

SUPPLEMENTARY MATERIAL

A Practical Method for Oxazole Synthesis by Cycloisomerization of Propargyl Amides

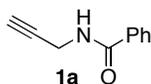
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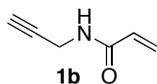
Experimental procedures and spectral data for **2a-e**, **3a,b,d,e**, **4a-d**, **5a,b**, **6a,b**, **7a,b**, **10**, **11**, **13**, **14**, **15**, **16**, **19**, **20**, **21**, **22**, **23**, including copies of ^1H and ^{13}C NMR spectra.

General: All reactions were performed in flamed-dried or oven-dried glassware under a dry nitrogen atmosphere. THF was distilled over Na/benzophenone ketyl; CH₂Cl₂ and Et₃N were distilled from CaH₂. Reactions were monitored by TLC analysis (EM Science pre-coated silica gel 60 F₂₅₄ plates, 250 μM layer thickness) and visualization was accomplished with a 254 nm UV light and basic KMnO₄ solution (1.5 g of KMnO₄, 10 g of K₂CO₃ and 2.5 mL of 5% aqueous NaOH in 150 mL of water). Flash chromatography on SiO₂ (Merck, EM-9385-9, 230-400 mesh) or preparative thin-layer chromatography (Analtech, Uniplate, silica gel GF, 20 x 20 cm, 1000 μm) was used to purify the crude reaction mixtures. Silica gel mediated reactions were performed on SiO₂ from E. Merck (EM-9385-9, 230-400 mesh). Melting points were determined using a Laboratory Devices Mel-Tem II. Infrared spectra were determined on a Nicolet Avatar 360 FT-IR spectrometer. NMR spectra were recorded in CDCl₃ (unless otherwise noted) at either 300 MHz (¹H NMR) or 75 MHz (¹³C NMR) using Bruker Avance 300 instruments with XWIN-NMR software. Mass spectra were obtained on a Micromass Autospec double focusing instrument.

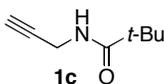


***N*-Prop-2-ynylbenzamide (1a).**¹ To a solution of 1.00 g (18.2 mmol) of propargylamine in 40 mL of CH₂Cl₂ was added 2.57 g (18.3 mmol) of benzoyl chloride and 3.00 mL (22.0 mmol) of Et₃N at 0 °C. The reaction mixture was stirred at rt for 1 h, poured into 1.0 M HCl (100 mL) and extracted with CHCl₃ (2 x 50 mL). The combined organic layers were washed with brine (100 mL), dried (MgSO₄), and concentrated under reduced pressure to afford 2.85 g (98%) of **1a** as a white solid: ¹H NMR δ 7.79 (d, 2 H, *J* = 7.3 Hz), 7.56-7.40 (m, 3 H), 6.35 (br, 1 H), 4.26 (dd, 2 H, *J* = 5.1, 2.5 Hz), 2.29 (t, 1 H, *J* = 2.4 Hz).

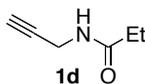
¹ Arcadi, A.; Cacchi, S.; Cascia, L.; Fabrizi, G.; Marinelli, F. *Org. Lett.* **2001**, 3, 2501-2504.



N-Prop-2-ynylacrylamide (1b). According to the procedure used for the preparation of **1a**, 0.995 g (18.0 mmol) of propargylamine, 35.0 mL (25.1 mmol) of Et₃N, and 1.45 mL (17.8 mmol) of acryloyl chloride gave 0.921 g (47%) of **1b** as a white solid: R_f 0.26 (65% hexanes:EtOAc); ¹H NMR δ 6.32 (d, 1 H, *J* = 16.9 Hz), 6.11 (dd, 1 H, *J* = 17.0, 10.2 Hz), 5.82 (br, 1 H), 5.70 (d, 1 H, *J* = 10.2 Hz), 4.14 (dd, 2 H, *J* = 5.1, 2.5 Hz), 2.25 (t, 1 H, *J* = 2.3 Hz); ¹³C NMR δ 165.4, 130.2, 127.3, 79.3, 71.7, 29.3; HRMS (EI) *m/z* calculated for C₆H₇NO (M-H) 109.0528, found 109.0524.



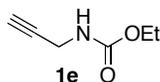
2,2-Dimethyl-N-prop-2-ynylpropionamide (1c).² According to the procedure used for the preparation of **1a**, 0.997 g (18.1 mmol) of propargylamine, 3.50 mL (25.1 mmol) of Et₃N, and 2.16 g (17.9 mmol) of trimethylacetyl chloride gave 2.16 g (87%) of **1c** as a white solid: ¹H NMR δ 5.89 (br, 1 H), 4.06 (dd, 2 H, *J* = 5.2, 2.5 Hz), 2.25 (q, 2 H, *J* = 7.6 Hz), 2.24 (t, 1 H, *J* = 2.5 Hz), 1.17 (t, 3 H, *J* = 7.5 Hz).



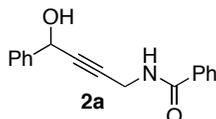
N-Prop-2-ynylpropionamide (1d).³ According to the procedure used for the preparation of **1a**, 1.02 g (18.2 mmol) of propargylamine, 3.50 mL (25.1 mmol) of Et₃N, and 1.50 mL (17.3 mmol) of propionyl chloride gave 1.73 g (90%) of **1d** as a pale yellow oil: R_f 0.20 (50% hexanes:EtOAc); ¹H NMR δ 6.19 (br, 1 H), 4.04 (dd, 2 H, *J* = 5.2, 2.6 Hz), 2.25 (t, 1 H, *J* = 2.6 Hz), 1.22 (s, 9 H).

² Gao, G.; Sanda, F.; Masuda, T. *Macromolecules* **2003**, *36*, 3938-3943.

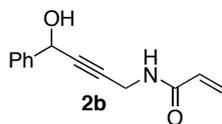
³ Nomura, R.; Tabei, J.; Masuda, T. *Macromolecules* **2002**, *35*, 2955-2961.



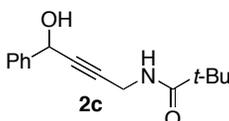
Prop-2-ynylcarbamic acid ethyl ester (1e): According to the procedure used for the preparation of **1a**, 1.01 g (18.3 mmol) of propargylamine, 2.00 g (18.5 mmol) of ethyl chloroformate, and 3.00 mL (21.5 mmol) of Et₃N gave 2.12 g (91%) of **1e** as a yellow oil: ¹H NMR δ 4.80 (br, 1 H), 4.15 (q, 2 H, *J* = 7.1 Hz), 3.98 (dd, 2 H, *J* = 5.3, 2.0 Hz), 2.24 (t, 1 H, *J* = 2.5 Hz), 1.25 (t, 3 H, *J* = 7.1 Hz).



***N*-(4-Hydroxy-4-phenylbut-2-ynyl)-benzamide (2a).** A solution of 2.85 g (17.9 mmol) of **1a** in 60 mL of THF was treated at -78 °C with 23.0 mL (36.8 mmol) of a 1.0 M solution of *n*-BuLi in THF, stirred for 30 min at -78 °C, treated with a solution of 1.59 g (15.0 mmol) of benzaldehyde in 10 mL of THF, warmed to 0 °C over 1 h, and stirred for 3.5 h. The reaction mixture was quenched with 1.0 M HCl (100 mL) and extracted with EtOAc (2 x 100 mL). The combined organic layers were washed with brine (100 mL), dried (MgSO₄), concentrated under reduced pressure, and chromatographed on SiO₂ (60% hexanes:EtOAc) to give 2.15 g (52%) of **2a** as a white solid: *R*_f 0.33 (50% hexanes:EtOAc); mp 91.5-92.0 °C (hexanes/EtOAc); IR (neat) 3370, 3217, 2244, 1634, 1538, 1435, 1304, 1125, 1015 cm⁻¹; ¹H NMR δ 7.74 (d, 2 H, *J* = 7.1 Hz), 7.58-7.28 (m, 8 H), 6.58 (br, 1 H), 5.47 (s, 1 H), 4.30-4.25 (m, 2 H, *J* = 5.1, 1.6 Hz), 3.21 (br 1 H); ¹³C NMR δ 167.3, 140.4, 133.6, 131.8, 128.6, 128.6, 128.4, 127.1, 126.6, 83.6, 82.1, 64.5, 30.1; MS (EI) *m/z* (rel intensity) 265 (M⁺, 0.8), 264 (4), 248 (4), 247 (5), 231 (3), 219 (2), 191 (4), 162 (2), 122 (18), 105 (100); HRMS (EI) *m/z* calculated for C₁₇H₁₄NO₂ (M-H) 264.1025, found 264.1028.

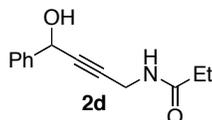


***N*-(4-Hydroxy-4-phenylbut-2-ynyl)acrylamide (2b)**. A solution of 0.511 g (4.68 mmol) of **1b** in 30 mL of THF was treated at $-78\text{ }^{\circ}\text{C}$ with 5.80 mL (9.30 mmol) of a 1.6 M solution of *n*-BuLi in hexane, stirred for 15 min at $-78\text{ }^{\circ}\text{C}$, treated with a solution of 0.481 g (4.53 mmol) of benzaldehyde in 1 mL of THF, warmed to $0\text{ }^{\circ}\text{C}$ over 1 h, and stirred for 1 h. The reaction mixture was quenched with 1.0 M HCl (40 mL) and extracted with EtOAc (2 x 25 mL). The combined organic layers were washed with brine (40 mL), dried (MgSO_4), concentrated under reduced pressure, and chromatographed on SiO_2 (65%-40% hexanes:EtOAc) to afford 0.697 g (71%) of **2b** as a colorless oil: R_f 0.15 (50% hexanes:EtOAc); IR (neat) 3313, 3066, 2924, 2243, 1655, 1542, 1451, 1248, 1133, 1004 cm^{-1} ; ^1H NMR δ 7.52 (dd, 2 H, $J = 7.8, 1.6$ Hz), 7.43-7.32 (m, 3 H), 6.32 (dd, 1 H, $J = 17.0, 1.3$ Hz), 6.08 (dd, 1 H, $J = 17.0, 10.3$ Hz), 5.70 (br, 1 H), 5.69 (dd, 1 H, $J = 10.3, 1.2$ Hz), 5.49 (br, 1 H), 4.25-4.23 (m, 2 H), 2.17 (br, 1 H); ^{13}C NMR δ 165.6, 140.4, 130.0, 128.6, 128.4, 127.4, 126.6, 83.5, 81.8, 64.3, 29.6; MS (EI) m/z (rel intensity) 215 (M^+ , 3), 214 (18), 198 (7), 197 (15), 169 (12), 168 (10), 144 (47), 143 (30), 131 (26), 116 (25), 115 (49), 105 (23); HRMS (EI) m/z calculated for $\text{C}_{13}\text{H}_{12}\text{NO}_2$ (M-H) 214.0868, found 214.0867.

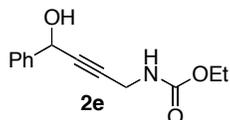


***N*-(4-Hydroxy-4-phenylbut-2-ynyl)-2,2-dimethylpropionamide (2c)**. A solution of 1.05 g (7.57 mmol) of **1c** in 30 mL of THF was treated at $-78\text{ }^{\circ}\text{C}$ with 10.0 mL (16.0 mmol) of a 1.6 M solution of *n*-BuLi in THF, stirred for 30 min at $-78\text{ }^{\circ}\text{C}$, treated with a solution of 0.852 g (8.03 mmol) of benzaldehyde, warmed to rt over 1 h, and stirred for 2.5 h. The reaction mixture was quenched with 1.0 M HCl (100 mL) and extracted with EtOAc (2 x 75 mL). The combined organic layers were washed with brine (50 mL), dried (MgSO_4), concentrated under reduced pressure, and chromatographed on SiO_2 (80%-35% hexanes:EtOAc) to give 0.932 g (50%) of **2c** as a yellow oil: R_f 0.32 (50% hexanes:EtOAc); IR (neat) 3357, 3063, 2967, 2249, 1655, 1528,

1452, 1208, 1130, 1002 cm^{-1} ; ^1H NMR δ 7.51 (d, 2 H, $J = 6.9$ Hz), 7.40-7.27 (m, 3 H), 6.01 (br, 1 H), 5.46 (s, 1 H), 4.08-4.06 (m, 2 H), 3.59 (br, 1 H), 1.17 (s, 9 H); ^{13}C NMR δ 178.5, 140.6, 128.5, 128.3, 126.6, 83.5, 82.2, 64.3, 38.6, 29.9, 27.4; MS (EI) m/z (rel intensity) 245 (M^+ , 0.7), 244 (5), 228 (3), 227 (4), 212 (5), 199 (2), 184 (3), 144 (18), 105 (43), 102 (32); HRMS (EI) m/z calculated for $\text{C}_{15}\text{H}_{18}\text{NO}_2$ (M-H) 244.1338, found 244.1336.

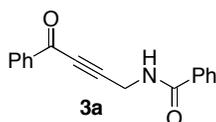


***N*-(4-Hydroxy-4-phenylbut-2-ynyl)-propionamide (2d)**. A solution of 0.0970 g (0.873 mmol) of **1d** in 5 mL of THF was treated at -78 °C with 1.80 mL (1.80 mmol) of a 1.0 M solution of LiHMDS in THF, stirred for 15 min at -78 °C, treated with a solution of 0.0967 mg (0.911 mmol) of benzaldehyde in 1 mL of THF, warmed to 0 °C over 1 h, and stirred for 1 h. The reaction mixture was quenched with 1.0 M HCl (10 mL) and extracted with EtOAc (2 x 10 mL). The combined organic layers were washed with brine (10 mL), dried (MgSO_4), concentrated under reduced pressure, and purified by chromatography on SiO_2 (85%-40% hexanes:EtOAc) to yield 0.0994 g (52%) of **2d** as a yellow oil: R_f 0.13 (50% hexanes:EtOAc); IR (neat) 3293, 3064, 2979, 2244, 1654, 1542, 1458, 1233, 1133, 1000 cm^{-1} ; ^1H NMR δ 7.50 (dd, 2 H, $J = 8.1, 1.7$ Hz), 7.40-7.27 (m, 3 H), 6.12 (br, 1 H), 5.45 (s, 1 H), 4.07-4.05 (m, 1 H), 3.75 (br, 1 H), 2.15 (q, 2 H, $J = 7.6$ Hz), 1.10 (t, 3 H, $J = 7.6$ Hz); ^{13}C NMR δ 173.9, 140.6, 128.6, 128.3, 126.6, 83.3, 82.1, 64.3, 29.5, 29.3, 9.6; MS (EI) m/z (rel intensity) 217 (M^+ , 6), 216 (47), 200 (17), 161 (15), 160 (10), 144 (88), 143 (100), 116 (47), 115 (77), 105 (43); HRMS (EI) m/z calculated for $\text{C}_{13}\text{H}_{14}\text{NO}_2$ (M-H) 216.1025, found 216.1023.

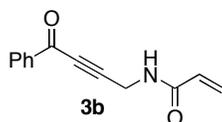


(4-Hydroxy-4-phenylbut-2-ynyl)-carbamic acid ethyl ester (2e). A solution of 2.12 g (16.7 mmol) of **1e** in 60 mL of THF was treated at -78 °C with 21.0 mL (33.6 mmol) of a 1.6 M

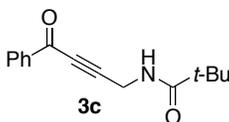
solution of *n*-BuLi in hexane, stirred for 15 min at $-78\text{ }^{\circ}\text{C}$, treated with a solution of 1.49 g (14.1 mmol) of benzaldehyde in 1 mL of THF, warmed to $0\text{ }^{\circ}\text{C}$ over 1 h, and stirred for 1 h. The reaction mixture was quenched with 1.0 M HCl (100 mL) and extracted with EtOAc (2 x 100 mL). The combined organic layers were washed with brine (100 mL), dried (MgSO_4), concentrated under reduced pressure, and purified by column chromatography on SiO_2 (65%-50% hexanes:EtOAc) to yield 2.73 g (83%) of **2e** as a pale yellow oil: R_f 0.39 (50% hexanes:EtOAc); IR (neat) 3337, 3063, 2982, 2238, 1686, 1560, 1451, 1254, 1117, 1041 cm^{-1} ; ^1H NMR δ 7.52 (dd, 2 H, $J = 7.8, 1.3$ Hz), 7.42-7.30 (m, 3 H), 5.47 (s, 1 H), 4.86 (br, 1 H), 4.11 (q, 2 H, $J = 7.1$ Hz), 4.06 (d, 2 H, $J = 5.0$ Hz), 2.42 (br, 1 H), 1.25 (t, 3 H, $J = 7.1$ Hz); ^{13}C NMR δ 156.4, 140.5, 128.6, 128.3, 126.6, 83.2, 82.4, 64.3, 61.4, 31.0, 14.6; MS (EI) m/z (rel intensity) 233 (M^+ , 10), 232 (52), 216 (18), 215 (21), 204 (34), 144 (99), 143 (44), 131 (49), 116 (53), 115 (100), 105 (78); HRMS (EI) m/z calculated for $\text{C}_{13}\text{H}_{14}\text{NO}_3$ (M-H) 232.0974, found 232.0975.



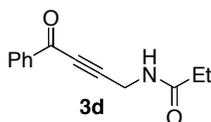
***N*-(4-Oxo-4-phenylbut-2-ynyl)-benzamide (3a)**. A mixture of 0.569 g (2.15 mmol) of **2a** and 1.00 g (2.36 mmol) of Dess-Martin reagent in 10 mL of CH_2Cl_2 was stirred at rt for 15 h, concentrated under reduced pressure, and purified by chromatography on SiO_2 (90%-65% hexanes:EtOAc) to yield 0.404 g (71%) of **3a** as a white solid and 0.085 g (*ca.* 15%) of crude oxazole **4a** as a yellow oil. **3a**: R_f 0.32 (65% hexanes:EtOAc); mp $111.0\text{-}111.5\text{ }^{\circ}\text{C}$ (hexanes/EtOAc); IR (neat) 3270, 2238, 2203, 1635, 1526, 1406, 1266, 966 cm^{-1} ; ^1H NMR δ 8.13 (dd, 2 H, $J = 7.8, 1.3$ Hz), 7.83 (dd, 2 H, $J = 7.7, 1.4$ Hz), 7.66-7.40 (m, 6 H), 6.61 (br, 1 H), 4.57 (d, 2 H, $J = 5.3$ Hz); ^{13}C NMR δ 177.7, 167.2, 136.3, 134.4, 133.4, 132.1, 129.7, 128.7, 128.7, 127.1, 89.8, 81.3, 30.0; MS (EI) m/z (rel intensity) 263 (M^+ , 6), 235 (6), 206 (6), 160 (27), 158 (35), 130 (7), 106 (14), 105 (100), 104 (9); HRMS (EI) m/z calculated for $\text{C}_{17}\text{H}_{13}\text{NO}_2$ 263.0946, found 263.0945.



***N*-(4-Oxo-4-phenylbut-2-ynyl)acrylamide (3b)**. A mixture of 0.249 g (1.16 mmol) of **2b** and 0.541 g (1.28 mmol) of Dess-Martin reagent in 5 mL of CH₂Cl₂ was stirred at rt for 15 h, concentrated under reduced pressure, and purified by chromatography on SiO₂ (80%-50% hexanes:EtOAc) to yield 0.210 g (84%) of **3b** as a white amorphous solid: R_f 0.43 (50% hexanes:EtOAc); IR (neat) 3257, 3052, 2244, 2198, 1634, 1621, 1538, 1504, 1405, 1312, 1270, 1239, 971 cm⁻¹; ¹H NMR δ 8.12 (dd, 2 H, *J* = 7.8, 1.4 Hz), 7.63 (tt, 1 H, *J* = 7.4, 1.3 Hz), 7.49 (t, 2 H, *J* = 7.8 Hz), 6.38 (dd, 1 H, *J* = 17.0, 1.4 Hz), 6.16 (dd, 1 H, *J* = 17.0, 10.2 Hz), 6.10 (br, 1 H), 5.75 (dd, 1 H, *J* = 10.2, 1.4 Hz), 4.46 (d, 2 H, *J* = 5.5 Hz); ¹³C NMR δ 177.7, 165.3, 136.2, 134.5, 129.8, 129.7, 128.7, 127.9, 89.8, 81.1, 29.5; MS (EI) *m/z* (rel intensity) 263 (M⁺, 6), 235 (6), 206 (6), 160 (27), 158 (35), 130 (7), 106 (14), 105 (100), 104 (9); HRMS (EI) *m/z* calculated for C₁₇H₁₃NO₂ 263.0946, found 263.0945.

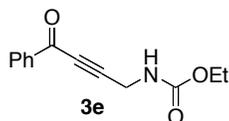


2,2-Dimethyl-*N*-(4-oxo-4-phenylbut-2-ynyl)propionamide (3c). A mixture of 0.527 g (2.15 mmol) of **2c** and 1.21 g (2.76 mmol) of Dess-Martin reagent in 10 mL of CH₂Cl₂ was stirred at rt for 24 h, concentrated under reduced pressure, and purified by chromatography on SiO₂ to yield 0.435 g (83%) of crude **3c** as a yellow oil that was used without further purification: R_f 0.55 (50% hexanes:EtOAc); ¹H NMR δ 8.12 (d, 2 H, *J* = 7.8 Hz), 7.62 (t, 1 H, *J* = 7.0 Hz), 7.48 (t, 2 H, *J* = 7.3 Hz), 6.07 (br, 1 H), 4.36 (d, 2 H, *J* = 5.3 Hz), 1.25 (s, 9 H).

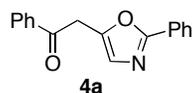


***N*-(4-Oxo-4-phenylbut-2-ynyl)propionamide (3d)**. A mixture of 0.247 g (1.14 mmol) of **2d** and 0.556 g (1.31 mmol) of Dess-Martin reagent in 5 mL of CH₂Cl₂ was stirred at rt for 15 h,

concentrated under reduced pressure, and purified by chromatography on SiO₂ to yield 0.172 g (70%) of **3d** as a yellow amorphous solid: R_f 0.19 (50% hexanes:EtOAc); IR (neat) 3292, 3068, 2976, 2238, 2218, 1643, 1539, 1455, 1260, 1096, 1025 cm⁻¹; ¹H NMR δ 8.12 (dd, 2 H, *J* = 7.8, 1.4 Hz), 7.63 (t, 1 H, *J* = 7.4 Hz), 7.49 (t, 2 H, *J* = 7.7 Hz), 5.83 (br, 1 H), 4.38 (d, 2 H, *J* = 5.4 Hz), 2.29 (q, 2 H, *J* = 7.6 Hz), 1.20 (t, 3 H, *J* = 7.6 Hz); ¹³C NMR δ 177.9, 174.0, 136.2, 134.4, 129.7, 128.6, 90.8, 80.7, 29.3, 29.3, 9.7; MS (EI) *m/z* (rel intensity) 215 (M⁺, 5), 187 (6), 160 (52), 159 (83), 158 (53), 130 (57), 115 (47), 105 (100); HRMS (EI) *m/z* calculated for C₁₃H₁₃NO₂ 215.0946, found 215.0938.

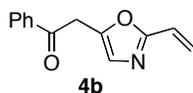


(4-Oxo-4-phenylbut-2-ynyl)-carbamic acid ethyl ester (3e). A mixture of 0.531 g (2.28 mmol) of **2e** and 1.06 g (2.50 mmol) of Dess-Martin reagent in 10 mL of CH₂Cl₂ was stirred at rt for 15 h, concentrated under reduced pressure, and purified by chromatography on SiO₂ (90%-80% hexanes:EtOAc) to yield 0.490 g (93%) of **3e** as a pale yellow oil: R_f 0.41 (65% hexanes:EtOAc); IR (neat) 3334, 2983, 2228, 1694, 1651, 1505, 1264, 1022 cm⁻¹; ¹H NMR δ 8.12 (dd, 2 H, *J* = 8.0, 1.5 Hz), 7.62 (tt, 1 H, *J* = 7.4, 1.1 Hz), 7.48 (t, 2 H, *J* = 7.3 Hz), 5.23 (br, 1 H), 4.29 (d, 2 H, *J* = 5.7 Hz), 4.18 (d, 2 H, *J* = 6.9 Hz), 1.27 (t, 3 H, *J* = 7.1 Hz); ¹³C NMR δ 177.7, 156.2, 136.3, 134.4, 129.7, 128.6, 90.3, 81.0, 61.6, 31.2, 14.6; MS (EI) *m/z* (rel intensity) 231 (M⁺, 7), 203 (19), 186 (12), 175 (20), 159 (28), 158 (66), 131 (25), 130 (60), 115 (54), 105 (100); HRMS (EI) *m/z* calculated for C₁₃H₁₃NO₃ 231.0895, found 231.0886.

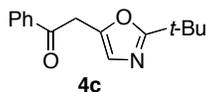


1-Phenyl-2-(2-phenyl-oxazol-5-yl)-ethanone (4a). A mixture of 0.0711 g (0.260 mmol) of **3a** and 0.214 g of SiO₂ in 5 mL of CH₂Cl₂ was stirred at rt for 24 h, filtered, concentrated under reduced pressure, and purified by chromatography on SiO₂ to yield 0.0711 g (>99%) of **4a** as a white amorphous solid.

Alternatively, a mixture of 0.103 g (0.386 mmol) of **2a** and 0.180 g (0.424 mmol) of Dess-Martin reagent in 5 mL of CH₂Cl₂ was stirred at rt for 15 h. The remaining Dess-Martin reagent and its by-products were removed by filtration through a short pad of SiO₂. The filtrate was stirred with 0.309 g of SiO₂ in 5 mL of CH₂Cl₂ at rt for 24 h, filtered, concentrated under reduced pressure, and purified by chromatography on SiO₂ (60% hexanes:EtOAc) to give 0.0866 g (85%) of **4a** as a white amorphous solid: R_f 0.35 (65% hexanes:EtOAc); IR (neat) 3061, 2917, 1690, 1603, 1552, 1489, 1454, 1339, 1212, 1126, 1062 cm⁻¹; ¹H NMR δ 8.03 (d, 2 H, *J* = 8.4 Hz), 8.02-7.96 (m, 2 H), 7.63-7.40 (m, 6 H), 7.13 (s, 1 H), 4.43 (s, 2 H); ¹³C NMR δ 193.7, 161.6, 145.5, 135.9, 133.8, 130.2, 128.8, 128.7, 128.5, 127.5, 126.8, 126.2, 36.0; MS (EI) *m/z* (rel intensity) 263 (M⁺, 15), 158 (18), 105 (100); HRMS (EI) *m/z* calculated for C₁₇H₁₃NO₂ 263.0946, found 263.0948.

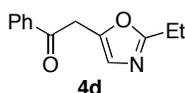


1-Phenyl-2-(2-vinyloxazol-5-yl)-ethanone (4b). A mixture of 0.0815 g (0.382 mmol) of **3b** and 0.245 g of SiO₂ in 5 mL of CH₂Cl₂ was stirred at rt for 24 h, filtered, concentrated under reduced pressure, and purified by chromatography on SiO₂ to yield 0.0710 g (87%) of **4b** as a yellow oil: R_f 0.33 (65% hexanes:EtOAc); IR (neat) 3073, 2917, 1694, 1597, 1449, 1207, 1112, 998 cm⁻¹; ¹H NMR δ 8.01 (dd, 2 H, *J* = 7.9, 1.5 Hz), 7.62 (tt, 1 H, *J* = 7.4, 1.3 Hz), 7.50 (t, 2 H, *J* = 7.9 Hz), 7.05 (s, 1 H), 6.56 (dd, 1 H, *J* = 17.7, 11.2 Hz), 6.11 (dd, 1 H, *J* = 17.7, 1.0 Hz), 5.58 (dd, 1 H, *J* = 11.2, 1.0 Hz), 4.38 (d, 2 H, *J* = 0.8 Hz); ¹³C NMR δ 193.7, 161.0, 145.2, 135.9, 133.8, 128.9, 128.5, 126.7, 123.4, 121.4, 35.9; MS (EI) *m/z* (rel intensity) 213 (M⁺, 15), 105 (100); HRMS (EI) *m/z* calculated for C₁₃H₁₁NO₂ 213.0790, found 213.0799.

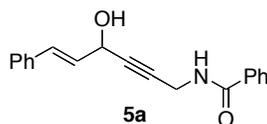


2-(2-tert-Butyloxazol-5-yl)-1-phenylethanone (4c). A mixture of 0.0915 g (0.373 mmol) of **2c** and 0.174 g (0.410 mmol) of Dess-Martin reagent in 5 mL of CH₂Cl₂ was stirred at rt for 15 h.

The remaining Dess-Martin reagent and its by-products were removed by filtration through a short pad of SiO₂. The filtrate was treated with 0.275 g of SiO₂ in 5 mL of CH₂Cl₂ at rt for 24 h, filtered, concentrated under reduced pressure, and purified by chromatography on SiO₂ to give 0.0780 g (86%) of **4c** as a colorless amorphous solid: R_f 0.40 (65% hexanes:EtOAc); IR (neat) 2972, 2934, 1686, 1560, 1449 cm⁻¹; ¹H NMR δ 8.03-7.99 (m, 2 H), 7.61 (tt, 1 H, *J* = 7.4, 2.0 Hz), 7.52-7.47 (m, 2 H), 6.88 (t, 1 H, *J* = 0.9 Hz), 4.34 (d, 2 H, *J* = 1.0 Hz), 1.36 (s, 9 H); ¹³C NMR δ 194.0, 171.0, 144.6, 136.0, 133.6, 128.8, 128.4, 124.8, 36.0, 33.7, 28.5; MS (EI) *m/z* (rel intensity) 243 (M⁺, 10), 106 (15), 105 (100); HRMS (EI) *m/z* calculated for C₁₅H₁₇NO₂ 243.1259, found 243.1269.

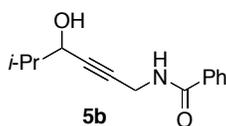


2-(2-Ethyloxazol-5-yl)-1-phenylethanone (4d). A mixture of 0.0810 g (0.382 mmol) of **3d** and 0.243 g of SiO₂ in 5 mL of CH₂Cl₂ was stirred at rt for 24 h, filtered, concentrated under reduced pressure, and purified by chromatography on SiO₂ (80%-65% hexanes:EtOAc) to yield 0.0259 g (32%) of **4d** as a pale yellow oil: R_f 0.38 (50% hexanes:EtOAc); IR (neat) 2980, 1688, 1558, 1450 cm⁻¹; ¹H NMR δ 8.00 (dd, 2 H, *J* = 7.8, 1.3 Hz), 7.60 (tt, 1 H, *J* = 7.2, 1.0 Hz), 7.49 (t, 2 H, *J* = 7.7 Hz), 6.89 (s, 1 H), 4.32 (s, 2 H), 2.77 (q, 2 H, *J* = 7.6 Hz), 1.32 (t, 3 H, *J* = 7.6 Hz); ¹³C NMR δ 194.0, 165.8, 144.8, 135.9, 133.7, 128.8, 128.4, 125.1, 35.8, 21.7, 11.1; MS (EI) *m/z* (rel intensity) 215 (M⁺, 21), 105 (100); HRMS (EI) *m/z* calculated for C₁₃H₁₃NO₂ 215.0946, found 215.0953.

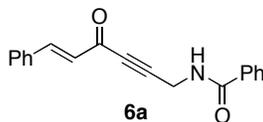


(5E)-N-(4-Hydroxy-6-phenylhex-5-en-2-ynyl)-benzamide (5a). A solution of 0.711 g (4.47 mmol) of **1a** in 30 mL of THF was treated at -78 °C with 9.83 mL (9.83 mmol) of a 1.0 M solution of LiHMDS in THF, stirred for 15 min at -78 °C, treated with a solution of 0.650 g (4.92 mmol) of *trans*-cinnamaldehyde in 1 mL of THF, warmed to 0 °C over 1 h, and stirred for 1

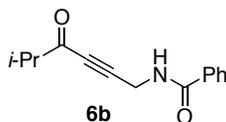
h. The reaction mixture was quenched with 1.0 M HCl (50 mL) and extracted with EtOAc (2 x 30 mL). The combined organic layers were washed with brine (50 mL), dried (MgSO₄), concentrated under reduced pressure, and purified by chromatography on SiO₂ to yield 0.937 g (72%) of **5a** as a pale yellow foam: *R*_f 0.27 (50% hexanes:EtOAc); IR (neat) 3308, 3028, 2924, 2222, 1645, 1538, 1448, 1293, 1157, 1070 cm⁻¹; ¹H NMR δ 7.78 (dd, 2 H, *J* = 7.8, 1.4 Hz), 7.57-7.22 (m, 8 H), 6.72 (d, 1 H, *J* = 15.8 Hz), 6.58 (br, 1 H), 6.27 (dd, 1 H, *J* = 15.8, 6.1 Hz), 5.07 (dd, 1 H, *J* = 6.0, 1.3 Hz), 4.31 (dd, 2 H, *J* = 5.2, 1.7 Hz), 2.91 (br, 1 H); ¹³C NMR δ 167.7, 136.0, 133.4, 131.8, 131.6, 128.5, 128.5, 128.5, 128.0, 127.2, 126.7, 82.7, 81.8, 62.6, 30.1; MS (EI) *m/z* (rel intensity) 291 (M⁺, 0.8), 290 (1), 273 (28), 169 (18), 142 (33), 105 (100); HRMS (EI) *m/z* calculated for C₁₉H₁₆NO₂ (M-H) 290.1181, found 290.1170.



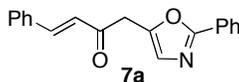
N-(4-Hydroxy-5-methylhex-2-ynyl)-benzamide (5b). A solution of 0.894 g (5.62 mmol) of **1a** in 30 mL of THF was treated at -78 °C with 12.0 mL (12.0 mmol) of a 1.0 M solution of LiHMDS in THF, stirred for 15 min at -78 °C, treated with a solution of 0.437 g (6.06 mmol) of isopropyl aldehyde in 1 mL of THF, warmed to 0 °C over 1 h, and stirred for 1 h. The reaction mixture was quenched with 1.0 M HCl (50 mL) and extracted with EtOAc (2 x 30 mL). The combined organic layers were washed with brine (50 mL), dried (MgSO₄), concentrated under reduced pressure, and purified by chromatography on SiO₂ to yield 1.22 g (94%) of **5b** as a colorless oil: *R*_f 0.39 (50% hexanes:EtOAc); IR (neat) 3307, 3068, 2963, 2246, 1651, 1538, 1487, 1307, 1158, 1027 cm⁻¹; ¹H NMR δ 7.79 (dd, 2 H, *J* = 8.1, 1.4 Hz), 7.53 (t, 1 H, *J* = 4.9 Hz), 7.55-7.51 (m, 1 H), 7.33 (t, 2 H, *J* = 7.9 Hz), 4.20 (dd, 2 H, *J* = 5.0, 1.0 Hz), 4.15-4.11 (m, 1 H), 3.98 (br, 1 H), 1.84-1.76 (m, 1 H), 0.95 (d, 3 H, *J* = 7.0 Hz), 0.92 (d, 3 H, *J* = 7.4 Hz); ¹³C NMR δ 167.6, 133.4, 131.5, 128.2, 127.1, 83.2, 80.7, 67.4, 34.2, 29.8, 18.0, 17.4; MS (EI) *m/z* (rel intensity) 231 (M⁺, 0.02), 230 (0.1), 214 (2), 189 (20), 188 (16), 173 (16), 105 (100); HRMS (EI) *m/z* calculated for C₁₄H₁₆NO₂ (M-H) 230.1181, found 230.1175.



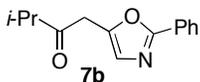
(5E)-N-(4-Oxo-6-phenylhex-5-en-2-ynyl)-benzamide (6a). A mixture of 0.536 g (2.19 mmol) of **5a** and 1.02 g (2.40 mmol) of Dess-Martin reagent in 10 mL of CH₂Cl₂ was stirred at rt for 15 h, filtered, concentrated under reduced pressure, and purified by chromatography on SiO₂ to yield 0.213 g (40%) of **6a** as a white solid: mp 129.0-130.0 °C (hexanes/EtOAc); IR (neat) 3280, 3053, 2223, 1633, 1531, 1312, 1253, 1209, 984 cm⁻¹; ¹H NMR δ 7.85 (d, 1 H, *J* = 16.0 Hz), 7.83 (d, 2 H, *J* = 7.1 Hz), 7.60-7.38 (m, 8 H), 6.77 (d, 1 H, *J* = 16.1 Hz), 6.69 (br, 1 H), 4.52 (d, 2 H, *J* = 5.4 Hz); ¹³C NMR δ 177.9, 167.2, 149.5, 133.9, 133.4, 132.1, 131.4, 129.1, 128.8, 128.8, 127.9, 127.1, 88.3, 80.9, 30.0; MS (EI) *m/z* (rel intensity) 289 (M⁺, 6), 288 (3), 184 (46), 131 (95), 105 (100), 103 (44); HRMS (EI) *m/z* calculated for C₁₉H₁₅NO₂ 289.1103, found 289.1100.



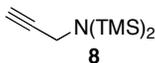
N-(5-Methyl-4-oxohex-2-ynyl)-benzamide (6b). A mixture of 0.128 g (0.553 mmol) of **5b** and 0.258 g (0.608 mmol) of Dess-Martin reagent in 10 mL of CH₂Cl₂ was stirred at rt for 15 h, filtered, concentrated under reduced pressure, and purified by chromatography on SiO₂ to yield 0.122 g (96%) of **6b** as a white solid: R_f 0.69 (65% hexanes:EtOAc); mp 69.5-70.5 °C (hexanes/EtOAc); IR (neat) 3271, 3068, 2976, 2218, 1688, 1634, 1538, 1488, 1408, 1298, 981 cm⁻¹; ¹H NMR δ 7.81 (dd, 2 H, *J* = 7.9, 1.6 Hz), 7.50 (tt, 1 H, *J* = 7.4, 1.2 Hz), 7.40 (t, 2 H, *J* = 7.8 Hz), 7.26 (br, 1 H), 4.41 (d, 2 H, *J* = 5.5 Hz), 2.62 (septet, 1 H, *J* = 7.0 Hz), 1.16 (d, 6 H, *J* = 6.9 Hz); ¹³C NMR δ 191.8, 167.4, 133.5, 131.9, 128.6, 127.2, 89.0, 81.2, 42.9, 29.8, 17.8; MS (EI) *m/z* (rel intensity) 229 (M⁺, 4), 201 (4), 186 (57), 172 (50), 159 (40), 105 (100); HRMS (EI) *m/z* calculated for C₁₄H₁₅NO₂ 229.1103, found 229.1076.



(3E) 4-Phenyl-1-(2-phenyloxazol-5-yl)-but-3-en-2-one (7a). A mixture of 0.0520 g (0.180 mmol) of **6a** and 0.156 g of SiO₂ in 5 mL of CH₂Cl₂ was stirred at rt for 24 h, filtered, concentrated under reduced pressure, and purified by chromatography on SiO₂ to yield 0.0516 g (>99%) of **7a** as a pale yellow amorphous solid: R_f 0.38 (65% hexanes:EtOAc); IR (neat) 3063, 2925, 1693, 1613, 1446, 1340, 1086 cm⁻¹; ¹H NMR δ 8.05-7.98 (m, 2 H), 7.70 (d, 1 H, *J* = 16.1 Hz), 7.59-7.53 (m, 2 H), 7.47-7.38 (m, 6 H), 7.13 (brs, 1 H), 6.84 (d, 1 H, *J* = 16.1 Hz), 4.10 (d, 2 H, *J* = 0.8 Hz); ¹³C NMR δ 193.3, 161.6, 145.5, 144.4, 134.0, 130.9, 130.2, 129.0, 128.7, 128.5, 127.4, 126.7, 126.2, 124.3, 38.3; MS (EI) *m/z* (rel intensity) 289 (M⁺, 4), 131 (100), 103 (27); HRMS (EI) *m/z* calculated for C₁₉H₁₅NO₂ 289.1103, found 289.1109.



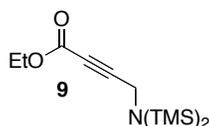
3-Methyl-1-(2-phenyloxazol-5-yl)-butan-2-one (7b). A mixture of 0.0732 g (0.319 mmol) of **6b** and 0.220 g of SiO₂ in 5 mL of CH₂Cl₂ was stirred at rt for 24 h, filtered, concentrated under reduced pressure, and purified by chromatography on SiO₂ to yield 0.0732 g (>99%) of **7b** as a pale yellow oil: R_f 0.56 (50% hexanes:EtOAc); IR (neat) 2971, 2933, 1721, 1603, 1552, 1445, 1358, 1122, 1040 cm⁻¹; ¹H NMR δ 8.03-7.97 (m, 2 H), 7.47-7.41 (m, 3 H), 7.08 (s, 1 H), 3.90 (s, 2 H), 2.77 (septet, 1 H, *J* = 6.9 Hz), 1.18 (d, 6 H, *J* = 6.8 Hz); ¹³C NMR δ 208.2, 161.5, 145.6, 130.2, 128.7, 127.4, 126.6, 126.2, 40.5, 37.6, 18.1; MS (EI) *m/z* (rel intensity) 229 (M⁺, 4), 229 (26), 160 (23), 159 (100), 158 (83), 130 (32), 104 (40); HRMS (EI) *m/z* calculated for C₁₄H₁₅NO₂ 229.1103, found 229.1102.



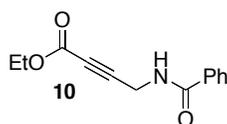
1,1,1,3,3,3-Hexamethyl-2-prop-2-ynyldisilazane (8).⁴ A mixture of 1.01 g (18.3 mmol) of propargylamine, 4.80 mL (37.8 mmol) of chlorotrimethylsilane, and 7.70 mL (55.2 mmol) of

⁴ Corriu, R. J. P.; Huynh, V.; Iqbal, J.; Moreau, J. J. E.; Vernhet, C. *Tetrahedron* **1992**, *48*, 6231-6244.

Et₃N in 30 mL of CH₂Cl₂ was stirred at rt for 12 h, quenched with brine (50 mL), and extracted with CHCl₃ (2 x 30 mL). The combined organic layers were washed with brine (50 mL), dried (MgSO₄), and concentrated under reduced pressure to give 2.11 g (58%) of **8** as a pale yellow oil: ¹H NMR δ 3.53 (d, 2 H, *J* = 2.4 Hz), 2.13 (t, 1 H, *J* = 2.4 Hz), 0.15 (s, 18 H).



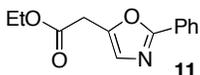
4-(1,1,1,3,3,3-Hexamethyldisilazan-2-yl)but-2-ynoic acid ethyl ester (9).⁵ A solution of 1.63 g (8.16 mmol) of **8** in 10 mL of THF was treated at -78 °C with 5.50 mL (8.80 mmol) of a 1.6 M solution of *n*-BuLi in hexane, stirred for 15 min at -78 °C, treated with a solution of 0.956 g (8.81 mmol) of ethyl chloroformate in 2 mL of THF, warmed to rt over 1 h, and stirred for 1 h. The reaction mixture was quenched with brine (30 mL) and extracted with EtOAc (2 x 30 mL). The combined organic layers were washed with brine (50 mL), dried (MgSO₄), and concentrated under reduced pressure to give 2.21 g of crude ester **9** as an orange oil: ¹H NMR δ 4.21 (q, 2 H, *J* = 7.0 Hz), 3.67 (s, 2 H), 1.30 (t, 3 H, *J* = 7.1 Hz), 0.16 (s, 18 H).



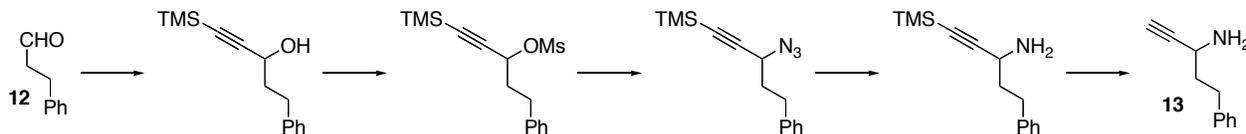
4-Benzoylamino but-2-ynoic acid ethyl ester (10). To a solution of 2.21 g of crude ester **9** and 1.26 g (8.96 mmol) of benzoyl chloride in 15 mL of THF was added 0.40 mL (0.40 mmol) of a 1.0 M solution of TBAF in THF. The reaction mixture was stirred at rt for 12 h, quenched with 1.0 M HCl (40 mL), and extracted with EtOAc (2 x 30 mL). The combined organic layers were washed with brine (40 mL), dried (MgSO₄), concentrated under reduced pressure, and purified by chromatography on SiO₂ to afford 1.26 g (67%, 2 steps from **8**) of **10** as a white solid: *R*_f 0.47 (50% hexanes:EtOAc); mp 103.8.-105.8 °C (hexanes/EtOAc); IR (neat) 3298, 2983, 2243, 1713, 1640, 1530, 1487, 1409, 1274, 1076, 984 cm⁻¹; ¹H NMR δ 7.78 (dd, 2 H, *J* = 7.8, 1.6 Hz), 7.54

⁵ Corriu, R. J. P.; Bolin, G.; Iqbal, J.; Moreau, J. E.; Vernhet, C. *Tetrahedron* **1993**, *49*, 4603-4618.

(tt, 1 H, $J = 7.3, 1.3$ Hz), 7.45 (t, 2 H, $J = 7.0$ Hz), 6.35 (br, 1 H), 4.42 (d, 2 H, $J = 5.4$ Hz), 4.24 (q, 2 H, $J = 7.1$ Hz), 1.31 (t, 3 H, $J = 7.1$ Hz); ^{13}C NMR δ 167.4, 153.3, 133.3, 132.0, 128.6, 127.2, 83.4, 75.1, 62.2, 29.5, 14.0; MS (EI) m/z (rel intensity) 231 (M^+ , 2), 187 (11), 186 (28), 160 (11), 159 (65), 158 (15), 130 (21), 105 (100); HRMS (EI) m/z calculated for $\text{C}_{13}\text{H}_{13}\text{NO}_3$ 231.0895, found 231.0890.



(2-Phenyloxazol-5-yl)-acetic acid ethyl ester (11). A mixture of 0.0444 g (0.192 mmol) of **10** and 0.145 g of SiO_2 in 4 mL of CH_2Cl_2 was stirred at rt for 72 h, filtered, concentrated under reduced pressure, and purified by chromatography on SiO_2 to yield 0.0401 g (90%) of **11** as a white amorphous solid: IR (neat) 2975, 1726, 1609, 1486, 1450, 1399, 1337, 1235, 1122, 1034, 994 cm^{-1} ; ^1H NMR δ 8.04-7.99 (m, 2 H), 7.46-7.42 (m, 3 H), 7.09 (s, 1 H), 4.22 (q, 2 H, $J = 7.1$ Hz), 3.79 (s, 2 H), 1.29 (t, 3 H, $J = 7.1$ Hz); ^{13}C NMR δ 168.4, 161.5, 145.0, 130.3, 128.7, 127.4, 126.4, 126.2, 61.5, 31.9, 14.1; MS (EI) m/z (rel intensity) 231 (M^+ , 46), 159 (22), 158 (100), 130 (21), 104 (23); HRMS (EI) m/z calculated for $\text{C}_{13}\text{H}_{13}\text{NO}_3$ 231.0895, found 231.0898.



5-Phenyl-1-trimethylsilylprop-1-yn-3-ol.⁶ A solution of 1.05 g (10.7 mmol) of (trimethylsilyl)acetylene in 30 mL of THF was treated at -78 °C with 6.40 mL (10.2 mmol) of a 1.6 M solution of *n*-BuLi in hexane, stirred for 15 min at -78 °C, treated with a solution of 1.43 g (9.59 mmol) of hydrocinnamaldehyde (**12**) in 2 mL of THF, warmed to rt over 1 h, and stirred for 1 h. The reaction mixture was quenched with 1N HCl (50 mL) and extracted with EtOAc (2 x 50 mL). The combined organic layers were washed with brine (50 mL), dried (MgSO_4), concentrated under reduced pressure, and purified by chromatography on SiO_2 to yield 1.93 g (81%) of the propargylic alcohol as a colorless oil: R_f 0.69 (50% hexanes:EtOAc); ^1H NMR δ

⁶ López, F.; Castedo, L.; Mascareñas, J. L. *J. Am. Chem. Soc.* **2002**, *124*, 4218-4219.

7.32-7.13 (m, 5 H), 4.36 (t, 1 H, $J = 6.5$ Hz), 2.79 (t, 2 H, $J = 8.0$ Hz), 2.06-1.96 (m, 2 H), 1.75 (br, 1 H), 0.18 (s, 9 H).

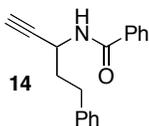
Methanesulfonic acid 1-phenethyl-3-trimethylsilanylprop-2-ynyl ester. A mixture of 1.79 g (7.69 mmol) of propargylic alcohol, 0.888 g (7.75 mmol) of methanesulfonyl chloride and 1.30 mL (9.33 mmol) of Et_3N in 20 mL of CH_2Cl_2 was stirred at rt for 12 h, quenched with 1N HCl (30 mL), and extracted with CHCl_3 (2 x 30 mL). The combined organic layers were washed with brine (40 mL), dried (MgSO_4), and concentrated under reduced pressure to give 2.39 g (>99%) of the mesylate as a colorless oil: IR (neat) 3029, 2960, 2177, 1604, 1497, 1455, 1361, 1252, 1176, 1093, 1032, 974 cm^{-1} ; ^1H NMR δ 7.35-7.15 (m, 5 H), 5.13 (t, 1 H, $J = 6.5$ Hz), 3.13 (s, 3 H), 2.90-2.70 (m, 2 H), 2.33-2.08 (m, 2 H), 0.20 (s, 9 H); ^{13}C NMR δ 140.0, 128.6, 128.5, 126.4, 100.2, 94.7, 71.6, 39.2, 37.2, 37.2, 31.0, -0.4; MS (EI) m/z (rel intensity) 310 (M^+ , 0.1), 215 (16), 214 (73), 199 (100), 183 (17), 153 (26), 141 (25), 91 (59); HRMS (EI) m/z calculated for $\text{C}_{14}\text{H}_{18}\text{Si}$ (M- $\text{CH}_3\text{SO}_3\text{H}$) 214.1178, found 214.1176.

(3-Azido-5-phenylpent-1-ynyl)-trimethylsilane. A mixture of 2.24 g (7.21 mmol) of the mesylate and 0.901 g (13.9 mmol) of NaN_3 in 15 mL of DMF was stirred at rt for 3 h, quenched with brine (50 mL), and extracted with EtOAc (2 x 70 mL). The combined organic layers were washed with brine (4 x 30 mL), dried (MgSO_4), and concentrated under reduced pressure to afford 1.67 g (90%) of the azide as a colorless oil: ^1H NMR δ 7.35-7.15 (m, 5 H), 4.05 (t, 1 H, $J = 6.9$ Hz), 2.80-2.70 (m, 2 H), 2.03-1.94 (m, 2 H), 0.21 (s, 9 H).

1-Phenethyl-3-trimethylsilanylprop-2-ynylamine. A mixture of 1.37 g (5.31 mmol) of the azide and 1.51 g (7.95 mmol) of SnCl_2 in 10 mL of MeOH was stirred at rt for 15 h, quenched with saturated aqueous NaHCO_3 (40 mL), filtered through a pad of Celite, and extracted with CHCl_3 (2 x 40 mL). The combined organic layers were washed with brine (40 mL), dried (MgSO_4), and concentrated under reduced pressure to give 1.04 g (85%) of the amine as an orange oil: IR (neat) 3380, 3298, 3027, 2957, 2163, 1603, 1496, 1455, 1250, 1045 cm^{-1} ; ^1H NMR δ 7.31-7.15 (m, 5 H), 3.52 (t, 1 H, $J = 7.0$ Hz), 2.88-2.70 (m, 2 H), 1.98-1.80 (m, 2 H), 1.57 (br, 2 H), 0.18 (s, 9 H); ^{13}C NMR δ 141.6, 128.5, 128.4, 126.0, 109.8, 86.9, 43.7, 39.7, 32.4, 0.1; MS

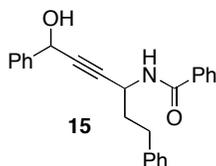
(EI) m/z (rel intensity) 231 (M^+ , 4), 230 (4), 158 (30), 153 (39), 126 (100), 98 (41), 91 (70); HRMS (EI) m/z calculated for $C_{14}H_{20}NSi$ (M-H) 230.1365, found 230.1356.

1-Phenethylprop-2-ynylamine (13). A mixture of 0.622 g (2.69 mmol) of the amine and 0.187 g (3.22 mmol) of KF in 5 mL of MeOH was stirred at rt for 15 h, quenched with saturated aqueous $NaHCO_3$ (30 mL), and extracted with EtOAc (2 x 30 mL). The combined organic layers were washed with brine (40 mL), dried ($MgSO_4$), concentrated under reduced pressure, and purified by chromatography on SiO_2 (95% $CHCl_3$:MeOH) to give 0.428 g (>99%) of **13** as an orange oil: R_f 0.18 (35% hexanes:EtOAc); IR (neat) 3291, 3067, 3026, 2923, 2110, 1602, 1496, 1454, 1317, 1035 cm^{-1} ; 1H NMR δ 7.33-7.15 (m, 5 H), 3.54 (td, 1 H, $J = 6.9, 2.0$ Hz), 2.89-2.70 (m, 2 H), 2.34 (d, 1 H, $J = 2.2$ Hz), 1.98-1.85 (m, 2 H), 1.47 (br, 2 H); ^{13}C NMR δ 141.2, 128.3, 128.3, 125.8, 87.4, 70.7, 42.6, 39.4, 32.0; MS (EI) m/z (rel intensity) 159 (M^+ , 9), 158 (25), 143 (14), 142 (47), 141 (29), 105 (14), 91 (27); HRMS (EI) m/z calculated for $C_{11}H_{12}N$ (M-H) 158.0970, found 158.0970.

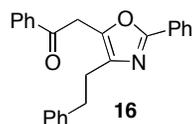


N-(1-Phenethylprop-2-ynyl)-benzamide (14). To a solution of 0.200 g (1.26 mmol) of **13** in 10 mL of CH_2Cl_2 were added 0.216 g (1.53 mmol) of benzoyl chloride and 0.30 mL (2.15 mmol) of Et_3N at 0 °C. The reaction mixture was stirred at rt for 2 h, poured into 1.0 M HCl (20 mL), and extracted with $CHCl_3$ (2 x 20 mL). The combined organic layers were washed with brine (30 mL), dried ($MgSO_4$), concentrated under reduced pressure, and purified by chromatography on SiO_2 to give 0.205 g (62%) of **14** as a white solid: R_f 0.51 (65% hexanes:EtOAc); mp 115.5-116.0 °C (hexanes/EtOAc); IR (neat) 3267, 2980, 2120, 1629, 1531, 1454, 1286 cm^{-1} ; 1H NMR δ 7.66 (d, 2 H, $J = 7.3$ Hz), 7.53-7.38 (m, 3 H), 7.33-7.18 (m, 5 H), 6.19 (bd 1 H, $J = 6.8$ Hz), 5.07-4.99 (m, 1 H), 2.97-2.77 (m, 2 H), 2.39 (d, 1 H, $J = 1.7$ Hz), 2.24-2.02 (m, 2 H); ^{13}C NMR δ 166.5, 140.9, 133.7, 131.6, 128.5, 128.4, 128.4, 127.1, 126.1, 82.8, 71.9, 41.6, 36.9, 31.9; MS (EI) m/z (rel intensity) 263 (M^+ , 6), 262 (10), 235 (16), 172 (13), 160 (16), 159 (83), 158 (24),

142 (18), 141 (23), 130 (36), 105 (100); HRMS (EI) m/z calculated for $C_{18}H_{16}NO$ (M-H) 262.1232, found 262.1230.

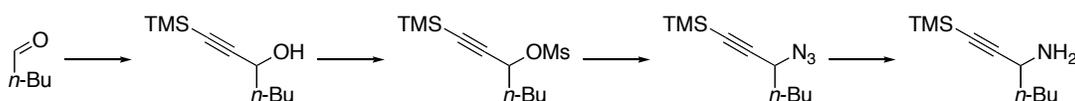


***N*-(4-Hydroxy-1-phenethyl-4-phenylbut-2-ynyl)-benzamide (15).** A solution of 0.0976 g (0.371 mmol) of **14** in 5 mL of THF was treated at -78 °C with 0.85 mL (0.85 mmol) of a 1.0 M solution of LiHMDS in THF, stirred for 15 min at -78 °C, treated with a solution of 0.0605 g (0.570 mmol) of benzaldehyde in 1 mL of THF, warmed to 0 °C over 1 h, and stirred for 1 h. The reaction mixture was quenched with 1.0 M HCl (10 mL) and extracted with EtOAc (2 x 10 mL). The combined organic layers were washed with brine (10 mL), dried ($MgSO_4$), concentrated under reduced pressure, and purified by chromatography on SiO_2 to give 0.0778 g (51%) of **15** as a pale yellow oil: R_f 0.30 (65% hexanes:EtOAc); IR (neat) 3368, 2980, 2243, 1634, 1533, 1440 cm^{-1} ; 1H NMR δ 7.63 (dd, 2 H, $J = 7.7, 1.4$ Hz), 7.53 (dd, 2 H, $J = 7.8, 1.6$ Hz), 7.47 (tt, 1 H, $J = 7.4, 1.3$ Hz), 7.40-7.14 (m, 10 H), 6.43 (d, 1 H, $J = 8.1$ Hz), 5.49 (s, 1 H), 5.11-5.02 (m, 1 H), 3.09 (br, 1 H), 2.89-2.72 (m, 2 H), 2.21-2.00 (m, 2 H); ^{13}C NMR δ 166.5, 141.0, 140.5, 133.7, 131.7, 128.7, 128.6, 128.5, 128.5, 128.4, 127.0, 126.6, 126.1, 85.3, 83.9, 64.5, 42.0, 37.1, 32.1; MS (EI) m/z (rel intensity) 369 (M^+ , 3), 368 (10), 352 (22), 341 (25), 265 (45), 248 (62), 105 (100); HRMS (EI) m/z calculated for $C_{25}H_{22}NO_2$ (M-H) 368.1651, found 368.1639.



2-(4-Phenethyl-2-phenyloxazol-5-yl)-1-phenylethanone (16). A mixture of 0.0721 g (0.195 mmol) of **15** and 0.0915 g (0.216 mmol) of Dess-Martin reagent in 5 mL of CH_2Cl_2 was stirred at rt for 15 h. The remaining Dess-Martin reagent and its by-products were removed by filtration through a short pad of SiO_2 . The filtrate was treated with 0.216 g of SiO_2 in 5 mL of CH_2Cl_2 at

rt for 24 h, filtered, concentrated under reduced pressure, and purified by chromatography on SiO₂ to give 0.0444 g (62%) of **16** as a pale yellow amorphous solid: R_f 0.69 (65% hexanes:EtOAc); IR (neat) 3027, 2926, 1694, 1598, 1556, 1486, 1454, 1330, 1213, 1070 cm⁻¹; ¹H NMR δ 8.02-7.96 (m, 2 H), 7.94 (dd, 2 H, *J* = 7.8, 1.5 Hz), 7.59 (tt, 1 H, *J* = 7.4, 1.3 Hz), 7.48 (t, 2 H, *J* = 7.8 Hz), 7.46-7.40 (m, 3 H), 7.30-7.15 (m, 5 H), 4.03 (s, 2 H), 2.99 (t, 2 H, *J* = 6.9 Hz), 2.83 (t, 2 H, *J* = 7.9 Hz); ¹³C NMR δ 194.0, 160.6, 141.5, 140.6, 138.2, 136.0, 133.6, 130.0, 128.7, 128.7, 128.7, 128.5, 128.4, 127.6, 126.2, 126.0, 35.1, 35.0, 28.3; MS (EI) *m/z* (rel intensity) 367 (M⁺, 21), 271 (33), 262 (35), 170 (100), 141 (90), 105 (55), 91 (46); HRMS (EI) *m/z* calculated for C₂₅H₂₁NO₂ 367.1572, found 367.1584.



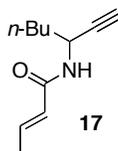
1-(Trimethylsilyl)hept-1-yn-3-ol.⁷ A solution of 1.51 mL (10.7 mmol) of (trimethylsilyl)acetylene in 30 mL of THF was treated at -78 °C with 6.3 mL (10.1 mmol) of a 1.6 M solution of *n*-BuLi in hexane, stirred for 15 min at -78 °C, treated with a solution of 1.01 mL (9.54 mmol) of valeraldehyde in 2 mL of THF, warmed to rt over 1 h, and stirred for 1 h. The clear, yellow reaction mixture was quenched with 10% HCl (50 mL) and extracted with EtOAc (3 x 20 mL). The combined organic layers were washed with brine (50 mL), dried (MgSO₄), concentrated under reduced pressure, and purified by chromatography on SiO₂ (90% hexanes:EtOAc) to give 1.42 g (81%) of the propargylic alcohol as a colorless oil: R_f 0.35 (90% hexanes:EtOAc); ¹H NMR δ 4.37-4.31 (m, 1 H), 1.90 (d, 1 H, *J* = 5.5 Hz), 1.71-1.65 (m, 2 H), 1.43-1.34 (m, 4 H), 0.91 (t, 3 H, *J* = 7.0 Hz), 0.16 (s, 9 H).

Methanesulfonic acid 1-butyl-3-(trimethylsilyl)prop-2-ynyl ester. A mixture of 6.22 g (33.7 mmol) of the propargylic alcohol, 2.87 mL (37.1 mmol) of methanesulfonyl chloride, and 5.69 mL (40.5 mmol) of Et₃N in 85 mL of CH₂Cl₂ was stirred at 0 °C for 15 min and allowed to warm to rt. After 20 h, the reaction mixture was quenched with 10% HCl (50 mL) and extracted with CH₂Cl₂ (2 x 100 mL). The organic layers were combined, washed with brine (50 mL), dried

(MgSO₄), and concentrated under reduced pressure to give the crude mesylate as a colorless oil: R_f 0.39 (80% hexanes:Et₂O).

(3-Azidohept-1-ynyl)-trimethylsilane. A mixture of the crude mesylate and 4.17 g (64.1 mmol) of NaN₃ in 67.5 mL of DMF was stirred at rt for 15 h, diluted with EtOAc (150 mL), washed with H₂O (7 x 75 mL) and brine (1 x 75 mL), dried (MgSO₄), and concentrated under reduced pressure to give 6.34 g (90%, 2 steps from the propargylic alcohol) of the azide as a yellow oil: R_f 0.90 (80% hexanes:Et₂O); ¹H NMR δ 4.05 (t, 1 H, *J* = 6.8 Hz), 1.72-1.65 (m, 2 H), 1.45-1.26 (m, 4 H), 0.92 (t, 3 H, *J* = 7.1 Hz), 0.21 (s, 9 H).

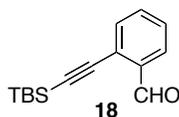
1-Butyl-3-(trimethylsilyl)-prop-2-ynylamine. A mixture of 6.34 g (30.3 mmol) of the azide and 8.61 g (45.4 mmol) of SnCl₂ was stirred in 60 mL of MeOH at rt for 14 h, quenched with saturated aqueous NaHCO₃ (250 mL) at 0 °C, and filtered through a pad of Celite. The solid was washed with MeOH (200 mL). The filtrate was concentrated under reduced pressure, diluted with H₂O (100 mL), and extracted with CH₂Cl₂ (3 x 250 mL). The combined organic layers were washed with brine (100 mL), dried (MgSO₄), and concentrated under reduced pressure to give 4.33 g (78%) of **18** as a tan oil: R_f 0.12 (50% hexanes:EtOAc); ¹H NMR δ 3.51 (t, 1 H, *J* = 6.6 Hz), 1.55-1.50 (m, 3 H), 1.46-1.24 (m, 5 H), 0.90 (t, 3 H, *J* = 7.0 Hz), 0.16 (s, 9 H).



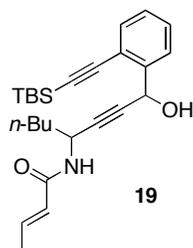
But-2-enoic acid (1-butylprop-2-ynyl)-amide (17). To a solution of 0.250 g (1.34 mmol) of the amine and 0.280 mL (2.00 mmol) of Et₃N in 5 mL of CH₂Cl₂ was added at 0 °C 0.140 mL (1.40 mmol) of *trans*-crotonyl chloride. The reaction mixture was allowed to warm to rt, stirred for 2 h, poured into 10% HCl (15 mL) and extracted with CHCl₃ (3 x 10 mL). The combined organic layers were washed with H₂O (5 mL), dried (Na₂SO₄), concentrated under reduced pressure, and purified by chromatography on SiO₂ (80% hexanes:EtOAc) to give the crude intermediate amide as a yellow oil which was used without further purification: R_f 0.70 (60%

⁷ Ivanov, I. V.; Romanov, S. G.; Groza, N. V.; Nigam, S.; Kuhn, H.; Myagkova, G. I. *Bioorg. Med. Chem.* **2002**,

hexanes:EtOAc). A mixture of 0.920 g of crude propargylic amide and NaOMe (prepared from 10.0 mg of sodium metal) was stirred in 1 mL of MeOH for 2 h. Then, activated Amberlite IR-120 Na ion-exchange resin was added until pH 7. The reaction mixture was filtered and concentrated under reduced pressure to give 0.630 g (96%, 2 steps from the propargyl amide) of crude **17** as an oil: R_f 0.53 (60% hexanes:EtOAc); $^1\text{H NMR}$ δ 6.87-6.77 (m, 1 H), 6.13 (d, 1 H, $J = 8.1$ Hz), 5.81 (dd, 1 H, $J = 15.1, 1.6$), 4.80-4.73 (m, 1 H), 2.22 (d, 1 H, $J = 2.3$ Hz), 1.82 (d, 3 H, $J = 6.8$ Hz), 1.67-1.63 (m, 2 H), 1.38-1.19 (m, 4 H), 0.86 (t, 3 H, $J = 7.0$ Hz).

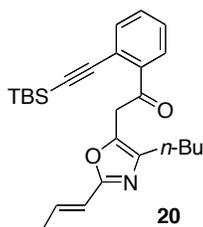


2-[(*tert*-Butyldimethylsilyl)-ethynyl]-benzaldehyde (18**).** A mixture of 1.11 mL (9.50 mmol) of 2-bromobenzaldehyde, 2.00 g (14.3 mmol) of (*tert*-butyldimethylsilyl)acetylene, 0.333 g (0.480 mmol) of $\text{PdCl}_2(\text{PPh}_3)_2$, and 0.180 g (0.950 mmol) of CuI was stirred in 20 mL of Et_3N at rt for 24 h, concentrated under reduced pressure to remove excess Et_3N , poured into 1.0 M HCl (150 mL), and extracted with CH_2Cl_2 (150 mL x 2). The combined organic layers were washed with brine (100 mL), dried (Na_2SO_4), concentrated under reduced pressure, and purified by chromatography on SiO_2 (95% hexanes: Et_2O) to give 2.24 g (96%) of **18** as a yellow oil: R_f 0.80 (80% hexanes: Et_2O); $^1\text{H NMR}$ δ 10.57 (s, 1 H), 7.90 (d, 1 H, $J = 7.7$ Hz), 7.57-7.43 (m, 3 H), 1.00 (s, 9 H), 0.22 (s, 6 H).



But-2-enoic acid (1-butyl-4-{2-[(*tert*-butyldimethylsilyl)-ethynyl]-phenyl}-4-hydroxybut-2-ynyl)-amide (19**).** A solution of 0.0780 g (0.435 mmol) of **17** in 3 mL of THF was treated

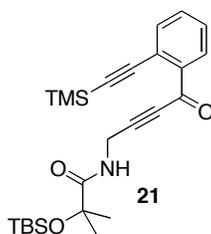
at $-78\text{ }^{\circ}\text{C}$ with 0.80 mL (1.31 mmol) of a 1.6 M solution of *n*-BuLi in hexane, stirred for 15 min at $-78\text{ }^{\circ}\text{C}$, treated with a solution of 0.117 g (0.479 mmol) of **18** in 1.5 mL of THF, warmed to $0\text{ }^{\circ}\text{C}$ over 1 h, and stirred for 2 h. The reaction mixture was quenched with 10% HCl (5 mL) and extracted with EtOAc (2 x 10 mL). The combined organic layers were washed with brine (5 mL), dried (Na_2SO_4), concentrated under reduced pressure, and purified by chromatography on SiO_2 (75% hexanes:EtOAc) to give 0.0830 g (46%) of **19** as a red oil: R_f 0.10 (80% hexanes:EtOAc); $^1\text{H NMR}$ δ 7.66 (d, 1 H, $J = 7.5$ Hz), 7.48 (d, 1 H, $J = 7.5$), 7.38-7.24 (m, 2 H), 6.89-6.78 (m, 1 H), 5.89 (s, 1 H), 5.76-5.69 (m, 2 H), 4.88 (q, 1 H, $J = 6.9$ Hz), 1.84 (d, 3 H, $J = 6.8$ Hz), 1.71-1.66 (m, 2 H), 1.41-1.22 (m, 4 H), 1.01 (s, 9 H), 0.90-0.87 (m, 3 H), 0.21 (s, 6 H).



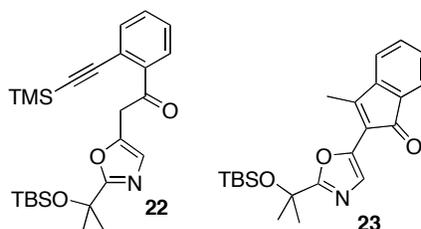
1-{2-[(*tert*-Butyldimethylsilyl)-ethynyl]-phenyl}-2-(4-butyl-2-propenyloxazol-5-yl)-

ethanone (20). A solution of 0.0510 g (0.120 mmol) of **19** and 0.0580 g (0.138 mmol) of Dess-Martin periodinane in 2 mL of CH_2Cl_2 was stirred at rt for 4 h. The light orange, cloudy reaction mixture was filtered through a plug of SiO_2 (80% hexanes:EtOAc). The filtrate was concentrated under reduced pressure to give the crude ketone as an orange oil: R_f 0.48 (60% hexanes:EtOAc); IR (NaCl, film) 3275, 3060, 2955, 2929, 2858, 2213, 2156, 1655, 1534, 1471, 1442, 1362, 1342, 1293, 1248, 1007, 966, 837, 807, 776, 758, 682 cm^{-1} ; $^1\text{H NMR}$ δ 7.60 (dd, 1H, $J = 7.5, 1.1$ Hz), 7.62-7.58 (m, 1 H), 7.52-7.41 (m, 2 H), 6.89 (sextet, 1 H, $J = 6.9$ Hz), 5.84-5.83 (m, 1H), 5.80-5.78 (m, 1 H), 5.13-5.06 (m, 1 H), 1.85 (dd, 3 H, $J = 6.8, 1.6$ Hz), 1.85-1.79 (m, 2 H), 1.50-1.26 (m, 4 H), 1.03 (s, 9 H), 0.92 (t, 3 H, $J = 7.1$ Hz), 0.21 (s, 3 H), 0.20 (s, 3 H); $^{13}\text{C NMR}$ δ 176.5, 164.9, 141.2, 137.9, 135.4, 132.4, 132.2, 128.0, 124.4, 122.7, 103.6, 99.7, 93.1, 81.5, 41.4, 35.1, 27.8, 26.2, 26.1, 25.5, 22.4, 22.2, 17.7, 13.8, -4.7, -4.8; HRMS (ES) m/z calculated for $\text{C}_{26}\text{H}_{35}\text{NO}_2\text{Si}$ 421.2437, found: 421.2433. The crude orange oil was stirred with 0.500 g (1,000%, w/w) of SiO_2 in 4 mL of CH_2Cl_2 at rt for 48 h, filtered, and concentrated under reduced

pressure to give 0.0290 g (58%, 2 steps from **19**) of **20** as a red oil: R_f 0.40 (80% hexanes:EtOAc); IR (NaCl, film) 2955, 2930, 2857, 2360, 2342, 2156, 1694, 1665, 1640, 1591, 1535, 1471, 1441, 1362, 1327, 1250, 1200, 1096, 1051, 964, 838, 808, 776, 761, 680 cm^{-1} ; ^1H NMR δ 7.60-7.57 (m, 2 H), 7.47-7.35 (m, 2 H), 6.61 (dq, 1 H, $J = 15.9, 6.9$ Hz), 6.21 (dq, 1 H, $J = 15.9, 1.5$ Hz), 4.49 (s, 2 H), 2.38 (t, 2 H, $J = 7.5$ Hz), 1.89 (dd, 3 H, $J = 6.9, 1.5$ Hz), 1.52 (quinted, 2 H, $J = 7.5$ Hz), 1.29 (sextet, 2 H, $J = 7.5$ Hz), 1.00 (s, 9 H), 0.88 (t, 3 H, $J = 7.2$ Hz), 0.21 (s, 6 H); ^{13}C NMR δ 197.8, 160.2, 140.7, 138.9, 134.5, 134.3, 131.3, 128.8, 128.6, 121.2, 118.1, 104.2, 100.2, 38.6, 31.1, 26.2, 25.7, 22.5, 18.5, 16.9, 14.0, -4.7; MS (EI) m/z (rel intensity) 421 (M^+ , 18), 365 (11), 243 (100), 194 (20), 187 (48), 178 (28), 143 (30), 136 (18); HRMS (EI) m/z calculated for $\text{C}_{26}\text{H}_{35}\text{NO}_2\text{Si}$ 421.2437, found 421.2433.



2-(tert-Butyldimethylsilyloxy)-2-methyl-N-[4-oxo-4-(2-trimethylsilylethynylphenyl)-but-2-ynyl]-propionamide (21). A mixture of 1.11 g (2.43 mmol) of the propargylic alcohol and 1.15 g (2.71 mmol) of Dess-Martin reagent in 40 mL of CH_2Cl_2 was stirred at rt for 15 h, filtered, concentrated under reduced pressure, and purified by chromatography on SiO_2 to yield 1.05 g (95%) of **21** as an orange oil: R_f 0.47 (75% hexanes:EtOAc); IR (neat) 3434, 3073, 2955, 2858, 2232, 2157, 1655, 1592, 1561, 1501, 1250, 1167, 1033 cm^{-1} ; ^1H NMR δ 8.05 (dd, 1 H, $J = 7.7, 1.0$ Hz), 7.60 (dd, 1 H, $J = 7.7, 1.2$ Hz), 7.50 (td, 1 H, $J = 7.5, 1.4$ Hz), 7.41 (td, 1 H, $J = 7.7, 1.4$ Hz), 7.13 (br, 1 H), 4.31 (d, 2 H, $J = 5.4$ Hz), 1.47 (s, 6 H), 0.92 (s, 9 H), 0.28 (s, 9 H), 0.18 (s, 6 H); ^{13}C NMR δ 176.4, 176.3, 138.0, 135.0, 132.4, 131.7, 128.1, 122.5, 102.9, 101.1, 89.6, 81.8, 76.8, 29.4, 27.8, 25.8, 17.9, -0.2, -2.4; MS (EI) m/z (rel intensity) 455 (M^+ , 2), 440 (6), 399 (42), 398 (99), 340 (27), 173 (100); HRMS (EI) m/z calculated for $\text{C}_{24}\text{H}_{34}\text{NO}_3\text{Si}_2$ ($\text{M}-\text{CH}_3$) 440.2077, found 440.2070.

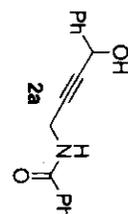
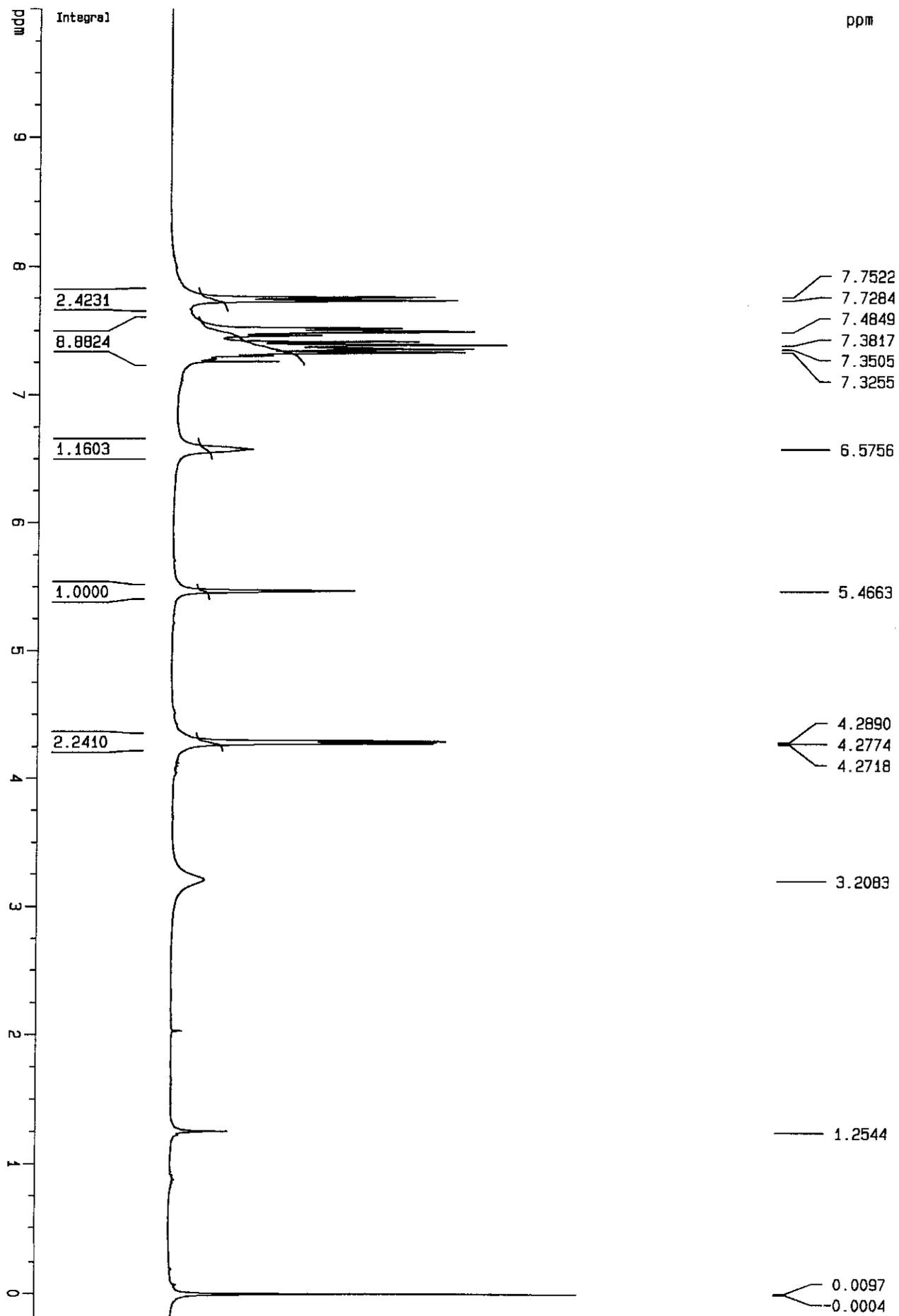


2-{2-[1-(*tert*-Butyldimethylsilyloxy)-1-methylethyl]-oxazol-5-yl}-1-(2-

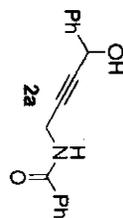
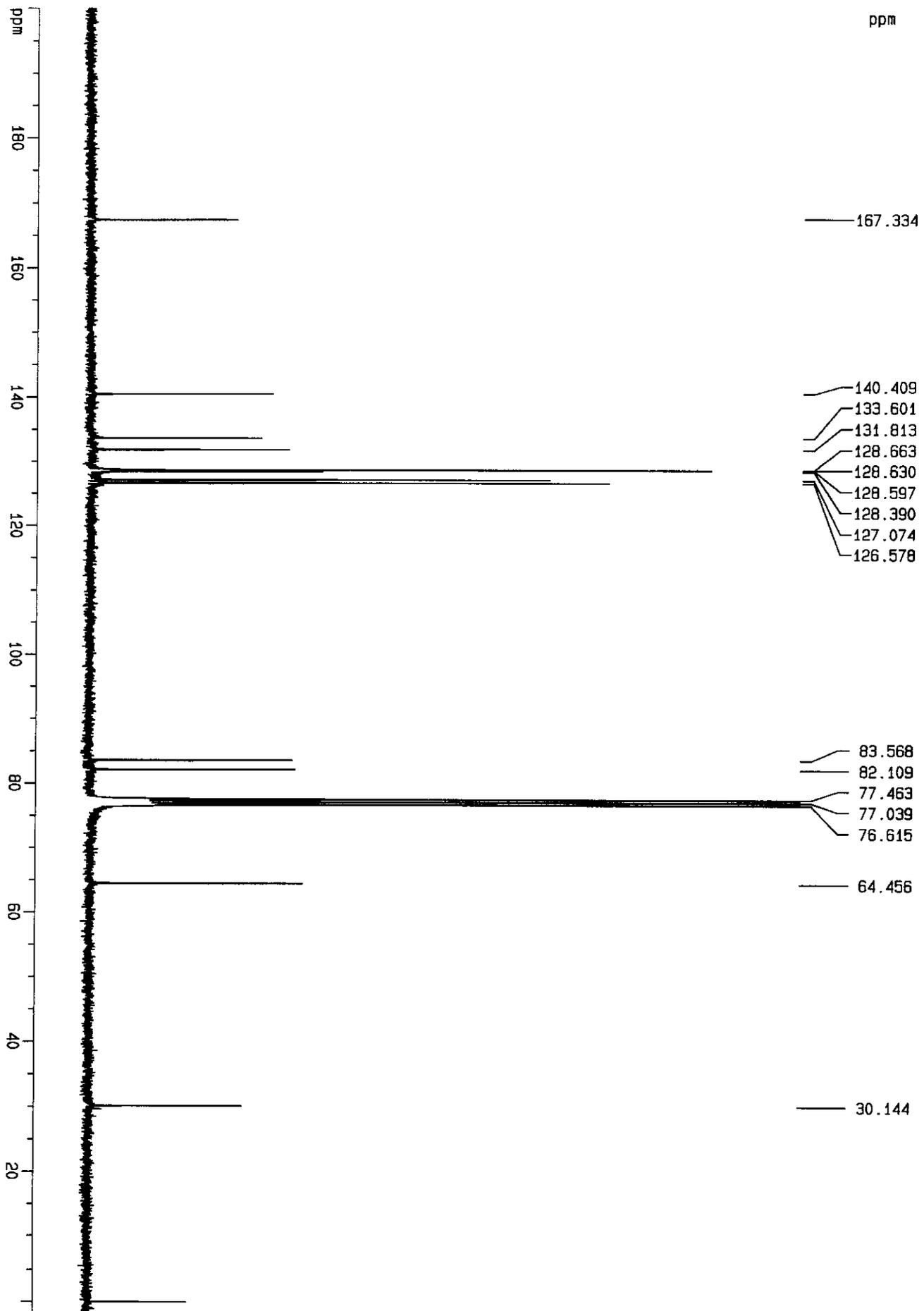
trimethylsilylphenyl)-ethanone (22). A mixture of 0.740 g (1.62 mmol) of **21** and 3.70 g of SiO₂ in 25 mL of CH₂Cl₂ was stirred at rt for 48 h, filtered, concentrated under reduced pressure, and purified by chromatography on SiO₂ to yield 0.112 g (18%) of **21** as a red amorphous solid, 0.255 g (34%) of **22** as an orange oil, and 0.234 g (32%) of **23**. **22**: R_f 0.54 (75% hexanes:EtOAc); IR (neat) 2957, 2857, 2157, 1694, 1592, 1556, 1473, 1251, 1172, 1134, 1044, 1004 cm⁻¹; ¹H NMR δ 7.66 (d, 1 H, *J* = 7.5 Hz), 7.57 (d, 1 H, *J* = 7.3 Hz), 7.48-7.35 (m, 2 H), 6.95 (s, 1 H), 4.61 (s, 2 H), 1.61 (s, 6 H), 0.84 (s, 9 H), 0.27 (s, 9 H), -0.12 (s, 6 H); ¹³C NMR δ 196.7, 167.3, 145.2, 140.3, 134.2, 131.4, 128.8, 128.7, 124.9, 121.1, 103.6, 101.7, 70.6, 39.2, 29.4, 25.7, 17.9, -0.4, -3.6; MS (EI) *m/z* (rel intensity) 455 (M⁺, 0.1), 442 (1), 441 (2), 440 (6), 400 (15), 399 (40), 398 (100), 341 (9), 340 (30), 201 (35); HRMS (EI) *m/z* calculated for C₂₄H₃₄NO₃Si₂ (M-CH₃) 440.2077, found 440.2081.

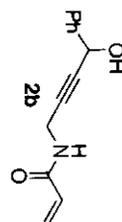
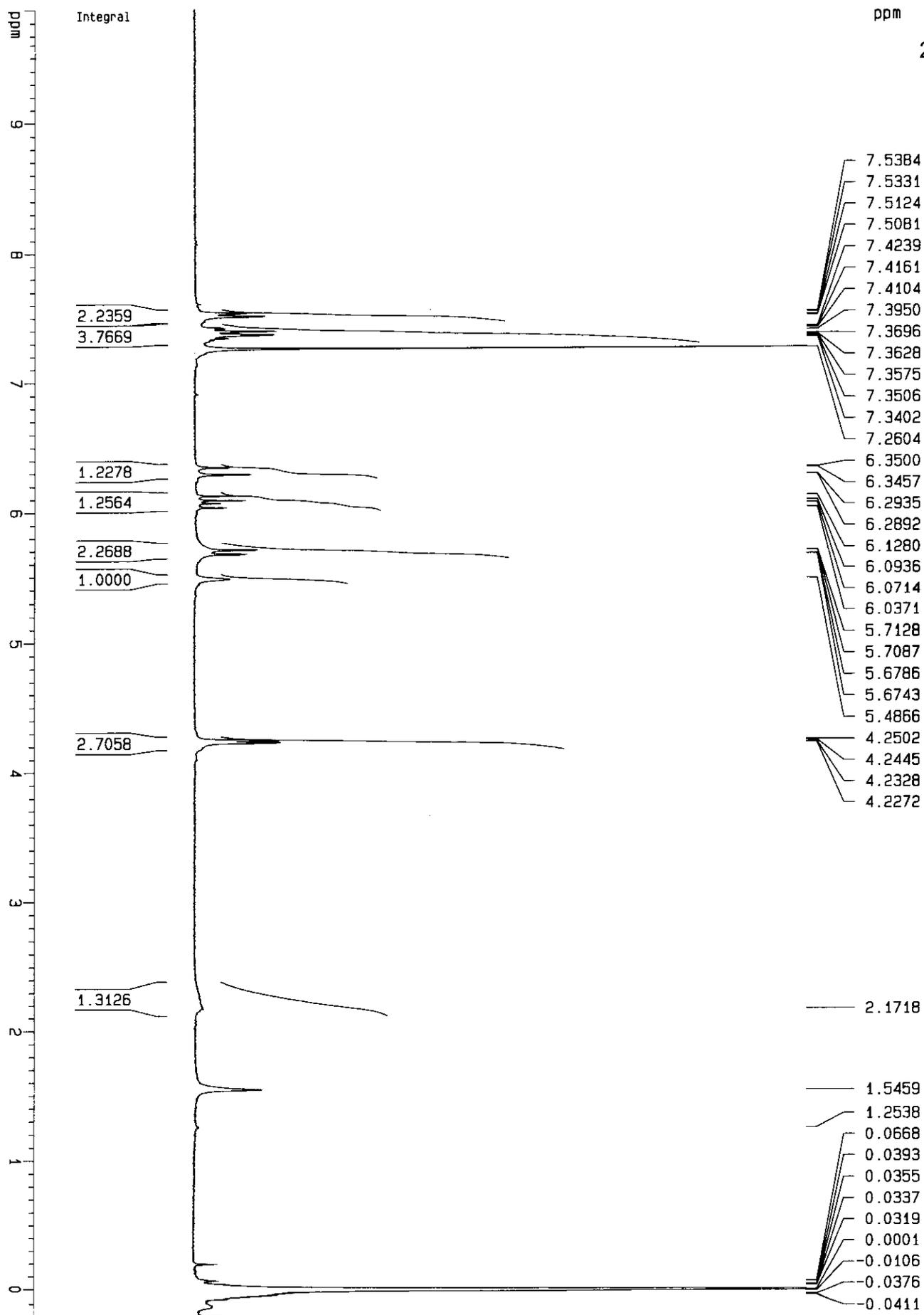
2-{2-[1-(*tert*-Butyldimethylsilyloxy)-1-methylethyl]-oxazol-5-yl}-3-methylinden-1-one

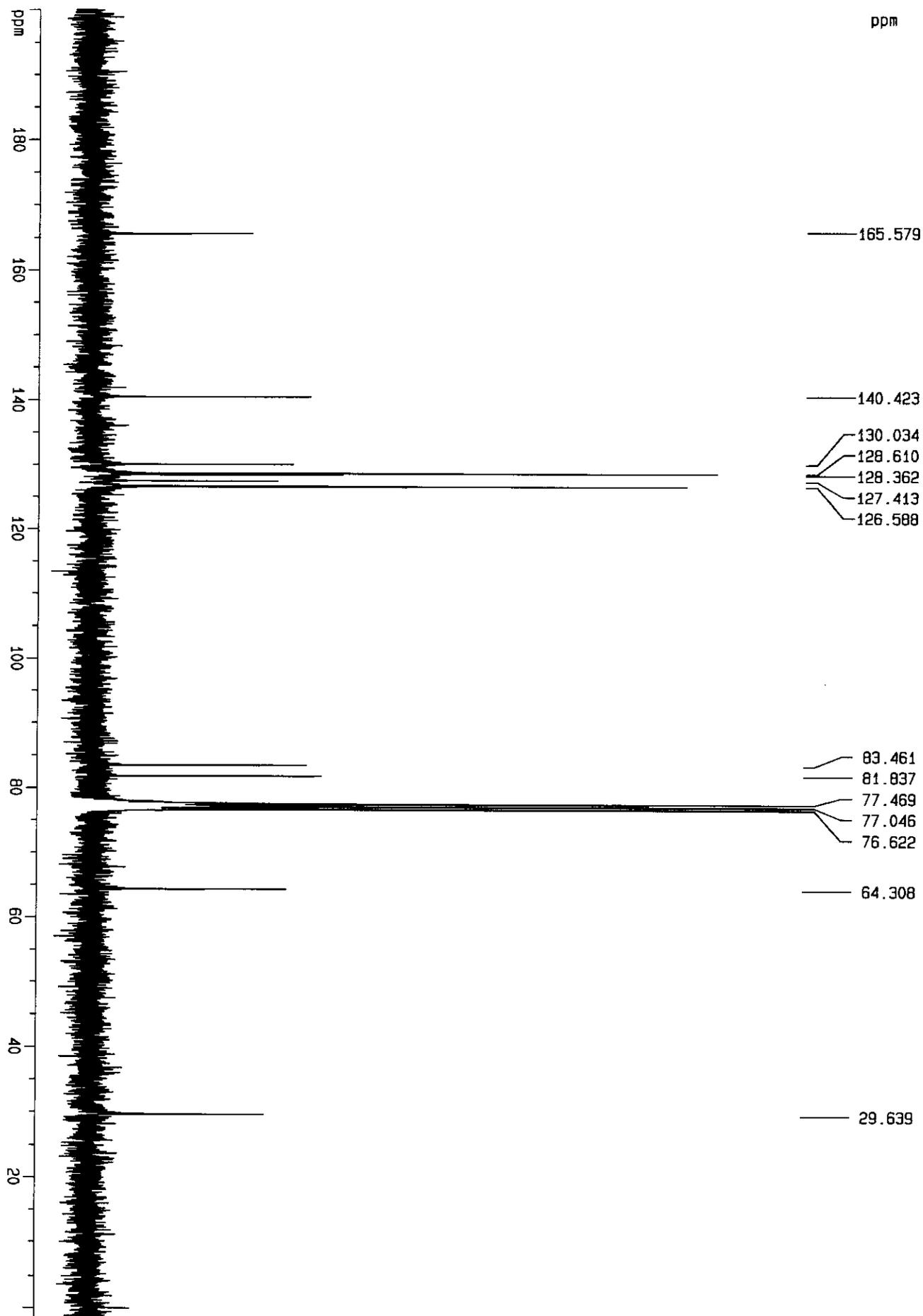
(23). R_f 0.67 (75% hexanes:EtOAc); IR (neat) 2960, 2928, 2858, 1714, 1603, 1562, 1455, 1383, 1367, 1260, 1173, 1037 cm⁻¹; ¹H NMR δ 7.63 (s, 1 H), 7.46 (t, 1 H, *J* = 7.2 Hz), 7.40 (d, 1 H, *J* = 7.8 Hz), 7.24 (t, 1 H, *J* = 7.3 Hz), 7.18 (d, 1 H, *J* = 7.2 Hz), 2.52 (s, 3 H), 1.71 (s, 6 H), 0.87 (s, 9 H), -0.07 (s, 6 H); ¹³C NMR δ 194.3, 167.4, 152.6, 146.6, 145.2, 134.3, 131.1, 129.5, 126.8, 122.7, 121.3, 120.3, 71.2, 29.7, 26.0, 18.3, 13.1, -3.0; MS (EI) *m/z* (rel intensity) 368 (15), 328 (11), 327 (35), 326 ([M-*t*-Bu]⁺, 100), 270 (5), 269 (20), 268 (76); HRMS (EI) *m/z* calculated for C₁₈H₂₀NO₃Si (M-C(CH₃)₃) 326.1212, found 326.1211.

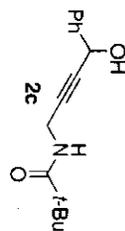
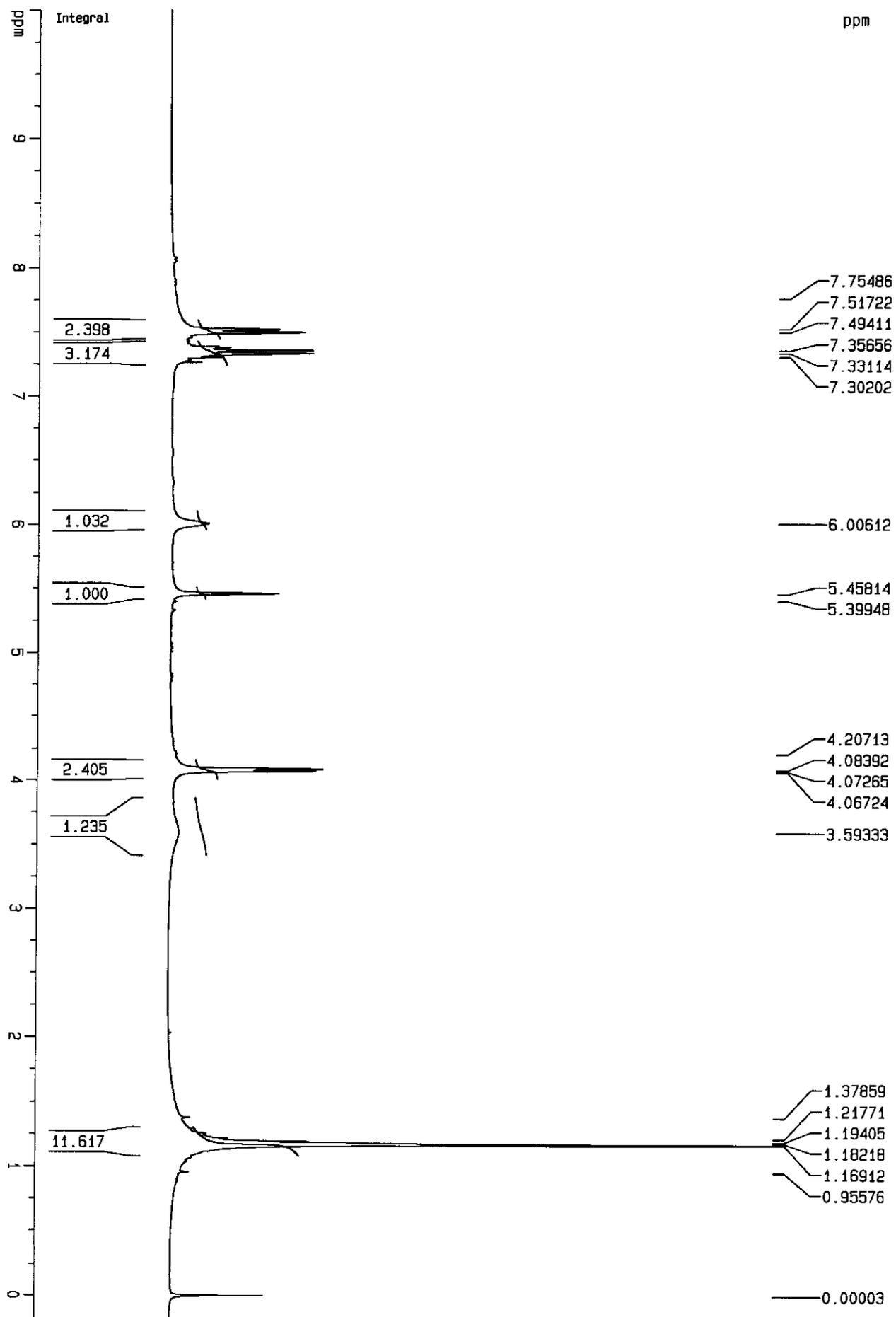


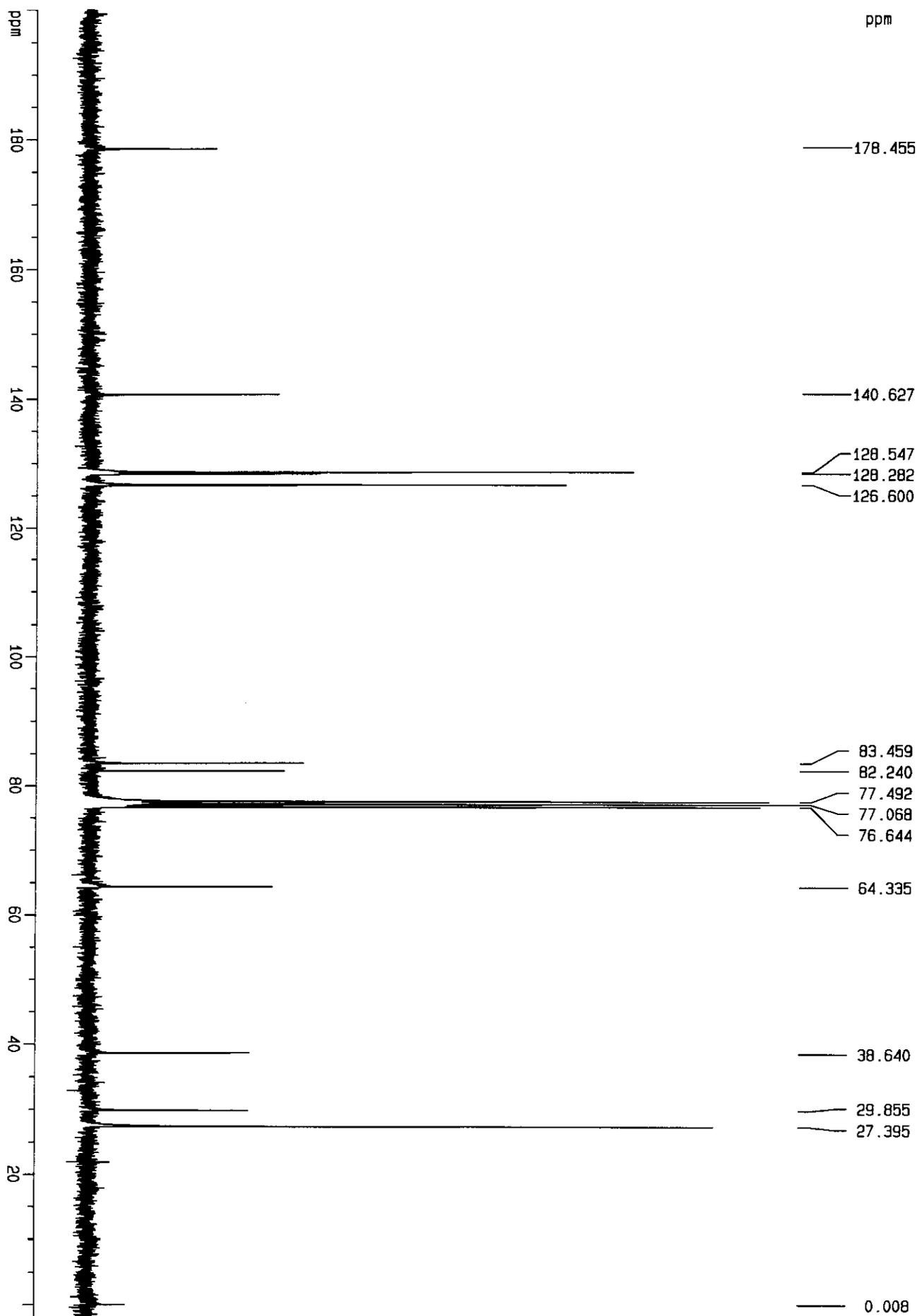
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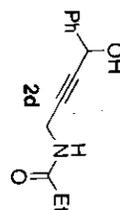
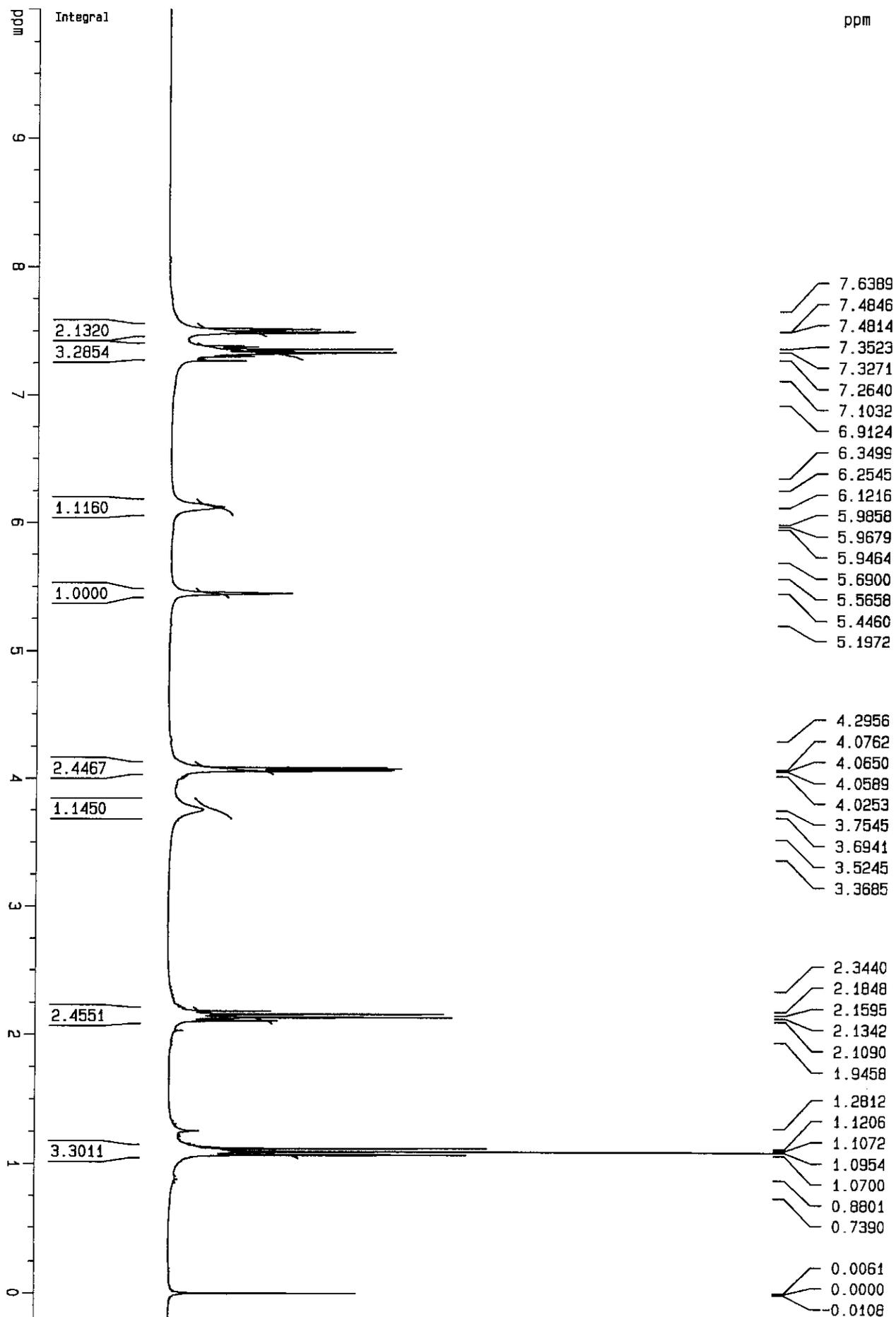




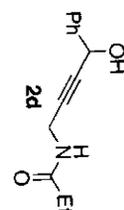
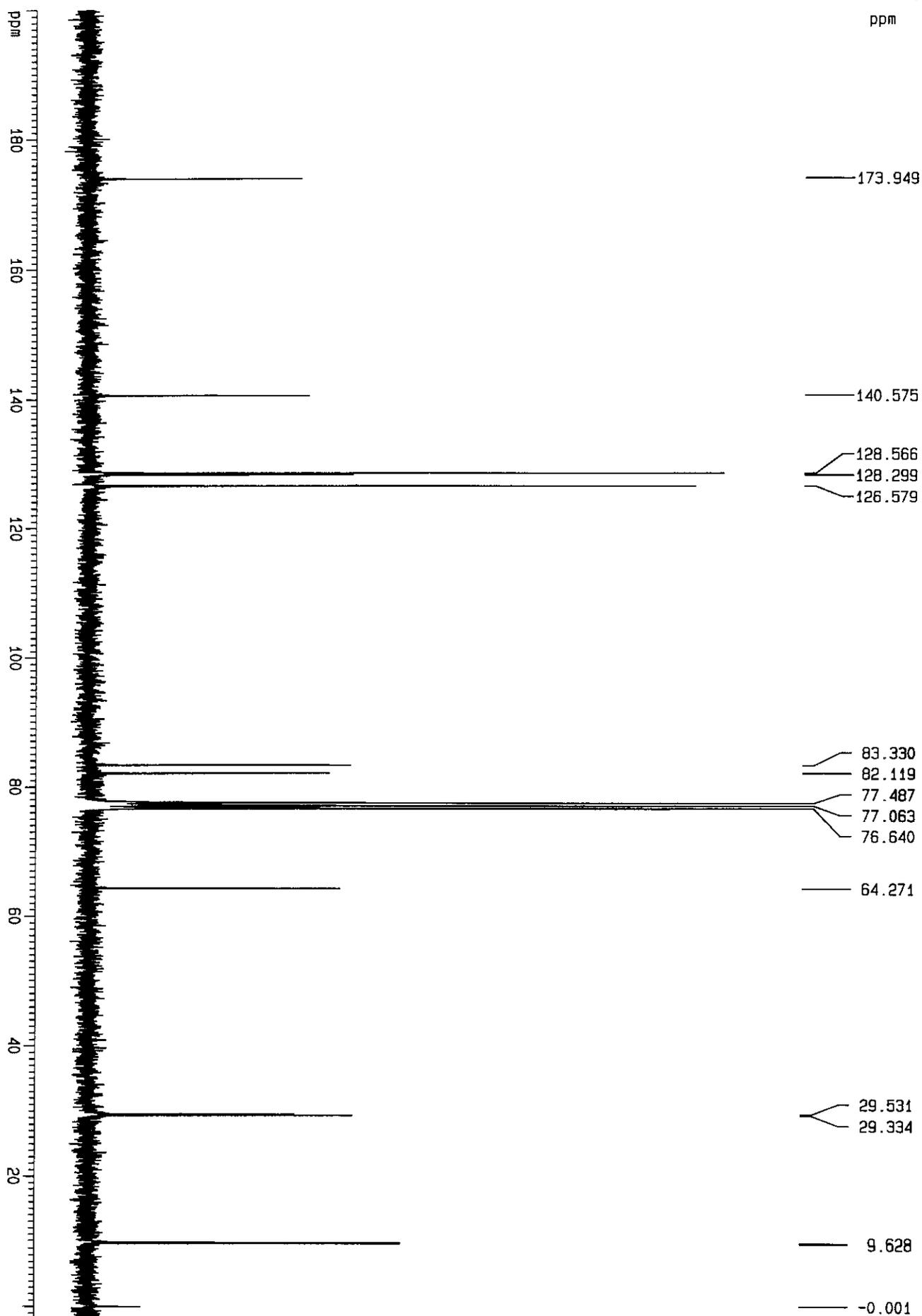


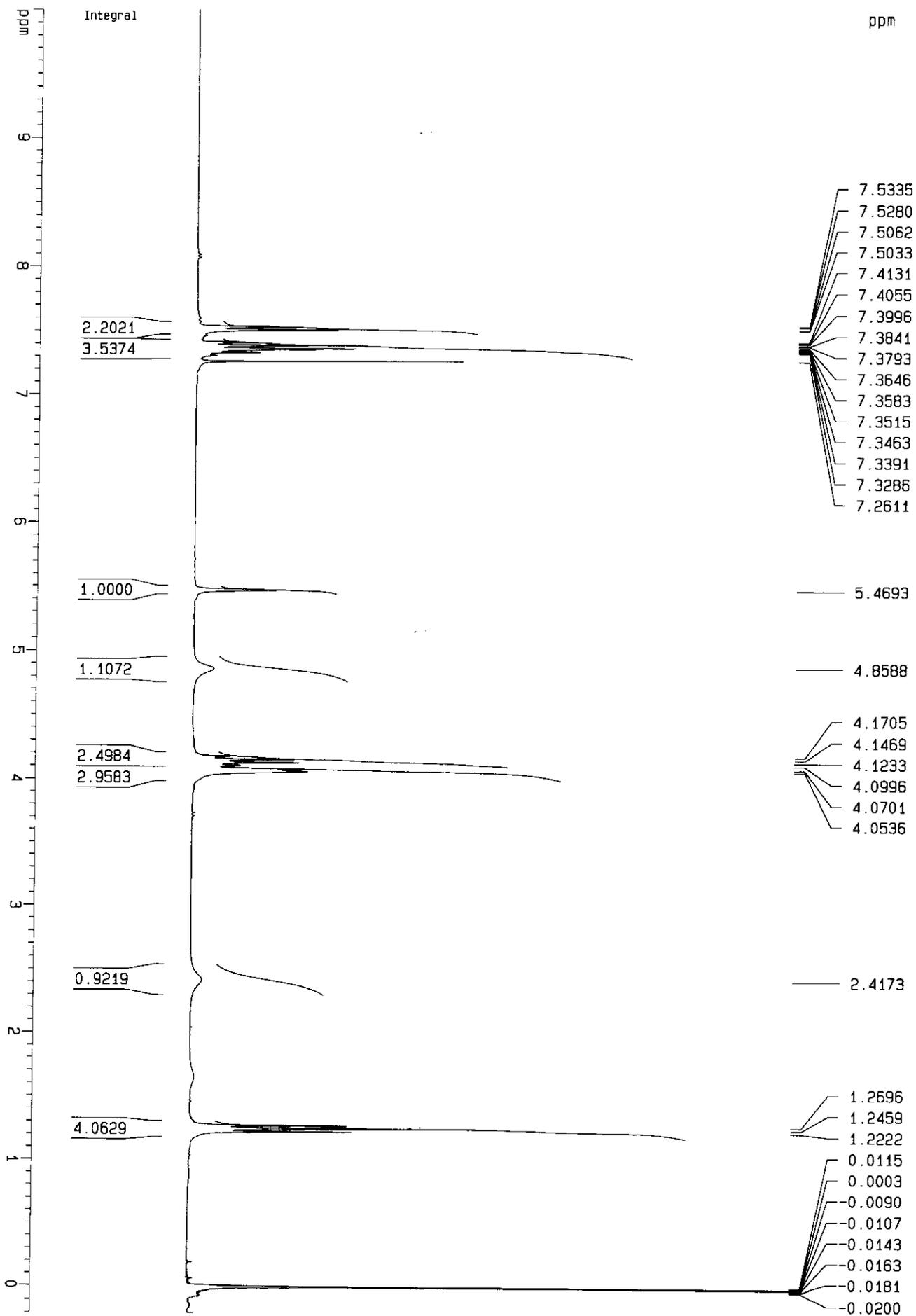




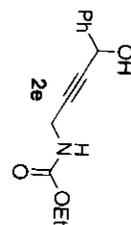
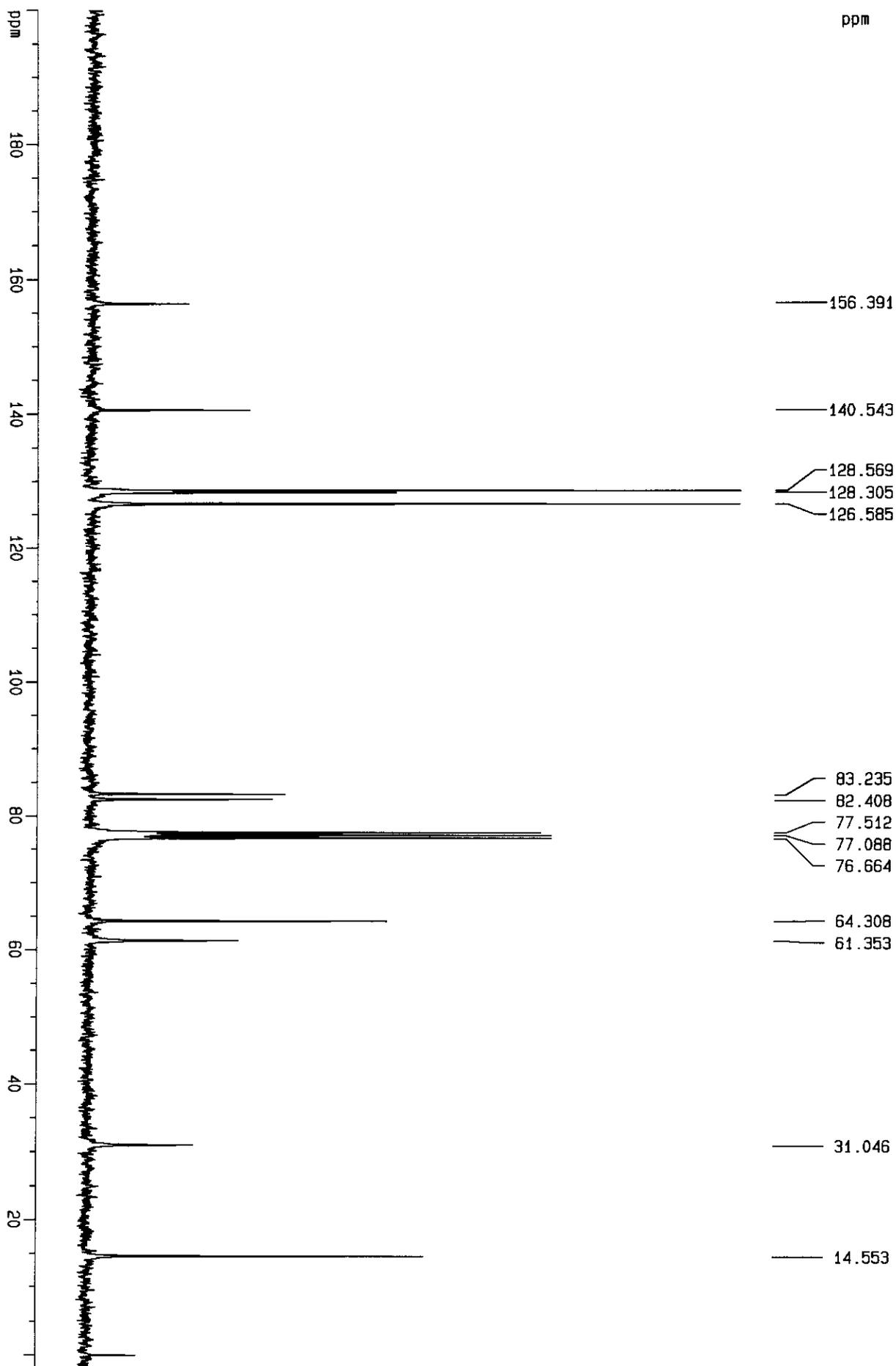


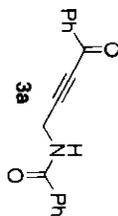
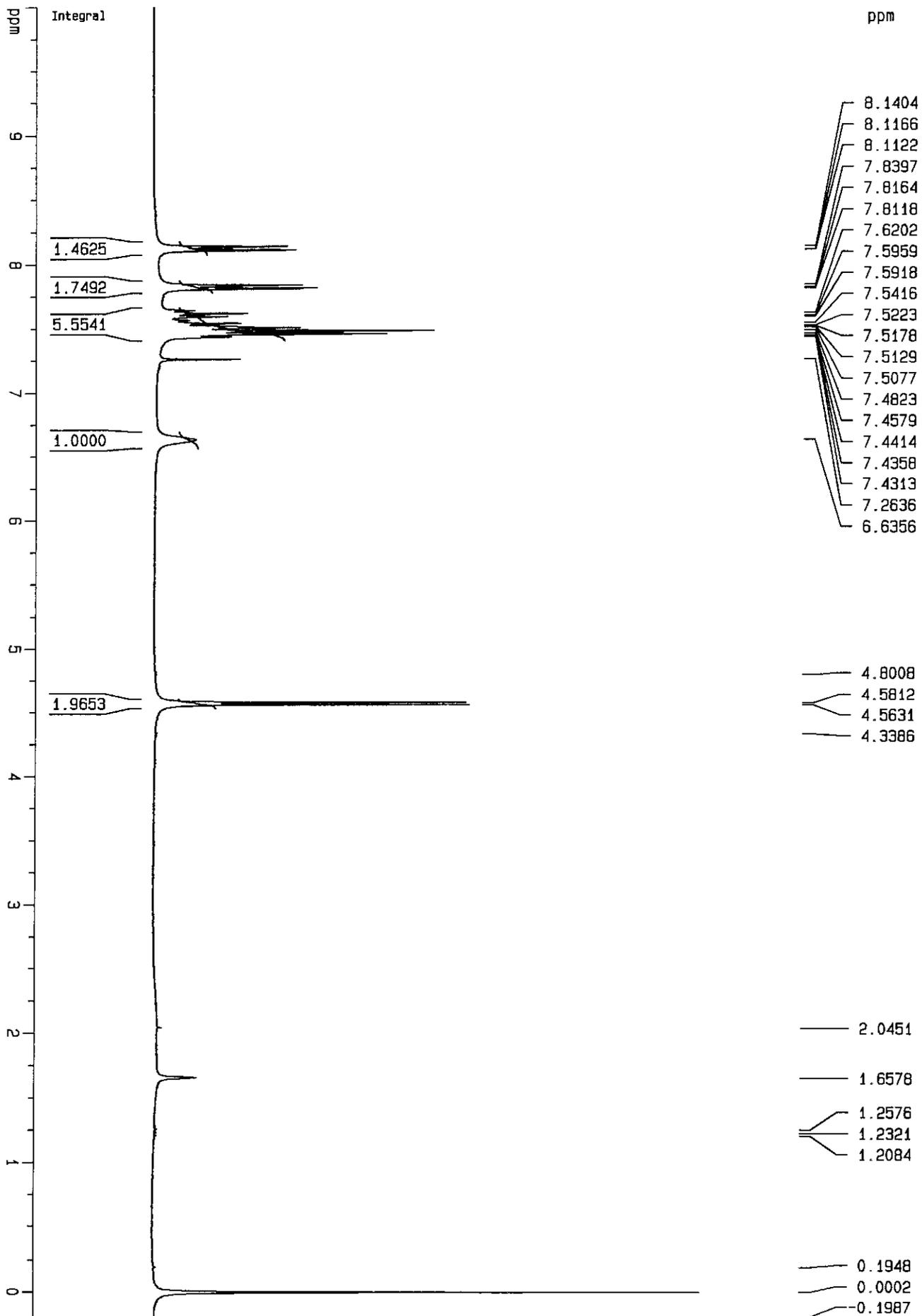
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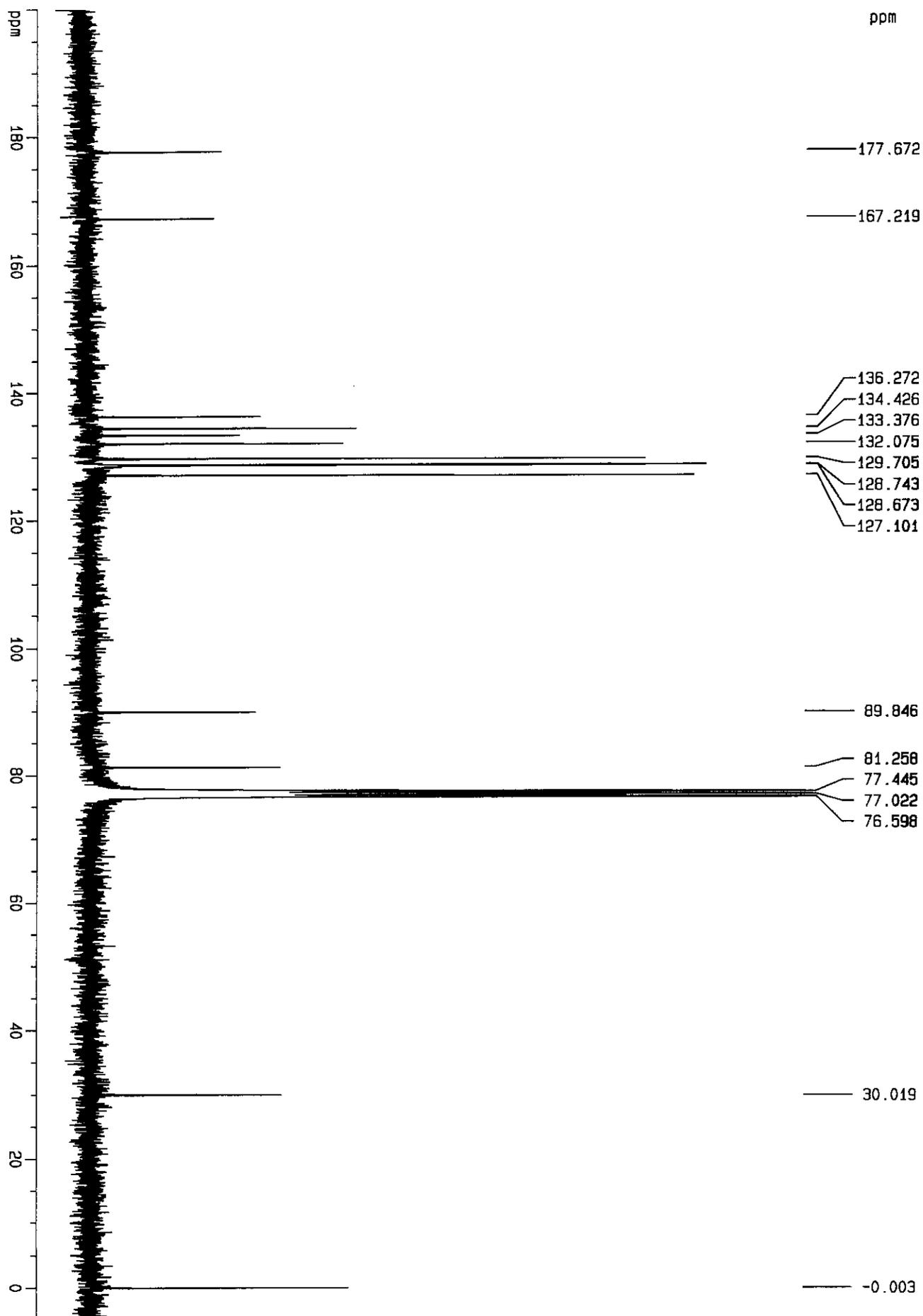


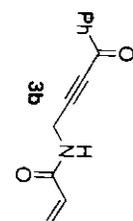
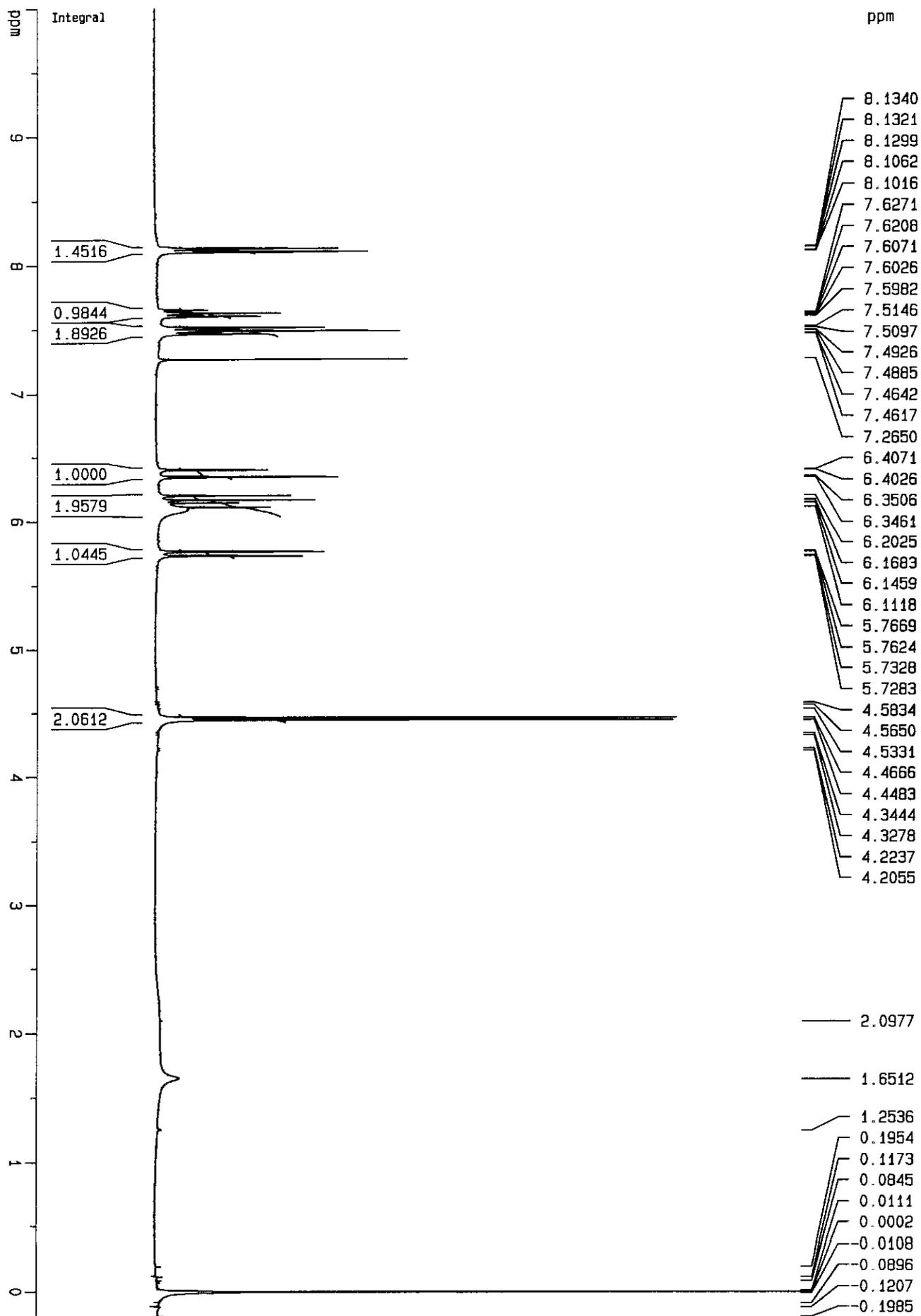
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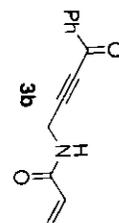
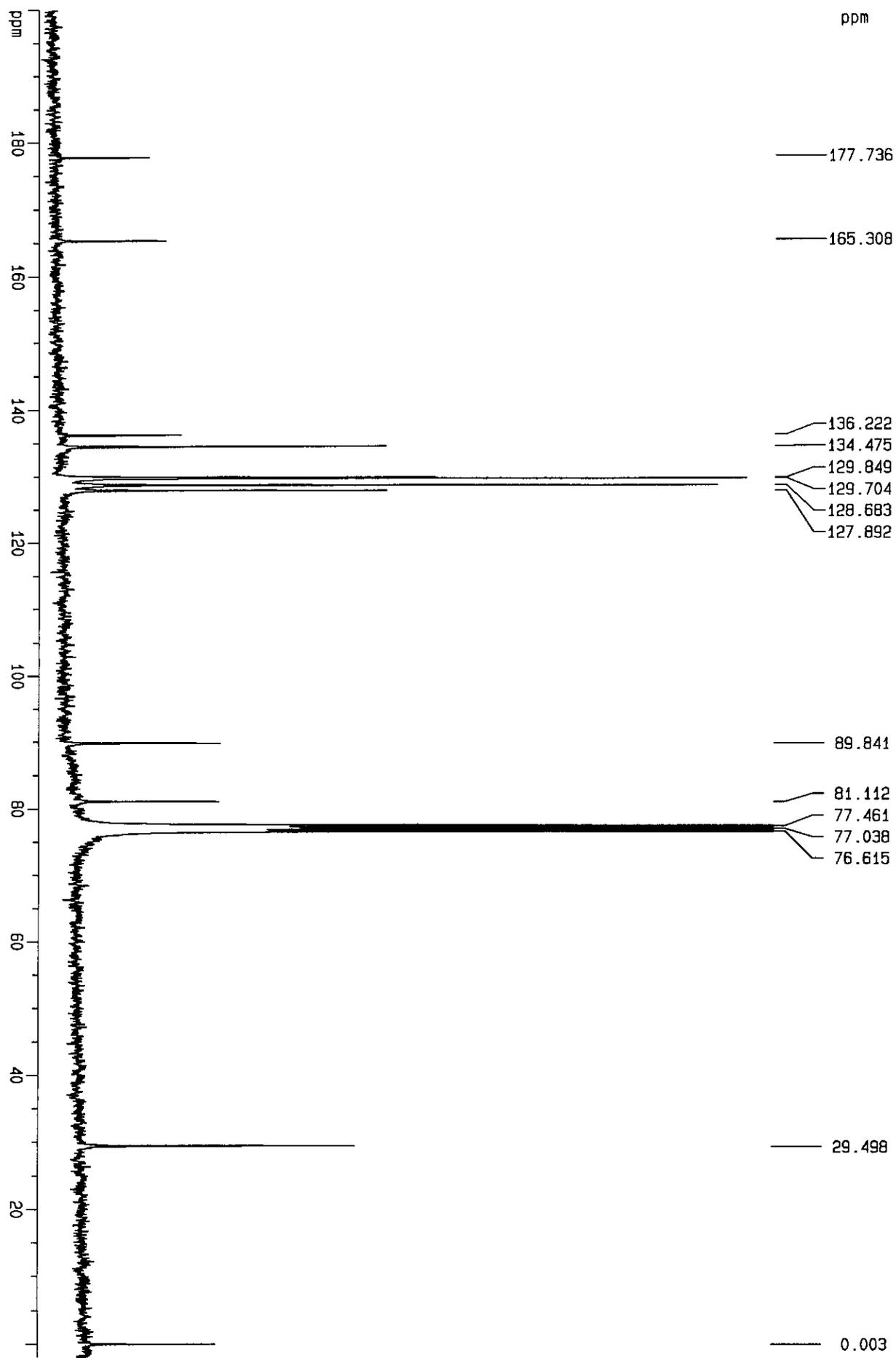


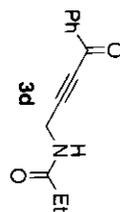
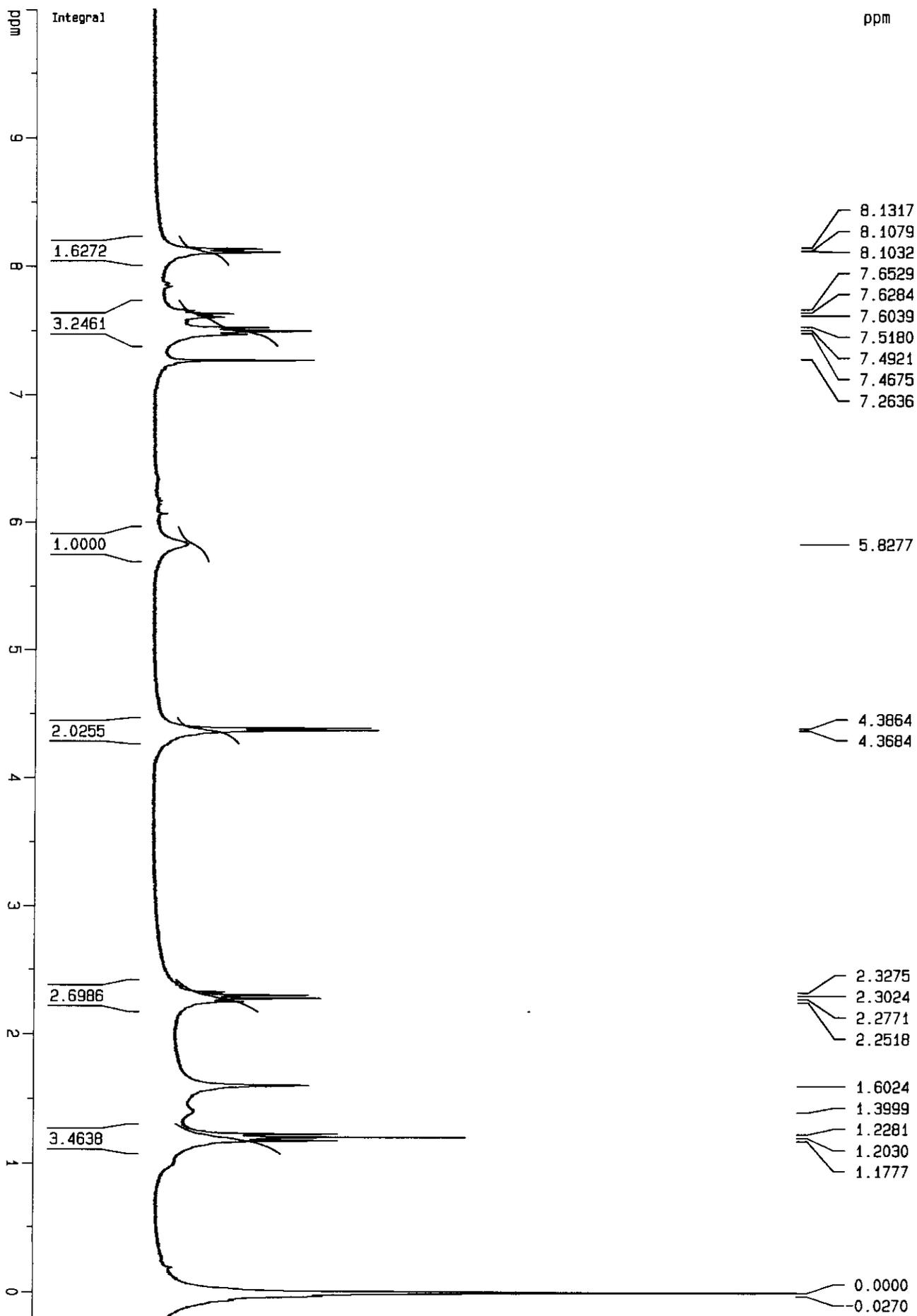


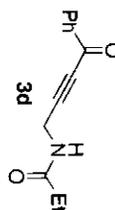
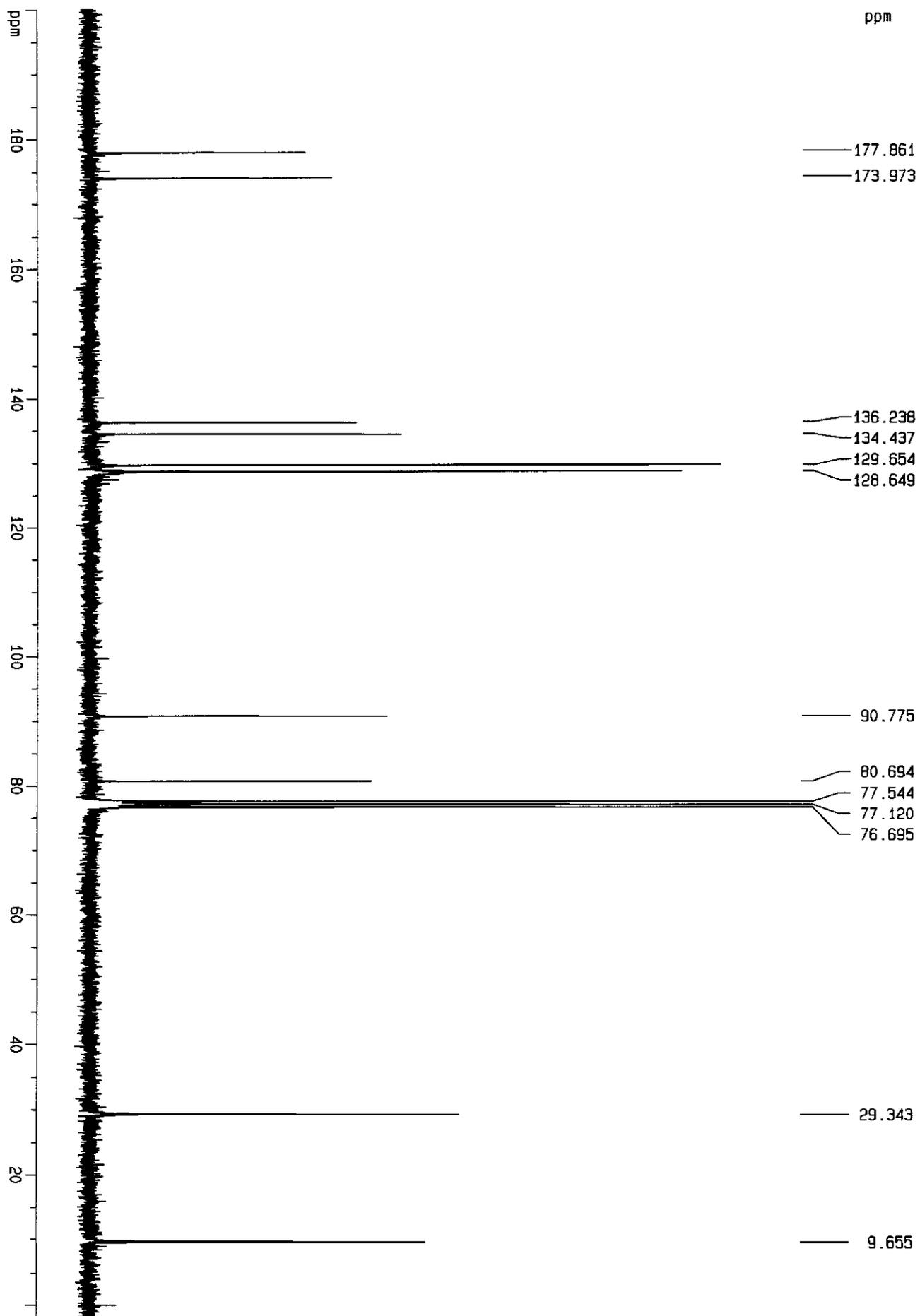
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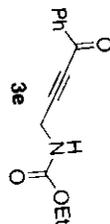
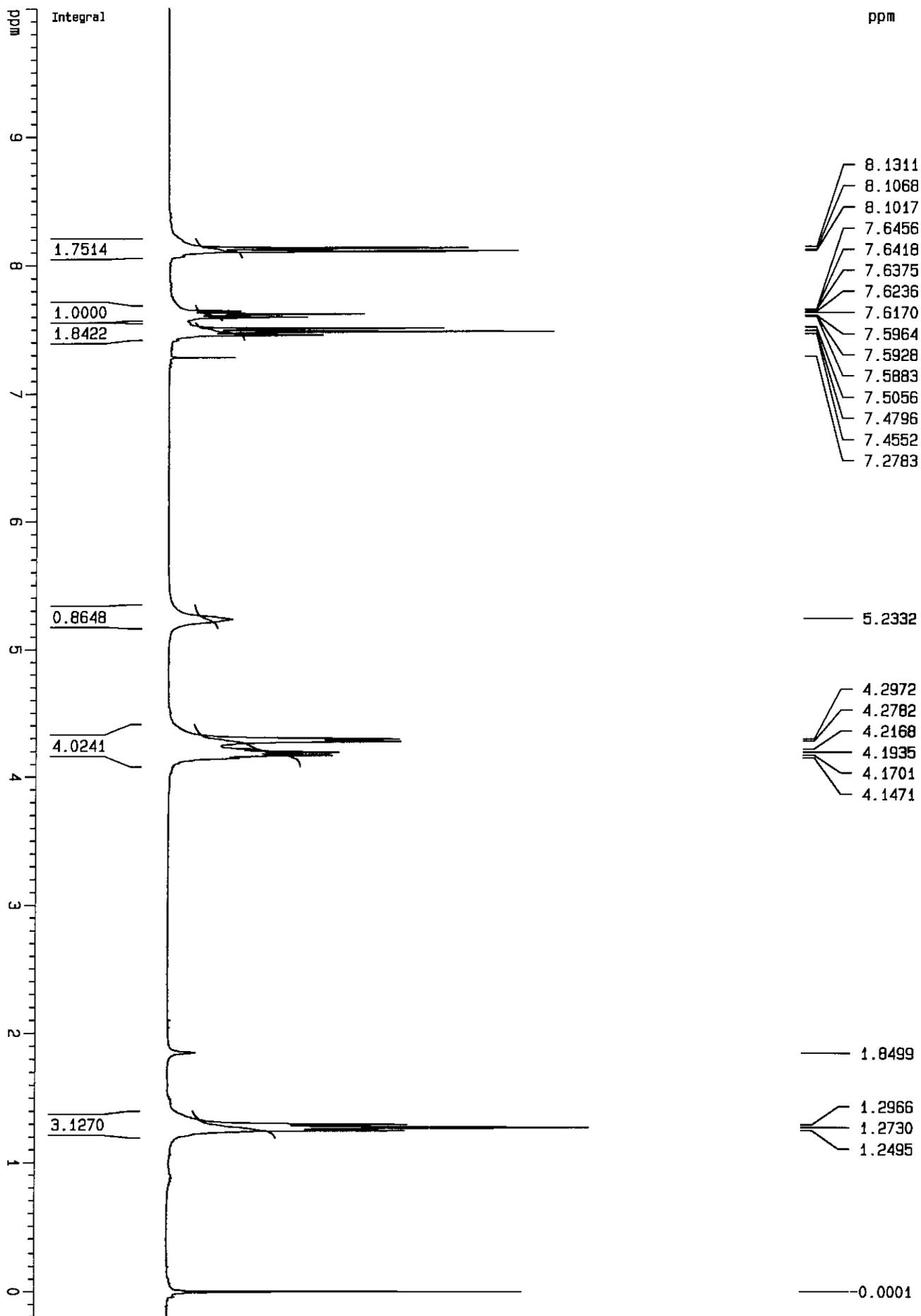




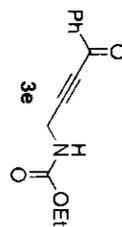
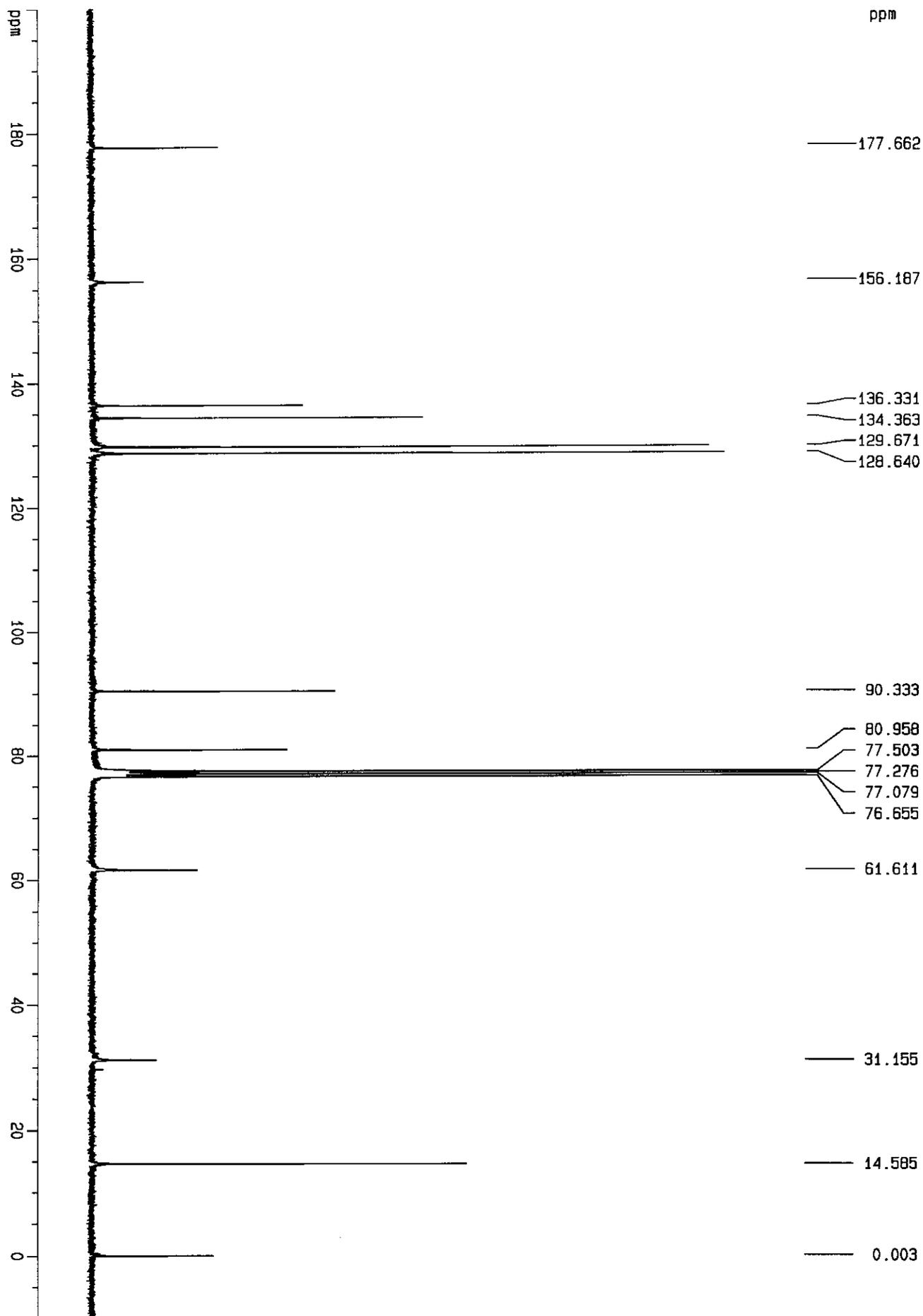


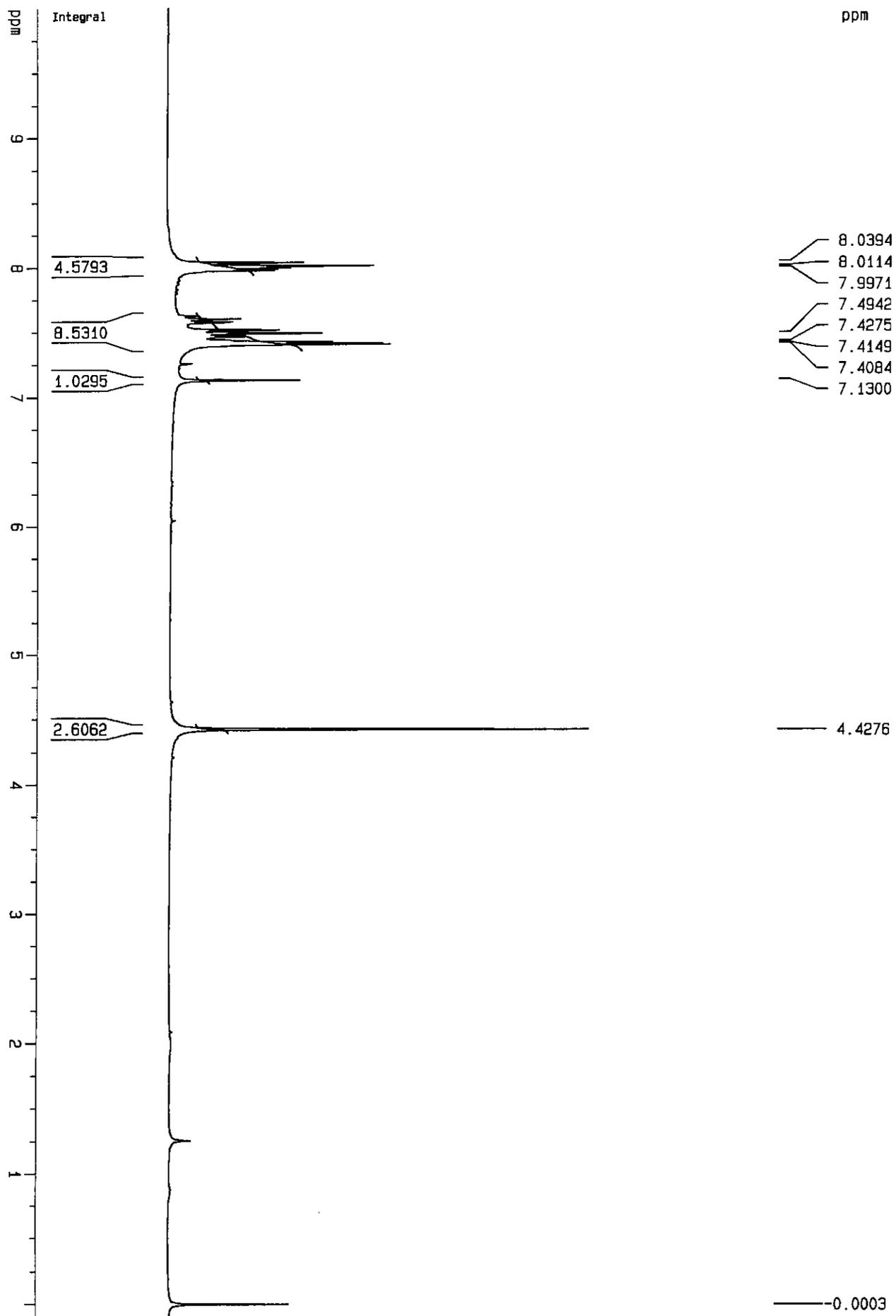


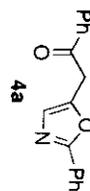
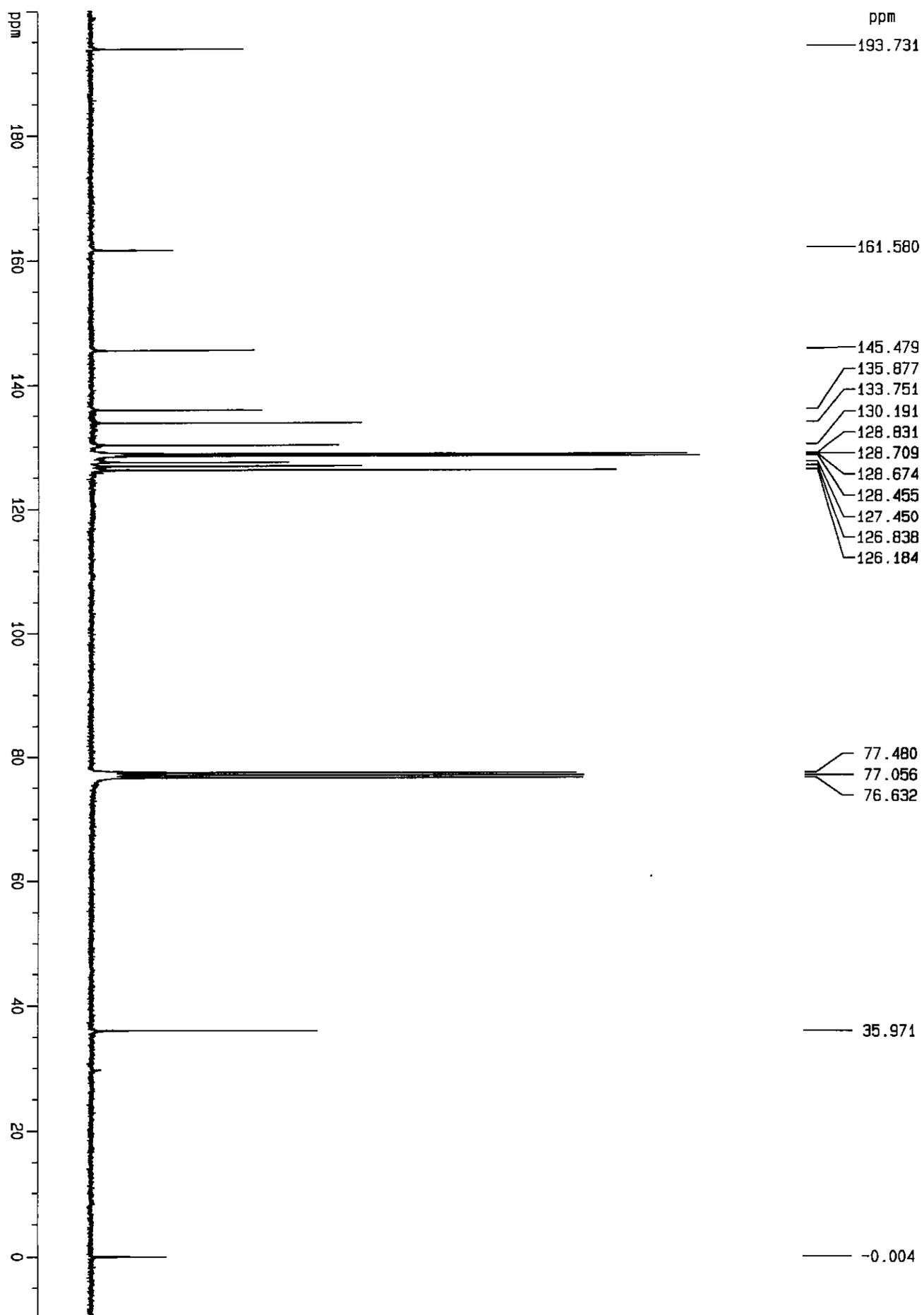


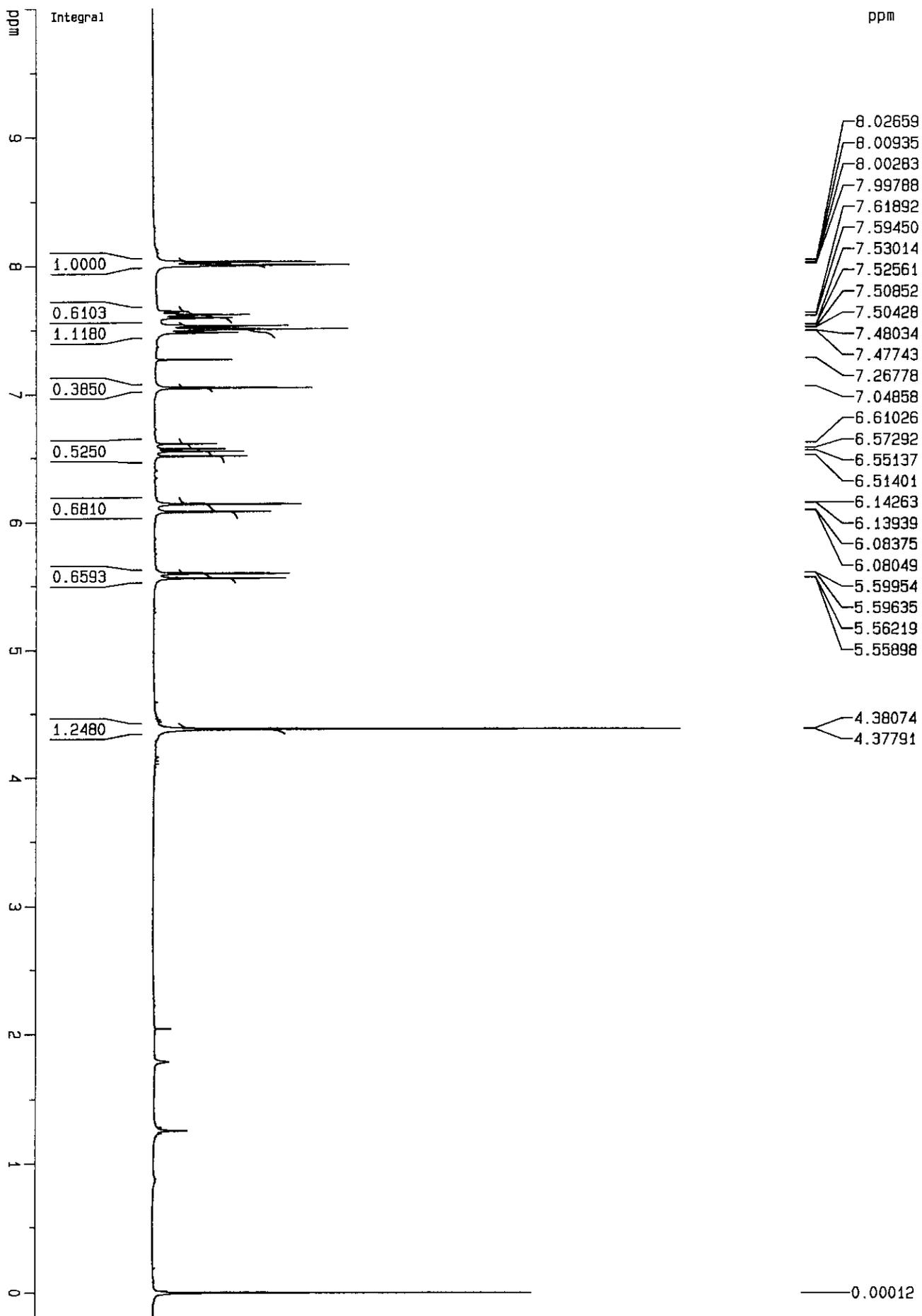


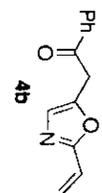
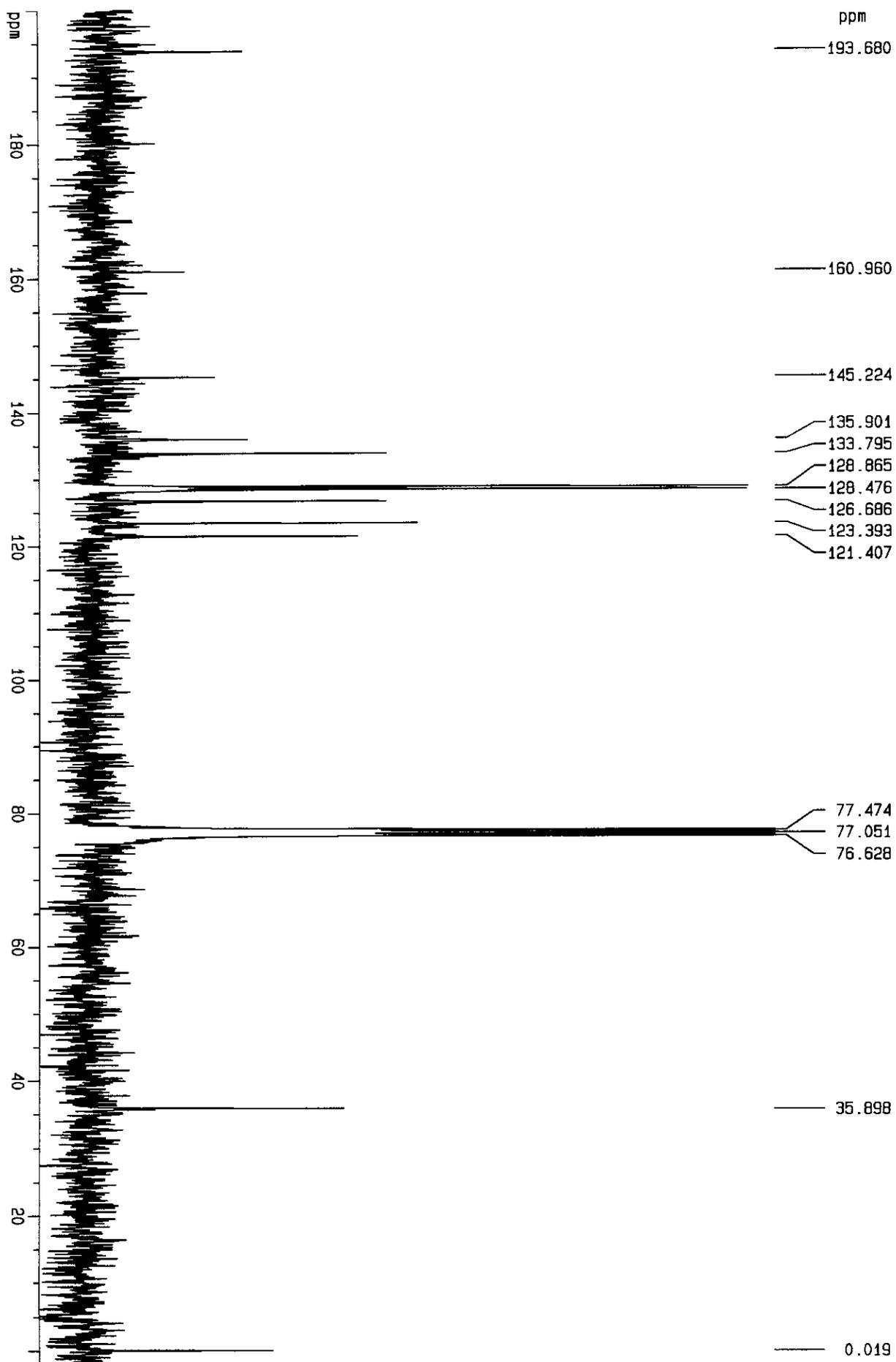
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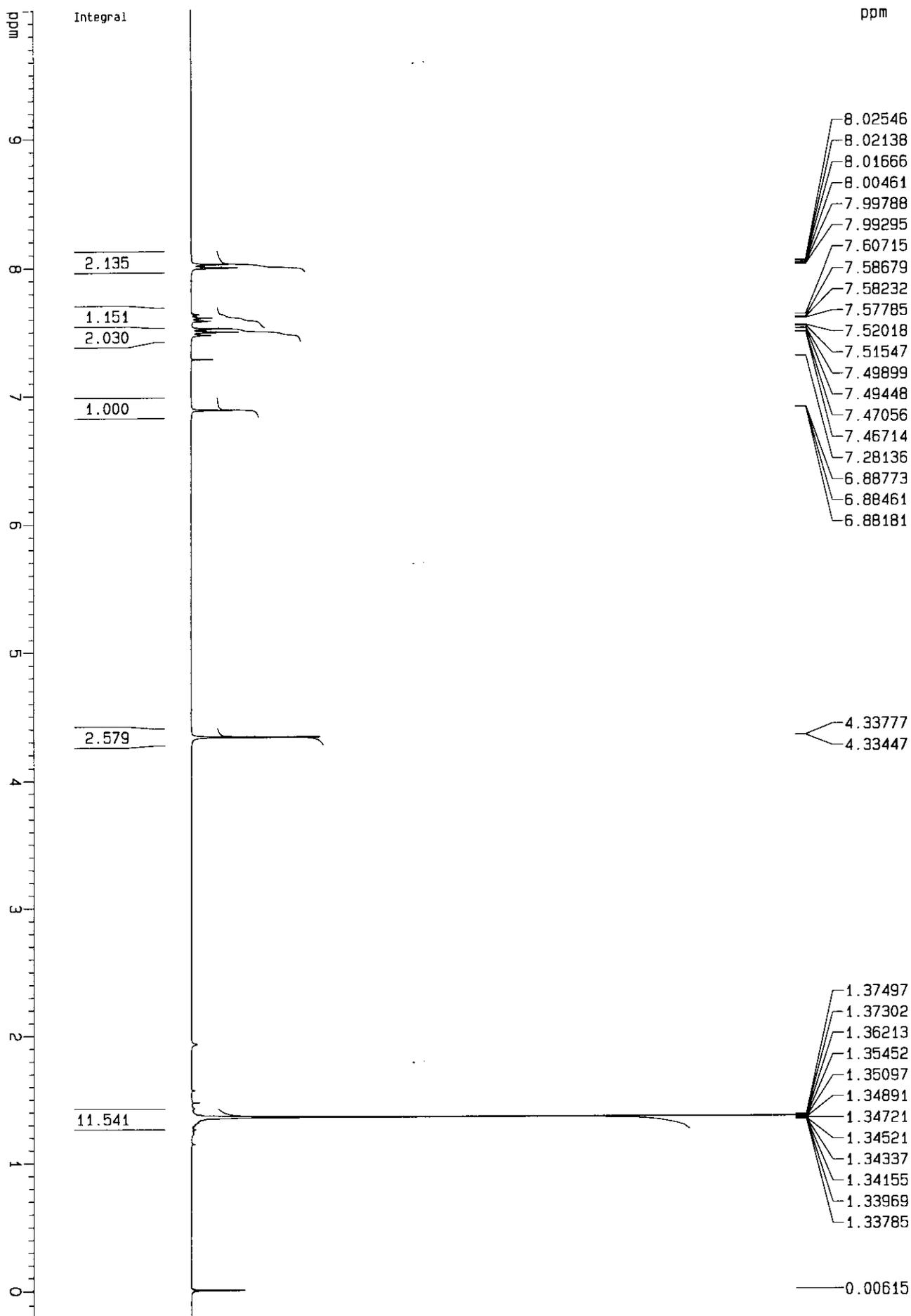


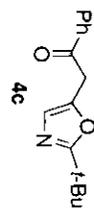
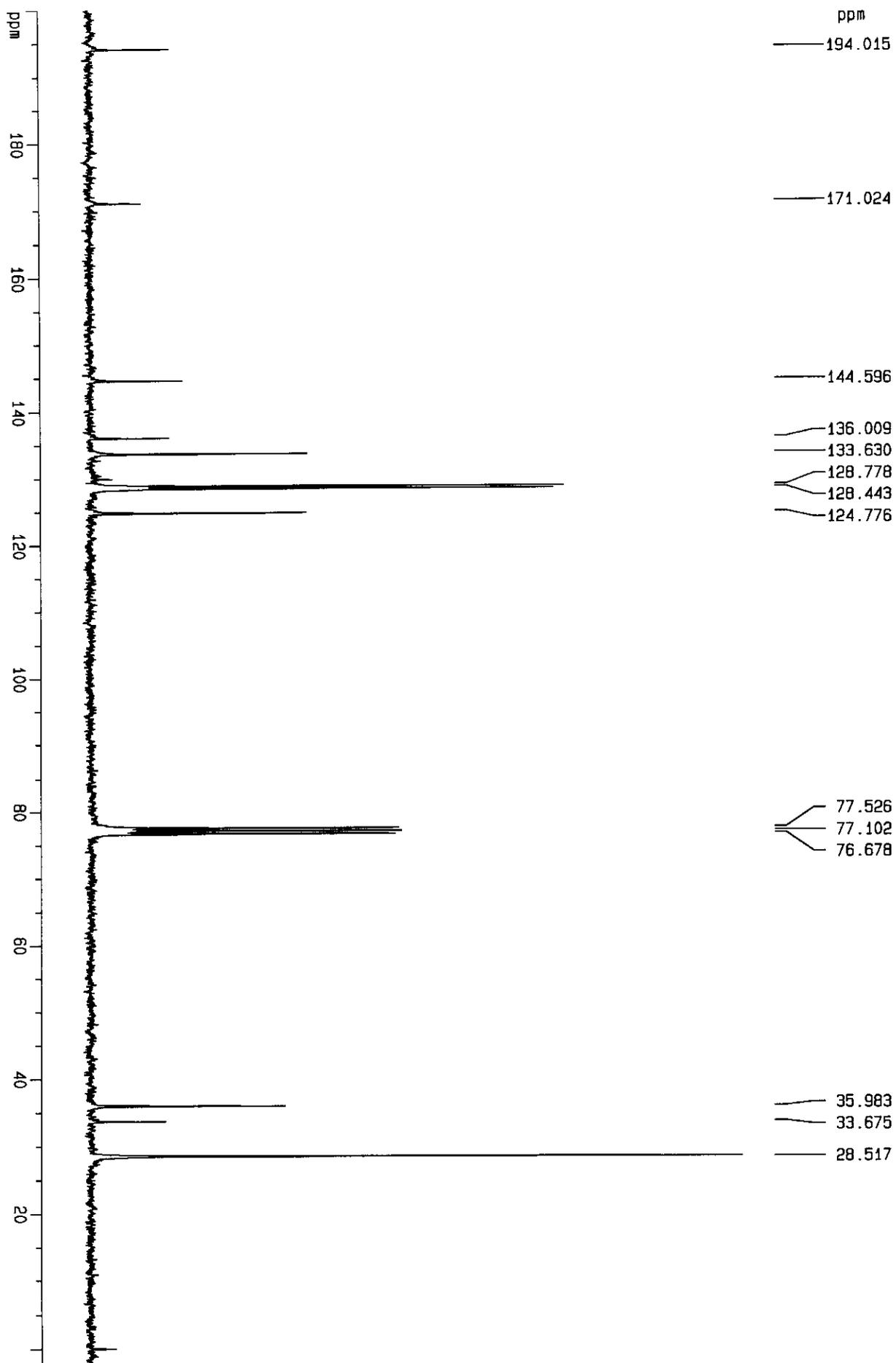


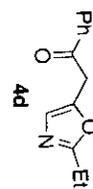
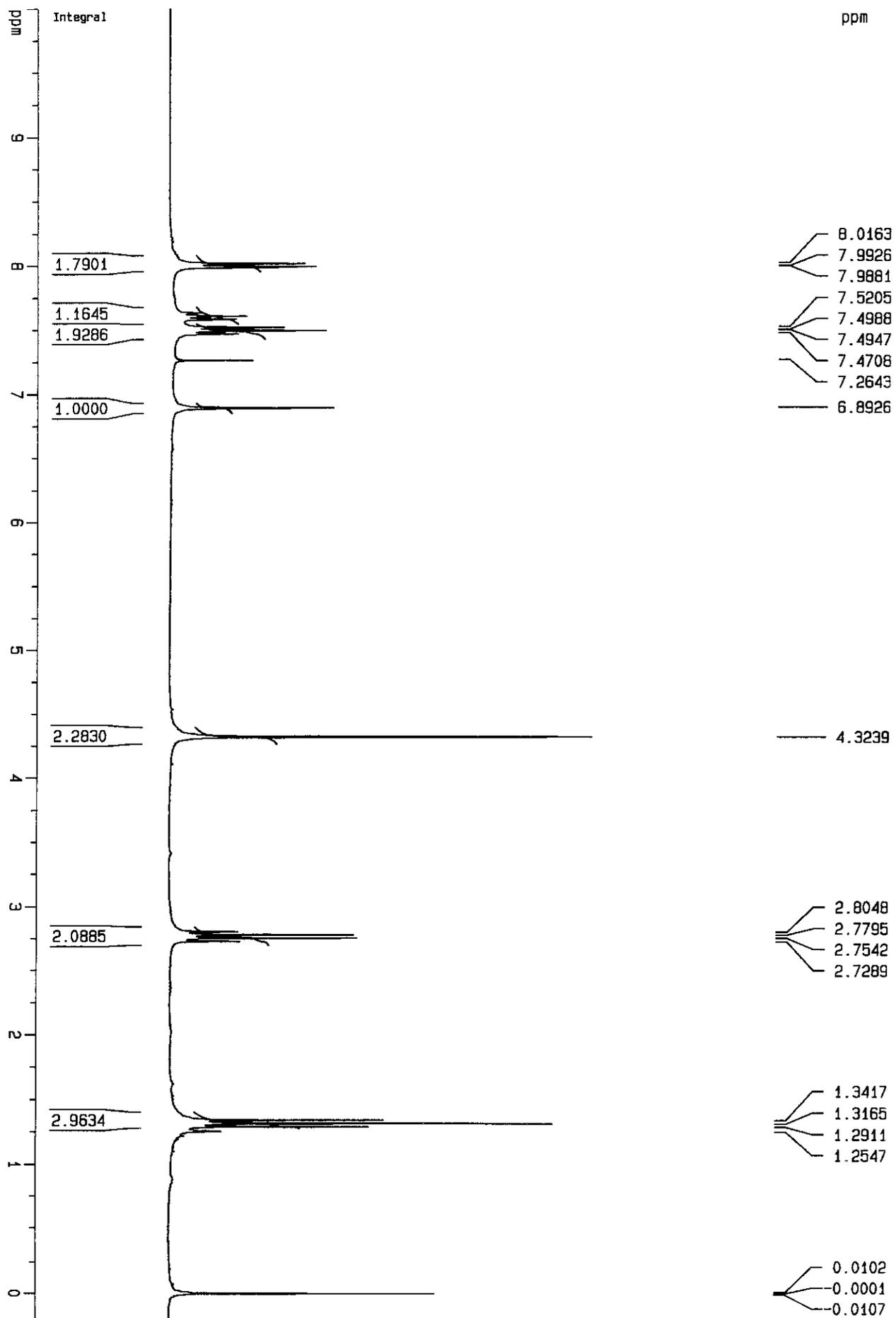


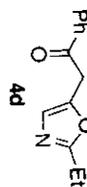
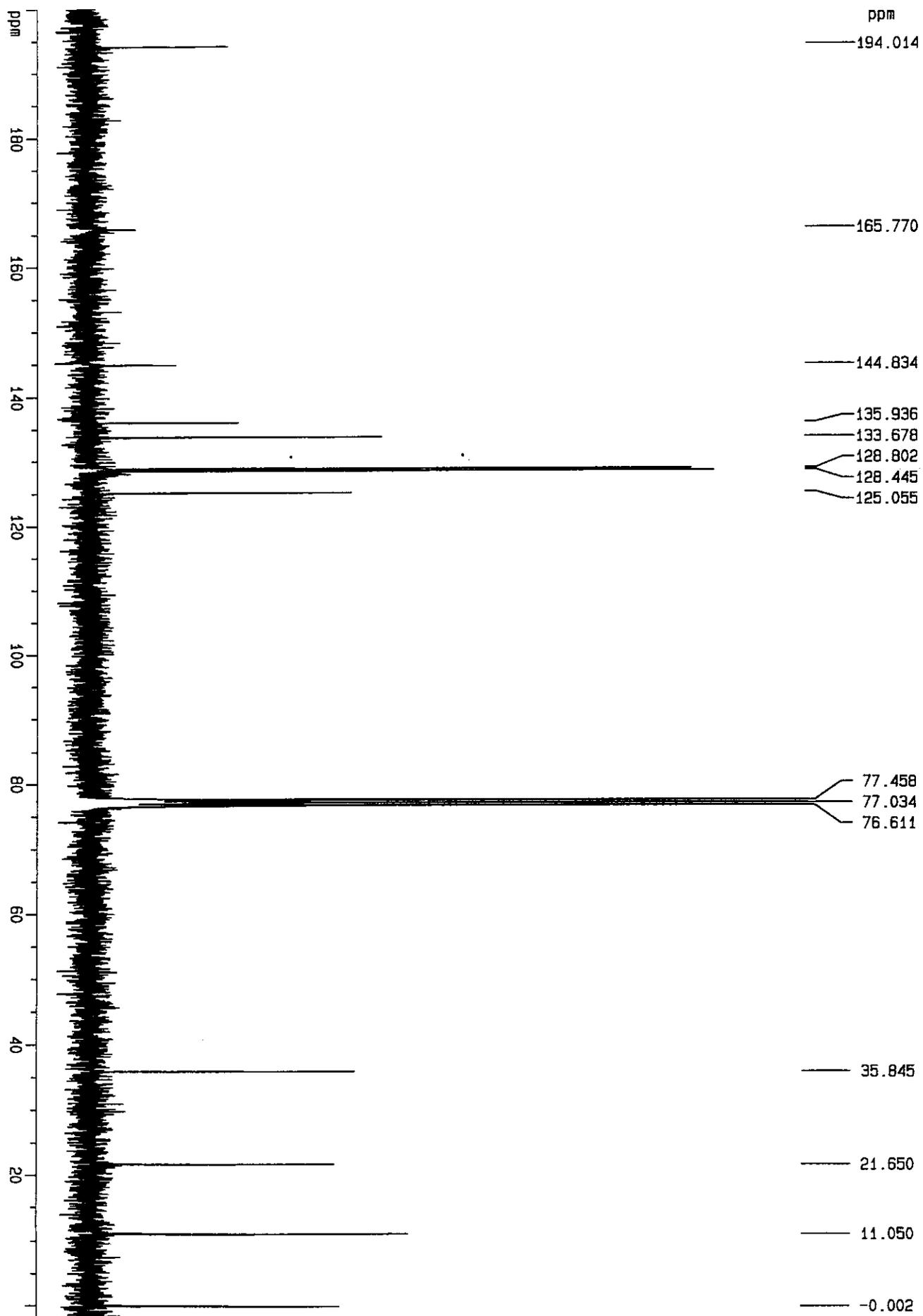


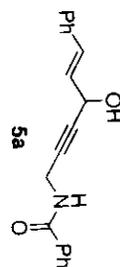
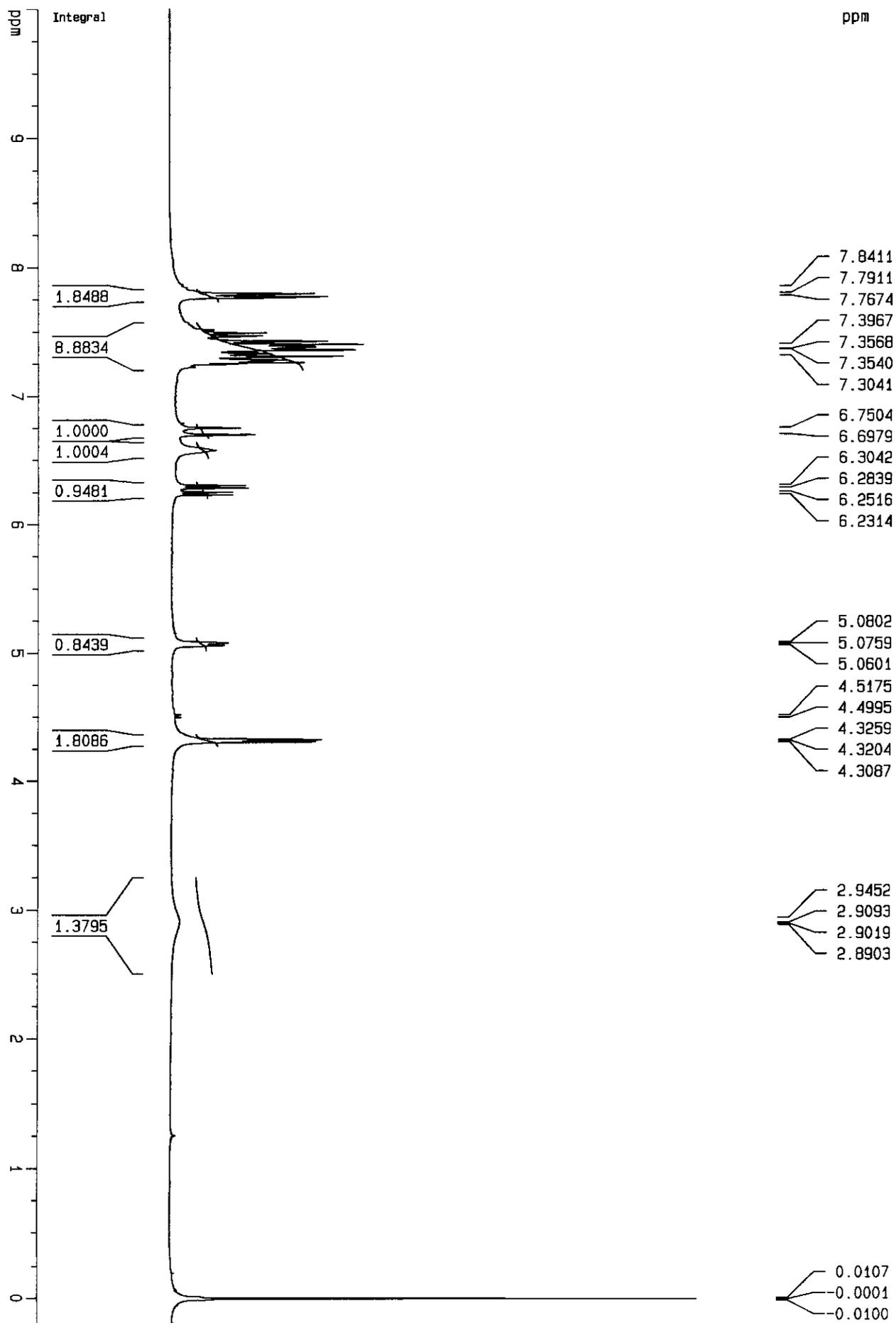




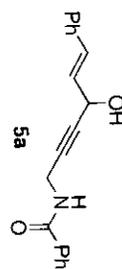
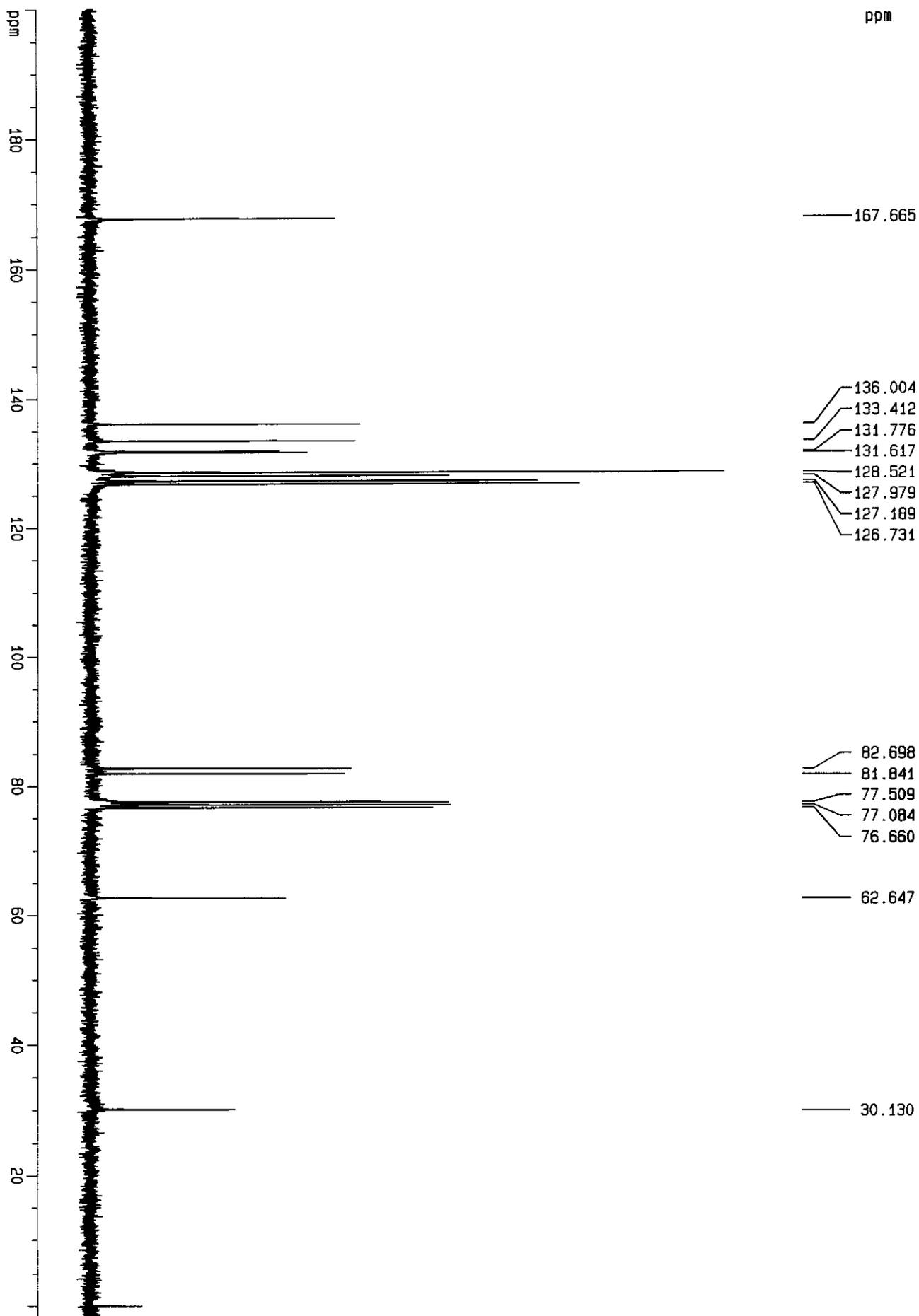


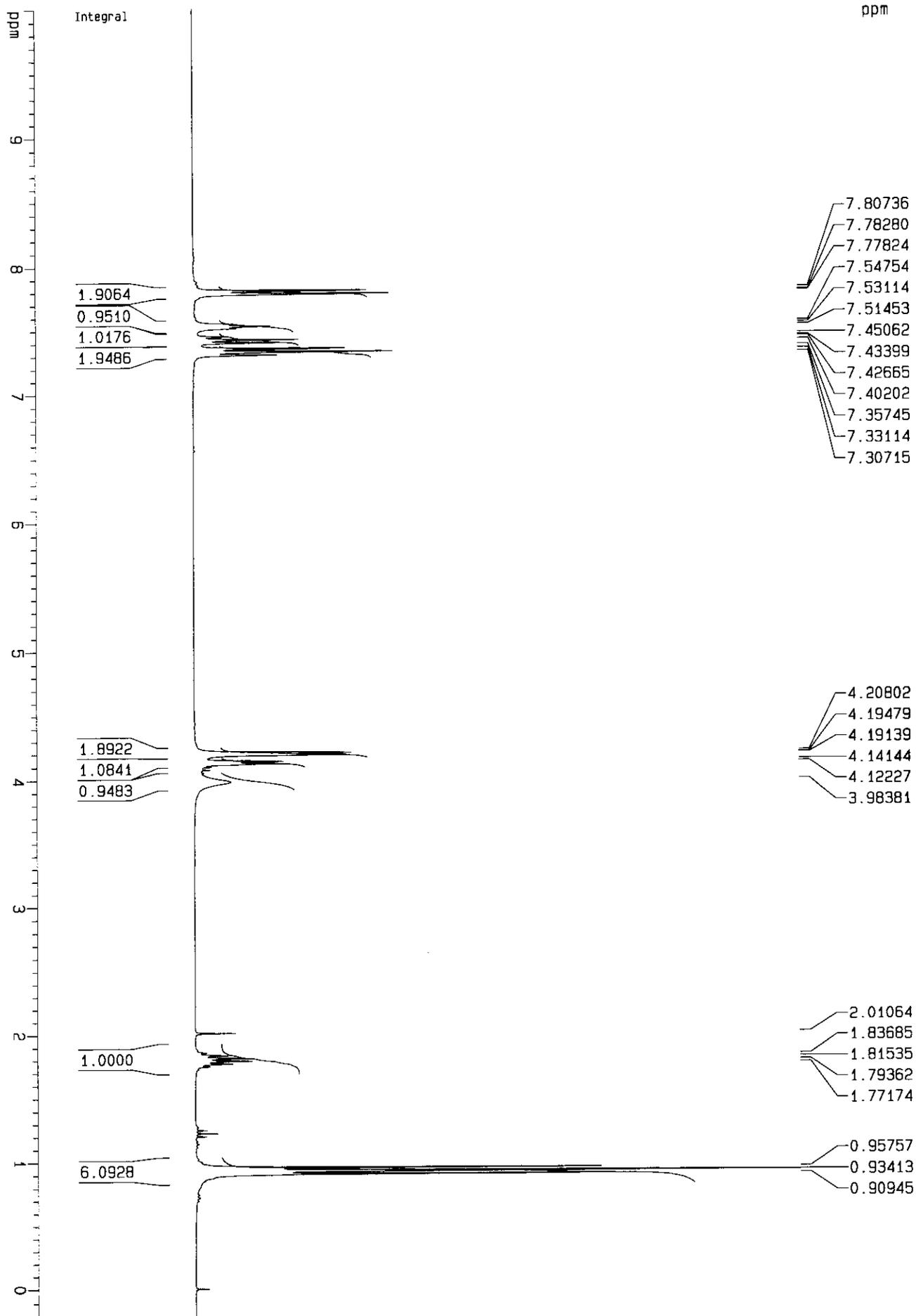


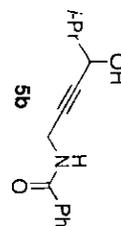
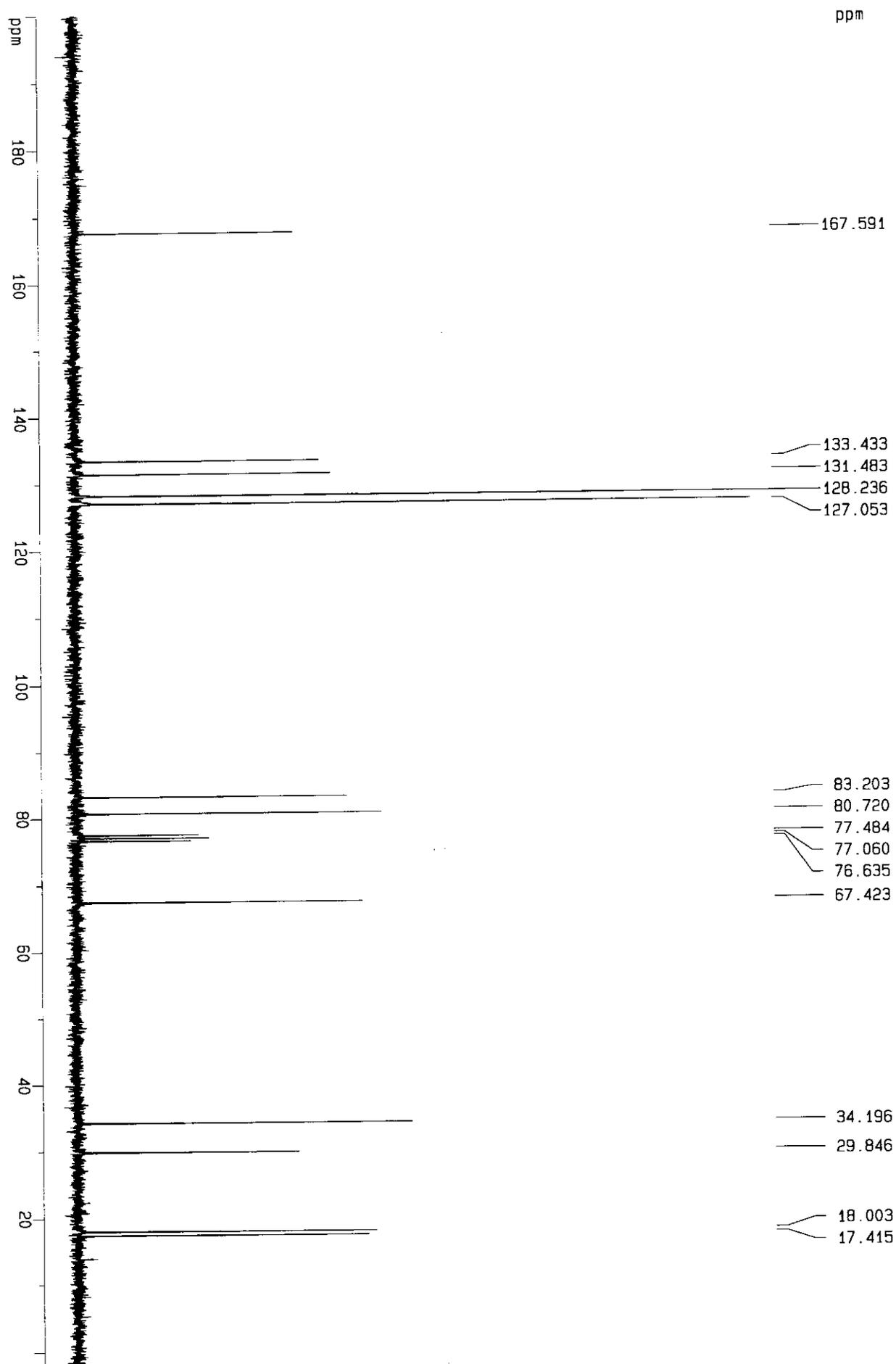


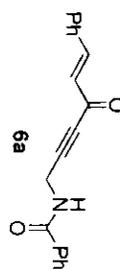
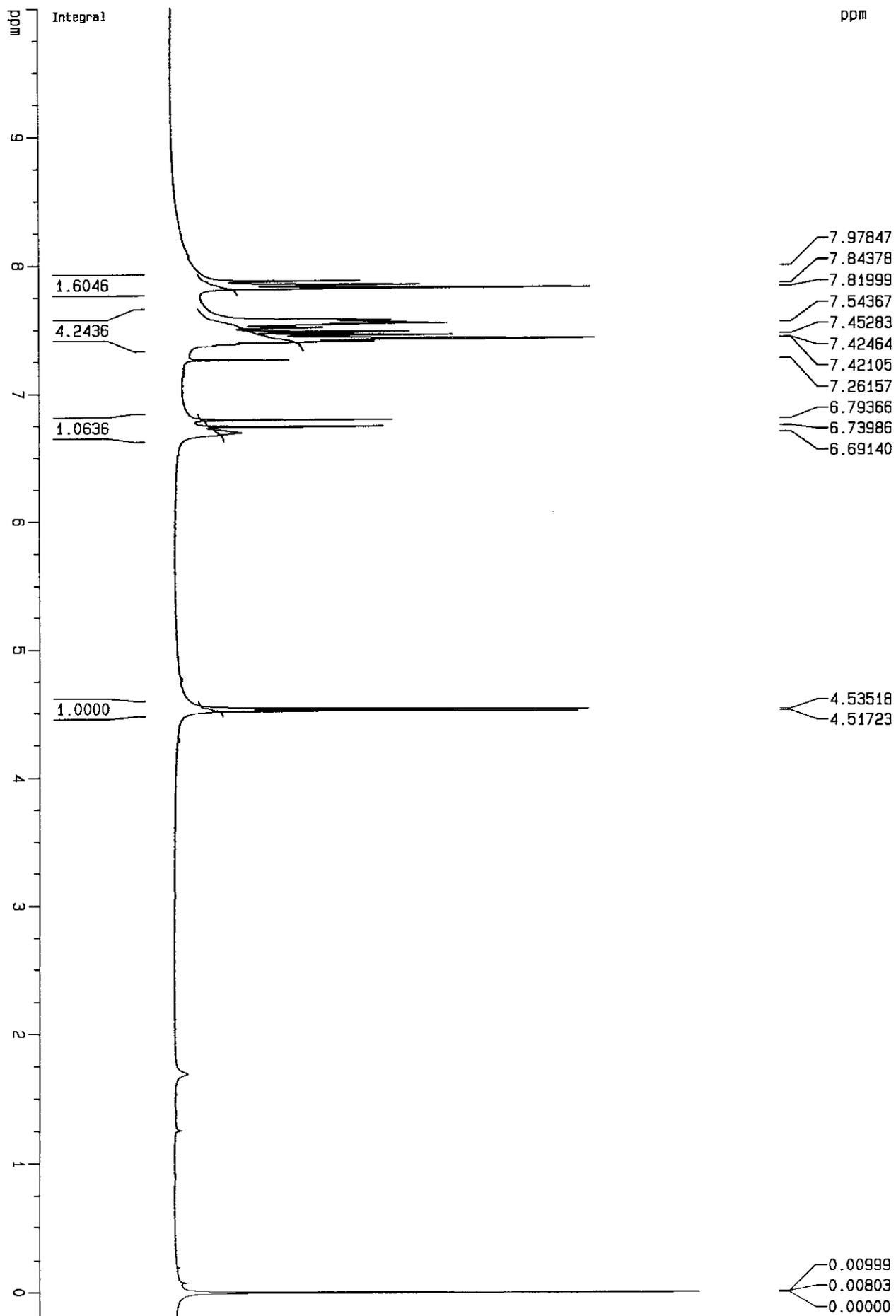


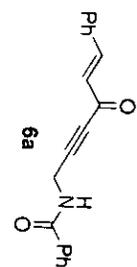
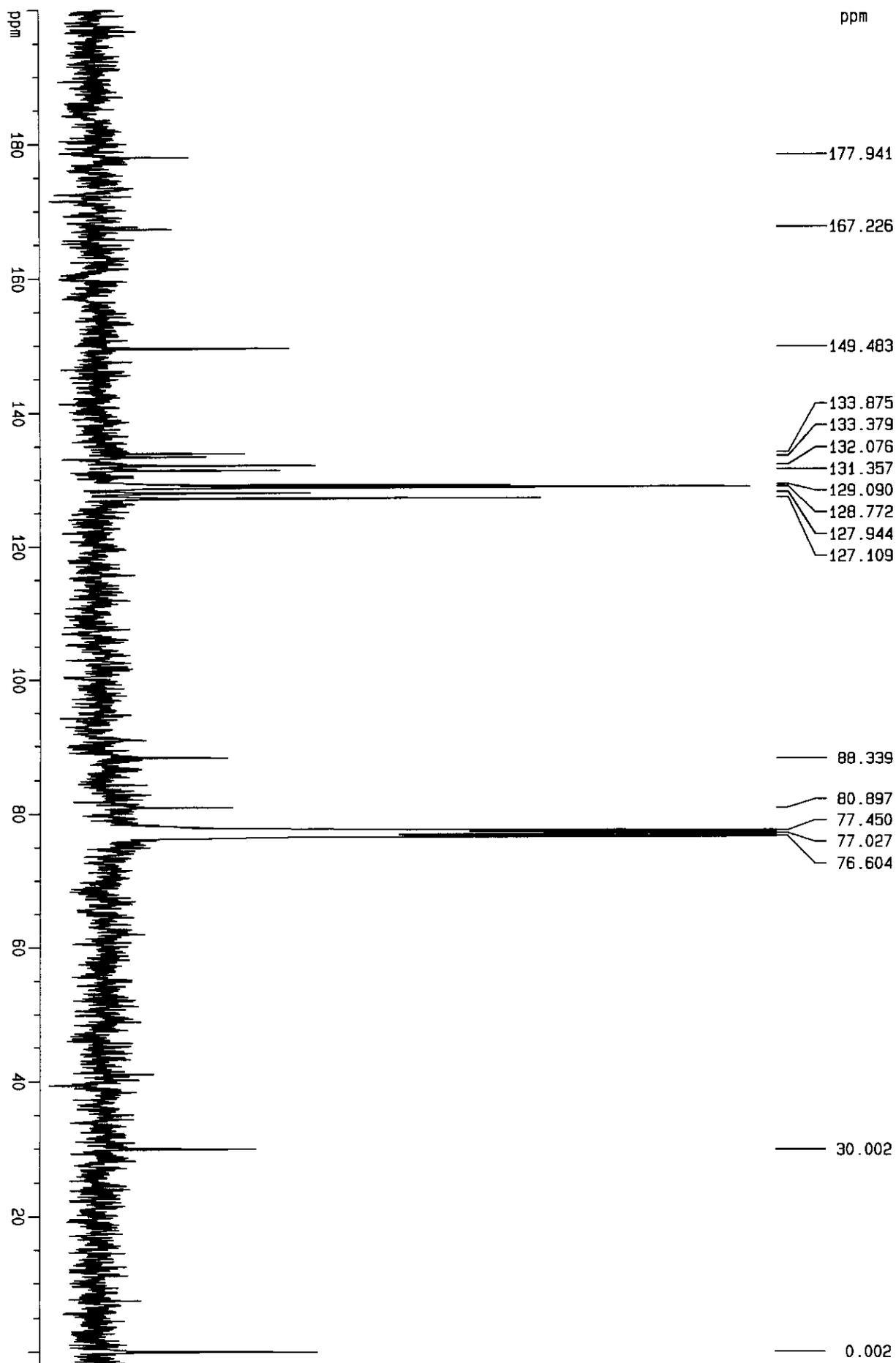
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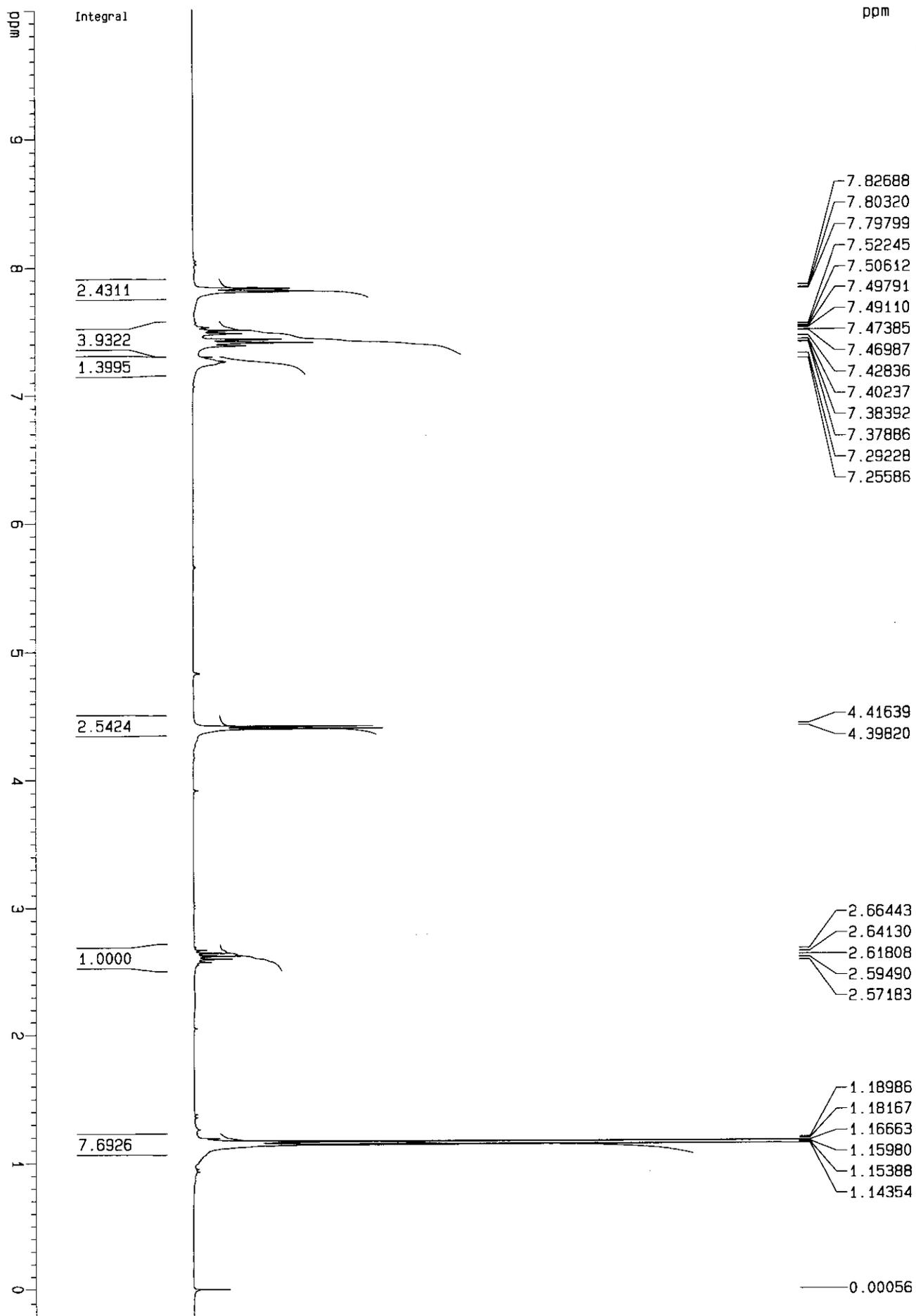


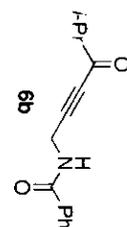
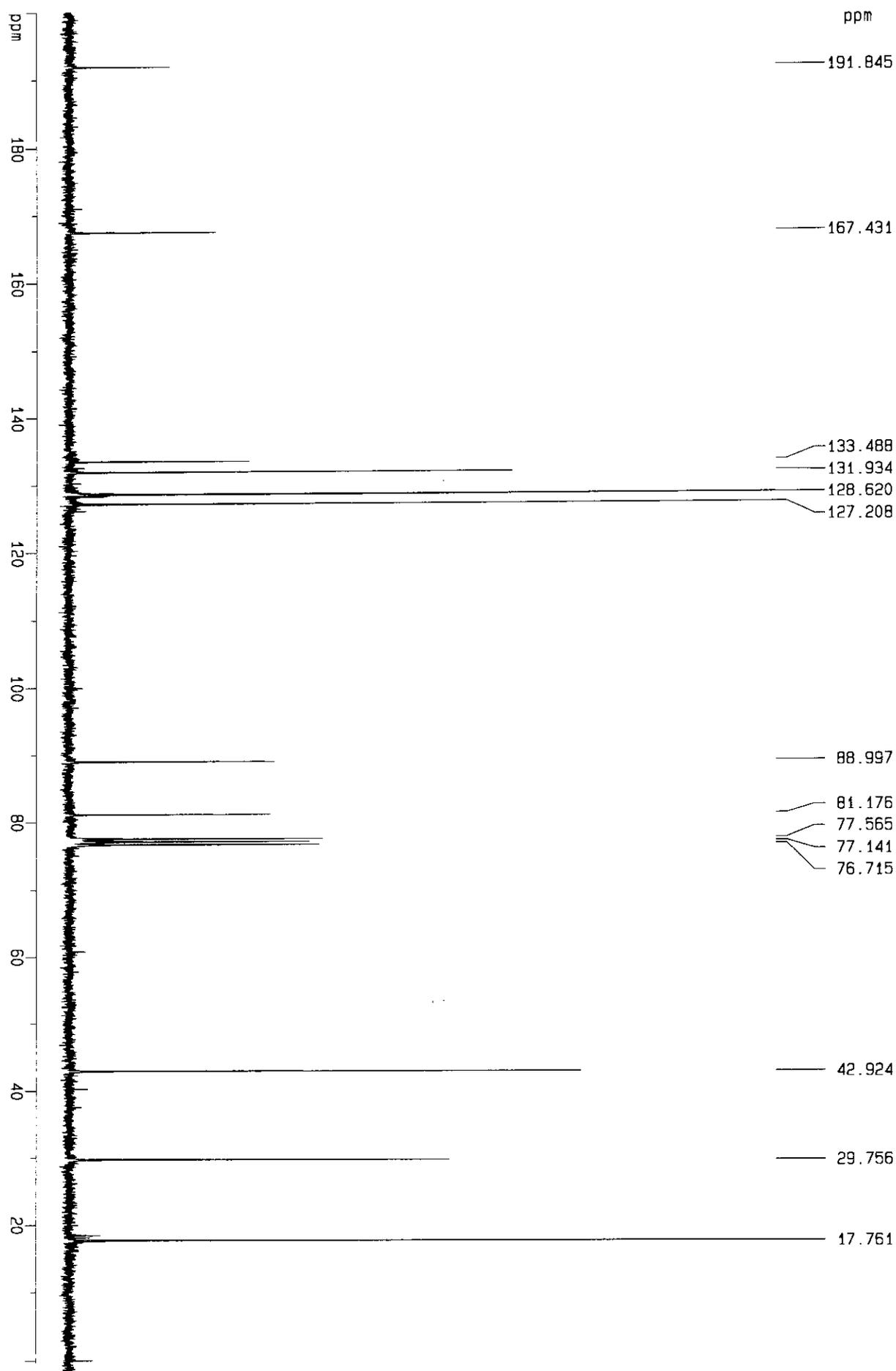








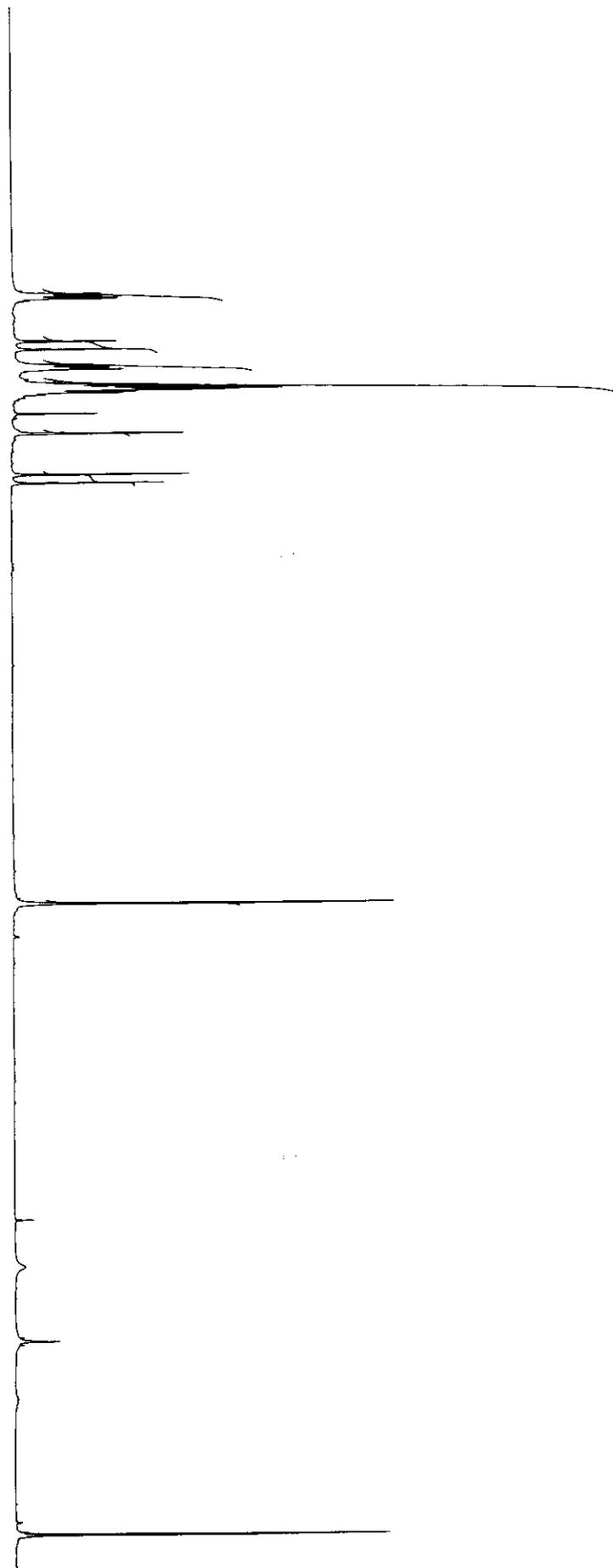




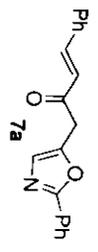
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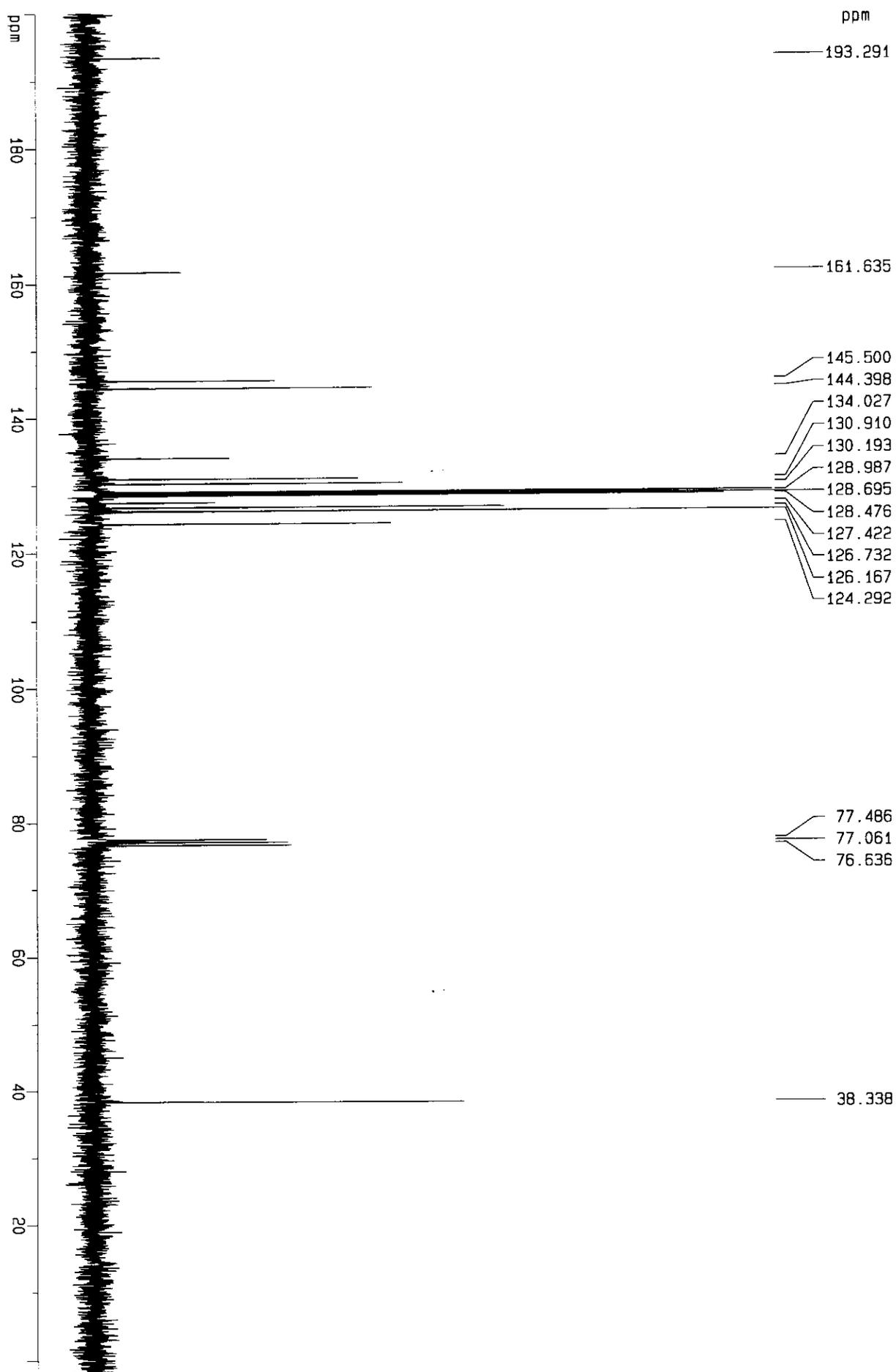
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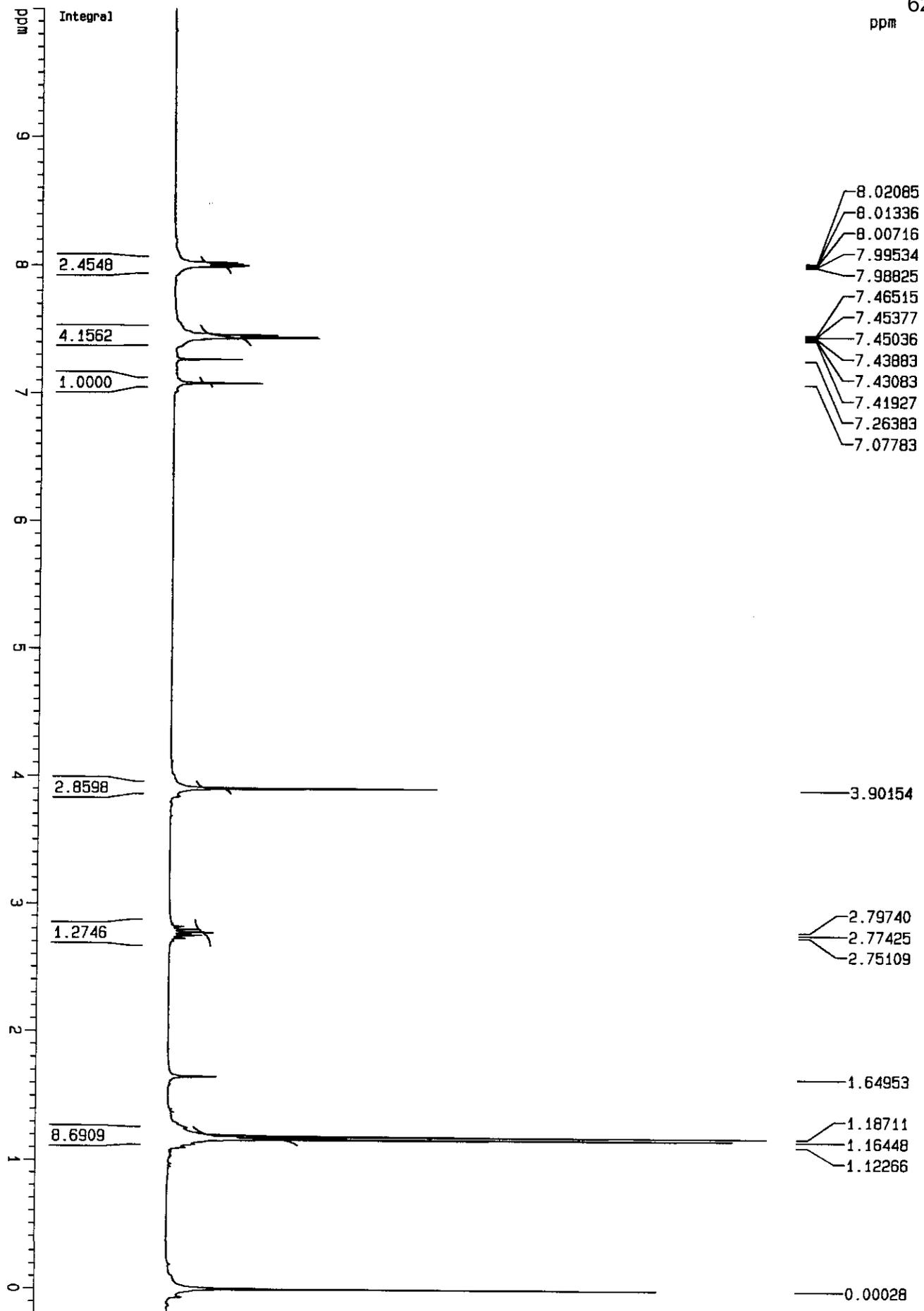


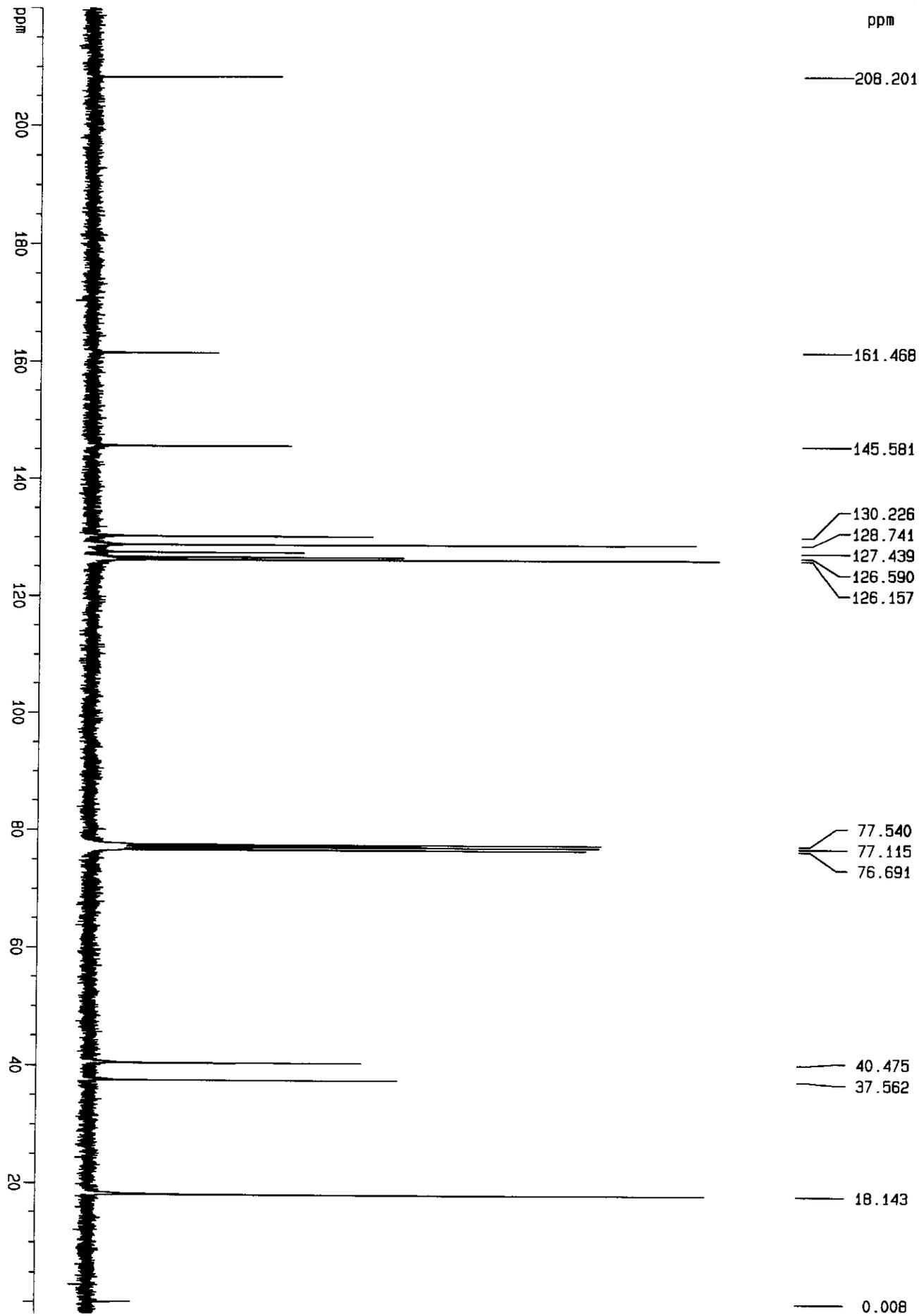
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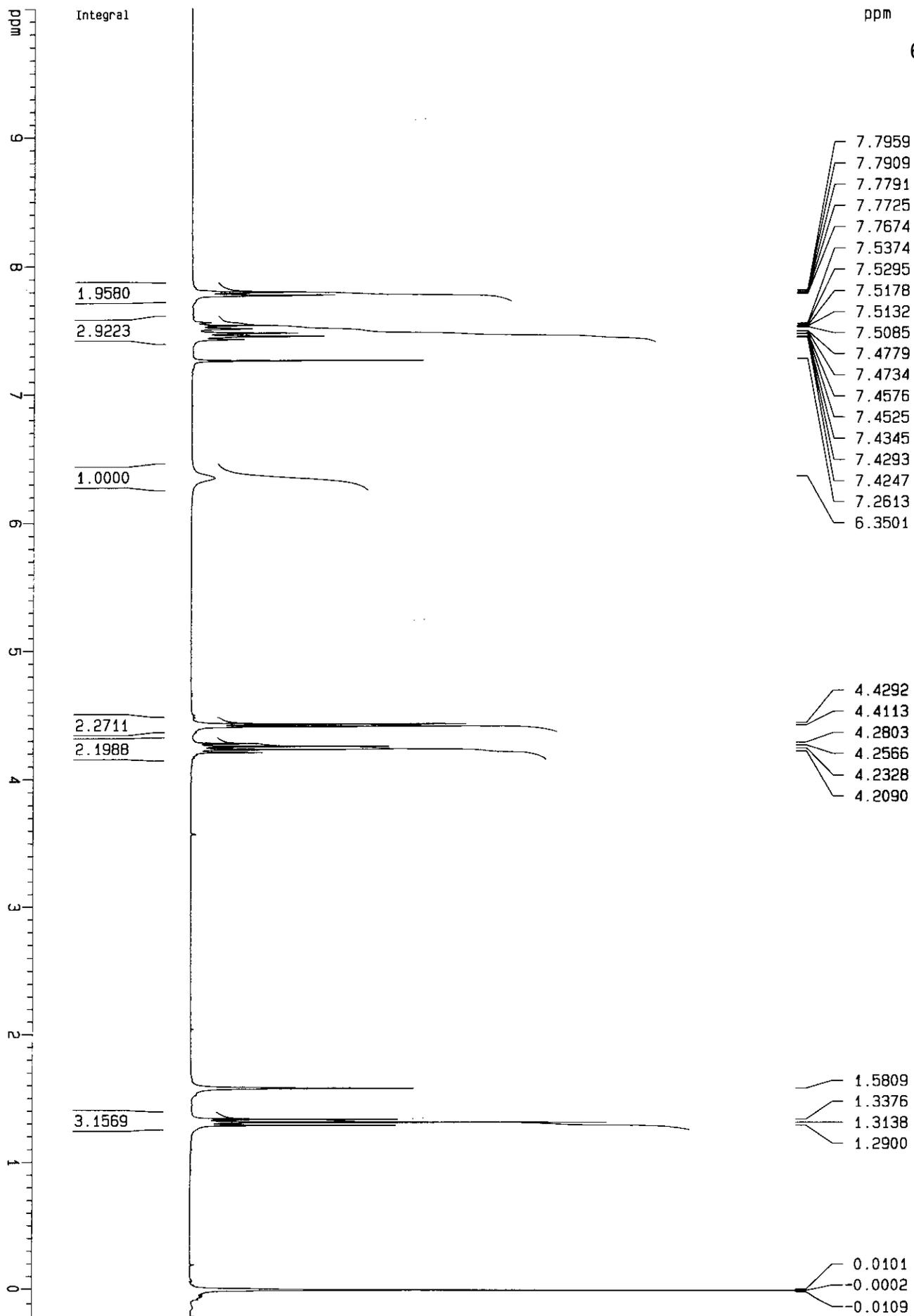




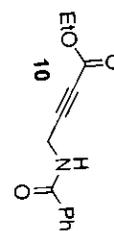
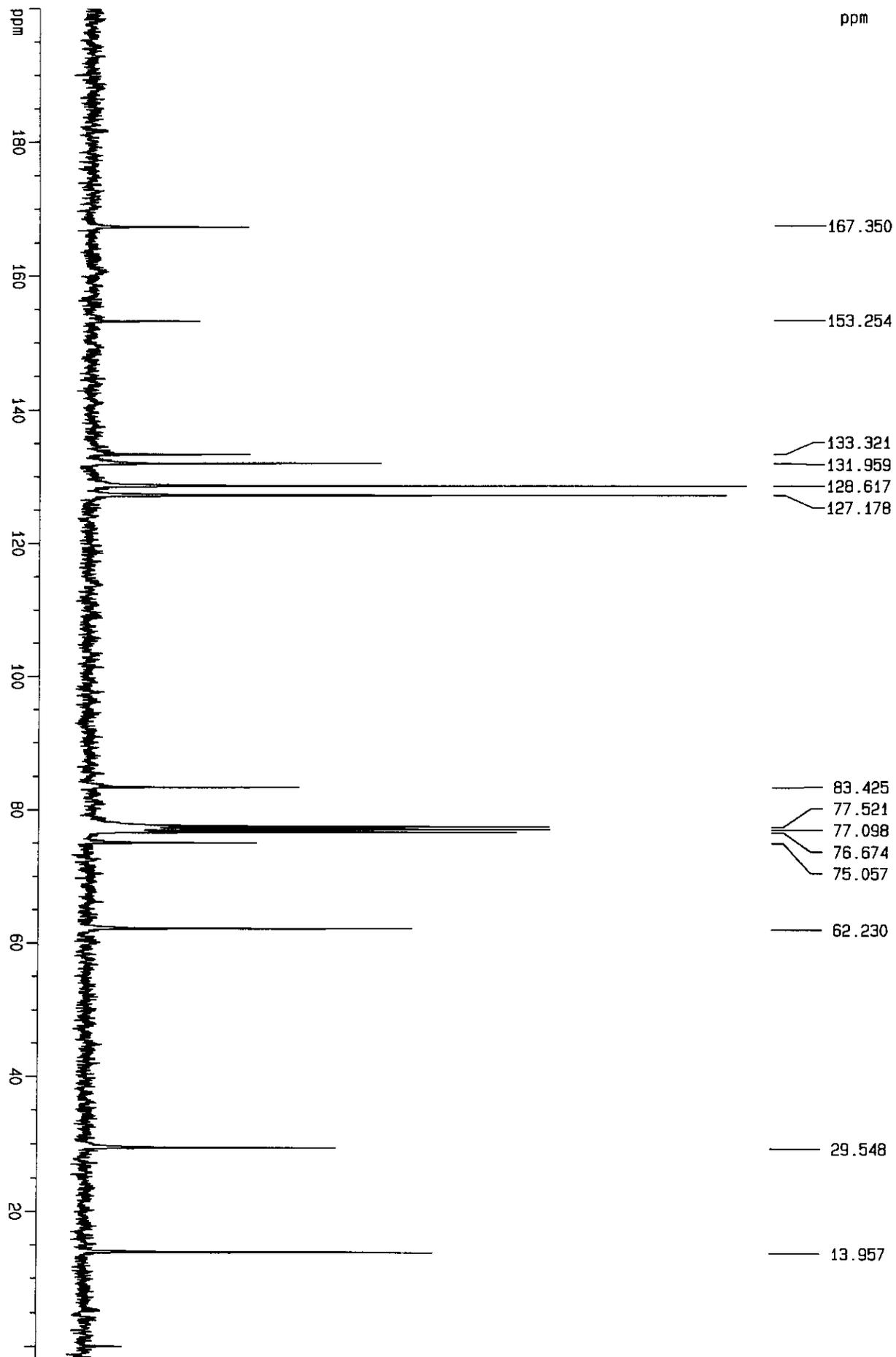
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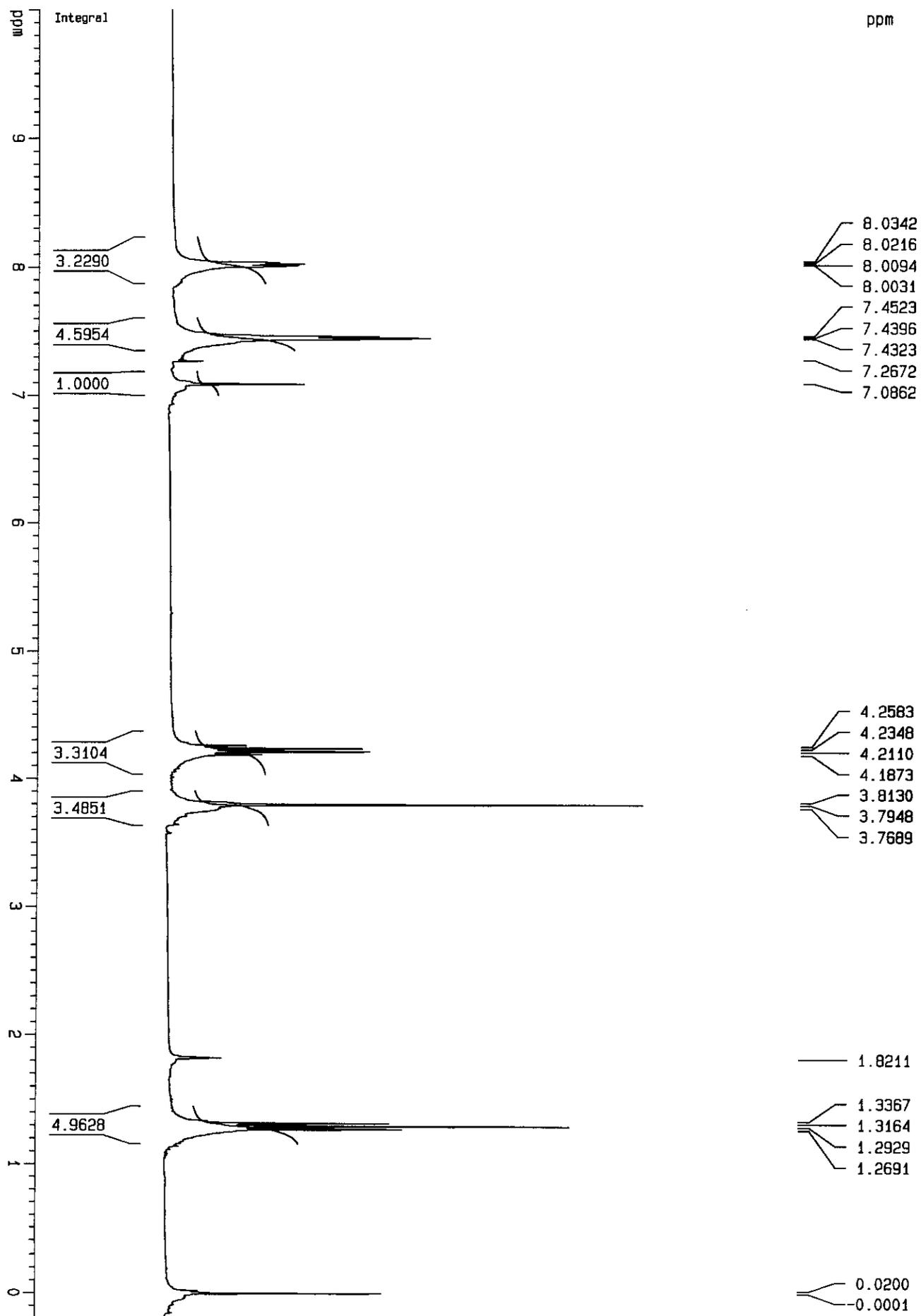




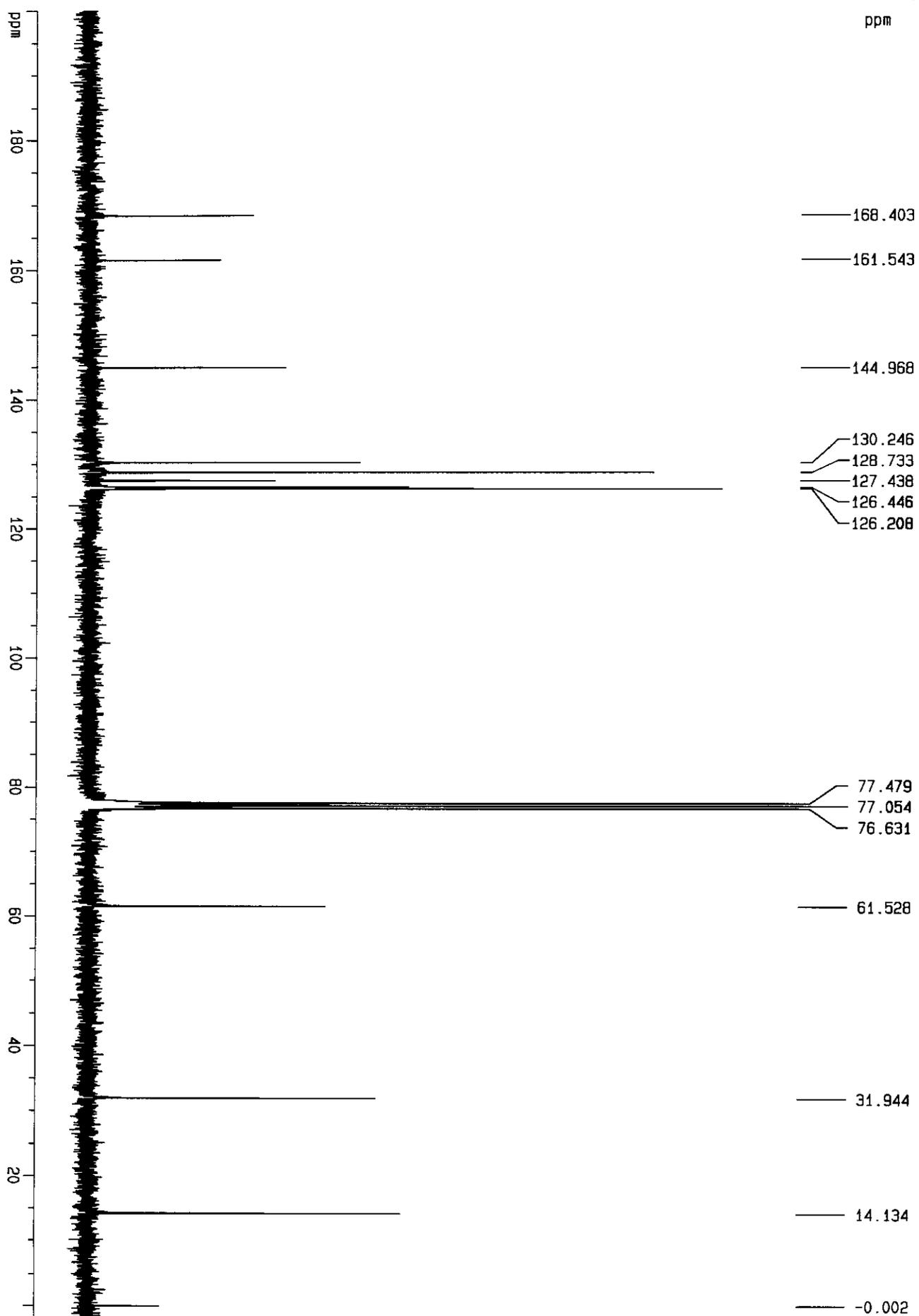


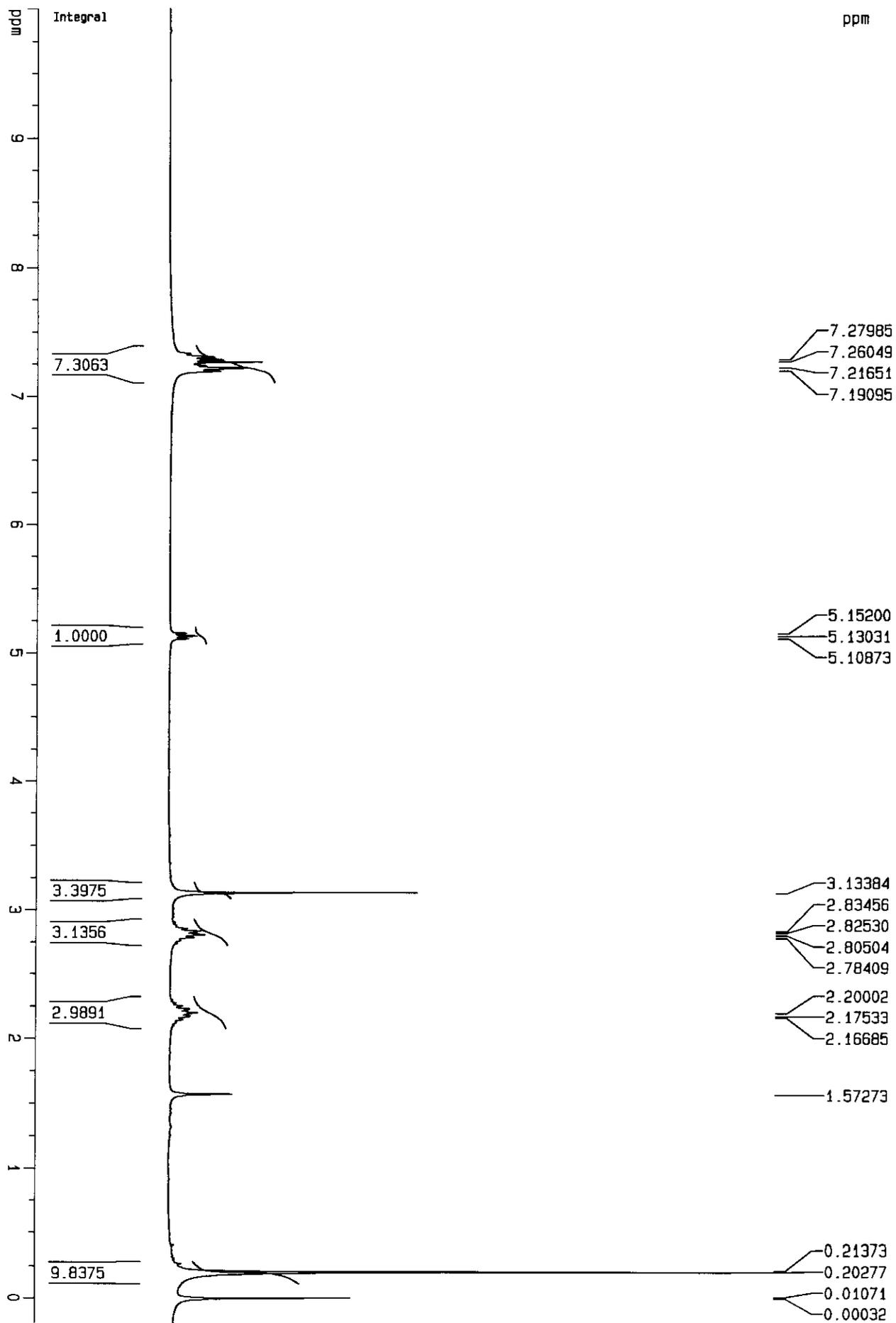
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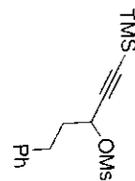
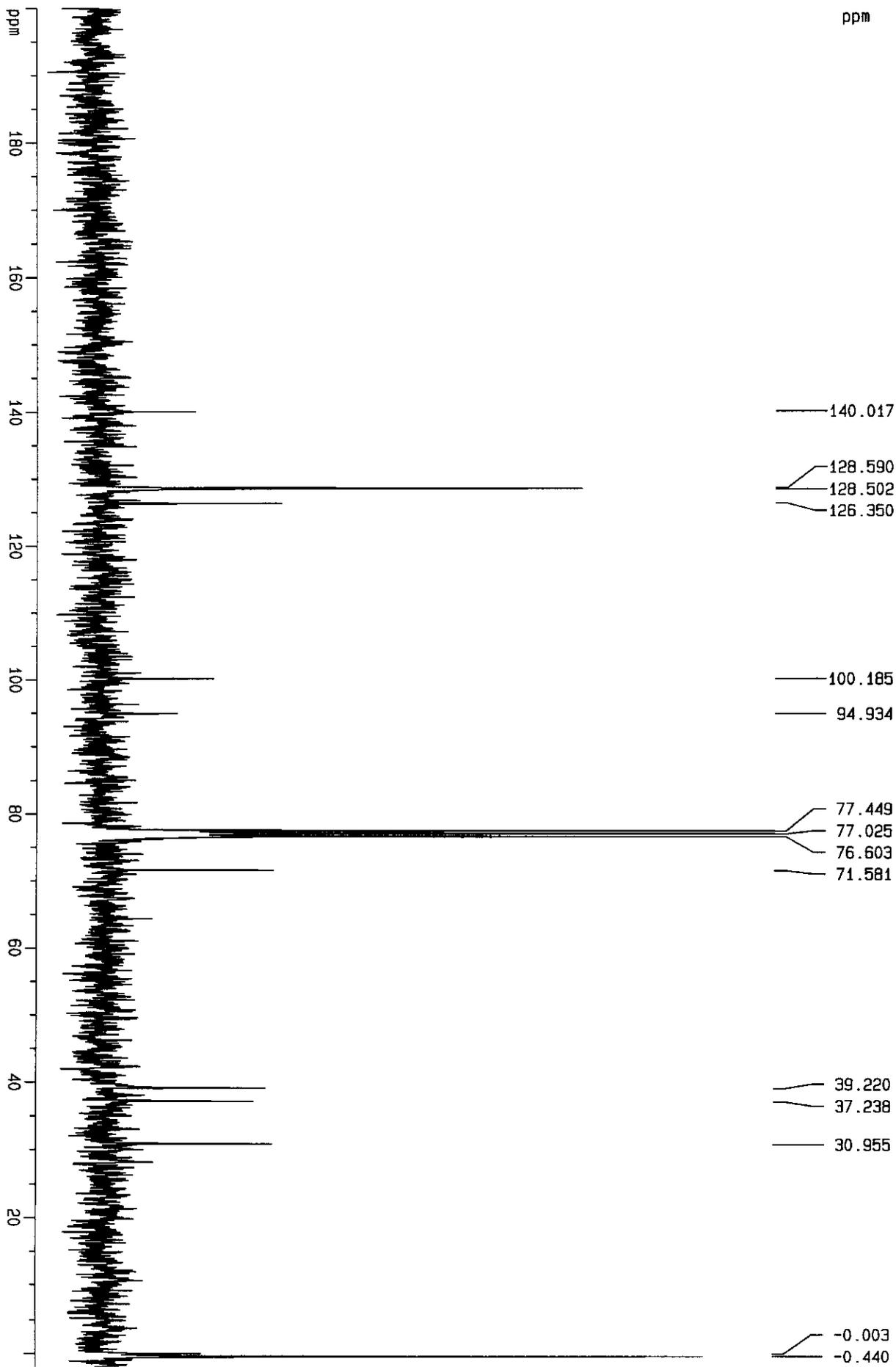


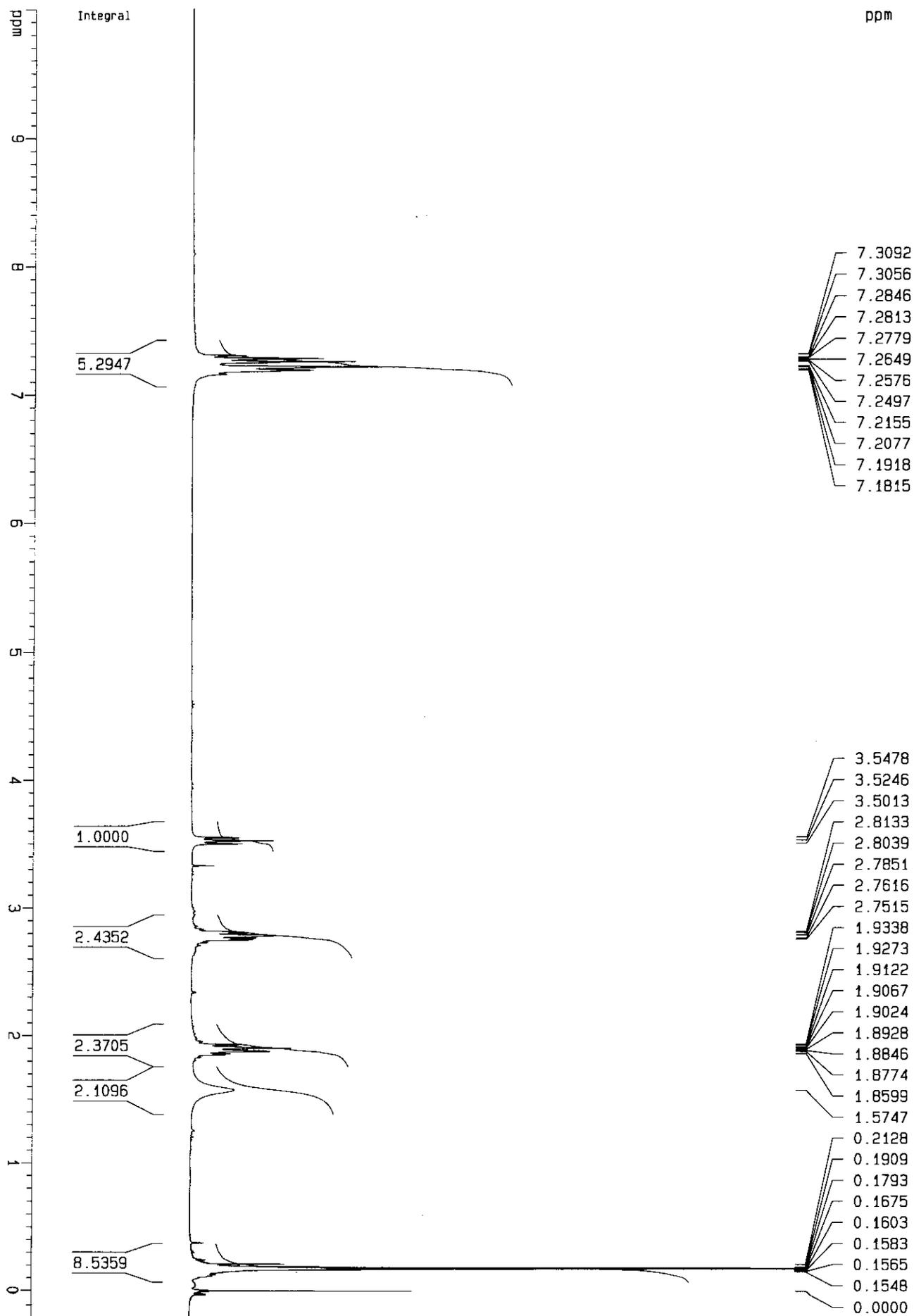
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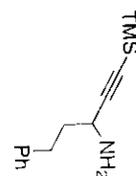
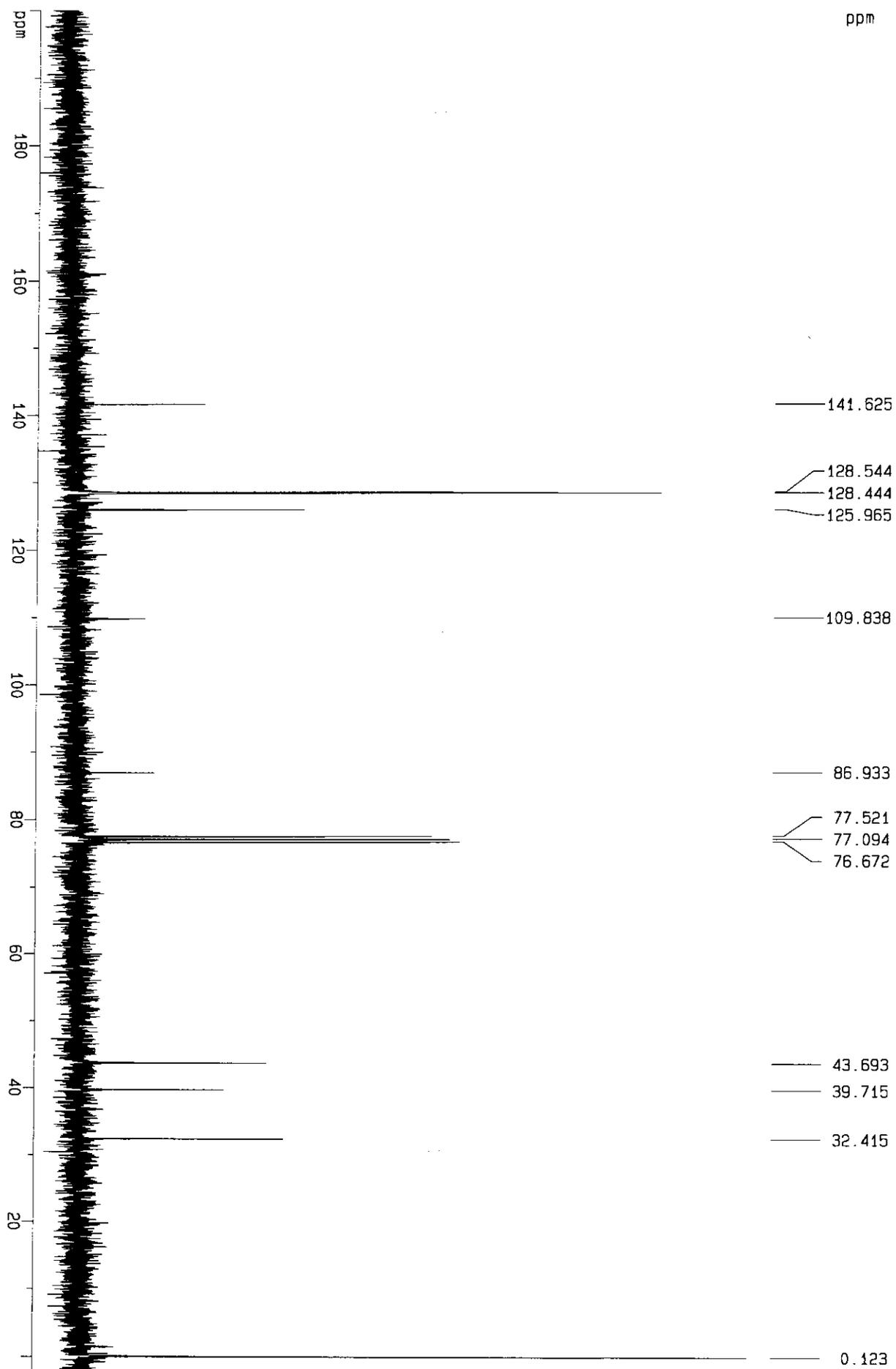


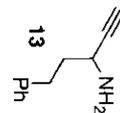
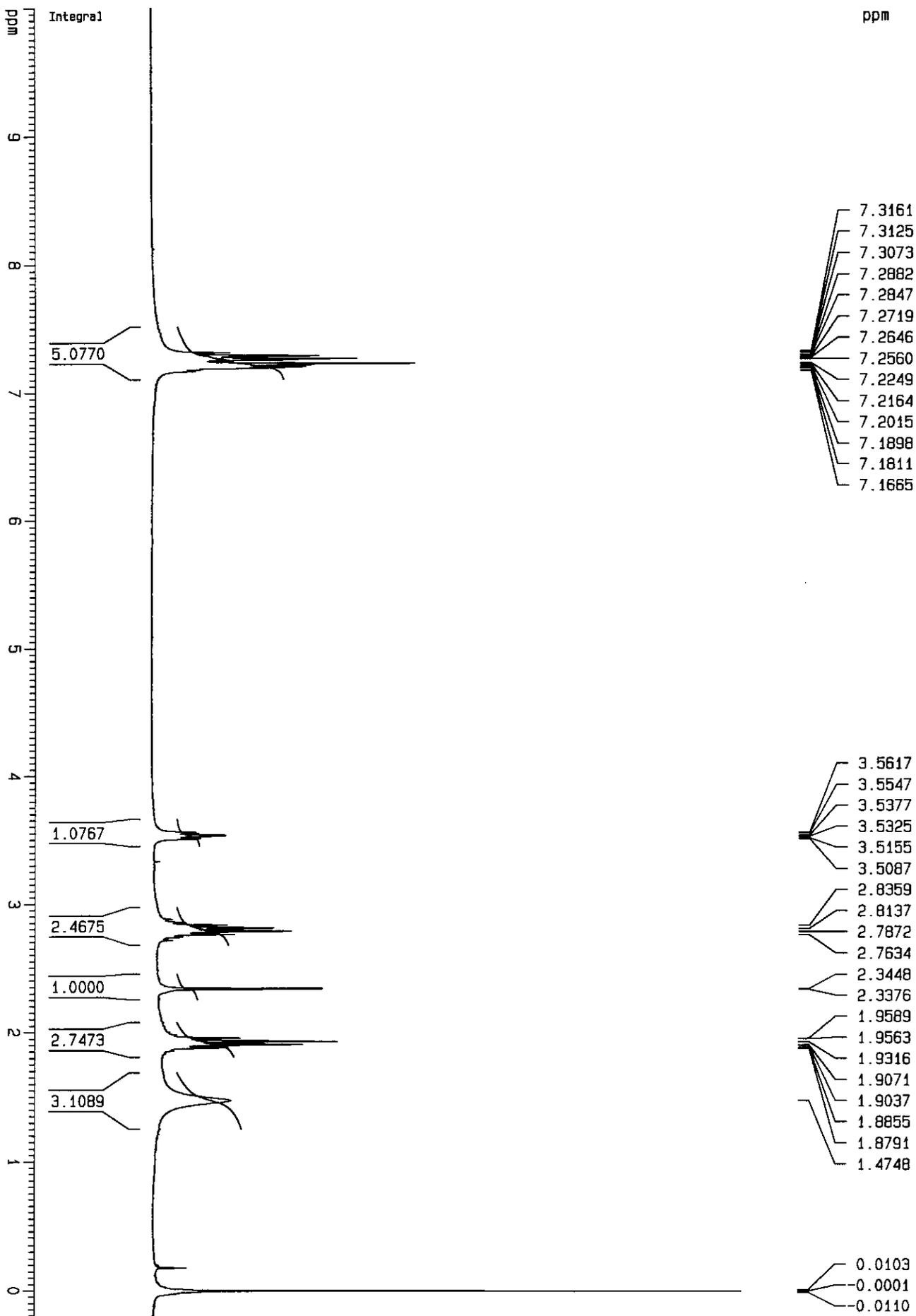


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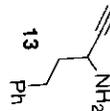
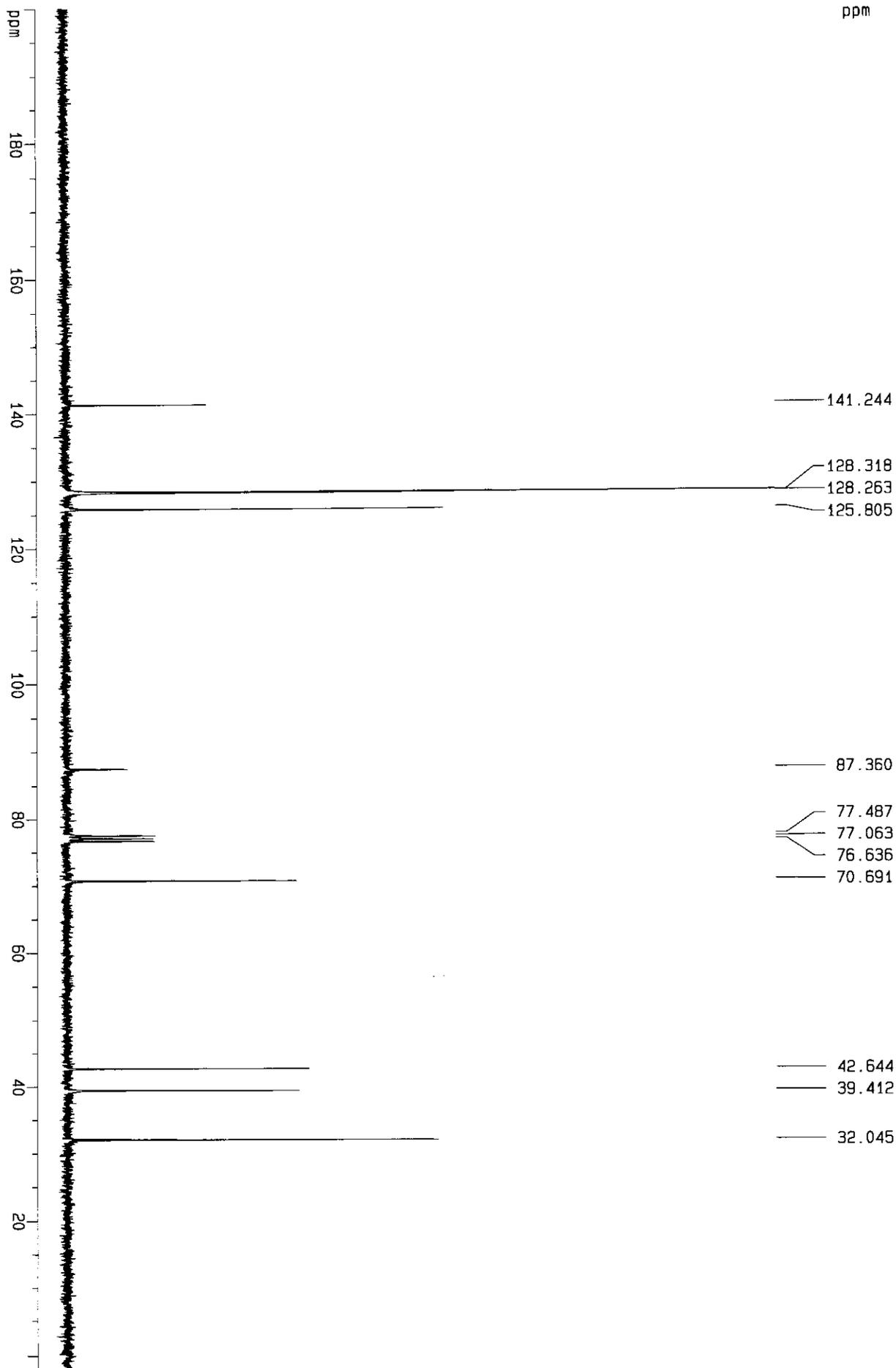


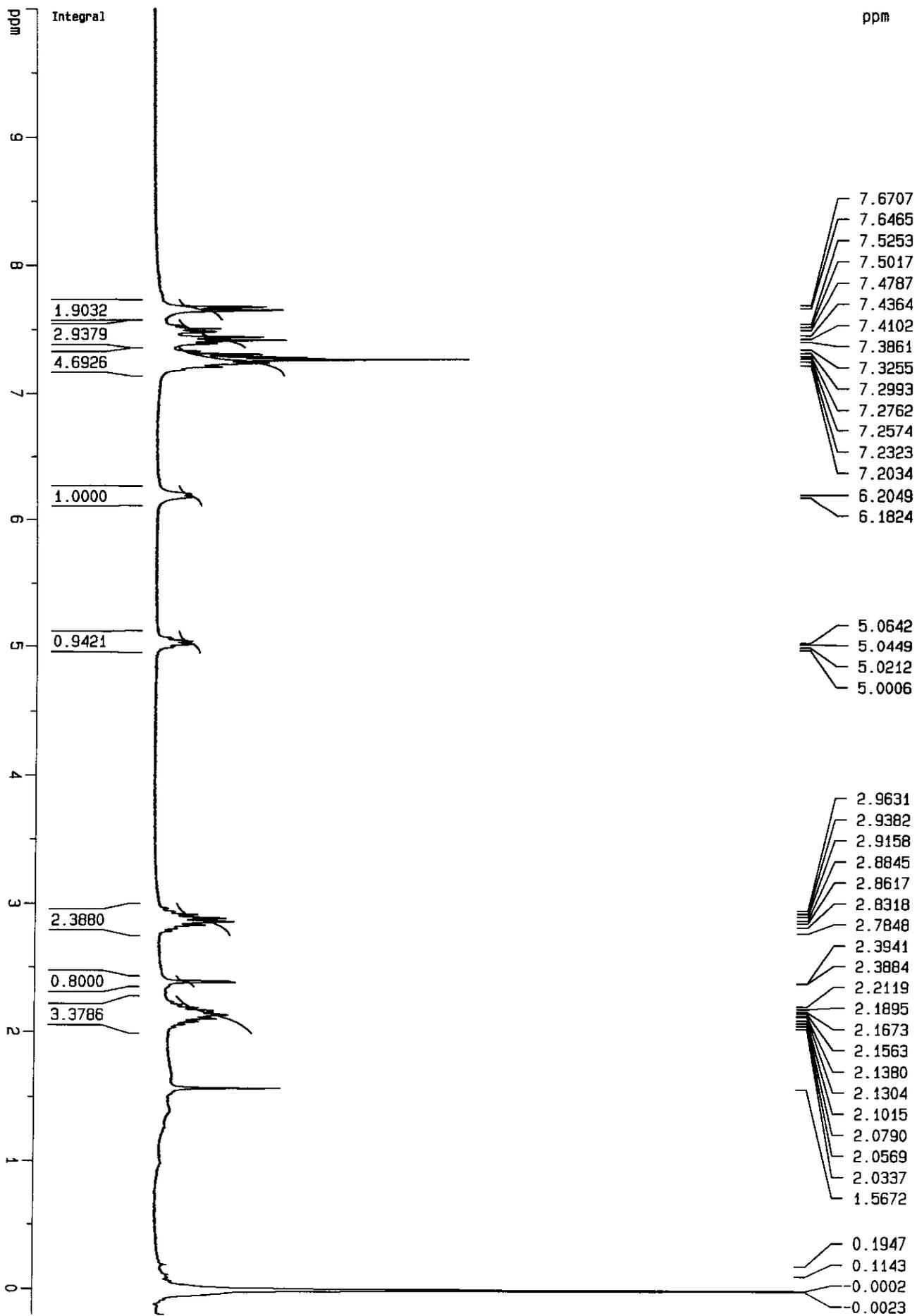




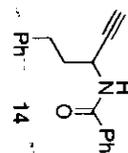
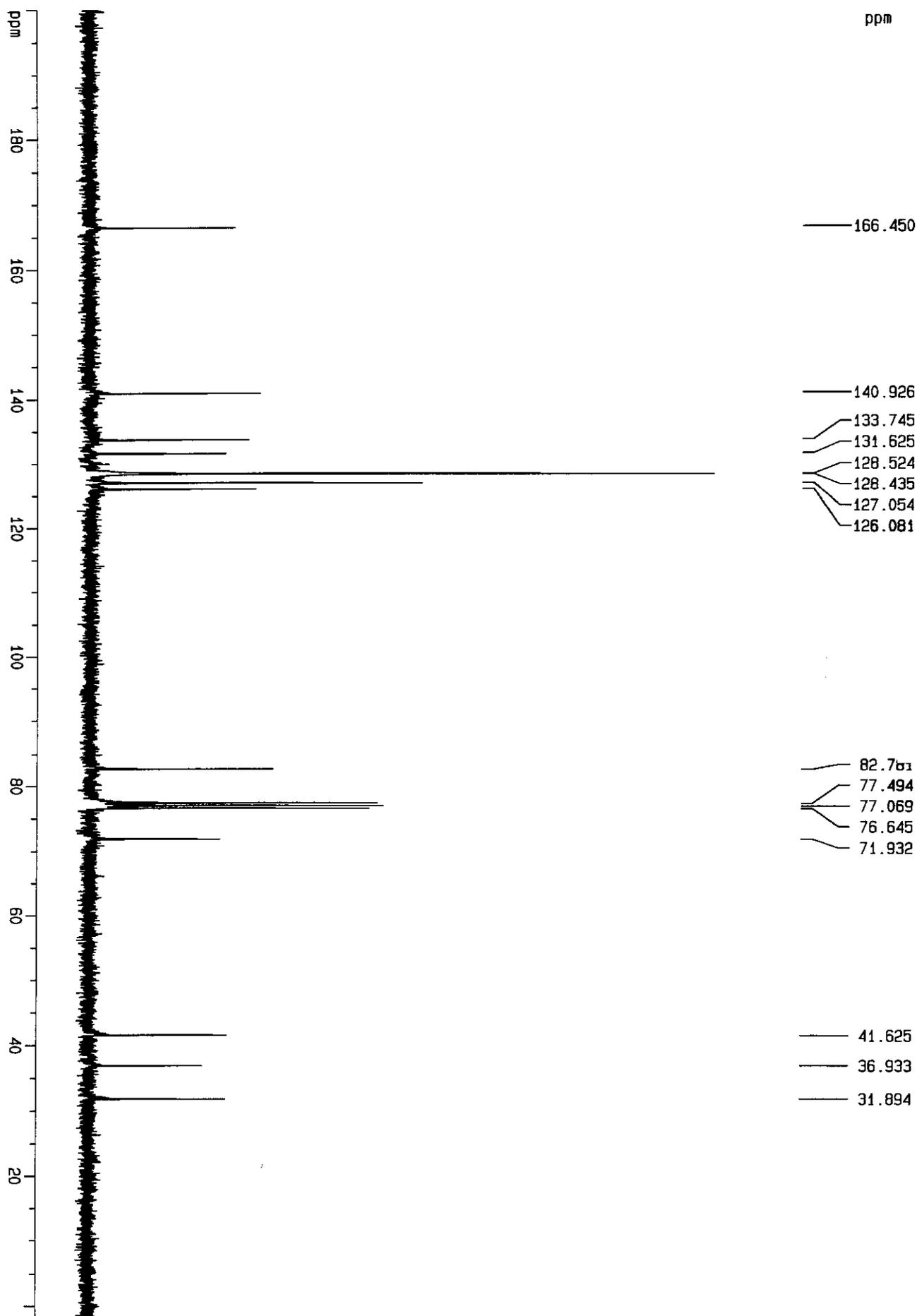
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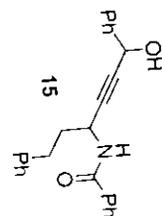
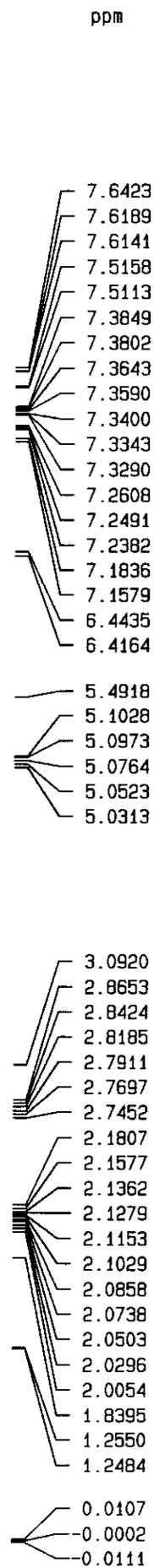
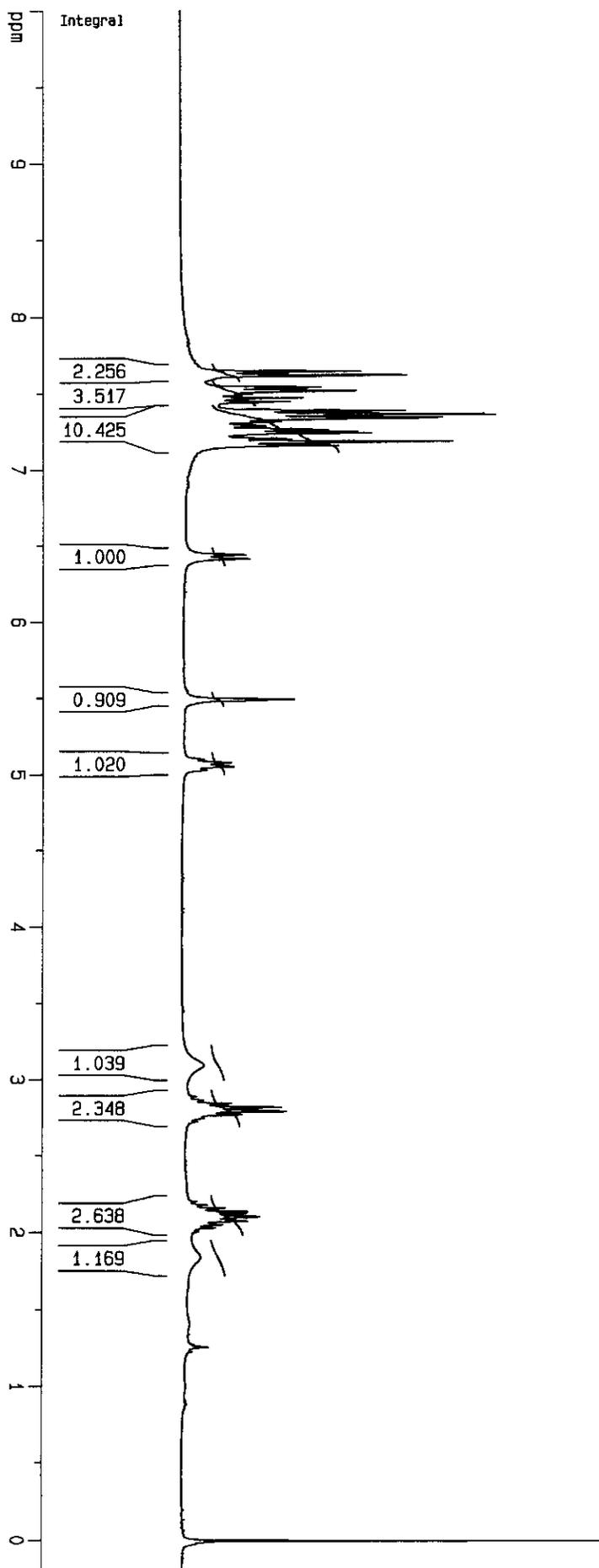
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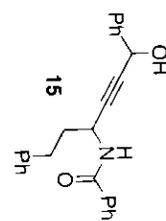
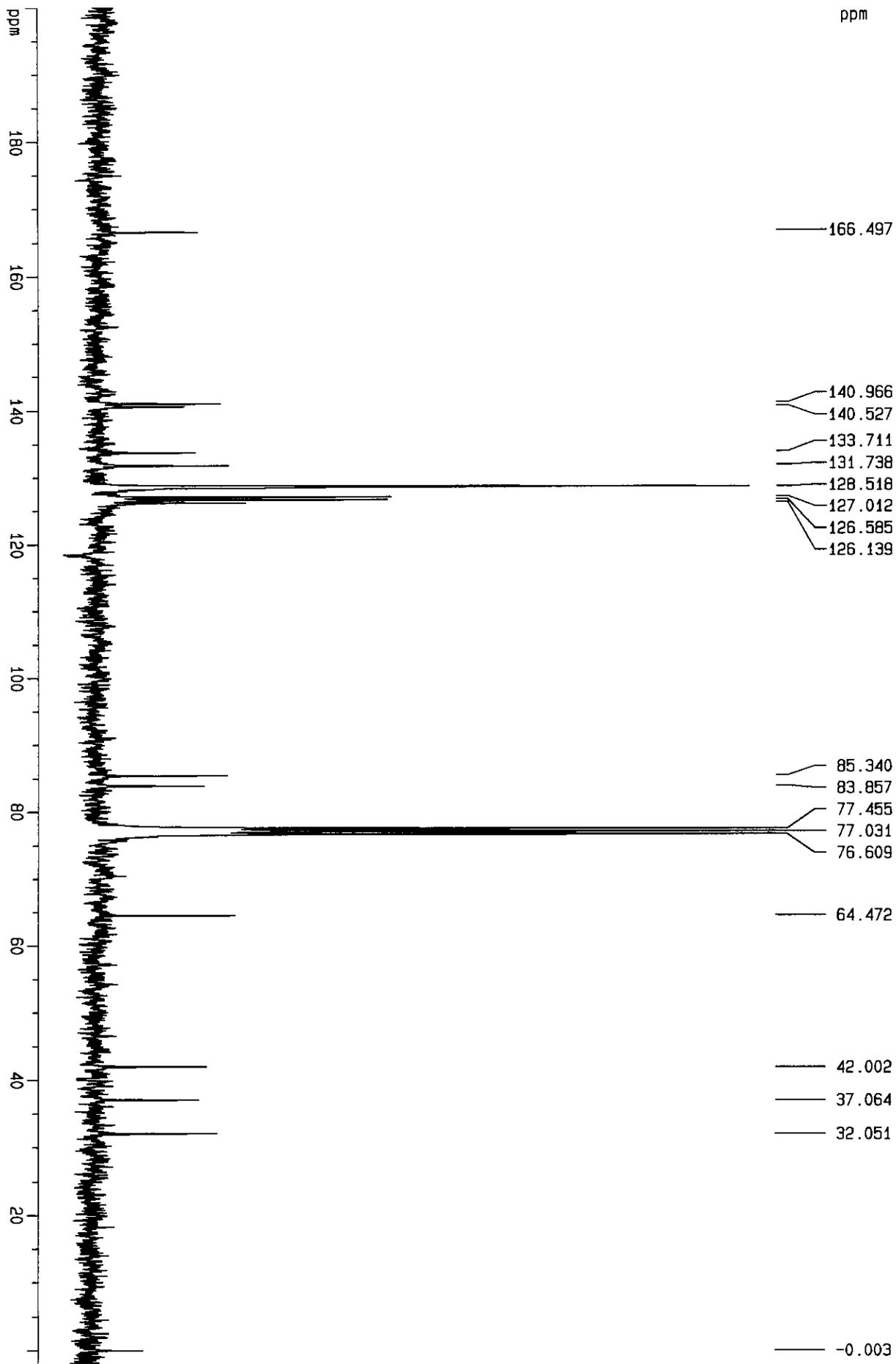


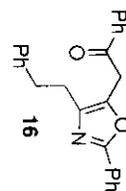
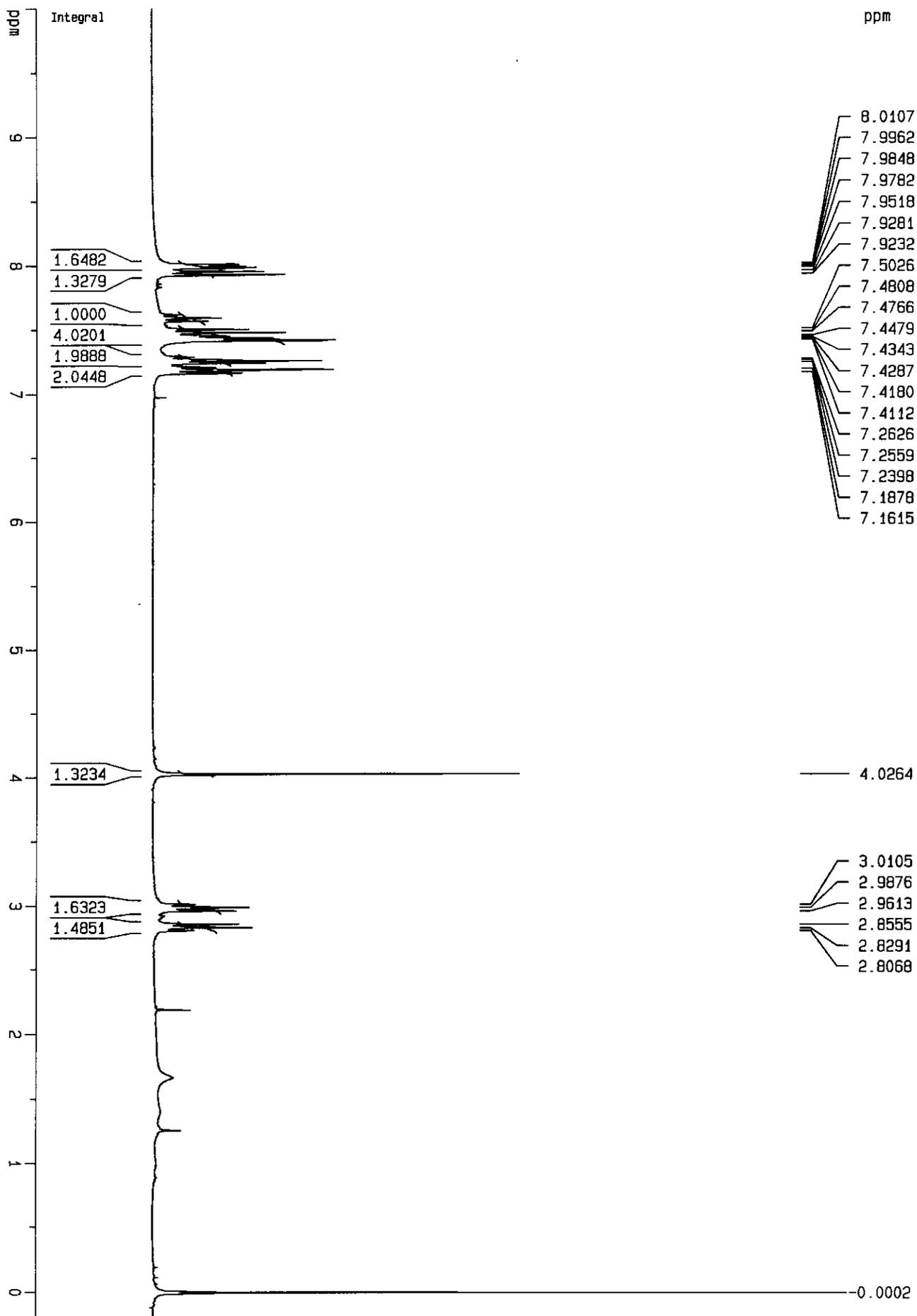
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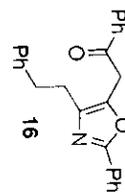
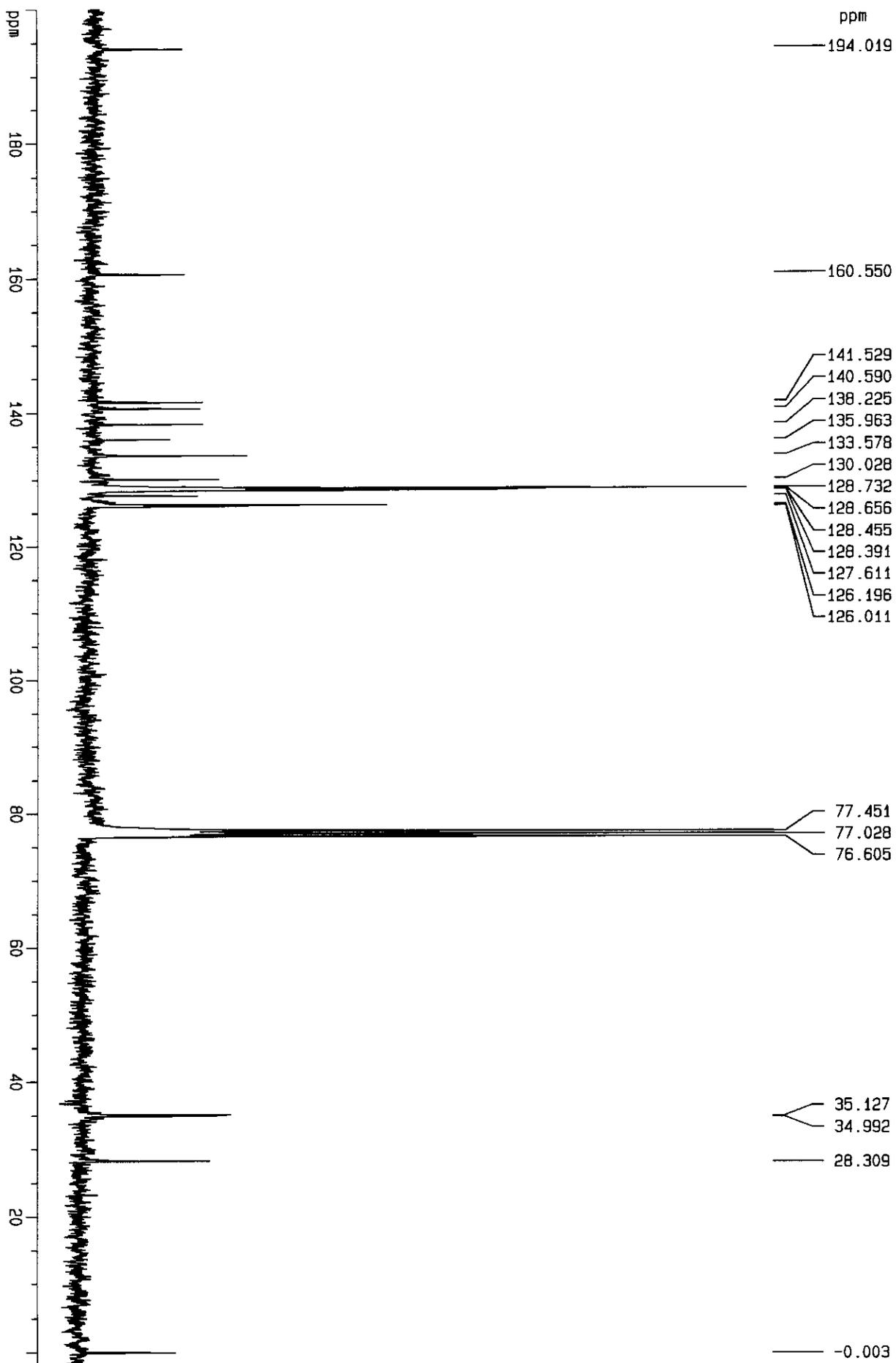


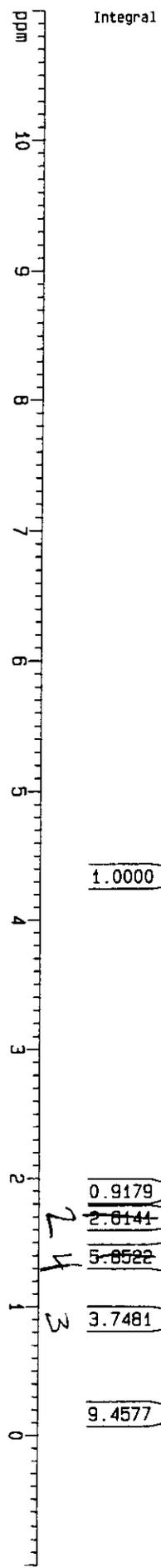


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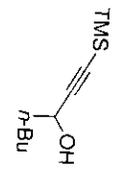








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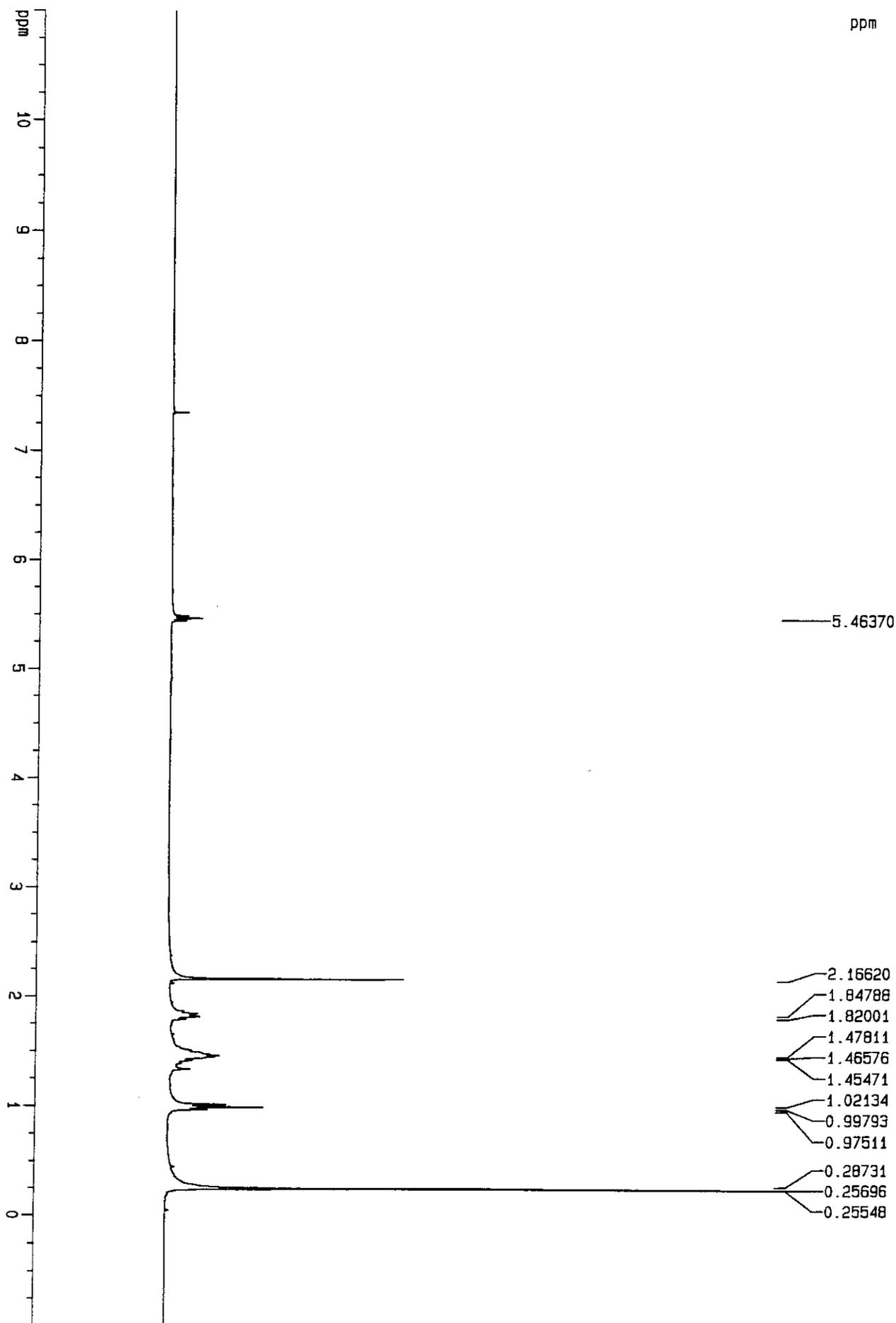
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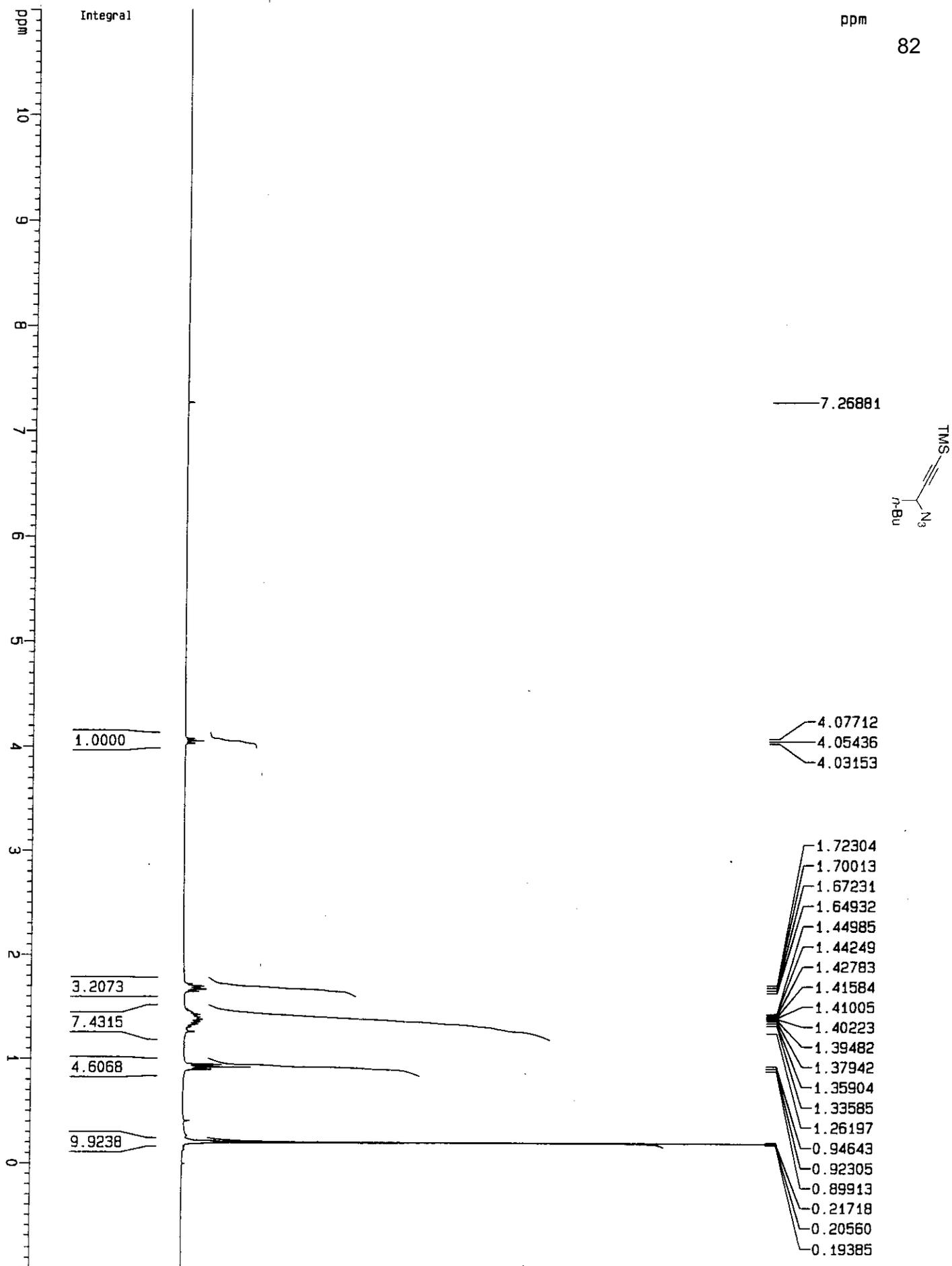
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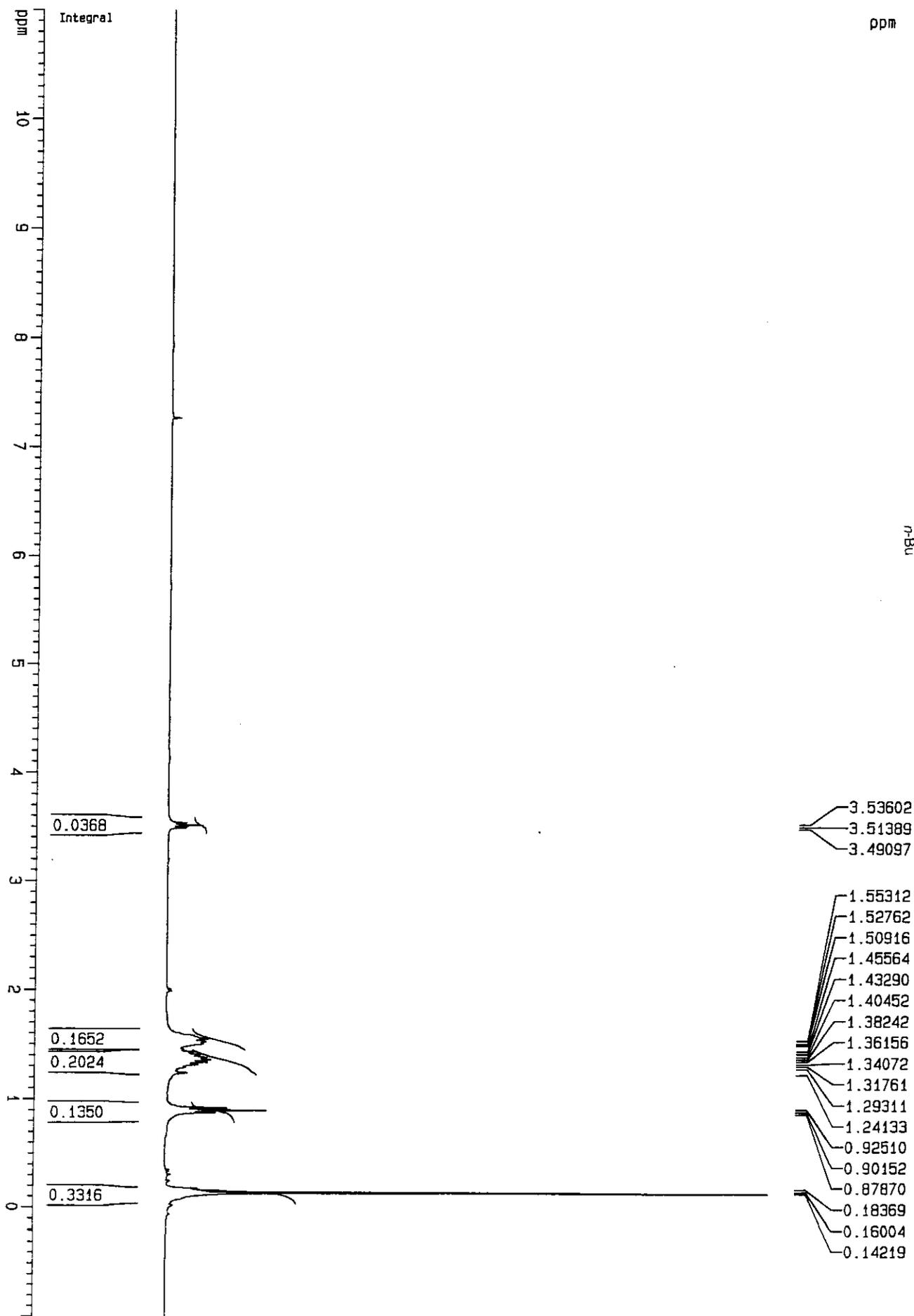
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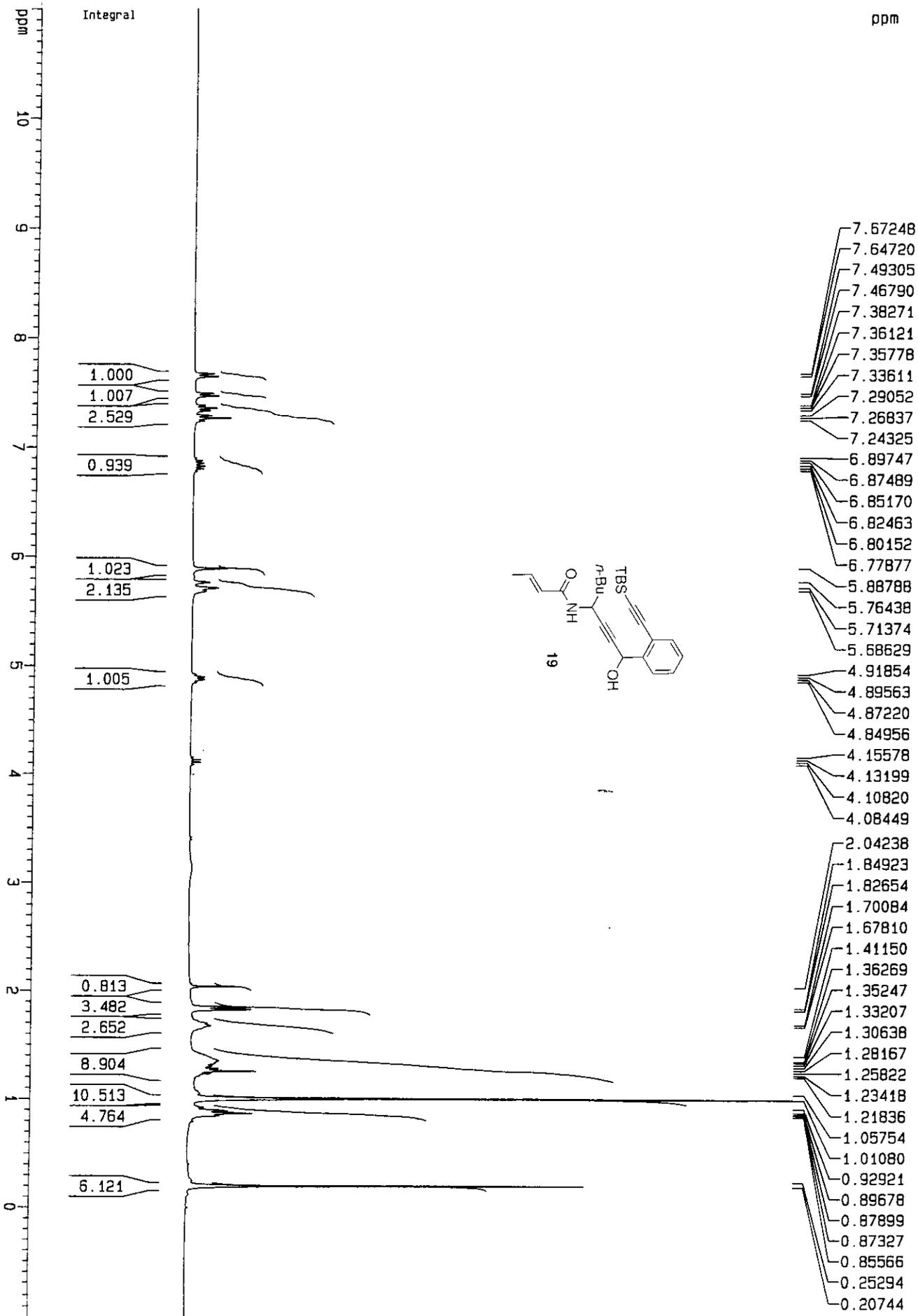
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