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# Supporting Information



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# Enantioselective Total Synthesis Of (+)-Homochelidonine Using a Pd(II)-Catalyzed Asymmetric Ring-Opening Reaction of a *meso*-Azabicyclic Alkene with an Arylboronic Acid

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## (I) General directions

All reactions were carried out under an argon atmosphere, in flame-dried roundbottom flasks fitted with rubber septa, with magnetic stirring unless otherwise noted. Air- or water-sensitive liquids and solutions were transferred via syringe. Organic solutions were concentrated by rotary evaporation at 23–40 °C at 40 Torr. Analytical thin layer chromatography (TLC) was performed with Silicycle™ normal phase glass plates (0.25 mm, 60-A pore size, 230-400 mesh). Visualization was accomplished with 254 nm UV light and/or by immersion in potassium permanganate or phosphomolybdic acid

solution, followed by brief heating using a heat gun. Purification of reaction products was generally done by flash chromatography with Silicycle™ Ultra-Pure 230-400 mesh silica gel. Tetrahydrofuran, 1,4-dioxane and toluene were purified by distillation under $N_2$ from Na/benzophenone immediately prior to use. Triethylamine was purified by distillation under $N_2$ from sodium hydroxide immediately prior to use. Diethyl ether and dichloromethane were purified by the method of Pangborn et al.[1] Proton nuclear magnetic resonance spectra ($^1$H NMR) spectra and carbon nuclear magnetic resonance spectra ($^{13}$C NMR) were recorded at 23 °C in $CDCl_3$ with a Varian 400 (400 MHz/100 MHz) NMR spectrometer equipped with ATB8123-400 probe, or a Varian Mercury 400 (400 MHz/100 MHz) NMR spectrometer equipped with a Nalorac4N-400 probe. Recorded shifts for protons are reported in parts per million (δ scale) downfield from tetramethylsilane and are referenced to residual proton in the NMR solvent ($CHCl_3$: δ 7.26). Chemical shifts for carbon resonances are reported in parts per million (δ scale) downfield from tetramethylsilane and are referenced to the carbon resonances of the solvent ($CDCl_3$: δ 77.0). Data are represented as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad), coupling constant (*J*, Hz) and integration. Infrared (IR) spectra were obtained using a Perkin-Elmer Spectrum 1000 FT-IR spectrometer as a neat film on a NaCl plate. Data is presented as follows: frequency of absorption ($cm^{-1}$) and intensity of absorption (s = strong, m = medium, w = weak, br = broad). High resolution mass spectra were obtained from a SI2 Micromass 70S-250 mass spectrometer (EI) or an ABI/Sciex Qstar mass spectrometer (ESI). Melting points were taken on a Fisher-Johns melting point apparatus and are uncorrected. Optical rotations were measured in a 5.0 or 10.0 cm cell with a Rudolph Autopol IV polarimeter digital polarimeter equipped with a sodium lamp source (589 nm), and are reported as follows: $[\alpha]_D^T$ °C (c = g/100 mL, solvent).

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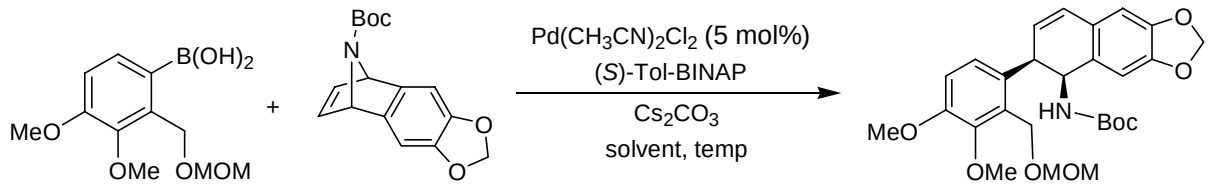
(1) A. B. Pangborn, M. A. Giardello, R. H. Grubbs, R. K. Rosen, F. J. Timmers, *Organometallics* **1996**, *15*, 1518.

## (II) Optimization of conditions for the asymmetric ring-opening reaction




**17** **10** **18**

**General procedure:** A 5 mL flame-dried round bottomed flask was charged with bis(acetonitrile)dichloropalladium(II) (2.2 mg, 0.0085 mmol) and (*S*)-Tol-BINAP (1.1−2.0 eq/Pd) and placed under an argon atmosphere. The appropriate solvent (0.5 mL) was added and the resulting catalyst mixture was stirred at rt for 1 h or until all solids went into solution. A solution of azabicycle **10** (50 mg, 0.17 mmol) and boronic acid **17** (67 mg, 0.26 mmol) in solvent (0.5 mL) was added by syringe followed by $Cs_2CO_3$ (0−1 eq). The resulting reaction mixture was then allowed to stir at the required temperature. At the end of the reaction, the mixture was adsorbed onto silica gel and subjected to flash column chromatography (25% hexane in EtOAc) to give dihydronaphthalene **18**. Enantioselectivity was determined by chiral HPLC analysis (Chiralpak AD, hexane/2-propanol 90:10, flow rate 1.0 mL/min): $T_r$: 15.24, 27.47 min.

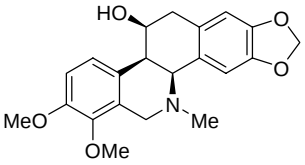
***Table 2.*** Optimization of the asymmetric ring-opening reaction.

| Entry | Solvent | Ligand (eq./Pd) | $Cs_2CO_3$ [eq.] | Rxn Time [h] | Temp. [°C] | Yield[a] [%] | *ee* [%][b] |
|---|---|---|---|---|---|---|---|
| 1[c] | MeOH | 1.1 | 1 | 24 | 60 | 72 | 80 |
| 2 | MeOH | 1.1 | 1 | 20 | 25 | 90 | 91 |
| 3[d] | MeOH | 1.1 | 1 | 20 | 25 | 70 | 82 |
| 4[e],[f] | MeOH | 1.1 | 1 (aq.) | 24 | 25 | 47 | 87 |
| 5[f] | MeOH | 1.1 | 1 | 2 | 25 | 81 | 90 |
| 6 | MeOH | 1.1 | 1 | 2 | 25 | 75 | 89 |
| 7[g] | MeOH | 1.1 | 1 | 2 | 25 | 82 | 91 |
| 8 | MeOH | 1.5 | 1 | 2 | 25 | 75 | 90 |
| 9 | MeOH | 2.0 | 1 | 2 | 25 | 77 | 90 |
| 10 | MeOH | 1.1 | 0.5 | 2 | 25 | 74 | 89 |
| 11[h] | MeOH | 1.1 | none | 18 | 25 | - | - |
| 12[i] | Toluene | 1.1 | 1 | 24 | 25 | n.d. | n.d. |
| 13[i] | THF | 1.1 | 1 | 24 | 25 | n.d. | n.d. |
| 14 | MeOH | 1.1 | 1 | 8 | 0 | 58 | 89 |
| 15 | MeOH | 1.1 | 1 | 27 | −20 | 51 | 92 |

[a] Isolated yields. [b] Enantioselectivites determined by chiral HPLC analysis ((Chiralpak AD, hexane/2-propanol 90:10, flow rate 1.0 mL/min): $T_r$: 15.24, 27.47 min). [c] 1 mol% catalyst used. [d] $Bu_4NBr$ (1 eq.) added. [e] 1 M (aq) $Cs_2CO_3$ used. [f] Undistilled MeOH used. [g] Dry and degassed MeOH used. [h] No reaction after 18 h. [i] Product impure and low yield after column chromatography.

## (III) Procedures and data for all compounds

1,2- (4b*R*,5*S*,11b*S*)-1,2-Dimethoxy-12-methyl-4b,5,6,11b,12,13-hexahydro[1,3]benzodioxolo[5,6-*c*]phenanthridin-5-ol [(+)-homochelidonine][2] **1**

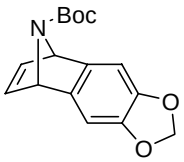



$LiAlH_4$ (36 mg, 0.96 mmol) was added in one portion to a solution of epoxide **27** (117 mg, 0.24 mmol) in 1,4-dioxane (5 mL) at 0 °C and stirred at this temp for 1 h. The reaction mixture was then heated to reflux for 12 h. After cooling to 0 °C the excess $LiAlH_4$ was destroyed by successive addition of acetone (1 mL), $Et_2O$ (1 mL) and $H_2O$ (10 mL). The layers were separated and the aqueous layer was extracted with EtOAc (2 x 20 mL); the combined organic layers were dried ($MgSO_4$) and solvent evaporated *in vacuo*. The residue was purified by column chromatography (30% EtOAc in hexane) to give (+)-homochelidonine **1** (77 mg, 87%) as a white solid with an *ee* of 99% as determined by chiral HPLC analysis (Chiralcel OD, hexane/2-propanol 85:15, flow rate 1.0 mL/min); $T_r$: 18.4 (minor), 26.6 (major). Mp 190−193 °C (lit.[3] mp 192−193.5 °C); $R_f$ = 0.26 (50% EtOAc in hexane); $^1$H NMR δ 7.75 (br s, 1H), 6.98 (d, *J* = 8.5, 1H), 6.88 (d, *J* = 8.5, 1H), 6.67 (s, 1H), 6.65 (s, 1H), 5.95 (d, *J* = 1.5, 1H), 5.94 (d, *J* = 1.5, 1H), 4.27−4.25 (m, 1H), 4.20 (d, *J* = 16, 1H), 3.88 (s, 3H), 3.86 (s, 3H), 3.54 (br d, *J* = 2, 1H), 3.44 (d, *J* = 16, 1H), 3.23 (dd, *J* = 17.5, 1, 1H), 3.09 (dd, *J* = 17.5, 4.5, 1H), 2.96 (t, *J* = 2.5, 1H), 2.30 (s, 3H); $^{13}$C NMR δ 150.6, 147.9, 145.1, 144.6, 130.3, 128.7 (x2), 125.7, 123.1, 111.9, 111.7, 109.4, 100.9, 71.9, 62.6, 60.1, 55.9, 55.1, 42.5, 41.8, 39.6; IR ($CH_2Cl_2$)/cm$^{-1}$ 2914s, 1486m, 1278s, 1230m, 1081m, 1041m, 937m; MS (EI): *m*/*z* 369 ($M^+$, 56), 351 (100), 320 (35), 336 (20), 204 (22), 192

(2) M. Hanaoka, S. Yoshida, C. Mukai, *Tetrahedron Lett*. **1985**, *26*, 5163.
(3) M. Nečas, J. Dostál, I. Kejnovská, M. Vorlíčková, J. Slavík, *Journal of Molecular Structure* **2005**, *734*, 1.

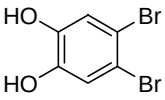
(19); HRMS (EI) calcd for $C_{21}H_{23}NO_5$ ($M^+$) 369.1576, found 369.1575; $[\alpha]_D^{25}$ = + 120 (*c*, 1.0 in EtOH) (lit.[5] $[\alpha]_D^{25}$ = + 128 (*c*, 1.0 in EtOH)).

*tert*-Butyl 5,8-dihydro-5,8-epiminonaphtho[2,3-*d*][1,3]dioxole-10-carboxylate **10**



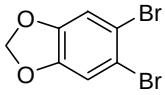

A mixture of dibromide **13** (5.99 g, 21.4 mmol) and freshly distilled *N*-Boc-pyrrole (5.35 mL, 32.0 mmol) in toluene (100 mL) was cooled to –78 °C. *n*-Butyllithium (1.6 M in hexane; 29.4 mL, 47.0 mmol) was added dropwise over a period of 2.5 h. The resulting bright orange solution was allowed to warm up to rt over 3 h and then left for a further 17 h at rt. The reaction mixture was then quenched with water (70 mL) and the phases were separated. The aqueous layer was extracted with EtOAc (3 x 80 mL) and the organic layers were combined, dried ($MgSO_4$) and concentrated *in vacuo* to give a brown oil. This was purified by column chromatography (10% EtOAc in hexane) to give azabicycle **10** (4.37 g, 71%) as an off-white solid. Mp 91−93 °C; $R_f$ = 0.10 (10% EtOAc in hexane); $^1$H NMR δ 6.97 (br s, 2H), 6.84, (s, 2H), 5.91 (d, *J* = 1.5, 1H), 5.87 (d, *J* = 1.5, 1H), 5.40 (br s, 2H), 1.38 (s, 9H); $^{13}$C NMR δ 154.8, 144.3, 142.5, [104.7, 104.2], 101.4, 80.6, [66.9, 66.3], 28.1; IR ($CH_2Cl_2$)/cm$^{-1}$ 3441w, 2975m, 2930m, 1705m 1461s, 1345m, 1367m, 1321s, 1293m,1252m, 1166m, 1037m MS (EI) *m*/*z* 287 ($M^+$, 17), 231 (24), 205 (52), 187 (21), 161 (41); HRMS (EI) calcd for $C_{16}H_{17}NO_4$ ($M^+$) 287.1157, found 287.1155.

Dibromocatechol[4] **12**




Boron tribromide (1 M in $CH_2Cl_2$; 100 mL, 0.10 mol) was added slowly to a solution of 4,5-dibromovertrole (10.0 g, 33.8 mmol) in $CH_2Cl_2$ (240 mL) and the resulting reaction mixture was stirred at rt for 2 h. The reaction was quenched by the addition of water (150 mL) and the resulting biphasic mixture was saturated with sodium chloride. The layers were separated and the aqueous phase was extracted with $CH_2Cl_2$ (3 x 125 mL). The combined organic layers were dried ($Na_2SO_4$) and concentrated *in vacuo* to give dibromocatechol **12** (9.1 g, quant.) as an off-white solid. Mp 97−98 °C (lit.[2] mp 119 °C); $R_f$ = 0.46 (50% EtOAc in hexane); $^1$H NMR δ 7.14 (s, 2H), 5.29 (s, 2H); $^{13}$C NMR δ 143.5, 119.9, 114.9; IR ($CH_2Cl_2$)/cm$^{-1}$ 3582m, 3354s, 1589m, 1495s, 1415s, 1267m, 1173m, 860m; MS (EI) *m*/*z* 268 ($M^+$, 100), 159 (17), 77 (14); HRMS (ESI) calcd for $C_6H_4O_2Br_2$ ($M^+$) 265.8578, found 265.8580.

5,6-Dibromobenzo[1,3]dioxole **13**

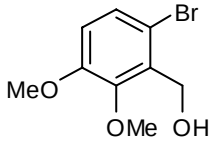



Bromochloromethane (4.24 mL, 65.3 mmol) was added to a stirred solution of catechol **12** (8.76 g, 32.7 mmol) and cesium carbonate (15.97 g, 49.0 mmol) in anhydrous DMF (50 mL). The resulting purple/brown suspension was then heated to 110 °C for 3h. After cooling to rt, the reaction mixture was filtered through a pad of celite which was then washed with EtOAc. Water (100 mL) was added to the filtrate, the organic layer was separated and the aqueous layer was extracted with EtOAc (2 x 100 mL). The combined organic layers were dried ($Na_2SO_4$) and concentrated *in vacuo* to give a brown solid. This was purified by column chromatography (10% EtOAc in hexane) to give

(4) C. F. van Nostrum, S. J. Picken, A.−J. Schouten, R. J. M. Nolte, *J. Am. Chem. Soc.* **1995**, *117*, 9957.

dibromide **13** (6.86 g, 75%) as a white solid. Mp 82−83 °C; $R_f$ = 0.50 (10% EtOAc in hexane); $^1$H NMR δ 7.07 (s, 2H), 6.00 (s, 2H); $^{13}$C NMR δ 147.9, 115.4, 113.2, 102.3; IR ($CH_2Cl_2$)/cm$^{-1}$ 3111s, 1700m, 1469m, 1378s, 1322m, 1138m, 930m; MS (ESI) *m*/*z* 280 ($M^+$, 100), 143 (29), 62 (37); HRMS (EI) calcd for $C_7H_4O_2Br_2$ ($M^+$) 277.8578, found 277.8580.

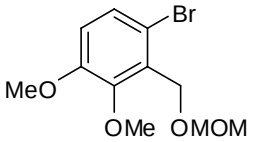
6-Bromo-2,3-dimethoxybenzyl alcohol[5] **15**




*N*-Bromosuccinimde (12.7 g, 71.4 mmol) was added to a solution of 2,3-dimethoxybenzyl alcohol (10.0 g, 59.5 mmol) in THF (45 mL) and stirred at rt until all the NBS had dissolved (approx. 30 min.). The THF was removed *in vacuo* and the residue was taken up in $Et_2O$ (110 mL). The resulting suspension was filtered to remove the insoluble succinimide and the filtrate was washed with 2 M NaOH (aq.) (2 x 100 mL). The organic phase was dried ($MgSO_4$) and concentrated under reduced pressure to give a pale yellow oil. This was purified by column chromatography (20% EtOAc in hexane) to give benzyl alcohol **15** (13.1 g, 83%) as a white solid. Mp 66−68 °C (lit.[3] 76 °C); $R_f$ = 0.08 (20% EtOAc in hexane); $^1$H NMR δ 7.27 (d, *J* = 8.5, 1H), 6.78 (d, *J* = 8.5, 1H), 4.83 (d, *J* = 7, 2H), 3.90 (s, 3H), 3.86 (s, 3H), 2.32 (t, *J* = 7, 1H); $^{13}$C NMR δ 152.3, 148.8, 133.9, 127.9, 114.8, 113.4, 61.7, 60.4, 56.0; IR ($CH_2Cl_2$)/cm$^{-1}$ 3426w, 2938m, 1576s, 1474m, 1413s, 1271s, 1229m, 1171m, 1079m, 1009m; MS *m*/*z* (EI) 269 (M+$Na^+$, 100), 127 (35), 79 (40); HRMS calcd for $C_9H_{11}BrO_3Na$ (M+$Na^+$) 268.9789, found 268.9780.

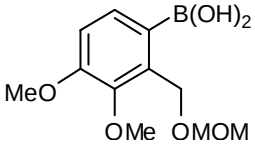
(5) A. M. Qandil, D. Ghosh, D. E. Nichols, *J. Org. Chem.* **1999**, *64*, 1407.

6-Bromo-2,3-dimethoxy(methoxymethoxy)methylbenzene[5] **16**




To a stirred solution of the benzyl alcohol **15** (10.0 g, 40.5 mmol) in dimethoxymethane (80 mL) was added lithium bromide (0.70 g, 8.09 mmol) and *p*-toluenesulfonic acid monohydrate (0.77 g, 4.05 mmol). The resulting reaction mixture was then stirred at rt for 14 h. Brine (100 mL) was added to the resulting white suspension and the mixture was extracted with $Et_2O$ (2 x 100 mL). The organic layers were combined, dried ($Na_2SO_4$) and concentrated *in vacuo* to give a yellow oil. This was purified by column chromatography (20% EtOAc in hexane) to give bromide **16** (8.49 g, 72%) as a colourless oil. $R_f$ = 0.35 (20% EtOAc in hexane); $^1$H NMR δ 7.29 (d, *J* = 9, 1H), 6.78 (d, *J* = 9, 1H), 4.76 (s, 2H), 4.74 (s, 2H), 3.88 (s, 3H), 3.85 (s, 3H), 3.46 (s, 3H); $^{13}$C NMR δ 152.3, 149.4, 131.2, 127.9, 116.2, 113.6, 96.4, 64.0, 61.7, 55.9, 55.4; IR ($CH_2Cl_2$)/cm$^{-1}$ 2940w, 1577w, 1475m, 1418m, 1378m, 1276s, 1232m, 1150m, 1101m, 1041m; MS *m*/*z* (EI) 313 (M+Na$^+$, 100), 244 (25), 64 (20) ; HRMS calcd for $C_{11}H_{15}BrO_4Na$ (M+Na$^+$) 313.0051, found 313.0048.

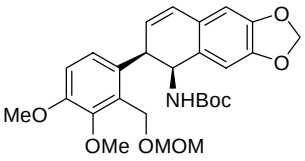
3,4-Dimethoxy-2-(methoxymethoxy)methylboronic acid[5] **17**




Aryl bromide **16** (3.81 g, 13.1 mmol) was dissolved in THF (14 mL) and cooled to −78 °C. *n*-Butyllithium (1.6 M in hexanes; 9.0 mL, 14.4 mmol) was added dropwise over a 20 min period and the reaction mixture was stirred at this temp for a further 15 min. Triisopropyl borate (9.0 mL, 39.2 mmol) was added in a single portion and the reaction mixture (a pale yellow suspension) was allowed to warm up to rt over 6 h and then stirred at this temperature for 12 h. The resulting orange suspension was cooled to 0 °C

and acidified to pH 5−6 with saturated aq. $NH_4Cl$. The solvent was removed *in vacuo* and the aqueous residue was extracted with $CH_2Cl_2$ (3 x 40 mL). The combined organic extracts were dried ($MgSO_4$) and concentrated under reduced pressure to give a yellow oil. This was purified by column chromatography (50% EtOAc in hexane) to give boronic acid **17** (2.11 g, 63%) as an off-white solid. Mp 63−65 °C; $R_f$ = 0.20 (50% EtOAc in hexane); $^1$H NMR δ 7.62 (d, *J* = 8, 1H), 6.92 (d, *J* = 8, 1H), 6.40 (br s, 2H), 4.87 (s, 2H), 4.72 (s, 2H), 3.89 (s, 3H), 3.83 (s, 3H), 3.41 (s, 3H); $^{13}$C NMR δ 154.4, 147.6, 134.1, 132.2, 126.8, 111.7, 95.3, 62.7, 61.3, 55.9, 55.6; IR ($CH_2Cl_2$)/cm$^{-1}$ 3380w, 2949s, 1589m, 1452m, 1419m, 1355m, 1279s, 1154m, 1098m, 1072m; MS (ESI): *m*/*z* 279 (M+Na$^+$, 100); HRMS (ESI) calcd for $C_{11}H_{17}BO_6Na$ (M+Na$^+$) 279.1010, found 279.1007.

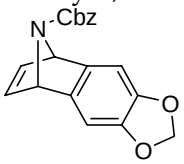
*tert*-Butyl (5*R*,6*S*)-6-(3,4-dimethoxy-2-((methoxymethoxy)methyl)phenyl)-5,6-dihydronaphtho[2,3-d][1,3]dioxol-5-ylcarbamate **18**




Following the general procedure (Table 2, p. 4, entry 2 conditions), the addition of the azabicycle **10** (1.0 g, 3.48 mmol) and boronic acid **17** (1.34 g, 5.22 mmol) in MeOH (10 mL) followed by cesium carbonate (1.13 g, 3.48 mmol) to bis(acetonitrile)dichloropalladium(II) (45 mg, 0.17 mmol) and (*S*)-Tol-BINAP (0.13 g, 0.19 mmol) gave dihydronaphthalene **18** (1.56 g, 90% yield) as a white solid with an *ee* of 91% as determined by chiral HPLC analysis (Chiralpak AD, hexane/2-propanol 90:10, flow rate 1.0 mL/min): $T_r$: 15.2 (minor), 27.5 (major) min. Mp 53−55 °C; $R_f$ 0.23 (20% EtOAc in hexane); $^1$H NMR δ 6.83 (d, *J* = 8.5, 1H), 6.75 (s, 1H), 6.73 (d, *J* = 8, 1H), 6.63 (s, 1H), 6.50 (d, *J* = 9.5, 1H), 5.98 (dd, *J* = 9.5, 4, 1H), 5.95 (d, *J* = 1.5, 1H), 5.92 (s, 1H), 5.17 (t, *J* = 8.5, 1H), 4.81 (d, *J* = 11, 1H), 4.72 (d, *J* = 6.5, 1H), 4.70 (d, *J* = 6.5, 1H), 4.68 (d, *J* = 11,

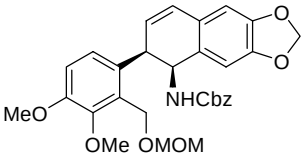
1H), 4.65 (d, *J* = 9, 1H), 4.20 (ddd, *J* = 7, 4.5, 2, 1H), 3.84 (s, 3H), 3.82 (s, 3H), 3.40 (s, 3H), 1.35 (s, 9H); $^{13}$C NMR δ 155.1, 151.6, 148.0, 147.1, 146.8, 130.9, 130.4, 129.7, 129.2, 127.4, 127.2, 124.7, 112.3, 107.3, 106.9, 101.0, 96.1, 79.3, 61.4, 60.4, 55.7, 55.5, 52.0, 39.6, 28.3; IR ($CH_2Cl_2$)/cm$^{-1}$ 3407w, 2923s, 1700s, 1507s, 1482s, 1364m, 1279s, 1244m, 1165m, 1037m; MS (ESI): *m*/*z* 522 (M+Na$^+$, 100); HRMS (ESI) calcd for $C_{27}H_{33}NO_8Na$ (M+Na$^+$) 522.2098, found 522.2116; $[\alpha]_D^{25}$ = + 90 (*c*, 1.0 in $CHCl_3$).

Benzy 5,8-dihydro-5,8-epiminonaphtho[2,3-*d*][1,3]dioxole-10-carboxylate **22**



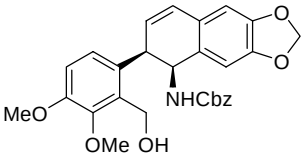

Iodotrimethylsilane (0.93 mL, 6.51 mmol) was added dropwise to a refluxing mixture of azabicycle **10** (1.70 g, 5.92 mmol) and triethylamine (0.99 mL, 7.10 mmol) in $CH_2Cl_2$ (25 mL). After 15 min, the reaction was cooled to 0 °C and anhydrous MeOH added (0.31 mL, 7.70 mmol). After 10 min freshly distilled benzyl chloroformate (1.10 mL, 7.70 mmol) was added and the reaction was left to stir for 3 h at rt. The reaction mixture was diluted with water (50 mL) and $CH_2Cl_2$ (25 mL) and the layers separated. The aqueous layer was extracted with EtOAc (2 x 50 mL), the combined organic layers were dried ($MgSO_4$) and solvent evaporated *in vacuo*. The residue was purified by column chromatography (10% EtOAc in hexane) to give azabicycle **22** (1.52 g, 80%) as a white solid. Mp 101 °C; $R_f$ = 0.33 (30% EtOAc in hexane); $^1$H NMR δ 7.34−7.25 (m, 5H), 6.98 (br s, 2H), 6.84 (br s, 2H), 5.90 (dd, *J* = 16, 1.5, 2H), 5.50 (s, 2H), 5.06 (s, 2H); $^{13}$C NMR δ 155.0, 144.4, [143.7, 142.8], 142.3, 136.2, 128.4, 128.0, 127.8, [104.7, 104.6], 101.1, 67.2, 66.4; IR ($CH_2Cl_2$)/cm$^{-1}$ 3053w, 2986w, 1711m, 1462m, 1265s, 1092w; MS *m*/*z* (EI) 321 (M$^+$, 25), 251 (12), 91 (100); HRMS calcd for $C_{19}H_{15}NO_4$ (M$^+$) 321.1001, found 321.1000.

Benzyl (5*R*,6*S*)-6-(3,4-dimethoxy-2-((methoxymethoxy)methyl)phenyl)-5,6-dihydronaphtho[2,3-d][1,3]dioxol-5-ylcarbamate **23**



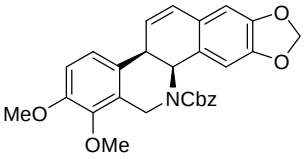

A flame-dried 100 mL flask was charged with bis(acetonitrile)dichloropalladium(II) (75 mg, 0.29 mmol) and (*S*)-Tol-BINAP (217 mg, 0.32 mmol) under an argon atmosphere. Anhydrous MeOH (16 mL) was added and the resulting catalyst mixture was stirred at rt for 1 h giving an orange solution. To this was added a solution of azabicycle **22** (1.88 g, 5.86 mmol) and boronic acid **17** (2.25 g, 8.79 mmol) in MeOH (16 mL) followed by cesium carbonate (1.91 g, 5.86 mmol) in one portion. The reaction mixture was allowed to stir for 6 h at rt and then dry loaded onto silica. Purification by flash column chromatography (10%→30% EtOAc in hexane) gave dihydronaphthalene **23** (2.78 g, 89%) as a pale brown foam with an *ee* of 90% as determined by chiral HPLC analysis (Chiralpak AD, hexane/2-propanol 85:15, flow rate 1.0 mL/min); $T_r$: 18.0 (minor), 59.4 (major). Recrystallization (2% $CH_2Cl_2$ in $Et_2O$) gave dihydronaphthalene **23** (2.50 g, 80%) as a white solid with an *ee* of 99% as determined by chiral HPLC analysis. Mp 106 °C; $R_f$ = 0.10 (20% EtOAc in hexane); $^1$H NMR δ 7.33−7.24 (m, 5H), 6.84 (d, *J* = 8.5, 1H), 6.76 (s, 1H), 6.69 (d, *J* = 8.5, 1H), 6.62 (s, 1H), 6.48 (d, *J* = 9.5, 1H), 5.96 (dd, *J* = 9.5, 4.5, 1H), 5.91 (d, *J* = 1.5, 1H), 5.89 (s, 1H), 5.23 (t, *J* = 9, 1H), 5.03 (d, *J* = 12.5, 1H), 5.04−5.02 (m, 2H), 4.73−4.61 (m, 4H), 4.23−4.20 (m, 1H), 3.82 (s, 3H), 3.78 (s, 3H), 3.40 (s, 3H); $^{13}$C NMR δ 155.7, 151.5, 148.0, 147.1, 146.8, 136.4, 130.8, 130.2, 129.4, 128.9, 128.3, 127.9, 127.4, 126.9, 124.5, 112.2, 107.3, 106.9, 100.9, 95.9, 77.2, 66.5, 61.3, 60.2, 55.5, 55.3, 52.6, 39.6; IR ($CH_2Cl_2$)/cm$^{-1}$ 3430w, 2919w, 2253m, 1711m, 1651w, 1484m, 1379w, 1277m, 1040m; MS *m*/*z* (EI) 556 (M+Na$^+$, 52), 322 (20), 321 (100), 290 (19); HRMS calcd for $C_{30}H_{31}NO_8Na$ (M+Na$^+$) 556.1962, found 556.1941; $[\alpha]_D^{25}$ = + 97 (*c*, 1.0 in $CHCl_3$).

Benzyl (5*R*,6*S*)-6-(2-(hydroxymethyl)-3,4-dimethoxyphenyl)-5,6-dihydronaphtho[2,3-d][1,3]dioxol-5-ylcarbamate **24**



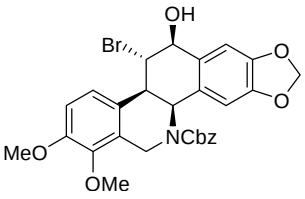

HCl (conc.) (17 mL) was added to a solution of dihydronaphthalene **23** (1.50 g, 2.81 mmol) in *i*PrOH (70 mL) and THF (70 mL) and the resulting mixture stirred at rt for 8 h. After careful quenching with excess sat. $NaHCO_3$ (aq) solution the layers were separated. The aqueous layer was extracted with EtOAc (2 x 100 mL), the combined organic layers were dried ($MgSO_4$) and solvent evaporated *in vacuo*. The residue was purified by column chromatography (20%→40% EtOAc in hexane) to give benzyl alcohol **24** (1.03 g, 75%) as a white solid. Mp 85 °C; $R_f$ = 0.30 (50% EtOAc in hexane); $^1$H NMR δ 7.35−7.28 (m, 3H), 7.19−7.16 (m, 2H), 6.91 (d, *J* = 8.5, 1H), 6.79 (s, 1H), 6.73 (d, *J* = 8.5, 1H), 6.64 (s, 1H), 6.53 (dd, *J* = 9.5, 2.5, 1H), 5.95−5.91 (m, 3H), 5.20 (dd, *J* = 10, 7, 1H), 4.95−4.69 (m, 4H), 4.30−4.28 (m, 1H), 3.90 (s, 3H), 3.82 (s, 3H), 2.98−2.94 (m, 1H), 1.80 (s, 1H); $^{13}$C NMR δ 155.7, 151.6, 147.9, 147.3, 147.1, 136.3, 133.5, 130.4, 128.7, 128.3, 128.0, 127.9, 127.8, 126.7, 125.1, 111.9, 108.4, 106.9, 101.1, 77.2, 66.6, 61.5, 56.5, 55.7, 52.5, 40.6; IR ($CH_2Cl_2$)/cm$^{-1}$ 3404br, 2938w, 1700s, 1484s, 1378m, 1276s, 1243s, 1082m, 1039s; MS *m*/*z* (EI) 512 (M+Na$^+$, 15), 321 (100), 291 (20); HRMS calcd for $C_{28}H_{27}NO_7Na$ (M+Na$^+$) 512.1679, found 512.1677; $[\alpha]_D^{22}$ = + 29 (*c*, 1.0 in $CHCl_3$).

Benzyl (4b*R*,11b*S*)-dimethoxy-4b,13-dihydro[1,3]benzodioxolo[5,6-*c*]phenanthridine-12(11b*H*)-carboxylate **25**



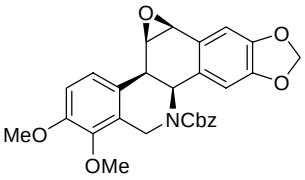

A solution of benzyl alcohol **24** (670 mg, 1.37 mmol) and triphenylphosphine (525 mg, 2.00 mmol) in $CH_2Cl_2$ (13 mL) was cooled to 0 °C. Carbon tetrabromide (663 mg, 2.00 mmol) was added in one portion and the resulting solution was stirred at this temp for 1 h. The reaction mixture was then diluted with $CH_2Cl_2$ (10 mL) and washed with water (2 x 20 mL). The solvent was evaporated *in vacuo* to give the crude benzyl bromide. This was dissolved in anhydrous DMF (23 mL) and cooled to 0 °C. Sodium hydride (60% in mineral oil; 80 mg, 2.00 mmol) was added in one portion and the suspension stirred at 0 °C for 90 min. The reaction mixture was quenched by the addition of cold water (15 mL) and the layers separated. The aqueous layer was extracted with EtOAc (2 x 20 mL), the combined organic layers were dried ($MgSO_4$) and solvent evaporated *in vacuo*. The residue was purified by column chromatography (20% EtOAc in hexane) to give dihydronaphthalene **25** (581 mg, 90%) as a white solid. Mp 169 °C; $R_f$ = 0.46 (50% EtOAc in hexane); $^1$H NMR δ 7.50−7.35 (m, 5H), 6.93 (d, *J* = 8.5, 1H), 6.72 (d, *J* = 8.5, 1H), 6.61 (s, 1H), 6.50 (s, 1H), 6.44−6.38 (m, 2H), 5.87 (s, 1H), 5.85 (s, 1H), 5.81 (br s, 1H), 5.28 (br s, 2H), 5.11 (d, *J* = 18, 1H), 4.41 (d, *J* = 18, 1H), 3.80 (s, 3H), 3.78 (s, 3H), 3.60 (br s, 1H); $^{13}$C NMR δ 156.2, 150.4, 147.2, 146.4, 144.3, 136.5, 128.4, 128.0, 127.9, 127.6 x2, 127.5, 127.3, 126.2, 121.8, 110.9, 107.5, 105.7, 100.8, 67.5, 60.0, 55.6, 53.4, 52.6, 39.1, 34.3; IR ($CH_2Cl_2$)/cm$^{-1}$ 2985w, 1699s, 1558m, 1482m, 1418m, 1278m, 1110m, 1037m; MS *m*/*z* (EI) 494 (M+Na$^+$, 90), 363 (20), 354 (21), 322 (22), 321 (100), 290 (21); HRMS calcd for $C_{28}H_{25}NO_6Na$ (M+Na$^+$) 494.1574, found 494.1579; $[\alpha]_D^{25}$ = + 119 (*c*, 1.0 in $CHCl_3$).

Benzyl (4b*R*,5*S*,6*S*,11b*S*)-5-bromo-6-hydroxy-1,2-dimethoxy-4b,6,11b,13-tetrahydro[1,3]benzodioxolo[5,6-c]phenanthridine-12(5*H*)-carboxylate **26**



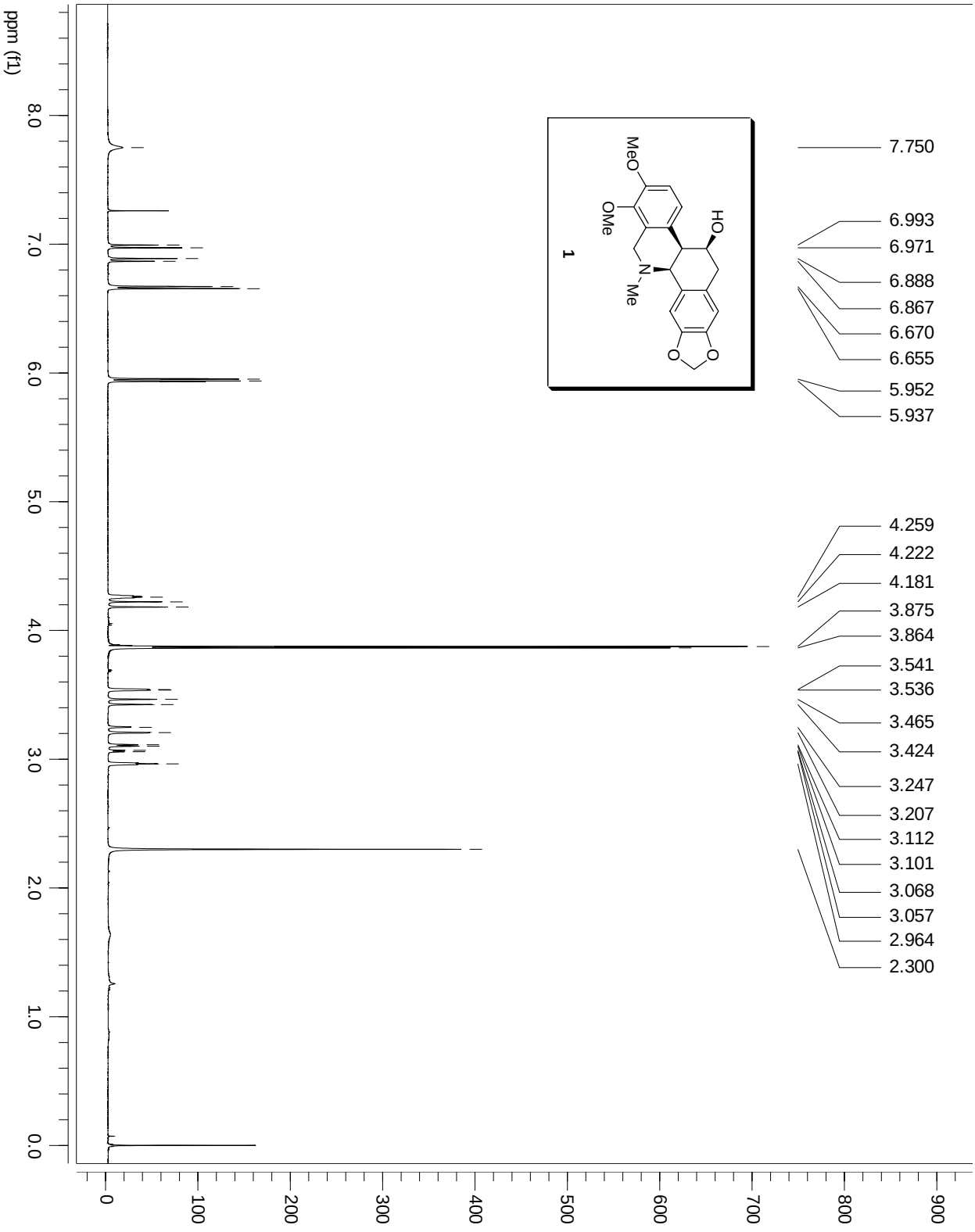

*N*-Bromosuccinimide (226 mg, 1.27 mmol) was added to a solution of dihydronaphthalene **25** (547 mg, 1.16 mmol) in THF (10.4 mL) and $H_2O$ (1.20 mL) and the resulting orange solution was allowed to stir at rt for 90 min. The reaction mixture was diluted with water (20 mL) and EtOAc (20 mL) and the layers separated. The aqueous layer was extracted with EtOAc (2 x 30 mL), the combined organic layers were dried ($MgSO_4$) and solvent evaporated *in vacuo*. The residue was purified by column chromatography (20% EtOAc in hexane) to give dihydronaphthalene **26** (495 mg, 75%) as a pale brown solid. Mp 75 °C; $R_f$ = 0.33 (50% EtOAc in hexane); $^1$H NMR δ 7.41−7.30 (m, 5H), 7.24 (d, *J* = 8.5, 1H), 6.82 (d, *J* = 8.5, 1H), 6.77 (s, 1H), 6.70 (s, 1H), 6.05 (d, *J* = 6.5, 1H), 5.92 (s, 2H), 5.30−5.27 (m, 3H), 5.14 (d, *J* = 18, 1H), 4.79 (d, *J* = 8.5, 1H), 3.92 (d, *J* =18, 1H), 3.88−3.85 (m, 1H), 3.80 (s, 3H), 3.77 (s, 3H), 1.37 (d, *J* = 10.5, 1H); $^{13}$C NMR δ 155.9, 150.8, 148.9, 147.6, 144.9, 136.4, 128.9, 128.5, 128.1, 128.1, 127.7, 127.6, 125.6, 124.0, 111.1, 109.5, 105.8, 101.3, 72.5, 67.7, 60.1, 55.7, 53.4, 49.8, 39.7, 38.6; IR ($CH_2Cl_2$)/cm$^{-1}$ 3443br, 291s, 1682s, 1606m, 1504s, 1242s, 1039s; MS (ESI) *m*/*z* 590 (M+Na$^+$, 100), 550 (45), 470 (26), 343 (20), 320 (25), 241 (35); HRMS calcd for $C_{28}H_{26}NO_7Na$ (M+Na$^+$) 590.0784, found 590.0783; $[\alpha]_D^{25}$ = + 103 (*c*, 1.0 in $CHCl_3$).

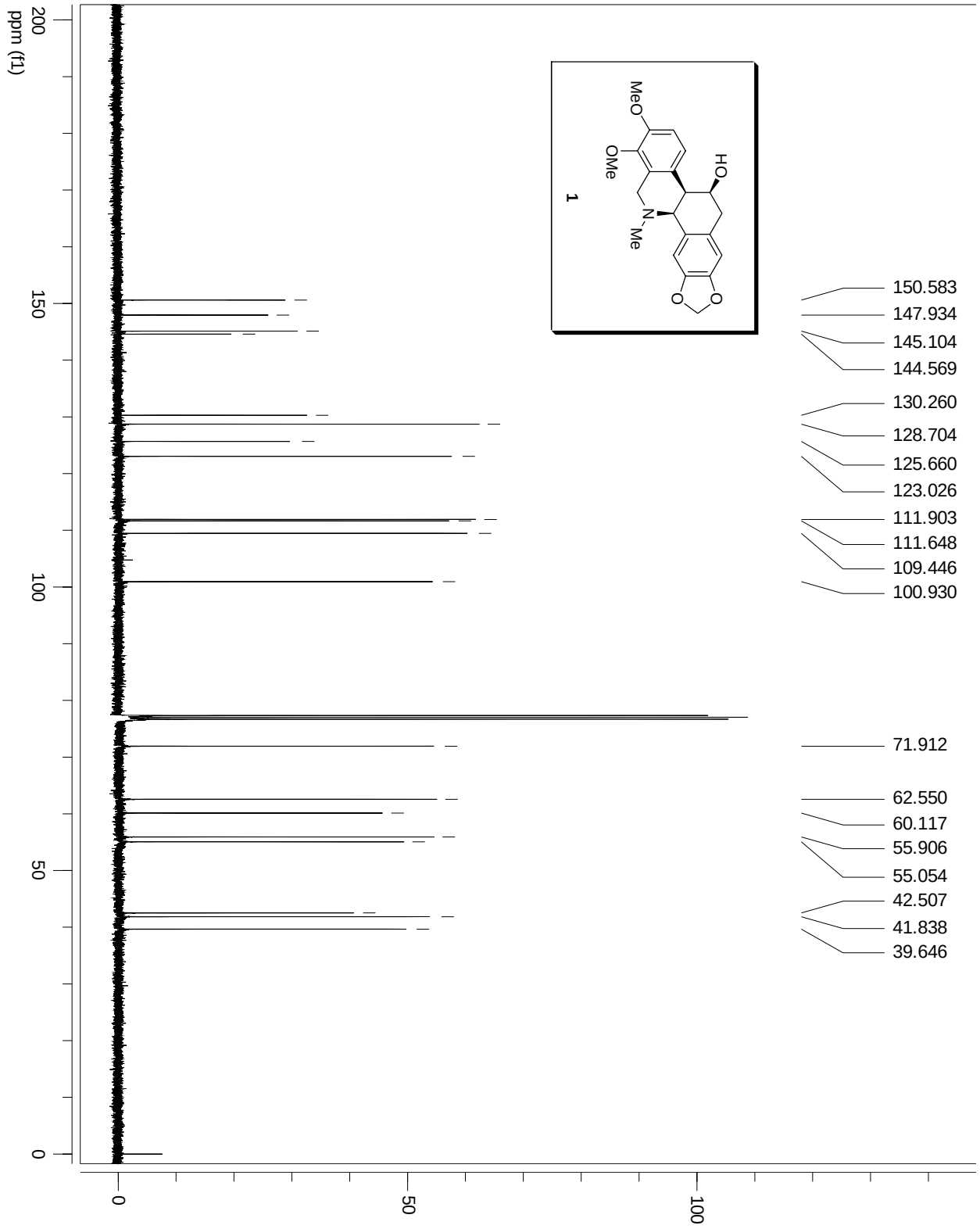
Benzyl (4b*R*,4c*R*,5a*S*,10b*S*)-1,2-dimethoxy-4b,5a,10b,12-tetrahydro[1,3]benzodioxolo[5,6-*c*]oxireno[*a*]phenanthndine-11(4c*H*)-carboxylate **27**




A solution of bromohydrin **26** (495 mg, 0.87 mmol) in THF (175 mL) was cooled to −78 °C under an argon atmosphere. A solution of potassium *tert*-butoxide (1 M in THF; 0.87 mL, 0.87 mmol) was added dropwise via a syringe and the resulting solution was stirred at this temp for 45 min. The reaction mixture was warmed to 0 °C and washed with cold water (100 mL). The layers were separated and the organic layer was dried ($Na_2SO_4$) and solvent evaporated *in vacuo* to give epoxide **27** (424 mg, quant.) as a pale yellow solid. Mp 60 °C; $R_f$ = 0.38 (50% EtOAc in hexane); $^1$H NMR δ 7.37−7.30 (m, 5H), 7.05 (d, *J* = 8.5, 1H), 6.89 (s, 1H), 6.84 (d, *J* = 8.5, 1H), 5.95 (d, *J* = 1.5, 1H), 5.91 (d, *J* = 1.5, 1H), 5.77 (d, *J* = 8, 1H), 5.20−5.16 (m, 2H), 5.15 (br s, 1H), 4.24 (d, *J* = 16, 1H), 3.84−3.68 (m, 10H); $^{13}$C NMR δ 156.6, 150.8, 148.6, 146.9, 144.6, 136.3, 128.7 x3, 128.4, 128.0, 127.9, 125.2, 124.2, 111.1, 110.0, 101.2, 67.6, 60.5, 55.7, 52.4, 51.1, 38.1, 36.8; IR ($CH_2Cl_2$)/cm$^{-1}$ 3441s, 1694s, 1488s, 1417m, 1236m, 1096m, 1036m; MS (ESI) *m*/*z* 510 (M+Na$^+$, 100), 488 (25); HRMS calcd for $C_{28}H_{25}NO_7Na$ (M+Na$^+$) 510.1523, found 510.1522; $[\alpha]_D^{25}$ = + 112 (*c*, 1.0 in $CHCl_3$).

## (IV) $^{1}H$ and $^{13}C$ NMR spectra for all compounds

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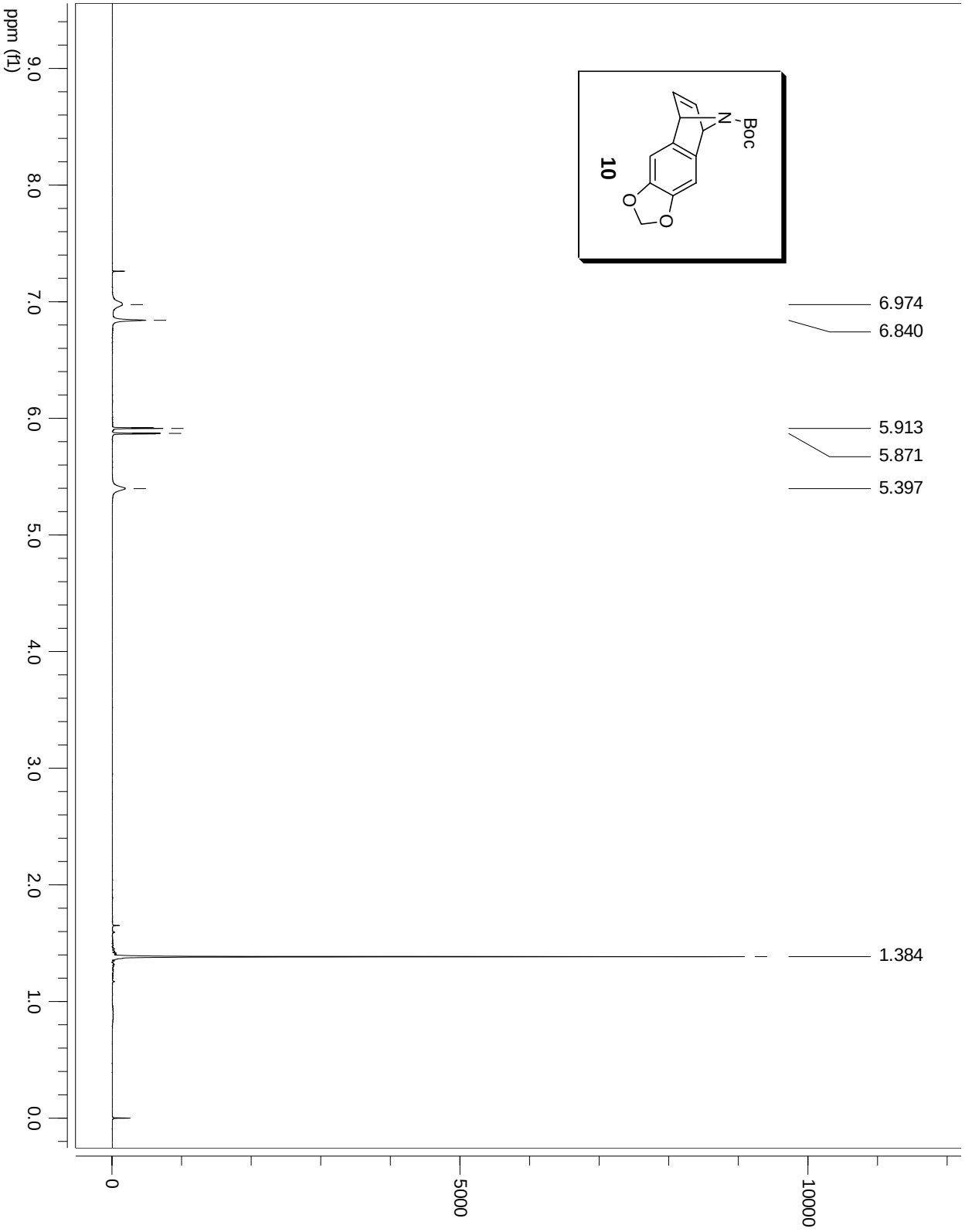
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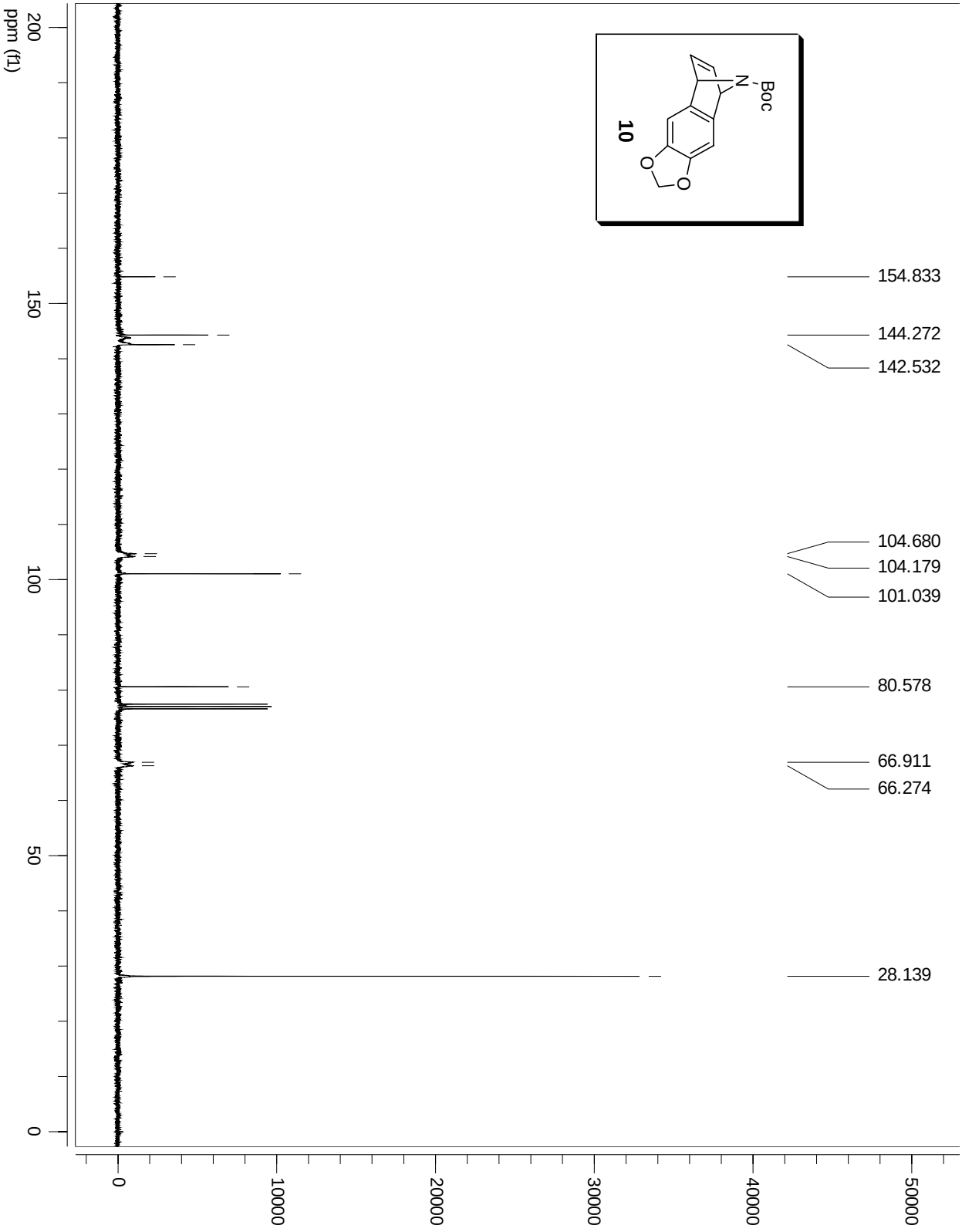
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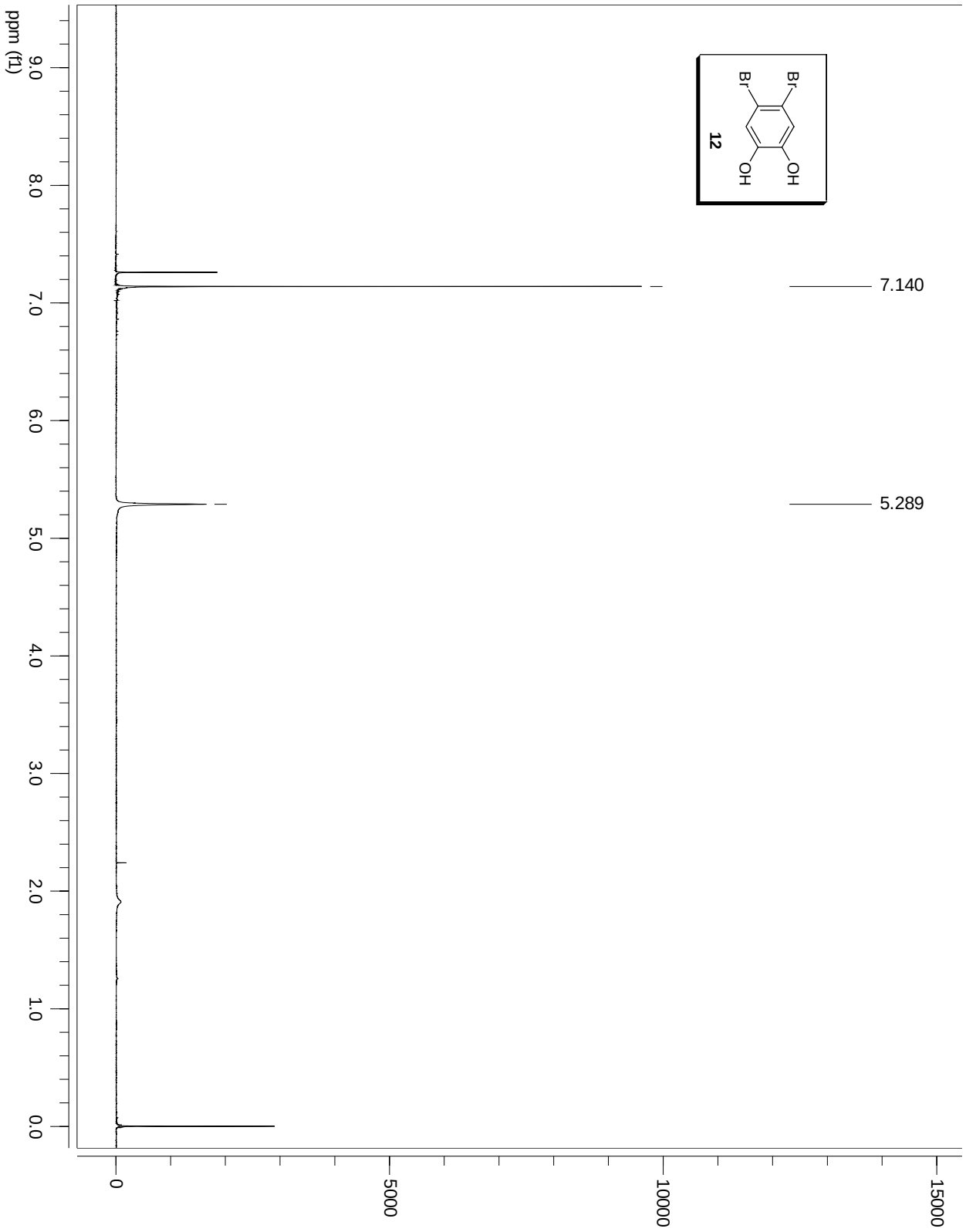
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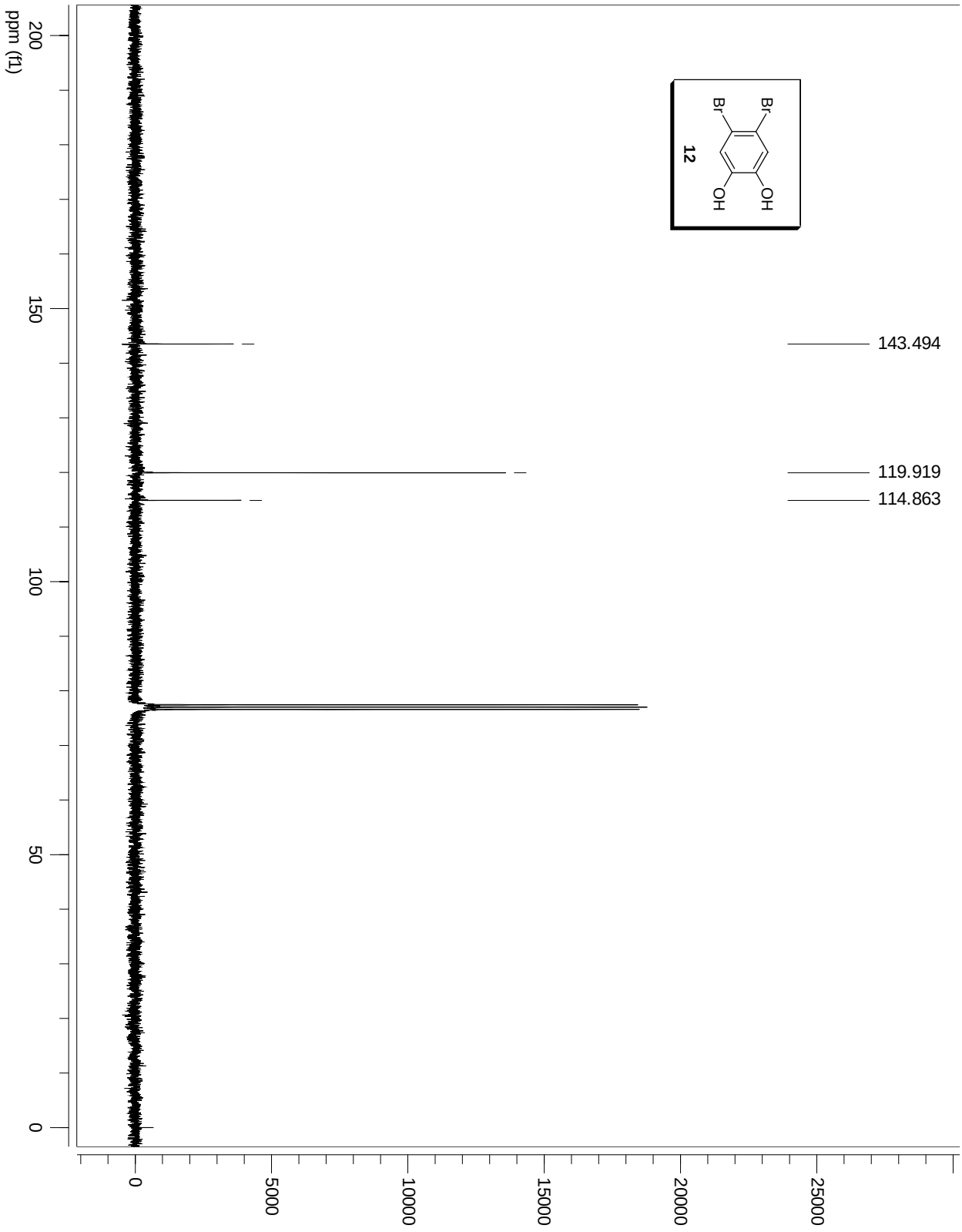
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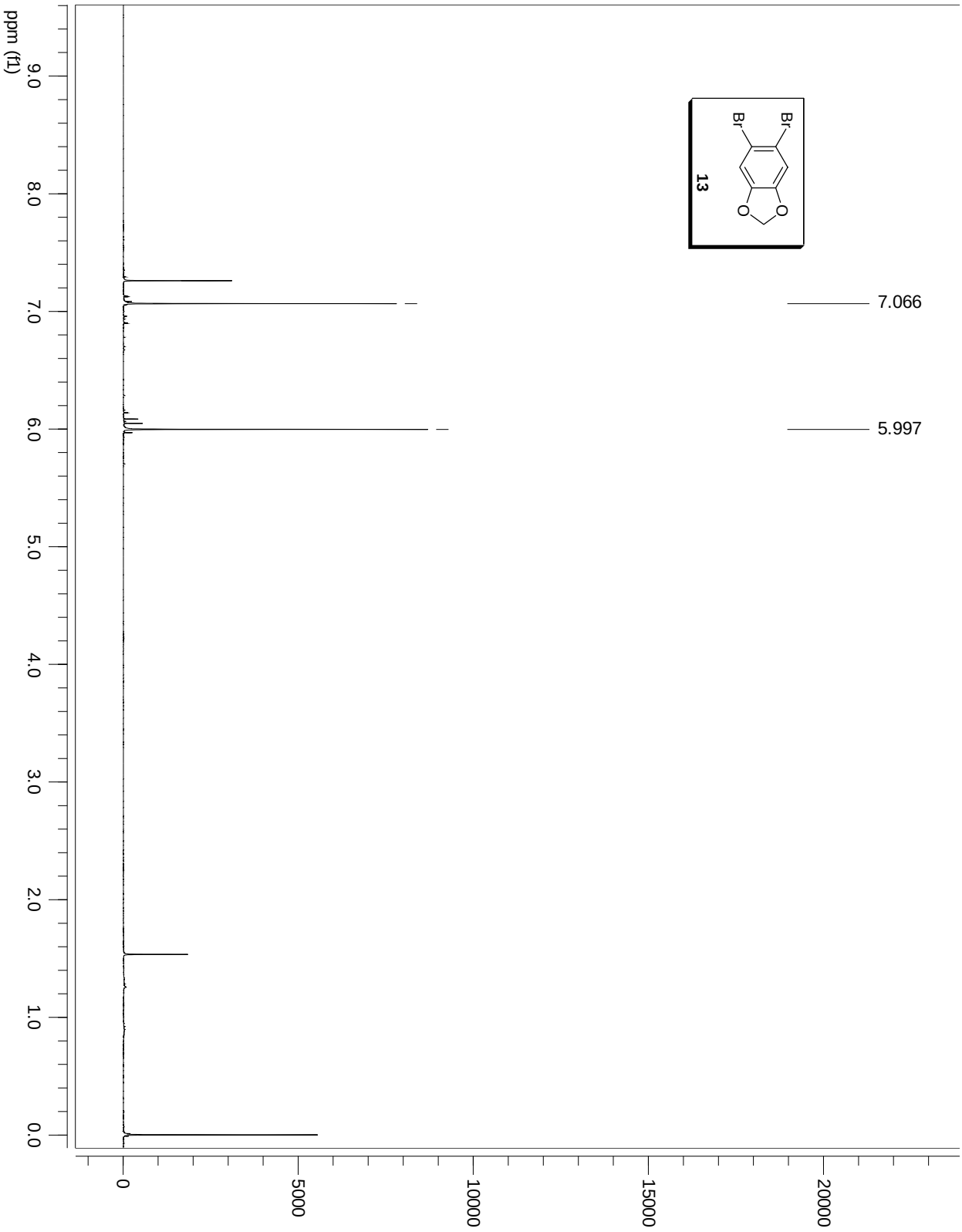
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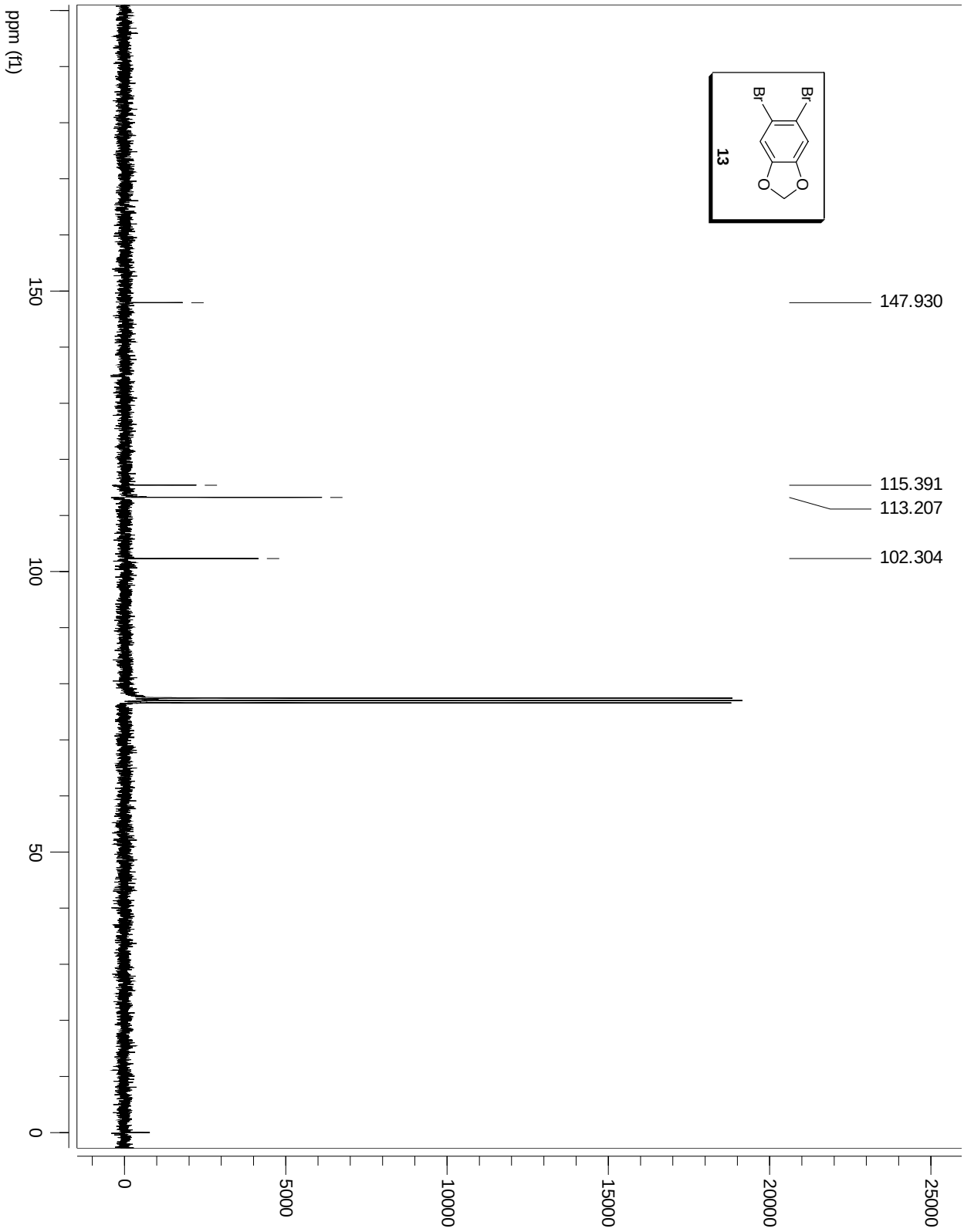
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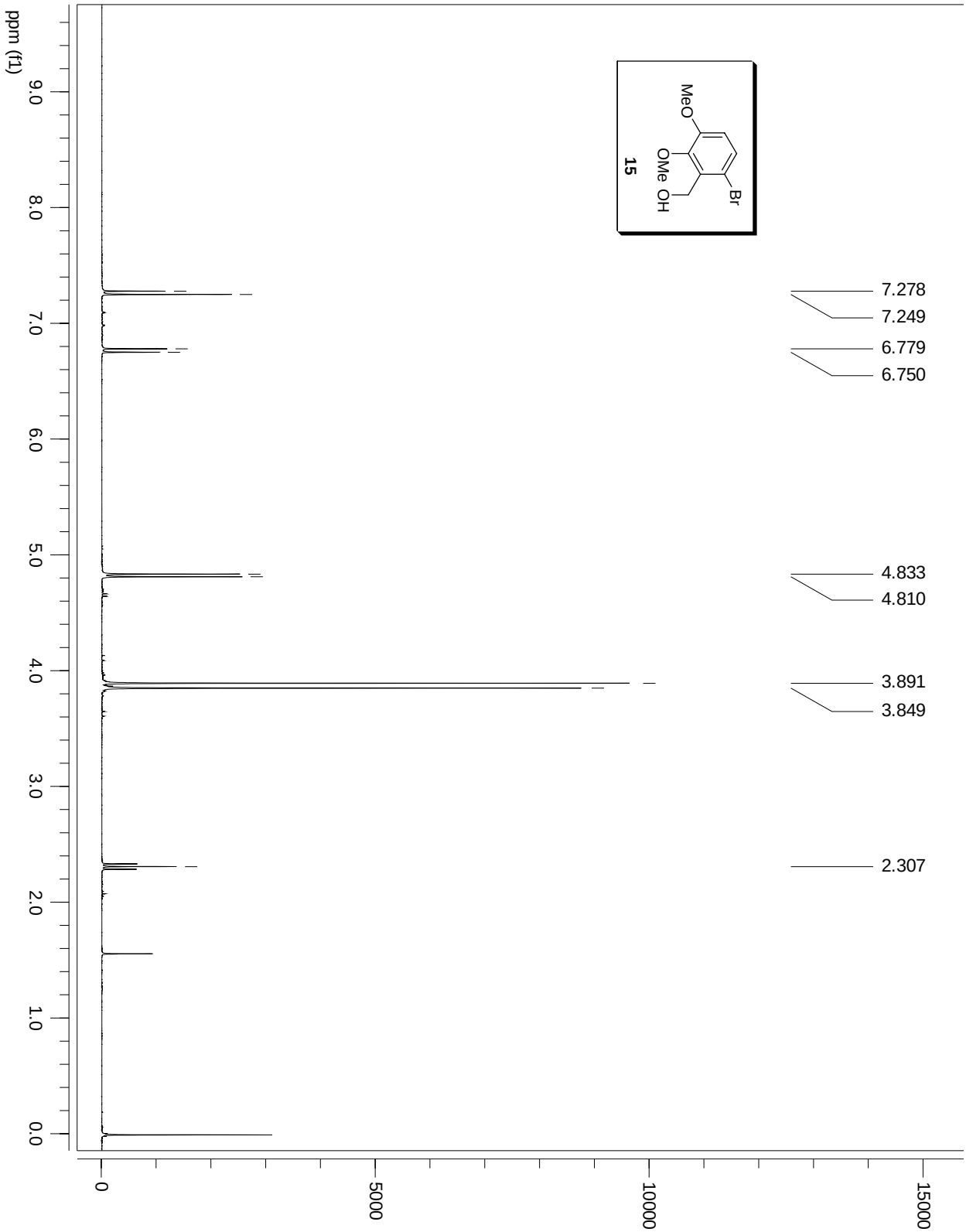
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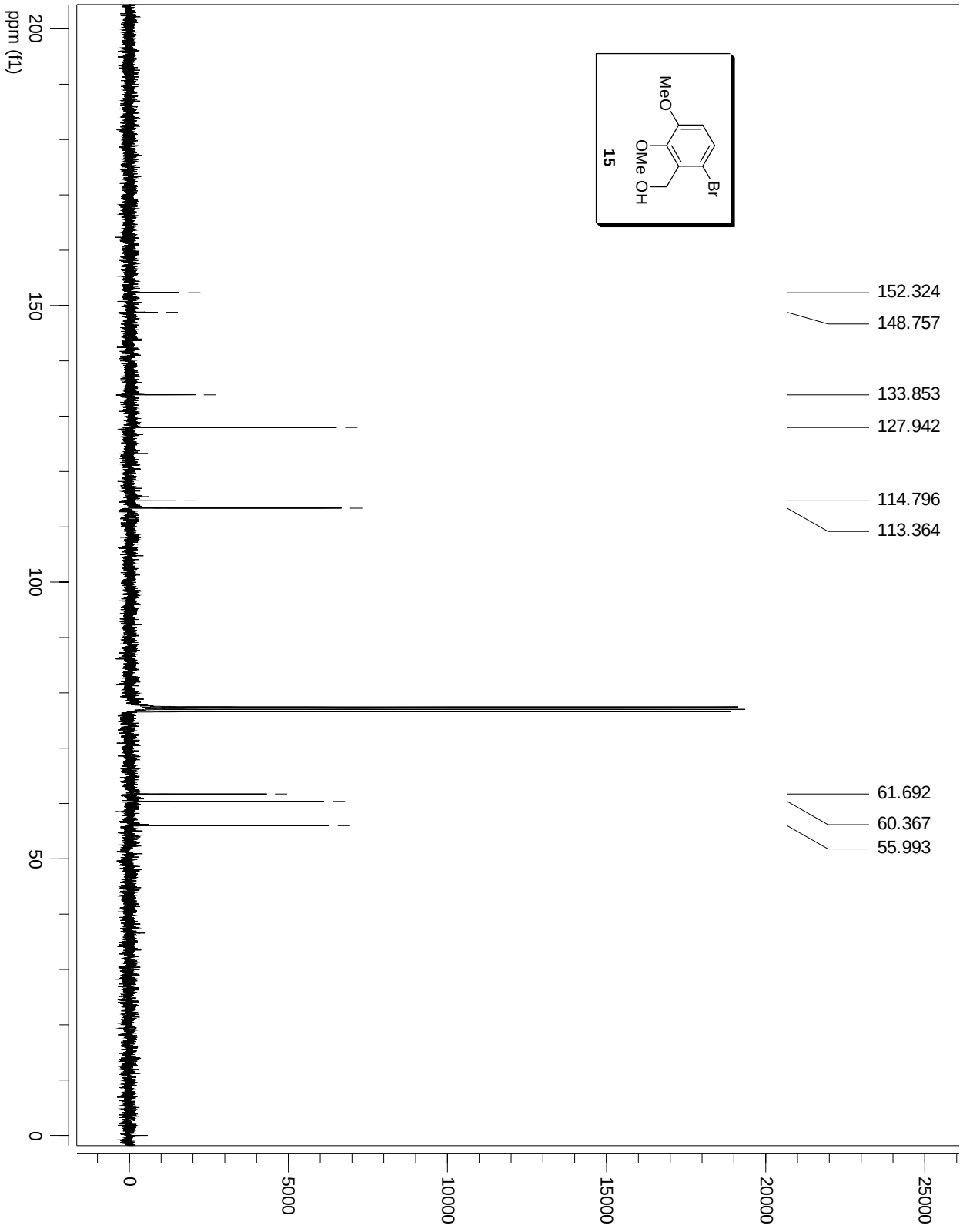
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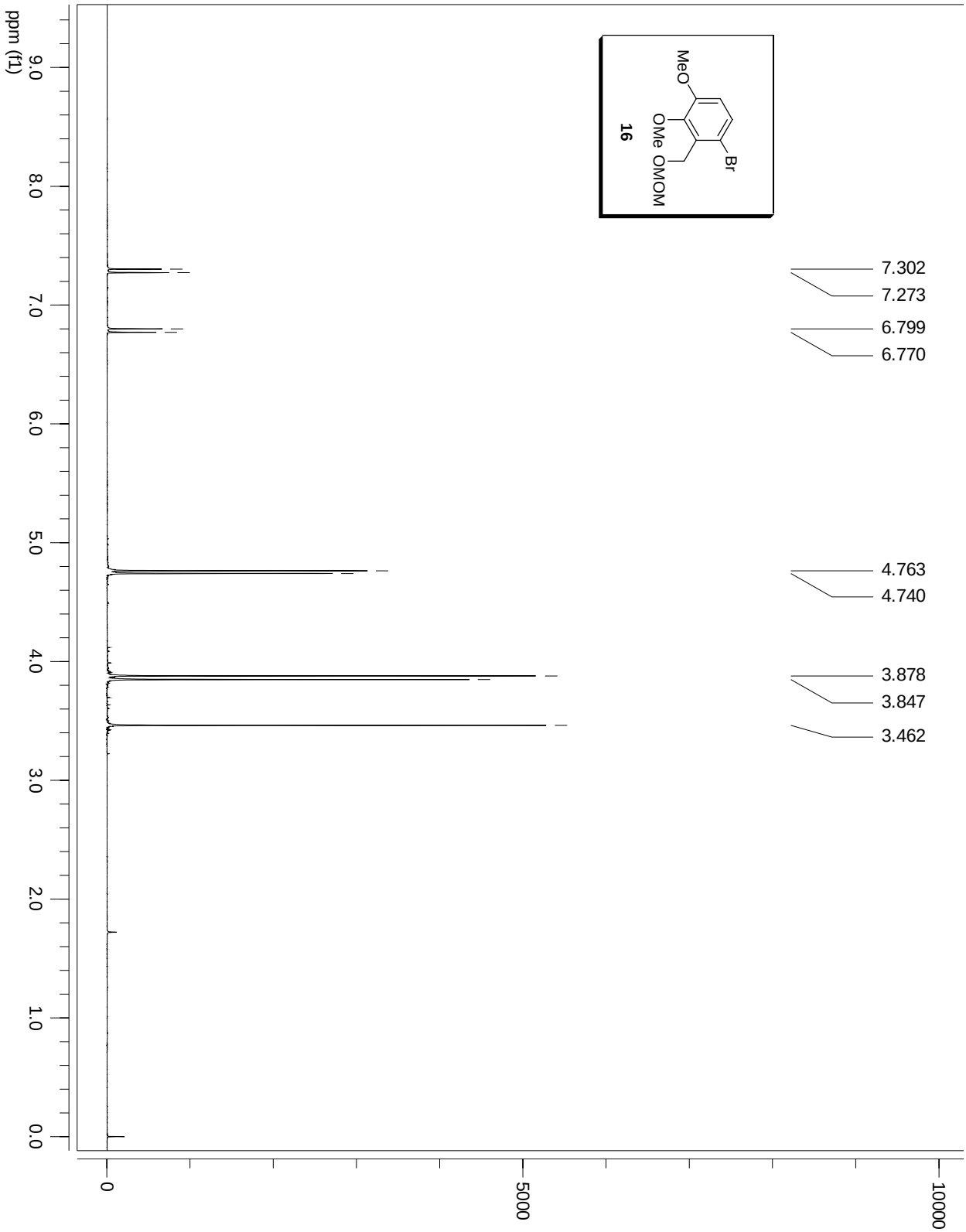
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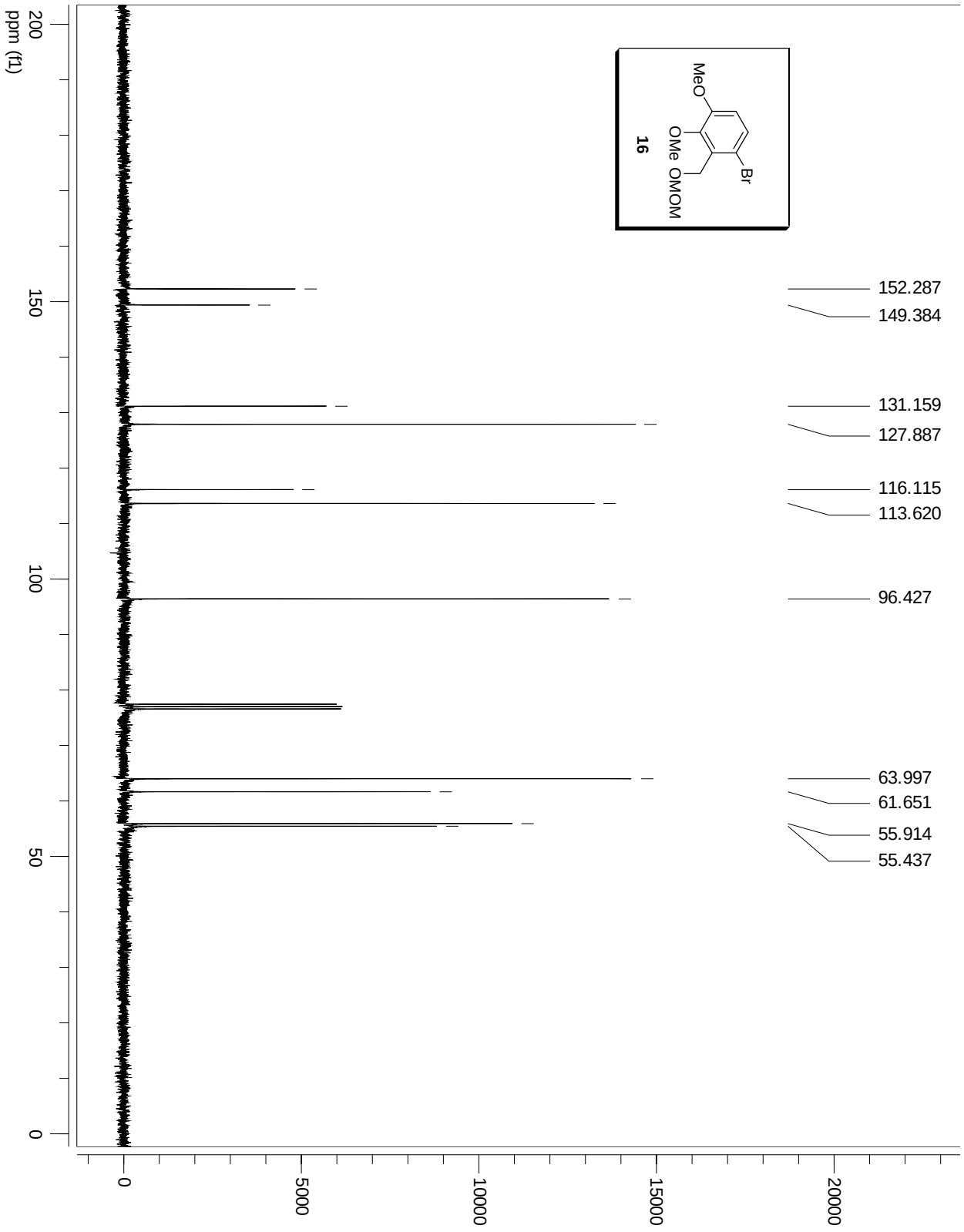
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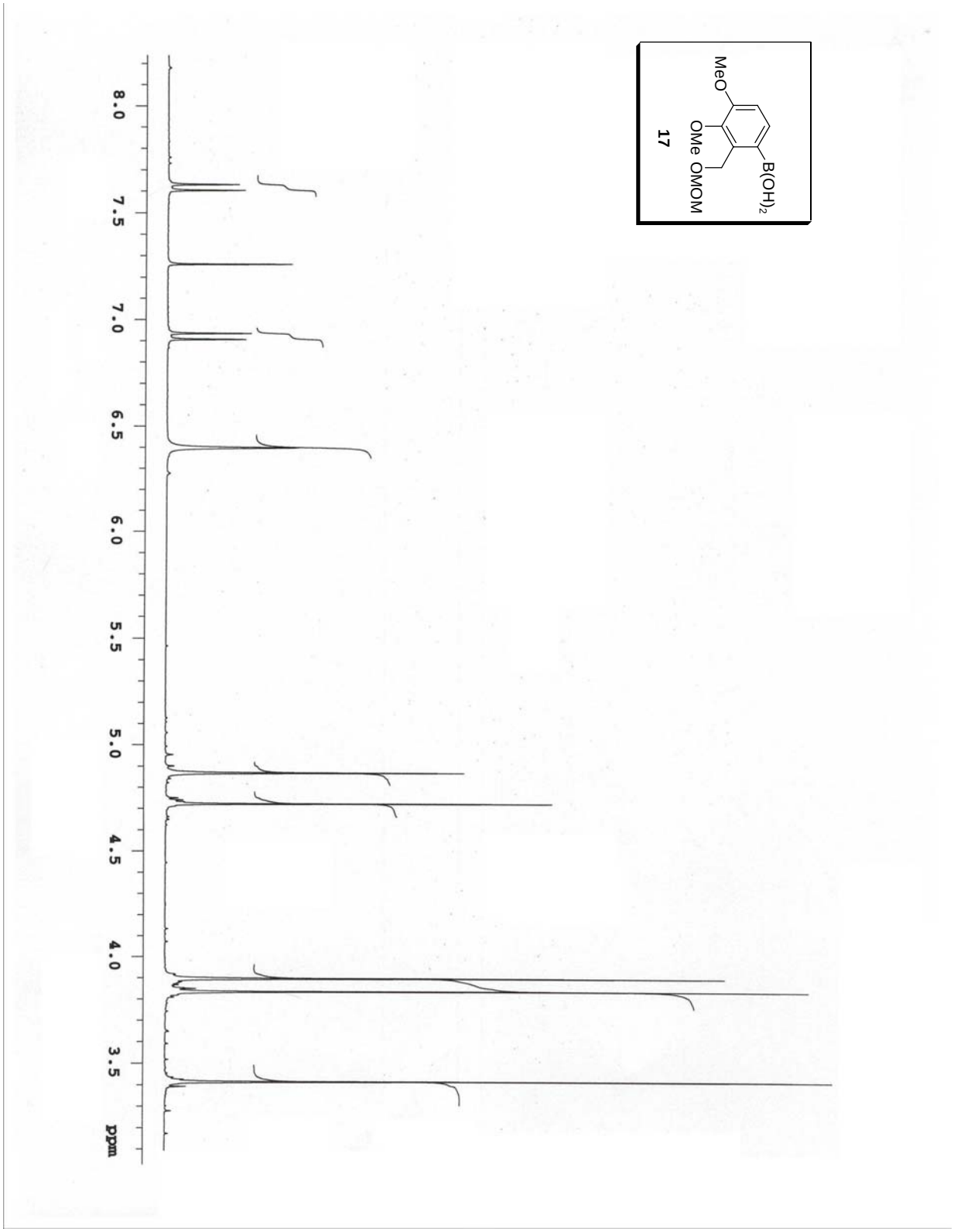
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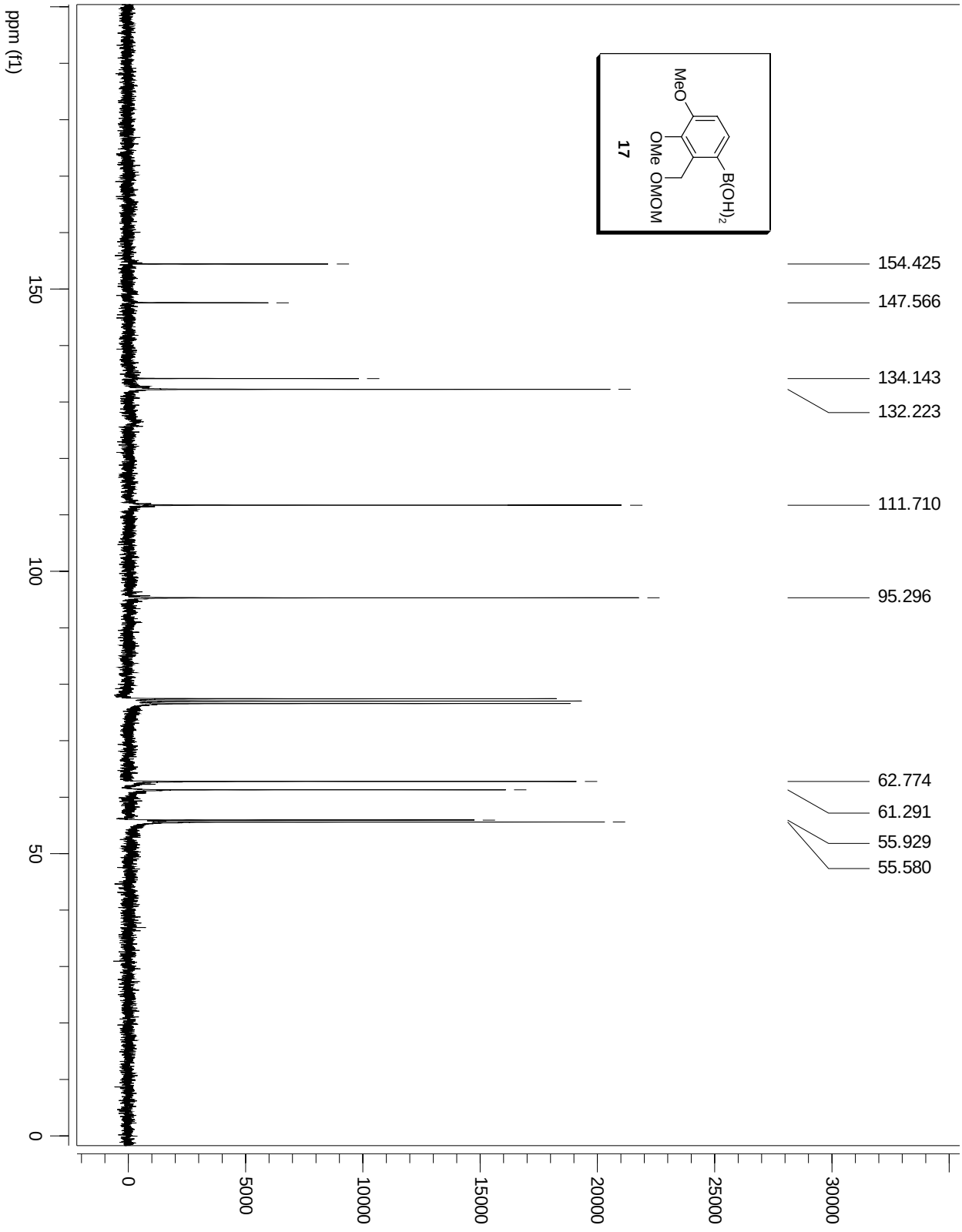
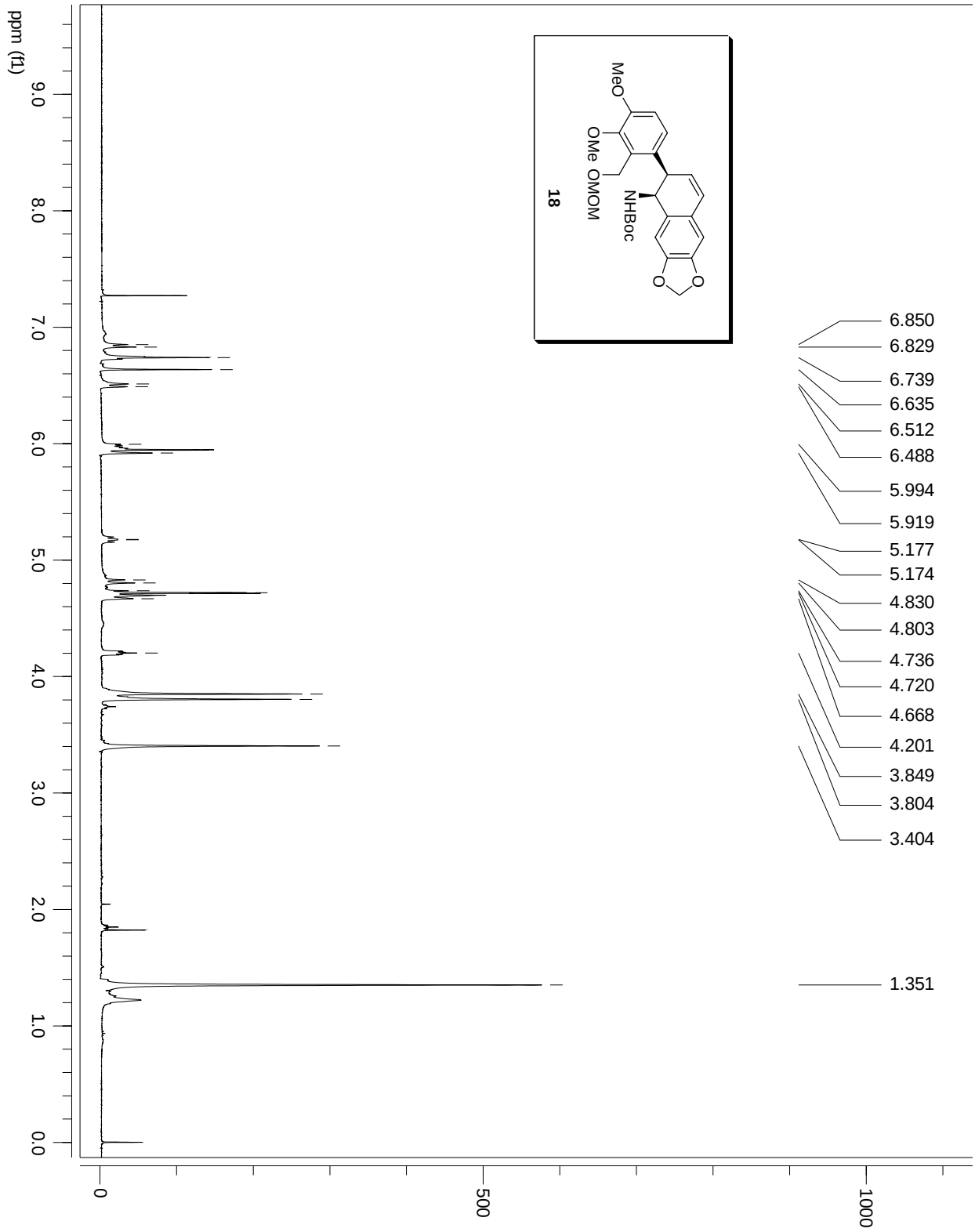
3.5
ppm


ppm (f1)
150
100
50
0
0
5000
10000
15000
20000
25000
30000
MeO
OMe OMOM
B(OH)2
17
154.425
147.566
134.143
132.223
111.710
95.296
62.774
61.291
55.929
55.580


18
MeO
OMe OMOM
NHBoc
O
O
O
6.850
6.829
6.739
6.635
6.512
6.488
5.994
5.919
5.177
5.174
4.830
4.803
4.736
4.720
4.668
4.201
3.849
3.804
3.404
1.351
ppm (f1)
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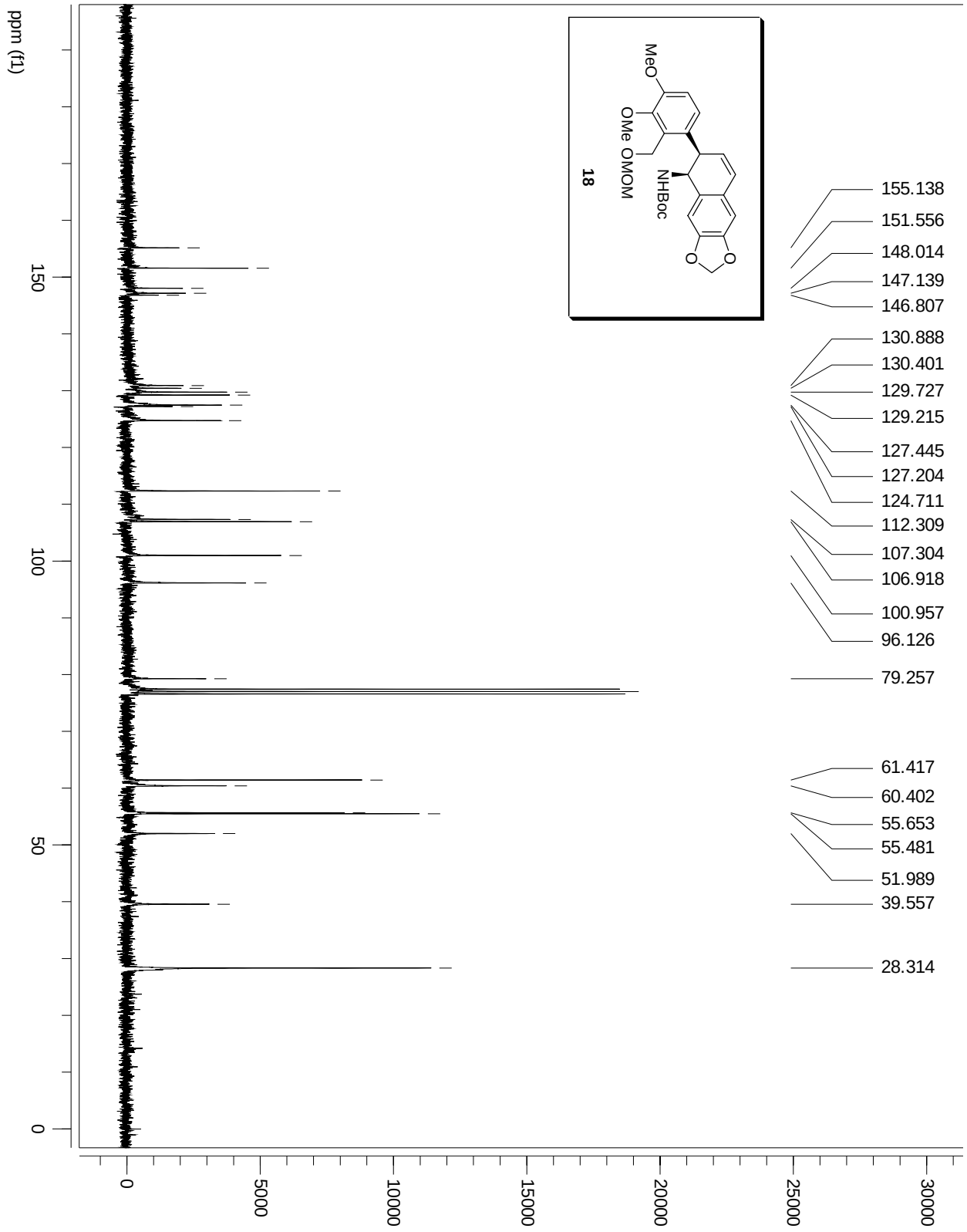
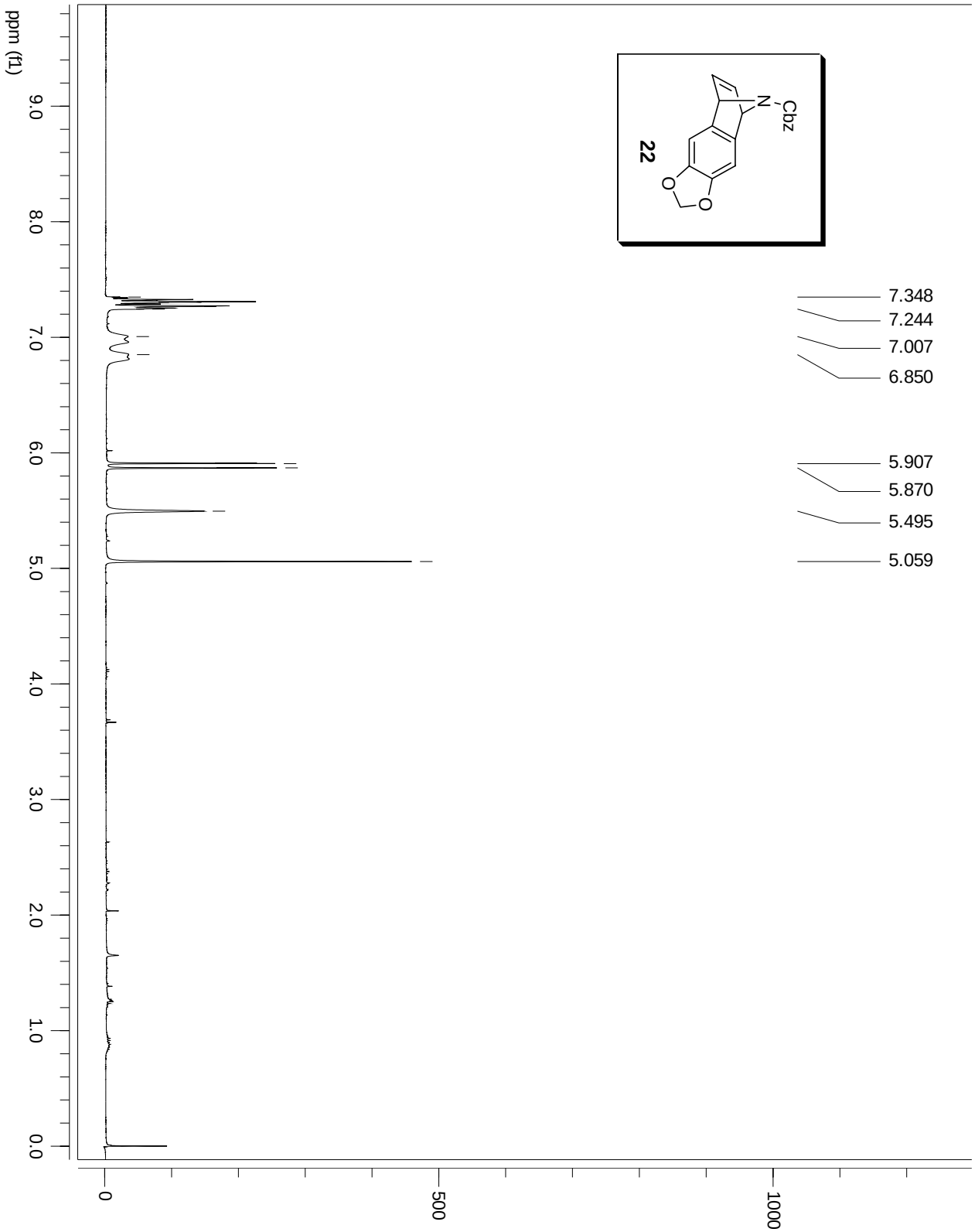
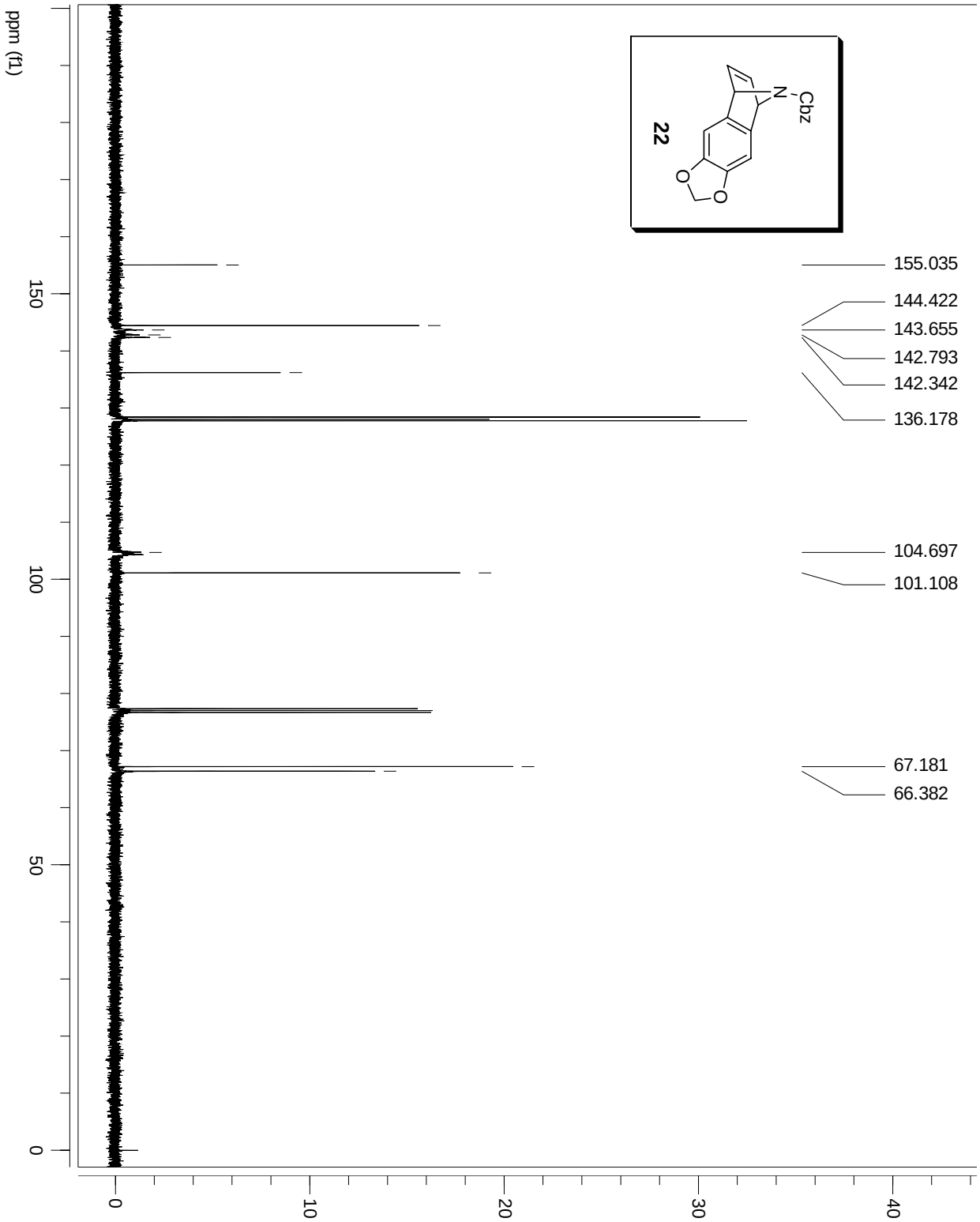
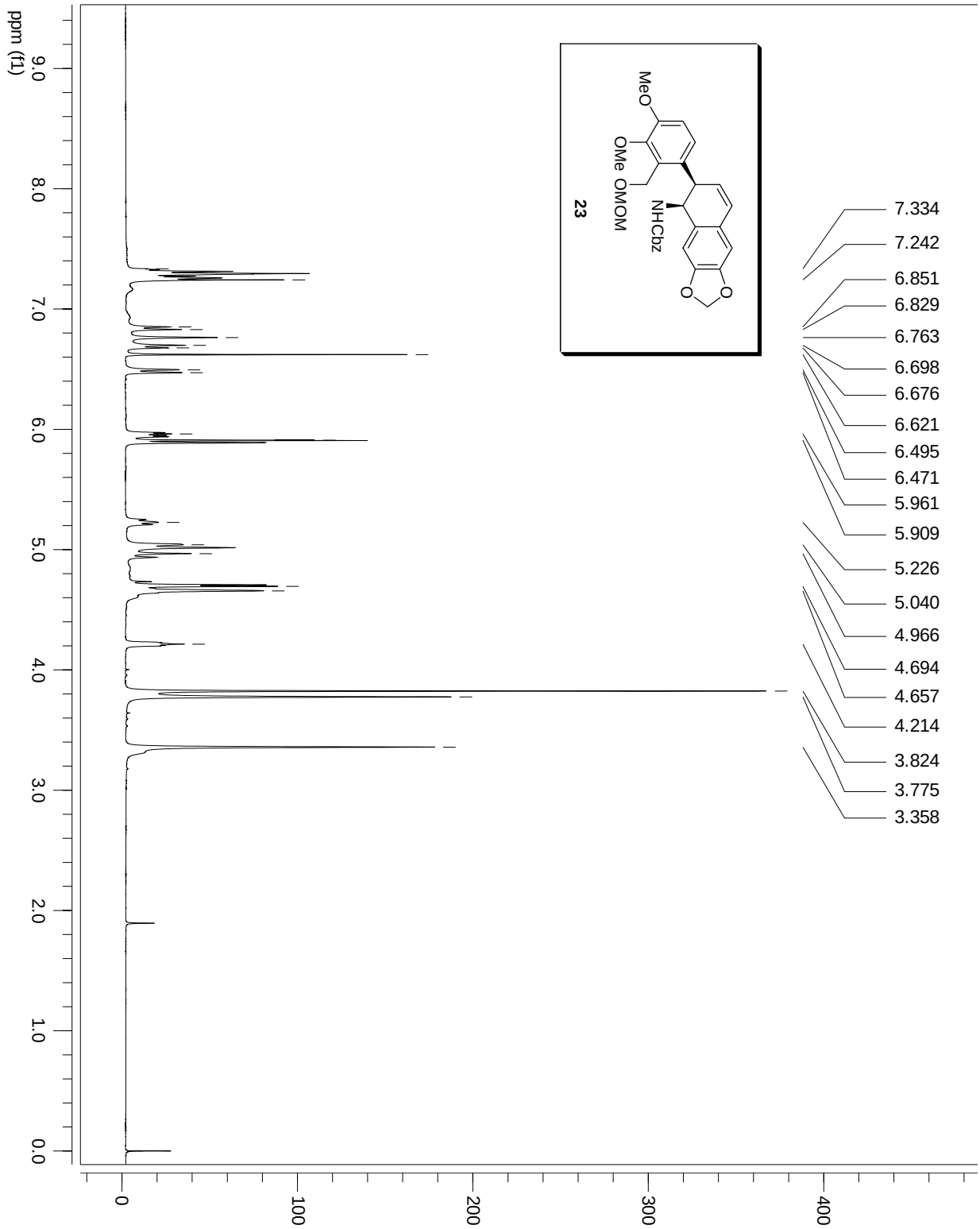
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
500
1000


ppm (f1)
0
50
100
150
0
5000
10000
15000
20000
25000
30000
MeO
OMe OMOM
NHBoc
O
O
O
18
155.138
151.556
148.014
147.139
146.807
130.888
130.401
129.727
129.215
127.445
127.204
124.711
112.309
107.304
106.918
100.957
96.126
79.257
61.417
60.402
55.653
55.481
51.989
39.557
28.314


22
N
Cbz
O
O
7.348
7.244
7.007
6.850
5.907
5.870
5.495
5.059
ppm (f1)
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
500
1000


22
N
Cbz
O
O
155.035
144.422
143.655
142.793
142.342
136.178
104.697
101.108
67.181
66.382
ppm (f1)
150
100
50
0
0
10
20
30
40


ppm (f1)
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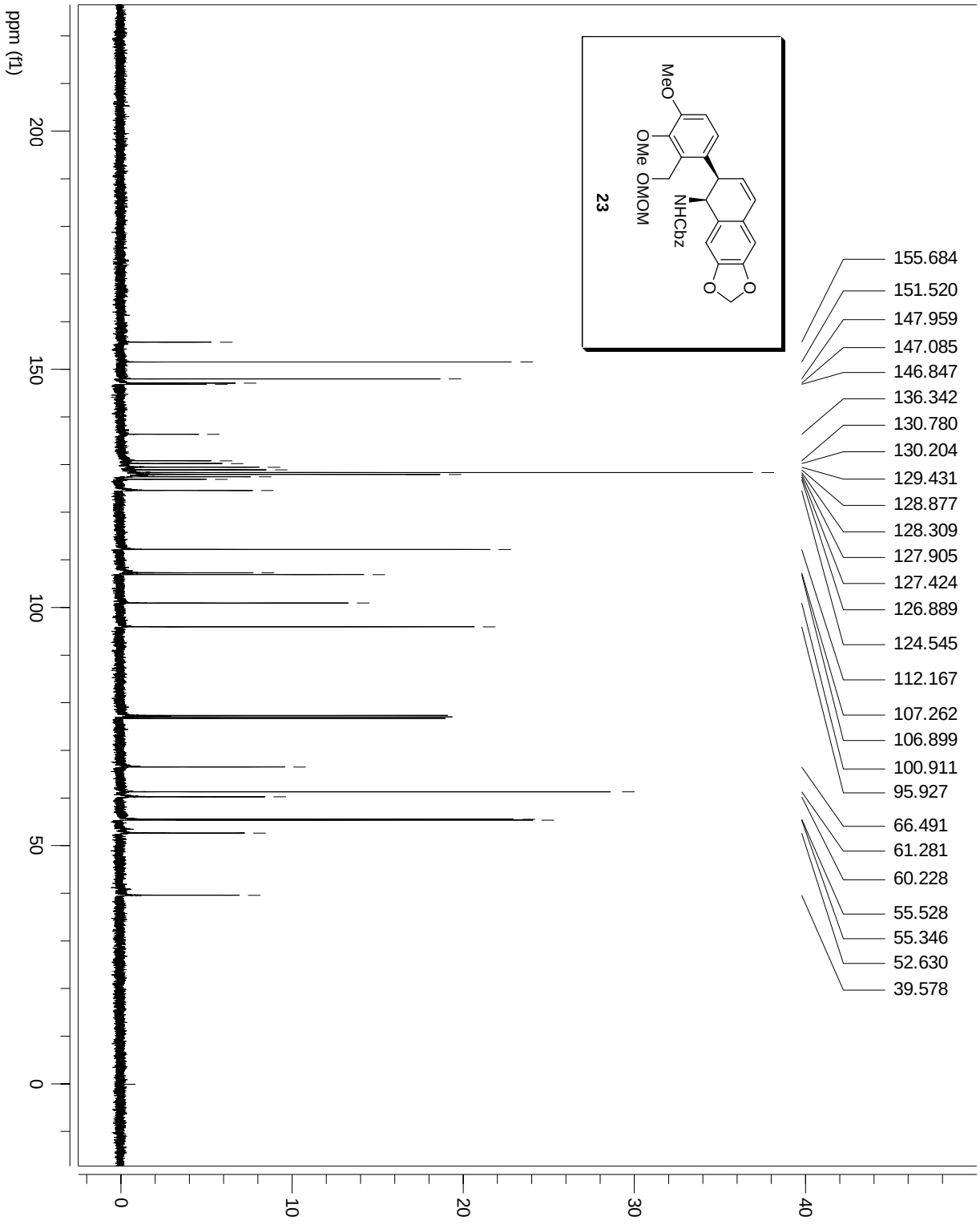
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
100
200
300
400
MeO
OMe OMOM
NHCbz
O
O
O
23
7.334
7.242
6.851
6.829
6.763
6.698
6.676
6.621
6.495
6.471
5.961
5.909
5.226
5.040
4.966
4.694
4.657
4.214
3.824
3.775
3.358


ppm (f1)
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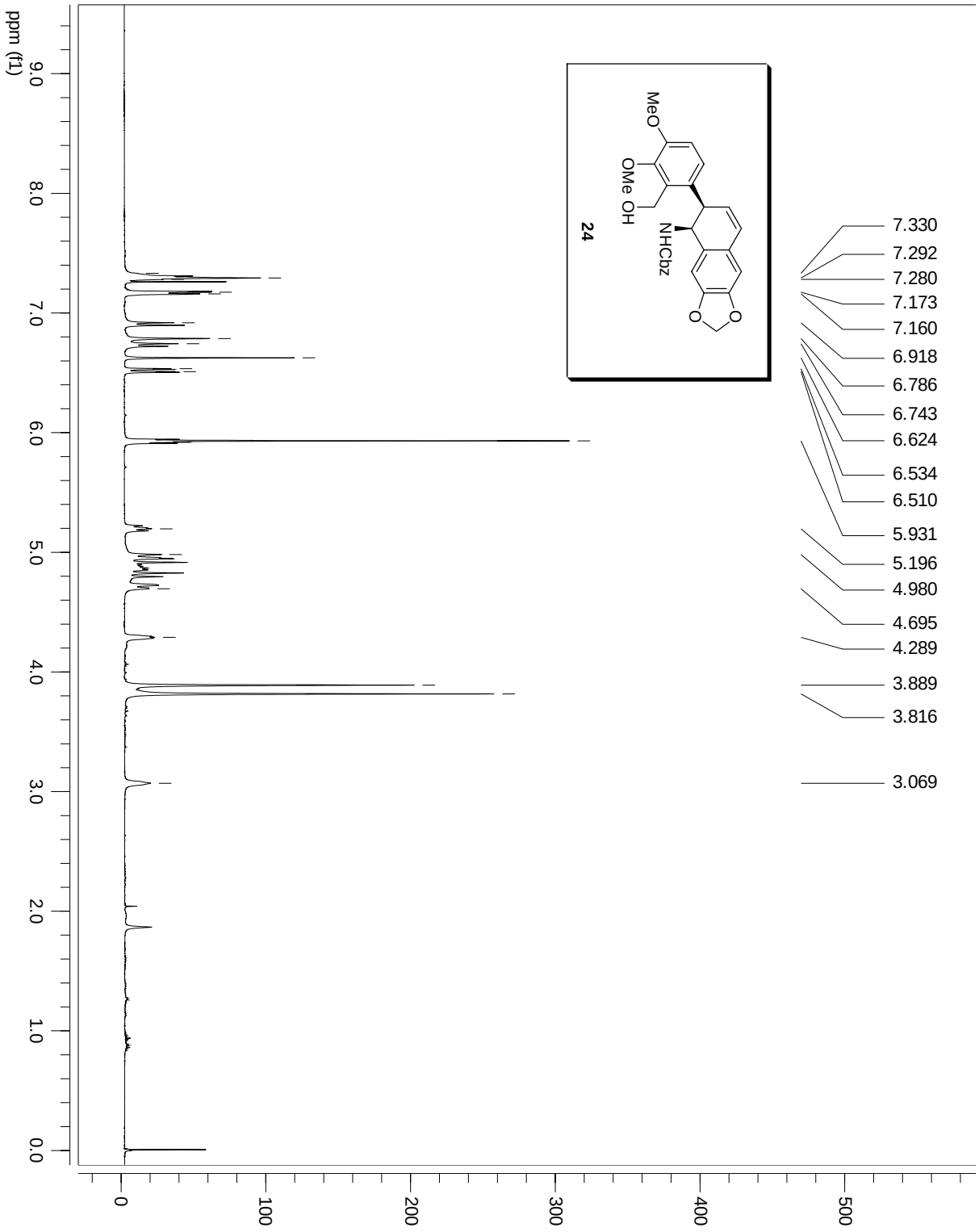
150
100
50
0
0
10
20
30
40
MeO
OMe OMOM
NHCbz
O
O
O
23
155.684
151.520
147.959
147.085
146.847
136.342
130.780
130.204
129.431
128.877
128.309
127.905
127.424
126.889
124.545
112.167
107.262
106.899
100.911
95.927
66.491
61.281
60.228
55.528
55.346
52.630
39.578


ppm (f1)
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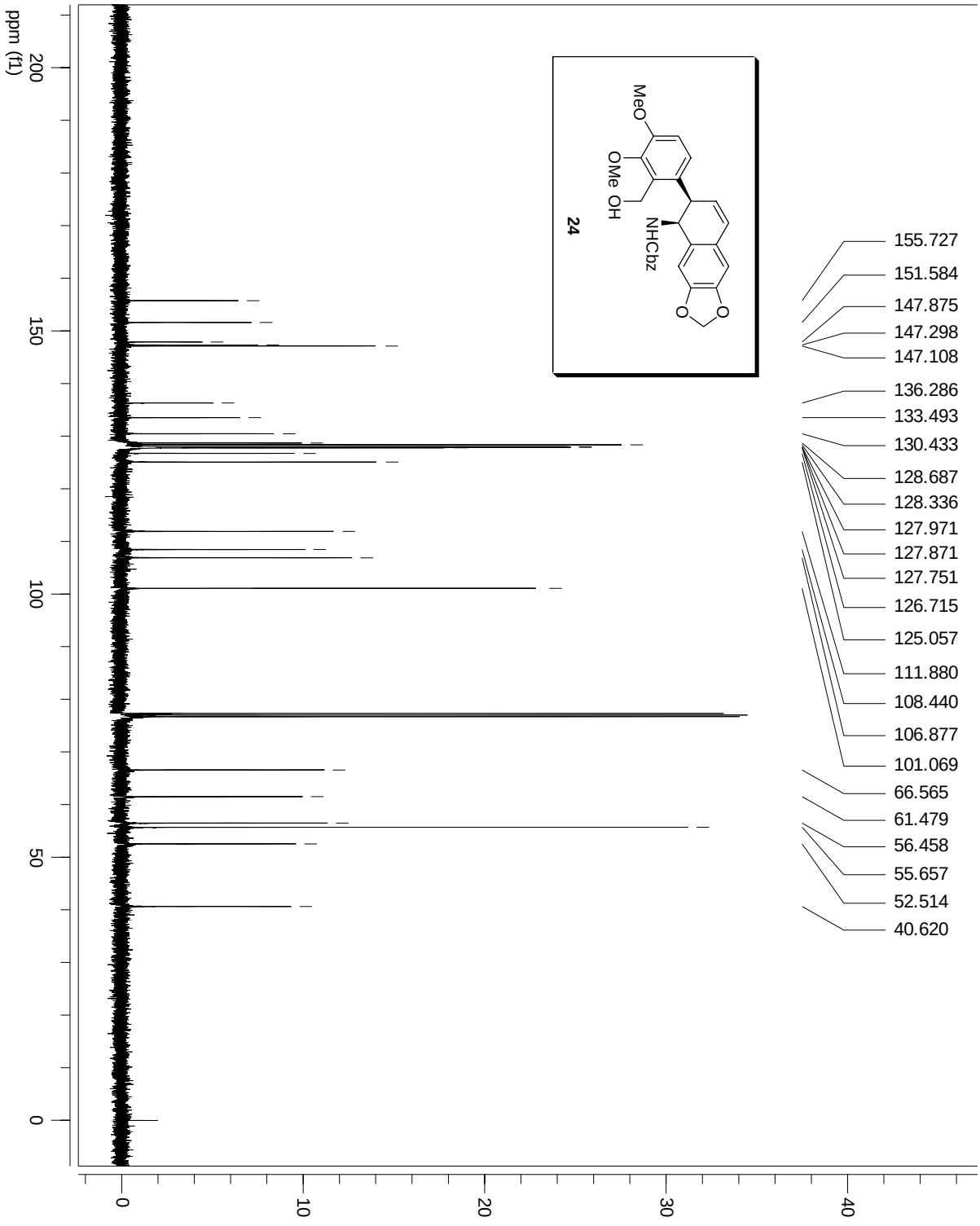
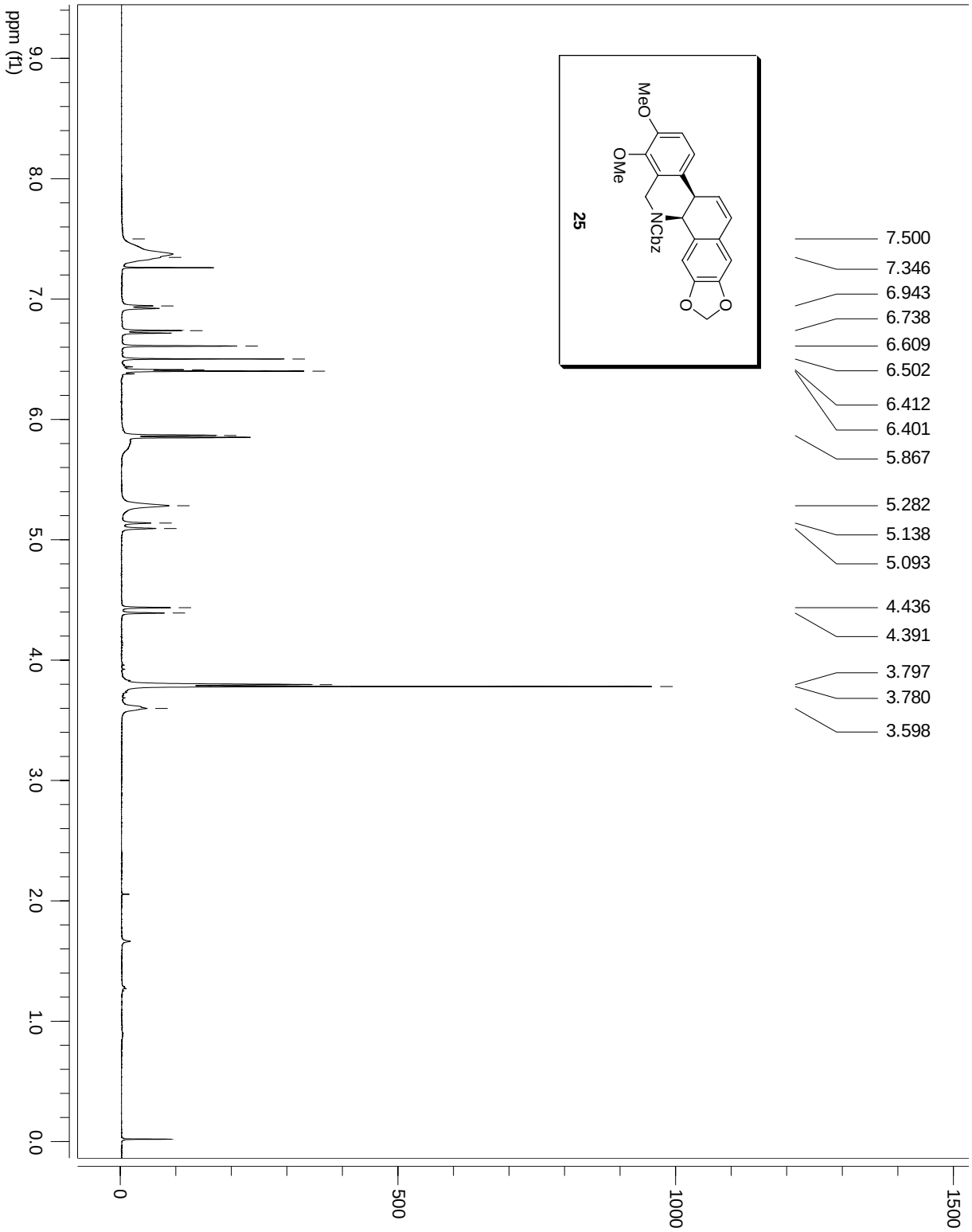
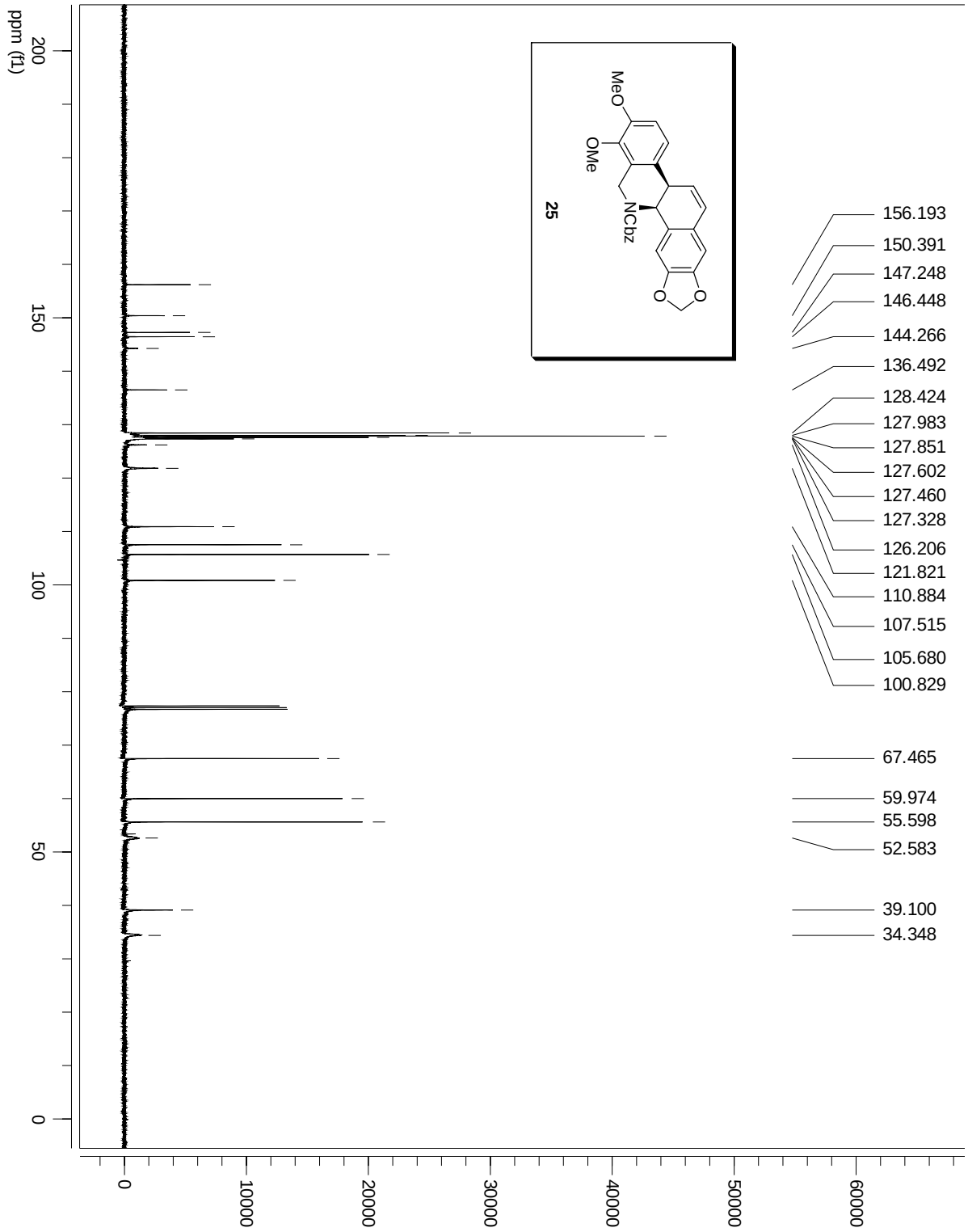
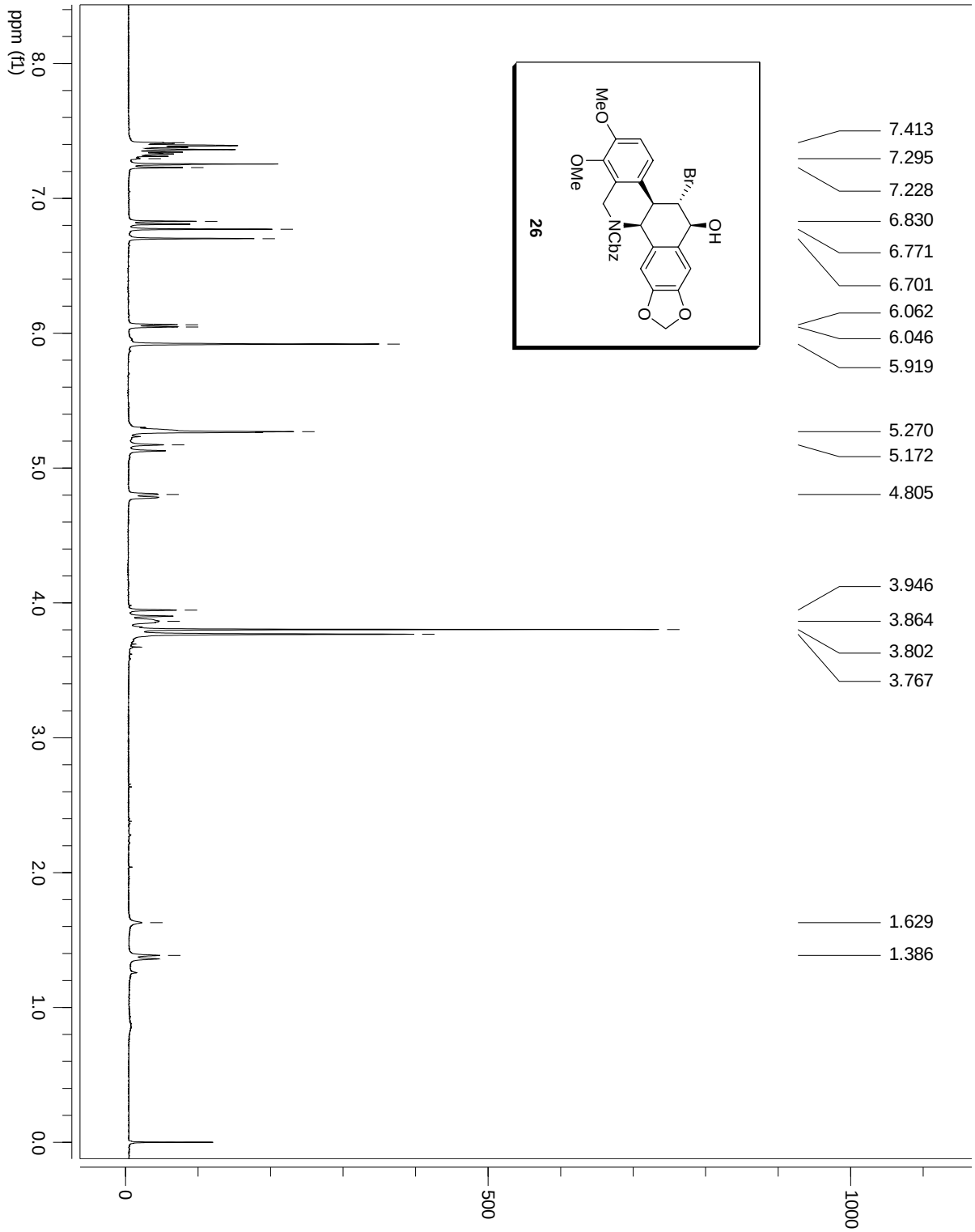
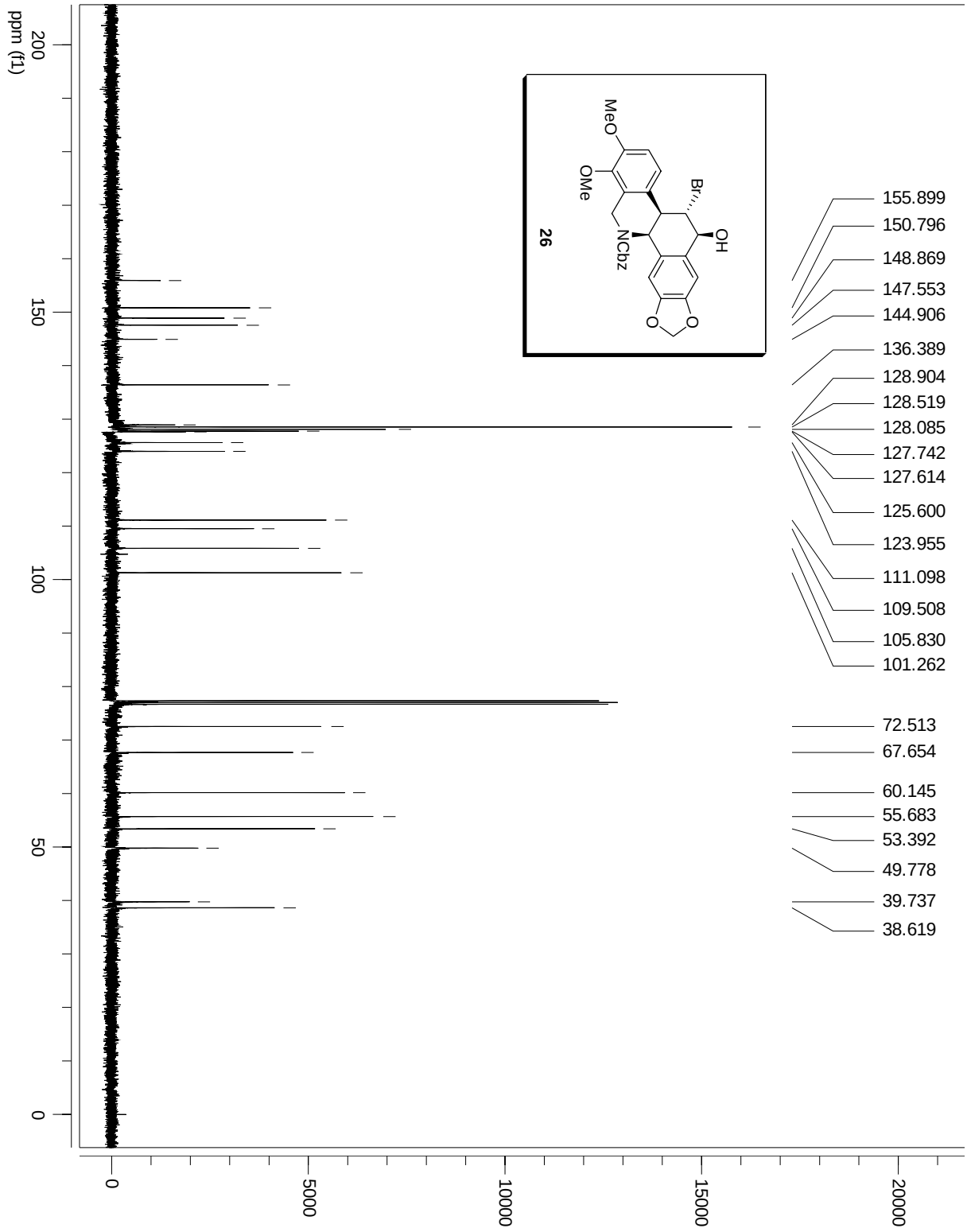
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
100
200
300
400
500
MeO
OMe OH
NHCbz
O
O
24
7.330
7.292
7.280
7.173
7.160
6.918
6.786
6.743
6.624
6.534
6.510
5.931
5.196
4.980
4.695
4.289
3.889
3.816
3.069

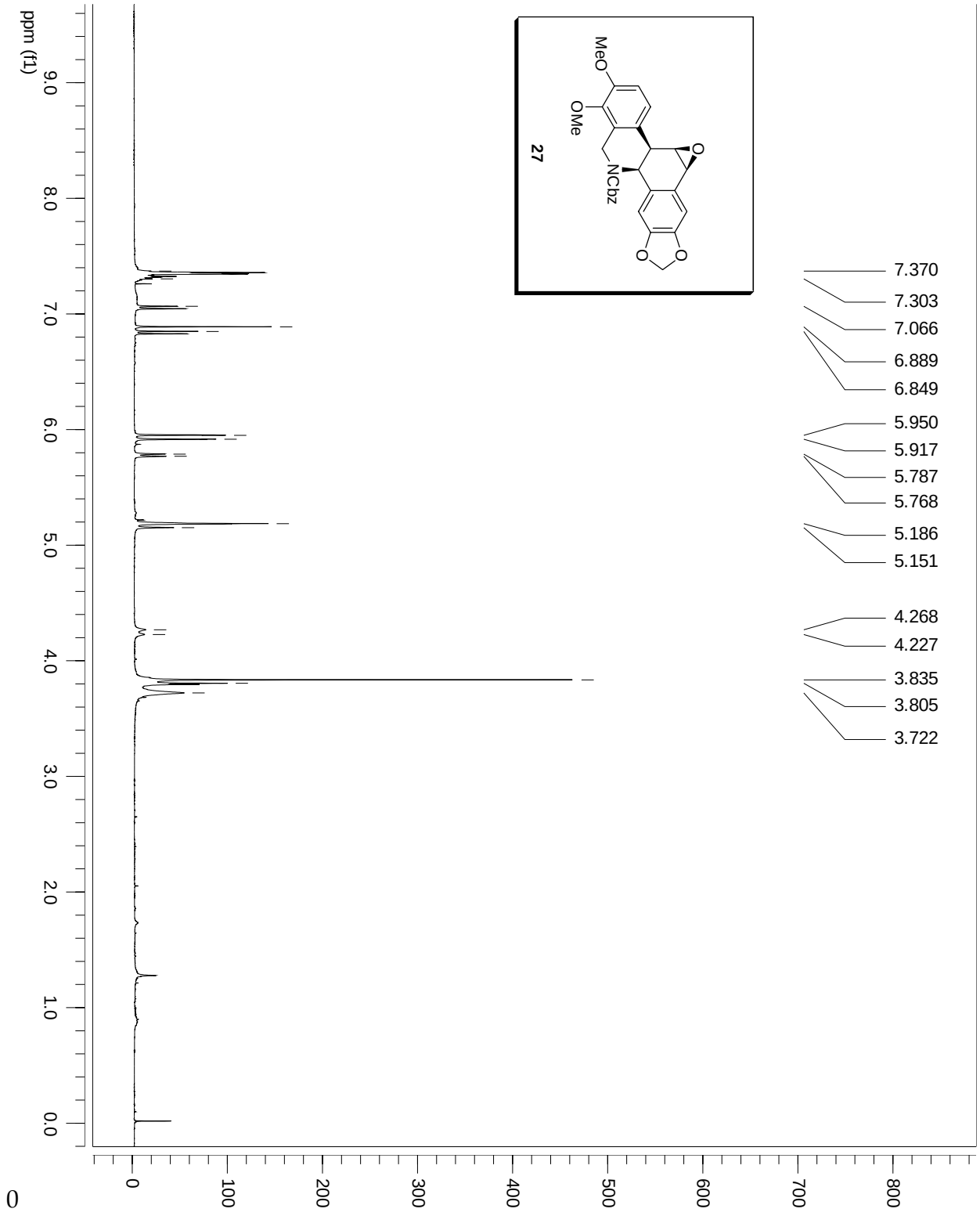

ppm (f1)
200
150
100
50
0
0
10
20
30
40
MeO
OMe OH
NHCbz
24
155.727
151.584
147.875
147.298
147.108
136.286
133.493
130.433
128.687
128.336
127.971
127.871
127.751
126.715
125.057
111.880
108.440
106.877
101.069
66.565
61.479
56.458
55.657
52.514
40.620

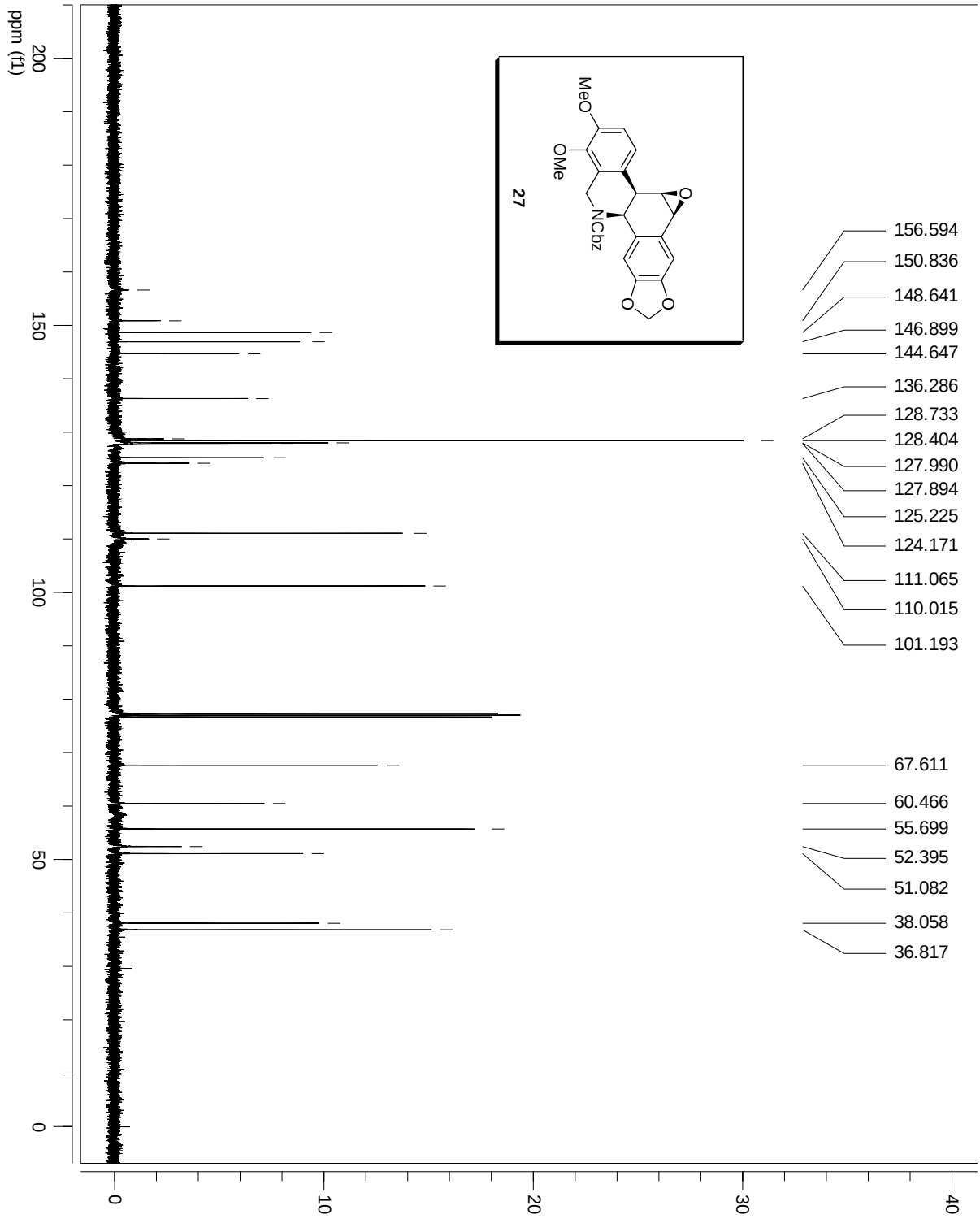

ppm (f1)
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
500
1000
1500
MeO
OMe
NCbz
O
O
25
7.500
7.346
6.943
6.738
6.609
6.502
6.412
6.401
5.867
5.282
5.138
5.093
4.436
4.391
3.797
3.780
3.598


ppm (f1)
200
150
100
50
0
0
10000
20000
30000
40000
50000
60000
MeO
OMe
NCbz
O
O
O
25
156.193
150.391
147.248
146.448
144.266
136.492
128.424
127.983
127.851
127.602
127.460
127.328
126.206
121.821
110.884
107.515
105.680
100.829
67.465
59.974
55.598
52.583
39.100
34.348

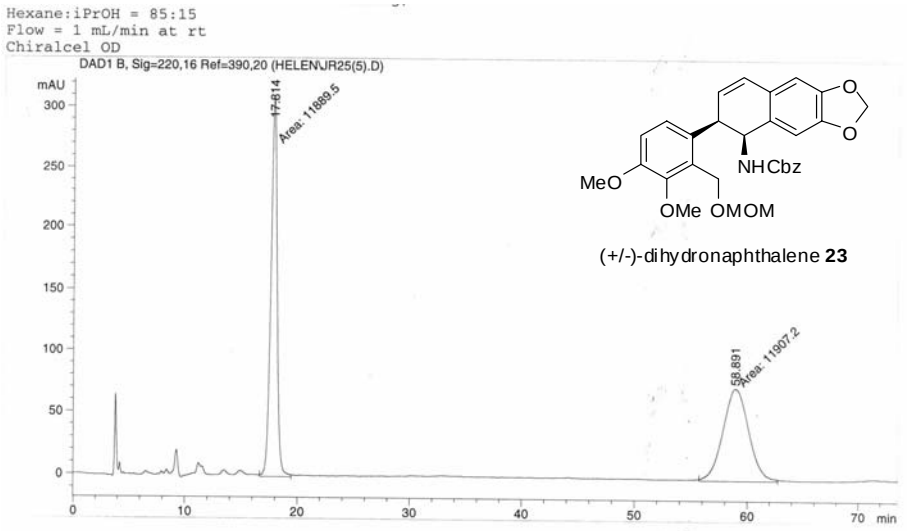

ppm (f1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
500
1000
MeO
OMe
Br
OH
NCbz
O
O
O
26
7.413
7.295
7.228
6.830
6.771
6.701
6.062
6.046
5.919
5.270
5.172
4.805
3.946
3.864
3.802
3.767
1.629
1.386


ppm (f1)
200
150
100
50
0
0
5000
10000
15000
20000
MeO
OMe
Br
OH
NCbz
O
O
O
26
155.899
150.796
148.869
147.553
144.906
136.389
128.904
128.519
128.085
127.742
127.614
125.600
123.955
111.098
109.508
105.830
101.262
72.513
67.654
60.145
55.683
53.392
49.778
39.737
38.619


MeO
OMe
NCbz
O
O
O
27
7.370
7.303
7.066
6.889
6.849
5.950
5.917
5.787
5.768
5.186
5.151
4.268
4.227
3.835
3.805
3.722
ppm (f1)
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
100
200
300
400
500
600
700
800

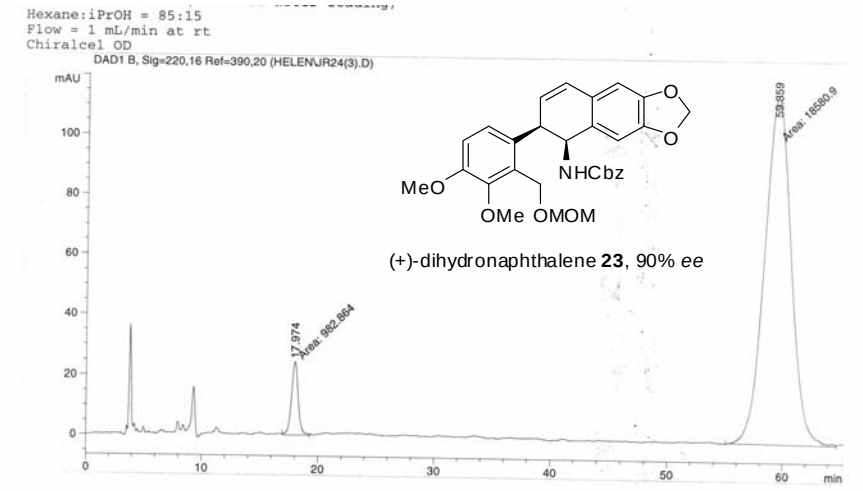

ppm (f1)
200
150
100
50
0
0
10
20
30
40
MeO
OMe
NCbz
O
O
O
27
156.594
150.836
148.641
146.899
144.647
136.286
128.733
128.404
127.990
127.894
125.225
124.171
111.065
110.015
101.193
67.611
60.466
55.699
52.395
51.082
38.058
36.817

# (V) HPLC traces for dihydronaphthalene 23 and homochelidonine 1



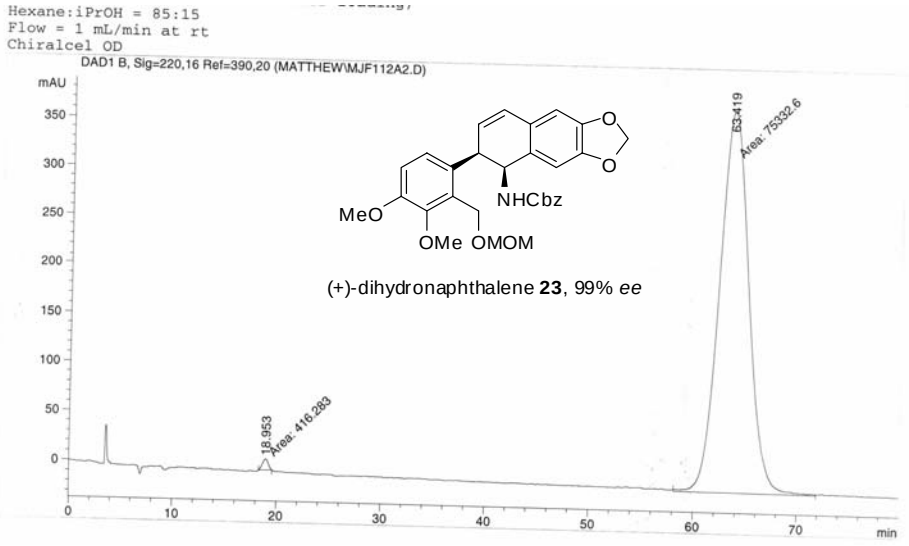

(+/-)-dihydronaphthalene **23**

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area % |
|---|---|---|---|---|---|---|
| 1 | 17.814 | MM | 0.6409 | 1.18895e4 | 309.17215 | 49.9627 |
| 2 | 58.891 | MM | 2.6756 | 1.19072e4 | 74.17054 | 50.0373 |
| Totals : | | | | 2.37967e4 | 383.34269 | |




(+)-dihydronaphthalene **23**, 90% *ee*

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area % |
|---|---|---|---|---|---|---|
| 1 | 17.974 | MM | 0.6729 | 982.86371 | 24.34346 | 5.0239 |
| 2 | 59.359 | MM | 2.6958 | 1.85809e4 | 114.87774 | 94.9761 |
| Totals : | | | | 1.95638e4 | 139.22120 | |

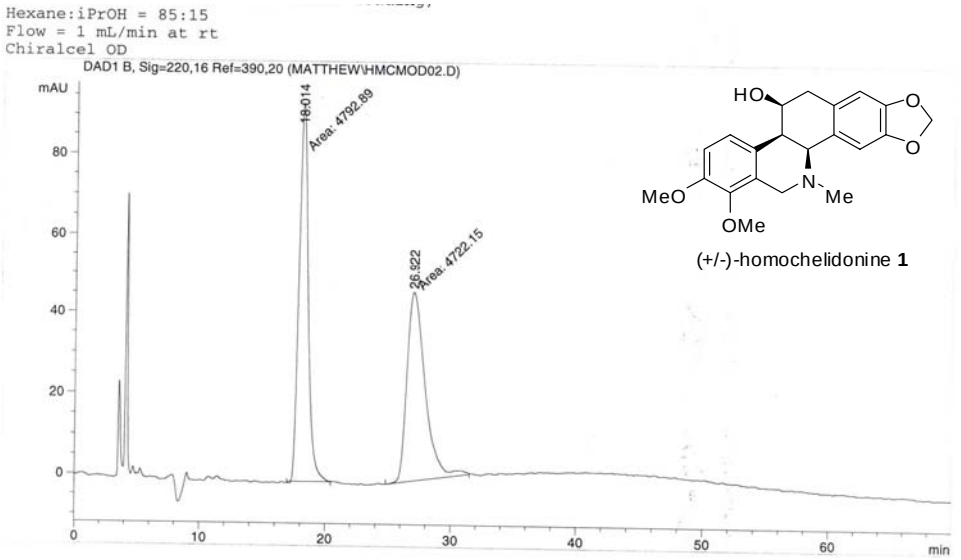






(+)-dihydronaphthalene **23**, 99% *ee*

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area % |
|---|---|---|---|---|---|---|
| 1 | 18.953 | MM | 0.6328 | 416.28345 | 10.96381 | 0.5496 |
| 2 | 63.419 | MM | 3.2515 | 7.53326e4 | 386.14160 | 99.4504 |





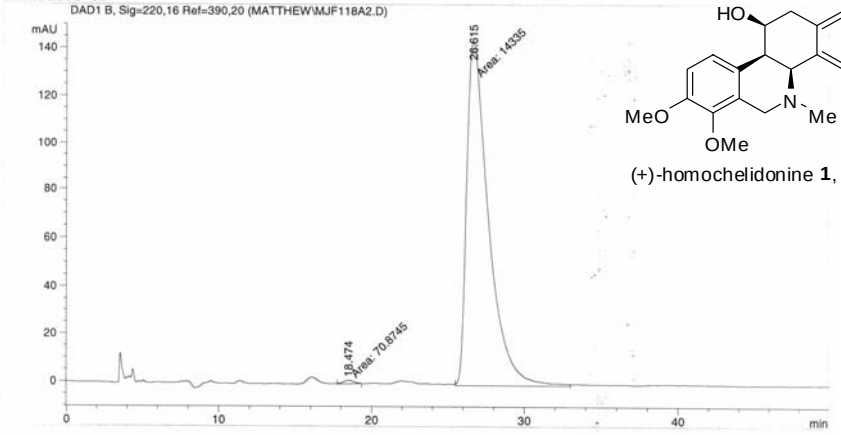


(+/-)-homochelidonine **1**

Signal 1: DAD1 B, Sig=220,16 Ref=390,20

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area % |
|---|---|---|---|---|---|---|
| 1 | 18.014 | MM | 0.8432 | 4792.89160 | 94.73311 | 50.3717 |
| 2 | 26.922 | MM | 1.6822 | 4722.15039 | 46.78588 | 49.6283 |
| Totals : | | | | 9515.04199 | 141.51899 | |

Hexane:iPrOH = 85:15
Flow = 1 mL/min at rt
Chiralcel OD







(+)-homochelidonine **1**, 99% *ee*

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area % |
|---|---|---|---|---|---|---|
| 1 | 18.474 | MM | 0.8053 | 70.87447 | 1.46689 | 0.4920 |
| 2 | 26.615 | MM | 1.6575 | 1.43350e4 | 144.14319 | 99.5080 |
| Totals : | | | | 1.44059e4 | 145.61007 | |