Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Supporting Information

Electrophilic *ipso*-Iodocyclization of *N*-benzyl-*N*-(1-naphthyl)propiolamides: Synthesis of Complex Polycyclic Lactams

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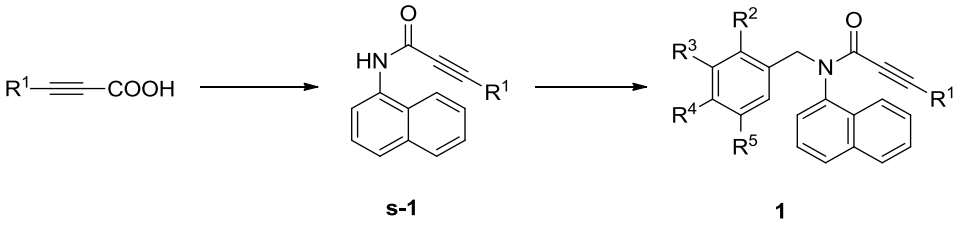
General Procedure A: Synthesis of *N*-Aryl Alkynylamides.

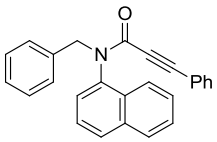


s-1 **1**

(a) For the synthesis of **s-1a**: To a stirred solution of 3-phenylpropiolic acid (438 mg, 3.00 mmol) and 4-methylmorpholine (455.2 mg, 4.50 mmol) in THF (20 mL) was added isobutyl chloroformate (491.7 mg, 3.60 mmol) in THF (3 mL) at 0 ℃, and the mixture was stirred at 0 ℃ for 0.5 h. 1-naphthylamine (515.5 mg, 3.60 mmol) in THF (2 mL) was added to the solution at 0 ℃, and the mixture was stirred at 0 ℃ for 1 h and at room temperature for 16 h. The reaction was quenched with water and extracted with CH_2Cl_2. The organic layer was washed with brine, dried over Na_2SO_4, and concentrated to furnish the corresponding crude amide **s-1a** (647.8 mg).

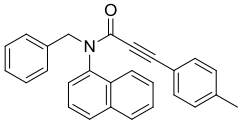
(b) For the synthesis of **1a**: To a suspension of 55% sodium hydride (130.9 mg, 3.00 mmol) in THF (12 mL) was added the crude amide **s-1a** (406.5 mg) in THF (12 mL) at 0 ℃, and the mixture was stirred at 0 ℃ for 0.5 h. Benzyl bromide (513.1 mg, 3.00 mmol) in THF (1.5 mL) was added to the solution at 0 ℃, and the mixture was stirred at 0 ℃ for 0.5 h and at room temperature for 3 h. The residue was purified by a silica gel column chromatography to give **1a** (509 mg, 1.41 mmol) in 78% yield from 3-phenylpropiolic acid. The above reactions were carried out under an atmosphere of argon.

Characterization Data of 1a-1q and 1aa



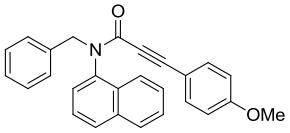
1a

***N*-benzyl-*N*-(naphthalen-1-yl)-3-phenylpropiolamide 1a.** Eluent: petroleum ether/ethyl acetate (10:1). Yield 509 mg (78%), light yellow solid, mp 75-77 ℃. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.92-7.83 (m, 3H), 7.53-7.51 (m, 2H), 7.37 (t, J = 8Hz, 1H), 7.25 (s, 5H), 7.18 (t, J = 7.6Hz, 1H), 7.09-7.04 (m, 3H), 6.73 (d, J = 7.2Hz, 2H), 5.70 (d, J = 14Hz, 1H), 4.46 (d, J = 14Hz, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 155.2, 137.5, 136.7, 134.4, 132.2, 130.8, 129.7, 129.3, 129.0, 128.4, 128.3, 128.0, 127.7, 127.7, 127.2, 126.4, 125.2, 122.6, 120.0, 90.8, 82.6, 52.0. IR (neat, cm^{-1}): 3436, 3060, 2215, 1637, 1491, 1384, 1301, 1211, 1078, 1030, 919, 778, 694, 604, 560. HRMS (ESI) Calcd for $C_{26}H_{20}NO$: $[M+H]^+$ = 362.1535. Found: 362.1545.



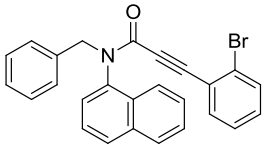
1b

***N*-benzyl-*N*-(naphthalen-1-yl)-3-(*p*-tolyl)propiolamide 1b.** Eluent: petroleum ether/ethyl acetate (10:1). Yield 338 mg (60%), light yellow solid, mp 98-100 ℃. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.92-7.83 (m, 3H), 7.51 (dd, J = 6.0Hz, 3.6Hz, 2H), 7.36 (t, J = 8Hz, 1H), 7.25 (s, 5H), 7.08 (d, J = 7.2Hz, 1H), 6.86 (d, J = 8Hz, 2H), 6.61 (d, J = 8Hz, 2H), 5.70 (d, J = 14Hz, 1H), 4.45 (d, J = 14Hz, 1H), 2.19 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 155.4, 140.2, 137.6, 136.8, 134.4, 132.2, 130.8,129.3, 128.9, 128.8, 128.4, 127.7, 127.6, 127.1, 126.4, 125.2, 122.6, 117.0, 91.3, 82.2, 52.0, 21.4. IR (neat, cm^{-1}): 3370, 3059, 2922, 2213, 1637, 1509, 1384, 1299, 1209, 1177, 1079, 976, 817, 777, 700, 663, 533. HRMS (ESI) Calcd for $C_{27}H_{22}NO$: $[M+H]^+$ = 376.1690. Found: 376.1701.



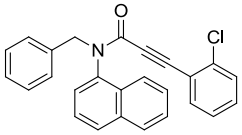
1c

***N*-benzyl-3-(4-methoxyphenyl)-*N*-(naphthalen-1-yl)propiolamide 1c.** Eluent: petroleum ether/ethyl acetate (10:1). Yield 411 mg (70%), light yellow solid, mp 90-92 ℃. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.92-7.83 (m, 3H), 7.51 (dd, J = 6.4Hz, 3.2Hz, 2H), 7.37 (t, J = 8Hz, 1H), 7.25 (s, 5H), 7.08 (d, J = 7.2Hz, 1H), 6.65 (d, J = 9.2Hz, 2H), 6.57 (d, J = 8.8Hz, 2H), 5.70 (d, J = 14Hz, 1H), 4.45 (d, J = 14Hz, 1H), 3.66 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 160.7, 155.5, 137.7, 136.9, 134.4, 134.0, 130.8, 129.3, 128.8, 128.4, 128.3, 127.7, 127.6, 127.1, 126.4, 125.2, 122.6, 113.7, 111.9, 91.6, 82.0, 55.1, 51.9. IR (neat, cm^{-1}): 3434, 3060, 2839, 2209, 1635, 1602, 1509, 1458, 1384, 1291, 1253, 1213, 1171, 1079, 1031, 976, 834, 728, 665, 540. HRMS (ESI) Calcd for $C_{27}H_{22}NO_2$: $[M+H]^+$ = 392.1651. Found: 392.1651.



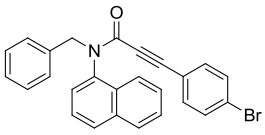
1d

***N*-benzyl-3-(2-bromophenyl)-*N*-(naphthalen-1-yl)propiolamide 1d.** Eluent: petroleum ether/ethyl acetate (10:1). Yield 383 mg (58%), light yellow solid, mp 111-113 ℃. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.90-7.83 (m, 3H), 7.54-7.51 (m, 2H), 7.35 (t, J = 8Hz, 1H), 7.25 (s, 6H), 7.08-7.04 (m, 4H), 5.72 (d, J = 14Hz, 1H), 4.39 (d, J = 14Hz, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 154.9, 137.1, 136.7, 134.7, 134.5, 132.1, 130.8, 129.4, 129.3, 129.0, 128.4, 128.3, 128.0, 127.9, 127.7, 127.2, 126.7, 126.5, 122.6, 88.2, 86.2, 52.2. IR (neat, cm^{-1}): 3394, 3060, 2923, 2360, 2216, 1636, 1597, 1468, 1385, 1302, 1213, 1078, 970, 776, 730, 606, 528. HRMS (ESI) Calcd for $C_{26}H_{18}BrNNaO$: $[M+Na]^+$ = 426.0475. Found: 426.0469.



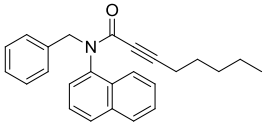
1e

***N*-benzyl-3-(2-chlorophenyl)-*N*-(naphthalen-1-yl)propiolamide 1e.** Eluent: petroleum ether/ethyl acetate (10:1). Yield 297 mg (50%), light yellow solid,. mp 110-112 ℃ ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.91-7.84 (m, 3H), 7.54-7.51 (m, 2H), 7.34 (t, J = 8Hz, 1H), 7.25 (s, 5H), 7.12-6.97 (m, 5H), 5.72 (d, J = 14Hz, 1H), 4.40 (d, J = 14Hz, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 154.9, 137.1, 136.7, 136.4, 134.6, 134.2, 130.8, 130.7, 129.4, 129.0, 128.9, 128.4, 128.3, 127.8, 127.7, 127.2, 126.4, 126.1, 125.4, 122.5, 120.5, 86.9, 86.6, 52.2. IR (neat, cm^{-1}): 3434, 3061, 2846, 2360, 2218, 1637, 1472, 1431, 1385, 1321, 1255, 1158, 1033, 911, 776, 700, 609, 535. HRMS (ESI) Calcd for $C_{26}H_{18}ClNNaO$: $[M+Na]^+$ = 418.0988. Found: 418.0975.



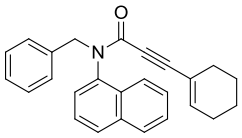
1f

***N*-benzyl-3-(4-bromophenyl)-*N*-(naphthalen-1-yl)propiolamide 1f.** Eluent: petroleum ether/ethyl acetate (10:1). Yield 561 mg (85%), light yellow liquid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.92-7.81 (m, 3H), 7.53-7.51 (m, 2H), 7.37 (t, J = 8Hz, 1H), 7.25 (s, 5H), 7.20-7.18 (m, 1H), 7.10-7.03 (m, 2H), 6.73-6.71 (m, 1H), 6.54 (d, J = 8.4Hz, 1H), 5.67 (d, J = 14Hz, 1H), 4.47 (d, J = 14Hz, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 154.9, 137.4, 136.6, 134.3, 133.4, 132.2, 131.4, 130.7, 129.7, 129.3, 129.0, 128.4, 128.0, 127.6, 127.2, 126.5, 125.1, 122.5, 119.0, 89.5, 83.5, 52.0. IR (neat, cm^{-1}): 3394, 3060, 2925, 2217, 1638, 1488, 1385, 1300, 1210, 1072, 1012, 912, 825, 730, 700, 614, 528. HRMS (ESI) Calcd for $C_{26}H_{18}BrNNaO$: $[M+Na]^+$ = 426.0475. Found: 426.0469.



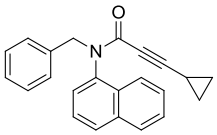
1g

***N*-benzyl-*N*-(naphthalen-1-yl)oct-2-ynamide 1g.** Eluent: petroleum ether/ethyl acetate (10:1). Yield 320 mg (60%), light yellow liquid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.89-7.77 (m, 3H), 7.52-7.50 (m, 2H), 7.32 (t, J = 8Hz, 1H), 7.25-7.21 (m, 6H), 6.99 (d, J = 7.2Hz, 1H), 5.64 (d, J = 14Hz, 1H), 4.35 (d, J = 14Hz, 1H), 1.82 (t, J = 6.8Hz, 2H), 0.93-0.79 (m, 5H), 0.67-0.56 (m, 4H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 155.3, 137.7, 136.9, 134.4, 130.7, 129.3, 128.7, 128.3, 128.2, 127.6, 127.5, 126.9, 126.3, 125.1, 122.6, 94.1, 74.9, 51.9, 30.0, 26.7, 21.8, 18.4, 13.6. IR (neat, cm^{-1}): 3439, 2929, 2360, 2245, 1639, 1508, 1383, 1290, 1223, 1116, 1024, 975, 805, 777, 700, 617, 546. HRMS (ESI) Calcd for $C_{25}H_{26}NO$: $[M+H]^+$ = 356.2002. Found: 356.2014.



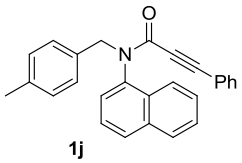
1h

***N*-benzyl-3-(cyclohex-1-en-1-yl)-*N*-(naphthalen-1-yl)propiolamide 1h.** Eluent: petroleum ether/ethyl acetate (10:1). Yield 345 mg (63%), yellow liquid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.89-7.78 (m, 3H), 7.52-7.50 (m, 2H), 7.33 (t, J = 8Hz, 1H), 7.23 (s, 5H), 7.01 (d, J = 7.2Hz, 1H), 5.66 (d, J = 14Hz, 1H), 4.40 (d, J = 14Hz, 1H), 1.84 (s, 2H), 1.33 (s, 6H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 155.6, 140.1, 137.7, 137.0, 134.4, 130.8, 129.3, 128.7, 128.3, 128.2, 127.6, 127.0, 126.3, 125.1, 122.6, 118.6, 93.0, 80.6, 51.9, 27.5, 25.6, 21.6, 20.8. IR (neat, cm^{-1}): 3388, 2927, 2858, 2360, 2203, 1638, 1509, 1383, 1295, 1254, 1183, 1077, 1018, 918, 778, 701, 669, 639. HRMS (ESI) Calcd for $C_{26}H_{24}NO$: $[M+H]^+$ = 366.1848. Found: 366.1858.



1i

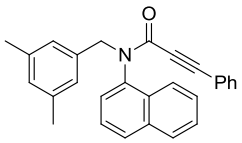
***N*-benzyl-3-cyclopropyl-*N*-(naphthalen-1-yl)propiolamide 1i.** Eluent: petroleum ether/ethyl acetate (10:1). Yield 390 mg (80%), light yellow solid, mp 82-84 ℃. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.89-7.74 (m, 3H), 7.53-7.48 (m, 2H), 7.34-7.30 (m, 1H), 7.24-7.20 (m, 5H), 6.98-6.96 (m, 1H), 5.60 (d, J = 14Hz, 1H), 4.38 (d, J = 14Hz, 1H), 0.85-0.78 (m, 1H), 0.49-0.37 (m, 2H), -0.07- -0.13 (m, 1H), -0.25- -0.31 (m, 1H). ^{13}C NMR ($CDCl_3$, 100MHz) δ 155.2, 137.8, 136.9, 134.3, 130.8, 129.2, 128.6, 128.3, 128.2, 127.5, 127.4, 126.9, 126.3, 125.1, 122.6, 97.9, 69.9, 51.8, 8.9, 8.7, -1.0. IR (neat, cm^{-1}): 3439, 3060, 2925, 2225, 1636, 1596, 1386, 1288, 1254, 1187, 1079, 948, 877, 778, 731, 667, 617. HRMS (ESI) Calcd for $C_{23}H_{20}NO$: $[M+H]^+$ = 326.1545. Found: 326.1545.



1j

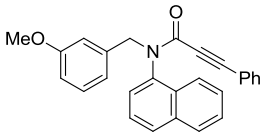
***N*-(4-methylbenzyl)-*N*-(naphthalen-1-yl)-3-phenylpropiolamide 1j.** Eluent: petroleum ether/ethyl acetate (10:1). Yield 422 mg (75%), light yellow solid, mp 116-118 ℃. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.93-7.85 (m, 3H), 7.54-7.52 (m, 2H), 7.37 (t, J = 8Hz, 1H), 7.24-7.12 (m, 3H), 7.09-7.04 (m, 5H), 6.73 (d, J = 6.8Hz, 2H), 5.69 (d, J = 14Hz, 1H), 4.39 (d, J = 14Hz, 1H), 2.30 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 155.2, 137.6, 137.3, 134.4, 133.8, 132.2, 130.8, 129.7, 129.3, 129.0, 128.9, 128.3, 128.0, 127.8, 127.2, 126.4, 125.2, 122.6, 120.1, 90.7, 82.6, 51.7, 21.1.

IR (neat, cm^{-1}): 3435, 3054, 2922, 2214, 1637, 1595, 1508, 1384, 1299, 1210, 1116, 1020, 804, 777, 689, 604, 533. HRMS (ESI) Calcd for $C_{27}H_{22}NO$: $[M+H]^+$ = 376.1690. Found: 376.1701.



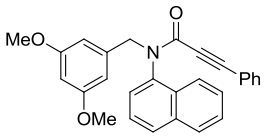
1k

***N*-(3,5-dimethylbenzyl)-*N*-(naphthalen-1-yl)-3-phenylpropiolamide 1k.** Eluent: petroleum ether/ethyl acetate (10:1). Yield 350 mg (60%), light yellow solid, mp 77-79 ℃. ^1H NMR ($CDCl_3$, 400 MHz) δ 7.93-7.85 (m, 3H), 7.55-7.52 (m, 2H), 7.40-7.36 (m, 1H), 7.20-7.16 (m, 1H), 7.11-7.04 (m, 3H), 6.87 (d, J = 8.8Hz, 3H), 6.74-6.72 (m, 2H), 5.69 (d, J = 14Hz, 1H), 4.32 (d, J = 14Hz, 1H), 2.23 (s, 6H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 155.2, 137.9, 137.6, 136.6, 134.4, 132.2, 130.8, 129.7, 129.2, 128.9, 128.3, 128.0, 127.8, 127.1, 127.0, 126.4, 125.1, 122.6, 120.1, 90.8, 82.7, 51.9, 21.1. IR (neat, cm^{-1}): 3439, 3056, 2918, 2359, 2217, 1638, 1489, 1382, 1304, 1211, 1116, 1039, 983, 832, 758, 602. HRMS (ESI) Calcd for $C_{28}H_{24}NO$: $[M+H]^+$ = 390.1847. Found: 390.1858.



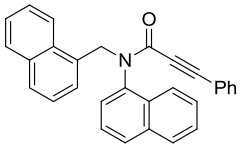
1l

***N*-(3-methoxybenzyl)-*N*-(naphthalen-1-yl)-3-phenylpropiolamide 1l.** Eluent: petroleum ether/ethyl acetate (10:1). Yield 381 mg (65%), light yellow solid, mp 109-111 ℃. ^1H NMR ($CDCl_3$, 400 MHz) δ 7.83-7.93 (m, 3H), 7.53-7.51 (m, 2H), 7.40-7.36 (m, 1H), 7.18-7.15 (m, 3h), 7.08-7.04 (m, 3H), 6.78 (d, J = 8.8Hz, 2H), 6.72 (d, J = 7.2Hz, 2H), 5.63 (d, J = 14Hz, 1H), 4.40 (d, J = 14Hz, 1H), 3.76 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 159.1, 155.1, 137.5, 134.4, 132.2, 130.8, 130.7, 129.7, 129.0, 128.9, 128.3, 128.0, 127.8, 127.1, 126.4, 125.2, 122.6, 120.1, 113.7, 90.7, 82.6, 55.1, 51.4. IR (neat, cm^{-1}): 3398, 2929, 2215, 1637, 1596, 1489, 1383, 1307, 1262, 1212, 1155, 1049, 919, 777, 691, 594. HRMS (ESI) Calcd for $C_{27}H_{22}NO_2$: $[M+H]^+$ = 392.1651. Found: 392.1651.



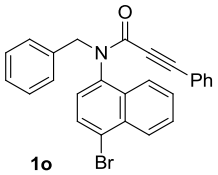
1m

***N*-(3,5-dimethoxybenzyl)-*N*-(naphthalen-1-yl)-3-phenylpropiolamide 1m.** Eluent: petroleum ether/ethyl acetate (10:1). Yield 600 mg (95%), light yellow solid, mp 85-87 ℃. ^1H NMR ($CDCl_3$, 400 MHz) δ 7.93-7.85 (m, 3H), 7.56-7.51 (m, 2H), 7.42-7.38 (m, 1H), 7.19 (t, J = 7.6Hz, 2H), 7.06 (t, J = 8Hz, 2H), 6.73 (d, J = 7.2Hz, 2H), 6.42 (d, J = 2.4Hz, 2H), 6.36 (t, J = 2.4Hz, 1H), 5.66 (d, J = 14Hz, 1H), 4.37 (d, J = 14Hz, 1H), 3.69 (s, 6H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 160.6, 155.2, 138.9, 137.5, 134.4, 132.2, 130.7, 129.7, 129.0, 128.3, 128.0, 127.7, 127.2, 126.4, 125.2, 122.5, 120.0, 107.0, 100.0, 90.9, 82.5, 55.2, 52.0. IR (neat, cm^{-1}): 3390, 2931, 2360, 2216, 1636, 1600, 1463, 1430, 1383, 1300, 1203, 1154, 1065, 833, 776, 689, 604. HRMS (ESI) Calcd for $C_{28}H_{24}NO_3$: $[M+H]^+$ = 422.1758. Found: 422.1756.



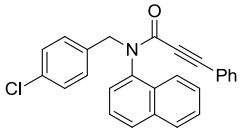
1n

***N*-(naphthalen-1-yl)-*N*-(naphthalen-1-ylmethyl)-3-phenylpropiolamide 1n.** Eluent: petroleum ether/ethyl acetate (10:1). Yield 543 mg (88%), light yellow solid, mp 52-54 ℃. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.26 (d, J = 8.4Hz, 1H), 7.86-7.77 (m, 4H), 7.70 (d, J = 8.4Hz, 1H), 7.55-7.42 (m, 4H), 7.19-7.11 (m, 3H), 7.03 (t, J = 8Hz, 2H), 6.96 (d, J = 8Hz, 1H), 6.77 (d, J = 7.2Hz, 1H), 6.69 (d, J = 7.2Hz, 2H), 6.17 (d, J = 14.4Hz, 1H), 5.04 (d, J = 14.4Hz, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 155.0, 136.7, 134.2, 133.6, 132.2, 132.0, 131.8, 131.0, 129.7, 128.9, 128.8, 128.7, 128.5, 128.3, 128.0, 127.8, 127.1, 126.6, 126.3, 125.8, 125.0, 124.8, 124.1, 122.4, 120.0, 90.9, 82.6, 49.1.IR (neat, cm^{-1}): 3394, 3054, 2924, 2216, 1634, 1575, 1489, 1386, 1285, 1204, 1160, 1053, 973, 910, 778, 731, 689, 604, 533. HRMS (ESI) Calcd for $C_{30}H_{21}NNaO$: $[M+Na]^+$ = 434.1527. Found: 434.1521.



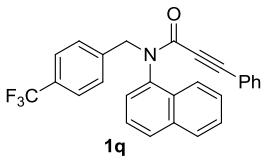
1o

***N*-benzyl-*N*-(4-bromonaphthalen-1-yl)-3-phenylpropiolamide 1o.** Eluent: petroleum ether/ethyl acetate (10:1). Yield 517 mg (80%), light yellow solid, mp 120-122 ℃. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.30 (d, J = 8.4Hz, 1H), 7.85 (d, J = 8.4Hz, 1H), 7.68 (d, J = 7.6Hz, 1H), 7.65-7.55 (m, 2H), 7.27-7.17 (m, 6H), 7.07 (t, J = 8Hz, 2H), 6.92 (d, J = 8Hz, 1H), 6.76-6.73 (m, 2H), 5.68 (d, J = 14Hz, 1H), 4.42 (d, J = 14Hz, 1H), . ^{13}C NMR ($CDCl_3$, 100 MHz) δ154.9, 137.3, 136.4, 132.7, 132.5, 132.1, 129.8, 129.3, 129.2, 128.5, 128.1, 127.9, 127.8, 127.8, 123.5, 123.1, 119.8, 91.2, 82.4, 52.0. HRMS (ESI) Calcd for $C_{26}H_{18}BrNNaO$: $[M+Na]^+$ = 426.0475. Found: 426.0469.



1p

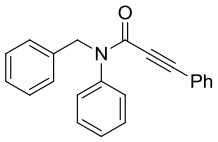
***N*-(4-chlorobenzyl)-*N*-(naphthalen-1-yl)-3-phenylpropiolamide 1p.** Eluent: petroleum ether/ethyl acetate (10:1). Yield 517 mg (87%), light yellow solid, mp 100-102 ℃. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.94-7.81 (m, 3H), 7.55-7.52 (m, 2H), 7.39 (t, J = 8Hz, 1H), 7.24-7.17 (m, 5H), 7.09-7.04 (m, 3H), 6.73 (d, J = 7.2Hz, 2H), 5.64 (d, J = 14.4Hz, 1H), 4.42 (d, J = 14.4Hz, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 155.2, 137.3, 135.3, 134.4, 133.6, 132.2, 130.7, 130.6, 129.8, 129.1, 128.6, 128.4, 128.1, 127.6, 127.3, 126.5, 125.2, 122.4, 119.9, 91.1, 82.4, 51.3. IR (neat, cm^{-1}): 3410, 3059, 2923, 2219, 1636, 1312, 1211, 1175, 1091, 978, 803, 735, 689, 603, 526. HRMS (ESI) Calcd for $C_{26}H_{18}ClNNaO$: $[M+Na]^+$ = 418.0988. Found: 418.0975.



1q

***N*-(naphthalen-1-yl)-3-phenyl-*N*-(4-(trifluoromethyl)benzyl)propiolamide 1q.** Eluent:

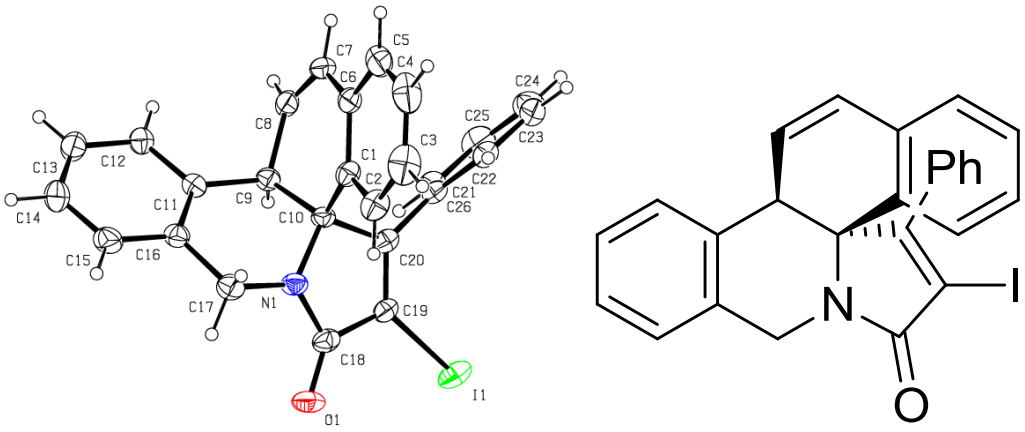
petroleum ether/ethyl acetate (10:1). Yield 515 mg (80%), light yellow solid, mp 107-109 ℃. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.95-7.82 (m, 3H), 7.56-7.52 (m, 4H), 7.40 (d, J = 7.6Hz, 3H), 7.20 (t, J = 7.2Hz, 1H), 7.12-7.05 (m, 3H), 6.74 (d, J = 7.6Hz, 2H), 5.72 (d, J = 14.4Hz, 1H), 4.51 (d, J = 14.4Hz, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 155.3, 140.8, 137.4, 134.5, 132.3, 130.6, 129.9, 129.6, 129.5, 129.2, 128.5, 128.1, 127.6, 127.4, 126.6, 125.4, 125.3, 125.2, 122.4, 119.9, 91.3, 82.3, 51.6. IR (neat, cm^{-1}): 3439, 3059, 2926, 2217, 1640, 1489, 1383, 1324, 1211, 1165, 1120, 1066, 1020, 857, 776, 688, 601, 515. HRMS (ESI) Calcd for $C_{27}H_{19}F_3NO$: $[M+H]^+$ = 430.1433. Found: 430.1419.



1aa

***N*-benzyl-*N*,3-diphenylpropiolamide 1aa.** Eluent: petroleum ether/ethyl acetate (10:1). Yield 401 mg (86%), yellow liquid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.35-7.34 (m, 3H), 7.30 (t, J = 2.4Hz, 1H), 7.28-7.25 (m, 5H), 7.22-7.19 (m, 2H), 7.17-7.15 (m, 2H), 7.10-7.08 (m, 2H), 5.00 (s, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 154.3, 141.6, 136.6, 132.4, 129.9, 128.9, 128.7, 128.6, 128.4, 128.2, 128.1, 127.5, 120.3, 91.4, 82.5, 52.2. IR (neat, cm^{-1}): 3435, 3060, 2924, 2216, 1635, 1592, 1491, 1388, 1316, 1202, 1075, 1024, 919, 757, 730, 694, 605, 527. HRMS (ESI) Calcd for $C_{22}H_{18}NO$: $[M+H]^+$ = 312.1377. Found: 312.1388.

X-ray Diffraction Analysis of Compound 2a



X-ray structure of 2a

Bond precision: C-C = 0.0064 A Wavelength=0.71070

Cell: a=9.6284(4) b=11.7266(6) c=19.1651(9)

alpha=75.073(5) beta=77.631(4) gamma=87.141(4)

Temperature: 294 K

	Calculated	Reported
Volume	2042.31(17)	2042.32(17)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C26 H18 I N O	C26 H18 I N O
Sum formula	C26 H18 I N O	C26 H18 I N O
Mr	487.31	487.31
Dx,g cm-3	1.585	1.585
Z	4	4
Mu (mm-1)	1.585	1.585
F000	968.0	968.0
F000’	966.43	
h,k,lmax	11,14,23	11,14,23
Nref	7758	7160
Tmin,Tmax	0.859,0.909	0.969,1.000
Tmin’	0.728	

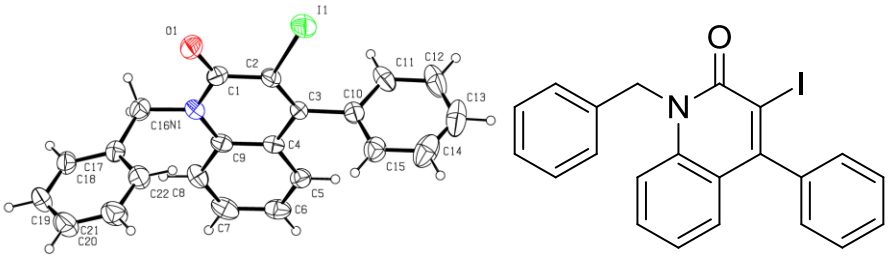
Correction method= MULTI-SCAN

Data completeness= 0.923 Theta(max)= 25.680

R(reflections)= 0.0393(5491) wR2(reflections)= 0.0810(7160)

S = 1.035 Npar= 523

X-ray Diffraction Analysis of Compound 3aa



X-ray structure of 3aa

Bond precision: C-C = 0.0123 A Wavelength=0.71070

Cell: a=10.5172(8) b=17.5717(6) c=20.0963(11)

alpha=90 beta=90 gamma=90

Temperature: 293 K

Calculated Reported

Volume 3713.9(4) 3713.9(4)

Space group P b c a P b c a

Hall group -P 2ac 2ab -P 2ac 2ab

Moiety formula C22 H16 I N O C22 H16 I N O

Sum formula C22 H16 I N O C22 H16 I N O

Mr 437.26 437.26

Dx,g cm-3 1.564 1.564

Z 8 8

Mu (mm-1) 1.733 1.733

F000 1728.0 1728.0

F000’ 1724.79

h,k,lmax 14,23,27 13,23,25

Nref 4733 4308

Tmin,Tmax 0.532,0.648 0.692,1.000

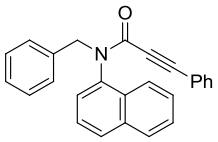
Tmin’ 0.521

Correction method= MULTI-SCAN

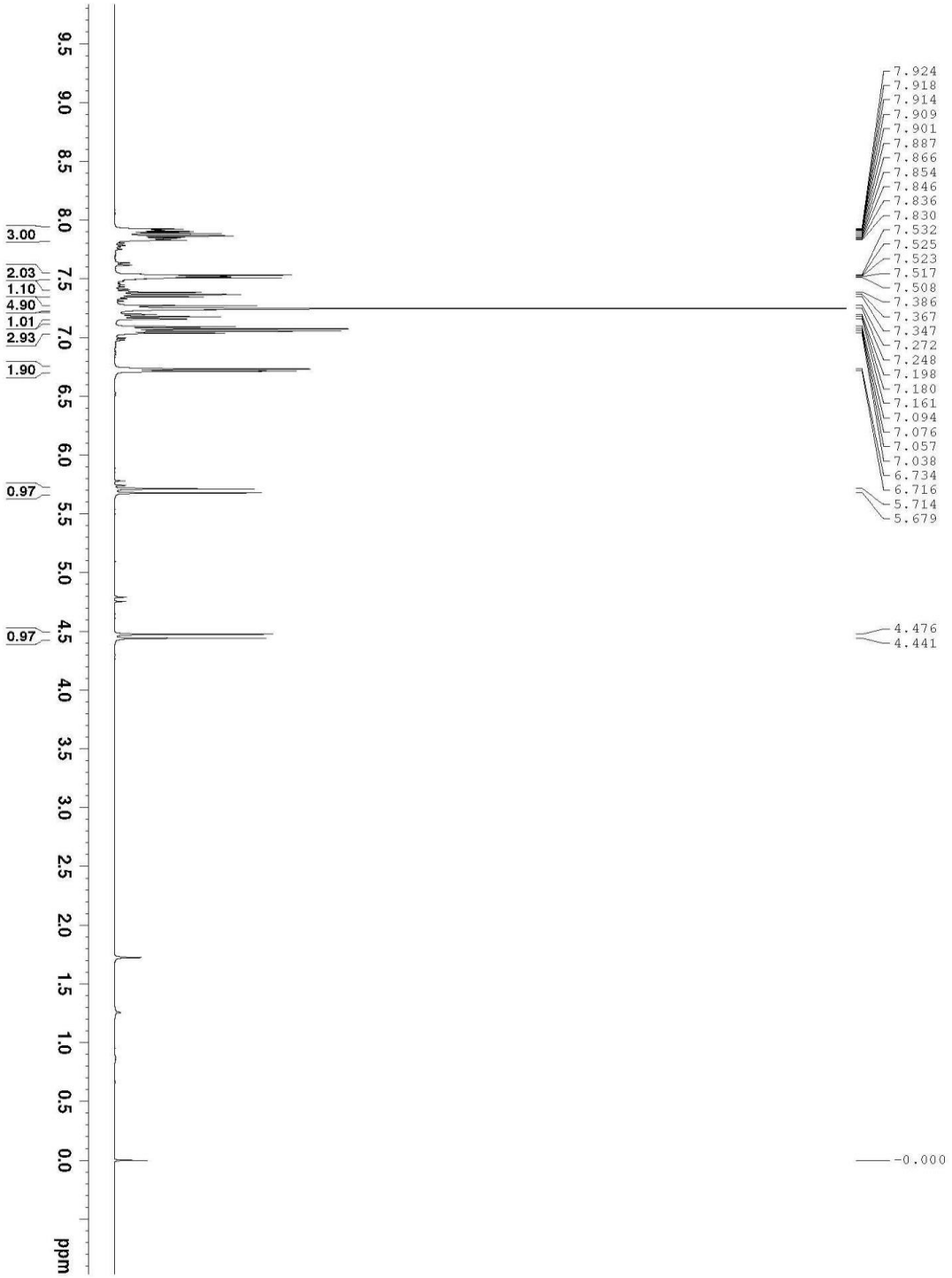
Data completeness= 0.910 Theta(max)= 28.550

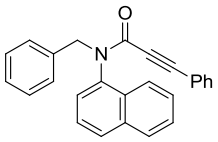
R(reflections)= 0.0741(2224) wR2(reflections)= 0.1546(4308)

S = 1.106 Npar= 226



1a





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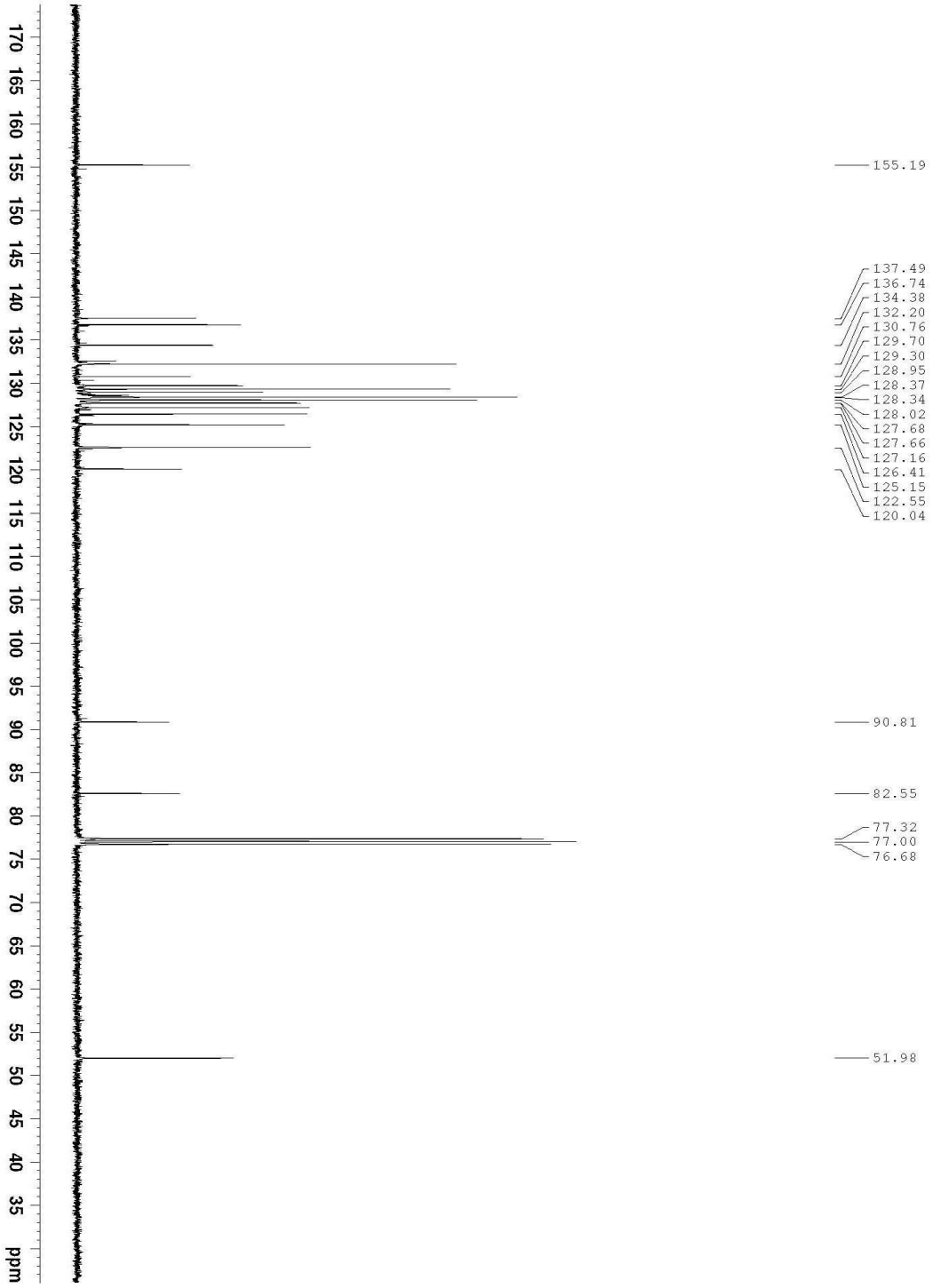

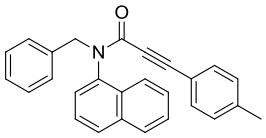

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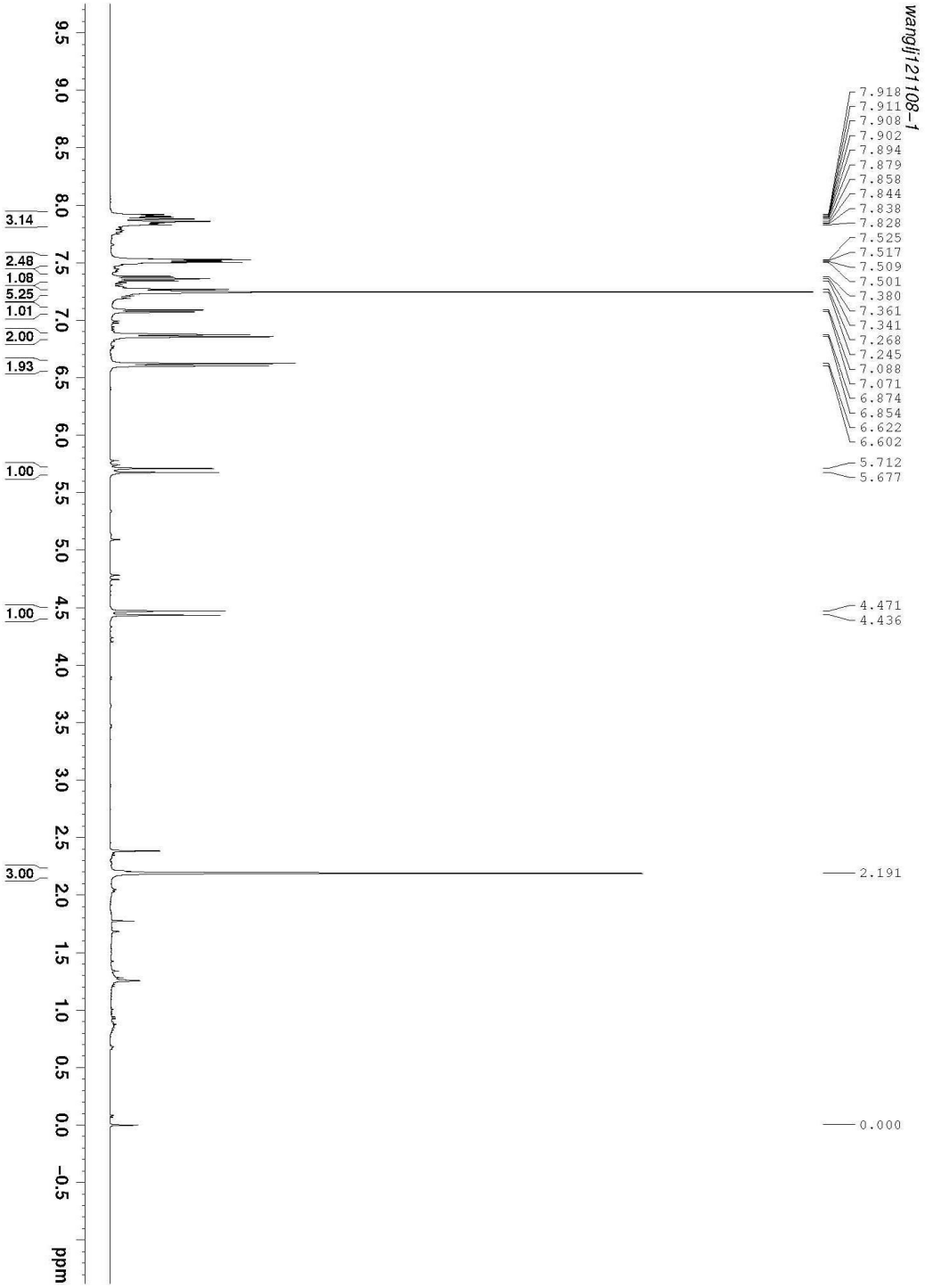

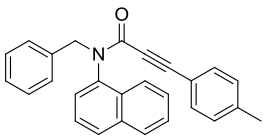

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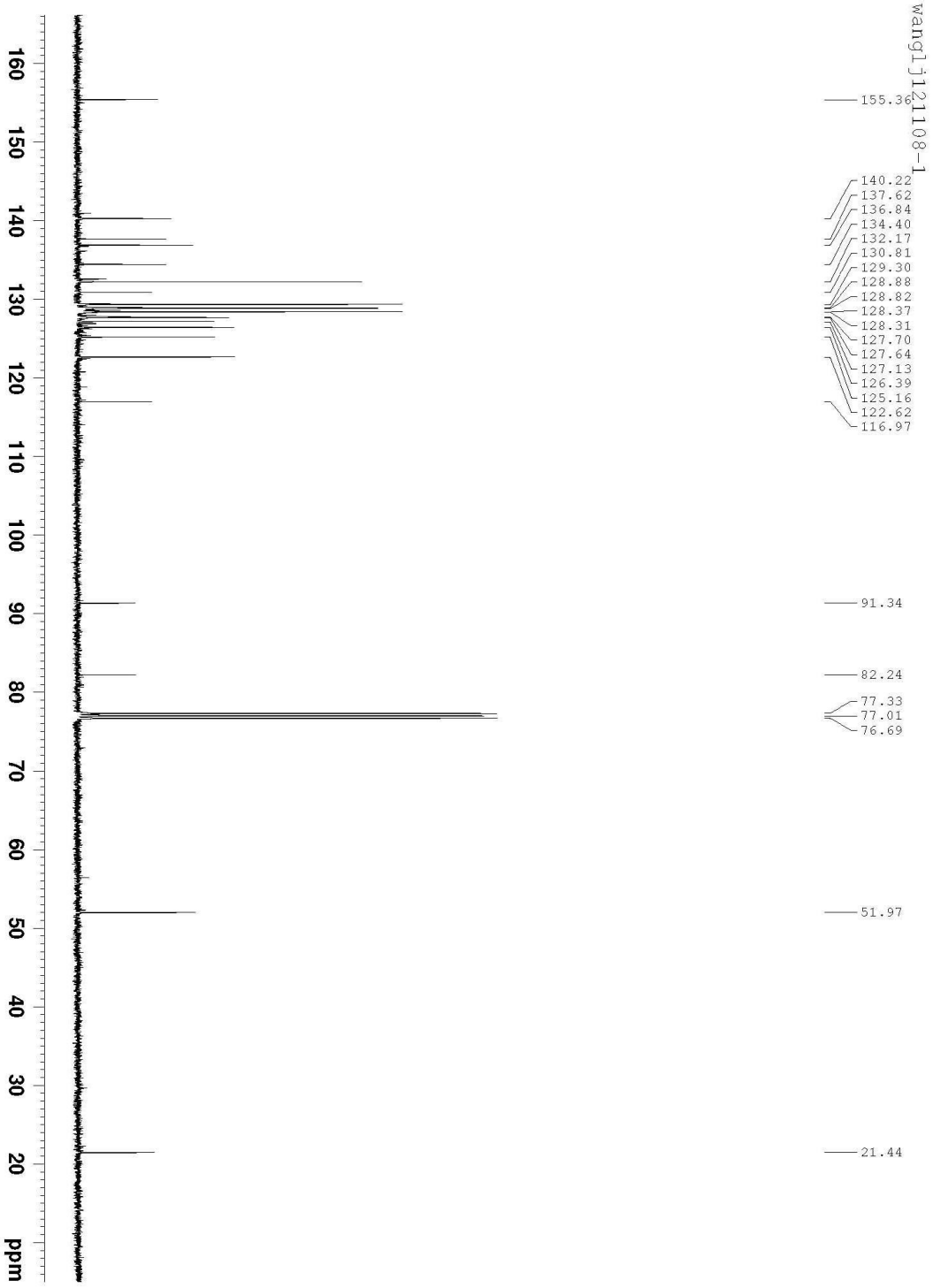

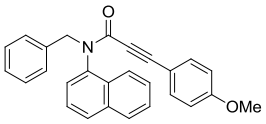

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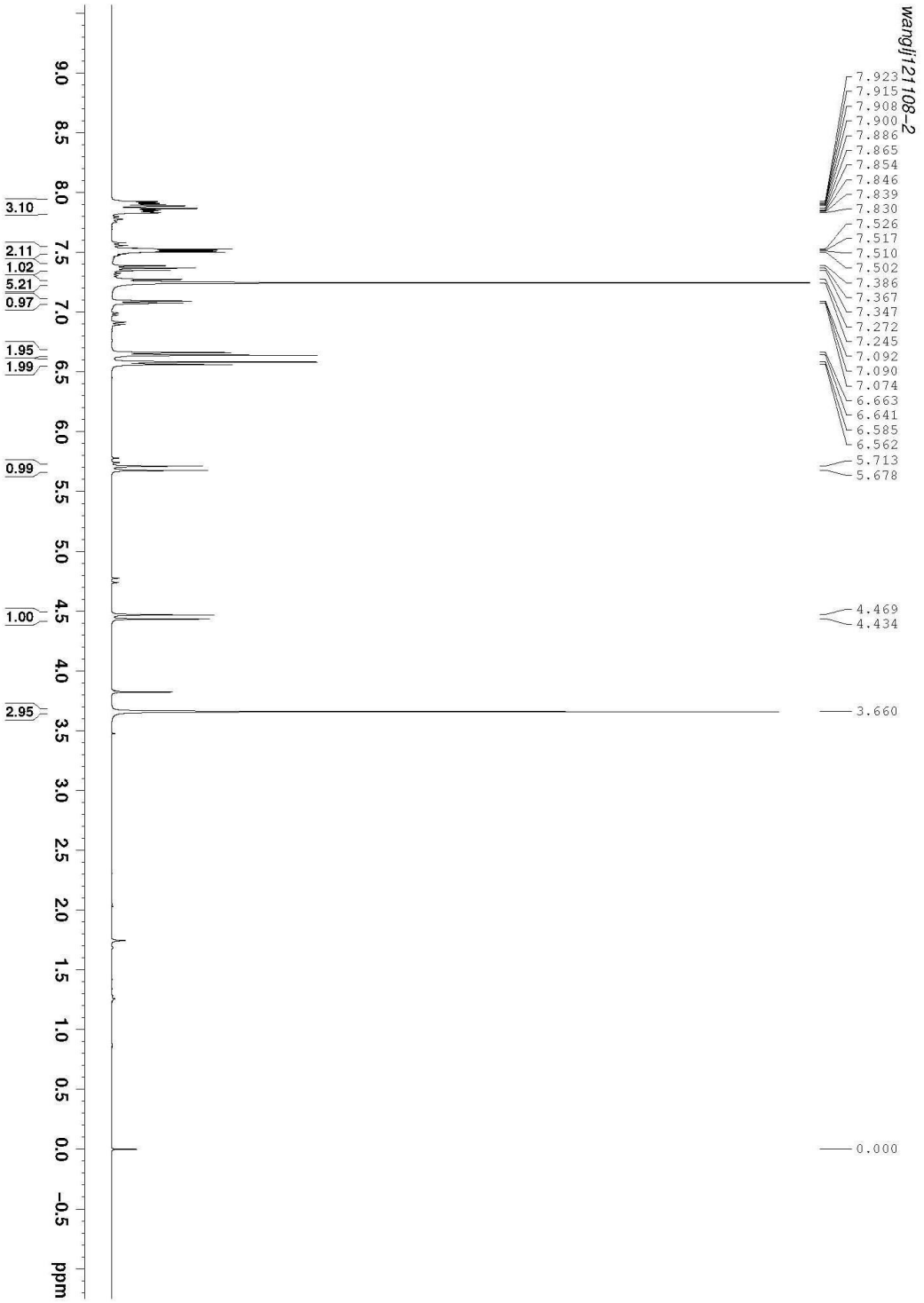

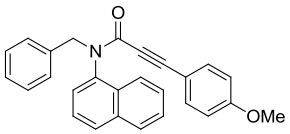

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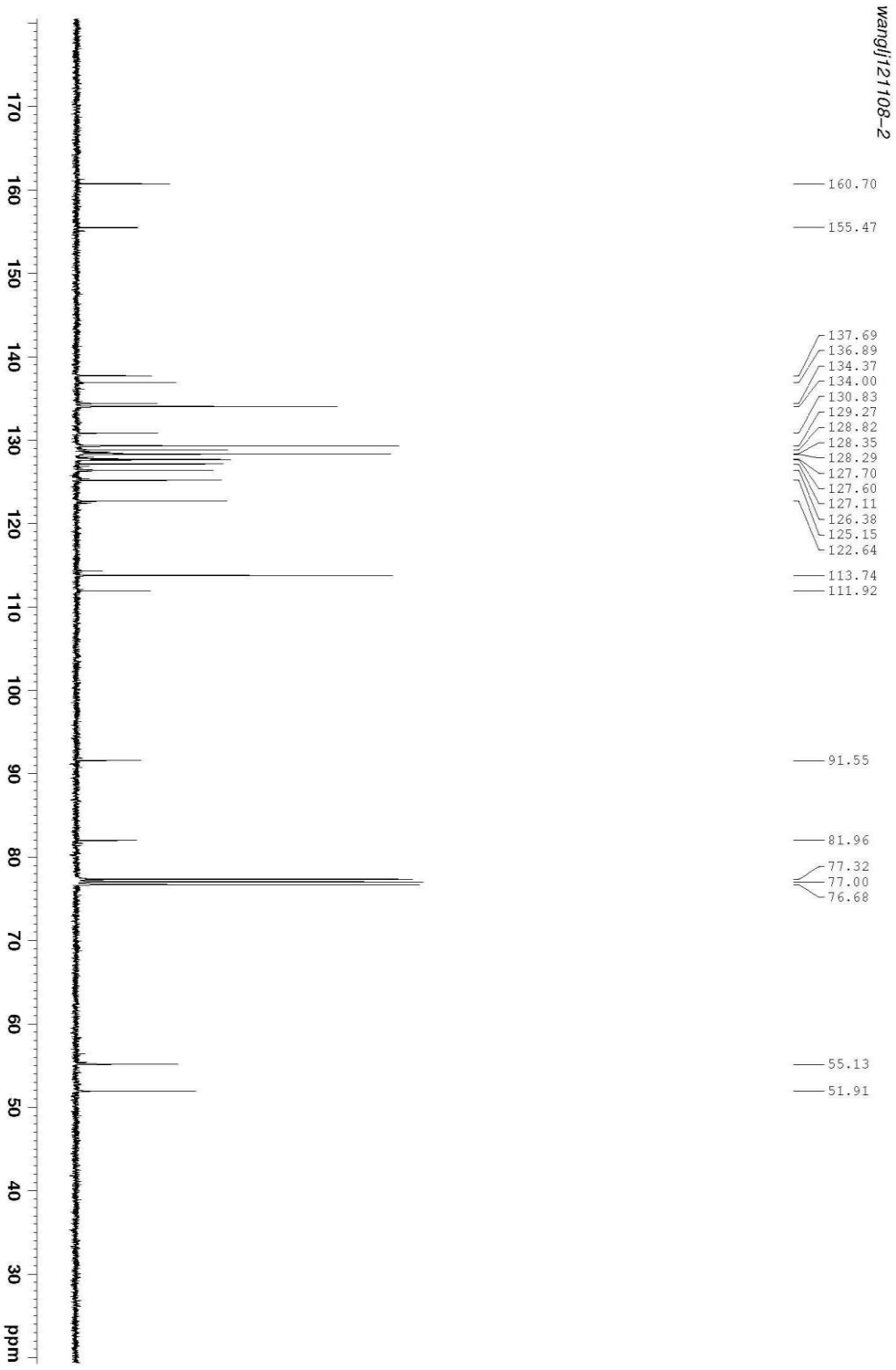

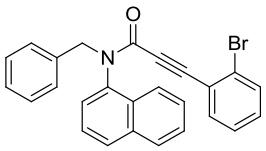

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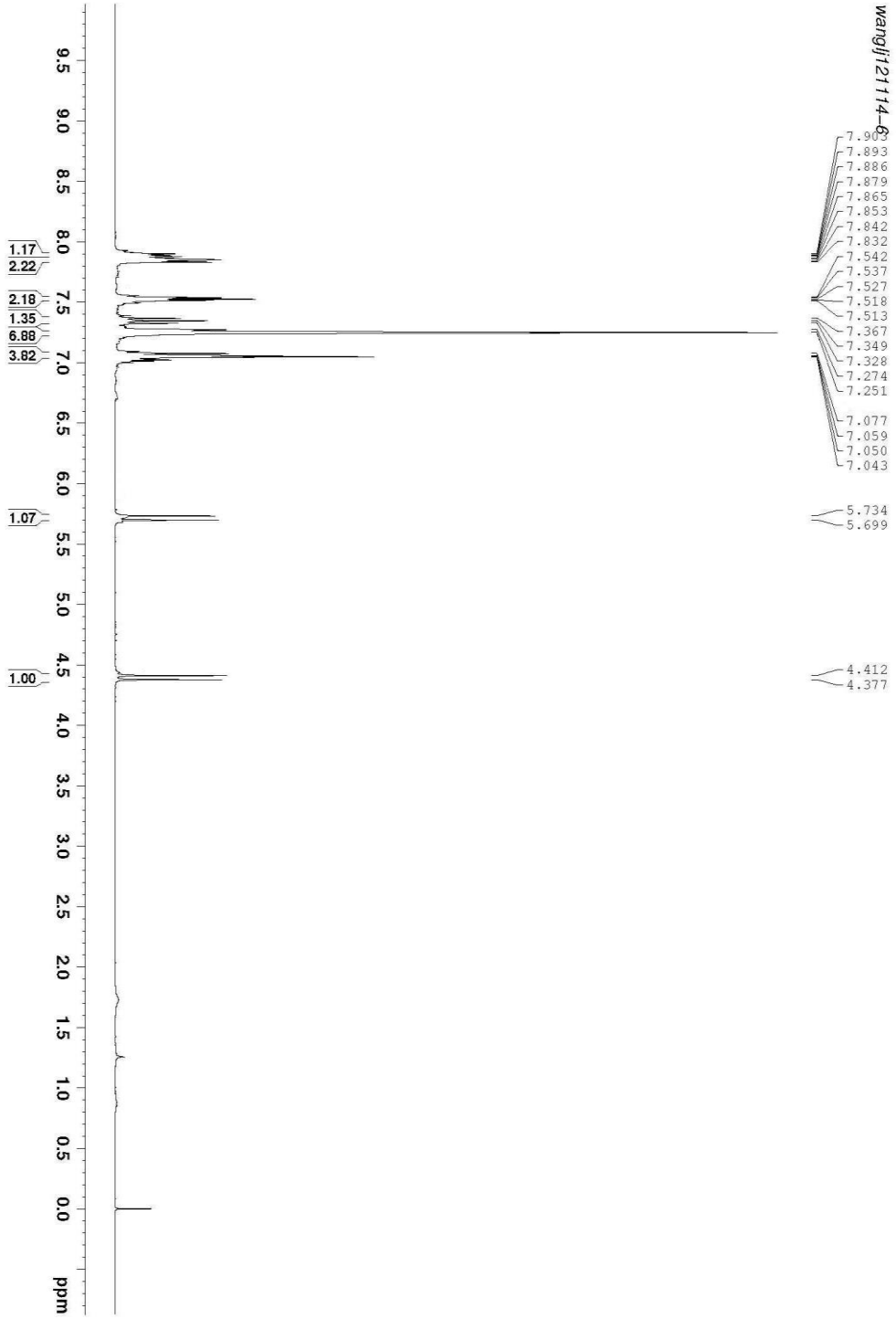

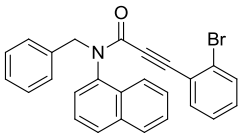

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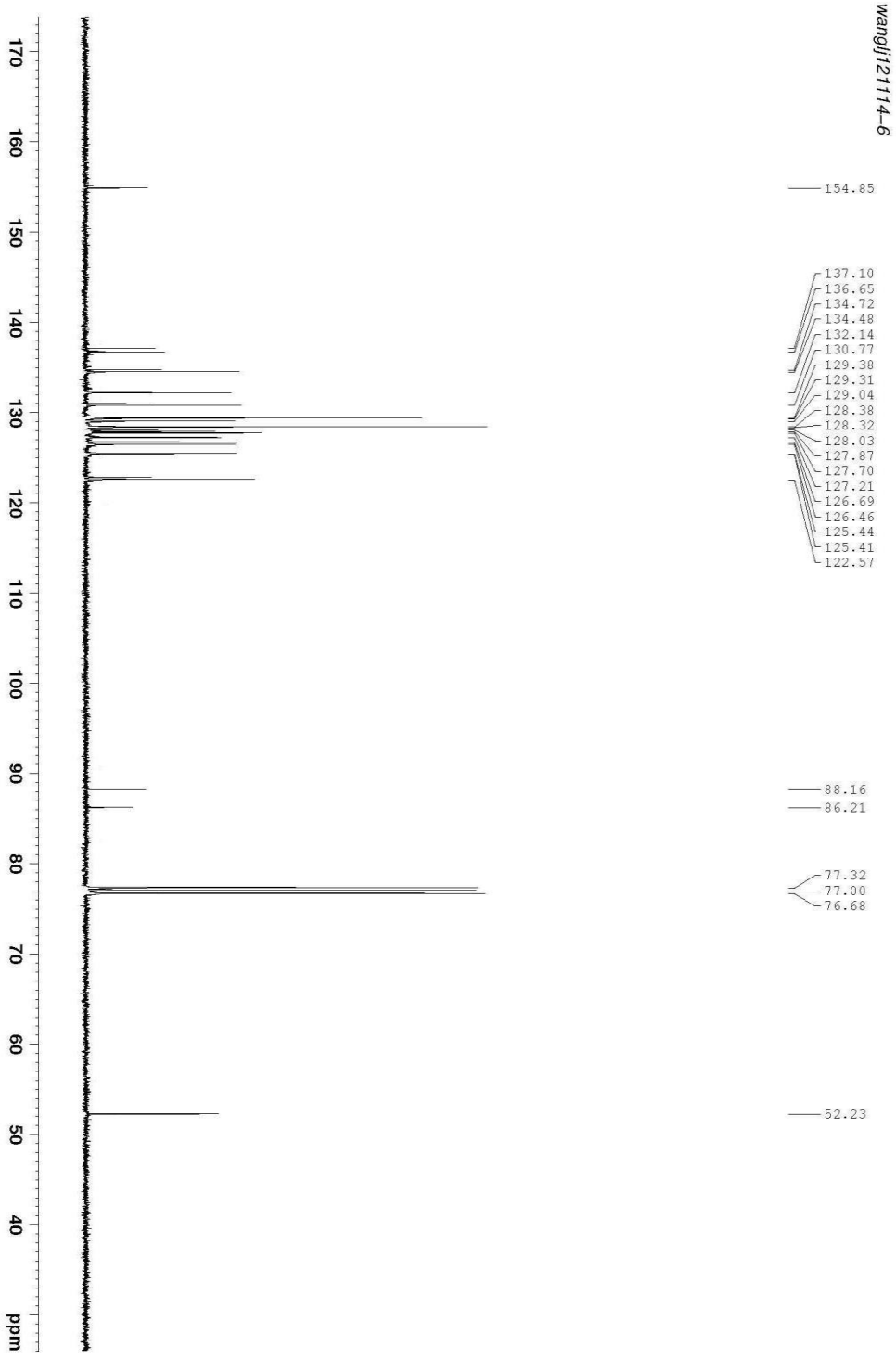

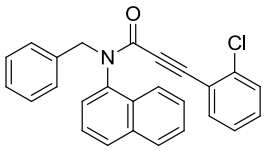

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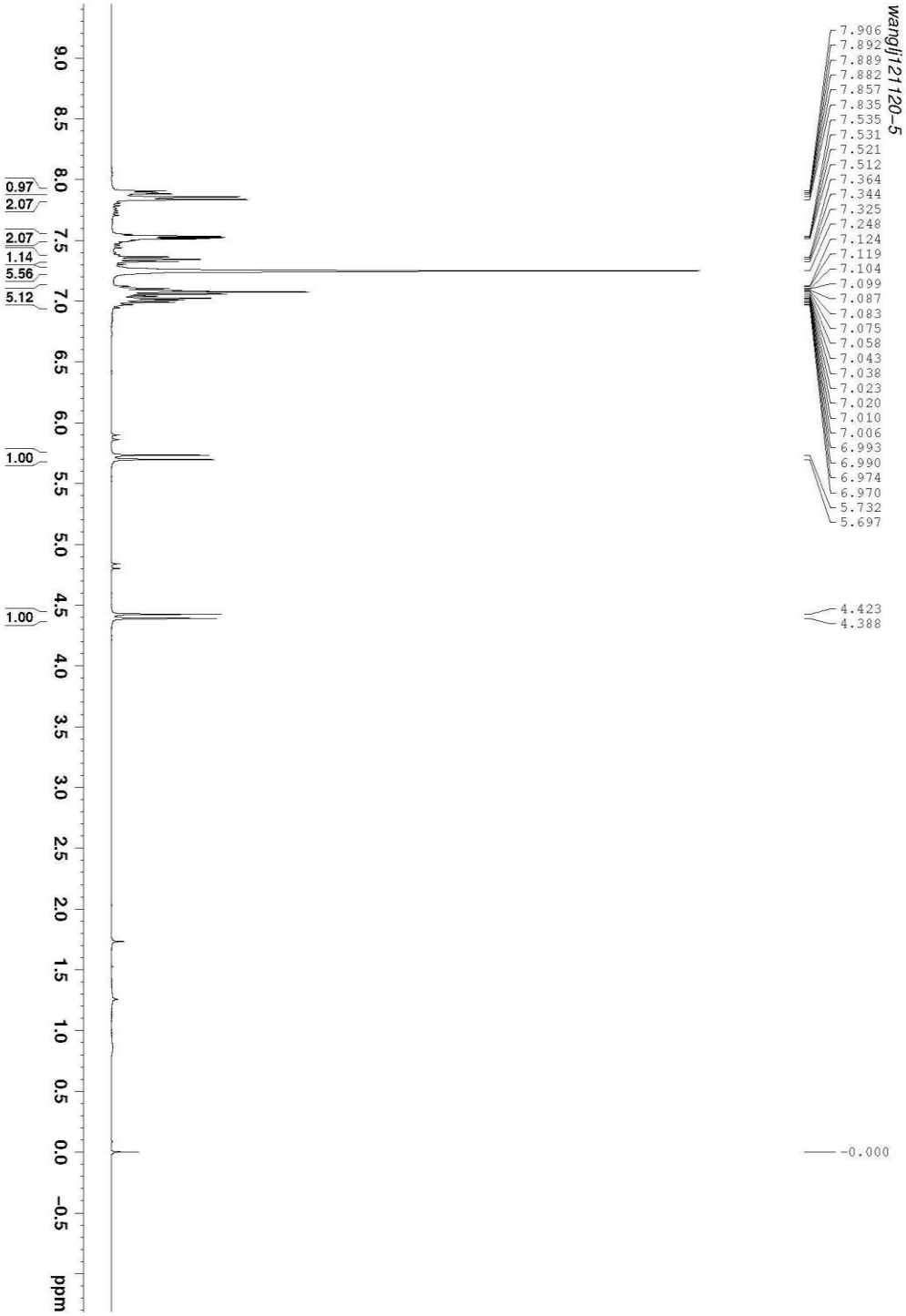

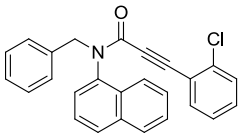

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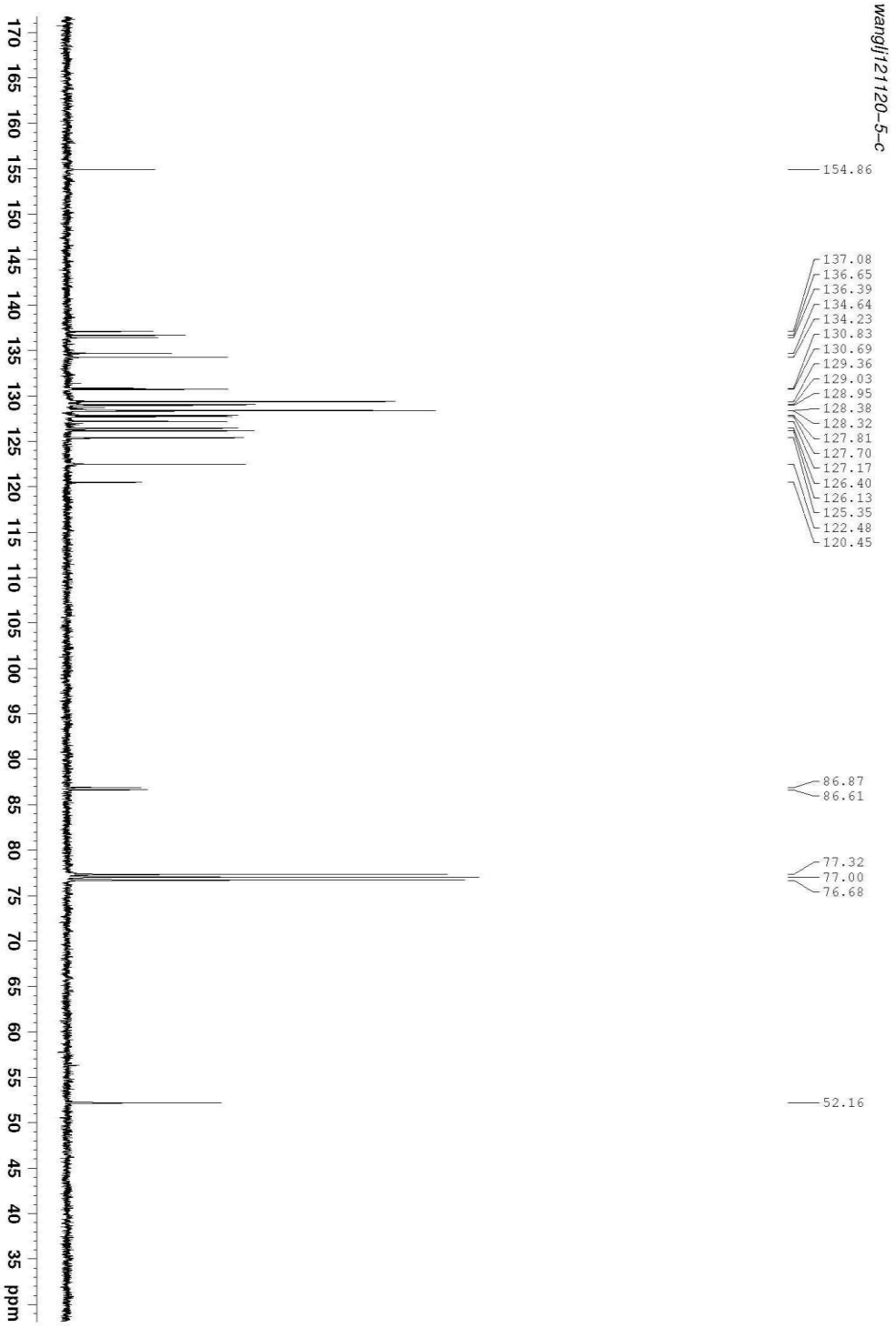

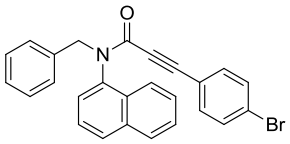

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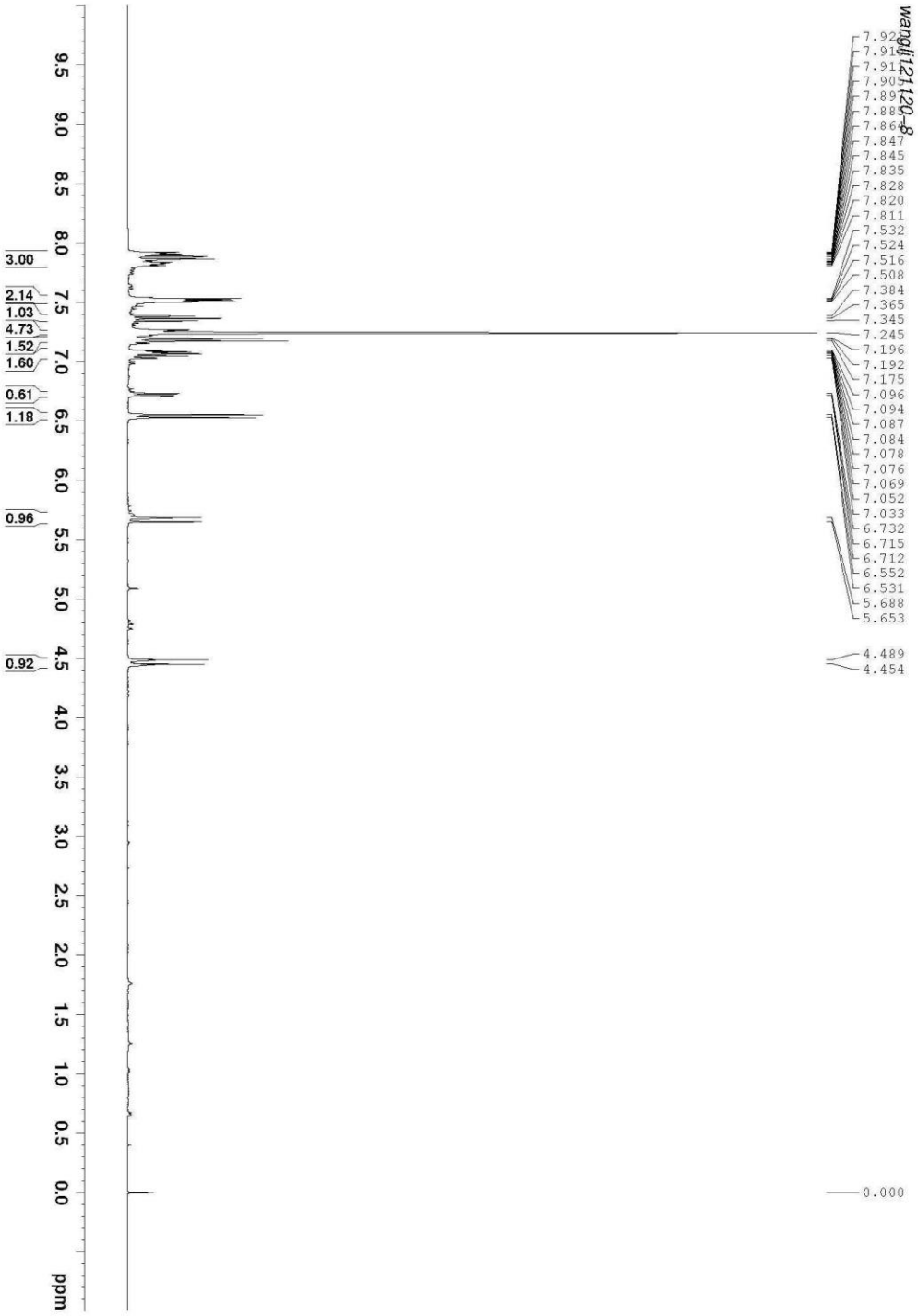

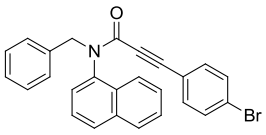

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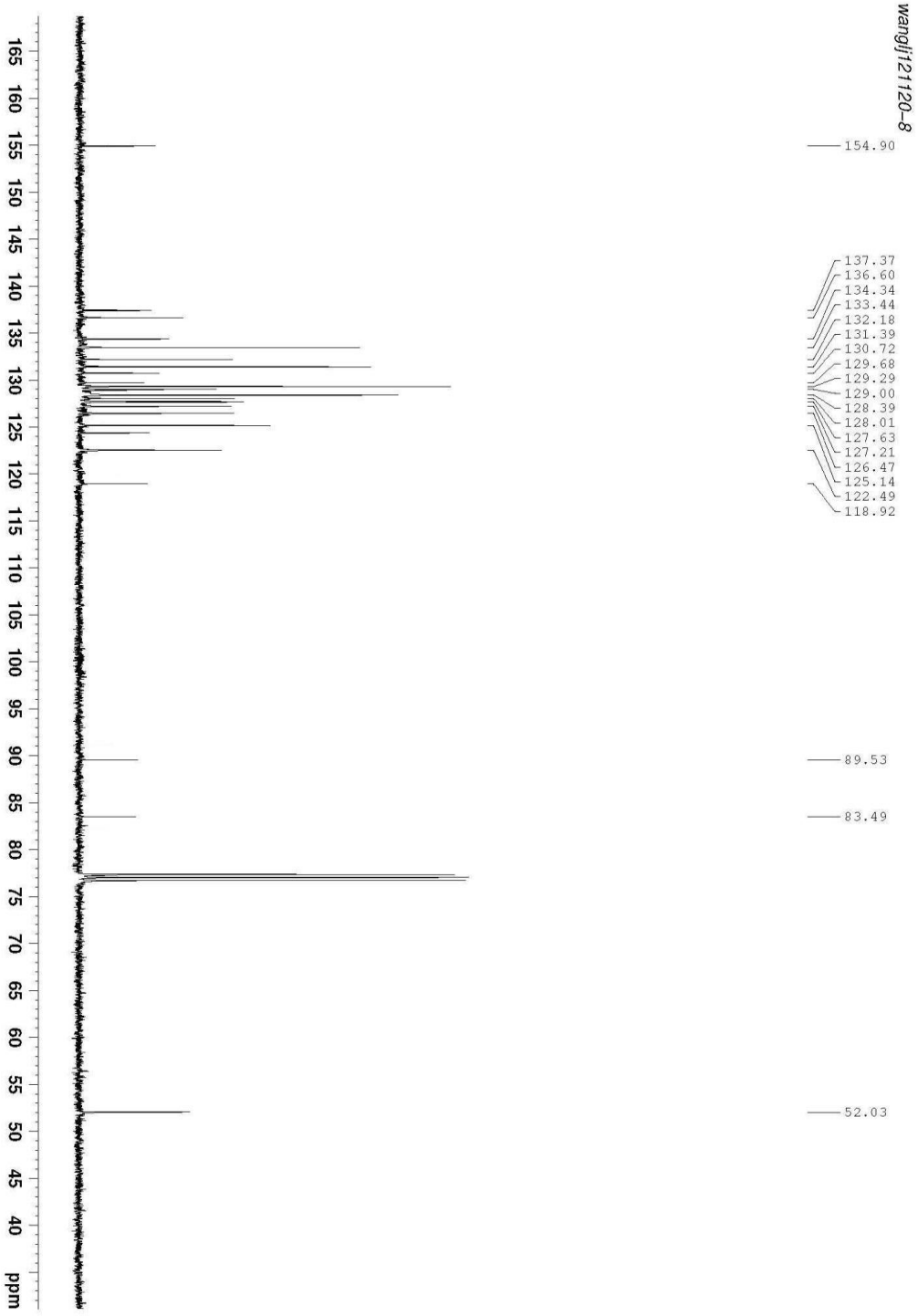

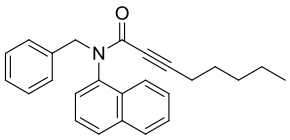

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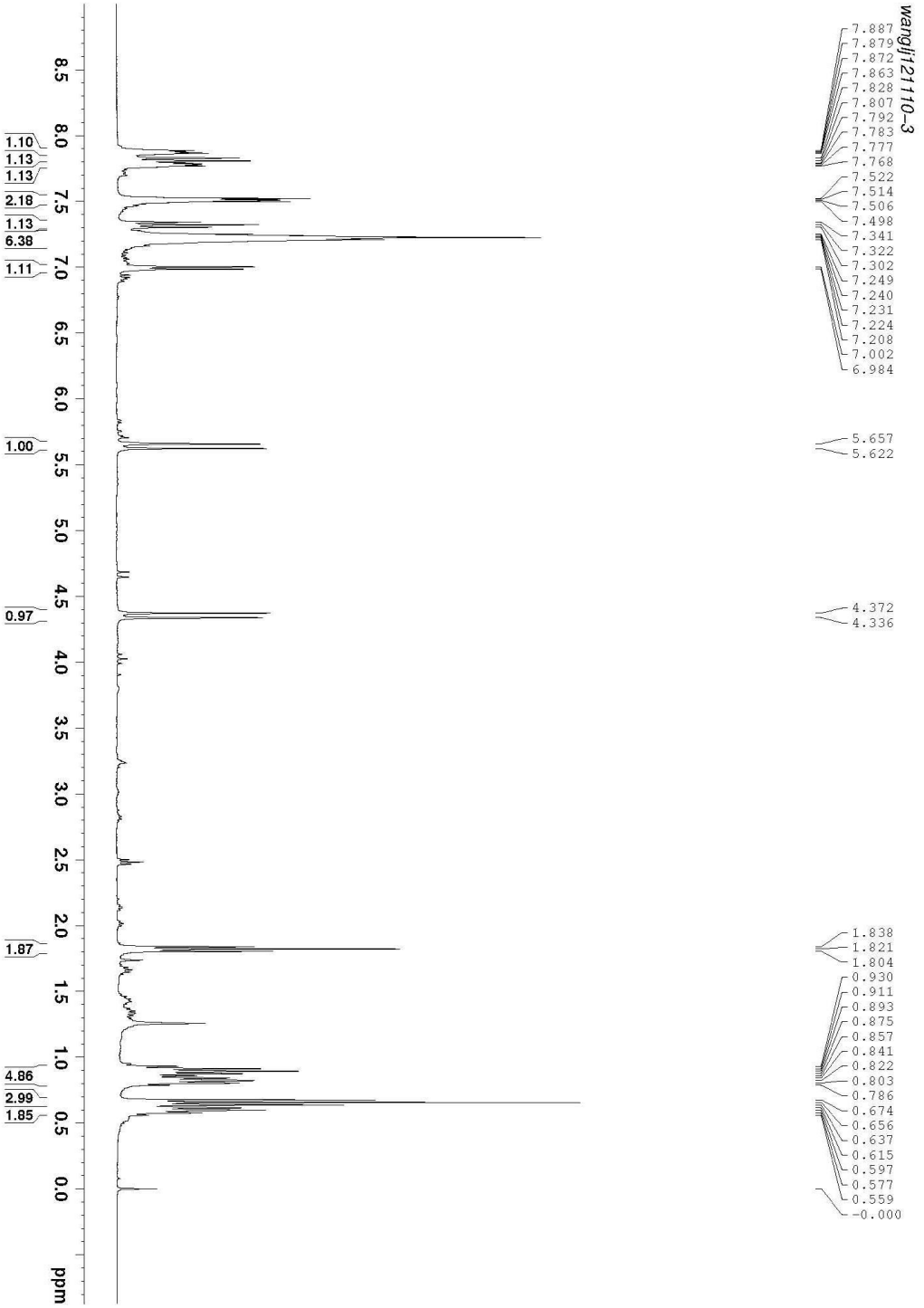
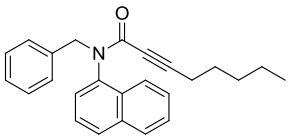
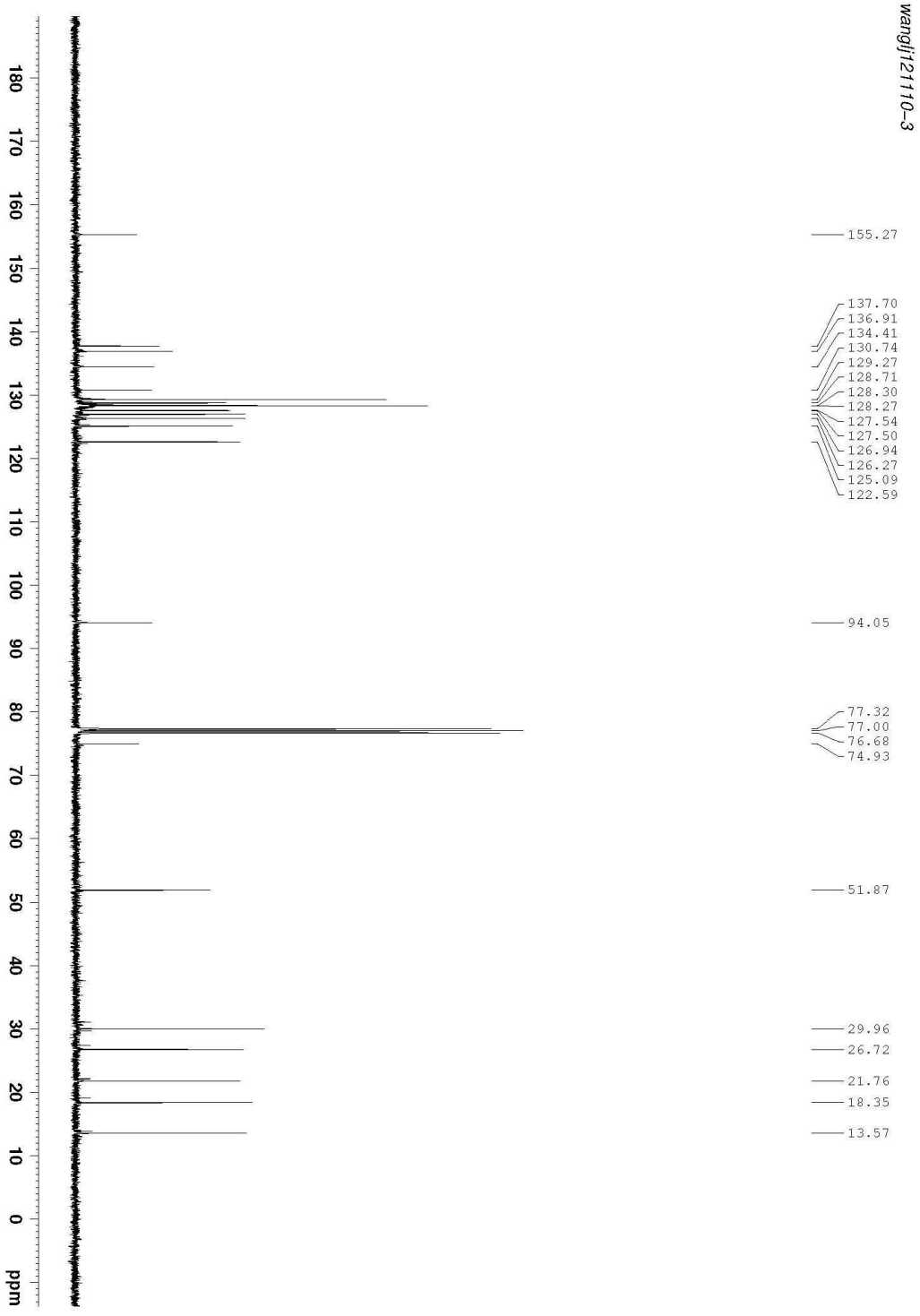
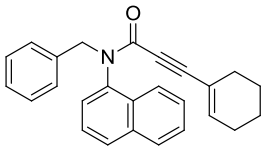




1g





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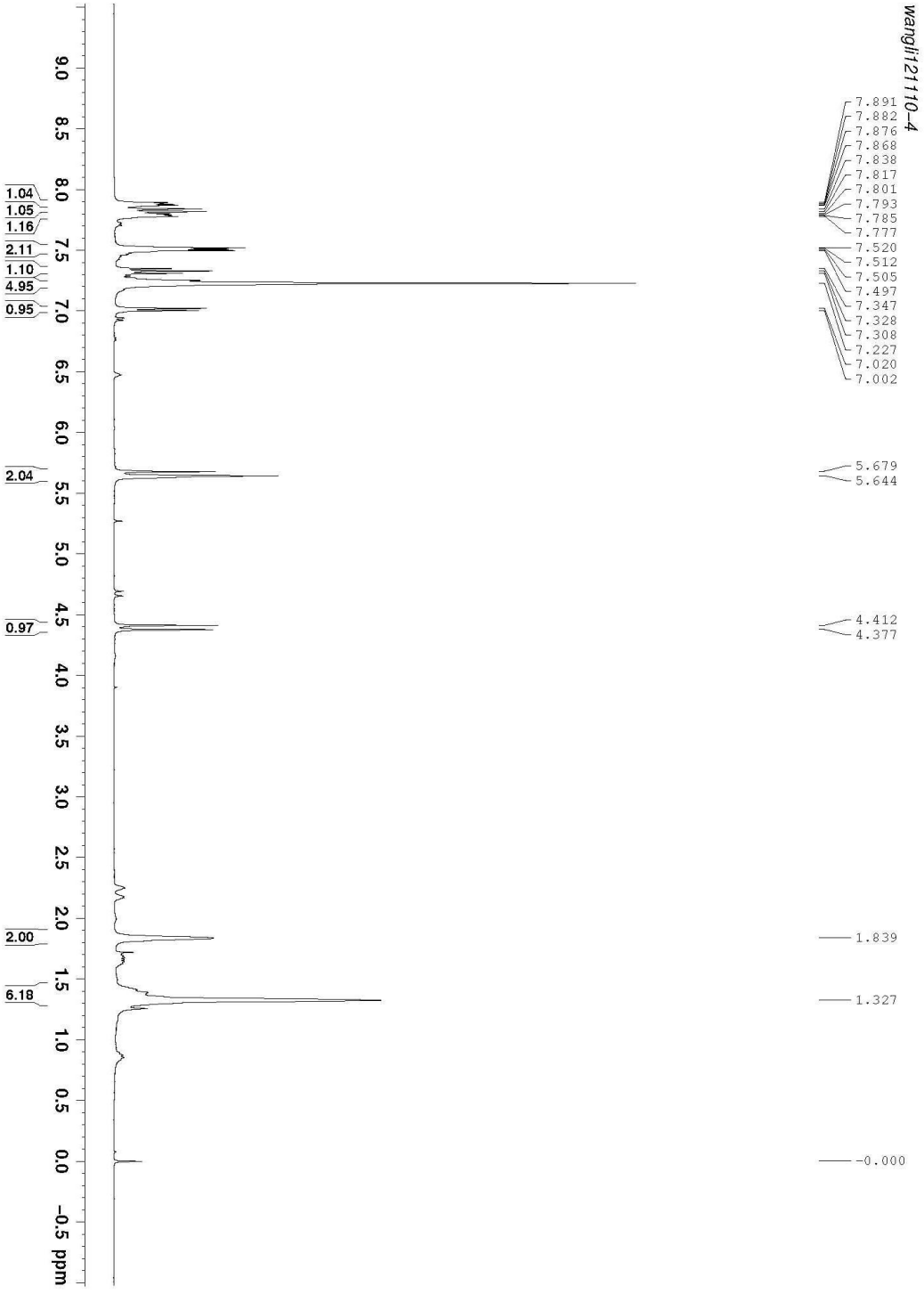

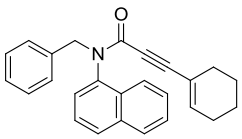

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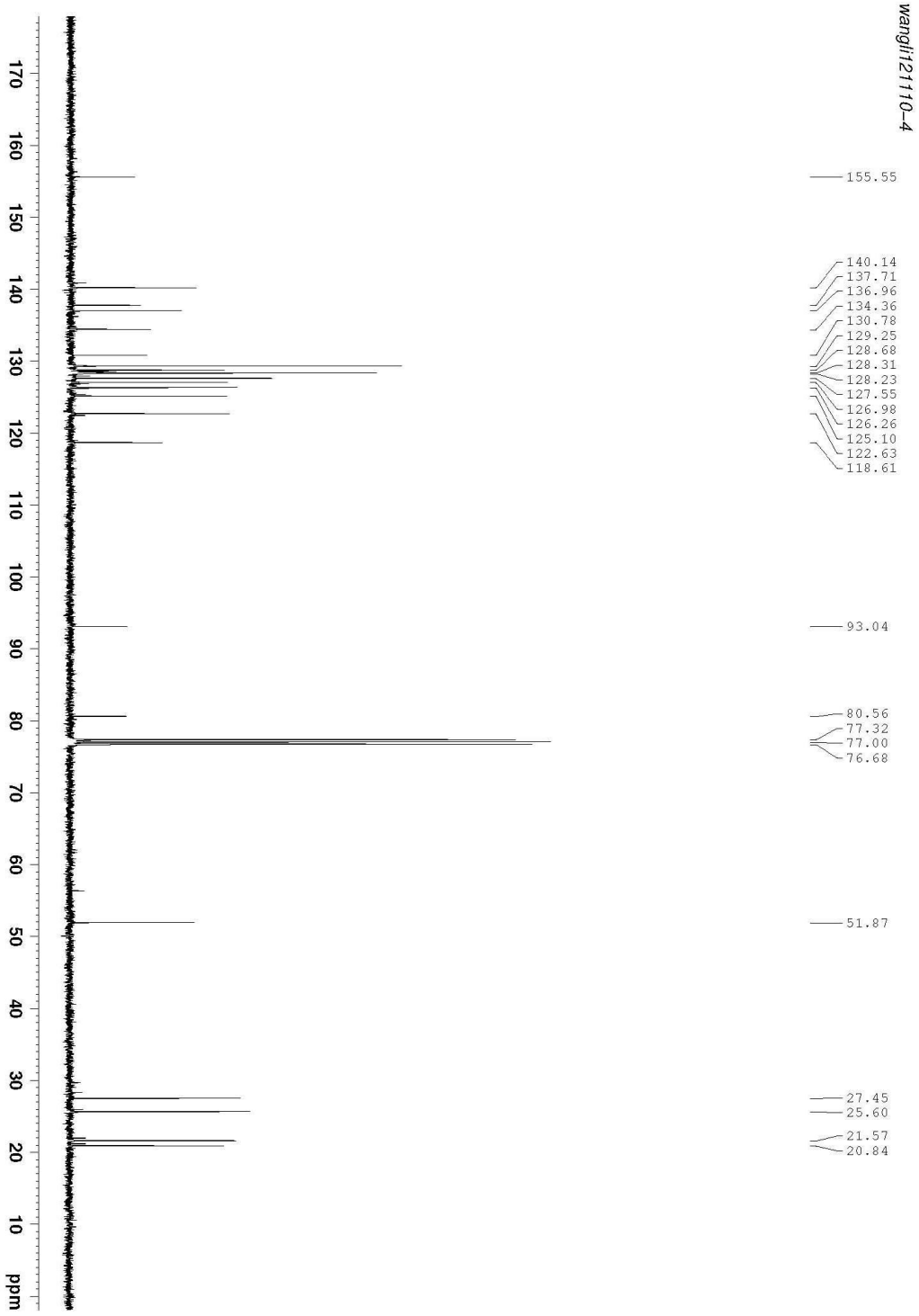

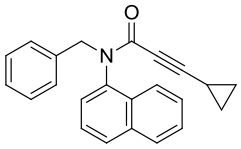

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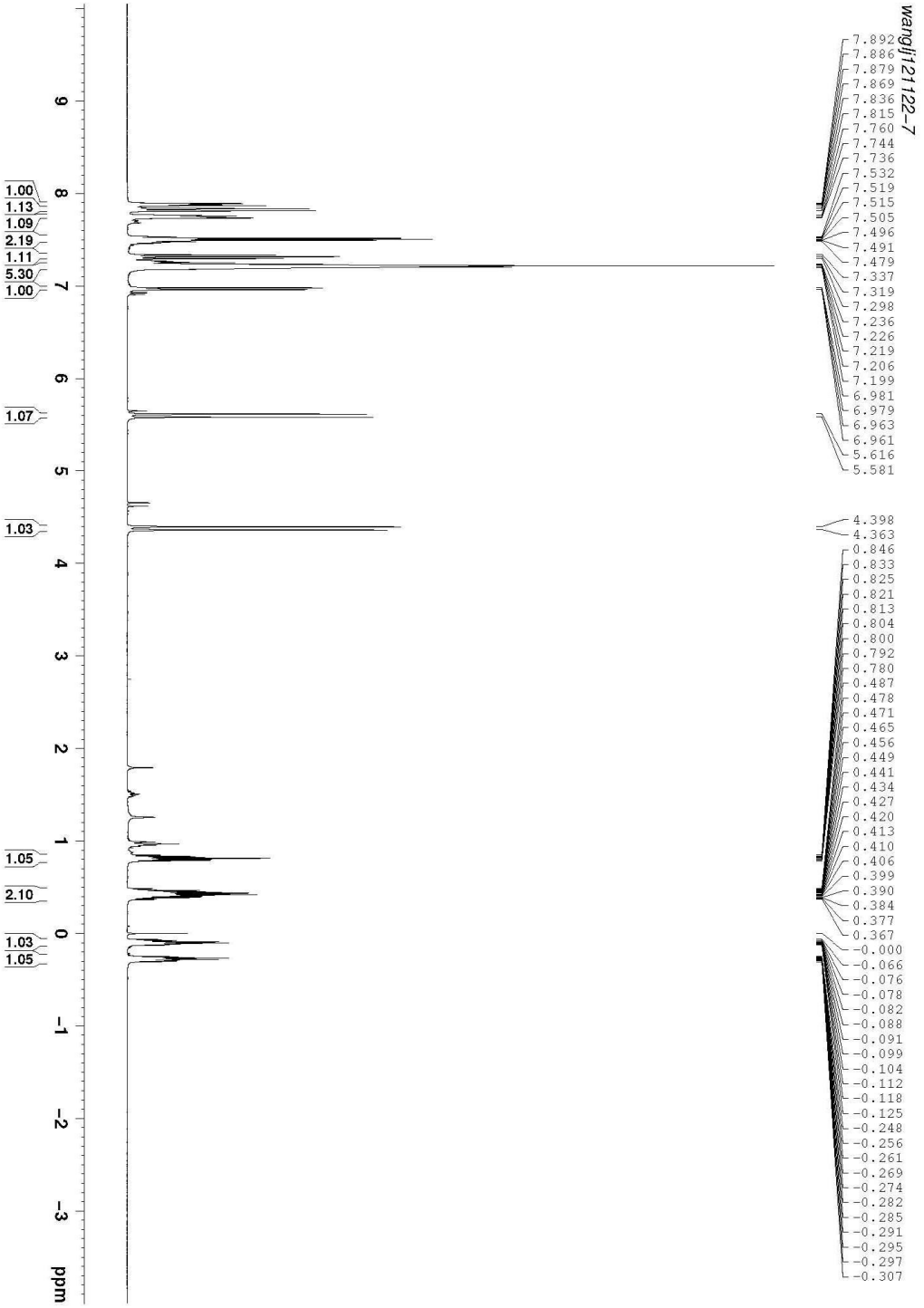

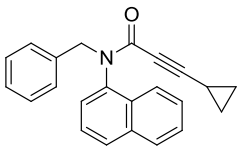

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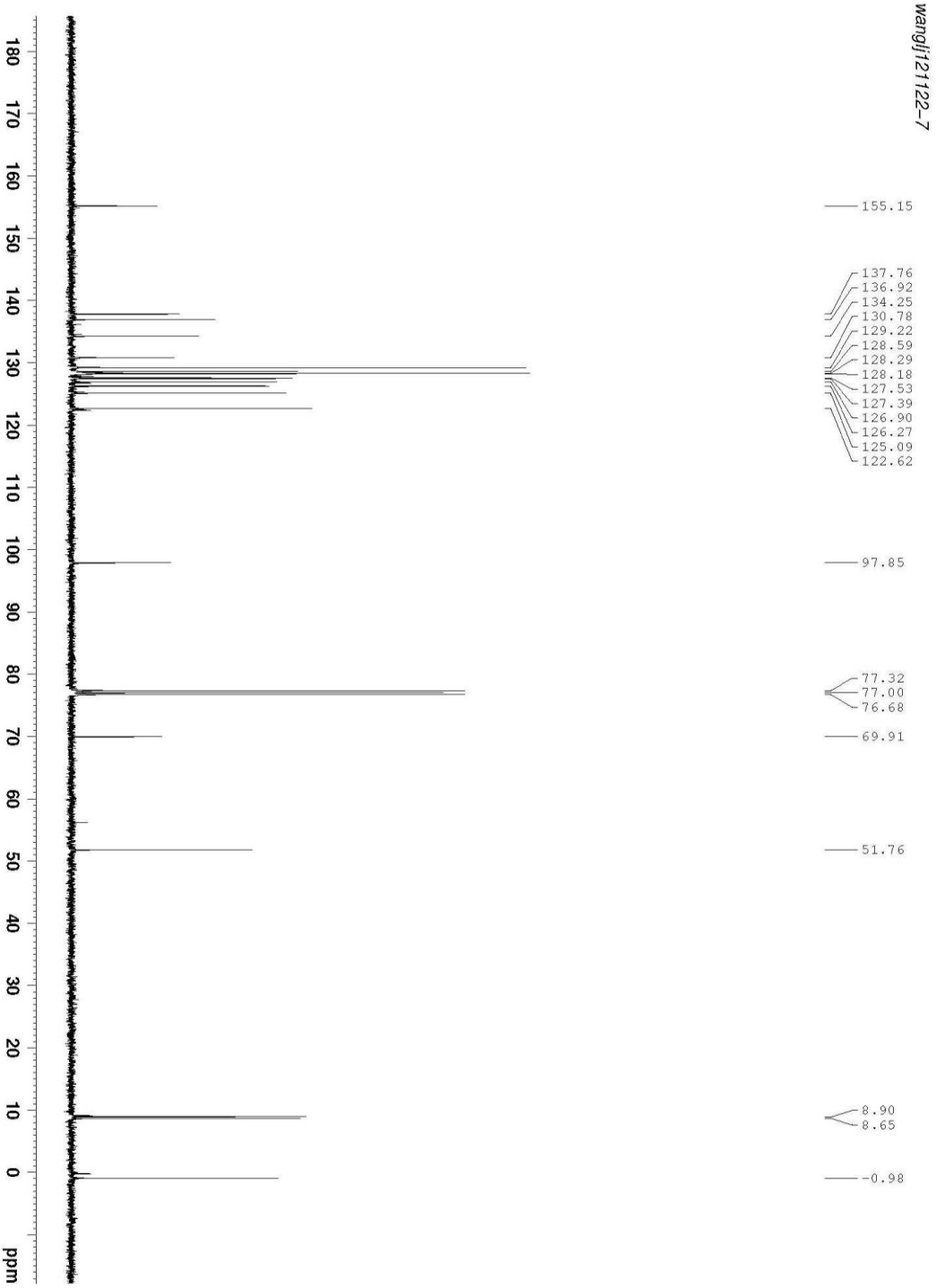

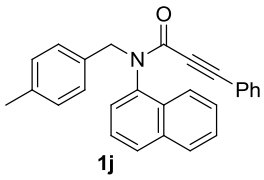

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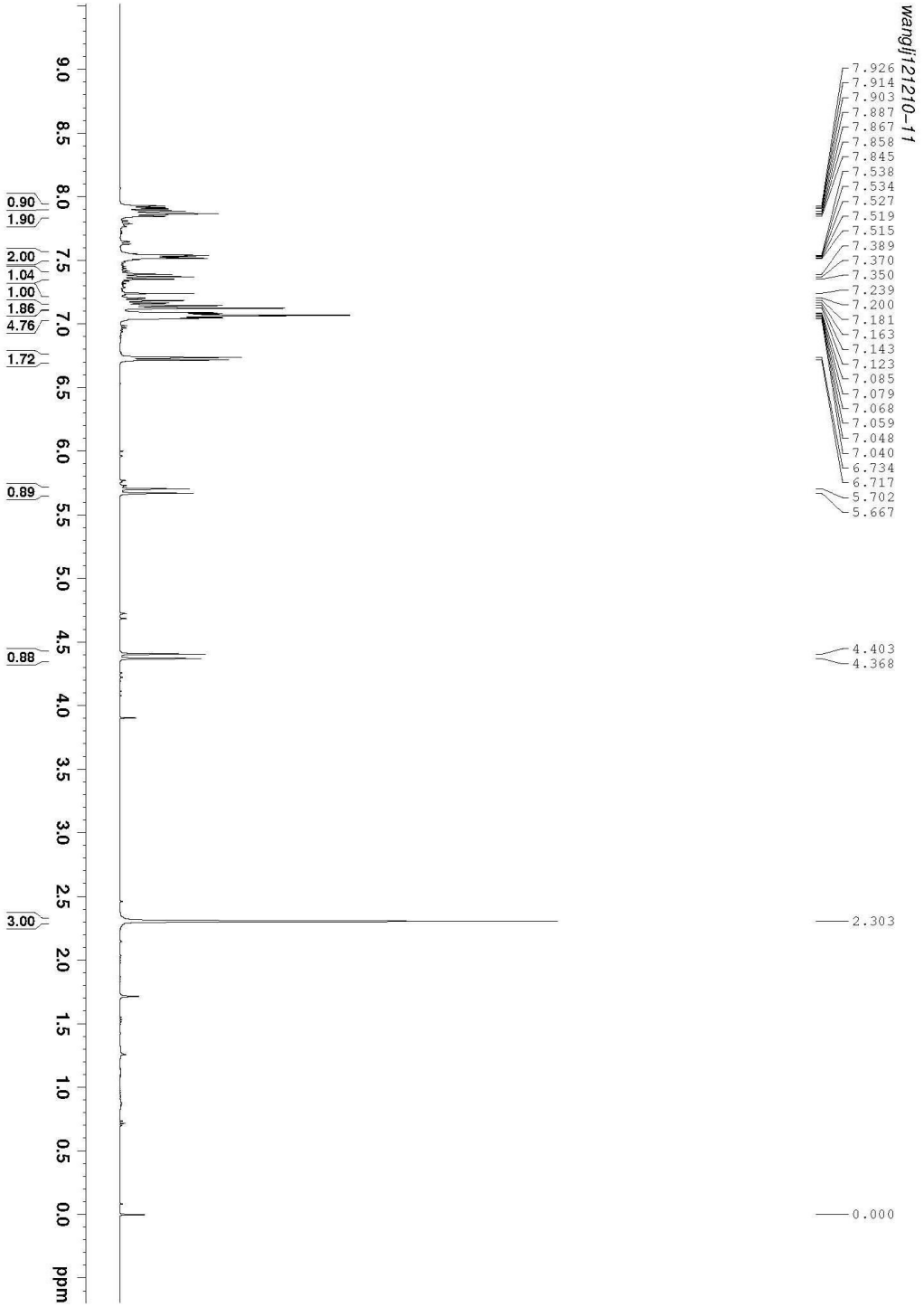

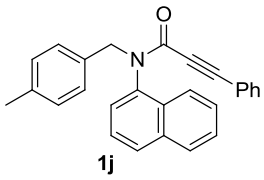

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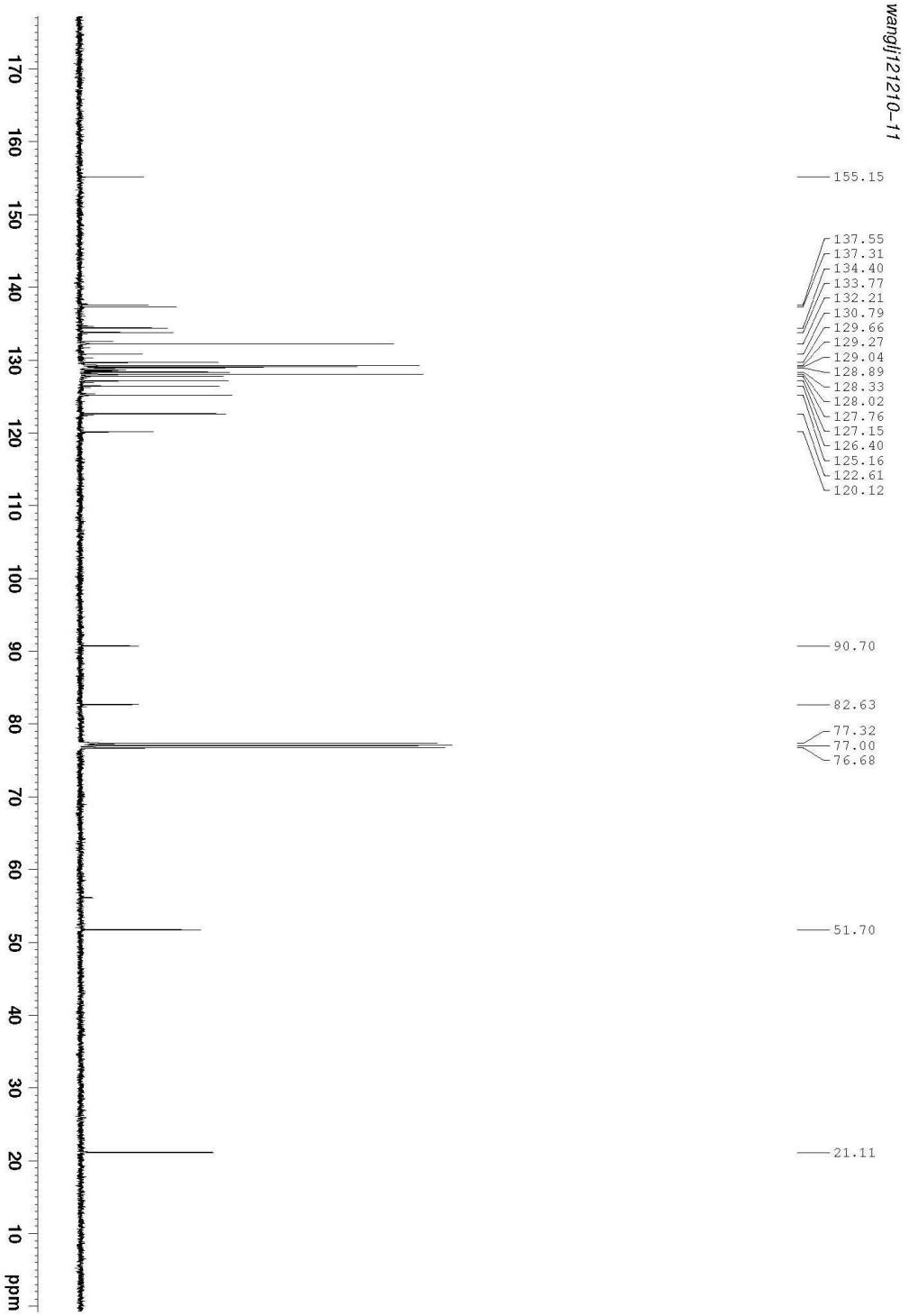

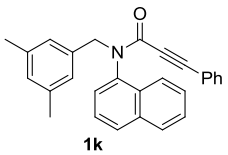

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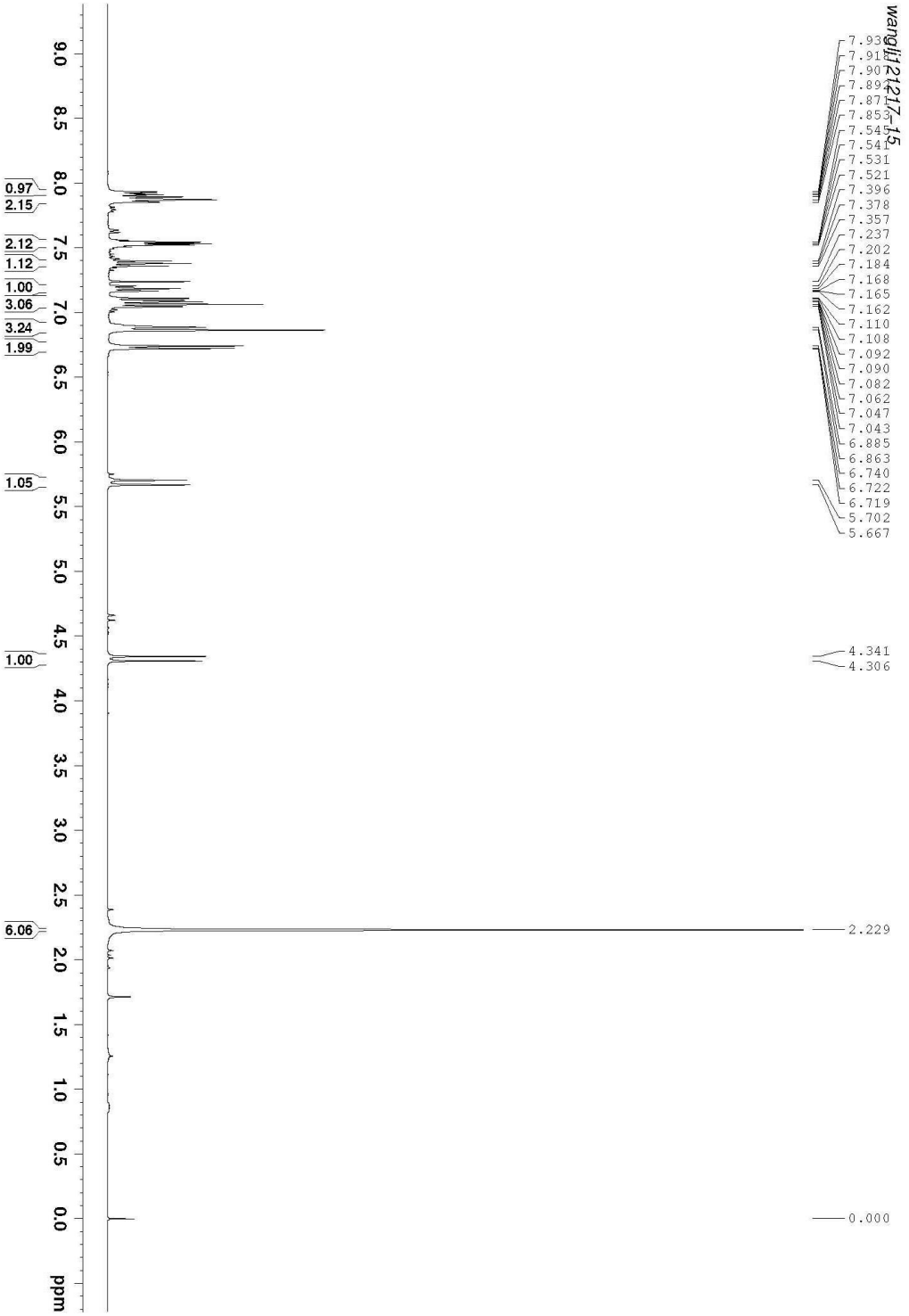

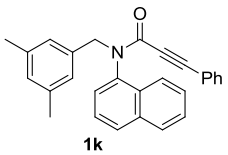

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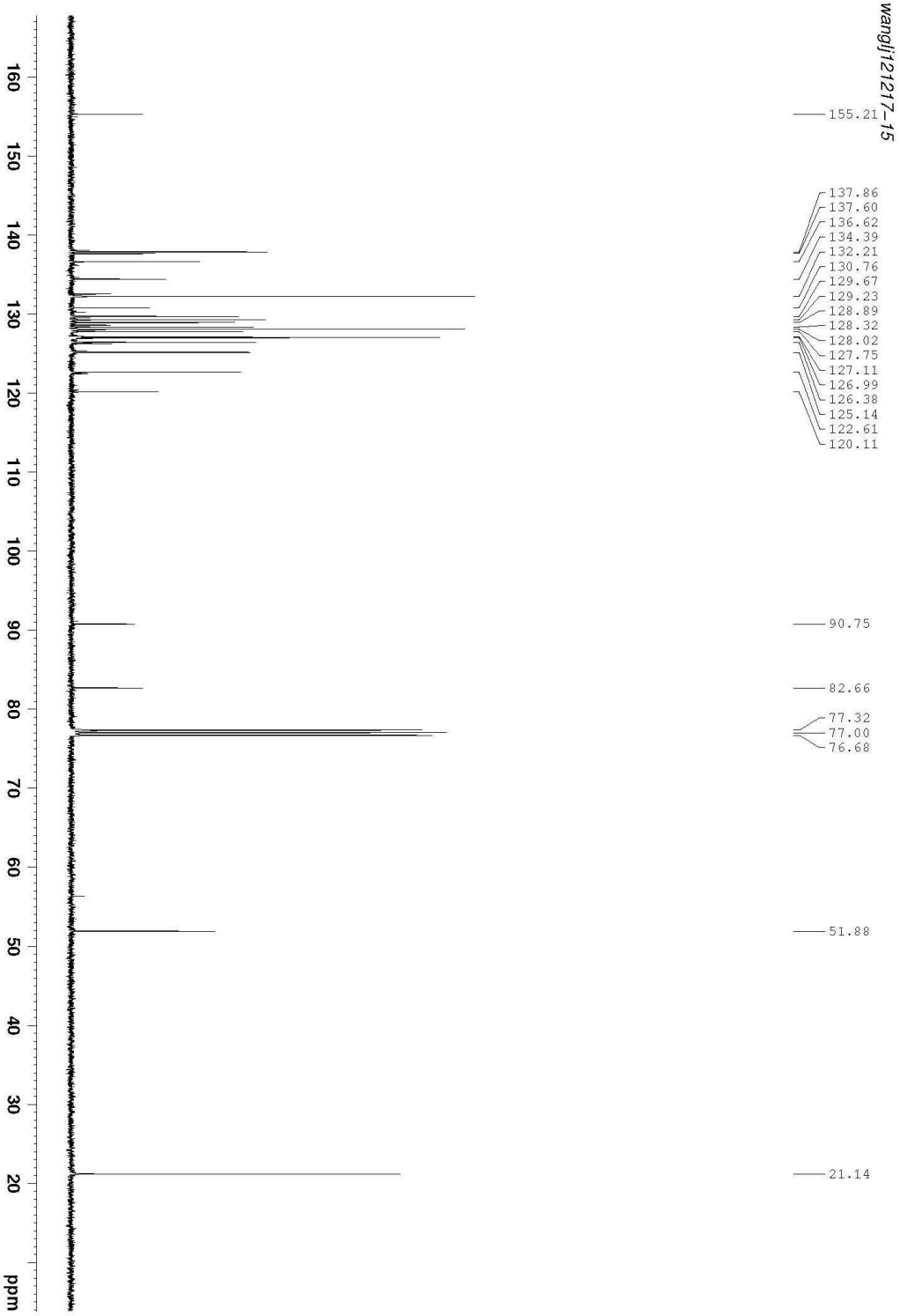

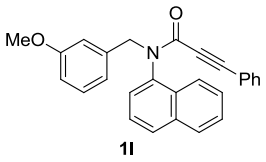

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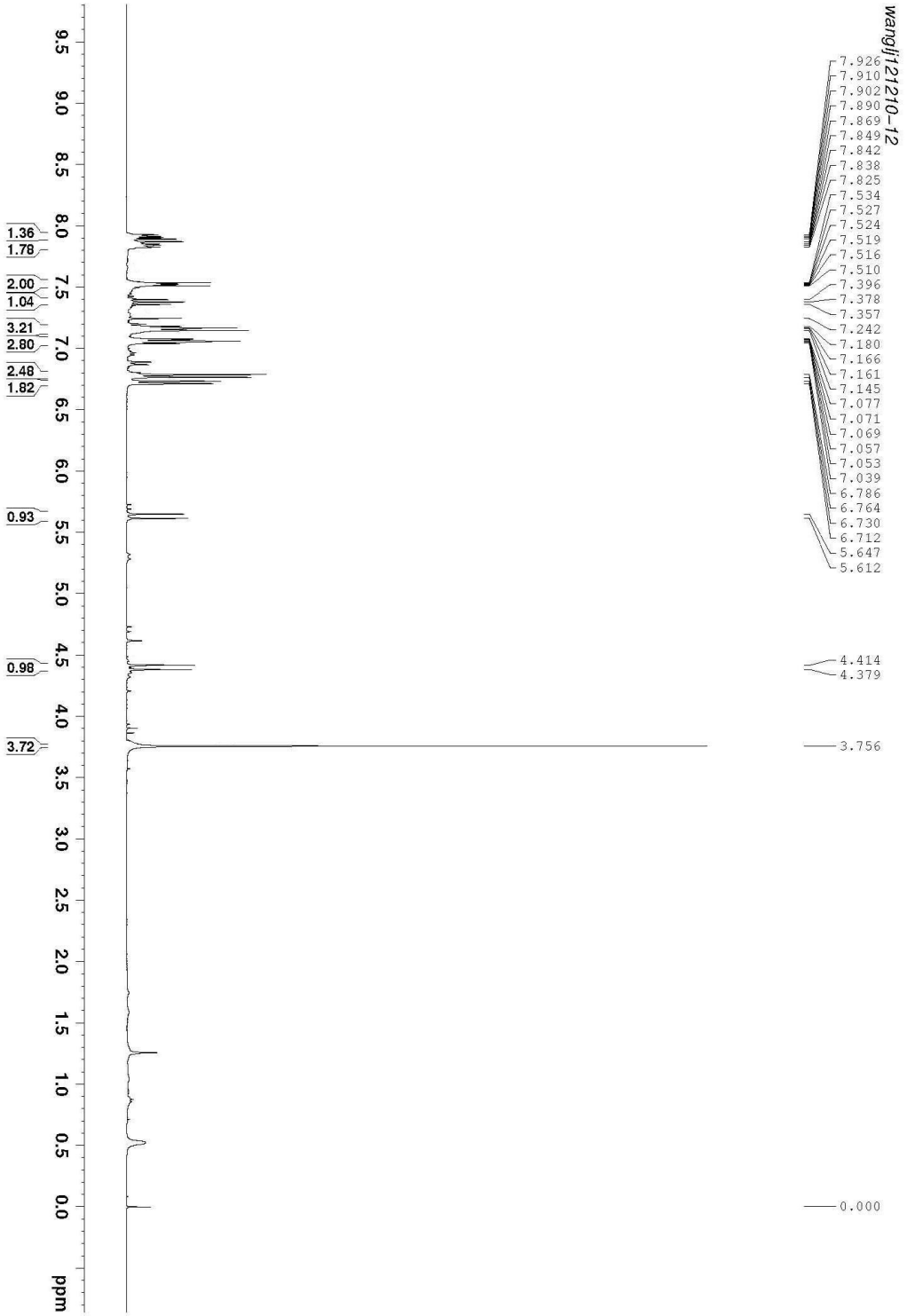

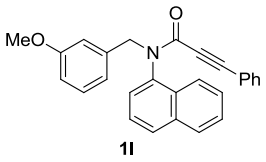

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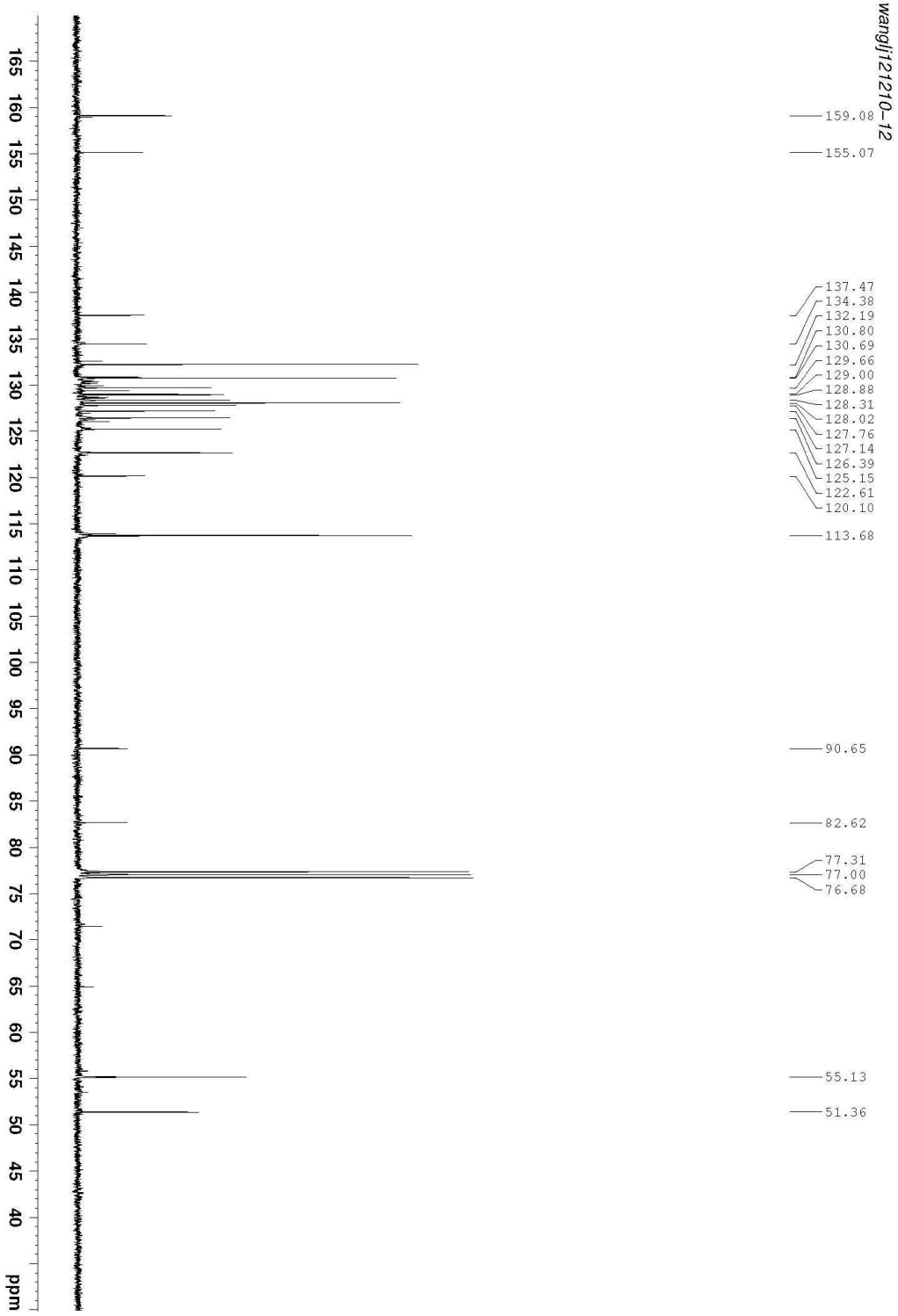

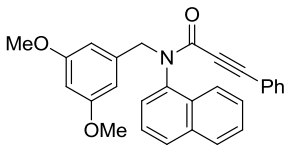

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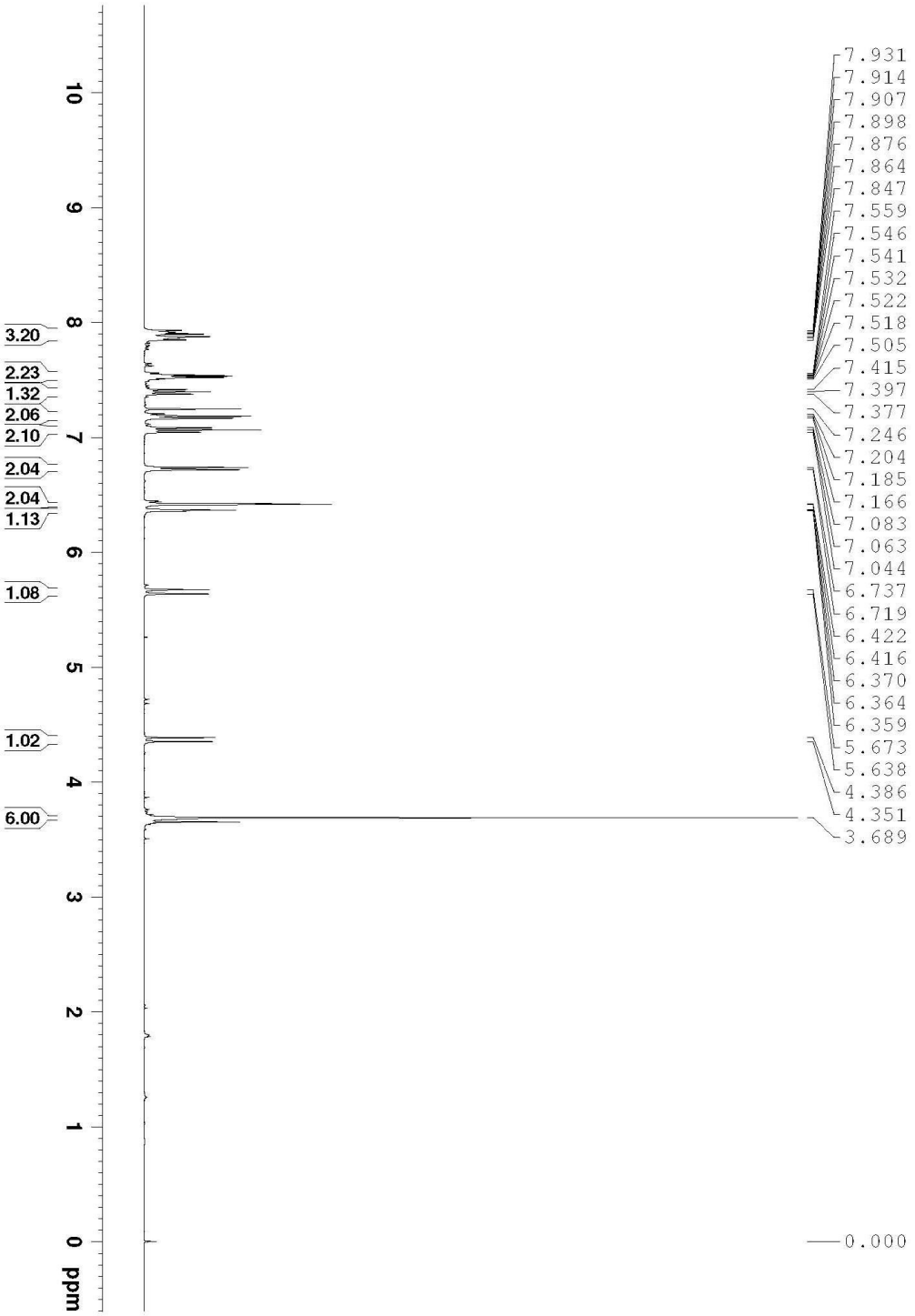

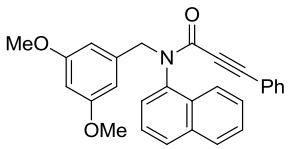

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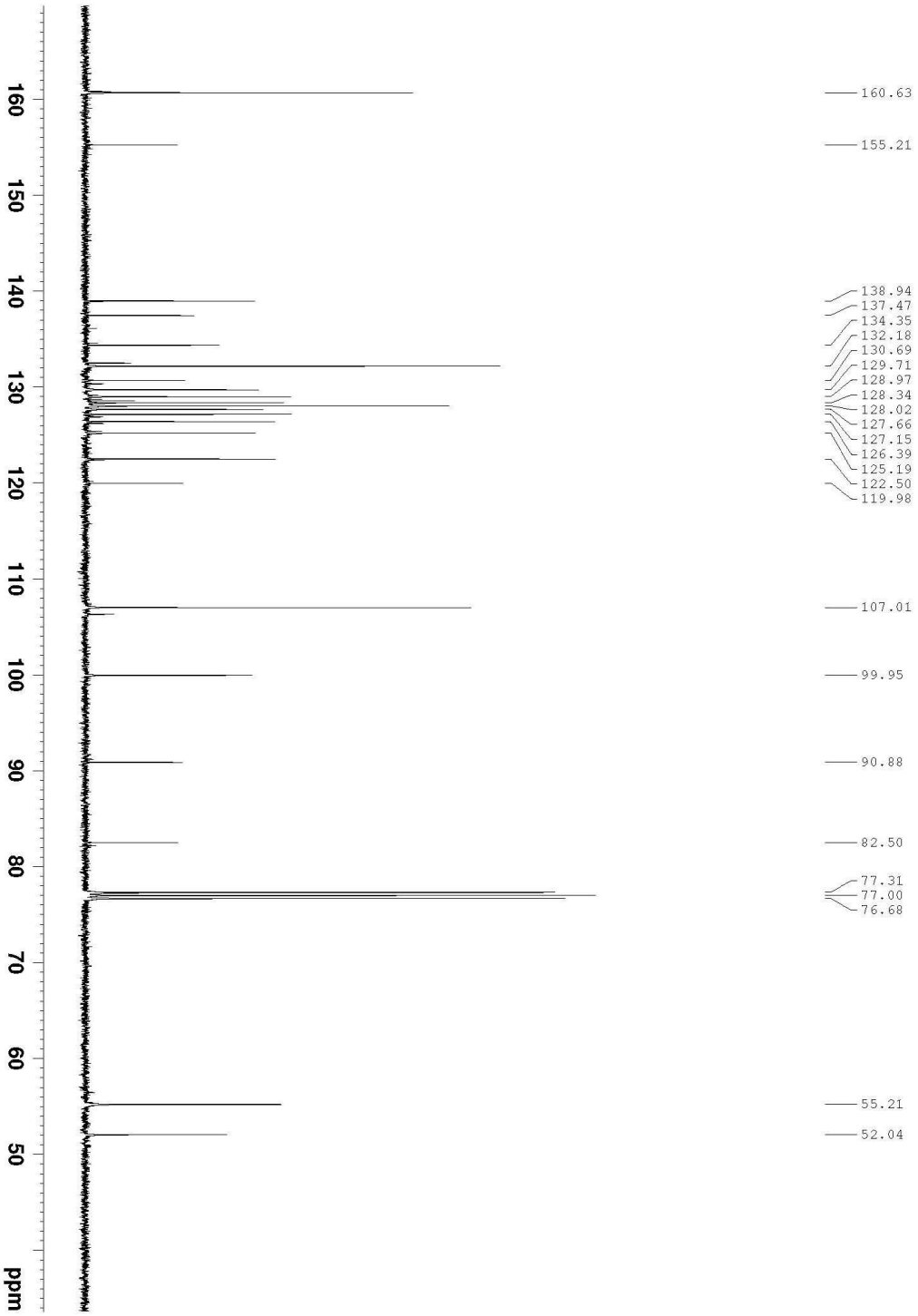

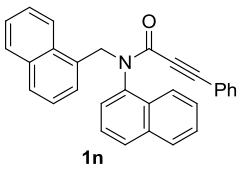

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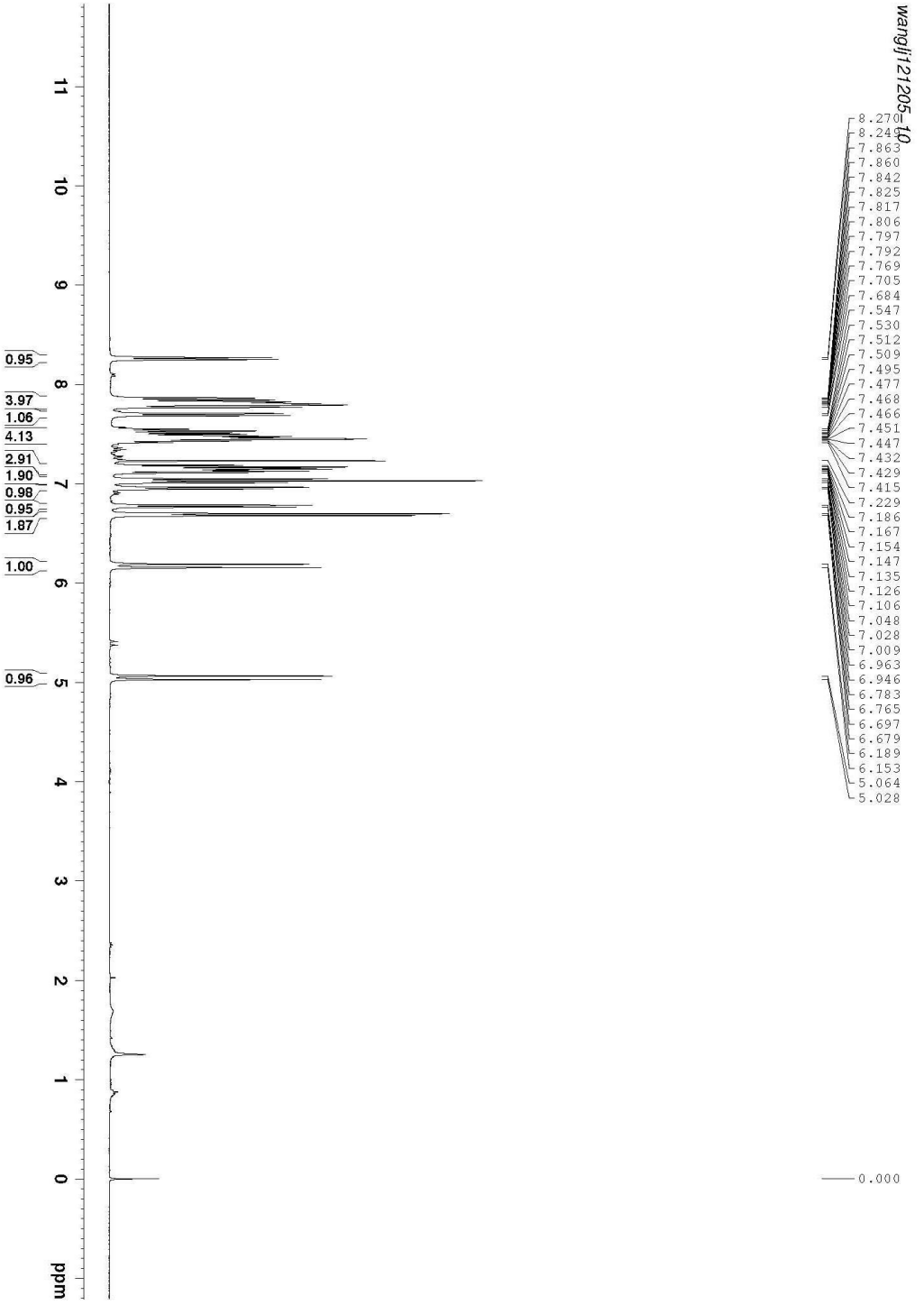

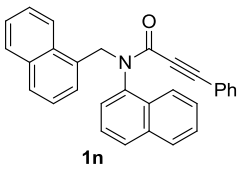
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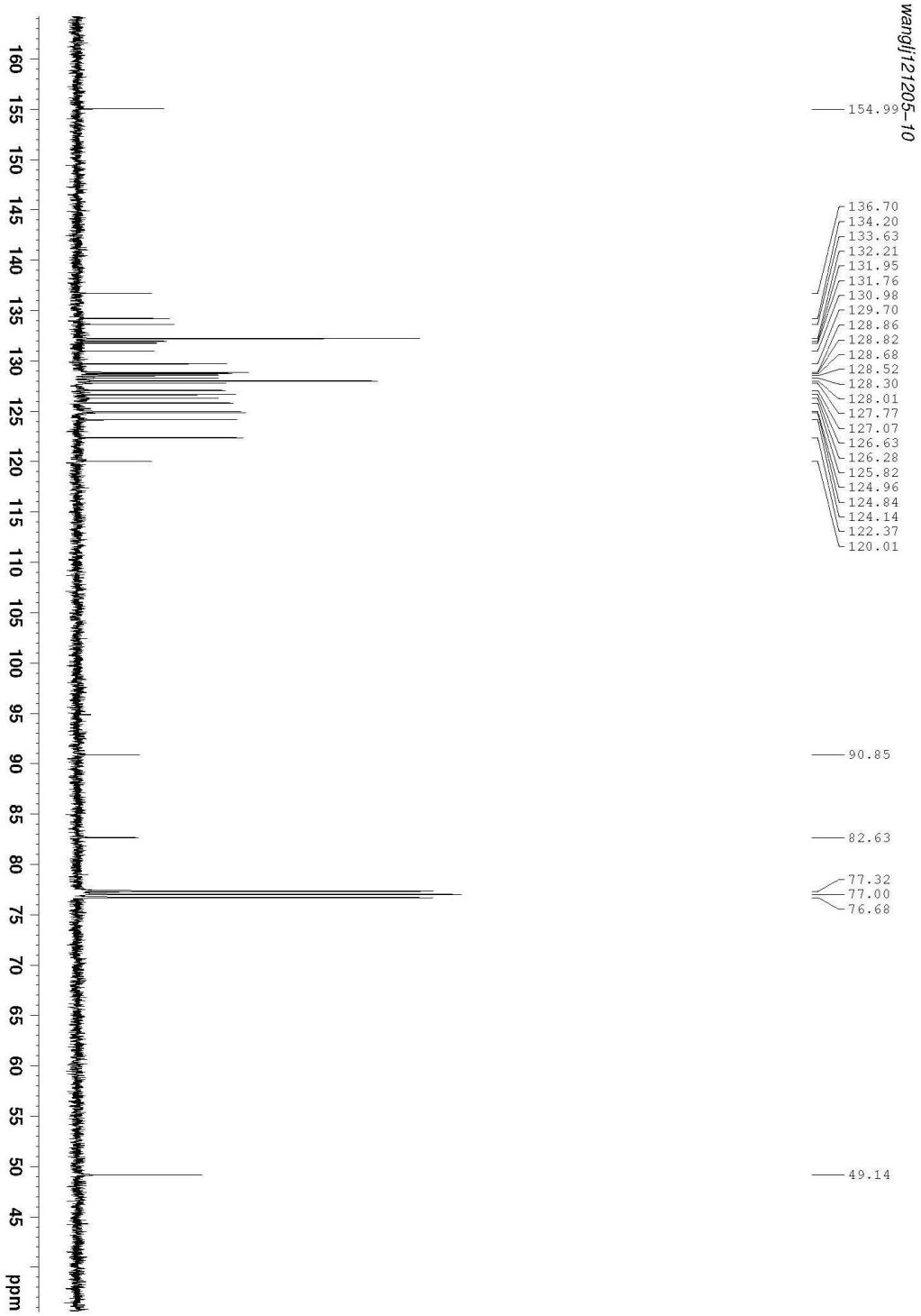
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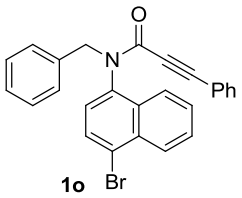

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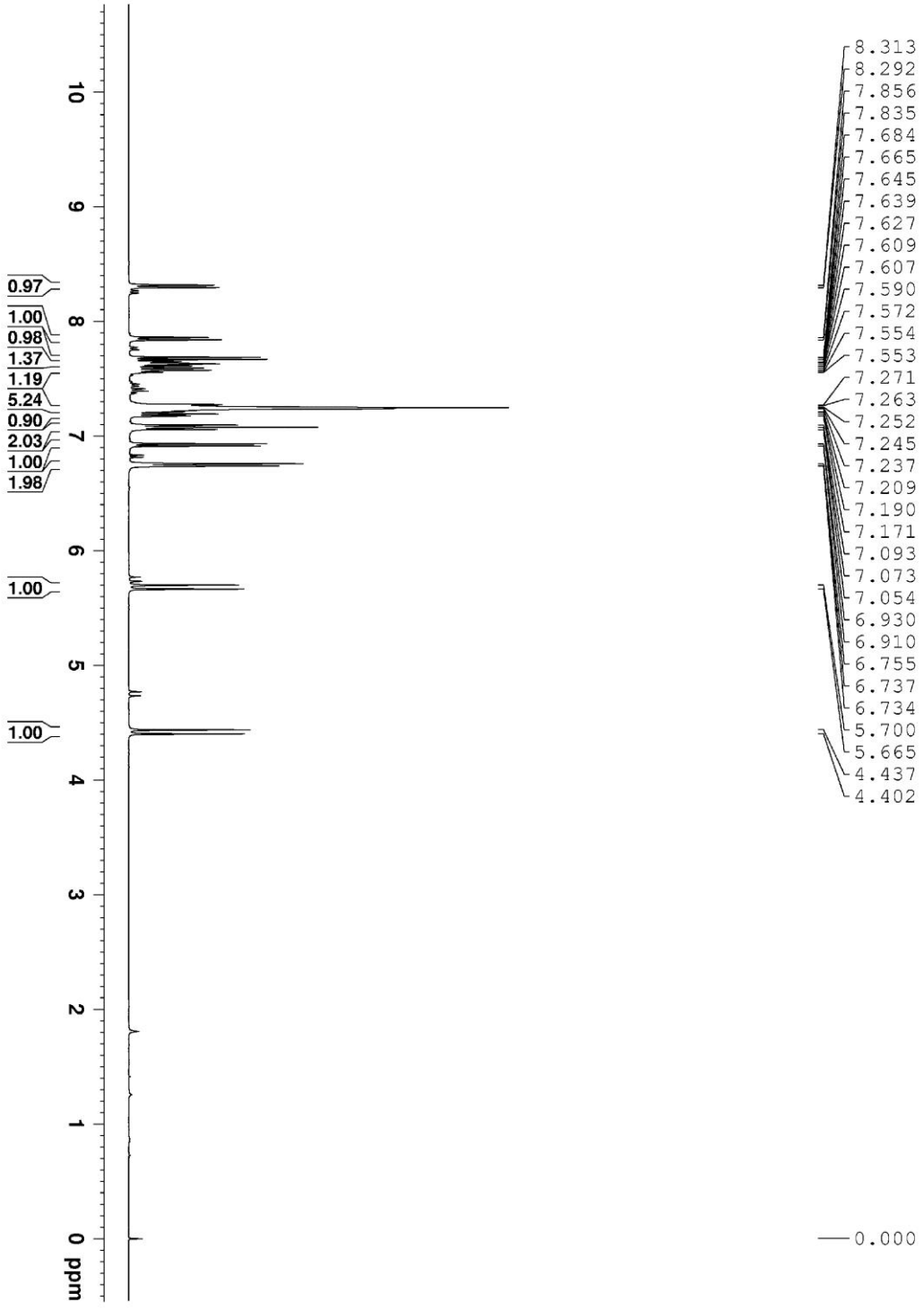

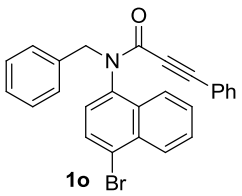

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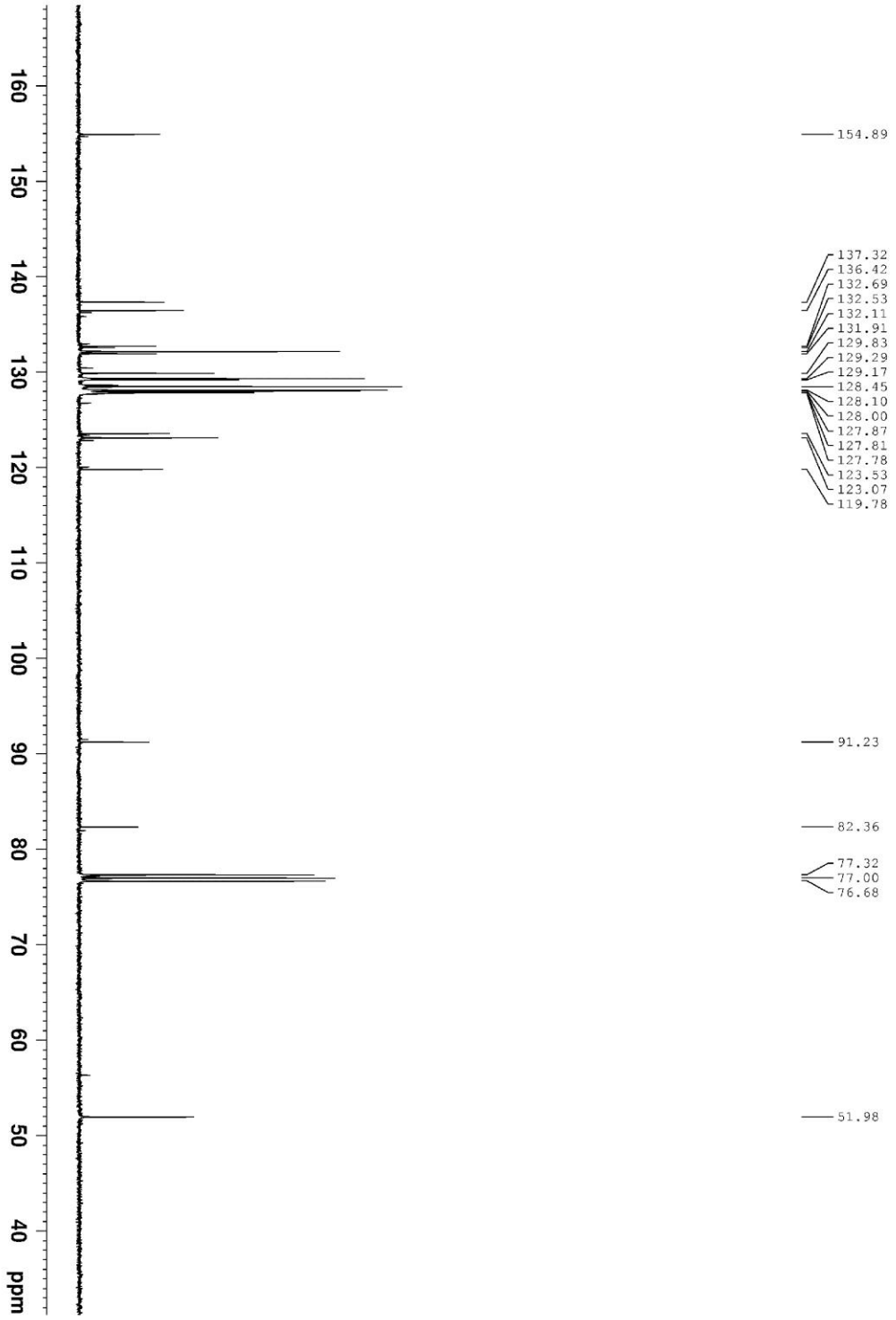

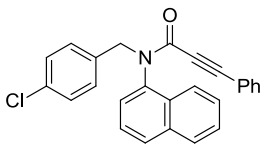

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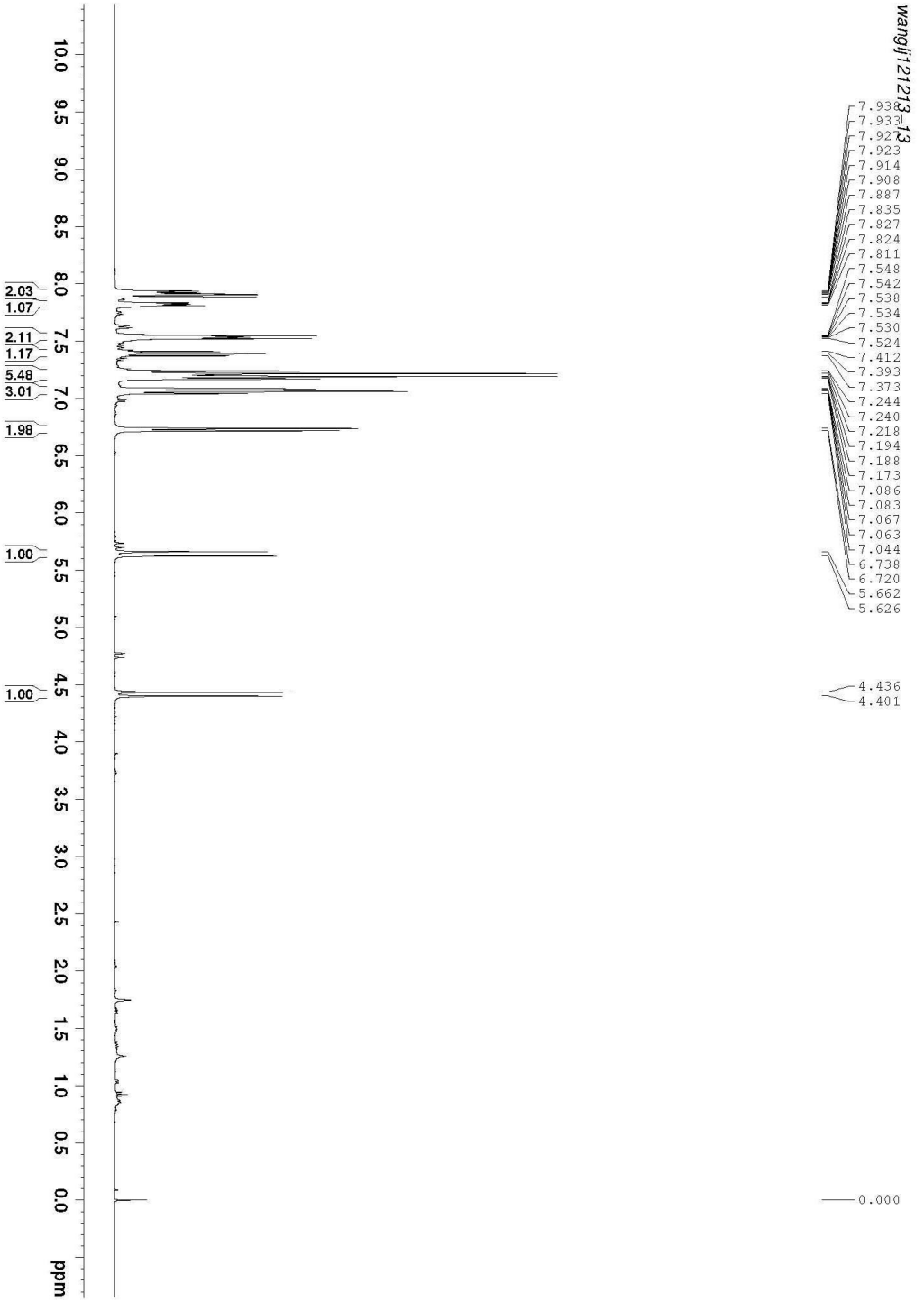

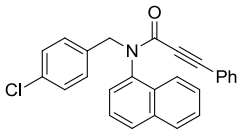

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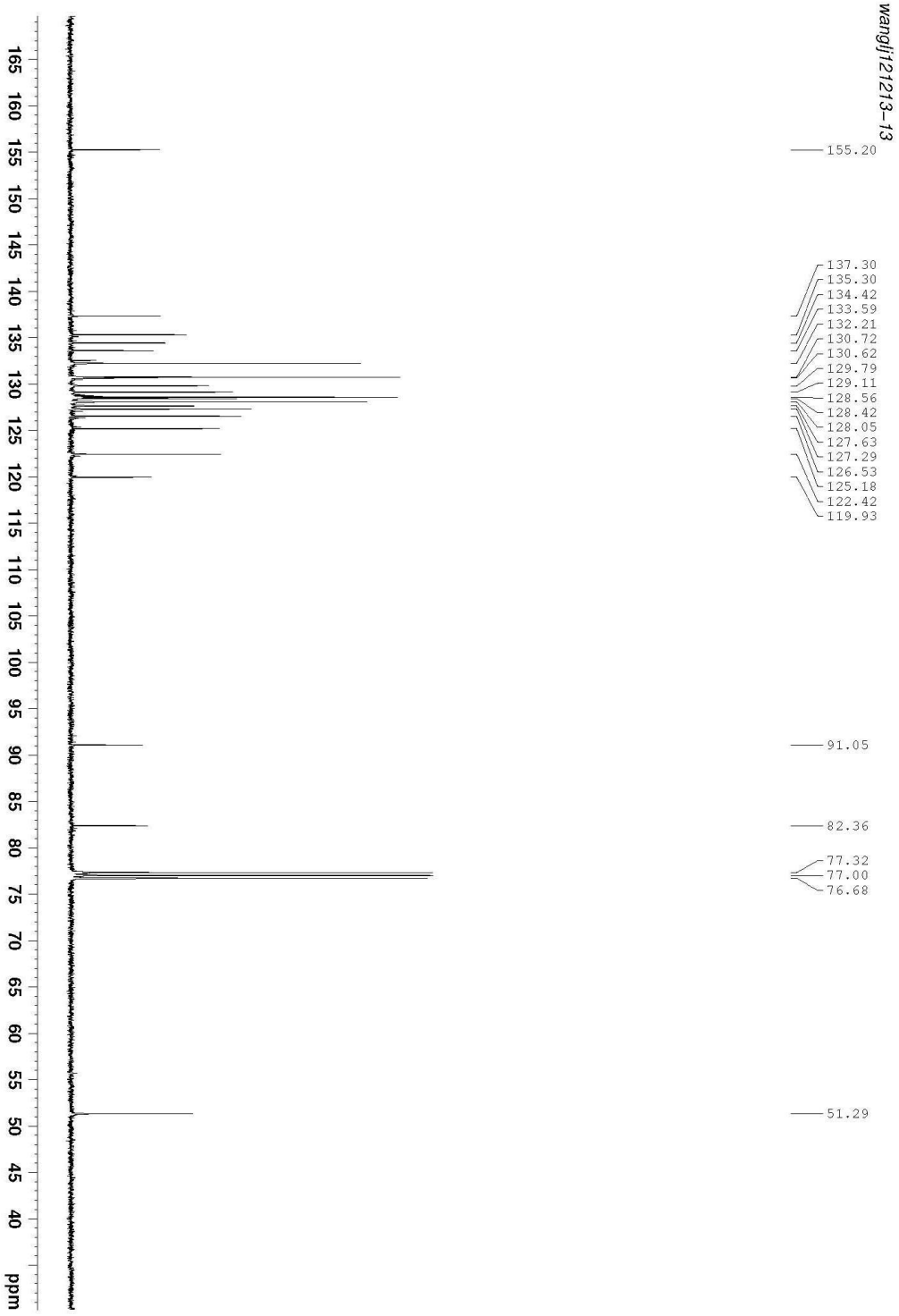

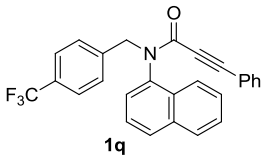

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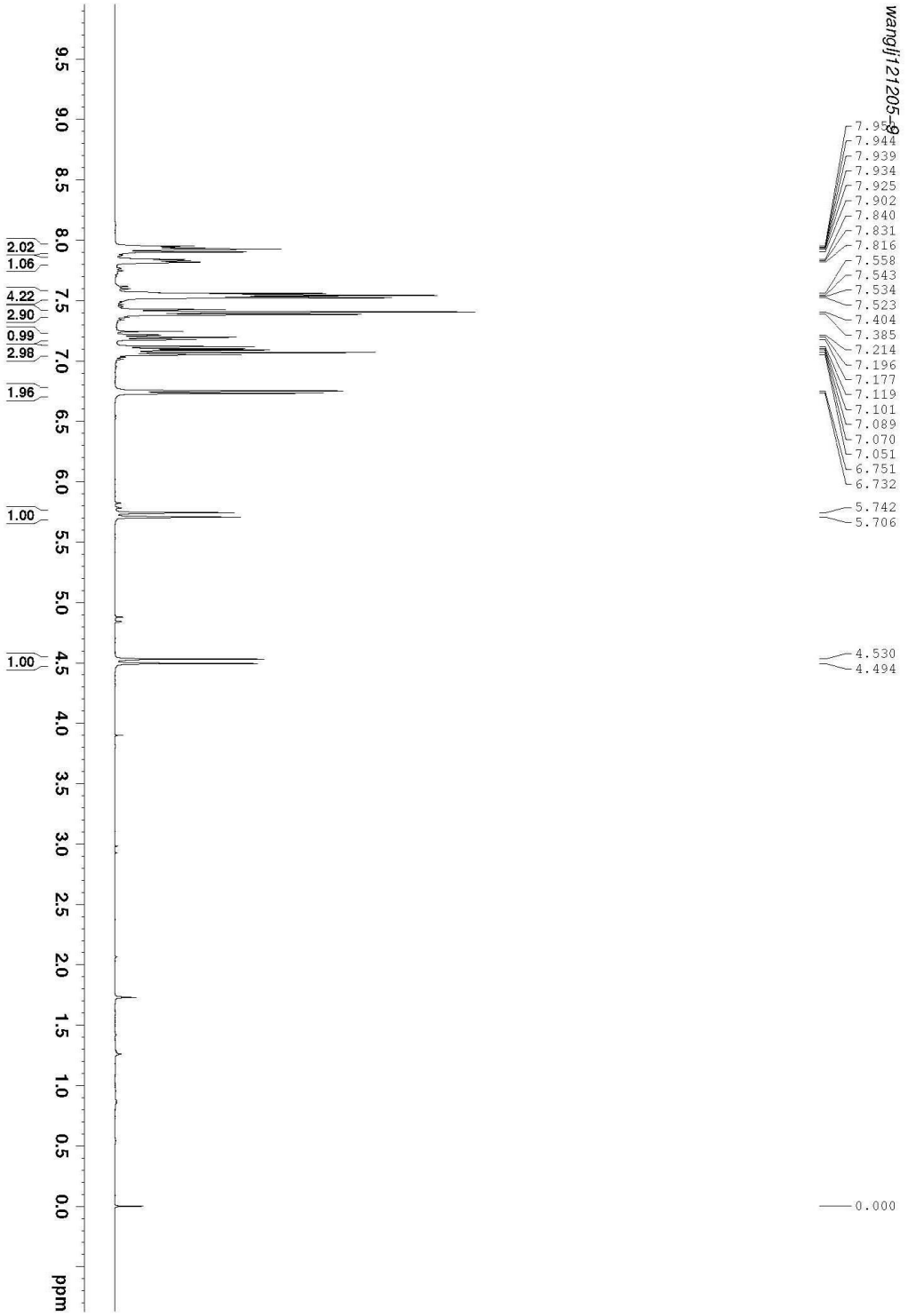

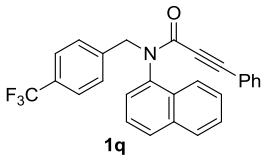

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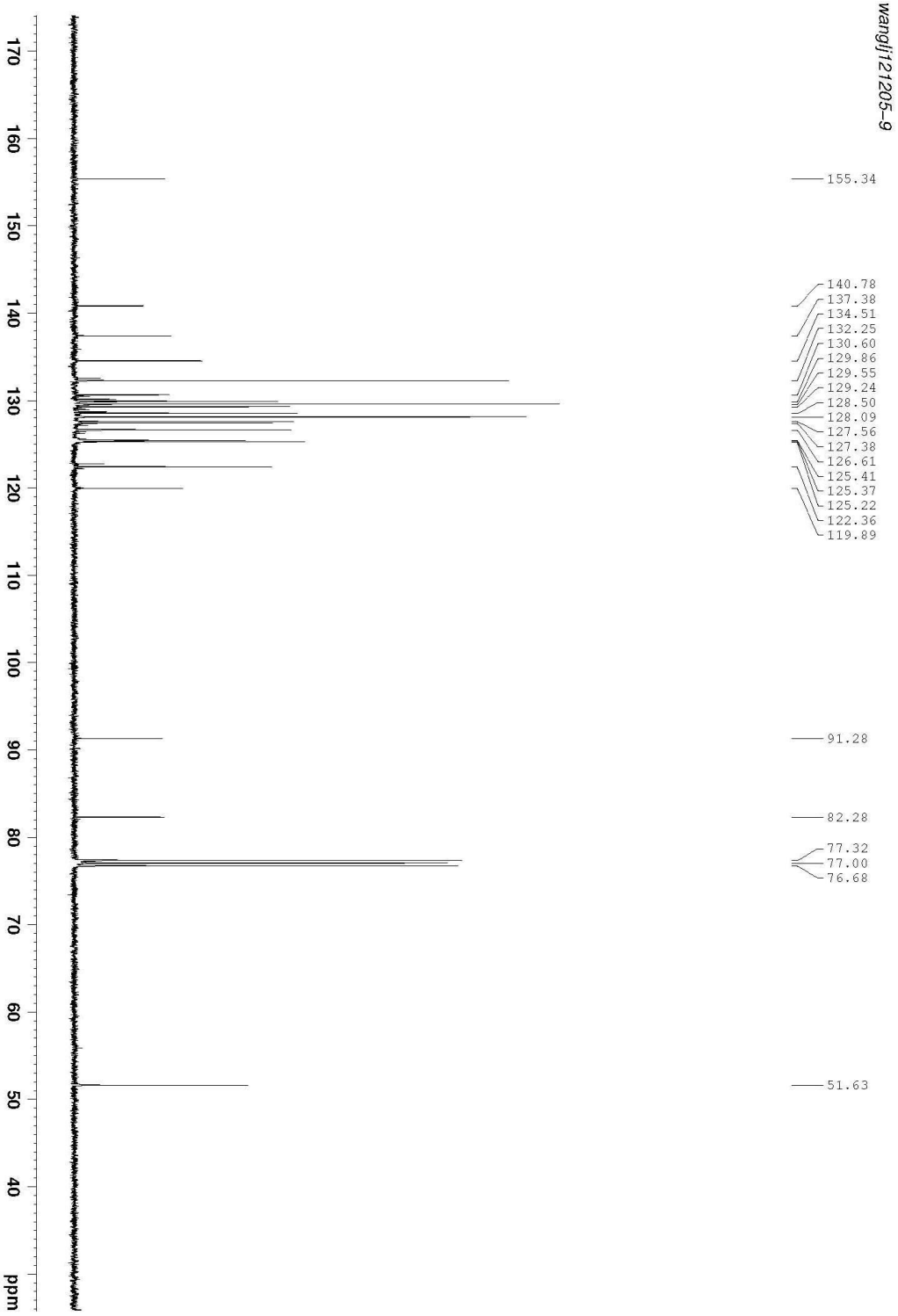

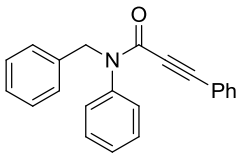

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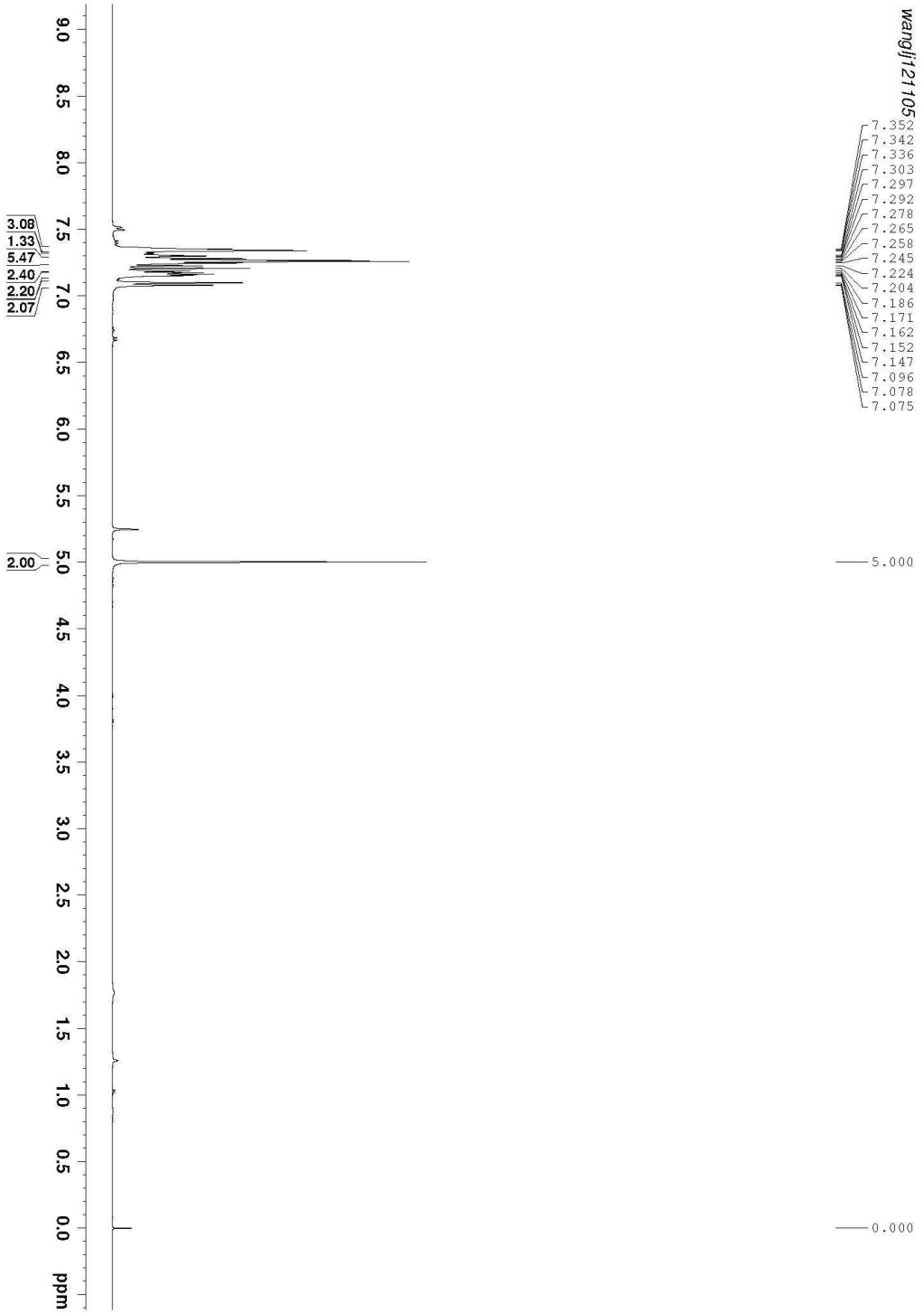

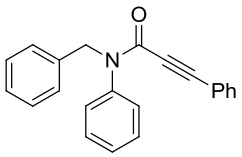

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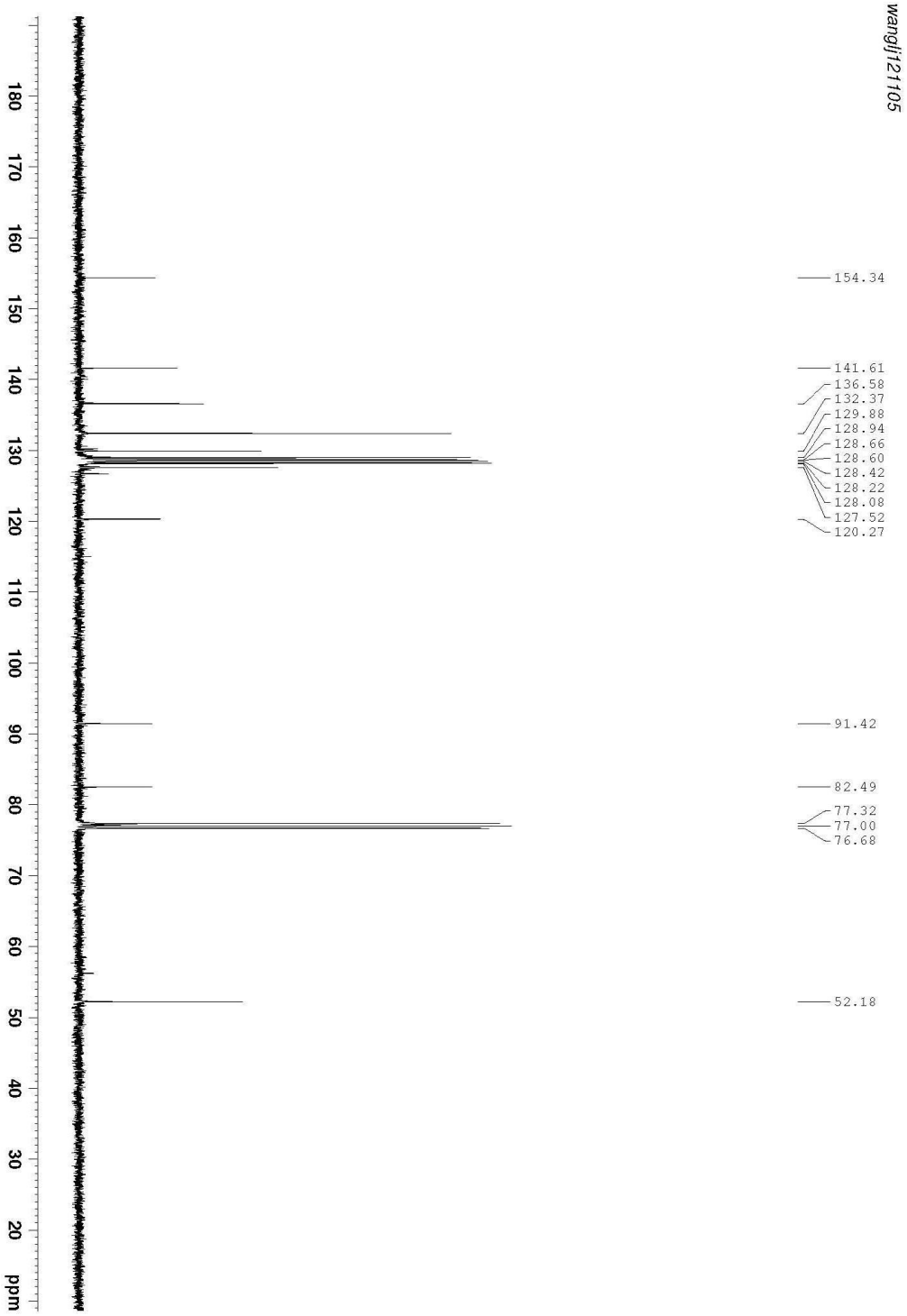

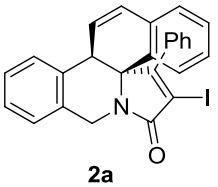

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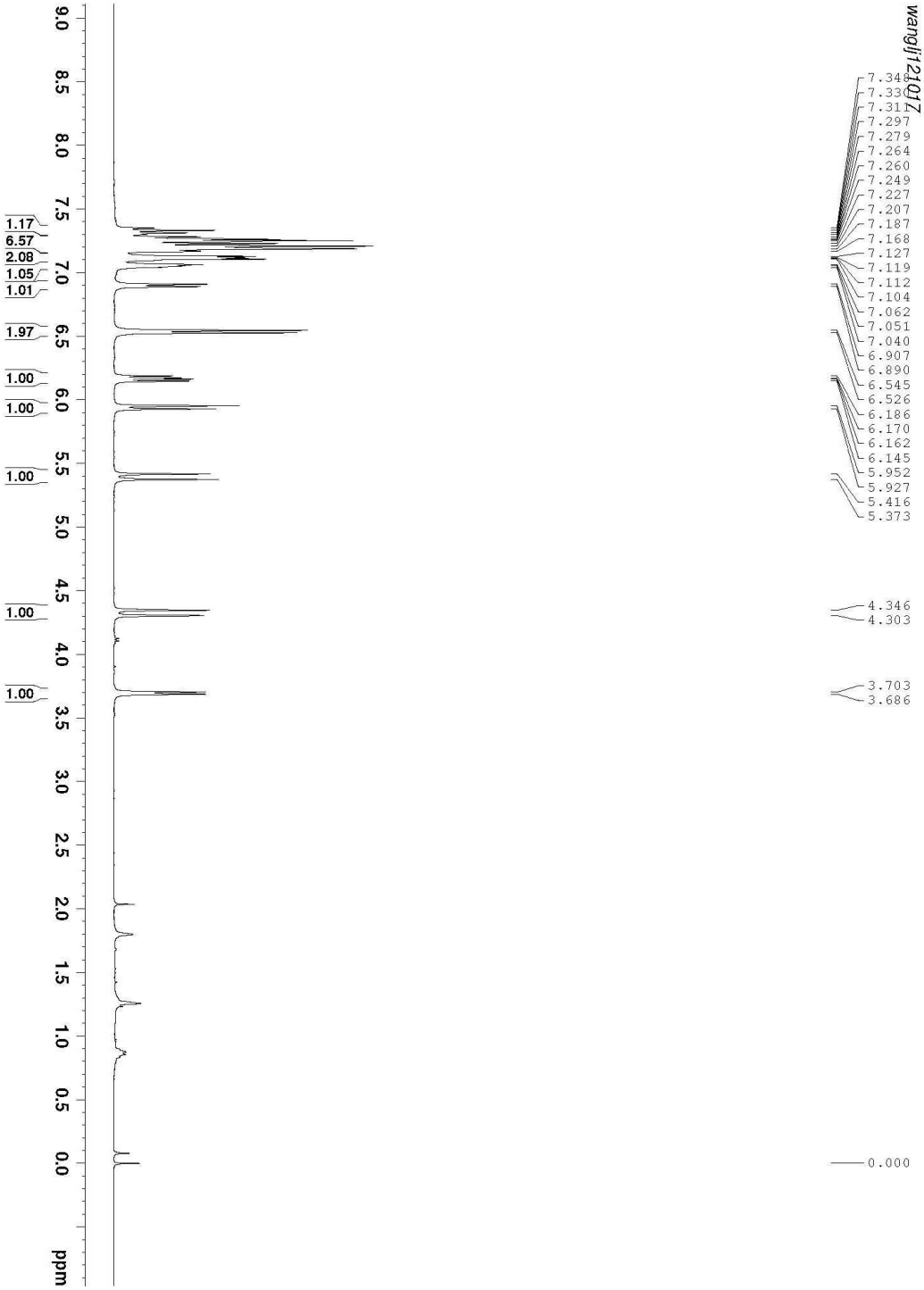

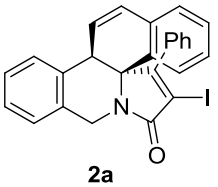

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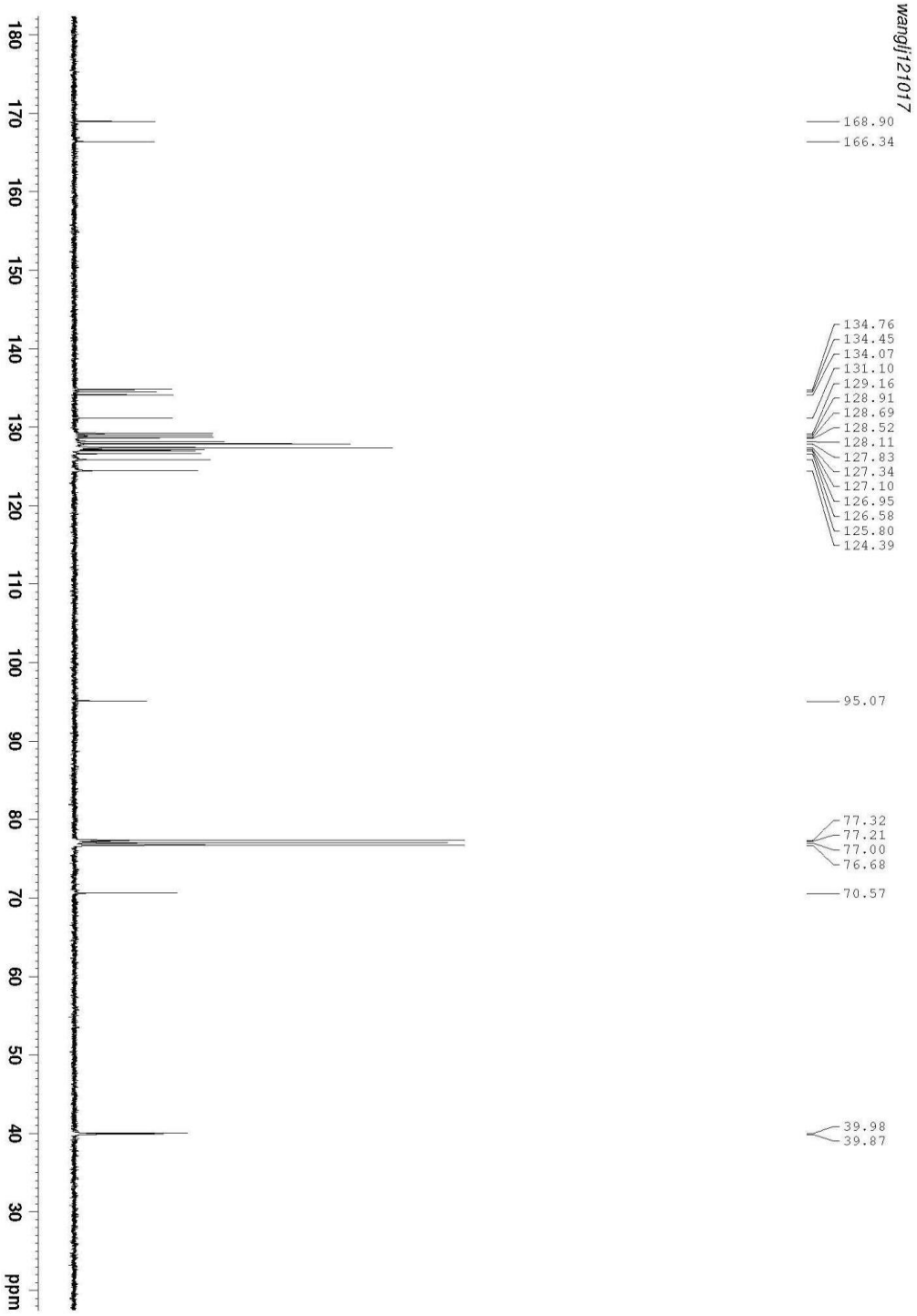

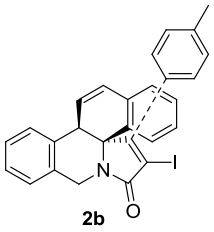

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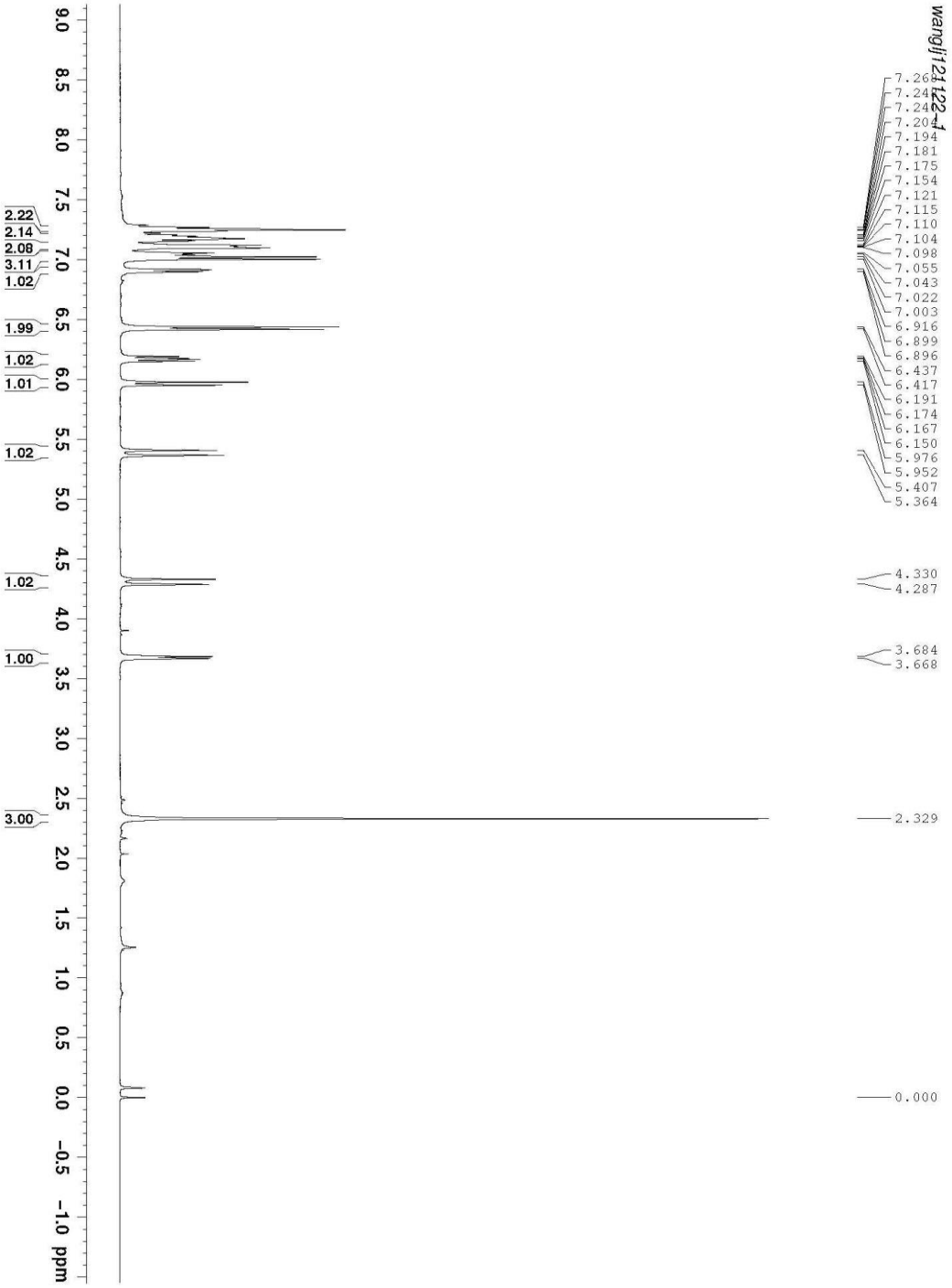

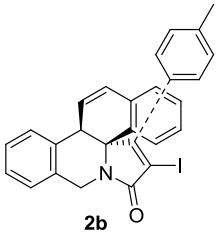

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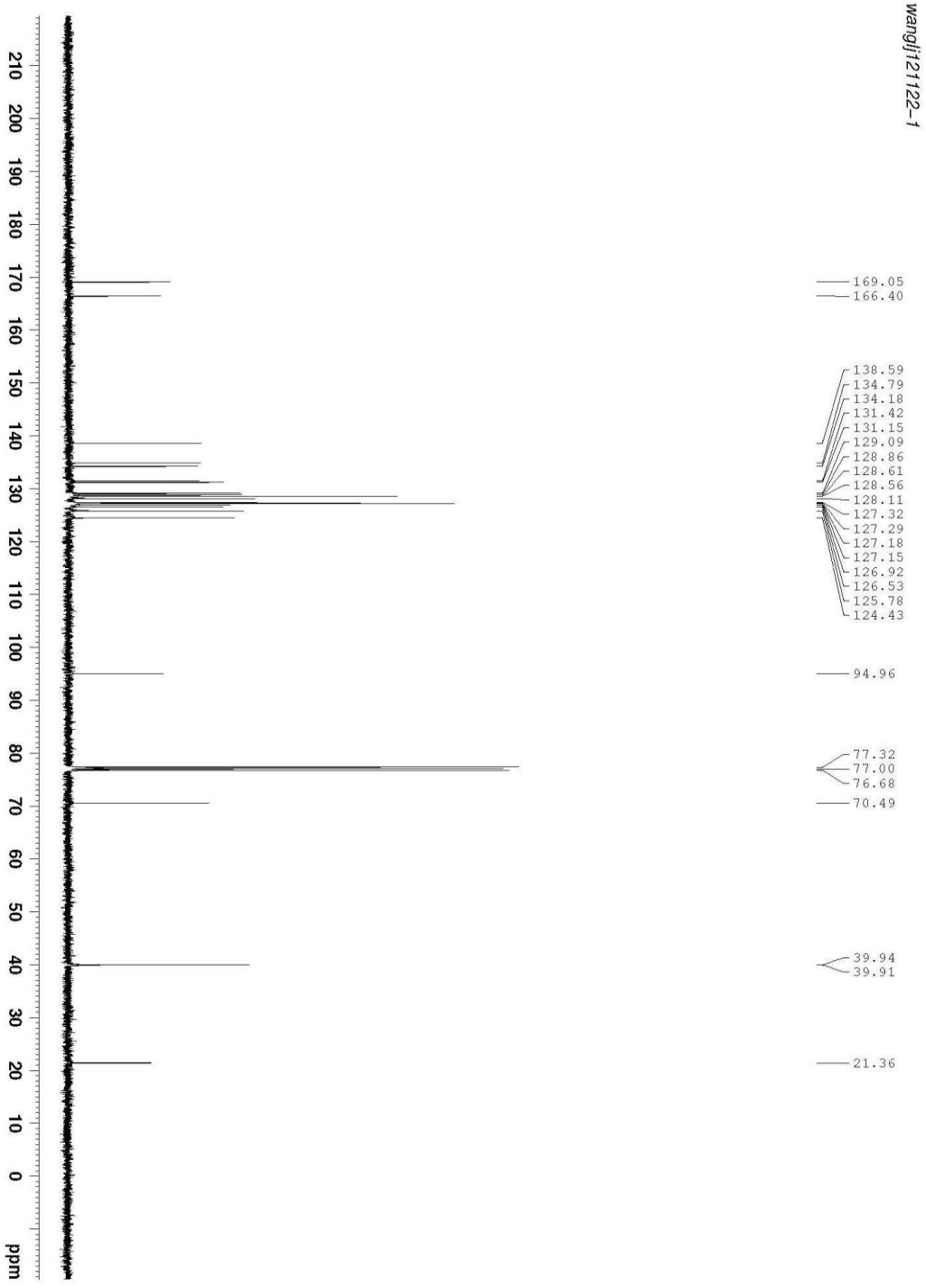

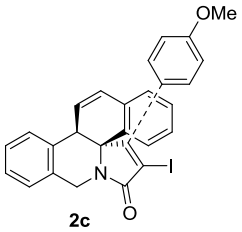

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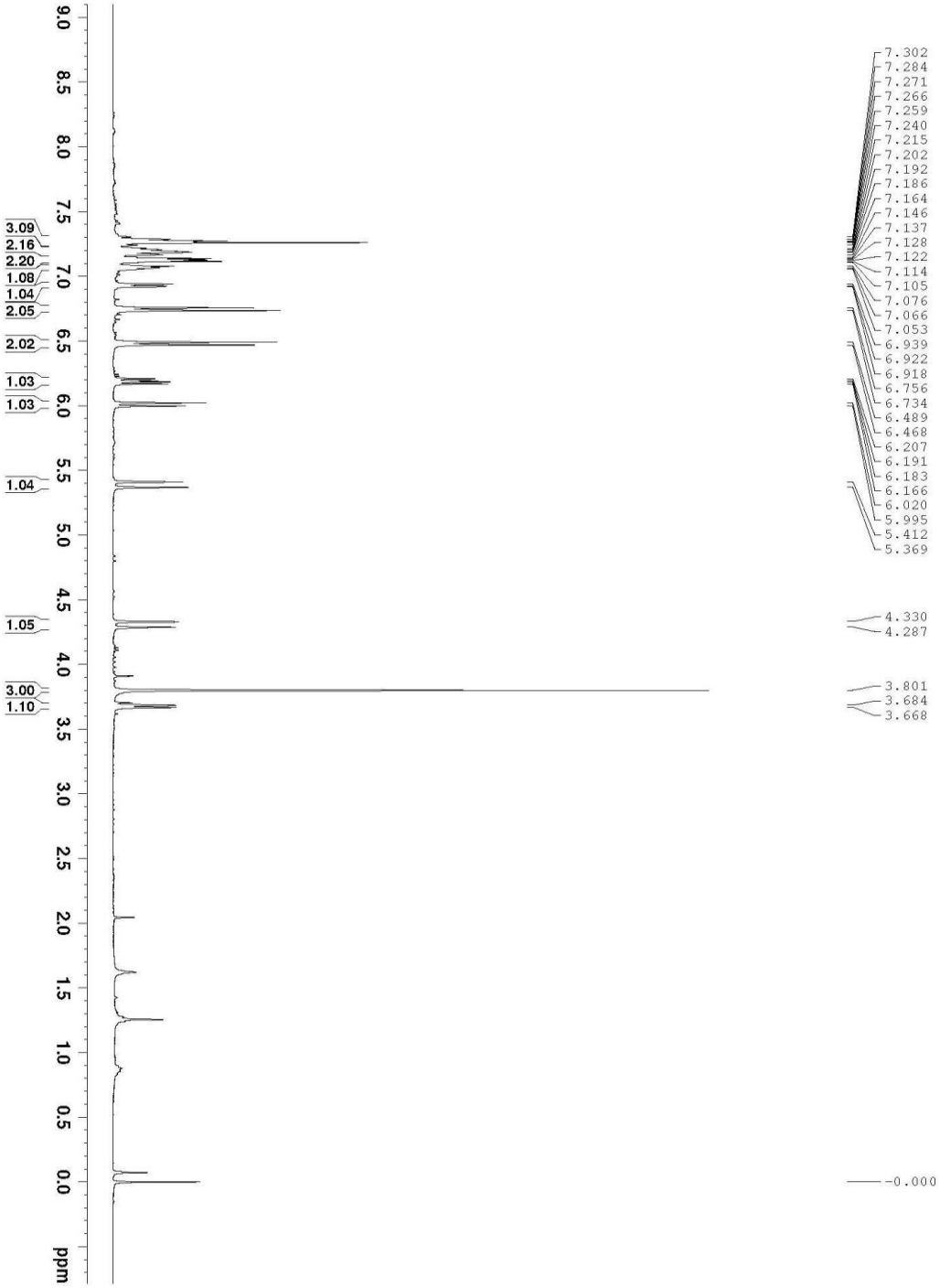

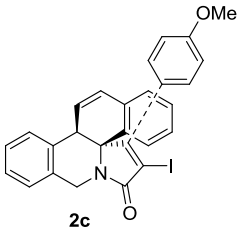

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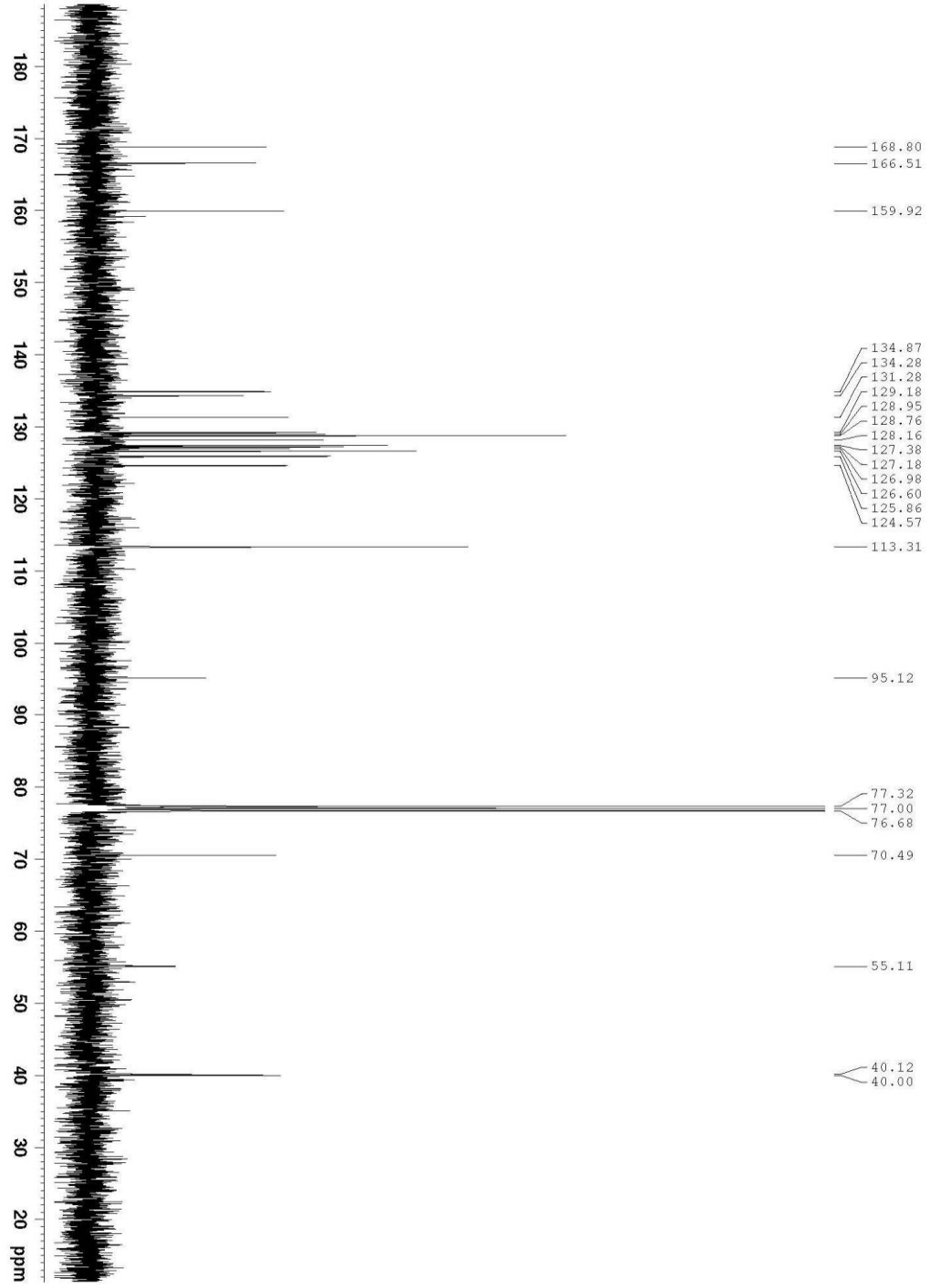

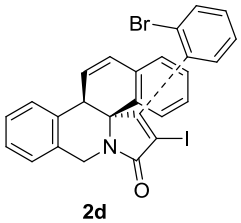

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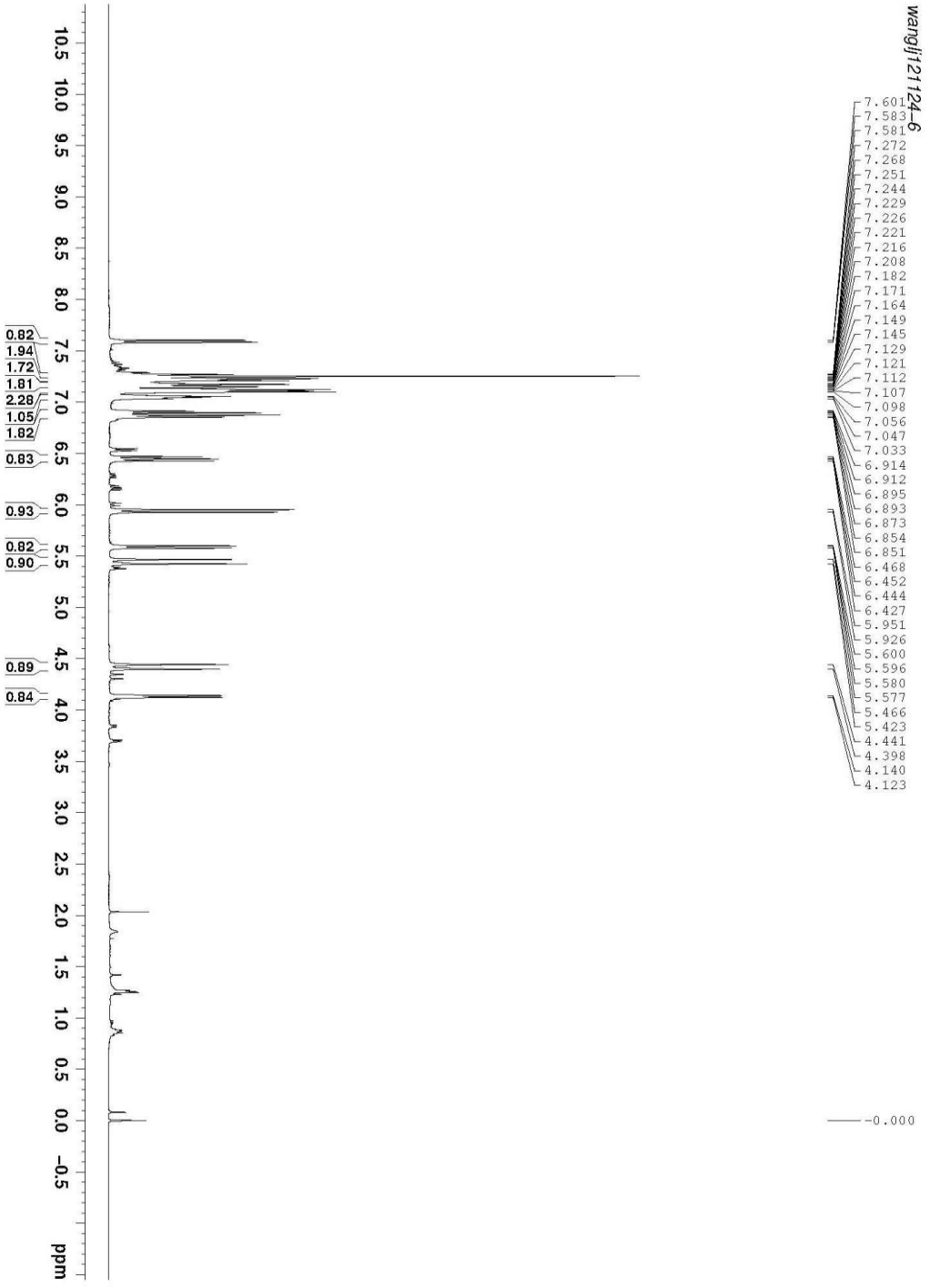

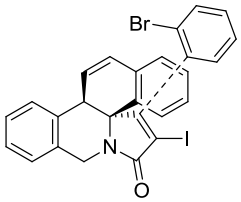

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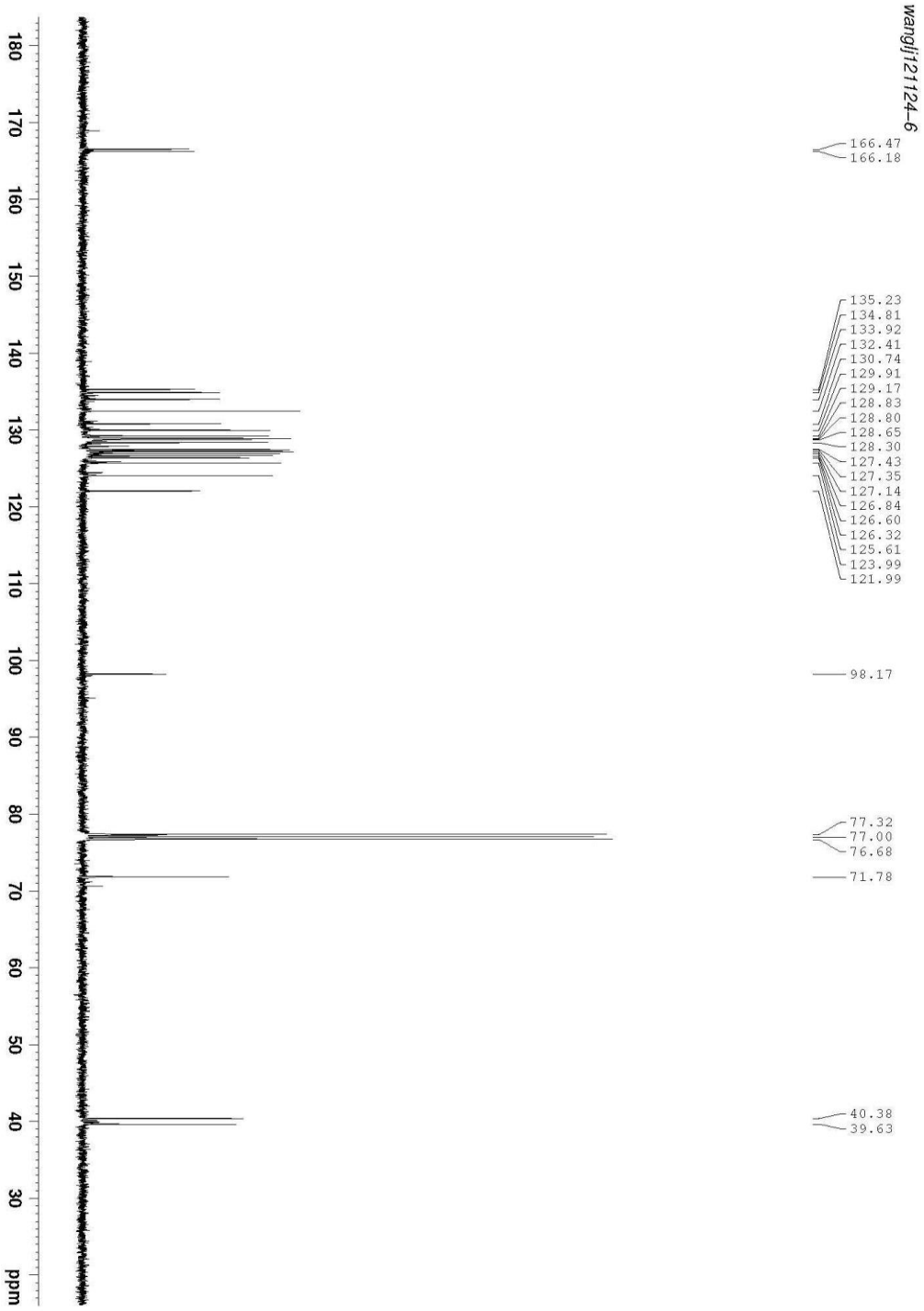

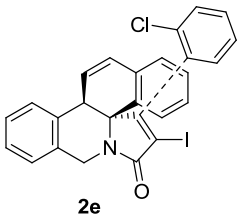
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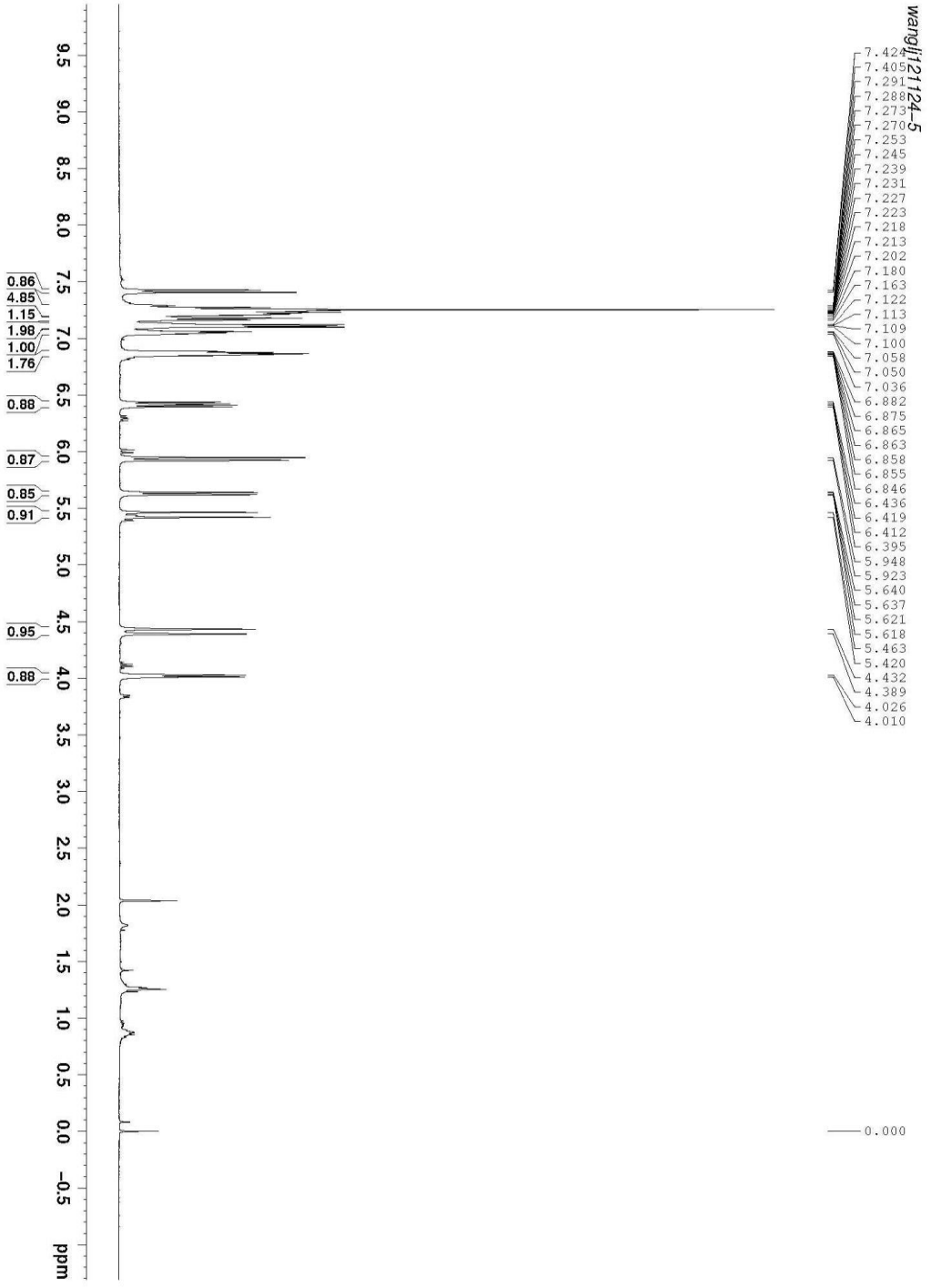
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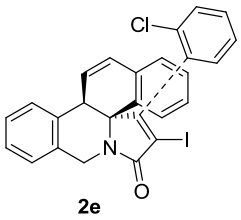

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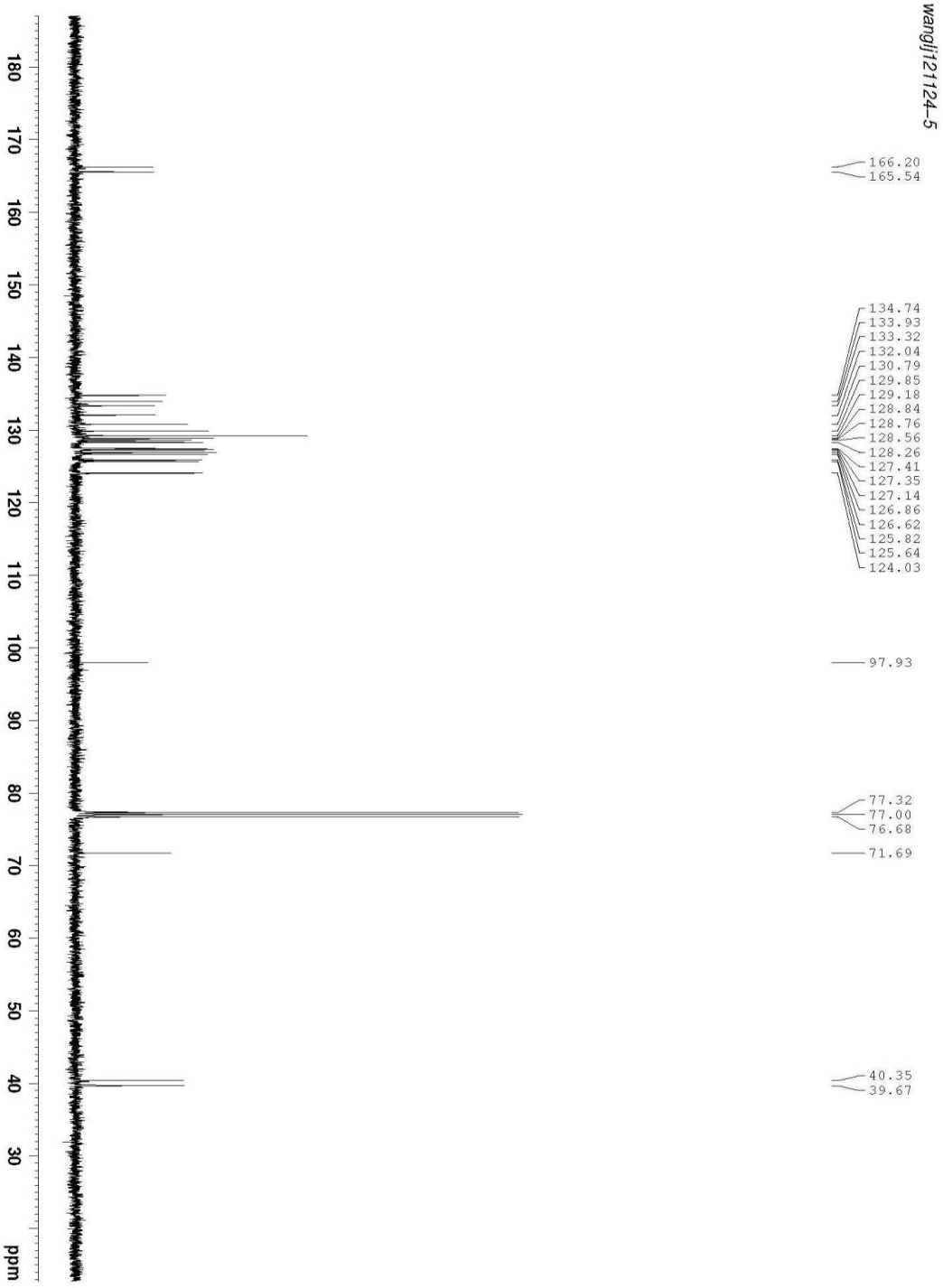

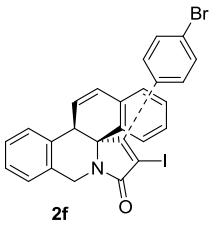

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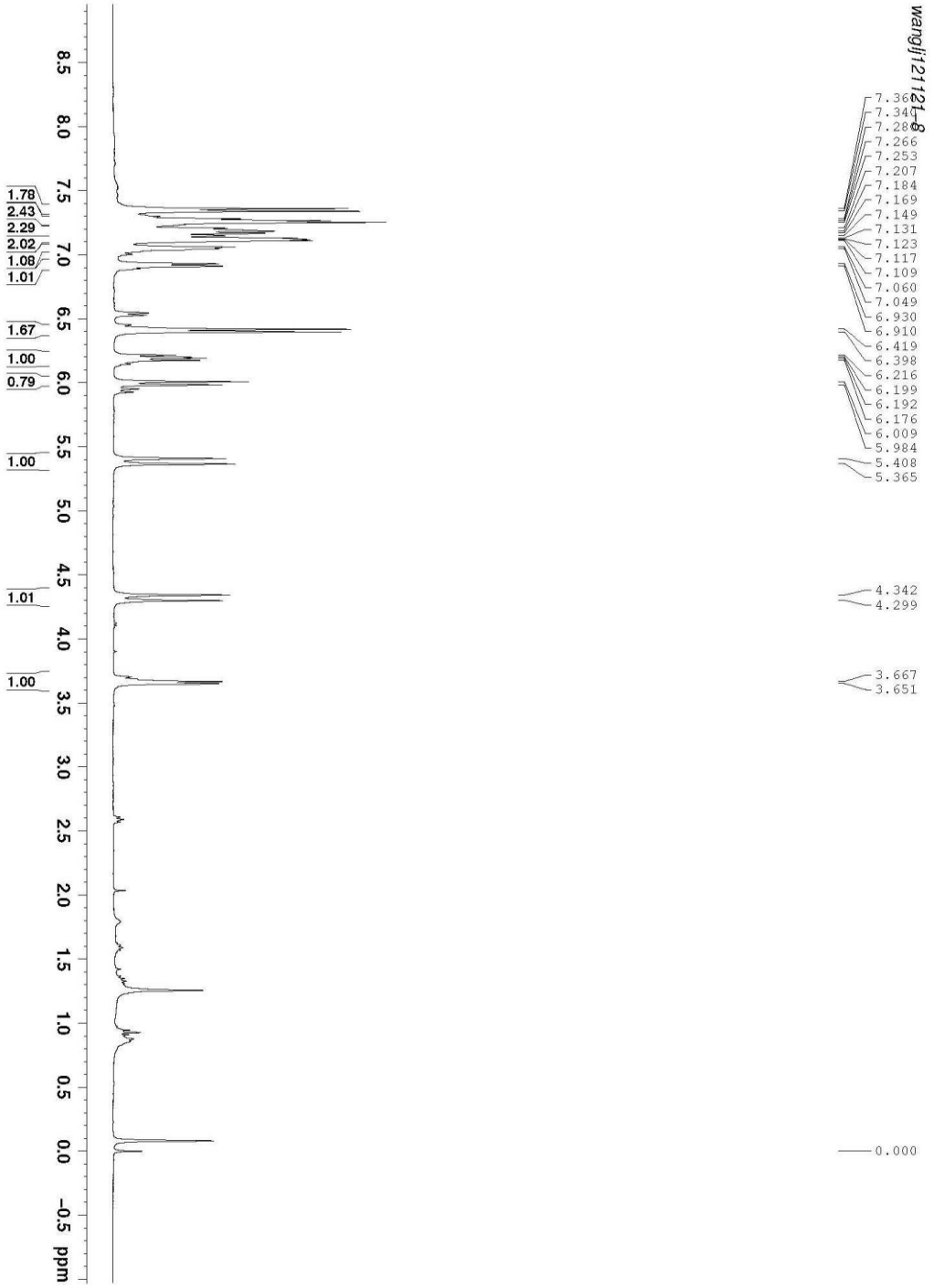

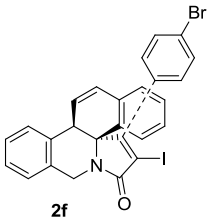

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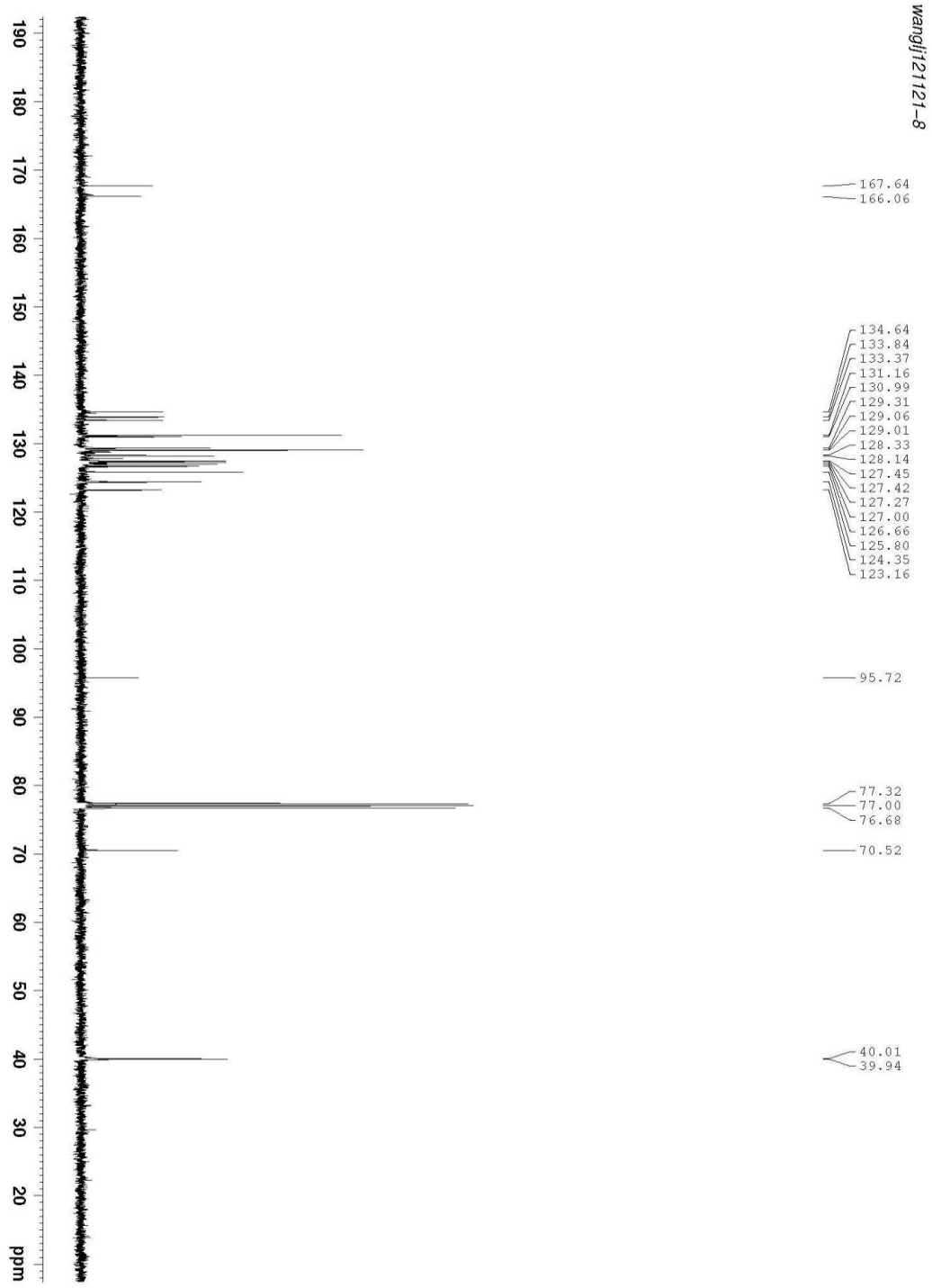

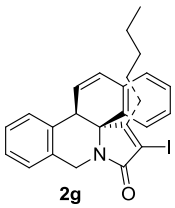

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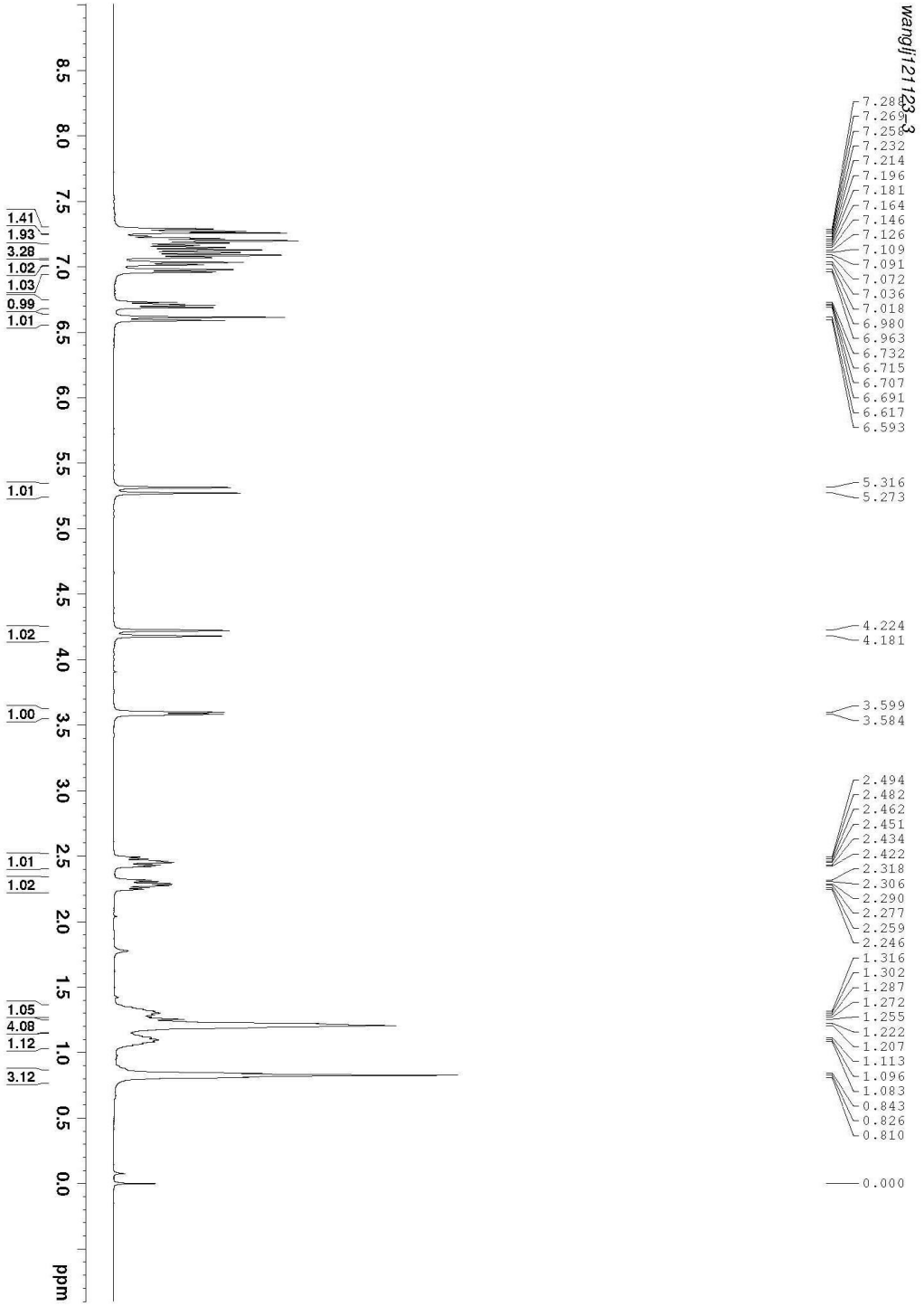

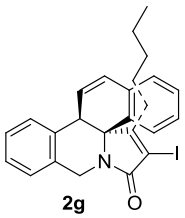

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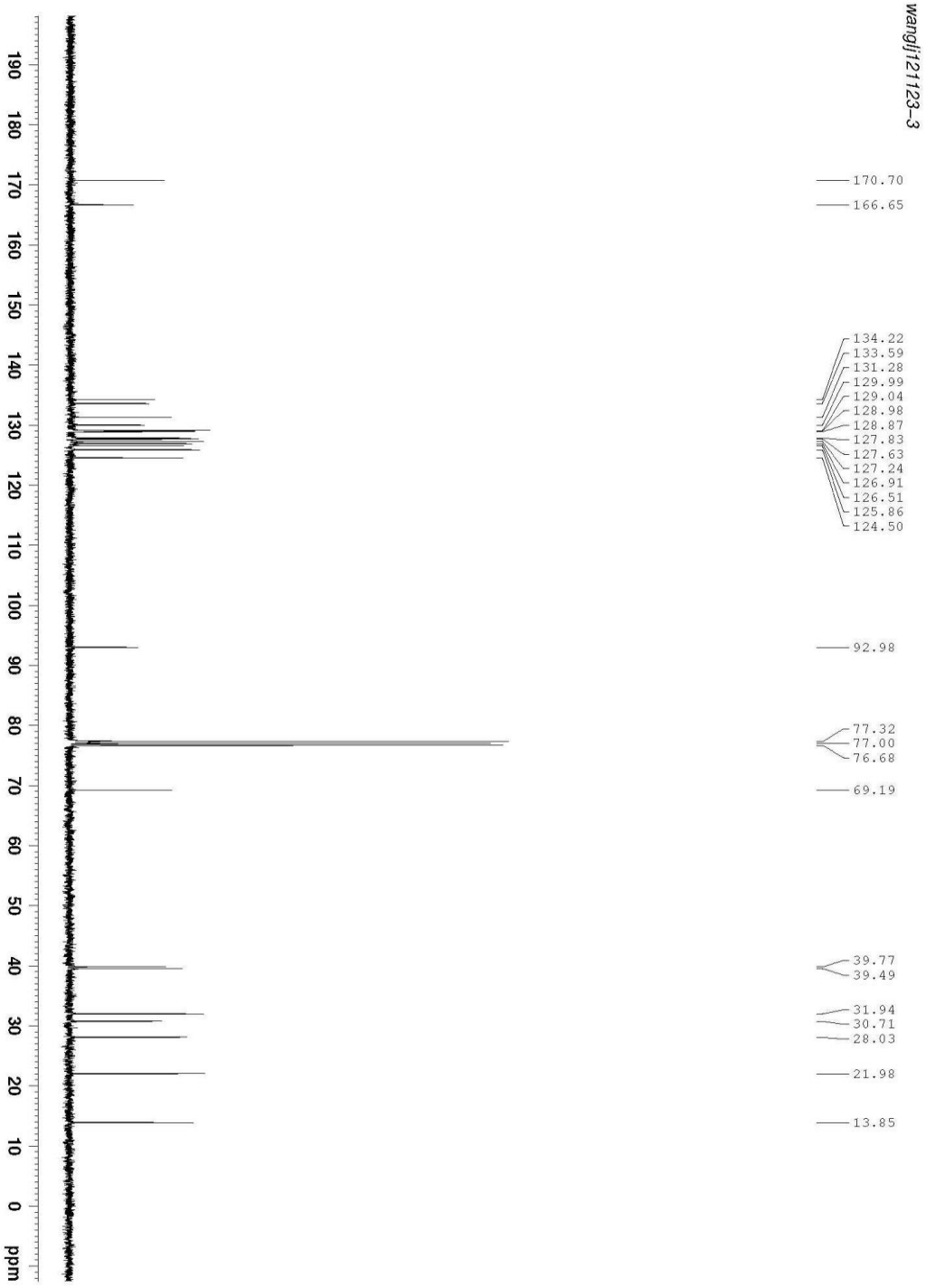

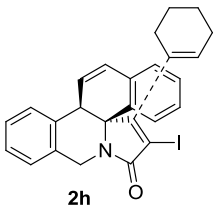

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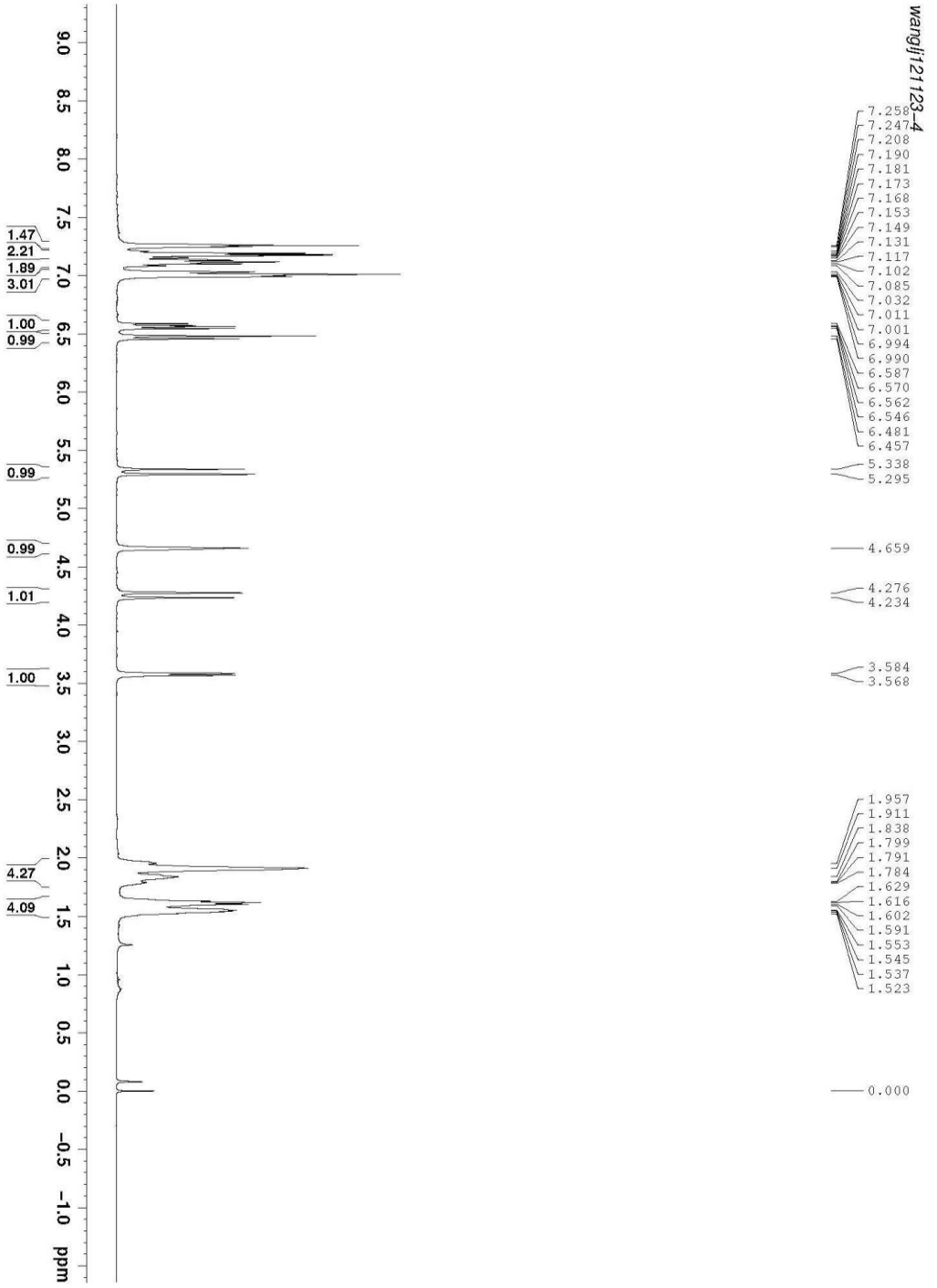

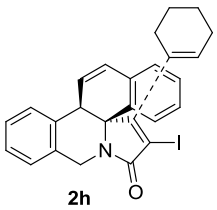

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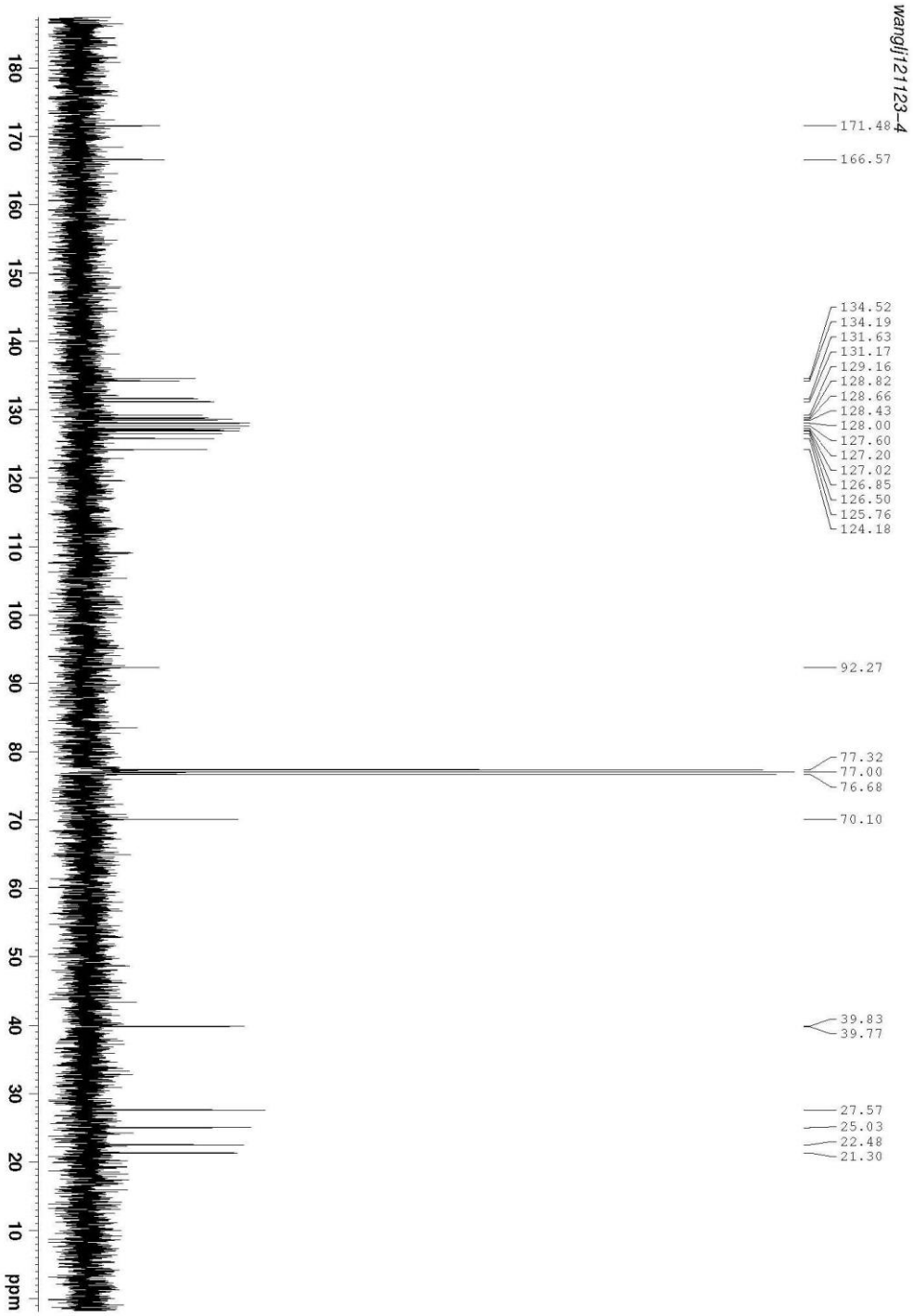

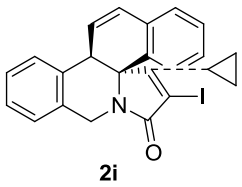

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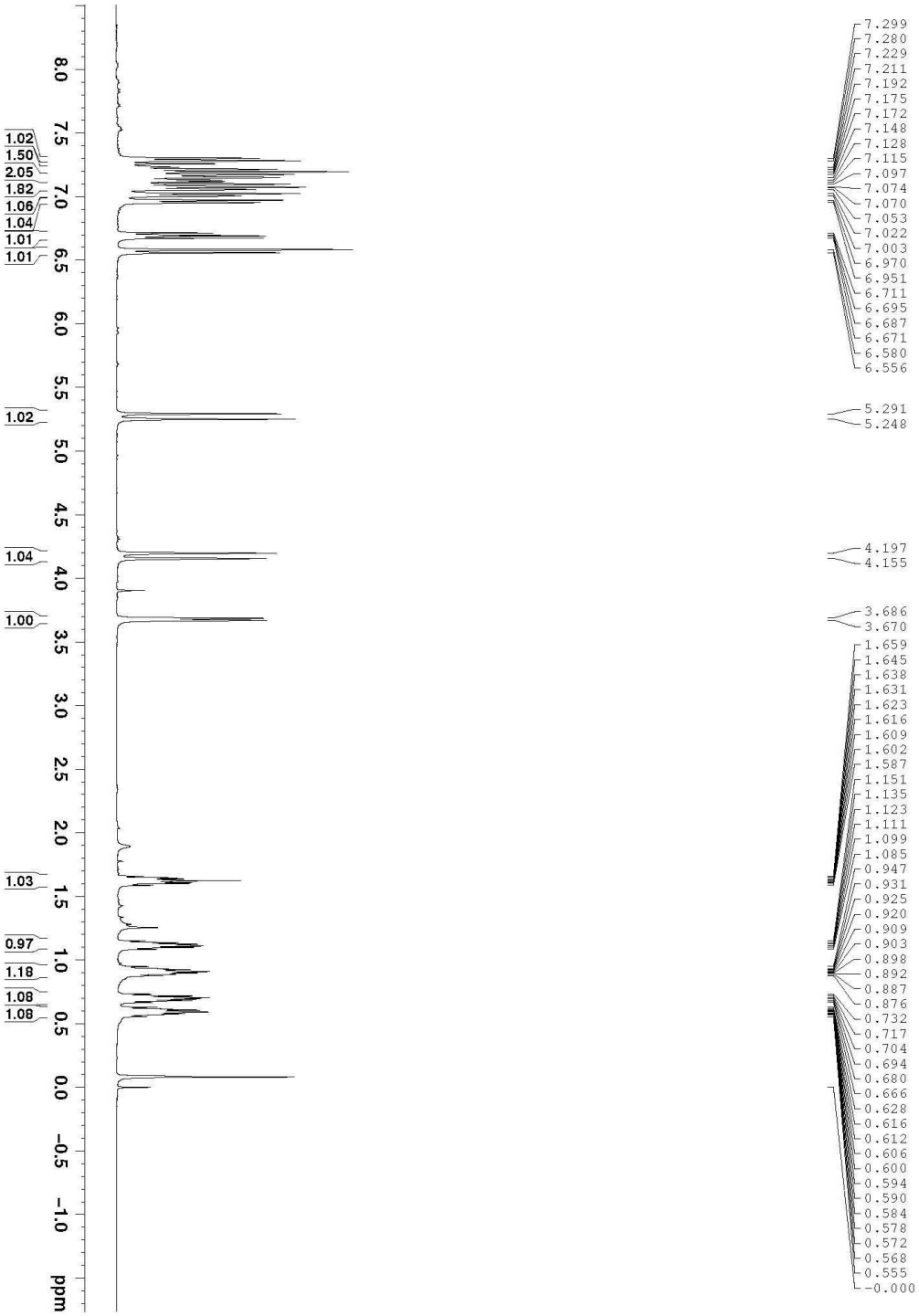

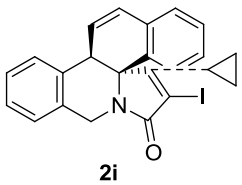

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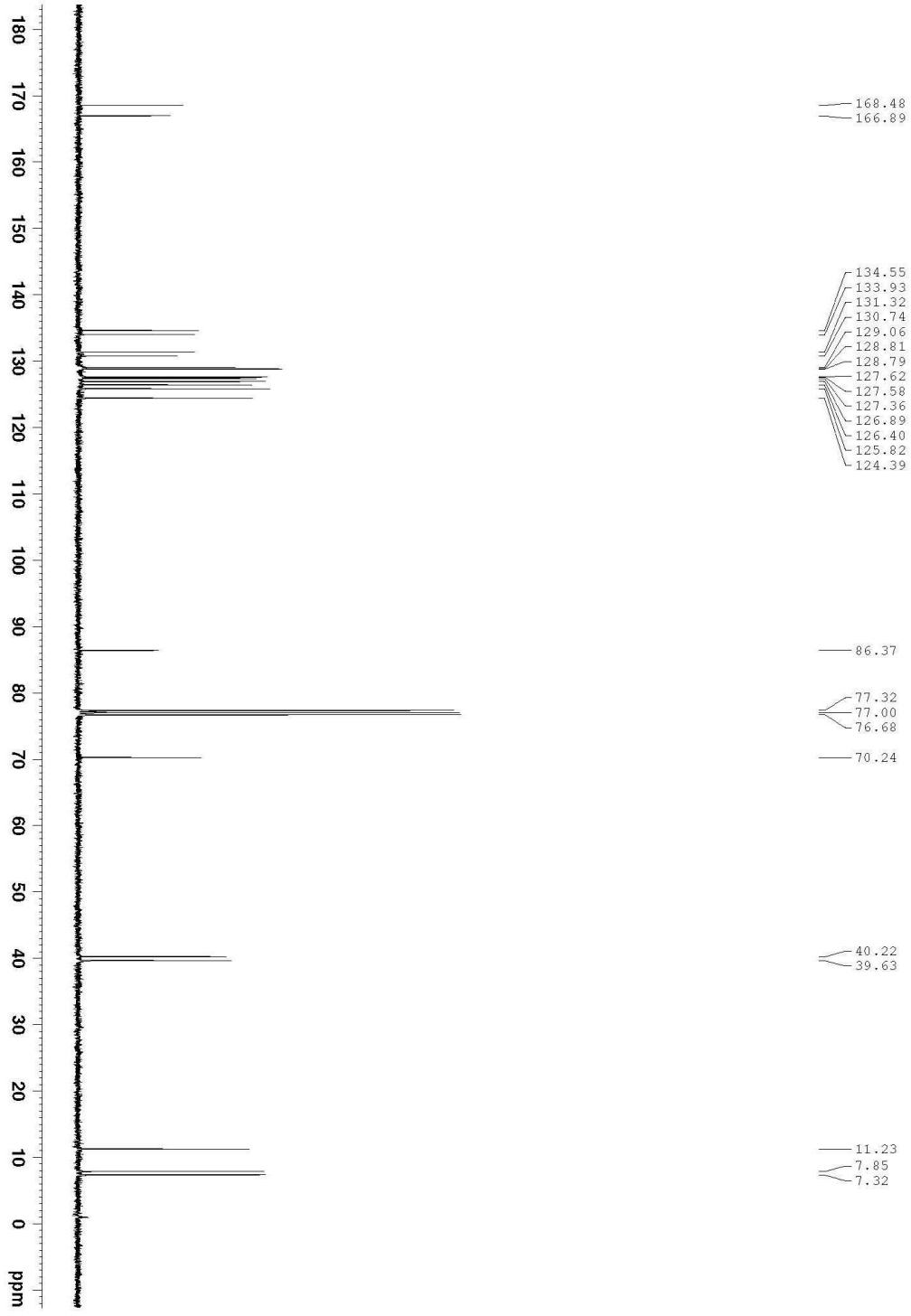

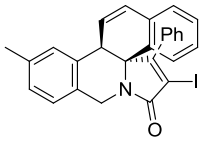

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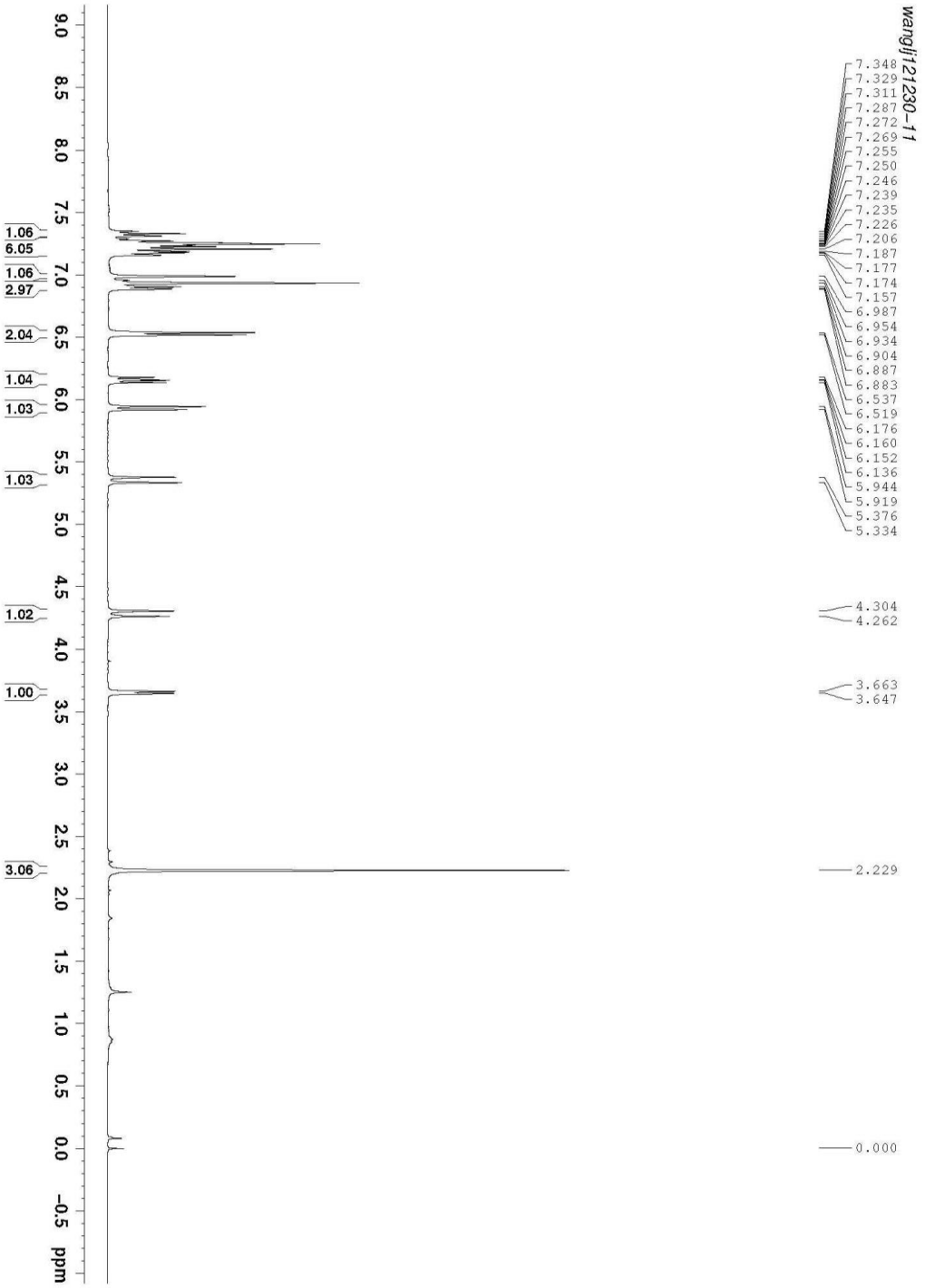

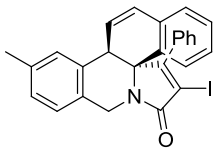

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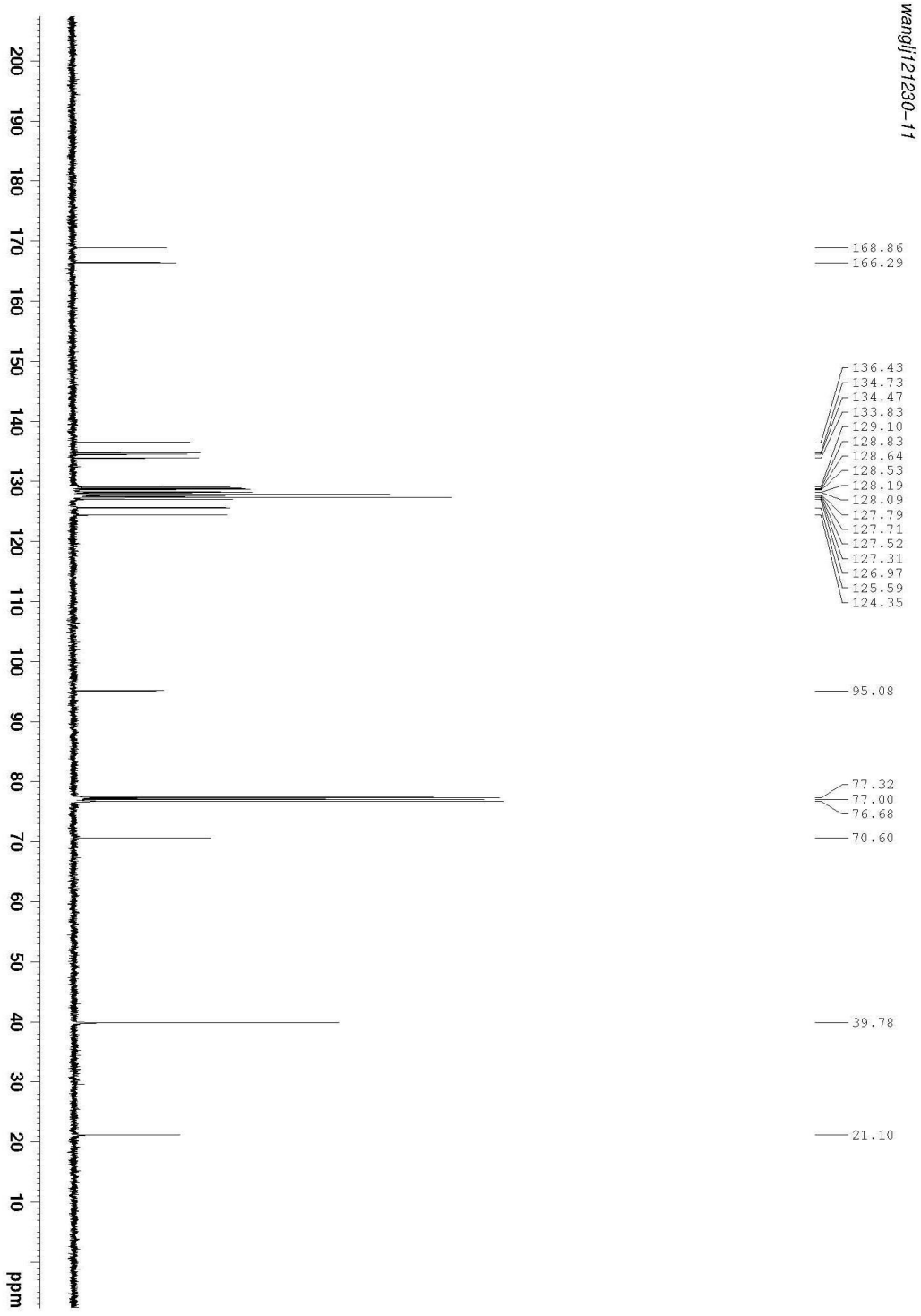

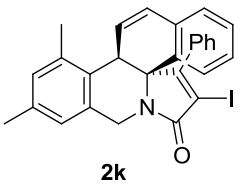

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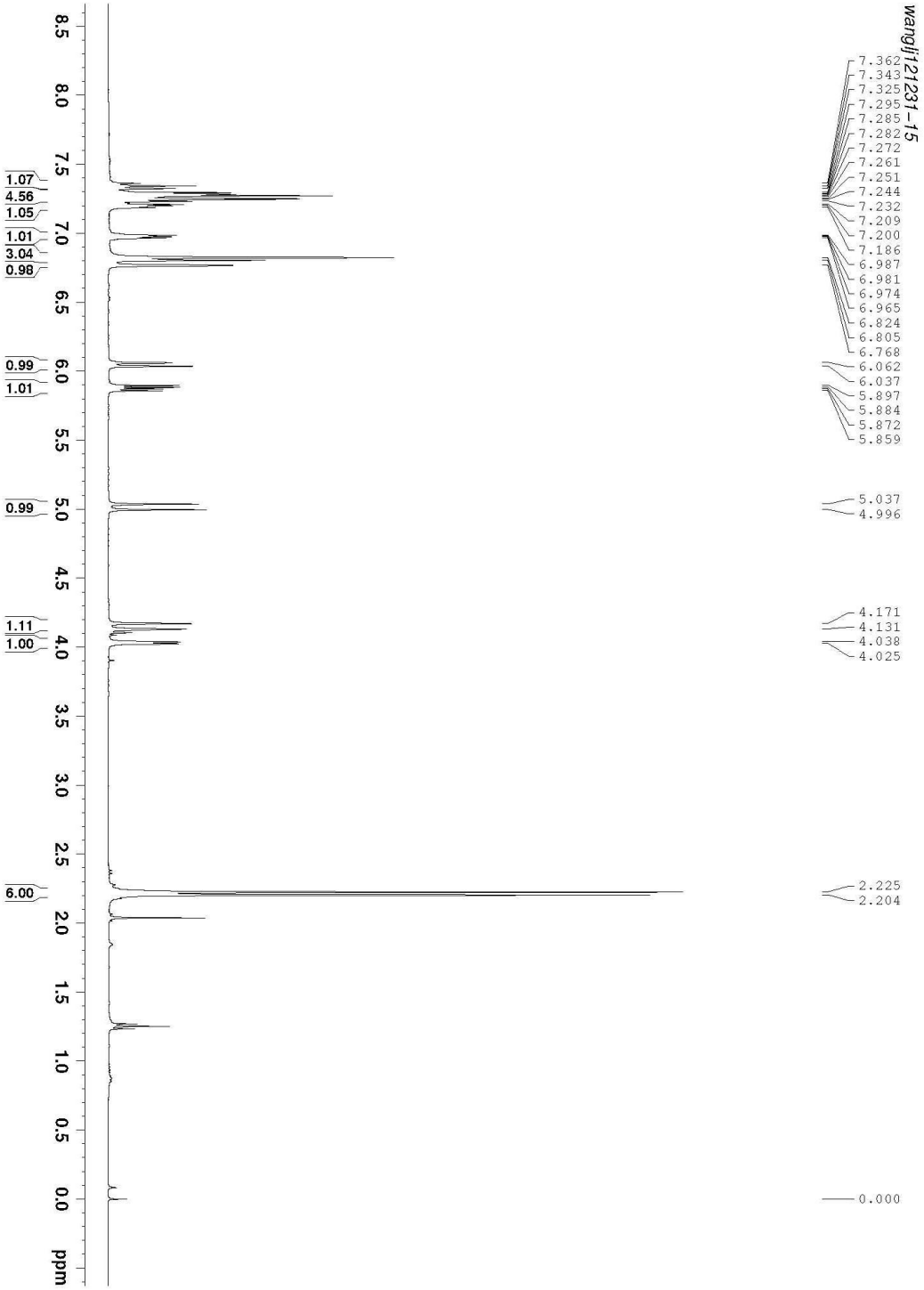

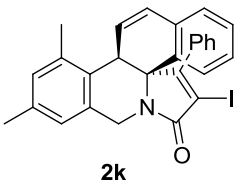

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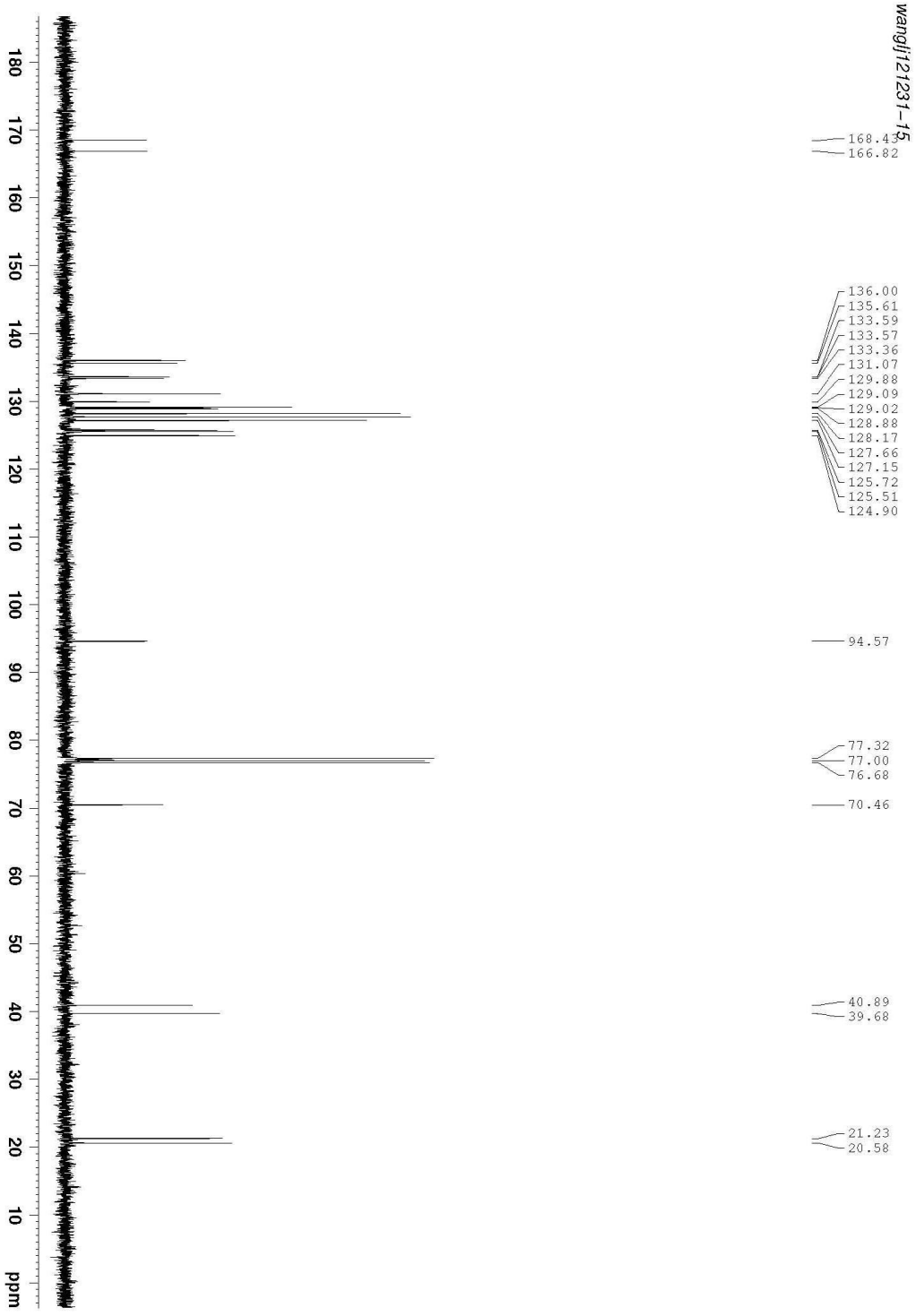

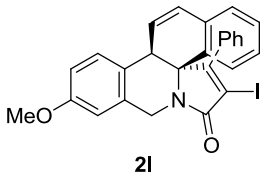

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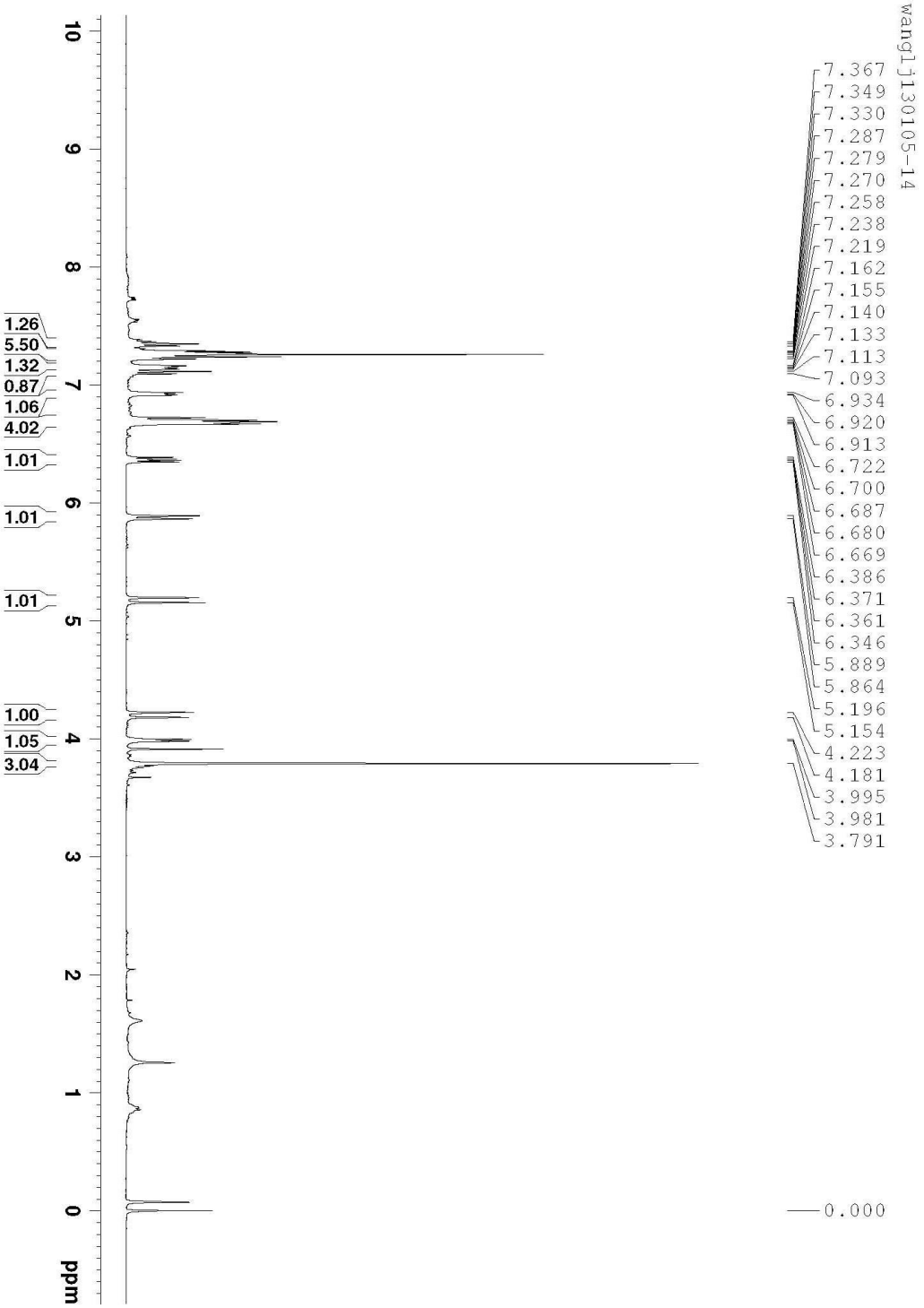

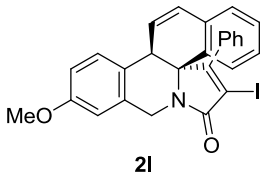

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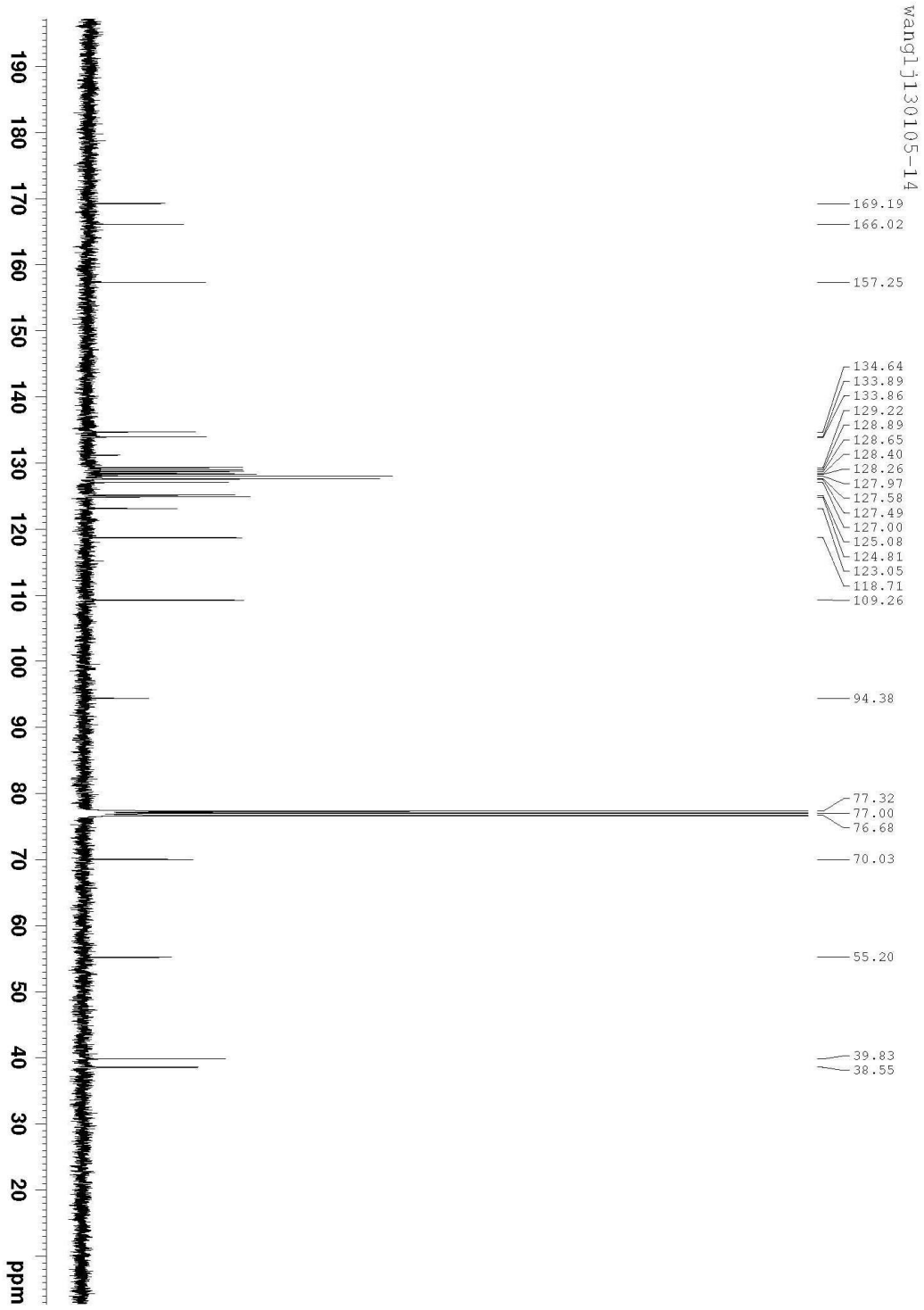

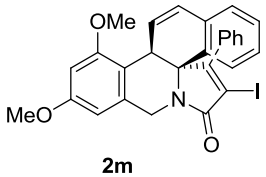

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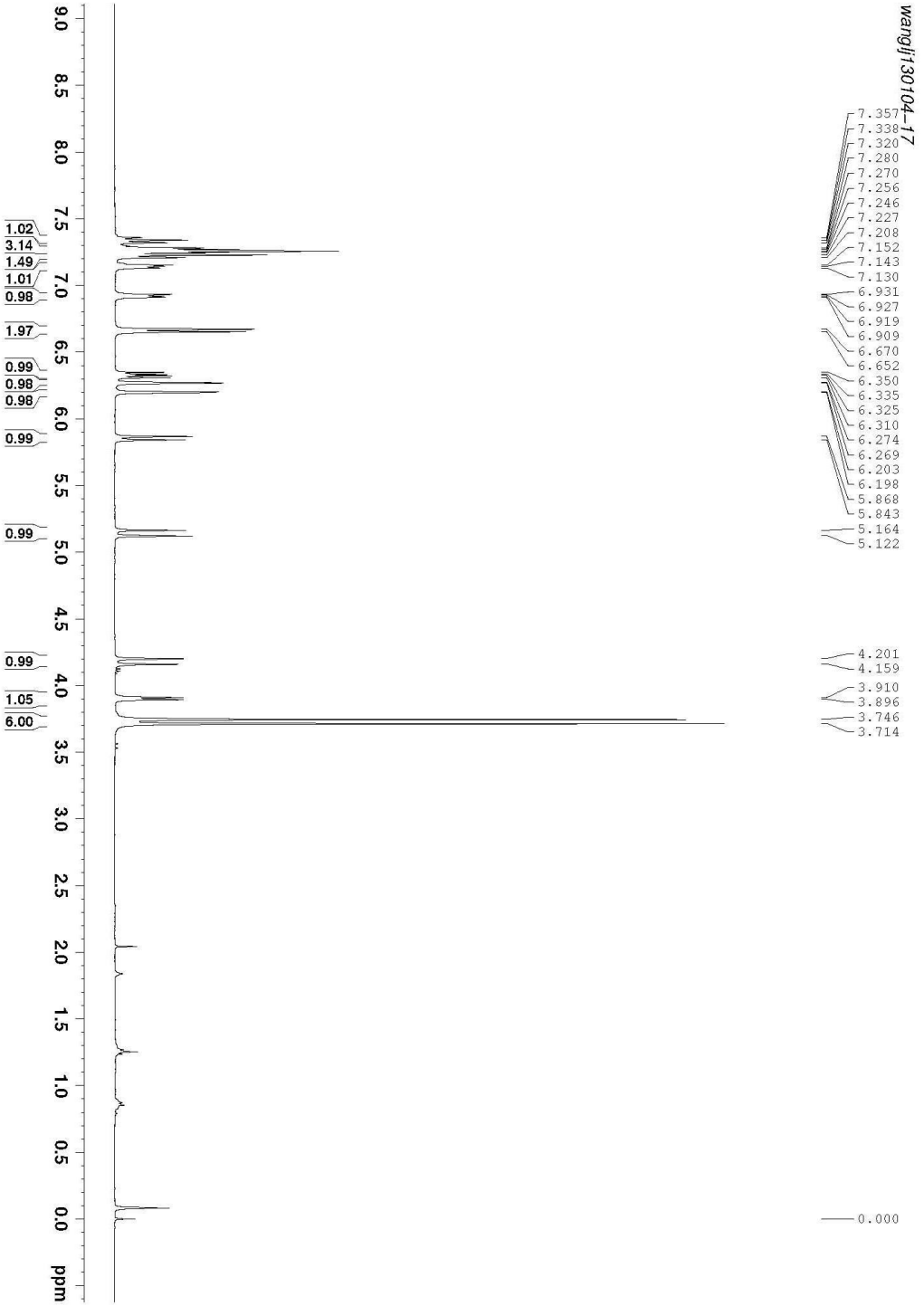

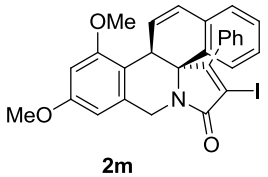

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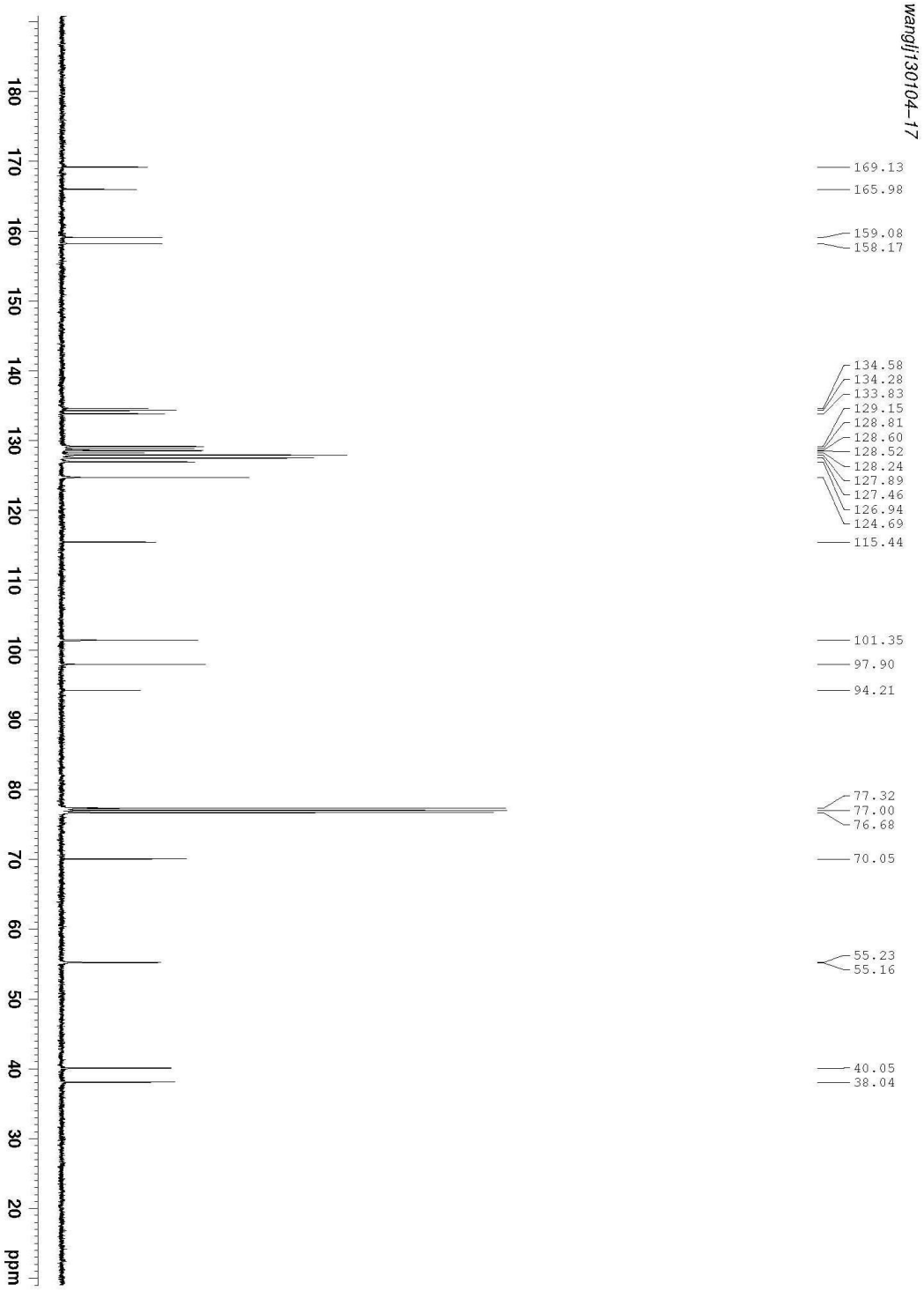

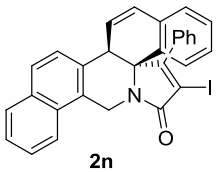

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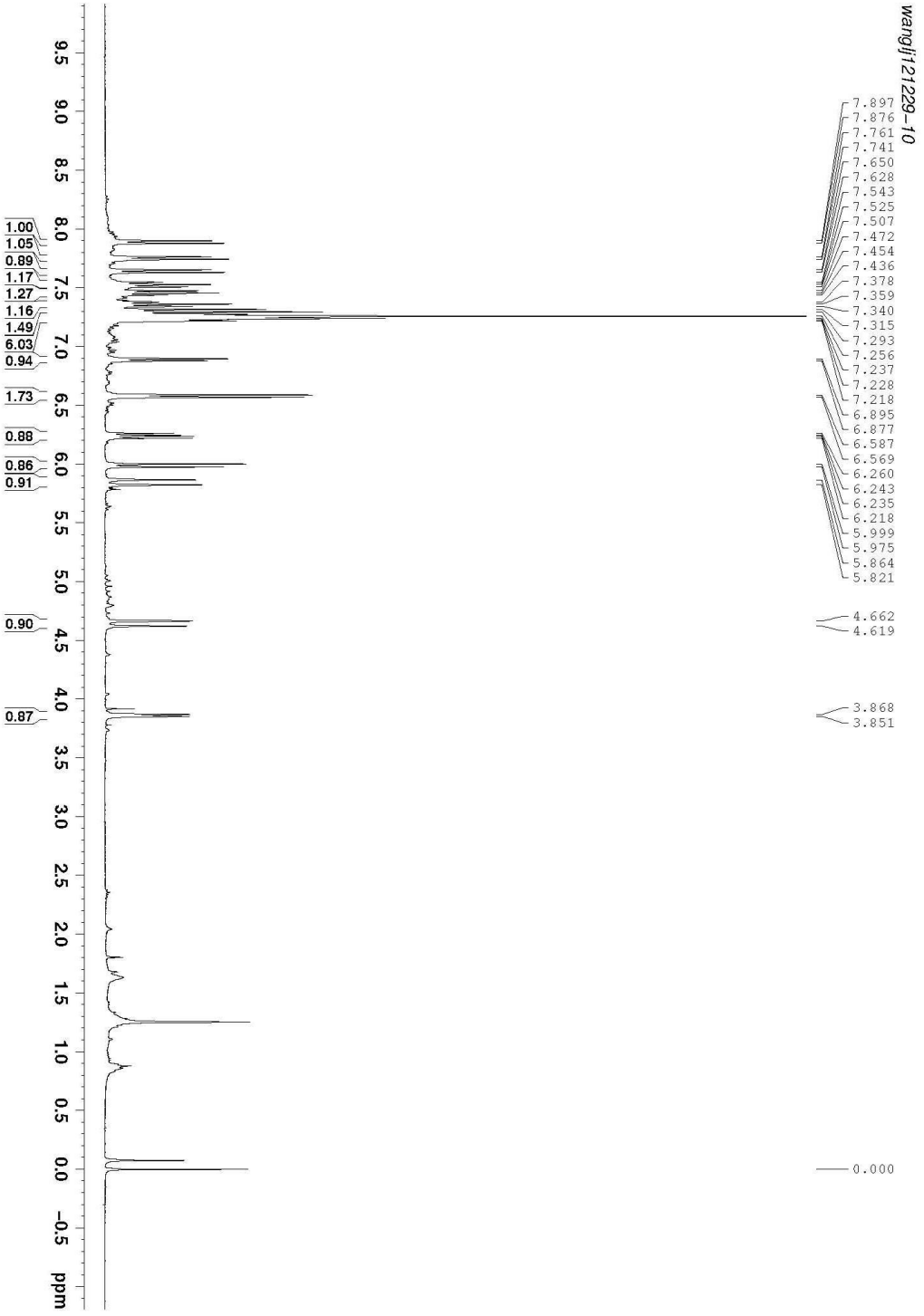

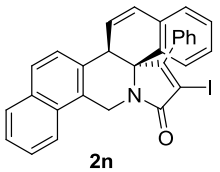

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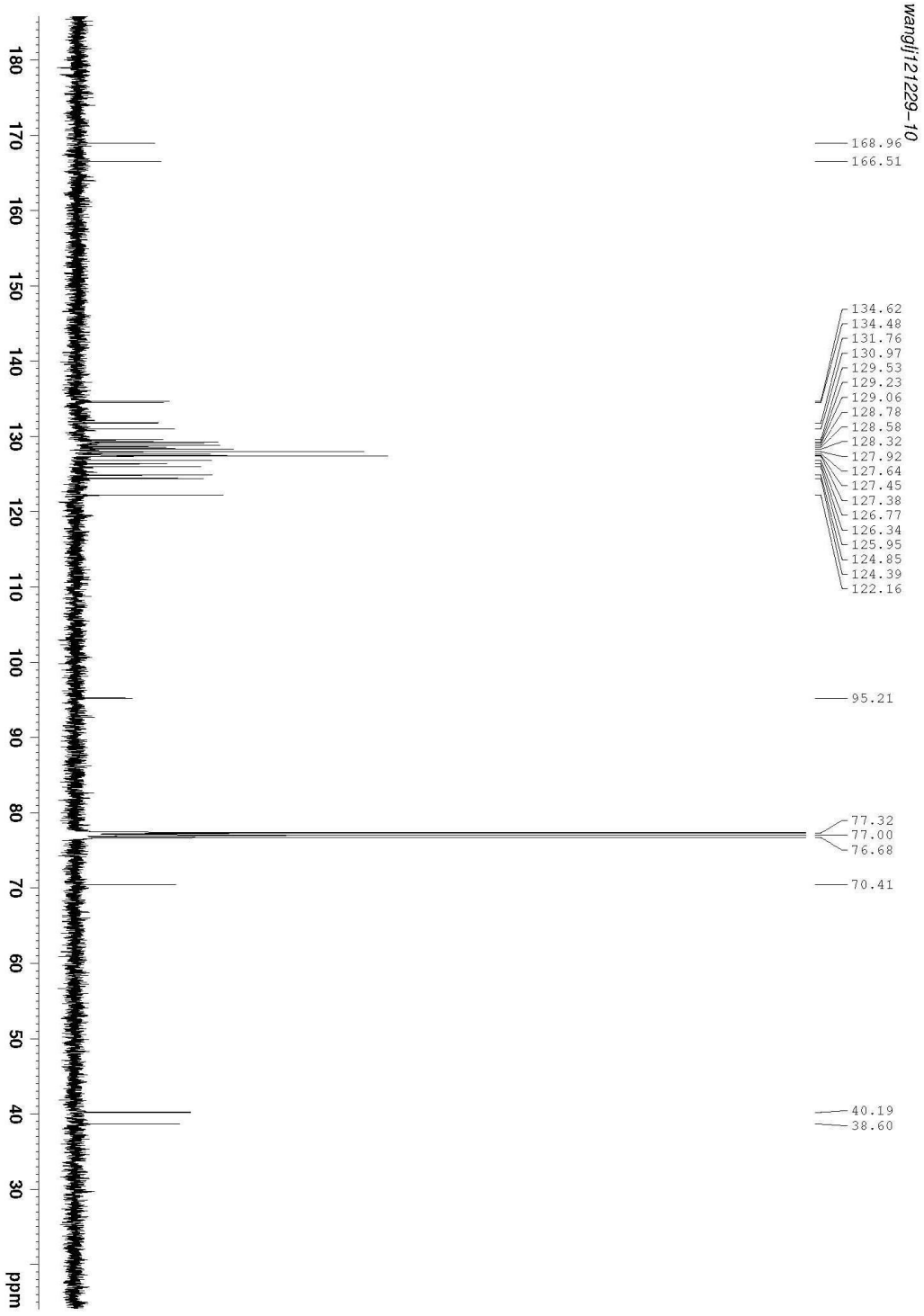

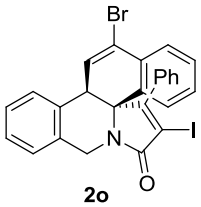

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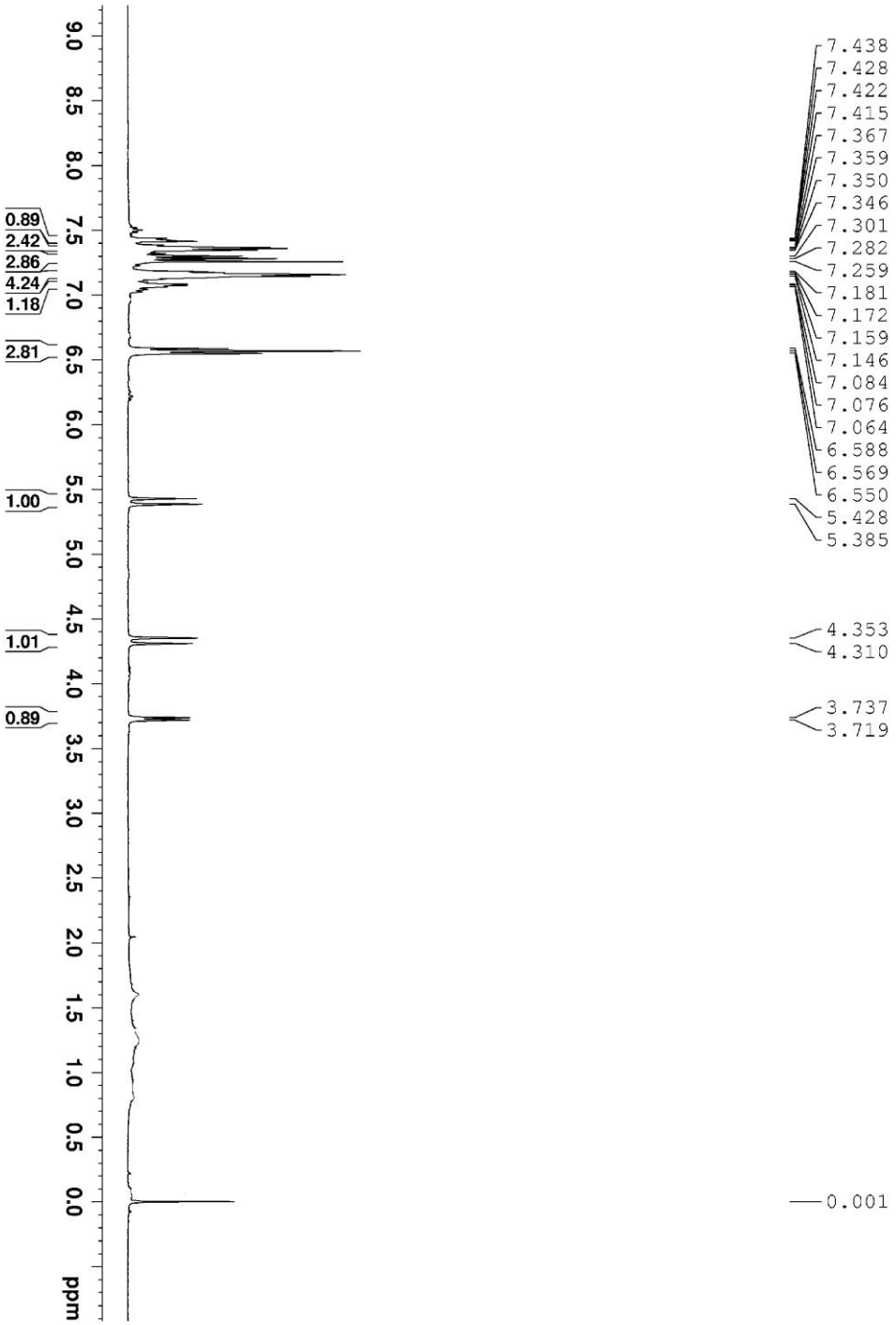

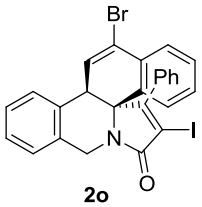

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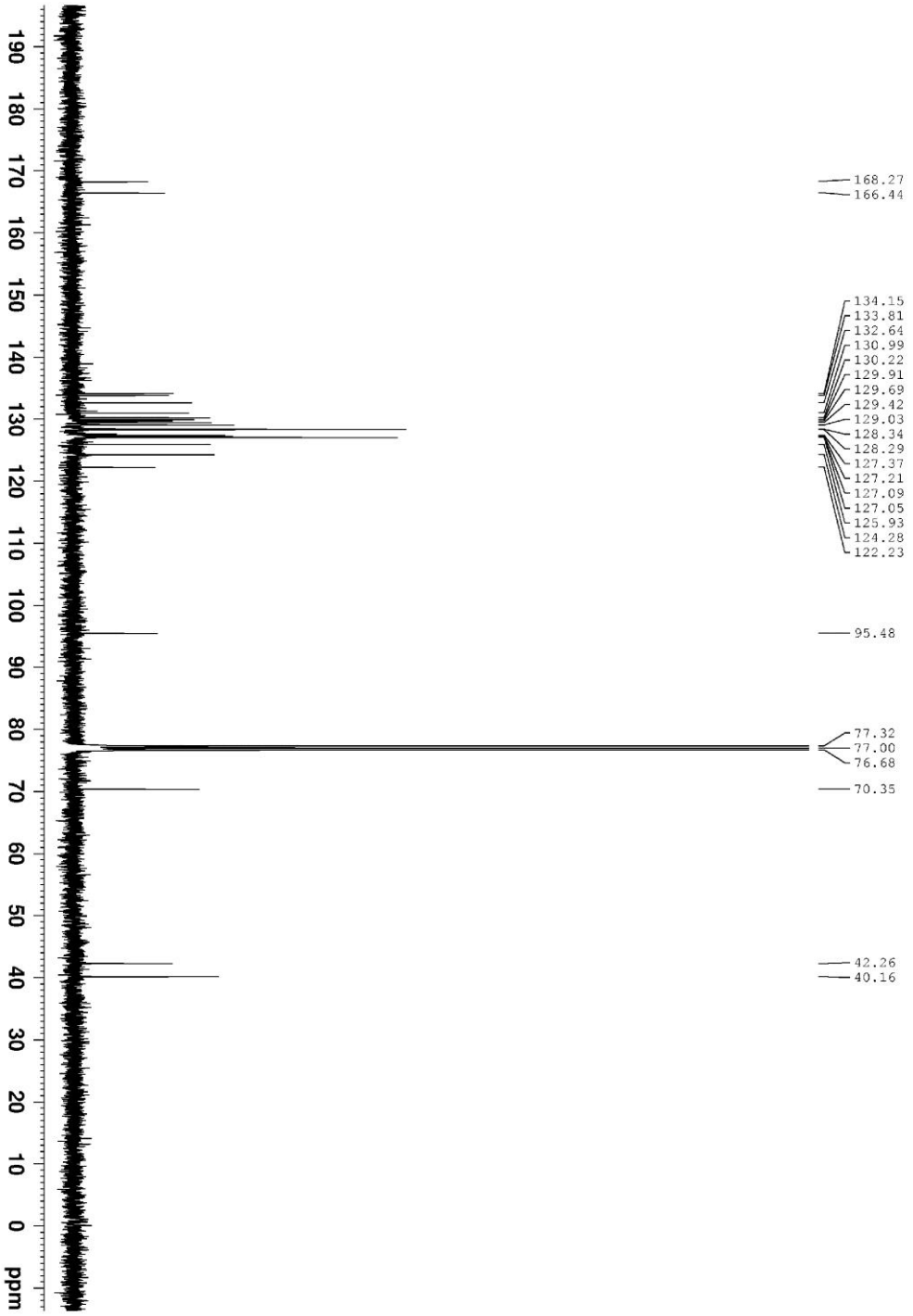

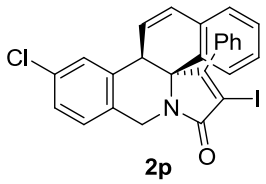

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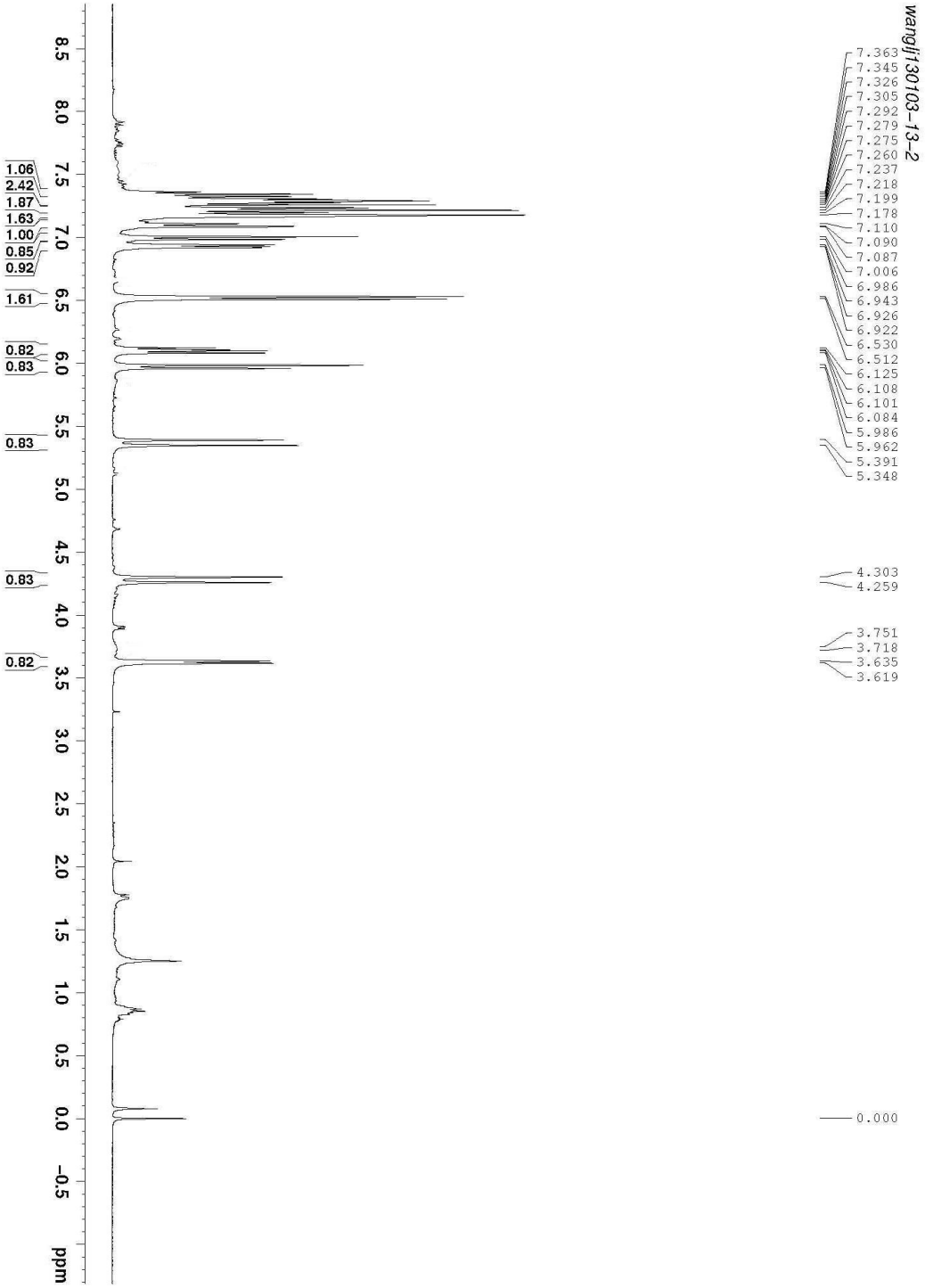

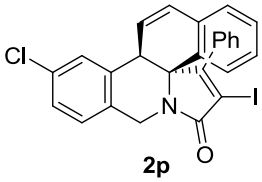

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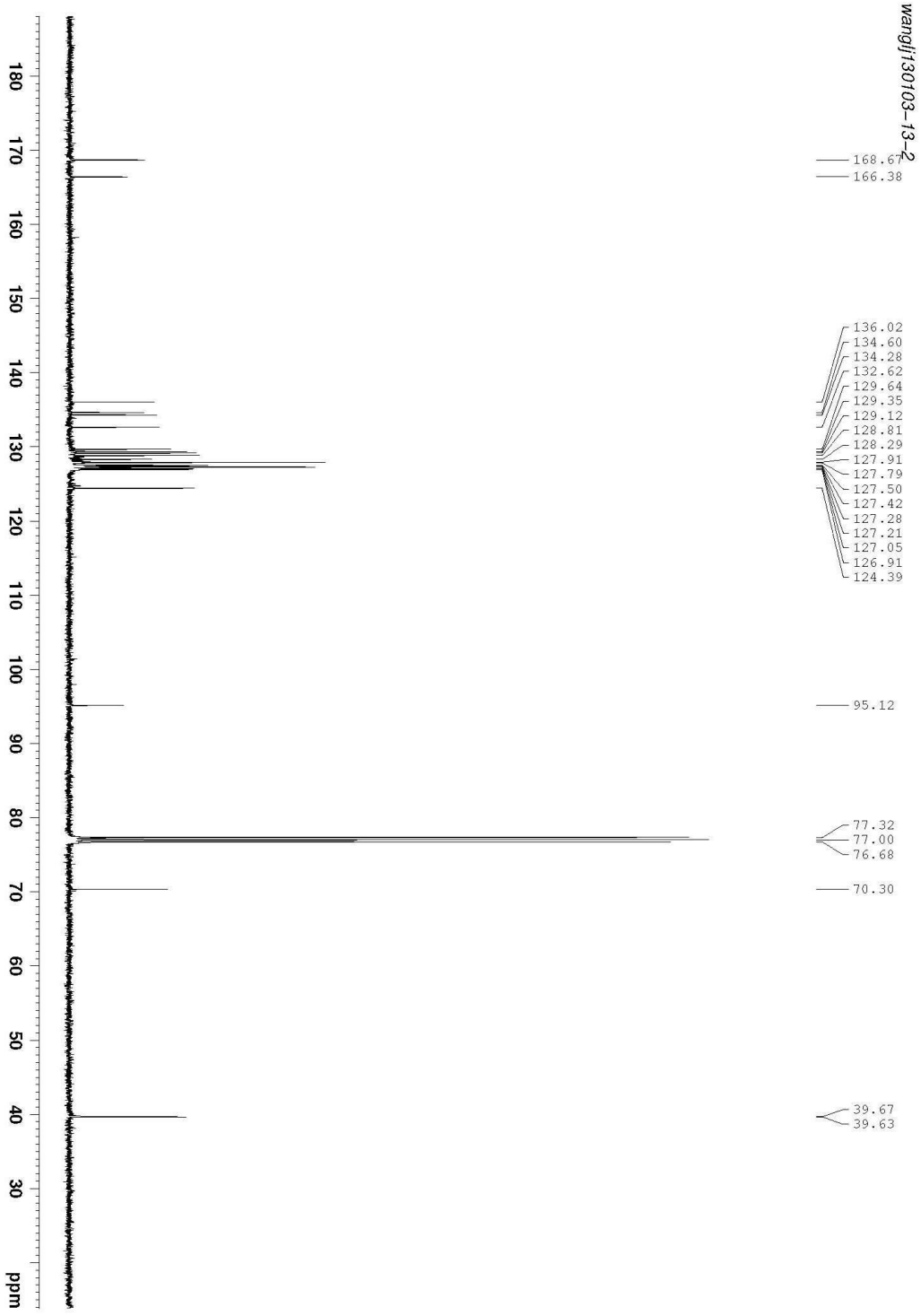

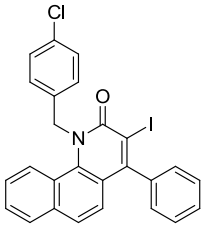

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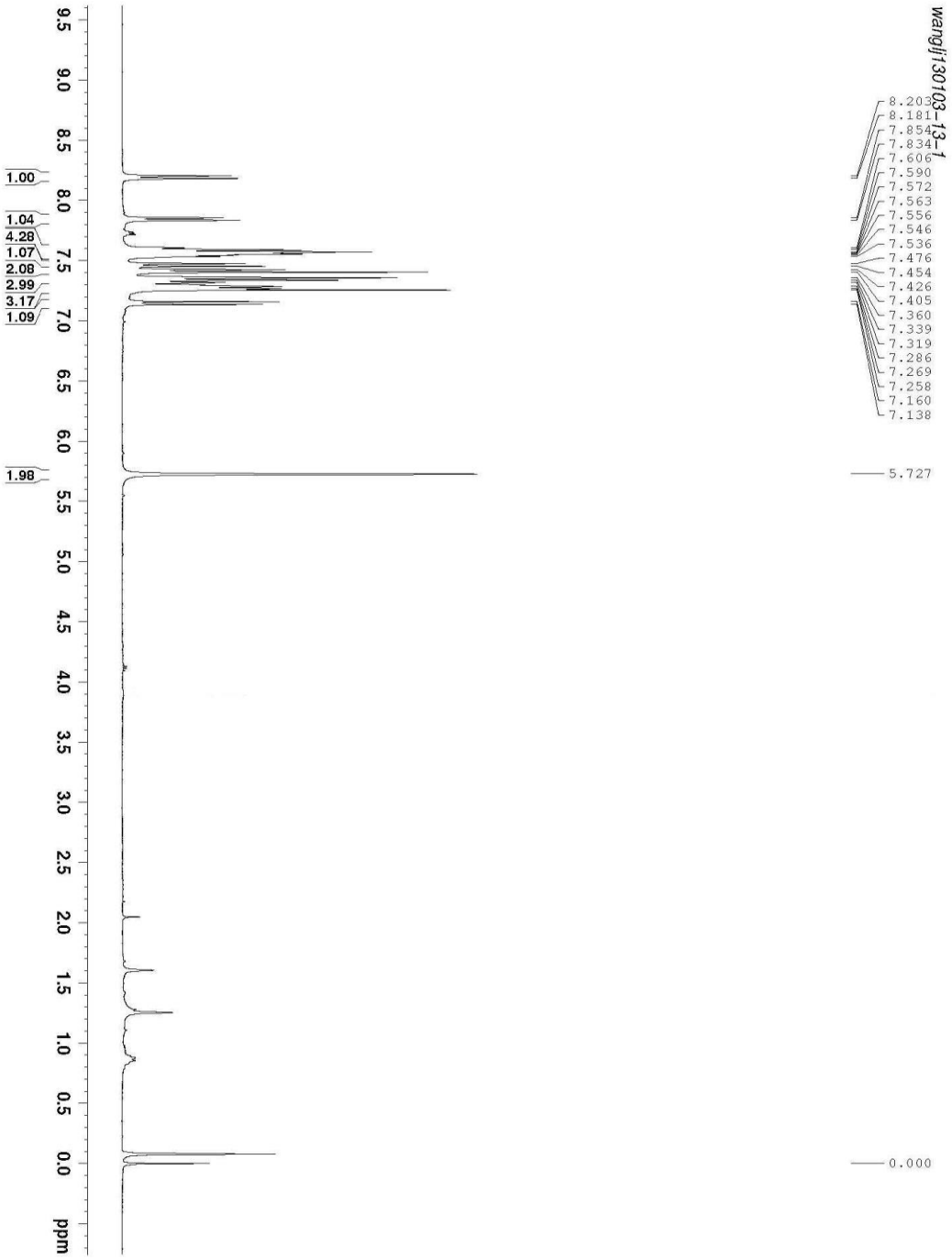

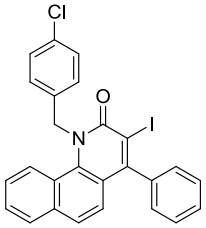

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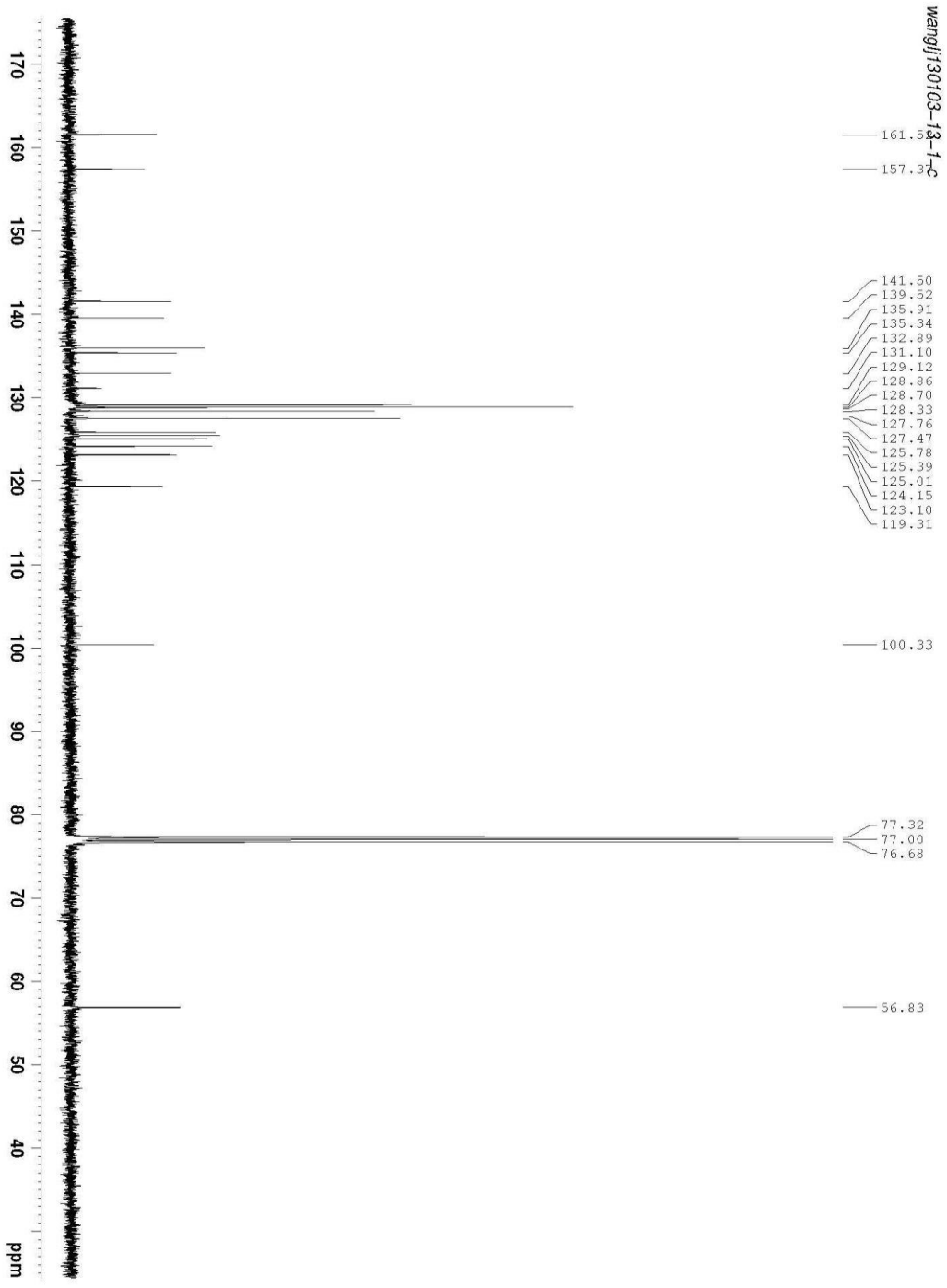

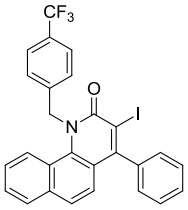

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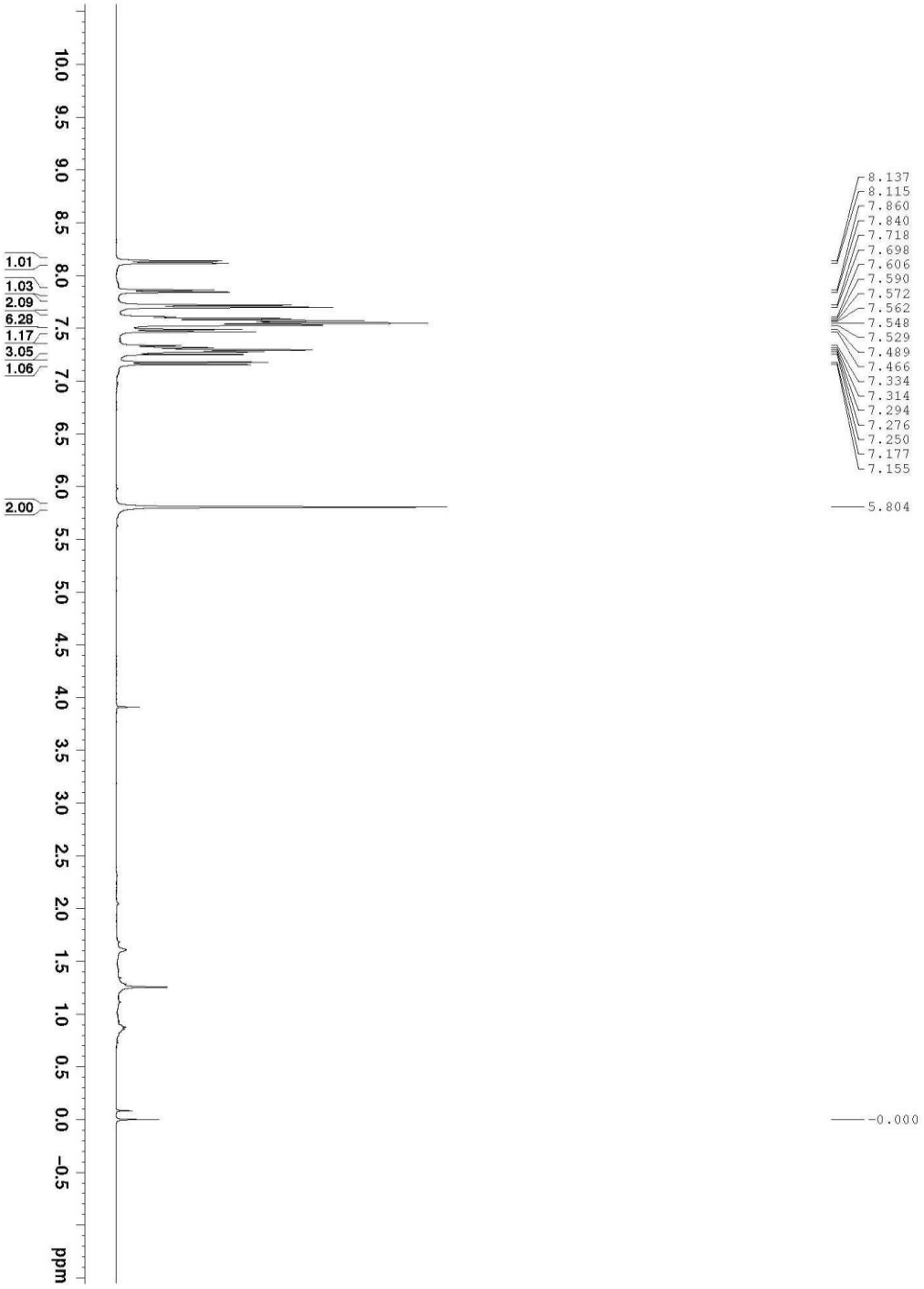

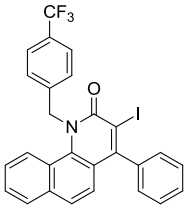

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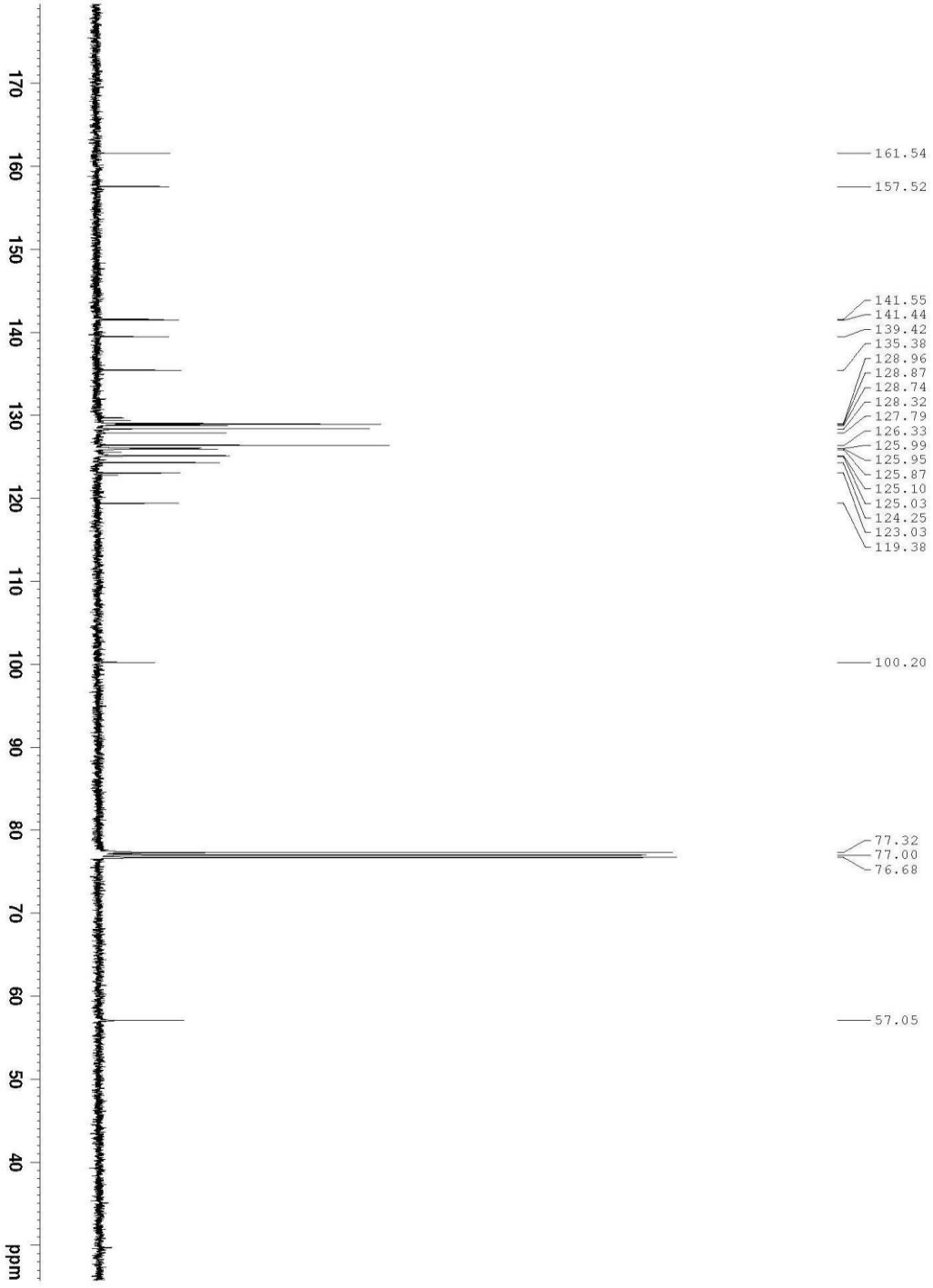

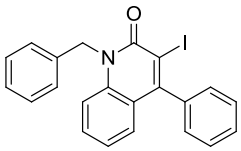

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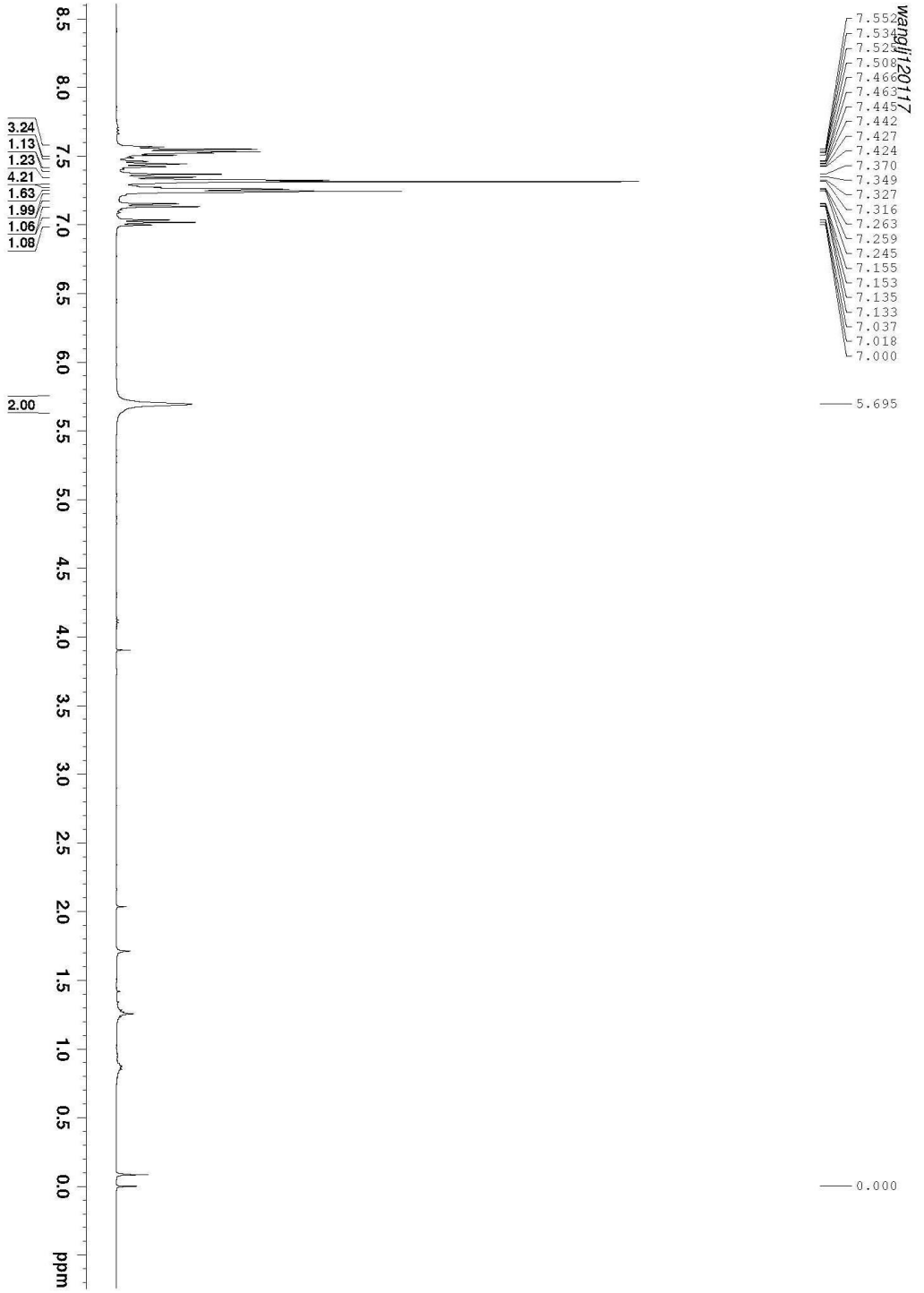

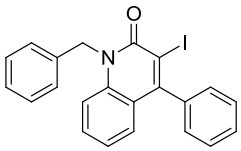

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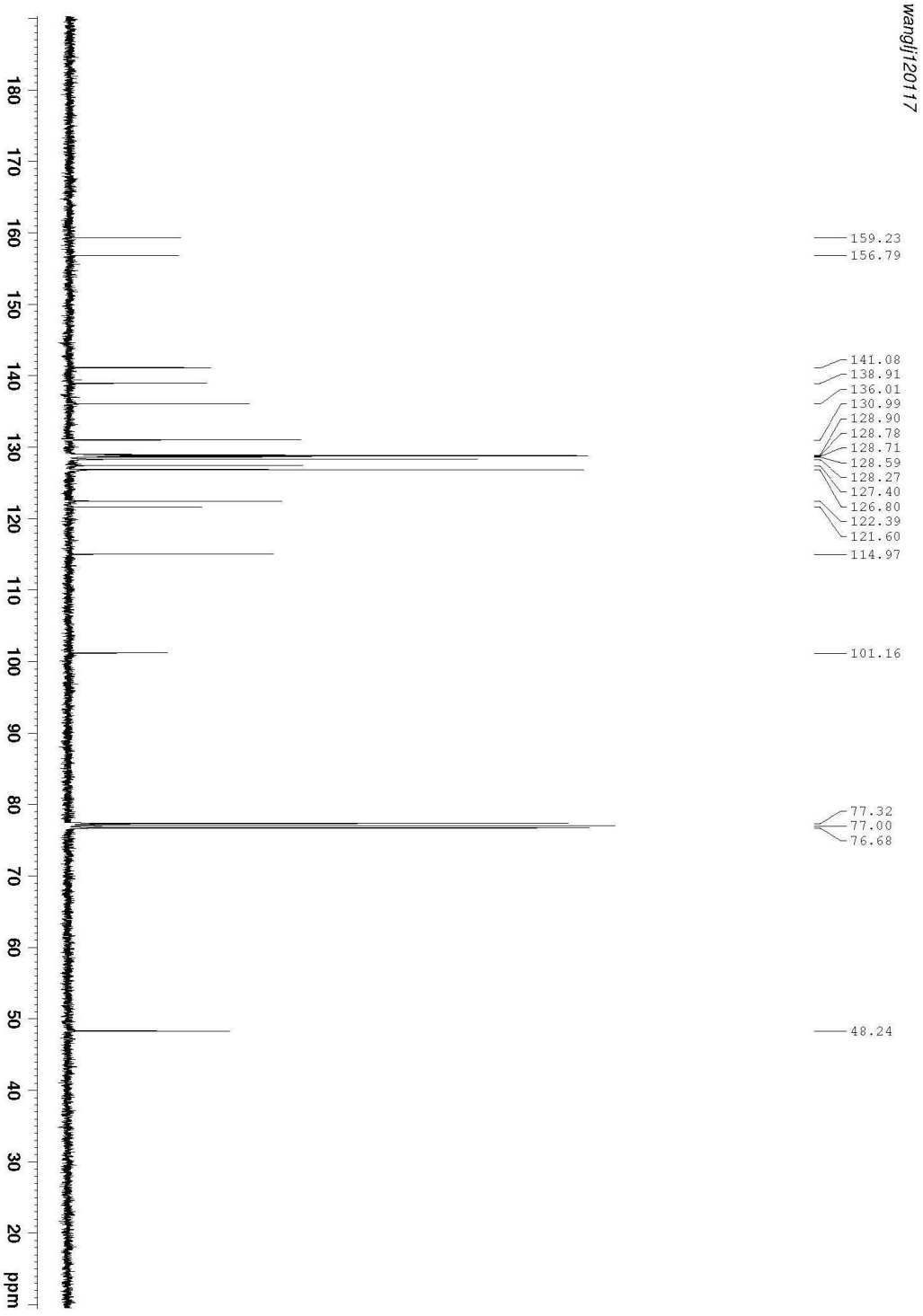

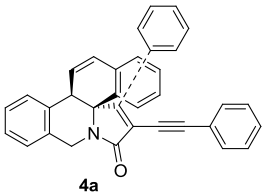

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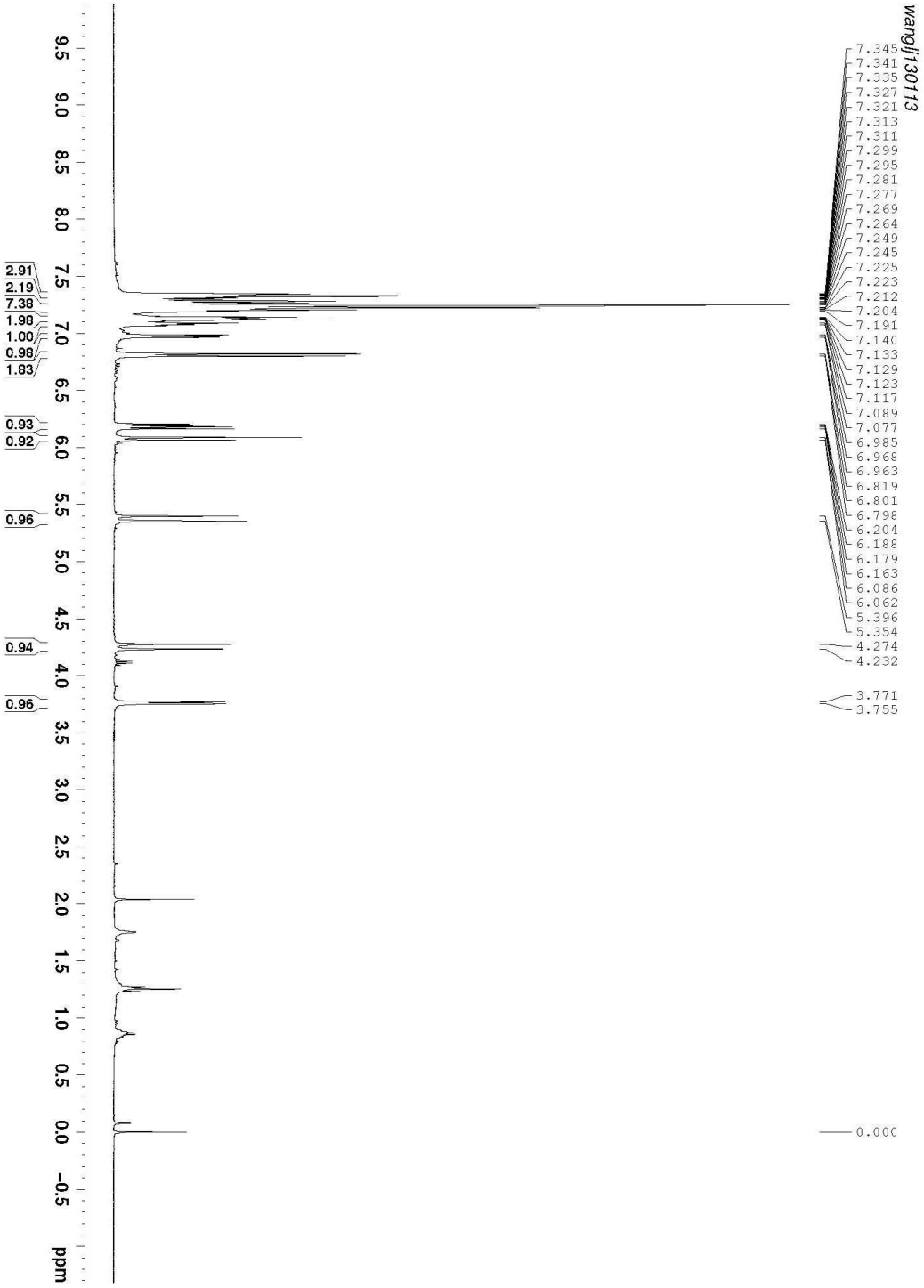

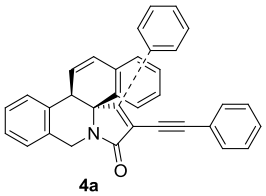

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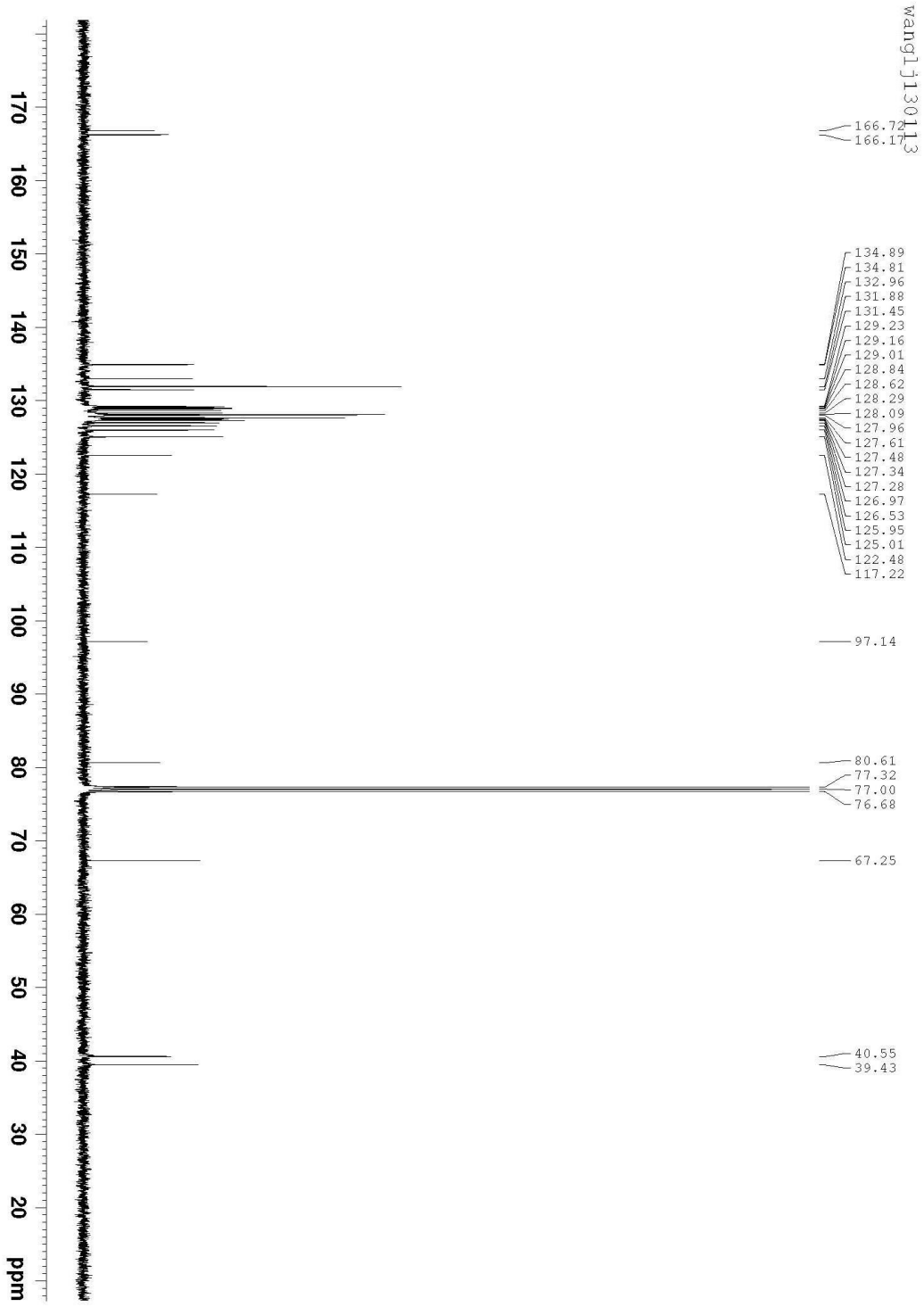

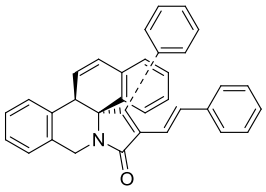
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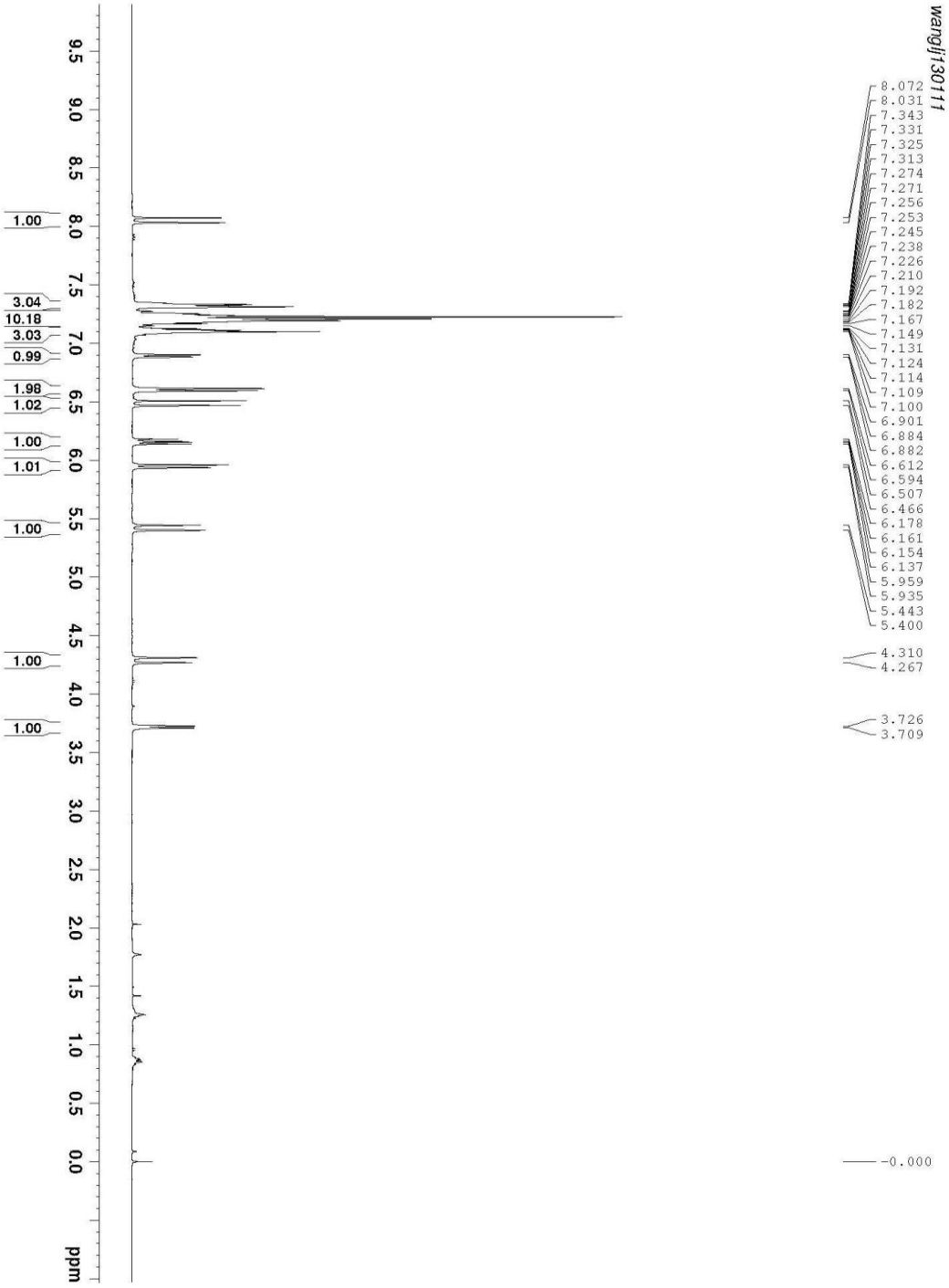
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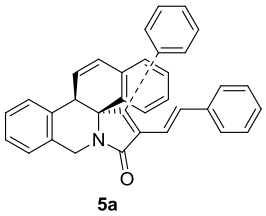

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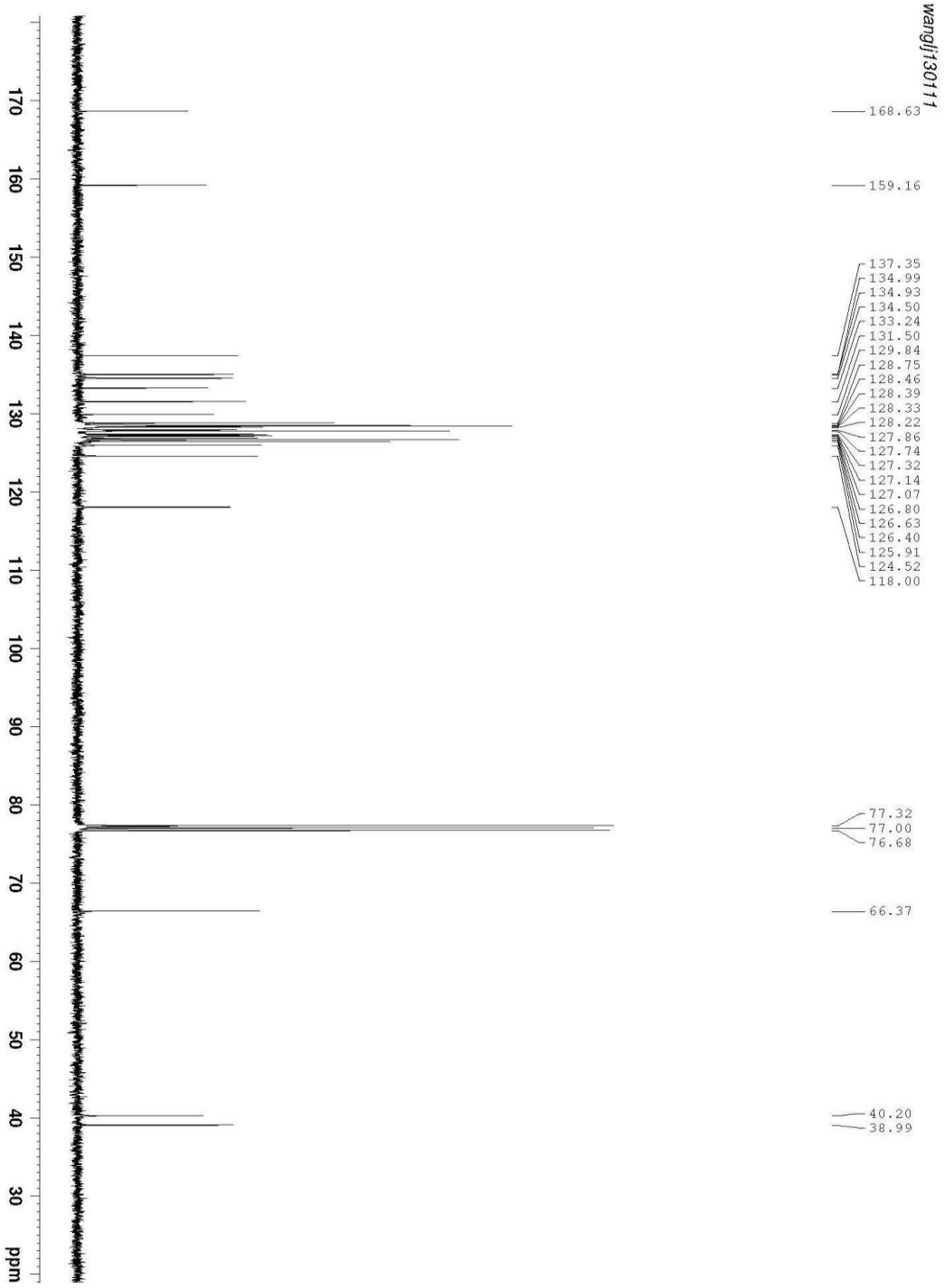

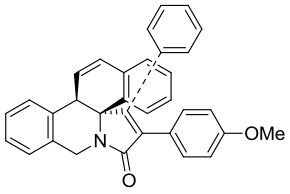

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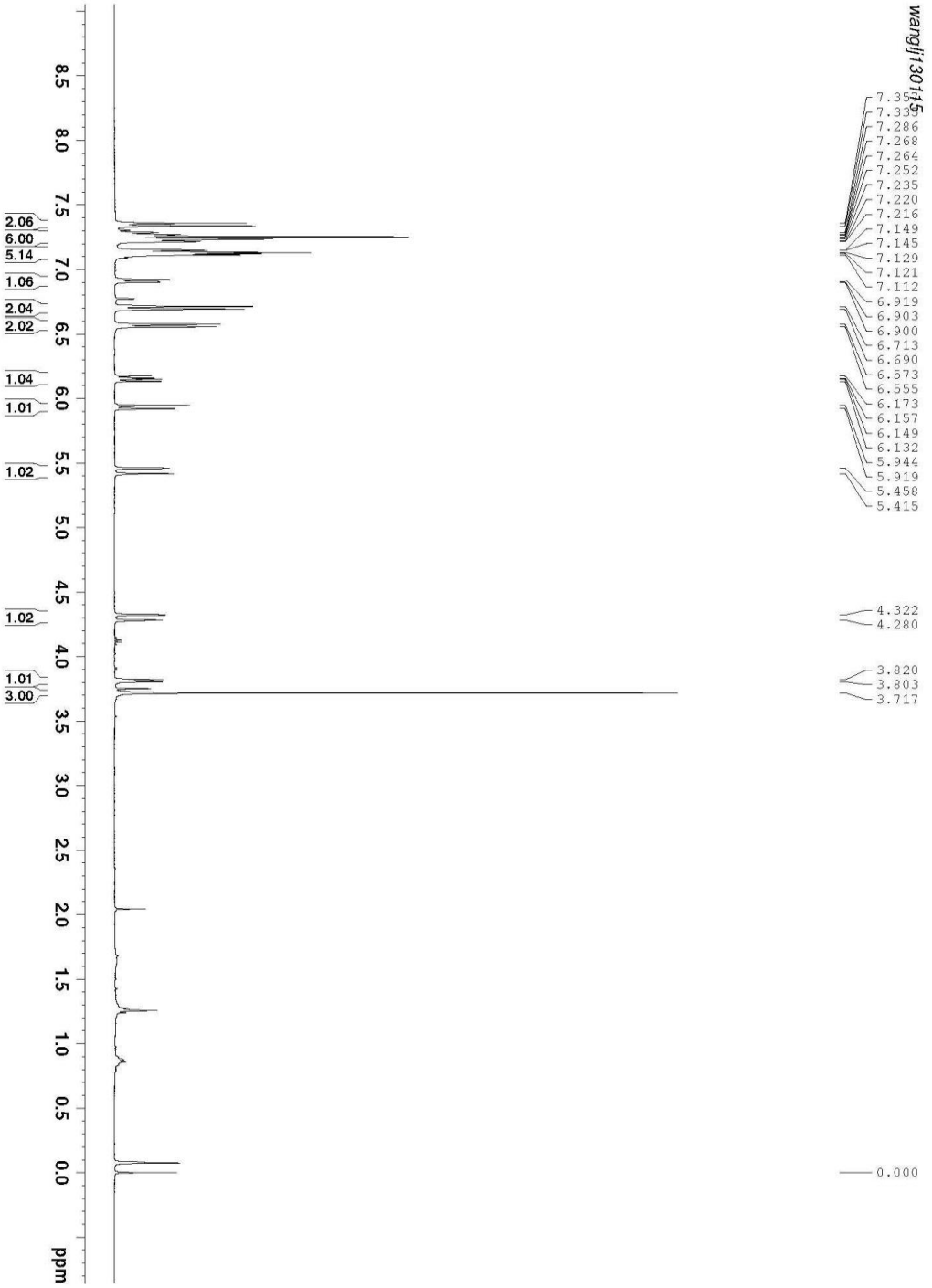

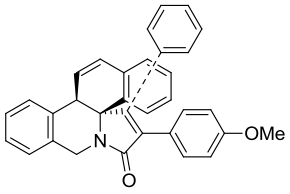

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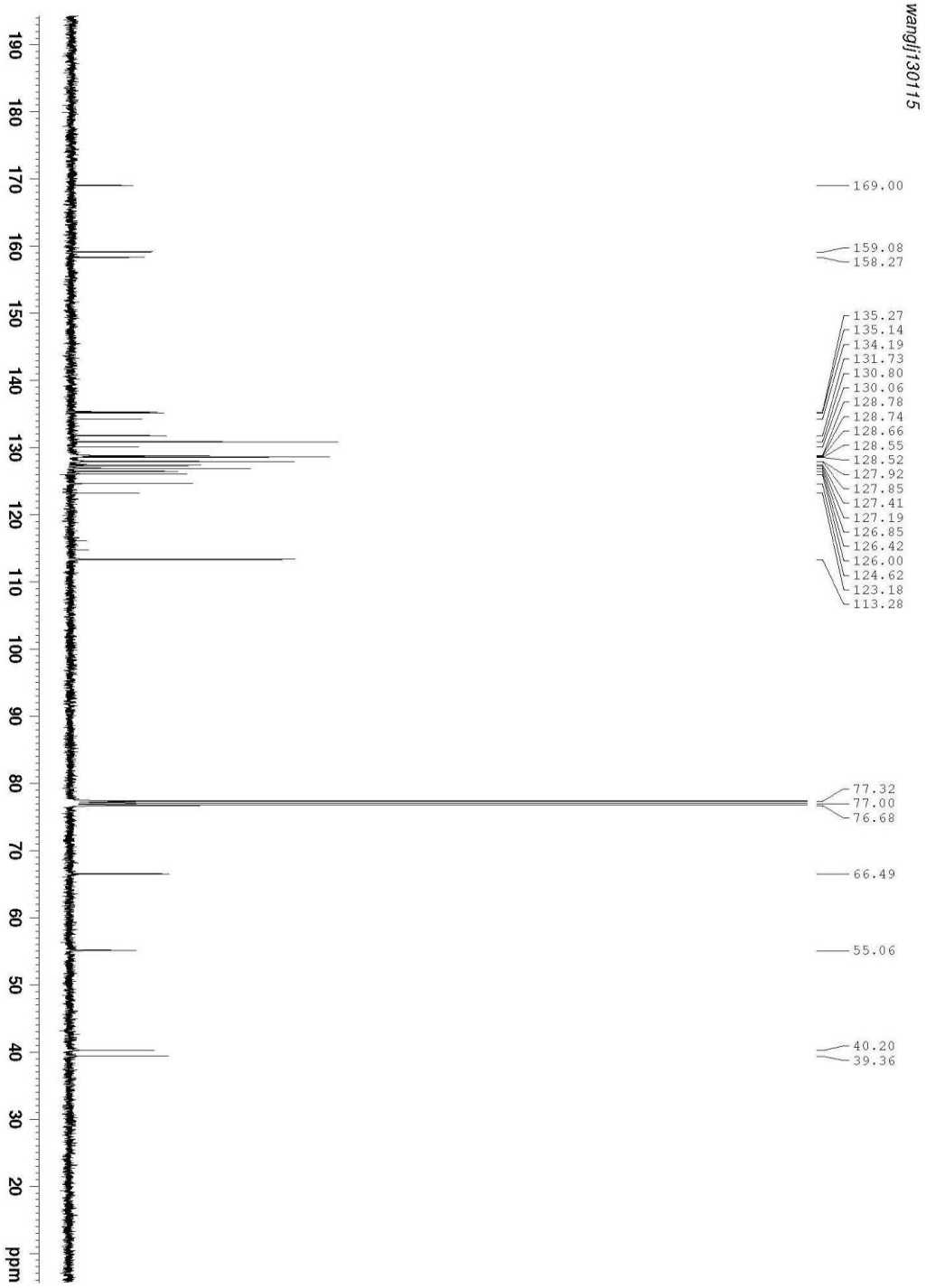

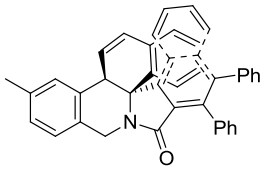

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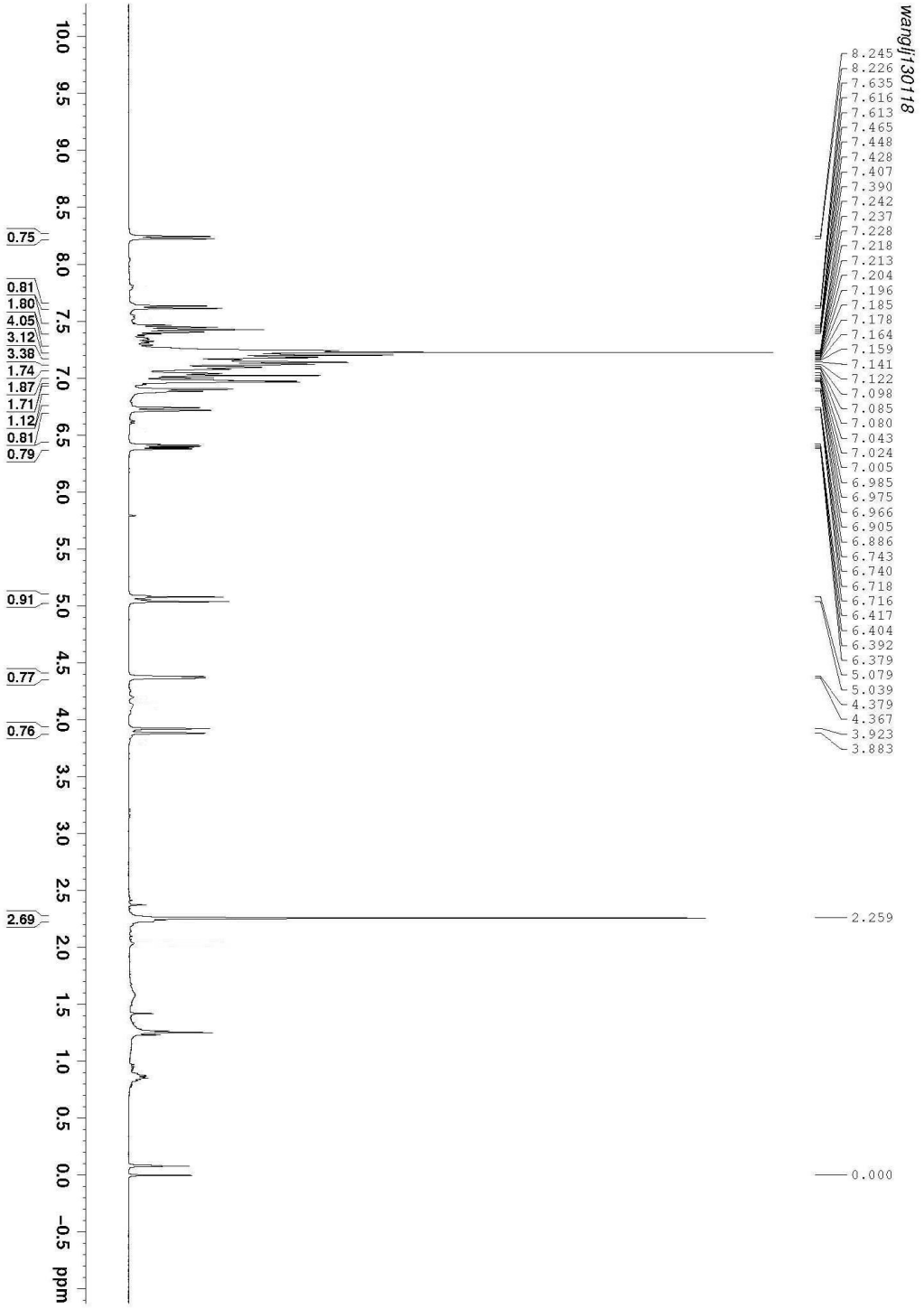

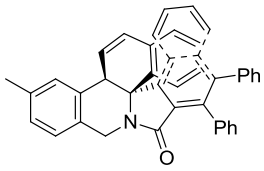

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