The *O*- Acylation of Ketone Enolates by Allyl 1*H*-imidazole-1-carboxylate Mediated with Boron Trifluoride Etherate---A Convenient Procedure for the Synthesis of Substituted Allyl Enol Carbonates

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Supplemental Information

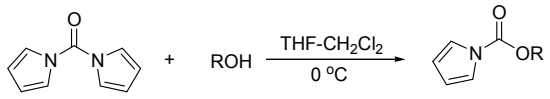
Experimental

General

All reactions were carried out under an atmosphere of nitrogen or argon in oven-dried glasswares with magnetic stirring, unless otherwise indicated. Solvents were from J. C. Meyer's Solvent Purification System. All other reagents were used as obtained unless otherwise noted.

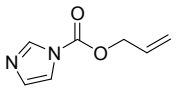
Flash Chromatography was performed with EM Science silica gel (0.040-0.063μm grade). Analytical thin-layer chromatography was performed with 0.25 mm coated commercial silica gel plates (E. Merck, DC-Plasrikfolien, kieselgel 60 F254). Melting points were obtained on a Thomas-Hoover apparatus in open capillary tubes and are uncorrected. Proton nuclear magnetic resonance (^{1}H-NMR) data were acquired on a Mercury 400 (400 MHz), a Varian 400 (400 MHz) or on a Varian Unity Inova-500 (500 MHz) spectrometer. Chemical shifts are reported in delta (δ) units, in parts per million (ppm) downfield from tetramethylsilane. Splitting patterns are designated as s, singlet; d, doublet; t, triplet; q, quartet; p, pentet, m, multiplet, br, broad. Carbon-13 nuclear magnetic resonance (^{13}C-NMR) data were acquired at 100 MHz on a Mercury 400 or at 125 MHz on a Varian Unity Inova 500 spectrometer. Chemical shifts are reported in ppm relative to the center line of a triplet at 77.1 ppm for chloroform-*d.* Infrared (IR) data were recorded as films on sodium chloride plates or a potassium bromide (KBr) pellets on a Perkin-Elmer Paragon 500 FT-IR spectrometer. Absorbance frequencies are reported in reciprocal centimeters (cm^{-1}). Elemental analyses (Anal.) were performed by M.-H.-W. Laboratories of Pheonix, AZ.

General Procedure for the Synthesis of 1[1]

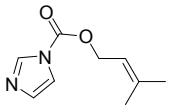


[1] Bertolini, G.; Pavich, G.; Vergani, B. *J. Org. Chem.* **1998**, *63*, 6031.

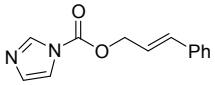
To a clean oven-dried 250 mL flask with a magnetic stirring bar was charged with 2.43 g 1,1'-carbonyldiimidazole (15 mmol) and 100 mL THF under nitrogen. The flask was cooled in a ice-water bath. A solution of allyl alcohol (10 mmol) in 30 mL methylene chloride was added slowly and stirred for 2 h. Most solvent was removed *in vacuo* by a rotaevaporator and the crude product was purified by silica gel column chromatography eluted with 1:1 ethyl acetate/petroleum ether.



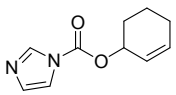
Allyl 1*H*-pyrrole-1-carboxylate (**1a**) :[1] Started from 580 mg allyl alcohol, 1.46 g **1a** was isolated (96%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.16 (bs, 2H), 7.45 (bs, 1H), 7.08 (bs, 1H), 6.02 (m, 1H), 5.47 (m, 1H), 5.39 (m, 1H), 4.90 (m, 2H).



3-Methylbut-2-en-1-yl 1*H*-pyrrole-1-carboxylate (**1b**):[2] Started from 1.72 g prenol, 2.90 g **1b** was isolated (80%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.14 (dd, *J* = 1.1, 1.1 Hz, 1H), 7.43 (dd, *J* = 1.7, 1.1 Hz, 1H), 7.06 (dd, *J* = 1.7, 1.0 Hz, 1H), 5.46 (m, 1H), 4.90 (m, 2H), 1.81 (m, 3H), 1.79 (m, 3H).

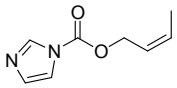


(2*E*)-3-phenylprop-2-en-1-yl 1*H*-pyrrole-1-carboxylate (**1c**) Started from 1.34 g cinnamyl alcohol (10 mmol), 2.08 g **1c** was isolated (91%) as a white solid. M.p. = 39-42 ^{o}C; R_f = 0.13 (30% ethyl acetate in petroleum ether); ^{1}H-NMR (400 MHz, $CDCl_3$): δ(ppm) = 8.22-8.13 (m, 1H), 7.52-7.27 (m, 6H), 7.08 (td, *J* = 1.7, 0.9 Hz, 1H), 6.79 (d, *J* = 15.8 Hz, 1H), 6.37 (dtd, *J* = 15.8, 6.8, 1.1 Hz, 1H), 5.06 (td, *J* = 6.8, 1.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 148.7, 137.2, 136.7, 135.6, 130.8, 128.8, 128.7, 126.9, 121.0, 117.2, 68.8. Anal. Calcd. for $C_{13}H_{12}N_2O_2$: C, 68.41; H, 5.30; N, 12.27; Found: C, 68.70; H, 5.45; N, 12.37.

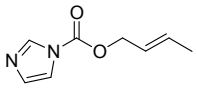


[2] Vatele, Jean-Michel; TETRAB; *Tetrahedron* **2004**, *60*, 4251.

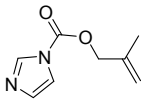
Cyclohex-2-en-1-yl 1*H*-imidazole-1-carboxylate (**1d**): started from 0.98 g 2-cyclohexen-1-ol (10 mmol), 1.87 g **1d** was isolated (97%) as a white solid. M.p. = 51-53 ^{o}C; R_f= 0.23 (30% ethyl acetate in petroleum ether); IR(neat): $\tilde{\nu}_{max}$ = 1755 cm^{-1}; ^{1}H-NMR (400 MHz, $CDCl_3$): 8.17-8.09 (m, 1H), 7.42 (m, 1H), 7.06 (dd, *J* = 1.6, 0.9 Hz, 1H), 6.14-6.03 (m, 1H), 5.88-5.78 (m, 1H), 5.52-5.40 (m, 1H), 2.25-1.65 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 137.1, 134.8, 130.5, 123.9, 117.2, 117.1, 72.8, 28.1, 24.8, 18.5. Anal. Calcd. for $C_{10}H_{12}N_2O_2$: C, 62.49; H, 6.29; N, 14.57; Found: C, 62.33; H, 5.98; N, 14.35.



(2*Z*)-But-2-en-1-yl 1*H*-imidazole-1-carboxylate (**1e**): started from 0.56 g (*Z*)-2-butene-1-ol (7.77 mmol), 1.1 g **1e** was isolated (97%) as a colorless oil. R_f= 0.17 (30% ethyl acetate in petroleum ether); IR(neat): $\tilde{\nu}_{max}$ = 1760 cm^{-1}; ^{1}H-NMR (400 MHz, $CDCl_3$): 8.11 (m, 1H), 7.45-7.35 (m, 1H), 7.08-7.00 (m, 1H), 5.91-5.77 (m, 1H), 5.69-5.55 (m, 1H), 4.98-4.89 (m, 2H), 1.75 (ddd, *J* = 4.6, 2.7, 1.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 148.8, 137.1, 132.0, 130.6, 122.4, 117.1, 63.5, 13.3. HRMS (EI): M^+ calcd. for $C_8H_{10}N_2O_2$ 166.0742, found 166.0741.

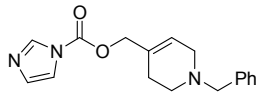


(2*E*)-But-2-en-1-yl 1*H*-imidazole-1-carboxylate (**1f**): started from 1.44 g (*E*)-2-butene-1-ol (20 mmol), 3.16 g **1f** was isolated (95%) as a colorless oil. R_f= 0.17 (30% ethyl acetate in petroleum ether); IR(neat): $\tilde{\nu}_{max}$ = 1760 cm^{-1}; ^{1}H-NMR (400 MHz, $CDCl_3$): 8.13 (s, 1H), 7.47-7.38 (m, 1H), 7.15-7.00 (m, 1H), 6.04-5.83 (m, 1H), 5.77-5.61 (m, 1H), 4.90-4.73 (m, 2H), 1.77 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 148.6, 137.1, 134.1, 130.5, 123.4, 117.1, 68.8, 17.8. Anal. Calcd. for $C_8H_{10}N_2O_2$: C, 57.82; H, 6.07; N, 16.86; Found: C, 57.72; H, 5.99; N, 17.06.



2-Methylprop-2-en-1-yl 1*H*-imidazole-1-carboxylate (**1g**): Started from 1.44 g (*E*)-2-methyl-prop-2-en-1-ol (20 mmol), 3.23 g **1g** was isolated (97%) as a white solid. M.p. = 36-38 ^{o}C; R_f= 0.18 (30% ethyl acetate in petroleum ether); IR(neat): $\tilde{\nu}_{max}$ = 1761 cm^{-1}; ^{1}H-NMR (400 MHz, $CDCl_3$): 8.17-8.11 (m, 1H), 7.43 (dd, *J* = 3.5, 2.1 Hz, 1H), 7.07 (dd, *J* = 1.5, 0.9 Hz, 1H), 5.08 (m,

1H), 5.04 (m, 1H), 4.80 (s, 2H), 1.82 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 148.5, 138.3, 137.1, 130.7, 117.1, 114.9, 71.2, 19.4. HRMS (EI): M^+ calcd. for $C_8H_{10}N_2O_2$ 166.0742, found 166.0737.

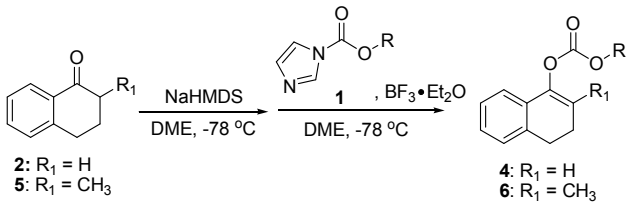


(1-Benzyl-1,2,3,6-tetrahydropyridin-4-yl)methyl 1*H*-imidazole-1-carboxylate (**1h**): Started from 2.03 g alcohol (10 mmol), 3.15 g **1h** was isolated (97%) as a slight brown solid. R_f= 0.18 (ethyl acetate); IR(neat): $\tilde{\nu}_{max}$ = 1770 cm^{-1}; ^{1}H-NMR (400 MHz, $CDCl_3$): 8.13 (s, 1H), 7.42 (t, *J* = 1.4 Hz, 1H), 7.38-7.20 (m, 5H), 7.11-7.02 (m, 1H), 5.88-5.79 (m, 1H), 4.81 (d, *J* = 0.6 Hz, 2H), 3.60 (s, 2H), 3.04 (td, *J* = 5.8, 2.1 Hz, 2H), 2.62 (t, *J* = 5.7 Hz, 2H), 2.22 (dt, *J* = 5.6, 3.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 148.7, 138.0, 137.1, 130.7, 130.1, 129.2, 128.3, 127.2, 126.3, 117.1, 71.2, 62.6, 52.4, 49.2, 26.9. Anal. Calcd. for $C_{17}H_{19}N_3O_2$: C, 68.67; H, 6.44; N, 14.13; Found: 69.00; H, 6.70; N, 14.38.

General Procedure A:

A clean oven-dried 50 mL flask was charged 460 mg sodium bis(trimethylsilyl)amide (2.4 mmol) under nitrogen. The flask was cooled to -78 ^{o}C in a dry-ice-acetone bath and was added 5 mL 1,2-dimethoxyethane (DME) through a syringe. The flask was taken out from the bath and let it warm up until the solid completely dissolved and then it was cooled to -78 ^{o}C. To the solution was added 317 mg 1-indanone (2.4 mmol) in 2 mL DME. The solution was stirred for 30 min before it was transferred into the solution of 384.4 mg **1d** (2 mmol). After 10 min the cooling bath was removed and the reaction was allowed to warm to room temperature and stirred at room temperature for 1 h. One portion of 10 mL saturated aqueous ammonium chloride was poured into the reaction mixture followed by 10 mL diethyl ether. The aqueous layer was extracted with 10 mL diethyl ether. The combined organic solution was washed with 10 mL saturated brine and dried over $MgSO_4$. After concentration the crude product was isolated with silica gel column chromatography.

General Procedure B and the Preparation of **4** and **6**



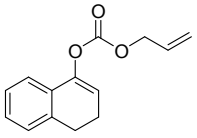
To the solution of 460 mg NaHMDS in 5 mL DME at -78 °C was added 292 mg 1-tetralone (2 mmol) in 2 mL DME. The solution was stirred at -78 °C for 30 min. Meanwhile, another clean oven-dried 50 mL flask was charged with 365 mg **1a** (2.0 mmol) and 5 mL DME. The solution cooled to -78 °C was added 0.30 mL boron trifluoride etherate (2.4 mmol). The solution was transferred into the solution of enolate through a cannula under nitrogen and stirred for 30 min. One portion of 10 mL saturated aqueous ammonium chloride was poured into the reaction mixture followed by 10 mL diethyl ether. The mixture was taken out from the bath and allowed to warm to ambient temperature. The organic layer was separated and the aqueous layer was extracted once with 10 mL diethyl ether. The organic layer was combined and dried over magnesium sulfate. After filtration and concentration *in vacuo* the crude material was purified by silica gel column chromatography eluted with 10% diethyl ether in petroleum ether to give 392 mg **4a** (85%).

Procedure C:

Follow the details in Procedure B but potassium *tert*-butoxide was used as base and THF as solvent.

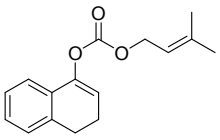
Procedure D:

Follow Procedure C but 1 equiv. 18-C-6 to the base was added to the enolate solution.

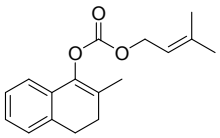


Allyl 3,4-dihydronaphthalen-1-yl carbonate (**4a**): colorless oil; R_f = 0.39 (Diethyl ether/petroleum ether 1:9); IR (film): 1760 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 1H NMR (400 MHz, *Solvent*) δ ppm 7.18 (m, 4H), 6.00 (tdd, *J* = 17.1, 10.4, 5.8 Hz, 1H), 5.82 (t, *J* = 4.7 Hz, 1H), 5.43 (qd, *J* = 17.2, 1.4 Hz, 1H), 5.33 (qd, *J* = 10.4, 1.2 Hz, 1H), 4.72 (td, *J* = 5.8, 1.3 Hz, 2H), 2.88 (t, *J* = 8.0 Hz, 2H), 2.52-2.38 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 153.4, 146.1,

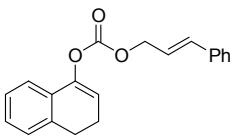
136.4, 131.2, 130.1, 128.1, 127.6, 126.5, 120.6, 119.4, 115.2, 69.1, 27.4, 22.0. Anal. Calcd. for $C_{14}H_{14}O_3$: C, 73.03; H, 6.13; Found: C, 73.58; H, 6.43.



3,4-Dihydronaphthalen-1-yl 3-methylbut-2-en-1-yl carbonate (**4b**) Started from 292 mg 1-tetralone (2.0 mmol) and 432.4 mg **1b** (2.4 mmol), 457 mg colorless oil was obtained after purification by silica gel chromatography (88%). R_f = 0.29 (Diethyl ether/petroleum ether 1:9); IR (film): 1760 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ = 7.22-7.12 (m, 4H), 5.81 (t, *J* = 4.6 Hz, 1H), 5.44 (m, 1H), 4.72 (d, *J* = 7.4 Hz, 2H), 2.86 (t, *J* = 8.1 Hz, 2H), 2.45 (m, 2H), 1.79 (s, broad, 3H), 1.75 (s, broad, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 153.7, 146.2, 140.7, 136.4, 130.3, 128.1, 127.6, 126.5, 120.7, 117.8, 115.2, 65.4, 27.4, 25.9, 22.1, 18.2. Anal. Calcd. for $C_{16}H_{18}O_3$: C, 74.39; H, 7.02; Found: C, 74.53; H, 7.03.

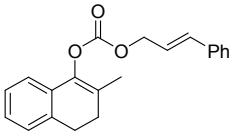


3-Methylbut-2-en-1-yl 2-methyl-3,4-dihydronaphthalen-1-yl carbonate (**6b**) Started from 384.5 mg 2-methyl-1-tetralone (2.4 mmol) and 360.4 mg **1b** (2.0 mmol), 568 mg colorless oil was obtained after purification by silica gel chromatography (100%). R_f = 0.29 (Diethyl ether/petroleum ether 1:9); IR (film): 1760 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.20-7.05 (m, 4H), 5.49-5.37 (m, 1H), 4.72 (d, *J* = 7.3 Hz, 2H), 2.85 (t, *J* = 8.1 Hz, 2H), 2.39 (ddd, *J* = 7.8, 6.3, 1.0 Hz, 2H), 1.82 (s, 3H), 1.79 (d, *J* = 0.8 Hz, 3H), 1.74 (d, *J* = 1.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 153.4, 140.7, 140.5, 135.3, 130.9, 127.3, 127.0, 126.4, 124.4, 120.0, 117.8, 65.3, 28.9, 27.4, 25.8, 18.1, 16.6. Anal. Calcd. for $C_{17}H_{20}O_3$: C, 74.97; H, 7.40; Found: C, 75.10; H, 7.18.

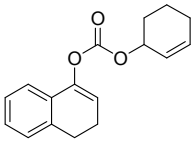


3,4-Dihydronaphthalen-1-yl (2*E*)-3-phenylprop-2-en-1-yl carbonate (**4c**) Started from 292 mg 1-tetralone (2.0 mmol) and 480 mg **1c** (2.4 mmol), 467 mg colorless oil was obtained after purification by silica gel chromatography (76%). R_f = 0.20 (Diethyl ether/petroleum ether 1:9);

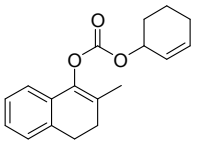
IR (film): 1770, 1748 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.45-7.10 (m, 9H), 6.73 (d, *J* = 16.0 Hz, 1H), 6.35 (dt, *J* = 15.9, 6.6 Hz, 1H), 5.82 (d, *J* = 4.7 Hz, 1H), 4.87 (dd, *J* = 6.4, 1.4 Hz, 2H), 2.87 (t, *J* = 8.1 Hz, 2H), 2.45 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 153.5, 146.2, 136.4, 136.0, 135.4, 130.2, 128.7, 128.4, 128.2, 127.7, 126.8, 126.6, 122.1, 115.3, 69.1, 27.4, 22.0. Anal. Calcd. for $C_{20}H_{18}O_3$: C, 78.41; H, 5.92; Found: C, 78.64; H, 5.70.



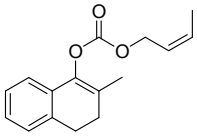
2-Methyl-3,4-dihydronaphthalen-1-yl (2*E*)-3-phenylprop-2-en-1-yl carbonate (**6c**) Started from 320 mg 2-methyl-1-tetralone (2.0 mmol) and 548 mg **1c** (2.4 mmol), 484 mg white crystals were obtained after purification by silica gel chromatography (76%). M.p. = 68-70 ^oC; R_f = 0.22 (Diethyl ether/petroleum ether 1:9); IR (film): 1769, 1749 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.45-7.04 (m, 9H), 6.74 (d, *J* = 15.9 Hz, 1H), 6.35 (dt, *J* = 15.9, 6.4 Hz, 1H), 4.88 (dd, *J* = 6.4, 1.4 Hz, 2H), 2.87 (t, *J* = 8.0 Hz, 2H), 2.41 (t, *J* = 8.0 Hz, 2H), 1.84 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 153.3, 140.8, 136.1, 135.3, 135.2, 130.9, 128.7, 128.4, 127.4, 127.2, 126.8, 126.5, 124.6, 122.2, 120.1, 69.1, 29.0, 27.5, 16.7. Anal. Calcd. for $C_{21}H_{20}O_3$: C, 78.73; H, 6.29; Found: C, 79.00; H, 6.15.



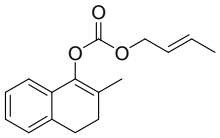
Cyclohex-2-en-1-yl 3,4-dihydronaphthalen-1-yl carbonate (**4d**) Started from 292 mg 1-tetralone (2.0 mmol) and 404 mg **1d** (2.1 mmol), 435 mg colorless oil was obtained after purification by silica gel chromatography (80%). R_f = 0.29 (Diethyl ether/petroleum ether 1:9); IR (film): 1756 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.21-7.09 (m, 4H), 6.03 (m, 1H), 5.83 (m, 1H), 5.80 (t, *J* = 4.7 Hz, 1H), 5.18 (m, 1H), 2.86 (t, *J* = 8.0 Hz, 2H), 2.45 (m, 2H), 2.20-1.60 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 153.4, 146.2, 136.4, 133.8, 130.4, 128.0, 127.6, 126.5, 124.7, 120.7, 115.2, 72.7, 28.2, 27.5, 24.9, 22.0, 18.6. Anal. Calcd. for $C_{17}H_{18}O_3$: C, 75.53; H, 6.71; Found: C, 75.60; H, 6.79.



Cyclohex-2-en-1-yl 2-methyl-3,4-dihydronaphthalen-1-yl carbonate (**6d**) Started from 320 mg 2-methyl-1-tetralone (2.0 mmol) and 461 mg **1d** (2.4 mmol), 536 mg colorless oil was obtained after purification by silica gel chromatography (94%). R_f = 0.22 (Diethyl ether/petroleum ether 1:9); IR (film): 1760 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.20-7.07 (m, 4H), 6.02 (m, 1H), 5.83 (m, 1H), 5.18 (m, 1H), 2.86 (t, *J* = 8.1 Hz, 2H), 2.39 (dt, *J* = 8.1, 1.0 Hz, 2H), 2.20-1.60 (m, 6H), 1.83 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 153.1, 140.8, 135.3, 133.7, 131.1, 127.3, 126.5, 124.8, 124.3, 120.1, 104.8, 72.7, 29.0, 28.2, 27.5, 24.9, 18.6, 16.7, Anal. Calcd. for $C_{18}H_{20}O_3$: C, 76.03; H, 7.09; Found: C, 75.93; H, 6.86.

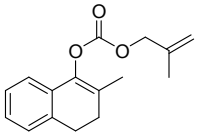


(2*Z*)-But-2-en-1-yl 2-methyl-3,4-dihydronaphthalen-1-yl carbonate (**6e**) Started from 385 mg 2-methyl-1-tetralone (2.4 mmol) and 479 mg **1e**(2.88 mmol), 550 mg colorless oil was obtained after purification by silica gel chromatography (89%). R_f = 0.33 (Diethyl ether/petroleum ether 1:9); IR (film): 1758 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.20-7.07 (m, 4H), 5.80 (m, 1H), 5.65 (m, 1H), 4.79 (m, 2H), 2.86 (t, *J* = 8.0 Hz, 2H), 2.39 (dt, *J* = 8.1, 1.0 Hz, 2H), 1.82 (t, *J* = 1.0 Hz, 3H), 1.74 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 153.4, 140.8, 135.3, 131.0, 130.8, 127.3, 127.1, 126.5, 124.5, 123.5, 120.1, 64.1, 29.0, 27.5, 16.6, 13.3. Anal. Calcd. for $C_{16}H_{18}O_3$: C, 74.39; H, 7.02; Found: C, 74.46; H, 6.86.

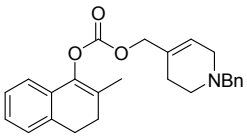


(2*E*)-But-2-en-1-yl 2-methyl-3,4-dihydronaphthalen-1-yl carbonate (**6f**) Started from 385 mg 2-methyl-1-tetralone (2.4 mmol) and 399 mg **1f** (2.4 mmol), 595 mg colorless oil was obtained after purification by silica gel chromatography (96%). R_f = 0.32 (Diethyl ether/petroleum ether 1:9); IR (film): 1760 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.20-7.07 (m, 4H), 5.89 (m, 1H), 5.67 (m, 1H), 4.64 (m, 2H), 2.86 (t, *J* = 8.0 Hz, 2H), 2.39 (dt, *J* = 8.1, 1.0 Hz, 2H), 1.82 (t, *J* = 1.0 Hz, 3H), 1.75 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 153.3, 140.7, 135.3, 132.8, 131.0,

127.3, 127.1, 126.5, 124.44, 124.36, 120.1, 69.3, 29.0, 27.5, 17.9, 16.7. Anal. Calcd. for $C_{16}H_{18}O_3$: C, 74.39; H, 7.02; Found: C, 74.40; H, 6.89.



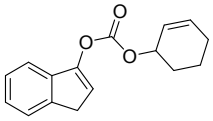
2-Methyl-3,4-dihydronaphthalen-1-yl 2-methylprop-2-en-1-yl carbonate (**6g**) Started from 320 mg 2-methyl-1-tetralone (2.0 mmol) and 399 mg **1g** (2.4 mmol), 472 mg colorless oil was obtained after purification by silica gel chromatography (92%). R_f = 0.34 (Diethyl ether/petroleum ether 1:9); IR (film): 1760 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.20-7.07 (m, 4H), 5.08 (m, 1H), 4.99 (m, 1H), 4.64 (m, 2H), 2.86 (t, *J* = 8.0 Hz, 2H), 2.39 (dt, *J* = 8.1, 1.0 Hz, 2H), 1.83 (t, *J* = 1.0 Hz, 3H), 1.80 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 153.2, 140.7, 139.3, 135.3, 130.9, 127.4, 127.1, 126.5, 124.5, 120.0, 113.7, 71.7, 29.0, 27.5, 19.4, 16.6. Anal. Calcd. for $C_{16}H_{18}O_3$: C, 74.39; H, 7.02; Found: C, 74.50; H, 7.12.



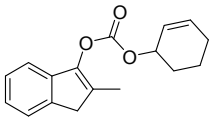
(1-Benzyl-1,2,3,6-tetrahydropyridin-4-yl)methyl 2-methyl-3,4-dihydronaphthalen-1-yl carbonate (**6h**) Started from 320 mg 2-methyl-1-tetralone (2.0 mmol), 714 mg **1h** (2.4 mmol) and 0.60 mL $BF_3{\cdot}Et_2O$ (4.8 mmol), 594 mg colorless oil was obtained after purification by silica gel chromatography (76%). R_f = 0.25 (30% EtOAc in petroleum ether); IR (film): 1766 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.40-7.05 (m, 9H), 5.78 (m, 1H), 4.62 (s, 2H), 3.59 (s, 2H), 3.01 (m, 2H), 2.84 (t, *J* = 8.0 Hz, 2H), 2.61 (t, *J* = 5.7 Hz, 2H), 2.38 (td, *J* = 8.0, 1.7 Hz, 2H), 2.20 (m, 2H), 1.81 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 153.2, 140.7, 138.2, 135.3, 130.89, 130.87, 129.1, 128.3, 127.3, 127.1, 127.1, 126.4, 124.9, 124.4, 120.0, 71.5, 62.6, 52.4, 49.4, 28.9, 27.4, 26.6, 16.6. HRMS (EI): M^+ calcd. for $C_{25}H_{27}NO_3$ 389.1991, found 389.1983.

Cyclohex-2-en-1-yl 1-oxoindane-2-carboxylate (**7**): Started from 317 mg 1-tetralone (2.4 mmol) and 384.4 mg **1d** (2 mmol), and followed the Procedure A, 323 mg white solid was isolated (63%). M.p. = 77-79 ^oC; R_f = 0.24 (Diethyl ether/petroleum ether 1:9); ^1H NMR (400 MHz,

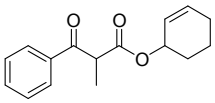
$CDCl_3$): δ = 10.45 (bs, 1H, enol), 7.77 (m, 1H, ketone), 7.62 (m, 1H, enol), 7.62 (m, 1H, ketone), 7.50 (m, 1H, ketone), 7.45 (m, 1H, enol), 7.39 (m, 2H, enol), 7.39 (m, 1H, ketone), 5.98 (m, 1H, enol), 5.98 (m, 1H, ketone), 5.81 (m, 1H, enol), 5.74 (m, 1H, ketone), 5.47 (m, 1H, enol), 5.35 (m, 1H, ketone), 3.71 (m, 1H, ketone), 3.55 (m, 1H, ketone), 3.55 (m, 2H, enol), 3.38 (m, 1H, ketone), 3.38 (m, 1H, enol), 2.20-1.60 (m, 6H, ketone), 2.20-1.60 (m, 6H, enol). Anal. Calcd. for $C_{16}H_{16}O_3$: C, 74.98; H, 6.29; Found: C, 75.07; H, 6.16.



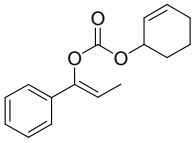
Cyclohex-2-en-1-yl 1*H*-inden-3-yl carbonate (**8**): Started from 317 mg 1-indanone (2.4 mmol) and 384.4 mg **1d** (2 mmol) by procedure D, 470 mg colorless oil was obtained after purification by silica gel chromatography (92%). R_f = 0.47 (Diethyl ether/petroleum ether 1:9); IR (film): 1756 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.41 (m, 2H), 7.30 (m, 1H), 7.24 (m, 1H), 6.34 (t, *J* = 2.4 Hz, 1H), 6.04 (m, 1H), 5.86 (m, 1H), 5.25 (m, 1H), 3.40 (d, *J* = 2.4 Hz, 2H), 2.20-1.60 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 152.3, 149.4, 141.8, 138.7, 134.0, 126.3, 125.7, 124.5, 124.1, 118.2, 114.3, 72.9, 34.9, 28.2, 24.9, 18.6. Anal. Calcd. for $C_{16}H_{16}O_3$: C, 74.98; H, 6.29; Found: C, 75.12; H, 6.11.



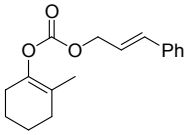
Cyclohex-2-en-1-yl 2-methyl-1*H*-inden-3-yl carbonate (**9**) Started from 350 mg 2-methyl-1-indanone (2.4 mmol) and 384.4 mg **1d** (2 mmol) by procedure C, 474 mg **1d** was obtained as a white solid after purification by silica gel chromatography (88%). M.p. = 66-67 ^oC; R_f = 0.30 (Diethyl ether/petroleum ether 1:9); IR (film): 1756 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.34 (m, 1H), 7.25 (m, 1H), 7.15 (m, 2H), 6.05 (m, 1H), 5.87 (m, 1H), 5.23 (m, 1H), 3.32 (s, 2H), 2.03 (s, 3H), 2.20-1.60 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 152.4, 144.6, 140.1, 139.6, 134.0, 128.3, 126.3, 124.6, 123.8, 117.3, 112.6, 73.1, 39.1, 28.2, 24.9, 18.6, 12.2. Anal. Calcd. for $C_{17}H_{18}O_3$: C, 75.53; H, 6.71; Found: C, 75.55; H, 6.52.



Cyclohex-2-en-1-yl 2-methyl-3-oxo-3-phenylpropanoate (**10**): mixture of 1:1 two diastereomers. Colorless oil; R_f = 0.19 (Diethyl ether/petroleum ether 1:9); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.90 (m, 2H), 7.61-7.52 (m, 1H), 7.50-7.41 (m, 2H), 5.94-5.83 (m, 1H), 5.59 (dtd, *J* = 10.1, 4.1, 2.1 Hz, 1H), 5.24 (tdt, *J* = 5.2, 3.6, 1.7 Hz, 1H), 4.34 (p, *J* = 7.0 Hz, 1H), 2.10-1.84 (m, 2H), 1.74 (tdd, *J* = 12.3, 9.6, 4.7 Hz, 1H), 1.67-1.51 (m, 3H), 1.49 (d, *J* = 1.6 Hz, 3H, one diastereomer), 1.48 (d, *J* = 1.6 Hz, 3H, the other diastereomer); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.0, 195.7, 170.7, 170.6, 136.0, 136.0, 133.4, 133.3, 133.2, 133.0, 128.7, 128.6, 128.6, 124.9, 69.1, 69.0, 48.8, 48.6, 28.0, 27.9, 24.8, 24.8, 18.6, 18.5, 13.7, 13.6. Anal. Calcd. for $C_{16}H_{18}O_3$: C, 74.39; H, 7.02; Found: C, 74.46; H, 7.09.

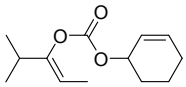


Cyclohex-2-en-1-yl (1*Z*)-1-phenylprop-1-en-1-yl carbonate(**11**) Started from 268 mg propiophenone (2.0 mmol) and 462 mg **1d** (2.4 mmol) by procedure B, 516 mg colorless oil was obtained after purification by silica gel chromatography (100%). R_f = 0.30 (Diethyl ether/petroleum ether 1:9); IR (film): 1757 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.46-7.24 (m, 5H), 6.01 (m, 1H), 5.85 (q, *J* = 7.0 Hz, 1H), 5.79 (m, 1H), 5.15 (m, 1H), 1.78 (s, 3H), 2.20-1.60 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 152.7, 147.5, 135.0, 133.8, 128.6, 128.2, 124.7, 124.4, 112.8, 72.7, 28.2, 24.9, 18.6, 11.4. Anal. Calcd. for $C_{16}H_{18}O_3$: C, 74.39; H, 7.02; Found: C, 74.20; H, 6.88.

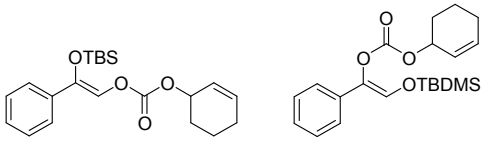


2-Methylcyclohex-1-en-1-yl (2*E*)-3-phenylprop-2-en-1-yl carbonate (**13**) To the solution of 269 mg 2-methyl-cycolohexan-1-one (2.4 mmol) in 5 mL DME at 0 ^{o}C was added a solution of 460 mg NaHMDS (2.4 mmol) in 2 mL DME under argon. The solution was stirred for 15 min before it was cooled to -78 oC. Meanwhile, a solution of 457 mg **1c** (2.0 mmol) in 5 mL DME at -78 ^{o}C was added 0.28 mL BF_3 etherate under argon. The enolate solution was transferred into the **1c**-BF3 solution through a cannula and the reaction mixture was stirred for 15 min before it was quenched with a portion of 20 mL saturated aqueous NH_4Cl. 415 mg colorless oil was obtained after purification by silica gel chromatography (99%). R_f = 0.31 (Diethyl ether/petroleum ether 1:9); IR (film): 1747 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.43-7.24 (m, 5H), 6.71 (d, *J* = 15.9

Hz, 1H), 6.32 (dt, *J* = 15.9, 6.4 Hz, 1H), 4.81 (dd, *J* = 6.6, 1.2 Hz, 2H), 2.17 (m, 2H), 2.04 (m, 2H), 1.78-1.57 (m, 7H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 153.3, 142.3, 136.1, 134.9, 128.7, 128.3, 126.8, 122.5, 121.0, 68.6, 30.2, 26.8, 23.2, 22.4, 15.9. Anal. Calcd. for $C_{17}H_{20}O_3$: C, 74.97; H, 7.40; Found: C, 75.17; H, 7.23.



Cyclohex-2-en-1-yl (1*Z*)-1-isopropylprop-1-en-1-yl carbonate (**15**): Started from 240.4 mg 2-methyl-3-pentanone (2.4 mmol) and 384.4 mg **1d** (2.0 mmol) by Procedure C, 392 mg colorless oil was obtained after purification by silica gel chromatography (93%). R_f = 0.48 (Diethyl ether/petroleum ether 1:9); IR (film): 1748 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 6.05-5.96 (m, 1H), 5.86-5.77 (m, 1H), 5.19-5.13 (m, 1H), 5.09 (dq, *J* = 6.8, 1.1 Hz, 1H), 2.53-2.38 (m, 1H), 2.19-1.61 (m, 6H), 1.53 (dd, *J* = 6.8, 1.3 Hz, 3H), 1.07 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 154.5, 152.7, 133.5, 124.8, 108.6, 72.2, 32.2, 28.2, 24.9, 20.2, 18.6, 10.6.

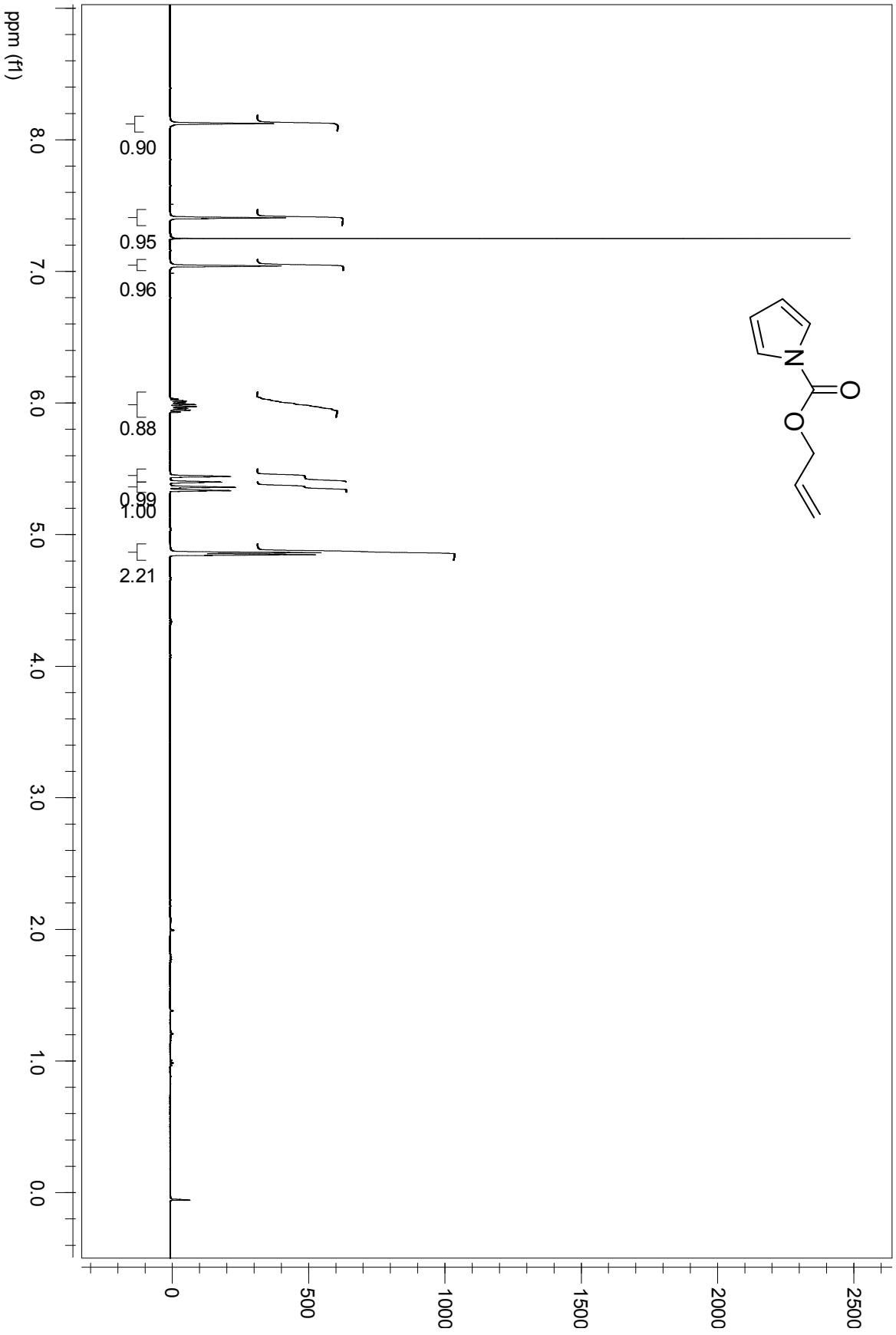


Started from 500 mg 2-{[*tert*-butyl(dimethyl)silyl]oxy}-1-phenylethanone (2 mmol) and 461 mg **1d** (2.4 mmol) by Procedure B, 415 mg **16** (55%) and 166 mg **17** (22%) were obtained after purification by silica gel chromatography.

(*Z*)-2-{[*tert*-Butyl(dimethyl)silyl]oxy}-2-phenylvinyl cyclohex-1-en-1-yl carbonate (**16**):[3] ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.49 (m, 2H), 7.30 (m, 3H), 7.12 (s, 1H), 6.00 (m, 1H), 5.80 (m, 1H), 5.19 (m, 1H), 2.20-1.60 (m, 6H), 0.97 (s, 9H), 0.14 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 152.6, 139.2, 135.7, 133.6, 128.3, 128.1, 125.1, 124.7, 121.4, 72.5, 28.2, 25.8, 24.9, 18.6, 18.5, -4.3.

(*Z*)-2-{[*tert*-Butyl(dimethyl)silyl]oxy}-1-phenylvinyl cyclohept-2-en-1-yl carbonate (**17**):[3] ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.4-7.2 (m, 5H), 6.74 (s, 1H), 6.00 (m, 1H), 5.81 (m, 1H), 5.17 (m, 1H), 2.2-1.6 (m, 6H), 0.96 (s, 9H), 0.21 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 152.7, 134.8, 133.5, 130.6, 128.7, 128.6, 127.3, 124.9, 123.2, 72.5, 28.2, 25.9, 25.5, 24.9, 18.6, -5.2.

[3] Trost, B. M.; Xu, J.; Markus, R. *J. Am. Chem. Soc.* **2007**, *129*, 282-283.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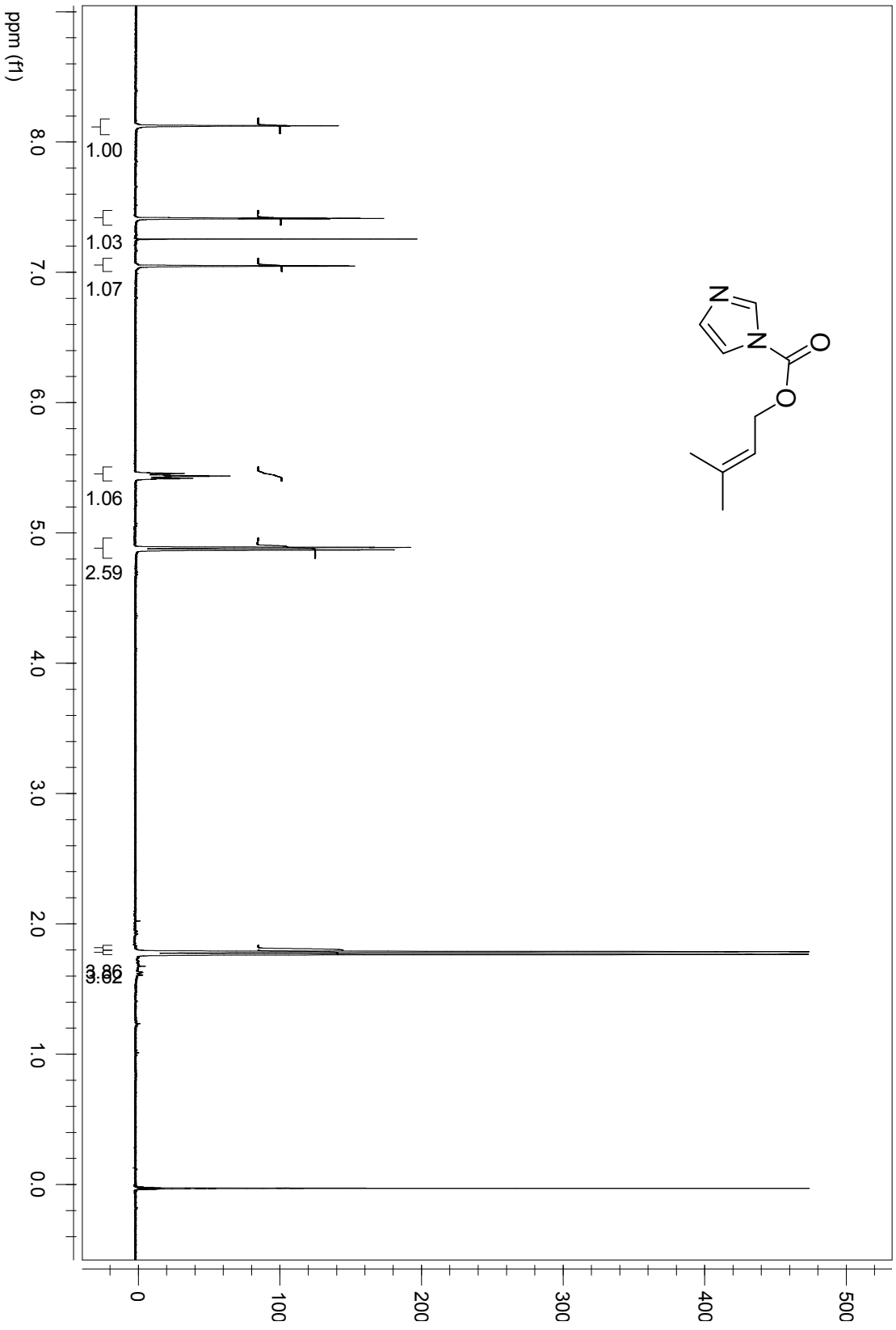
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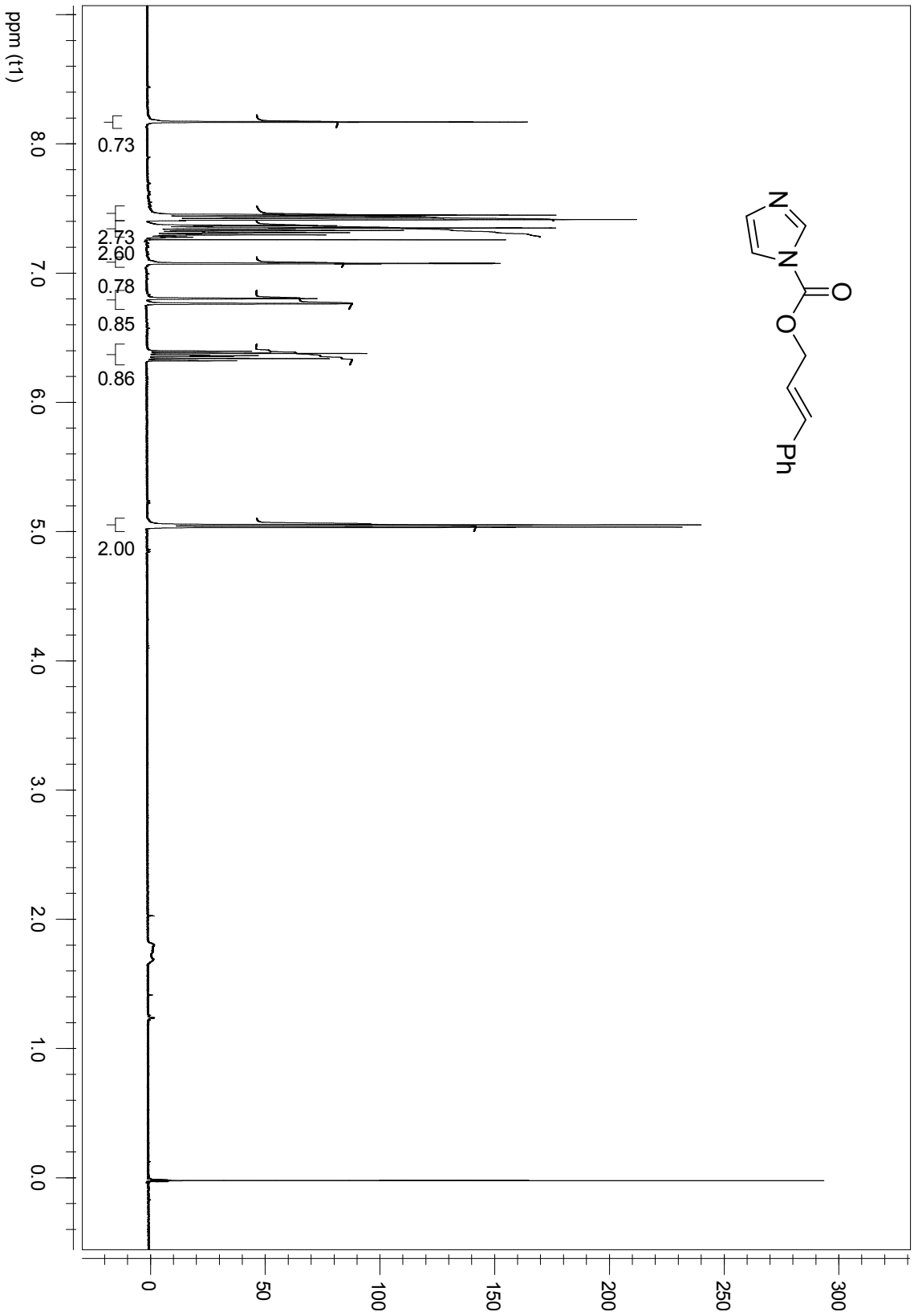
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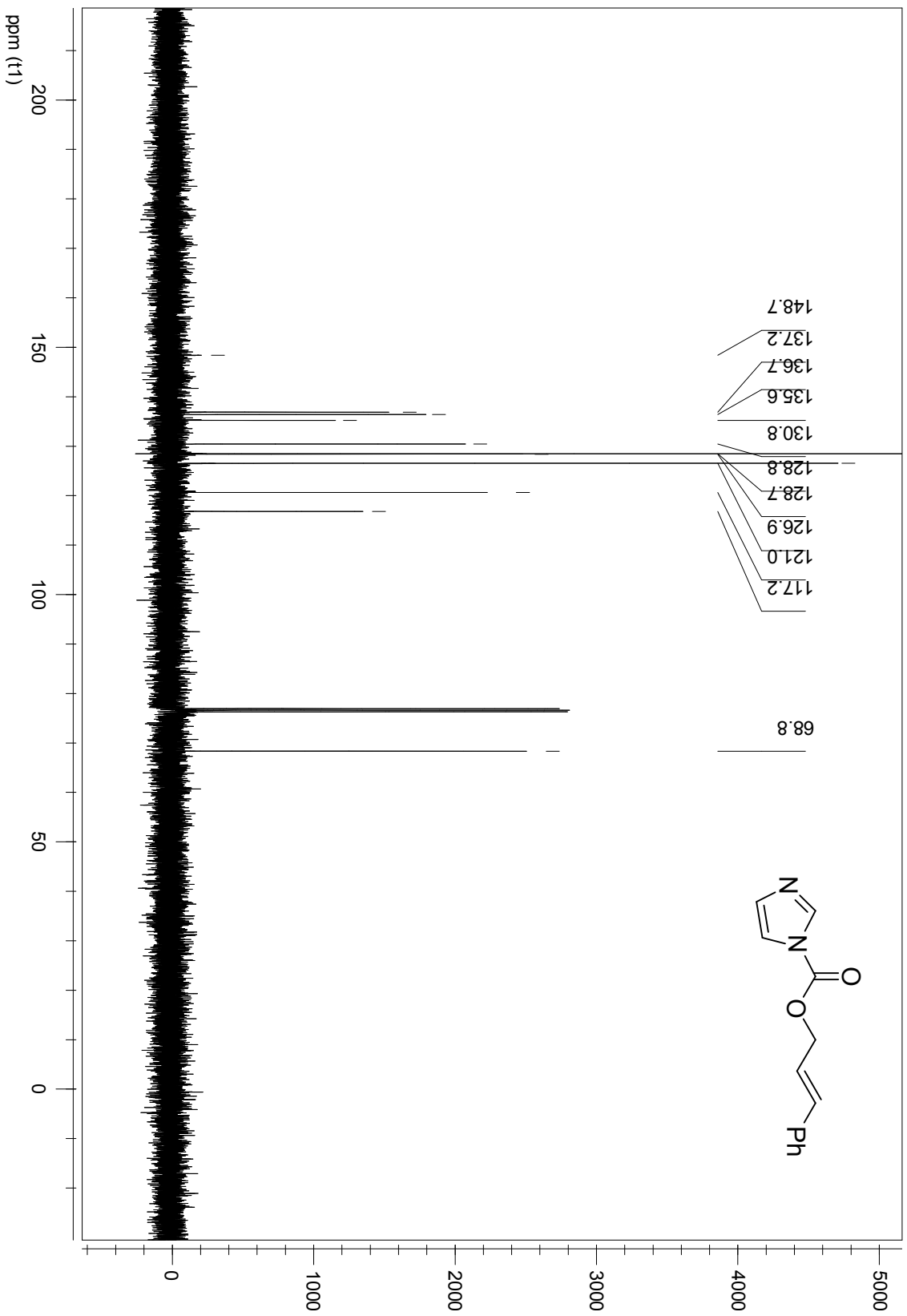
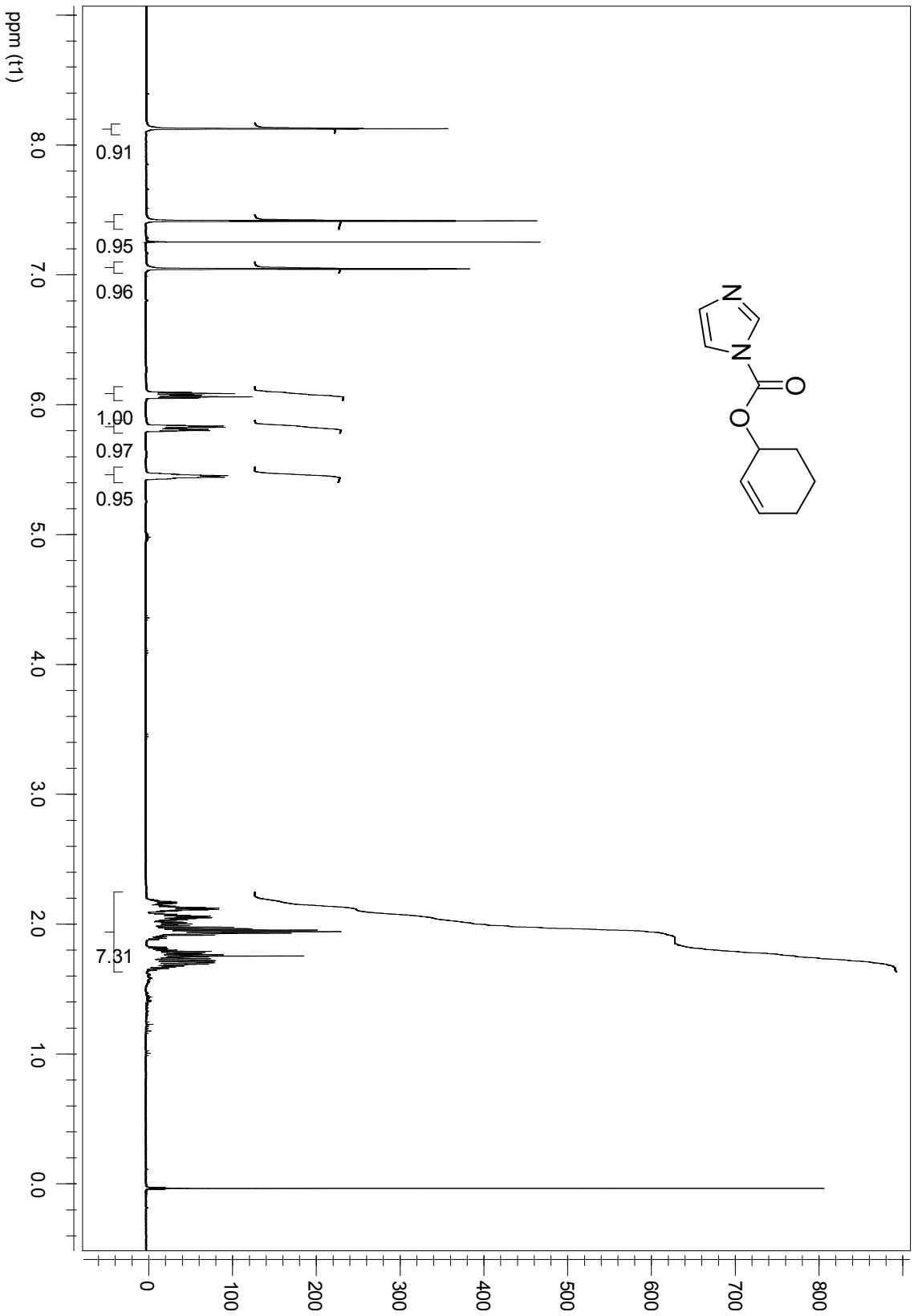
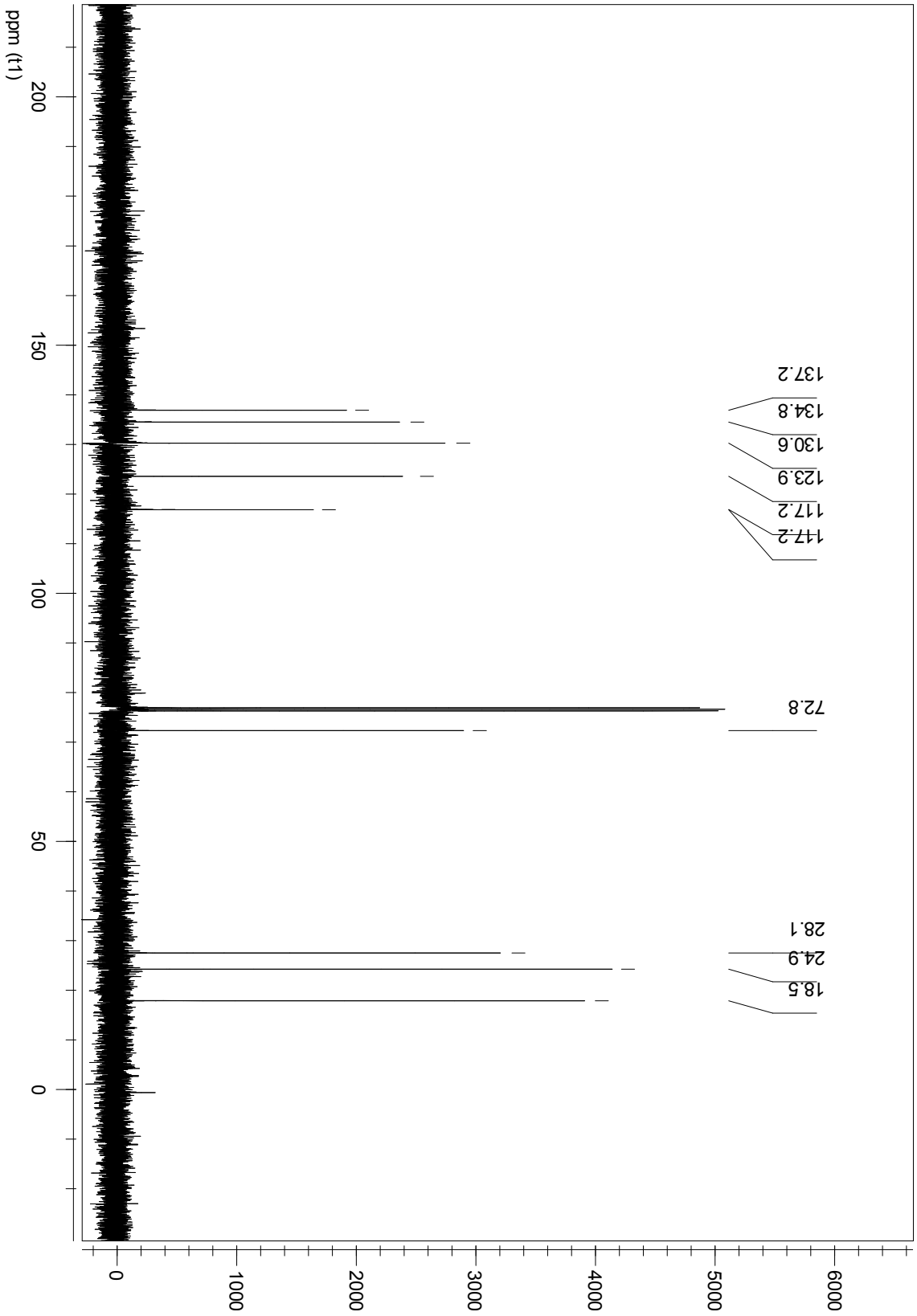
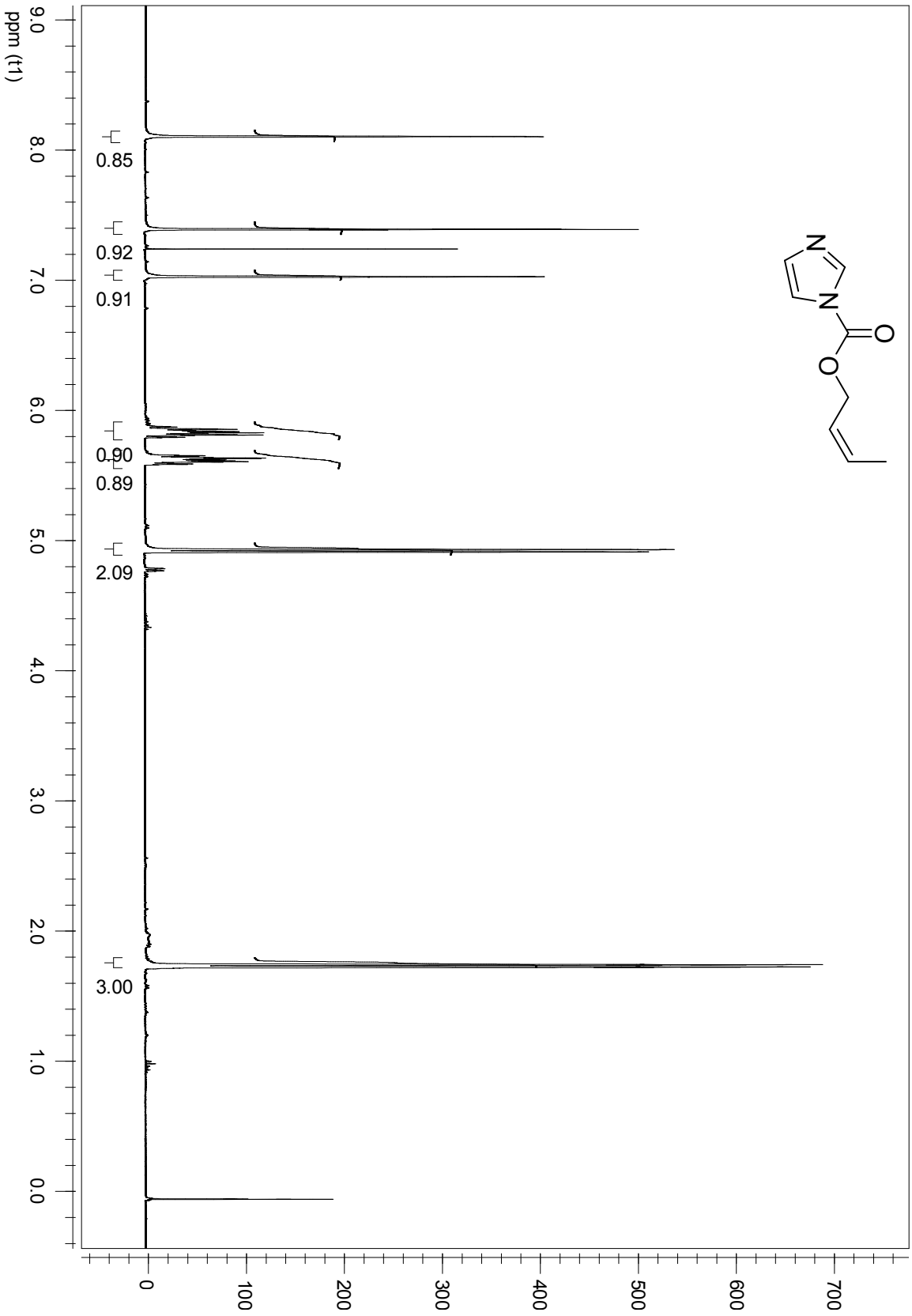
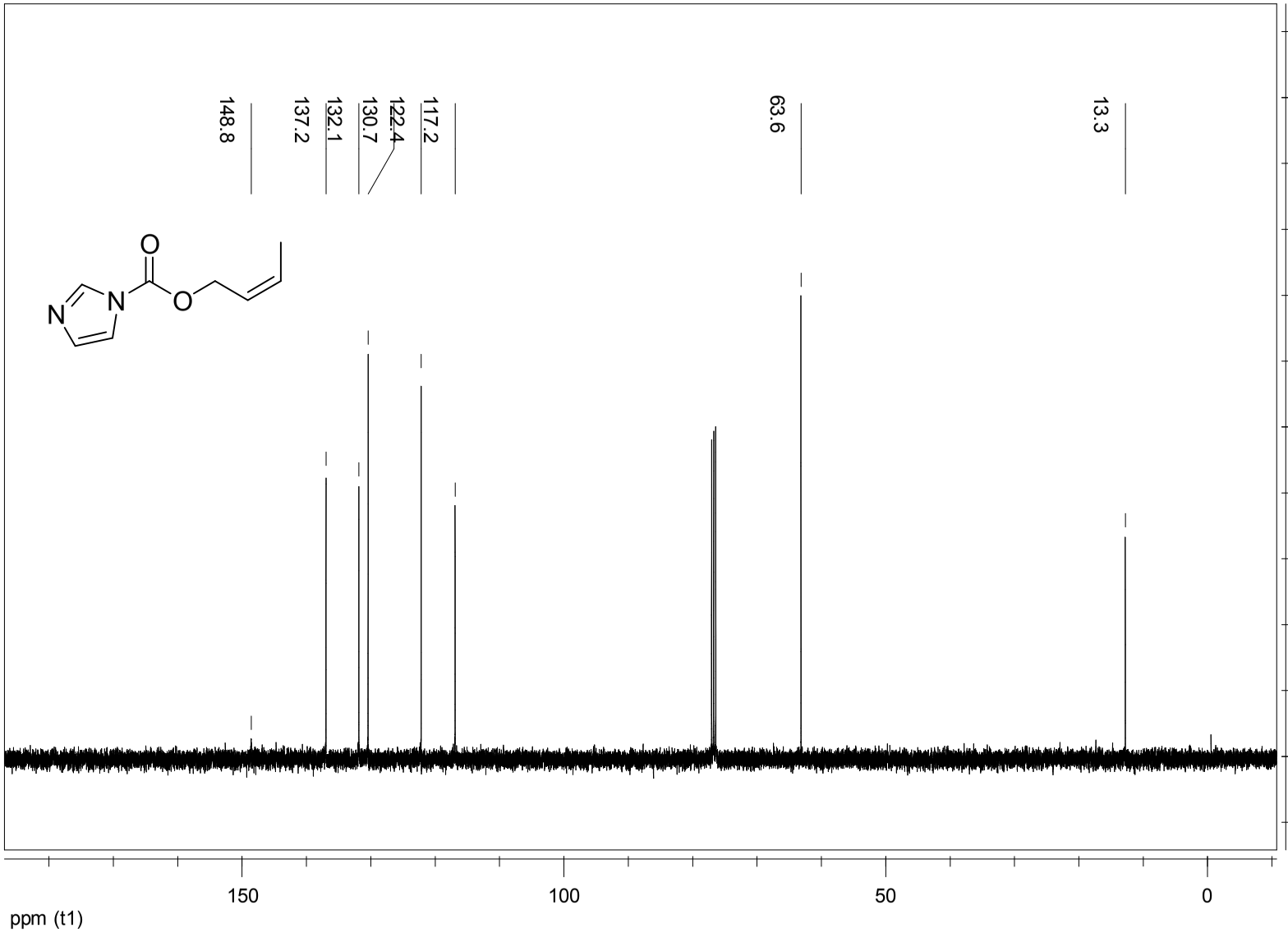
O
Ph

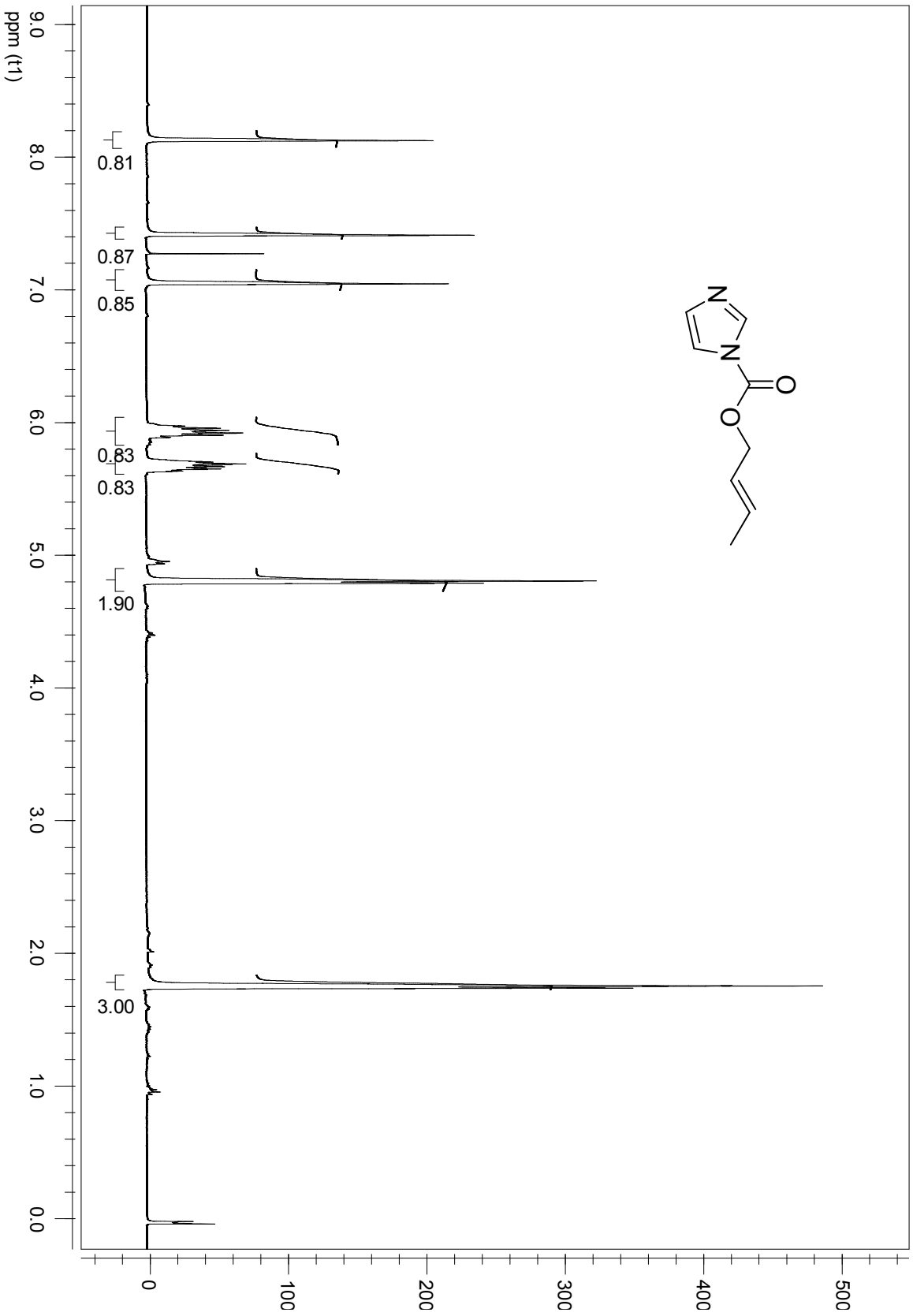

ppm (t1)
200
150
100
50
0
0
1000
2000
3000
4000
5000
148.7
137.2
136.7
135.6
130.8
128.8
128.7
126.9
121.0
117.2
68.8
N
N
O
O
Ph

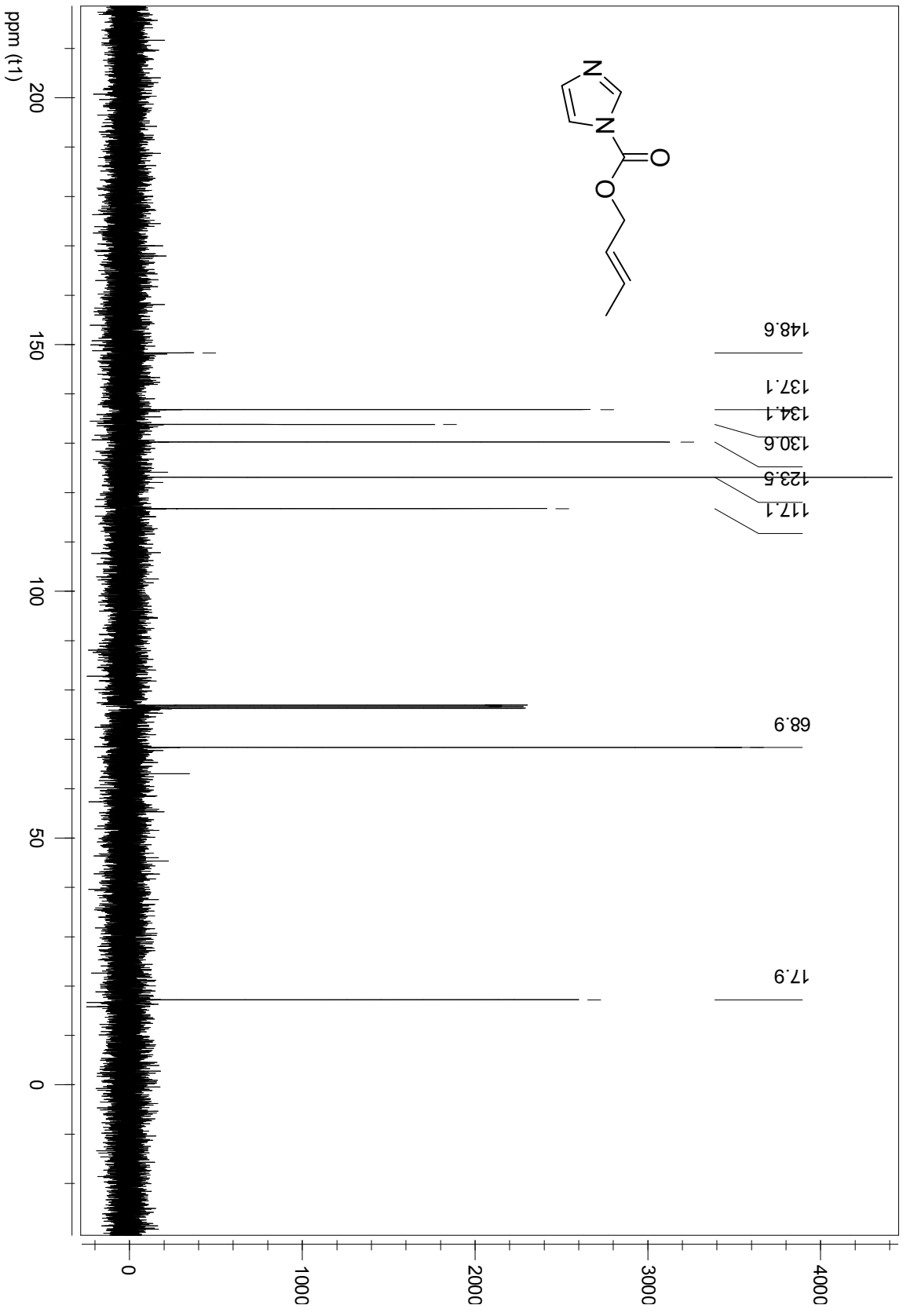

ppm (t1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0.91
0.95
0.96
1.00
0.97
0.95
7.31
N
N
O
O
0
100
200
300
400
500
600
700
800

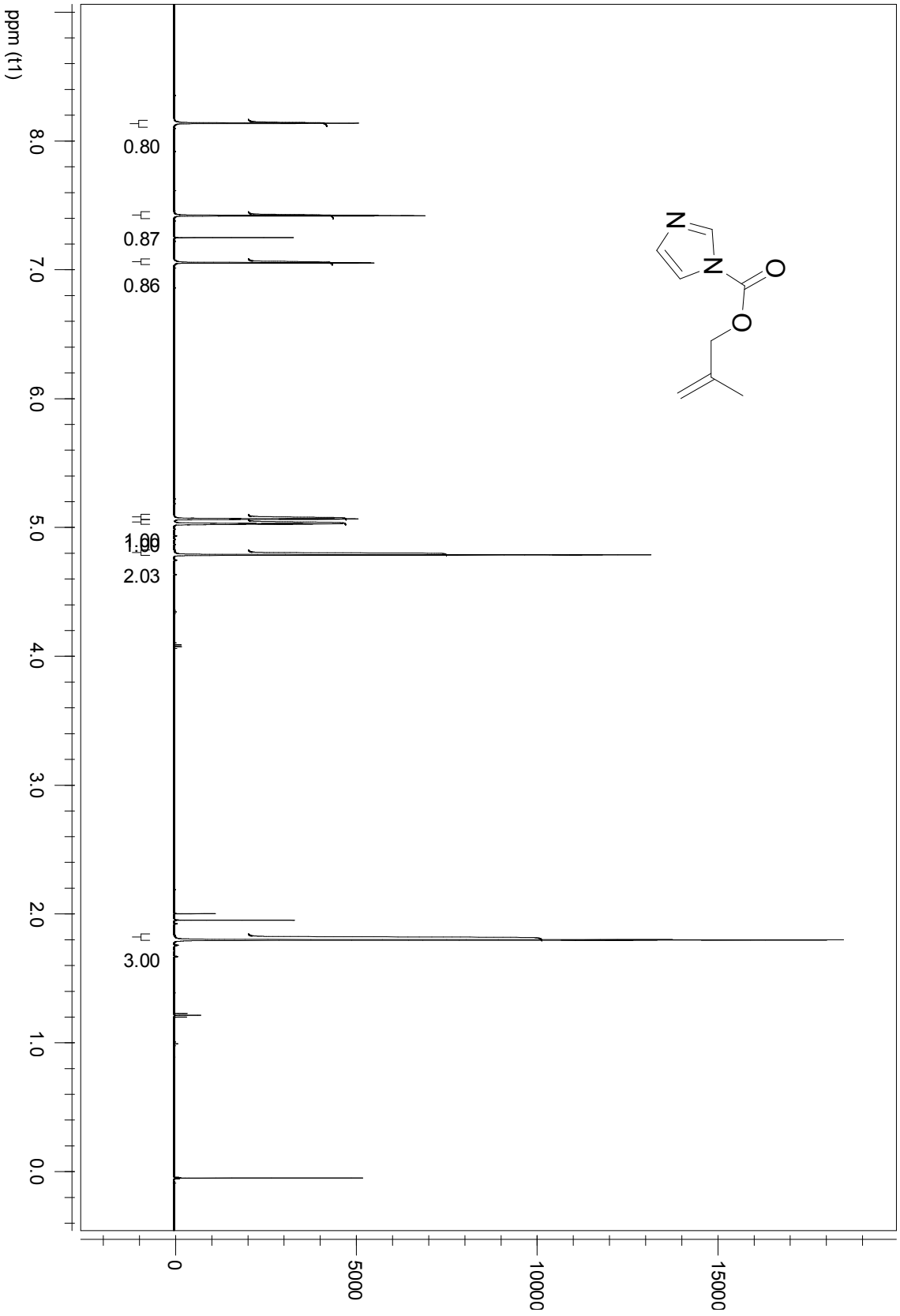

ppm (t1)
0
50
100
150
200
0
1000
2000
3000
4000
5000
6000
137.2
134.8
130.6
123.9
117.2
117.2
72.8
28.1
24.9
18.5

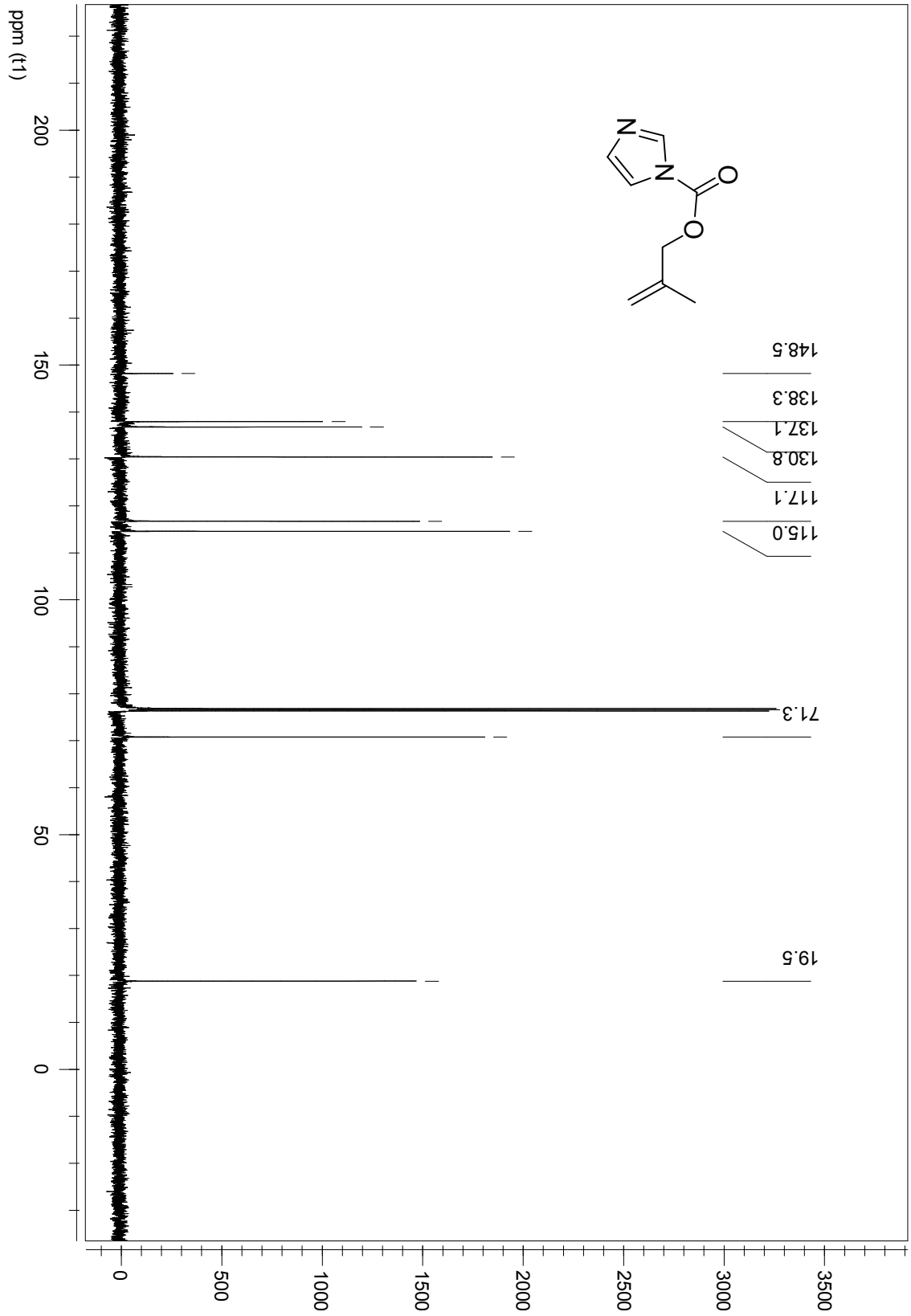

ppm (t1)
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0.85
0.92
0.91
0.90
0.89
2.09
3.00
0
100
200
300
400
500
600
700
N
N
O
O

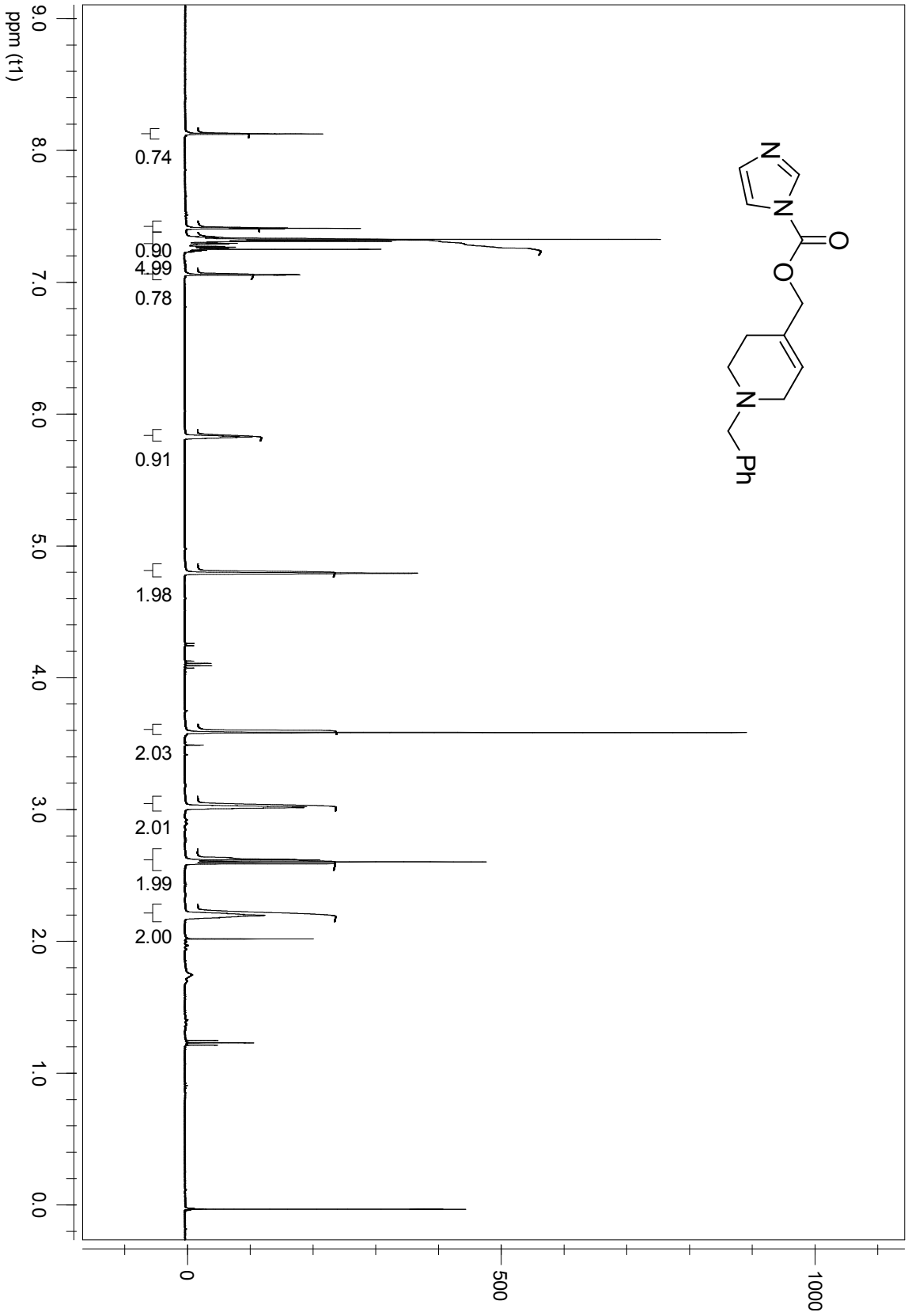

148.8
137.2
132.1
130.7
122.4
117.2
63.6
13.3
O
N
N
O
10000
5000
0
150
100
50
0
ppm (t1)

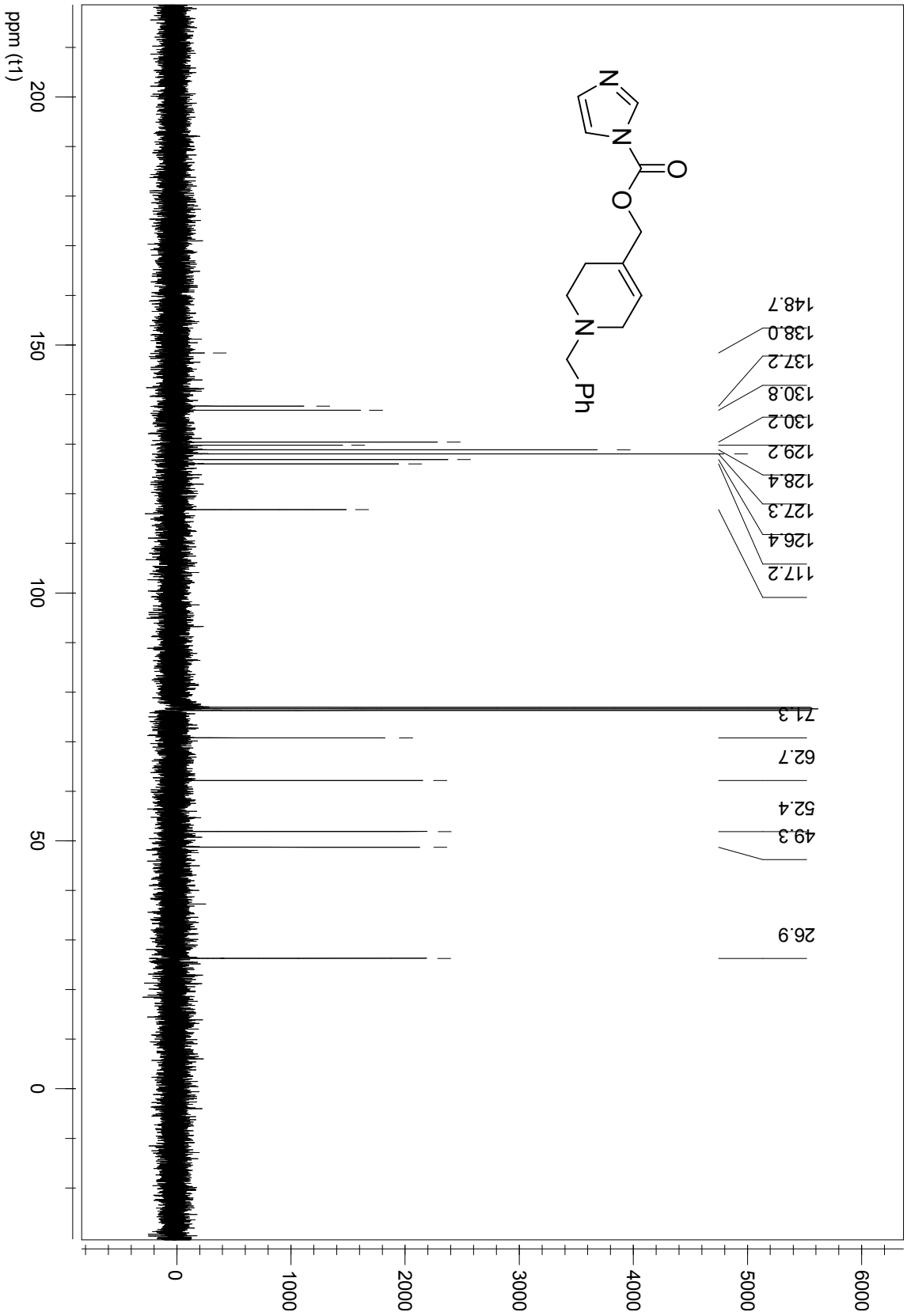

ppm (t1)
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0.81
0.87
0.85
0.83
0.83
1.90
3.00
0
100
200
300
400
500
N
N
O
O

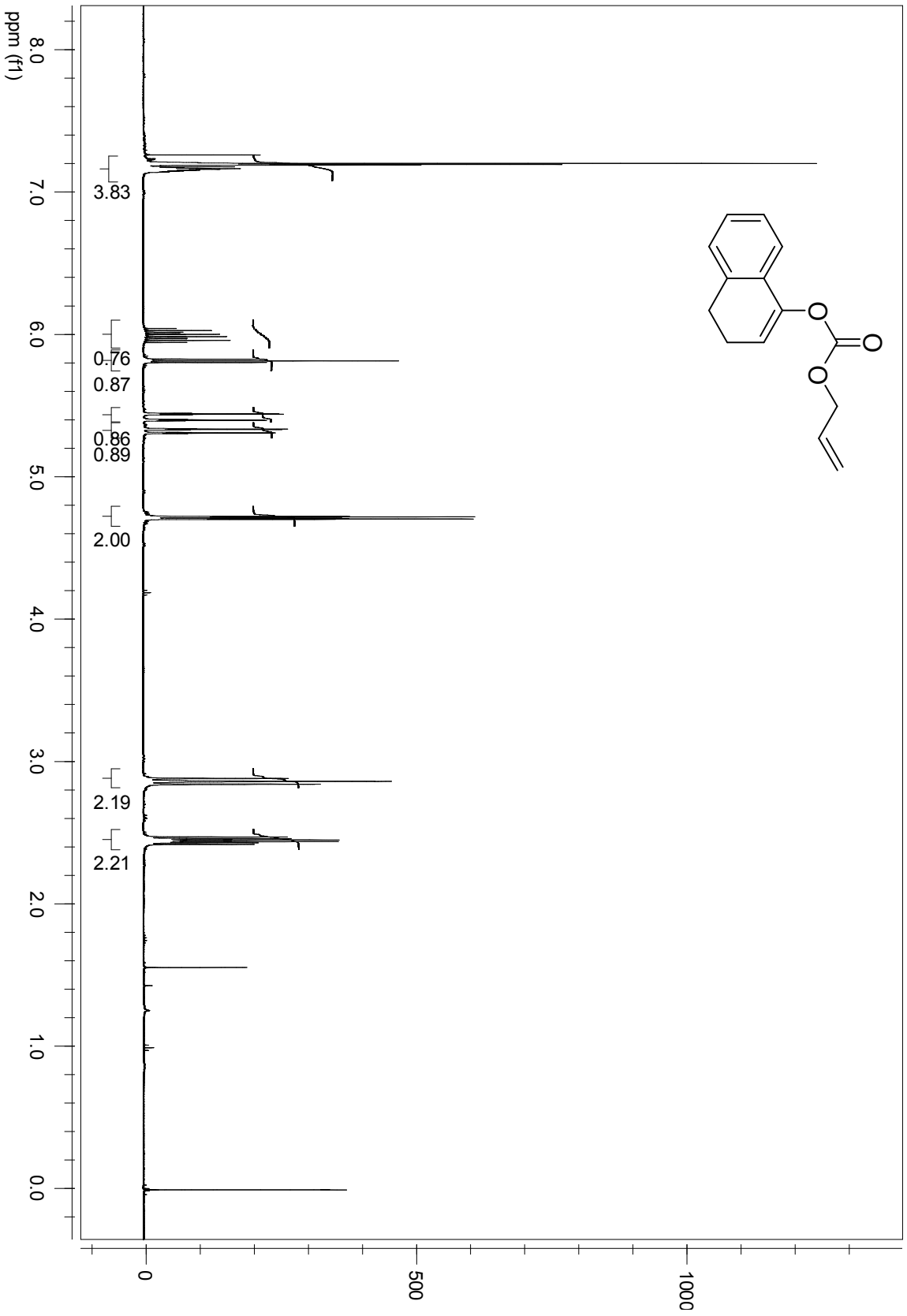

ppm (t1)
200
150
100
50
0
0
1000
2000
3000
4000
148.6
137.1
134.1
130.6
123.6
117.1
68.9
17.9
N
N
O
O

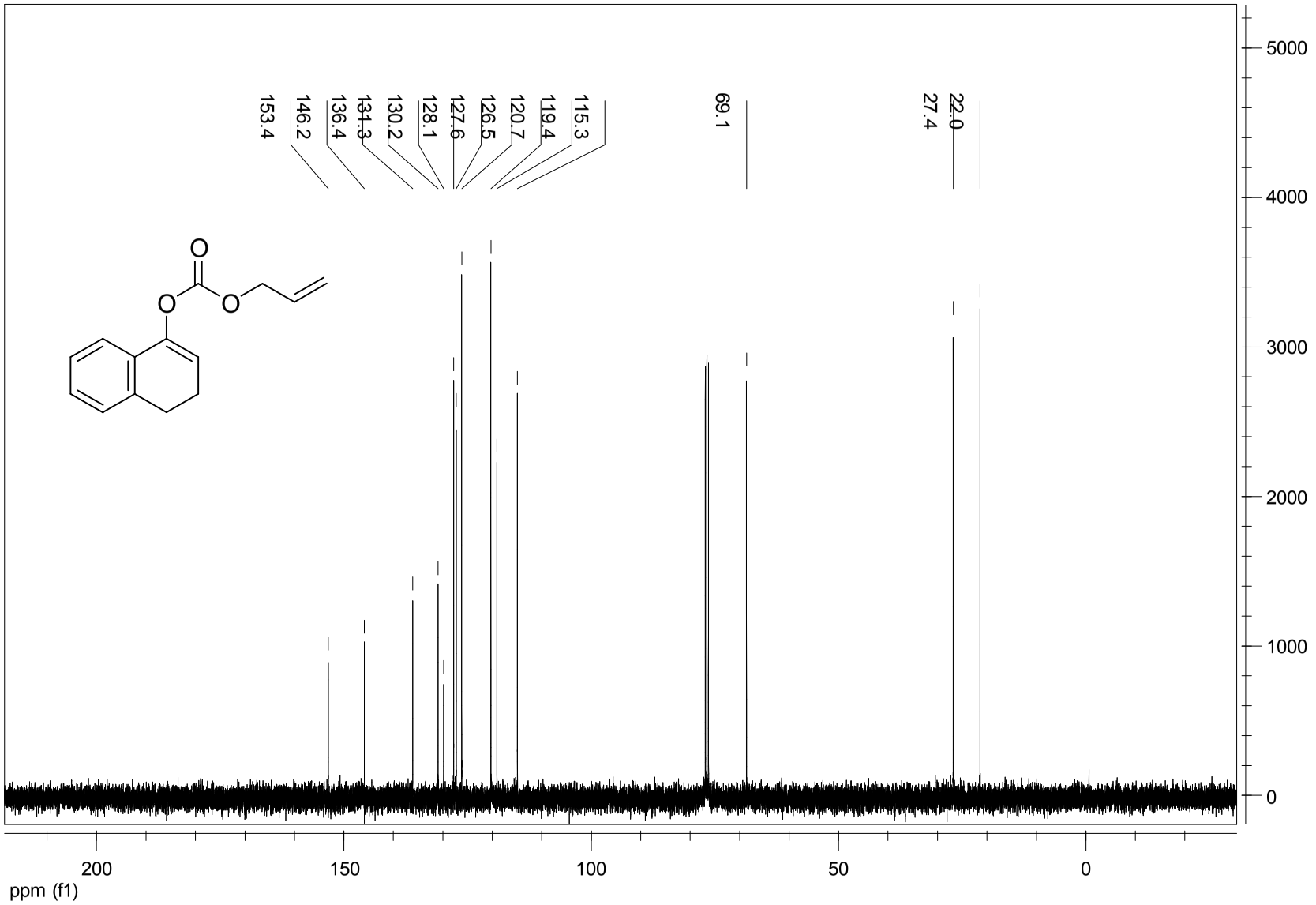

ppm (t1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0.80
0.87
0.86
1.00
2.03
3.00
0
5000
10000
15000
N
N
O
O

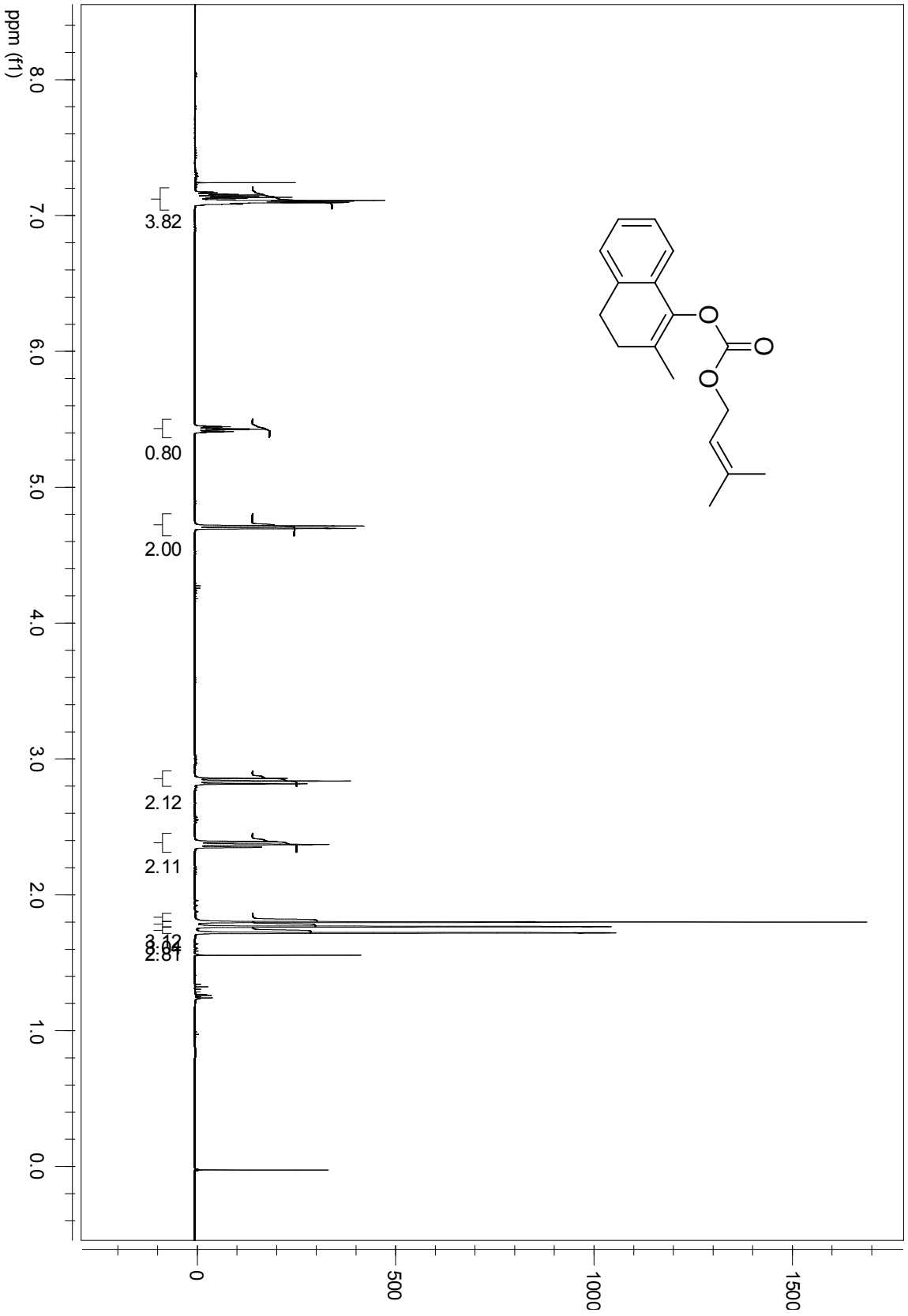

ppm (t1)
0
50
100
150
200
0
500
1000
1500
2000
2500
3000
3500
19.5
71.3
115.0
117.1
130.8
137.1
138.3
148.5
N
N
O
O

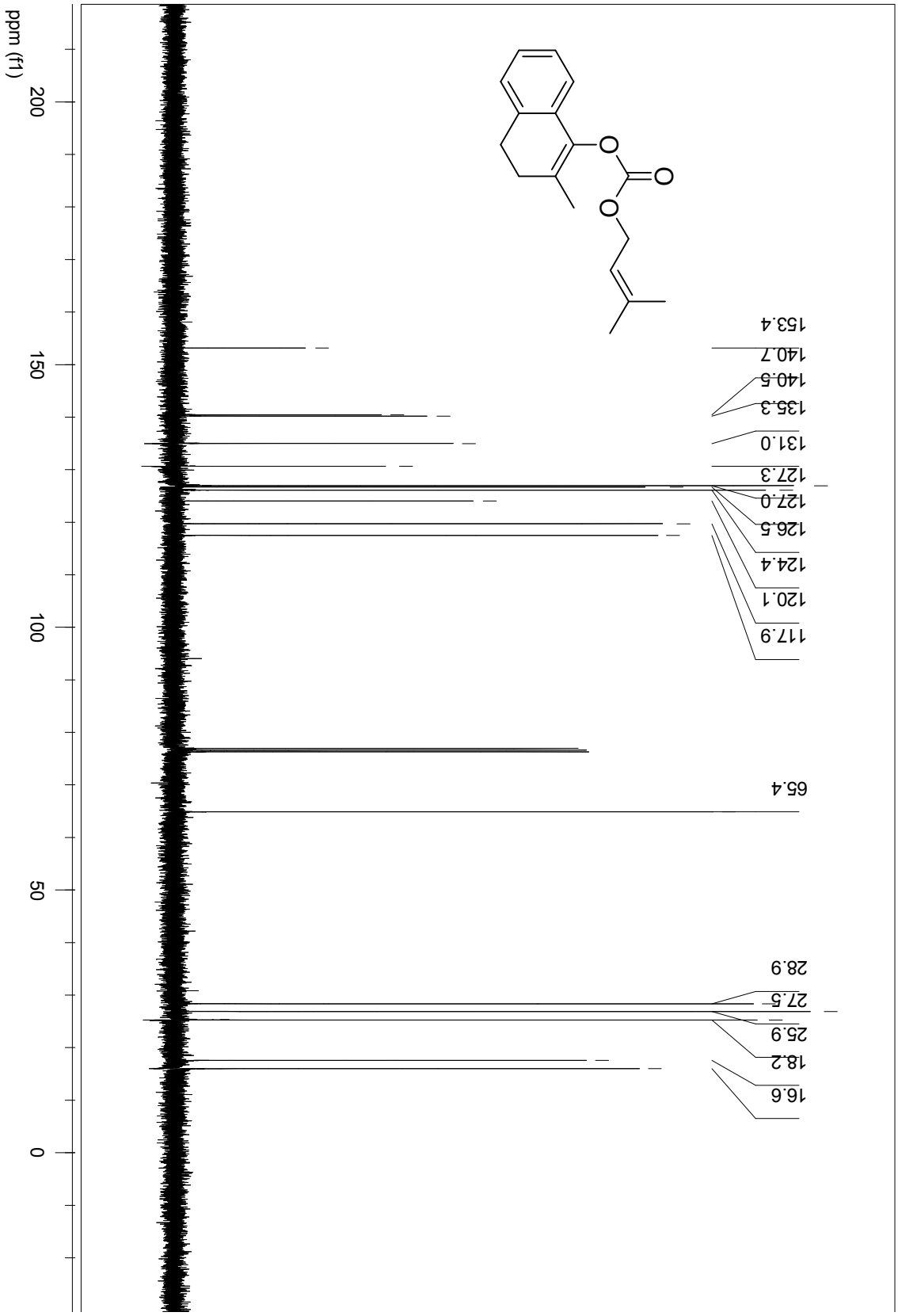

ppm (t1)
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0.74
0.90
4.99
0.78
0.91
1.98
2.03
2.01
1.99
2.00
0
500
1000
N
N
O
O
N
Ph

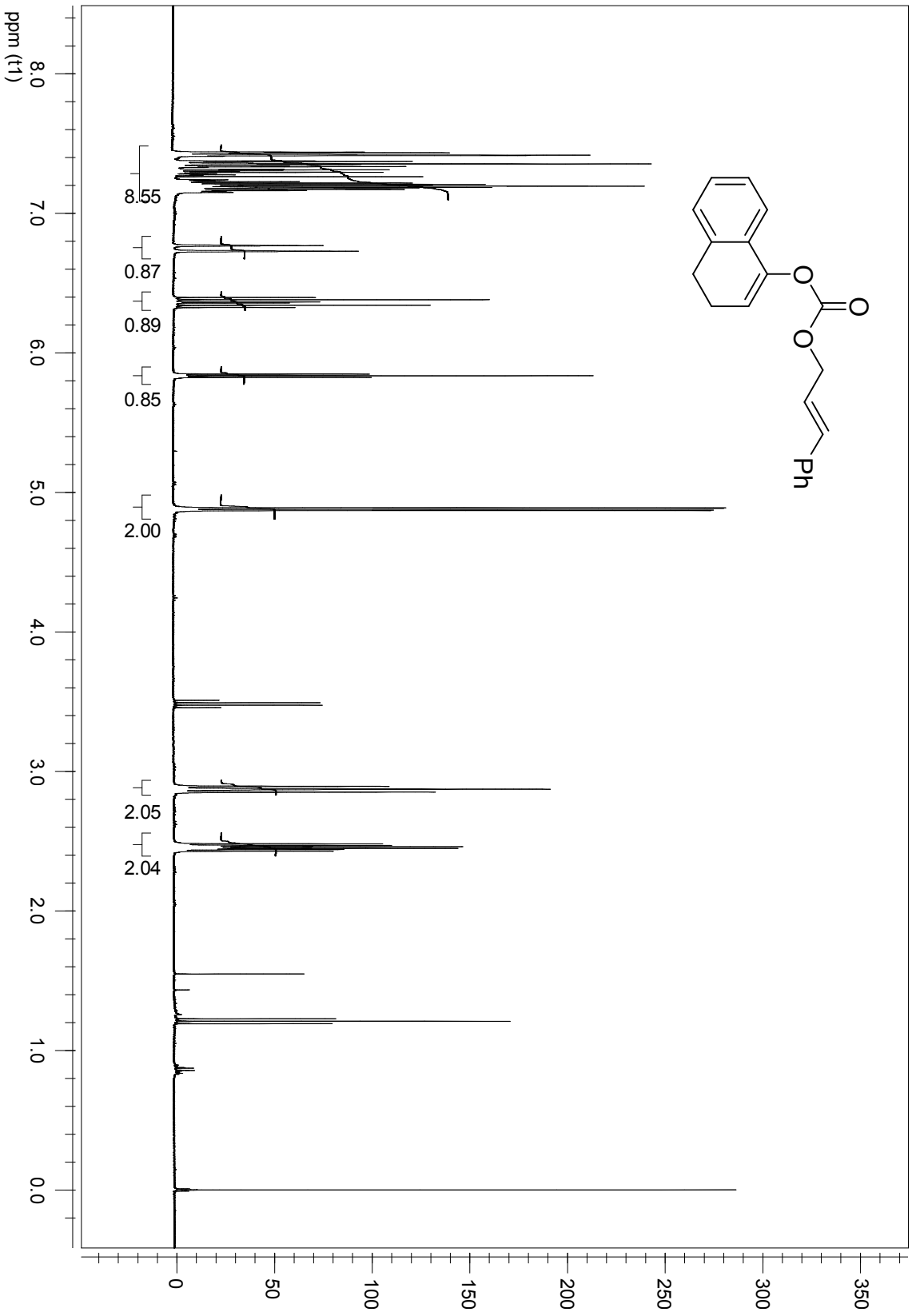

ppm (t1)
200
150
100
50
0
0
1000
2000
3000
4000
5000
6000
148.7
138.0
137.2
130.8
130.2
129.2
128.4
127.3
126.4
117.2
71.3
62.7
52.4
49.3
26.9
N
N
O
O
N
Ph

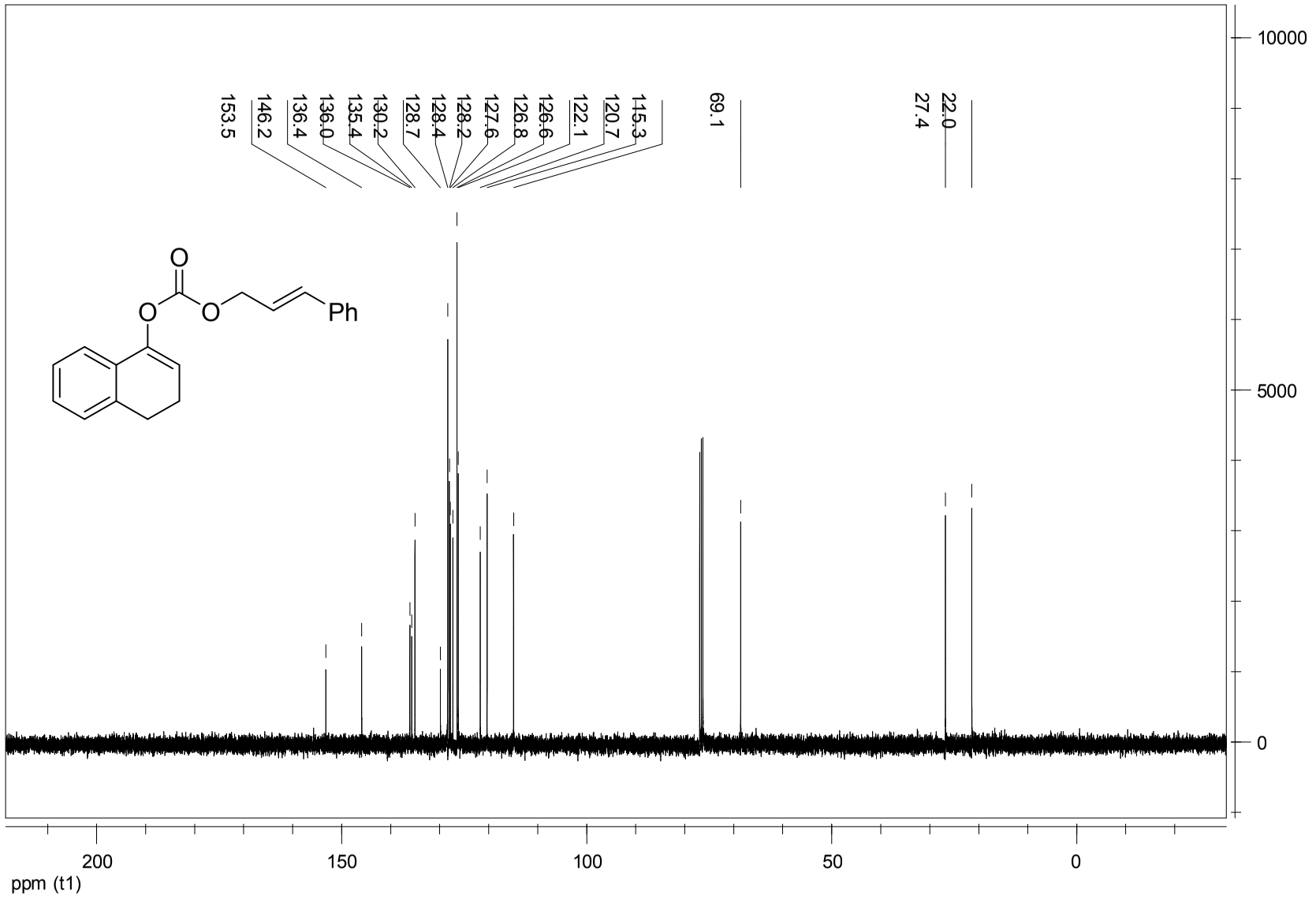

ppm (f1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
3.83
0.76
0.87
0.86
0.89
2.00
2.19
2.21
0
500
1000
O
O
O

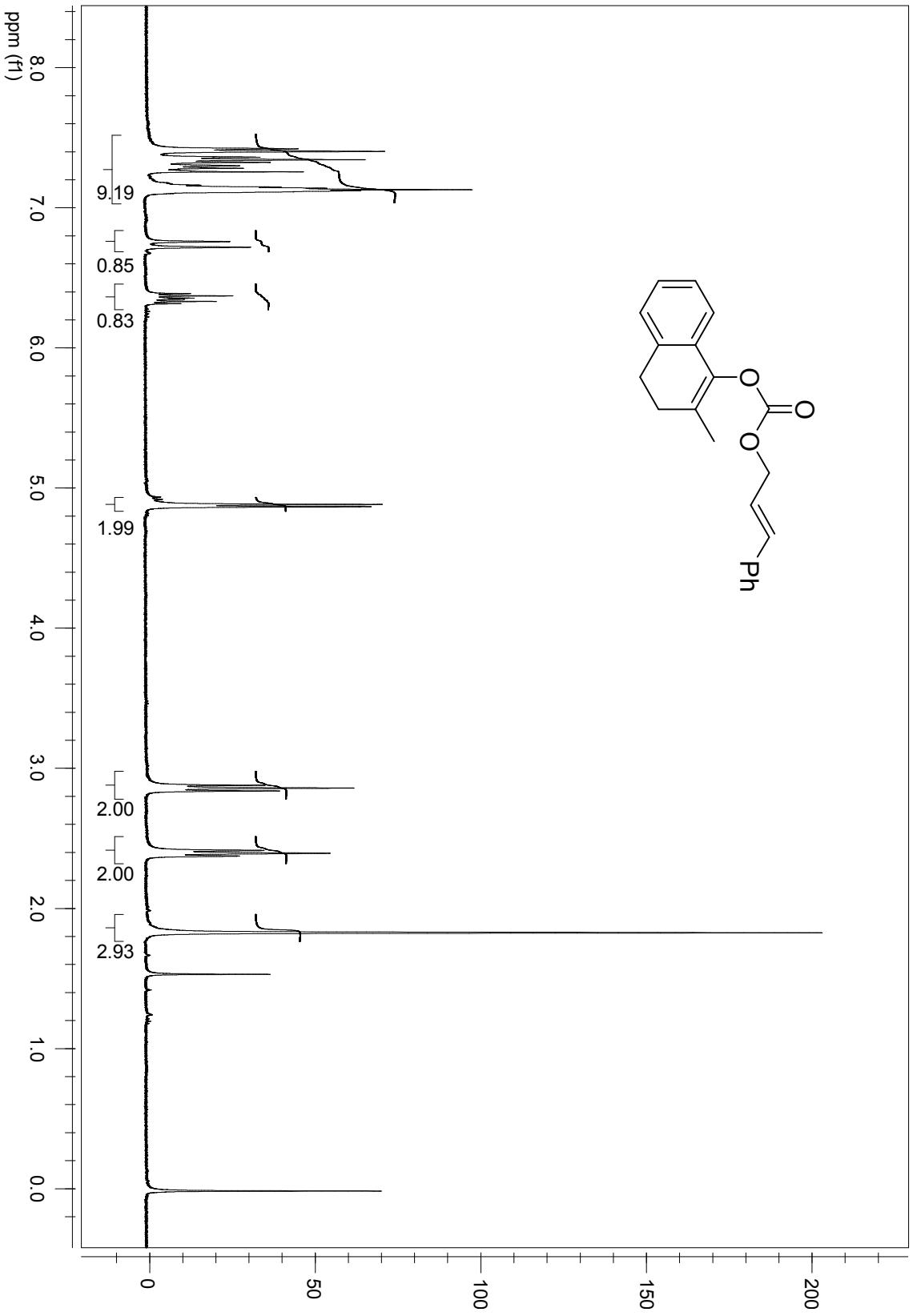

153.4
146.2
136.4
131.3
130.2
128.1
127.6
126.5
120.7
119.4
115.3
69.1
27.4
22.0
O
O
O
5000
4000
3000
2000
1000
0
200
150
100
50
0
ppm (f1)

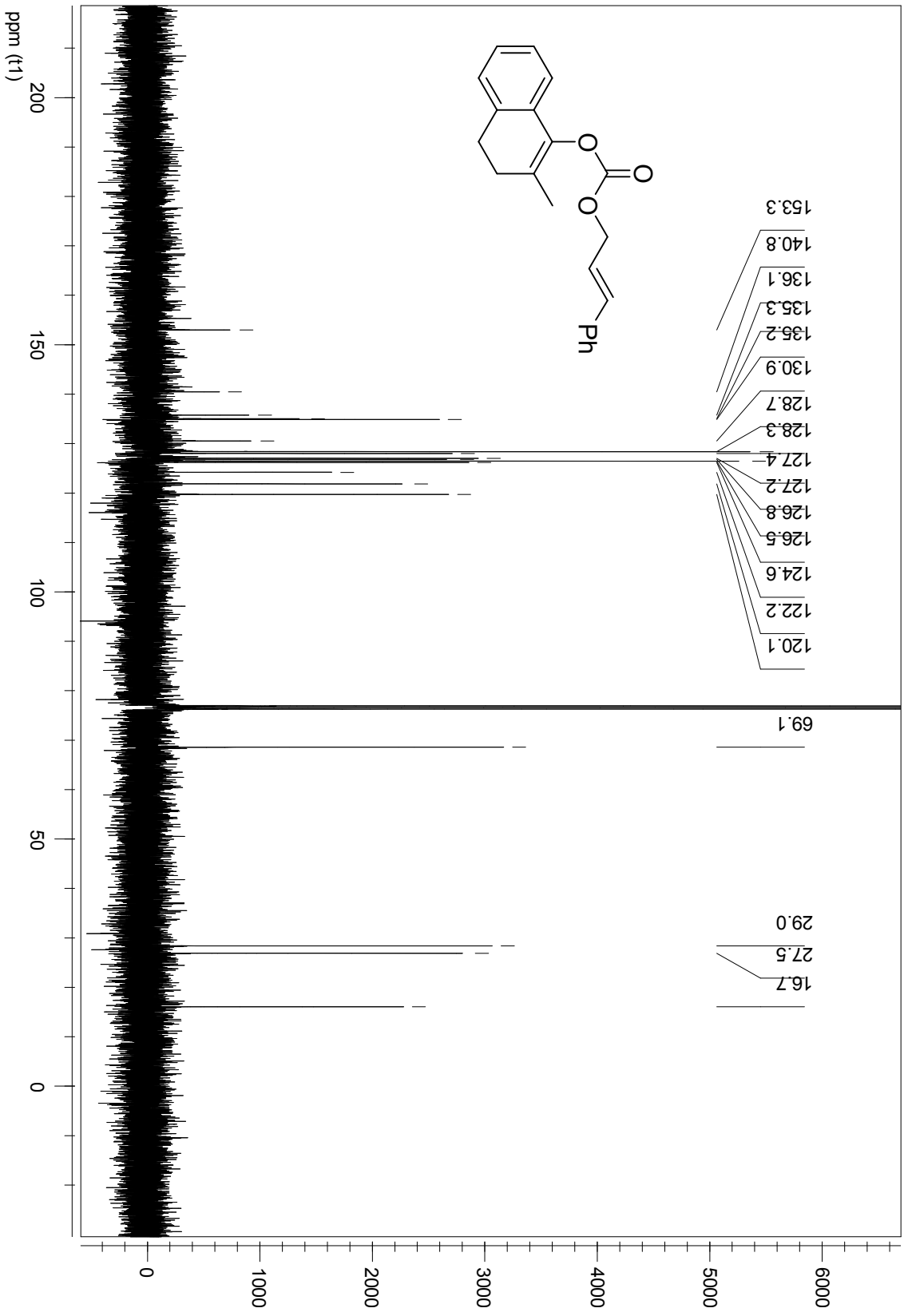

ppm (f1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
3.82
0.80
2.00
2.12
2.11
2.81
0
500
1000
1500

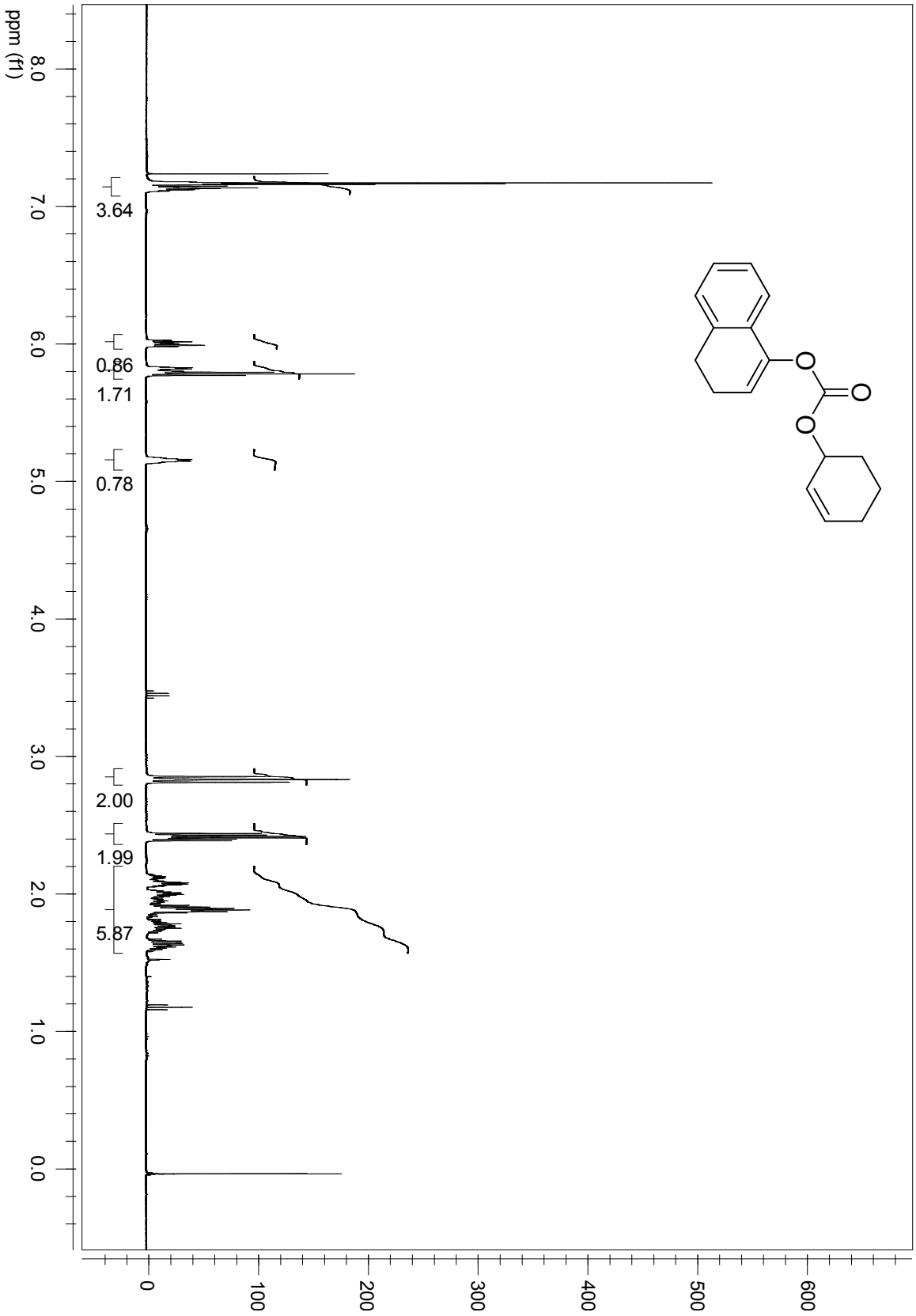

ppm (f1)
200
150
100
50
0
153.4
140.7
140.5
135.3
131.0
127.3
127.0
126.5
124.4
120.1
117.9
65.4
28.9
27.5
25.9
18.2
16.6
O
O
O

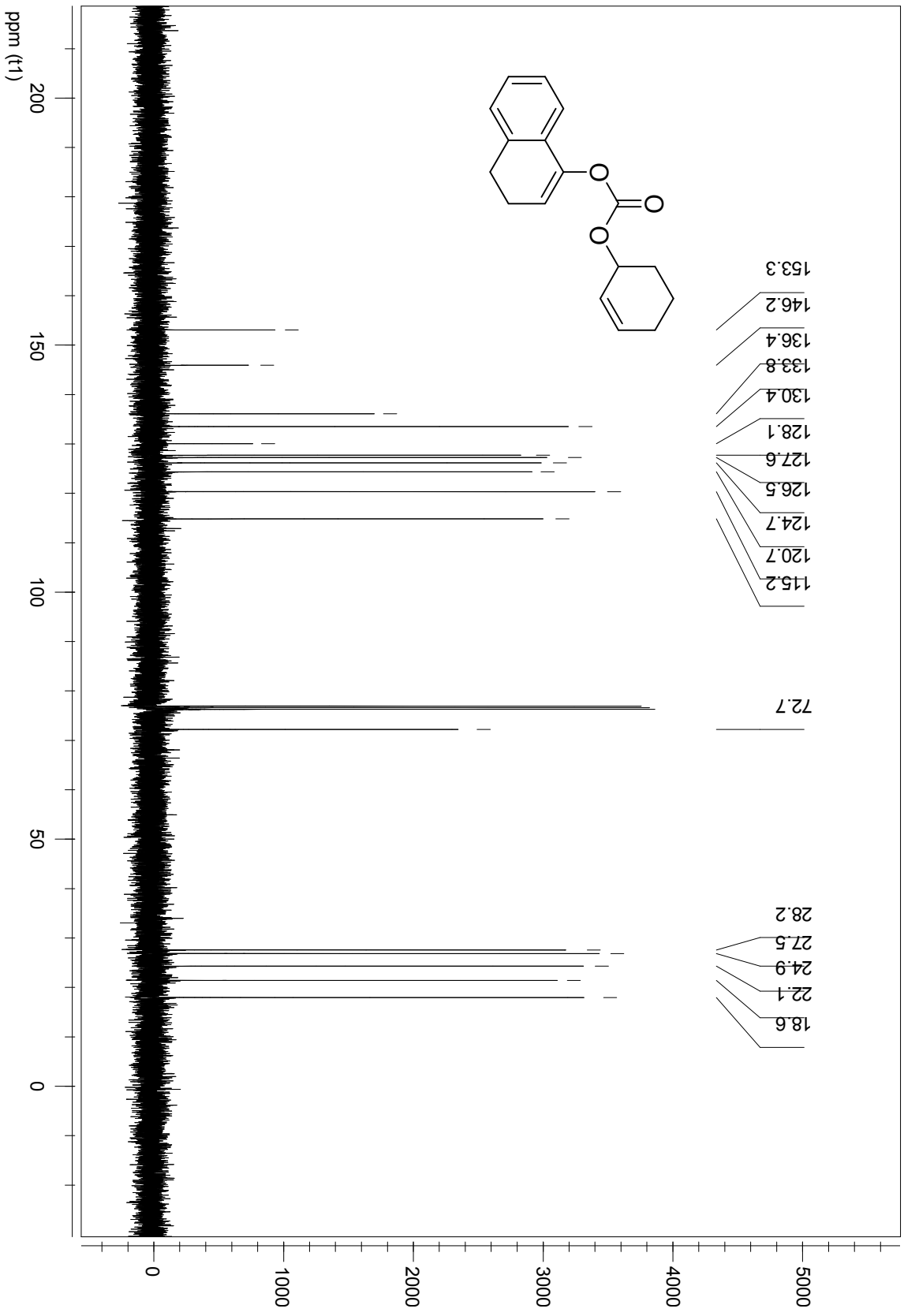

ppm (t1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
8.55
0.87
0.89
0.85
2.00
2.05
2.04
0
50
100
150
200
250
300
350
O
O
O
Ph

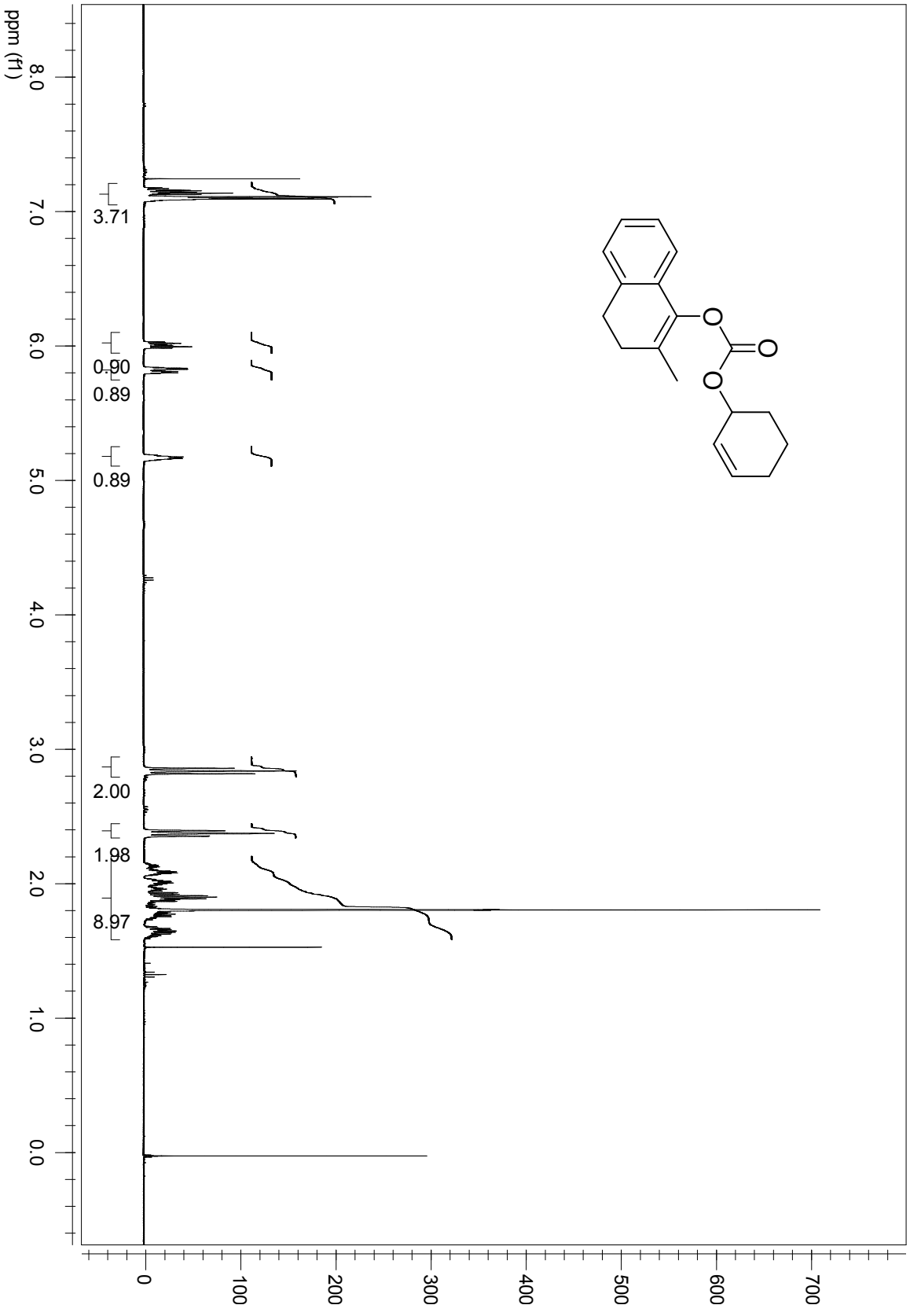

153.5
146.2
136.4
136.0
135.4
130.2
128.7
128.4
128.2
127.6
126.8
126.6
122.1
120.7
115.3
69.1
27.4
22.0
O
O
O
Ph
10000
5000
0
200
150
100
50
0
ppm (t1)

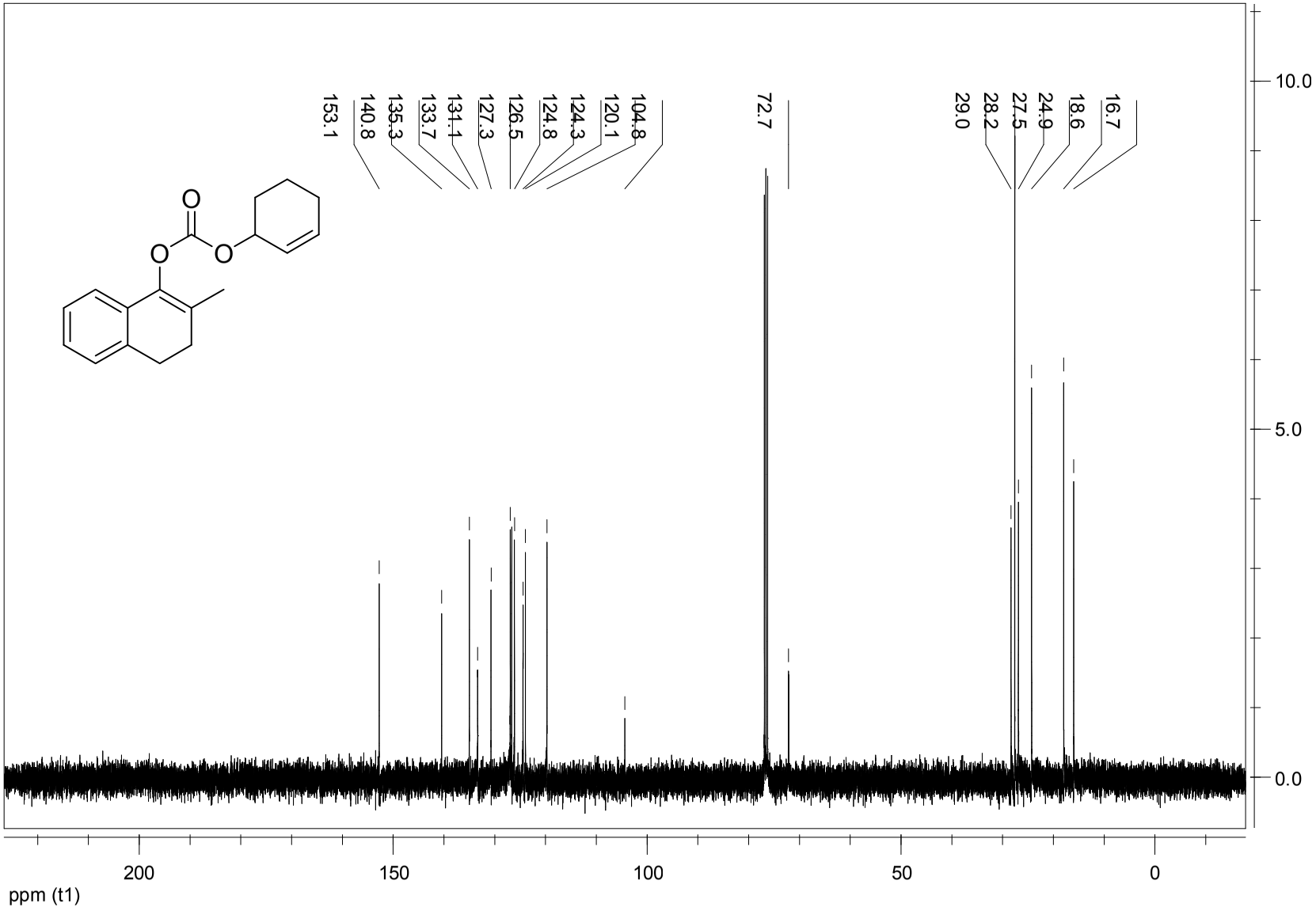

ppm (f1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
9.19
0.85
0.83
1.99
2.00
2.00
2.93
O
O
O
Ph
0
50
100
150
200

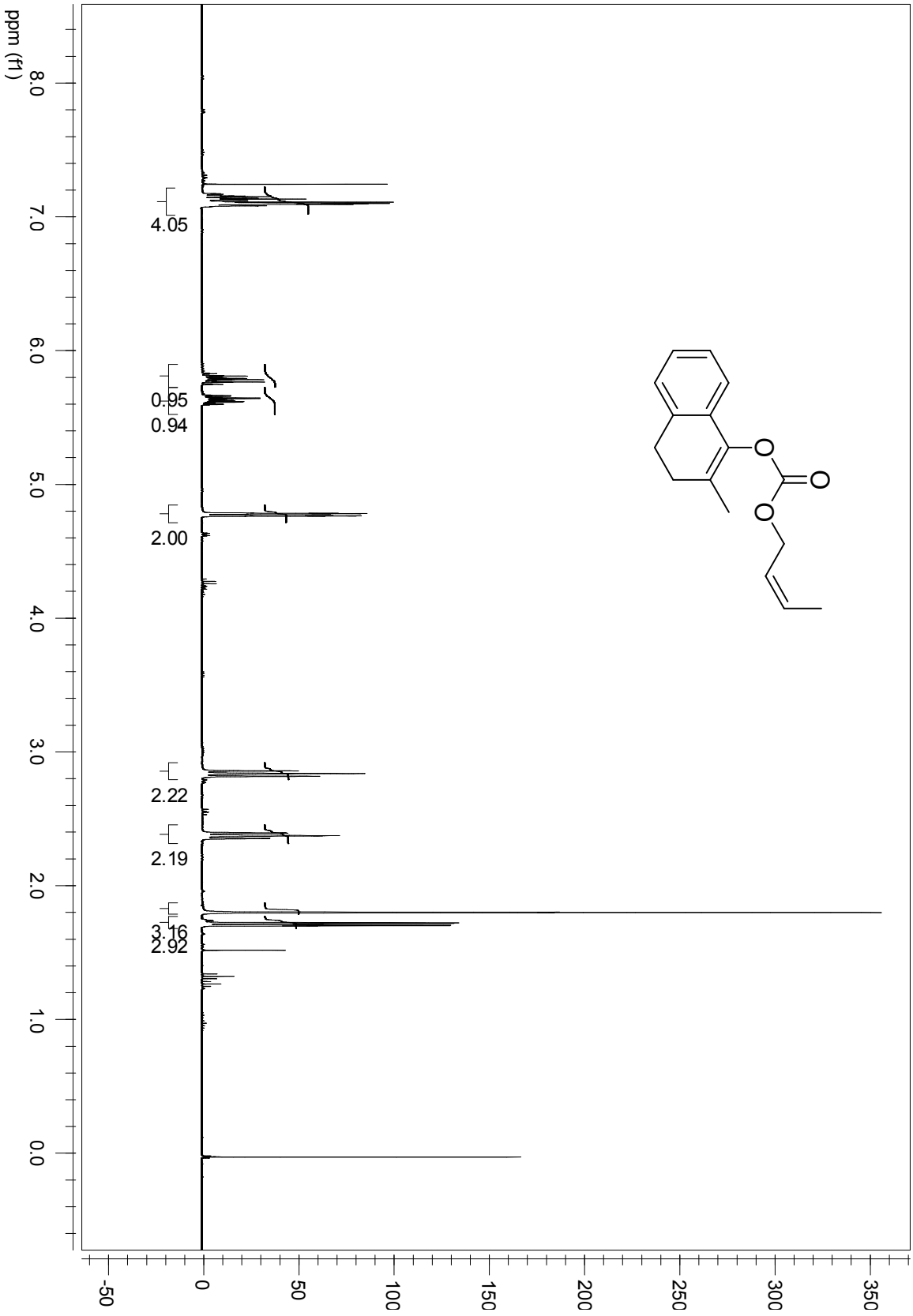

ppm (t1)
0
50
100
150
200
0
1000
2000
3000
4000
5000
6000
O
O
O
Ph
153.3
140.8
136.1
135.3
135.2
130.9
128.7
128.3
127.4
127.2
126.8
126.5
124.6
122.2
120.1
69.1
29.0
27.5
16.7

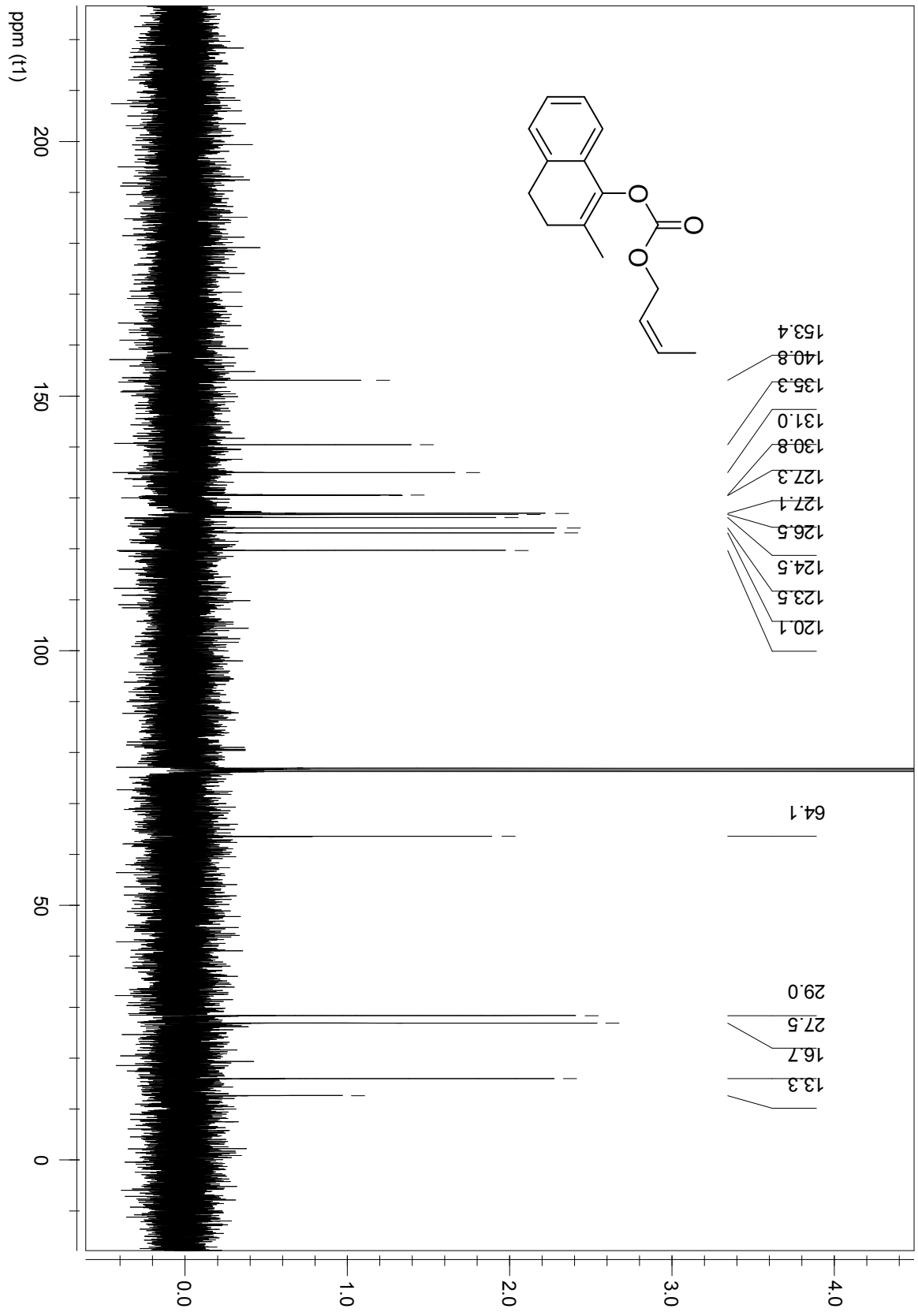

ppm (f1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
3.64
0.86
1.71
0.78
2.00
1.99
5.87
0
100
200
300
400
500
600
O
O
O

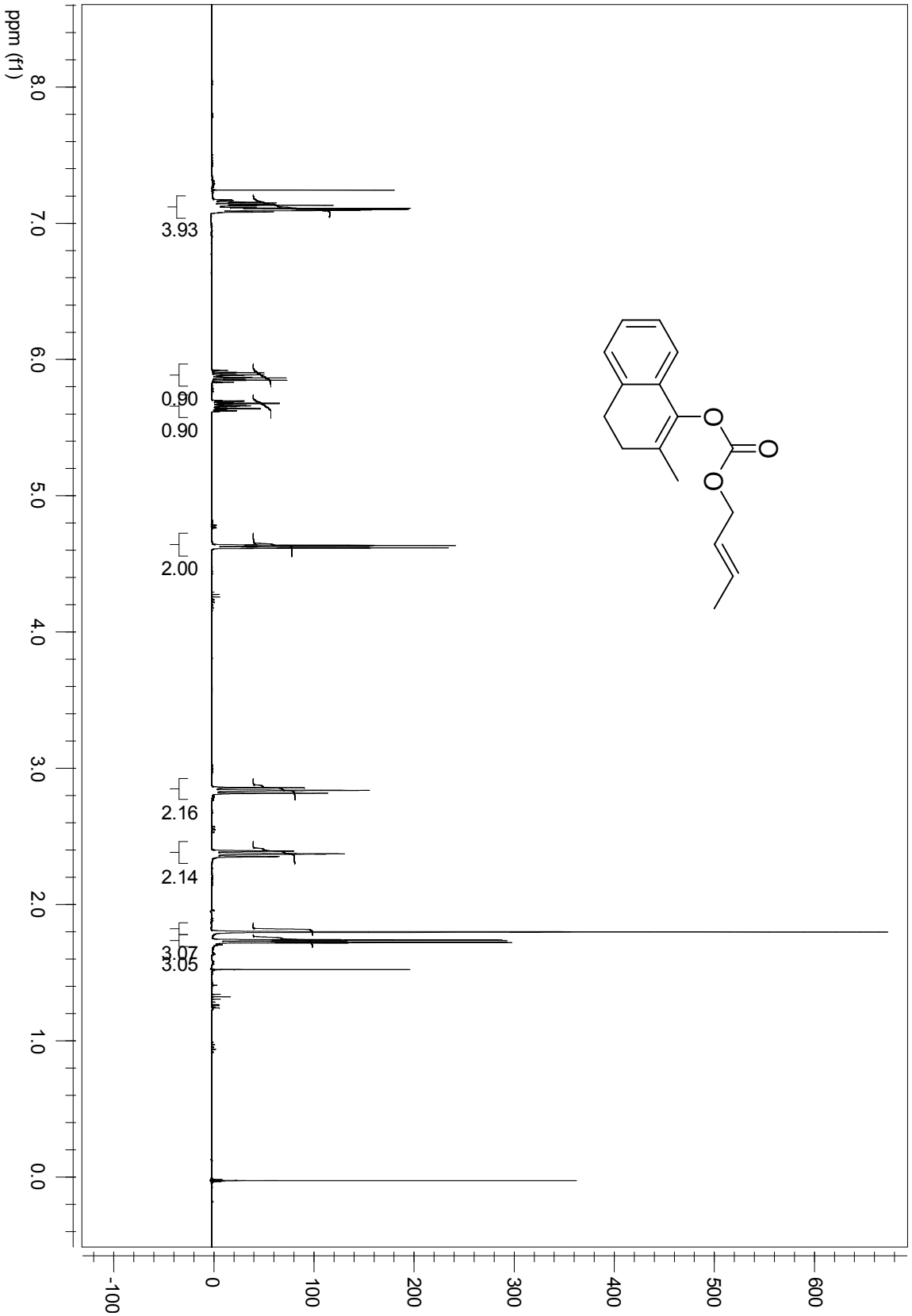

ppm (t1)
200
150
100
50
0
0
1000
2000
3000
4000
5000
153.3
146.2
136.4
133.8
130.4
128.1
127.6
126.5
124.7
120.7
115.2
72.7
28.2
27.5
24.9
22.1
18.6

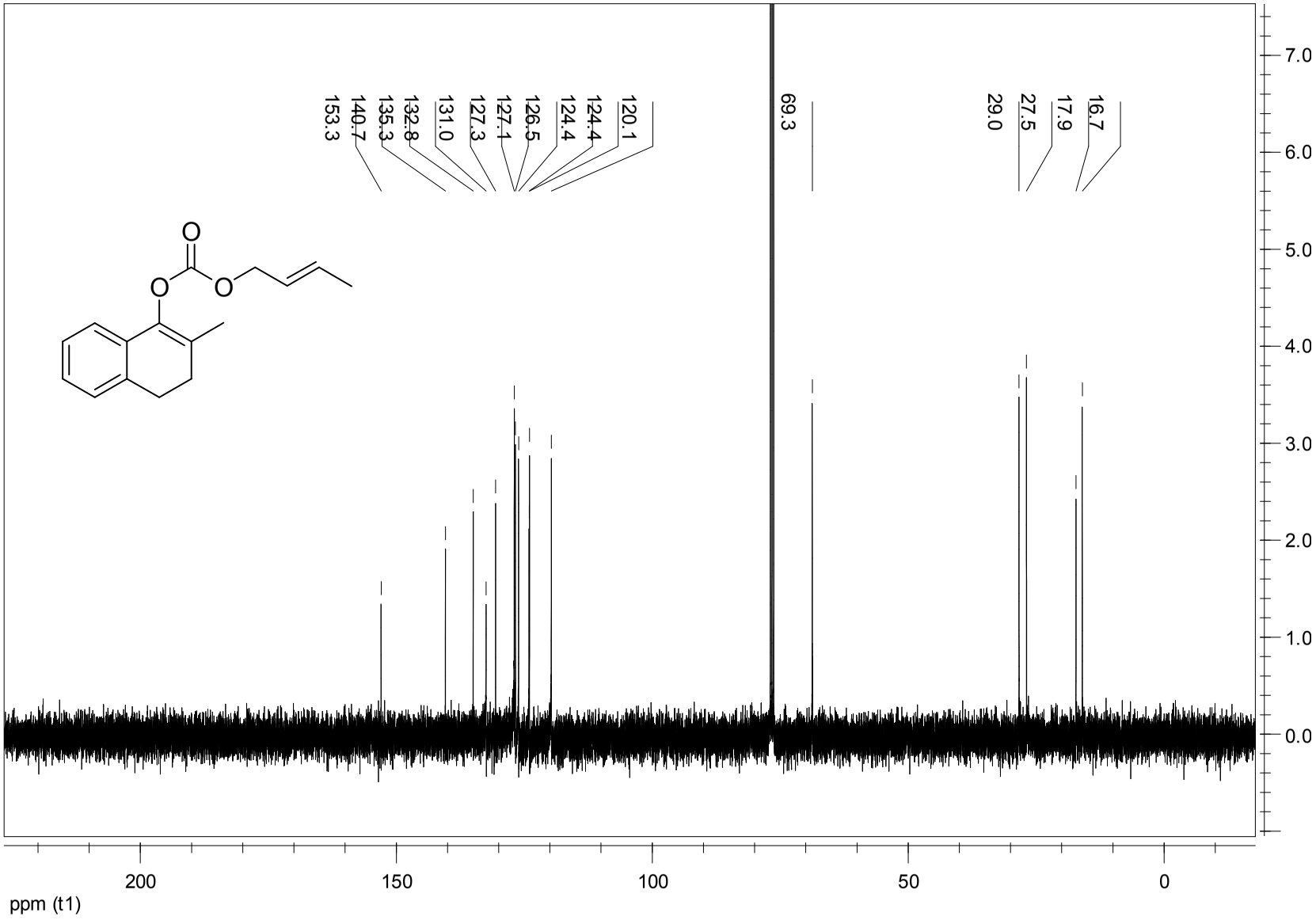

ppm (f1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
3.71
0.90
0.89
0.89
2.00
1.98
8.97
0
100
200
300
400
500
600
700

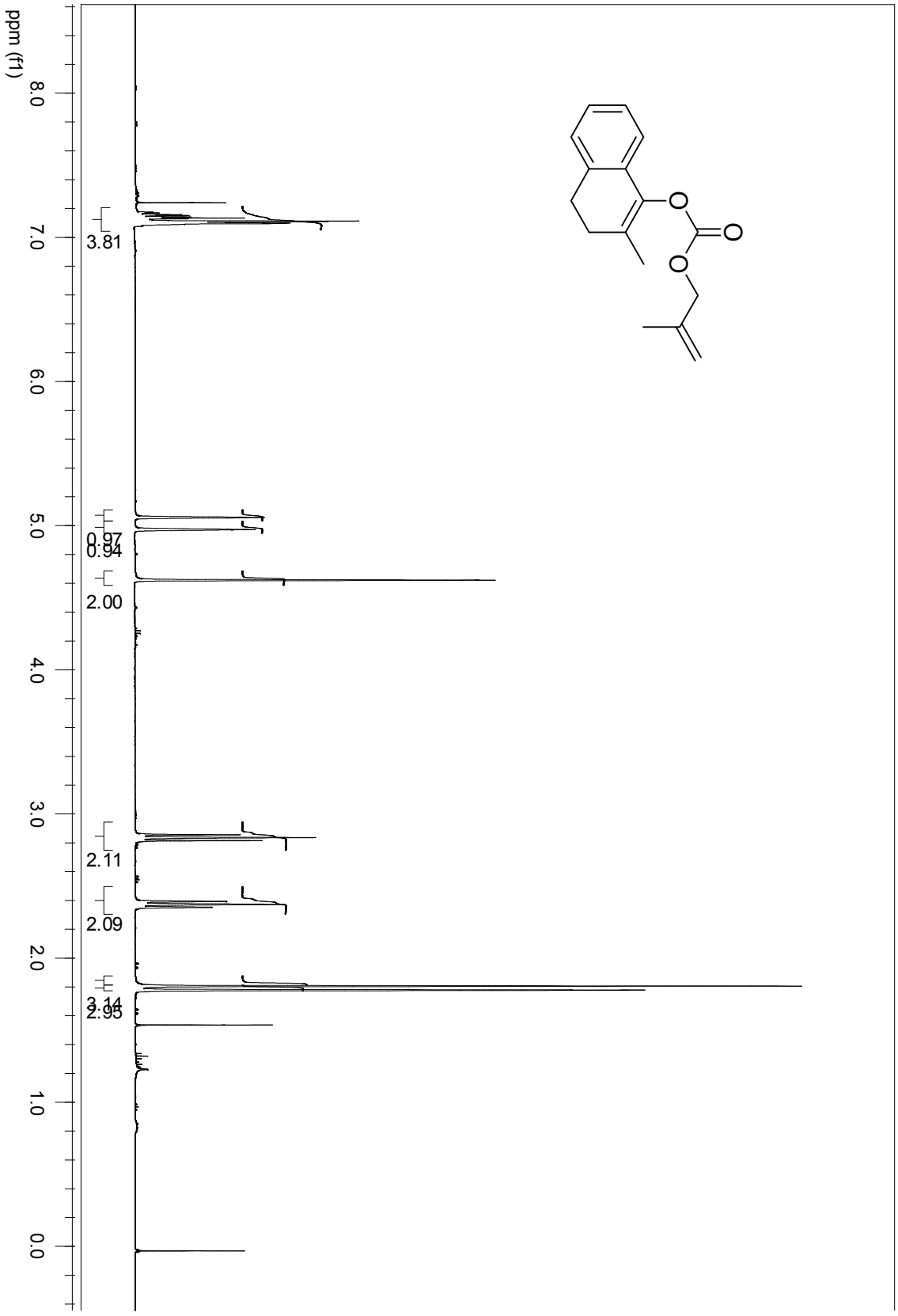

153.1
140.8
135.3
133.7
131.1
127.3
126.5
124.8
124.3
120.1
104.8
72.7
29.0
28.2
27.5
24.9
18.6
16.7
O
O
O
10.0
5.0
0.0
200
150
100
50
0
ppm (t1)


ppm (f1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
4.05
0.95
0.94
2.00
2.22
2.19
3.16
2.92
-50
0
50
100
150
200
250
300
350

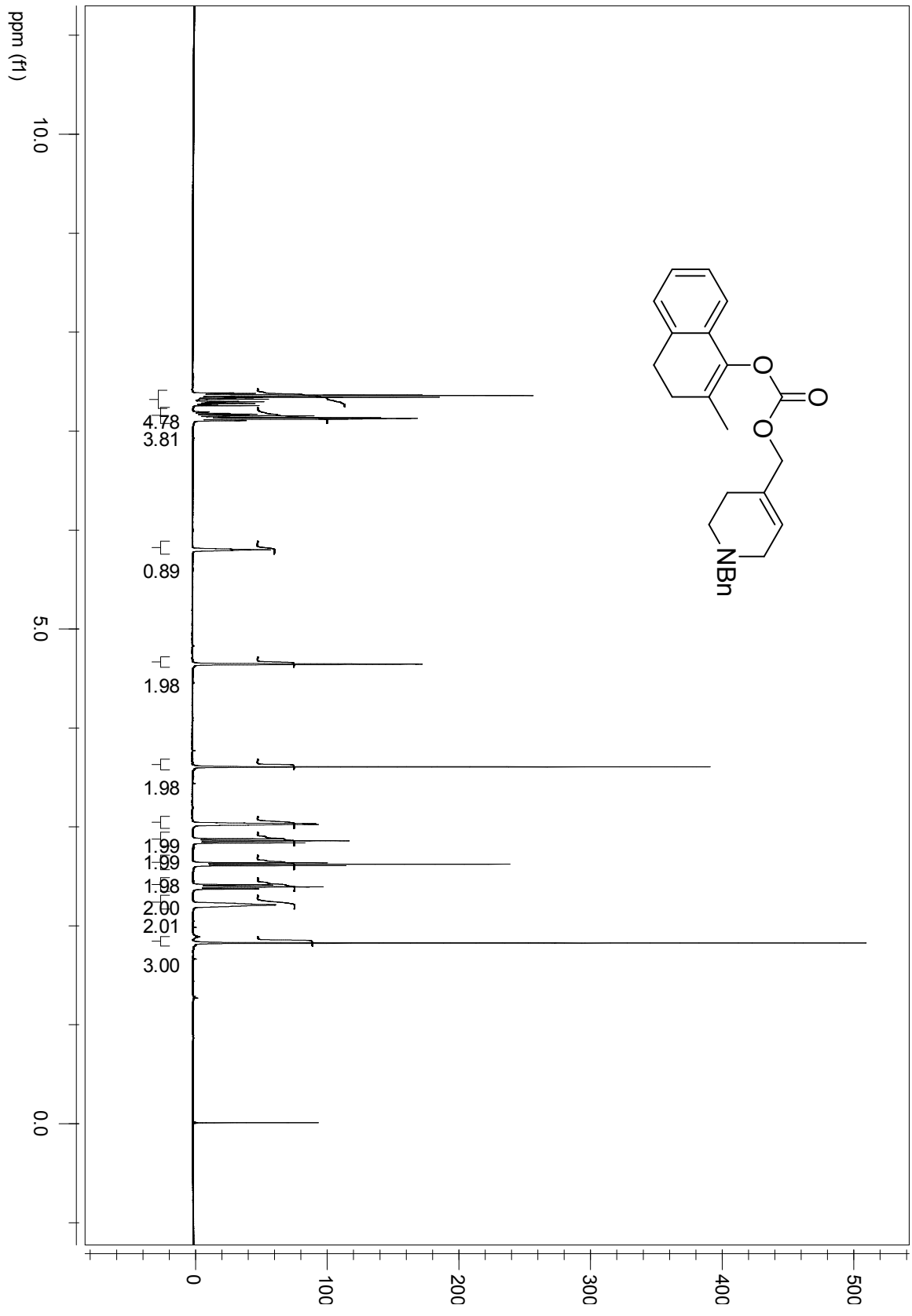

ppm (t1)
0
50
100
150
200
0.0
1.0
2.0
3.0
4.0
64.1
29.0
27.5
16.7
13.3
120.1
123.5
124.5
126.5
127.1
127.3
130.8
131.0
135.3
140.8
153.4
O
O
O

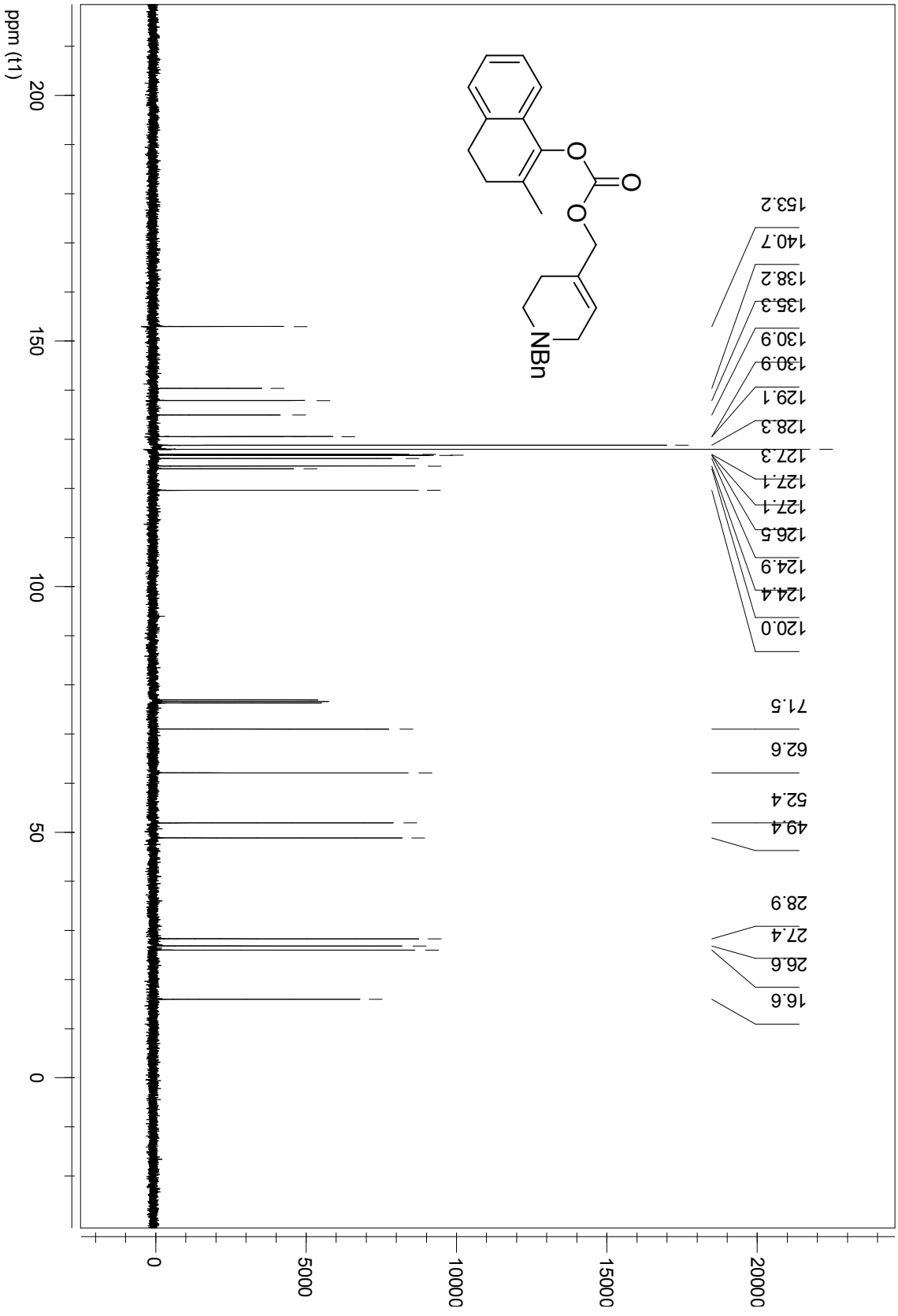

ppm (f1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
3.93
0.90
0.90
2.00
2.16
2.14
3.07
3.05
-100
0
100
200
300
400
500
600

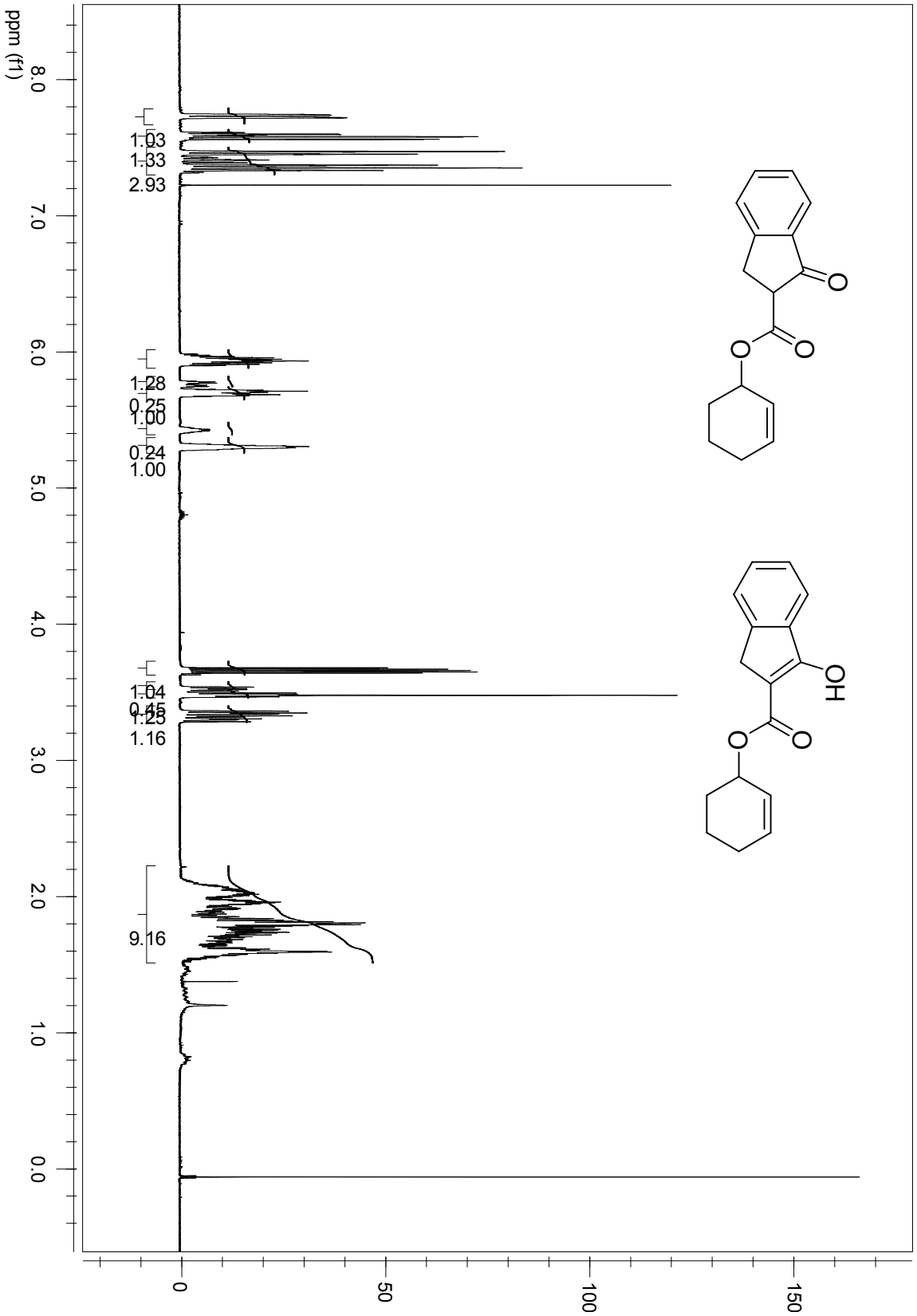

153.3
140.7
135.3
132.8
131.0
127.3
127.1
126.5
124.4
124.4
120.1
69.3
29.0
27.5
17.9
16.7
O
O
O
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
200
150
100
50
0
ppm (t1)

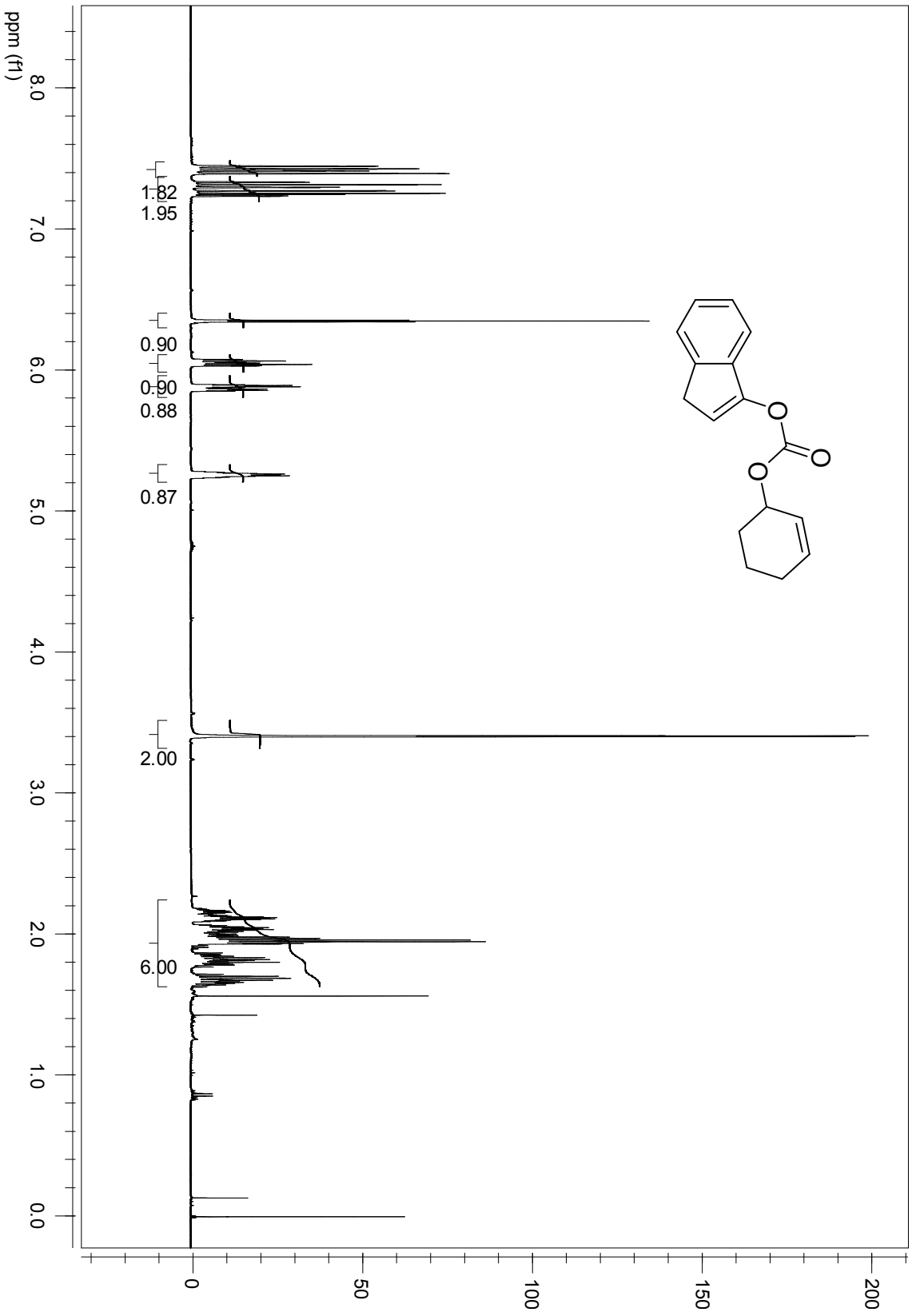

ppm (f1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
3.81
0.97
0.94
2.00
2.11
2.09
3.14
2.95
O
O
O

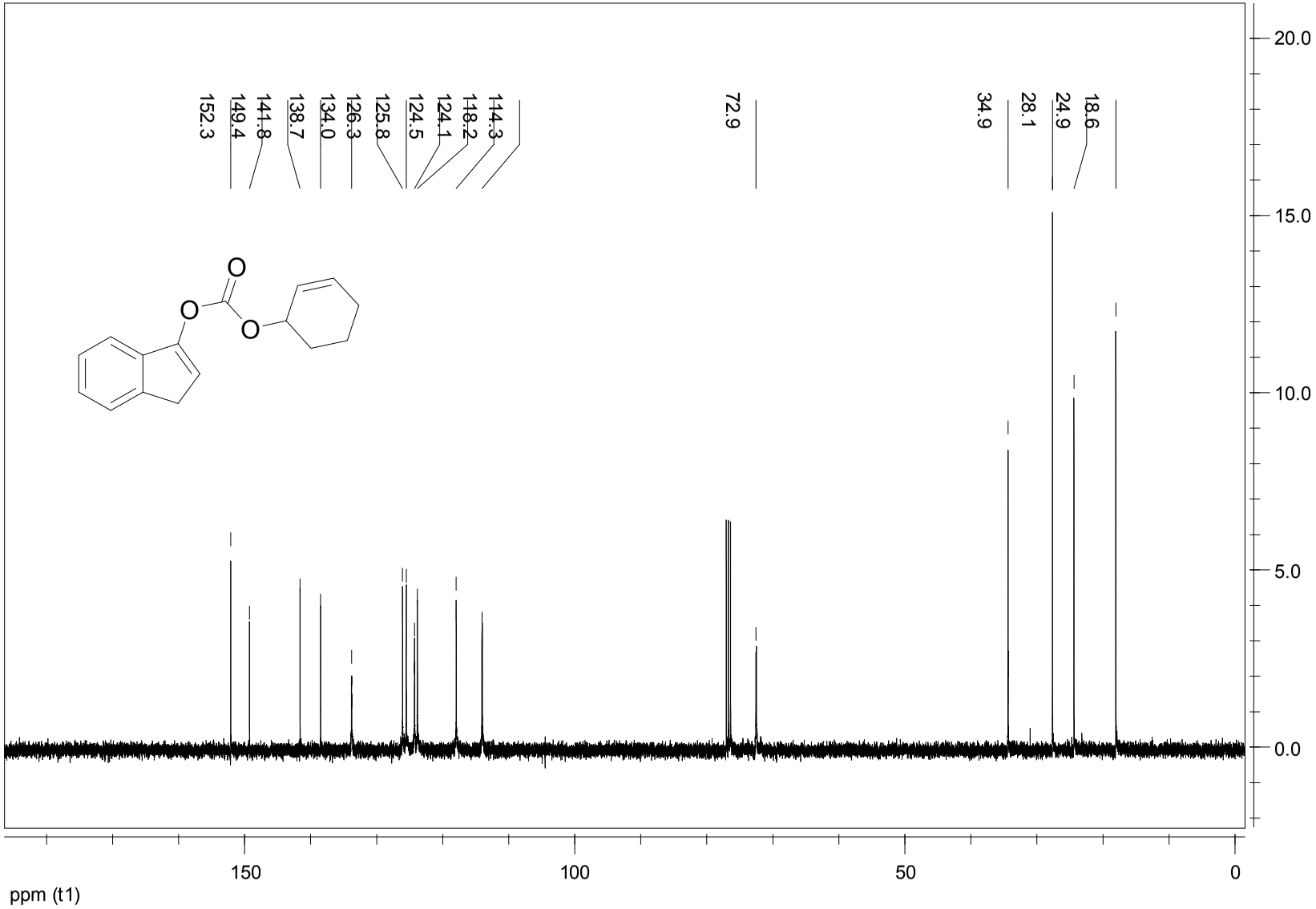
ppm (t1)
200
150
100
50
0
0
1000
2000
3000
4000
5000
6000
153.2
140.7
139.3
135.3
130.9
127.4
127.1
126.5
124.5
120.0
113.7
71.7
29.0
27.5
19.4
16.6
O
O
O

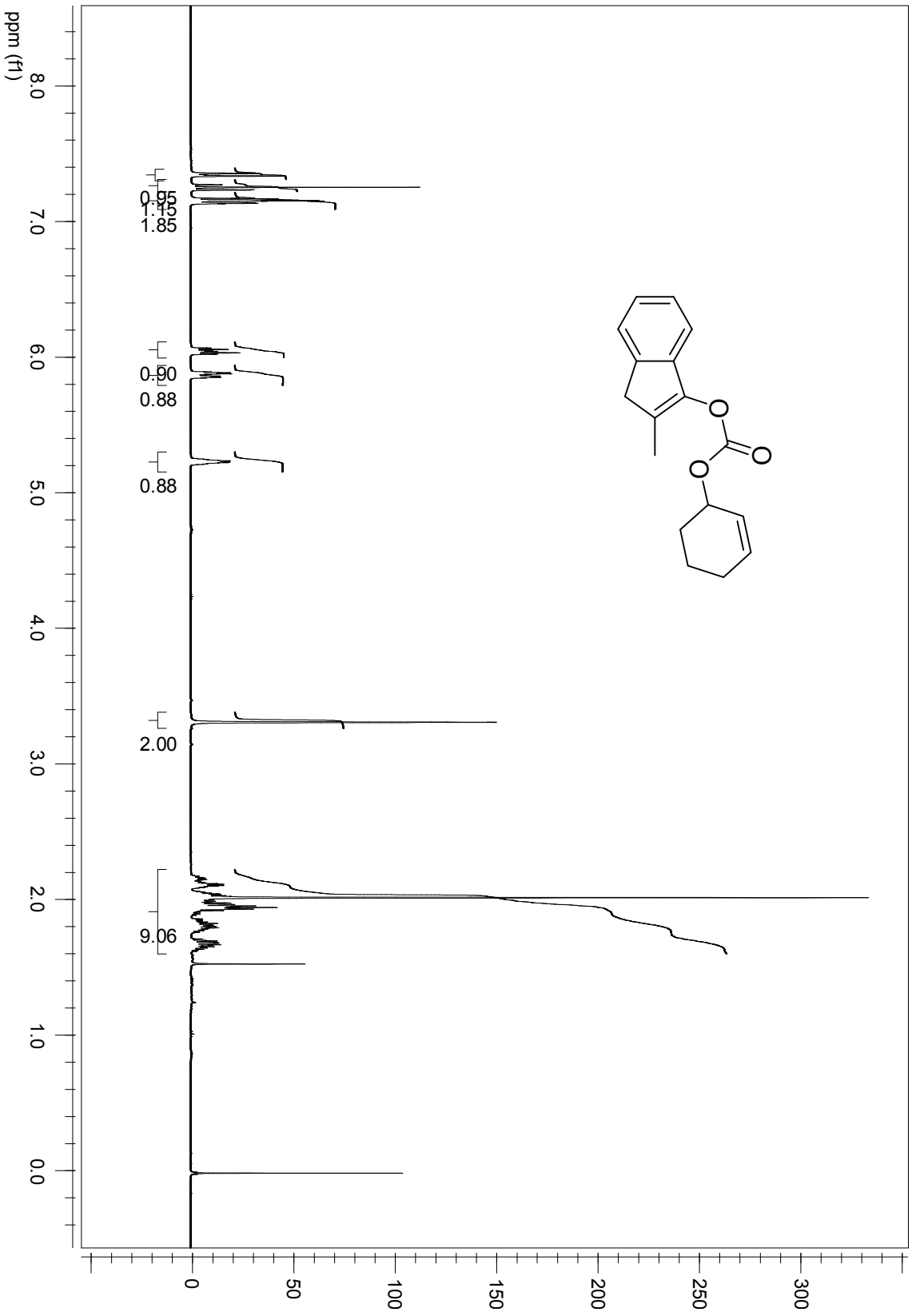

ppm (f1)
10.0
5.0
0.0
0
100
200
300
400
500
4.78
3.81
0.89
1.98
1.98
1.99
1.99
1.98
2.00
2.01
3.00
O
O
O
NBn

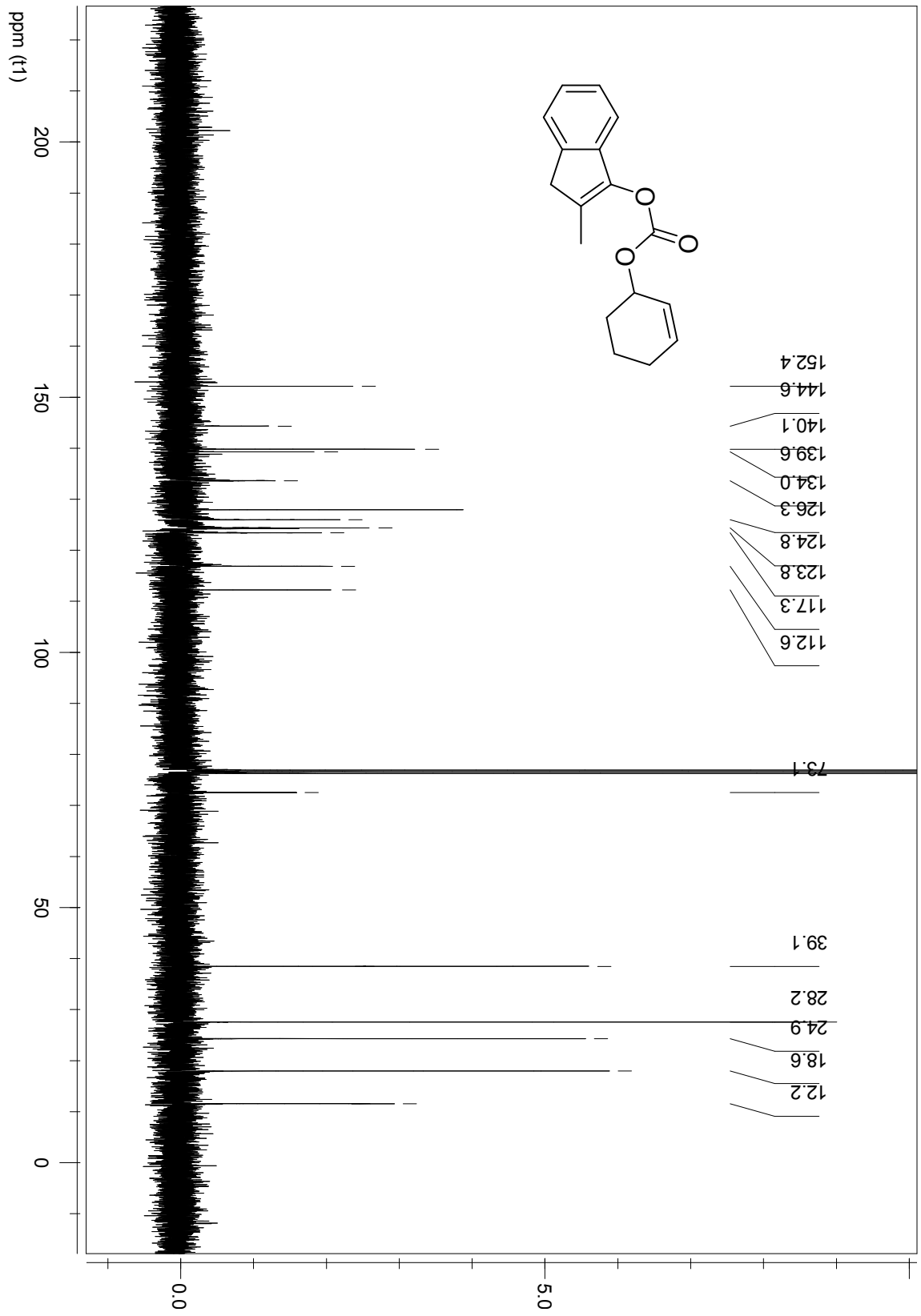

ppm (t1)
200
150
100
50
0
0
5000
10000
15000
20000
153.2
140.7
138.2
135.3
130.9
130.9
129.1
128.3
127.3
127.1
127.1
126.5
124.9
124.4
120.0
71.5
62.6
52.4
49.4
28.9
27.4
26.6
16.6
NBn
O
O
O

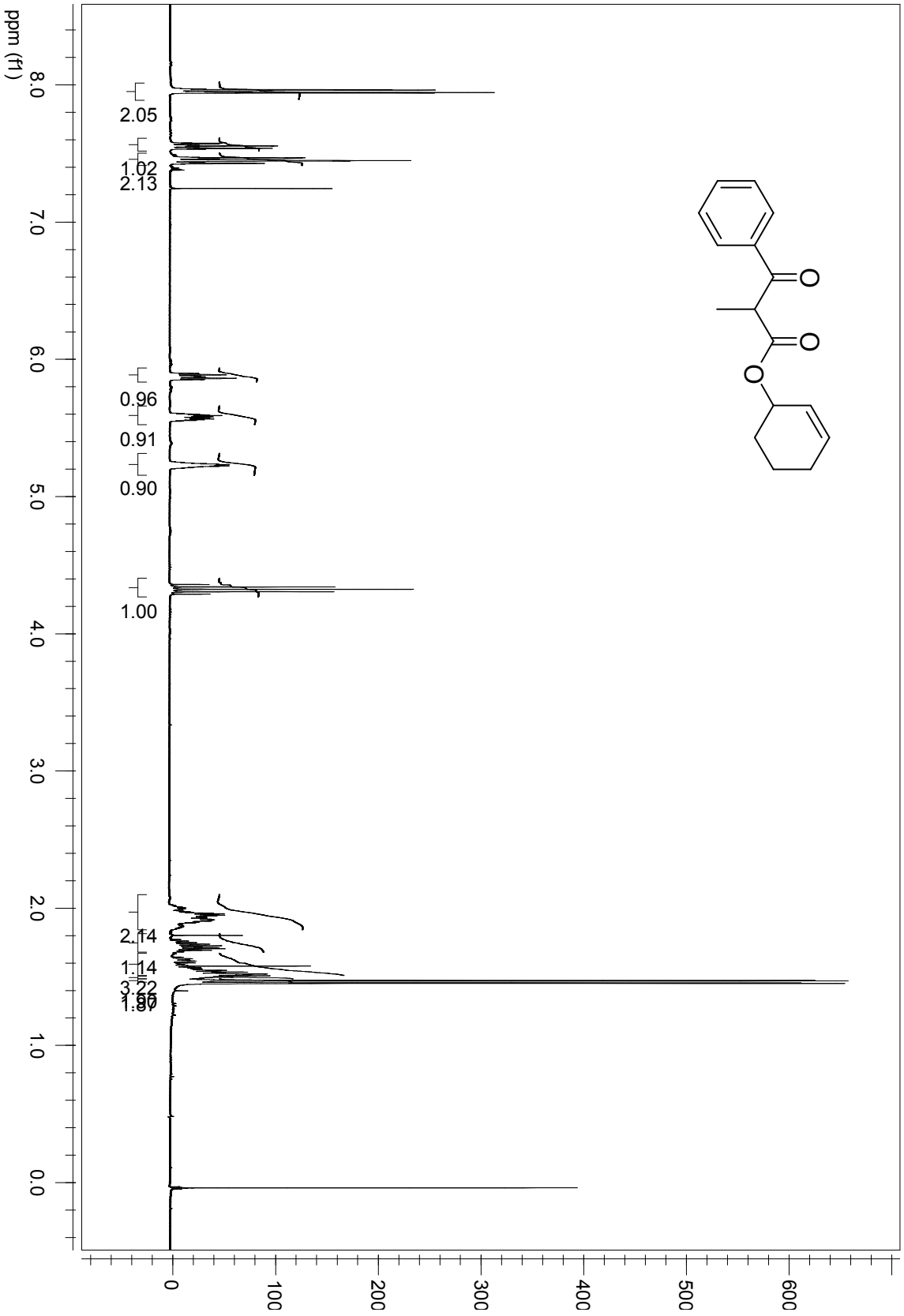

ppm (f1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
1.03
1.33
2.93
1.28
0.25
1.00
0.24
1.00
1.04
0.45
1.23
1.16
9.16
O
O
O
OH
O
O
0
50
100
150

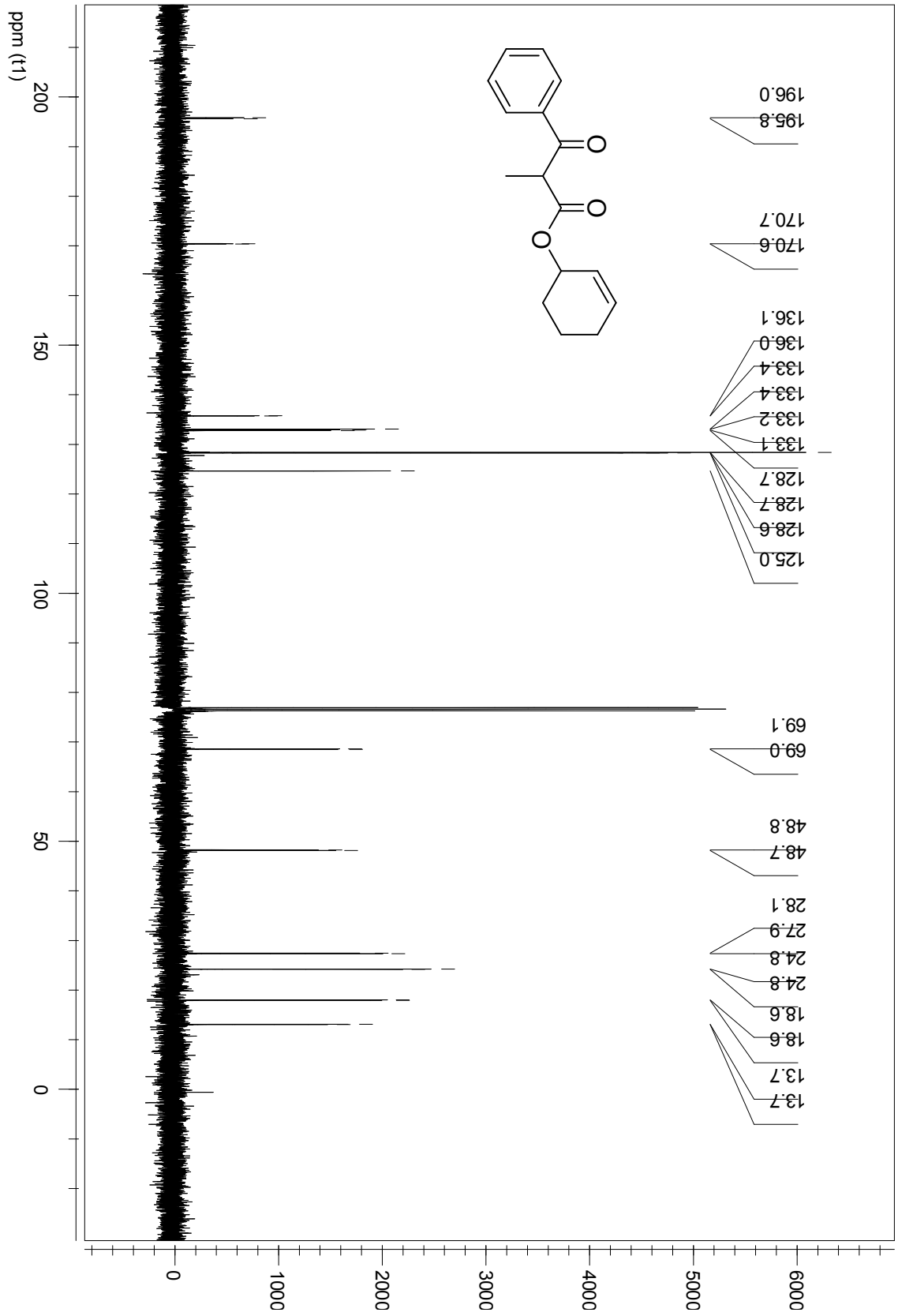

ppm (f1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
1.82
1.95
0.90
0.90
0.88
0.87
2.00
6.00
0
50
100
150
200
O
O
O

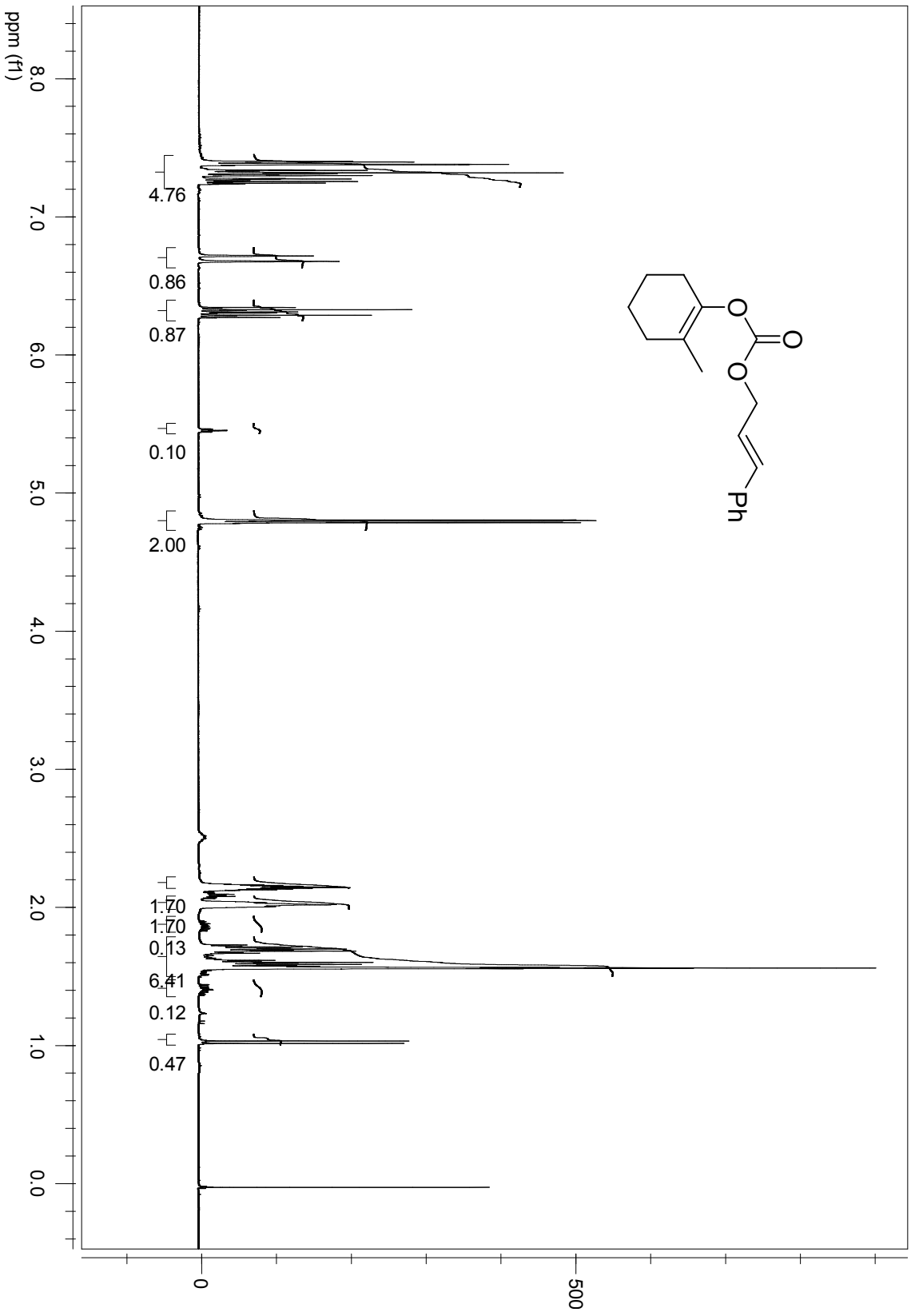

152.3
149.4
141.8
138.7
134.0
126.3
125.8
124.5
124.1
118.2
114.3
72.9
34.9
28.1
24.9
18.6
O
O
O
20.0
15.0
10.0
5.0
0.0
150
100
50
0
ppm (t1)

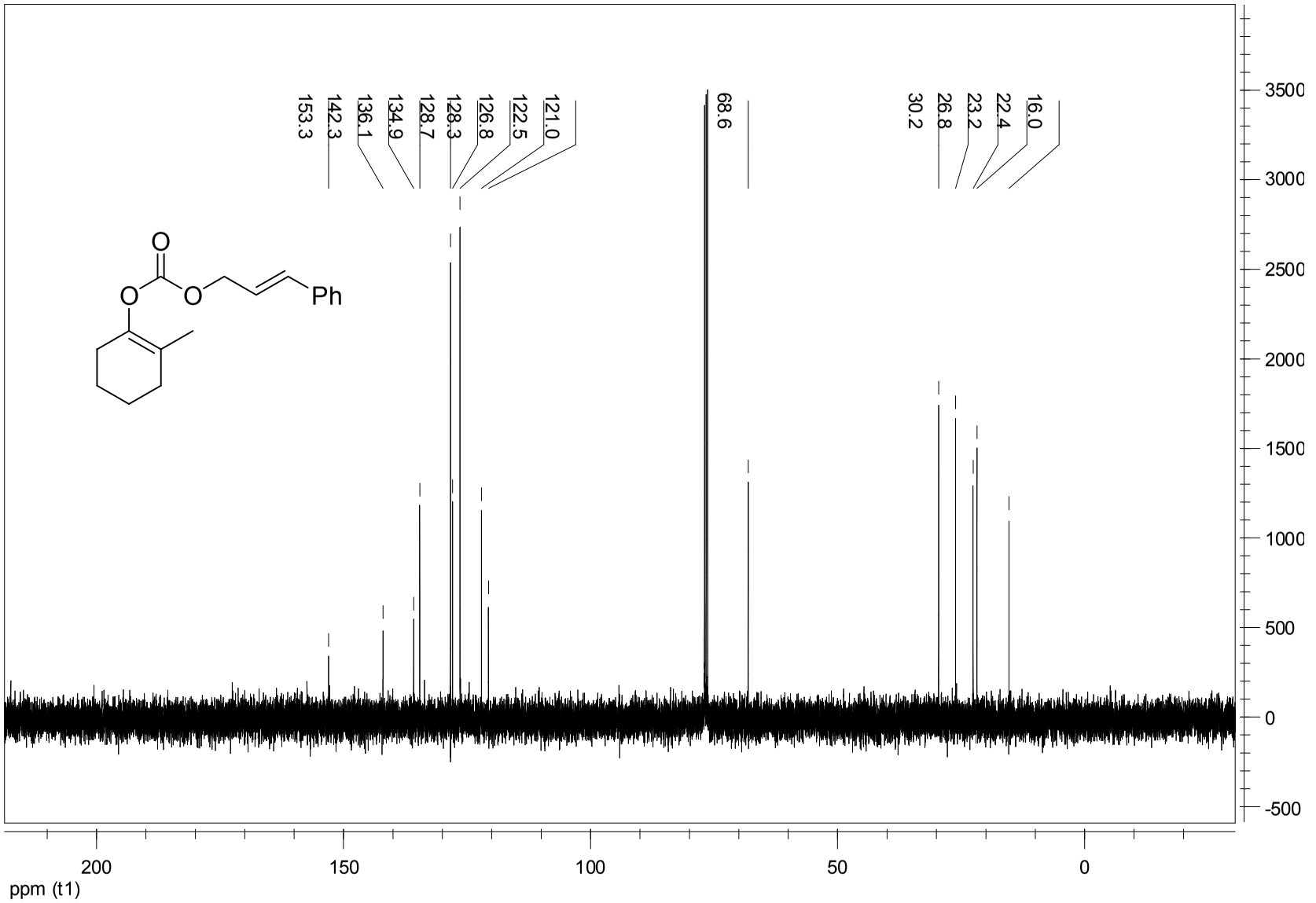

ppm (f1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0.95
1.85
0.90
0.88
0.88
2.00
9.06
0
50
100
150
200
250
300

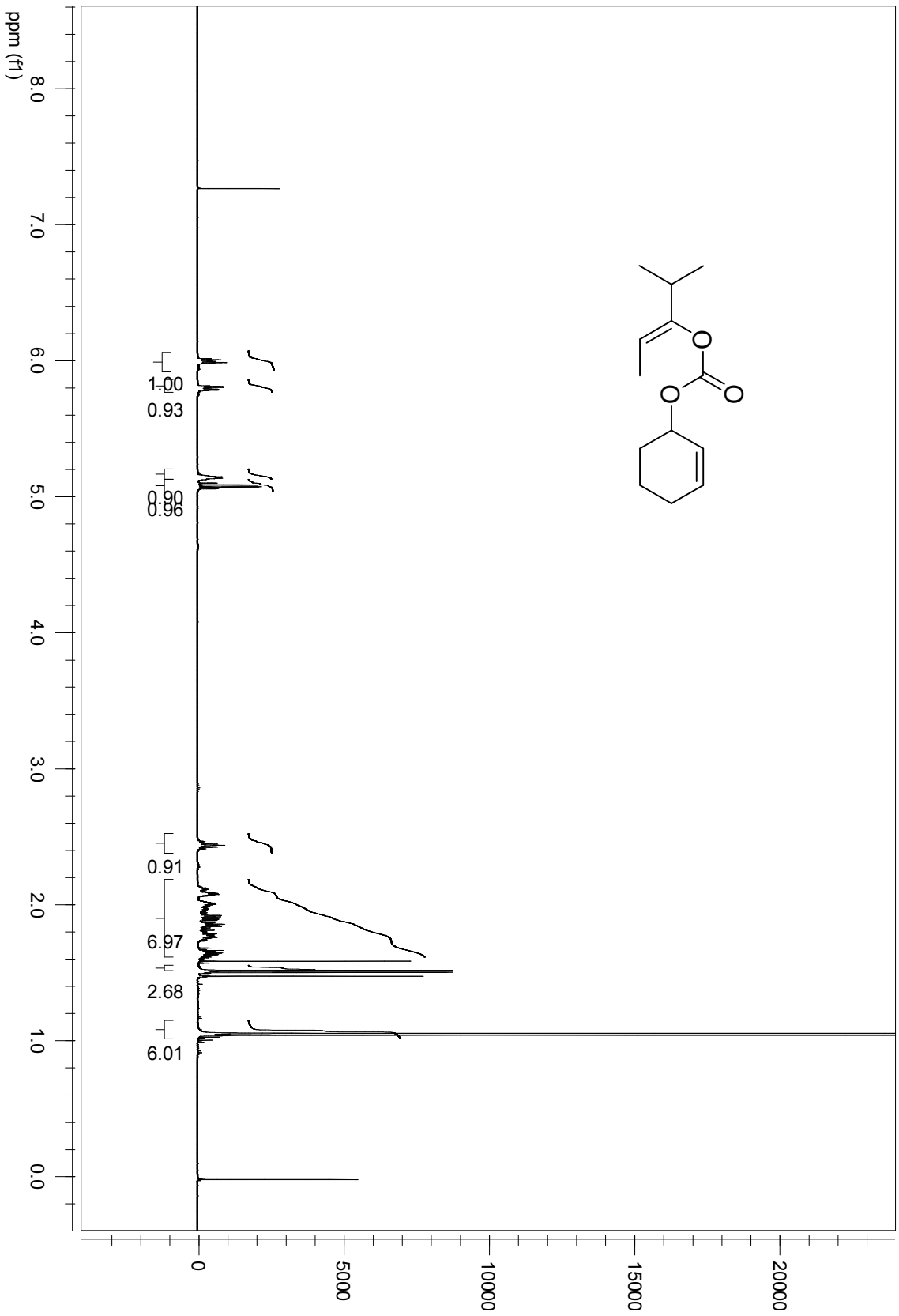

ppm (t1)
200
150
100
50
0
0.0
5.0
152.4
144.6
140.1
139.6
134.0
126.3
124.8
123.8
117.3
112.6
79.1
39.1
28.2
24.9
18.6
12.2

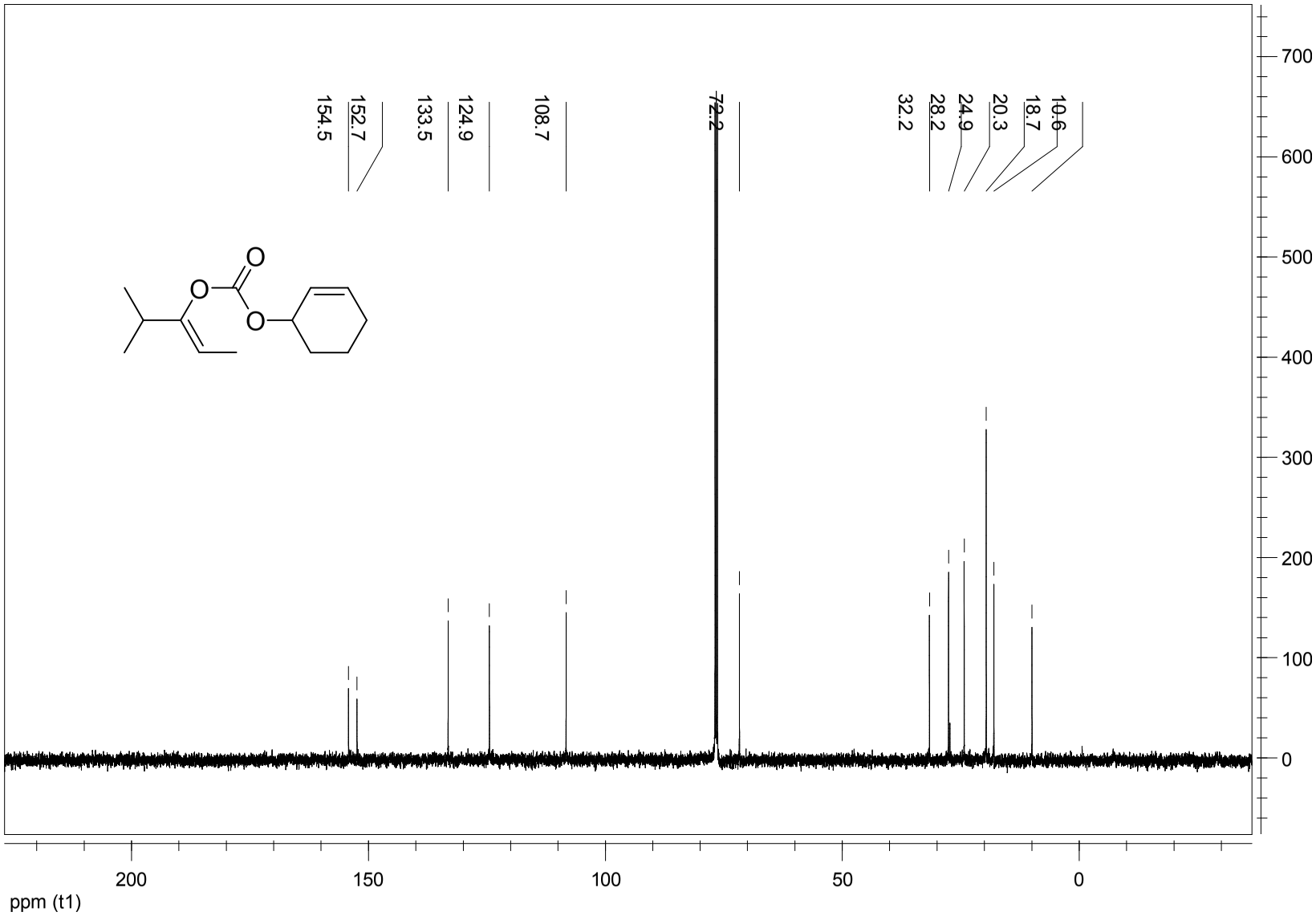

ppm (f1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
2.05
1.02
2.13
0.96
0.91
0.90
1.00
2.14
1.14
3.22
0
100
200
300
400
500
600
O
O
O

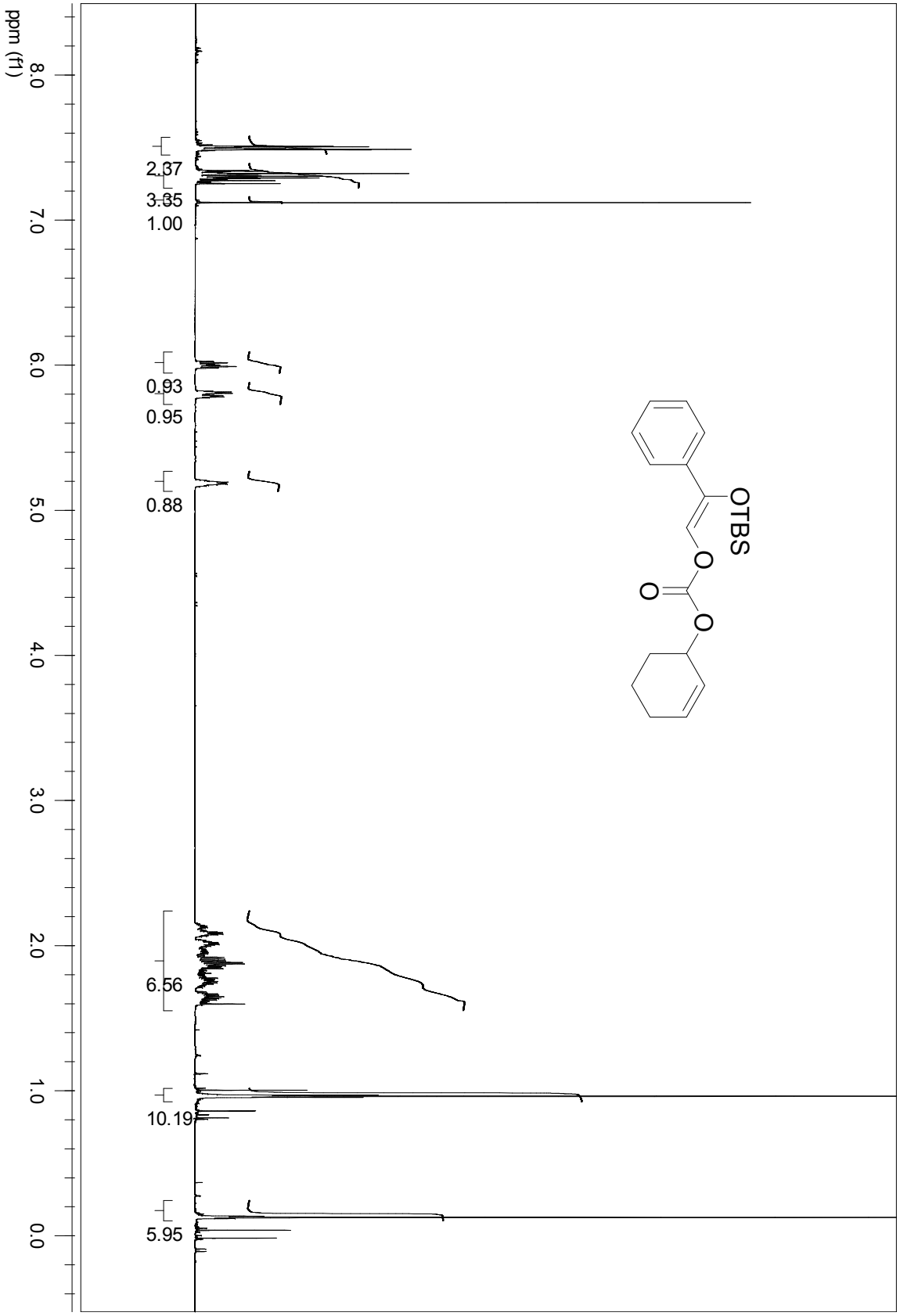

ppm (t1)
200
150
100
50
0
0
1000
2000
3000
4000
5000
6000
O
O
O
196.8
196.0
170.6
170.7
136.1
136.0
133.4
133.4
133.2
133.1
128.7
128.7
128.6
125.0
69.0
69.1
48.7
48.8
28.1
27.9
24.8
24.8
18.6
18.6
13.7
13.7

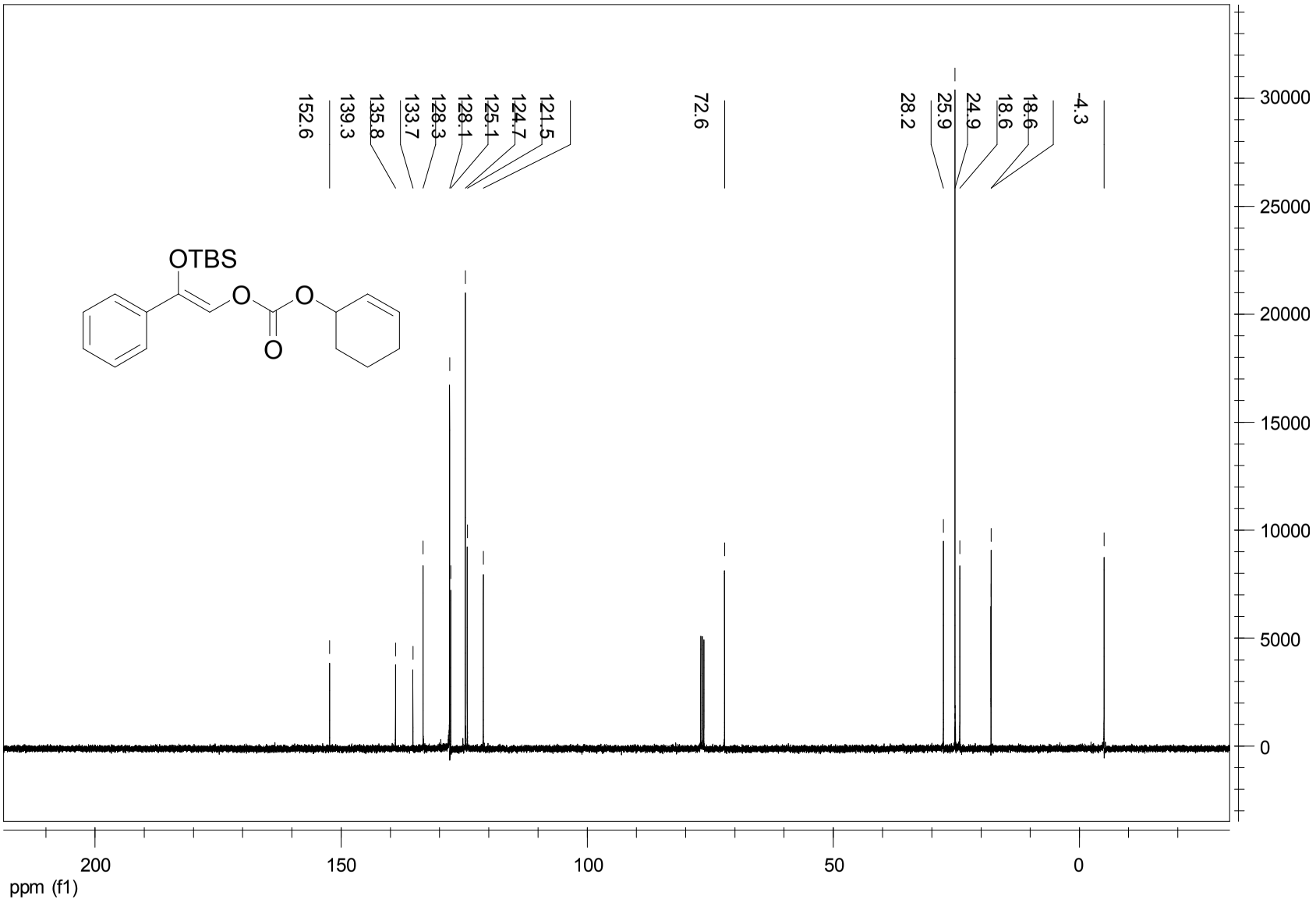

ppm (f1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
4.76
0.86
0.87
0.10
2.00
1.70
1.70
0.13
6.41
0.12
0.47
O
O
O
Ph
0
500

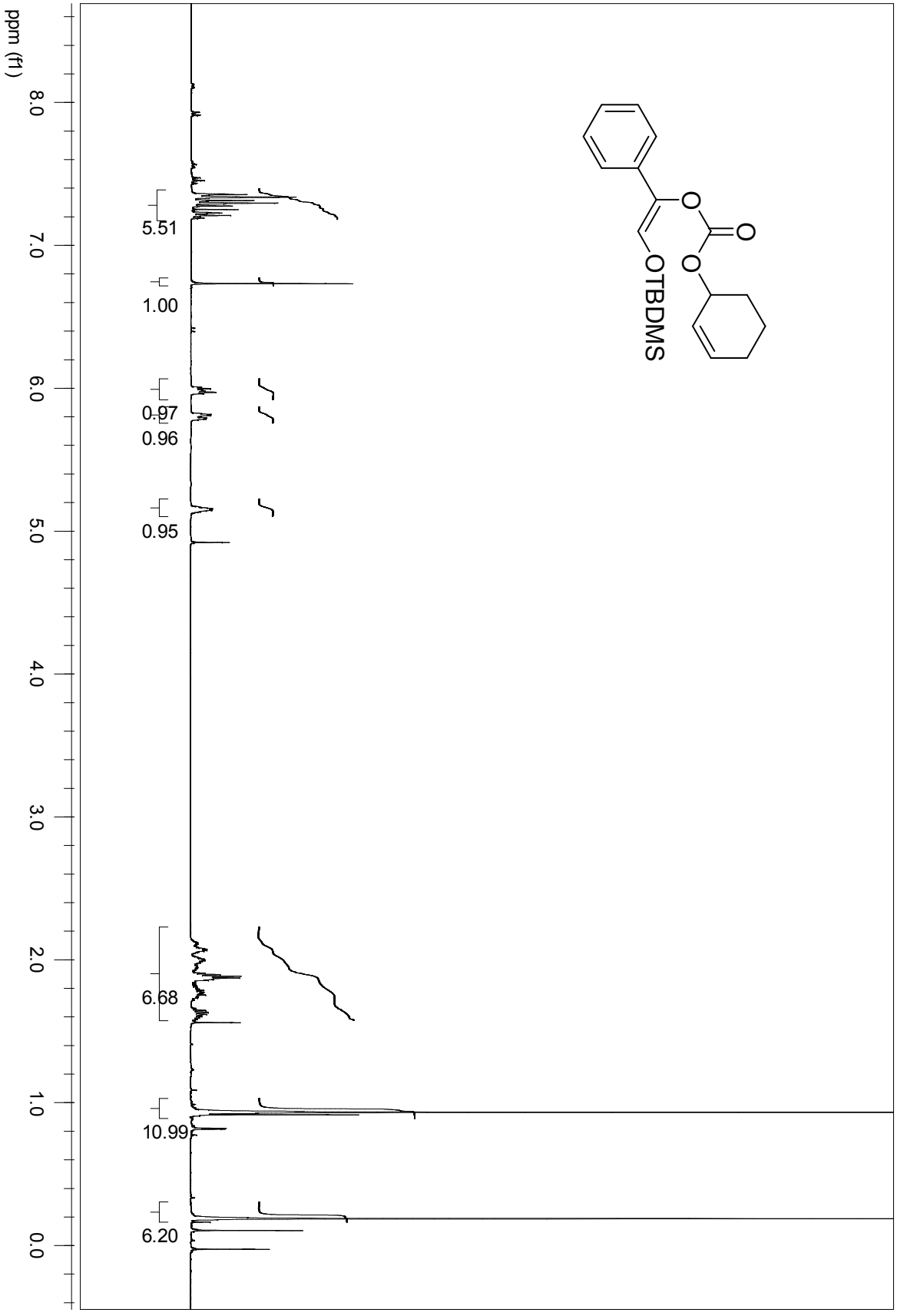

153.3
142.3
136.1
134.9
128.7
128.3
126.8
122.5
121.0
68.6
30.2
26.8
23.2
22.4
16.0
O
O
O
Ph
3500
3000
2500
2000
1500
1000
500
0
-500
200
150
100
50
0
ppm (t1)


ppm (f1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
1.00
0.93
0.90
0.96
0.91
6.97
2.68
6.01
0
5000
10000
15000
20000
O
O
O


154.5
152.7
133.5
124.9
108.7
72.2
32.2
28.2
24.9
20.3
18.7
10.6
O
O
O
700
600
500
400
300
200
100
0
200
150
100
50
0
ppm (t1)


ppm (f1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
2.37
3.35
1.00
0.93
0.95
0.88
6.56
10.19
5.95
OTBS
O
O
O


152.6
139.3
135.8
133.7
128.3
128.1
125.1
124.7
121.5
72.6
28.2
25.9
24.9
18.6
18.6
-4.3
OTBS
O
O
O
30000
25000
20000
15000
10000
5000
0
200
150
100
50
0
ppm (f1)


ppm (f1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
5.51
1.00
0.97
0.96
0.95
6.68
10.99
6.20
OTBDMS
O
O
O

ppm (f1)
200
150
100
50
0
0
5000
OTBDMS
O
O
O
-5.159
18.237
18.594
24.925
25.515
28.205
72.478
123.233
124.916
127.250
128.547
130.593
133.470
133.500
134.738
152.658