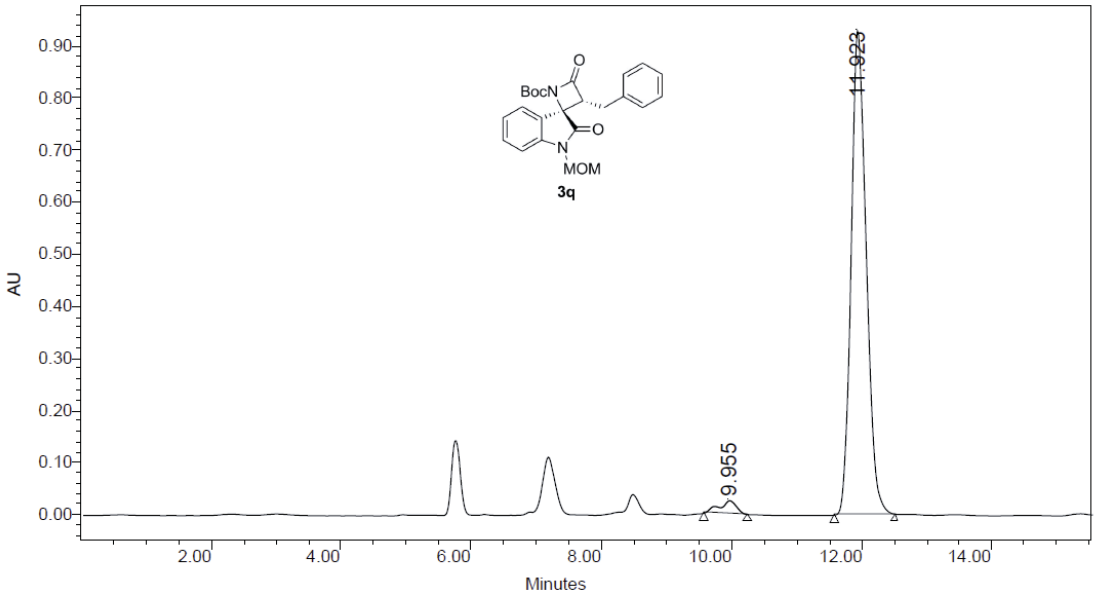
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	9.631	Unknown	1989186	50.97	158816	55.20	bb	36	9.317	9.933	9.317
2	12.131	Unknown	1913283	49.03	128878	44.80	bb	39	11.833	12.500	11.833



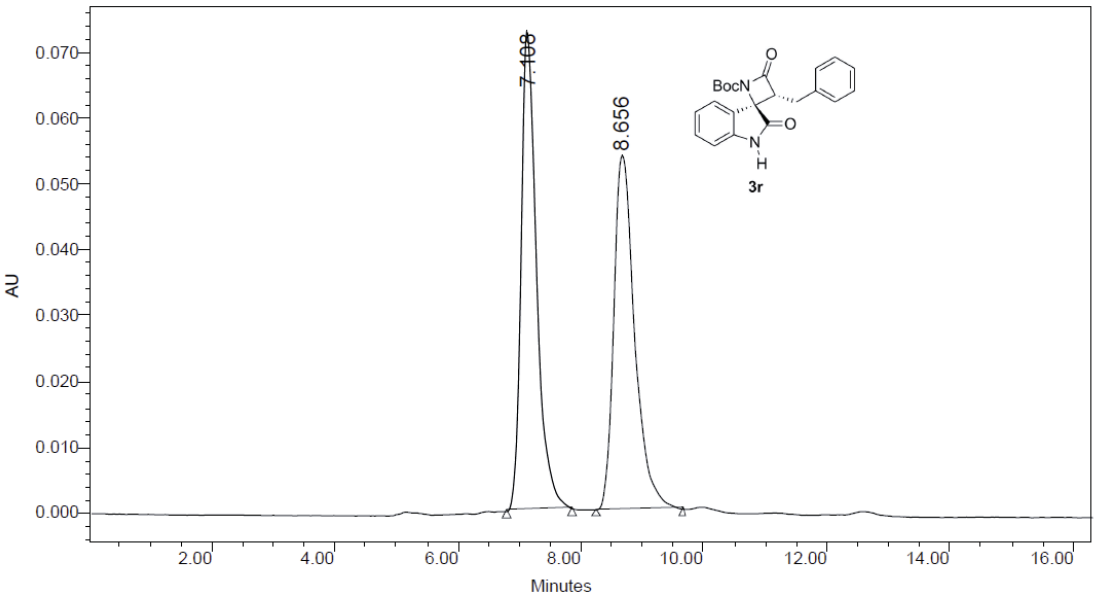
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)
1	9.577	Unknown	547707	1.54	42187	1.92	bb	23	9.383	9.783
2	12.064	Unknown	35118671	98.46	2153704	98.08	bb	44	11.717	12.450



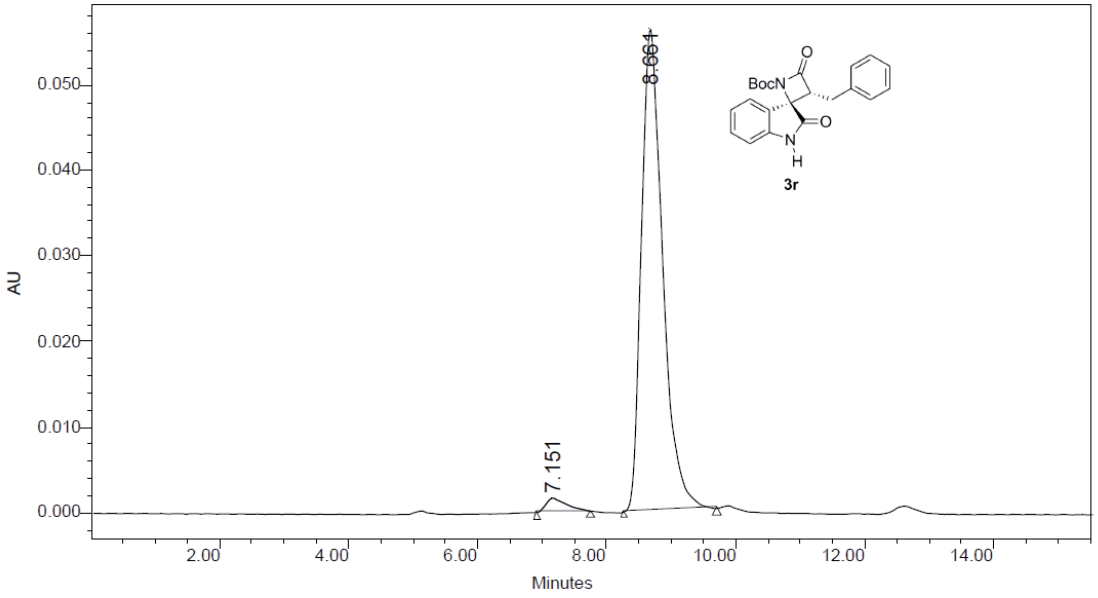
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	10.003	Unknown	920614	49.76	70742	54.69	bb	43	9.700	10.417	9.700
2	11.970	Unknown	929606	50.24	58607	45.31	bb	54	11.533	12.433	11.533



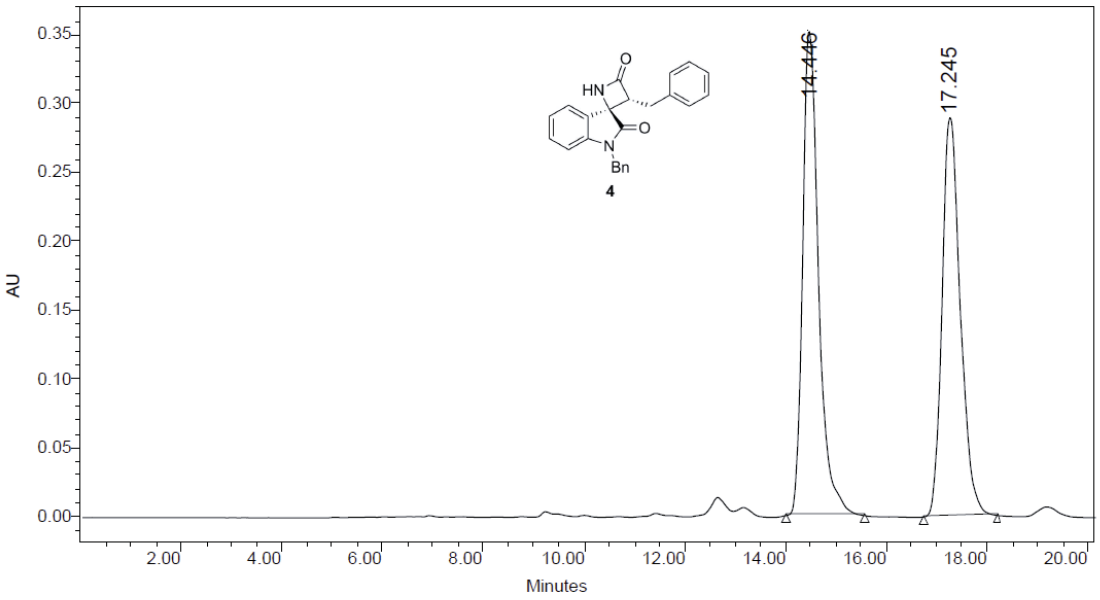
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	9.955	Unknown	474548	3.07	25601	2.67	bb	40	9.567	10.250	9.567
2	11.923	Unknown	14973794	96.93	931927	97.33	bb	54	11.583	12.500	11.583



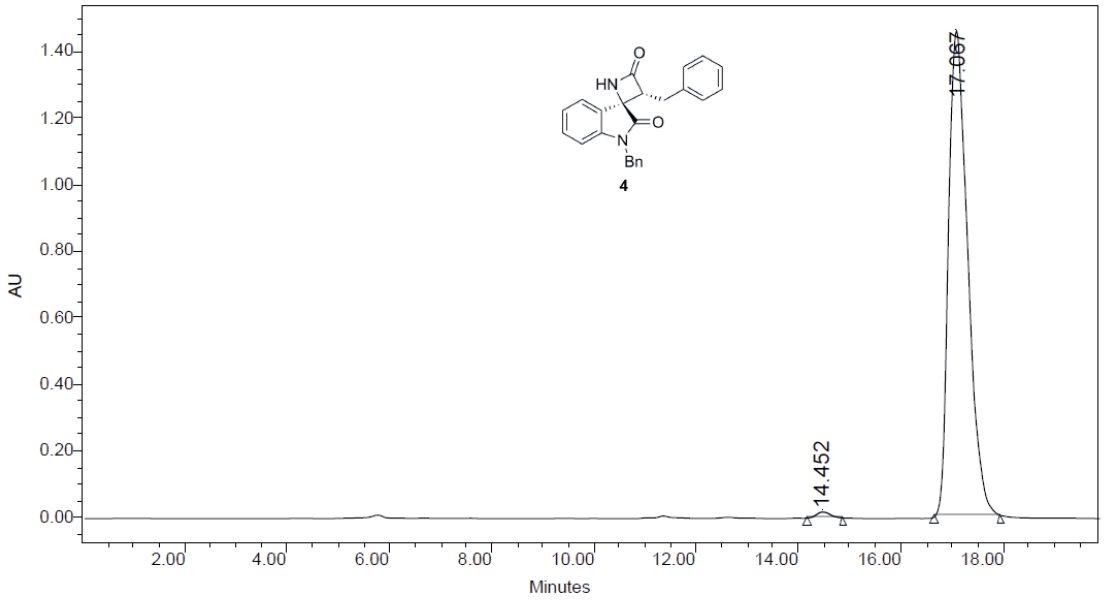
	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	7.108	Unknown	1276907	50.03	73099	57.56	bb	63	6.800	7.867	6.800
2	8.656	Unknown	1275273	49.97	53907	42.44	bb	84	8.250	9.650	8.250



	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	7.151	Unknown	36219	2.73	1623	2.80	bb	50	6.917	7.750	6.917
2	8.661	Unknown	1291047	97.27	56259	97.20	bb	86	8.267	9.700	8.267



	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)	Baseline Start (min)
1	14.446	Unknown	7734970	51.42	351578	54.84	bb	92	14.017	15.567	14.017
2	17.245	Unknown	7307082	48.58	289550	45.16	bb	86	16.750	18.200	16.750



	RT (min)	Peak Type	Area (*sec)	% Area	Height ( )	% Height	Integration Type	Points Across Peak	Start Time (min)	End Time (min)
1	14.452	Unknown	337943	0.88	17859	1.21	bb	41	14.167	14.867
2	17.067	Unknown	38006839	99.12	1458811	98.79	bb	78	16.650	17.950