Supporting Information for:

Decarbonylative Cycloaddition of Phthalimides with 1,3-Dienes

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Contents

Instrumentation and Chemicals

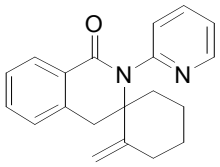
All manipulations of oxygen- and moisture-sensitive materials were conducted in a dry box or with a standard Schlenk technique under a purified argon atmosphere. Nuclear magnetic resonance spectra were taken on Varian UNITY INOVA 500 (^{1}H, 500 MHz; ^{13}C, 125.7 MHz) spectrometer using tetramethylsilane (^{1}H) as an internal standard. ^{1}H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, sext = sextet, sept = septet, br = broad, m = multiplet), coupling constants (Hz), integration, and identification. GC-MS analyses and High-resolution mass spectra were obtained with a JEOL JMS-700 spectrometer by electron ionization at 70 eV. Preparative recycling gel permeation chromatography (GPC) was performed with JAI LC-908 equipped with JAIGEL-1H and -2H columns (toluene as an eluent). Elemental analyses were carried out with a YANAKO MT2 CHN CORDER machine at Kyoto University Elemental Analysis Center. Infrared spectra (IR) spectra were determined on a SHIMADZU FTIR-8200PC spectrometer. In-situ IR spectra were obtained with Mettler Toledo ReactIR 45M equipped with AgX Fiber (9.5 mm). Melting points were determined using a YANAKO MP-500D. TLC analyses were performed by means of Merck Kieselgel 60 F_{254} (0.25 mm) Plates. Visualization was accomplished with UV light (254 nm) and/or an aqueous alkaline $KMnO_4$ solution followed by heating. Flash column chromatography was carried out using Kanto Chemical silica gel (spherical, 40–50 μm). Unless otherwise noted, commercially available reagents were used without purification. Toluene was purchased from Wako Pure Chemical Co. stored over slices of sodium. Bis(1,5-cyclooctadiene)nickel and trimethylphosphine were purchased from Strem Chemicals, Inc. *N*-Arylphthalimides were readily prepared by dehydration of aryl amine and phthalic anhydride.

Experimental Procedure for the Nickel-catalyzed Cycloaddition.

General procedure. The reaction was performed in a 50 mL round-bottomed flask equipped with a Teflon-coated magnetic stirrer bar and Dimrotho reflux condenser. The top of condenser was connected with a balloon filled with argon gas (ca. 1 atm). An *N*-Arylphthalimide (0.5 mmol) and an 1,2-dimethylenecyclohexane (0.55 mmol) were added to a solution of Bis(1,5-dicyclooctadiene)nickel (14 mg, 0.05 mmol) and trimethylphosphine (15 mg, 0.20 mmol) in 1,4-dioxane (20 mL) in a dry box. The flask was taken outside the dry box and heated at 100 ℃ for the indicated time under argon atmosphere. The resulting reaction mixture was cooled to ambient temperature and filtered through a silica gel pad, concentrated in vacuo. The residue was purified by flash silica gel column chromatography (20 g, 2x15 cm, hexane/ethyl acetate = 5:1) to give the corresponding cycloaddition products.

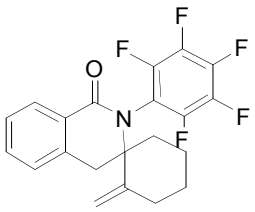
Characterization Data for Products.

2-Methylene-2'-(pyridin-2-yl)-2',4'-dihydro-1'H-spiro[cyclohexane-1,3'-isoquinolin]-1'-one (3aa).



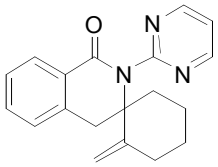
Yield: 78%, white powder. Mp. 65 °C (hexane/ethyl acetate). TLC: R_f 0.46 (hexane/ethyl acetate = 5:1). ^{1}H NMR ($CDCl_3$) δ 8.62 (d, *J* = 6.5 Hz, 1H; Ar–H), 8.06 (d, *J* = 8.0 Hz, 1H; Ph–H), 7.77 (dd, *J* = 8.0, 8.0 Hz, 1H ; Ar–H), 7.45 (dd, *J* = 7.5, 7.5 Hz, 1H ; Ar–H), 7.36–7.28 (m, 2H ; Ph–H), 7.27 (dd, *J* = 7.0, 7.0 Hz, 1H ; Ph–H), 7.15 (d, *J* = 7.0 Hz, 1H ; Ph–H), 5.13 (s, 1H), 4.94 (s, 1H), 3.41 (dd, *J* = 145.0, 15.5 Hz), 2.28–2.17 (m, 2H), 1.80–1.68 (m, 4H), 1.37–1.23 (m, 2H). ^{13}C NMR ($CDCl_3$) δ 166.3 (C=O), 153.3 (C), 149.1 (CH), 146.7 (C), 137.5 (CH), 135.9 (CH), 132.1 (CH), 129.6 (C), 128.1 (CH), 127.1 (CH_2), 127.0 (CH), 125.1 (CH), 122.7 (CH), 112.4 (C), 65.8 (C), 38.6 (CH_2), 38.1 (CH_2), 33.0 (CH_2), 26.9 (CH_2), 22.4 (CH_2). IR (KBr): 3510, 2936, 1654, 1369 cm^{-1}. MS m/z (%): 305/304/303 (11/40/11 [M^+], 249/248/247(28/100/30), 84(47), 78(13). HRMS Calcd for $C_{20}H_{20}N_2O$; [M^+], 304.1576. Found: m/z 304.1580.

2-Methylene-2'-(perfluorophenyl)-2',4'-dihydro-1'H-spiro[cyclohexane-1,3'-isoquinoli n]-1'-one (3ca).



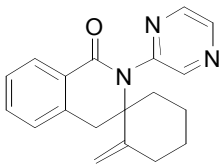
Yield: 59%, colorless oil. TLC :R_f 0.37 (hexane/ethyl acetate = 10:1). ^{1}H NMR ($CDCl_3$) δ 8.02 (d, *J* = 7.5 Hz, 1H ; Ph–H,), 7.47 (dd, *J* = 6.5, 6.5 Hz, 1H ; Ph–H,), 7.37 (dd, *J* = 8.5, 8.5 Hz, 1H ; Ph–H,), 7.18 (d, *J* = 7.5 Hz, 1H ; Ph–H,), 4.99 (s, 1H), 4.85 (s, 1H), 3.39 (dd, *J* = 155, 15.5 Hz, 2H), 2.25–2.11 (m, 2H), 1.83–1.77 (m, 4H), 1.42–1.24 (m, 2H). ^{13}C NMR ($CDCl_3$) δ 165.9 (C=O), 146.2 (dd, *J* =249, 7.6 Hz; C–F), 144.7 (C), 141.1 (dt, *J* = 255, 13.8 Hz; C–F), 137.9 (dt, *J* = 224, 13.0 Hz; C–F), 136.2 (C), 132.7 (CH), 128.9 (C), 128.7 (CH), 128.4 (CH), 127.3 (CH), 127.3 (CH), 112.9 (d, *J* = 8.2 Hz; C–CF), 65.6 (C), 38.3 (CH_2), 37.6 (CH_2), 33.2 (CH_2), 26.9 (CH_2), 22.6 (CH_2). IR (neat): 2940, 1679, 1516, 1356 cm^{-1}. MS m/z (%): 394/393 (85/22) [M^+], 118(100). HRMS Calcd for $C_{21}H_{26}F_5NO$; [M^+], 393.1152. Found: m/z 393.1152.

2-Methylene-2'-(pyrimidin-2-yl)-2',4'-dihydro-1'H-spiro[cyclohexane-1,3'-isoquinolin] -1'-one (3da).



Yield: 69%, white powder. Mp. 105 °C (hexane/ethyl acetate). TLC: R_f 0.20 (hexane/ethyl acetate = 1:5). ^{1}H NMR ($CDCl_3$) δ 8.55 (d, *J* = 5.0 Hz, 2H; Ar–H), 8.07 (d, *J* = 6.5 Hz, 1H; Ph–H), 7.45 (dd, *J* = 8.0, 8.0 Hz, 1H ; Ar–H), 7.33 (dd, *J* = 7.5, 7.5 Hz, 1H ; Ph–H), 7.16 (d, *J* = 7.5 Hz, 1H ; Ph–H), 5.28 (s, 1H), 4.95 (s, 1H), 3.45 (dd, *J* = 171.5, 15.5 Hz), 2.30–2.21 (m, 2H), 1.91–1.72 (m, 4H), 1.38–1.24 (m, 2H). ^{13}C NMR ($CDCl_3$) δ 166.4 (C=O), 160.1 (C), 158.5 (CH), 146.0 (CH), 136.1 (CH), 132.3 (CH), 129.2 (C), 128.2 (CH_2), 127.1 (CH), 119.7 (CH), 112.9 (CH), 109.7 (C), 66.3 (C), 38.3 (CH_2), 37.6 (CH_2), 33.0 (CH_2), 27.0 (CH_2), 22.4 (CH_2). IR (KBr): 3657, 2941, 1654, 1355 cm^{-1}. MS m/z (%): 306/305/304 (11/62/18) [M^+], 262(70), 207(88), 149(85). HRMS Calcd for $C_{19}H_{19}N_3O$; [M^+], 305.1528. Found: m/z 305.1526.

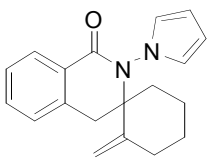
2-Methylene-2'-(pyrazin-2-yl)-2',4'-dihydro-1'H-spiro[cyclohexane-1,3'-isoquinolin]-1'-one (3ea).



Yield: 82%, white powder. Mp. 105 °C (hexane/ethyl acetate). TLC: R_f 0.27 (hexane/ethyl acetate = 1:1). ^{1}H NMR ($CDCl_3$) δ 8.63 (s, 1H; Ar–H), 8.57 (d, *J* = 4.0 Hz, 1H; Ar–H), 8.50 (d, *J* =4.0 Hz, 1H ; Ar–H), 8.05 (d, *J* = 7.5 Hz, 1H ; Ph–H), 7.47 (dd, *J* = 9.0, 9.0 Hz, 1H ; Ph–H), 7.35 (dd, *J* = 7.0, 7.0 Hz, 1H ; Ph–H), 7.17 (d, *J* = 7.5 Hz, 1H ; Ph–H), 5.09 (s, 1H), 4.94 (s, 1H), 3.43 (dd, *J* = 178.6, 15.5 Hz, 2H), 2.29–2.17 (m, 2H), 1.81–1.69 (m, 4H), 1.46–1.27 (m, 2H). ^{13}C NMR ($CDCl_3$) δ 166.6 (C=O), 150.2 (C), 146.7 (CH), 146.4 (CH), 143.2 (C), 142.7 (C), 135.9 (CH), 132.5 (CH_2), 128.9 (C), 128.2 (CH), 127.2 (CH_2), 127.2 (CH), 112.6 (CH), 66.3 (C), 38.5 (CH_2), 38.2 (CH_2), 32.9 (CH_2), 26.8 (CH_2), 22.4 (CH_2)

IR (KBr): 3587, 2934, 1651, 1377 cm^{-1} . MS m/z (%): 305/304/303 (15/55/12) [M^+], 290(62), 262/263(90,13) , 250/249/248(17/100/30), 84(35), 78(10). HRMS Calcd for $C_{19}H_{19}N_3O$; [M^+], 304.1528. Found: m/z 304.1526.

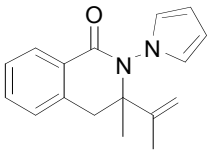
2-methylene-2'-(1H-pyrrol-1-yl)-2',4'-dihydro-1'H-spiro[cyclohexane-1,3'-isoquinolin]-1'-one (3fa).



Yield: 99%, white powder. Mp. 170 °C (cyclohexane/ethyl acetate). TLC: R_f 0.21 (cyclohexane/ethyl acetate = 5:1). ^{1}H NMR ($CDCl_3$) δ 8.01 (d, *J* = 7.5 Hz , 1H; Ph–H), 7.47 (dd, *J* = 7.5, 7.5 Hz, 1H; Ph–H), 7.35 (dd, *J* =7.5, 7.5 Hz, 1H ; Ph–H), 7.15 (d, *J* = 7.5 Hz, 1H ; Ph–H), 6.86 (d, *J* = 7.0 Hz, 2H ; Ar–H), 6.64 (d, *J* = 7.0 Hz, 2H ; Ar–H), 4.91 (s, 1H), 4.88 (s, 1H), 3.40 (dd, *J* = 150.0, 16.0 Hz, 2H), 2.32–2.15 (m, 2H), 1.93–1.66 (m, 4H), 1.30–1.23 (m, 2H). ^{13}C NMR ($CDCl_3$) δ 164.9 (C=O), 145.8 (C), 135.3 (C), 132.6(C), 128.6 (CH), 128.5 (CH), 127.3 (CH), 127.2 (CH), 124.0 (CH), 120.8 (CH_2), 111.8 (CH), 107.2 (CH), 106.5 (CH), 66.5 (C), 39.3 (CH_2), 35.6 (CH_2), 33.0 (CH_2), 26.5 (CH_2), 22.2 (CH_2)

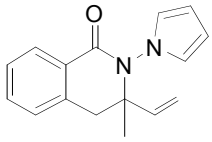
IR (KBr): 2937, 1671, 1458, 1362 cm^{-1} . MS m/z (%): 293/292/291 (31/100/21) [M^+], 277 (30), 249 (51), 226 (35), 90 (56). HRMS Calcd for $C_{20}H_{20}N_2O$; [M^+], 292.1576. Found: m/z 292.1575.

3-methyl-3-(prop-1-en-2-yl)-2-(1H-pyrrol-1-yl)-3,4-dihydroisoquinolin-1(2H)-one (3fb).



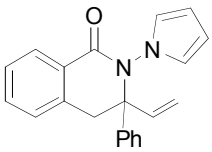
Yield: 89%, white powder. Mp. 125 °C (hexane/ethyl acetate). TLC: R_f 0.52 (hexane/ethyl acetate = 3:1). ^{1}H NMR ($CDCl_3$) δ 8.00 (d, *J* = 8.5 Hz, 1H; Ph–H), 7.37 (dd, *J* = 7.5, 7.5 Hz, 1H; Ph–H), 7.27 (dd, *J* =8.5, 8.5 Hz, 1H ; Ph–H), 7.05 (d, *J* = 7.5 Hz, 1H ; Ph–H), 6.64 (d, *J* = 53.0Hz, 2H ; Ar–H), 6.10 (d, *J* = 5.0 Hz, 2H; Ar–H), 4.91 (s, 1H), 4.87 (s, 1H), 3.21 (dd, *J* = 60.0, 16.0 Hz, 2H), 1.65 (s, 3H), 1.31 (s, 3H). ^{13}C NMR ($CDCl_3$) δ 164.4 (C=O), 144.3 (C), 135.8 (C), 132.7(CH), 128.6 (CH), 128.2 (C), 127.1 (CH_2), 127.1 (CH), 123.6 (CH), 120.7 (CH), 114.6 (CH), 107.1 (CH), 106.6 (CH), 67.6 (C), 41.2 (CH_2), 23.7 (CH_3), 19.8 (CH_3). IR (KBr): 2978, 1668, 1447, 1288 cm^{-1} . MS m/z (%): 267/266/265 (9/41/18) [M^+], 252/251/240(9/100/18), 107(60), 90(48). HRMS Calcd for $C_{17}H_{18}N_2O$; [M^+], 266.1419. Found: m/z 266.1411.

3-methyl-2-(1H-pyrrol-1-yl)-3-vinyl-3,4-dihydroisoquinolin-1(2H)-one (3fc).



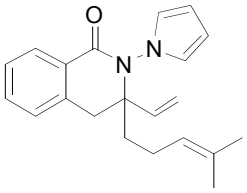
Yield: 86%, white powder. Mp. 110 °C (hexane/ethyl acetate). TLC: R_f 0.44 (hexane/ethyl acetate = 3:1). ^{1}H NMR ($CDCl_3$) δ 8.13 (d, *J* = 7.5 Hz, 1H; Ph–H), 7.50 (dd, *J* = 7.5, 7.5 Hz, 1H; Ph–H), 7.38 (dd, *J* =8.0, 8.0 Hz, 1H ; Ph–H), 7.20 (d, *J* = 8.0 Hz, 1H ; Ph–H), 6.67 (d, *J* = 4.5 Hz, 2H ; Ar–H), 6.20 (d, *J* = 9.5 Hz, 2H ; Ar–H), 5.98 (dd, 1H, *J* = 17.0, 11.0 Hz, 2H), 5.18 (d, *J* = 17.0 Hz, 1H), 5.13 (d, *J* = 11.0 Hz, 1H), 3.27 (dd, *J* = 51.3, 15.5 Hz, 2H), 1.39 (s, 3H). ^{13}C NMR ($CDCl_3$) δ 164.2 (C=O), 139.1 (C), 135.8 (CH), 132.9 (CH), 128.9 (CH_2), 127.8 (C), 127.2 (CH), 127.2 (CH), 122.7 (CH), 121.5 (CH), 115.8 (CH), 107.1 (CH), 106.8 (CH), 64.5 (C), 42.4 (CH_2), 23.4 (CH_3). IR (KBr): 2973, 1675, 1462, 1361 cm^{-1} . MS m/z (%): 251/252/253 (21/67/12) [M^+], 237 (100), 186 (90), 160 (95), 90 (80). HRMS Calcd for $C_{16}H16N_2O$; [M^+], 252.1263 . Found: m/z 252.1284.

3-phenyl-2-(1H-pyrrol-1-yl)-3-vinyl-3,4-dihydroisoquinolin-1(2H)-one (3fd).



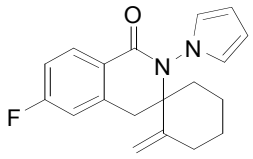
Yield: 56%, white powder. Mp. 110 °C (hexane/ethyl acetate). TLC: R_f 0.42 (hexane/ethyl acetate = 3:1). ^{1}H NMR ($CDCl_3$) δ 8.14 (d, *J* = 6.5 Hz, 1H; Ph–H), 7.40–7.20 (m, 8H; Ph–H), 7.04 (d, *J* =7.5 Hz, 1H ; Ph–H), 6.60 (d, *J* = 66.0 Hz, 2H; Ar–H), 6.06 (d, *J* = 38.0 Hz, 2H; Ar–H), 5.82 (dd, 1H, *J* = 17.5, 11.0 Hz, 2H), 5.43 (d, *J* = 17.5 Hz, 1H), 5.37 (d, *J* = 11.0 Hz, 1H), 3.67 (dd, *J* = 50.0, 15.5 Hz, 2H). ^{13}C NMR ($CDCl_3$) δ 164.6 (C=O), 139.8 (C), 137.4 (CH), 135.3 (C), 132.9 (CH_2), 128.7 (CH), 128.6 (CH), 128.3 (CH), 128.2 (CH), 127.9 (C), 127.3 (CH), 126.7 (CH), 117.5 (CH) , 109.7 (CH), 109.7 (CH), 70.8 (C), 41.9 (CH_2). IR (KBr): 1681, 1559 cm^{-1} . MS m/z (%): 315/314/313 (18/40/18) [M^+], 249/248/247(14/45/43), 215(100),118 (89), 90 (55). HRMS Calcd for $C_{21}H_{18}N_2O$; [M^+], 314.1419. Found: m/z 314.1419.

3-(4-methylpent-3-enyl)-2-(1H-pyrrol-1-yl)-3-vinyl-3,4-dihydroisoquinolin-1(2H)-one (3fe).



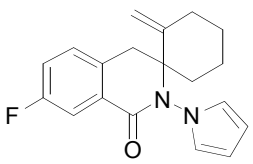
Yield: 91%,colorless oil. TLC: R_f 0.5 (hexane/ethyl acetate = 5:1). ^{1}H NMR ($CDCl_3$) δ 8.10 (d, *J* = 7.0 Hz, 1H; Ph–H), 7.50 (dd, *J* = 8.0, 8.0 Hz, 1H; Ph–H), 7.39 (dd, *J* =8.0, 8.0 Hz, 1H; Ph–H), 7.23 (d, *J* = 7.5 Hz, 1H ; Ph–H), 6.67 (d, *J* = 15.0 Hz, 2H; Ar–H), 6.21 (d, *J* = 13.0 Hz, 2H ; Ar–H), 5.76 (dd, 1H, *J* = 17.5, 11.0 Hz, 2H), 5.21 (d, *J* = 11.0 Hz, 1H), 5.17 (d, *J* = 17.5 Hz, 1H), 4.95 (m, 1H), 3.33 (dd, *J* = 25.0, 16.0 Hz, 2H), 1.98–1.65 (m, 4H), 1.63 (s, 3H), 1.51 (s, 3H). ^{13}C NMR ($CDCl_3$) δ 164.4 (C=O), 137.3 (CH), 135.9 (CH), 132.9 (CH), 132.4 (C), 128.9 (CH_2), 128.0 (CH), 127.5 (C), 127.2 (CH), 123.0 (CH), 122.9 (CH), 122.3 (CH), 107.2 (CH), 107.1 (CH), 66.9 (C), 37.6 (CH_2), 36.6 (CH_3), 25.5 (CH_2), 23.0 (CH_3),17.6 (CH_2). IR (neat): 2967, 1685 cm^{-1} . MS m/z (%): 321/320/319 (6/15/7) [M^+], 251/250(100/19), 215(100), 207 (18), 90 (25). HRMS Calcd for $C_{21}H_{24}N_2O$; [M^+], 320.1889. Found: m/z 320.1864.

6'-fluoro-2-methylene-2'-(1H-pyrrol-1-yl)-2',4'-dihydro-1'H-spiro[cyclohexane-1,3'-isoquinolin]-1'-one (3ga1).



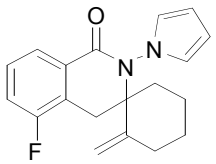
Yield: 54%, white powder. Mp. 152 °C (hexane/ethyl acetate). TLC: R_f 0.37 (hexane/ethyl acetate = 5:1). ^{1}H NMR ($CDCl_3$) δ 8.11 (dd, *J* = 9.0, 6.0 Hz , 1H; Ph–H), 7.15 (d, *J* = 8.0, 1H; Ph–H), 7.03 (dd, *J* =9.0, 9.0 Hz, 1H; Ph–H), 6.85 (d, *J* = 7.5 Hz, 2H; Ar–H), 6.21 (d, *J* = 7.5 Hz, 2H; Ar–H), 4.93 (s, 1H), 4.88 (s, 1H), 3.38 (dd, *J* = 141.5, 15.5 Hz, 2H), 2.35–2.13 (m, 2H), 1.91–1.70 (m, 4H), 1.30–1.22 (m, 2H). ^{13}C NMR ($CDCl_3$) δ 166.3 (C=O), 145.6 (C), 138.1 (CH), 131.5 (CH), 129.0 (C), 124.0 (CH), 120.8 (CH), 115.2 (d, *J* = 23.5 Hz, C–CF), 114.4 (CH), 113.1 (d, *J* = 284 Hz; C–F), 112.0 (CH), 107.4 (CH), 106.7 (CH), 68.5 (C), 39.3 (CH_2), 35.6 (CH_2), 33.1 (CH_2), 26.5 (CH_2), 22.2 (CH_2) . ^{19}F NMR ($CDCl_3$) δ –106.3 . IR (KBr): 2951, 1670, 1491, 1361 cm^{-1} . MS m/z (%): 311/310/309 (17/62/21) [M^+], 267(35), 84(92), 57 (100). HRMS Calcd for $C_{19}H_{19}FN_2O$; [M^+], 310.1481. Found: m/z 310.1481.

7'-fluoro-2-methylene-2'-(1H-pyrrol-1-yl)-2',4'-dihydro-1'H-spiro[cyclohexane-1,3'-isoquinolin]-1'-one (3ga2).



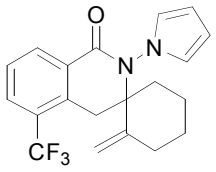
Yield: 14%, white powder. Mp. 152 °C (hexane/ethyl acetate). TLC: R_f 0.37 (hexane/ethyl acetate = 5:1). ^{1}H NMR ($CDCl_3$) δ 7.77 (dd, *J* = 9.0, 2.5 Hz , 1H; Ph–H), 7.17 (d, *J* = 8.0 Hz, 1H; Ph–H), 7.03 (dd, *J* =9.0, 9.0 Hz, 1H; Ph–H), 6.62 (d, *J* = 7.5 Hz, 2H; Ar–H), 6.21 (d, *J* = 7.5 Hz, 2H; Ar–H), 4.93 (s, 1H), 4.86 (s, 1H), 3.36 (dd, *J* = 166.0, 16.0 Hz, 2H), 2.35–2.13 (m, 2H), 1.91–1.70 (m, 4H), 1.30–1.22 (m, 2H). ^{13}C NMR ($CDCl_3$) δ 164.3 (C=O), 145.2 (CH), 138.1 (CH), 131.4 (CH), 129.0 (C), 124.9 (CH), 124.0 (CH), 120.7 (CH), 119.6 (d, *J* = 21.0 Hz; C–CF), 113.2 (d, *J* = 284 Hz; C–F), 111.9 (C), 107.4 (CH), 106.6 (CH), 68.7 (C), 38.6 (CH_2), 35.5 (CH_2), 33.1 (CH_2), 26.5 (CH_2), 22.2 (CH_2). ^{19}F NMR ($CDCl_3$) δ –114.3 . IR (KBr): 2951, 1670, 1491, 1361 cm^{-1} . MS m/z (%): 311/310/309 (17/87/25) [M^+], 295 (23), 267(38), 84 (60), 57 (100). HRMS Calcd for $C_{19}H_{19}FN_2O$; [M^+], 310.1481. Found: m/z 310.1486.

5'-fluoro-2-methylene-2'-(1H-pyrrol-1-yl)-2',4'-dihydro-1'H-spiro[cyclohexane-1,3'-isoquinolin]-1'-one (3ha).



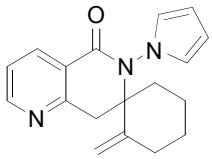
Yield: 99%, white powder. Mp. 130 °C (hexane/ethyl acetate). TLC: R_f 0.45 (hexane/ethyl acetate = 5:1). ^{1}H NMR ($CDCl_3$) δ 7.95 (d, *J* = 7.5 Hz , 1H; Ph–H), 7.32 (dd, *J* = 8.0, 6.5 Hz, 1H; Ph–H), 7.21 (dd, *J* =9.0, 9.0 Hz, 1H; Ph–H), 6.84 (d, *J* = 7.5 Hz, 2H ;Ar–H), 6.21 (d, *J* = 7.5 Hz, 2H ; Ar–H), 4.93 (s, 1H), 4.87 (s, 1H), 3.42 (dd, *J* = 463, 16.0 Hz, 2H), 2.32–2.17 (m, 2H), 1.95–1.77 (m, 4H), 1.31–1.22 (m, 2H). ^{13}C NMR ($CDCl_3$) δ 164.9 (C=O), 159.2 (d, *J* = 245 Hz; C–F), 145.9 (CH), 130.6 (C), 128.2 (CH), 124.3 (CH), 122.4 (C), 122.3 (CH), 120.7 (CH), 119.3 (d, *J* = 21.9 Hz; C–CF), 111.8 (CH), 107.4 (CH), 106.6 (CH), 68.3 (C), 35.7 (CH_2), 33.0 (CH_2), 31.5 (CH_2), 26.5 (CH_2), 22.1 (CH_2). ^{19}F NMR ($CDCl_3$) δ −121.1 . IR (KBr): 2955, 1684, 1473 cm^{-1} . MS m/z (%): 311/310/309 (21/100/25) [M^+], 295 (25) ,267(40), 173 (51), 108 (68), 84(39), 57 (25). HRMS Calcd for $C_{19}H_{19}FN_2O$; [M^+], 310.1481. Found: m/z 310.1488.

2-methylene-2'-(1H-pyrrol-1-yl)-5'-(trifluoromethyl)-2',4'-dihydro-1'H-spiro[cyclohexane-1,3'-isoquinolin]-1'-one (3ia).



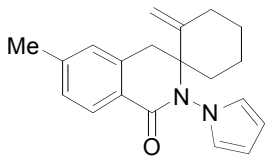
Yield: 99%, white powder. Mp. 123 °C (hexane/ethyl acetate). TLC: R_f 0.43 (hexane/ethyl acetate = 3:1). ^{1}H NMR ($CDCl_3$) δ 8.30 (d, *J* = 7.5 Hz , 1H; Ph–H), 7.80 (d, *J* = 7.5 Hz, 1H; Ph–H), 7.47 (dd, *J* =8.0, 8.0 Hz, 1H; Ph–H), 6.85 (d, *J* = 4.5 Hz, 2H; Ar–H), 6.22 (d, *J* = 4.5 Hz, 2H; Ar–H), 4.92 (s, 1H), 4.86 (s, 1H), 3.58 (dd, *J* = 402, 16.5 Hz, 2H), 2.28–2.14 (m, 2H), 1.95–1.75 (m, 4H), 1.32–1.21 (m, 2H). ^{13}C NMR ($CDCl_3$) δ 163.8 (C=O), 145.8 (C), 134.1 (CH), 132.3 (CH), 130.8 (CH), 129.6 (q, *J* = 147 Hz; C–CF_3), 129.5 (CH), 127.1 (CH), 126.2 (q, *J* = 274 Hz; CF_3), 123.9 (CH), 120.6 (CH), 112.0 (CH), 107.5 (CH), 106.8 (CH), 67.7 (C), 35.7 (CH_2), 35.5 (CH_2), 32.7 (CH_2), 26.5 (CH_2), 22.3 (CH_2). ^{19}F NMR ($CDCl_3$) δ −60.4 . IR (KBr): 2951, 1683, 1559, 1321 cm^{-1} . MS m/z (%): 361/360/359 (17/100/21) [M^+], 317(49), 173 (54), 158(60). HRMS Calcd for $C_{20}H_{19}F_3N_2O$; [M^+], 360.1449. Found: m/z 360.1451.

2-methylene-6'-(1H-pyrrol-1-yl)-6',8'-dihydro-5'H-spiro[cyclohexane-1,7'-[1,6]naphthyridin]-5'-one (3ja).



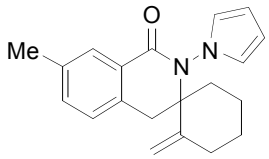
Yield: 47%, yellow oil. TLC: R_f 0.43 (hexane/ethyl acetate = 1:2). ^{1}H NMR ($CDCl_3$) δ 8.63 (d, *J* = 4.5 Hz , 1H; Ph–H), 8.34 (d, *J* = 7.5 Hz, 1H; Ph–H), 7.32 (dd, *J* =7.5, 4.5 Hz, 1H; Ph–H), 6.86 (d, *J* = 4.5 Hz,2H; Ar–H), 6.63 (d, *J* = 4.5 Hz, Ar–H, 2H), 4.92 (s, 1H), 4.88 (s, 1H), 3.59 (dd, *J* = 241.5, 16.5 Hz, 2H), 2.30–2.27 (m, 2H), 1.95–1.75 (m, 4H), 1.32–1.21 (m, 2H). ^{13}C NMR ($CDCl_3$) δ 164.3 (C=O), 155.6 (C), 152.7 (CH), 146.1 (C), 136.1 (CH), 124.2 (C), 123.9 (CH_2), 122.6 (CH), 120.7 (CH), 111.7 (CH), 107.5 (CH), 106.8 (CH), 68.4 (C), 42.2 (CH_2), 35.6 (CH_2), 32.9 (CH_2), 26.4 (CH_2), 22.1 (CH_2) . IR (neat): 2943, 1676, 1540, 1295 cm^{-1} . MS m/z (%): 294/293/292 (22/100/65) [M^+], 265(27), 173 (61), 160 (39) ,84(45), 57 (92). HRMS Calcd for $C_{18}H_{19}N_3O$; [M^+], 293.1528. Found: m/z 293.1526.

6'-methyl-2-methylene-2'-(1H-pyrrol-1-yl)-2',4'-dihydro-1'H-spiro[cyclohexane-1,3'-isoquinolin]-1'-one (3ka1).



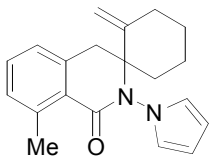
Yield: 45%, white powder. Mp. 138 °C (hexane/ethyl acetate). TLC: R_f 0.51 (hexane/ethyl acetate = 5:1). ^{1}H NMR ($CDCl_3$) δ 7.98 (d, *J* = 8.0 Hz , 1H; Ph–H), 7.27 (d, *J* = 8.0 Hz, 1H; Ph–H), 6.94 (s, Ph–H, 1H), 6.86 (d, *J* = 6.0 Hz, 2H; Ar–H), 6.20 (d, *J* = 6.0 Hz, 2H; Ar–H), 4.91 (s, 1H), 4.88 (s, 1H), 3.35 (dd, *J* = 149.0, 20.0 Hz, 2H), 2.38 (s, 3H), 2.30–2.14 (m, 2H), 1.91–1.71 (m, 4H), 1.28–1.21 (m, 2H). ^{13}C NMR ($CDCl_3$) δ 165.1 (C=O), 146.0 (C), 143.2 (C), 137.0 (CH), 133.4 (CH), 129.0 (C), 128.3 (CH), 128.0 (C), 124.1 (CH_2), 120.9 (CH), 111.8 (CH), 107.2 (CH), 106.4 (CH), 68.6 (C), 39.3 (CH_2), 35.7 (CH_2), 33.1 (CH_2), 26.6 (CH_3), 22.3 (CH_2), 21.6 (CH_2) . IR (KBr): 2945, 1672, 1458, 1288 cm^{-1} .MS m/z (%): 307/306/305 (25/80/21) [M^+], 291 (30), 263 (45), 173 (55), 133/132 (40/36) , 71 (61), 57 (100). HRMS Calcd for $C_{20}H_{22}N_2O$; [M^+], 306.1732 Found: m/z 306.1733.

7'-methyl-2-methylene-2'-(1H-pyrrol-1-yl)-2',4'-dihydro-1'H-spiro[cyclohexane-1,3'-isoquinolin]-1'-one (3ka2).



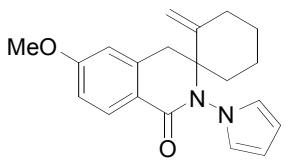
Yield: 45%, white powder. Mp. 138 °C (hexane/ethyl acetate). TLC: R_f 0.51 (hexane/ethyl acetate = 5:1). ^{1}H NMR ($CDCl_3$) δ 7.90 (s, 1H; Ph–H), 7.27 (d, *J* = 8.0 Hz, 1H; Ph–H), 7.05 (d, *J* = 8.0 Hz, 1H; Ph–H), 6.63 (d, *J* = 6.0 Hz, 2H; Ar–H), 6.20 (d, *J* = 6.0 Hz, 2H; Ar–H,), 4.91 (s, 1H), 4.87 (s, 1H), 3.35 (dd, *J* = 148.0, 11.5 Hz, 2H), 2.37 (s, 3H), 2.30–2.14 (m, 2H), 1.91–1.71 (m, 4H), 1.28–1.21 (m, 2H). ^{13}C NMR ($CDCl_3$) δ 165.0 (C=O), 145.9 (C), 143.2 (C), 135.2 (CH), 133.4 (CH), 128.7 (C), 128.1 (CH), 127.2 (C), 124.1 (CH_2), 120.8 (CH), 111.7 (CH), 107.2 (CH), 106.4 (CH), 68.5 (C), 39.0 (CH_2), 35.7 (CH_2), 33.1 (CH_2), 26.6 (CH_3), 22.3 (CH_2), 21.0 (CH_2) . IR (KBr): 2945, 1672, 1458, 1288 cm^{-1} . MS m/z (%): 307/306/305 (28/100/29) [M^+], 291 (35), 263 (45), 173 (83), 133/132 (65/56) , 57 (92). HRMS Calcd for $C_{20}H_{22}N_2O$; [M^+], 306.1732 Found: m/z 306.1733.

8'-methyl-2-methylene-2'-(1H-pyrrol-1-yl)-2',4'-dihydro-1'H-spiro[cyclohexane-1,3'-isoquinolin]-1'-one (3la).



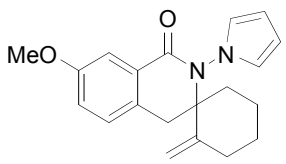
Yield: 69%, white powder. Mp. 185 °C (hexane/ethyl acetate). TLC: R_f 0.40 (hexane/ethyl acetate = 5:1). ^{1}H NMR ($CDCl_3$) δ 7.29 (dd, *J* = 8.0, 8.0 Hz, 1H; Ph–H), 7.14 (d, *J* = 8.0 Hz, 1H; Ph–H), 6.97 (d, *J* = 8.0 Hz, 1H; Ph–H), 6.82 (d, *J* = 4.5 Hz, 2H; Ar–H), 6.20 (d, *J* = 4.5 Hz, 2H; Ar–H), 4.87 (s, 1H), 4.48 (s, 1H), 3.36 (dd, *J* = 134.5 15.5 Hz, 2H), 2.66 (s, 3H), 2.29–2.09 (m, 2H), 1.90–1.69 (m, 4H), 1.28–1.21 (m, 2H). ^{13}C NMR ($CDCl_3$) δ 165.3 (C=O), 146.0 (C), 141.7 (C), 136.3 (CH), 131.6 (CH), 131.1 (CH_2), 126.6 (C), 125.4 (CH), 124.4 (CH), 120.9 (CH), 111.4 (CH), 107.2 (CH), 106.5 (CH), 67.6 (C), 40.3 (CH_2), 35.7 (CH_3), 33.0 (CH_2), 26.7 (CH_2), 22.3 (CH_2), 22.3 (CH_2) . IR (KBr): 2969, 1684, 1574 cm^{-1} . MS m/z (%): 307/306/305 (25/100/28) [M^+], 263 (55), 173 (85), 133/132 (40/36) , 71 (61), 57 (100). HRMS Calcd for $C_{20}H_{22}N_2O$; [M^+], 306.1732 Found: m/z 306.1731.

6'-methoxy-2-methylene-2'-(1H-pyrrol-1-yl)-2',4'-dihydro-1'H-spiro[cyclohexane-1,3'-isoquinolin]-1'-one (3ma1).



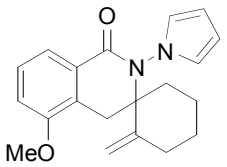
Yield: 70%, white powder. Mp. 142 °C (hexane/ethyl acetate). TLC: R_f 0.22 (toluene/ethyl acetate = 20:1). ^{1}H NMR ($CDCl_3$) δ 8.04 (d, *J* = 8.5 Hz, 1H; Ph–H), 6.86 (dd, *J* = 8.0, 8.0 Hz, 1H; Ph–H), 6.83 (s, 1H; Ph–H), 6.63 (d, *J* = 5.0 Hz, 2H; Ar–H), 6.20 (d, *J* = 6.0 Hz, 2H; Ar–H), 4.92 (s, 1H), 4.89 (s, 1H), 3.84 (s, 3H), 3.35 (dd, *J* = 136.5, 15.5 Hz, 2H), 2.31–2.16 (m, 2H), 1.90–1.70 (m, 4H), 1.29–1.23 (m, 2H). ^{13}C NMR ($CDCl_3$) δ 164.8 (C=O), 163.0 (C), 145.8 (C), 137.4 (CH), 130.8 (CH), 124.1 (CH_2), 121.2 (C), 121.0 (CH), 112.7 (CH), 112.3 (CH), 111.8 (CH), 107.1 (CH), 106.4 (CH), 68.4 (C), 55.3 (CH_3), 39.5 (CH_2), 35.7 (CH_2), 33.1 (CH_2), 26.5 (CH_2), 22.2 (CH_2) . IR (KBr): 2930, 1658, 1499, 1288 cm^{-1} . MS m/z (%): 323/322/321 (21/72/34) [M^+], 279 (58), 207 (40), 173 (40), 149/148 (56/56) , 71 (65), 57 (100). HRMS Calcd for $C_{20}H_{22}N_2O_2$; [M^+], 322.1681 Found: m/z 322.1681.

7'-methoxy-2-methylene-2'-(1H-pyrrol-1-yl)-2',4'-dihydro-1'H-spiro[cyclohexane-1,3'-isoquinolin]-1'-one (3ma2).



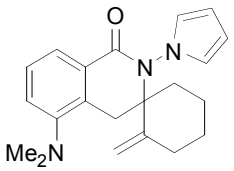
Yield: 30%, white powder. Mp. 115 °C (hexane/ethyl acetate). TLC: R_f 0.22 (toluene/ethyl acetate = 20:1). ^{1}H NMR ($CDCl_3$) δ 7.61 (s, 1H; Ph–H), 7.05 (dd, *J* = 8.5, 8.5 Hz, 1H; Ph–H), 7.00 (d, *J* = 8.5 Hz, 1H; Ph–H), 6.63 (d, *J* = 8.5 Hz, 2H; Ar–H), 6.20 (d, *J* = 8.5 Hz, 2H; Ar–H), 4.92 (s, 1H), 4.87 (s, 1H), 3.83 (s, 3H), 3.33 (dd, *J* = 156.5, 15.5 Hz, 2H), 2.30–2.18 (m, 2H), 1.91–1.67 (m, 4H), 1.28–1.22 (m, 2H). ^{13}C NMR ($CDCl_3$) δ 164.9 (C=O), 158.8 (C), 146.0 (CH), 129.5 (CH), 128.5 (CH), 127.5 (C), 124.0 (C), 120.8 (CH_2), 119.8 (CH), 112.1 (CH), 111.7 (CH), 107.2 (CH), 106.5 (CH), 68.8 (C), 55.5 (CH_3), 38.5 (CH_2), 35.6 (CH_2), 33.1 (CH_2), 26.5 (CH_2), 22.3 (CH_2) . IR (KBr): 2942, 1664, 1507, 1284 cm^{-1} . MS m/z (%): 323/322/321 (19/55/17) [M^+], 307 (21), 279 (22), 173 (21), 149 (47) , 85 (50), 71 (62), 57 (100). HRMS Calcd for $C_{20}H_{22}N_2O_2$; [M^+], 322.1681 Found: m/z 322.1686.

5'-methoxy-2-methylene-2'-(1H-pyrrol-1-yl)-2',4'-dihydro-1'H-spiro[cyclohexane-1,3'-isoquinolin]-1'-one (3na).



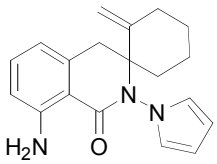
Yield: 99%, white powder. Mp. 118 °C (hexane/ethyl acetate). TLC: R_f 0.50 (hexane/ethyl acetate = 3:1). ^{1}H NMR ($CDCl_3$) δ 7.71 (d, *J* = 8.0 Hz, 1H; Ph–H), 7.29 (dd, *J* = 8.0, 8.0 Hz, 1H; Ph–H), 7.02 (d, *J* = 8.0 Hz, 1H; Ph–H), 6.63 (d, *J* = 5.0 Hz, 2H; Ar–H), 6.20 (d, *J* =5.0 Hz, 2H; Ar–H,), 4.90 (s, 1H), 4.88 (s, 1H), 3.86 (s, 3H), 3.34 (dd, *J* = 633.0, 16.0 Hz, 2H), 2.28–2.20 (m, 2H), 1.93–1.65 (m, 4H), 1.29–1.24 (m, 2H). ^{13}C NMR ($CDCl_3$) δ 164.9 (C=O), 155.8 (C), 146.4 (C), 129.7 (C), 127.5 (CH), 124.0 (CH), 120.8 (CH_2), 120.4 (CH), 120.4 (CH),114.0 (CH), 111.3 (CH), 107.2 (CH), 106.4 (CH), 68.2 (C), 55.6 (CH_3), 35.9 (CH_2), 33.1 (CH_2), 32.1 (CH_2), 26.6 (CH_2), 22.1 (CH_2) . IR (KBr): 2940, 1678, 1482, 1266 cm^{-1} . MS m/z (%): 323/322/321 (11/44/16) [M^+], 307 (16), 279 (24), 149 (27) , 85 (50), 71 (64), 57 (100). HRMS Calcd for $C_{20}H_{22}N_2O_2$; [M^+], 322.1681 Found: m/z 322.1687.

5'-(dimethylamino)-2-methylene-2'-(1H-pyrrol-1-yl)-2',4'-dihydro-1'H-spiro[cyclohexane-1,3'-isoquinolin]-1'-one (3oa).



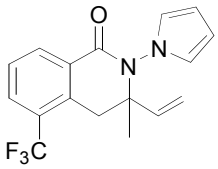
Yield: 31%, white powder. Mp. 212 °C (hexane/ethyl acetate). TLC: R_f 0.25 (hexane/ethyl acetate = 5:1). ^{1}H NMR ($CDCl_3$) δ 7.89 (d, *J* = 7.0 Hz, 1H; Ph–H), 7.26 (m, 2H; Ph–H), 7.02 (d, *J* = 8.0 Hz, 1H; Ph–H), 6.75 (d, *J* = 107.5 Hz, 2H; Ar–H,), 6.20 (d, *J* = 8.5 Hz, 2H ; Ar–H), 4.85 (s, 1H), 4.84 (s, 1H), 3.44 (dd, *J* = 587.0, 16.0 Hz, 2H), 2.69 (s, 6H), 2.23–2.10 (m, 2H), 1.96–1.75 (m, 4H), 1.30–1.22 (m, 2H). ^{13}C NMR ($CDCl_3$) δ 165.4 (C=O), 152.5 (C), 146.4 (CH), 130.0 (C), 129.6 (CH), 127.3 (CH), 124.1 (CH_2), 123.0 (C), 122.8 (CH), 120.9 (CH), 111.2 (CH), 107.2 (CH), 106.5 (CH), 68.1 (C), 44.5 (CH_2), 35.9 (CH_3), 34.6 (CH_2), 33.1 (CH_2), 26.7 (CH_2), 22.3 (CH_2) . IR (KBr): 2944, 1675, 1366 cm^{-1} . MS m/z (%): 335/334/333 (26/100/16) [M^+], 320(19), 292 (24), 255 (50) , 85 (45), 71 (58), 57 (94). HRMS Calcd for $C_{21}H_{25}N_3O$; [M^+], 335.1998 Found: m/z 335.1993.

8'-amino-2-methylene-2'-(1H-pyrrol-1-yl)-2',4'-dihydro-1'H-spiro[cyclohexane-1,3'-isoquinolin]-1'-one (3pa).



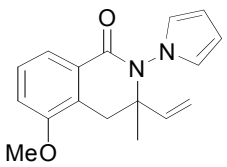
Yield: 92%, white powder. Mp. 140 °C (hexane/ethyl acetate). TLC: R_f 0.41 (hexane/ethyl acetate = 3:1). ^{1}H NMR ($CDCl_3$) δ 7.13 (dd, *J* = 8.0, 8.0 Hz, 1H; Ph–H), 6.75 (d, *J* = 122.0 Hz, 2H; Ar–H), 6.51 (d, *J* = 8.0 Hz, 1H; Ph–H), 6.34 (d, *J* = 8.0 Hz, 1H; Ph–H,), 6.23–6.20 (m, 2H; Ar–H), 6.01 (br, 2H; NH_2), 4.94 (s, 1H), 4.89 (s, 1H), 3.28 (dd, *J* = 143.5, 15.5 Hz, 2H), 2.31–2.05 (m, 2H), 1.88–1.67 (m, 4H), 1.31–1.21 (m, 2H). ^{13}C NMR ($CDCl_3$) δ 167.3 (C=O), 150.3 (C), 145.6 (C), 136.7 (CH), 133.3 (CH), 124.2 (CH_2), 121.0 (CH), 115.5 (C), 115.4 (CH), 111.3 (CH), 109.5 (C), 107.2 (CH), 106.4 (CH), 67.8 (C), 40.0 (CH_2), 35.8 (CH_2), 32.9 (CH_2), 26.5 (CH_2), 22.3 (CH_2) . IR (KBr): 2937, 1654, 1362 cm^{-1} . MS m/z (%): 308/307/306 (23/100/16) [M^+], 279 (13), 264 (28), 227 (41) , 174/173 (38/37), 84 (60), 57 (34). HRMS Calcd for $C_{19}H_{21}N_3O$; [M^+], 307.1685 Found: m/z 306.1687.

3-methyl-2-(1H-pyrrol-1-yl)-5-(trifluoromethyl)-3-vinyl-3,4-dihydroisoquinolin-1(2H)-one (3ic).



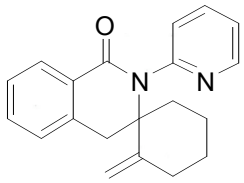
Yield: 58%, colorless oil. TLC: R_f 0.25 (hexane/ethyl acetate = 5:1). ^{1}H NMR ($CDCl_3$) δ 8.35 (d, *J* =7.5 Hz, 1H; Ph–H), 7.84 (d, *J* = 7.5 Hz, 1H; Ar–H), 7.50 (dd, *J* = 7.5 ,7.5 Hz, 1H; Ph–H), 6.80 (d, *J* = 28.5 Hz, 2H; Ar–H,), 6.22 (d, *J* = 4.0 Hz, 2H; Ar–H), 5.86 (dd, *J* = 17.0, 11.0 Hz, 2H), 5.19 (d, *J* = 17.0 Hz, 1H), 5.15 (d, *J* = 11.0 Hz, 1H), 3.56 (dd, *J* = 101.0 , 7.0 Hz, 2H), 1.41 (s, 3H). ^{13}C NMR ($CDCl_3$) δ 163.1 (C=O), 141.0 (C), 138.5 (CH), 134.7 (C), 132.6 (CH), 132.3 (CH), 129.9 (q, *J* = 78.1 Hz; C-CF_3), 127.2 (CH), 122.7 (q, *J* = 251 Hz; CF_3), 120.6 (CH_2), 116.3 (CH), 115.6 (CH), 107.5 (CH), 107.2 (CH), 66.3 (C), 38.9 (CH_2), 23.7 (CH_3) . ^{19}F NMR ($CDCl_3$) δ –60.98 . IR (neat): 2975, 1683, 1527 cm^{-1} . MS m/z (%): 321/320/319 (23/100/28) [M^+], 207 (48), 264 (28), 84 (70). HRMS Calcd for $C_{17}H_{15}F_3N_2O$; [M^+], 320.1136 Found: m/z 306.1123.

5-methoxy-3-methyl-2-(1H-pyrrol-1-yl)-3-vinyl-3,4-dihydroisoquinolin-1(2H)-one (3nc).



Yield: 64%, white powder. Mp. 118 °C (hexane/ethyl acetate). TLC: R_f 0.14 (hexane/ethyl acetate = 5:1). ^{1}H NMR ($CDCl_3$) δ 7.70 (d, *J* =8.0 Hz, 1H; Ph–H), 7.33 (dd, *J* = 8.0, 8.0 Hz, 1H; Ph–H), 7.05 (d, *J* = 8.0 Hz, 1H; Ph–H), 6.69 (d, *J* = 26.5 Hz, 2H; Ar–H,), 6.22 (d, *J* = 4.0 Hz, 2H; Ar–H), 5.98 (dd, *J* = 17.5, 11.5 Hz, 2H), 5.16 (d, *J* = 17.5 Hz, 1H), 5.11 (d, *J* = 11.5 Hz, 1H), 3.87 (s, 3H), 3.25 (dd, *J* = 99.0 , 17.0 Hz, 2H), 1.38 (s, 3H). ^{13}C NMR ($CDCl_3$) δ 164.2 (C=O), 155.9 (C), 142.1 (C), 139.4 (CH), 129.0 (C), 127.6 (CH), 122.8 (CH), 121.5 (CH), 120.7 (CH_2), 115.2 (CH), 114.2 (CH), 107.1 (CH), 106.9 (CH), 66.9 (C), 55.7 (CH_3), 35.5 (CH_2), 23.8 (CH_3). IR (KBr): 2936, 1675, 1559 cm^{-1} . MS m/z (%): 283/282/281 (5/33/16) [M^+], 267 (26), 254 (21), 216 (41), 148/149 (49/26), 57 (100). HRMS Calcd for $C_{17}H_{18}N_2O_2$; [M^+], 282.1368 Found: m/z 282.1365.

^{1}H NMR and ^{13}C NMR Spectra of Products



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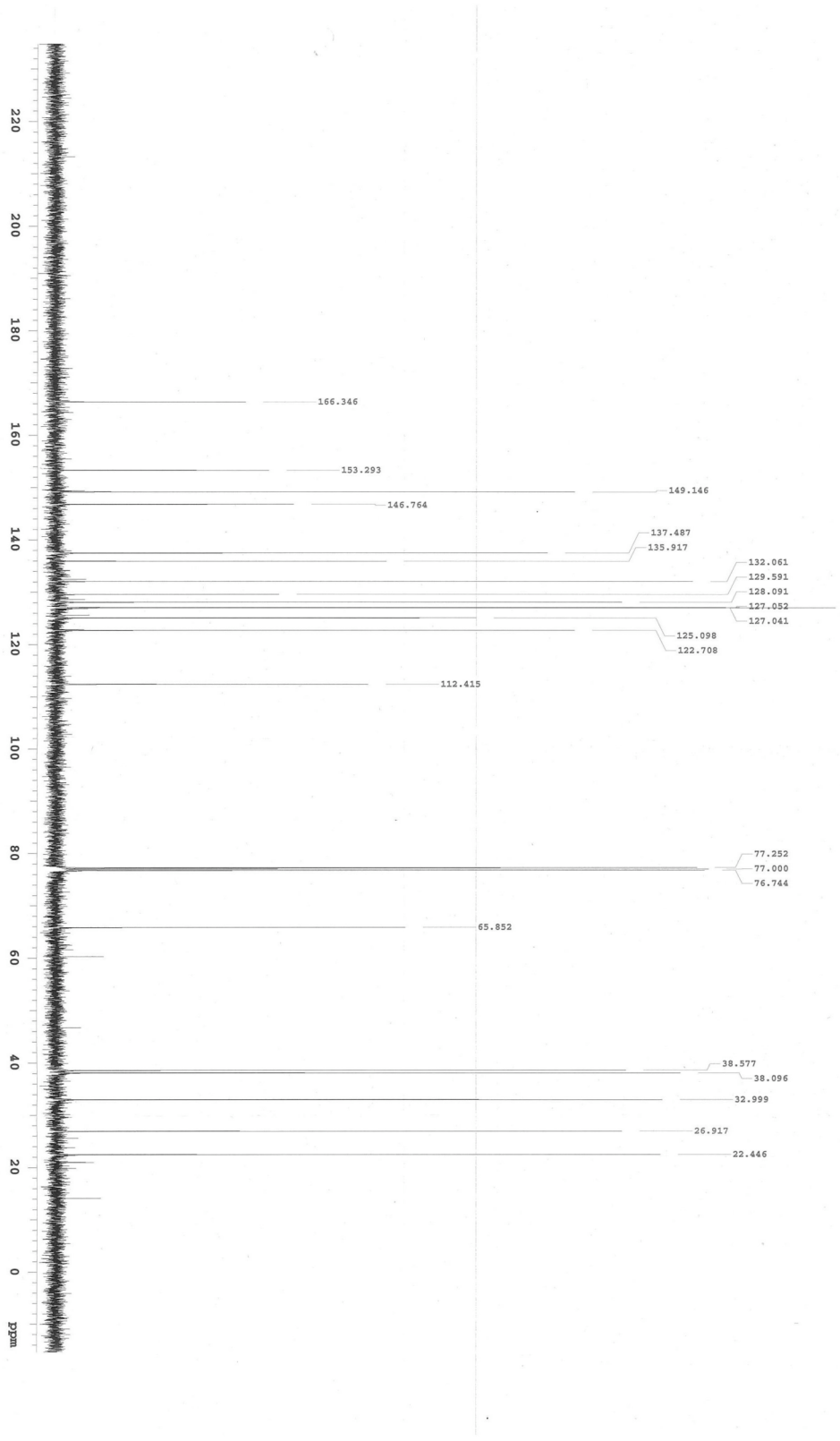
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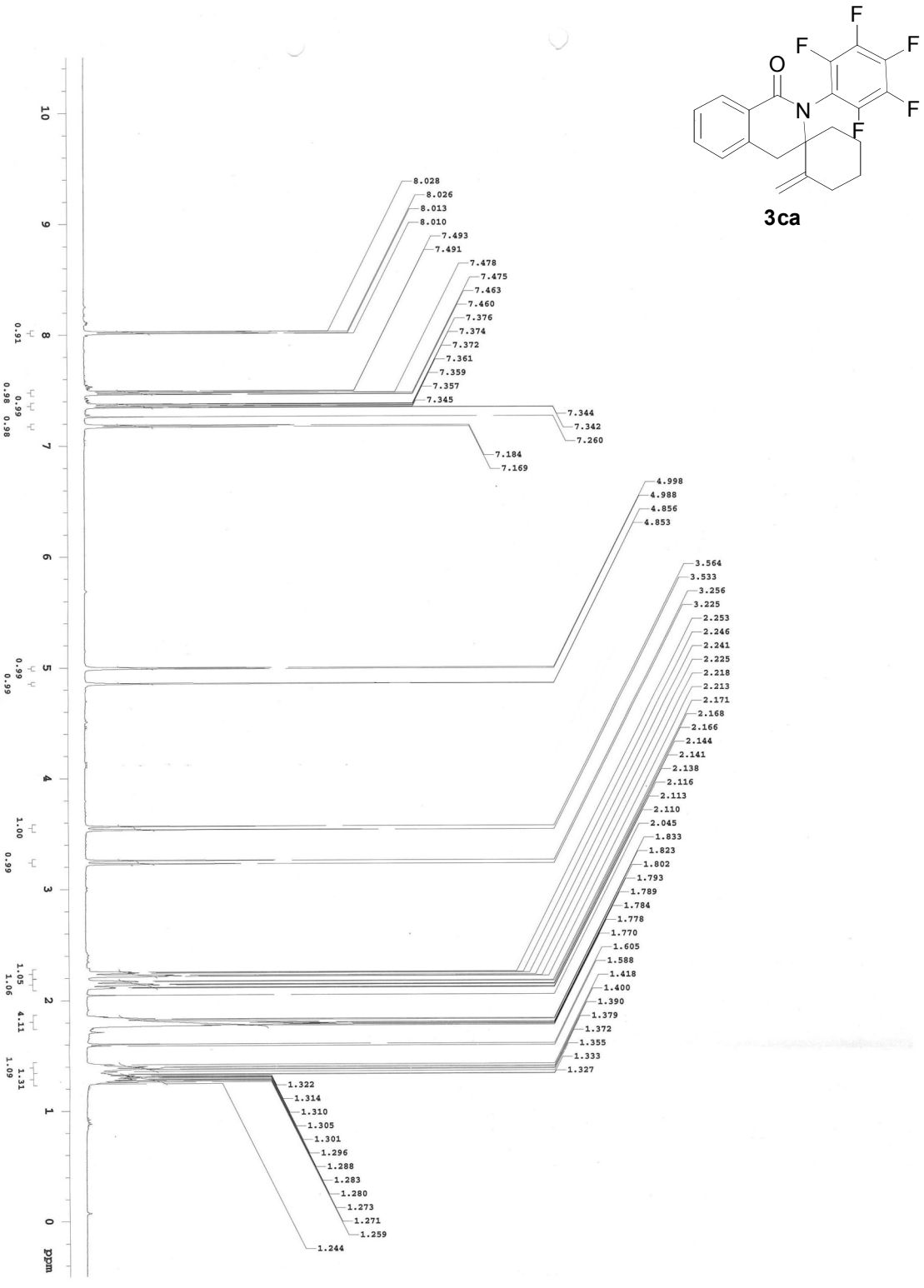
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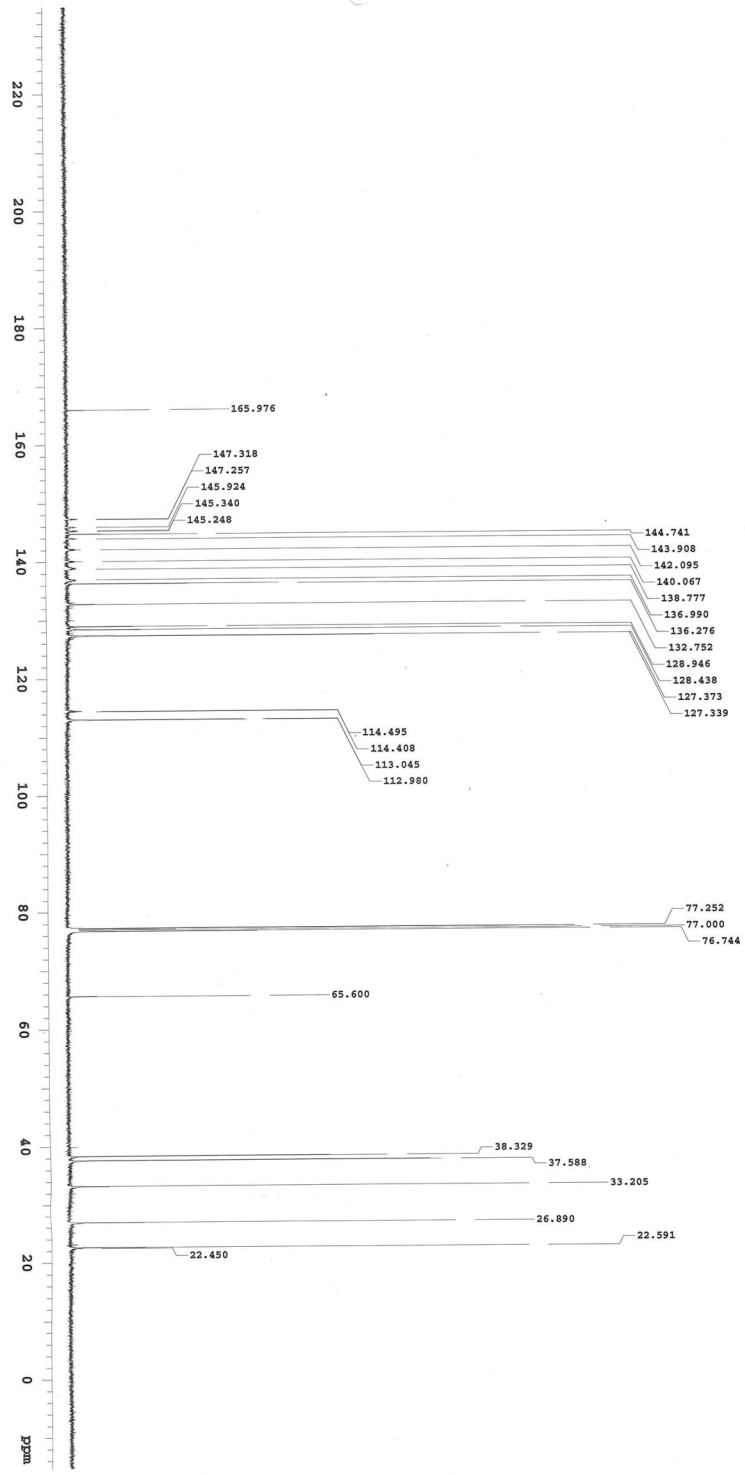
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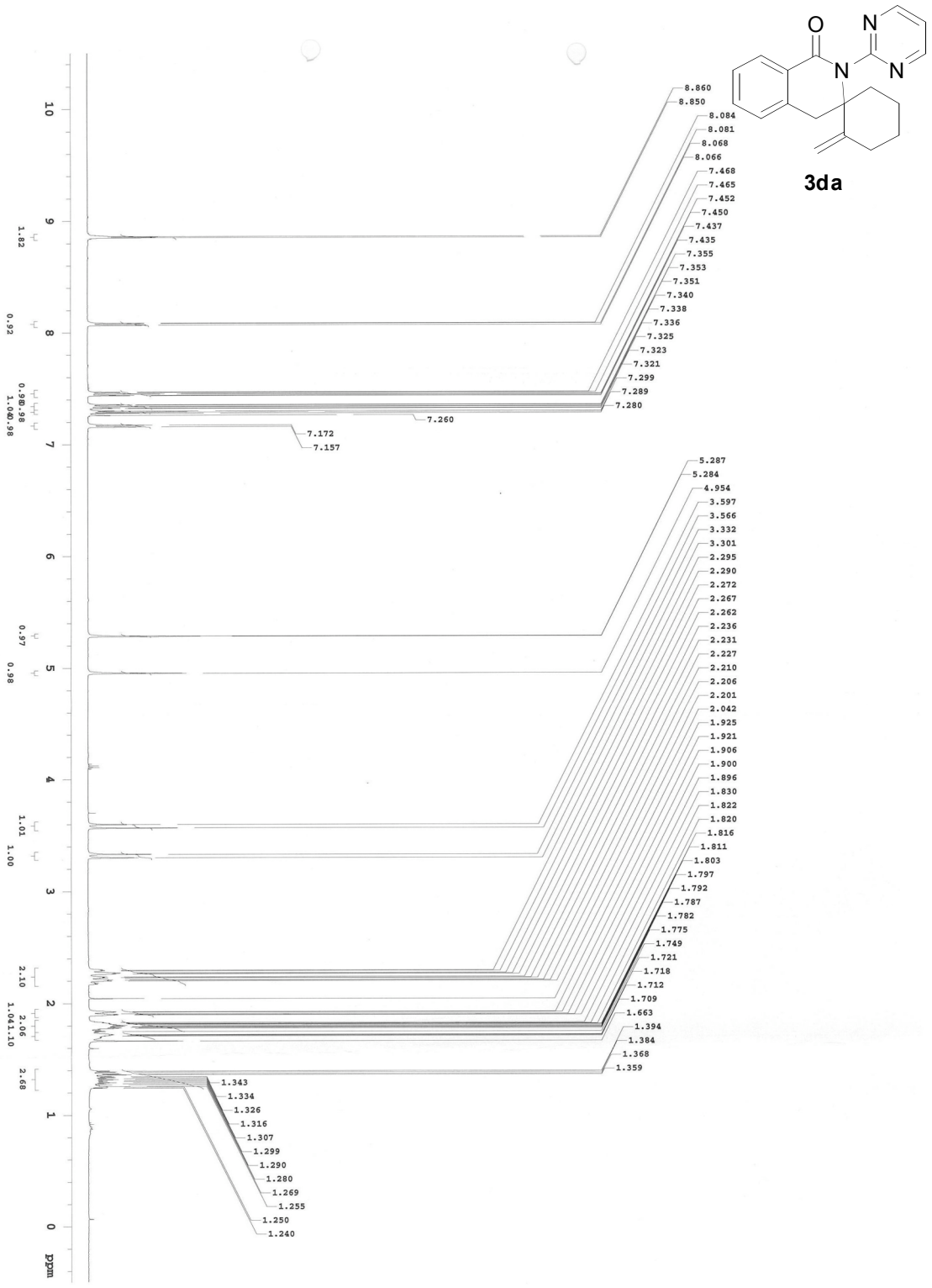
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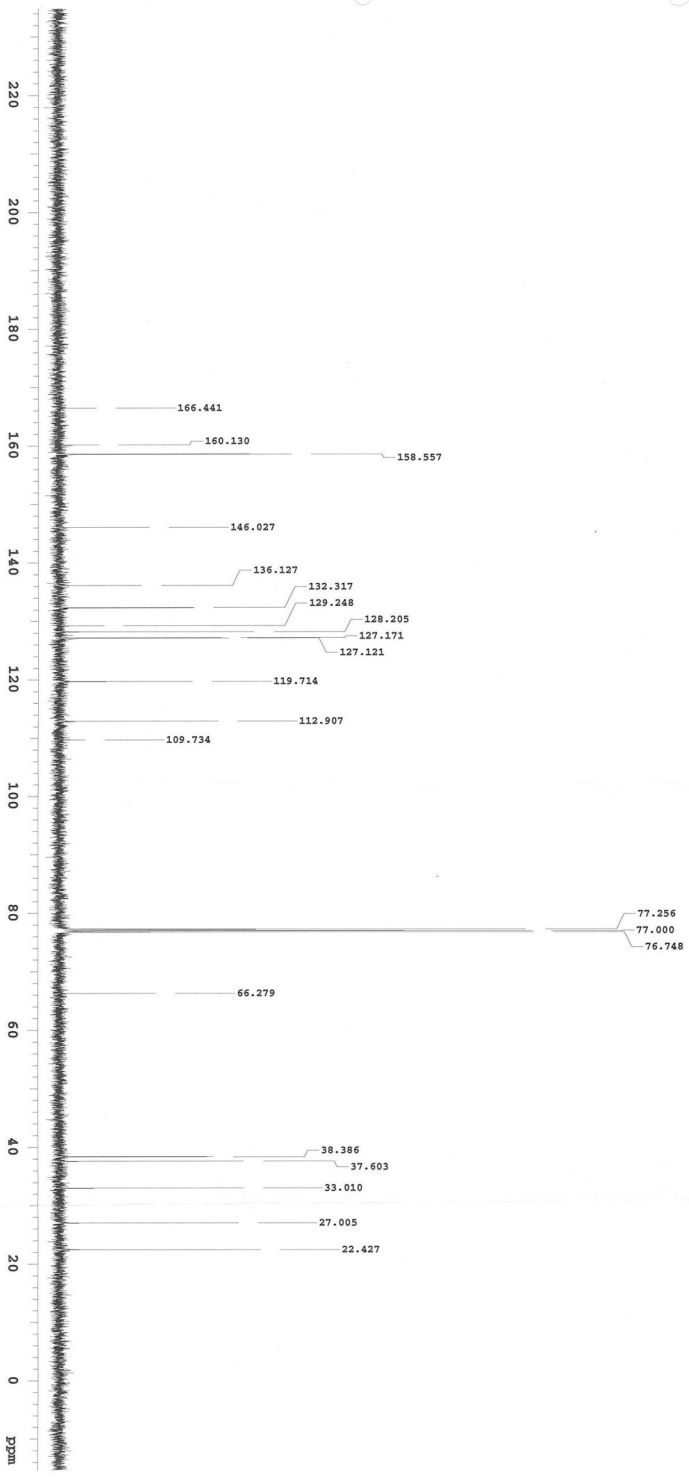
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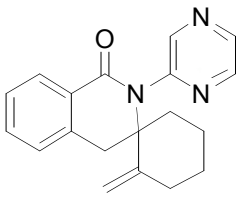
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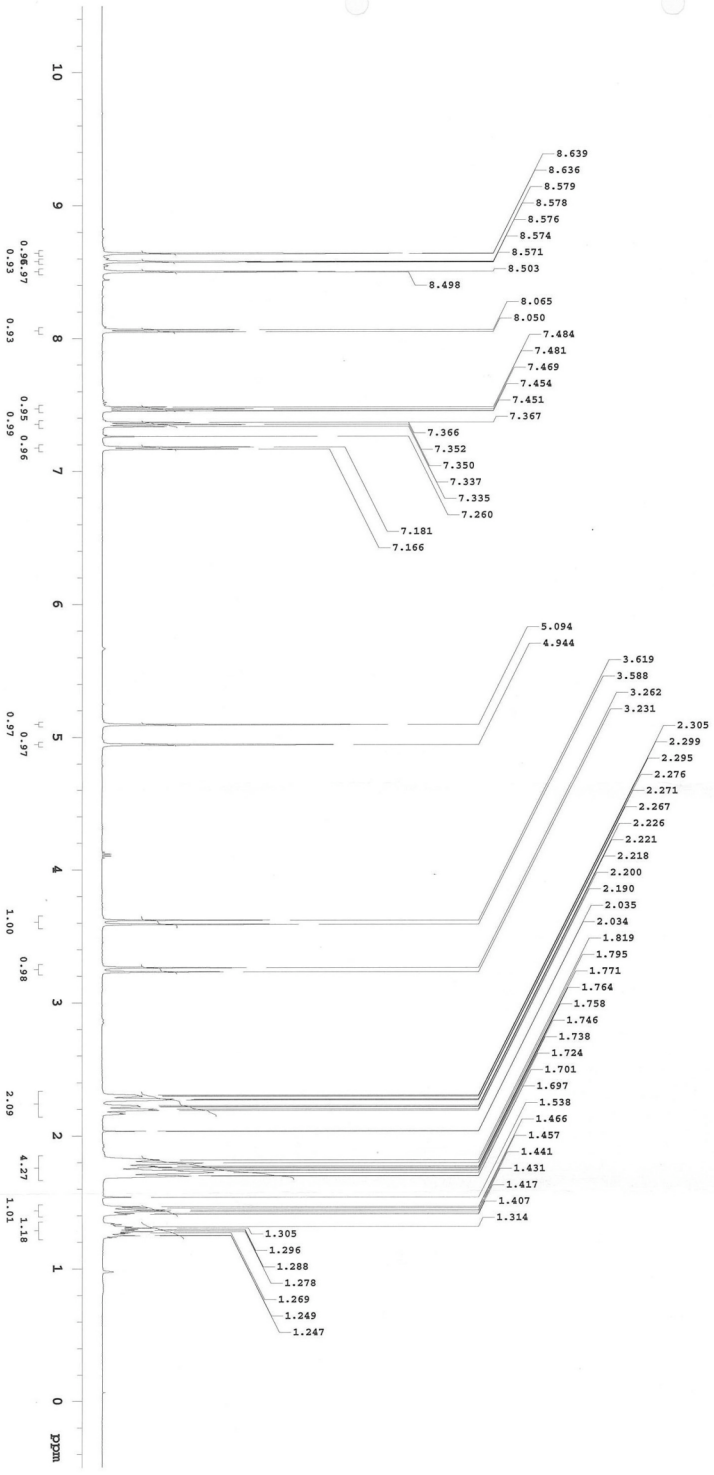
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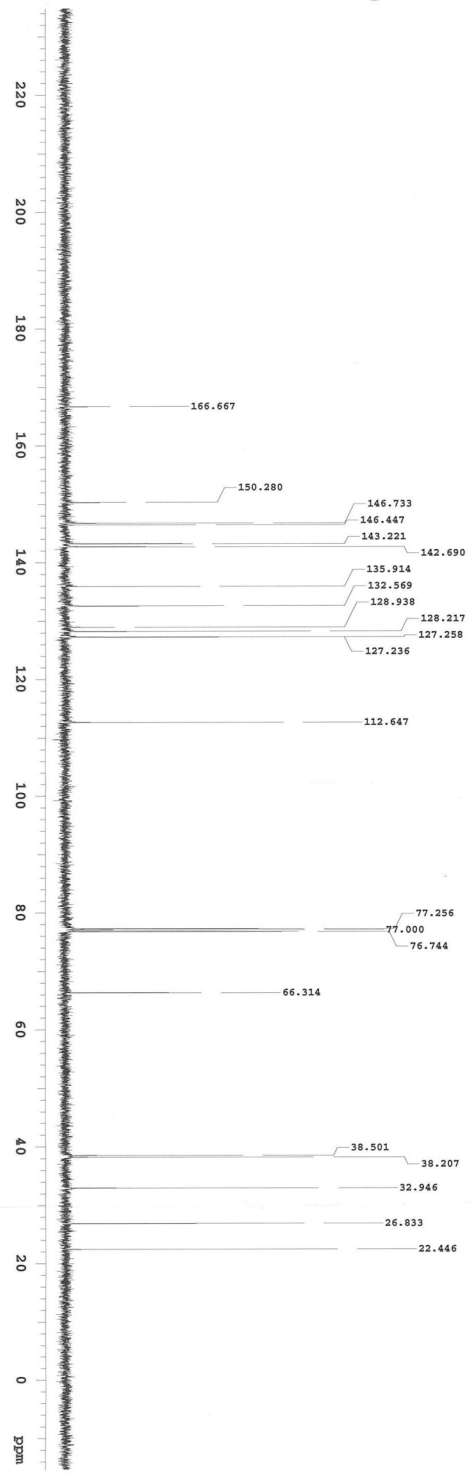
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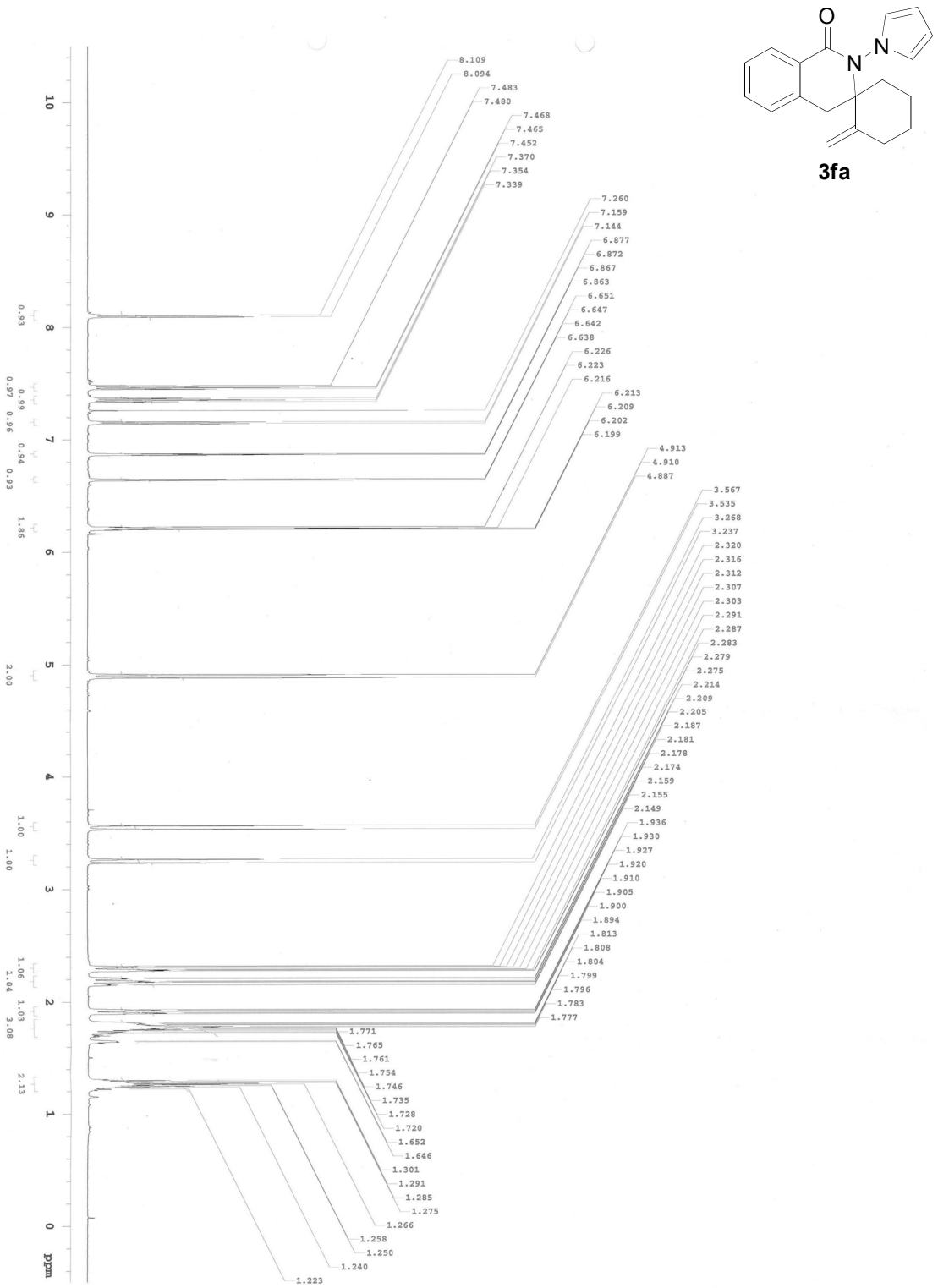
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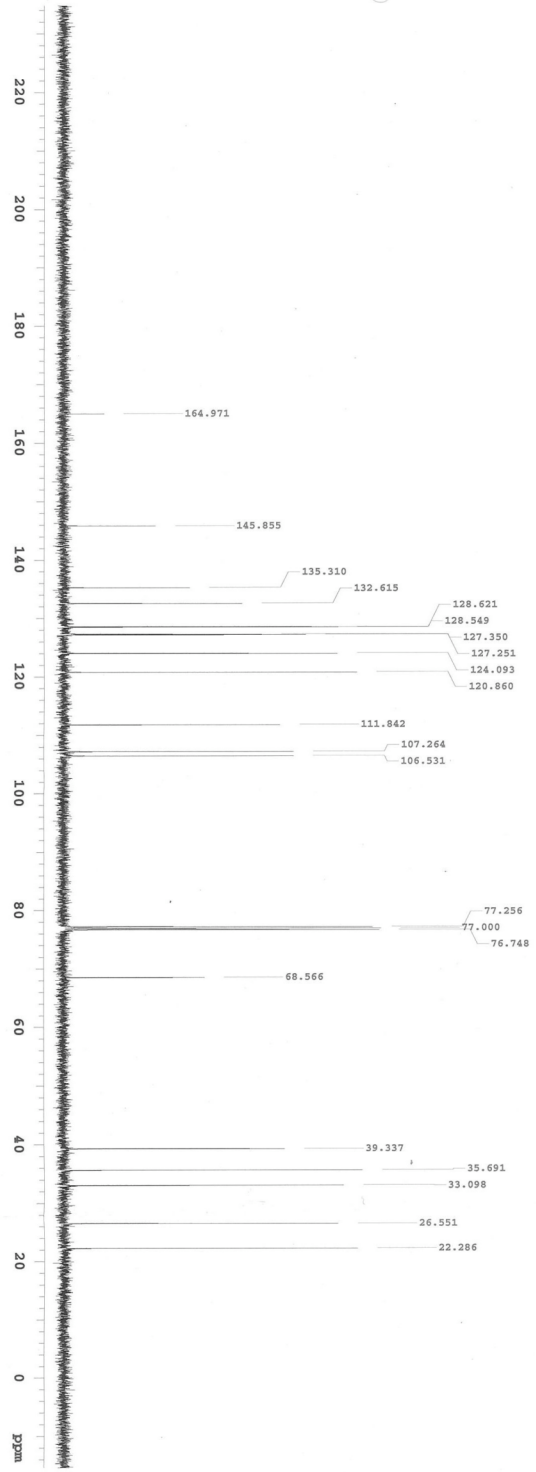
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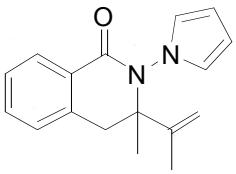

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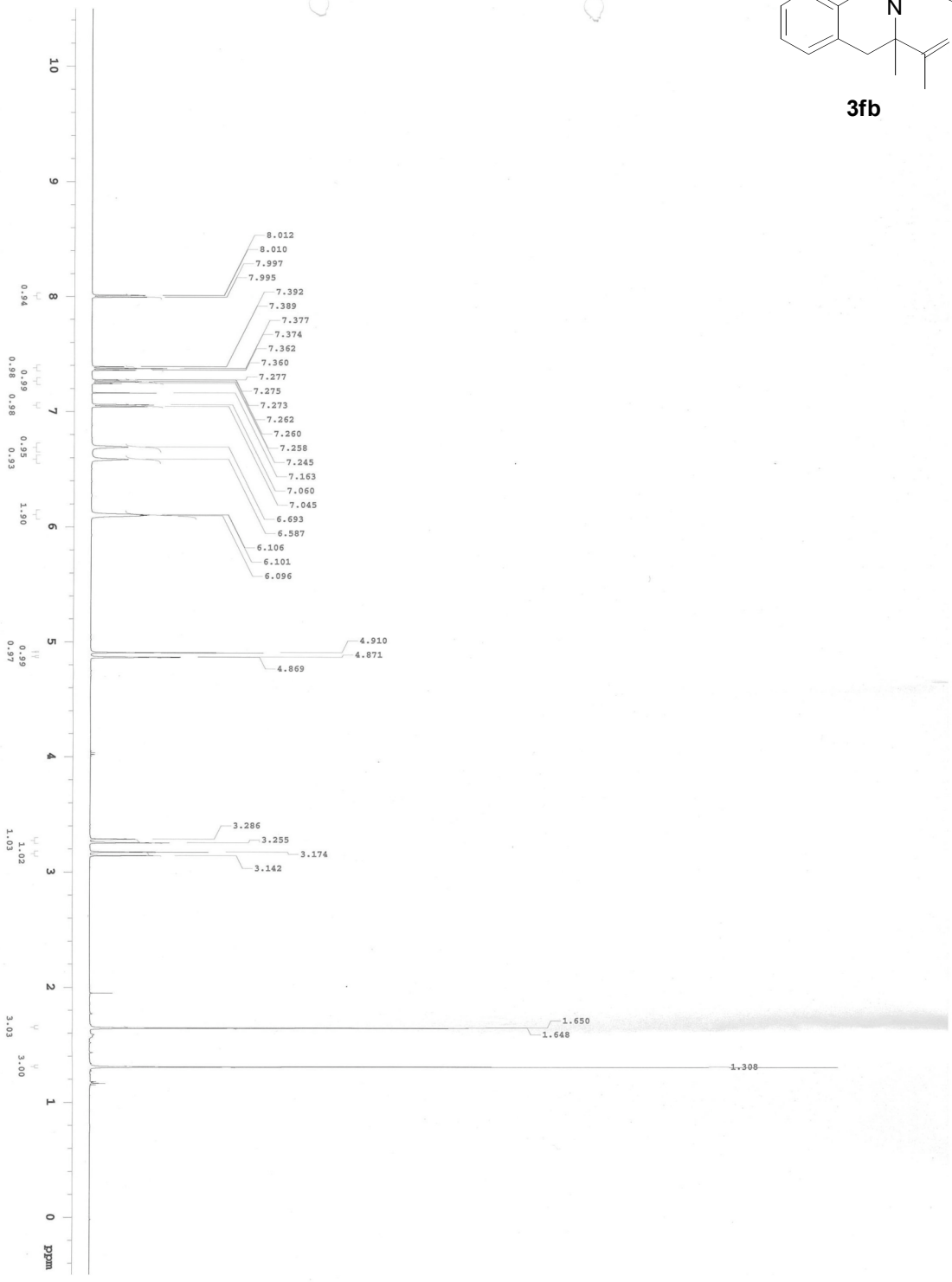
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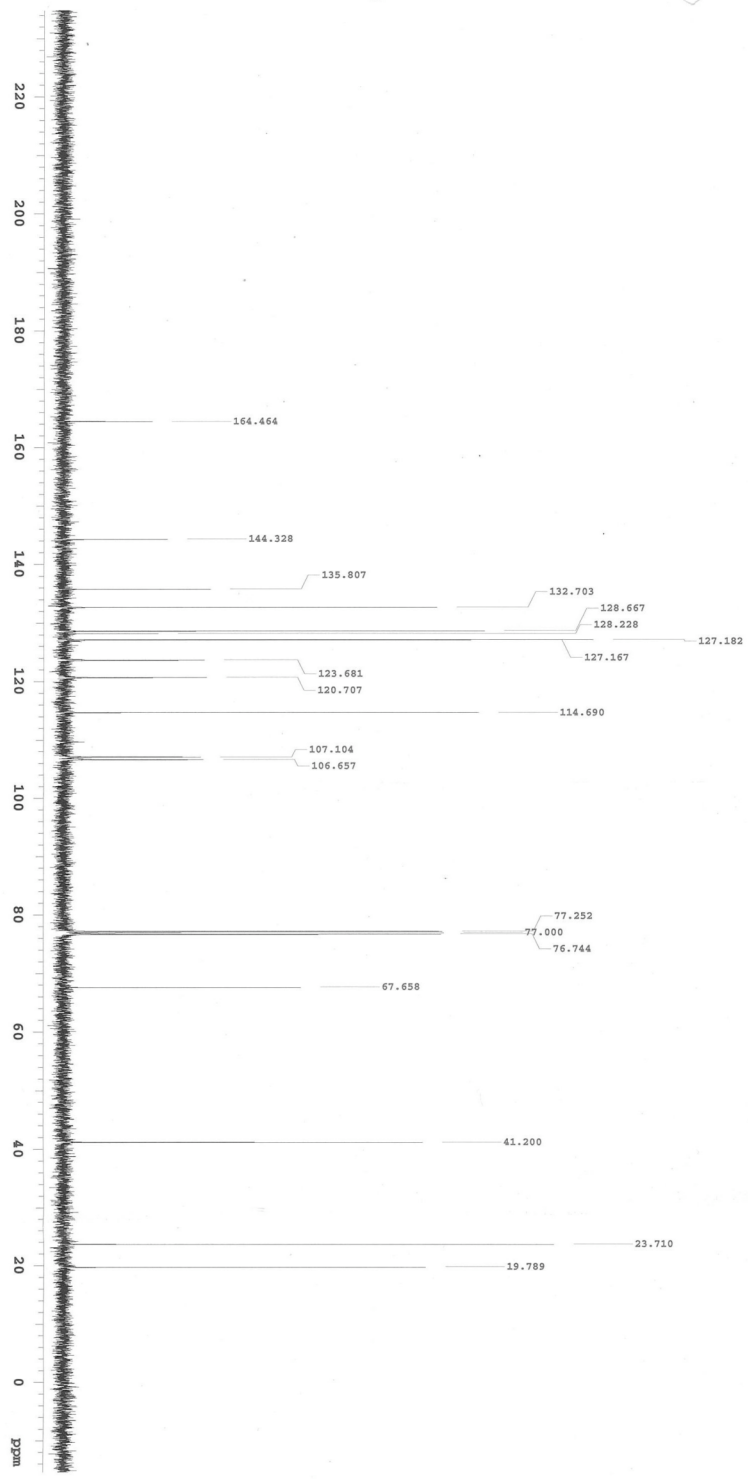
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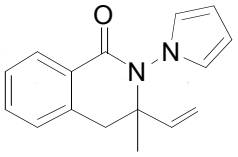

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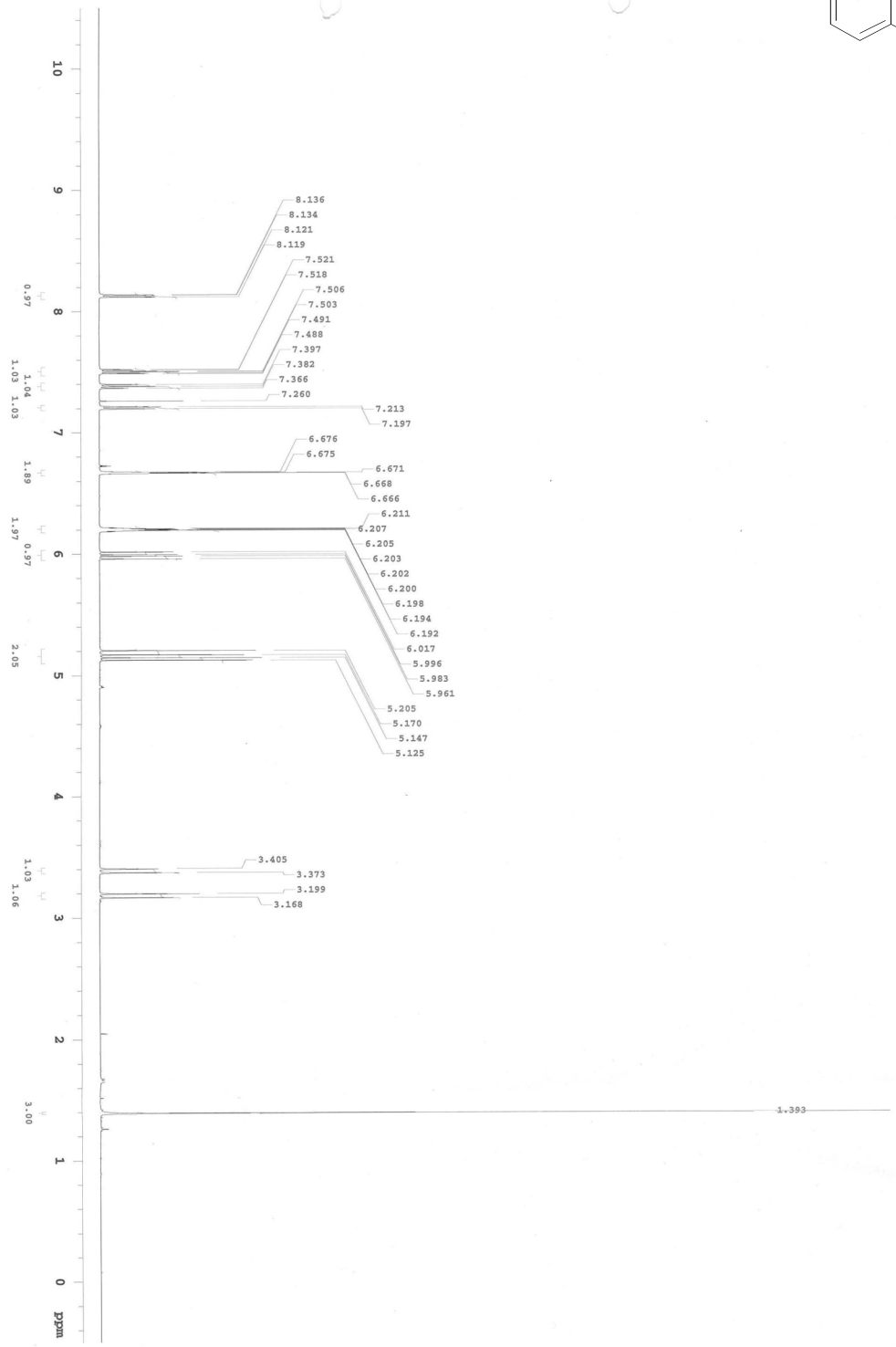
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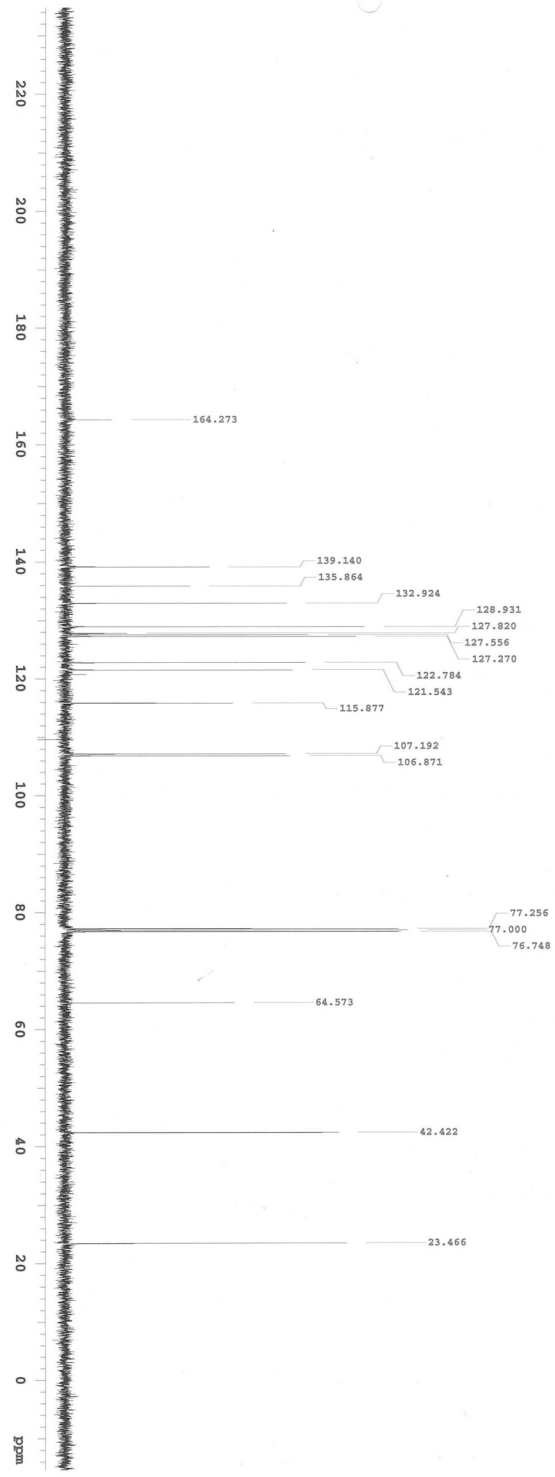
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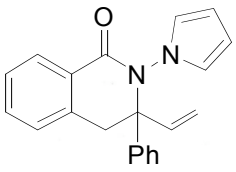
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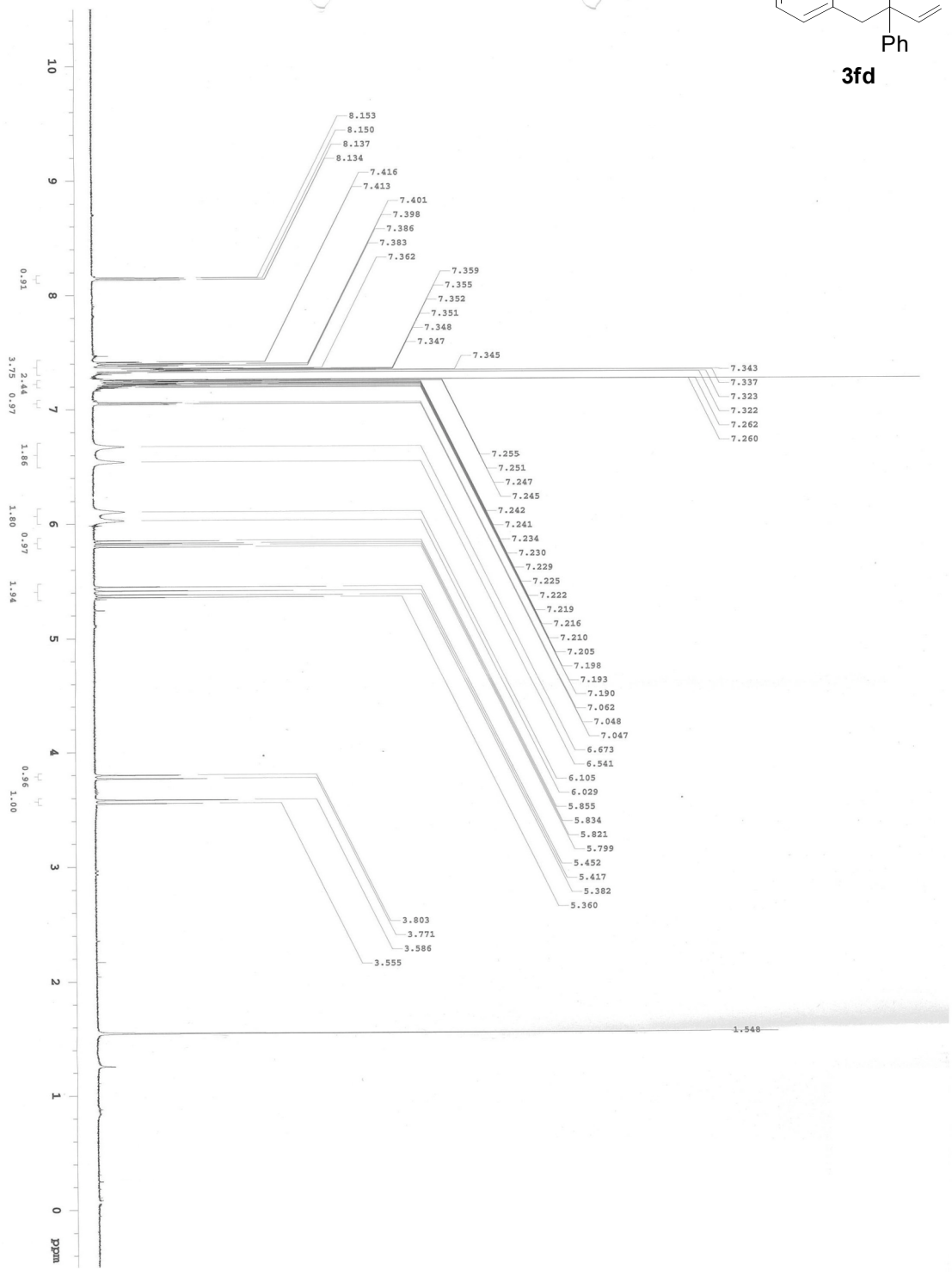
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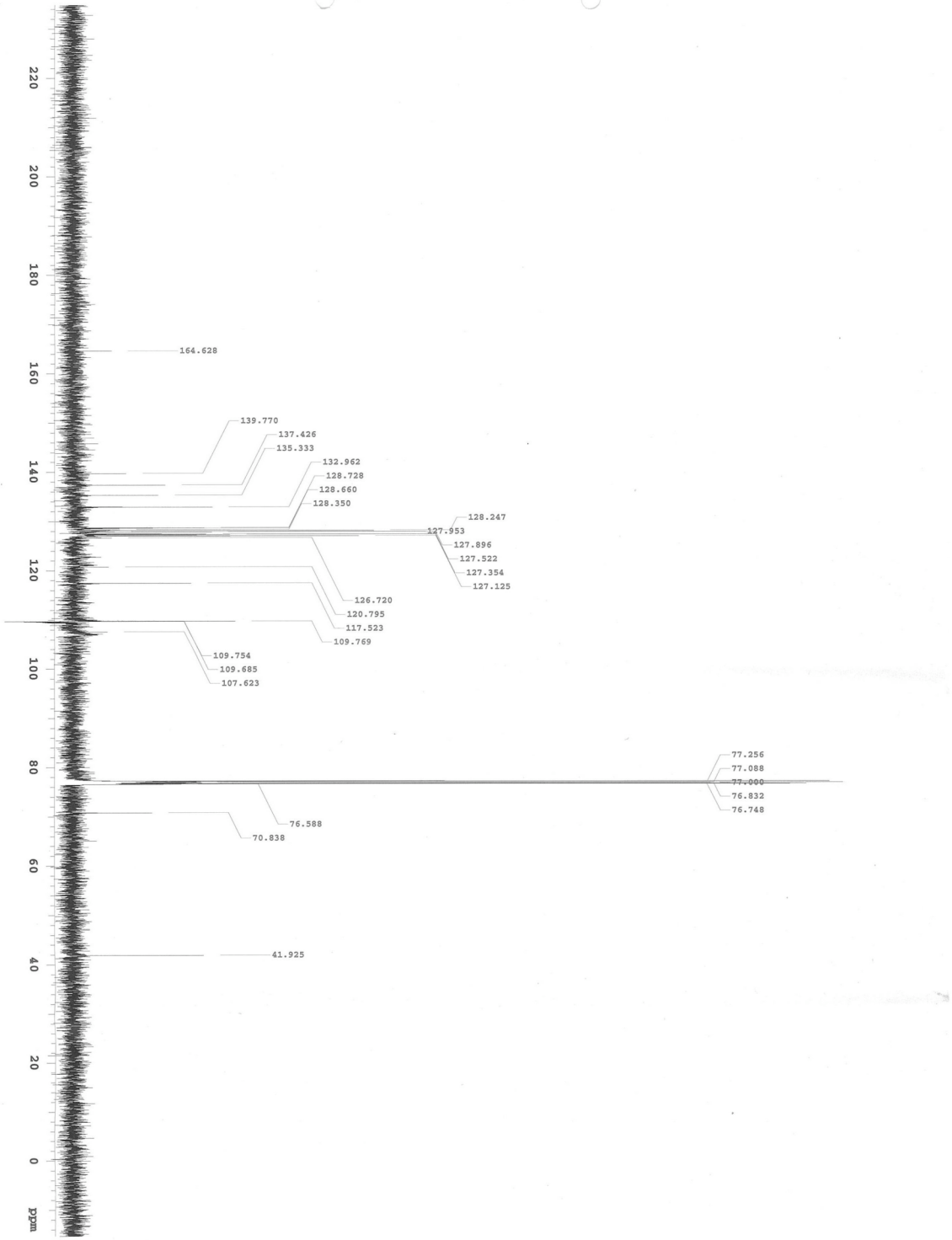
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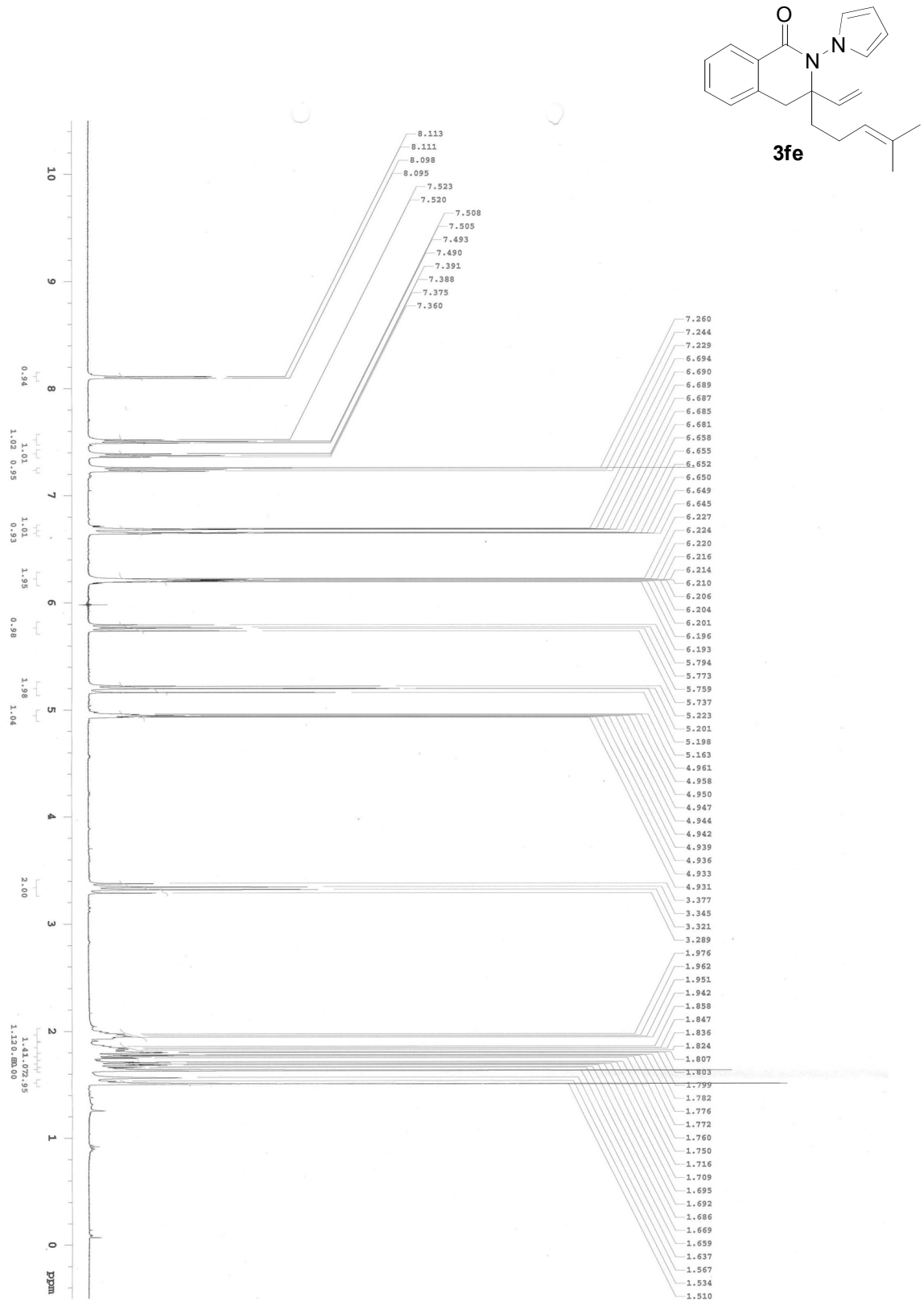
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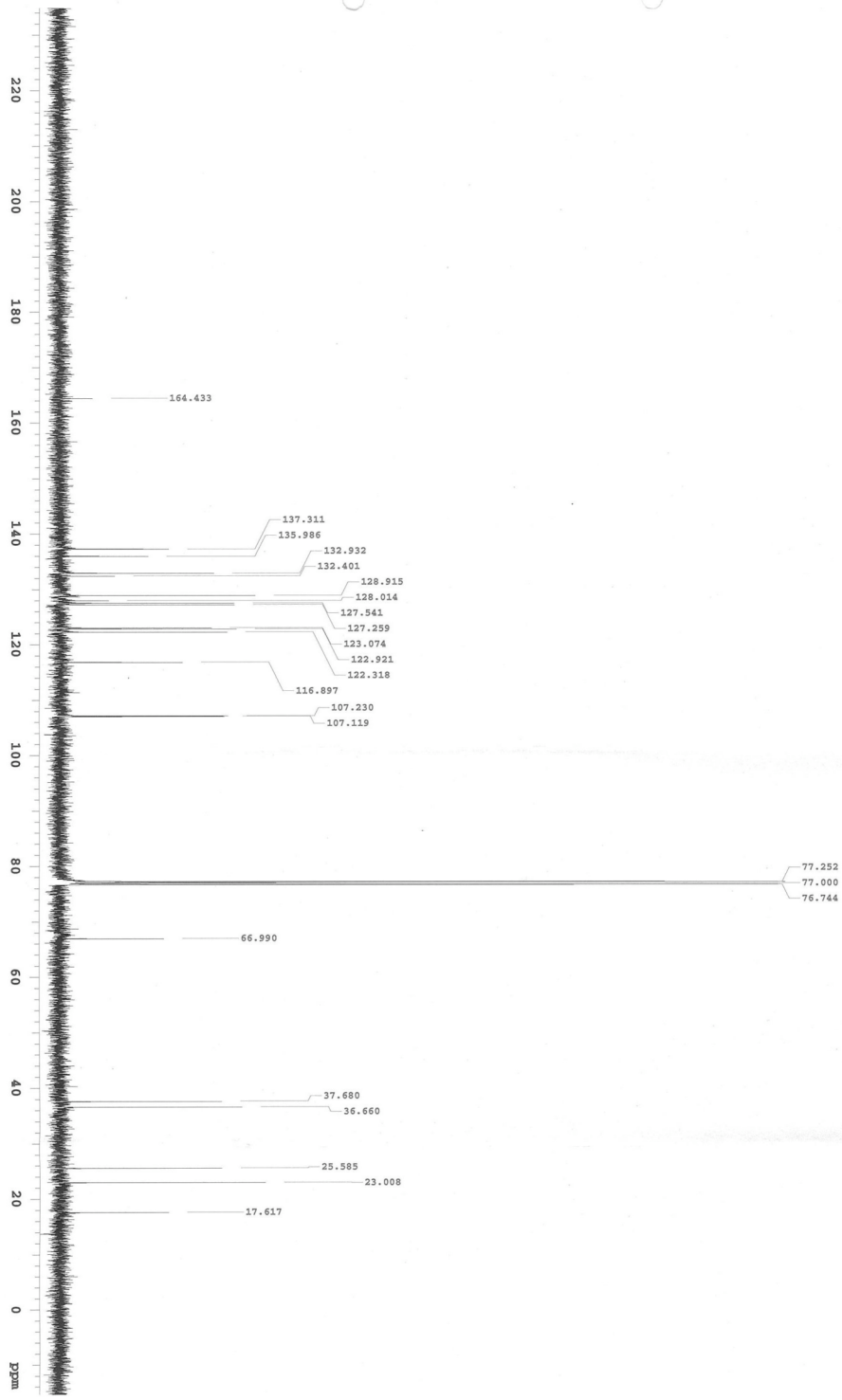
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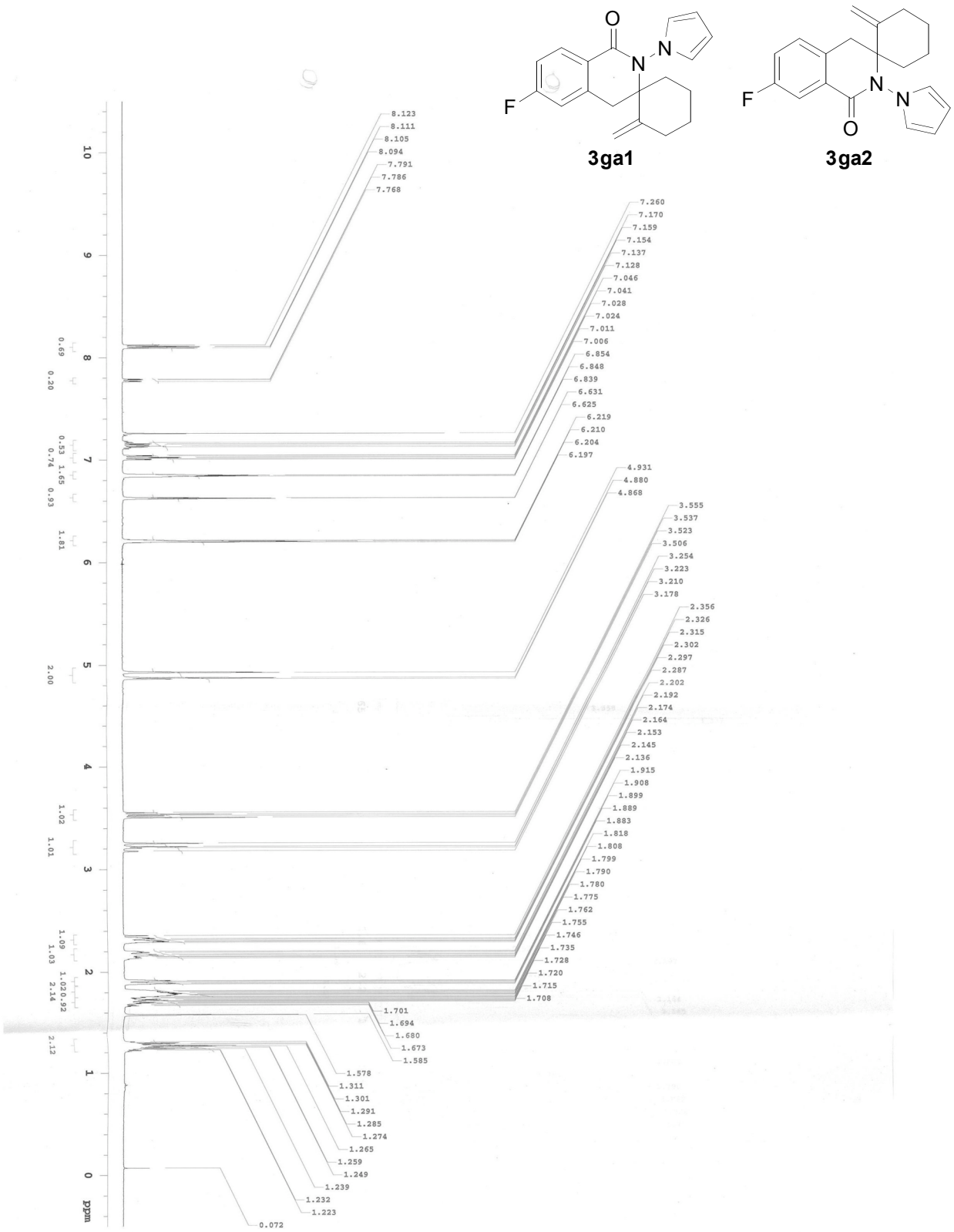
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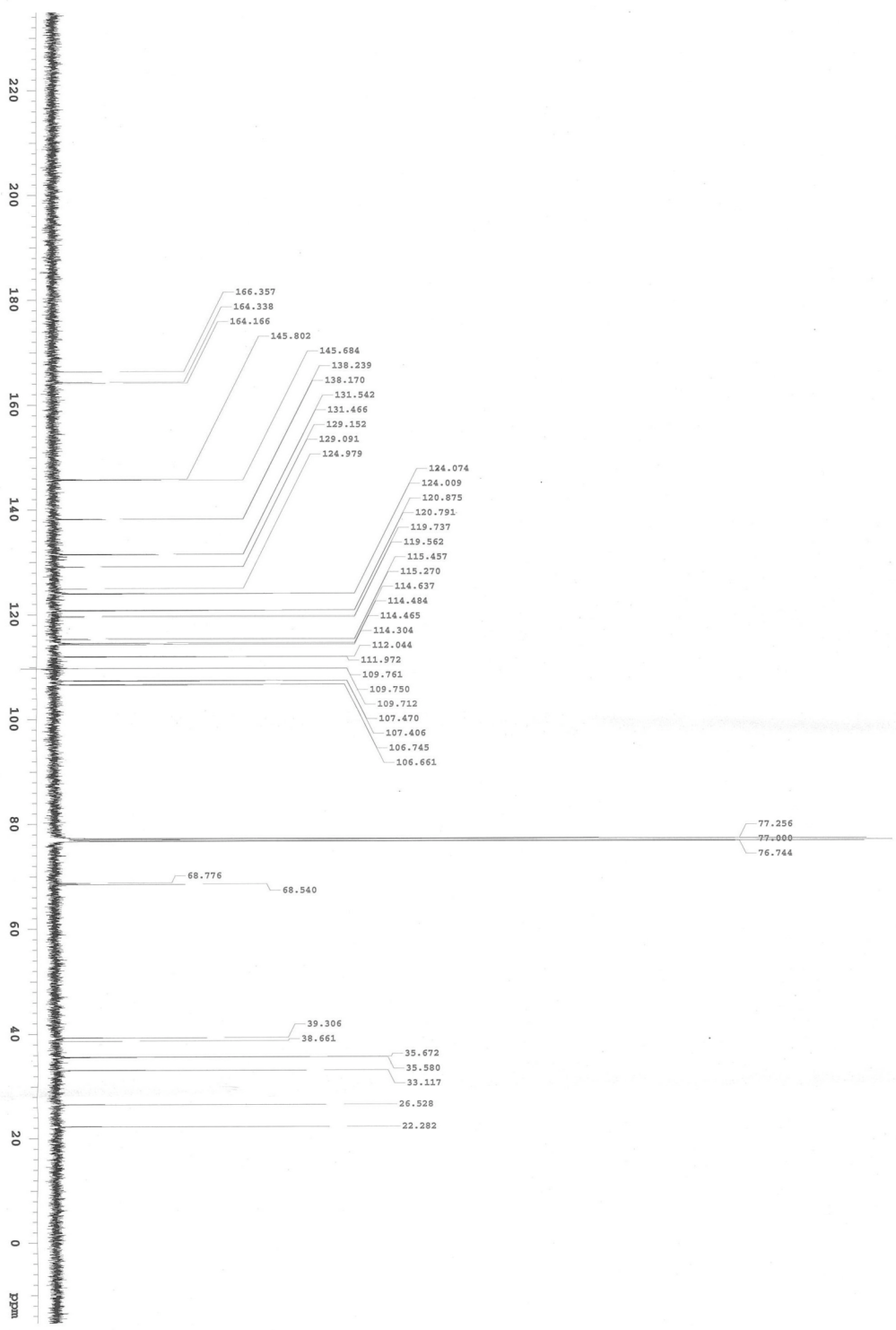
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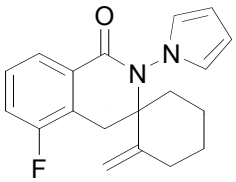
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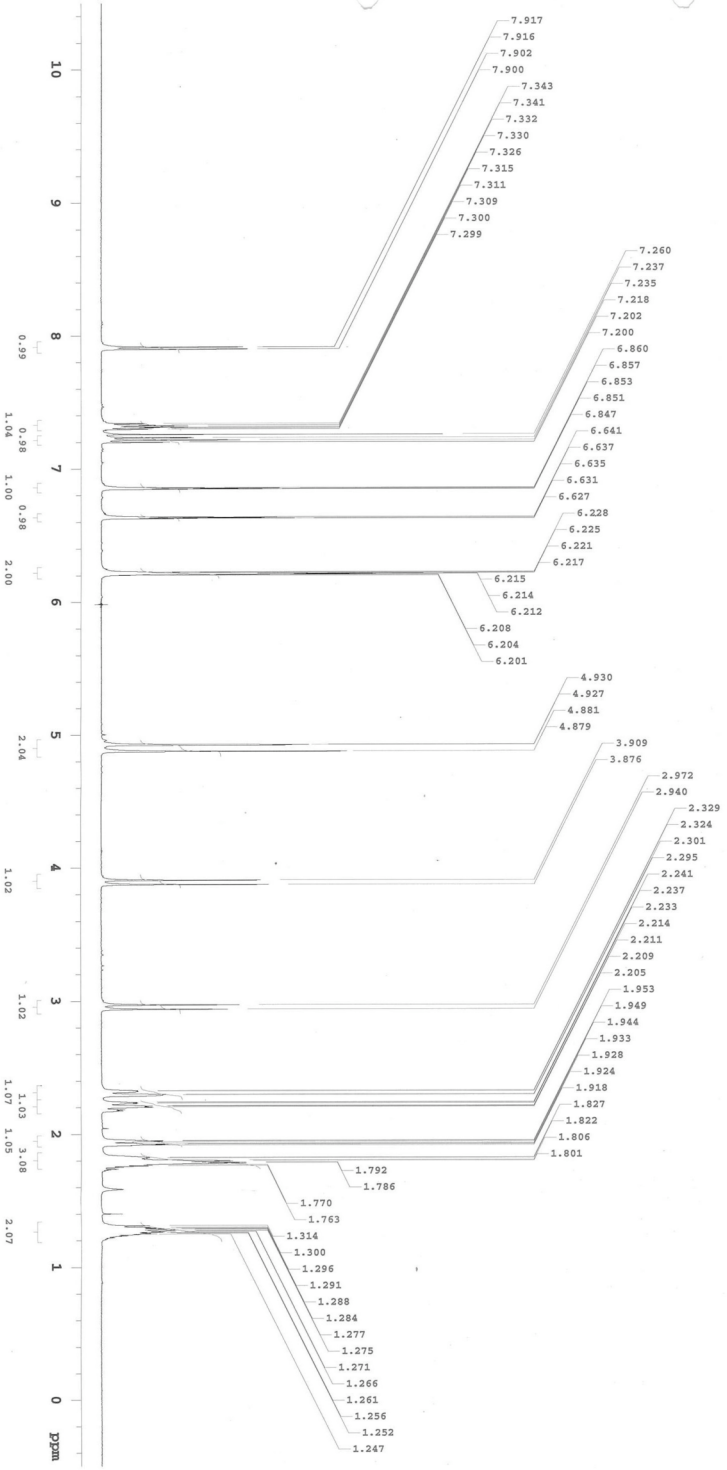
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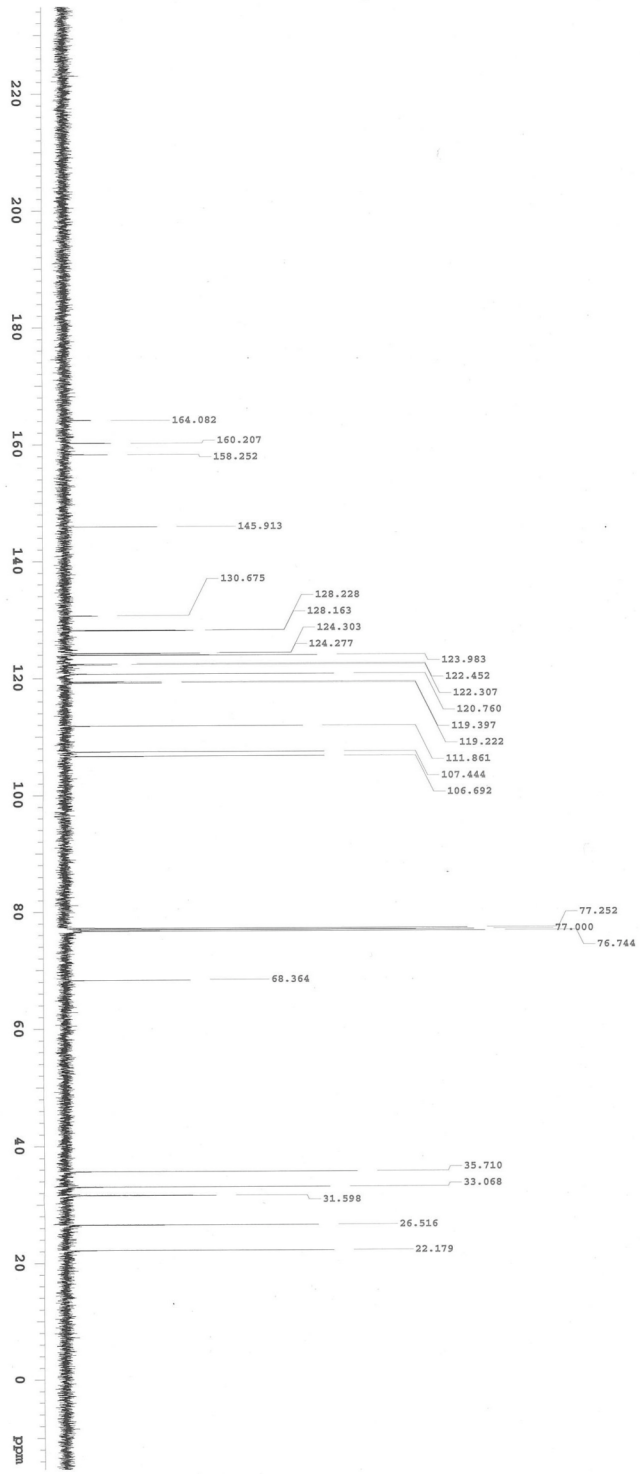
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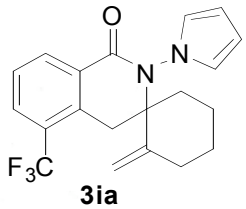
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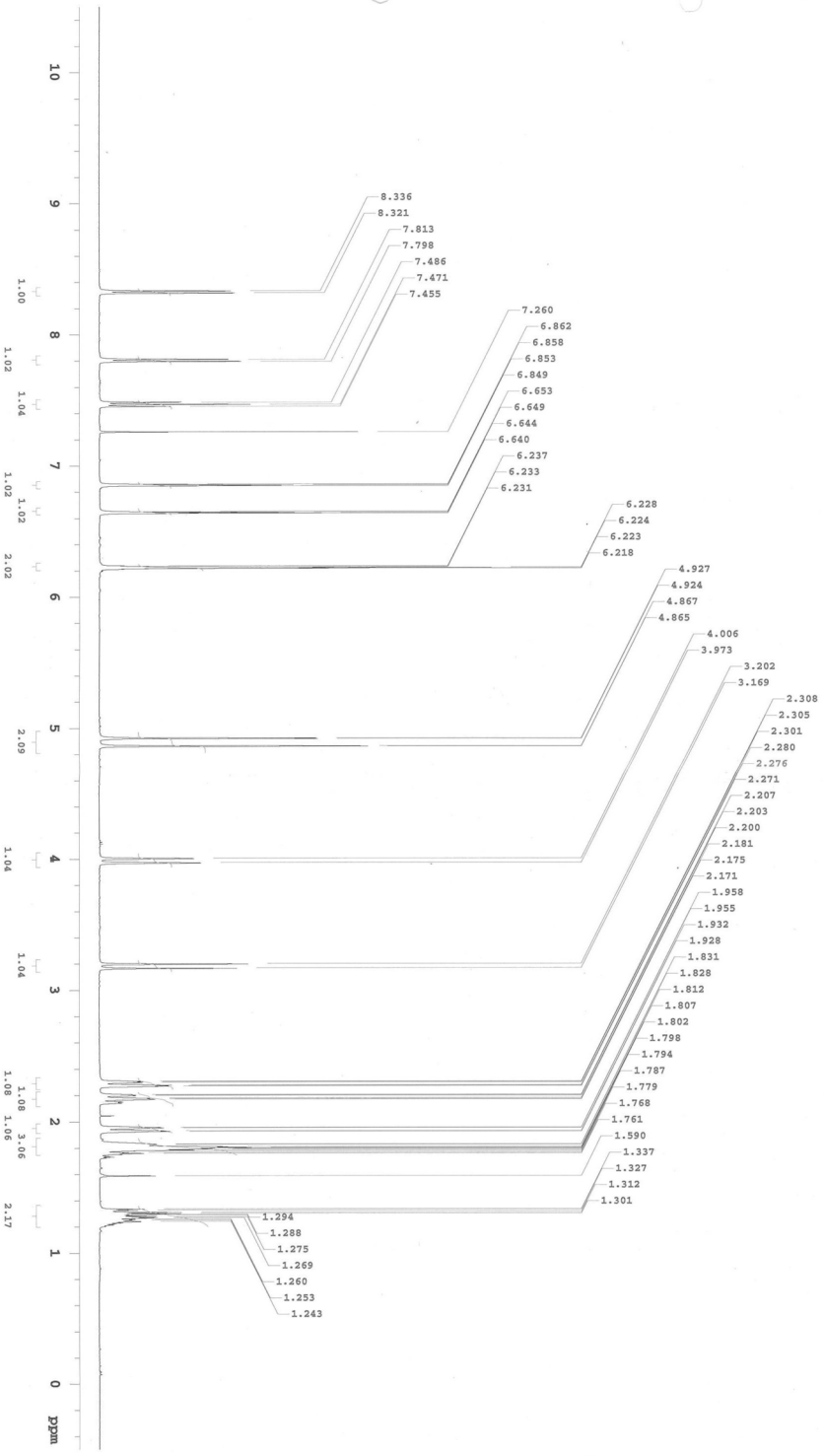
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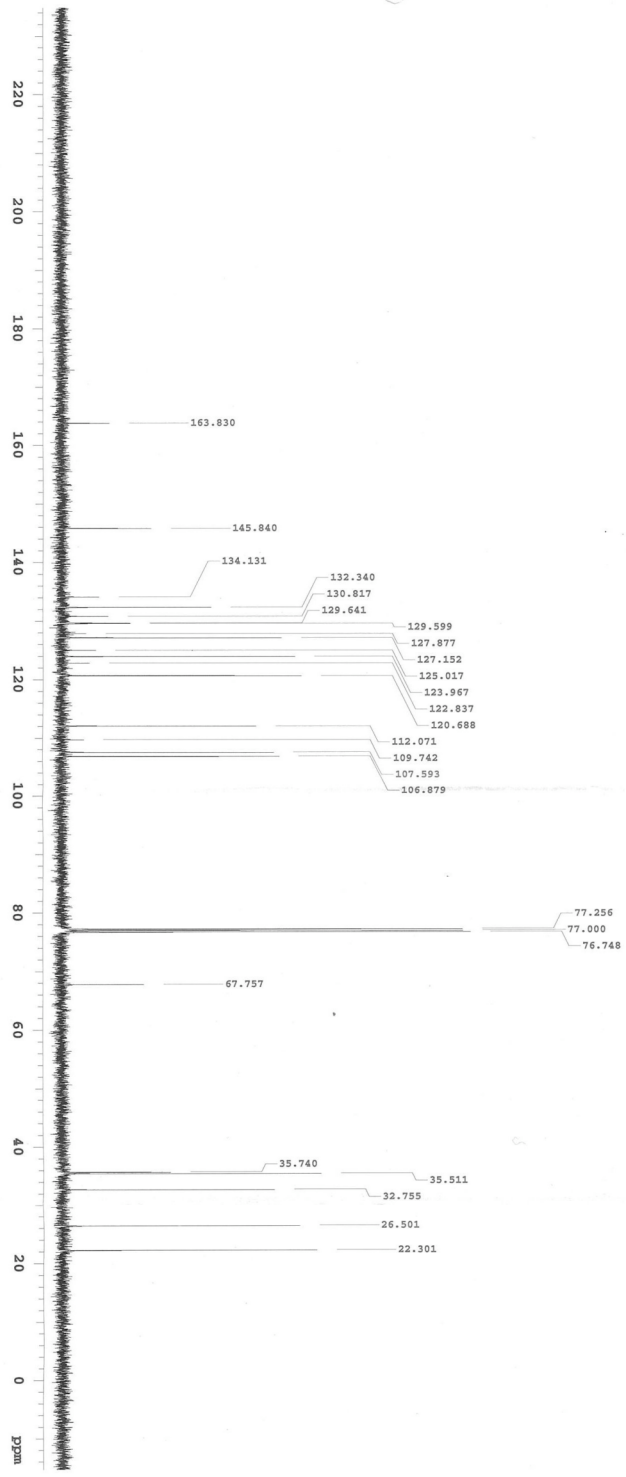
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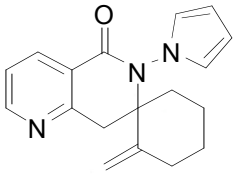
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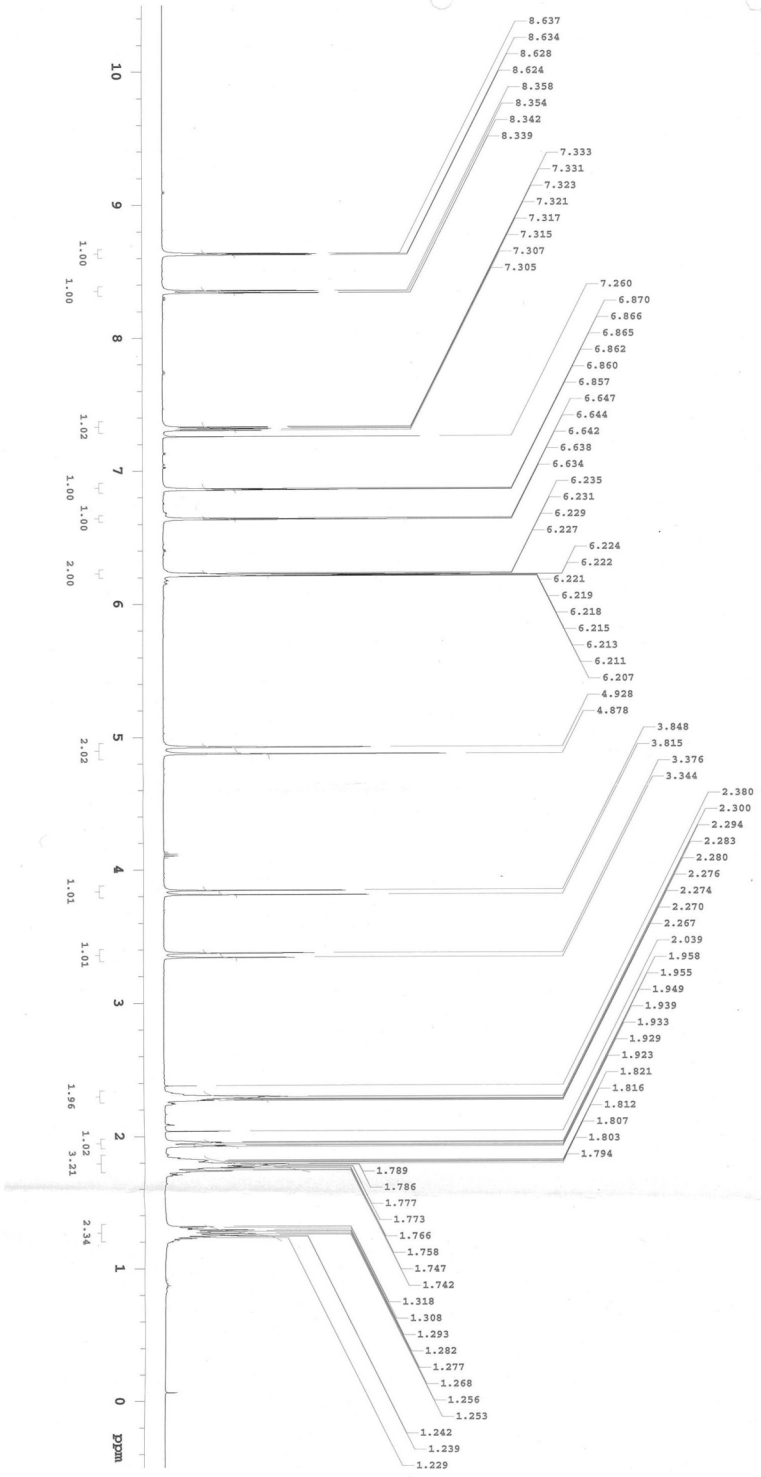
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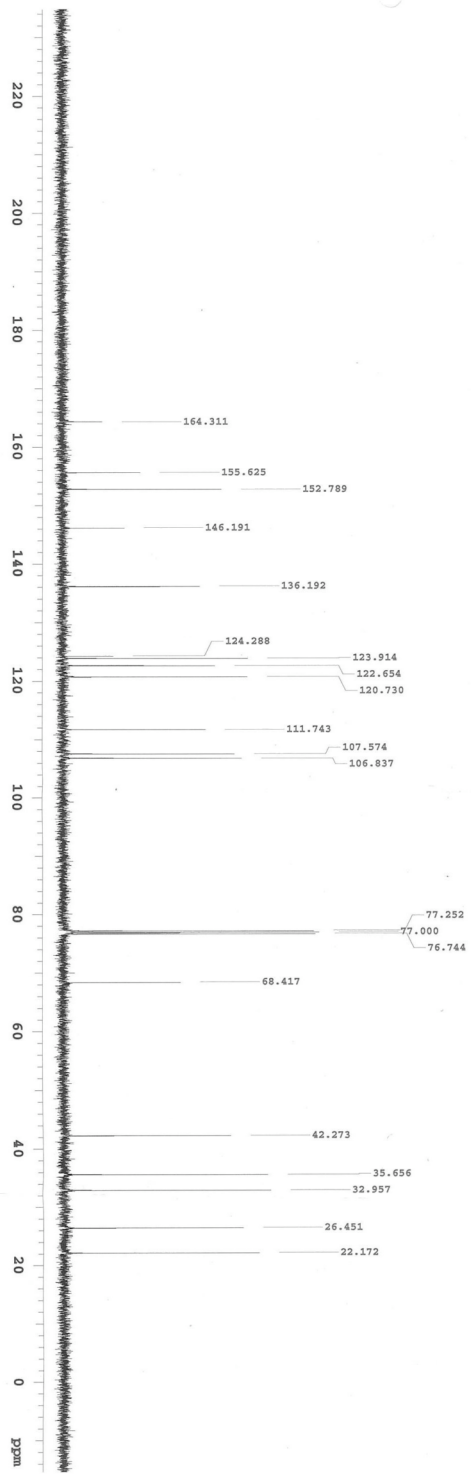
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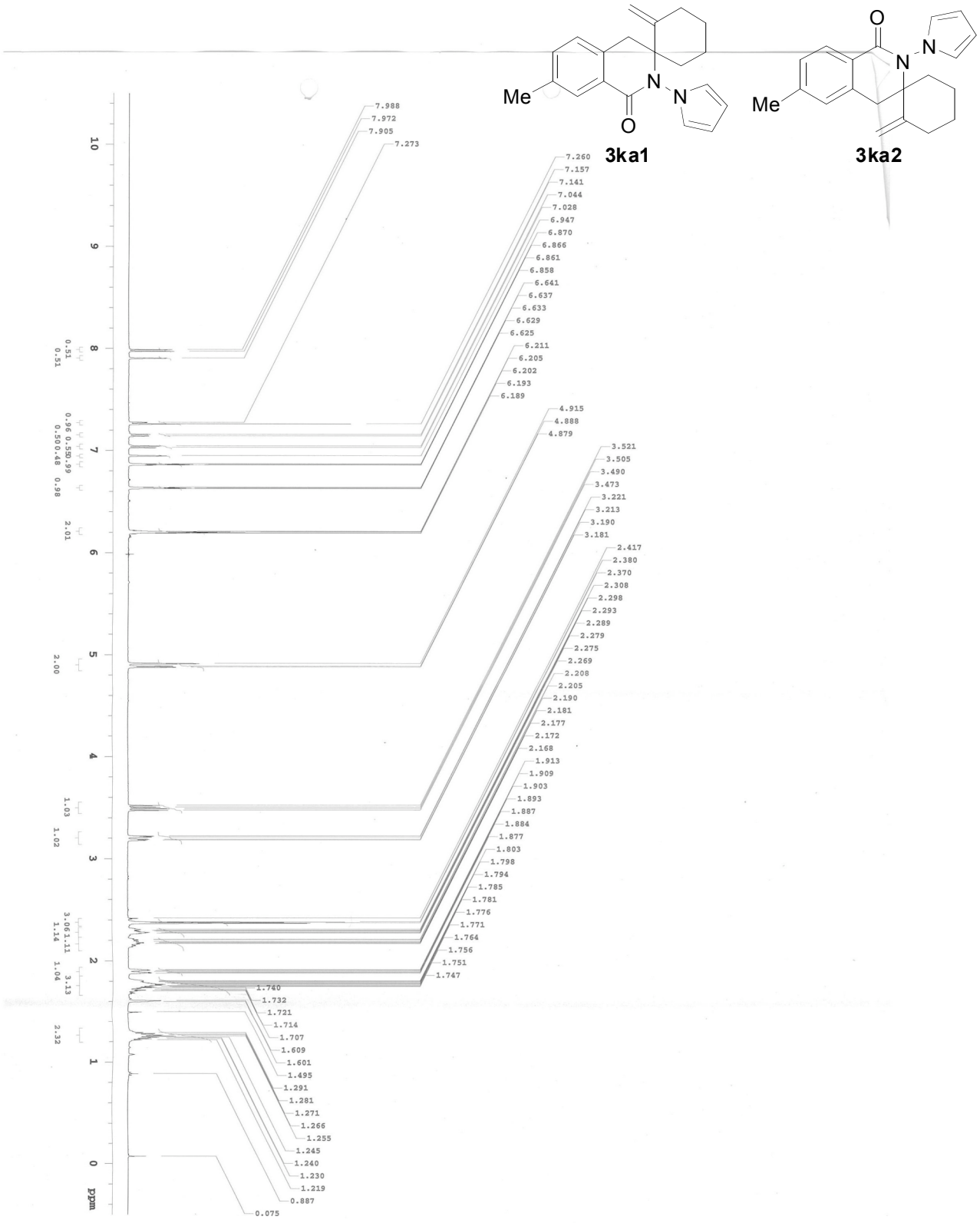
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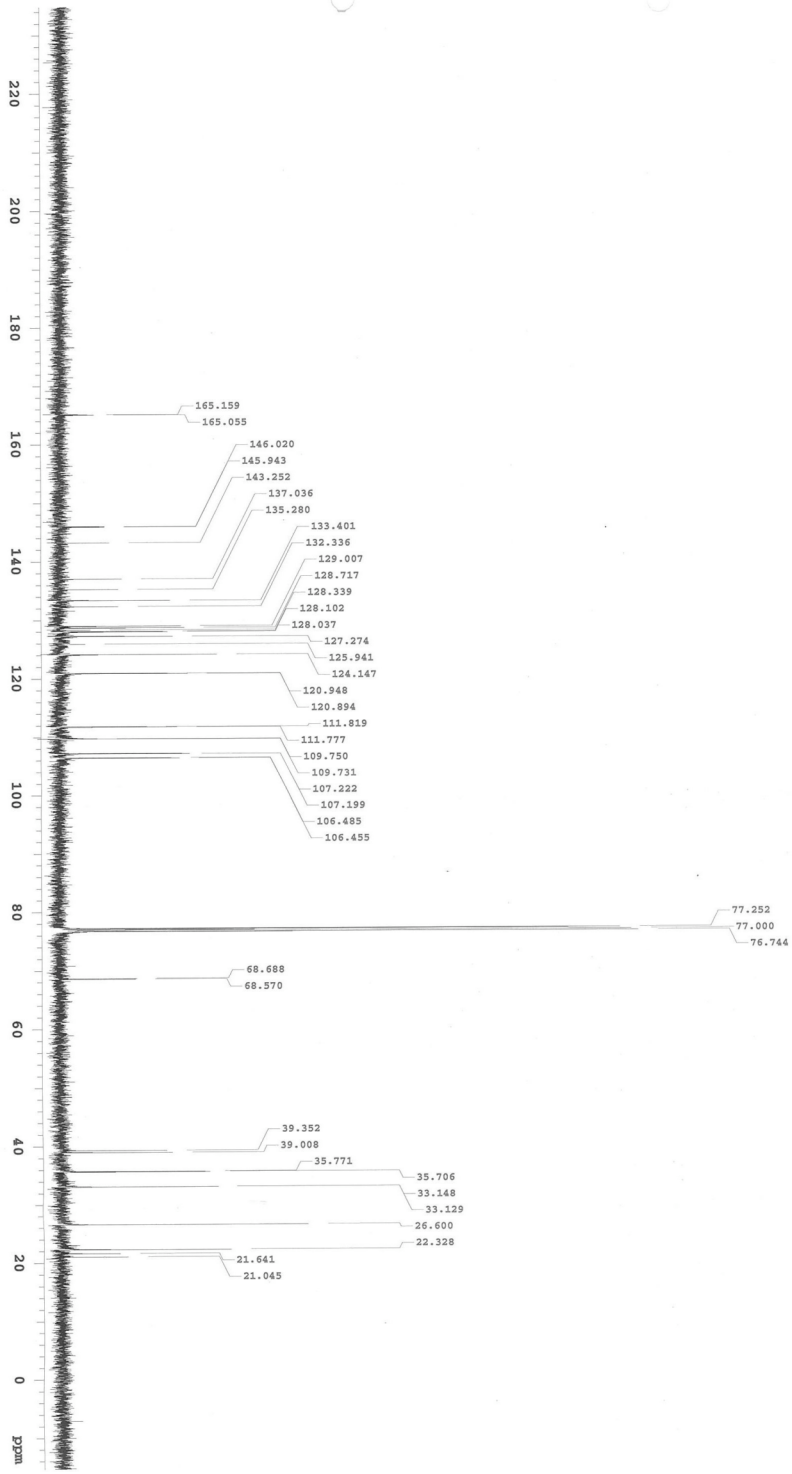
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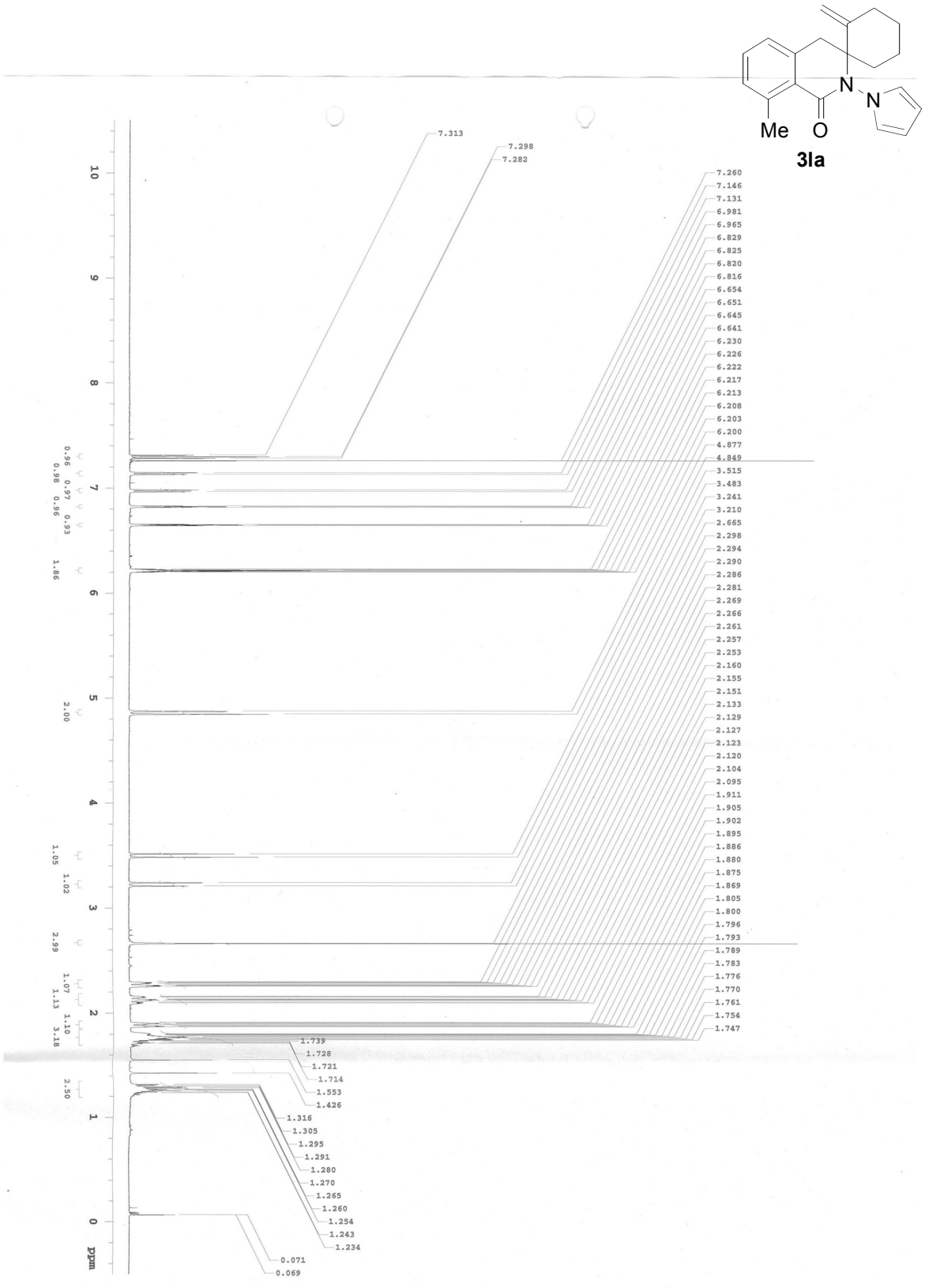
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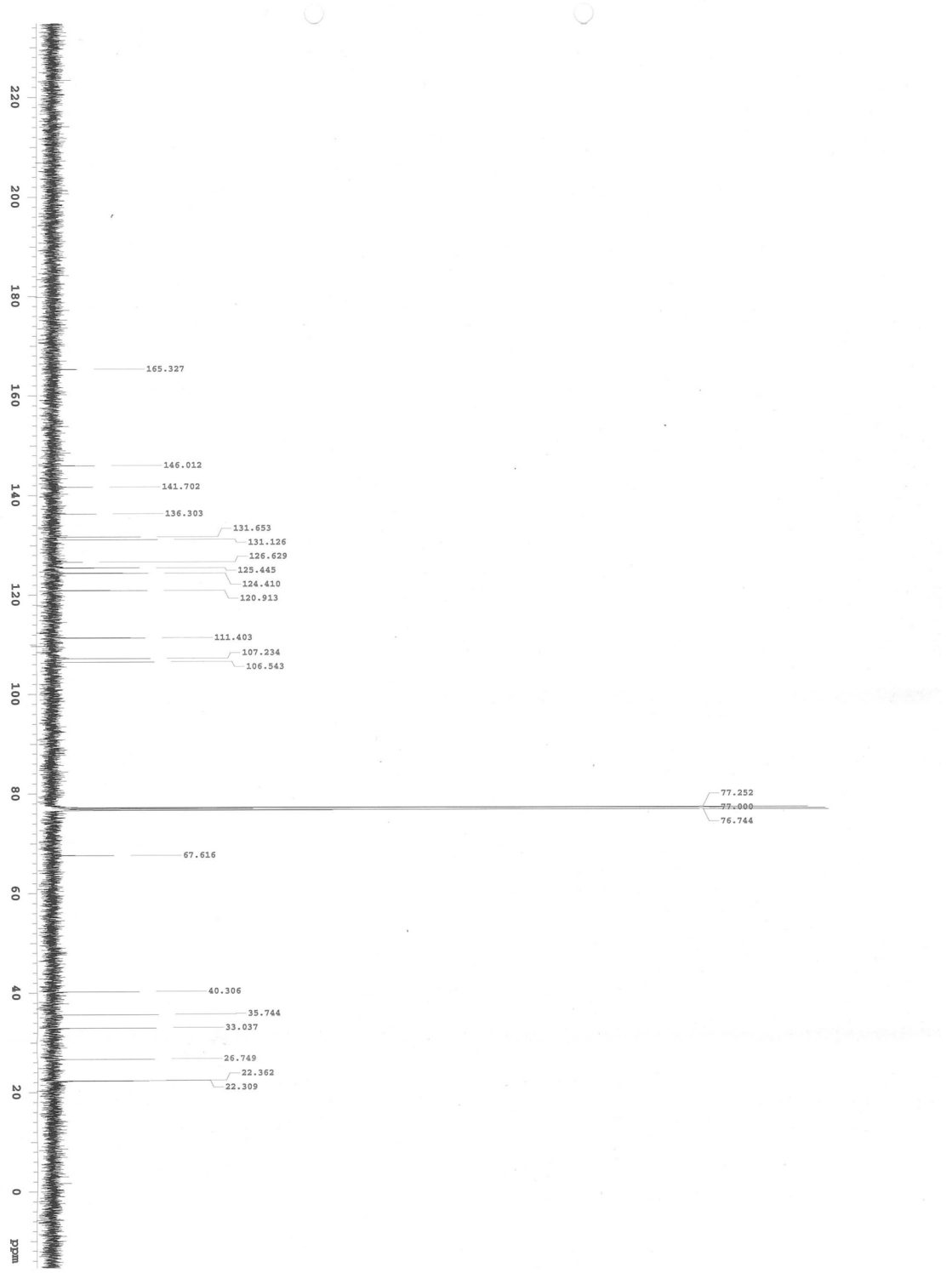
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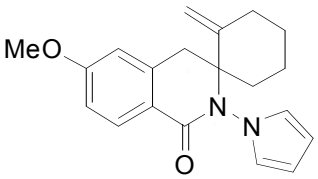
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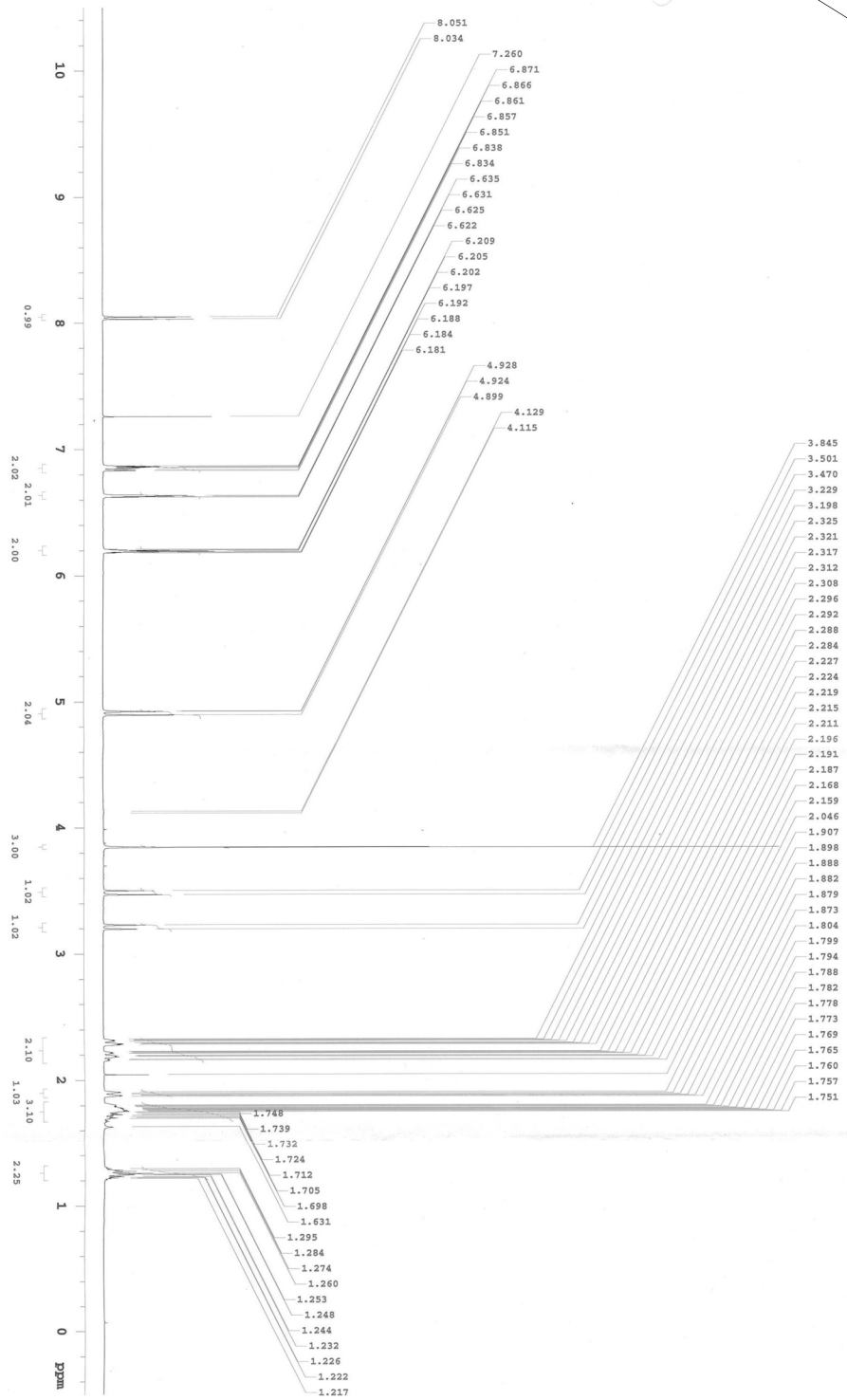
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2.227
2.224
2.219
2.215
2.211
2.196
2.191
2.187
2.168
2.159
2.046
1.907
1.898
1.888
1.882
1.879
1.873
1.804
1.799
1.794
1.788
1.782
1.778
1.773
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1.765
1.760
1.757
1.751
1.748
1.739
1.732
1.724
1.712
1.705
1.698
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1.284
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1.260
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1.244
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1.226
1.222
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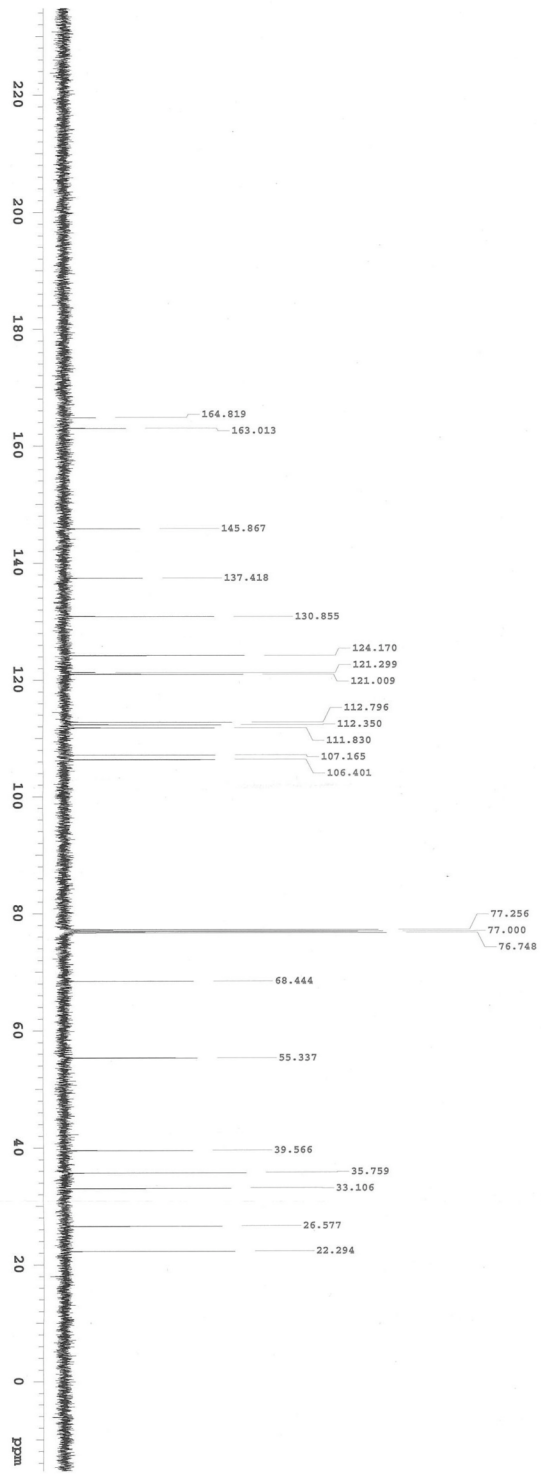
3.10
2.25
ppm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112.350
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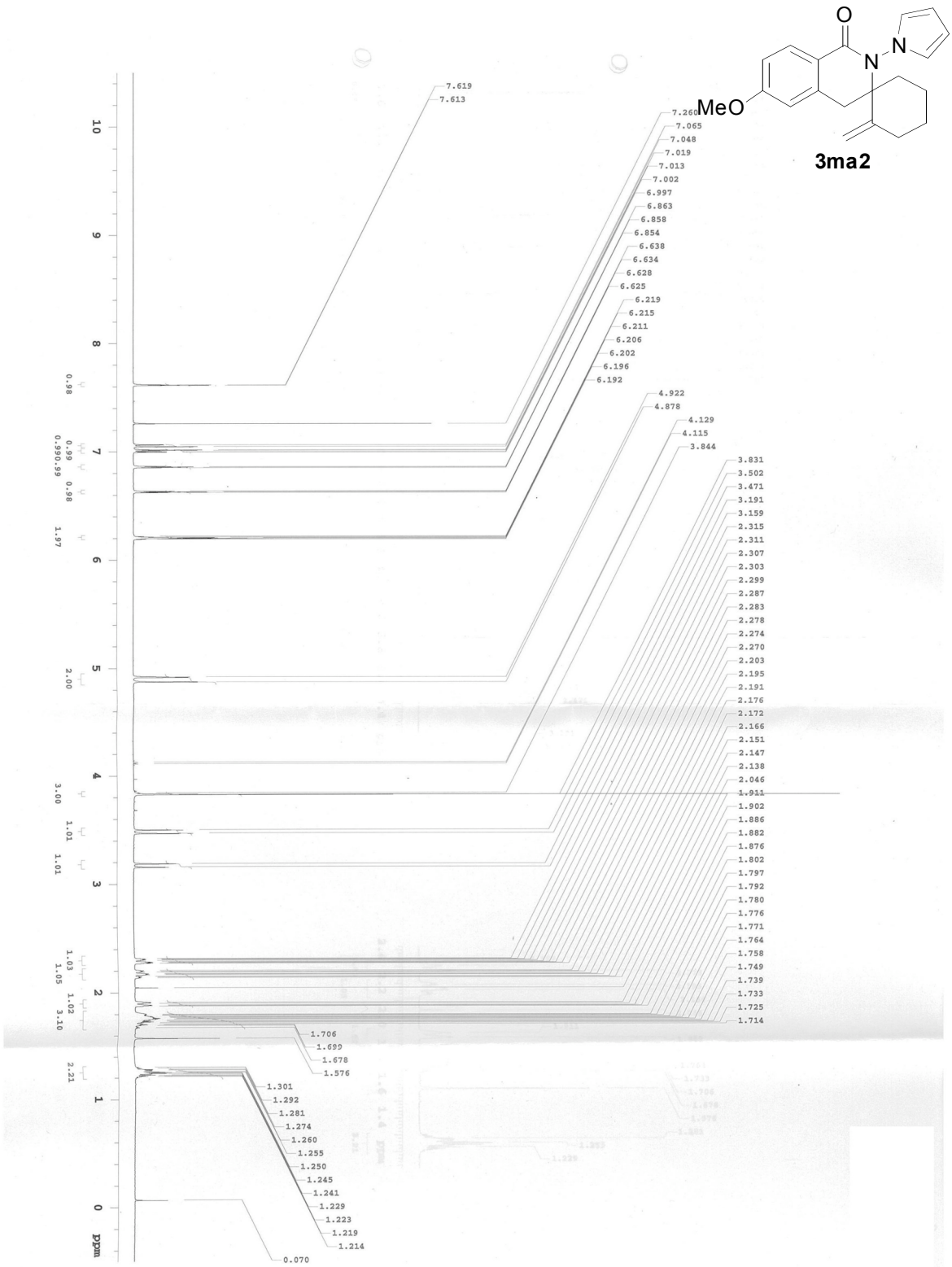
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N
MeO
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6.211
6.206
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4.115
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3.831
3.502
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3.191
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2.311
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2.303
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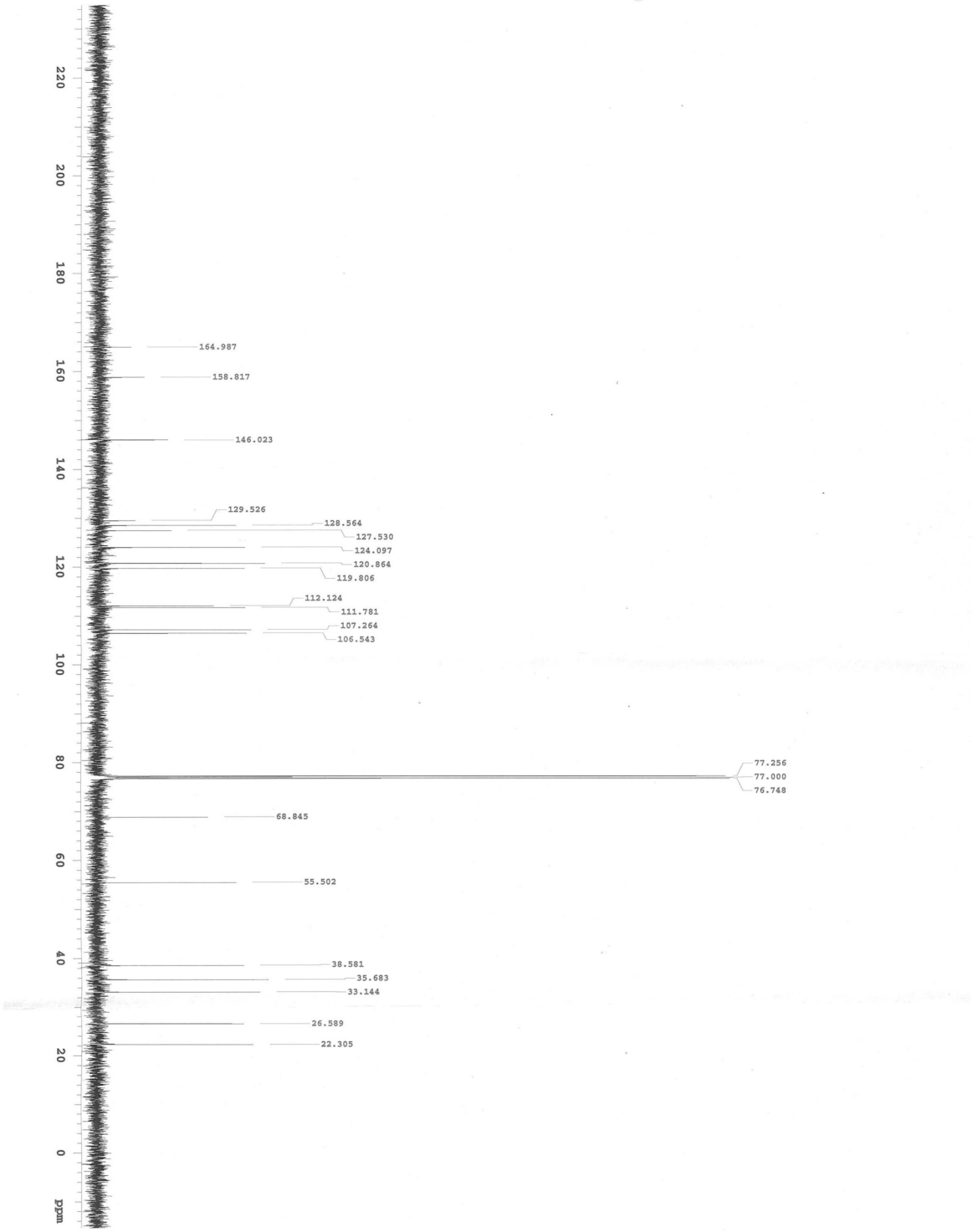
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111.781
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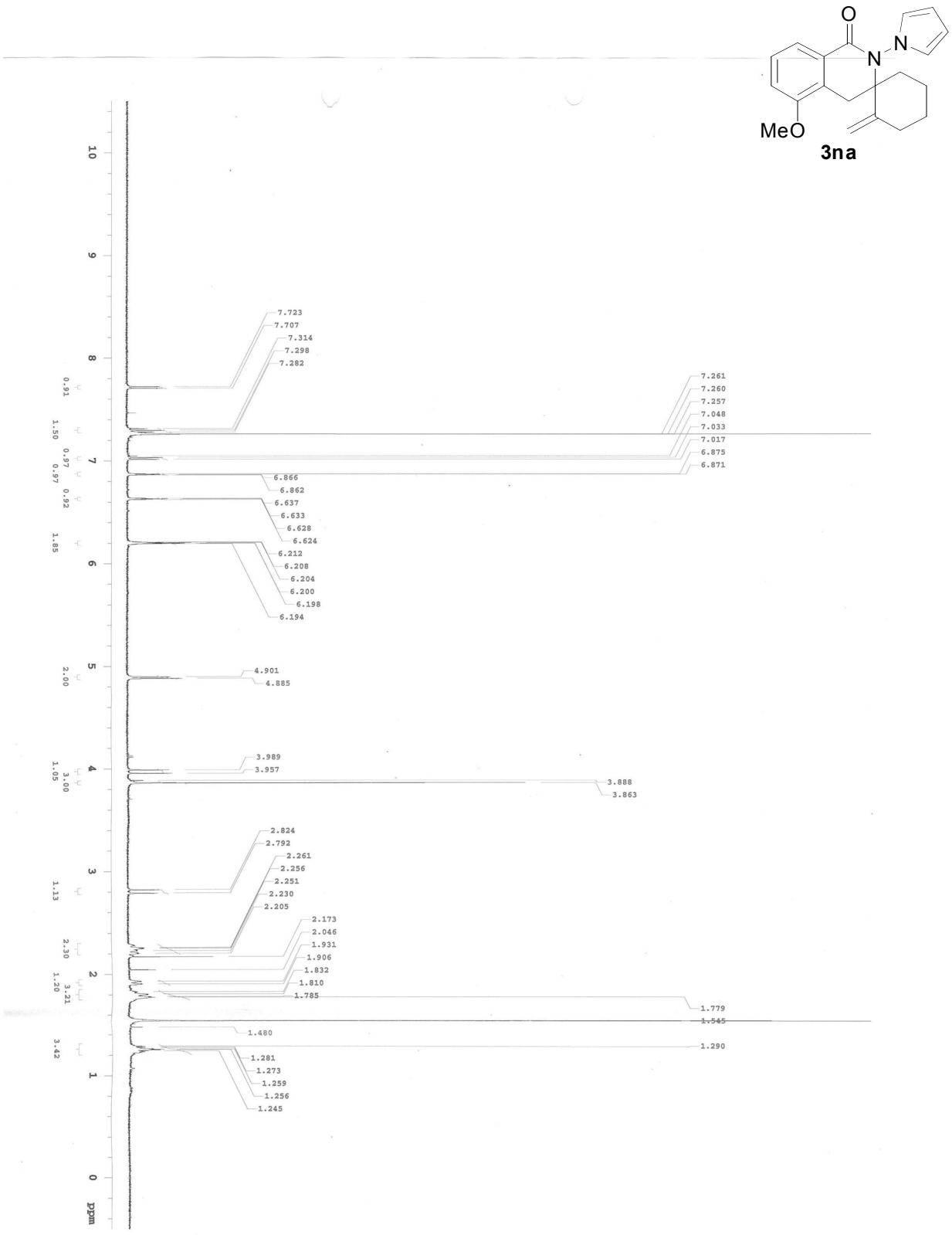
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6.637
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6.628
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6.208
6.204
6.200
6.198
6.194
4.901
4.885
3.989
3.957
3.888
3.863
2.824
2.792
2.261
2.256
2.251
2.230
2.205
2.173
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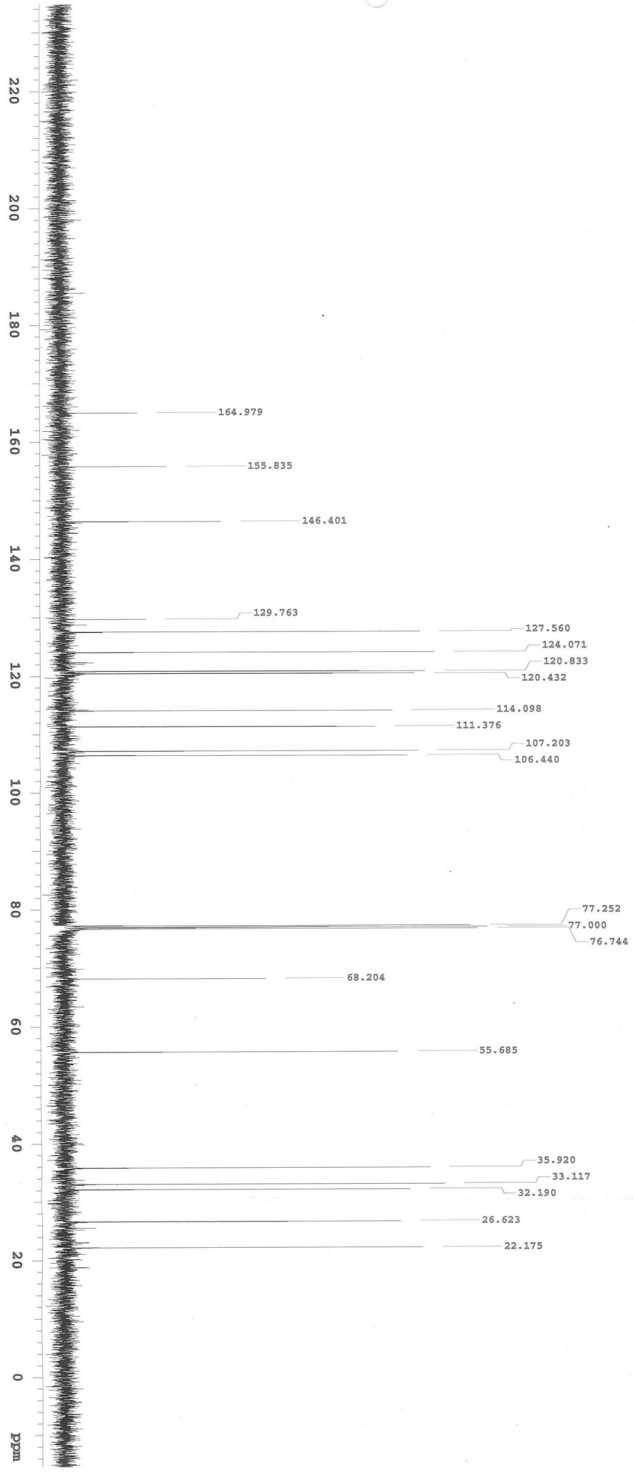
1.245
ppm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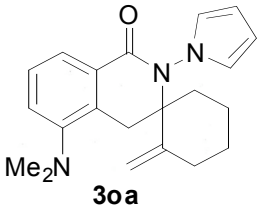
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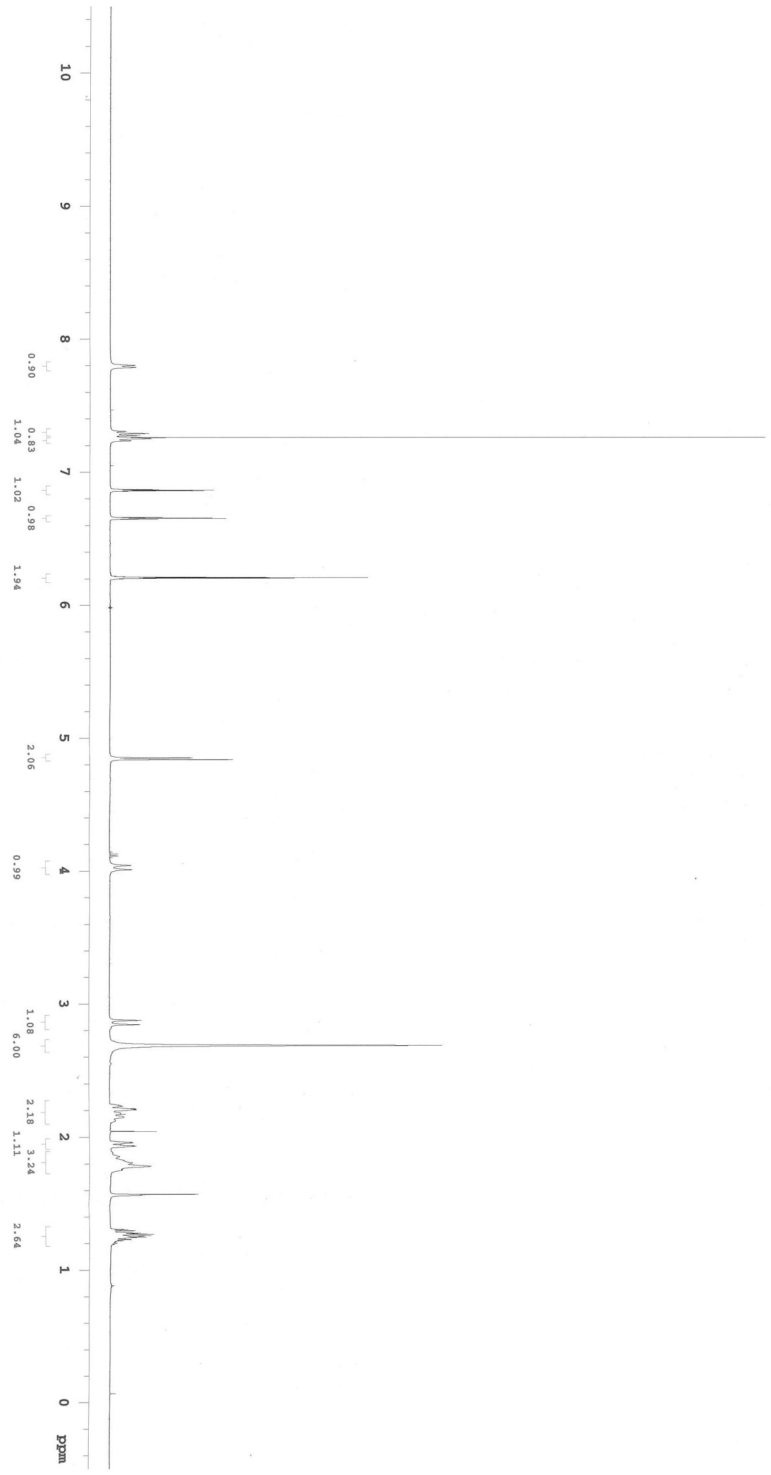
N
Me2N

3oa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8
7
6
5
4
3
2
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0
ppm
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1.94
2.06
0.99
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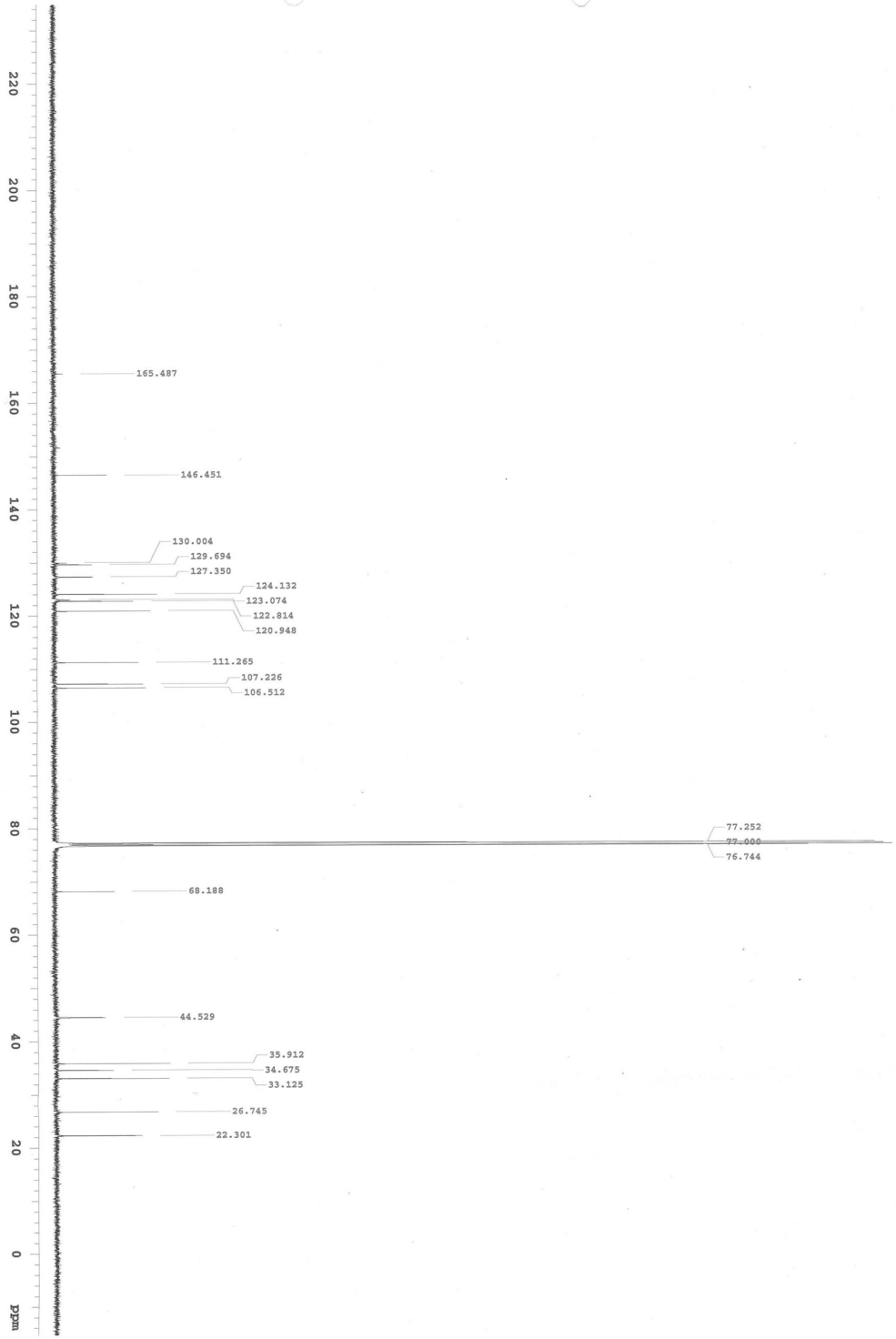
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20
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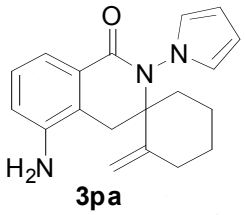
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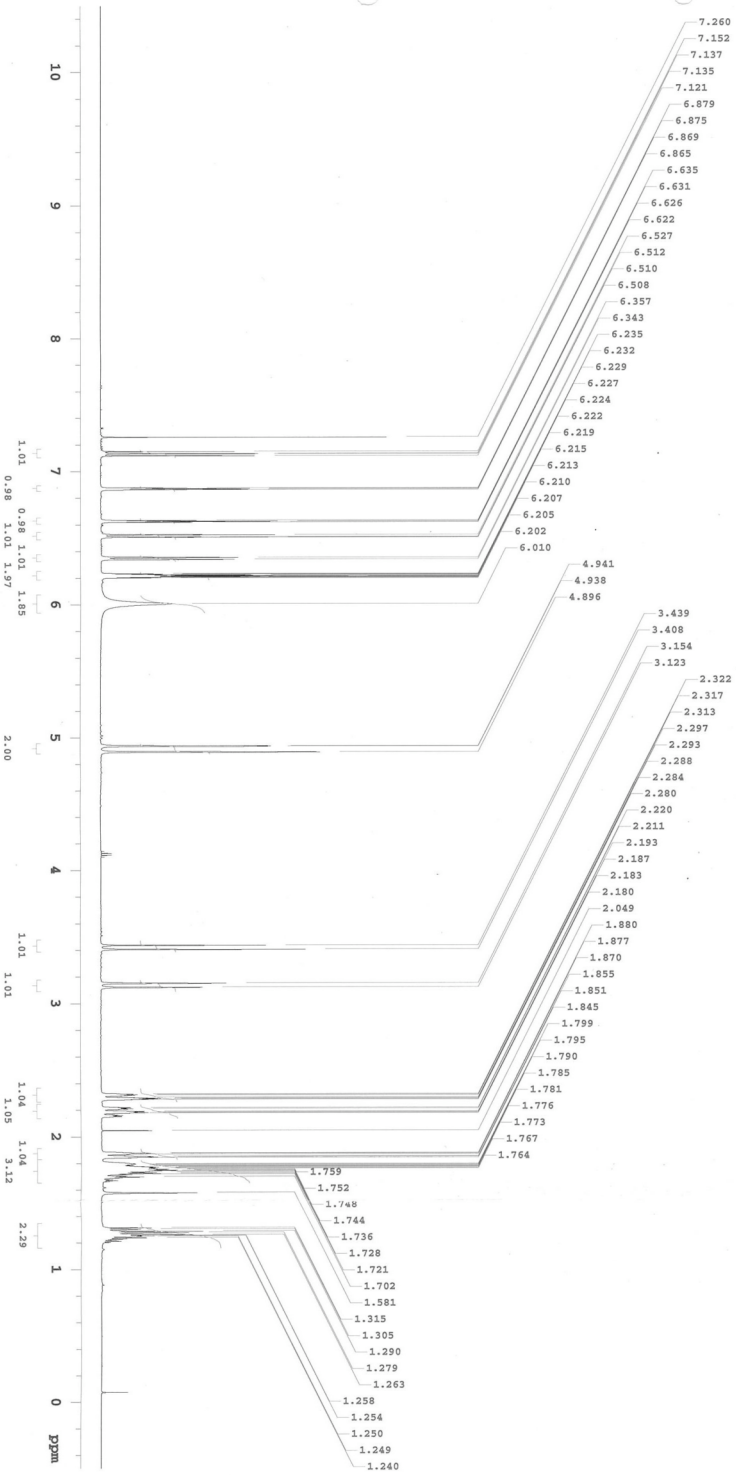
N
N
H2N

3pa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6.527
6.512
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6.227
6.224
6.222
6.219
6.215
6.213
6.210
6.207
6.205
6.202
6.010
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4.938
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2.293
2.288
2.284
2.280
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2.211
2.193
2.187
2.183
2.180
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1.855
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1.845
1.799
1.795
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1.773
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0.98
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1.01
1.97
1.85
2.00
1.01
1.01
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7
6
5
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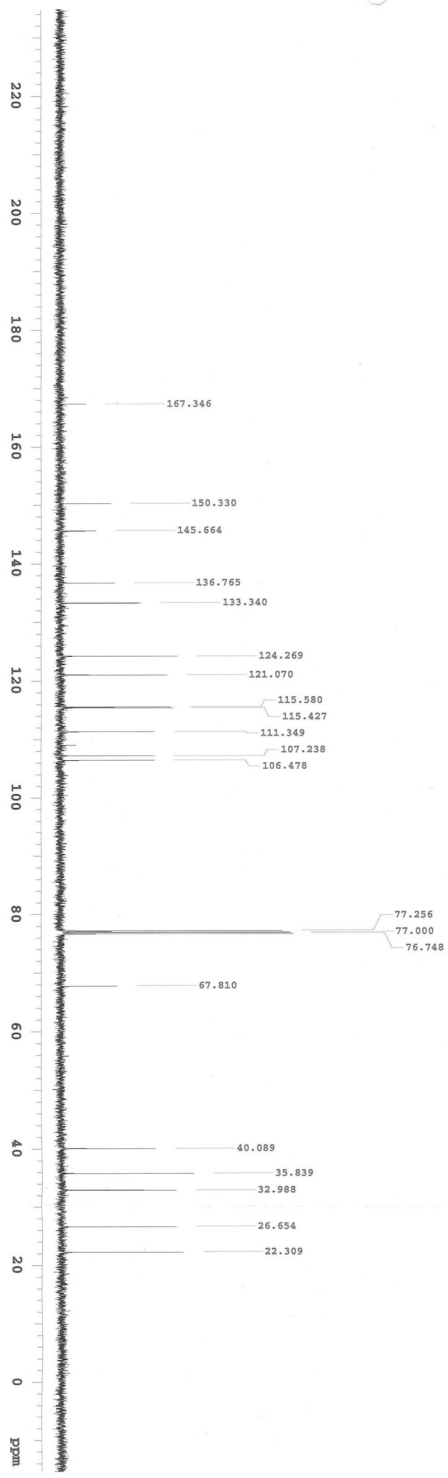
ppm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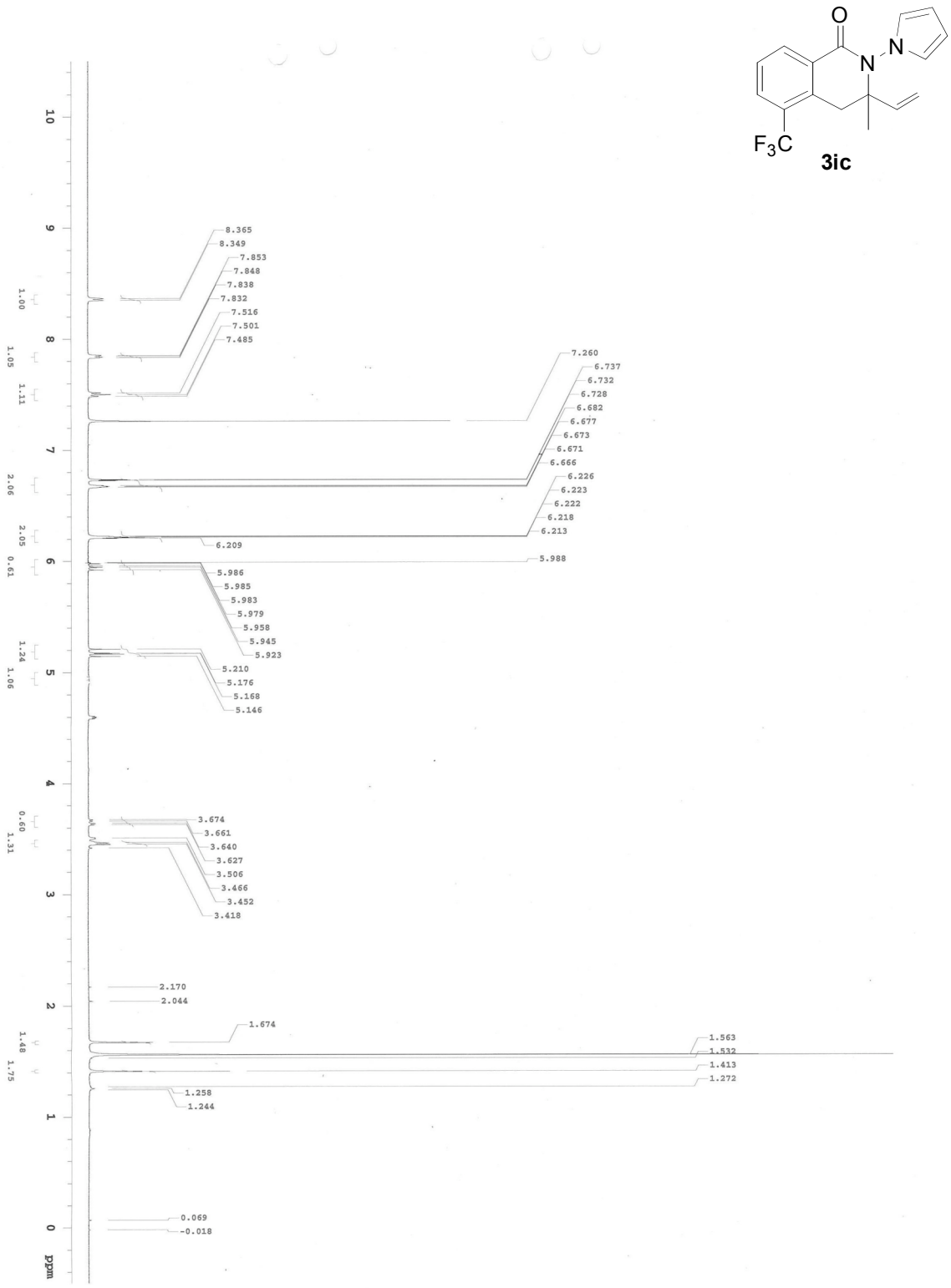
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N
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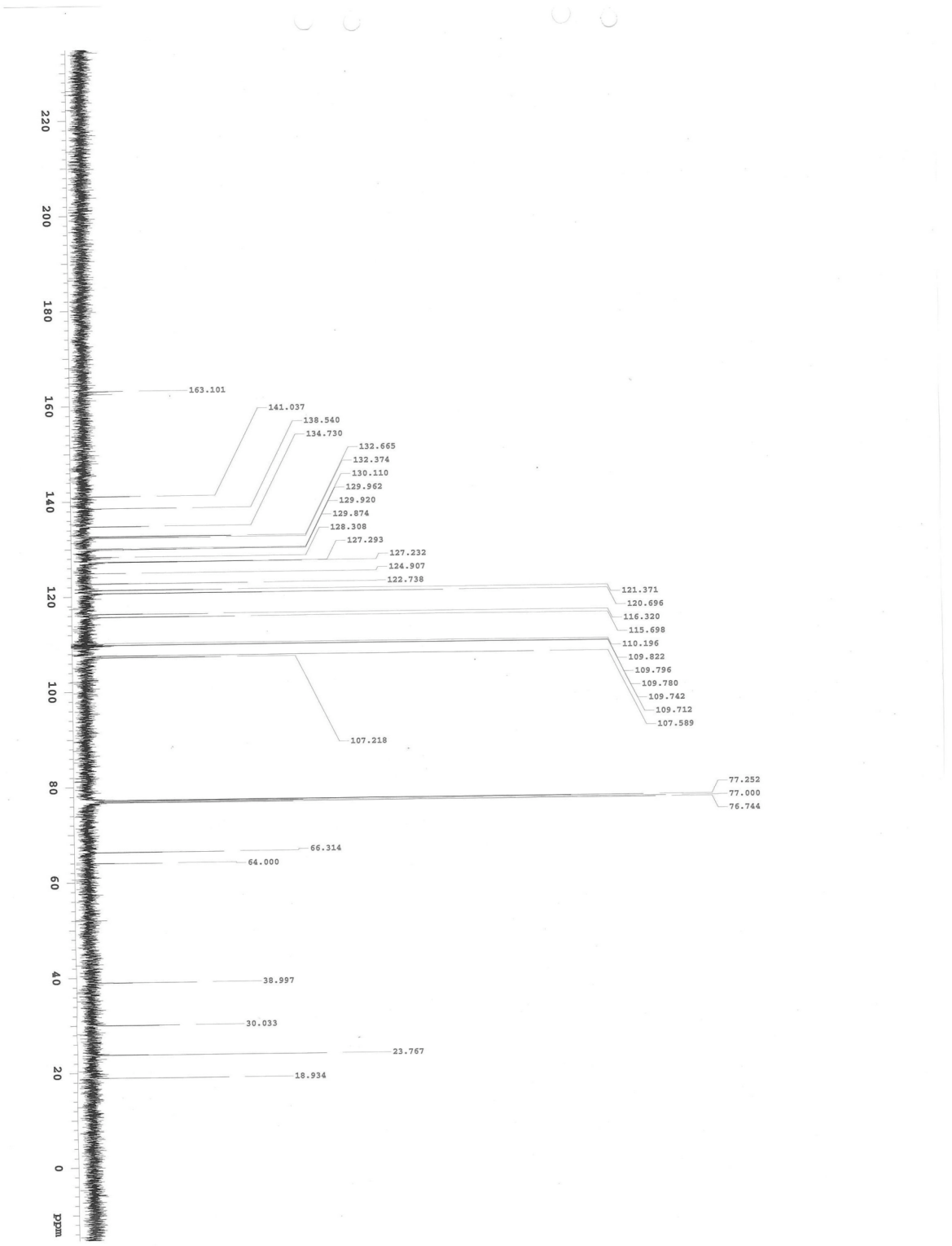
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ppm
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122.738
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115.698
110.196
109.822
109.796
109.780
109.742
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107.589
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23.767
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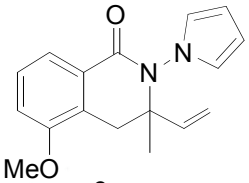
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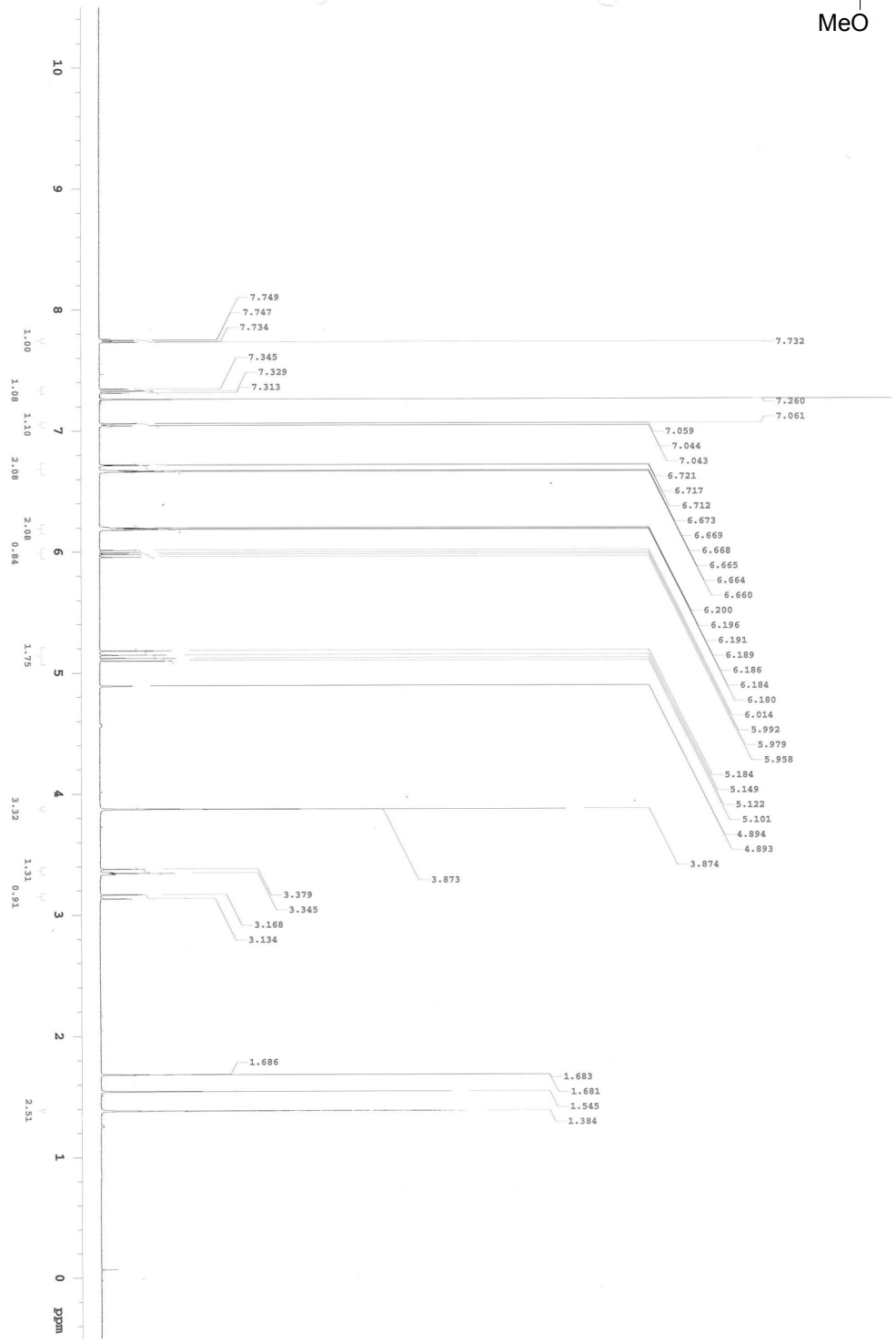
N
MeO

3nc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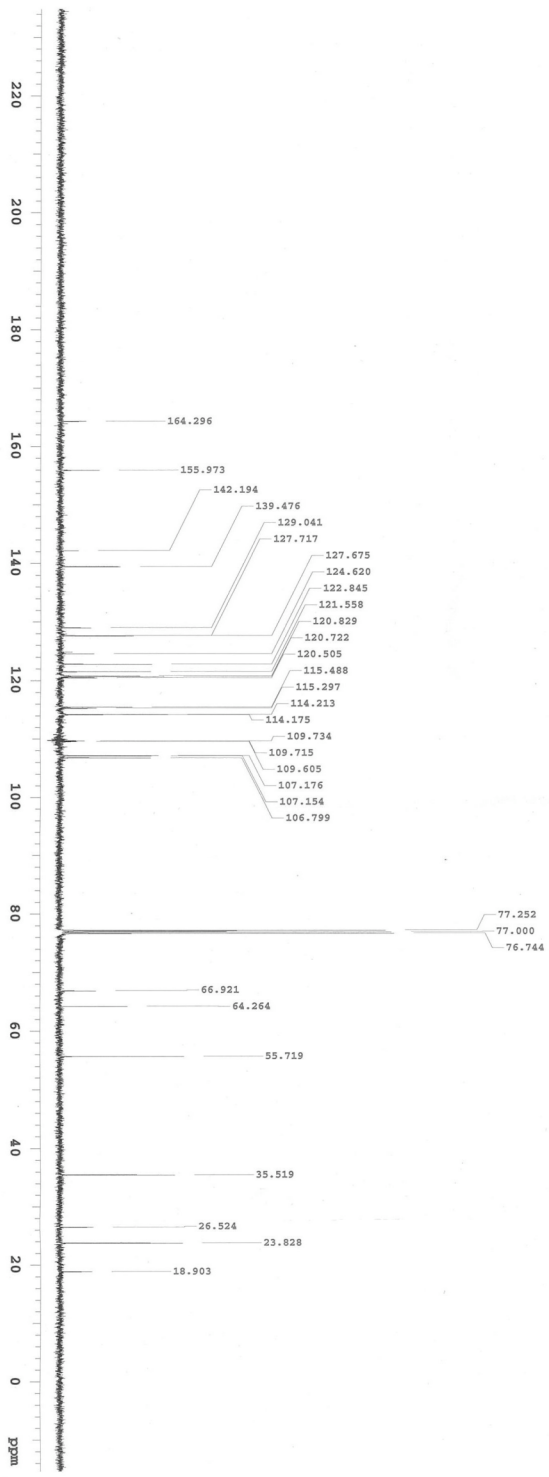
ppm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109.605
107.176
107.154
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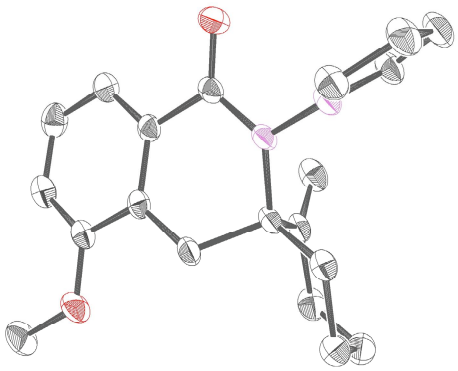
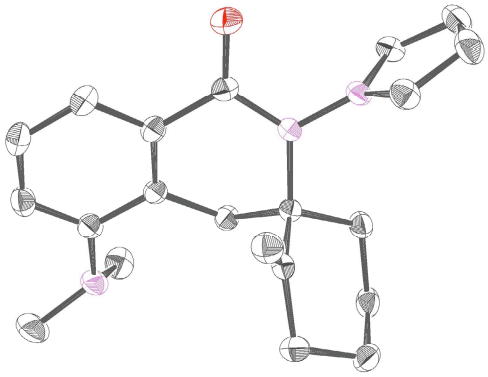
18.903

ORTEP Drawing of 3na



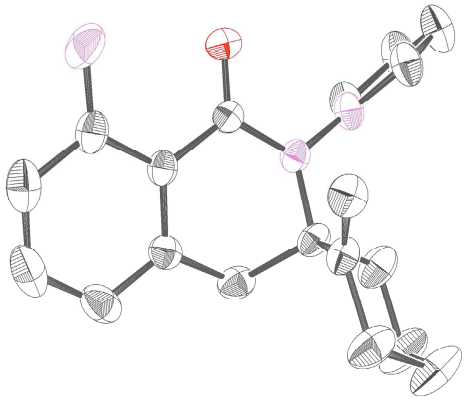
Empirical formula	C20 H22 N2 O2	
Formula weight	322.40	
Temperature	273(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 9.174(4) Å	α = 90°.
	b = 17.881(8) Å	β = 97.844(8)°.
	c = 10.373(4) Å	γ = 90°.
Volume	1685.7(12) Å^3	
Z	4	
Density (calculated)	1.270 Mg/m^3	
Absorption coefficient	0.083 mm^{-1}	
F(000)	688	
Crystal size	0.60 x 0.30 x 0.20 mm^3	
Theta range for data collection	2.24 to 27.09°.	
Index ranges	-11<=h<=11, -22<=k<=22, -7<=l<=13	
Reflections collected	9696	
Independent reflections	3657 [R(int) = 0.0843]	
Completeness to theta = 27.09°	98.3 %	
Absorption correction	None	
Max. and min. transmission	0.9837 and 0.9521	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3657 / 0 / 218	
Goodness-of-fit on F^2	0.913	
Final R indices [I>2sigma(I)]	R1 = 0.0574, wR2 = 0.1330	
R indices (all data)	R1 = 0.1267, wR2 = 0.1659	
Largest diff. peak and hole	0.254 and -0.304 e.Å^{-3}	

ORTEP Drawing of 3oa



Empirical formula	C21 H25 N3 O	
Formula weight	335.44	
Temperature	273(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 9.6129(10) Å	α = 90°.
	b = 15.9000(16) Å	β = 109.191(2)°.
	c = 12.4424(13) Å	γ = 90°.
Volume	1796.1(3) Å^3	
Z	4	
Density (calculated)	1.241 Mg/m^3	
Absorption coefficient	0.078 mm^{-1}	
F(000)	720	
Crystal size	1.00 x 0.50 x 0.10 mm^3	
Theta range for data collection	2.16 to 27.01°.	
Index ranges	-12<=h<=12, -20<=k<=16, -15<=l<=11	
Reflections collected	10825	
Independent reflections	3913 [R(int) = 0.0218]	
Completeness to theta = 27.01°	99.7 %	
Absorption correction	None	
Max. and min. transmission	0.9923 and 0.9264	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3913 / 0 / 228	
Goodness-of-fit on F^2	1.026	
Final R indices [I>2sigma(I)]	R1 = 0.0479, wR2 = 0.1304	
R indices (all data)	R1 = 0.0611, wR2 = 0.1411	
Largest diff. peak and hole	0.286 and -0.158 e.Å^{-3}	

ORTEP Drawing of 3pa



Empirical formula	C19 H21 N3 O	
Formula weight	307.39	
Temperature	273(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 10.6196(11) Å	α = 91.696(2)°.
	b = 11.8470(12) Å	β = 104.085(2)°.
	c = 13.8430(15) Å	γ = 97.966(2)°.
Volume	1669.3(3) Å^3	
Z	4	
Density (calculated)	1.223 Mg/m^3	
Absorption coefficient	0.077 mm^{-1}	
F(000)	656	
Crystal size	0.80 x 0.60 x 0.20 mm^3	
Theta range for data collection	1.52 to 27.04°.	
Index ranges	-11<=h<=13, -15<=k<=15, -15<=l<=17	
Reflections collected	10279	
Independent reflections	7096 [R(int) = 0.0146]	
Completeness to theta = 27.04°	96.8 %	
Absorption correction	None	
Max. and min. transmission	0.9847 and 0.9407	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7096 / 0 / 415	
Goodness-of-fit on F^2	1.034	
Final R indices [I>2sigma(I)]	R1 = 0.0520, wR2 = 0.1354	
R indices (all data)	R1 = 0.0743, wR2 = 0.1502	
Largest diff. peak and hole	0.229 and -0.175 e.Å^{-3}	