Supplementary Information

Atropselective Synthesis of *N*-Aryl Pyridones *via* Dynamic Kinetic Resolution Enabled by Non-Covalent Interactions

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Contents

1 General Experimental

1.1 Naming and Numbering of Compounds

Systematic compound names are those generated by ChemBioDraw™ Ultra version 15.1.0.144 (Perkin Elmer) following IUPAC nomenclature.

1.2 Solvents and Reagents

Reactions were carried out under a nitrogen atmosphere in oven-dried glassware unless otherwise stated. Standard inert atmosphere techniques were used in handling all air- and moisture-sensitive reagents. Where necessary toluene and DMF (from commercial sources) were degassed prior to use by sparging with argon or nitrogen (15 min). Anhydrous and oxygen-free THF was obtained by distillation from Na/benzophenone. Other solvents and reagents were used directly as received from commercial suppliers.

1.3 Chromatography

Flash column chromatography was carried out using Fluorochem 60 40-63 micron silica gel. Thin-layer chromatography was carried out using Merck Kieselgel 60 F254 (230-400 mesh) fluorescent treated silica, visualized under UV light (254 nm) or by staining with aqueous potassium permanganate solution, ninhydrin or ceric ammonium molybdate solutions.

1.4 Analytical Techniques

^{1}H and ^{13}C NMR spectra were recorded using a Bruker Ascend 600 or Avance 400 MHz spectrometer running TopSpin™ 3.6.3 software and are quoted in ppm for measurement against tetramethylsilane. Where no tetramethylsilane was present, spectra are referenced relative to the residual non-deuterated solvent peaks. Unless otherwise stated spectra were acquired at 298 K. Topspin™ was used for processing and viewing NMR data. Chemical shifts (δ) are given in parts per million (ppm), and coupling constants (*J*) are given in Hertz (Hz). The ^{1}H NMR spectra are reported as follows: δ / ppm (number of protons, multiplicity, coupling constant *J* / Hz (where appropriate), assignment). Multiplicity is abbreviated as follows: s = singlet, br = broad, d = doublet, t = triplet, q = quartet, m = multiplet. The numbering scheme used for NMR assignment is arbitrary and does not follow any particular convention. The ^{13}C NMR spectra are reported in δ / ppm. Where necessary or appropriate, two-dimensional (COSY, HSQC, HMBC, NOESY or ROESY) NMR experiments were used to assist the assignment of signals in the ^{1}H and ^{13}C NMR spectra. In some cases, complete assignment of spectra was not possible (in particular, aromatic CHs corresponding to multiple phenyl groups overlapped significantly); in these cases only a partial assignment is reported.

High-performance liquid chromatography (HPLC) was conducted using an Agilent 1220 Infinity II instrument using an isocratic acetonitrile/water eluent mixture.

Liquid chromatography-mass spectrometry (LCMS) analysis was conducted using an instrument comprising an Agilent 1260 HPLC (equipped with Infinity II quaternary pump, vial sampler, integrated column compartment and variable wavelength detector) and 6125C MSD single quadrupole mass spectrometer. Samples were analysed using an Agilent Infinitylab poroshell 120 column (2.7 µm, 2.1 x 150 mm) under an acetonitrile/water gradient with 0.1% HCOOH additive.

Infra-red (IR) spectra were recorded on an Agilent Cary 630 spectrometer equipped with an attenuated total refractance (ATR) accessory. Samples were deposited on the ATR as a thin film. Only selected maximum absorbances (ν_{max}) of the most intense peaks are reported (cm^{-1}).

High resolution mass spectra (HRMS) were recorded by Analytical Services and Environmental Projects (ASEP) at Queen's University Belfast on a Waters LCT Premier ToF mass spectrometer using the electrospray ionisation (ESI) technique.

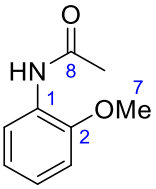
Optical rotations were recorded at the sodium D-line (589 nm) using a Perkin Elmer 341 polarimeter at a temperature of 20 °C and are reported in degrees using concentrations (*c*) in g·100 mL^{-1}. Reported values are the average of eight readings.

Melting points were determined for compounds where a preparative recrystallization was carried out. These were acquired on a Stuart SMP10 digital melting point apparatus. Values are given in °C and are uncorrected.

2 Experimental Procedures and Characterization Data

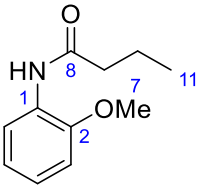
2.1 Acetamides (1)

N-(2-Methoxyphenyl)acetamide (1a)



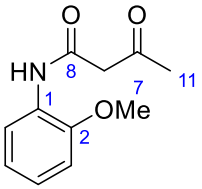
To a solution of 2-aminoanisole (4.58 g, 40.6 mmol, 1.0 equiv.) in dichloromethane (150 mL) was added Ac_2O (4.6 mL, 48.7 mmol, 1.2 equiv.), flushed with Ar and was stirred for 20 mins at RT. The reaction mixture was quenched with saturated $NaHCO_3$ aqueous solution (*ca*. 3 x 10 mL/mmol), extracted with dichloromethane (*ca*. 3 x 10 mL/mmol) and dried over anhydrous magnesium sulfate. The solvent was removed *in vacuo* affording **1a** as an off-white fluffy solid (6.51 g, 97%). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 8.35 (1H, dd, *J* = 8.1, 1.3 Hz, H6), 7.76 (1H, s, NH), 7.03 (1H, td, *J* = 7.8, 1.5 Hz, H5), 6.95 (1H, td, *J* = 7.9, 1.3 Hz, H4), 6.87 (1H, dd, *J* = 7.9, 1.0 Hz, H3), 3.87 (3H, s, H7), 2.20 (3H, s, H9). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 168.1 (C8), 147.6 (C1), 127.7 (C2), 123.6 (C6), 121.1 (C5), 118.8 (C4), 109.9 (C3), 55.7 (C7), 25.0 (C9). **IR**: ν_{max} (thin film): 3246, 2967, 1654, 1595, 1461, 1248, 1113, 1021, 745, 708 cm^{-1}. **HRMS** (ES+): found 166.0868; $C_9H_{12}NO_2$, $[M+H]^+$ requires 166.0868. The data obtained are consistent with those reported in the literature.[1]

N-(2-Methoxyphenyl)butyramide (1b)



To a solution of *o*-anisidine (3.55 g, 28.9 mmol, 1.0 equiv.) in pyridine (35 mL) was added dropwise a solution of butyryl chloride (3.59 mL, 34.6 mmol, 1.2 equiv.) in THF (175 mL) at 0 °C. The mixture was stirred at 0 °C for 3 hours. The reaction mixture was reduced *in vacuo* and crude residue extracted with EtOAc (100 mL), washed with H_2O (75 mL), followed by 1M HCl aqueous solution (75 mL), saturated $NaHCO_3$ aqueous solution (75 mL) and saturated NaCl aqueous solution (75 mL). The organic extracts were dried over anhydrous magnesium sulfate and reduced *in vacuo* to an orange residue. The crude product was purified by flash column chromatography (silica gel 50 g, EtOAc/PE 10 → 20%) affording **1b** as a yellow oil (5.11 g, 91%). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 8.40 (1H, dd, *J* = 7.9, 1.3 Hz, H6), 7.76 (1H, s, NH), 7.03 (1H, td, *J* = 7.6, 1.6 Hz, H5), 6.96 (1H, td, *J* = 7.8, 1.5 Hz, H4), 6.87 (1H, dd, *J* = 8.0, 1.5 Hz, H3), 3.88 (3H, s, H7), 2.37 (2H, t, *J* = 7.3 Hz, H9), 1.77 (2H, sex, *J* = 7.4 Hz, H10) 1.01 (3H, t, *J* = 7.4 Hz, H11). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 171.1 (C8), 147.6 (C1), 127.8 (C2), 123.4 (C6), 121.1 (C5), 119.7(C4), 109.8 (C3), 55.7 (C7), 40.0 (C9), 19.11 (C10), 13.8 (C11). **IR**: ν_{max} (thin film): 3317, 2959, 1669, 1599, 1520, 1457, 1248, 1025, 745 cm^{-1}. **HRMS** (ES+): found 194.1185; $C_{11}H_{16}NO_2$, $[M+H]^+$ requires 194.1181. The data obtained are consistent with those reported in the literature.[2]

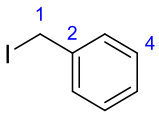
***N*-(2-Methoxyphenyl)-3-oxobutanamide (1c)**



A neat mixture of 2-aminoanisole (2.72 g, 22 mmol) and ethyl acetoacetate (5.6 mL, 44 mmol) and potassium *tert*-butoxide (240 mg, 2.2 mmol) was heated to 130 °C. After 2 h the reaction mixture was cooled to room temperature and the crude residue was purified by flash column chromatography (silica gel, 0% → 20% EtOAc/dichloromethane) to afford the product **1c** as an off-white solid (1.94 g, 42%). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 9.22 (1H, s, NH), 8.32 (1H, dd, *J* = 7.9, 1.5 Hz, H6), 7.06 (1H, td, *J* = 8.0, 1.5 Hz, H5), 6.95 (1H, td, *J* = 8.0, 1.2 Hz, H4), 6.89 (1H, dd, *J* = 8.1, 1.0 Hz, H3), 3.92 (3H, s, H7), 3.60 (2H, s, H9), 2.33 (3H, s, H11). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 204.4 (C10), 163.2 (C8), 148.3 (C1), 127.4 (C2), 124.1 (C6), 121.0 (C5), 120.1 (C4), 110.1 (C3), 55.8 (C7), 50.9 (C9), 25.0 (C11). **IR**: ν_{max} (thin film): 3295, 2940, 1714, 1669, 1599, 1524, 1461, 1356, 1252, 1118, 1025, 745 cm^{-1}. **HRMS** (ES+): found 208.0965; $C_{11}H_{14}NO_3$, $[M+H]^+$ requires 208.0974. The data obtained are consistent with those reported in the literature.[3]

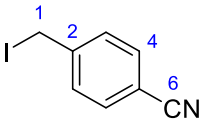
2.2 Benzyl Iodides

(Iodomethyl)benzene (S1)



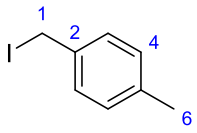
NaI (4.50 g, 30.0 mmol, 2.0 equiv.) was dissolved in acetone (20 mL) at 0 °C. To the solution, was added BnBr (1.80 mL, 15.0 mmol, 1.0 equiv.) and the resulting mixture was stirred at 0 °C for 6 h. The reaction mixture was washed with brine (50 mL) and extracted with Et_2O (3 x 50 mL), dried over anhydrous magnesium sulfate and the solvent was removed *in vacuo* giving a pink oil. PE (*ca*. 1 mL) was added and crude product placed on ice as pink crystals formed. Mother liquor was removed via pipette at 0 °C affording **S1** as a pink solid (3.06 g, 94%). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.39-7.36 (2H, m, H3), 7.31-7.21 (3H, m, H4, H5), 4.46 (2H, s, H1). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 139.3 (C2), 128.8 (C3), 128.7 (C4), 127.9 (C5), 5.7 (C1). **IR**: ν_{max} (thin film): 3026, 1699, 1453, 1211, 1155, 749, 689 cm^{-1}. **HRMS** (ES+): found 218.9705; C_7H_8I, $[M+H]^+$ requires 218.9796. The data obtained are consistent with those reported in the literature.[4]

4-(Iodomethyl)benzonitrile (S2)



NaI (3.0 g, 20.0 mmol, 2.0 equiv.) was dissolved in acetone (12 mL) at 0 °C. To the solution, was added 4-cyanobenzyl bromide (1.96 g, 10.0 mmol, 1.0 equiv.) and the resulting mixture was stirred at 0 °C for 4 h. The reaction mixture was washed with brine (30 mL) and extracted with Et_2O (3 x 30 mL), dried over anhydrous magnesium sulfate and the solvent was removed *in vacuo*. The residue was purified by trituration (petroleum ether) to afford **S2** as a pale yellow solid (2.26 g, 93%). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.60-7.57 (2H, m, H4), 7.48-7.45 (2H, m, H3), 4.43 (2H, s, H1). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 144.7 (C2), 132.6 (2C, C4), 129.7 (2C, C3), 118.5 (C6), 111.6 (C5), 2.8 (C1). **IR**: ν_{max} (thin film): 2221, 1707, 1602, 1505, 1159, 842, 734 cm^{-1}. **HRMS** (ES+): found 243.9889; C_8H_7IN, $[M+H]^+$ requires 243.9798. The data obtained are consistent with those reported in the literature.[5]

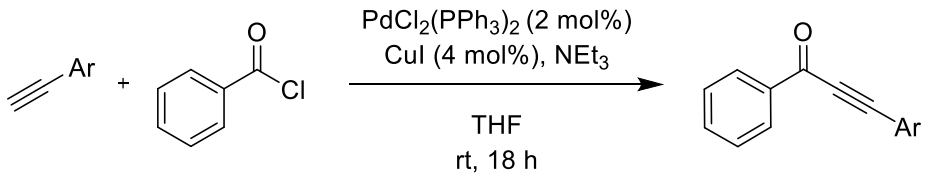
1-(Iodomethyl)-4-methylbenzene (S3)



NaI (3.0 g, 20.0 mmol, 2.0 equiv.) was dissolved in acetone (12 mL) at 0 °C. To the solution, was added 4-methylbenzyl bromide (1.85 g, 10.0 mmol, 1.0 equiv.) and the resulting mixture was stirred at 0 °C for 4 h. The reaction mixture was washed with brine (30 mL) and extracted with Et_2O (3 x 30 mL), dried over anhydrous magnesium sulfate and the solvent was removed *in vacuo.* The crude residue was purified by recrystallization from hot petroleum ether to afford **S3** (1.41 g, 68%) as a white crystalline solid. **M.p.** = 46-48 °C (petroleum ether), **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.28 (2H, d, *J* = 8.1 Hz, H3), 7.11 (2H, d, *J* = 7.8 Hz, H4), 4.46 (2H, s, H1), 2.32 (3H, s, H6). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 137.8 (C2), 136.3 (C5), 129.6 (2C, C4), 128.7 (2C, C3), 21.3 (C6), 6.2 (C1). **IR**: ν_{max} (thin film): 2911, 1610, 1513, 1427, 1207, 1148, 812 cm^{-1}. **HRMS** (ES+): found 232.9787; $C_8H_{10}I$, $[M+H]^+$ requires 232.9782. The data obtained are consistent with those reported in the literature.[5]

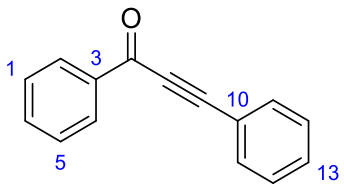
2.3 Alkynones (2)

General Procedure A (*Preparation of Prop-2-yn-1-ones*)



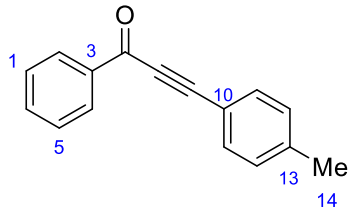
A flask was charged with benzoyl chloride, $PdCl_2(PPh_3)_2$, and Et_3N, THF was added, and the resulting solution stirred for 10 min after which CuI was added changing the colour (substrate dependent) upon stirring for an additional 10 min. Arylacetylene was added and the flask was sealed and left to stir at room temperature for 18 h. The reaction mixture was quenched with 0.1 N HCl (*ca.* 2 x 10 mL/mmol), washed with saturated NH_4Cl aqueous solution (*ca.* 10 mL/mmol), extracted with dichloromethane (*ca.* 3 x 10 mL/mmol), and dried over anhydrous magnesium sulfate. The solvent was removed *in vacuo* resulting in a crude solid which was purified by column chromatography (silica gel, 2.5% → 5% Et_2O/PE) affording the desired alkynone.

1,3-Diphenylprop-2-yn-1-one (2a)



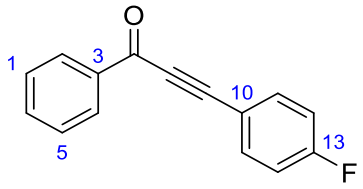
Prepared according to **General Procedure A** using benzoyl chloride (3.18 g, 27.3 mmol, 1.2 equiv.), $PdCl_2(PPh_3)_2$ (319 mg, 460 µmol, 2.0 mol%) and Et_3N (3.63 mL, 27.3 mmol, 1.2 equiv.), THF (40 mL), CuI (174 mg, 0.91 mmol, 4.0 mol%) and phenylacetylene (2.50 g, 22.8 mmol, 1.0 equiv.) **2a** was obtained as a yellow oil (4.21 g, 90%). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 8.23 (2H, dd, *J* = 8.6, 1.4 Hz, PhH), 7.69 (2H, m, PhH), 7.64 (1H, t, *J* = 7.6 Hz, PhH), 7.54-7.47 (3H, m, PhH) 7.45-7.41 (2H, m, PhH). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 178.0, 136.9, 134.1, 133.1 (2C), 130.8, 129.6 (2C), 128.7 (2C), 128.6 (2C), 120.1, 93.1, 86.9. **IR**: ν_{max} (thin film): 3060, 2195, 1636, 1490, 1446, 1312, 1282, 1207, 756, 689 cm^{-1}. **HRMS** (ES+): found 207.0815; $C_{15}H_{11}O$, $[M+H]^+$ requires 207.0810. The data obtained are consistent with those reported in the literature.[6]

1-Phenyl-3-(*p*-tolyl)prop-2-yn-1-one (2b)



Prepared according to **General Procedure A** using benzoyl chloride (1.32 mL, 11.4 mmol, 1.5 equiv.), $PdCl_2(PPh_3)_2$ (105 mg, 150 µmol, 2.0 mol%) and Et_3N (1.30 mL, 9.4 mmol, 1.25 equiv.), THF (16 mL), CuI (29 mg, 0.15 mmol, 2.0 mol%) and 4-methylphenylacetylene (0.96 mL, 7.58 mmol, 1.0 equiv.) **2b** was obtained as a yellow solid (1.39 g, 84%). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 8.24-8.22 (2H, m, PhH), 7.65-7.58 (3H, m, PhH, ArH), 7.52 (2H, t, *J* = 7.8 Hz, ArH), 7.23 (2H, d, *J* = 7.9 Hz, ArH), 2.41 (3H, s, CH_3). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 178.0, 141.5, 137.0, 134.0, 133.1 (2C), 129.6 (2C), 129.5 (2C), 128.6 (2C), 117.1, 93.8, 86.8, 21.8. **IR**: ν_{max} (thin film): 2191, 1632, 1599, 1312, 1170, 1006, 812, 697 cm^{-1}. **HRMS** (ES+): found 221.0966; $C_{16}H_{13}O$, $[M+H]^+$ requires 221.0966. The data obtained are consistent with those reported in the literature.[7]

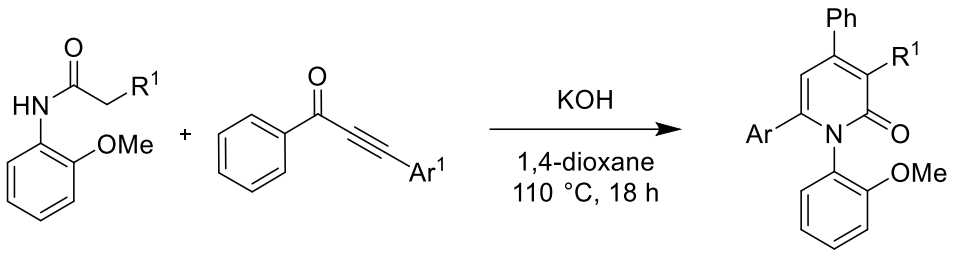
3-(4-Fluorophenyl)-1-phenylprop-2-yn-1-one (2c)



Prepared according to **General Procedure A** using benzoyl chloride (0.58 mL, 5.0 mmol, 1.5 equiv.), $PdCl_2(PPh_3)_2$ (46 mg, 66 µmol, 2.0 mol%), Et_3N (0.69 mL, 4.9 mmol, 1.5 equiv.), THF (8 mL), CuI (13 mg, 66 µmol, 2.0 mol%) and 4-fluorophenyl acetylene (398 mg, 3.31 mmol, 1.0 equiv.). **2c** was obtained as a lime green foam (598 mg, 81%). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 8.21 (2H, dd, *J* = 8.5, 1.5 Hz, PhH), 7.72-7.62 (3H, m, PhH, ArH), 7.52 (2H, t, *J* = 7.8 Hz, ArH), 7.13 (2H, t, *J* = 8.5 Hz, ArH). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 177.9, 164.1 (d, $^1J_{CF}$ = 250.1 Hz), 136.8, 135.4 (2C, d, $^3J_{CF}$ = 8.9 Hz), 134.2, 129.6 (2C), 128.7 (2C), 116.3 (d, $^4J_{CF}$ = 3.4 Hz), 116.3 (2C, d, $^2J_{CF}$ = 22.4 Hz), 92.0, 86.8. **^{19}F NMR** (376 MHz, $CDCl_3$): δ_F = -106.0. **IR:** ν_{max} (thin film): 2199, 1640, 1595, 1233, 1010, 834, 693 cm^{-1}. **HRMS** (ES+): found 225.0712; $C_{15}H_{10}FO$, $[M+K]^+$ requires 225.0716.[7]

2.4 2′-Methoxyphenyl-2-pyridones

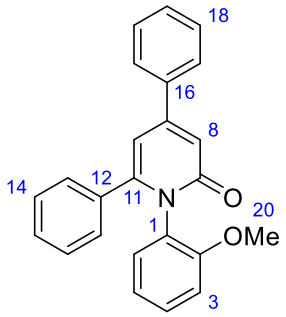
General Procedure B (*Preparation of 2′-methoxyphenyl-2-pyridones*)



This reaction was conducted by analogy to the method reported by Tang and Pen.[8] An oven-dried microwave tube was charged with prop-2-yn-1-one derivatives (1.0 equiv.), 1,4-dioxane (*ca*. 0.3 M), amide (1.0 equiv.) and KOH (1.0 equiv., freshly ground up *via* pestle and mortar). The tube was sealed and stirred at 80 °C for 24-36 h, then allowed to cool to RT. The reaction mixture was diluted with H_2O (*ca*. 5 mL/mmol) and extracted with EtOAc (*ca*. 3 x 5 mL/mmol) and dried

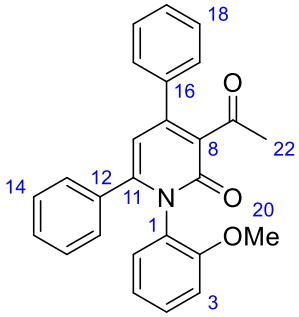
over anhydrous magnesium sulfate. The solvent was removed *in vacuo* resulting in a crude oil which was purified by column chromatography (silica gel, 2.5% → 5% MeOH/dichloromethane) affording the desired product.

1-(2-Methoxyphenyl)-4,6-diphenylpyridin-2(1*H*)-one (S4)



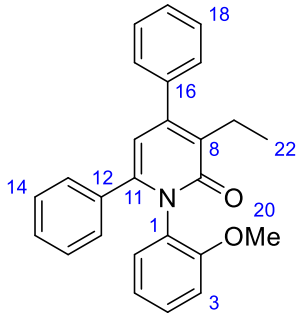
Prepared according to **General Procedure B** using alkynone **2a** (1.77 g, 8.60 mmol, 1.2 equiv.), amide **1a** (1.18 g, 7.17 mmol, 1.0 equiv.), KOH (402 mg, 7.17 mmol, 1.0 equiv.) and 1,4-dioxane (28 mL). Crude mixture purified by flash column chromatography (silica gel, 2.5% → 5% MeOH/dichloromethane) affording **S4** as a cream foam (1.24 g, 49%). Impure fractions were recrystallized from $CHCl_3$/PE giving off-white crystals (112 mg, 4%), in total 1.35 g, 53%. M.p. = 147-149 °C, **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.68 (2H, dd, *J* = 7.9 Hz, H13), 7.50-7.42 (3H, m, H14, 15), 7.23-7.14 (6H, m, H4, 17, 18, 19), 7.09 (1H, dd, *J* = 7.7, 1.5 Hz, H6), 6.93 (1H, d, *J* = 2.0 Hz, H10), 6.87 (1H, td, *J* = 7.6, 1.0 Hz, H5), 6.80 (1H, d, *J* = 8.2 Hz, H3), 6.52 (1H, d, *J* = 1.6 Hz, H8), 3.71 (3H, s, H20). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 163.3 (C7), 154.5 (C9), 151.3 (C2), 150.2 (C11), 137.8 (C16), 135.8 (C12), 130.2 (C19), 129.9 (C15), 129.4 (C4), 129.0 (2C, C18), 128.6 (2C, C17), 128.5 (C5), 127.6 (C1), 127.5 (2C, C14), 126.9 (2C, C13), 120.6 (C3), 116.1 (C6), 111.8 (C8), 107.0 (C10), 55.5 (C20). **IR**: ν_{max} (thin film): 1654,1587, 1498, 1364, 1271, 1118, 1025, 861, 760, 700 cm^{-1}. **HRMS** (ES+): found 354.1431; $C_{24}H_{20}NO_2$, $[M+H]^+$ requires 354.1494. The data obtained are consistent with those reported in the literature.[8]

3-Acetyl-1-(2-methoxyphenyl)-4,6-diphenylpyridin-2(1*H*)-one (S5)



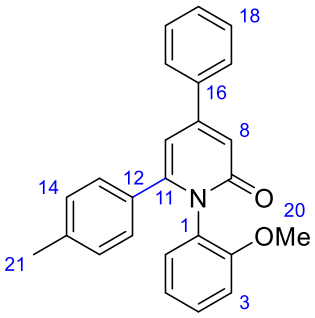
Prepared according to **General Procedure B** using alkynone **2a** (450 mg, 2.18 mmol, 1.2 equiv.), amide **1c** (377 mg, 1.82 mmol, 1.0 equiv.), KOH (102 mg, 1.82 mmol, 1.0 equiv.) and 1,4-dioxane (7 mL). Crude mixture purified by flash column chromatography (silica gel, 2.5% → 5% MeOH/dichloromethane) affording **S5** as a yellow foam (306 mg, 43%). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.44-7.39 (5H, m, H17, 18, 19), 7.24-7.16 (6H, m, H13, 14, 15), 7.12 (1H, dd, *J* = 7.9, 1.8 Hz, H6), 6.89 (1H, td, *J* = 7.6, 1.2 Hz, H5), 6.79 (1H, dd, *J* = 8.5, 1.2 Hz, H3), 6.30 (1H, s, H10), 3.71 (3H, s, H20), 2.49 (3H, s, H22). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 202.4 (C21), 160.6 (C7), 154.4 (C2), 150.8 (C11), 150.8 (C16), 150.8 (C9), 138.0 (C8), 135.0 (C9), 130.1 (C19), 130.0 (C15), 129.4 (C12), 129.0 (C4), 128.9 (C5), 128.6 (2C, C17), 128.4 (2C, C18), 128.0 (2C, C14), 127.6 (2C, C13), 126.9 (C1), 120.6 (C3), 111.8 (C6), 109.7 (C10), 55.5 (C20), 31.8 (C22). **IR**: ν_{max} (thin film): 3060, 2243, 1699, 1640, 1572, 1528, 1271, 1025, 909, 756, 730 cm^{-1}. **HRMS** (ES+): found 396.1593; $C_{26}H_{22}NO_3$, $[M+H]^+$ requires 396.1600.

3-Ethyl-1-(2-methoxyphenyl)-4,6-diphenylpyridin-2(1*H*)-one (S6)



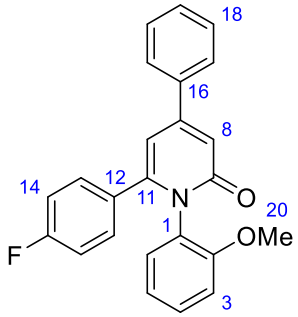
Prepared according to **General Procedure B** using alkynone **2a** (450 mg, 2.18 mmol, 1.2 equiv.), amide **1b** (352 mg, 1.82 mmol, 1.0 equiv.), KOH (102 mg, 1.82 mmol, 1.0 equiv.) and 1,4-dioxane (7 mL). Crude mixture purified by flash column chromatography (silica gel, 2.5% → 5% MeOH/dichloromethane) affording **S6** as an orange foam (359 mg, 52%). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.47-7.37 (5H, m, H17, 18, 19), 7.22-7.09 (7H, m, H6, 13, 14, 15), 6.87 (1H, td, *J* = 7.6, 1.2 Hz, H5), 6.80 (1H, dd, *J* = 8.3, 1.1 Hz, H3), 6.17 (1H, s, H10), 3.71 (3H, s, H20), 2.66-2.52 (2H, m, H21), 1.18 (3H, t, *J* = 7.4 Hz, H22). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 163.0 (C7), 154.6 (C2), 149.2 (C11), 146.2 (C9), 139.9 (C12), 135.8 (C16), 130.8 (C8), 130.2 (C19), 129.6 (C4), 128.6 (2C, C18), 128.3 (2C, C17), 128.2 (C1), 128.2 (C15), 128.1 (2C, C14), 127.8 (C5), 127.4 (2C, C13), 120.5 (C3), 111.7 (C6), 109.7 (C10), 55.5 (C20), 21.7 (C21), 13.6 (C22). **IR**: ν_{max} (thin film): 2929, 2236, 1643, 1498, 1271, 1237, 1025, 909, 726, 700 cm^{-1}. **HRMS** (ES+): found 382.1809; $C_{26}H_{24}NO_2$, $[M+H]^+$ requires 382.1807.

1-(2-Methoxyphenyl)-4-phenyl-6-(*p*-tolyl)pyridin-2(1*H*)-one (S7)



Prepared according to **General Procedure B** using alkynone **2b** (600 mg, 2.68 mmol, 1.2 equiv.), amide **1a** (368 mg, 2.23 mmol, 1.0 equiv.), KOH (125 mg, 2.23 mmol, 1.0 equiv.) and 1,4-dioxane (9 mL). Crude mixture purified by flash column chromatography (silica gel, 2.5% → 5% MeOH/dichloromethane) affording **S7** as an off-white foam (711 mg, 86 %). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.68-7.65 (2H, m, H17), 7.49-7.41 (3H, m, H18, 19), 7.22 (1H, td, *J* = 8.2, 1.6 Hz, H6), 7.09-7.06 (3H, m, H4, 13), 6.96 (2H, d, *J* = 8.1 Hz, H14), 6.90-6.86 (2H, m, H5, 10), 6.81 (1H, dd, *J* = 8.2, 1.1 Hz, H3), 6.49 (1H, d, *J* = 2.1 Hz, H8), 3.72 (3H, s, H20), 2.26 (3H, s, H21). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 163.4 (C7), 154.7 (C9), 151.3 (C2), 150.4 (C11), 138.4 (C16), 137.9 (C15), 133.0 (C12), 130.2 (C19), 129.8 (C4), 129.4 (C5), 128.9 (2C, C13), 128.5 (2C, C14), 128.3 (2C, C17), 127.8 (C1), 126.9 (2C, C18), 120.6 (C3), 115.9 (C6), 111.9 (C8), 109.0 (C10), 55.6 (C20), 21.2 (C21). **IR**: ν_{max} (thin film): 2922, 2236, 1654, 1572, 1498, 1364, 1267, 1025, 905, 723 cm^{-1}. **HRMS** (ES+): found 368.1649; $C_{25}H_{23}NO_2$, $[M+H]^+$ requires 368.1651.

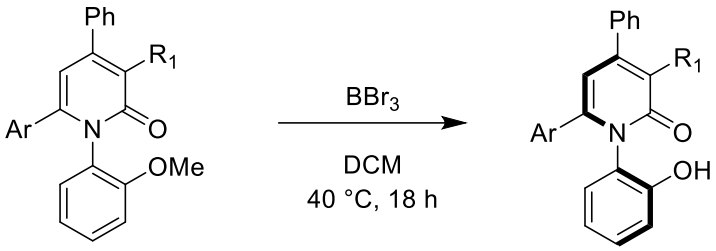
6-(4-Fluorophenyl)-1-(2-methoxyphenyl)-4-phenylpyridin-2(1*H*)-one (S8)



Prepared according to **General Procedure B** using alkynone **2c** (450 mg, 2.01 mmol, 1.2 equiv.), amide **1a** (276 mg, 1.67 mmol, 1.0 equiv.), KOH (94 mg, 1.67 mmol, 1.0 equiv.) and 1,4-dioxane (7 mL). Crude mixture purified by flash column chromatography (silica gel, 2.5% → 5% MeOH/dichloromethane) affording **S8** as a cream foam (530 mg, 85%). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.68-7.65 (2H, m, H17), 7.50-7.42 (3H, m, H18, 19), 7.26-7.15 (3H, m, H4, 13), 7.09 (1H, dd, *J* = 7.8, 1.7 Hz, H6), 6.92-6.80 (5H, m, H3, 5, 10, 14), 6.48 (1H, d, *J* = 1.9 Hz, H8), 3.72 (3H, s, H20). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 163.2 (C7), 162.5 (d, $^{1}J_{CF}$ = 249.0 Hz, C15), 154.4 (C2), 151.3 (C9), 149.1 (C11), 137.6 (C16), 131.8 (d, $^{4}J_{CF}$ = 3.6 Hz, C12), 130.5 (d, $^{3}J_{CF}$ = 8.5 Hz, C13), 130.1 (C19), 130.1 (C4), 129.5 (C5), 129.0 (2C, C17), 127.4 (C1), 126.4 (2C, C18), 120.7 (C3), 116.4 (C6), 114.7 (d, $^{2}J_{CF}$ = 21.7, C14), 111.9 (C8), 107.1 (C10), 55.5 (C20). **IR**: ν_{max} (thin film): 2922, 2236, 1654, 1606, 1498, 1364, 1222, 1159, 1025, 723, 697 cm^{-1}. **HRMS** (ES+): found 743.2714; $C_{24}H_{19}FNO_2$, $[2M+H]^+$ requires 743.2710.

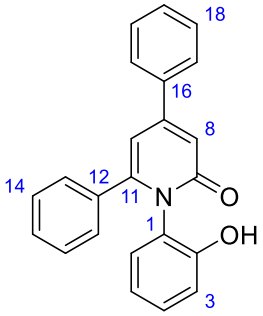
2.5 2′-Hydroxyphenyl-2-pyridones (3)

General Procedure C (*Preparation of 2′-hydroxyphenyl-2-pyridones*)



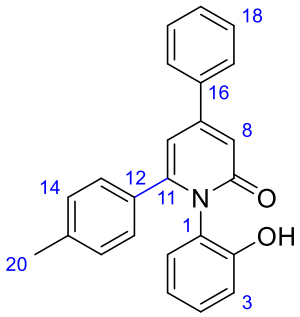
An oven-dried microwave tube was charged with pyridone derivatives (1.0 equiv.) and flushed with Ar. To this was added dichloromethane (*ca*. M) followed by BBr_3 (5.3 equiv.) and the tube was sealed and stirred at 40 °C for 18 h, then allowed to cool to RT. In some cases, as the tube cooled down the desired 2′-hydroxyphenyl-2-pyridone crashed out of solution. The solid was collected by filtration and washed with cold dichloromethane affording the desired 2′-hydroxyphenyl-2-pyridone derivative. Each mother liquor was reduced *in vacuo* and a further crop of desired enamine was retained either by recrystallization or flash column chromatography. If no crystallisation occurred purification was carried out by flash column chromatography.

1-(2-Hydroxyphenyl)-4,6-diphenylpyridin-2(1*H*)-one (3a)



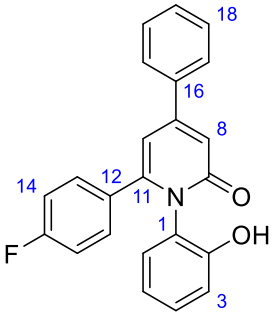
Prepared according to **General Procedure C** using pyridone **S4** (1.00 g, 2.83 mmol, 1.0 equiv.), BBr_3 (15.0 mL, 15.0 mmol, 5.3 equiv., 1M in dichloromethane), and dichloromethane (32 mL). **3a** obtained via trituration with dichloromethane as a beige solid (876 mg, 92%). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 8.16 (1H, s, OH), 7.72-7.66 (2H, m, H17), 7.54-7.45 (3H, m, H18, 19), 7.26-7.16 (5H, m, H13, 14, 15), 7.05-6.99 (2H, m, H4, 10), 6.94 (1H, dd, *J* = 8.2, 1.1 Hz, H6), 6.73 (1H, d, *J* = 2.2 Hz, H8), 6.64 (1H, dd, *J* = 7.9, 1.8 Hz, H3), 6.57 (1H, td, *J* = 7.3, 1.5 Hz, H5). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 164.8 (C7), 153.4 (C9), 152.6 (C2), 150.6 (C11), 137.0 (C16), 135.9 (C3), 130.0 (C4), 129.9 (C19), 129.2 (3C, C1, 17), 128.8 (C15), 128.3 (2C, C14), 128.2 (2C, C18), 126.9 (2C, C13), 120.5 (C5), 119.7 (C6), 115.3 (C8), 110.1 (C10). **IR**: ν_{max} (thin film): 3056, 1654, 1531, 1587, 1498, 1271, 1025, 760, 700 cm^{-1}. **HRMS** (ES+): found 701.2413; $C_{23}H_{17}NaNO_2$, $[2M+Na]^+$ requires 701.2416.

1-(2-Hydroxyphenyl)-4-phenyl-6-(*p*-tolyl)pyridin-2(1*H*)-one (3b)



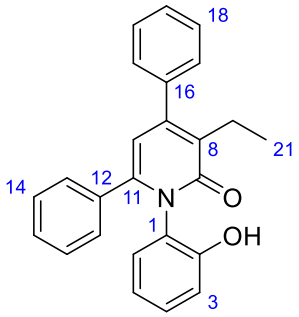
Prepared according to **General Procedure C** using pyridone **S7** (500 mg, 1.36 mmol, 1.0 equiv.), BBr_3 (7.21 mL, 7.21 mmol, 5.3 equiv., 1M in dichloromethane), and dichloromethane (16 mL). Crude mixture purified by flash column chromatography (silica gel, 1% → 5% MeOH/dichloromethane) affording **3b** as a white foam (374 mg, 78%). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 8.03 (1H, s, OH), 7.69-7.66 (2H, m, H17), 7.52-7.45 (3H, m, H18, 19), 7.11-7.05 (3H, m, H4, 13), 7.0-6.97 (4H, m, H6, 10, 14), 6.71 (1H, d, *J* = 2.0 Hz, H8), 6.64 (1H, dd, *J* = 7.9, 1.8 Hz, H3), 6.60 (1H, td, *J* = 7.2, 1.6 Hz, H5), 2.26 (3H, s, H20). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 164.8 (C7), 153.5 (C2), 152.6 (C9), 150.8 (C11), 138.9 (C15), 137.2 (C16), 133.0 (C12), 129.9 (C3), 129.7 (C4), 129.1 (2C, C17), 129.0 (C19), 128.9 (2C, C18), 128.8 (C1), 128.2 (2C, C13), 126.9 (2C, C14), 120.3 (C5), 119.3 (C6), 115.0 (C10), 109.8 (C8), 21.3 (C20). **IR**: ν_{max} (thin film): 3058, 2713, 1640, 1602, 1505, 1457, 1362, 1278, 915, 723, 697 cm^{-1}. **HRMS** (ES+): found 354.1457; $C_{24}H_{20}NO_2$, $[M+H]^+$ requires 354.1494.

6-(4-Fluorophenyl)-1-(2-hydroxyphenyl)-4-phenylpyridin-2(1*H*)-one (3c)



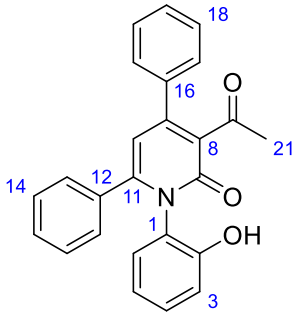
Prepared according to **General Procedure C** using pyridone **S8** (500 mg, 1.35 mmol, 1.0 equiv.), BBr_3 (7.13 mL, 7.13 mmol, 5.3 equiv., 1M in dichloromethane), and dichloromethane (16 mL). Crude mixture purified by flash column chromatography (silica gel, 1% → 5% MeOH/dichloromethane) affording **3c** as an off-white solid (473 mg, 98%). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 8.28 (1H, s, OH), 7.69-7.65 (2H, m, H17), 7.53-7.46 (3H, m, H18, 19), 7.25-7.21 (2H, m, H13), 7.04-6.99 (2H, m, H4, 10), 6.90-6.85 (3H, m, H6, 14), 6.69 (1H, dd, *J* = 2.0 Hz, H8), 6.65-6.59 (2H, m, H3, 5). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 164.6 (C7), 162.7 (d, $^1J_{CF}$ = 249.9 Hz, C15), 153.5 (C2), 152.6 (C9), 149.7 (C11), 137.1 (C16), 131.8 (d, $^4J_{CF}$ = 3.5 Hz, C12), 130.3 (d, $^3J_{CF}$ = 8.6 Hz, C13), 129.9 (C3), 129.9 (C4), 129.1 (2C, C17), 128.8 (C19), 127.8 (C1), 126.9 (2C, C18), 120.2 (C5), 118.9 (C6), 115.6 (C8), 115.3 (d, $^2J_{CF}$ = 21.6 Hz, C14), 109.6 (C10). **IR**: ν_{max} (thin film): 3060, 1643, 1602, 1558, 1502, 1367, 1230, 909, 831 cm^{-1}. **HRMS** (ES+): found 715.2405; $C_{23}H_{17}FNO_2$, $[2M+H]^+$ requires 715.2408.

3-Ethyl-1-(2-hydroxyphenyl)-4,6-diphenylpyridin-2(1*H*)-one (3d)



Prepared according to **General Procedure C** using pyridone **S6** (330 mg, 865 µmol, 1.0 equiv.), BBr_3 (4.58 mL, 4.58 mmol, 5.3 equiv., 1M in dichloromethane), and dichloromethane (10 mL). Crude mixture purified by flash column chromatography (silica gel, 1% → 5% MeOH/dichloromethane) affording **3d** as an orange foam (301 mg, 95%). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.85 (1H, s, OH), 7.50-7.38 (5H, m, H13, 14, 15), 7.21-7.06 (7H, m, H4, 6, 17, 18, 19), 6.63 (1H, dd, *J* = 8.0, 1.6 Hz, H3), 6.59-6.55 (1H, m, H5), 6.42 (1H, s, H8), 2.75-7.54 (2H, m, H20), 1.20 (3H, t, *J* = 7.5 Hz, H21). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 164.7 (C7), 153.6 (C2), 150.7 (C9), 146.5 (C11), 139.1 (C12), 135.9 (C16), 130.6 (C1), 129.9 (C8), 129.6 (C3), 129.4 (C4), 128.5 (2C, C17), 128.4 (C19), 128.3 (C15), 128.2 (4C, C14, 18), 128.0 (2C, C13), 128.0 (2C, C13), 120.5 (C5), 119.8 (C6), 113.1 (C10), 21.6 (C20), 13.6 (C21). **IR**: ν_{max} (thin film): 2963, 1632, 1599, 1565, 1457, 1259, 1025, 905, 726, 697 cm^{-1}. **HRMS** (ES+): found 368.1645; $C_{25}H_{22}NO_2$, $[M+H]^+$ requires 368.1651.

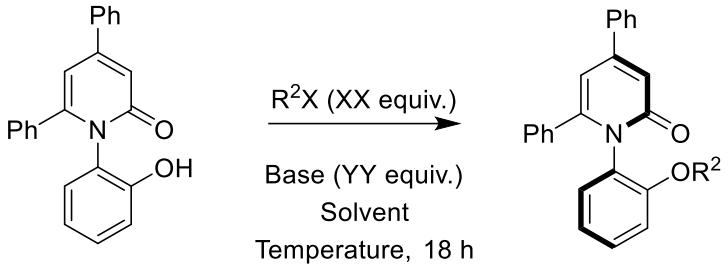
3-Acetyl-1-(2-hydroxyphenyl)-4,6-diphenylpyridin-2(1*H*)-one (3e)



Prepared according to **General Procedure C** using pyridone **S5** (300 mg, 759 μmol, 1.0 equiv.), BBr_3 (4.02 mL, 4.02 mmol, 5.3 equiv., 1M in dichloromethane), and dichloromethane (10 mL). Crude mixture purified by flash column chromatography (silica gel, 1% → 5% MeOH/dichloromethane) affording **3e** as a yellow foam (236 mg, 82%). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.52-7.43 (5H, m, H13, 14, 15), 7.40 (1H, s, OH), 7.25-7.17 (5H, m, H17, 18, 19), (5H, m, H13, 14, 15), 7.117.07 (1H, m, H4), 7.00 (1H, dd, *J* = 8.2, 1.3 Hz, H6), 6.65 (1H, dd, *J* = 8.0, 1.8 Hz, H8), 6.61 (1H, td, *J* = 7.1, 1.4 Hz, H3), 6.53 (1H, s, H10), 2.44 (3H, s, H21). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 201.9 (C20), 161.8 (C7), 153.4 (C2), 151.1 (C9), 150.8 (C11), 137.1 (C16), 135.1 (C12), 130.1 (C3), 129.5 (C19), 129.2 (C15), 129.0 (C4), 129.0 (C8), 128.9 (2C, 17), 128.3 (2C, C18), 128.3 (C1), 128.2 (2C, C14), 128.0 (2C, C13), 120.7 (C5), 119.5 (C6), 112.2 (C10), 31.8 (C21). **IR**: ν_{max} (thin film): 3183, 1699, 1628, 1565, 1528, 1367, 1103, 909, 756 cm^{-1}. **HRMS** (ES+): found 763.2812; $C_{25}H_{20}NO_3$, $[2M+H]^+$ requires 763.2808.

2.6 *O*-Benzylated *N*-(2-phenoxy)pyridones (4)

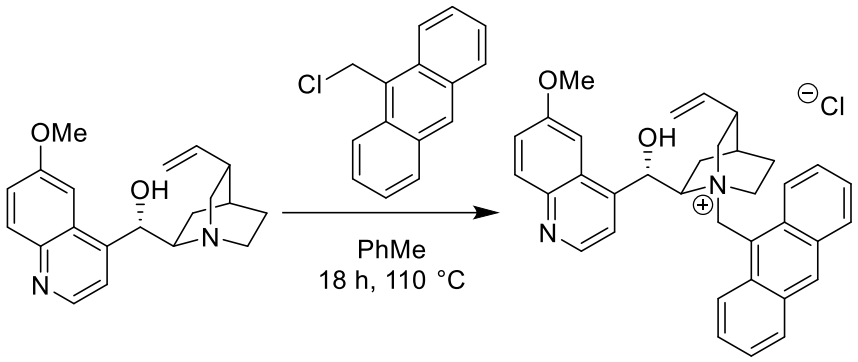
2.6.1 Optimisation method using chiral catalyst



Sample vials were charged with *N*-(2-phenoxy)pyridone (14 mg, 41.0 μmol, 1.0 equiv.) and catalyst (10-15 mol%). Solvent (0.5-1.5 mL) was added followed by 50% aqueous base (5.0 equiv.) and R^2X (X = Br, I or OTs, 0.6-5.0 equiv.) as the vial was sealed and stirred at the desired temperature for the time stated. At various intervals, the resulting solution was subjected to preparative TLC and enantiomeric excess determined by HPLC (Chiralpak, 60% MeCN/H_2O).

2.6.2 Catalyst synthesis

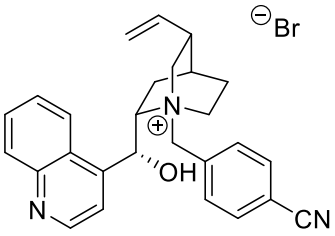
Various catalysts were synthesised and used during optimisation. *O*-Alkylation was performed as described by Corey *et al.*[9] and hydrogenation was performed as described by Tang *et al.*[10]



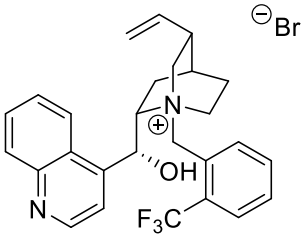
A flask was charged with quinidine (200 mg, 616 µmol, 1.0 equiv.), 9-chloromethyl anthracene (139 mg, 616 µmol, 1.0 equiv.) and toluene (10 mL). The reaction mixture was stirred at 110 °C for 18 h as a cream precipitate formed from a yellow solution. The crude product was removed from the heat, allowed to cool and then Et_2O (3 x 25 mL) was added and resulting mixture stirred for 10 mins. Product purified by filtration, washing with cold Et_2O (3 x 25 mL), affording QD-1 as a yellow solid (178 mg, 52%). The data obtained are consistent with those reported in the literature.[11]

^{1}H NMR (400 MHz, $CDCl_3$): δ_H = 9.07 (1H, d, *J* = 9.0 Hz), 8.50 (1H, d, *J* = 8.9 Hz), 8.30-8.24 (2H, m), 8.12-8.09 (1H, m), 8.00 (1H, s, OH), 7.95-7.91 (1H, m), 7.65 (1H, d, *J* = 8.8 Hz), 7.57 (1H, d, *J* = 8.3 Hz), 7.38-7.26 (3H, m), 7.20-7.12 (2H, m), 7.07-7.05 (1H, m), 6.95 (1H, d, *J* = 13.2 Hz), 6.56 (1H, d, *J* = 13.3 Hz), 5.74-5.65 (1H, m), 5.09 (1H, d, *J* = 10.5 Hz), 4.98-4.94 (1H, m), 4.75-4.70 (1H, m), 4.57-4.51 (1H, m), 4.14 (1H, t, *J* = 11.4 Hz), 3.84 (3H, s, OMe), 2.81-2.70 (1H, m), 2.35-2.21 (2H, m), 1.87-1.72 (3H, m), 1.63 (1H, s), 1.44-1.38 (1H, m), 1.00-0.88 (1H, m). **^{13}C NMR** (101 MHz, $CDCl_3$): δ_C = 157.7, 147.2, 144.5, 143.2, 135.8, 133.1, 132.9, 131.5, 131.0, 130.5, 130.3, 128.7, 128.5, 127.8, 127.6, 127.3, 126.1, 125.0, 124.9, 124.8, 121.4, 120.5, 118.3, 117.4, 104.1, 68.3, 65.5, 56.6, 56.2, 54.5, 54.3, 38.1, 26.3, 24.2, 22.6. **IR**: ν_{max} (thin film): 3652, 3052, 2199, 1621, 1505, 1449, 1241, 1028, 905, 723 cm^{-1}. **HRMS** (ES+): found 515.2687; $C_{35}H_{35}N_2O_2$, $[M\text{-}Cl^-]^+$ requires 515.2699. $[\boldsymbol{\alpha}]_{\boldsymbol{D}}^{\boldsymbol{293}}$ +380.2 (*c* = 0.3, $CHCl_3$).

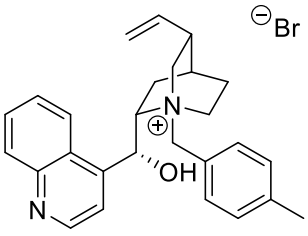
2.6.3 Chiral catalyst library



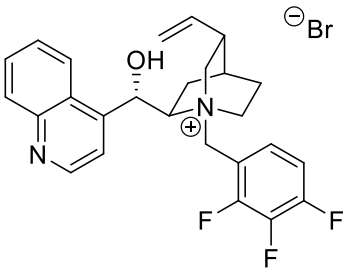
CD-1



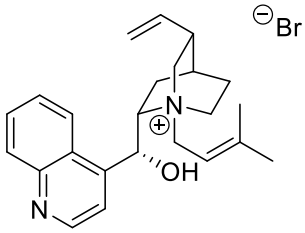
CD-2



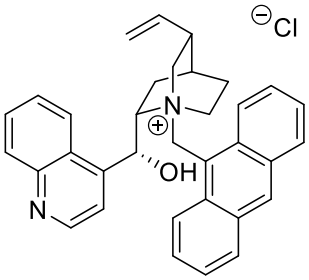
CD-3



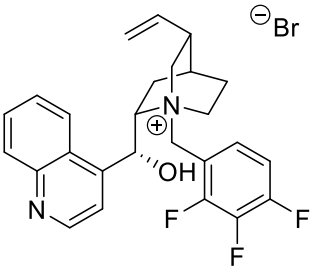
CN-1



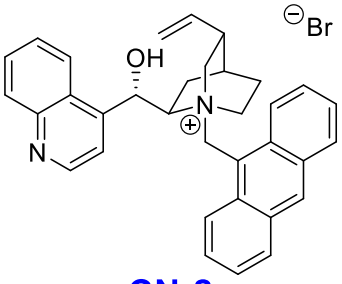
CD-4



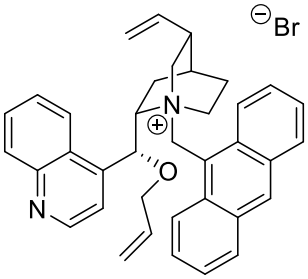
CD-5
(C1)



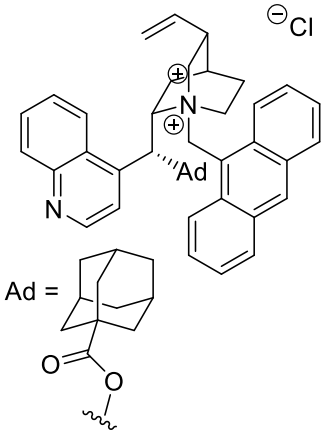
CD-6



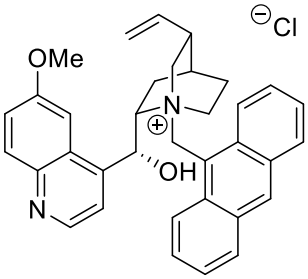
CN-2



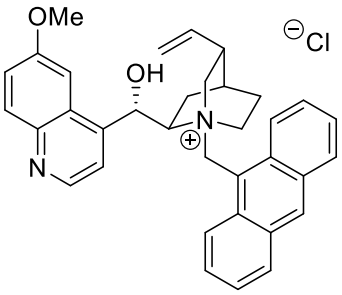
CD-7
(C2)



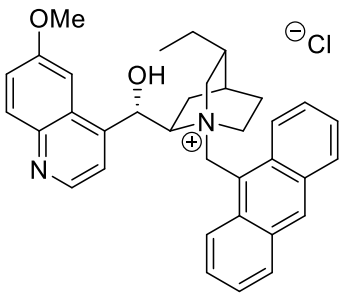
CD-8



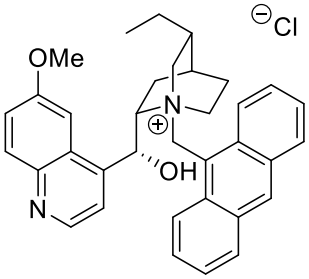
QN-1
(C3)



QD-1
(C4)



QD-2



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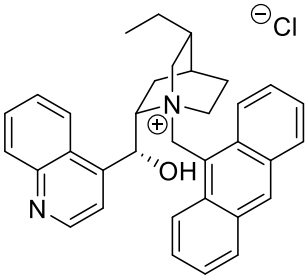
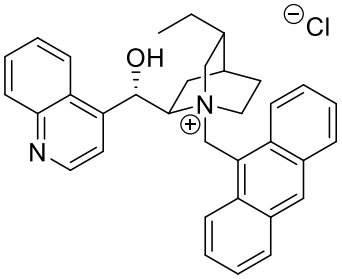
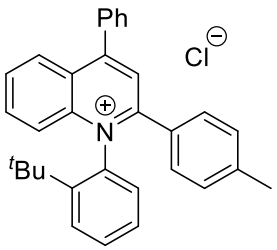


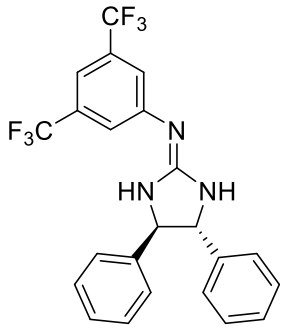
CD-9



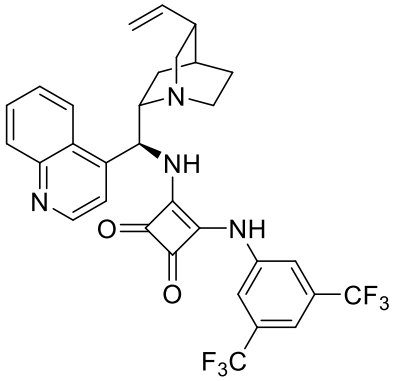
CN-3



Quinolinium-1



Cat-1



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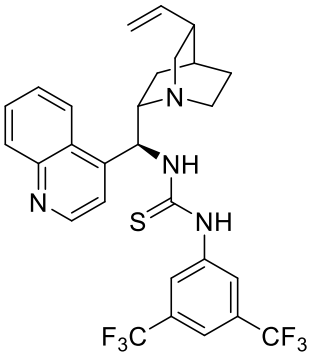
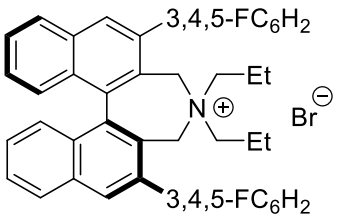
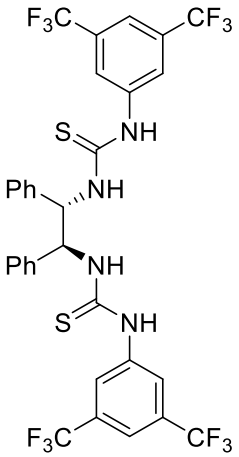


Cat-3



Maruoka-1



Cat-4

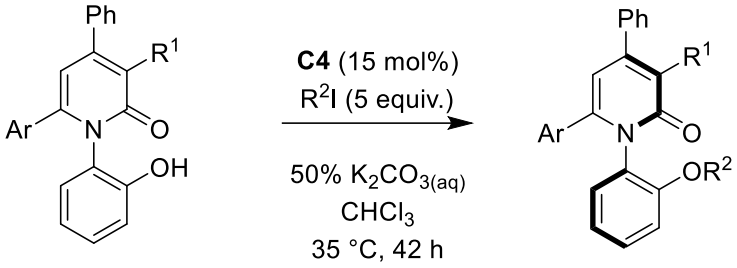
2.6.4 Optimisation results

Table S1. Optimisation of dynamic kinetic resolution of pyridones. For reaction conditions see section 2.6.1 above. In each case, base was always 50% aqueous solution. Enantiomeric excesses were determined after 18 h by HPLC on a chiral stationary phase.

Entry	Solvent	Base	Bn-X (eq.)	TLC Conversion (%)	Catalyst	Temp/ °C	*ee*
1	PhH	K_3PO_4	BnBr (1.2 eq.)	<25	CD-1	rt	**16%**
2	PhH	K_3PO_4	BnBr (1.2 eq.)	<25	CD-2	rt	**11%**
3	PhH	K_3PO_4	BnBr (1.2 eq.)	<25	CD-3	rt	**16%**
4	PhH	K_3PO_4	BnBr (1.2 eq.)	<25	CD-4	rt	**1%**
5	PhH	K_3PO_4	BnBr (1.2 eq.)	<25	CD-5	rt	**-12%**
6	PhH	K_3PO_4	BnBr (1.2 eq.)	<25	CD-6	rt	**9%**
7	PhH	K_3PO_4	BnBr (0.6 eq.)	<25	CN-1	rt	**-3%**
8	PhH	K_3PO_4	BnBr (0.6 eq.)	<25	Maruoka-1	rt	**24%**
9	PhH	K_3PO_4	BnBr (5.0 eq.)	<25	Maruoka-1	rt	**23%**
10	$CHCl_3$	K_3PO_4	BnBr (0.6 eq.)	<25	Cat-1	rt	**0%**
11	$CHCl_3$	K_3PO_4	BnBr (0.6 eq.)	<25	Cat-2	rt	**6%**
12	$CHCl_3$	K_3PO_4	BnBr (0.6 eq.)	<25	Cat-3	rt	**18%**
13	$CHCl_3$	K_3PO_4	BnBr (0.6 eq.)	<25	Cat-4	rt	**28%**
14	$CHCl_3$	K_3PO_4	BnBr (0.6 eq.)	<25	Quinolinium-1	rt	**-7%**
15	$CHCl_3$	K_3PO_4	BnBr (0.6 eq.)	<25	Maruoka-1	rt	**29%**
17	$CHCl_3$	K_3PO_4	BnBr (0.6 eq.)	25-50	CD-8	rt	**-2%**
18	$CHCl_3$	K_3PO_4	BnBr (0.6 eq.)	<25	CD-5	rt	**-58%**
19	$CHCl_3$	KOH	BnBr (0.6 eq.)	<25	CD-5	rt	**-8%**
20	$CHCl_3$	Cs_2CO_3	BnBr (0.6 eq.)	<25	CD-5	rt	**-66%**
21	$CHCl_3$	K_2CO_3	BnBr (0.6 eq.)	<25	CD-5	rt	**-66%**
22	DCE	K_2CO_3	BnBr (0.6 eq.)	<25	CD-5	rt	**-40%**
23	Et_2O	K_2CO_3	BnBr (0.6 eq.)	<25	CD-5	rt	**-2%**
24	*m*-xylene	K_2CO_3	BnBr (0.6 eq.)	<25	CD-5	rt	**-30%**
25	THF	K_2CO_3	BnBr (0.6 eq.)	<25	CD-5	rt	**-8%**
26	PhMe	K_2CO_3	BnBr (0.6 eq.)	<25	CD-5	rt	**-22%**
27	$CHCl_3$	K_2CO_3	BnBr (0.6 eq.)	<25	CN-2	rt	**68%**
28	$CHCl_3$	K_2CO_3	BnOTs (0.6 eq.)	25-50	CD-5	rt	**-51%**
29	$CHCl_3$	K_2CO_3	BnI (0.6 eq.)	<25	CD-5	rt	**-73%**
30	$CHCl_3$	K_2CO_3	BnI (0.6 eq.)	<25	CD-7	rt	**-33%**
31	$CHCl_3$	K_2CO_3	BnI (0.6 eq.)	50-75	CD-5	50	**-45%**
32	$CHCl_3$	K_2CO_3	BnI (0.6 eq.)	<25	CD-5	0	**-54%**
33	$CHCl_3$	K_2CO_3	BnI (5.0 eq.)	<25	QN-1	rt	**-76%**
34	$CHCl_3$	K_2CO_3	BnI (5.0 eq.)	<25	QD-1	rt	**85%**
35	$CHCl_3$	K_2CO_3	BnI (5.0 eq.)	<25	QD-2	rt	**57%**
36	$CHCl_3$	K_2CO_3	BnI (5.0 eq.)	50-75	QD-1	35	**80%**

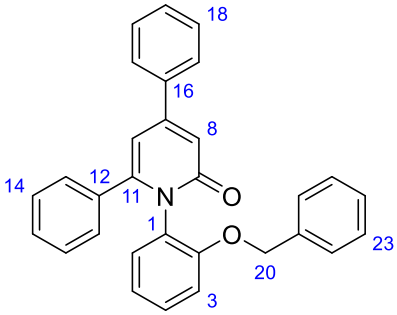
2.6.5 Enantioselective Synthesis of 4a-4o

General Procedure D (Preparation of 2-arylpyridones)



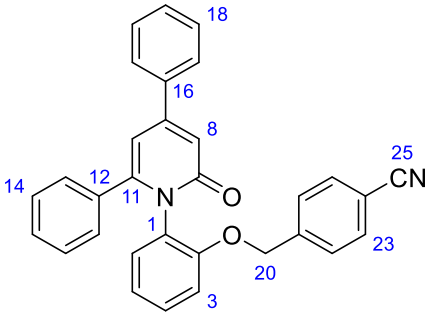
A round-bottomed flask was charged with 2′-hydroxyphenyl-2-pyridone (1.0 equiv.) and QD-1 (10 mol%). $CHCl_3$ (*ca*. 0.3 M) was added followed by 50% aqueous K_2CO_3 (5.0 equiv.) and R^2I (5.0 equiv.) as the flask was sealed and stirred at 35 °C for 42 h. The reaction mixture was subjected to preparative TLC and enantiomeric excess analysis determined by HPLC (CHIRALPAK, 60% MeCN/H_2O,) after 18 h, at which time an additional portion of QD-1 (5 mol%) was added. Piperidine (4.0 equiv.) was added and reaction mixture was stirred for 30 mins before addition of 3M HCl (*ca*. 1 mL). Crude product was extracted with EtOAc (3 x10 mL/mmol), dried over anhydrous magnesium sulfate and the solvent removed *in vacuo* resulting in a crude solid. Purification by column chromatography (silica gel, 1% → 2.5% MeOH/dichloromethane) afforded the desired product.

1-(2-(Benzyloxy)phenyl)-4,6-diphenylpyridin-2(1*H*)-one (4a)



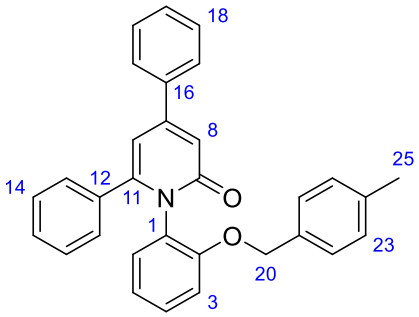
General Procedure D: Pyridone **3a** (100 mg, 295 µmol, 1.0 equiv.), QD-1 (24.4 mg, 44.3 µmol, 15 mol%). $CHCl_3$ (11 mL) was added followed by 50% aqueous K_2CO_3 (205 µL, 1.48 mmol, 5.0 equiv.) and R^2I-**S1** (184 µL, 1.48 mmol, 5.0 equiv.). Crude mixture purified by flash column chromatography (silica gel, 1% → 2.5% MeOH/dichloromethane) affording **4a** as a yellow foam (98 mg, 77%, 75% *ee*). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.69-7.66 (2H, m, H17), 7.50-7.44 (3H, m, H18, 19), 7.33-7.24 (5H, m, H13, 14, 15), 7.21-7.11 (7H, m, H4, 6, 22, 23, 24), 6.94 (1H, d, *J* = 2.0 Hz, H10), 6.9. (1H, td, *J* = 7.6, 1.2 Hz, H5), 6.84 (1H, dd, *J* = 8.4, 1.1 Hz, H3), 6.49 (1H, d, *J* = 2.1 Hz, H8), 5.09-4.93 (2H, m, H20). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 163.4 (C7), 153.8 (C9), 151.4 (C2), 150.3 (C11), 137.8 (C16), 136.8 (C21), 135.7 (C12), 130.4 (C19), 129.9 (C4), 129.4 (C15), 129.0 (2C, C17), 128.8 (2C, C23), 128.5 (C24), 128.5 (2C, C18), 128.1 (C1), 127.7 (C6), 127.6 (2C, C14), 126.8 (2C, C13), 126.7 (2C, C22), 120.9 (C5), 116.1 (C10), 113.4 (C3), 107.0 (C8), 70.2 (C20). **HPLC**: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 36.4 min, t_R (minor) = 15.5 min. **IR**: ν_{max} (thin film): 3060, 2236, 1654, 1572, 1531, 1490, 1364, 1267, 1230, 909, 730, 697 cm^{-1}. **HRMS** (ES+): found 430.1810; $C_{30}H_{24}NO_2$, $[M+H]^+$ requires 430.1807. $[\alpha]_D^{293}$ +33.3 (*c* = 0.6, $CHCl_3$).

4-((2-(2-Oxo-4,6-diphenylpyridin-1(2*H*)-yl)phenoxy)methyl)benzonitrile (4b)



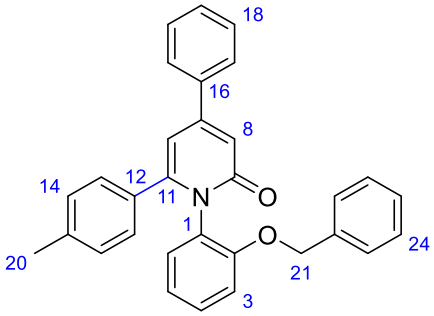
General Procedure D: Pyridone **3a** (50.0 mg, 141 µmol, 1.0 equiv.), QD-1 (11.7 mg, 21.2 µmol, 15 mol%). $CHCl_3$ (6 mL) was added followed by 50% aqueous K_2CO_3 (98.0 µL, 707 µmol, 5.0 equiv.) and R^2I-**S2** (89 mg, 707 µmol, 5.0 equiv.). Crude mixture purified by flash column chromatography (silica gel, 1% → 2.5% MeOH/dichloromethane) affording **4b** as a yellow solid (40 mg, 80%, 67% *ee*). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.69-7.66 (2H, m, H17), 7.60 (2H, d, *J* = 8.4 Hz, H23), 7.52-7.46 (3H, m, H18, 19), 7.42 (2H, d, *J* = 8.6 Hz, H22), 7.24-7.13 (7H, m, H4, 6, 13, 14, 15), 6.94-6.91 (2H, m, H5, 10), 6.78 (1H, dd, *J* = 8.3, 1.0 Hz, H3), 6.53 (1H, d, *J* = 2.0 Hz, H8), 5.16-5.05 (2H, m, H20). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 163.3 (C7), 153.3 (C9), 151.6 (C2), 150.2 (C11), 142.2 (C21), 137.6 (C16), 135.6 (C12), 132.4 (2C, C23), 130.6 (C4), 130.0 (C19), 129.7 (C15), 129.1 (2C, C17), 128.7 (C5), 128.6 (2C, C14), 128.3 (C1), 127.8 (2C, C17), 127.1 (2C, C13), 126.8 (2C, C22), 121.6 (C6), 118.7 (C25), 115.9 (C10), 113.3 (C3), 111.6 (C24), 107.2 (C8), 69.3 (C20). **HPLC**: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 33.4 min, t_R (minor) = 13.7 min. **IR**: ν_{max} (thin film): 3056, 2228, 1651, 1490, 1449, 1271, 909, 820, 726, 697 cm^{-1}. **HRMS** (ES+): found 455.1785; $C_{31}H_{23}N_2O_2$, $[M+H]^+$ requires 455.1760. $[\alpha]_D^{293}$ +43.6 (*c* = 0.5, $CHCl_3$).

1-(2-((4-Methylbenzyl)oxy)phenyl)-4,6-diphenylpyridin-2(1*H*)-one (4c)



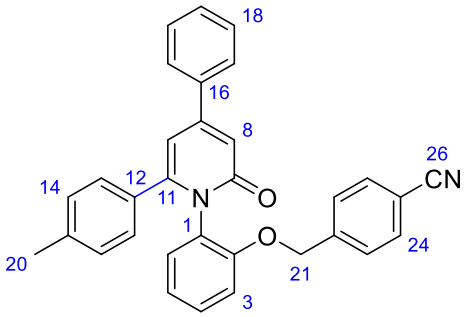
General Procedure D: 2-hydroxypyridone **3a** (60 mg, 177 µmol, 1.0 equiv.), QD-1 (14.6 mg, 26.6 µmol, 15 mol%). $CHCl_3$ (6 mL) was added followed by 50% aqueous K_2CO_3 (122 µL, 884 µmol, 5.0 equiv.) and R^2I-**S3** (205 mg, 884 µmol, 5.0 equiv.). Crude mixture purified by flash column chromatography (silica gel, 1% → 2.5% MeOH/dichloromethane) affording **4c** as an off-white solid (60 mg, 76%, 70% *ee*). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.69-7.65 (2H, m, H17), 7.50-7.45 (3H, m, H18, 19), 7.22-7.11 (11H, m, H4, 6, 13, 14, 15, 22, 23), 6.94 (1H, d, *J* = 2.0 Hz, H10), 6.89 (1H, td, *J* = 7.5, 1.1 Hz, H5), 6.84 (1H, dd, *J* = 8.4, 1.0 Hz, H3), 6.48 (1H, d, *J* = 2.0 Hz, H8), 5.05-4.88 (2H, m, H20), 2.33 (3H, s, H25). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 163.4 (C7), 153.9 (C9), 151.3 (C2), 150.3 (C11), 137.8 (C16), 137.4 (C24), 135.7 (C12), 133.8 (C21), 130.4 (C4), 129.8 (C19), 129.4 (C15), 129.1 (2C, C23), 129.0 (2C, C17), 128.8 (2C, C18), 128.5 (C5), 128.1 (C1), 127.6 (2C, C14), 126.9 (2C, C13), 126.9 (2C, C22), 120.8 (C6), 116.1 (C8), 113.5 (C3), 107.0 (C10), 70.2 (C20), 21.2 (C25). **HPLC**: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 53.5 min, t_R (minor) = 19.9 min. **IR**: ν_{max} (thin film): 3056, 2922, 2236, 1654, 1572, 1531, 1490, 1364, 1267, 998, 801, 697, 723 cm^{-1}. **HRMS** (ES+): found 444.1976; $C_{31}H_{26}NO_2$, $[M+H]^+$ requires 444.1964. $[\alpha]_D^{293}$ +27.4 (*c* = 0.4, $CHCl_3$).

1-(2-(Benzyloxy)phenyl)-4-phenyl-6-(*p*-tolyl)pyridin-2(1*H*)-one (4d)



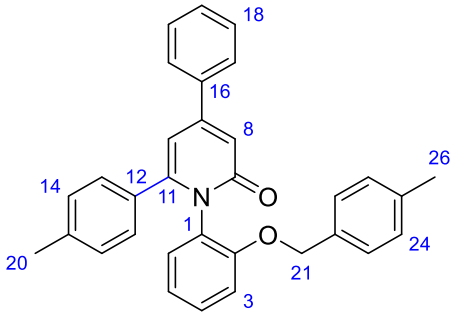
General Procedure D: Pyridone **3d** (100 mg, 262 µmol, 1.0 equiv.), QD-1 (21.6 mg, 39.3 µmol, 15 mol%). $CHCl_3$ (11 mL) was added followed by 50% aqueous K_2CO_3 (182 µL, 1.31 mmol, 5.0 equiv.) and R^2I-**S1** (163 µL, 1.31 mmol, 5.0 equiv.). Crude mixture purified by flash column chromatography (silica gel, 1% → 2.5% MeOH/dichloromethane) affording **4d** as a yellow foam (82 mg, 70%, 77% *ee*). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.68-7.65 (2H, m, H17), 7.50-7.44 (3H, m, H18, 19), 7.33-7.24 (5H, m, H23, 24, 25), 7.22-7.14 (2H, m, H4, 6), 7.06-7.04 (2H, m, H13), 6.95-6.89 (4H, m, H5, 10, 14), 6.85 (1H, dd, *J* = 8.4, 1.2 Hz, H3), 6.48 (1H, d, *J* = 2.0 Hz, H8), 5.10-4.94 (2H, m, H21), 2.26 (3H, s, H20). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 163.5 (C7), 153.8 (C9), 151.4 (C2), 150.4 (C11), 138.4 (C16), 137.9 (C12), 136.9 (C21), 132.9 (C15), 130.4 (C19), 129.8 (C4), 129.4 (C25), 129.0 (2C, C13), 128.7 (2C, C17), 128.4 (2C, C24), 128.3 (2C, C14), 128.3 (C1), 127.7, 126.9 (2C, C18), 126.7 (2C, C23), 120.9 (C5), 115.9 (C10), 113.5 (C3), 107.1 (C8), 70.2 (C20), 21.2 (C20). **HPLC**: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) =40.2 min, t_R (minor) = 18.2 min. **IR**: ν_{max} (thin film): 3030, 2236, 1654, 1572, 1498, 1364, 1267, 905, 723 cm^{-1}. **HRMS** (ES+): found 444.1956; $C_{31}H_{26}NO_2$, $[M+H]^+$ requires 444.1964. $[\alpha]_D^{293}$ +43.8 (*c* = 0.6, $CHCl_3$).

4-((2-(2-Oxo-4-phenyl-6-(*p*-tolyl)pyridin-1(2*H*)-yl)phenoxy)methyl)benzonitrile (4e)



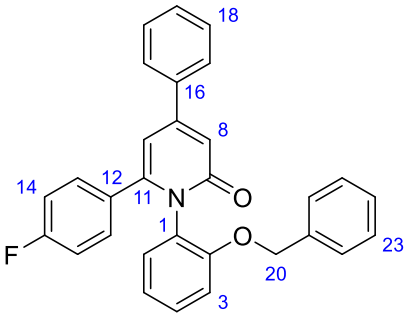
General Procedure D: Pyridone **3d** (100 mg, 283 µmol, 1.0 equiv.), QD-1 (23.4 mg, 42.5 µmol, 15 mol%). $CHCl_3$ (11 mL) was added followed by 50% aqueous K_2CO_3 (196 µL, 1.42 mmol, 5.0 equiv.) and R^2I-**S2** (344 mg, 1.42 mmol, 5.0 equiv.). Crude mixture purified by flash column chromatography (silica gel, 1% → 2.5% MeOH/dichloromethane) affording **4e** as a white foam (114 mg, 86%, 63% *ee*). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.68-7.65 (2H, m, H17), 7.60 (2H, d, *J* = 8.1 Hz, H24), 7.52-7.46 (3H, m, H18, 19), 7.42 (2H, d, *J* = 8.2 Hz, H23), 7.21 (1H, td, *J* = 8.3, 1.7 Hz, H4), 7.14 (1H, dd, *J* = 7.8, 1.7 Hz, H6), 7.04 (2H, d, *J* = 8.2 Hz, H13), 6.98-6.92 (4H, m, H5, 10, 14), 6.79 (1H, dd, *J* = 8.3, 1.0 Hz, H3), 6.51 (1H, d, *J* = 2.0 Hz, H8), 5.15-5.02 (2H, m, H21), 2.26 (3H, s, H20). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 163.4 (C7), 153.3 (C9), 151.6 (C2), 150.3 (C11), 142.2 (C22), 138.6 (C16), 137.7 (C15), 132.7 (C12), 132.3 (2C, C24), 130.6 (C3), 129.8 (C4), 129.6 (C19), 129.1 (2C, C13), 128.5 (2C, C17), 128.4 (3C, C1, 14), 127.1 (2C, C18), 126.8 (2C, C23), 121.6 (C5), 118.7 (C26), 115.7 (C8), 113.4 (C3), 111.6 (C25), 107.1 (C10), 69.4 (C21), 21.2 (C20). **^{19}F NMR** (376 MHz, $CDCl_3$): δ_F = -111.7. **HPLC**: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 39.6 min, t_R (minor) = 16.6 min. **IR**: ν_{max} (thin film): 3056, 2922, 2228, 1654, 1572, 1498, 1453, 1364, 1267, 998, 909, 816 723 cm^{-1}. **HRMS** (ES+): found 469.1940; $C_{32}H_{25}N_2O_2$, $[M+H]^+$ requires 469.1916. $[\alpha]_D^{293}$ +53.1 (*c* = 0.4, $CHCl_3$).

1-(2-((4-Methylbenzyl)oxy)phenyl)-4-phenyl-6-(*p*-tolyl)pyridin-2(1*H*)-one (4f)



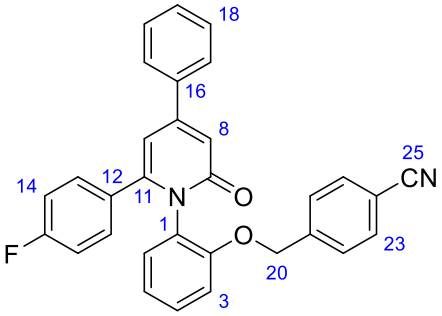
General Procedure D: Pyridone **3d** (100 mg, 283 µmol, 1.0 equiv.), QD-1 (23.4 mg, 42.5 µmol, 15 mol%). $CHCl_3$ (11 mL) was added followed by 50% aqueous K_2CO_3 (196 µL, 1.42 mmol, 5.0 equiv.) and R^2I-**S3** (328 mg, 1.42 mmol, 5.0 equiv.). Crude mixture purified by flash column chromatography (silica gel, 1% → 2.5% MeOH/dichloromethane) affording **4f** as a yellow foam (86 mg, 66%, 58% *ee*). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.68-7.65 (2H, m, H17), 7.50-7.44 (3H, m, H18, 19), 7.21-7.10 (6H, m, H4, 6, 13, 14), 7.07-7.04 (2H, m, H23), 6.95-6.85 (5H, m, H3, 5, 11, 24), 6.47 (1H, d, *J* = 2.0 Hz, H8), 5.05-4.89 (2H, m, H21), 2.33 (3H, s, H20), 2.26 (3H, s, H26). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 162.4 (C7), 152.9 (C9), 150.3 (C2), 149.4 (C11), 137.4 (C16), 136.9 (C15), 136.3 (C25), 132.8 (C22), 131.8 (C12), 129.3 (C4), 128.7 (C19), 128.3 (C5), 128.1 (2C, C24), 127.9 (2C, C13), 127.6 (2C, C17), 127.3 (2C, C14), 127.2 (C1), 125.8 (2C, C18), 125.8 (2C, C23), 119.8 (C6), 114.8 (C8), 112.5 (C3), 105.6 (C10), 69.2 (C21), 20.2 (C20), 20.1 (C26). **HPLC**: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 62.8 min, t_R (minor) = 23.8 min. **IR**: ν_{max} (thin film): 3026, 2922, 2236, 1654, 1498, 1453, 1364, 1263, 998, 801, 723, 697 cm^{-1}. **HRMS** (ES+): found 458.2122; $C_{32}H_{28}NO_2$, $[M+H]^+$ requires 458.2120. $[\alpha]_D^{293}$ +39.2 (*c* = 0.5, $CHCl_3$).

1-(2-(Benzyloxy)phenyl)-6-(4-fluorophenyl)-4-phenylpyridin-2(1*H*)-one (4g)



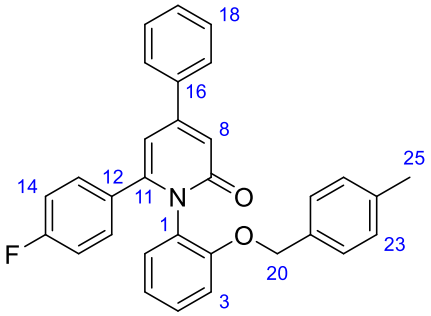
General Procedure D: Pyridone **3e** (50.0 mg, 141 µmol, 1.0 equiv.), QD-1 (11.7 mg, 21.2 µmol, 15 mol%). $CHCl_3$ (6 mL) was added followed by 50% aqueous K_2CO_3 (98.0 µL, 707 µmol, 5.0 equiv.) and R^2I-**S1** (89 µL, 707 µmol, 5.0 equiv.). Crude mixture purified by flash column chromatography (silica gel, 1% → 2.5% MeOH/dichloromethane) affording **4g** as a yellow solid (40 mg, 64%, 65% *ee*). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.68-7.65 (2H, m, H17), 7.51-7.45 (3H, m, H18, 19), 7.35-7.27 (5H, m, H23, 24, 25), 7.24-7.19 (1H, m, H4), 7.16-7.11 (3H, m, H6, 13), 6.95-6.90 (2H, m, H5, 10), 6.88-6.80 (3H, m, H3, H14), 6.46 (1H, d, *J* = 1.9 Hz, H8), 509-4.92 (2H, m, H21). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 163.5 (C7), 162.5 (d, $^1J_{CF}$ = 249.0 Hz, C15), 153.6 (C9), 151.4 (C2), 149.2 (C11), 137.7 (C16), 136.7 (C21), 131.8 (d, $^4J_{CF}$ = 3.6 Hz, C12), 130.7 (d, $^3J_{CF}$ = 8.4 Hz, C13), 130.4 (C19), 130.0 (C4), 129.5 (C24), 129.0 (2C, C17), 128.5 (2C, C23), 128.0 (C1), 127.8 (C6), 126.8 (2C, C18), 126.8 (2C, C23), 121.1 (C5), 116.3 (C10), 114.7 (d, $^2J_{CF}$ = 21.7 Hz, C14), 113.6 (C3), 107.1 (C8), 70.2 (C20). **^{19}F NMR** (376 MHz, $CDCl_3$): δ_F = -112.1. **HPLC**: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) =41.8 min, t_R (minor) = 16.8 min. **IR**: ν_{max} (thin film): 3060, 2922, 2236, 1654, 1605, 1498, 1271, 1222, 1159, 998, 730, 697 cm^{-1}. **HRMS** (ES+): found 448.1711; $C_{30}H_{23}FNO_2$, $[M+H]^+$ requires 448.1713. $[\alpha]_D^{293}$ +29.6 (*c* = 0.5, $CHCl_3$).

4-((2-(6-(4-Fluorophenyl)-2-oxo-4-phenylpyridin-1(2*H*)-yl)phenoxy)methyl)benzonitrile (4h)



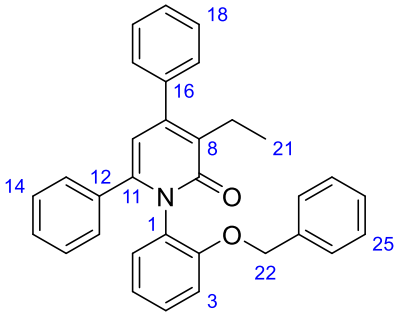
General Procedure D: Pyridone **3e** (100 mg, 280 µmol, 1.0 equiv.), QD-1 (23.3 mg, 42.0 µmol, 15 mol%). $CHCl_3$ (11 mL) was added followed by 50% aqueous K_2CO_3 (193 µL, 1.40 mmol, 5.0 equiv.) and R^2I-**S2** (340 mg, 1.40 mmol, 5.0 equiv.). Crude mixture purified by flash column chromatography (silica gel, 1% → 2.5% MeOH/dichloromethane) affording **4h** as a yellow solid (114 mg, 86%, 57% *ee*). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.68-7.65 (2H, m, H17), 7.62-7.59 (2H, m, H23), 7.52-7.46 (3H, m, H18, 19), 7.43 (2H, d, *J* = 8.6 Hz, H22), 7.22 (1H, td, *J* = 8.0, 1.8 Hz, H4), 7.16-7.11 (3H, m, H6, 13), 6.97-6.93 (2H, m, H5, 10), 6.87-6.80 (3H, m, H3,14), 6.50 (1H, d, *J* = 2.0 Hz, H8), 5.14-5.02 (2H, m, H20). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 163.9 (C7), 162.6 (d, $^1J_{CF}$ = 249.0 Hz, C15), 153.2 (C9), 151.6 (C2), 149.1 (C11), 142.0 (C21), 137.4 (C16), 132.3 (2C, C23), 131.6 (d, $^4J_{CF}$ = 3.5 Hz, C12), 130.6 (C6), 130.6 (d, $^3J_{CF}$ = 8.1 Hz, C13), 130.1 (C4), 129.7 (C19), 129.1 (2C, C17), 128.2 (C1), 127.1 (2C, C18), 126.8 (2C, C22), 121.8 (C5), 118.7 (C25), 116.2 (C8), 114.9 (d, $^2J_{CF}$ = 21.7 Hz, C14), 113.5 (C3), 111.7 (C25), 107.2 (C10), 69.4 (C20) . **^{19}F NMR** (376 MHz, $CDCl_3$): δ_F = -111.7. **HPLC**: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 38.7 min, t_R (minor) = 14.5 min. **IR**: ν_{max} (thin film): 3060, 2922, 2228, 1654, 1606, 1498, 1364, 1222, 909, 820, 726, 697 cm^{-1}. **HRMS** (ES+): found 473.1670; $C_{31}H_{22}FN_2O_2$, $[M+H]^+$ requires 473.1665. $[\alpha]_D^{293}$ +42.9 (*c* = 0.6, $CHCl_3$).

6-(4-Fluorophenyl)-1-(2-((4-methylbenzyl)oxy)phenyl)-4-phenylpyridin-2(1*H*)-one (4i)



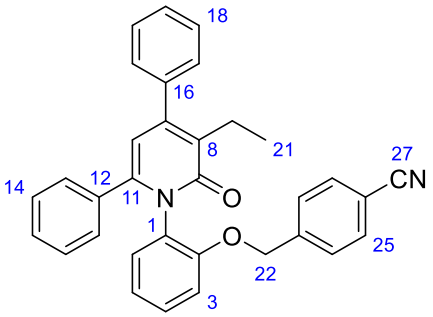
General Procedure D: Pyridone **3e** (100 mg, 280 µmol, 1.0 equiv.), QD-1 (23.3 mg, 42.0 µmol, 15 mol%). $CHCl_3$ (11 mL) was added followed by 50% aqueous K_2CO_3 (193 µL, 1.40 mmol, 5.0 equiv.) and R^2I-**S3** (324 mg, 1.40 mmol, 5.0 equiv.). Crude mixture purified by flash column chromatography (silica gel, 1% → 2.5% MeOH/dichloromethane) affording **4i** as an off-white foam (81 mg, 63%, 51% *ee*). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.68-7.65 (2H, m, H17), 7.51-7.44 (3H, m, H18, 19), 7.23-7.19 (3H, m, H4, 22), 7.15-7.11 (5H, m, H6, 13, 23), 6.94-6.80 (5H, m, H3, 5, 10, 14), 6.45 (1H, d, *J* = 2.0 Hz, H8), 5.05-4.88 (2H, m, H20), 2.33 (3H, s, H25). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 163.8 (C7), 162.5 (d, $^1J_{CF}$ = 248.7 Hz, C15), 153.8 (C9), 151.3 (C2), 149.2 (C11), 137.7 (C16), 137.6 (C24), 133.7 (C22), 131.8 (d, $^4J_{CF}$ = 3.6 Hz, C12), 130.7 (d, $^3J_{CF}$ = 8.5 Hz, C13), 130.3 (C4), 130.0 (C19), 129.5 (C5), 129.2 (2C, C23), 129.0 (2C, C17), 128.0 (C1), 126.9 (2C, C18), 126.8 (2C, C22), 121.0 (C6), 116.3 (C8), 114.7 (d, $^2J_{CF}$ = 21.6 Hz, C14), 112.5 (C3), 107.1 (C10), 70.3 (C20), 21.2 (C25). **^{19}F NMR** (376 MHz, $CDCl_3$): δ_F = -112.2. **HPLC**: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 61.4 min, t_R (minor) = 21.6 min. **IR**: ν_{max} (thin film): 3056, 2922, 2236, 1654, 1608, 1498 1364, 1222, 909, 726, 697 cm^{-1}. **HRMS** (ES+): found 462.1872; $C_{31}H_{25}FNO_2$, $[M+H]^+$ requires 462.1869. $[\alpha]_D^{293}$ +24.7 (*c* = 0.6, $CHCl_3$).

1-(2-(Benzyloxy)phenyl)-3-ethyl-4,6-diphenylpyridin-2(1*H*)-one (4j)



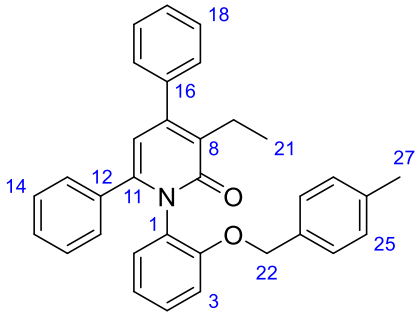
General Procedure D: Pyridone **3c** (100 mg, 272 µmol, 1.0 equiv.), QD-1 (22.5 mg, 40.8 µmol, 15 mol%). $CHCl_3$ (11 mL) was added followed by 50% aqueous K_2CO_3 (188 µL, 1.36 mmol, 5.0 equiv.) and R^2I-**S1** (169 µL, 1.36 mmol, 5.0 equiv.). Crude mixture purified by flash column chromatography (silica gel, 1% → 2.5% MeOH/dichloromethane) affording **4j** as a yellow foam (66 mg, 53%, 40% *ee*). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.48-7.37 (5H, m, H17, 18, 19), 7.36-7.27 (5H, m, H13, 14, 15), 7.21-7.09 (7H, m, H4, 6, 24, 25, 26), 6.92-6.85 (2H, m, H3, 5), 6.14 (1H, s, H10), 5.11-4.92 (2H, m, H22), 2.64-2.55 (2H, m, H20), 2.45 (3H, t, *J* = 7.3 Hz, H21). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 163.1 (C7), 153.9 (C2), 149.2 (C16), 146.3 (C11), 140.0 (C16), 137.0 (C23), 135.7 (C12), 130.8 (C8), 130.5 (C4), 129.5 (C26), 129.0 (C1), 128.8 (2C, C17), 128.4 (2C, C18), 128.3 (2C, C25), 128.2 (C19), 128.0 (2C, C14), 127.8 (C15), 127.6 (C6), 127.5 (2C, C13), 126.7 (2C, C24), 120.9 (C5), 113.4 (C3), 109.8 (C10), 70.2 (C22), 21.7 (C20), 13.6 (C21). **HPLC**: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 69.9 min, t_R (minor) = 30.2 min. **IR**: ν_{max} (thin film): 2929, 2240, 1643, 1602, 1494, 1449, 1375, 1271, 1226, 1025, 723, 697 cm^{-1}. **HRMS** (ES+): found 458.2115; $C_{32}H_{28}NO_2$, $[M+H]^+$ requires 458.2120. $[\alpha]_D^{293}$ +6.0 (*c* = 0.5, $CHCl_3$).

4-((2-(3-Ethyl-2-oxo-4,6-diphenylpyridin-1(2*H*)-yl)phenoxy)methyl)benzonitrile (4k)



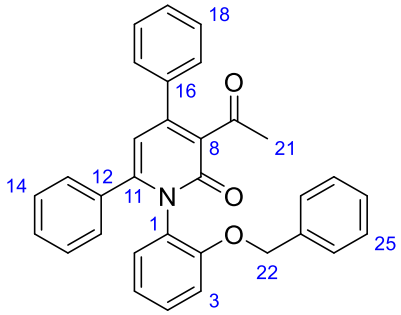
General Procedure D: Pyridone **3c** (50.0 mg, 136 µmol, 1.0 equiv.), QD-1 (11.3 mg, 20.4 µmol, 15 mol%). $CHCl_3$ (6 mL) was added followed by 50% aqueous K_2CO_3 (94.0 µL, 680 µmol, 5.0 equiv.) and R^2I-**S2** (165 mg, 680 µmol, 5.0 equiv.). Crude mixture purified by flash column chromatography (silica gel, 1% → 2.5% MeOH/dichloromethane) affording **4k** as a yellow foam (46 mg, 70%, 28% *ee*). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.64-7.61 (2H, m, H17), 7.50-7.38 (7H, m, H18, 19, 24, 25), 7.22-7.09 (7H, m, H4, 6, 13, 14, 15), 6.93 (1H, td, *J* = 7.6, 1.2 Hz, H5), 6.81 (1H, dd, *J* = 8.4, 1.1 Hz, H3), 6.19 (1H, s, H10), 5.17-5.02 (2H, m, H22), 2.66-2.53 (2H, m, H20), 1.16 (1H, t, *J* = 7.5 Hz, H21). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 163.0 (C7), 153.4 (C2), 149.4 (C9), 146.2 (C11), 142.4 (C23), 139.7 (C12), 135.6 (C16), 132.3 (2C, C25), 130.8 (C8), 130.6 (C19), 129.6 (C4), 129.0 (C1), 128.7 (2C, C18), 128.5 (2C, C14), 128.3 (C15), 128.0 (C5), 127.9 (2C, C24), 127.6 (2C, C13), 127.0 (2C, C17), 121.5 (C6), 118.7 (C27), 113.2 (C3), 111.5 (C26), 110.0 (C10), 69.2 (C22), 21.7 (C20), 13.6 (C21). **HPLC**: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 22.3 min, t_R (minor) = 17.4 min. **IR**: ν_{max} (thin film): 3056, 2929, 2228, 1640, 1605, 1539, 1494, 1446, 1274, 1021, 909, 726 cm^{-1}. **HRMS** (ES+): found 483.2078; $C_{33}H_{27}N_2O_2$, $[M+H]^+$ requires 483.2073. $[\alpha]_D^{293}$ +12.0 (*c* = 0.4, $CHCl_3$).

3-Ethyl-1-(2-((4-methylbenzyl)oxy)phenyl)-4,6-diphenylpyridin-2(1*H*)-one (4l)



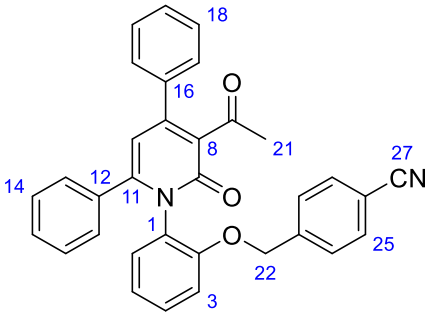
General Procedure D: Pyridone **3c** (50 mg, 136 µmol, 1.0 equiv.), QD-1 (11.3 mg, 20.4 µmol, 15 mol%). $CHCl_3$ (6 mL) was added followed by 50% aqueous K_2CO_3 (95.0 µL, 680 µmol, 5.0 equiv.) and R^2I-**S3** (158 mg, 680 µmol, 5.0 equiv.). Crude mixture purified by flash column chromatography (silica gel, 1% → 2.5% MeOH/dichloromethane) affording **4l** as a yellow foam (21 mg, 33%, 41% *ee*). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.48-7.37 (5H, m, H17, 18, 19), 7.23-7.07 (11H, m, H4, 6, 13, 14, 15, 22, 23), 6.90-6.86 (2H, m, H3, 5), 6.14 (1H, s, H10), 5.07-4.88 (2H, m, H22), 2.65-2.56 (2H, m, H20), 2.36 (3H, s, H27), 1.89 (3H, t, *J* = 7.3 Hz, H21). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 163.1 (C7), 154.0 (C2), 149.2 (C9), 146.3 (C11), 140.0 (C26), 137.3 (C12), 135.8 (C23), 134.0 (C16), 130.8 (C8), 130.5 (C4) 129.5 (C19), 129.0 (2C, C25), 129.0 (C1), 128.8 (2C, C17), 128.3 (2C, C14), 128.1 (C5), 128.1 (2C, C18), 127.8 (C15), 127.5 (2C, C13), 126.9 (2C, C24), 120.8 (C6), 113.4 (C3), 109.8 (C10), 70.2 (C22), 21.7 (C20), 21.2 (C27), 13.6 (C21). **HPLC**: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 26.9 min, t_R (minor) = 25.0 min. **IR**: ν_{max} (thin film): 3026, 2922, 2240, 1643, 1606, 1539, 1474, 1271, 1226, 909, 726, 697 cm^{-1}. **HRMS** (ES+): found 472.2289; $C_{33}H_{30}NO_2$, $[M+H]^+$ requires 472.2277. $[\alpha]_D^{293}$ +8.54 (*c* = 0.4, $CHCl_3$).

3-Acetyl-1-(2-(benzyloxy)phenyl)-4,6-diphenylpyridin-2(1*H*)-one (4m)



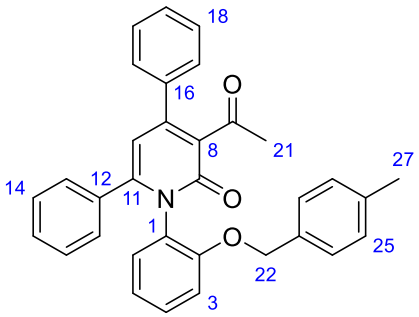
General Procedure D: Pyridone **3b** (100 mg, 262 µmol, 1.0 equiv.), QD-1 (21.6 mg, 39.3 µmol, 15 mol%). $CHCl_3$ (11 mL) was added followed by 50% aqueous K_2CO_3 (182 µL, 1.31 mmol, 5.0 equiv.) and R^2I-**S1** (163 µL, 1.31 mmol, 5.0 equiv.). Crude mixture purified by flash column chromatography (silica gel, 1% → 2.5% MeOH/dichloromethane) affording **4m** as a yellow foam (82 mg, 66%, 6% *ee*). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.44-7.40 (5H, m, H17, 18, 19), 7.37-7.31 (5H, m, H13, 14, 15), 7.23-7.18 (2H, m, H4, 26), 7.16-7.13 (5H, m, H6, 24, 25), 6.90-6.86 (2H, m, H3, 5), 6.27 (1H, s, H10), 5.11-4.95 (2H, m, H22), 2.45 (3H, s, H21). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 202.2 (C2), 160.6 (C7), 153.8 (C9), 150.8 (C2), 150.7 (C11), 137.9 (C16), 136.7 (C21), 135.0 (C12), 130.3 (C19), 130.1 (C4), 129.3 (C8), 129.0 (C15), 128.9 (C26), 128.6 (2C, C17), 128.6 (2C, C18), 128.5 (2C, C24), 128.0 (2C, C14), 127.8 (C6), 127.7 (2C, C13), 127.5 (C1), 126.9 (2C, C24), 121.0 (C5), 113.4 (C3), 109.7 (C8), 70.4 (C22), 31.7 (C21). **HPLC**: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 11.3 min, t_R (minor) = 9.4 min. **IR**: ν_{max} (thin film): 3060, 2243, 1699, 1640, 1528, 1490, 1367, 1271, 909, 752, 730, 697 cm^{-1}. **HRMS** (ES+): found 472.1904; $C_{32}H_{26}NO_3$, $[M+H]^+$ requires 472.1913. $[\alpha]_D^{293}$ +1.3 (*c* = 0.5, $CHCl_3$).

4-((2-(3-Acetyl-2-oxo-4,6-diphenylpyridin-1(2*H*)-yl)phenoxy)methyl)benzonitrile (4n)



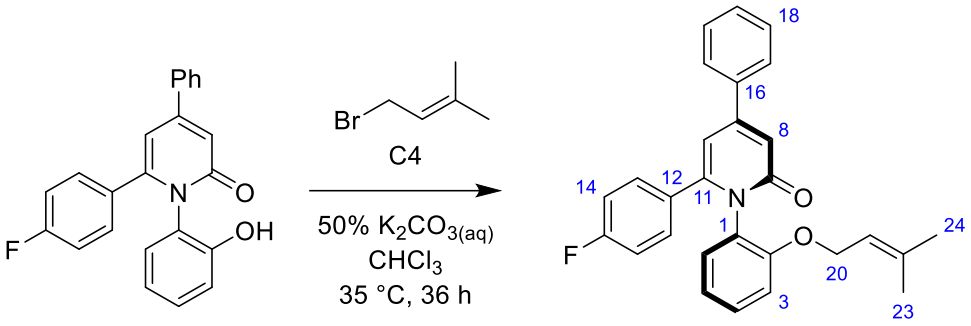
General Procedure D: Pyridone **3b** (50.0 mg, 131 µmol, 1.0 equiv.), QD-1 (10.8 mg, 19.7 µmol, 15 mol%). $CHCl_3$ (6 mL) was added followed by 50% aqueous K_2CO_3 (91.0 µL, 655 µmol, 5.0 equiv.) and R^2I-**S2** (159 mg, 655 µmol, 5.0 equiv.). Crude mixture purified by flash column chromatography (silica gel, 1% → 2.5% MeOH/dichloromethane) affording **4n** as a yellow foam (45 mg, 69%, 3% *ee*). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.67-7.64 (2H, m, H17), 7.46-7.40 (7H, m, H18, 19, 24, 25), 7.24-7.12 (7H, m, H4, 6, 13, 14, 15), 6.93 (1H, td, *J* = 7.6, 1.2 Hz, H5), 6.81 (1H, dd, *J* = 8.4, 1.2 Hz, H3), 6.31 (1H, s, H10), 5.16-5.05 (2H, m, H22). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 202.13 (C20), 160.5 (C7), 153.4 (C2), 150.6 (C11), 150.5 (C9), 142.0 (C23), 137.6 (C16), 134.9 (C12), 132.5 (2C, C25), 130.4 (C4), 130.2 (C19), 129.5 (C8), 129.2 (C15), 129.0 (C5), 128.8 (2C, C18), 128.5 (2C, C14), 127.9 (2C, C24), 127.9 (2C, C13), 127.6 (C1), 127.3 (2C, C17), 121.6 (C6), 118.7 (C27), 113.3 (C3), 111.8 (C26), 109.8 (C10), 69.5 (C22), 31.7 (C21). **HPLC**: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 13.7 min, t_R (minor) = 10.9 min. **IR**: ν_{max} (thin film): 3060, 2922, 2228, 1699, 1640, 1602, 1528, 1271, 909, 756, 726 cm^{-1}. **HRMS** (ES+): found 497.1862; $C_{33}H_{25}N_2O_3$, $[M+H]^+$ requires 497.1865. $[\alpha]_D^{293}$ +0.8 (*c* = 0.5, $CHCl_3$).

3-Acetyl-1-(2-((4-methylbenzyl)oxy)phenyl)-4,6-diphenylpyridin-2(1*H*)-one (4o)



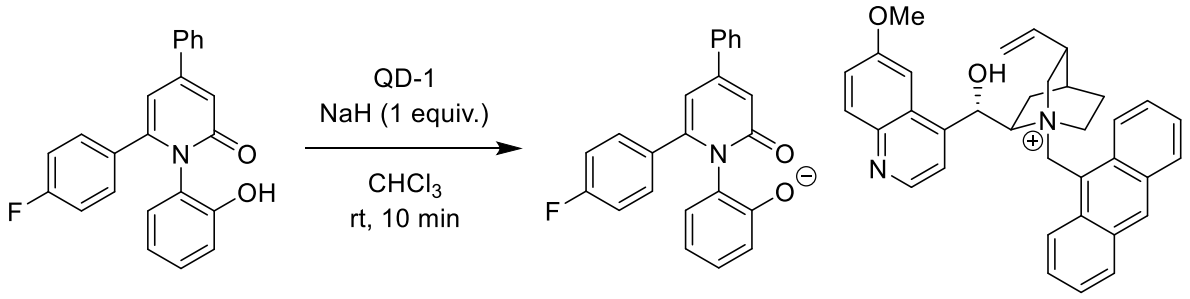
General Procedure D: Pyridone **3b** (50.0 mg, 131 µmol, 1.0 equiv.), QD-1 (10.8 mg, 19.7 µmol, 15 mol%). $CHCl_3$ (6 mL) was added followed by 50% aqueous K_2CO_3 (91.0 µL, 655 µmol, 5.0 equiv.) and R^2I-**S3** (152 mg, 655 µmol, 5.0 equiv.). Crude mixture purified by flash column chromatography (silica gel, 1% → 2.5% MeOH/dichloromethane) affording **4o** as an off-white foam (32 mg, 54%, 5% *ee*). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.42-7.39 (5H, m, H17, 18, 19), 7.24-7.11 (11H, m, H4, 6, 13, 14, 15, 22, 23), 6.90-6.87 (2H, m, H3, 5), 6.26 (1H, s, H10), 5.06-4.91 (2H, m, H22), 2.47 (3H, s, H21), 2.36 (3H, s, H27). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 202.2 (C20), 160.6 (C7), 153.9 (C2), 150.8 (C11), 150.7 (C16), 138.0 (C9), 137.6 (C26), 135.0 (C12), 133.7 (C23), 130.2 (C4), 130.0 (C5) 129.3 (C8), 129.2 (2C, C17), 129.0 (C19), 128.9 (C15), 128.6 (2C, C25), 128.6 (2C, C18), 128.0 (2C, C14), 127.7 (2C, C13), 127.5 (C1), 127.0 (2C, C24), 120.9 (C6), 113.4 (C3), 109.7 (C10), 70.4 (C22), 31.7 (C21), 21.2 (C27). **HPLC**: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 17.5 min, t_R (minor) = 15.6 min. **IR**: ν_{max} (thin film): 3056, 2922, 1699, 1643, 1602, 1531, 1494, 1267, 1028, 801, 760 cm^{-1}. **HRMS** (ES+): found 486.2066; $C_{33}H_{28}NO_3$, $[M+H]^+$ requires 486.2069. $[\alpha]_D^{293}$ +0.9 (*c* = 0.6, $CHCl_3$).

2.7 6-(4-Fluorophenyl)-1-(2-((3-methylbut-2-en-1-yl)oxy)phenyl)-4-phenylpyridin-2(1H)-one (4p)

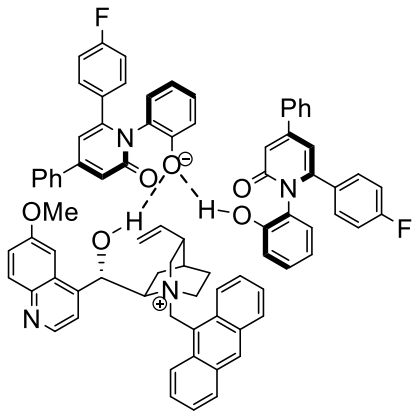


A round-bottomed flask was charged with 2′-hydroxyphenyl-2-pyridone **3e** (50 mg, 140 µmol, 1.0 equiv.), **C4** (7.7 mg, 14.0 µmol, 15 mol%). $CHCl_3$ (6 mL) was added followed by 50% aqueous K_2CO_3 (97.0 µL, 700 µmol, 5.0 equiv.) and 3,3-dimethylallyl bromide (81 µL, 700 µmol, 5.0 equiv.) as the flask was sealed and stirred at 35 °C for 18 h. Piperidine (4.0 equiv.) was added and reaction mixture was stirred for 30 mins before addition of 3M HCl (*ca*. 1 mL). Crude product was extracted with EtOAc (3 x10 mL/mmol), dried over anhydrous magnesium sulfate and the solvent removed *in vacuo* resulting in a crude solid. Crude mixture purified by flash column chromatography (silica gel, 1% → 2.5% MeOH/dichloromethane) affording **4p** as a yellow foam (47 mg, 59%, 47% *ee*). **^{1}H NMR** (400 MHz, $CDCl_3$): δ_H = 7.69-7.66 (2H, m, H17), 7.50-7.42 (3H, m, H18, 19), 7.22-7.16 (3H, m, H4, 22), 7.10 (1H, dd, *J* = 7.7, 1.7 Hz, H6), 6.91-6.78 (5H, m, H3, 5, 10, 14), 6.47 (1H, d, *J* = 2.0 Hz, H8), 5.31-5.27 (1H, m, H21), 4.51-4.35 (2H, m, H20), 1.74 (3H, s, H24), 1.68 (3H, s, H23). **^{13}C NMR** (101 MHz $CDCl_3$): δ_C = 163.8 (C7), 162.5 (d, $^1J_{CF}$ = 248.7 Hz, C15), 153.7 (C9), 151.1 (C2), 149.2 (C11), 137.7 (C16), 137.0 (C22), 131.9 (d, $^4J_{CF}$ = 3.6 Hz, C12), 130.7 (d, $^3J_{CF}$ = 8.4 Hz, C13), 130.3 (C4), 129.9 (C19), 129.5 (C5), 129.0 (2C, C17), 127.7 (C1), 126.8 (2C, C18), 120.5 (C5), 119.8 (C21), 116.3 (C8), 114.6 (d, $^2J_{CF}$ = 21.8 Hz, C14), 113.2 (C3), 106.9 (C10), 65.5 (C20), 25.7 (C24), 18.3 (C23). **^{19}F NMR** (376 MHz, $CDCl_3$): δ_F = -112.3. **HPLC**: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 29.5 min, t_R (minor) = 13.7 min. **IR**: ν_{max} (thin film): 3060, 2922, 1658, 1605, 1498, 1364, 1267, 1222, 998, 842, 749, 697 cm^{-1}. **HRMS** (ES+): found 426.1867; $C_{28}H_{25}NO_2$, $[M+H]^+$ requires 426.1869. $[\alpha]_D^{293}$ +19.5 (*c* = 0.4, $CHCl_3$).

2.8 Catalyst-substrate co-crystal (6)

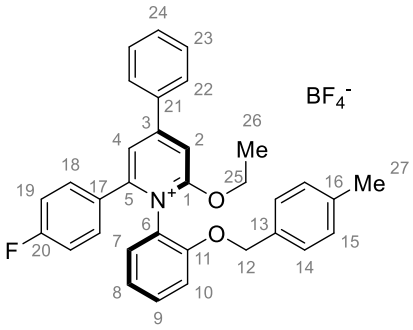


crystals formed:



A flask was charged with pyridone **3c** (25 mg, 70.0 µmol, 1.0 equiv.) and evacuated/backfilled with Ar under vacuum. THF (3mL) was added followed by NaH (2.0 mg, 70.0 µmol, 1.0 equiv.) as the flask was sealed and stirred at room temperature for 10 minutes. Under Ar, QD-1 was added and reaction mixture left to stir for a further 10 minutes. The reaction mixture was washed through a celite pad with EtOAc (25 mL) and reduced in vacuo affording a yellow residue. Recrystallization from hot EtOAc was attempted but with no success, the solution was reduced to dryness and $CHCl_3$ was added. A small portion (ca. 0.2 mL) was removed and subjected to vapour diffusion, using n-hexane (ca. 1 mL) as the anti-solvent, affording yellow crystals.

2.9 Synthesis of Pyridinium Salt 7



Triethyloxonium tetrafluoroborate (13 mg, 0.068 mmol) was added in one portion to a stirred solution of pyridone **4i** (15 mg, 0.032 mmol) in anhydrous dichloromethane (1 mL). After 1 h the reaction mixture was diluted with dichloromethane (5 mL) and washed with sodium bicarbonate (sat. aq., 5 mL). The aqueous phase was extracted with dichloromethane (3 x 5 mL). The organic extracts were combined, dried over anhydrous magnesium sulfate, filtered and concentrated *in vacuo*. The crude residue was purified by flash column chromatography (SiO_2, 2% MeOH in dichloromethane) to afford the product **7** salt as a beige solid (17 mg, 91%). δ_H ($CDCl_3$, 600 MHz): 7.93-7.89 (2H, m, ArC), 7.65 (1H, s, H2), 7.60-7.54 (4H, m, ArH), 7.50 (1H, s, H4), 7.33 (1H, t, *J* 7.9, ArH), 7.29-7.27 (2H, m, ArH), 7.14 (2H, d, *J* 7.8, H15), 7.07 (2H, d, *J* 7.8, H14) 6.99 (1H, t, *J* 7.7, ArH), 6.94-6.89 (3H, m, ArH), 5.02 (1H, dd, *J* 12.0, H12), 4.96 (1H, dd, *J* 12.0, H12'), 4.75-4.69 (1H, m, H25), 4.57-4.50 (1H, m, H25'), 2.34 (3H, s, H27), 1.30 (3H, t, *J* 7.0, H26); δ_C ($CDCl_3$, 151 Hz) 163.6 (d, *J* 252.6), 160.9, 160.4, 153.2, 152.2 (C11), 138.5, 135.0, 132.5, 132.03, 132.01, 131.4 (d, *J* 8.8, C18), 129.73, 129.68, 129.5 (C15), 128.4, 128.3, 127.0 (C14), 125.0, 121.7, 118.4 (C4), 115.5 (d, *J* 22.1, C19), 113.0, 107.3 (C2), 70.5 (C12), 69.9 (C25), 21.9 (C27), 13.9 (C26); δF ($CDCl_3$, 564 MHz): -108.7 (1F, s, Ar-F), 153.4 (4F, s, BF_4^-); **HRMS (ES$^+$)** found 490.2165 ($C_{33}H_{29}NO_2F^+$, $[M\text{-}BF_4^-]^+$ requires 490.2182), **HRMS (ES$^-$)** found 86.9950 ($[BF_4]^-$ requires 87.0029); 664.2233 ($C_{33}H_{29}B_2NO_2F_9^-$, $[M+BF_4]^-$ requires 664.2241). **HPLC**: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 54.9 min, t_R (minor) = 20.9 min, 49% *ee*; $[\boldsymbol{\alpha}]_{\boldsymbol{D}}^{\boldsymbol{293}}$ +49.6 (*c* = 0.4, $CHCl_3$).

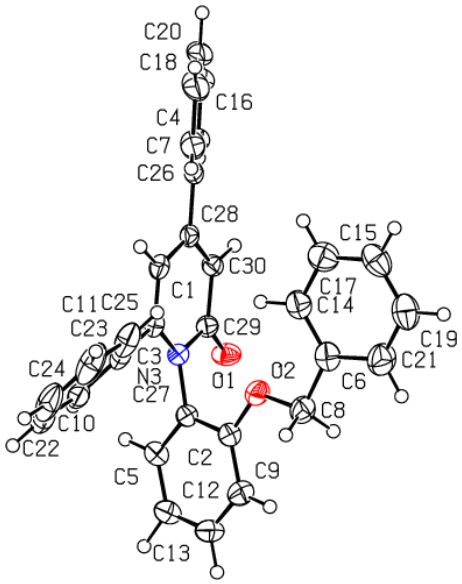
3 X-Ray Crystallography

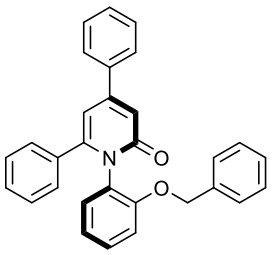
Low temperature[12] and room temperature single crystal X-ray diffraction studies for **4a**, **4b**, **4j**, **4m** were carried out using CuK$_\alpha$ radiation on an Agilent Supernova diffractometer equipped with an area detector and graphite monochromator. Raw frame data were reduced using CrysAlisPRO[13] and solved using SHELXT 2015.[14] Full-matrix least-squares refinement of the structures were carried out using CRYSTALS.[15] The absolute stereochemistry of **4b** was found using the method of Flack.[16]

X-ray diffraction studies for **3a** were conducted using CuK$_\alpha$ on a Rigaku 007HF equipped with Varimax confocal mirrors and an AFC11 goniometer and HyPix 6000 detector, at the National Crystallography Service in the University of Southampton. Raw frame data were reduced using CrysAlisPRO[2] and solved using SHELXT.[14] Full-matrix least-squared refinement of the structure was carried out using SHELXL[17] and visualised using Olex 1.3.[18] A void containing disordered solvent molecules necessitated the use of a solvent mask. A mask was calculated, and 1559 electrons were found in a volume of 5917 Å^3 in 1 void per unit cell. This is consistent with the presence of 3[$CHCl_3$] per asymmetric unit. One of the pyridone phenol moieties is disordered, so was refined over two positions (occupancies 0.63 and 0.37).

Full refinement details are given in the supplementary material (CIF). CCDC 2124211 (**4a**), 2124210 (**4b**), 2124208 (**4j**), 2124209 (**4m**) and 2124143 (**6**) contain the supplementary crystallographic data for this paper. These data are provided free of charge by The Cambridge Crystallographic Data Centre and copies can be obtained free of charge *via* www.ccdc.cam.ac.uk/data_request/cif.

(±)-4a (CCDC 2124211)

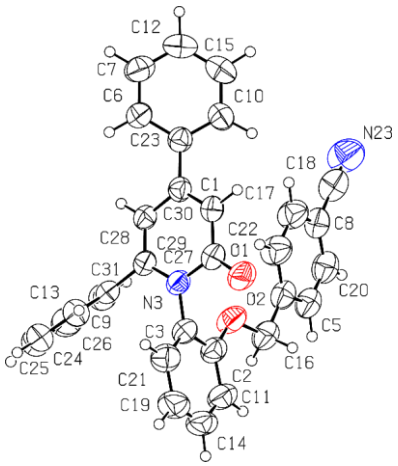


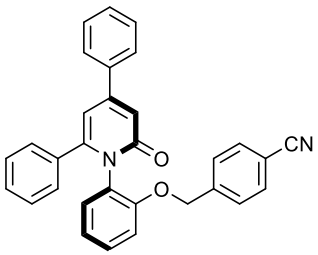


(±)

Crystal data	
Chemical formula	$C_{30}H_{23}NO_2$
M_r	429.52
Crystal system, space group	Monoclinic, *I*2/*a*
Temperature (K)	100
a, *b*, *c* (Å)	18.54, 10.37, 24.28
β (°)	103.312 (3)
V (Å^3)	4542.64 (6)
Z	8
Radiation type	Cu *K*α
μ (mm^{-1})	0.62
Crystal size (mm)	0.20 × 0.16 × 0.16
Data collection	
Diffractometer	Oxford Diffraction SuperNova
Absorption correction	Multi-scan *CrysAlis PRO* (Rigaku Oxford Diffraction, 2017)
T_{min}, T_{max}	0.78, 0.91
No. of measured, independent and observed [$I > 2.0\sigma(I)$] reflections	22074, 4384, 3995
R_{int}	0.019
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.615
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.048, 0.125, 1.00
No. of reflections	4384
No. of parameters	299
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.05, −0.17

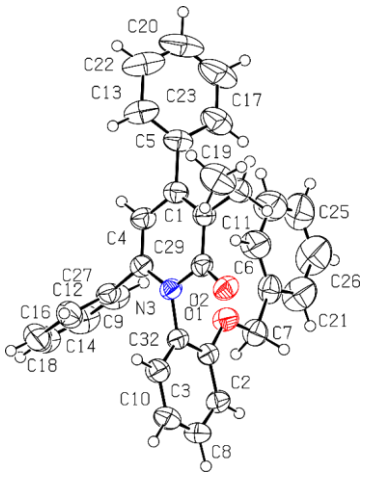
(+)-4b (CCDC 2124210)

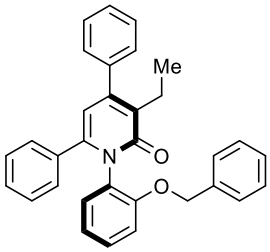




Crystal data	
Chemical formula	$C_{62}H_{44}N_4O_4$
M_r	909.06
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	299
a, b, c (Å)	9.3537 (3), 12.1471 (4), 11.0218 (3)
β (°)	104.661 (3)
V (Å^3)	1211.52 (6)
Z	1
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.62
Crystal size (mm)	0.16 × 0.16 × 0.12
Data collection	
Diffractometer	Oxford Diffraction SuperNova
Absorption correction	Multi-scan *CrysAlis PRO* (Rigaku Oxford Diffraction, 2017)
T_{min}, T_{max}	0.76, 0.93
No. of measured, independent and observed [$I > 2.0\sigma(I)$] reflections	6917, 4528, 4016
R_{int}	0.000
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.615
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.037, 0.094, 0.95
No. of reflections	4528
No. of parameters	317
No. of restraints	1
H-atom treatment	H-atom parameters not refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.27
Absolute structure	Parsons, Flack & Wagner (2013), 1957 Friedel Pairs
Absolute structure parameter	0.02 (15)

(±)-4j (CCDC 2124208)

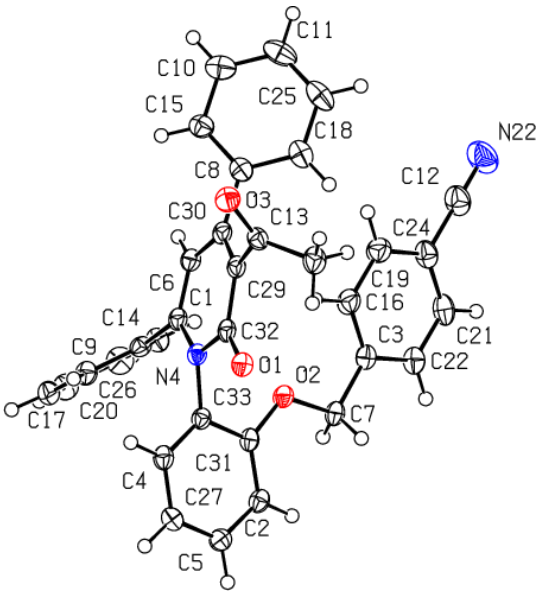


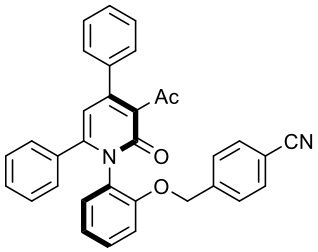


(±)

Crystal data	
Chemical formula	$C_{32}H_{27}NO_2$
M_r	457.55
Crystal system, space group	Monoclinic, *I*2/*a*
Temperature (K)	293
a, *b*, *c* (Å)	21.811899 (14), 9.84050 (1), 23.848099 (14)
β (°)	101.3470 (18)
V (Å^3)	5018.70 (3)
Z	8
Radiation type	Cu *K*α
μ (mm^{-1})	0.59
Crystal size (mm)	0.30 × 0.28 × 0.18
Data collection	
Diffractometer	Oxford Diffraction SuperNova
Absorption correction	Multi-scan *CrysAlis PRO* (Rigaku Oxford Diffraction, 2017)
T_{min}, T_{max}	0.67, 0.90
No. of measured, independent and observed [$I > 2.0\sigma(I)$] reflections	45155, 4846, 4399
R_{int}	0.021
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.615
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.041, 0.110, 0.88
No. of reflections	4846
No. of parameters	316
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.13, −0.18

(±)-4m (CCDC 2124209)

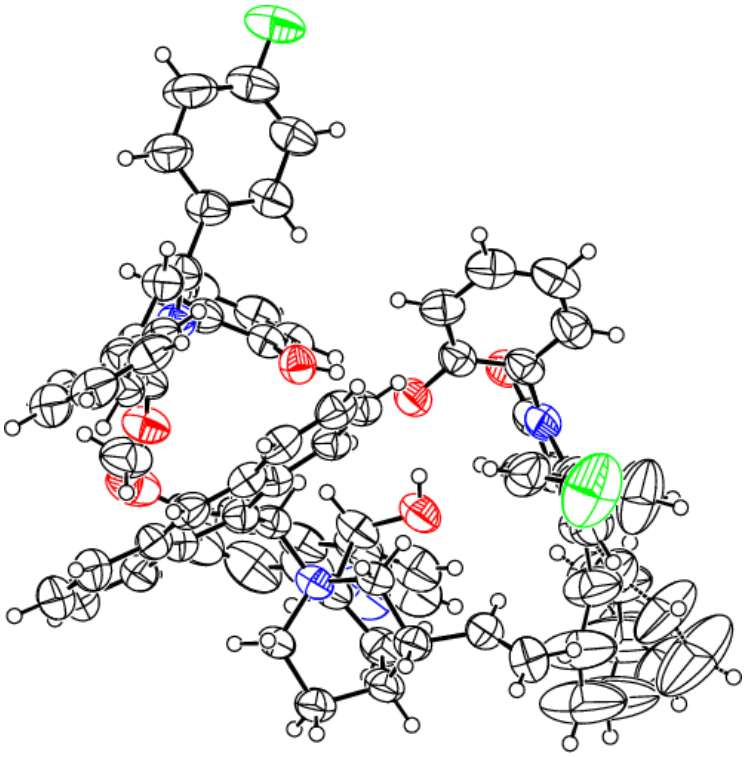


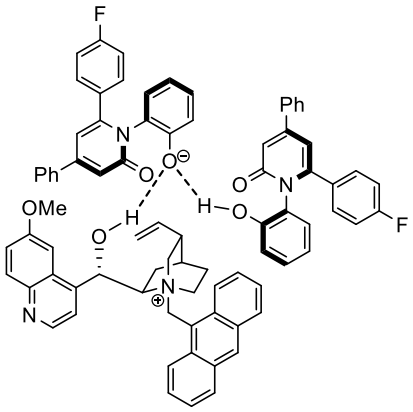


(±)

Crystal data	
Chemical formula	$C_{132}H_{96}N_8O_{12}$
M_r	1986.26
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	100
a, b, c (Å)	14.759600 (14), 9.835999 (10), 17.417799 (14)
β (°)	94.4720 (18)
V (Å^3)	2520.94 (1)
Z	1
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.67
Crystal size (mm)	0.30 × 0.10 × 0.10
Data collection	
Diffractometer	Oxford Diffraction SuperNova
Absorption correction	Multi-scan *CrysAlis PRO* (Rigaku Oxford Diffraction, 2017)
T_{min}, T_{max}	0.93, 0.93
No. of measured, independent and observed [$I > 2.0\sigma(I)$] reflections	23616, 4859, 4283
R_{int}	0.122
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.615
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.052, 0.138, 0.96
No. of reflections	4859
No. of parameters	343
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.33, −0.30

6 (CCDC 2124143)





Crystal data	
Chemical formula	$C_{23}H_{16}FNO_2 \cdot C_{23}H_{15}FNO_2 \cdot C_{35}H_{35}N_2O_2$
M_r	1229.37
Crystal system, space group	Trigonal, *R*3
Temperature (K)	100
a, *c* (Å)	28.7071 (4), 25.9528 (4)
V (Å^3)	18522.2 (6)
Z	9
Radiation type	Cu *K*α
μ (mm^{-1})	0.53
Crystal size (mm)	0.22 × 0.04 × 0.04
Data collection	
Diffractometer	Rigaku 007HF diffractometer equipped with Varimax confocal mirrors, an AFC11 goniometer and HyPix 6000 detector
Absorption correction	Multi-scan *CrysAlis PRO* 1.171.41.113a (Rigaku Oxford Diffraction, 2021) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.
T_{min}, T_{max}	0.393, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	72024, 14182, 10313

R_{int}	0.051
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.040, 0.110, 1.02
No. of reflections	14182
No. of parameters	896
No. of restraints	841
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.15, −0.11
Absolute structure	Flack x determined using 3880 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).
Absolute structure parameter	0.01 (8)

4 Measurement of Rotational Barriers

The phenol starting materials **3a-e** underwent rapid racemisation at room temperature, necessitating the separation of racemic mixtures by HPLC on a chiral stationary phase immediately prior to the thermal racemisation analysis.

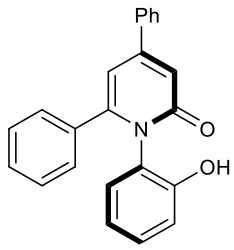
General Procedure E – Measurement of Barriers in Phenols 3a-3e

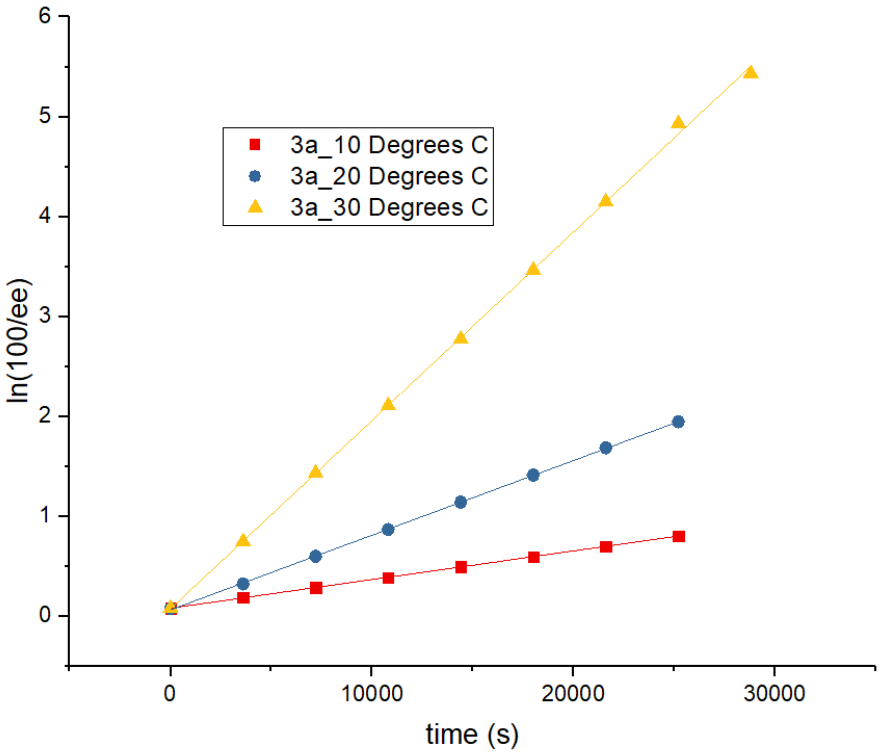
A sample of racemic 2′-hydroxyphenyl-2-pyridone was separated on HPLC (3a, d and e, Chiralpak OD-RH, 60:40 MeCN/H_2O, 0.75 mL/min) or (3b and c, SB-Cellulose, 20:80 IPA/hexane, 1 mL/min) and each enantiomer semi-preparatively collected to ensure two enantioenriched samples of each enantiomer of the respective 2′-hydroxyphenyl-2-pyridone. The samples were stored at 0 °C until sampled as soon as possible for HPLC analysis (under the same conditions required for initial enantiomer separation) over 7-12 hours with the column and the sample tray held at the desired temperature (10, 20 or 30 °C) until analysis was complete. Compounds were sampled in either MeCN (60 µL, 3a, d and e) or IPA (60 µL, 3b and c).

General Procedure F – Measurement of Barriers in Products 4a-4p

A sample of the benzylated product (1-2 mg) was dissolved in DMSO (80 µL), placed in a glass vial and immersed in a temperature-controlled oil bath (t = 0) set to the desired temperature (60, 70 and 80 °C). Samples were removed using the tip of a pasteur pipette at a series of time-points (e.g. t = 1 h, 2 h, 3 h, 4 h, 6 h, 16 h, 30 h, 54 h, 76 h). Each sample was added to an HPLC vial containing acetonitrile (1 mL) and its *ee* measured by HPLC on a chiral stationary phase (Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min).

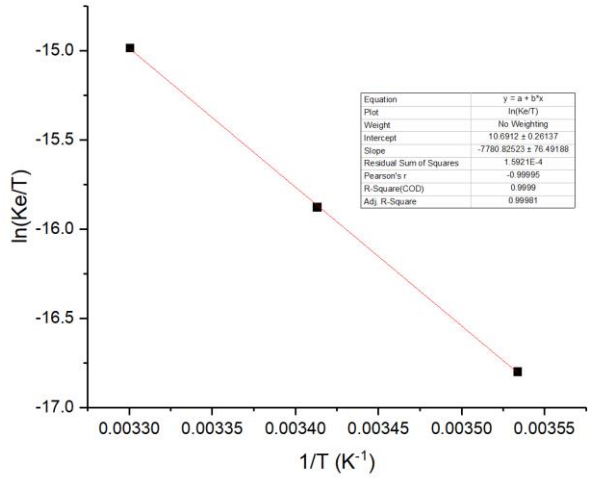
3a





Equation	y = a + b*x		
Plot	ln(100/ee)	ln(100/ee)	ln(100/ee)
Weight	No Weighting		
Intercept	0.08202 ± 0.00118	0.06561 ± 0.00469	0.07507 ± 0.02999
Slope	2.86714E-5 ± 7.8339E-8	7.47416E-5 ± 3.11499E-7	1.88598E-4 ± 1.74965E-6
Residual Sum of Squares	2.00429E-5	3.16897E-4	0.01666
Pearson's r	0.99998	0.99995	0.9997
R-Square(COD)	0.99996	0.9999	0.9994
Adj. R-Square	0.99995	0.99988	0.99931

	T (K)		
	283	**293**	**303**
k_{rac} (s^{-1})	2.867E-05	7.474E-05	1.886E-04
k_{ent} (s^{-1})	1.434E-05	3.737E-05	9.430E-05
$\Delta G_T^{\ddagger}$ (kJ mol^{-1})	95.43	96.56	97.60
$t_{1/2}(rac)_T$ (min)	402.93	154.57	61.25

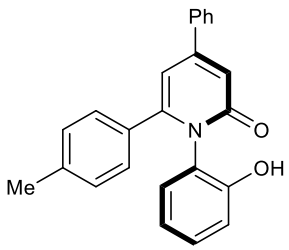


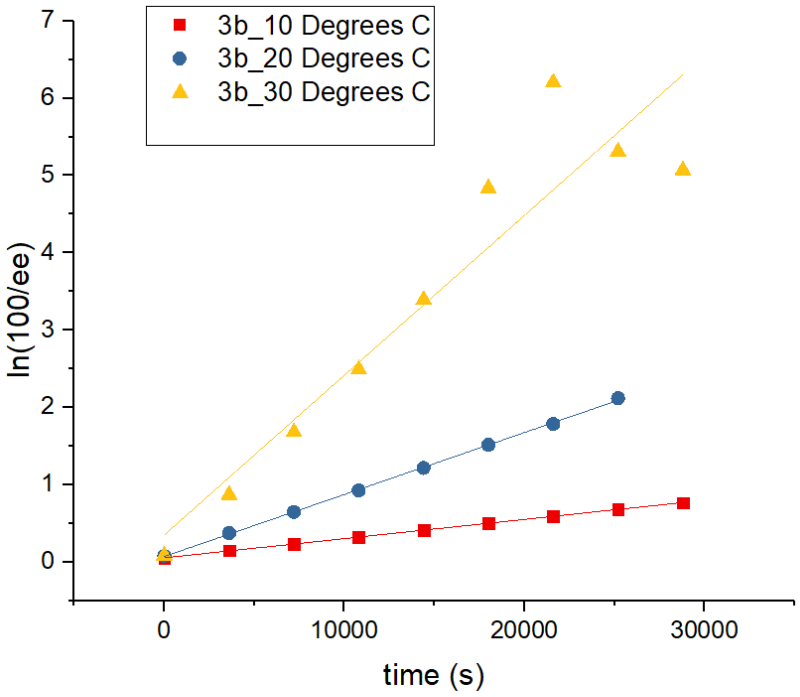
Equation	y = a + b*x
Plot	ln(Ke/T)
Weight	No Weighting
Intercept	10.6912 ± 0.26137
Slope	-7780.82523 ± 76.49188
Residual Sum of Squares	1.5921E-4
Pearson's r	-0.99995
R-Square(COD)	0.9999
Adj. R-Square	0.99981

$\Delta H_{ent}^{\ddagger} = 64.7$ kJ mol^{-1}

$\Delta S_{ent}^{\ddagger} = -108.7$ J K^{-1} mol^{-1}

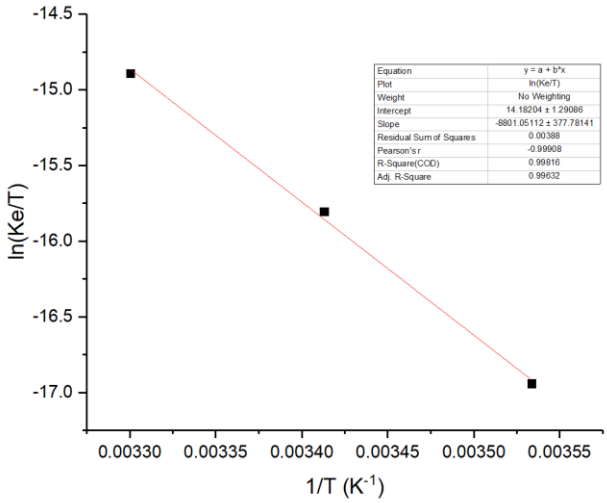
3b





Equation	y = a + b*x		
Plot	ln(100/ee)	ln(100/ee)	ln(100/ee)
Weight		No Weighting	
Intercept	0.05367 ± 0.00242	0.07136 ± 0.01036	0.34948 ± 0.47954
Slope	2.4848E-5 ± 1.4133E-7	8.01546E-5 ± 6.8824E-7	2.06711E-4 ± 2.79789E-5
Residual Sum of Squares	1.08723E-4	0.00155	4.26104
Pearson's r	0.99989	0.99978	0.94145
R-Square(COD)	0.99977	0.99956	0.88633
Adj. R-Square	0.99974	0.99948	0.8701

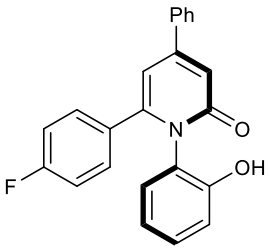
	T (K)		
	283	**293**	**303**
k_{rac} (s^{-1})	2.485E-05	8.015E-05	2.067E-04
k_{ent} (s^{-1})	1.242E-05	4.008E-05	1.034E-04
$\Delta G_T^{\ddagger}$ (kJ mol^{-1})	95.77	96.39	97.37
$t_{1/2}(rac)_T$ (min)	464.92	144.13	55.89

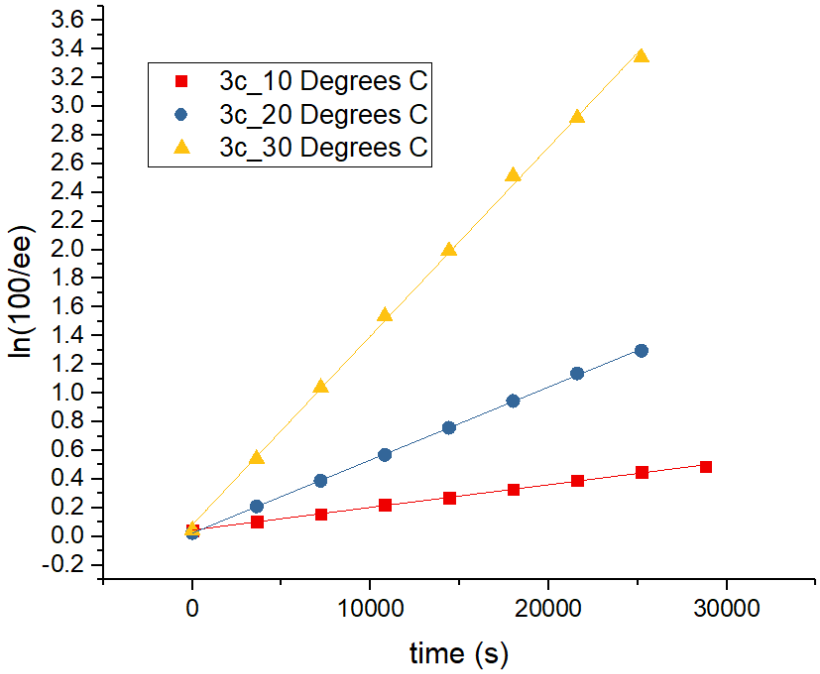


$\Delta H_{ent}^{\ddagger} = 73.2$ kJ mol^{-1}

$\Delta S_{ent}^{\ddagger} = -79.6$ J K^{-1} mol^{-1}

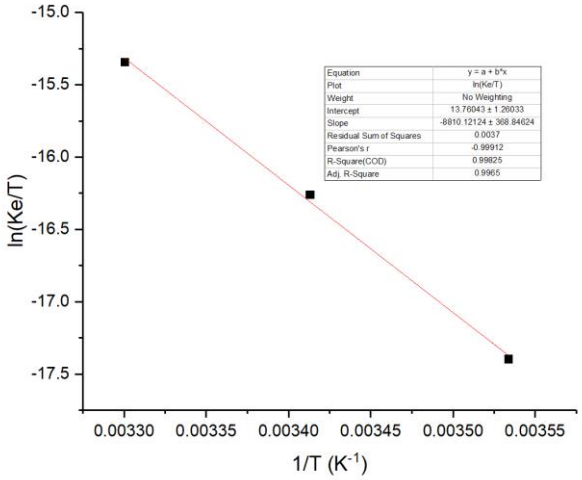
3c





Equation	y = a + b*x		
Plot	ln(100/ee)	ln(100/ee)	ln(100/ee)
Weight		No Weighting	
Intercept	0.04601 ± 0.00428	0.025 ± 0.00493	0.08217 ± 0.02716
Slope	1.57896E-5 ± 2.49711E-7	5.09242E-5 ± 3.27242E-7	1.31733E-4 ± 1.80372E-6
Residual Sum of Squares	3.39413E-4	3.4974E-4	0.01063
Pearson's r	0.99913	0.99988	0.99944
R-Square(COD)	0.99825	0.99975	0.99888
Adj. R-Square	0.998	0.99971	0.99869

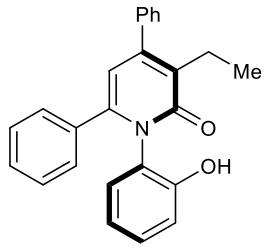
	T (K)		
	283	**293**	**303**
k_{rac} (s^{-1})	1.579E-05	5.092E-05	1.317E-04
k_{ent} (s^{-1})	7.895E-06	2.546E-05	6.587E-05
$\Delta G_T^{\ddagger}$ (kJ mol^{-1})	96.84	97.49	98.51
$t_{1/2}(rac)_T$ (min)	731.65	226.86	87.70

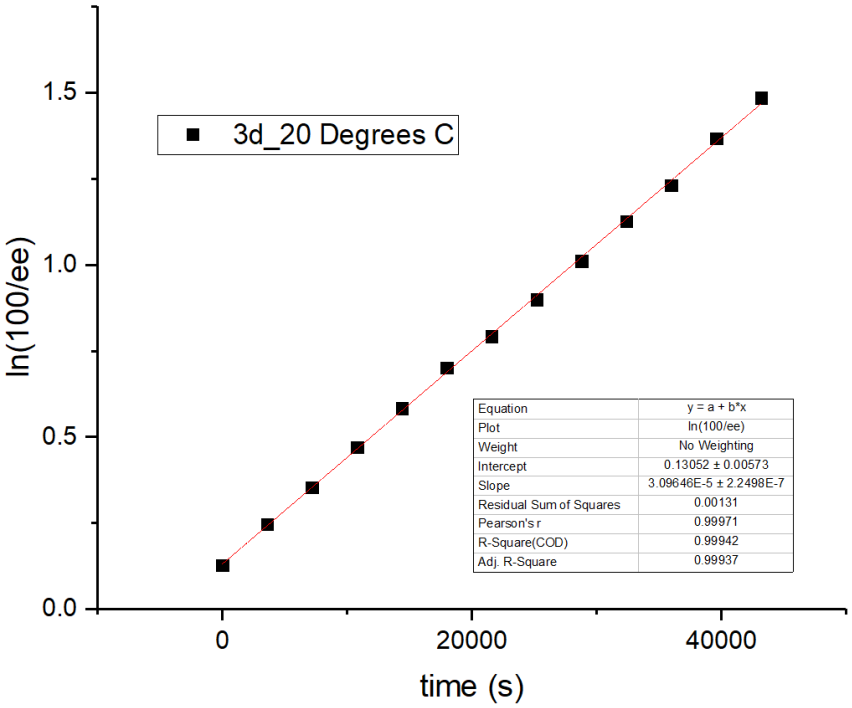


$\Delta H_{ent}^{\ddagger} = 73.3$ kJ mol^{-1}

$\Delta S_{ent}^{\ddagger} = -83.1$ J K^{-1} mol^{-1}

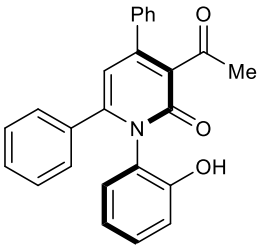
3d

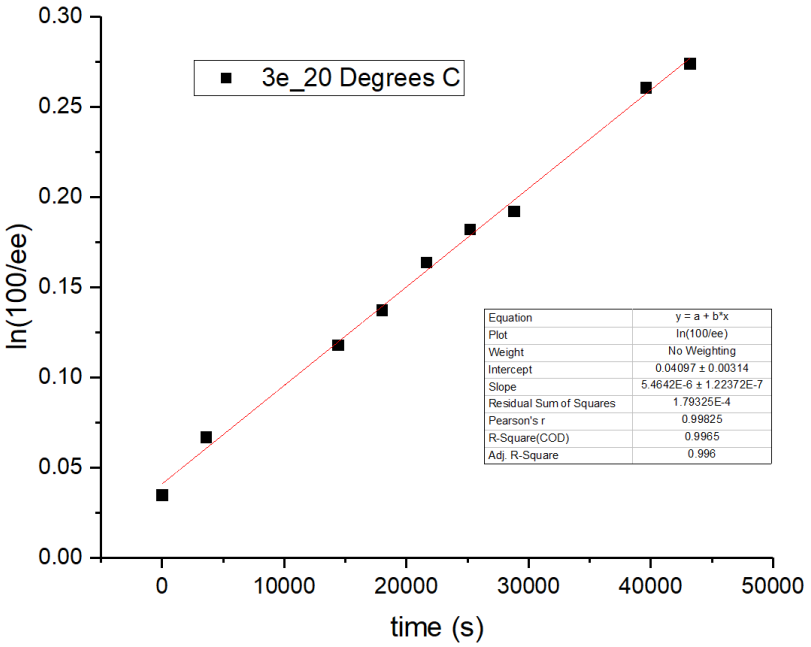




	T (K)
	393
k_{rac} (s^{-1})	3.096E-05
k_{ent} (s^{-1})	1.548E-05
$\Delta G_T^{\ddagger}$ (kJ mol^{-1})	98.70
$t_{1/2}(rac)_T$ (min)	373.09

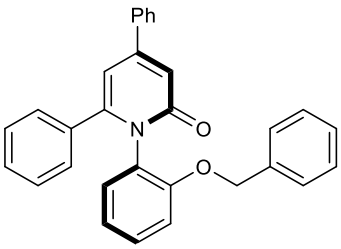
3e

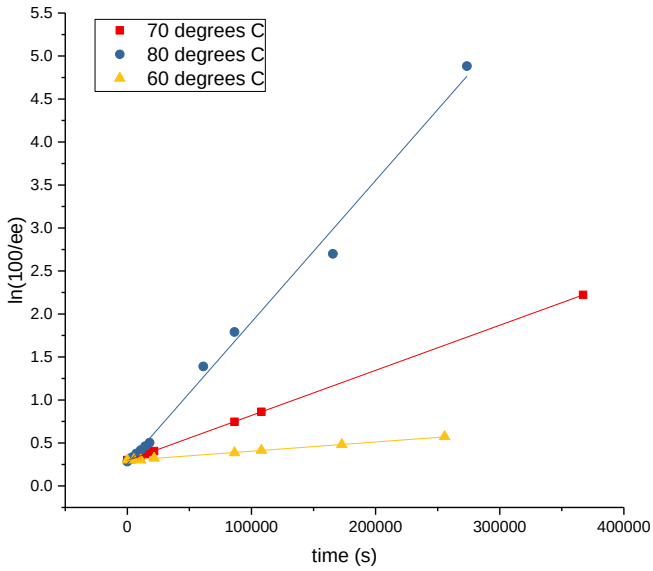




	T (K)
	393
k_{rac} (s^{-1})	5.464E-06
k_{ent} (s^{-1})	2.732E-06
$\Delta G_T^{\ddagger}$ (kJ mol^{-1})	102.93
$t_{1/2}(rac)_T$ (min)	2114.21

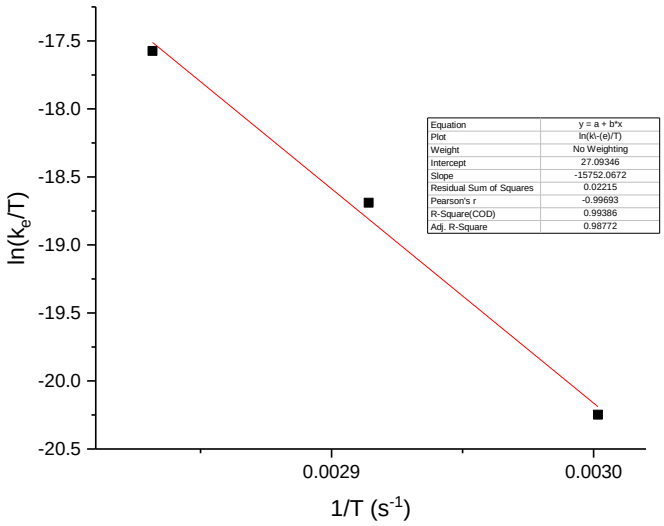
4a





Equation	y = a + b*x		
Plot	ln(100/ee)	ln(100/ee)	ln(100/ee)
Weight	No Weighting		
Intercept	0.2954	0.25744	0.29711
Slope	5.24593E-6	1.64779E-5	1.0719E-6
Residual Sum of Square	7.70709E-5	0.12878	1.33402E-4
Pearson's r	0.99999	0.99677	0.99904
R-Square(COD)	0.99998	0.99356	0.99808
Adj. R-Square	0.99997	0.99275	0.99776

	T (K)		
	333	**343**	**353**
k_{rac} (s^{-1})	1.072E-06	5.246E-06	1.648E-05
k_{ent} (s^{-1})	5.360E-07	2.623E-06	8.239E-06
$\Delta G_T^{\ddagger}$ (kJ mol^{-1})	121.84	121.06	121.31
$t_{1/2}(rac)_T$ (min)	10777.55	2202.17	701.09



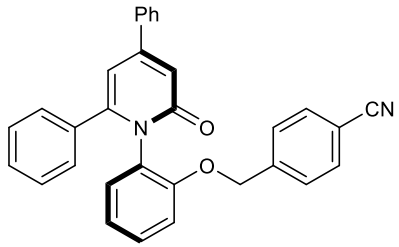
Equation	y = a + b*x
Plot	ln(k-(e)/T)
Weight	No Weighting
Intercept	27.09346
Slope	-15752.0672
Residual Sum of Squares	0.02215
Pearson's r	-0.99693
R-Square(COD)	0.99386
Adj. R-Square	0.98772

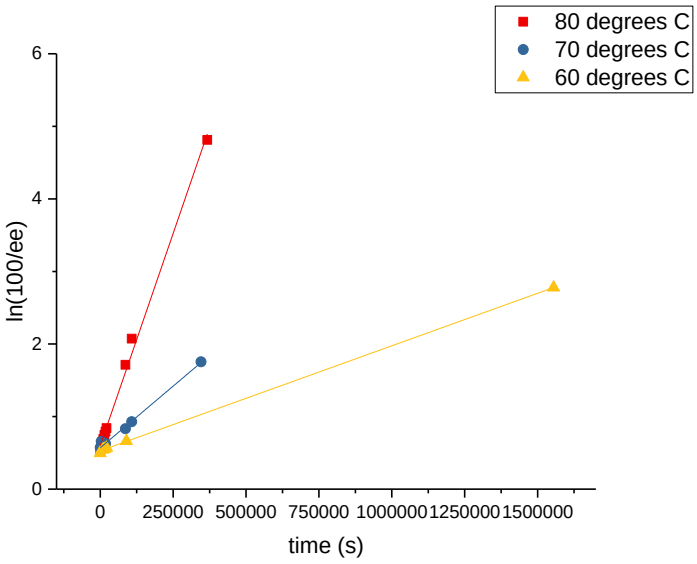
$\Delta H_{ent}^{\ddagger} = 131.0$ kJ mol^{-1}

$\Delta S_{ent}^{\ddagger} = +27.7$ J K^{-1} mol^{-1}

Extrapolated half-life of racemization at 20°C: 14.3 y

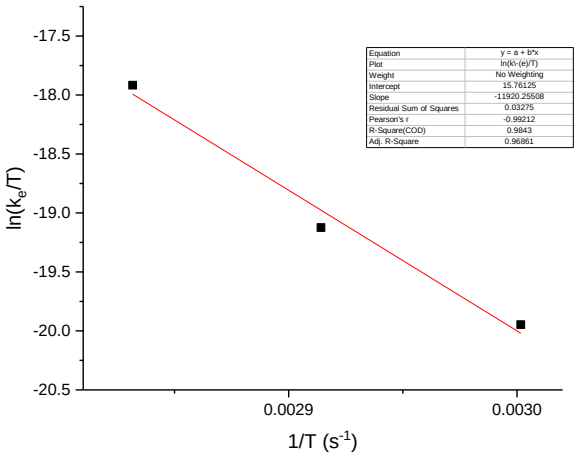
4b





Equation	y = a + b*x		
Plot	ln(100/ee)	ln(100/ee)	ln(100/ee)
Weight		No Weighting	
Intercept	0.60148	0.57021	0.52778
Slope	1.16899E-5	3.39693E-6	1.44743E-6
Residual Sum of Squares	0.0704	0.00759	0.00132
Pearson's r	0.99776	0.99673	0.99984
R-Square(COD)	0.99552	0.99347	0.99969
Adj. R-Square	0.99496	0.99254	0.99963

	T (K)		
	333		**333**
k_{rac} (s^{-1})	1.447E-06	k_{rac} (s^{-1})	1.447E-06
k_{ent} (s^{-1})	7.237E-07	k_{ent} (s^{-1})	7.237E-07
$\Delta G_T^{\ddagger}$ (kJ mol^{-1})	121.01	$\Delta G_T^{\ddagger}$ (kJ mol^{-1})	121.01
$t_{1/2}(rac)_T$ (min)	7981.36	$t_{1/2}(rac)_T$ (min)	7981.36



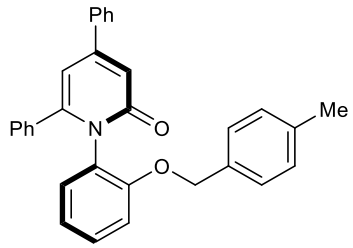
Equation	y = a + b*x
Plot	ln(k\-(e)/T)
Weight	No Weighting
Intercept	15.76125
Slope	-11920.25508
Residual Sum of Squares	0.03275
Pearson's r	-0.99212
R-Square(COD)	0.9843
Adj. R-Square	0.96861

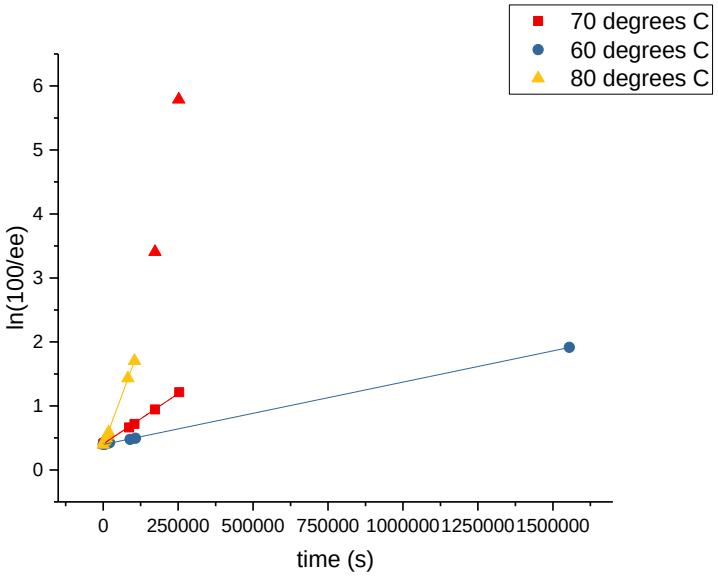
$\Delta H_{ent}^{\ddagger} = 99.1$ kJ mol^{-1}

$\Delta S_{ent}^{\ddagger} = -66.5$ J K^{-1} mol^{-1}

Extrapolated half-life of racemization at 20°C: 2.5 y

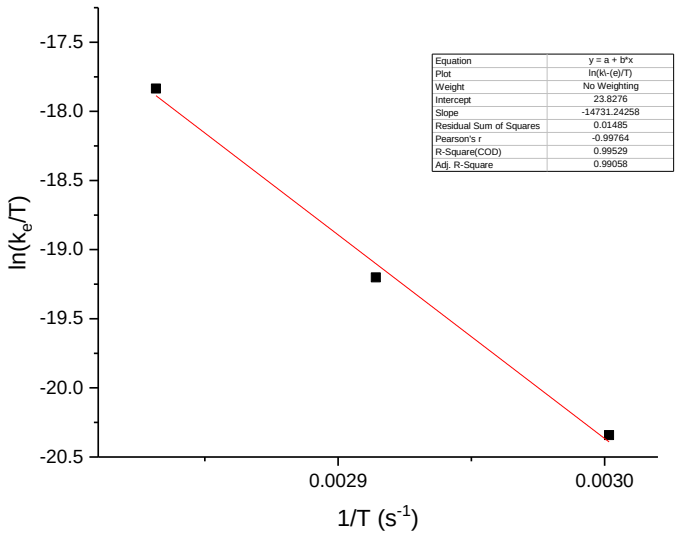
4c





Equation	y = a + b*x		
Plot	ln(100/ee)	ln(100/ee)	ln(100/ee)
Weight		No Weighting	
Intercept	0.40512	0.39669	0.37596
Slope	3.14403E-6	9.75417E-7	1.26864E-5
Residual Sum of Squares	0.0012	1.63651E-4	0.00197
Pearson's r	0.99911	0.99996	0.99945
R-Square(COD)	0.99822	0.99992	0.99891
Adj. R-Square	0.998	0.9999	0.99872

			T (K)
	333		**333**
k_{rac} (s^{-1})	9.754E-07	k_{rac} (s^{-1})	9.754E-07
k_{ent} (s^{-1})	4.877E-07	k_{ent} (s^{-1})	4.877E-07
$\Delta G_T^{\ddagger}$ (kJ mol^{-1})	122.11	$\Delta G_T^{\ddagger}$ (kJ mol^{-1})	122.11
$t_{1/2}(rac)_T$ (min)	11843.60	$t_{1/2}(rac)_T$ (min)	11843.60



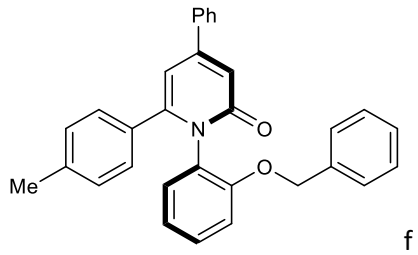
Equation	y = a + b*x
Plot	ln(k\-(e)/T)
Weight	No Weighting
Intercept	23.8276
Slope	-14731.24258
Residual Sum of Squares	0.01485
Pearson's r	-0.99764
R-Square(COD)	0.99529
Adj. R-Square	0.99058

$\Delta H_{ent}^{\ddagger} = 122.5$ kJ mol^{-1}

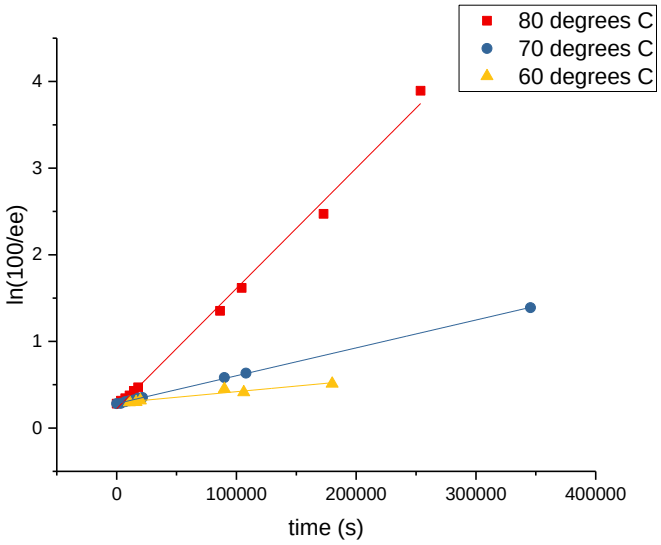
$\Delta S_{ent}^{\ddagger} = +0.6$ J K^{-1} mol^{-1}

Extrapolated half-life of racemization at 20°C: 11.5 y

4d

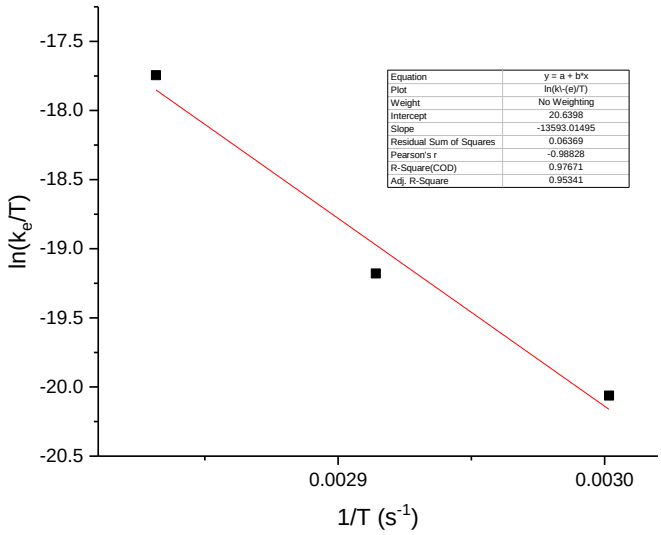


f



Equation	y = a + b*x		
Plot	ln(100/ee)	ln(100/ee)	ln(100/ee)
Weight		No Weighting	
Intercept	0.22285	0.28198	0.29059
Slope	1.38817E-5	3.21633E-6	1.29122E-6
Residual Sum of Squares	0.05757	2.68552E-4	0.00249
Pearson's r	0.99782	0.99987	0.96873
R-Square(COD)	0.99564	0.99975	0.93845
Adj. R-Square	0.9951	0.99971	0.92306

	T (K)		
	333	**343**	**353**
k_{rac} (s^{-1})	1.291E-06	3.216E-06	1.388E-05
k_{ent} (s^{-1})	6.456E-07	1.608E-06	6.941E-06
$\Delta G_T^{\ddagger}$ (kJ mol^{-1})	121.33	122.45	121.82
$t_{1/2}(rac)_T$ (min)	8946.93	3591.81	832.21



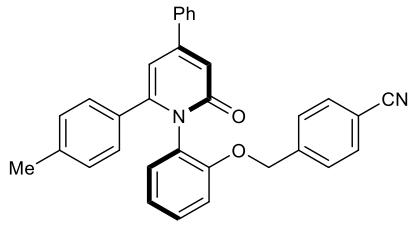
Equation	y = a + b*x
Plot	ln(k-(e)/T)
Weight	No Weighting
Intercept	20.6398
Slope	-13593.01495
Residual Sum of Squares	0.06369
Pearson's r	-0.98828
R-Square(COD)	0.97671
Adj. R-Square	0.95341

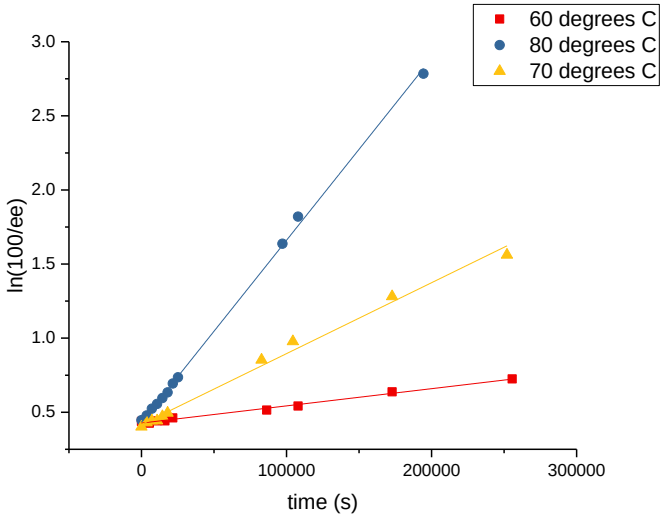
$\Delta H_{ent}^{\ddagger} = 113.0$ kJ mol^{-1}

$\Delta S_{ent}^{\ddagger} = -25.9$ J K^{-1} mol^{-1}

Extrapolated half-life of racemization at 20°C: 5.7 y

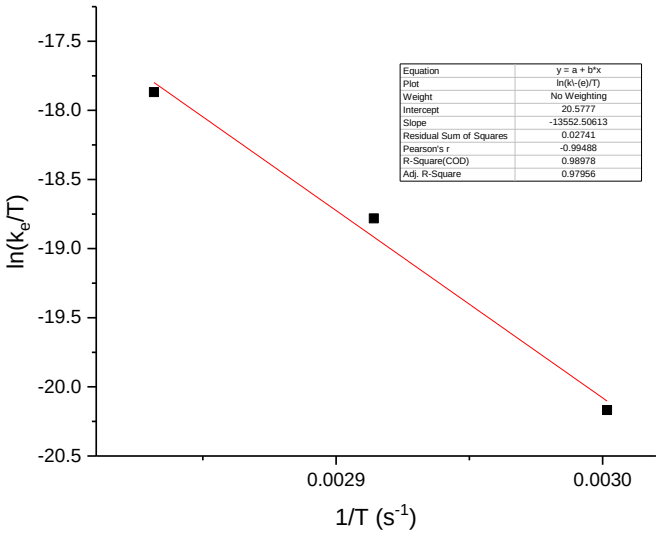
4e





Equation	y = a + b*x		
Plot	ln(100/ee)	ln(100/ee)	ln(100/ee)
Weight		No Weighting	
Intercept	0.42686	0.43287	0.41714
Slope	1.16124E-6	1.22767E-5	4.7849E-6
Residual Sum of Squares	6.6832E-4	0.00607	0.01187
Pearson's r	0.99617	0.99947	0.99618
R-Square(COD)	0.99235	0.99894	0.99238
Adj. R-Square	0.99126	0.99882	0.99142

	T (K)		
	333	**343**	**353**
k_{rac} (s^{-1})	1.161E-06	4.785E-06	1.228E-05
k_{ent} (s^{-1})	5.806E-07	2.392E-06	6.138E-06
$\Delta G_T^{\ddagger}$ (kJ mol^{-1})	121.62	121.32	122.18
$t_{1/2}(rac)_T$ (min)	9948.38	2414.36	941.01



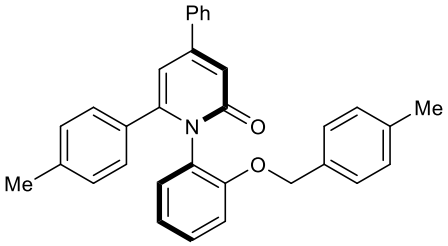
Equation	y = a + b*x
Plot	ln(k\-(e)/T)
Weight	No Weighting
Intercept	20.5777
Slope	-13552.50613
Residual Sum of Squares	0.02741
Pearson's r	-0.99488
R-Square(COD)	0.98978
Adj. R-Square	0.97956

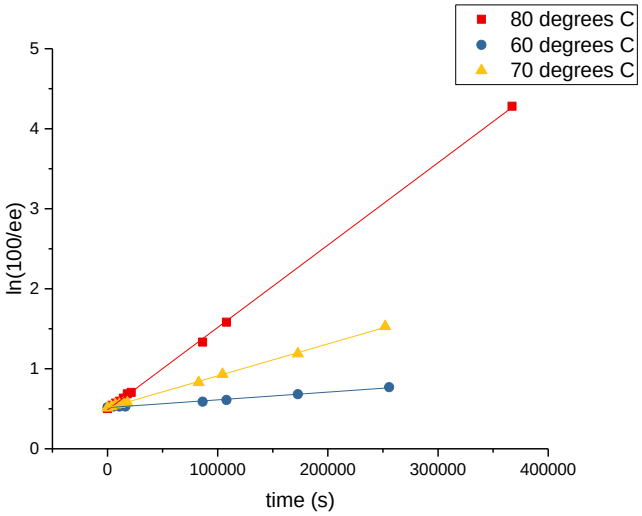
$\Delta H_{ent}^{\ddagger} = 112.7$ kJ mol^{-1}

$\Delta S_{ent}^{\ddagger} = -26.5$ J K^{-1} mol^{-1}

Extrapolated half-life of racemization at 20°C: 5.3 y

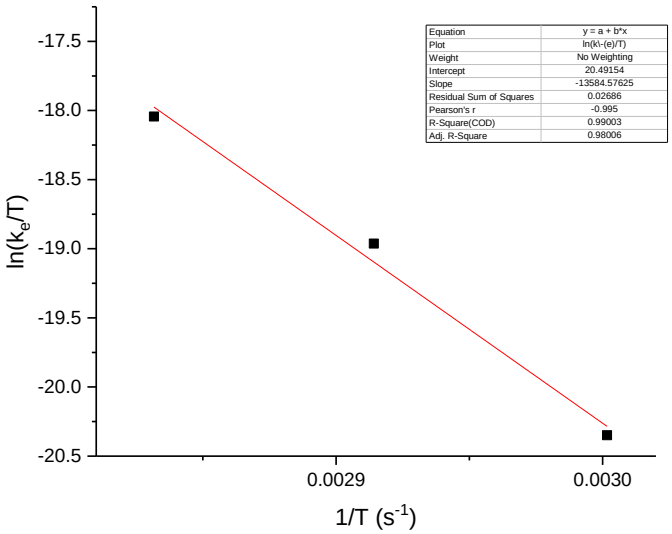
4f





Equation	y = a + b*x		
Plot	ln(100/ee)	ln(100/ee)	ln(100/ee)
Weight		No Weighting	
Intercept	0.4873	0.51488	0.51182
Slope	1.02927E-5	9.68233E-7	3.99273E-6
Residual Sum of Squares	0.00329	3.03691E-4	6.12457E-4
Pearson's r	0.99986	0.99736	0.99972
R-Square(COD)	0.99973	0.99472	0.99943
Adj. R-Square	0.9997	0.99384	0.99936

	T (K)		
	333	**343**	**353**
k_{rac} (s^{-1})	9.682E-07	3.993E-06	1.029E-05
k_{ent} (s^{-1})	4.841E-07	1.996E-06	5.146E-06
$\Delta G_T^{\ddagger}$ (kJ mol^{-1})	122.13	121.84	122.69
$t_{1/2}(rac)_T$ (min)	11931.48	2893.37	1122.39



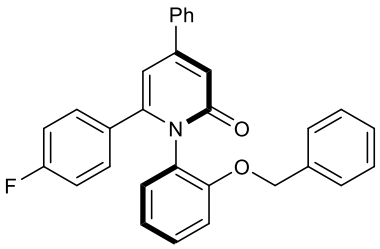
Equation	y = a + b*x
Plot	ln(k\-(e)/T)
Weight	No Weighting
Intercept	20.49154
Slope	-13584.57625
Residual Sum of Squares	0.02686
Pearson's r	-0.995
R-Square(COD)	0.99003
Adj. R-Square	0.98006

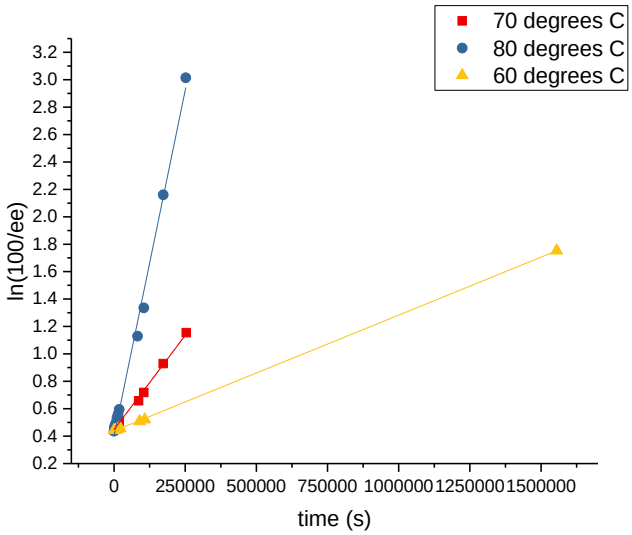
$\Delta H_{ent}^{\ddagger} = 112.9$ kJ mol^{-1}

$\Delta S_{ent}^{\ddagger} = -27.2$ J K^{-1} mol^{-1}

Extrapolated half-life of racemization at 20°C: 6.5 y

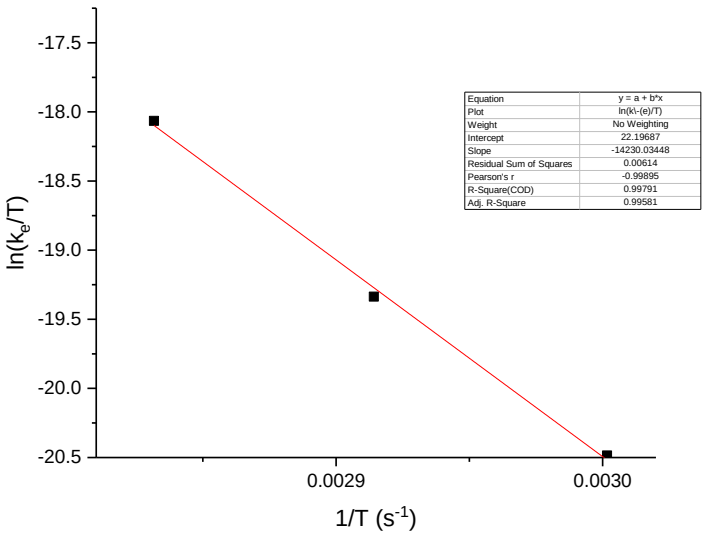
4g





Equation	y = a + b*x		
Plot	ln(100/ee)	ln(100/ee)	ln(100/ee)
Weight		No Weighting	
Intercept	0.44743	0.40233	0.43808
Slope	2.74858E-6	1.00819E-5	8.44969E-7
Residual Sum of Squares	0.00133	0.03525	1.56816E-4
Pearson's r	0.99872	0.99744	0.99995
R-Square(COD)	0.99744	0.99489	0.99989
Adj. R-Square	0.99712	0.99425	0.99987

	T (K)		
	333	**343**	**353**
k_{rac} (s^{-1})	8.450E-07	2.749E-06	1.008E-05
k_{ent} (s^{-1})	4.225E-07	1.374E-06	5.041E-06
$\Delta G_T^{\ddagger}$ (kJ mol^{-1})	122.50	122.90	122.70
$t_{1/2}(rac)_T$ (min)	13672.04	4203.06	1145.86



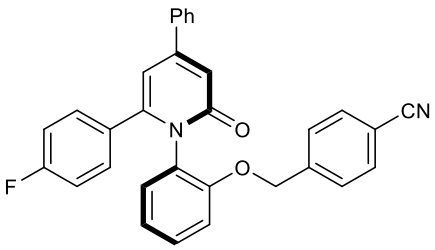
Equation	y = a + b*x
Plot	ln(k\-(e)/T)
Weight	No Weighting
Intercept	22.19687
Slope	-14230.03448
Residual Sum of Squares	0.00614
Pearson's r	-0.99895
R-Square(COD)	0.99791
Adj. R-Square	0.99581

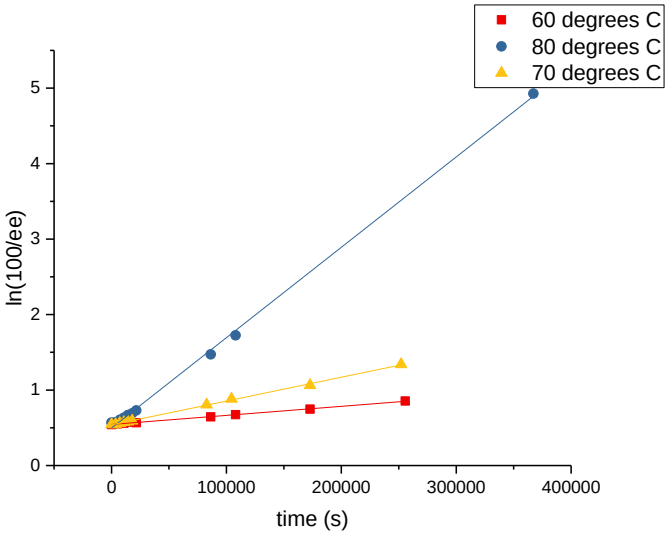
$\Delta H_{ent}^{\ddagger} = 118.3$ kJ mol^{-1}

$\Delta S_{ent}^{\ddagger} = -13.0$ J K^{-1} mol^{-1}

Extrapolated half-life of racemization at 20°C: 10.6 y

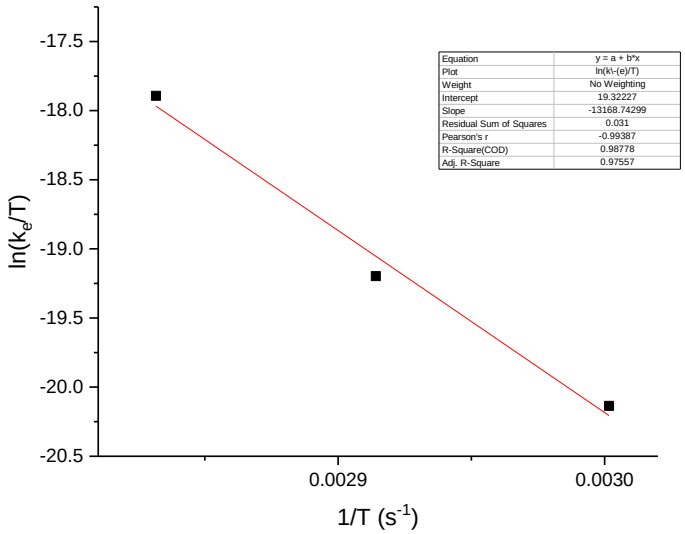
4h





Equation	y = a + b*x		
Plot	ln(100/ee)	ln(100/ee)	ln(100/ee)
Weight		No Weighting	
Intercept	0.54455	0.49695	0.5388
Slope	1.19874E-6	1.19706E-5	3.15503E-6
Residual Sum of Squares	1.83607E-4	0.01693	7.72388E-4
Pearson's r	0.99901	0.99948	0.99943
R-Square(COD)	0.99802	0.99897	0.99885
Adj. R-Square	0.99773	0.99884	0.99871

	T (K)		
	333	**343**	**353**
k_{rac} (s^{-1})	1.199E-06	3.155E-06	1.197E-05
k_{ent} (s^{-1})	5.994E-07	1.578E-06	5.985E-06
$\Delta G_T^{\ddagger}$ (kJ mol^{-1})	121.53	122.51	122.25
$t_{1/2}(rac)_T$ (min)	9637.16	3661.60	965.07



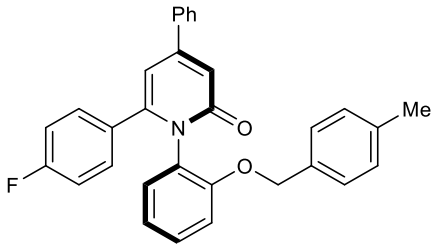
Equation	y = a + b*x
Plot	ln(k\-(e)/T)
Weight	No Weighting
Intercept	19.32227
Slope	-13168.74299
Residual Sum of Squares	0.031
Pearson's r	-0.99387
R-Square(COD)	0.98778
Adj. R-Square	0.97557

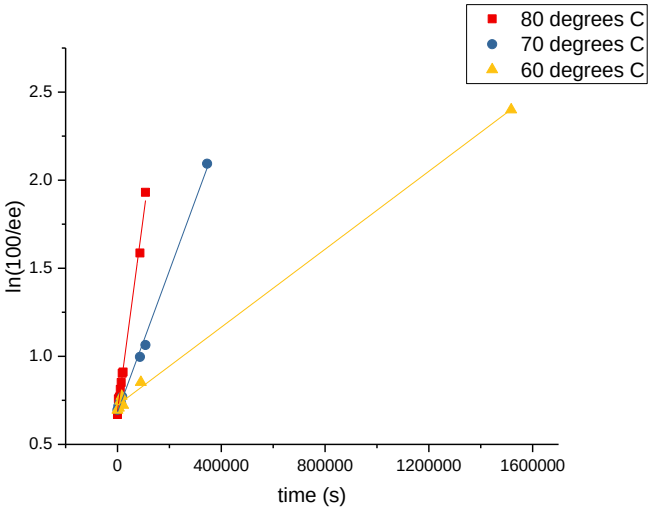
$\Delta H_{ent}^{\ddagger} = 109.5$ kJ mol^{-1}

$\Delta S_{ent}^{\ddagger} = -36.9$ J K^{-1} mol^{-1}

Extrapolated half-life of racemization at 20°C: 5.0 y

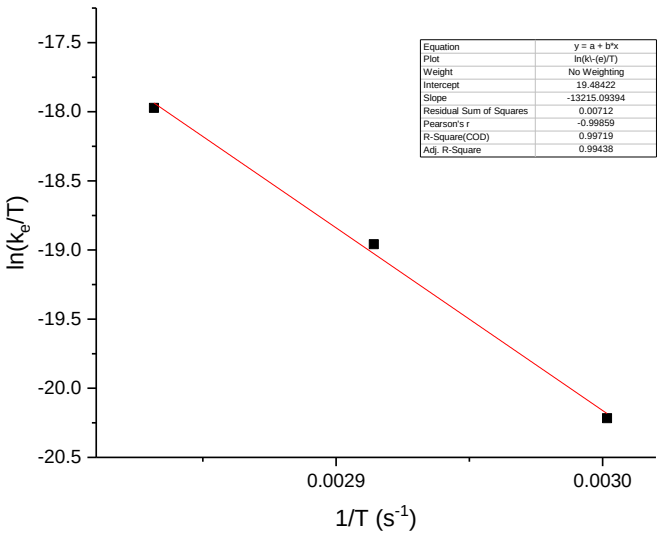
4i





Equation	y = a + b*x		
Plot	ln(100/ee)	ln(100/ee)	ln(100/ee)
Weight	No Weighting		
Intercept	0.68945	0.68403	0.7229
Slope	1.10648E-5	4.00925E-6	1.10622E-6
Residual Sum of Squares	0.00747	0.00574	0.004
Pearson's r	0.99751	0.99822	0.99915
R-Square(COD)	0.99503	0.99644	0.9983
Adj. R-Square	0.99432	0.99593	0.99796

	T (K)		
	333	**343**	**353**
k_{rac} (s^{-1})	1.106E-06	4.009E-06	1.106E-05
k_{ent} (s^{-1})	5.531E-07	2.005E-06	5.532E-06
$\Delta G_T^{\ddagger}$ (kJ mol^{-1})	121.76	121.83	122.48
$t_{1/2}(rac)_T$ (min)	10443.18	2881.45	1044.07



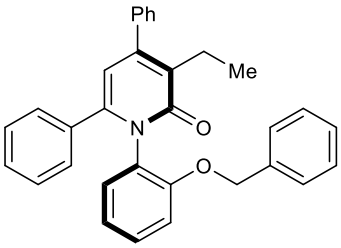
Equation	y = a + b*x
Plot	ln(kl-(e)/T)
Weight	No Weighting
Intercept	19.48422
Slope	-13215.09394
Residual Sum of Squares	0.00712
Pearson's r	-0.99859
R-Square(COD)	0.99719
Adj. R-Square	0.99438

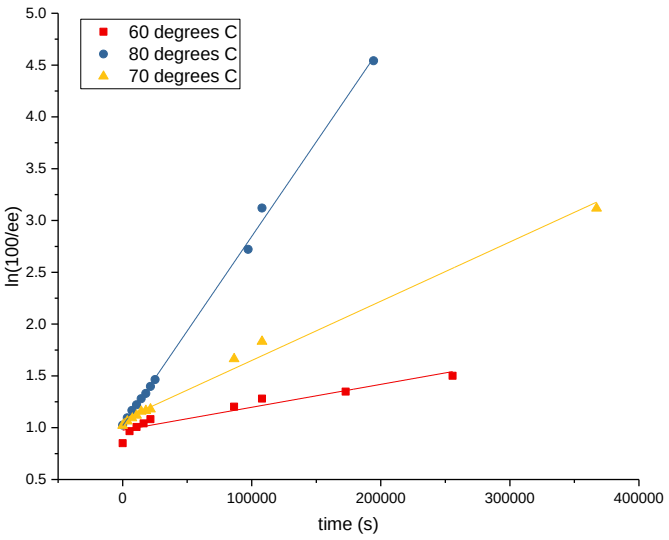
$\Delta H_{ent}^{\ddagger} = 109.9$ kJ mol^{-1}

$\Delta S_{ent}^{\ddagger} = -35.6$ J K^{-1} mol^{-1}

Extrapolated half-life of racemization at 20°C: 5.0 y

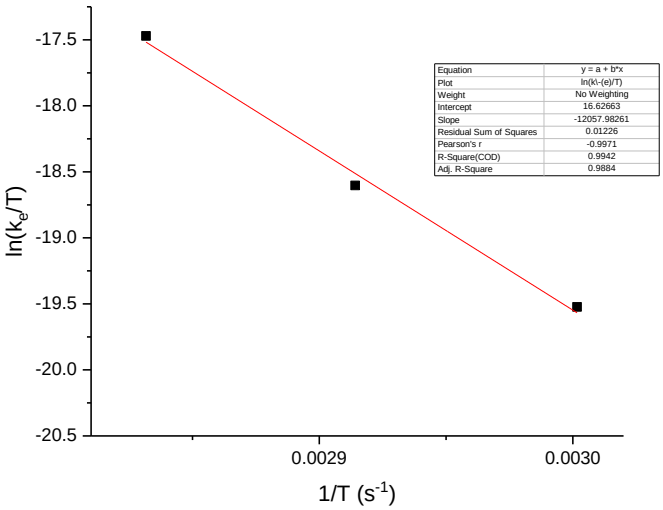
4j





Equation	y = a + b*x		
Plot	ln(100/ee)	ln(100/ee)	ln(100/ee)
Weight		No Weighting	
Intercept	0.97592	1.02045	1.07696
Slope	2.21257E-6	1.82572E-5	5.71927E-6
Residual Sum of Squares	0.02814	0.02391	0.03735
Pearsons r	0.9581	0.99806	0.99525
R-Square(COD)	0.91795	0.99811	0.99013
Adj. R-Square	0.90622	0.9979	0.98889

	T (K)		
	333	**343**	**353**
k_{rac} (s^{-1})	2.213E-06	5.719E-06	1.826E-05
k_{ent} (s^{-1})	1.106E-06	2.860E-06	9.129E-06
$\Delta G_T^{\ddagger}$ (kJ mol^{-1})	119.84	120.81	121.01
$t_{1/2}(rac)_T$ (min)	5221.28	2019.92	632.76



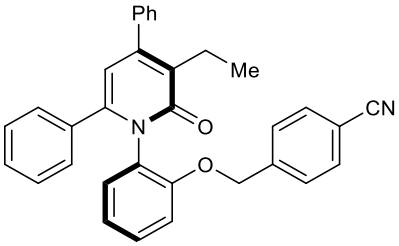
Equation	y = a + b*x
Plot	ln(k\-(e)/T)
Weight	No Weighting
Intercept	16.62663
Slope	-12057.98261
Residual Sum of Squares	0.01226
Pearson's r	-0.9971
R-Square(COD)	0.9942
Adj. R-Square	0.9884

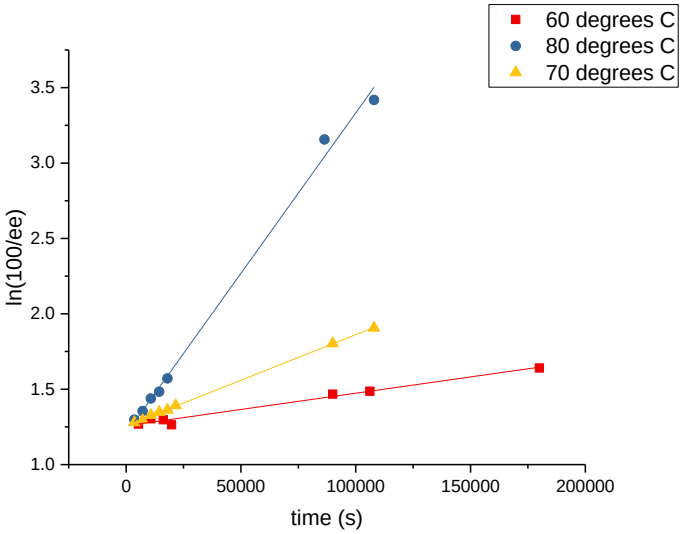
$\Delta H_{ent}^{\ddagger} = 100.3$ kJ mol^{-1}

$\Delta S_{ent}^{\ddagger} = -59.3$ J K^{-1} mol^{-1}

Extrapolated half-life of racemization at 20°C: 1.7 y

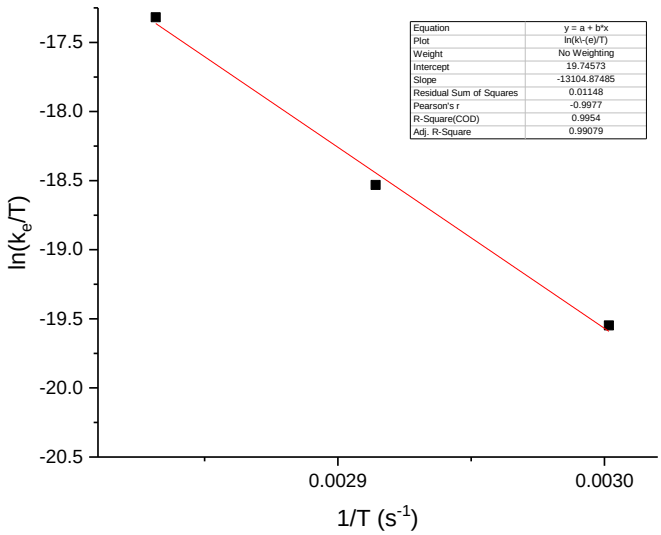
4k





Equation	y = a + b*x		
Plot	ln(100/ee)	ln(100/ee)	ln(100/ee)
Weight		No Weighting	
Intercept	1.25769	1.20502	1.25774
Slope	2.15851E-6	2.1275E-5	6.03728E-6
Residual Sum of Squares	0.00214	0.02115	1.27596E-4
Pearson's r	0.99141	0.99789	0.99985
R-Square(COD)	0.9829	0.99578	0.9997
Adj. R-Square	0.97948	0.99494	0.99965

	T (K)		
	333	**343**	**353**
k_{rac} (s^{-1})	2.159E-06	6.143E-06	2.128E-05
k_{ent} (s^{-1})	1.079E-06	3.071E-06	1.064E-05
$\Delta G_T^{\ddagger}$ (kJ mol^{-1})	119.91	120.61	120.56
$t_{1/2}(rac)_T$ (min)	5352.05	1880.65	543.01



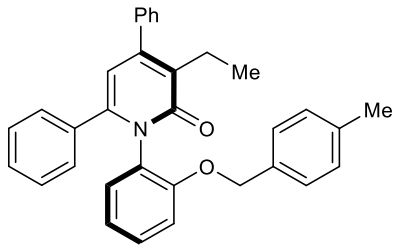
Equation	y = a + b*x
Plot	ln(k-(e)/T)
Weight	No Weighting
Intercept	19.74573
Slope	-13104.87485
Residual Sum of Squares	0.01148
Pearson's r	-0.9977
R-Square(COD)	0.9954
Adj. R-Square	0.99079

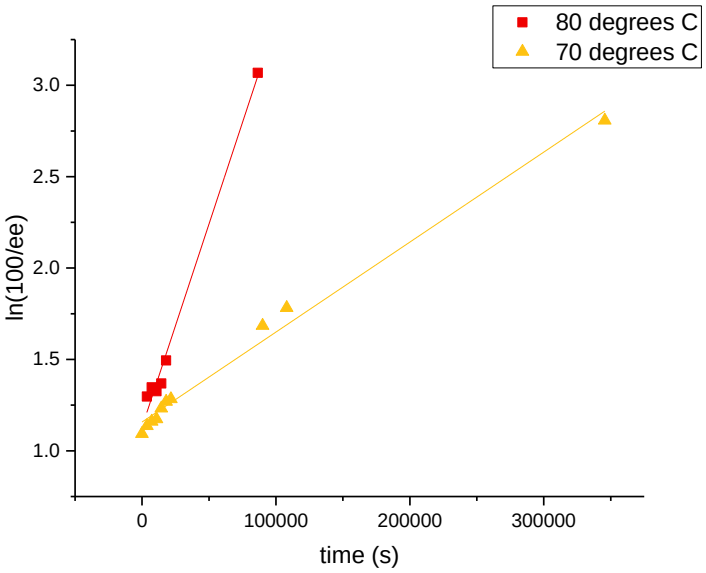
$\Delta H_{ent}^{\ddagger} = 109.0$ kJ mol^{-1}

$\Delta S_{ent}^{\ddagger} = -33.4$ J K^{-1} mol^{-1}

Extrapolated half-life of racemization at 20°C: 2.6 y

4l

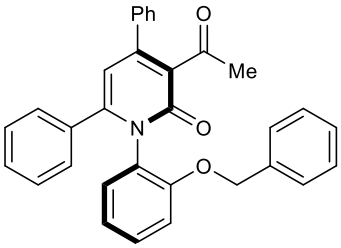


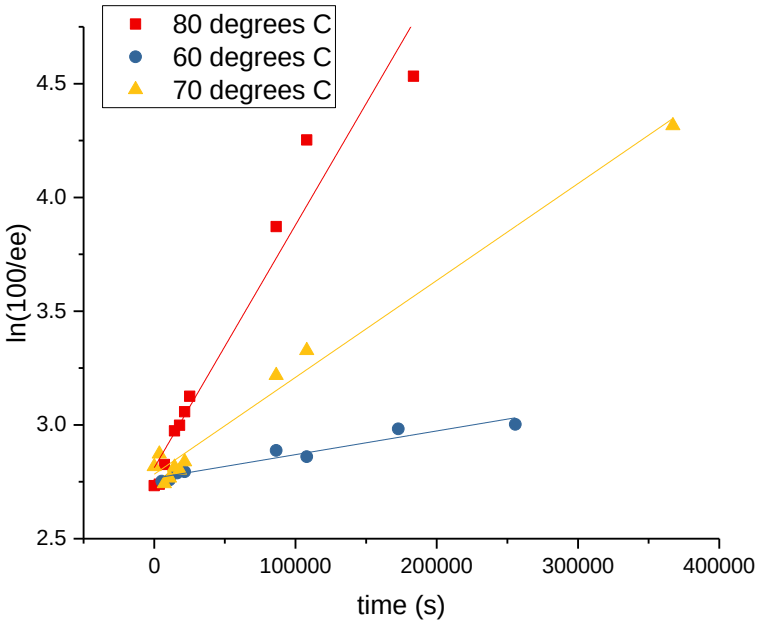


Equation	y = a + b*x		
Plot	ln(100/ee)	ln(100/ee)	ln(100/ee)
Weight	No Weighting		
Intercept	1.13025	--	1.15755
Slope	2.22156E-5	--	4.92286E-6
Residual Sum of Squares	0.02095	--	0.02741
Pearson's r	0.99569	--	0.9945
R-Square(COD)	0.9914	--	0.98904
Adj. R-Square	0.98925	--	0.98767

	T (K)		
	333	**343**	**353**
k_{rac} (s^{-1})	*not measured*	4.923E-06	2.222E-05
k_{ent} (s^{-1})	-	2.461E-06	1.111E-05
$\Delta G_T^{\ddagger}$ (kJ mol^{-1})	-	121.24	120.44
$t_{1/2}(rac)_T$ (min)	-	2346.70	520.02

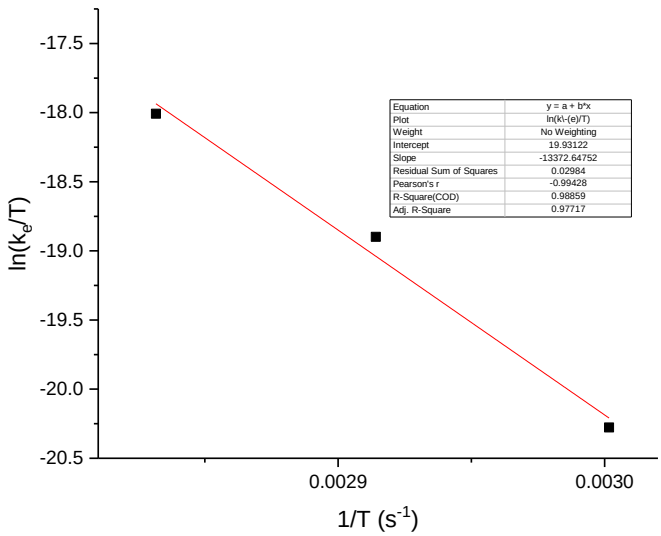
4m





Equation	y = a + b*x		
Plot	ln(100/ee)	ln(100/ee)	ln(100/ee)
Weight		No Weighting	
Intercept	2.81188	2.76545	2.78334
Slope	1.06692E-5	1.04072E-6	4.25857E-6
Residual Sum of Squares	0.18368	0.00434	0.03506
Pearson's r	0.97616	0.96709	0.99192
R-Square(COD)	0.95288	0.93526	0.98391
Adj. R-Square	0.94699	0.92447	0.98212

	T (K)		
	333	**343**	**353**
k_{rac} (s^{-1})	1.041E-06	4.259E-06	1.067E-05
k_{ent} (s^{-1})	5.204E-07	2.129E-06	5.335E-06
$\Delta G_T^{\ddagger}$ (kJ mol^{-1})	121.93	121.65	122.59
$t_{1/2}(rac)_T$ (min)	11100.44	2712.75	1082.79



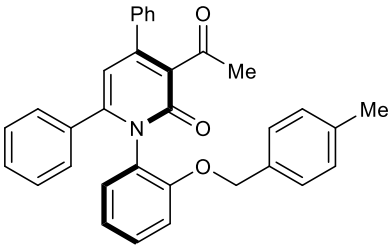
Equation	y = a + b*x
Plot	ln(k\-(e)/T)
Weight	No Weighting
Intercept	19.93122
Slope	-13372.64752
Residual Sum of Squares	0.02984
Pearson's r	-0.99428
R-Square(COD)	0.98859
Adj. R-Square	0.97717

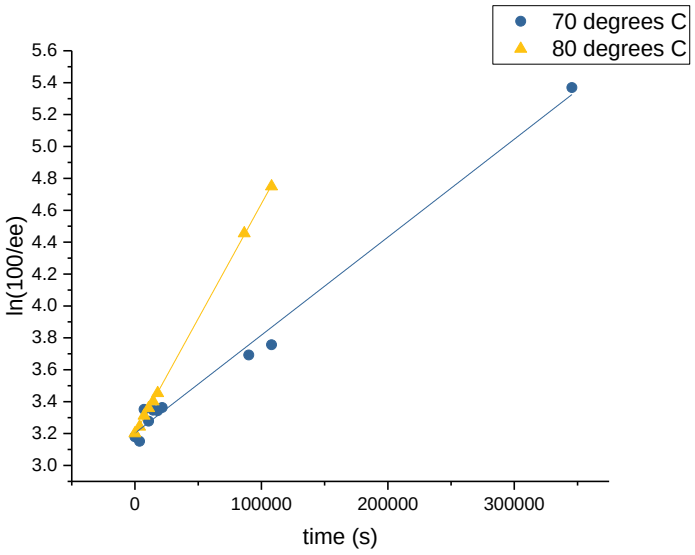
$\Delta H_{ent}^{\ddagger} = 111.2$ kJ mol^{-1}

$\Delta S_{ent}^{\ddagger} = -31.8$ J K^{-1} mol^{-1}

Extrapolated half-life of racemization at 20°C: 5.5 y

4o

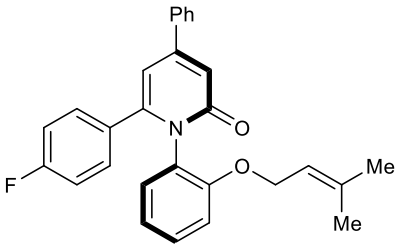


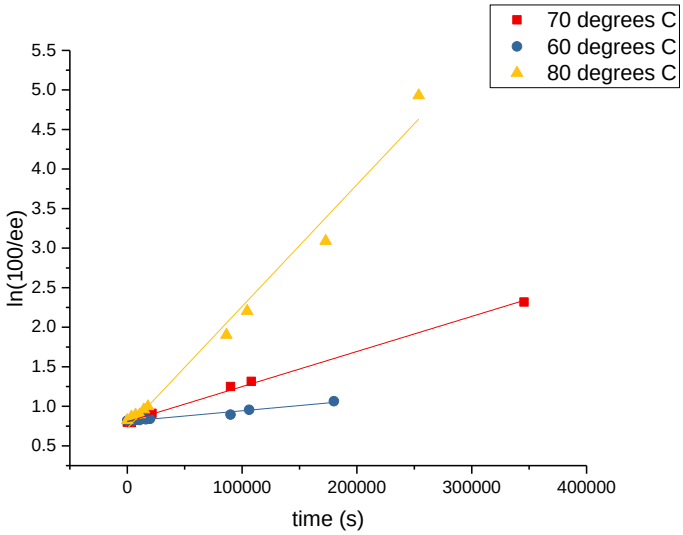


Equation		
Plot	ln(100/ee)	ln(100/ee)
Weight		
Intercept	3.20285	3.19908
Slope	6.14279E-6	1.44216E-5
Residual Sum of Squares	0.03945	3.71975E-4
Pearson's r	0.99492	0.99993
R-Square(COD)	0.98986	0.99985
Adj. R-Square	0.98859	0.99983

	T (K)		
	333	**343**	**353**
k_{rac} (s^{-1})	*not measured*	6.143E-06	1.442E-05
k_{ent} (s^{-1})	-	3.071E-06	7.211E-06
$\Delta G_T^{\ddagger}$ (kJ mol^{-1})	-	120.61	121.70
$t_{1/2}(rac)_T$ (min)	-	1880.65	801.05

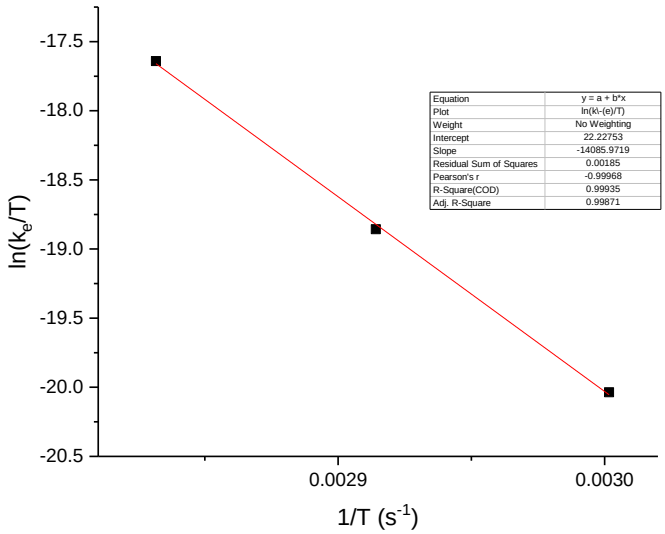
4p





Equation	y = a + b*x		
Plot	ln(100/ee)	ln(100/ee)	ln(100/ee)
Weight	No Weighting		
Intercept	0.80576	0.81113	0.72264
Slope	4.43721E-6	1.32408E-6	1.54021E-5
Residual Sum of Squares	0.00534	0.00159	0.23766
Pearson's r	0.99867	0.98503	0.99274
R-Square(COD)	0.99735	0.97029	0.98554
Adj. R-Square	0.99702	0.96534	0.98373

	T (K)		
	333	**343**	**353**
k_{rac} (s^{-1})	1.324E-06	4.437E-06	1.540E-05
k_{ent} (s^{-1})	6.620E-07	2.219E-06	7.701E-06
$\Delta G_T^{\ddagger}$ (kJ mol^{-1})	121.26	121.54	121.51
$t_{1/2}(rac)_T$ (min)	8724.89	2603.54	750.06



Equation	y = a + b*x
Plot	ln(k-(e)/T)
Weight	No Weighting
Intercept	22.22753
Slope	-14085.9719
Residual Sum of Squares	0.00185
Pearson's r	-0.99968
R-Square(COD)	0.99935
Adj. R-Square	0.99871

$\Delta H_{ent}^{\ddagger} = 117.1$ kJ mol^{-1}

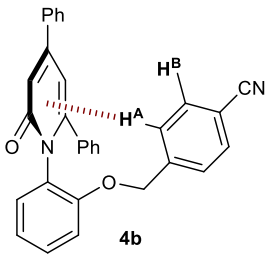
$\Delta S_{ent}^{\ddagger} = -12.7$ J K^{-1} mol^{-1}

j

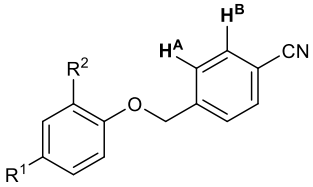
5 NMR Analysis of CH···π Interaction

Singe crystal data and quantum chemical calculations both indicate the presence of a CH···π interaction present in the ground state of the benzylated products **4**. To demonstrate that this effect is present in solution as well as solid phase, the chemical shift values of $\mathbf{H^A}$ and $\mathbf{H^B}$ in 4-cyanobenzyl compound **4b** were compared to literature values for structurally related compounds (see manuscript, figure 2B). In addition to the comparison with *O*-(*para*-cyanobenzyl)phenol provided in the manuscript, below is given comparison with a large number of literature compounds.

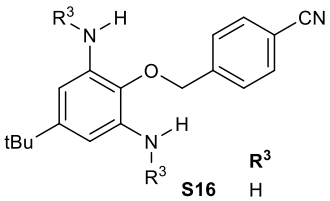
Table S2. Comparison of ^{1}H chemical shift values of **4b** with structurally similar compounds previously reported. All ^{1}H signals are reported in $CDCl_3$.



4b



	$\mathbf{R^1}$	$\mathbf{R^2}$
5	H	H
S9	Cl	H
S10	OMe	H
S11	CF_3	H
S12	OCF_3	H
S13	H	Cl
S14	H	OMe
S15	H	OCF_3



	$\mathbf{R^3}$
S16	H
S17	Boc
S18	Me

Compound	H^A (ppm)	H^B (ppm)	Reference
4b	7.42	7.60	
5	7.56	7.69	[19]
S9	7.53	7.68	[20]
S10	7.54	7.67	[20]
S11	7.54	7.69	[20]
S12	7.56	7.7	[20]
S13	7.60	7.68	[20]
S14	7.56	7.66	[20]
S15	7.55	7.69	[20]
S16	7.58	7.68	[21]
S17	7.54	-*	[21]
S18	7.55	7.69	[21]

* Co-incident with another ^{1}H signal.

6 Density Functional Theory Calculations

6.1 Methods

All optimisation, frequency, and single-point calculations were performed with *Gaussian 16*, rev C.01.[22] Structures were generated with CYLview.[23] All non-covalent interaction surfaces were calculated with and displayed using Jmol.[24] The B3LYP functional was used for all geometry optimisations with the 6-31G(d,p) basis set on all atoms. Grimme's DFT-D3[25] correction was included in the optimisation procedure as well as single point calculations. All optimised structures were confirmed as minima by the absence of imaginary frequencies while transition states were characterised by the presence of a single imaginary frequency. All transition states were further analysed via intrinsic reaction coordinate calculations. Further refinements to the electronic energies were made through single point calculations on the optimised geometries using the B3LYP functional with the 6-311+G(d,p) basis set for all atoms. Grimme's DFT-D3 correction and further corrections for bulk solvation through a polarisable continuum model (CPCM) were also incorporated. Free energies were determined from thermochemical corrections of the geometries applied to electronic energies.

6.2 Summary of Results

Three starting materials, and their corresponding products, were chosen for study each with a different (R^3) substituent at the pyridine 3-position: **3a**, **3d**, and **3e** leading to **4a**, **4j**, and **4m** respectively (addition of a benzyl group). Barriers were calculated for starting material, **3**, the deprotonated intermediate, **3(int)**, and the reaction products, **4**. Calculated rotational barriers are summarised in Table S3, showing good agreement with experimentally determined barriers. Computational barriers for **3** and **3(int)** were calculated in acetonitrile but for **4** were calculated in DMSO to reflect experimental procedures (see section 4 above).

Table S3: Calculated *vs*. experimental barriers for **3**, **3int**, and **4**. Calculated at B3LYP-D3-CPCM/6-311+G(d,p)//B3LYP-D3/6-31G(d,p).

	dG_{DFT} (kJ mol^{-1})	dG_{Exp} (kJ mol^{-1})
3a	95.4	96.6
3d	97.1	98.7
3e	97.5	102.9
3(int)a	123.0	-
3(int)d	125.9	-
3(int)e	123.4	-
4a	123.0	121.3
4j	124.7	121.0
4m	121.3	122.6

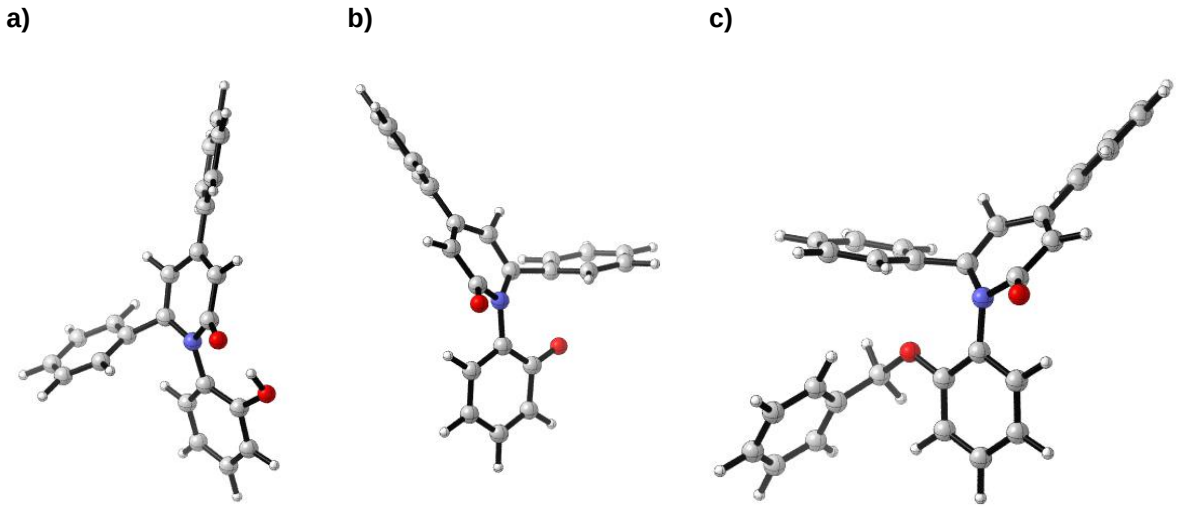


Figure S1. Representative enantiomerisation transition state geometries for: a) **3a**; b) **3(int)a**; c) **4a**. Calculated at B3LYP-D3-CPCM$_{DMSO}$/6-311+G(d,p)//B3LYP-D3/6-31G(d,p).

For starting material **3a**, the lowest barrier transition state was found with the phenol and carbonyl groups *syn* to allow for the favourable OH···O hydrogen bond, Figure S1a. The alternative transition state, with phenol and carbonyl groups *anti* was found to be *ca*. 10 kcal mol^{-1} higher in energy.

A non-covalent interaction descriptor based on electron density, developed by the Johnson and Contreras-Garcia groups,[26] allows a qualitative visual analysis of NCIs and is shown here to be a useful tool. The coloration of the NCI surface allows identification and characterization of attractive and repulsive interactions; a strong attractive interaction is in blue, van der Waals and dispersion interactions are in green, and destabilizing steric interactions are in red. In the enantiomerisation transition state of **3a**, the strong hydrogen bond between the phenol and 2-pyridone oxygens is observed as a deep blue surface, Figure S2a. The stabilising OH···O hydrogen bond is also observed in the minimum, S2b. Rotating the phenol group out of reach of an OH···O hydrogen bond and reoptimizing the structure results in a large 6 kJ mol^{-1} energy penalty

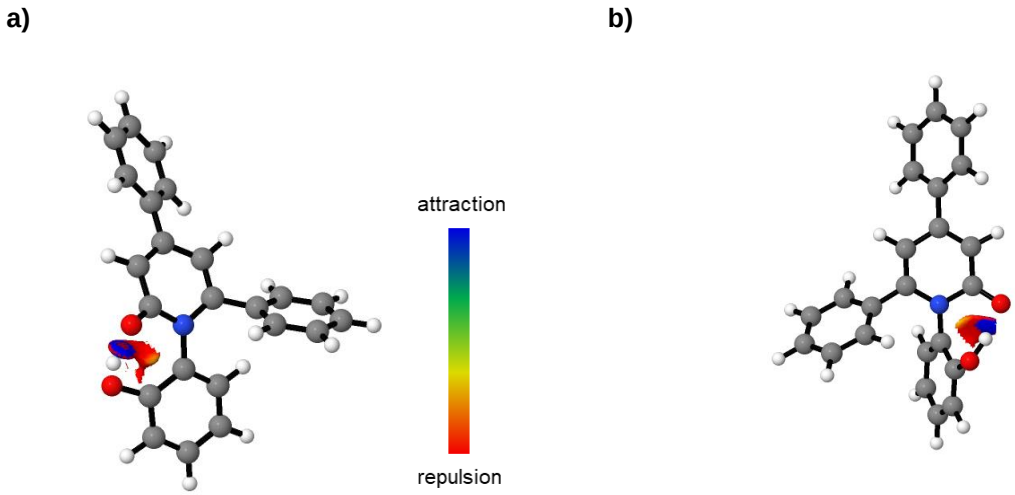


Figure S2. Non-covalent interaction surface of the starting material enantiomerisation transition state of **3a** showing a strong hydrogen bond in deep blue. The colour spectrum ranges from blue (strongly attractive) to green (weekly attractive) to yellow (mildly repulsive) to red (strongly repulsive), geometries optimised at B3LYP-D3/6-31G(d,p).

Without the proton to participate in the hydrogen bond, the enantiomerisation transition state barrier for **3(int)a** is significantly higher, by 28 kJ mol^{-1} (Table S3) with the lowest energy transition state found with the phenoxide and carbonyl groups *anti*, Figure S1b. The *syn* transition state could not be found.

Addition of the benzyl group raises the transition state barrier for **4a** similarly to that of the deprotonated intermediate, Table S3. The lowest barrier to enantiomerisation was found with the benzyl ether and carbonyl group *anti*, Figure S1c.

The crystal structures and NMR shifts of the products, **4**, showed a CH-π non-covalent interaction between the *ortho*-CH of the benzyl group and the π-system of the 2-pyridone which was replicated in the solution phase *via* DFT. A non-covalent interaction surface shows this CH-π interaction as an area of light blue, Figure S3. The non-planar geometry around the 2-pyridone nitrogen, and tilt of this group, is also replicated.

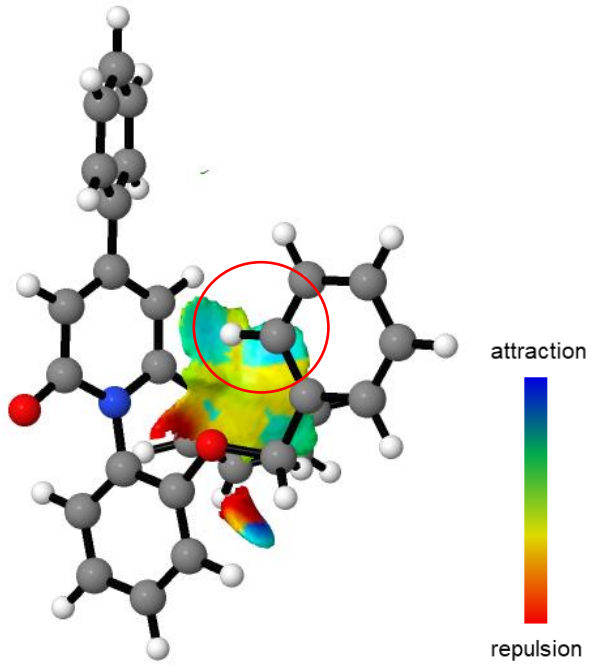


Figure S3. Non-covalent interaction surface of the minima of 4a showing a stabilising CH-π interaction in light blue (circled). The colour spectrum ranges from blue (strongly attractive) to green (weekly attractive) to yellow (mildly repulsive) to red (strongly repulsive), geometries optimised at B3LYP-D3/6-31G(d,p).

In all structures examined, the minima that included the CH-π interaction were lower in energy than those without, Table S4, in which the benzyl group lies face on to the 2-pyridone.

Table S4. Relative energies of product minima with and without CH-π interaction as identified in the crystal structures of products 4. B3LYP-D3-CPCM$_{DMSO}$/6-311+G(d,p)//B3LYP-D3/6-31G(d,p).

	With CH-π interaction (kJ mol^{-1})	**Without CH-π interaction (kJ mol^{-1})**
4a	0.0	+7.1
4j	0.0	+4.2
4m	0.0	+2.1

6.3 Cartesian Coordinates

3a – Minimum

C	4.67967	-1.22837	1.09847
C	4.20245	-1.88348	-0.03237
C	2.848	-1.81327	-0.38189
C	1.98287	-1.04515	0.41937
C	2.46119	-0.42349	1.5761
C	3.8067	-0.50727	1.91848
H	5.73348	-1.29568	1.35219
H	4.85516	-2.47125	-0.66876
H	1.76992	0.14693	2.18779
H	4.16939	-0.01467	2.81448
C	0.10808	0.46504	-0.05847
C	-0.25331	-1.95559	-0.06958
C	-1.24546	0.68217	-0.05975
C	-1.66084	-1.68486	-0.01702
C	-2.16557	-0.4066	0.00469
H	-1.60486	1.69103	-0.21407
H	-2.29805	-2.55978	0.01945
C	1.05447	1.58649	-0.27623
C	2.06283	1.48731	-1.24902
C	0.90711	2.78289	0.43791
C	2.90656	2.56638	-1.49383
H	2.17471	0.56615	-1.81149
C	1.75678	3.86119	0.19149
H	0.13688	2.85608	1.19988
C	2.75916	3.75489	-0.77306
H	3.68079	2.4801	-2.25005
H	1.63943	4.78	0.75821
H	3.42347	4.59257	-0.963
C	-3.62346	-0.14453	0.05103
C	-4.12475	0.96748	0.74808
C	-4.53404	-0.99869	-0.59288
C	-5.49535	1.2118	0.80868
H	-3.4374	1.62711	1.26894
C	-5.90381	-0.75126	-0.5354
H	-4.16256	-1.84349	-1.16414
C	-6.38985	0.35372	0.16658
H	-5.86507	2.07026	1.36153
H	-6.59216	-1.41748	-1.04679
H	-7.45764	0.54607	0.21026
O	2.41968	-2.44818	-1.49876
H	1.55557	-2.86152	-1.2472
O	0.23449	-3.09478	-0.17486
N	0.60131	-0.82991	0.04191

3a – Transition State

C	4.65174	-1.66841	1.30736
C	3.87256	-2.6547	0.73423
C	2.58312	-2.40997	0.22045
C	2.0861	-1.06694	0.1992
C	2.88581	-0.09917	0.84471
C	4.13503	-0.37494	1.38026
H	5.63283	-1.90533	1.7074
H	4.20484	-3.68597	0.68469
H	2.52771	0.90728	0.96018
H	4.69015	0.42499	1.85929
C	0.19976	0.58855	-0.06205
C	-0.08933	-1.60706	-0.97724
C	-1.15512	0.72688	0.06096
C	-1.49999	-1.48846	-0.72156
C	-2.0499	-0.3653	-0.16458
H	-1.5552	1.7267	0.17248
H	-2.08959	-2.34446	-1.02605
C	1.00195	1.82987	-0.22434
C	1.85414	1.96211	-1.3337
C	0.83638	2.91818	0.64416
C	2.52804	3.15816	-1.56121
H	1.98582	1.11984	-2.00529
C	1.51588	4.11407	0.41299
H	0.19283	2.8132	1.51226
C	2.36342	4.23781	-0.68875
H	3.1825	3.24928	-2.42279
H	1.38734	4.94624	1.09859
H	2.89451	5.16792	-0.86641
C	-3.497	-0.2195	0.09906
C	-3.94067	0.57313	1.17126
C	-4.45541	-0.86455	-0.70072
C	-5.30061	0.70636	1.4436
H	-3.21205	1.0643	1.80926
C	-5.81472	-0.72769	-0.42969
H	-4.13365	-1.45218	-1.55461
C	-6.24254	0.05656	0.64395
H	-5.62464	1.3141	2.28316
H	-6.54194	-1.22666	-1.06319
H	-7.30266	0.1631	0.85332
O	1.92146	-3.52738	-0.12812
H	1.30071	-3.31629	-0.87729
O	0.36877	-2.48662	-1.72771
N	0.76098	-0.68629	-0.29548

3(int)a – Intermediate Minimum

C	-4.76149	-1.7555	-0.20808
C	-4.13721	-1.52897	1.00783
C	-2.73795	-1.19417	1.11914
C	-2.06786	-1.12472	-0.16162
C	-2.69951	-1.36702	-1.37467
C	-4.06042	-1.67239	-1.42364
H	-5.8247	-1.99918	-0.22209
H	-4.69168	-1.58883	1.94187
H	-2.11034	-1.31196	-2.28849
H	-4.55598	-1.85345	-2.3733
C	-0.14255	0.40845	-0.10973
C	0.21343	-2.01474	-0.13541
C	1.21505	0.64246	-0.0718
C	1.63136	-1.72312	-0.06639
C	2.1306	-0.44598	-0.04388
H	1.56543	1.66634	-0.07255
H	2.2762	-2.59256	-0.00648
C	-1.06254	1.58147	-0.14651
C	-1.99519	1.81709	0.87507
C	-0.9239	2.51563	-1.18252
C	-2.77678	2.97079	0.84098
H	-2.08974	1.0839	1.6756
C	-1.71259	3.66716	-1.21115
H	-0.20154	2.32638	-1.97153
C	-2.64198	3.89758	-0.19665
H	-3.50094	3.1443	1.63272
H	-1.60198	4.37823	-2.02615
H	-3.25946	4.79257	-0.21521
C	3.59206	-0.19001	0.02249
C	4.10487	0.87202	0.78647
C	4.50173	-1.00183	-0.6758
C	5.47711	1.10931	0.8552
H	3.41838	1.49771	1.34833
C	5.87376	-0.76529	-0.60784
H	4.11938	-1.81166	-1.28935
C	6.36921	0.29222	0.15803
H	5.85069	1.93117	1.46053
H	6.55746	-1.40371	-1.16144
H	7.43865	0.47818	0.2098
O	-2.15822	-0.92555	2.21609
O	-0.24563	-3.15129	-0.19936
N	-0.64202	-0.86649	-0.15332

3(int)a – Intermediate Transition State

C	4.40566	-2.60492	-1.11183
C	4.3608	-1.23712	-0.96767
C	3.20769	-0.51813	-0.46745
C	2.09072	-1.35329	-0.06097
C	2.15884	-2.74566	-0.253
C	3.28508	-3.37935	-0.76125
H	5.30344	-3.08492	-1.50171
H	5.2013	-0.60612	-1.24574
H	1.3135	-3.36451	0.01428
H	3.28491	-4.4586	-0.88776
C	0.28434	0.44422	0.06136
C	-0.05454	-1.70334	1.13391
C	-1.08074	0.5821	-0.08829
C	-1.46184	-1.57692	0.83627
C	-1.9846	-0.47428	0.20802
H	-1.46735	1.57554	-0.27695
H	-2.07342	-2.40946	1.16516
C	1.03084	1.73423	0.11589
C	0.85374	2.70202	-0.88014
C	1.65994	2.10956	1.31728
C	1.33834	3.99934	-0.70613
H	0.35891	2.42071	-1.80502
C	2.1272	3.40445	1.49351
H	1.78699	1.36396	2.09494
C	1.97489	4.35986	0.48055
H	1.21429	4.72977	-1.5025
H	2.62063	3.67387	2.42435
H	2.34789	5.37153	0.61951
C	-3.42925	-0.32957	-0.07942
C	-3.86309	0.40832	-1.19567
C	-4.4084	-0.91899	0.74027
C	-5.21945	0.54494	-1.4853
H	-3.12089	0.8575	-1.84845
C	-5.76457	-0.78514	0.44957
H	-4.09743	-1.46571	1.62489
C	-6.17912	-0.05245	-0.6652
H	-5.52767	1.11454	-2.35831
H	-6.50166	-1.246	1.10231
H	-7.23698	0.05395	-0.88997
O	3.21713	0.74528	-0.42418
O	0.38841	-2.56664	1.89163
N	0.82204	-0.80207	0.40649

4a – Product Minimum

O	-0.21381	-2.69452	-2.53727

O	-1.59289	0.75879	-1.10729
N	-0.61045	-1.66135	-0.51115
C	-1.99866	-1.53576	-0.86171
C	-2.50808	-0.24923	-1.11442
C	0.25832	-2.17184	-1.53403
C	-0.13571	-1.08458	0.65515
C	1.66854	-1.98697	-1.27399
C	-1.10042	-0.60906	1.68002
C	2.14461	-1.38447	-0.13853
C	-3.87252	-0.08822	-1.36771
C	-2.82376	-2.64965	-0.96011
C	1.2121	-0.95583	0.85708
C	3.59673	-1.16317	0.07163
C	-0.78369	2.97395	-0.76036
C	-2.00721	2.08706	-0.80465
C	-4.70034	-1.21107	-1.42785
C	-0.97547	0.68975	2.19537
C	-4.18072	-2.49206	-1.24293
C	-2.11865	-1.44014	2.17223
C	4.17596	-1.32885	1.34019
C	-1.86036	1.15277	3.16943
C	0.4982	2.44435	-0.57212
C	-2.99674	-0.97739	3.14908
C	4.42008	-0.77064	-0.99641
C	-2.87467	0.32195	3.64736
C	6.34887	-0.73033	0.46262
C	5.54048	-1.11891	1.53249
C	5.78327	-0.55586	-0.80209
C	1.60395	3.29177	-0.48378
C	1.44192	4.67447	-0.57959
H	-4.28439	0.89999	-1.53455
H	-2.39149	-3.63116	-0.80153
H	1.55791	-0.50338	1.77696
H	-2.51785	2.08267	0.17005
H	-2.71428	2.46452	-1.55596
H	-5.75834	-1.07783	-1.63293
H	-0.20218	1.34391	1.80803
H	-4.82558	-3.3618	-1.31139
H	-2.22035	-2.44814	1.78692
H	3.55789	-1.64955	2.17334
H	-1.7577	2.16505	3.54919
H	0.63055	1.37088	-0.51836
H	-3.77835	-1.63279	3.5214
H	3.97961	-0.61192	-1.97582
H	-3.5657	0.68298	4.40332
H	7.41109	-0.56256	0.61373
H	5.97336	-1.26307	2.5181
H	6.40303	-0.24462	-1.63788
H	2.5928	2.86395	-0.34428
H	2.30286	5.33282	-0.51029
C	-0.93851	4.36017	-0.8724
C	0.1658	5.20688	-0.77655
H	-1.92834	4.78111	-1.03466
H	0.03081	6.28101	-0.86495
H	2.33147	-2.36145	-2.0446

3d – Minimum

C	-4.80422	-1.40783	0.92349
C	-4.27149	-1.94709	-0.24328
C	-2.92272	-1.75654	-0.56828
C	-2.12189	-0.98603	0.29455
C	-2.65275	-0.48384	1.48557
C	-3.99167	-0.68693	1.80339
H	-5.85271	-1.56682	1.15759
H	-4.87441	-2.53517	-0.92677
H	-2.00989	0.09074	2.14421
H	-4.39608	-0.28533	2.72653
C	-0.36456	0.68961	-0.06155
C	0.17267	-1.68899	-0.22501
C	0.97059	0.99009	-0.06652
C	1.58337	-1.35302	-0.18895
C	1.96572	-0.03068	-0.09544
H	1.27045	2.02853	-0.12639
C	-1.38839	1.75627	-0.17562
C	-1.3148	2.89244	0.64109
C	-2.39959	1.6744	-1.14701
C	-2.23956	3.92668	0.49711
H	-0.54026	2.95225	1.39983
C	-3.31936	2.70897	-1.28862
H	-2.45593	0.80051	-1.78775
C	-3.24498	3.83651	-0.4659
H	-2.17786	4.79804	1.14221
H	-4.09563	2.63598	-2.04421
H	-3.96786	4.63931	-0.57587
C	3.38939	0.40833	-0.05721
C	3.8599	1.14487	1.03943
C	4.26461	0.14485	-1.12119
C	5.18168	1.58838	1.08237
H	3.18504	1.35816	1.86345
C	5.58396	0.59412	-1.08097
H	3.90053	-0.4028	-1.98514
C	6.0474	1.31336	0.02255
H	5.53458	2.14863	1.94324

H	6.24876	0.38631	-1.91419
H	7.07576	1.66074	0.05408
O	-2.43766	-2.2859	-1.71724
H	-1.55415	-2.65971	-1.47502
O	-0.24267	-2.85595	-0.37154
N	-0.75676	-0.64337	-0.05103
C	2.51968	-2.53797	-0.17108
H	2.18852	-3.25199	-0.9316
H	3.53416	-2.22613	-0.42335
C	2.52269	-3.23814	1.20058
H	2.8614	-2.55322	1.98492
H	1.51784	-3.58763	1.45061
H	3.19374	-4.10293	1.18999

3d – Transition State

C	4.61507	-1.81562	-1.42665
C	3.77671	-2.72869	-0.81469
C	2.53661	-2.36942	-0.25344
C	2.15645	-0.99013	-0.22846
C	3.01024	-0.09692	-0.90982
C	4.21022	-0.48286	-1.48932
H	5.55528	-2.13869	-1.86278
H	4.02165	-3.78431	-0.76857
H	2.73294	0.93609	-1.0186
H	4.81397	0.26343	-1.99536
C	0.4385	0.83027	0.08513
C	-0.03281	-1.33659	0.97499
C	-0.90102	1.06581	-0.0248
C	-1.45077	-1.15993	0.67912
C	-1.87733	0.03367	0.14973
H	-1.23612	2.09084	-0.12465
C	1.34793	1.99203	0.2564
C	1.25174	3.11406	-0.57964
C	2.24662	2.01943	1.33642
C	2.04067	4.23957	-0.34346
H	0.57308	3.08969	-1.42672
C	3.03165	3.14494	1.56801
H	2.32546	1.15149	1.98316
C	2.93309	4.25878	0.72936
H	1.96262	5.09833	-1.00347
H	3.72098	3.15443	2.40692
H	3.54988	5.13378	0.91025
C	-3.29219	0.36062	-0.16955
C	-3.61786	0.83578	-1.44911
C	-4.3088	0.26159	0.79278
C	-4.93204	1.17609	-1.76779
H	-2.83422	0.92382	-2.19616
C	-5.62143	0.60842	0.47564
H	-4.06072	-0.07017	1.79618
C	-5.93797	1.0616	-0.80659
H	-5.16997	1.53089	-2.76628
H	-6.39607	0.53102	1.23282
H	-6.96107	1.32933	-1.05311
O	1.79608	-3.42496	0.14374
H	1.23925	-3.1519	0.92057
O	0.36013	-2.23072	1.74467
N	0.88724	-0.49195	0.3022
C	-2.30329	-2.38079	0.93784
H	-2.10439	-2.74111	1.95161
H	-3.36123	-2.12254	0.87808
C	-1.99692	-3.50411	-0.07237
H	-2.25054	-3.18632	-1.08878
H	-0.93789	-3.7751	-0.05697
H	-2.58141	-4.39961	0.16071

3(int)d – Intermediate Minimum

C	-4.84668	-1.80445	-0.43445
C	-4.23048	-1.68634	0.8011
C	-2.85617	-1.27392	0.95324
C	-2.20132	-1.00848	-0.30898
C	-2.82372	-1.13922	-1.54324
C	-4.16211	-1.52649	-1.63027
H	-5.89137	-2.1153	-0.47926
H	-4.77367	-1.89603	1.72004
H	-2.24618	-0.93268	-2.4429
H	-4.65195	-1.62208	-2.59521
C	-0.3853	0.64667	-0.13272
C	0.12762	-1.73333	-0.30729
C	0.95432	0.95373	-0.09939
C	1.5502	-1.38909	-0.24008
C	1.94047	-0.07157	-0.15674
H	1.25297	1.99033	-0.00774
C	-1.38118	1.74947	-0.03833
C	-2.31885	1.79961	1.00529
C	-1.31727	2.80491	-0.95855
C	-3.17865	2.89174	1.10657
H	-2.3532	0.97709	1.71904
C	-2.18317	3.89478	-0.85113
H	-0.59175	2.7585	-1.76578
C	-3.11739	3.94016	0.18377

H	-3.90539	2.92143	1.91421
H	-2.12918	4.70197	-1.57751
H	-3.79538	4.78599	0.27027
C	3.36785	0.35618	-0.09438
C	3.832	1.10043	1.00103
C	4.26474	0.07548	-1.13669
C	5.15619	1.53545	1.06172
H	3.14255	1.3265	1.80909
C	5.58844	0.51122	-1.07896
H	3.90919	-0.48176	-1.99813
C	6.04071	1.24134	0.02227
H	5.49765	2.10352	1.92319
H	6.2663	0.28482	-1.89796
H	7.0722	1.58045	0.06815
O	-2.28667	-1.09602	2.07355
O	-0.26642	-2.89444	-0.42175
N	-0.79559	-0.65471	-0.26618
C	2.47684	-2.57966	-0.18427
H	2.16003	-3.29573	-0.95059
H	3.50656	-2.28439	-0.40109
C	2.42111	-3.27872	1.18694
H	2.75338	-2.60087	1.98083
H	1.39576	-3.5885	1.40005
H	3.06589	-4.16588	1.20135

3(int)d – Intermediate Transition State

C	4.39952	-2.6446	-1.21085
C	4.44645	-1.28028	-1.03371
C	3.34316	-0.49686	-0.51936
C	2.17292	-1.2642	-0.13193
C	2.14586	-2.65144	-0.36091
C	3.22785	-3.34903	-0.88292
H	5.26458	-3.17453	-1.61
H	5.32869	-0.70125	-1.29518
H	1.25952	-3.21817	-0.11328
H	3.15406	-4.42241	-1.03522
C	0.50985	0.66349	0.02922
C	-0.0083	-1.47649	1.02281
C	-0.83735	0.89761	-0.12983
C	-1.42303	-1.27127	0.71589
C	-1.82415	-0.09685	0.1178
H	-1.15787	1.91767	-0.30106
C	1.34788	1.89044	0.14784
C	1.24945	2.91459	-0.80208
C	1.99066	2.16389	1.36966
C	1.8233	4.16418	-0.56362
H	0.74481	2.71326	-1.74234
C	2.54798	3.41212	1.60945
H	2.05671	1.37579	2.11233
C	2.47341	4.42267	0.64231
H	1.75844	4.93814	-1.32518
H	3.05063	3.60151	2.55504
H	2.91621	5.39745	0.83111
C	-3.23492	0.24955	-0.20559
C	-3.5655	0.67192	-1.50361
C	-4.25122	0.23139	0.76372
C	-4.87122	1.03813	-1.82934
H	-2.78246	0.70359	-2.25549
C	-5.55725	0.59857	0.44052
H	-4.00256	-0.05683	1.78034
C	-5.87465	0.99957	-0.85904
H	-5.1053	1.35401	-2.84272
H	-6.3268	0.58055	1.20803
H	-6.89236	1.28565	-1.11101
O	3.43564	0.76258	-0.45076
O	0.36381	-2.39217	1.76172
N	0.94154	-0.63155	0.34355
C	-2.32706	-2.42654	1.08324
H	-2.1812	-2.66266	2.14368
H	-3.37424	-2.14721	0.9478
C	-2.03738	-3.6932	0.25755
H	-2.16576	-3.49706	-0.81238
H	-1.0123	-4.02757	0.42978
H	-2.71678	-4.50644	0.53986

4j – Product Minimum

C	2.35700	3.43200	-1.54800
C	2.66400	2.09000	-1.36500
C	1.76400	1.21300	-0.74300
C	0.52400	1.68400	-0.24300
C	0.23500	3.04200	-0.47600
C	1.12500	3.90300	-1.10800
H	3.07400	4.09200	-2.02600
H	3.62500	1.71100	-1.68900
H	-0.70200	3.45500	-0.13900
H	0.84200	4.94100	-1.25000
C	-0.72100	-0.52600	0.15000
C	-1.60200	1.56100	0.99600
C	-1.99600	-0.95200	-0.06900
C	-2.95900	1.14200	0.61500

C	-3.14000	-0.08200	0.02400
H	-2.17400	-2.01800	-0.14800
C	0.29300	-1.55500	0.49300
C	1.01100	-1.44000	1.69400
C	0.44300	-2.71400	-0.27900
C	1.87000	-2.45600	2.10200
H	0.89100	-0.54400	2.29600
C	1.30700	-3.73100	0.13200
H	-0.10200	-2.80400	-1.21300
C	2.02700	-3.60500	1.32100
H	2.42000	-2.35300	3.03300
H	1.42000	-4.62000	-0.48300
H	2.70200	-4.39400	1.63800
C	-4.43800	-0.62000	-0.45900
C	-5.61200	-0.58200	0.31000
C	-4.48800	-1.23200	-1.72400
C	-6.80200	-1.12000	-0.18000
H	-5.58600	-0.15000	1.30300
C	-5.67800	-1.76300	-2.21600
H	-3.58400	-1.27300	-2.32500
C	-6.84100	-1.70700	-1.44500
H	-7.69800	-1.08600	0.43300
H	-5.69800	-2.22000	-3.20100
H	-7.76900	-2.12400	-1.82500
O	-1.39200	2.53900	1.70400
N	-0.50300	0.85400	0.38300
O	2.03900	-0.11200	-0.61400
C	3.13700	-0.71200	-1.29500
H	2.89200	-1.77800	-1.27300
H	3.15900	-0.39100	-2.34600
C	4.47500	-0.47200	-0.62700
C	5.64900	-0.46400	-1.38800
C	4.55500	-0.29600	0.75800
C	6.89000	-0.29200	-0.77200

4j – Product Transition State

C	2.35700	3.43200	-1.54800
C	2.66400	2.09000	-1.36500
C	1.76400	1.21300	-0.74300
C	0.52400	1.68400	-0.24300
C	0.23500	3.04200	-0.47600
C	1.12500	3.90300	-1.10800
H	3.07400	4.09200	-2.02600
H	3.62500	1.71100	-1.68900
H	-0.70200	3.45500	-0.13900
H	0.84200	4.94100	-1.25000
C	-0.72100	-0.52600	0.15000
C	-1.60200	1.56100	0.99600
C	-1.99600	-0.95200	-0.06900
C	-2.95900	1.14200	0.61500
C	-3.14000	-0.08200	0.02400
H	-2.17400	-2.01800	-0.14800
C	0.29300	-1.55500	0.49300
C	1.01100	-1.44000	1.69400
C	0.44300	-2.71400	-0.27900
C	1.87000	-2.45600	2.10200
H	0.89100	-0.54400	2.29600
C	1.30700	-3.73100	0.13200
H	-0.10200	-2.80400	-1.21300
C	2.02700	-3.60500	1.32100
H	2.42000	-2.35300	3.03300
H	1.42000	-4.62000	-0.48300
H	2.70200	-4.39400	1.63800
C	-4.43800	-0.62000	-0.45900
C	-5.61200	-0.58200	0.31000
C	-4.48800	-1.23200	-1.72400
C	-6.80200	-1.12000	-0.18000
H	-5.58600	-0.15000	1.30300
C	-5.67800	-1.76300	-2.21600
H	-3.58400	-1.27300	-2.32500
C	-6.84100	-1.70700	-1.44500
H	-7.69800	-1.08600	0.43300
H	-5.69800	-2.22000	-3.20100
H	-7.76900	-2.12400	-1.82500
O	-1.39200	2.53900	1.70400
N	-0.50300	0.85400	0.38300
O	2.03900	-0.11200	-0.61400
C	3.13700	-0.71200	-1.29500
H	2.89200	-1.77800	-1.27300
H	3.15900	-0.39100	-2.34600
C	4.47500	-0.47200	-0.62700
C	5.64900	-0.46400	-1.38800
C	4.55500	-0.29600	0.75800
C	6.89000	-0.29200	-0.77200

3e – Minimum

C	4.8619	-1.52496	0.98473
C	4.28561	-2.05889	-0.16374
C	2.94096	-1.81429	-0.4674
C	2.19141	-0.9968	0.39725

C	2.76391	-0.49742	1.5698
C	4.09829	-0.75442	1.86659
H	5.9063	-1.72653	1.20345
H	4.85003	-2.68343	-0.84767
H	2.1582	0.11459	2.22993
H	4.53705	-0.35671	2.77552
C	0.50319	0.74268	0.00943
C	-0.14573	-1.60847	-0.05791
C	-0.81679	1.11095	0.01756
C	-1.52481	-1.19294	0.01676
C	-1.86398	0.14716	0.07752
H	-1.06058	2.16324	-0.04813
C	1.57403	1.75531	-0.15255
C	2.55865	1.59526	-1.14136
C	1.57138	2.91572	0.63262
C	3.52356	2.57953	-1.33171
H	2.55863	0.70195	-1.75736
C	2.54301	3.89793	0.44074
H	0.81727	3.03494	1.40469
C	3.52145	3.73135	-0.53973
H	4.27876	2.44799	-2.10049
H	2.53816	4.78857	1.06178
H	4.27988	4.49425	-0.68729
C	-3.26344	0.65294	0.10272
C	-3.661	1.61483	-0.84208
C	-4.18682	0.2308	1.07211
C	-4.95569	2.12925	-0.83004
H	-2.95601	1.94242	-1.60075
C	-5.47795	0.75752	1.08702
H	-3.88768	-0.51087	1.80154
C	-5.8677	1.7033	0.13781
H	-5.25182	2.86092	-1.57589
H	-6.18055	0.42523	1.84529
H	-6.87589	2.10679	0.15131
O	2.41111	-2.33609	-1.60089
H	1.51896	-2.67062	-1.33824
O	0.20931	-2.79528	-0.2066
N	0.83531	-0.60218	0.07021
C	-2.53945	-2.31777	-0.03386
O	-3.23732	-2.57966	0.92767
C	-2.65248	-3.05783	-1.34602
H	-1.66985	-3.43135	-1.6441
H	-3.36225	-3.88013	-1.24396
H	-2.99948	-2.36223	-2.1201

3e – Transition State

C	4.62371	-1.91083	-1.53454
C	3.72972	-2.81027	-0.98419
C	2.50898	-2.41401	-0.40685
C	2.20706	-1.01998	-0.30318
C	3.11653	-0.13579	-0.92094
C	4.29745	-0.55519	-1.51581
H	5.54755	-2.26001	-1.98503
H	3.91404	-3.87894	-1.00089
H	2.90076	0.91652	-0.96504
H	4.94888	0.18421	-1.97001
C	0.57715	0.86377	0.10979
C	-0.01657	-1.31564	0.85461
C	-0.74766	1.17874	-0.00134
C	-1.4106	-1.03617	0.56618
C	-1.79111	0.20367	0.10154
H	-1.01968	2.22558	-0.05011
C	1.54411	1.96499	0.35025
C	1.51821	3.13244	-0.42636
C	2.42735	1.88567	1.44022
C	2.36395	4.19887	-0.12277
H	0.85051	3.18982	-1.28049
C	3.26764	2.95361	1.74009
H	2.44968	0.98269	2.04177
C	3.24074	4.11286	0.95965
H	2.3415	5.09401	-0.7369
H	3.94377	2.88255	2.5866
H	3.90078	4.94247	1.19363
C	-3.16453	0.62317	-0.27182
C	-3.33673	1.3442	-1.46787
C	-4.28566	0.37607	0.53722
C	-4.599	1.78245	-1.85983
H	-2.47492	1.54052	-2.09938
C	-5.54562	0.82745	0.14533
H	-4.16072	-0.16494	1.46573
C	-5.70886	1.52556	-1.05183
H	-4.71614	2.32378	-2.79396
H	-6.4025	0.63379	0.78358
H	-6.69354	1.87102	-1.35274
O	1.70557	-3.44586	-0.07134
H	1.17151	-3.18966	0.72675
O	0.2993	-2.27739	1.57776
N	0.95902	-0.48789	0.24507
C	-2.31721	-2.22731	0.79488
O	-3.24305	-2.19729	1.58364
C	-2.04853	-3.43767	-0.07698
H	-0.98407	-3.60562	-0.25078
H	-2.50923	-4.3201	0.36994
H	-2.51899	-3.25212	-1.05138

3(int)e – Intermediate Minimum

C	-4.8972	-1.885	-0.27486
C	-4.26268	-1.71273	0.94467
C	-2.8989	-1.25649	1.05741
C	-2.2785	-1.00865	-0.22495
C	-2.91651	-1.19537	-1.44412
C	-4.24367	-1.62331	-1.49195
H	-5.9326	-2.22724	-0.29046
H	-4.78217	-1.91103	1.87947
H	-2.36205	-1.0005	-2.36049
H	-4.74878	-1.76226	-2.4434
C	-0.51064	0.68798	-0.11789
C	0.07414	-1.68327	-0.24406
C	0.82894	1.03595	-0.09678
C	1.48581	-1.29299	-0.20585
C	1.83813	0.05287	-0.13016
H	1.09207	2.07855	0.02422
C	-1.52959	1.77024	-0.03964
C	-2.45743	1.82711	1.01156
C	-1.49388	2.79974	-0.99009
C	-3.34097	2.9022	1.08879
H	-2.46386	1.02605	1.74996
C	-2.38698	3.8691	-0.90857
H	-0.77158	2.74891	-1.79992
C	-3.31313	3.92209	0.13347
H	-4.0603	2.93984	1.90241
H	-2.35882	4.65544	-1.65848
H	-4.01036	4.75366	0.20094
C	3.23755	0.55253	0.01036
C	4.04171	0.15853	1.08974
C	3.72859	1.52142	-0.87367
C	5.30788	0.70743	1.26913
H	3.66212	-0.58361	1.78459
C	5.00268	2.06471	-0.70131
H	3.10751	1.83743	-1.7067
C	5.79692	1.66112	0.37163
H	5.91627	0.39126	2.11233
H	5.37178	2.80606	-1.40534
H	6.78716	2.08666	0.51103
O	-2.31032	-1.02864	2.15855
O	-0.32957	-2.83747	-0.34763
N	-0.88287	-0.61398	-0.21434
C	2.52754	-2.35428	-0.33334
O	3.63444	-2.10272	-0.80362
C	2.22162	-3.76438	0.13793
H	1.63245	-3.76923	1.05813
H	1.60447	-4.27202	-0.6075
H	3.17188	-4.28879	0.2664

3(int)e – Intermediate Transition State

C	4.39291	-2.74668	-1.25331
C	4.53928	-1.40659	-0.98603
C	3.46491	-0.5559	-0.5111
C	2.21216	-1.24678	-0.24426
C	2.08813	-2.61231	-0.57066
C	3.14181	-3.36598	-1.06163
H	5.23755	-3.32582	-1.6254
H	5.48379	-0.89295	-1.14565
H	1.132	-3.10557	-0.4476
H	2.98887	-4.41264	-1.30838
C	0.61275	0.72557	0.0345
C	0.01724	-1.45194	0.87352
C	-0.74784	0.99486	-0.09816
C	-1.36292	-1.26845	0.43746
C	-1.7398	0.00464	-0.02108
H	-1.04597	2.03231	-0.17626
C	1.4837	1.91522	0.20009
C	1.36604	3.01104	-0.66279
C	2.24853	2.05278	1.37239
C	2.02455	4.20942	-0.38256
H	0.77744	2.90944	-1.56969
C	2.88264	3.25358	1.66105
H	2.33612	1.20442	2.04159
C	2.78047	4.33802	0.78185
H	1.94253	5.04284	-1.07593
H	3.47391	3.34292	2.56867
H	3.28761	5.27323	1.0054
C	-3.1362	0.45371	-0.27698
C	-3.42458	1.19336	-1.43268
C	-4.15944	0.26644	0.66615
C	-4.70169	1.71324	-1.65263
H	-2.63804	1.34833	-2.16524
C	-5.42887	0.79314	0.45443
H	-3.94588	-0.29967	1.56616
C	-5.70911	1.51736	-0.70919
H	-4.90544	2.27345	-2.56164
H	-6.20568	0.63863	1.19887
H	-6.70317	1.92452	-0.87481
O	3.65191	0.68405	-0.37344
O	0.40306	-2.29483	1.67424

N	1.00991	-0.58547	0.2238
C	-2.29669	-2.4183	0.454
O	-3.36438	-2.38298	-0.1599
C	-1.93201	-3.68817	1.20905
H	-0.99201	-4.11249	0.84466
H	-1.76296	-3.48151	2.26858
H	-2.75054	-4.39869	1.07435

4m – Product Minimum

O	-0.2949	-3.0466	-1.5741
O	-1.6507	0.5954	-1.2491
N	-0.8719	-1.5713	0.078
C	-2.2075	-1.5351	-0.459
C	-2.615	-0.3541	-1.1059
C	0.1021	-2.2726	-0.7087
C	-0.5337	-0.7276	1.1119
C	1.4979	-1.9644	-0.4117
C	-1.6019	-0.0363	1.875
C	1.8193	-1.0715	0.6
C	-3.931	-0.2401	-1.5594
C	-3.0711	-2.6169	-0.3528
C	0.7884	-0.5032	1.3956
C	3.1923	-0.5539	0.8538
C	-0.7517	2.7956	-1.4314
C	-2.015	1.9647	-1.3987
C	-4.8038	-1.3211	-1.4165
C	-1.5378	1.3565	2.0307
C	-4.3777	-2.5135	-0.8317
C	-2.663	-0.7421	2.4618
C	3.7487	-0.6042	2.1374
C	-2.5255	2.0315	2.7481
C	0.4549	2.3138	-0.9108
C	-3.6424	-0.0658	3.1845
C	3.8924	0.1068	-0.167
C	-3.58	1.3228	3.3254
C	5.686	0.6238	1.3703
C	4.9944	-0.0295	2.3907
C	5.1285	0.6939	0.0909
C	1.5961	3.118	-0.9096
C	1.5423	4.4132	-1.426
H	-4.2694	0.6736	-2.0334
H	-2.712	-3.5262	0.116
H	1.0431	0.17	2.2036
H	-2.65	2.2537	-0.5477
H	-2.5917	2.123	-2.3202
H	-5.8235	-1.228	-1.7779
H	-0.7286	1.908	1.5637
H	-5.0567	-3.3548	-0.7426
H	-2.7167	-1.8191	2.3501
H	3.2102	-1.1115	2.9325
H	-2.4704	3.111	2.8518
H	0.506	1.3026	-0.529
H	-4.4564	-0.6236	3.6375
H	3.457	0.1616	-1.16
H	-4.3496	1.8481	3.883
H	6.6525	1.0772	1.5695
H	5.4225	-0.0907	3.3869
H	5.6583	1.2045	-0.7079
H	2.5244	2.7213	-0.5078
H	2.4293	5.0399	-1.4253
C	-0.7951	4.0898	-1.9619
C	0.3428	4.8966	-1.9541
H	-1.723	4.47	-2.3834
H	0.2949	5.8993	-2.369
C	2.3074	-3.0595	-2.6359
H	1.695	-3.9644	-2.6595
H	1.7356	-2.2942	-3.1676
H	3.2683	-3.2426	-3.1204
C	2.5732	-2.6651	-1.198
O	3.654	-2.9001	-0.6781

4m – Product Transition State

O	-0.29498	-3.04641	-1.57447
O	-1.65068	0.59558	-1.24904
N	-0.87192	-1.57132	0.07782
C	-2.20753	-1.53498	-0.45912
C	-2.61501	-0.35394	-1.1059
C	0.10199	-2.27244	-0.70888
C	-0.53377	-0.72767	1.11184
C	1.49783	-1.96438	-0.41188
C	-1.60197	-0.0365	1.87501
C	1.81922	-1.07164	0.59995
C	-3.93101	-0.2398	-1.55945
C	-3.07119	-2.61675	-0.35302
C	0.78837	-0.50338	1.39563
C	3.19225	-0.55409	0.8538
C	-0.75157	2.79576	-1.43123
C	-2.01495	1.96489	-1.39852
C	-4.80387	-1.32084	-1.41665
C	-1.53778	1.35635	2.03095

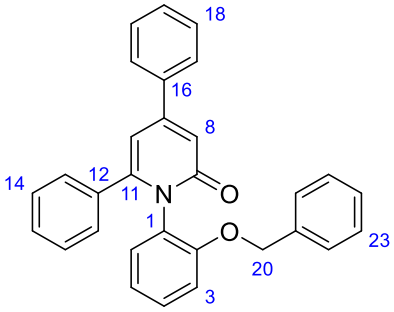
C	-4.37781	-2.51326	-0.83198
C	-2.66305	-0.74232	2.46172
C	3.74864	-0.60452	2.13746
C	-2.5255	2.03123	2.74837
C	0.45499	2.31389	-0.91044
C	-3.64247	-0.06613	3.18453
C	3.89241	0.10676	-0.16683
C	-3.58004	1.32244	3.32565
C	5.68602	0.6235	1.37049
C	4.9944	-0.02994	2.39078
C	5.12849	0.69379	0.09112
C	1.59618	3.11804	-0.90928
C	1.54254	4.41322	-1.42592
H	-4.2694	0.67394	-2.03337
H	-2.71215	-3.5261	0.11568
H	1.04308	0.1697	2.2037
H	-2.6498	2.25387	-0.54736
H	-2.59173	2.12333	-2.31987
H	-5.82355	-1.22761	-1.77805
H	-0.72863	1.90781	1.56397
H	-5.05685	-3.35454	-0.74293
H	-2.71681	-1.81928	2.34995
H	3.21017	-1.11198	2.93243
H	-2.47034	3.11076	2.8522
H	0.50595	1.30278	-0.5286
H	-4.45641	-0.62391	3.63751
H	3.45701	0.16176	-1.15983
H	-4.34963	1.84777	3.88327
H	6.65246	1.07683	1.56974
H	5.42242	-0.09127	3.38704
H	5.65831	1.2045	-0.70761
H	2.52447	2.72137	-0.50743
H	2.42953	5.03986	-1.42526
C	-0.7949	4.08986	-1.96185
C	0.34314	4.89665	-1.95407
H	-1.7227	4.47012	-2.38353
H	0.29527	5.89921	-2.36918
C	2.30738	-3.05918	-2.63618
H	1.69492	-3.964	-2.65998
H	1.73556	-2.29372	-3.16781
H	3.26827	-3.24222	-3.12075
C	2.57317	-2.66502	-1.1983
O	3.65397	-2.9001	-0.67842

7 HPLC data

7.1 *O*-Benzylated *N*-(2-phenoxy)pyridones (4)

Racemic traces were generated *via* thermal racemisation of the enantio-enriched materials, using the end-point trace generated according to General Procedure F (see page S37 below).

1-(2-(Benzyloxy)phenyl)-4,6-diphenylpyridin-2(1*H*)-one (4a)



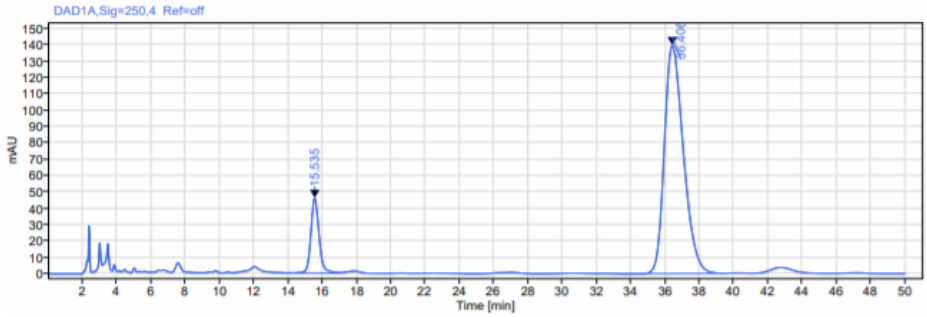
Conditions: **HPLC**: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 36.4 min, t_R (minor) = 15.5 min.

Asymmetric trace: 75% ee

Single Injection Report

Agilent Technologies

Data file:	JS-4-289 rot. bar. 80 oC t=0h 60% MeCN-H2O20210423 122213.dx		
Sequence Name:	SingleSample	**Project Name:**	JS
Sample name:	JS-4-289 rot. bar. 80 oC t=0h 60% MeCN/H2O	**Operator:**	SYSTEM
Instrument:	1100HPLC	**Injection date:**	2021-04-23 12:23:21+01:00
Inj. volume:	10.000	**Location:**	43
Acq. method:	60% MeCN-H2O 45 MIN.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	0.00
Manually modified:	Manual Integration		



Signal: DAD1A,Sig=250,4 Ref=off

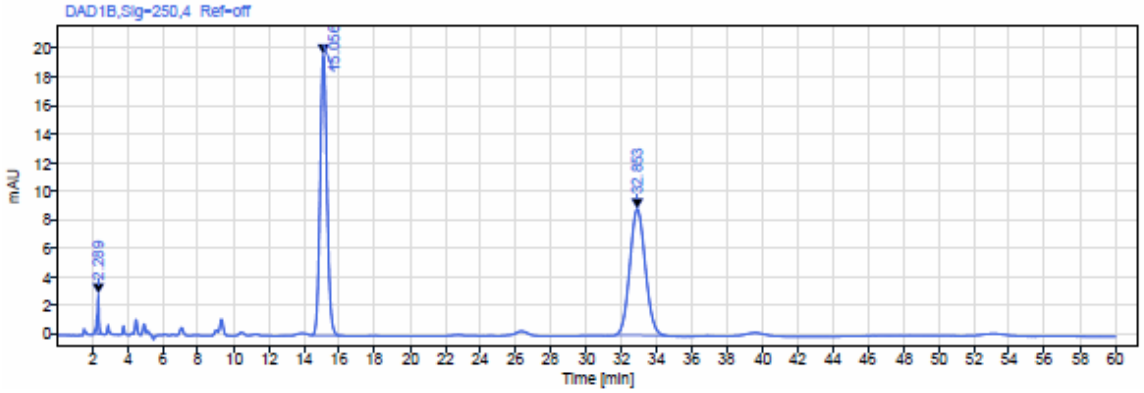
RT [min]	Type	Width [min]	Area	Height	Area%	Name
15.535	BV	2.53	1522.20	45.83	12.35	
36.406	BB	4.83	10804.04	139.60	87.65	
		Sum	12326.23			

Racemic trace

Single Injection Report

Agilent Technologies

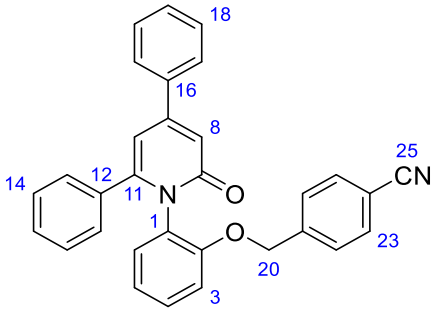
Data file:	JS #289 S10 76H.dx		
Sequence Name:	SingleSample	**Project Name:**	Agilent
Sample name:	JS #289 S10 76H	**Operator:**	Agilent
Instrument:	1220LC	**Injection date:**	2021-07-08 17:43:02+01:00
Inj. volume:	5.000	**Location:**	2
Acq. method:	Atropisomer barrier measurement.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	1.00
Manually modified:	None		



Signal: DAD1B,Sig=250,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
2.289	VB	0.23	12.81	2.80	1.18	
15.056	BB	1.69	531.33	19.66	49.03	
32.853	BB	2.87	539.44	8.83	49.78	
		Sum	1083.58			

4-((2-(2-Oxo-4,6-diphenylpyridin-1(2*H*)-yl)phenoxy)methyl)benzonitrile (4b)



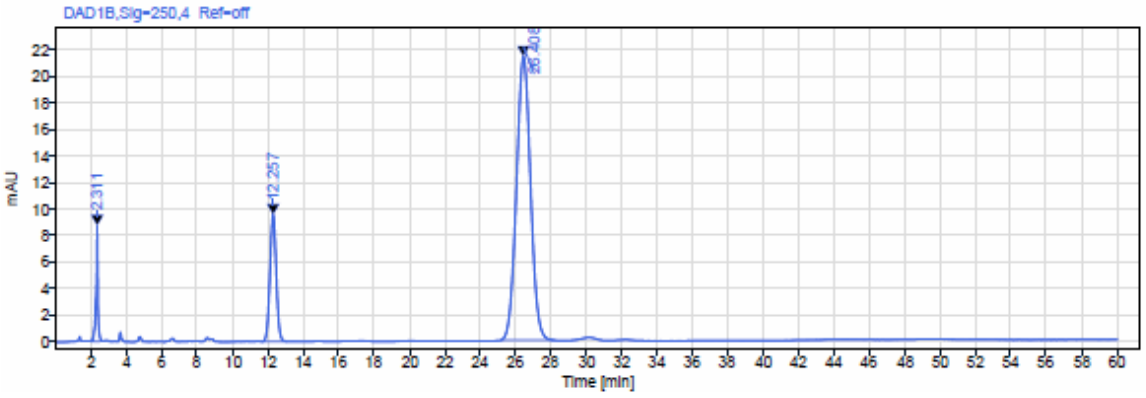
Conditions: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 26.4 min, t_R (minor) = 12.3 min.

Asymmetric trace: 67% *ee*

Single Injection Report

Agilent Technologies

Data file:	JS #302 80C Origin.dx		
Sequence Name:	SingleSample	**Project Name:**	Agilent
Sample name:	JS #302 80C Origin	**Operator:**	Agilent
Instrument:	1220LC	**Injection date:**	2021-07-23 23:39:50+01:00
Inj. volume:	5.000	**Location:**	21
Acq. method:	Atropisomer barrier measurement.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	1.00
Manually modified:	None		



Signal: DAD1B,Sig=250,4 Ref=off

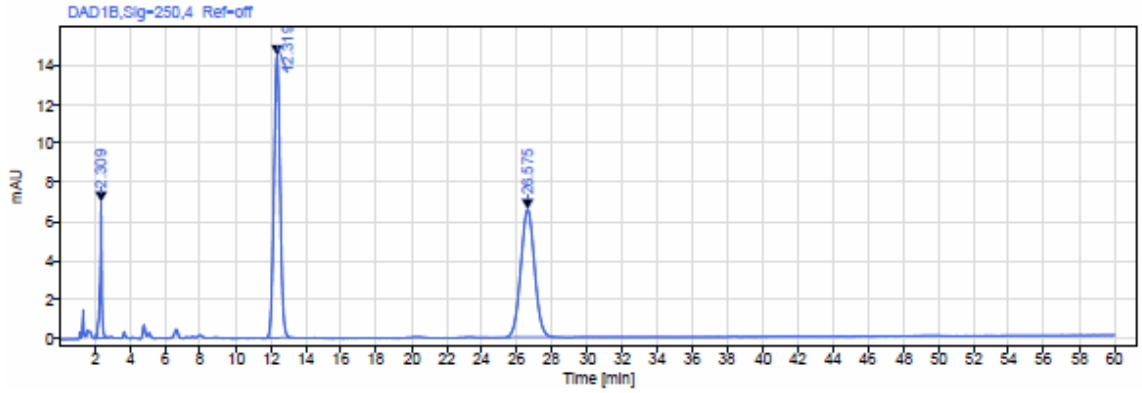
RT [min]	Type	Width [min]	Area	Height	Area%	Name
2.311	VB	0.61	55.44	8.71	3.77	
12.257	BB	1.37	234.75	9.55	15.96	
26.408	BB	2.96	1180.71	21.35	80.27	
		Sum	1470.89			

Racemic trace

Single Injection Report

Agilent Technologies

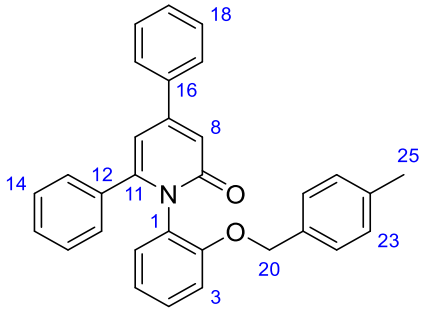
Data file:	JS #298 80C S9 102H.dx		
Sequence Name:	SingleSample	**Project Name:**	Agilent
Sample name:	JS #298 80C S9 102H	**Operator:**	Agilent
Instrument:	1220LC	**Injection date:**	2021-07-22 20:27:57+01:00
Inj. volume:	5.000	**Location:**	64
Acq. method:	Atropisomer barrier measurement.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	1.00
Manually modified:	None		



Signal: DAD1B,Sig=250,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
2.309	VB	0.63	44.97	6.96	5.99	
12.319	BB	1.34	349.81	14.45	46.62	
26.575	BB	2.70	355.54	6.53	47.39	
		Sum	750.31			

1-(2-((4-Methylbenzyl)oxy)phenyl)-4,6-diphenylpyridin-2(1*H*)-one (4c)

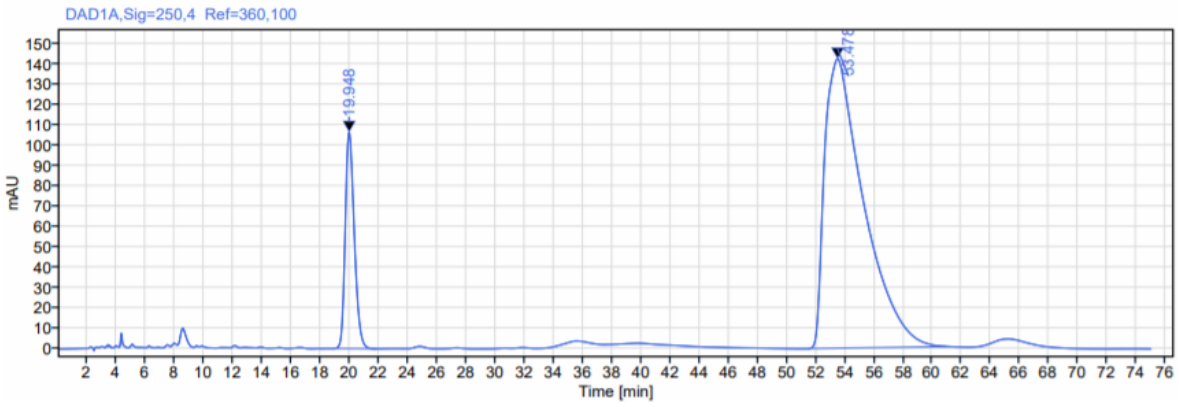


Conditions: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 53.5 min, t_R (minor) = 19.9 min.

Asymmetric trace: 70% *ee*

Single Injection Report — Agilent Technologies

Data file:	JS-4-303 col 60% MeCN-H2O20210504 142035.dx		
Sequence Name:	SingleSample	**Project Name:**	JS
Sample name:	JS-4-303 col 60% MeCN/H2O	**Operator:**	SYSTEM
Instrument:	1100HPLC	**Injection date:**	2021-05-04 14:21:44+01:00
Inj. volume:	5.000	**Location:**	17
Acq. method:	60% MeCN-H2O 45 MIN.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	0.00
Manually modified:	Manual Integration		



Signal: DAD1A,Sig=250,4 Ref=360,100

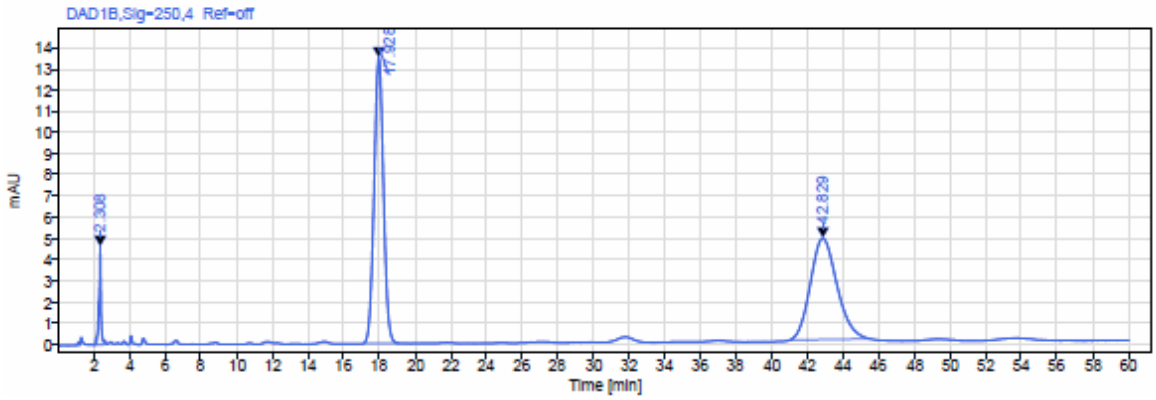
RT [min]	Type	Width [min]	Area	Height	Area%	Name
19.948	BB	3.00	4592.01	106.31	14.78	
53.478	BB	9.53	26471.31	142.11	85.22	
		Sum	31063.32			

Racemic trace

Single Injection Report

Agilent Technologies

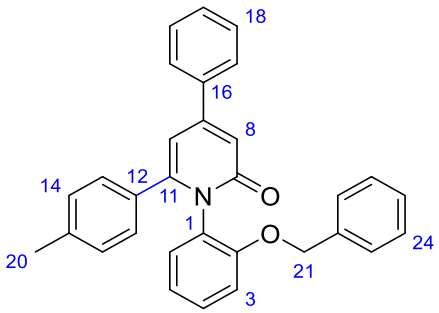
Data file:	JS #303 80C S9 70H.dx		
Sequence Name:	SingleSample	**Project Name:**	Agilent
Sample name:	JS #303 80C S9 70H	**Operator:**	Agilent
Instrument:	1220LC	**Injection date:**	2021-07-24 18:59:09+01:00
Inj. volume:	5.000	**Location:**	80
Acq. method:	Atropisomer barrier measurement.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	1.00
Manually modified:	None		



Signal: DAD1B,Sig=250,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
2.308	VV	0.40	32.21	4.63	3.16	
17.928	BB	1.97	491.37	13.45	48.27	
42.829	BB	4.44	494.39	4.78	48.57	
		Sum	1017.96			

1-(2-(Benzyloxy)phenyl)-4-phenyl-6-(*p*-tolyl)pyridin-2(1*H*)-one (4d)

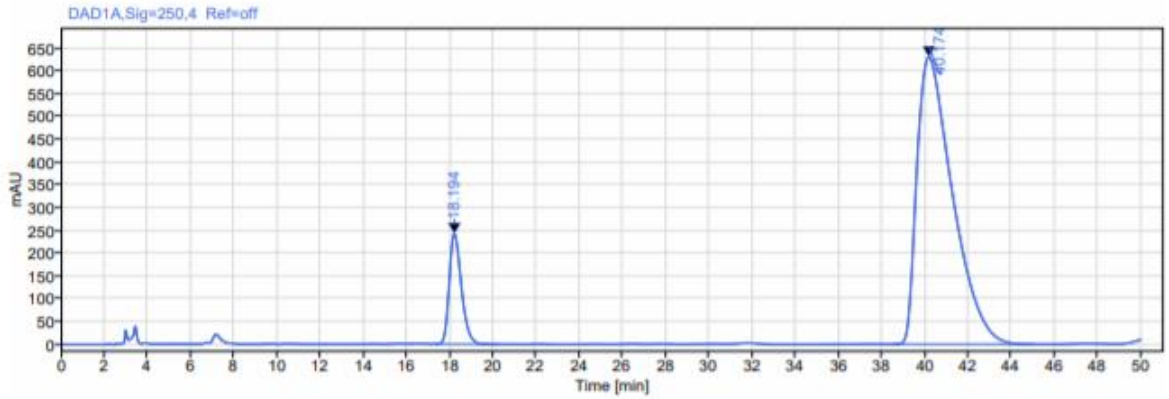


Conditions: **HPLC**: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) =40.2 min, t_R (minor) = 18.2 min.

Asymmetric trace: 77% *ee*

Single Injection Report — Agilent Technologies

Data file:	JS-5-308 col 60% MeCN-H2O20210430 153237.dx		
Sequence Name:	SingleSample	**Project Name:**	JS
Sample name:	JS-5-308 col 60% MeCN/H2O	**Operator:**	SYSTEM
Instrument:	1100HPLC	**Injection date:**	2021-04-30 15:49:26+01:00
Inj. volume:	10.000	**Location:**	15
Acq. method:	60% MeCN-H2O 45 MIN.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	0.00
Manually modified:	Manual Integration		



Signal: DAD1A,Sig=250,4 Ref=off

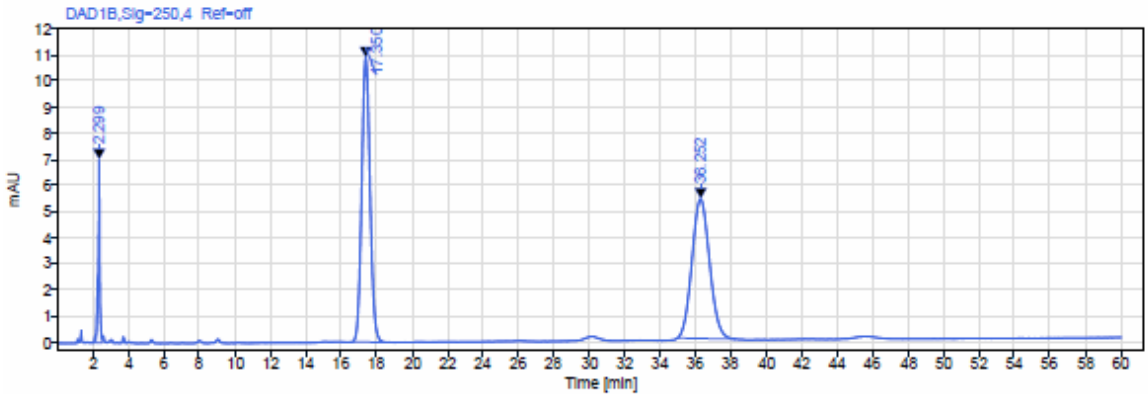
RT [min]	Type	Width [min]	Area	Height	Area%	Name
18.194	BB	2.89	9504.28	242.00	11.63	
40.174	BB	6.55	72200.43	630.27	88.37	
		Sum	81704.70			

Racemic trace

Single Injection Report

Agilent Technologies

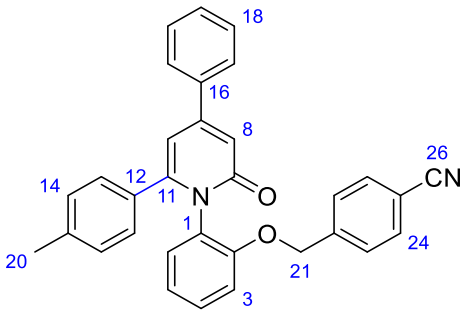
Data file:	JS #308 80C S9 70.5H.dx		
Sequence Name:	SingleSample	**Project Name:**	Agilent
Sample name:	JS #308 80C S9 70.5H	**Operator:**	Agilent
Instrument:	1220LC	**Injection date:**	2021-07-31 22:02:06+01:00
Inj. volume:	5.000	**Location:**	55
Acq. method:	Atropisomer barrier measurement.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	1.00
Manually modified:	None		



Signal: DAD1B,Sig=250,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
2.299	VV	0.37	40.37	7.01	5.28	
17.350	BB	1.85	354.39	10.88	46.39	
36.252	BB	3.27	369.14	5.34	48.32	
		Sum	763.89			

4-((2-(2-Oxo-4-phenyl-6-(*p*-tolyl)pyridin-1(2*H*)-yl)phenoxy)methyl)benzonitrile (4e)



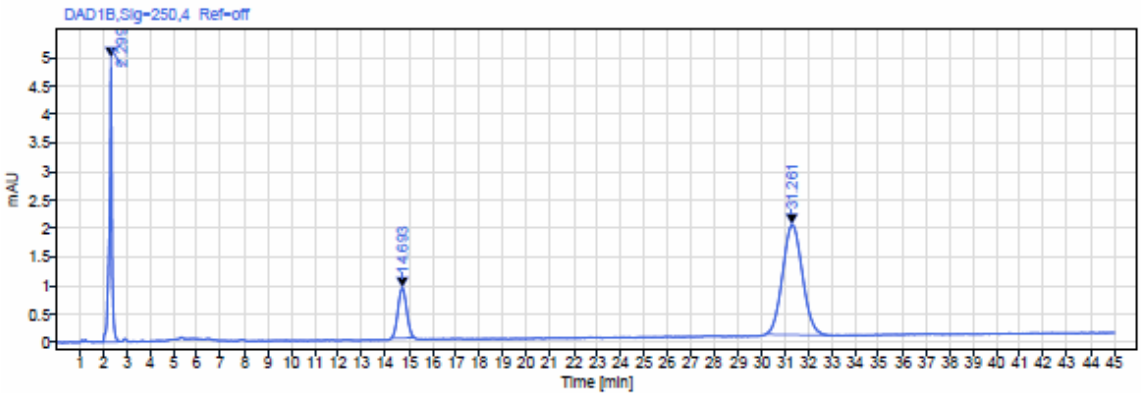
Conditions: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 31.3 min, t_R (minor) = 14.7 min.

Asymmetric trace: 67% *ee*

Single Injection Report

Agilent Technologies

Data file:	JS #292 70C Origin.dx		
Sequence Name:	SingleSample	**Project Name:**	Agilent
Sample name:	JS #292 70C Origin	**Operator:**	Agilent
Instrument:	1220LC	**Injection date:**	2021-07-26 20:40:36+01:00
Inj. volume:	5.000	**Location:**	1
Acq. method:	(45min) Atropisomer barrier measurement.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	1.00
Manually modified:	Manual Integration		



Signal: DAD1B,Sig=250,4 Ref=off

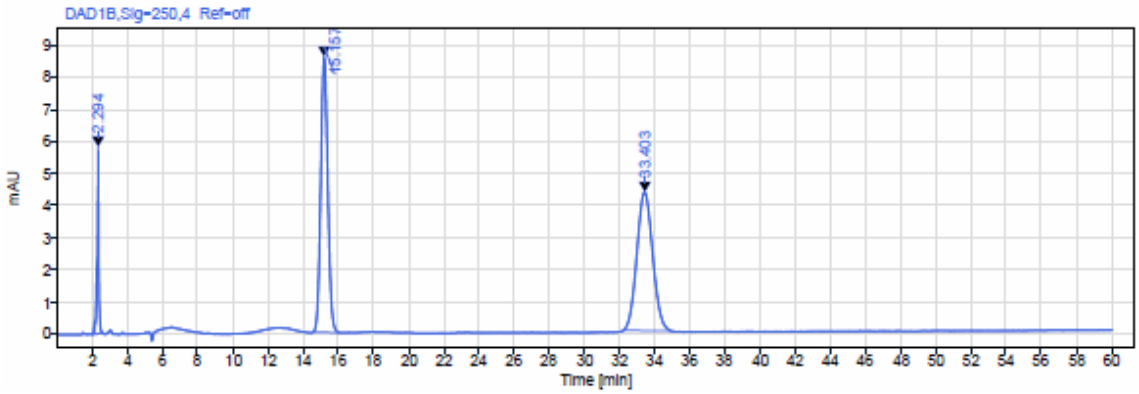
RT [min]	Type	Width [min]	Area	Height	Area%	Name
2.299	BB	0.76	36.37	4.99	21.12	
14.693	MM m	0.34	22.46	0.88	13.04	
31.261	BB	2.69	113.35	1.93	65.83	
		Sum	172.18			

Racemic trace

Single Injection Report

Agilent Technologies

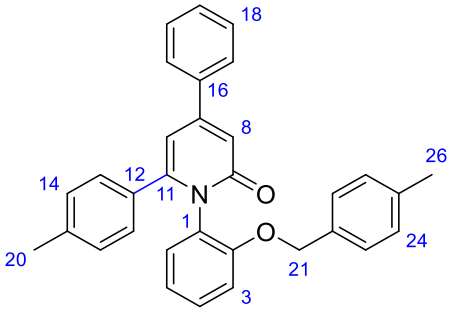
Data file:	JS #292 80C S10 54H (07.09).dx		
Sequence Name:	SingleSample	**Project Name:**	Agilent
Sample name:	JS #292 80C S10 54H (07.09)	**Operator:**	Agilent
Instrument:	1220LC	**Injection date:**	2021-07-09 20:40:05+01:00
Inj. volume:	5.000	**Location:**	32
Acq. method:	Atropisomer barrier measurement.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	1.00
Manually modified:	None		



Signal: DAD1B,Sig=250,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
2.294	VB	0.39	32.98	5.81	6.11	
15.157	BB	1.44	237.55	8.57	44.04	
33.403	BB	2.70	268.82	4.31	49.84	
		Sum	539.35			

1-(2-((4-methylbenzyl)oxy)phenyl)-4-phenyl-6-(*p*-tolyl)pyridin-2(1*H*)-one (4f)

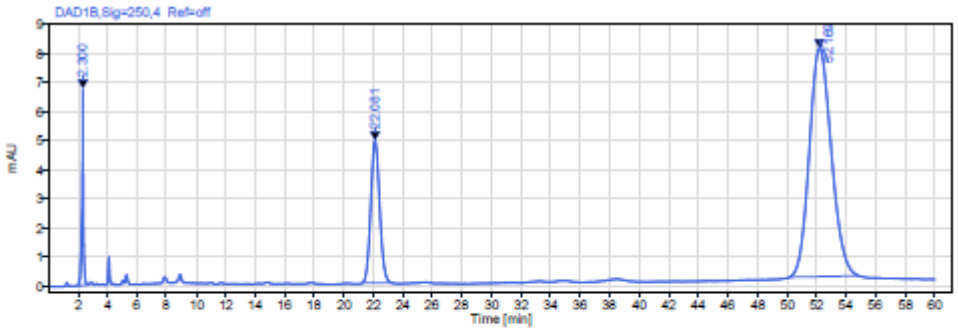


Conditions: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 52.8 min, t_R (minor) = 22.1 min.

Asymmetric trace: 58% *ee*

Single Injection Report — Agilent Technologies

Data file:	JS #296 70C Origin.dx		
Sequence Name:	SingleSample	**Project Name:**	Agilent
Sample name:	JS #296 70C Origin	**Operator:**	Agilent
Instrument:	1220LC	**Injection date:**	2021-07-27 12:02:34+01:00
Inj. volume:	5.000	**Location:**	21
Acq. method:	Atropisomer barrier measurement.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	1.00
Manually modified:	Manual Integration		



Signal: DAD1B,Sig=250,4 Ref=off

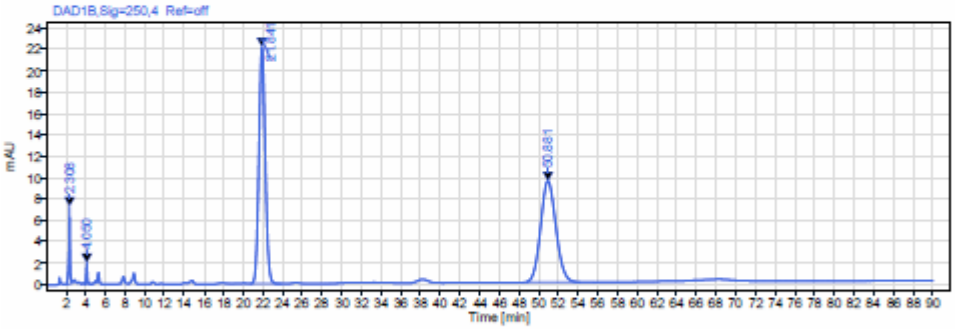
RT [min]	Type	Width [min]	Area	Height	Area%	Name
2.300	VB	0.54	40.96	6.77	3.87	
22.081	BB	2.00	204.03	4.90	19.28	
52.169	MM m	1.21	813.43	7.85	76.85	
		Sum	1058.42			

Racemic Trace

Single Injection Report

Agilent Technologies

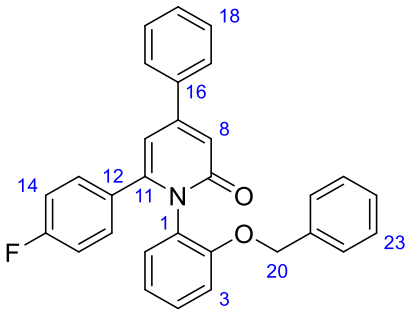
Data file:	JS #296 80C S9 102H.dx		
Sequence Name:	SingleSample	**Project Name:**	Agilent
Sample name:	JS #296 80C S9 102H	**Operator:**	Agilent
Instrument:	1220LC	**Injection date:**	2021-07-22 17:25:41+01:00
Inj. volume:	5.000	**Location:**	62
Acq. method:	(90min) atropisomer barrier measurement.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	1.00
Manually modified:	None		



Signal: DAD1B,Sig=250,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
2.308	VV	0.46	52.32	7.29	2.52	
4.050	BB	0.43	13.59	1.99	0.65	
21.841	BB	3.05	993.05	22.23	47.75	
50.881	BB	5.47	1020.89	9.48	49.08	
		Sum	2079.84			

1-(2-(Benzyloxy)phenyl)-6-(4-fluorophenyl)-4-phenylpyridin-2(1*H*)-one (4g)

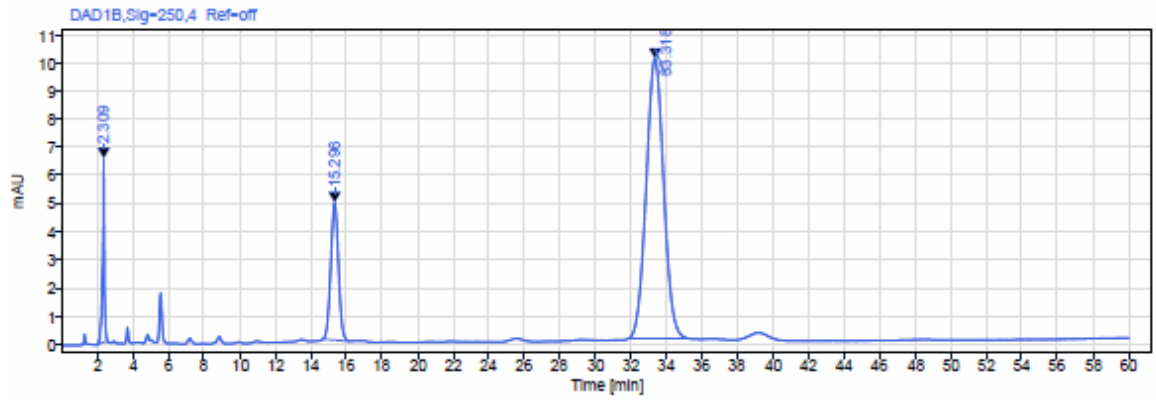


Conditions: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) =33.3 min, t_R (minor) = 15.3 min.

Asymmetric trace: 65% *ee*

Single Injection Report — Agilent Technologies

Data file:	JS #301 80C Origin.dx		
Sequence Name:	SingleSample	**Project Name:**	Agilent
Sample name:	JS #301 80C Origin	**Operator:**	Agilent
Instrument:	1220LC	**Injection date:**	2021-07-23 13:29:44+01:00
Inj. volume:	5.000	**Location:**	11
Acq. method:	Atropisomer barrier measurement.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	1.00
Manually modified:	Manual Integration		



Signal: DAD1B,Sig=250,4 Ref=off

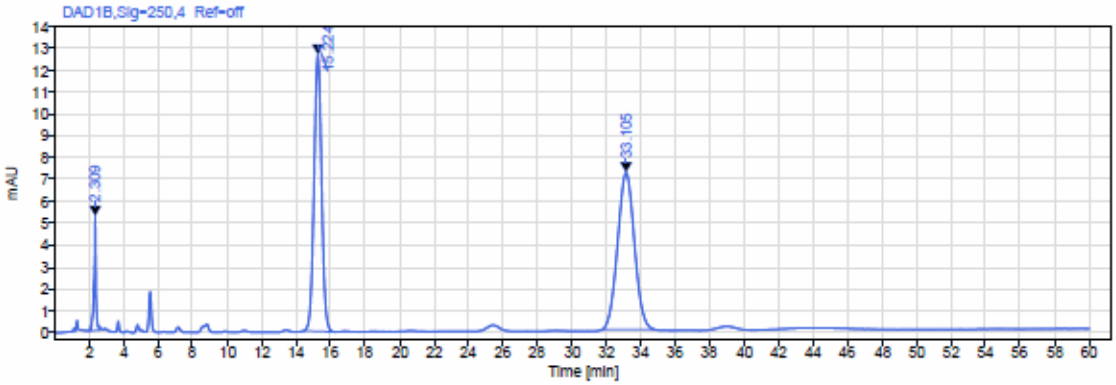
RT [min]	Type	Width [min]	Area	Height	Area%	Name
2.309	MM m	0.09	42.16	6.50	4.86	
15.296	BB	1.50	145.65	4.86	16.80	
33.318	BB	3.31	678.99	9.91	78.33	
		Sum	866.80			

Racemic trace

Single Injection Report

Agilent Technologies

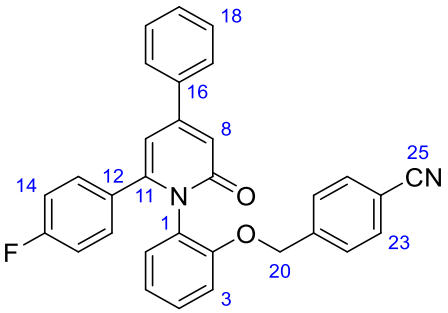
Data file:	JS #301 80C S8 48H.dx		
Sequence Name:	SingleSample	**Project Name:**	Agilent
Sample name:	JS #301 80C S8 48H	**Operator:**	Agilent
Instrument:	1220LC	**Injection date:**	2021-07-23 21:37:46+01:00
Inj. volume:	5.000	**Location:**	19
Acq. method:	Atropisomer barrier measurement.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	1.00
Manually modified:	Manual Integration		



Signal: DAD1B,Sig=250,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
2.309	VV	0.44	36.47	5.23	4.02	
15.224	BB	1.78	384.75	12.65	42.46	
33.105	BB	3.00	485.02	7.17	53.52	
		Sum	906.24			

4-((2-(6-(4-Fluorophenyl)-2-oxo-4-phenylpyridin-1(2*H*)-yl)phenoxy)methyl)benzonitrile (4h)

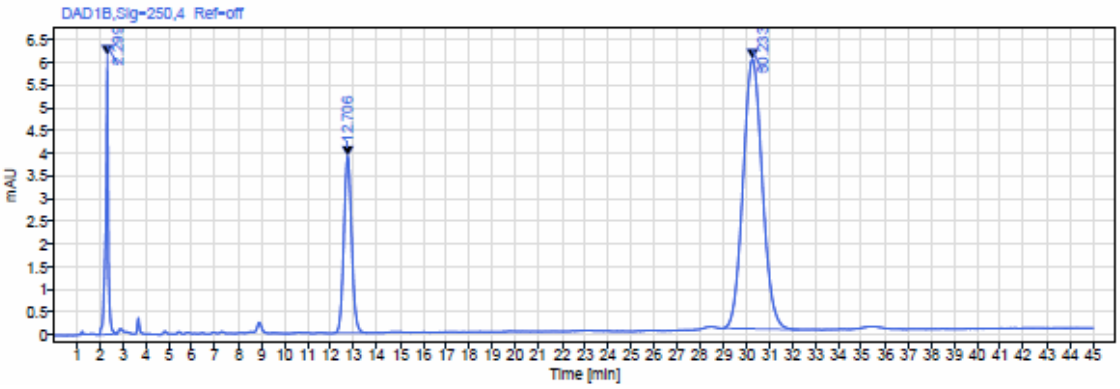


Conditions: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 30.3 min, t_R (minor) = 12.7 min.

Asymmetric trace: 58% *ee*

Single Injection Report — Agilent Technologies

Data file:	JS #293 70C Origin.dx		
Sequence Name:	SingleSample	**Project Name:**	Agilent
Sample name:	JS #293 70C Origin	**Operator:**	Agilent
Instrument:	1220LC	**Injection date:**	2021-07-27 04:21:37+01:00
Inj. volume:	5.000	**Location:**	11
Acq. method:	(45min) Atropisomer barrier measurement.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	1.00
Manually modified:	None		



Signal: DAD1B,Sig=250,4 Ref=off

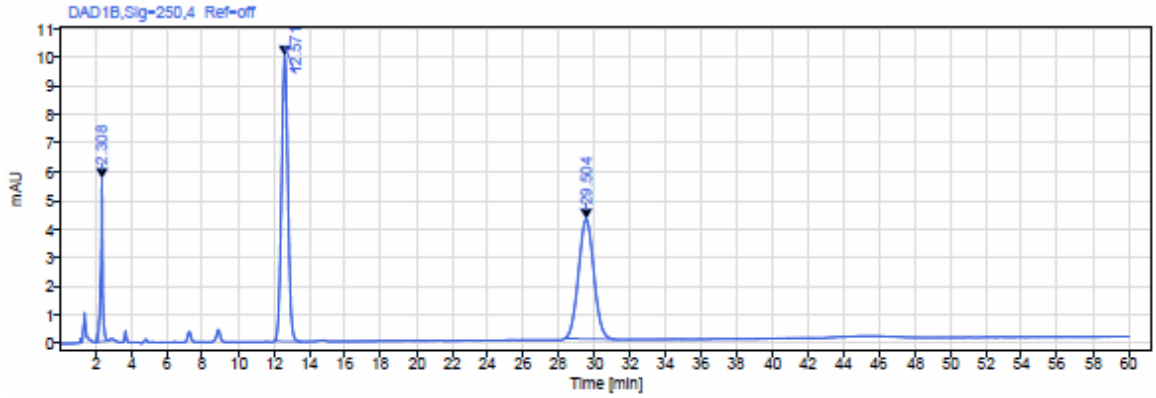
RT [min]	Type	Width [min]	Area	Height	Area%	Name
2.299	BB	0.78	43.74	6.13	9.14	
12.706	BB	1.36	91.24	3.87	19.07	
30.233	BB	3.24	343.41	5.92	71.79	
		Sum	478.39			

Racemic trace

Single Injection Report

Agilent Technologies

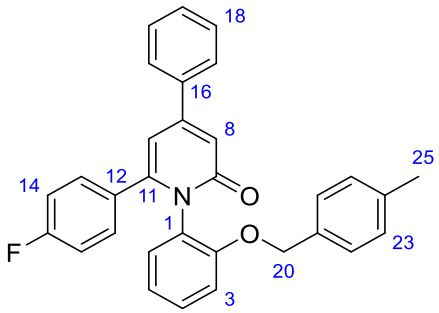
Data file:	JS #293 80C S9 102H.dx		
Sequence Name:	SingleSample	**Project Name:**	Agilent
Sample name:	JS #293 80C S9 102H	**Operator:**	Agilent
Instrument:	1220LC	**Injection date:**	2021-07-22 16:24:22+01:00
Inj. volume:	5.000	**Location:**	61
Acq. method:	Atropisomer barrier measurement.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	1.00
Manually modified:	None		



Signal: DAD1B,Sig=250,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
2.308	VB	0.57	39.42	5.71	7.31	
12.571	BB	1.48	247.95	10.01	46.01	
29.504	BB	2.81	251.57	4.19	46.68	
		Sum	538.94			

6-(4-Fluorophenyl)-1-(2-((4-methylbenzyl)oxy)phenyl)-4-phenylpyridin-2(1*H*)-one (4i)

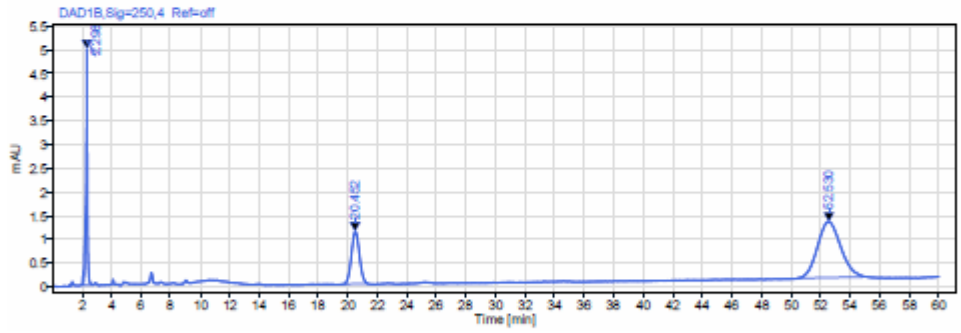


Conditions: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 52.3 min, t_R (minor) = 20.5 min.

Asymmetric trace: 51% *ee*

Single Injection Report — Agilent Technologies

Data file:	JS #297 80C Origin.dx		
Sequence Name:	SingleSample	**Project Name:**	Agilent
Sample name:	JS #297 80C Origin	**Operator:**	Agilent
Instrument:	1220LC	**Injection date:**	2021-07-17 01:22:17+01:00
Inj. volume:	5.000	**Location:**	17
Acq. method:	Atropisomer barrier measurementLamx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	1.00
Manually modified:	Manual Integration		



Signal: DAD1B,Sig=250,4 Ref=off

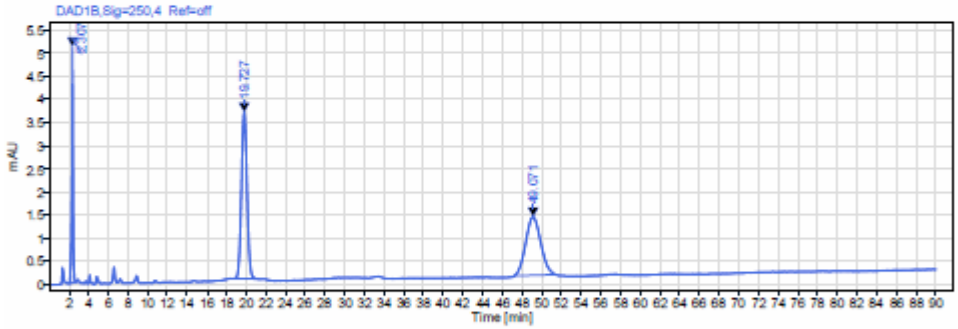
RT [min]	Type	Width [min]	Area	Height	Area%	Name
2.298	VB	0.60	30.51	5.01	15.15	
20.452	MM m	0.44	41.71	1.11	20.70	
52.530	MM m	1.27	129.23	1.19	64.15	
		Sum	201.45			

Racemic Trace

Single Injection Report

Agilent Technologies

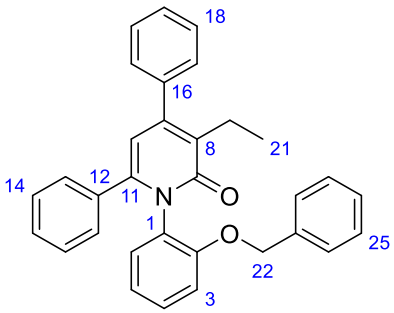
Data file:	JS #297 80C S9 102H.dx		
Sequence Name:	SingleSample	**Project Name:**	Agilent
Sample name:	JS #297 80C S9 102H	**Operator:**	Agilent
Instrument:	1220LC	**Injection date:**	2021-07-22 18:56:47+01:00
Inj. volume:	5.000	**Location:**	63
Acq. method:	(90min) atropisomer barrier measurement.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	1.00
Manually modified:	Manual Integration		



Signal: DAD1B,Sig=250,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
2.307	VB	0.53	38.58	5.11	12.30	
19.727	BB	2.30	140.96	3.61	44.94	
49.071	MM m	1.24	134.16	1.27	42.77	
		Sum	313.69			

1-(2-(Benzyloxy)phenyl)-3-ethyl-4,6-diphenylpyridin-2(1*H*)-one (4j)

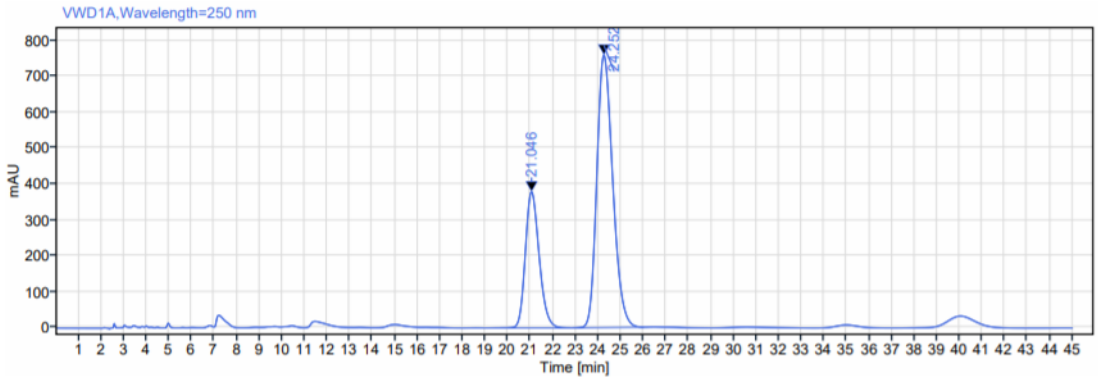


Conditions: **HPLC**: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 69.9 min, t_R (minor) = 30.2 min.

Asymmetric trace: 40% *ee*

Single Injection Report — Agilent Technologies

Data file:	JS-4-291 col 2 60% MeCN-H2O20210330 115119.dx		
Sequence Name:	SingleSample	**Project Name:**	Walkup
Sample name:	JS-4-291 col 2 60% MeCN:H2O	**Operator:**	admin account
Instrument:	LCMS	**Injection date:**	2021-03-30 12:33:57+01:00
Inj. volume:	5.000	**Location:**	P1-a3
Acq. method:	60% MeCN in H2O OD-RH column 40 mins MS.amx	**Type:**	Sample
Processing method:	LCMS Purity.pmx	**Sample amount:**	0.00
Manually modified:	Manual Integration		



Signal: VWD1A,Wavelength=250 nm

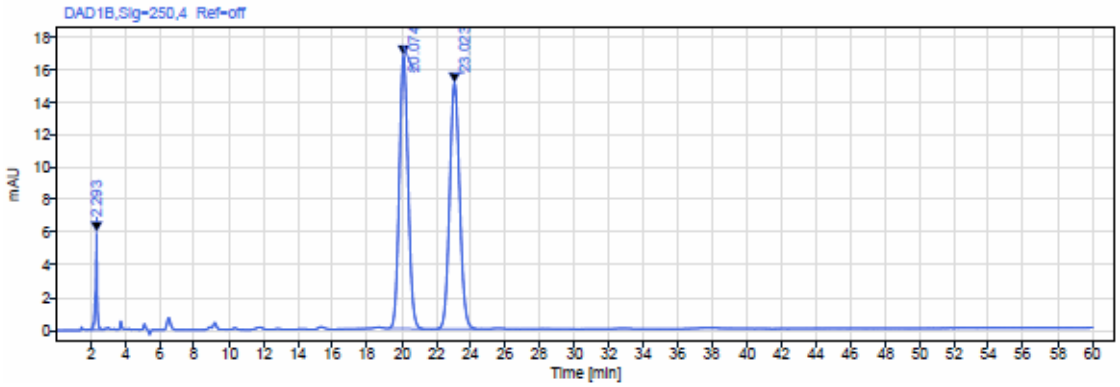
RT [min]	Type	Width [min]	Area	Height	Area%	Name
21.046	BB	3.08	16050.69	378.81	30.15	
24.252	BB	3.14	37179.46	758.83	69.85	
		Sum	53230.15			

Racemic trace

Single Injection Report

Agilent Technologies

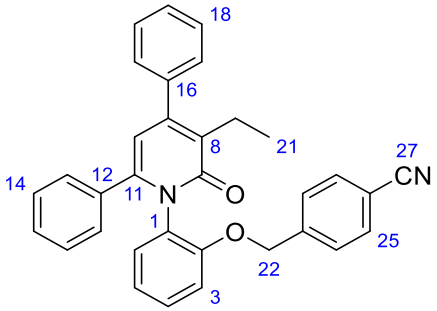
Data file:	JS #291 80C S10 54H (07.09).dx		
Sequence Name:	SingleSample	**Project Name:**	Agilent
Sample name:	JS #291 80C S10 54H (07.09)	**Operator:**	Agilent
Instrument:	1220LC	**Injection date:**	2021-07-09 19:39:03+01:00
Inj. volume:	5.000	**Location:**	31
Acq. method:	Atropisomer barrier measurement.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	1.00
Manually modified:	None		



Signal: DAD1B,Sig=250,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
2.293	VB	0.59	35.16	6.04	2.84	
20.074	BB	1.97	595.66	16.83	48.06	
23.023	BB	2.26	608.50	15.18	49.10	
		Sum	1239.32			

4-((2-(3-Ethyl-2-oxo-4,6-diphenylpyridin-1(2*H*)-yl)phenoxy)methyl)benzonitrile (4k)

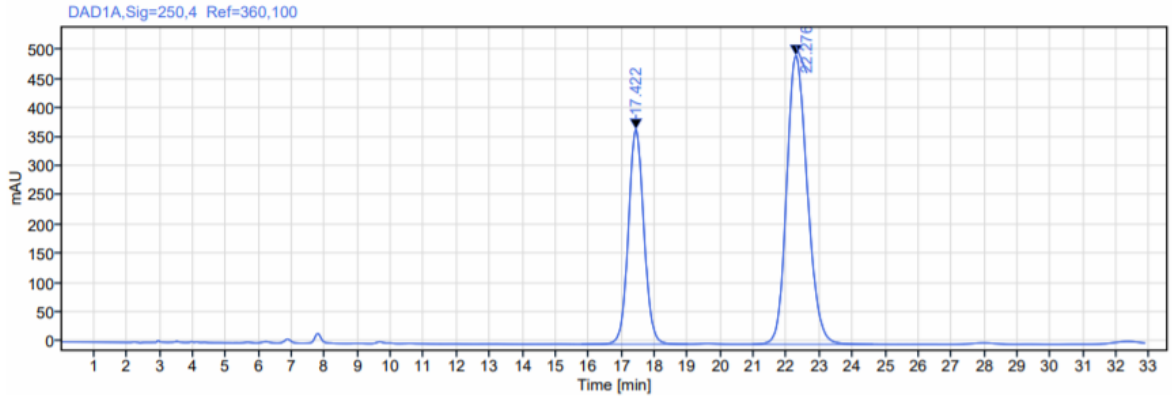


Conditions: CHIRALPAK OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = min, t_R (minor) = min.

Asymmetric trace: 28% *ee*

Single Injection Report — Agilent Technologies

Data file:	JS-4-306 col 60% MeCN-H2O20210520 211751.dx		
Sequence Name:	SingleSample	**Project Name:**	JS
Sample name:	JS-4-306 col 60% MeCN/H2O	**Operator:**	SYSTEM
Instrument:	1100HPLC	**Injection date:**	2021-05-20 21:18:55+01:00
Inj. volume:	2.000	**Location:**	37
Acq. method:	60% MeCN-H2O 45 MIN.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	0.00
Manually modified:	Manual Integration		



Signal: DAD1A,Sig=250,4 Ref=360,100

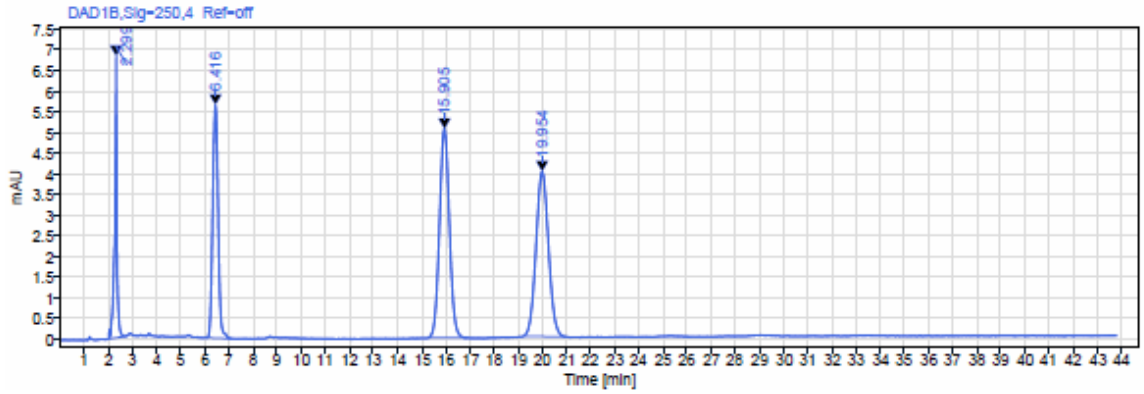
RT [min]	Type	Width [min]	Area	Height	Area%	Name
17.422	BB	3.23	12011.16	366.40	35.82	
22.276	BB	3.66	21520.34	494.41	64.18	
		Sum	33531.50			

Racemic trace*

Single Injection Report

Agilent Technologies

Data file:	JS #306 80C S8 96H (07.26).dx		
Sequence Name:	SingleSample	**Project Name:**	Agilent
Sample name:	JS #306 80C S8 96H (07.26)	**Operator:**	Agilent
Instrument:	1220LC	**Injection date:**	2021-07-26 15:51:27+01:00
Inj. volume:	5.000	**Location:**	83
Acq. method:	Atropisomer barrier measurement.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	1.00
Manually modified:	None		

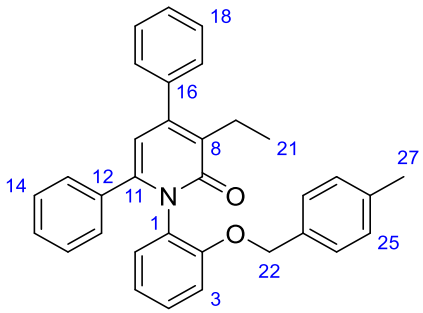


Signal: DAD1B,Sig=250,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
2.299	VB	0.63	40.68	6.82	9.80	
6.416	BB	1.01	88.00	5.64	21.20	
15.905	BB	1.56	143.69	5.05	34.61	
19.954	BB	1.73	142.80	3.99	34.40	
		Sum	415.18			

* Peaks at 2.3 and 6.4 min are an impurity carried-over from a previous sample.

3-Ethyl-1-(2-((4-methylbenzyl)oxy)phenyl)-4,6-diphenylpyridin-2(1*H*)-one (4l)



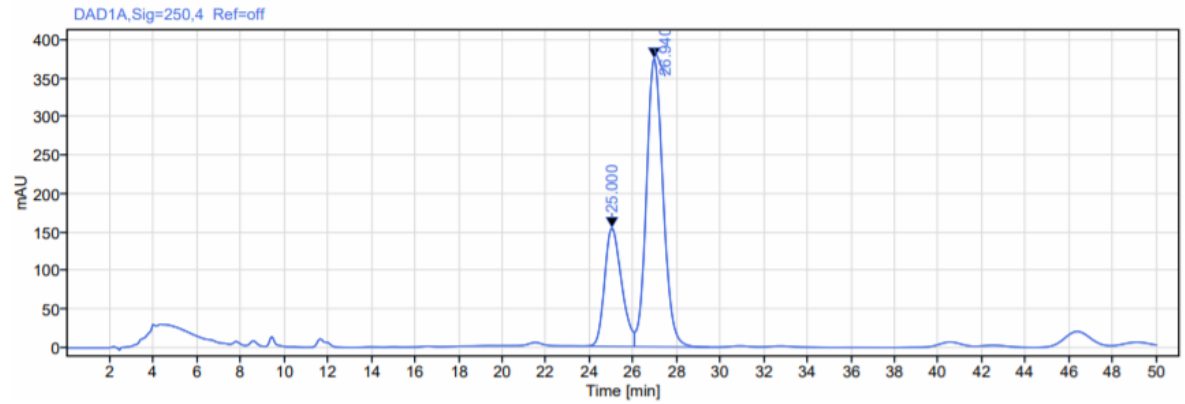
Conditions: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 26.9 min, t_R (minor) = 25.0 min.

Asymmetric trace: 41% *ee*

Single Injection Report

Agilent Technologies

Data file:	JS-4-307 col 60% MeCN-H2O20210423 183713.dx		
Sequence Name:	SingleSample	**Project Name:**	JS
Sample name:	JS-4-307 col 60% MeCN/H2O	**Operator:**	SYSTEM
Instrument:	1100HPLC	**Injection date:**	2021-04-24 09:33:53+01:00
Inj. volume:	10.000	**Location:**	54
Acq. method:	60% MeCN-H2O 45 MIN.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	0.00
Manually modified:	Manual Integration		



Signal: DAD1A,Sig=250,4 Ref=off

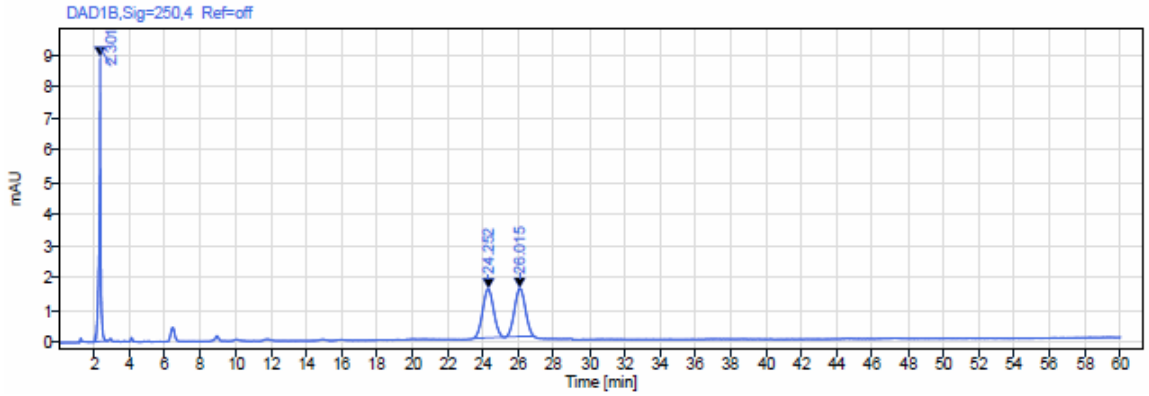
RT [min]	Type	Width [min]	Area	Height	Area%	Name
25.000	BV	2.28	8345.52	153.68	29.57	
26.940	VB	3.42	19874.06	374.57	70.43	
		Sum	28219.58			

Racemic Trace

Single Injection Report

Agilent Technologies

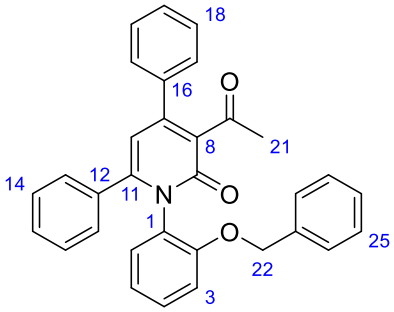
Data file:	JS #307 80C S8 96H (07.26).dx		
Sequence Name:	SingleSample	**Project Name:**	Agilent
Sample name:	JS #307 80C S8 96H (07.26)	**Operator:**	Agilent
Instrument:	1220LC	**Injection date:**	2021-07-26 16:36:16+01:00
Inj. volume:	5.000	**Location:**	84
Acq. method:	Atropisomer barrier measurement.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	1.00
Manually modified:	Manual Integration		



Signal: DAD1B,Sig=250,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
2.301	VB	0.59	50.01	8.92	27.78	
24.252	MM m	0.51	65.80	1.52	36.55	
26.015	MM m	0.50	64.22	1.51	35.67	
		Sum	180.04			

3-Acetyl-1-(2-(benzyloxy)phenyl)-4,6-diphenylpyridin-2(1*H*)-one (4m)

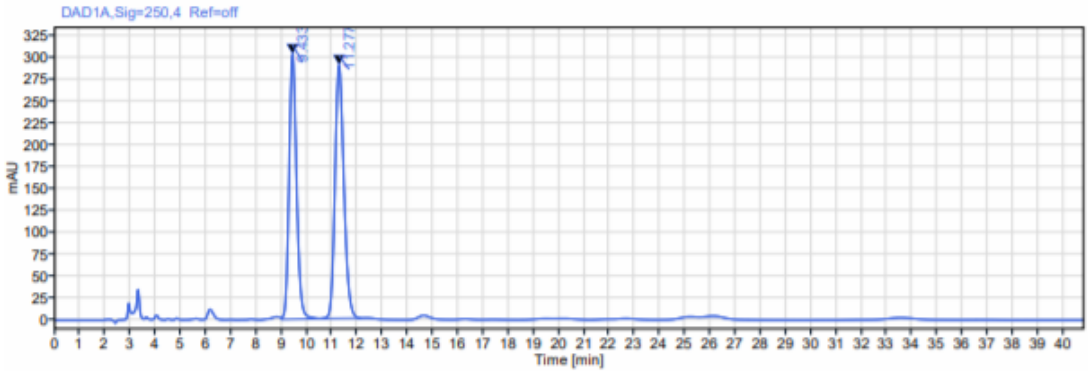


Conditions: **HPLC**: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 11.3 min, t_R (minor) = 9.4 min.

Asymmetric trace: 6% *ee*

Single Injection Report — Agilent Technologies

Data file:	JS-4-290 col 60% MeCN-H2O20210329 164450.dx		
Sequence Name:	SingleSample	**Project Name:**	JS
Sample name:	JS-4-290 col 60% MeCN/H2O	**Operator:**	SYSTEM
Instrument:	1100HPLC	**Injection date:**	2021-03-29 16:46:43+01:00
Inj. volume:	10.000	**Location:**	13
Acq. method:	60% MeCN-H2O 45 MIN.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	0.00
Manually modified:	Manual Integration		



Signal: DAD1A,Sig=250,4 Ref=off

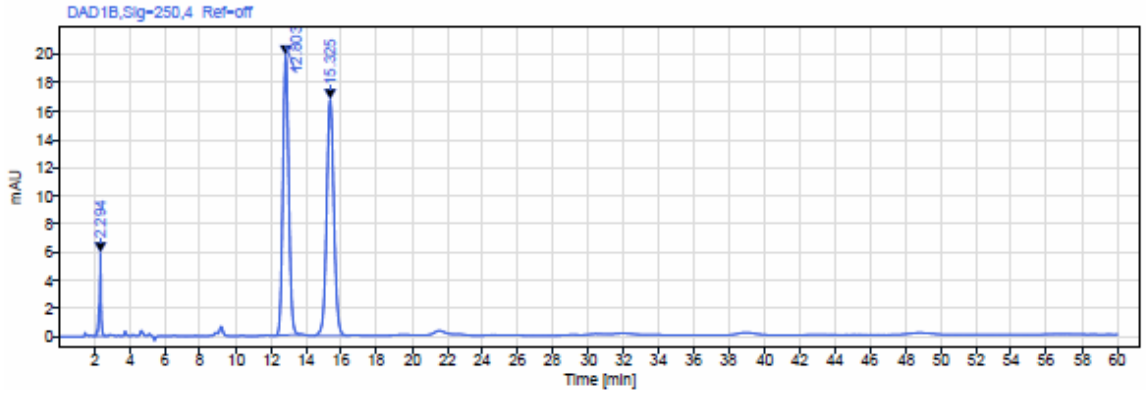
RT [min]	Type	Width [min]	Area	Height	Area%	Name
9.433	VB	1.50	6239.89	302.83	46.76	
11.277	BB	1.63	7104.91	289.41	53.24	
		Sum	13344.80			

Racemic trace

Single Injection Report

Agilent Technologies

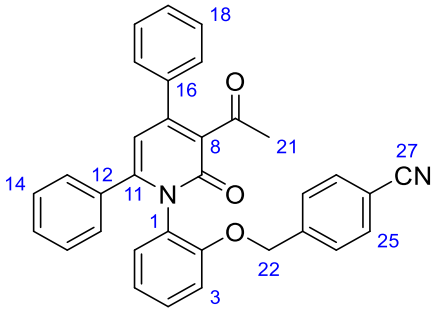
Data file:	JS #290 S11 76H (07.09).dx		
Sequence Name:	SingleSample	**Project Name:**	Agilent
Sample name:	JS #290 S11 76H (07.09)	**Operator:**	Agilent
Instrument:	1220LC	**Injection date:**	2021-07-09 18:38:04+01:00
Inj. volume:	5.000	**Location:**	6
Acq. method:	Atropisomer barrier measurement.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	1.00
Manually modified:	None		



Signal: DAD1B,Sig=250,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
2.294	VB	0.48	34.18	5.91	3.52	
12.803	BB	1.44	460.14	19.85	47.35	
15.325	BB	1.81	477.52	16.76	49.14	
		Sum	971.83			

4-((2-(3-Acetyl-2-oxo-4,6-diphenylpyridin-1(2*H*)-yl)phenoxy)methyl)benzonitrile (4n)

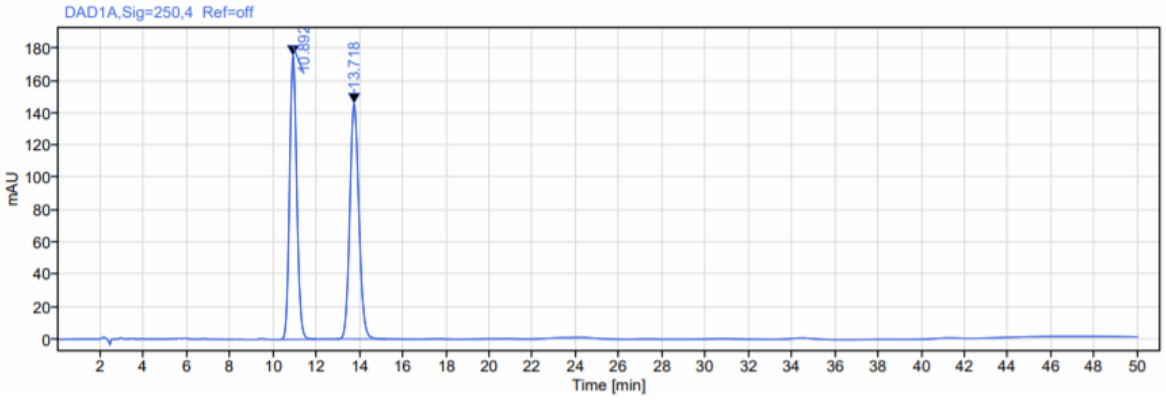


Conditions: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 13.7 min, t_R (minor) = 10.9 min.

Asymmetric trace: 3% *ee*

Single Injection Report — Agilent Technologies

Data file:	JS-4-304 col 60% MeCN-H2O20210422 135035.dx		
Sequence Name:	SingleSample	**Project Name:**	JS
Sample name:	JS-4-304 col 60% MeCN/H2O	**Operator:**	SYSTEM
Instrument:	1100HPLC	**Injection date:**	2021-04-22 15:34:32+01:00
Inj. volume:	10.000	**Location:**	83
Acq. method:	60% MeCN-H2O 45 MIN.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	0.00
Manually modified:	Manual Integration		



Signal: DAD1A,Sig=250,4 Ref=off

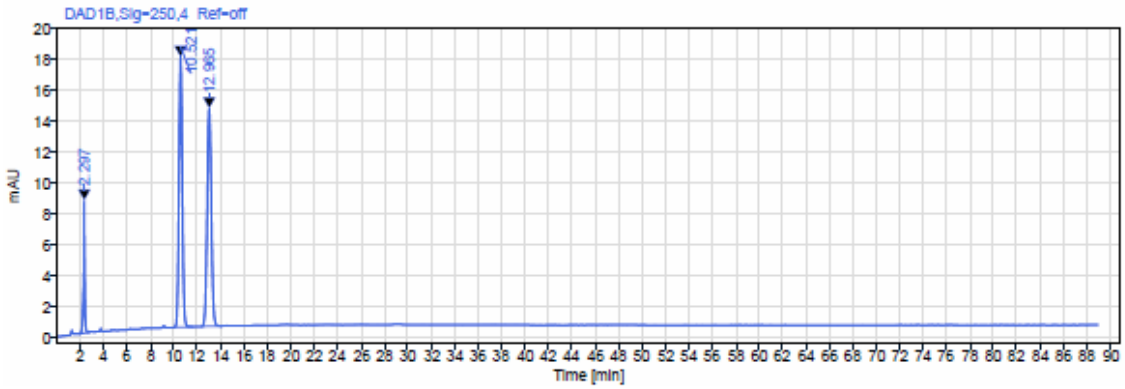
RT [min]	Type	Width [min]	Area	Height	Area%	Name
10.892	BB	1.72	4046.90	175.25	48.65	
13.718	BB	2.30	4272.14	145.40	51.35	
		Sum	8319.04			

Racemic trace

Single Injection Report

Agilent Technologies

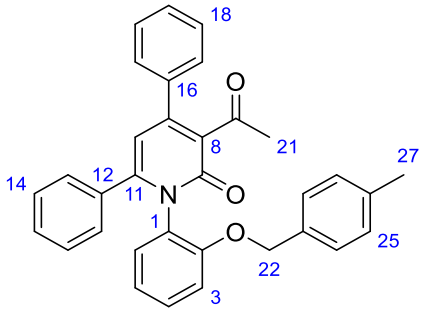
Data file:	JS #304 80C S8 96H.dx		
Sequence Name:	SingleSample	**Project Name:**	Agilent
Sample name:	JS #304 80C S8 96H	**Operator:**	Agilent
Instrument:	1220LC	**Injection date:**	2021-07-26 13:39:25+01:00
Inj. volume:	5.000	**Location:**	81
Acq. method:	(90min) atropisomer barrier measurement.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	1.00
Manually modified:	None		



Signal: DAD1B,Sig=250,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
2.297	BB	0.73	54.92	8.63	7.35	
10.521	BB	1.60	345.12	17.55	46.22	
12.965	BB	1.74	346.71	14.17	46.43	
		Sum	746.76			

3-Acetyl-1-(2-((4-methylbenzyl)oxy)phenyl)-4,6-diphenylpyridin-2(1*H*)-one (4o)

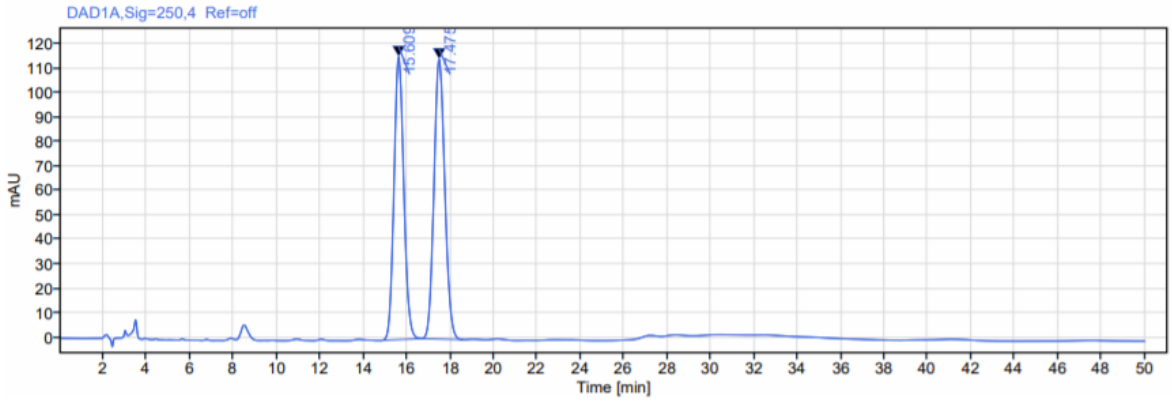


Conditions: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 17.5 min, t_R (minor) = 15.6 min.

Asymmetric trace: 5% *ee*

Single Injection Report — Agilent Technologies

Data file:	JS-4-305 col 60% MeCN-H2O20210422 135043.dx		
Sequence Name:	SingleSample	**Project Name:**	JS
Sample name:	JS-4-305 col 60% MeCN/H2O	**Operator:**	SYSTEM
Instrument:	1100HPLC	**Injection date:**	2021-04-22 16:25:40+01:00
Inj. volume:	10.000	**Location:**	84
Acq. method:	60% MeCN-H2O 45 MIN.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	0.00
Manually modified:	Manual Integration		



Signal: DAD1A,Sig=250,4 Ref=off

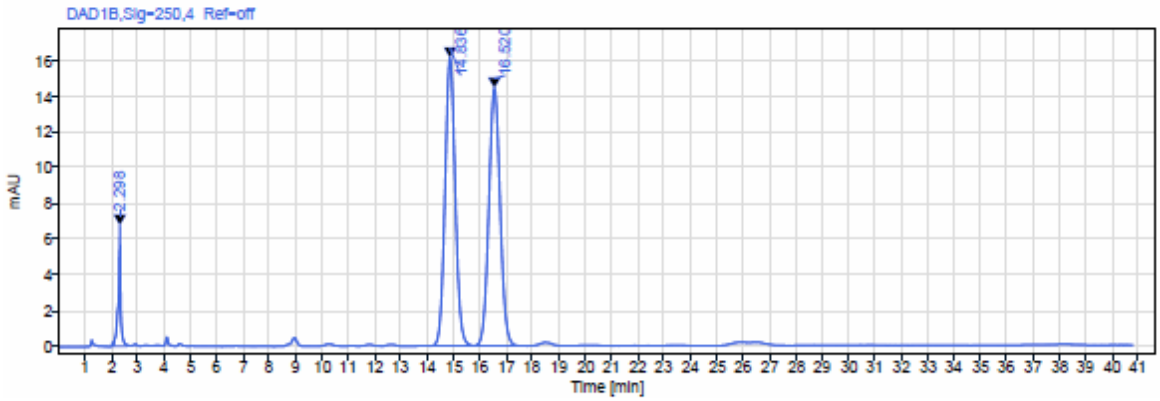
RT [min]	Type	Width [min]	Area	Height	Area%	Name
15.609	BB	1.89	3621.20	115.52	47.71	
17.475	BB	1.95	3968.93	114.33	52.29	
		Sum	7590.13			

Racemic Trace

Single Injection Report

Agilent Technologies

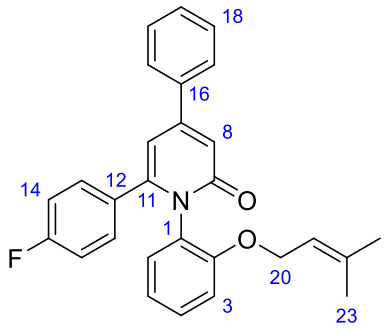
Data file:	JS #305 80C S8 96H (07.26).dx		
Sequence Name:	SingleSample	**Project Name:**	Agilent
Sample name:	JS #305 80C S8 96H (07.26)	**Operator:**	Agilent
Instrument:	1220LC	**Injection date:**	2021-07-26 15:09:40+01:00
Inj. volume:	5.000	**Location:**	82
Acq. method:	Atropisomer barrier measurement.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	1.00
Manually modified:	None		



Signal: DAD1B,Sig=250,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
2.298	VB	0.67	40.74	6.76	4.52	
14.836	BB	1.56	432.90	16.11	47.99	
16.520	BB	1.65	428.50	14.42	47.50	
		Sum	902.14			

6-(4-Fluorophenyl)-1-(2-((3-methylbut-2-en-1-yl)oxy)phenyl)-4-phenylpyridin-2(1*H*)-one (4p)

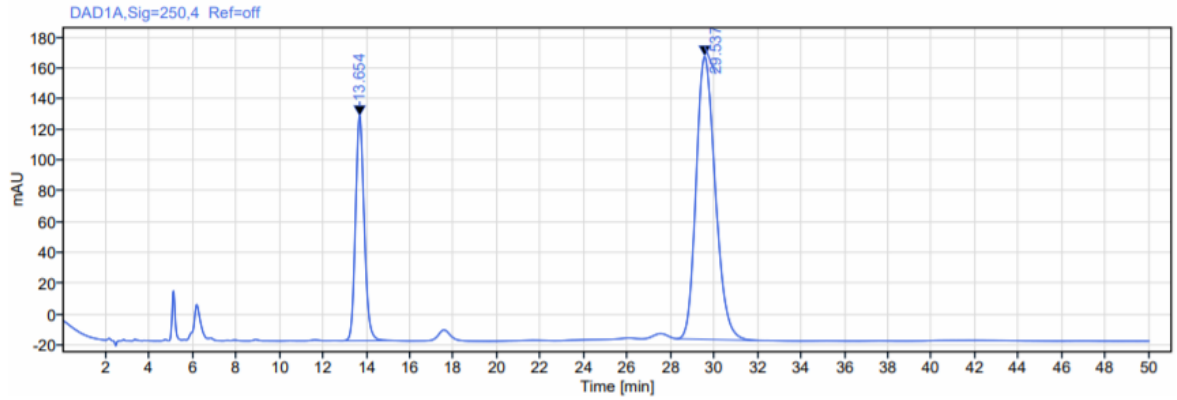


Conditions: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 29.5 min, t_R (minor) = 13.7 min.

Asymmetric trace: 47% *ee*

Single Injection Report — Agilent Technologies

Data file:	JS-5-310 col 60% MeCN-H2O20210430 153951.dx		
Sequence Name:	SingleSample	**Project Name:**	JS
Sample name:	JS-5-310 col 60% MeCN/H2O	**Operator:**	SYSTEM
Instrument:	1100HPLC	**Injection date:**	2021-04-30 16:40:37+01:00
Inj. volume:	10.000	**Location:**	16
Acq. method:	60% MeCN-H2O 45 MIN.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	0.00
Manually modified:	Manual Integration		



Signal: DAD1A,Sig=250,4 Ref=off

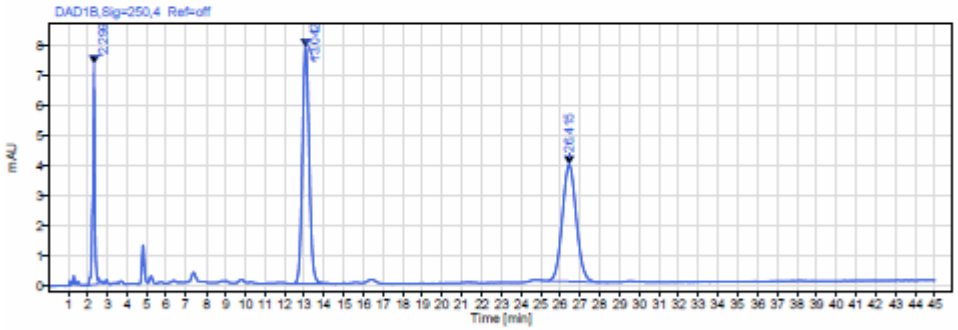
RT [min]	Type	Width [min]	Area	Height	Area%	Name
13.654	BB	2.22	4059.51	145.79	26.52	
29.537	BB	3.78	11249.32	183.73	73.48	
		Sum	15308.84			

Racemic Trace

Single Injection Report

Agilent Technologies

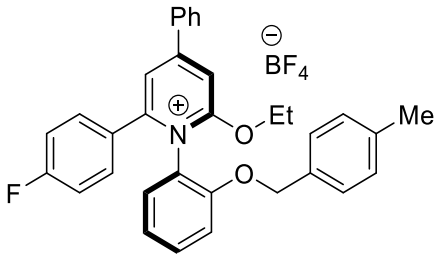
Data file:	JS #310 80C S9 70.5H.dx		
Sequence Name:	SingleSample	**Project Name:**	Agilent
Sample name:	JS #310 80C S9 70.5H	**Operator:**	Agilent
Instrument:	1220LC	**Injection date:**	2021-07-31 23:03:26+01:00
Inj. volume:	5.000	**Location:**	56
Acq. method:	(45min) Atropisomer barrier measurement.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	1.00
Manually modified:	None		



Signal: DAD1B,Sig=250,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
2.299	BV	0.51	48.37	7.34	11.30	
13.042	BB	1.71	188.50	7.86	44.03	
26.415	BB	2.63	191.24	3.88	44.67	
		Sum	428.11			

7.2 Pyridinium salt (7)

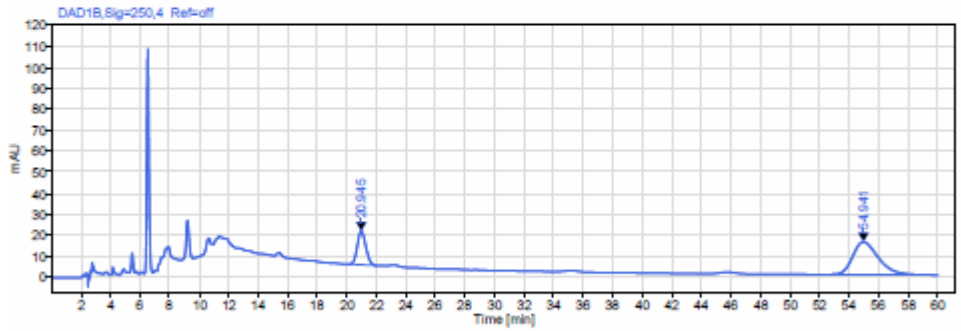


Conditions: Chiralpak OD-RH, 60% MeCN/H_2O, 0.75 mL/min, λ = 250 nm, t_R (major) = 54.9 min, t_R (minor) = 20.9 min.

Asymmetric trace: 49% *ee*

Single Injection Report — Agilent Technologies

Data file:	pck 3.79.dx		
Sequence Name:	SingleSample	Project Name:	Agilent
Sample name:	pck 3.79	Operator:	Agilent
Instrument:	1220LC	Injection date:	2021-07-08 07:26:05+01:00
Inj. volume:	5.000	Location:	91
Acq. method:	Atropisomer barrier measurement.amx	Type:	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	Sample amount:	1.00
Manually modified:	Manual Integration		



Signal: DAD1B,Sig=250,4 Ref=off

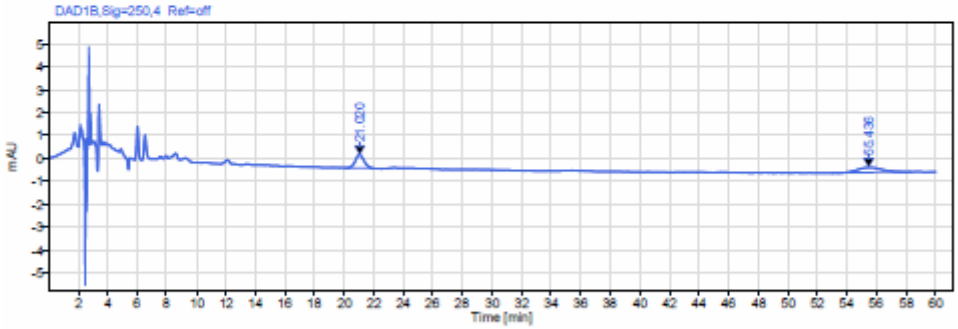
RT [min]	Type	Width [min]	Area	Height	Area%	Name
20.945	BB	1.84	639.57	16.07	25.56	
54.941	BB	6.15	1862.37	15.55	74.44	
		Sum	2501.94			

Racemic Trace

Single Injection Report

Agilent Technologies

Data file:	pck 3.80.dx		
Sequence Name:	SingleSample	**Project Name:**	Agilent
Sample name:	pck 3.80	**Operator:**	Agilent
Instrument:	1220LC	**Injection date:**	2021-07-08 08:27:03+01:00
Inj. volume:	5.000	**Location:**	92
Acq. method:	Atropisomer barrier measurement.amx	**Type:**	Sample
Processing method:	3D UV Quantitative_DefaultMethod.pmx	**Sample amount:**	1.00
Manually modified:	Manual Integration		

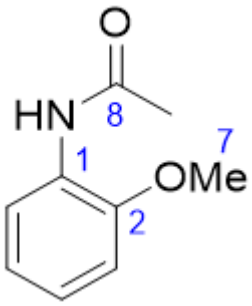


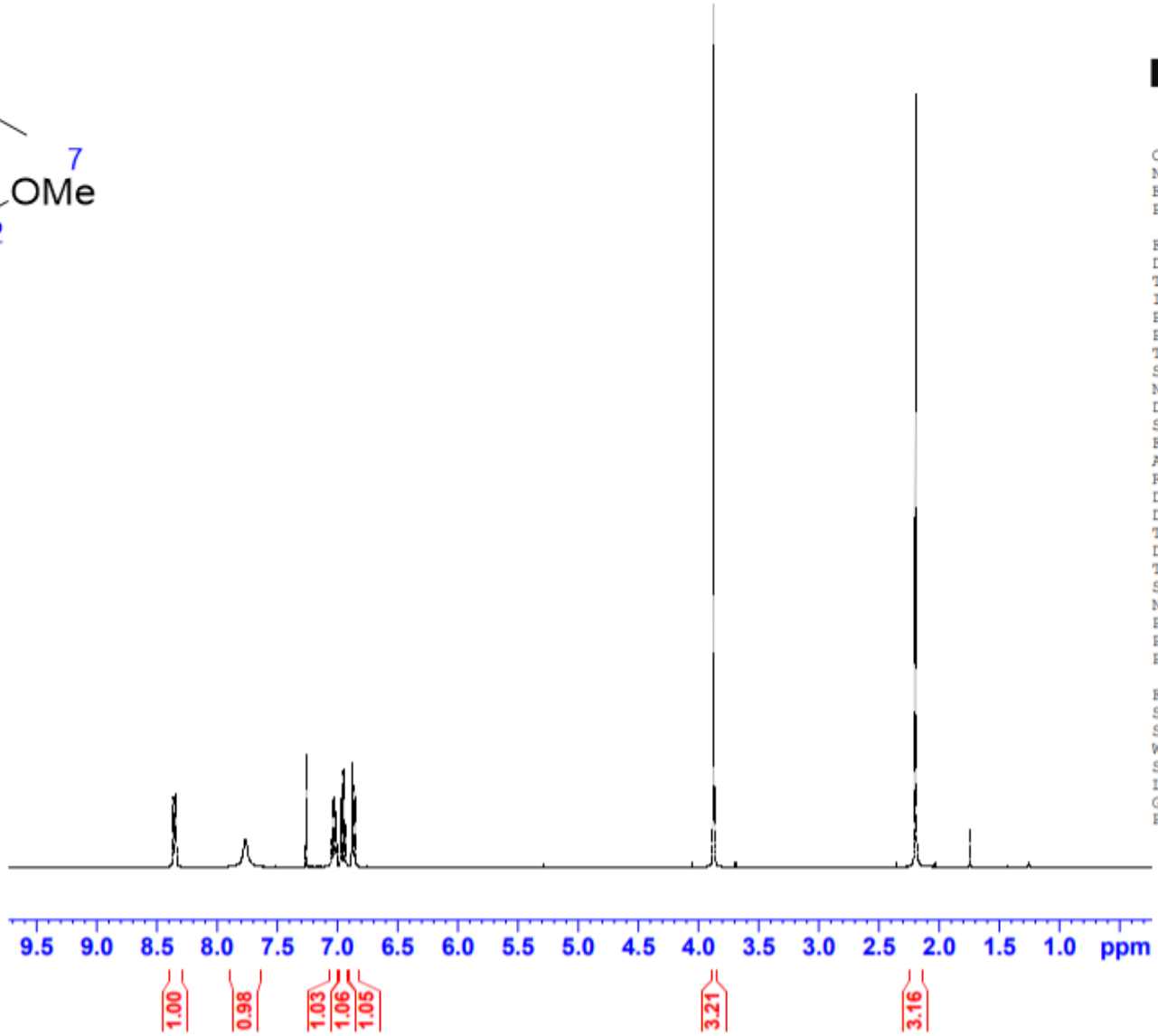
Signal: DAD1B,Sig=250,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
21.020	MM m	0.49	24.36	0.59	49.70	
55.436	MM m	1.29	24.65	0.22	50.30	
		Sum	49.01			

8 NMR data

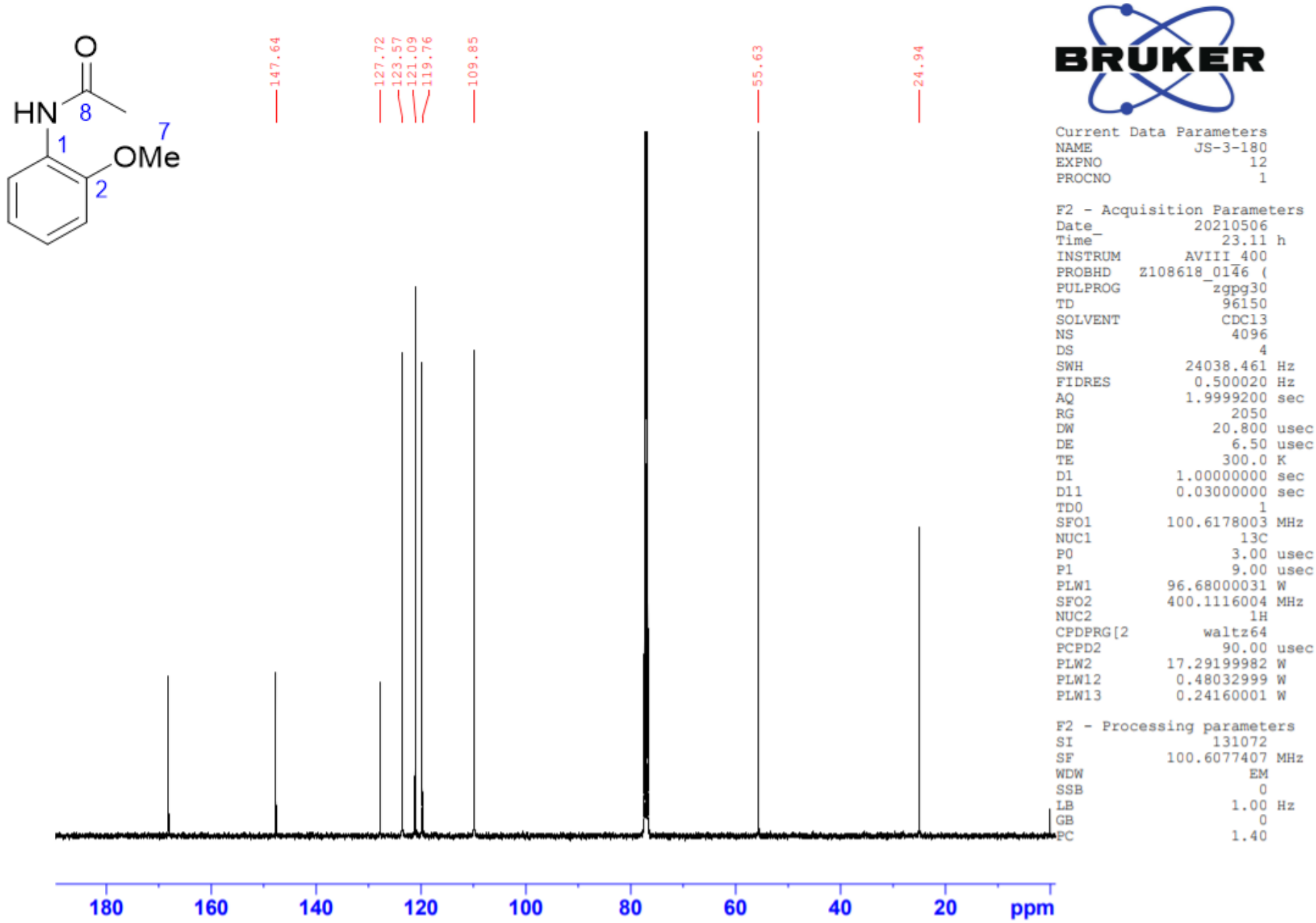
1a - ^{1}H NMR, 400 MHz, $CDCl_3$





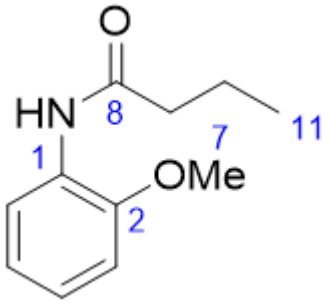

```
Current Data Parameters
NAME                  JS-3-180
EXPNO                       14
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20210506
Time                     23.30 h
INSTRUM               AVIII_400
PROBHD    Z108618_0146 (
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                   8223.685 Hz
FIDRES                0.250967 Hz
AQ                    3.9845889 sec
RG                         144
DW                      60.800 usec
DE                      17.42 usec
TE                      300.0 K
D1                 1.00000000 sec
TD0                          1
SFO1               400.1124708 MHz
NUC1                        1H
P0                       5.00 usec
P1                      15.00 usec
PLW1               17.29199982 W

F2 - Processing parameters
SI                       32768
SF                 400.1100098 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```

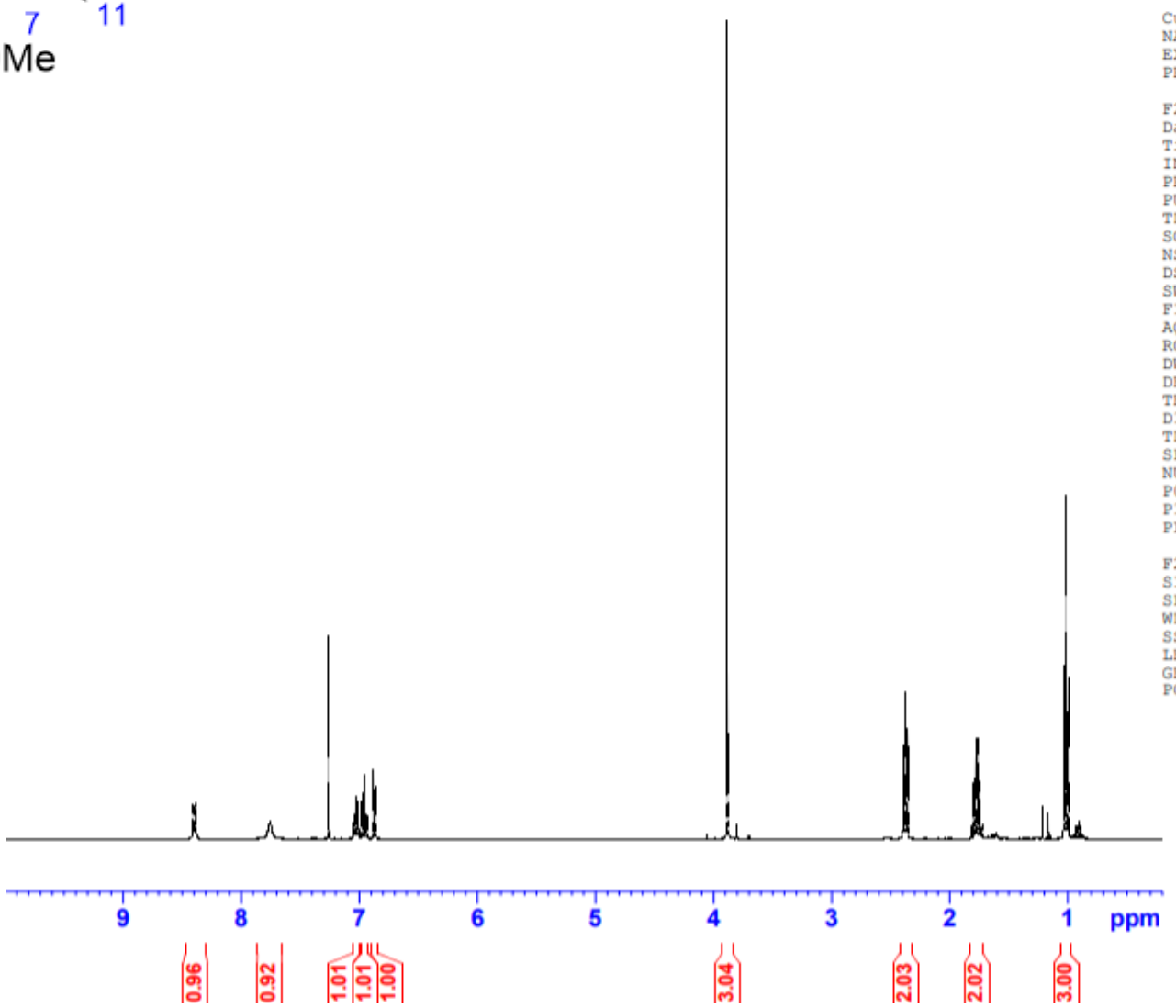
1a - ^{13}C NMR, 101 MHz, $CDCl_3$




```
Current Data Parameters
NAME                  JS-3-180
EXPNO                       12
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20210506
Time                     23.11 h
INSTRUM               AVIII 400
PROBHD   Z108618_0146 (
PULPROG                 zgpg30
TD                       96150
SOLVENT                  CDCl3
NS                        4096
DS                           4
SWH                  24038.461 Hz
FIDRES                0.500020 Hz
AQ                    1.9999200 sec
RG                        2050
DW                      20.800 usec
DE                       6.50 usec
TE                      300.0 K
D1                    1.00000000 sec
D11                   0.03000000 sec
TD0                          1
SFO1                100.6178003 MHz
NUC1                        13C
P0                       3.00 usec
P1                       9.00 usec
PLW1                96.68000031 W
SFO2                400.1116004 MHz
NUC2                        1H
CPDPRG[2                waltz64
PCPD2                   90.00 usec
PLW2                17.29199982 W
PLW12                0.48032999 W
PLW13                0.24160001 W

F2 - Processing parameters
SI                       131072
SF                  100.6077407 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

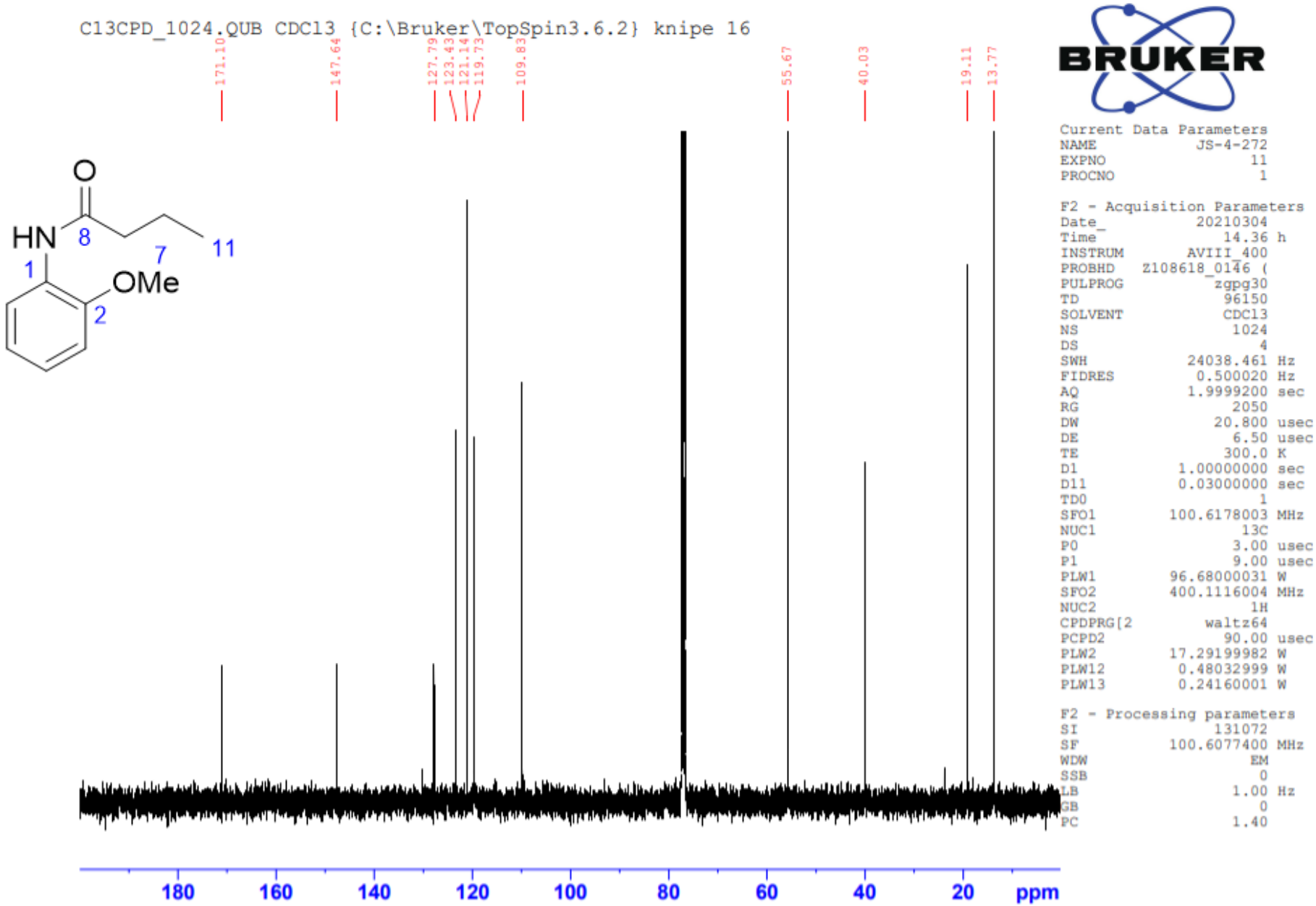
1b - ^{1}H NMR, 400 MHz, $CDCl_3$



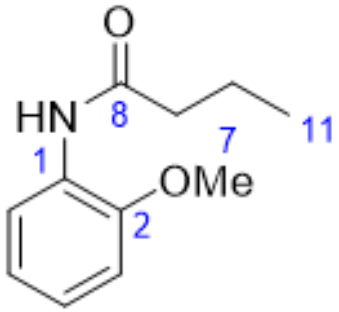


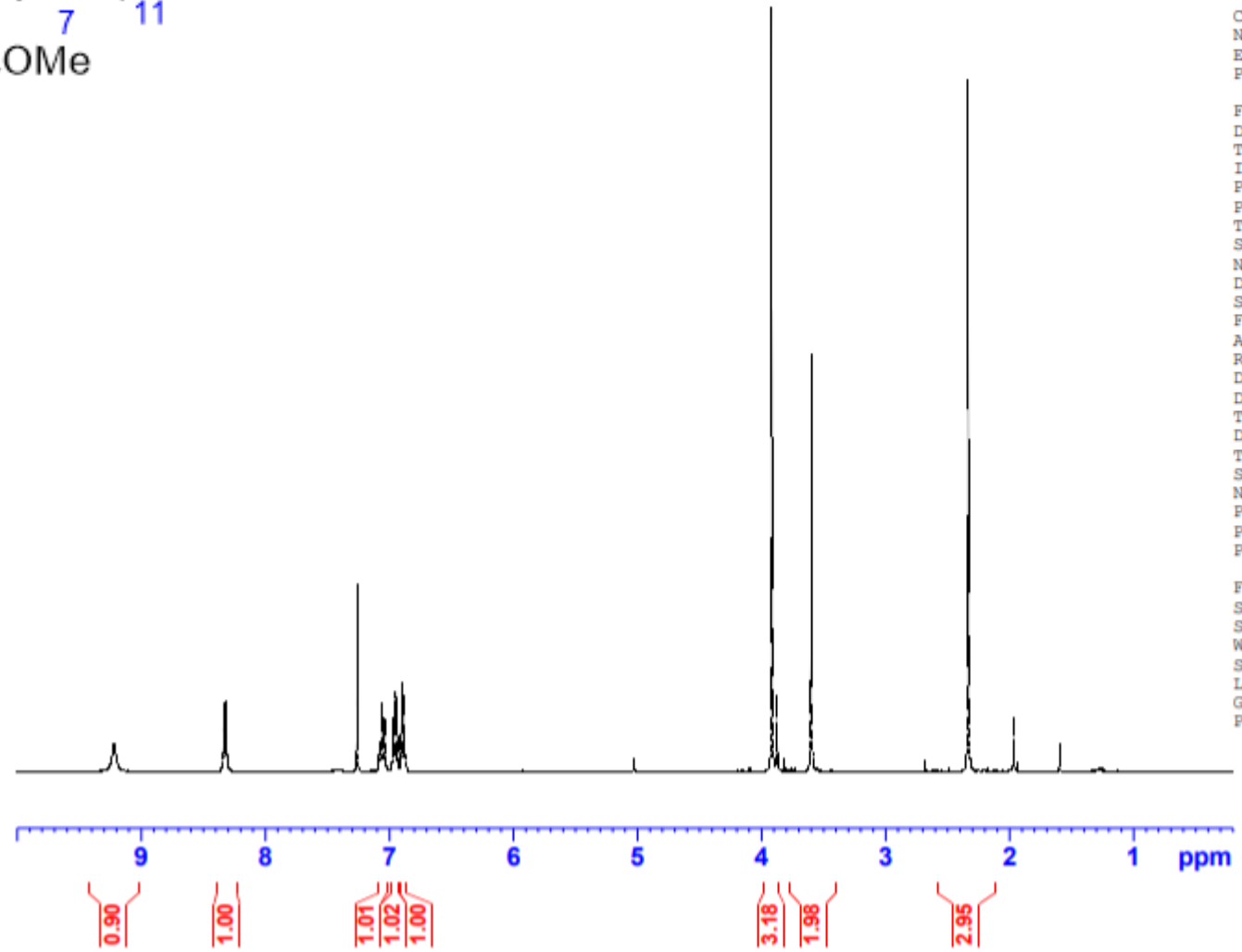

```
Current Data Parameters
NAME                  JS-4-272
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20210304
Time                     11.43 h
INSTRUM               AVIII_400
PROBHD    Z108618_0146 (
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                   8223.685 Hz
FIDRES                0.250967 Hz
AQ                    3.9845889 sec
RG                         256
DW                      60.800 usec
DE                      17.42 usec
TE                      300.0 K
D1                    1.00000000 sec
TD0                          1
SFO1                400.1124708 MHz
NUC1                        1H
P0                       5.00 usec
P1                      15.00 usec
PLW1                17.29199982 W

F2 - Processing parameters
SI                          32768
SF                  400.1100099 MHz
WDW                            EM
SSB                             0
LB                           0.30 Hz
GB                              0
PC                           1.00
```

1b - ^{13}C NMR, 101 MHz, $CDCl_3$



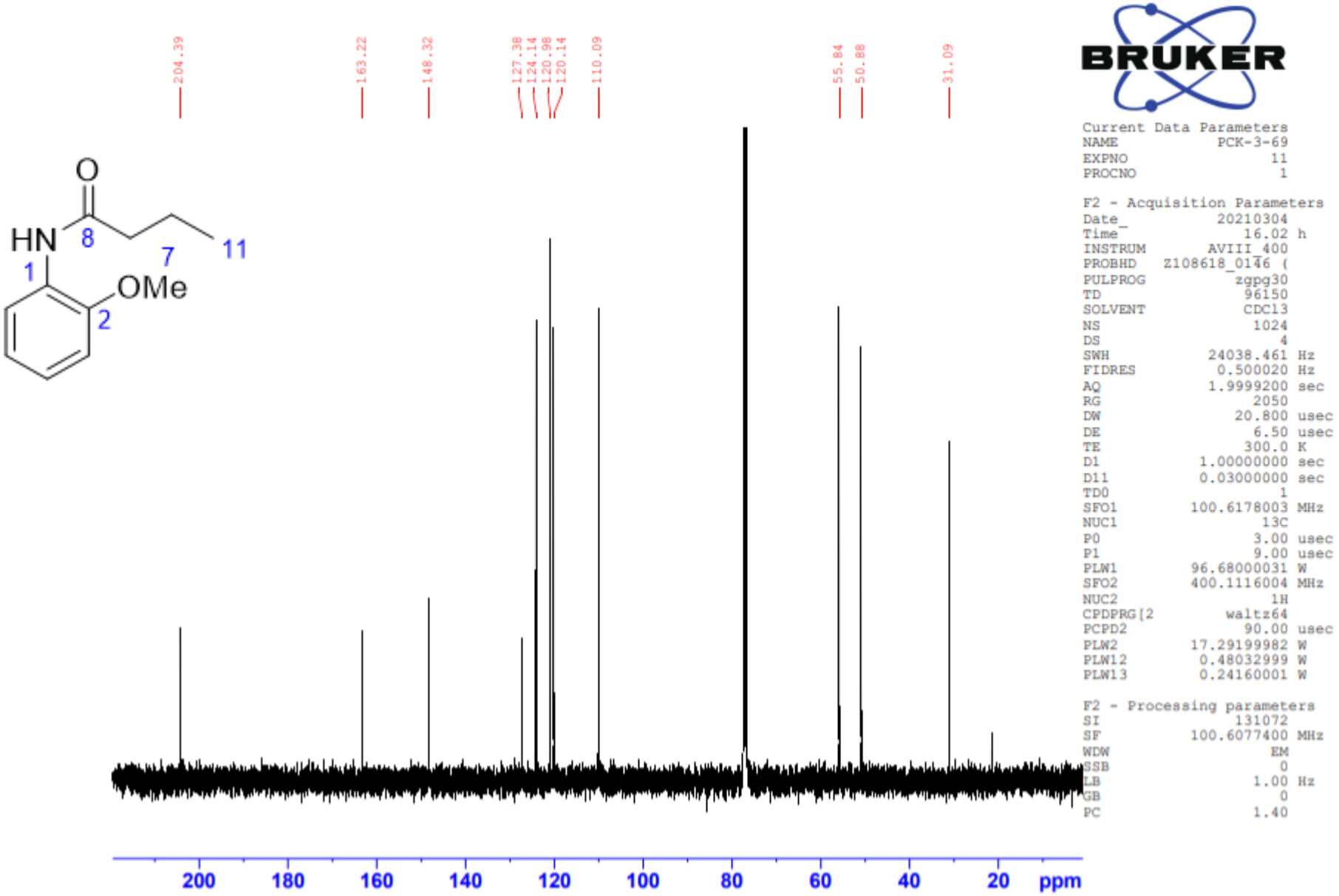
1c - ^{1}H NMR, 400 MHz, $CDCl_3$





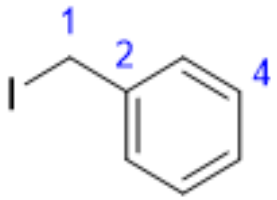

```
Current Data Parameters
NAME                  PCK-3-69
EXPNO                       12
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20210304
Time                     16.07 h
INSTRUM               AVIII_400
PROBHD    Z108618_0146 (
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                   8223.685 Hz
FIDRES                0.250967 Hz
AQ                    3.9845889 sec
RG                         287
DW                       60.800 usec
DE                       17.42 usec
TE                       300.0 K
D1                    1.00000000 sec
TD0                          1
SFO1                400.1124708 MHz
NUC1                        1H
P0                        5.00 usec
P1                       15.00 usec
PLW1                17.29199982 W

F2 - Processing parameters
SI                       32768
SF                  400.1100100 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```

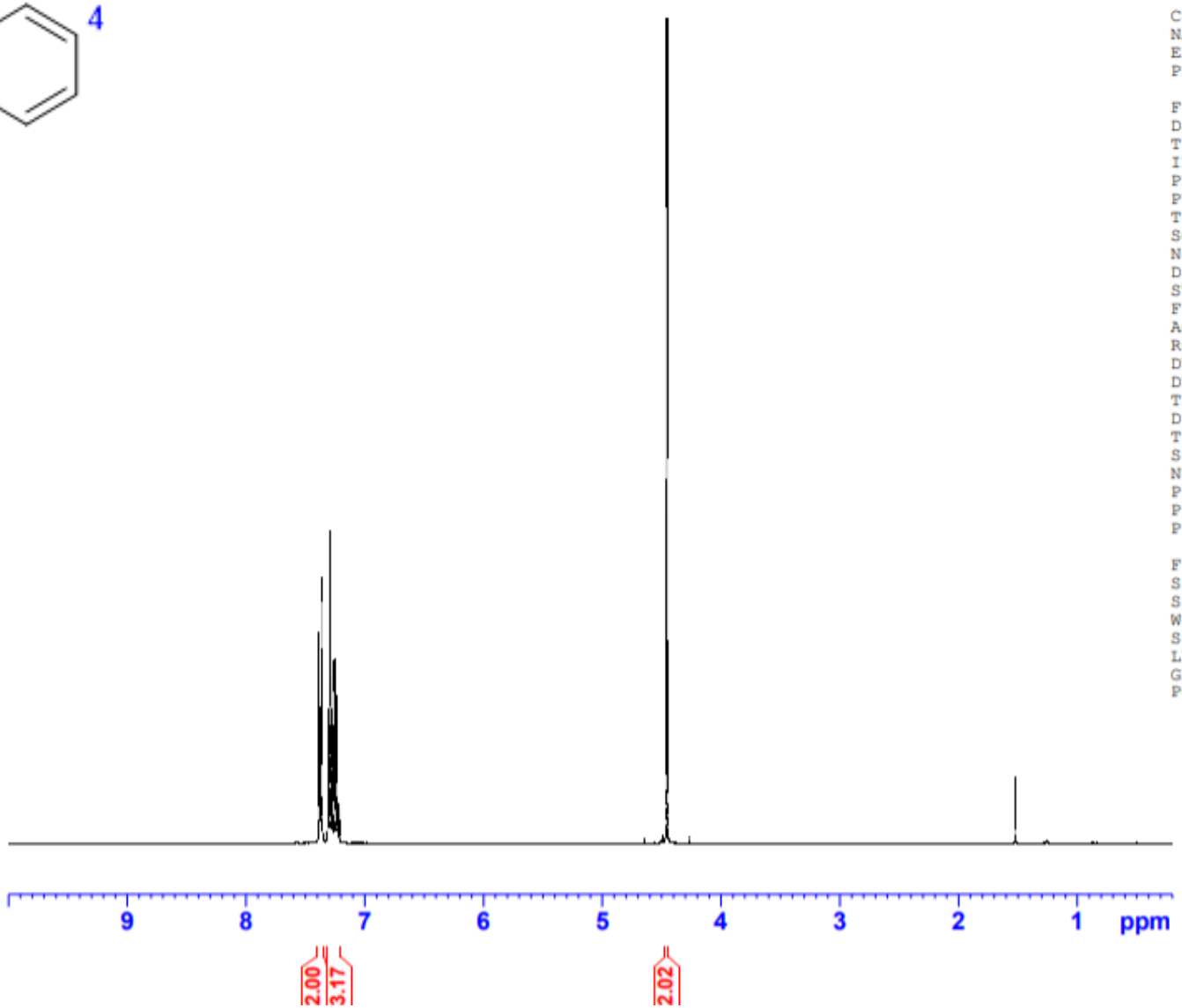
1c - ^{13}C NMR, 101 MHz, $CDCl_3$




```
Current Data Parameters
NAME            PCK-3-69
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210304
Time              16.02 h
INSTRUM        AVIII 400
PROBHD    Z108618_0146 (
PULPROG          zgpg30
TD                96150
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.500020 Hz
AQ             1.9999200 sec
RG                 2050
DW               20.800 usec
DE                6.50 usec
TE               300.0 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                   1
SFO1         100.6178003 MHz
NUC1               13C
P0                3.00 usec
P1                9.00 usec
PLW1          96.68000031 W
SFO2         400.1116004 MHz
NUC2               1H
CPDPRG[2       waltz64
PCPD2            90.00 usec
PLW2          17.29199982 W
PLW12          0.48032999 W
PLW13          0.24160001 W

F2 - Processing parameters
SI               131072
SF           100.6077400 MHz
WDW                EM
SSB                   0
LB                1.00 Hz
GB                    0
PC                1.40
```

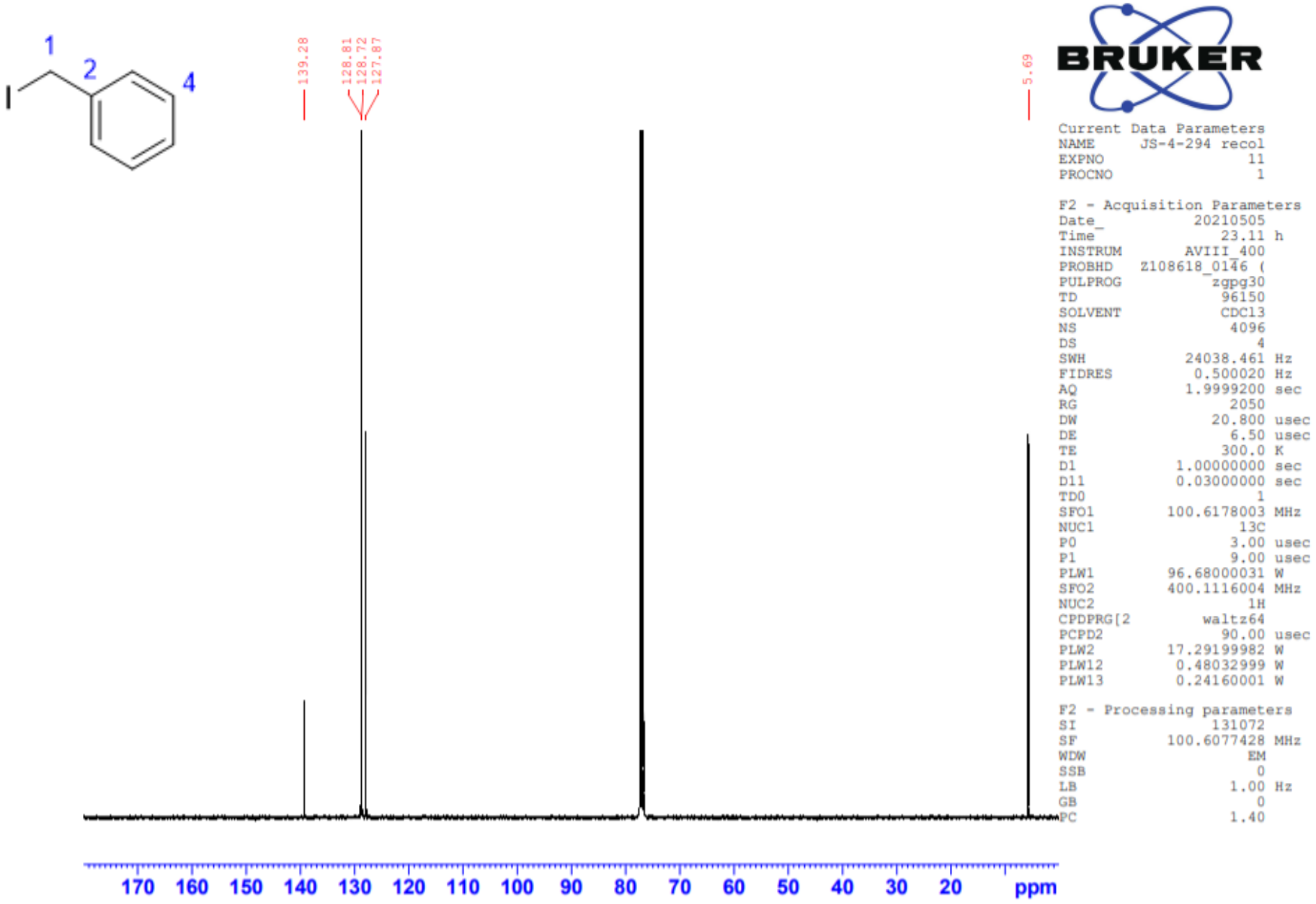
S1 - ^{1}H NMR, 400 MHz, $CDCl_3$



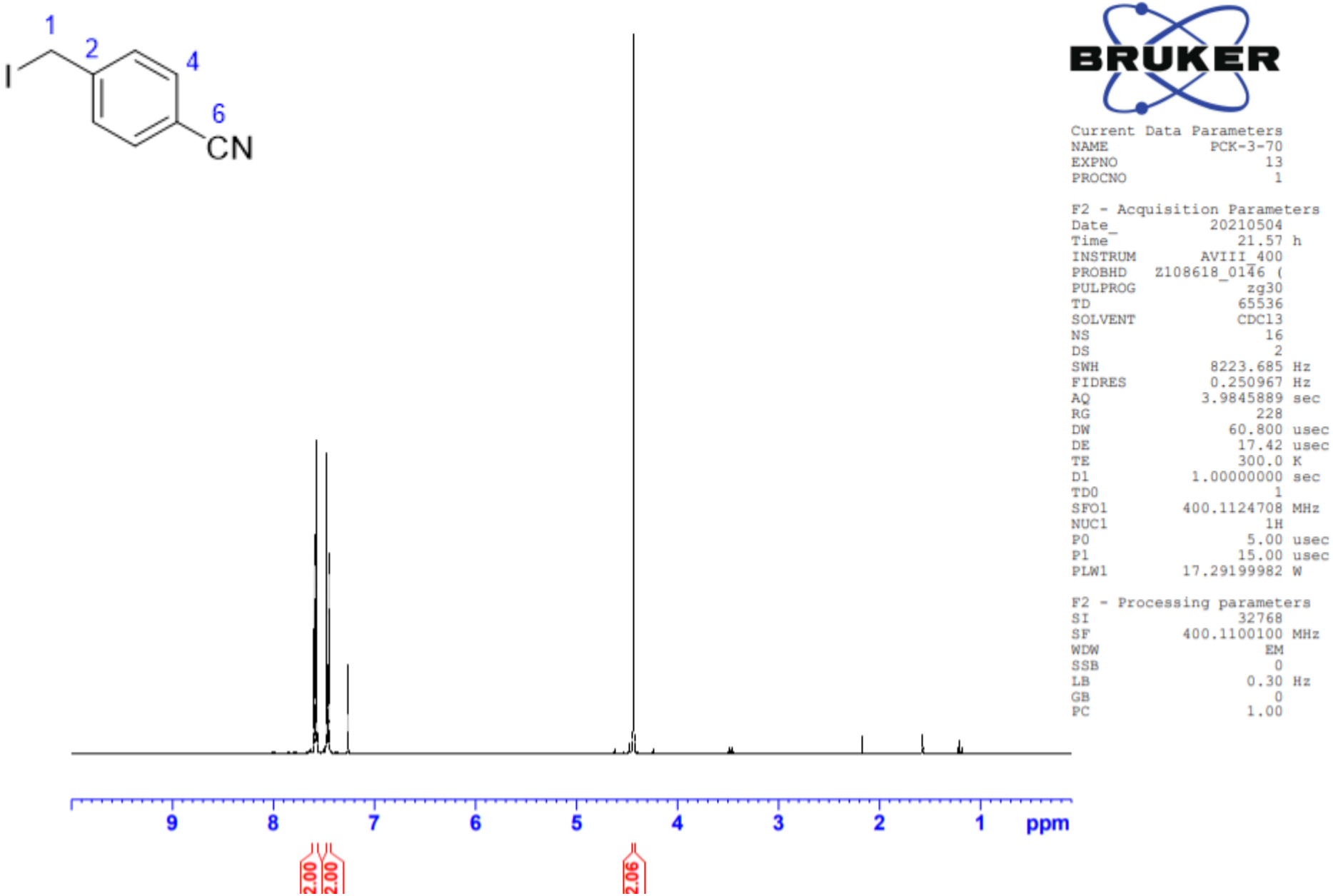



```
Current Data Parameters
NAME      JS-4-294 recol
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20210505
Time              14.38 h
INSTRUM       AVIII_400
PROBHD   Z108618_0146 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ              3.9845889 sec
RG                   256
DW                60.800 usec
DE                17.42 usec
TE                300.0 K
D1           1.00000000 sec
TD0                    1
SFO1        400.1124708 MHz
NUC1                  1H
P0                 5.00 usec
P1                15.00 usec
PLW1        17.29199982 W

F2 - Processing parameters
SI                 32768
SF          400.1100135 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

S1 - ^{13}C NMR, 101 MHz, $CDCl_3$

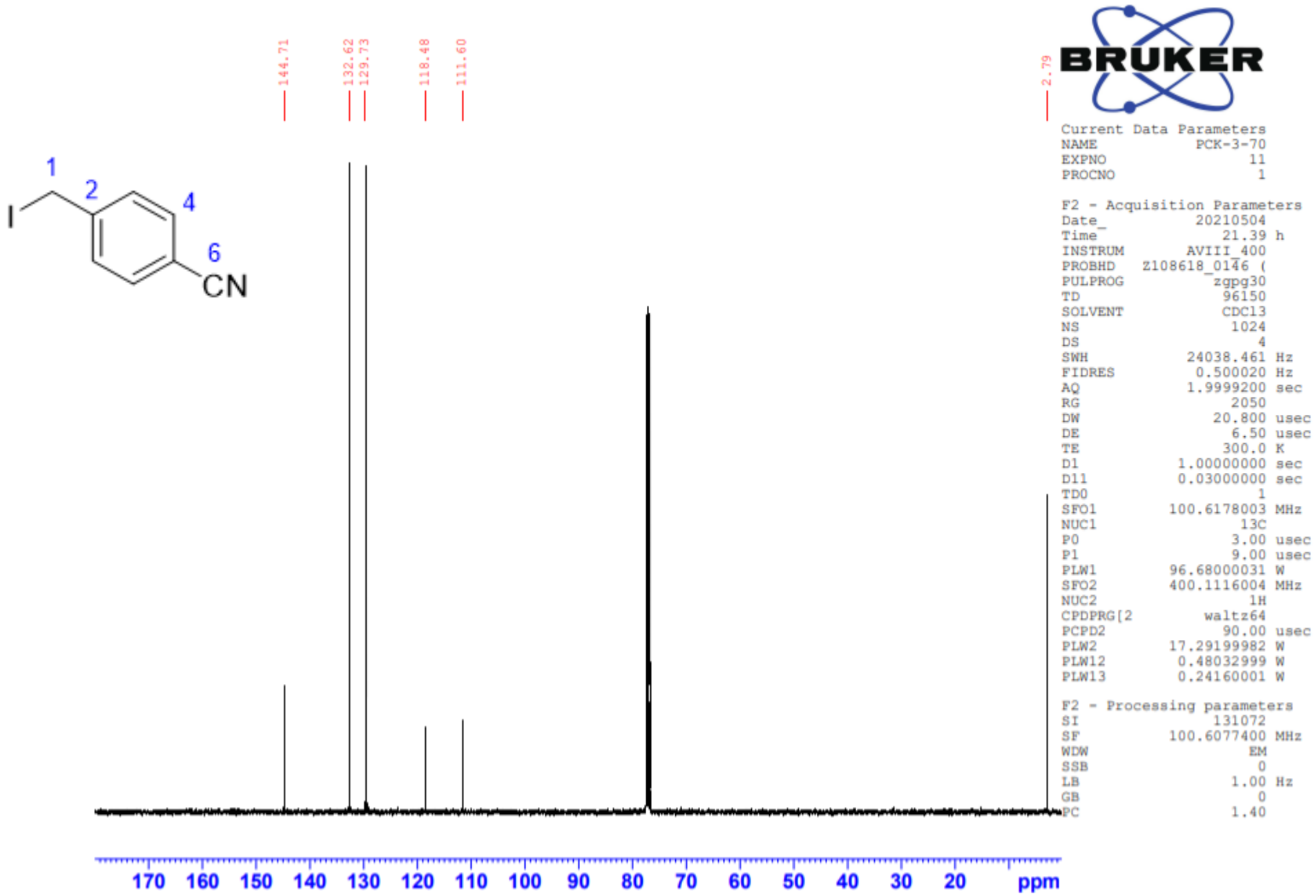


S2 - ^{1}H NMR, 400 MHz, $CDCl_3$




```
Current Data Parameters
NAME                 PCK-3-70
EXPNO                      13
PROCNO                      1

F2 - Acquisition Parameters
Date_               20210504
Time                   21.57 h
INSTRUM             AVIII 400
PROBHD    Z108618_0146 (
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 8223.685 Hz
FIDRES              0.250967 Hz
AQ                  3.9845889 sec
RG                       228
DW                    60.800 usec
DE                    17.42 usec
TE                    300.0 K
D1                  1.00000000 sec
TD0                        1
SFO1             400.1124708 MHz
NUC1                      1H
P0                     5.00 usec
P1                    15.00 usec
PLW1             17.29199982 W

F2 - Processing parameters
SI                     32768
SF               400.1100100 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                      1.00
```

S2 - ^{13}C NMR, 101 MHz, $CDCl_3$



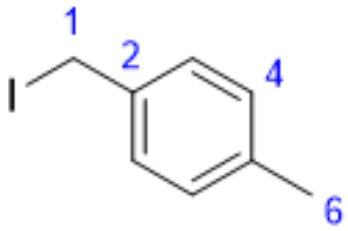
BRUKER

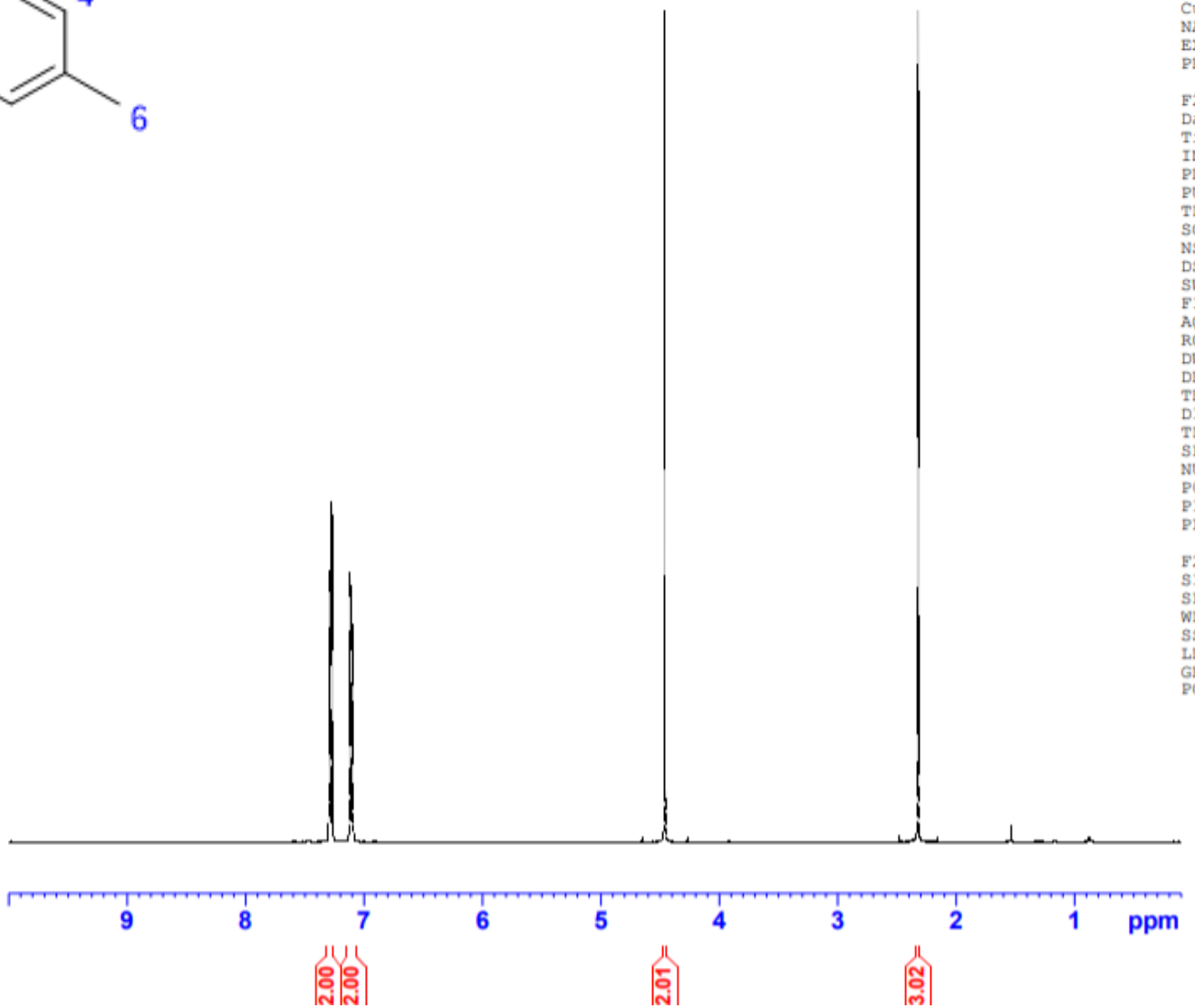
Current Data Parameters
NAME PCK-3-70
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210504
Time 21.39 h
INSTRUM AVIII_400
PROBHD Z108618_0146 (
PULPROG zgpg30
TD 96150
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.500020 Hz
AQ 1.9999200 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6178003 MHz
NUC1 13C
P0 3.00 usec
P1 9.00 usec
PLW1 96.68000031 W
SFO2 400.1116004 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 17.29199982 W
PLW12 0.48032999 W
PLW13 0.24160001 W

F2 - Processing parameters
SI 131072
SF 100.6077400 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

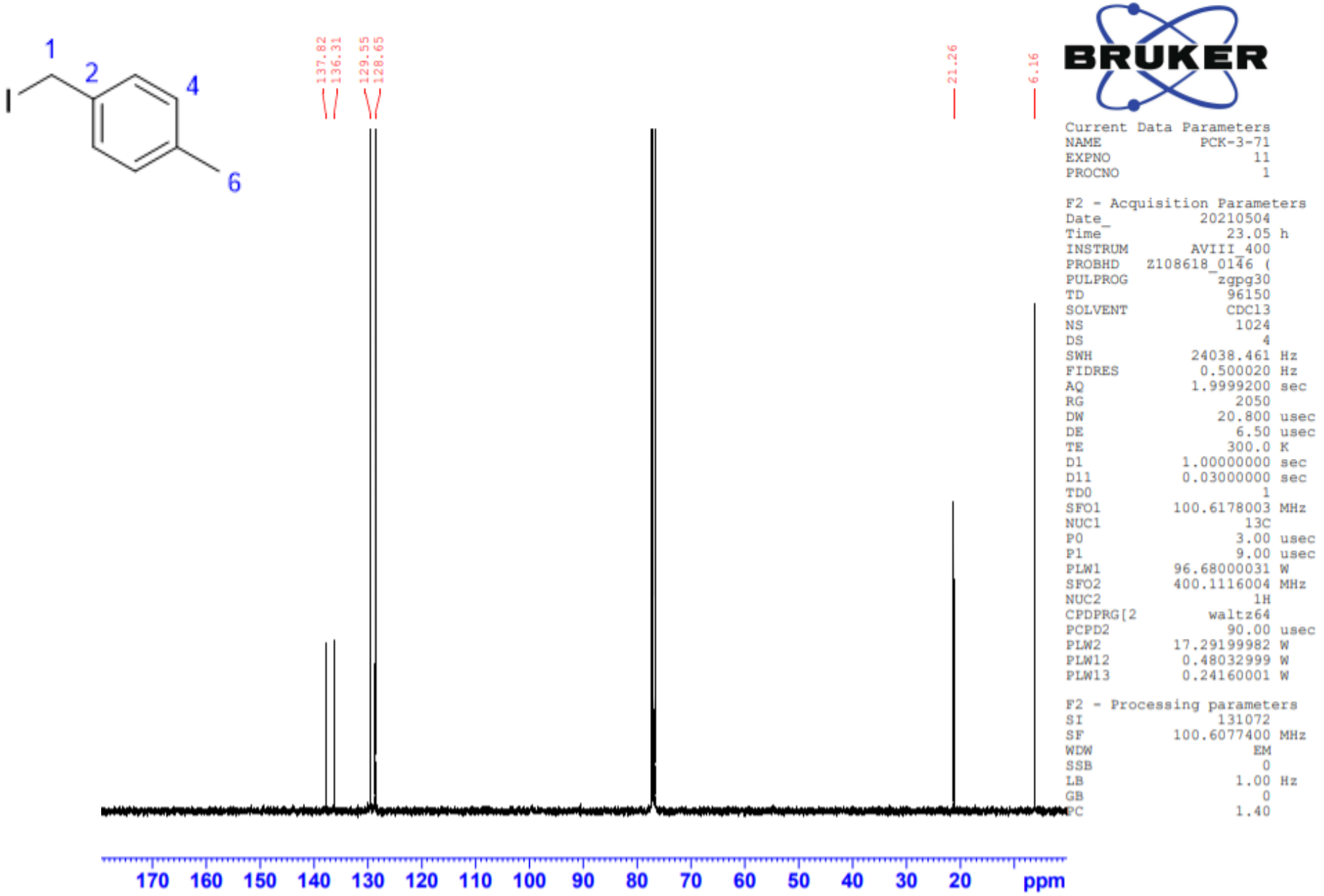
S3 - ^{1}H NMR, 400 MHz, $CDCl_3$





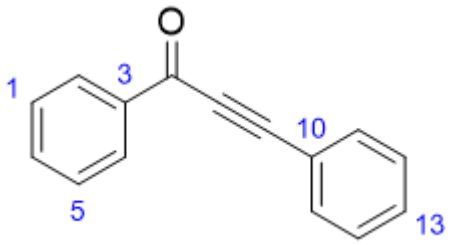

```
Current Data Parameters
NAME                  PCK-3-71
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20210504
Time                     16.25 h
INSTRUM               AVIII_400
PROBHD    Z108618_0146 (
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                   8223.685 Hz
FIDRES                0.250967 Hz
AQ                    3.9845889 sec
RG                         161
DW                      60.800 usec
DE                      17.42 usec
TE                      300.0 K
D1                  1.00000000 sec
TD0                          1
SFO1                400.1124708 MHz
NUC1                        1H
P0                       5.00 usec
P1                      15.00 usec
PLW1                17.29199982 W

F2 - Processing parameters
SI                       32768
SF                  400.1100097 MHz
WDW                         EM
SSB                          0
LB                       0.30 Hz
GB                           0
PC                       1.00
```

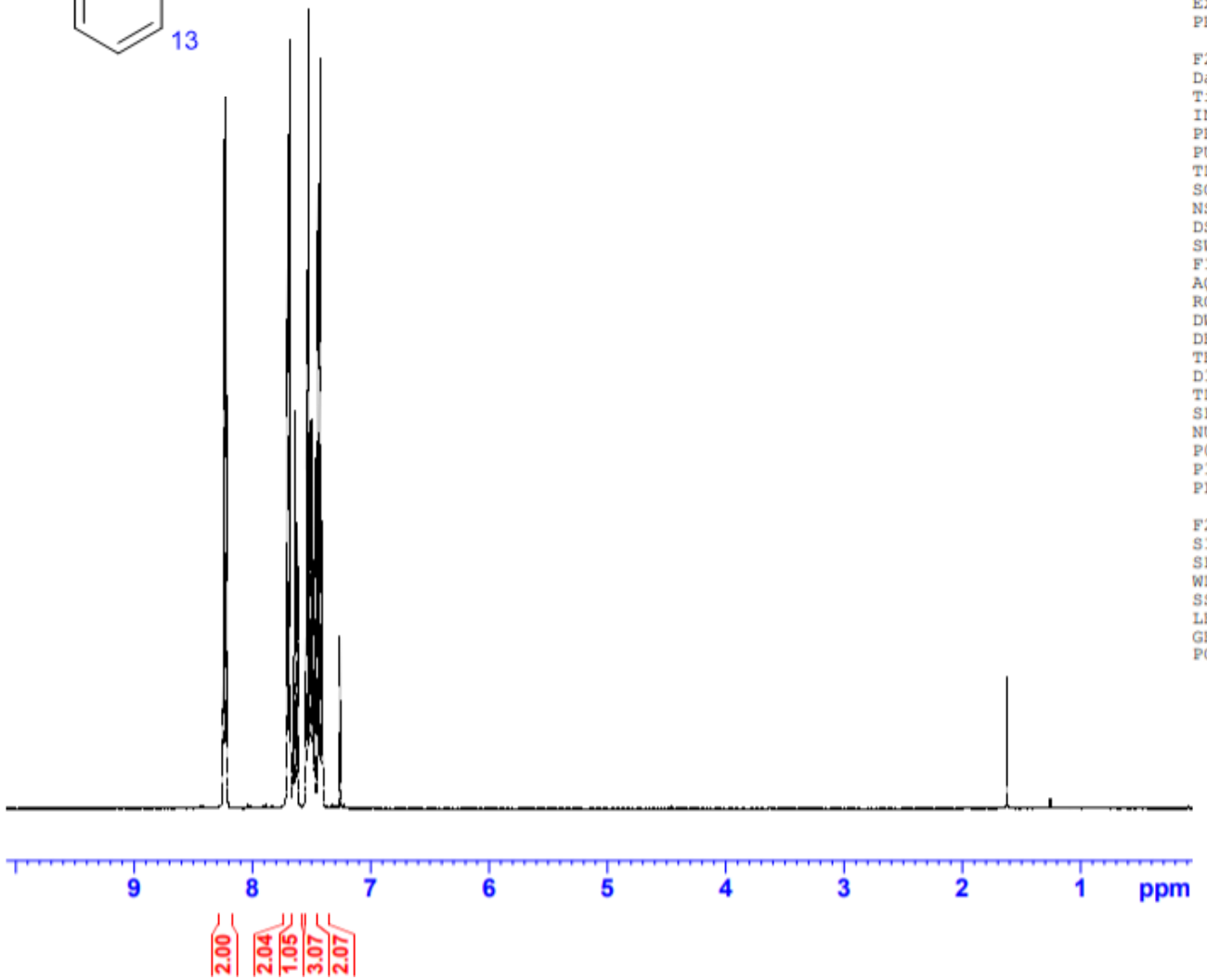
S3 - ^{13}C NMR, 101 MHz, $CDCl_3$




```
Current Data Parameters
NAME                 PCK-3-71
EXPNO                      11
PROCNO                      1

F2 - Acquisition Parameters
Date_               20210504
Time                   23.05 h
INSTRUM              AVIII_400
PROBHD     Z108618_0146 (
PULPROG                zgpg30
TD                      96150
SOLVENT                 CDCl3
NS                       1024
DS                          4
SWH                 24038.461 Hz
FIDRES                0.500020 Hz
AQ                    1.9999200 sec
RG                       2050
DW                     20.800 usec
DE                      6.50 usec
TE                     300.0 K
D1                 1.00000000 sec
D11                0.03000000 sec
TD0                         1
SFO1              100.6178003 MHz
NUC1                      13C
P0                     3.00 usec
P1                     9.00 usec
PLW1               96.68000031 W
SFO2              400.1116004 MHz
NUC2                      1H
CPDPRG[2              waltz64
PCPD2                  90.00 usec
PLW2               17.29199982 W
PLW12               0.48032999 W
PLW13               0.24160001 W

F2 - Processing parameters
SI                       131072
SF                100.6077400 MHz
WDW                        EM
SSB                         0
LB                     1.00 Hz
GB                          0
PC                     1.40
```

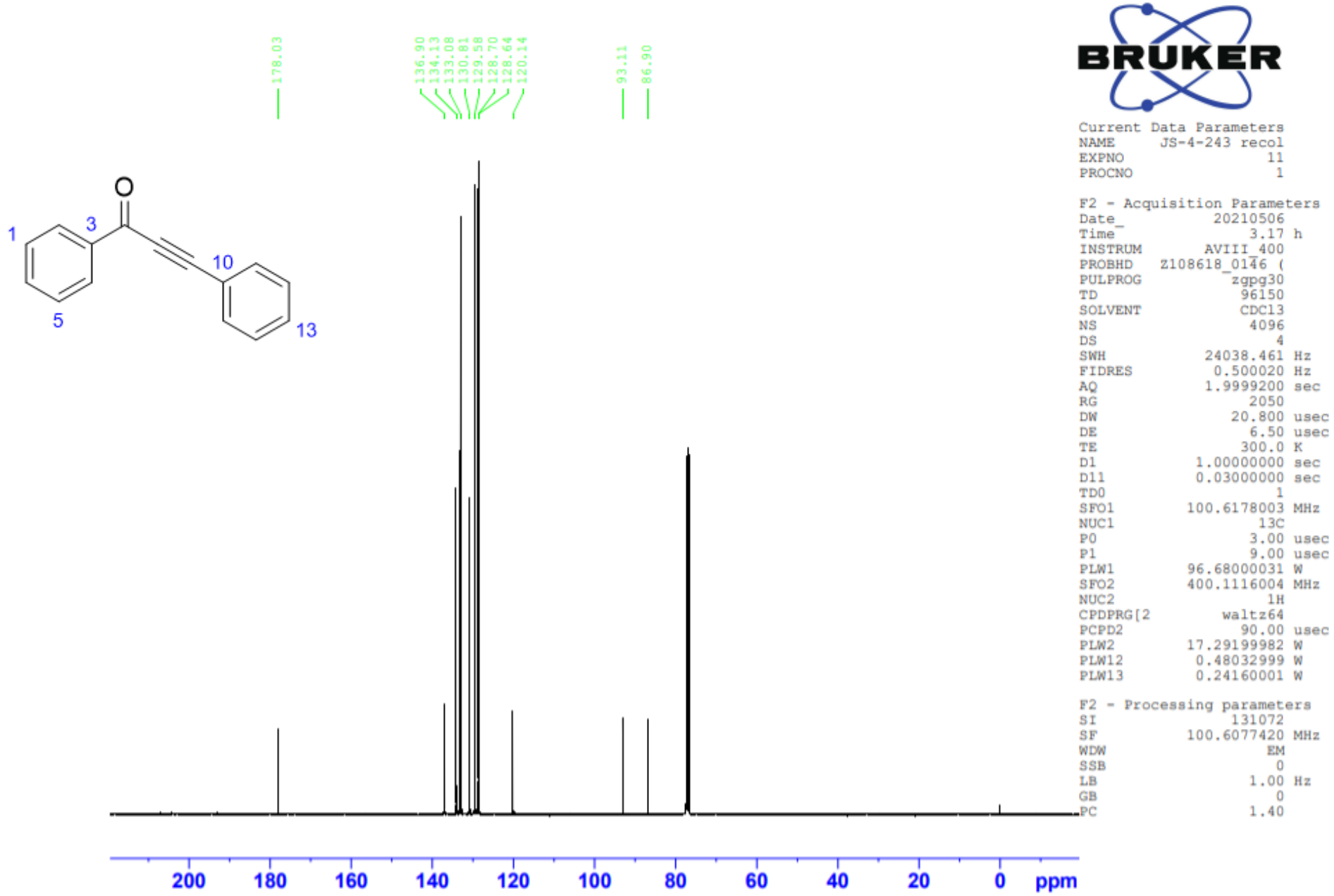
2a - ^{1}H NMR, 400 MHz, $CDCl_3$





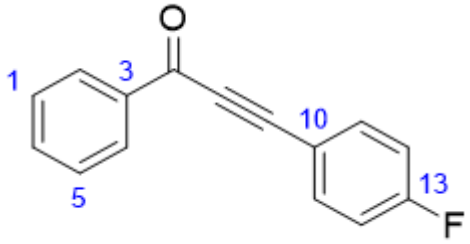

```
Current Data Parameters
NAME      JS-4-243 recol
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210505
Time              16.17 h
INSTRUM        AVIII 400
PROBHD   Z108618_0146 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ              3.9845889 sec
RG                   128
DW                60.800 usec
DE                17.42 usec
TE                300.0 K
D1           1.00000000 sec
TD0                    1
SFO1        400.1124708 MHz
NUC1                  1H
P0                 5.00 usec
P1                15.00 usec
PLW1        17.29199982 W

F2 - Processing parameters
SI                 32768
SF          400.1100099 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```

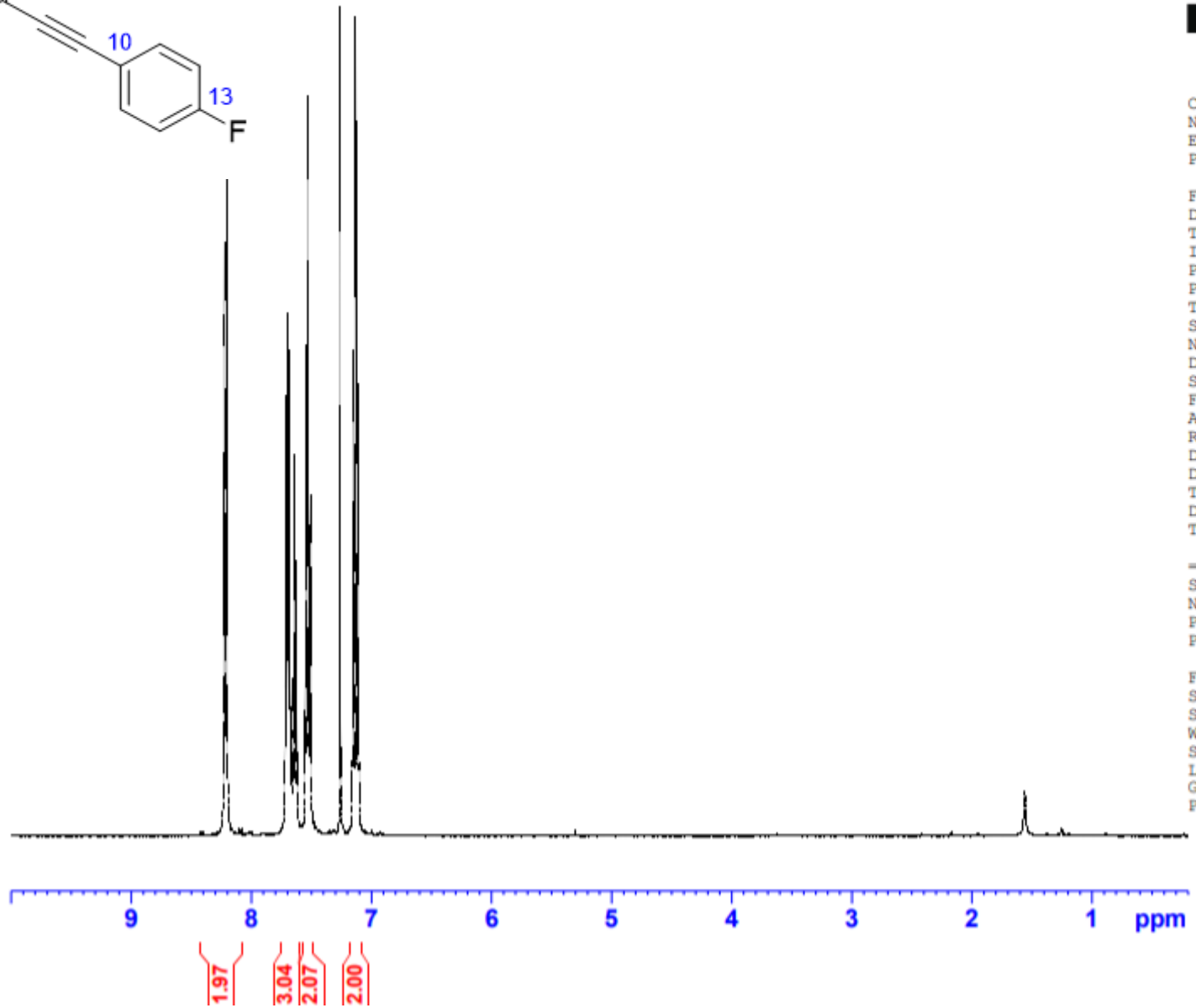
2a - ^{13}C NMR, 101 MHz, $CDCl_3$




```
Current Data Parameters
NAME      JS-4-243 recol
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210506
Time               3.17 h
INSTRUM       AVIII_400
PROBHD   Z108618_0146 (
PULPROG          zgpg30
TD                96150
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           24038.461 Hz
FIDRES         0.500020 Hz
AQ             1.9999200 sec
RG                 2050
DW               20.800 usec
DE                6.50 usec
TE               300.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1        100.6178003 MHz
NUC1                13C
P0                3.00 usec
P1                9.00 usec
PLW1        96.68000031 W
SFO2        400.1116004 MHz
NUC2                1H
CPDPRG[2        waltz64
PCPD2            90.00 usec
PLW2        17.29199982 W
PLW12        0.48032999 W
PLW13        0.24160001 W

F2 - Processing parameters
SI                131072
SF          100.6077420 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

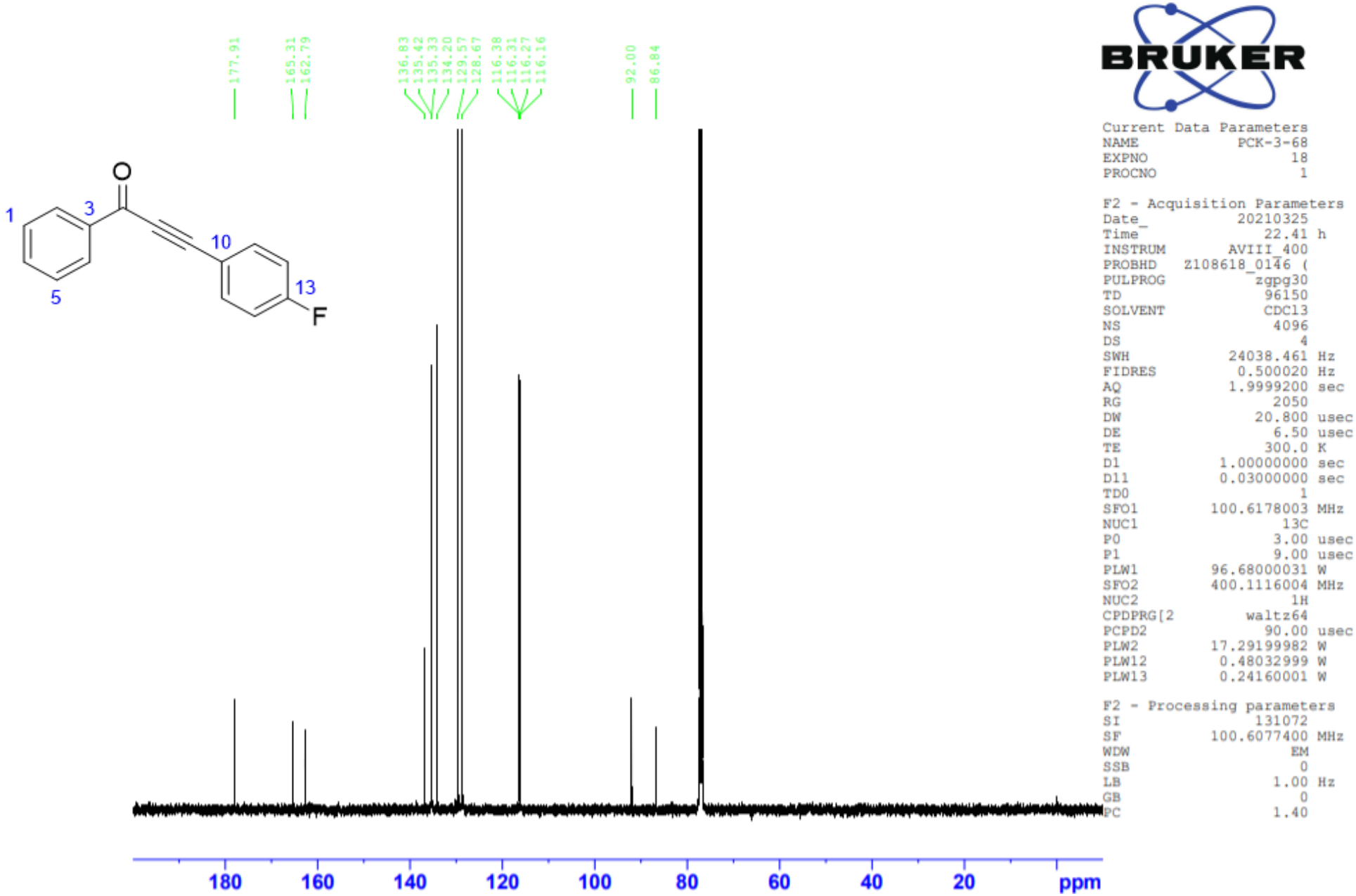
2b - ^{1}H NMR, 400 MHz, $CDCl_3$





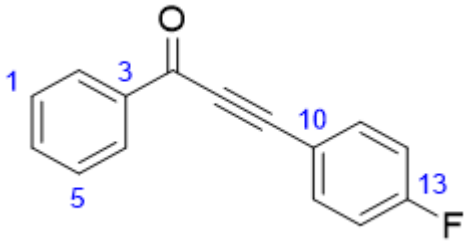

```
Current Data Parameters
NAME                  PCK-3-68
EXPNO                       12
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20210309
Time                     20.01
INSTRUM              AVIII_400
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                   8223.685 Hz
FIDRES                0.125483 Hz
AQ                    3.9845889 sec
RG                         80.6
DW                       60.800 usec
DE                        6.50 usec
TE                       296.2 K
D1                  1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               399.9124696 MHz
NUC1                       1H
P1                       15.00 usec
PLW1               17.29199982 W

F2 - Processing parameters
SI                      32768
SF                 399.9100097 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```

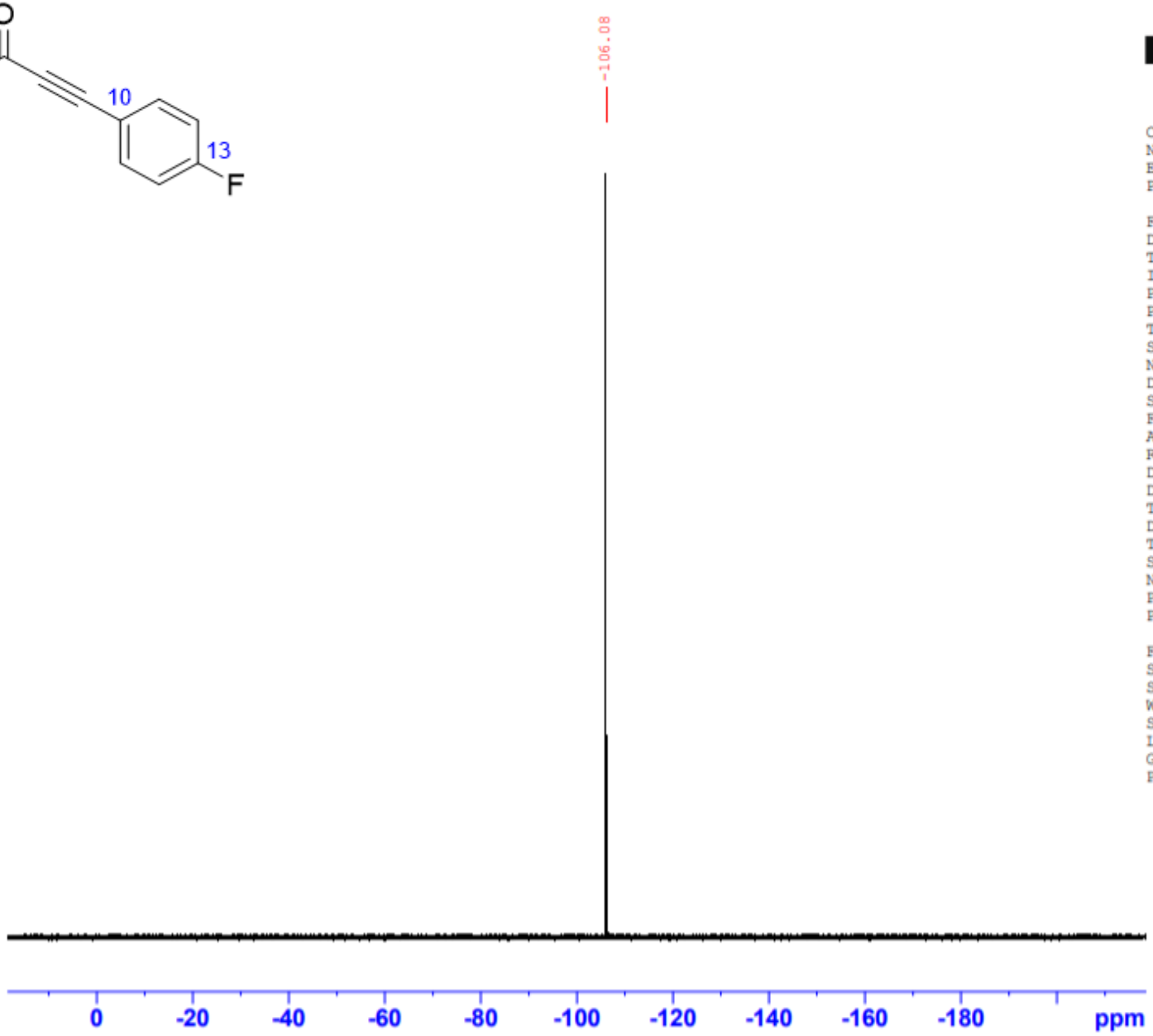
2b - ^{13}C NMR, 101 MHz, $CDCl_3$




```
Current Data Parameters
NAME                 PCK-3-68
EXPNO                      18
PROCNO                      1

F2 - Acquisition Parameters
Date_               20210325
Time                   22.41 h
INSTRUM             AVIII_400
PROBHD    Z108618_0146 (
PULPROG               zgpg30
TD                     96150
SOLVENT                CDCl3
NS                      4096
DS                         4
SWH                24038.461 Hz
FIDRES             0.500020 Hz
AQ                 1.9999200 sec
RG                      2050
DW                    20.800 usec
DE                     6.50 usec
TE                    300.0 K
D1                 1.00000000 sec
D11                0.03000000 sec
TD0                        1
SFO1              100.6178003 MHz
NUC1                     13C
P0                    3.00 usec
P1                    9.00 usec
PLW1              96.68000031 W
SFO2              400.1116004 MHz
NUC2                     1H
CPDPRG[2              waltz64
PCPD2                 90.00 usec
PLW2              17.29199982 W
PLW12              0.48032999 W
PLW13              0.24160001 W

F2 - Processing parameters
SI                      131072
SF                400.6077400 MHz
WDW                        EM
SSB                        0
LB                    1.00 Hz
GB                         0
PC                    1.40
```

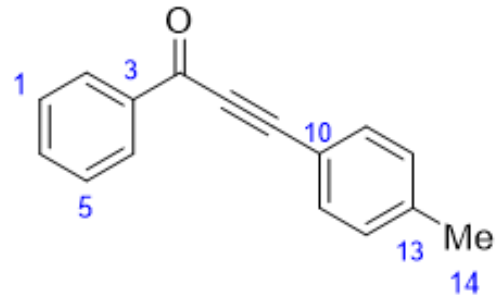
2b - ^{19}F NMR, 376 MHz, $CDCl_3$

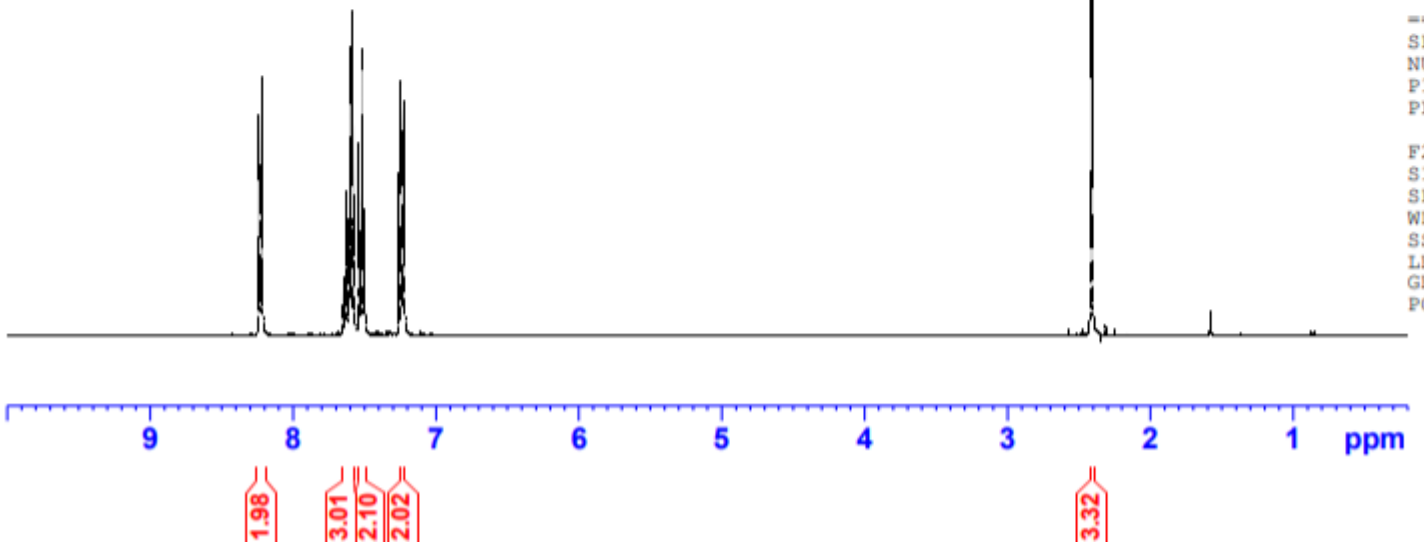





```
Current Data Parameters
NAME                 PCK-3-68
EXPNO                      31
PROCNO                      1

F2 - Acquisition Parameters
Date_               20210326
Time                   10.55 h
INSTRUM              AVIII_400
PROBHD     Z108618_0146 (
PULPROG                    zg
TD                     261992
SOLVENT                 CDCl3
NS                         16
DS                          4
SWH                89285.711 Hz
FIDRES             0.681591 Hz
AQ                 1.4671552 sec
RG                        575
DW                      5.600 usec
DE                      7.11 usec
TE                     300.0 K
D1                 1.00000000 sec
TD0                         1
SFO1            376.4418995 MHz
NUC1                      19F
P1                     11.80 usec
PLW1            32.96500015 W

F2 - Processing parameters
SI                     262144
SF              376.4795470 MHz
WDW                        EM
SSB                         0
LB                      0.30 Hz
GB                          0
PC                      1.00
```

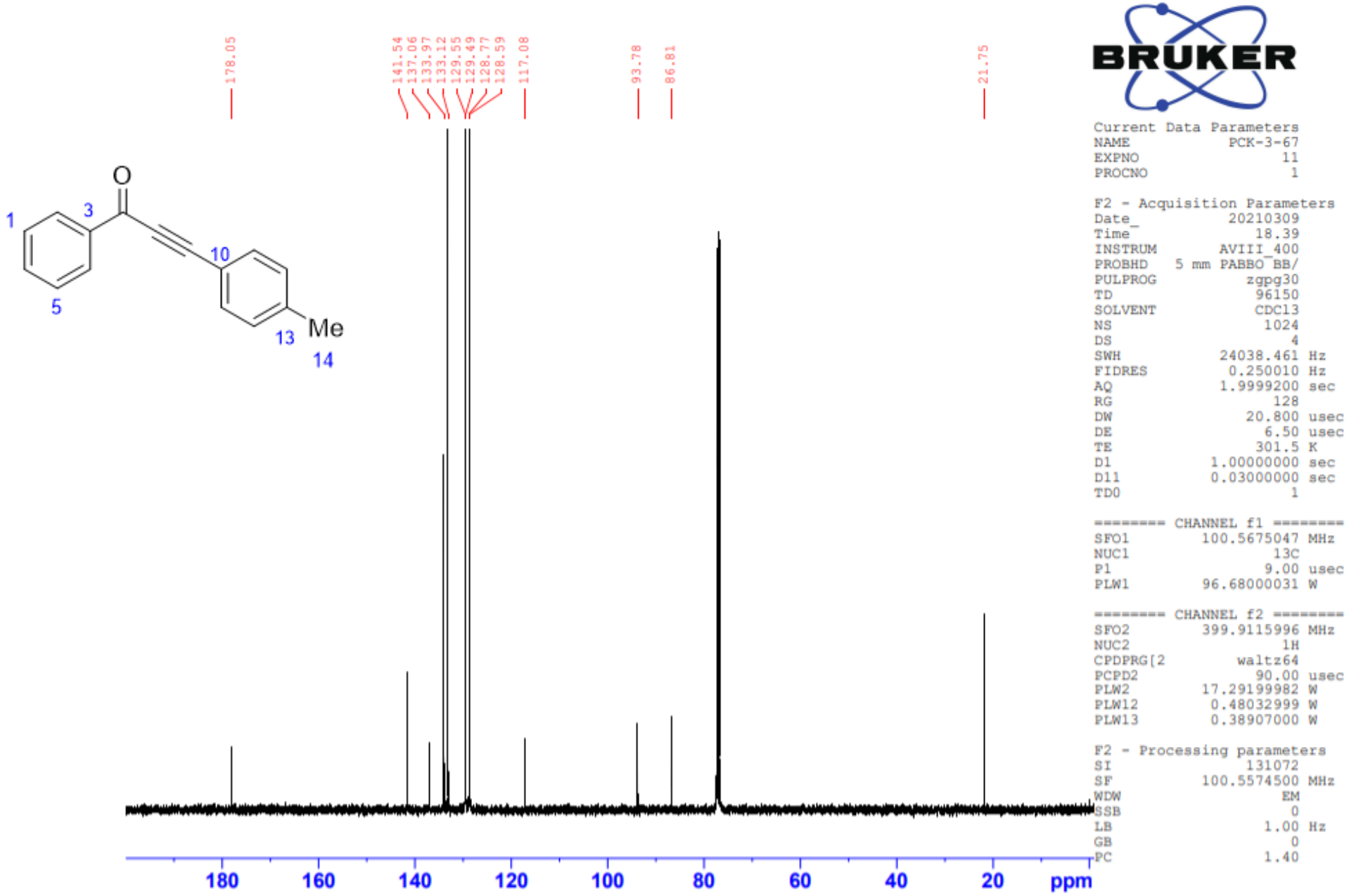
2c - ^{1}H NMR, 400 MHz, $CDCl_3$





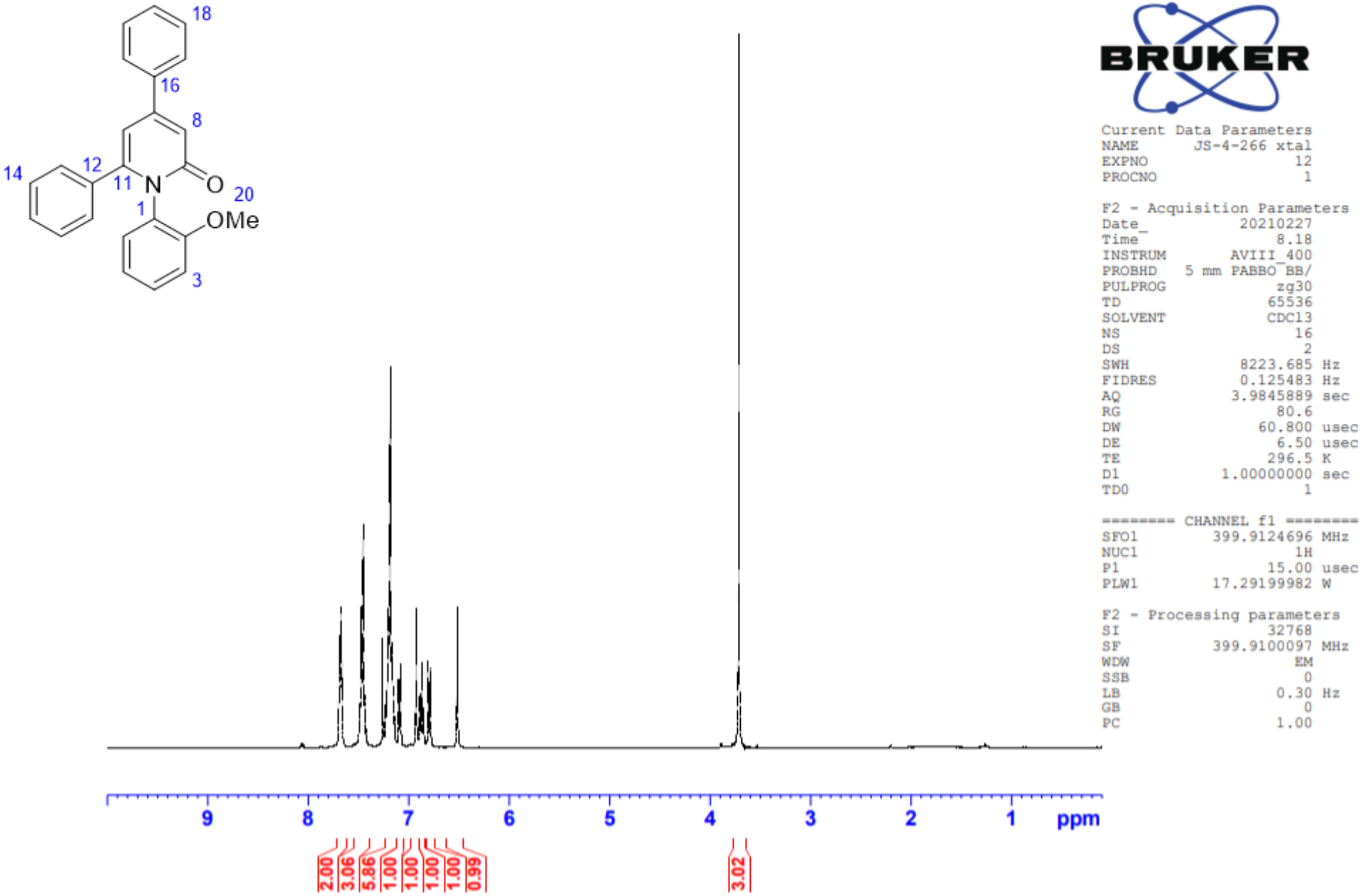

```
Current Data Parameters
NAME                  PCK-3-67
EXPNO                       12
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20210309
Time                     18.42
INSTRUM               AVIII_400
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                   8223.685 Hz
FIDRES                0.125483 Hz
AQ                    3.9845889 sec
RG                         80.6
DW                       60.800 usec
DE                        6.50 usec
TE                       297.0 K
D1                    1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1                399.9124696 MHz
NUC1                        1H
P1                       15.00 usec
PLW1                17.29199982 W

F2 - Processing parameters
SI                          32768
SF                  399.9100098 MHz
WDW                            EM
SSB                             0
LB                           0.30 Hz
GB                              0
PC                           1.00
```

2c - ^{13}C NMR, 101 MHz, $CDCl_3$



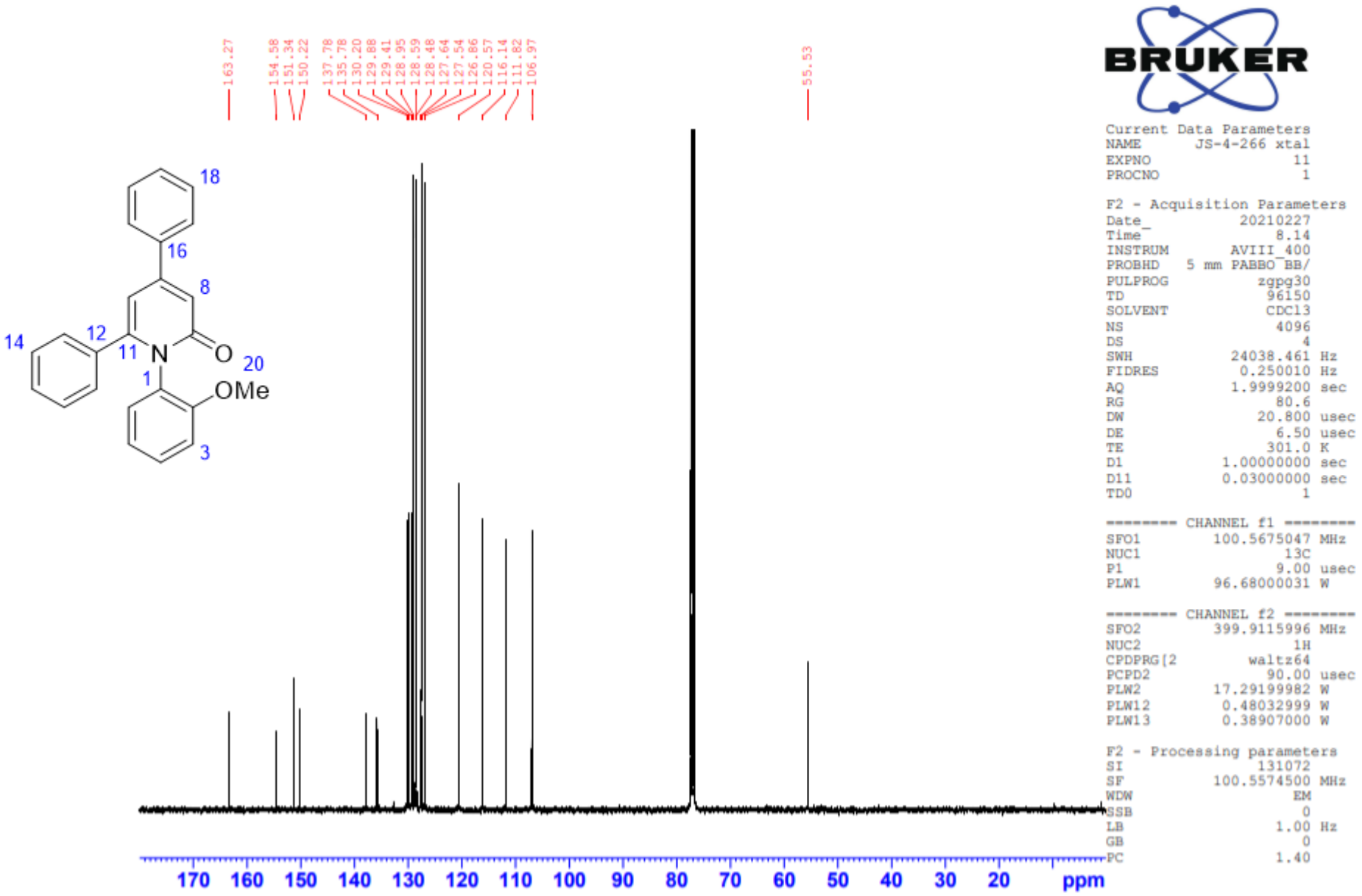

```
Current Data Parameters
NAME                  PCK-3-67
EXPNO                       11
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20210309
Time                     18.39
INSTRUM               AVIII_400
PROBHD    5 mm PABBO BB/
PULPROG                zgpg30
TD                      96150
SOLVENT                 CDCl3
NS                       1024
DS                          4
SWH                 24038.461 Hz
FIDRES               0.250010 Hz
AQ                   1.9999200 sec
RG                         128
DW                      20.800 usec
DE                       6.50 usec
TE                      301.5 K
D1                   1.00000000 sec
D11                  0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               100.5675047 MHz
NUC1                       13C
P1                        9.00 usec
PLW1               96.68000031 W

======== CHANNEL f2 ========
SFO2               399.9115996 MHz
NUC2                       1H
CPDPRG[2                waltz64
PCPD2                    90.00 usec
PLW2               17.29199982 W
PLW12               0.48032999 W
PLW13               0.38907000 W

F2 - Processing parameters
SI                        131072
SF                 100.5574500 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

S4 - ^{1}H NMR, 400 MHz, $CDCl_3$

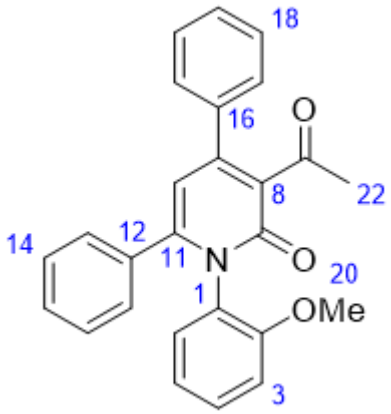


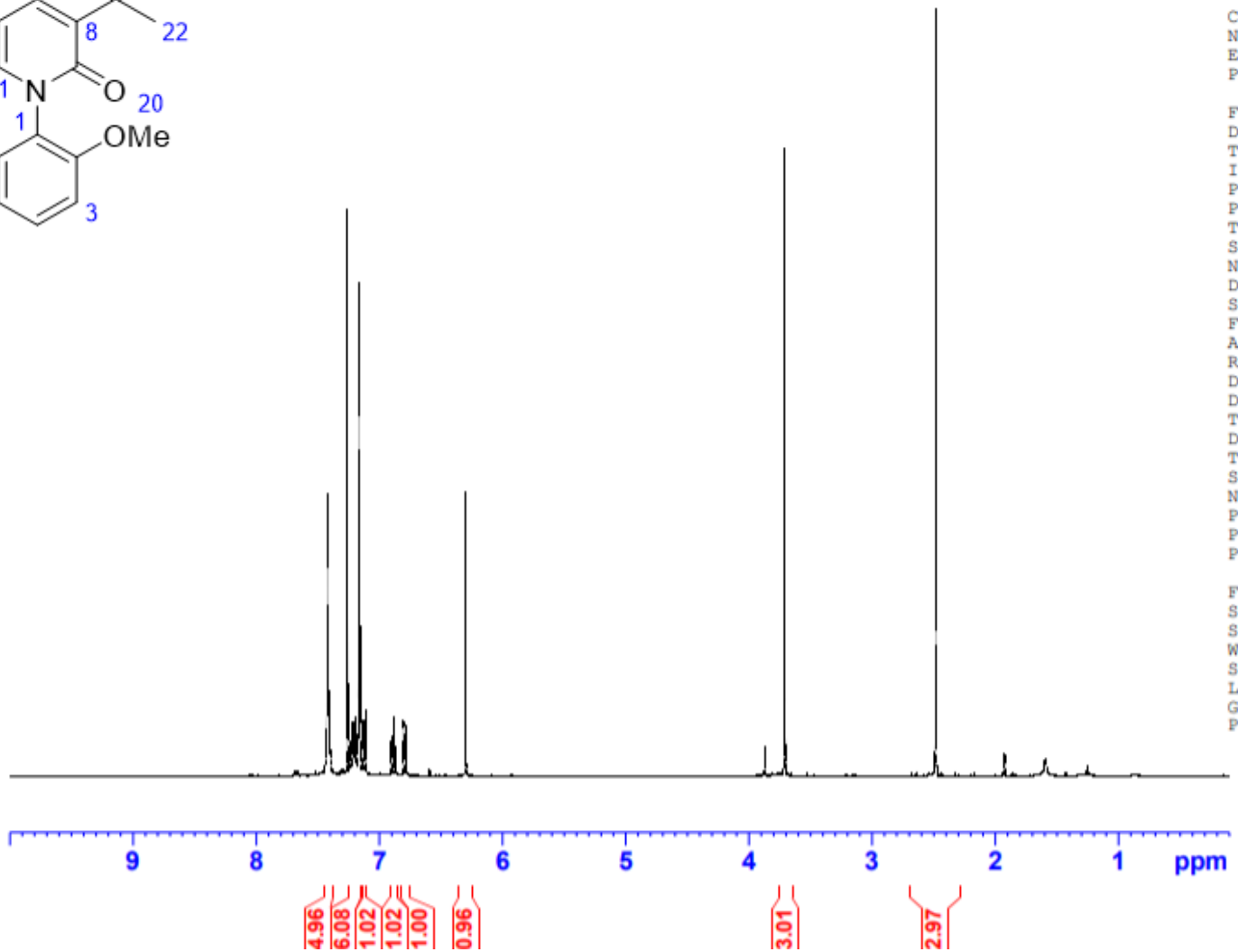

```
Current Data Parameters
NAME       JS-4-266 xtal
EXPNO                12
PROCNO                1

F2 - Acquisition Parameters
Date_          20210227
Time               8.18
INSTRUM       AVIII_400
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ            3.9845889 sec
RG                 80.6
DW                60.800 usec
DE                 6.50 usec
TE                296.5 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        399.9124696 MHz
NUC1                   1H
P1                 15.00 usec
PLW1        17.29199982 W

F2 - Processing parameters
SI                 32768
SF          399.9100097 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```

S4 - ^{13}C NMR, 101 MHz, $CDCl_3$



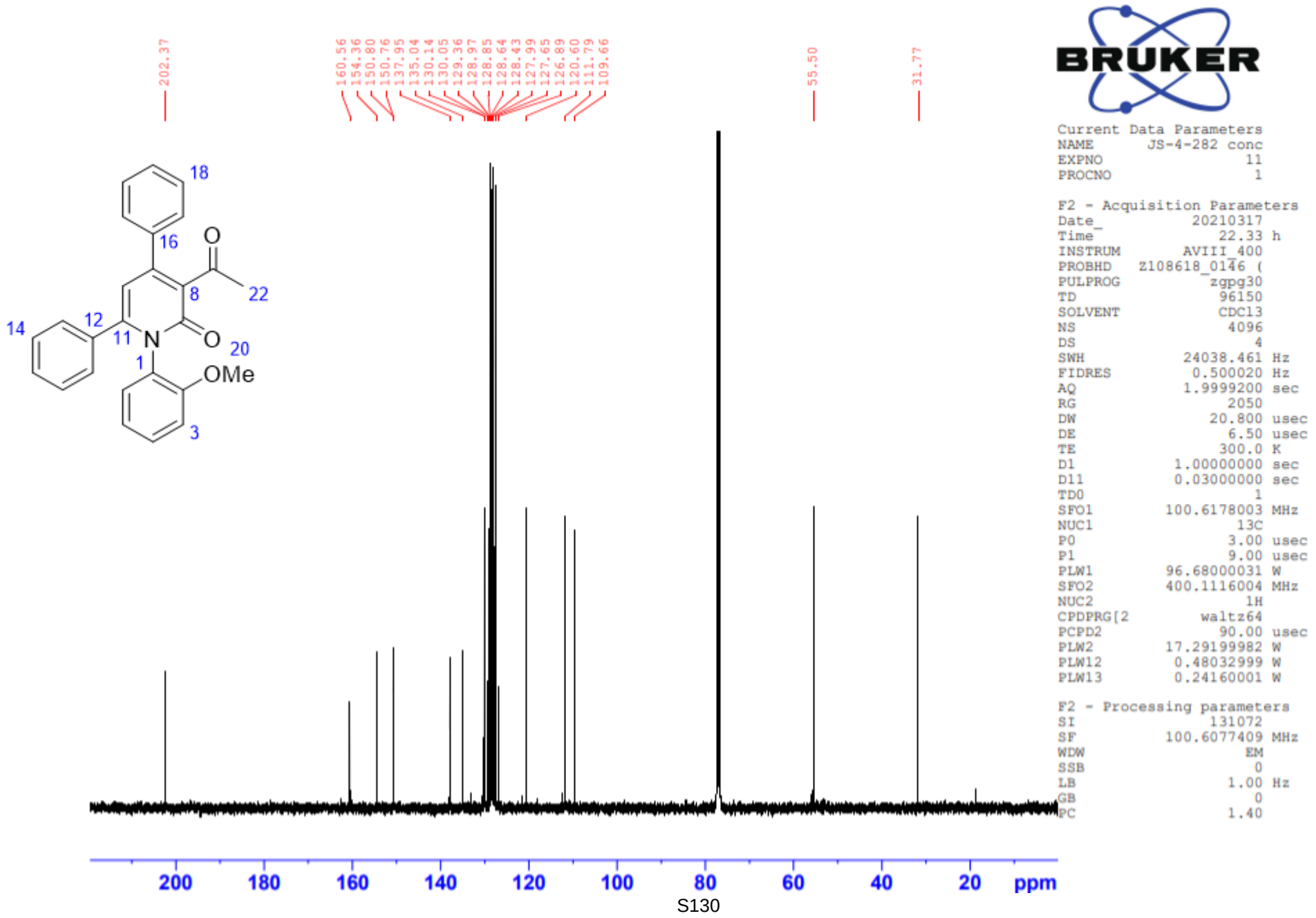
S5 - ^{1}H NMR, 400 MHz, $CDCl_3$






```
Current Data Parameters
NAME      JS-4-282 conc
EXPNO               12
PROCNO               1

F2 - Acquisition Parameters
Date_          20210317
Time             22.36 h
INSTRUM       AVIII_400
PROBHD   Z108618_0146 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.250967 Hz
AQ             3.9845889 sec
RG                  256
DW               60.800 usec
DE               17.42 usec
TE                300.0 K
D1           1.00000000 sec
TD0                  1
SFO1         400.1124708 MHz
NUC1                 1H
P0                5.00 usec
P1               15.00 usec
PLW1         17.29199982 W

F2 - Processing parameters
SI                32768
SF           400.1100099 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                 1.00
```

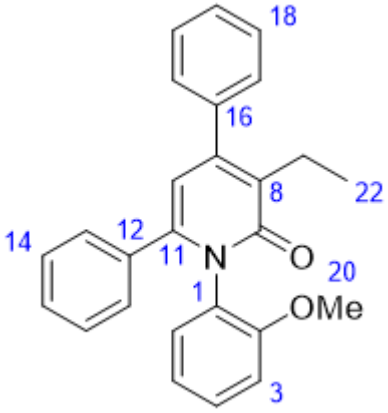
S5 - ^{13}C NMR, 101 MHz, $CDCl_3$

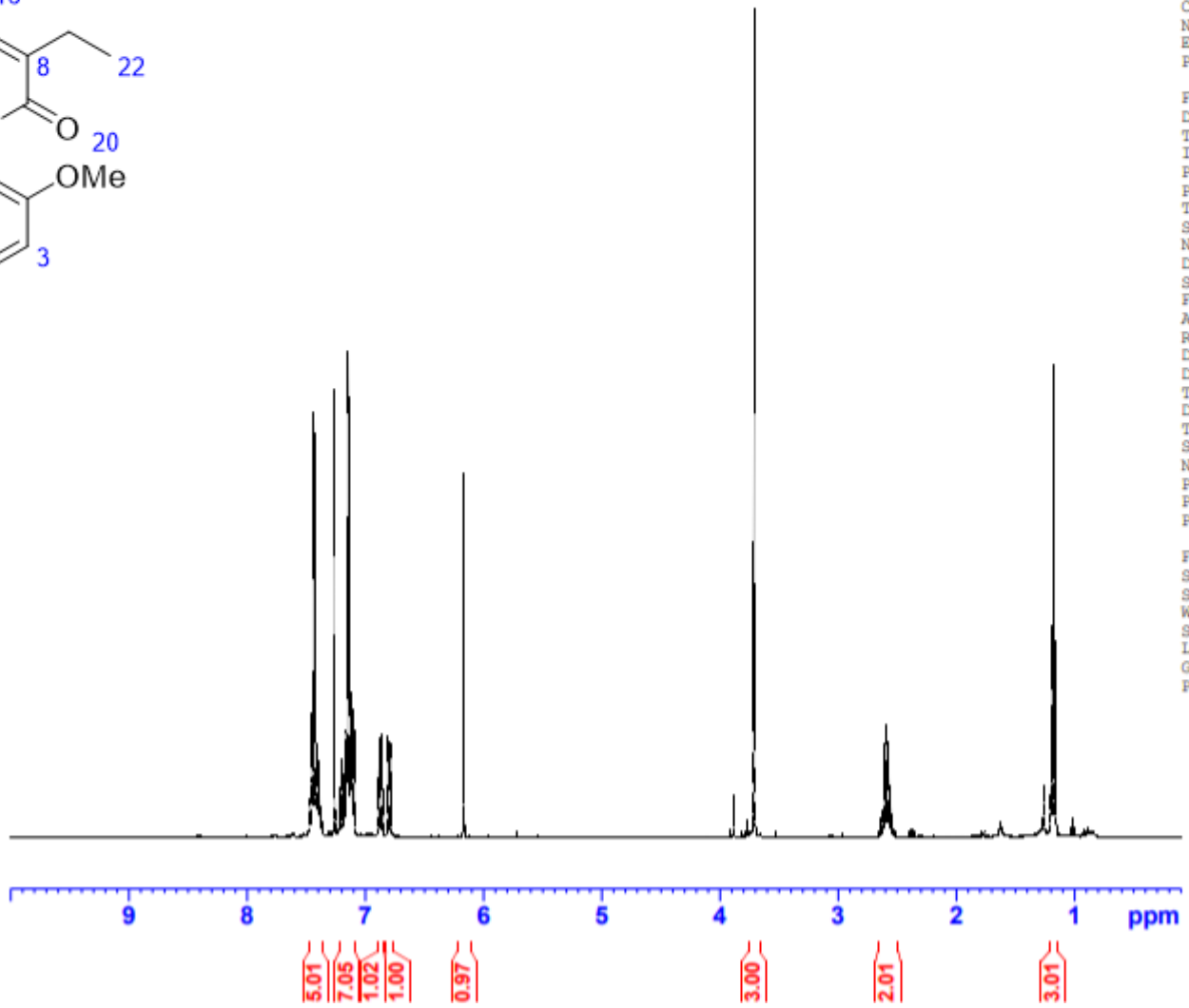


BRUKER

```
Current Data Parameters
NAME       JS-4-282 conc
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210317
Time              22.33 h
INSTRUM        AVIII_400
PROBHD    Z108618_0146 (
PULPROG          zgpg30
TD                96150
SOLVENT           CDC13
NS                 4096
DS                    4
SWH           24038.461 Hz
FIDRES         0.500020 Hz
AQ             1.9999200 sec
RG                 2050
DW               20.800 usec
DE                6.50 usec
TE               300.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         100.6178003 MHz
NUC1                13C
P0                3.00 usec
P1                9.00 usec
PLW1         96.68000031 W
SFO2         400.1116004 MHz
NUC2                1H
CPDPRG[2         waltz64
PCPD2             90.00 usec
PLW2         17.29199982 W
PLW12         0.48032999 W
PLW13         0.24160001 W

F2 - Processing parameters
SI                131072
SF         100.6077409 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

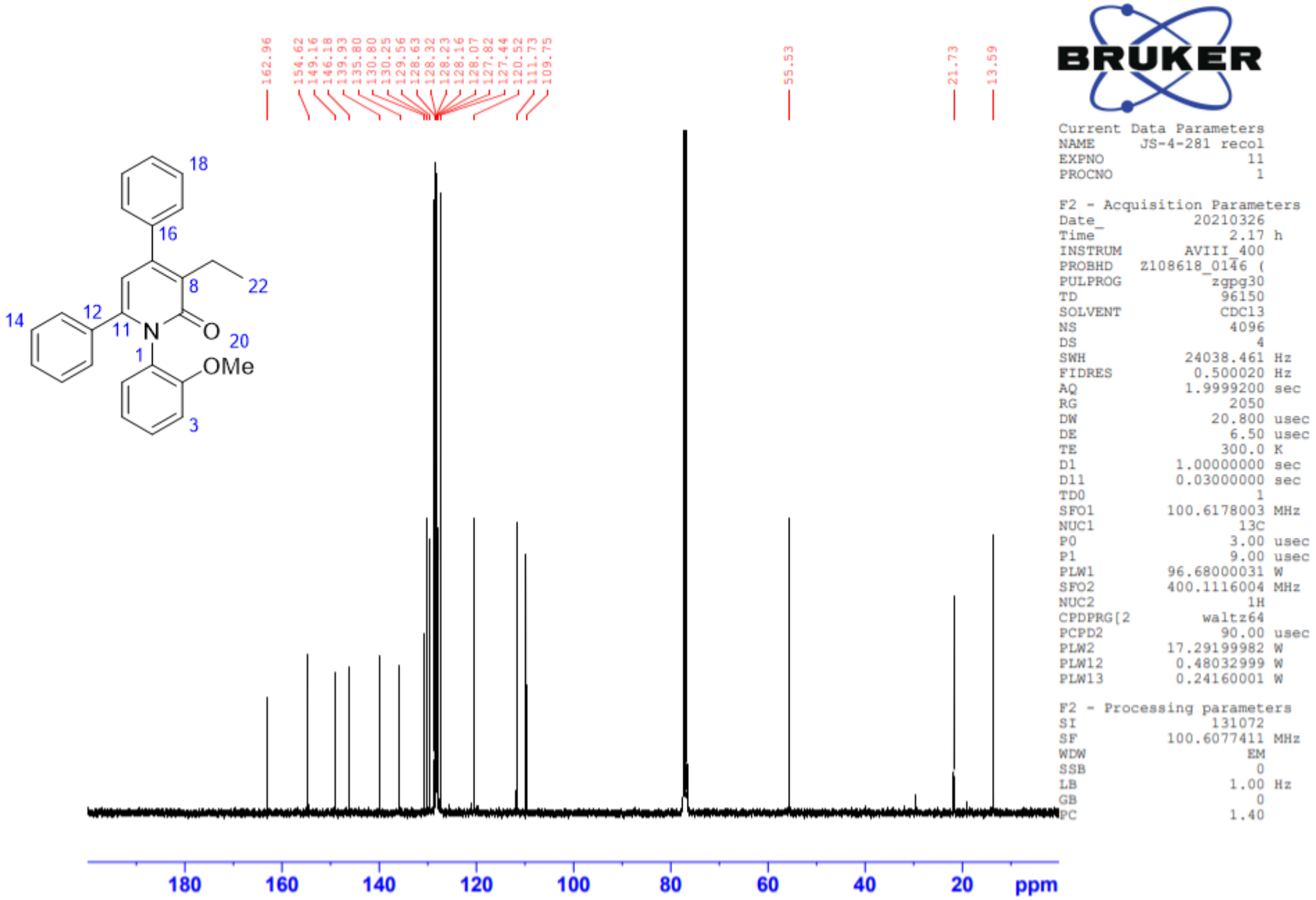
S6 - ^{1}H NMR, 400 MHz, $CDCl_3$





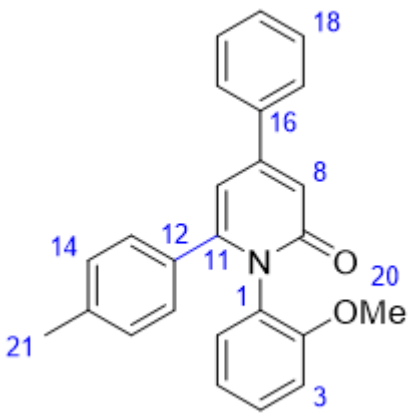

```
Current Data Parameters
NAME      JS-4-281 recol
EXPNO               10
PROCNO               1

F2 - Acquisition Parameters
Date_          20210325
Time             16.38 h
INSTRUM       AVIII 400
PROBHD   Z108618_0146 (
PULPROG            zg30
TD                65536
SOLVENT           CDC13
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES        0.250967 Hz
AQ            3.9845889 sec
RG                  161
DW               60.800 usec
DE               17.42 usec
TE               300.0 K
D1            1.00000000 sec
TD0                  1
SFO1        400.1124708 MHz
NUC1                1H
P0               5.00 usec
P1              15.00 usec
PLW1        17.29199982 W

F2 - Processing parameters
SI                32768
SF        400.1100100 MHz
WDW                  EM
SSB                   0
LB               0.30 Hz
GB                    0
PC                 1.00
```

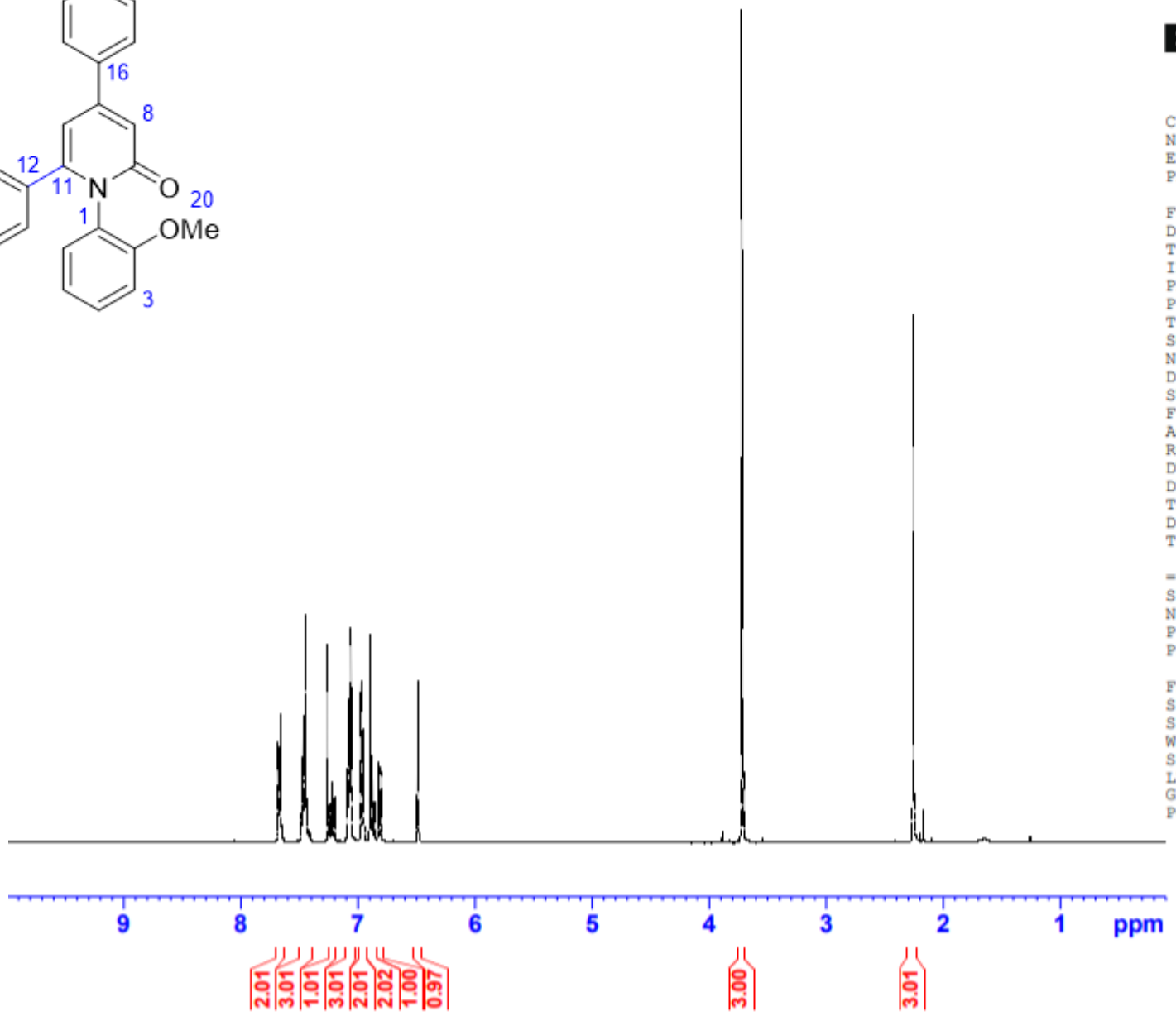
S6 - ^{13}C NMR, 101 MHz, $CDCl_3$




```
Current Data Parameters
NAME      JS-4-281 recol
EXPNO                11
PROCNO                1

F2 - Acquisition Parameters
Date_          20210326
Time               2.17 h
INSTRUM       AVIII 400
PROBHD   Z108618_0146 (
PULPROG         zgpg30
TD               96150
SOLVENT          CDCl3
NS                4096
DS                   4
SWH          24038.461 Hz
FIDRES        0.500020 Hz
AQ            1.9999200 sec
RG                2050
DW              20.800 usec
DE               6.50 usec
TE              300.0 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                  1
SFO1        100.6178003 MHz
NUC1                13C
P0                3.00 usec
P1                9.00 usec
PLW1         96.68000031 W
SFO2        400.1116004 MHz
NUC2                 1H
CPDPRG[2         waltz64
PCPD2            90.00 usec
PLW2         17.29199982 W
PLW12         0.48032999 W
PLW13         0.24160001 W

F2 - Processing parameters
SI                131072
SF          100.6077411 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

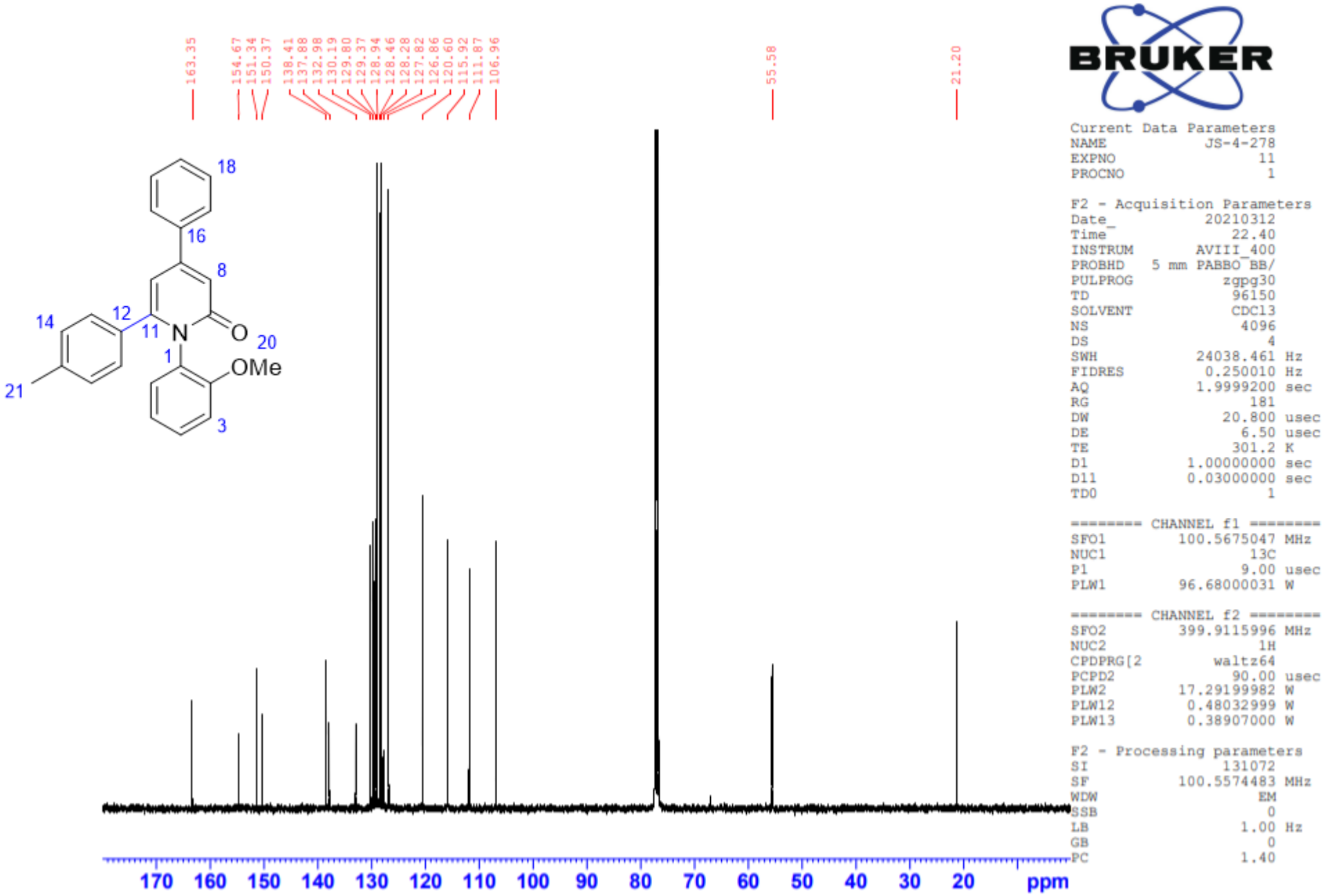
S7 - ^{1}H NMR, 400 MHz, $CDCl_3$





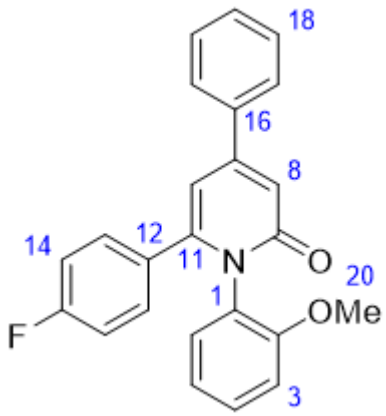

```
Current Data Parameters
NAME            JS-4-278
EXPNO                 12
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210312
Time               22.44
INSTRUM        AVIII_400
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9845889 sec
RG                  80.6
DW                60.800 usec
DE                 6.50 usec
TE                296.8 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         399.9124696 MHz
NUC1                   1H
P1                 15.00 usec
PLW1         17.29199982 W

F2 - Processing parameters
SI                 32768
SF           399.9100097 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```

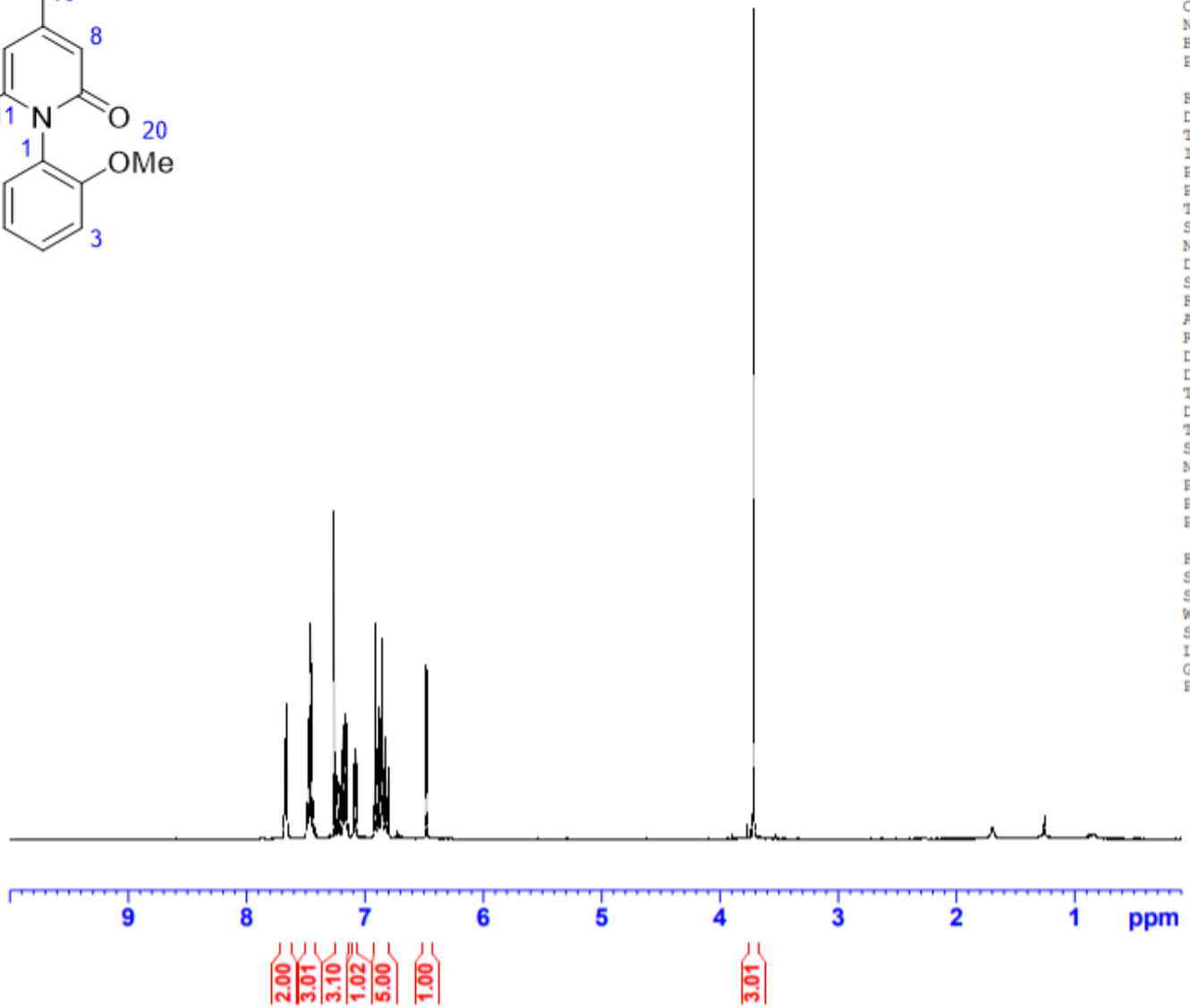
S7 - ^{13}C NMR, 101 MHz, $CDCl_3$




```
Current Data Parameters
NAME                  JS-4-278
EXPNO                       11
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20210312
Time                     22.40
INSTRUM               AVIII_400
PROBHD   5 mm PABBO_BB/
PULPROG                zgpg30
TD                      96150
SOLVENT                 CDCl3
NS                       4096
DS                          4
SWH                24038.461 Hz
FIDRES              0.250010 Hz
AQ                  1.9999200 sec
RG                        181
DW                     20.800 usec
DE                      6.50 usec
TE                     301.2 K
D1                  1.00000000 sec
D11                 0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               100.5675047 MHz
NUC1                       13C
P1                        9.00 usec
PLW1               96.68000031 W

======== CHANNEL f2 ========
SFO2               399.9115996 MHz
NUC2                       1H
CPDPRG[2                waltz64
PCPD2                    90.00 usec
PLW2               17.29199982 W
PLW12               0.48032999 W
PLW13               0.38907000 W

F2 - Processing parameters
SI                        131072
SF                 100.5574483 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

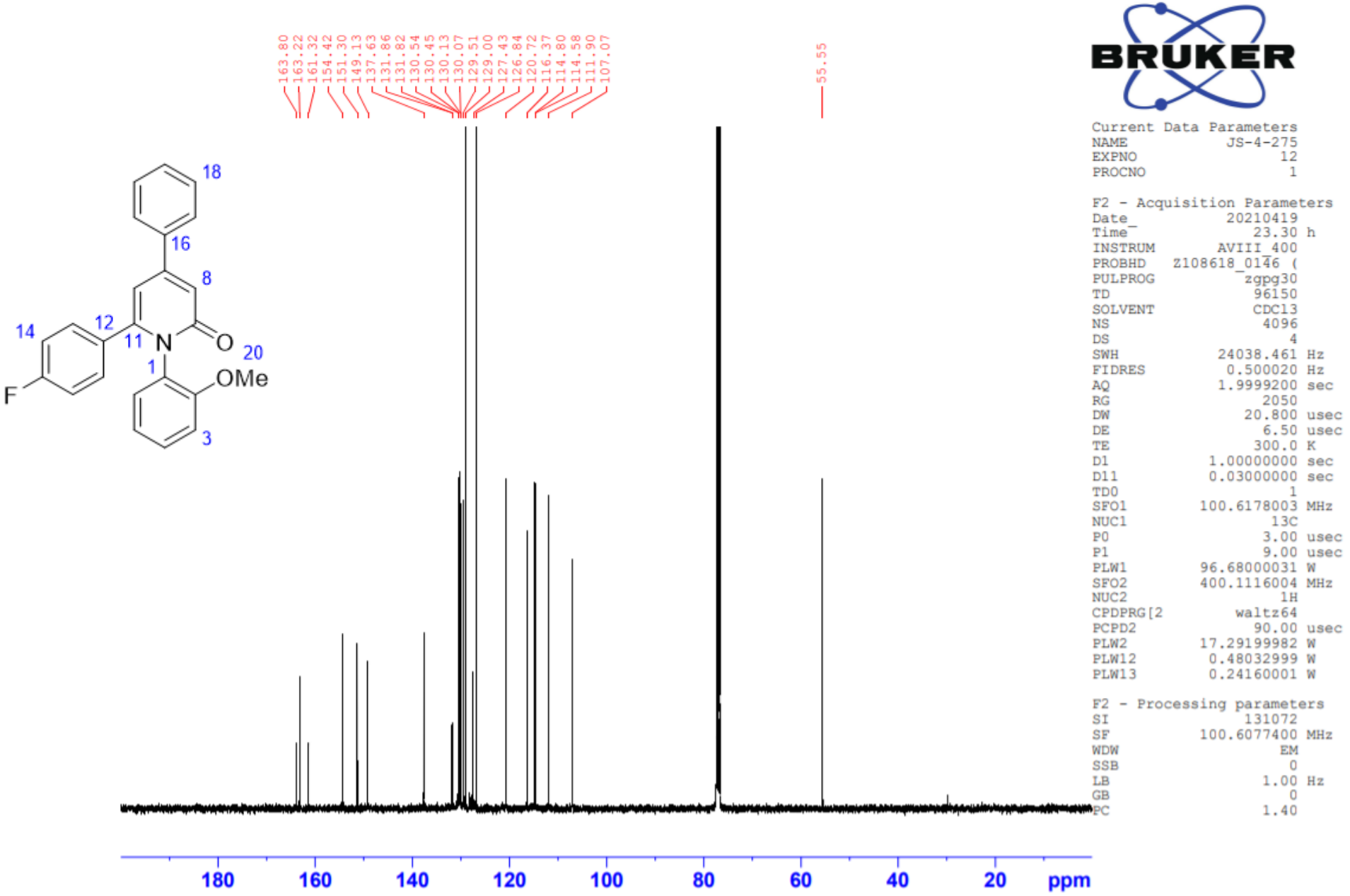
S8 - ^{1}H NMR, 400 MHz, $CDCl_3$





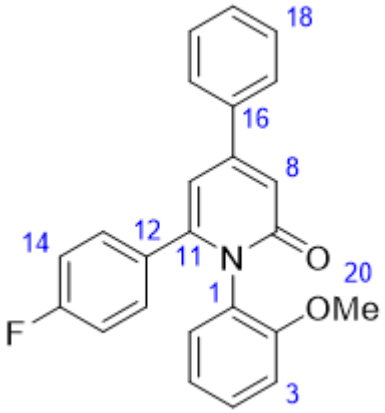

```
Current Data Parameters
NAME      JS-4-275 recol
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210326
Time              10.44 h
INSTRUM        AVIII_400
PROBHD   Z108618_0146 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ              3.9845889 sec
RG                   161
DW                60.800 usec
DE                17.42 usec
TE                300.0 K
D1           1.00000000 sec
TD0                    1
SFO1         400.1124708 MHz
NUC1                  1H
P0                 5.00 usec
P1                15.00 usec
PLW1        17.29199982 W

F2 - Processing parameters
SI                 32768
SF          400.1100100 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.00
```

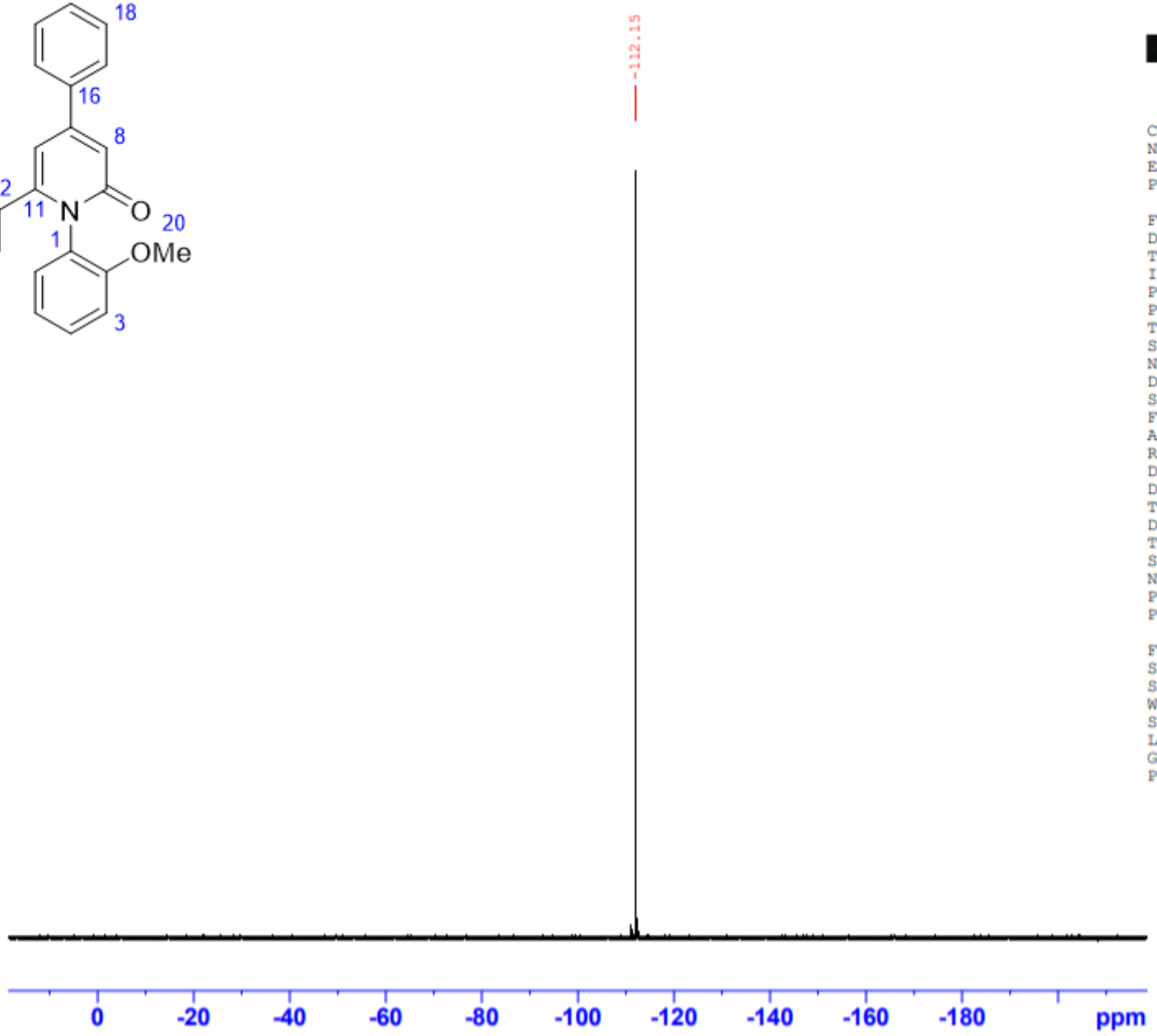
S8 - ^{13}C NMR, 101 MHz, $CDCl_3$




```
Current Data Parameters
NAME                  JS-4-275
EXPNO                       12
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20210419
Time                     23.30 h
INSTRUM              AVIII_400
PROBHD    Z108618_0146 (
PULPROG                zgpg30
TD                      96150
SOLVENT                 CDCl3
NS                       4096
DS                          4
SWH                 24038.461 Hz
FIDRES               0.500020 Hz
AQ                   1.9999200 sec
RG                        2050
DW                      20.800 usec
DE                       6.50 usec
TE                      300.0 K
D1                   1.00000000 sec
D11                  0.03000000 sec
TD0                         1
SFO1               100.6178003 MHz
NUC1                       13C
P0                      3.00 usec
P1                      9.00 usec
PLW1               96.68000031 W
SFO2               400.1116004 MHz
NUC2                       1H
CPDPRG[2                waltz64
PCPD2                   90.00 usec
PLW2               17.29199982 W
PLW12               0.48032999 W
PLW13               0.24160001 W

F2 - Processing parameters
SI                        131072
SF                 100.6077400 MHz
WDW                         EM
SSB                          0
LB                      1.00 Hz
GB                           0
PC                      1.40
```

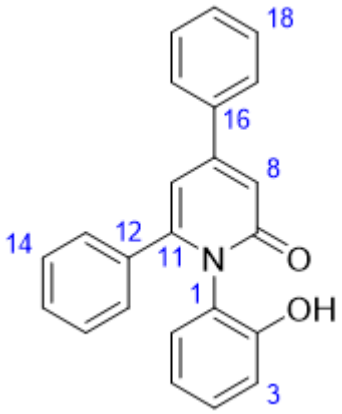
S8 - ^{19}F NMR, 376 MHz, $CDCl_3$

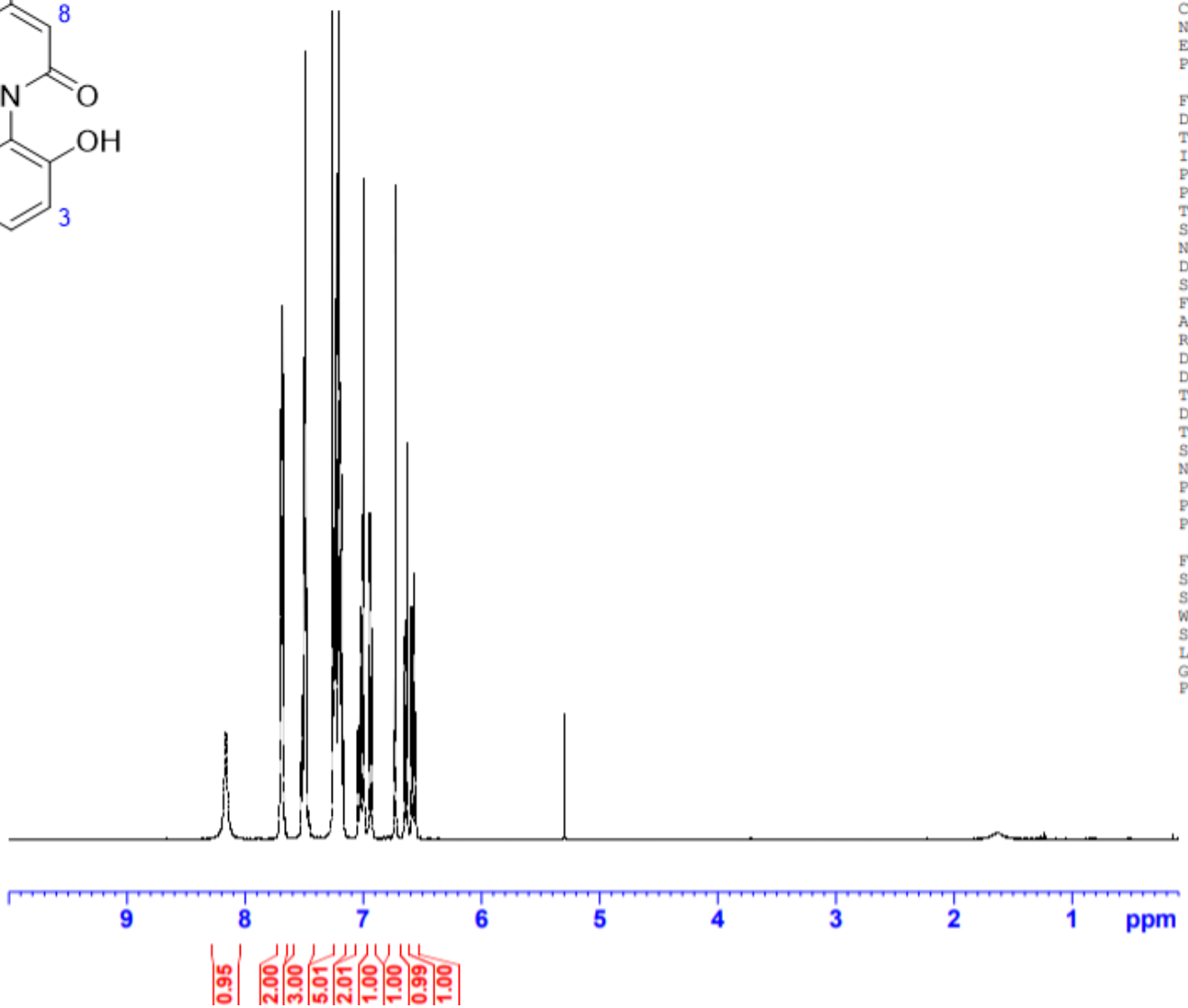





```
Current Data Parameters
NAME                  JS-4-275
EXPNO                       16
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20210420
Time                      0.01 h
INSTRUM              AVIII_400
PROBHD   Z108618_0146 (
PULPROG                     zg
TD                      261992
SOLVENT                  CDCl3
NS                          16
DS                           4
SWH                  89285.711 Hz
FIDRES                0.681591 Hz
AQ                    1.4671552 sec
RG                          575
DW                        5.600 usec
DE                        7.11 usec
TE                        300.0 K
D1                  1.00000000 sec
TD0                          1
SFO1                376.4418995 MHz
NUC1                        19F
P1                       11.80 usec
PLW1                32.96500015 W

F2 - Processing parameters
SI                      262144
SF                  376.4795470 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```

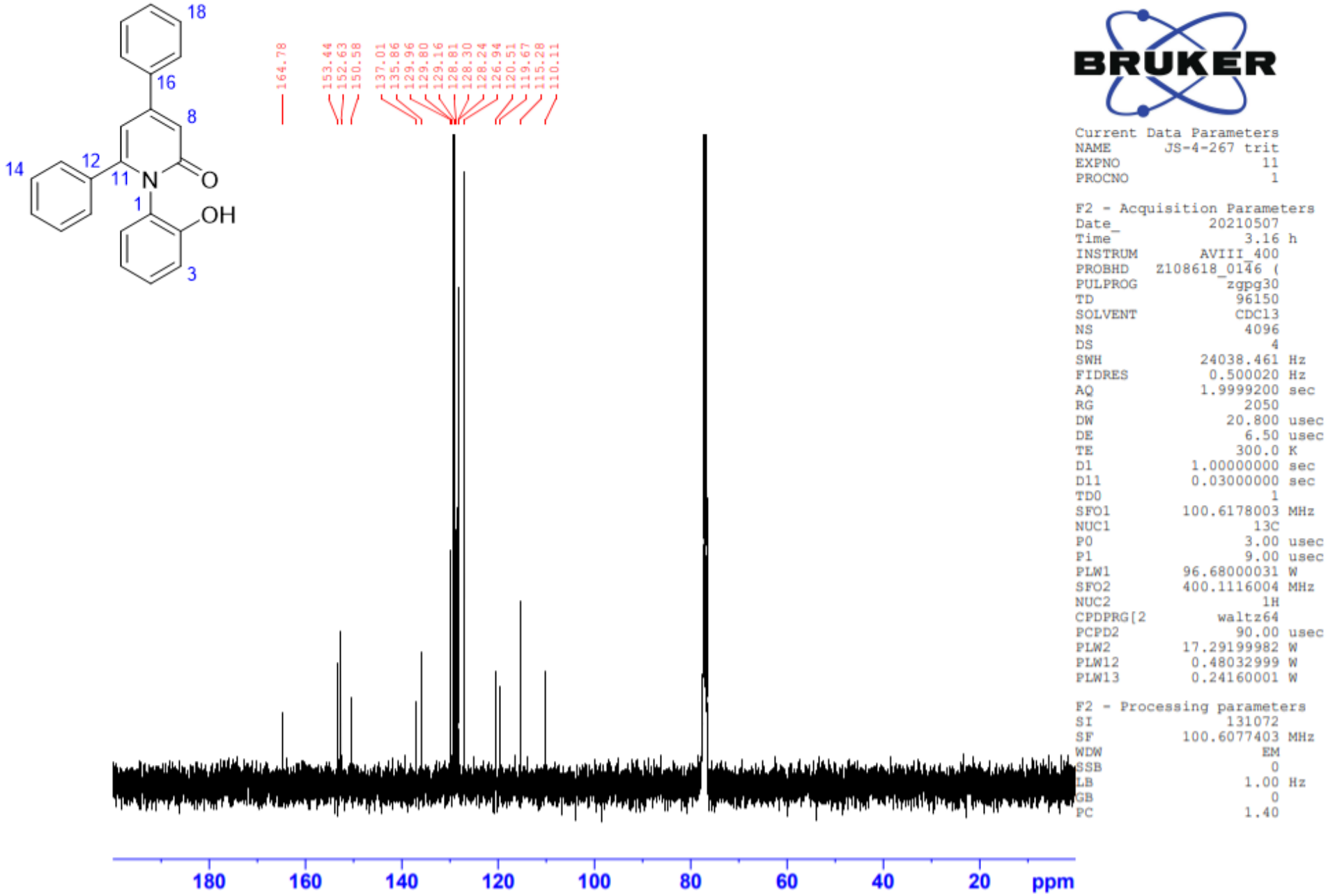
3a - ^{1}H NMR, 400 MHz, $CDCl_3$






```
Current Data Parameters
NAME       JS-4-267 trit
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20210506
Time               12.51 h
INSTRUM       AVIII 400
PROBHD   Z108618_0146 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ              3.9845889 sec
RG                   256
DW                60.800 usec
DE                17.42 usec
TE                300.0 K
D1           1.00000000 sec
TD0                    1
SFO1         400.1124708 MHz
NUC1                  1H
P0                 5.00 usec
P1                15.00 usec
PLW1         17.29199982 W

F2 - Processing parameters
SI                 32768
SF           400.1100099 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.00
```

3a - ^{13}C NMR, 101 MHz, $CDCl_3$



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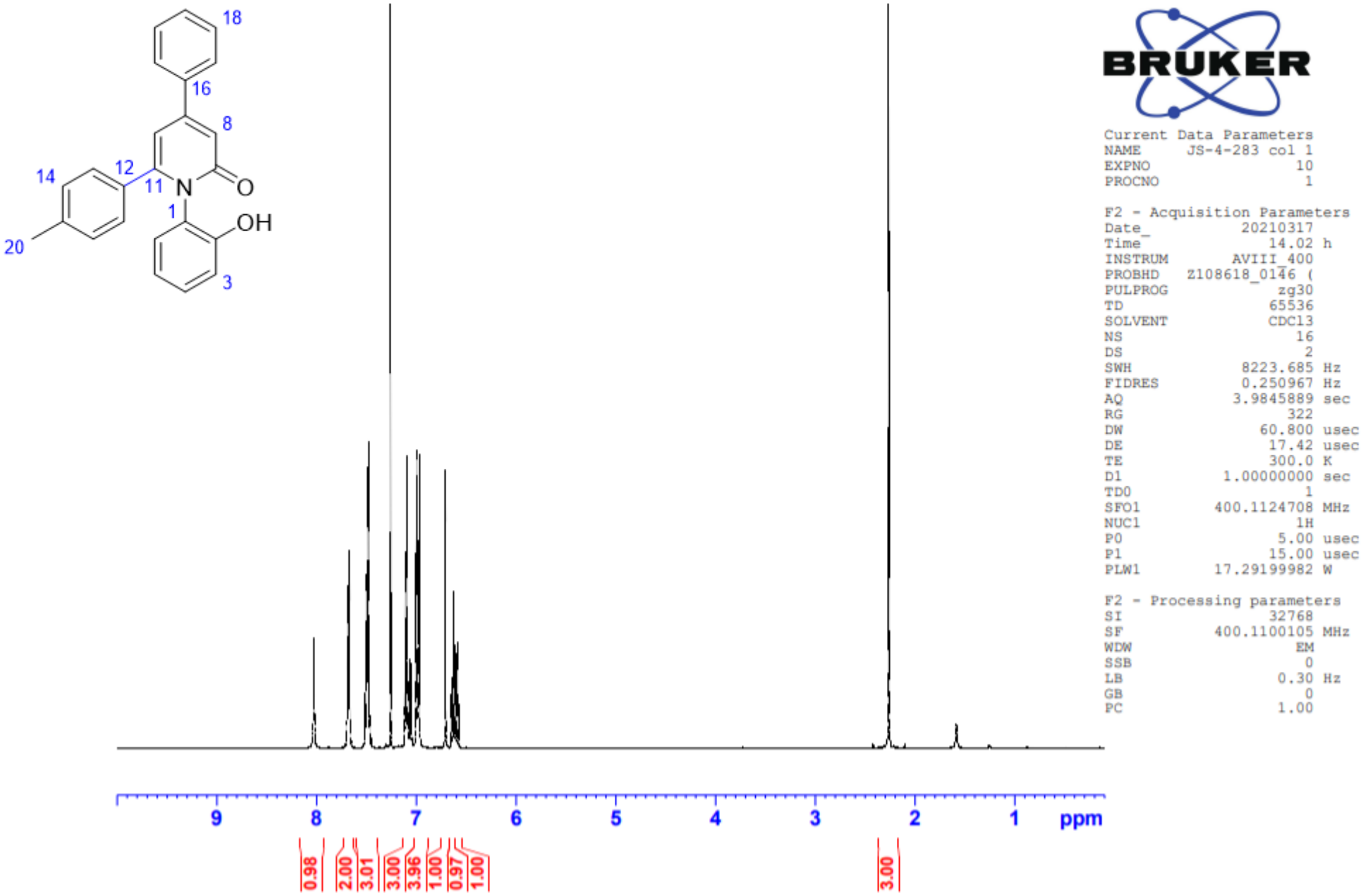
```
Current Data Parameters
NAME       JS-4-267 trit
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210507
Time                3.16 h
INSTRUM        AVIII 400
PROBHD   Z108618_0146 (
PULPROG          zgpg30
TD                96150
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           24038.461 Hz
FIDRES         0.500020 Hz
AQ             1.9999200 sec
RG                 2050
DW               20.800 usec
DE                6.50 usec
TE               300.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1        100.6178003 MHz
NUC1               13C
P0                3.00 usec
P1                9.00 usec
PLW1         96.68000031 W
SFO2        400.1116004 MHz
NUC2               1H
CPDPRG[2        waltz64
PCPD2             90.00 usec
PLW2         17.29199982 W
PLW12         0.48032999 W
PLW13         0.24160001 W

F2 - Processing parameters
SI                131072
SF          100.6077403 MHz
WDW                 EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

3b - ^{1}H NMR, 400 MHz, $CDCl_3$

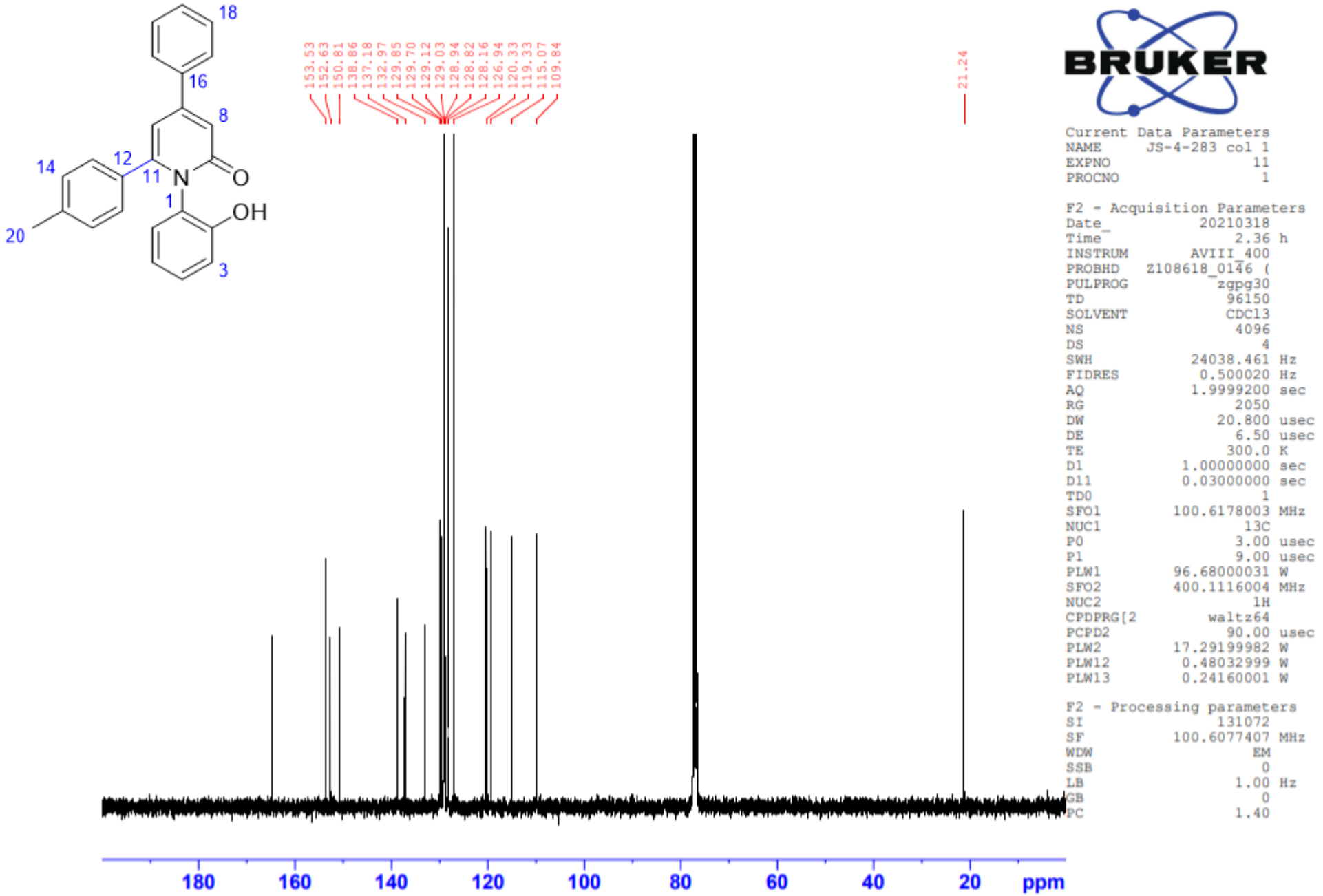



```
Current Data Parameters
NAME      JS-4-283 col 1
EXPNO               10
PROCNO               1

F2 - Acquisition Parameters
Date_          20210317
Time             14.02 h
INSTRUM       AVIII_400
PROBHD   Z108618_0146 (
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8223.685 Hz
FIDRES        0.250967 Hz
AQ           3.9845889 sec
RG                 322
DW              60.800 usec
DE              17.42 usec
TE              300.0 K
D1           1.00000000 sec
TD0                  1
SFO1       400.1124708 MHz
NUC1               1H
P0              5.00 usec
P1             15.00 usec
PLW1       17.29199982 W

F2 - Processing parameters
SI               32768
SF         400.1100105 MHz
WDW                EM
SSB                 0
LB              0.30 Hz
GB                  0
PC               1.00
```

3b - ^{13}C NMR, 101 MHz, $CDCl_3$

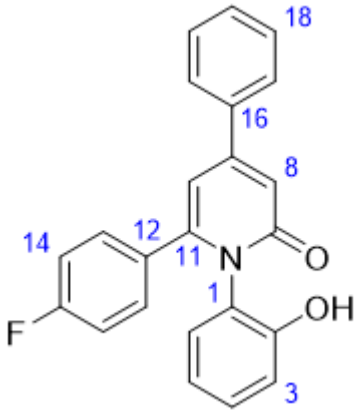


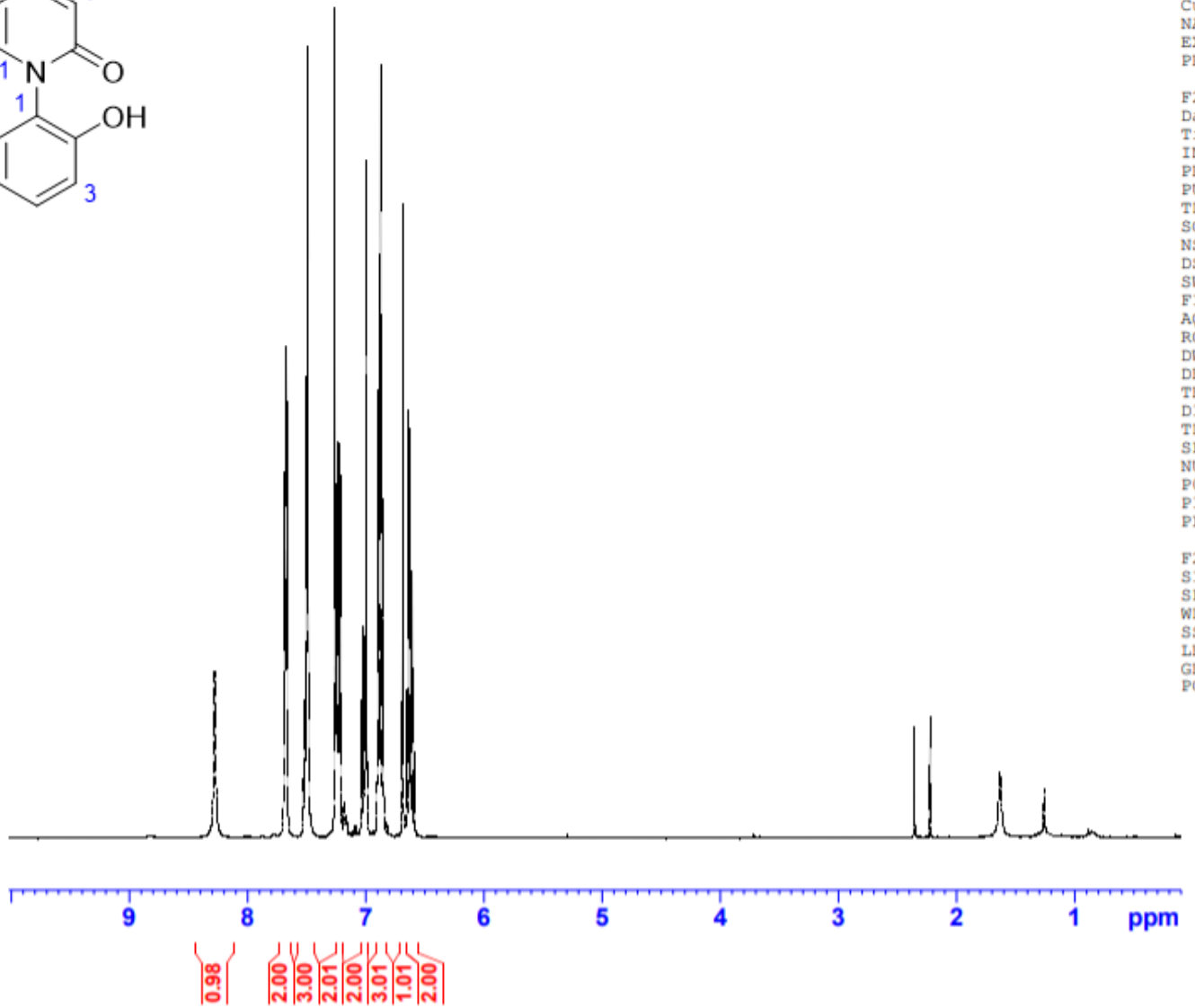

```
Current Data Parameters
NAME      JS-4-283 col 1
EXPNO               11
PROCNO               1

F2 - Acquisition Parameters
Date_          20210318
Time              2.36 h
INSTRUM       AVIII_400
PROBHD   Z108618_0146 (
PULPROG          zgpg30
TD                96150
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           24038.461 Hz
FIDRES         0.500020 Hz
AQ             1.9999200 sec
RG                 2050
DW               20.800 usec
DE                6.50 usec
TE               300.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1        100.6178003 MHz
NUC1               13C
P0                3.00 usec
P1                9.00 usec
PLW1         96.68000031 W
SFO2        400.1116004 MHz
NUC2               1H
CPDPRG[2       waltz64
PCPD2            90.00 usec
PLW2         17.29199982 W
PLW12         0.48032999 W
PLW13         0.24160001 W

F2 - Processing parameters
SI                131072
SF          100.6077407 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

3c - ^{1}H NMR, 400 MHz, $CDCl_3$



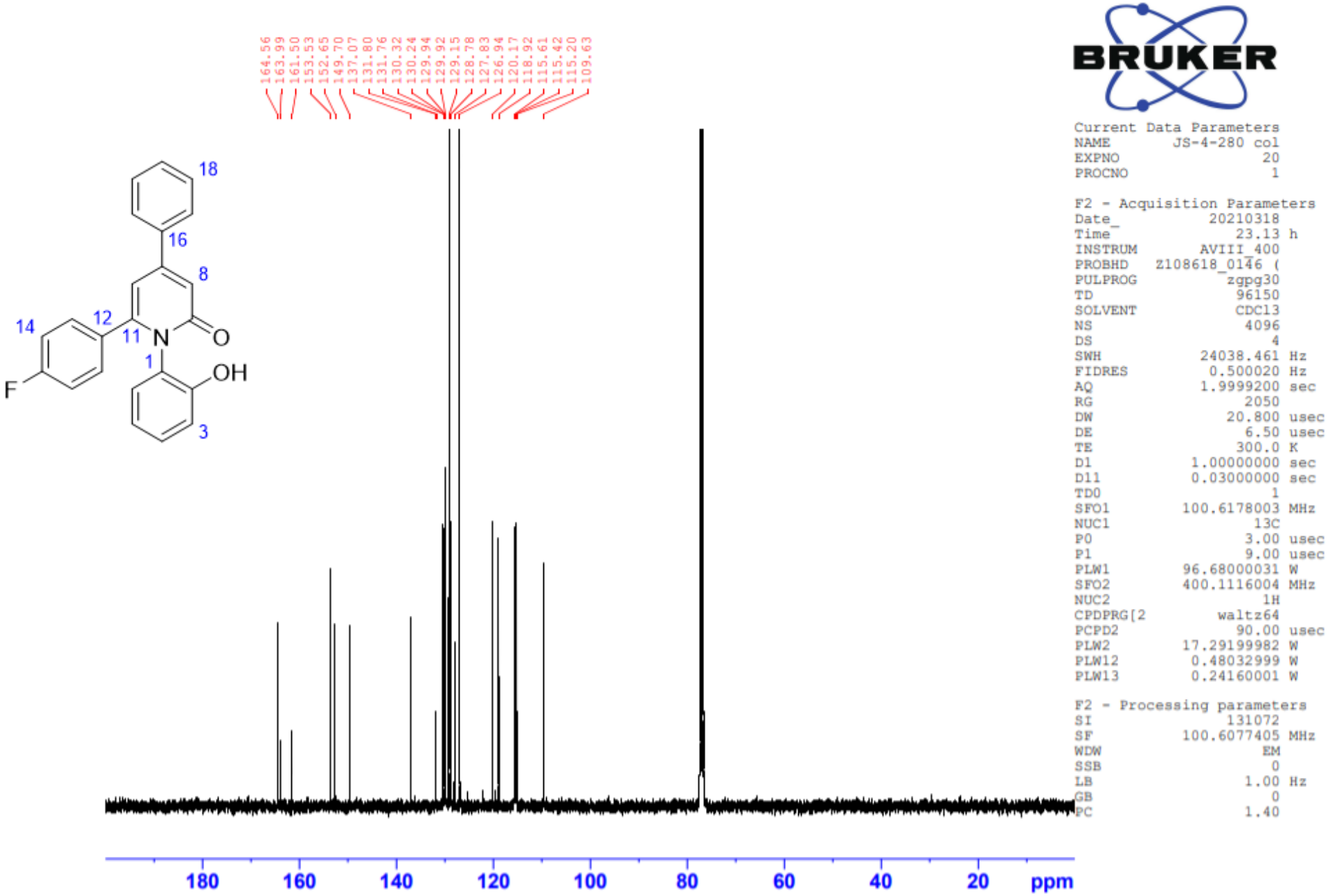



```
Current Data Parameters
NAME        JS-4-280 col
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20210317
Time             17.30 h
INSTRUM       AVIII_400
PROBHD   Z108618_0146 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           8223.685 Hz
FIDRES        0.250967 Hz
AQ           3.9845889 sec
RG                  256
DW               60.800 usec
DE               17.42 usec
TE               300.0 K
D1           1.00000000 sec
TD0                   1
SFO1       400.1124708 MHz
NUC1                 1H
P0                5.00 usec
P1               15.00 usec
PLW1       17.29199982 W

F2 - Processing parameters
SI                32768
SF         400.1100097 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                1.00
```

3c - ^{13}C NMR, 101 MHz, $CDCl_3$



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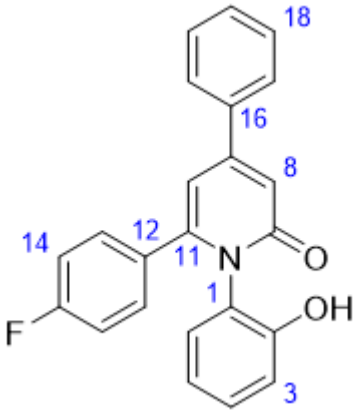
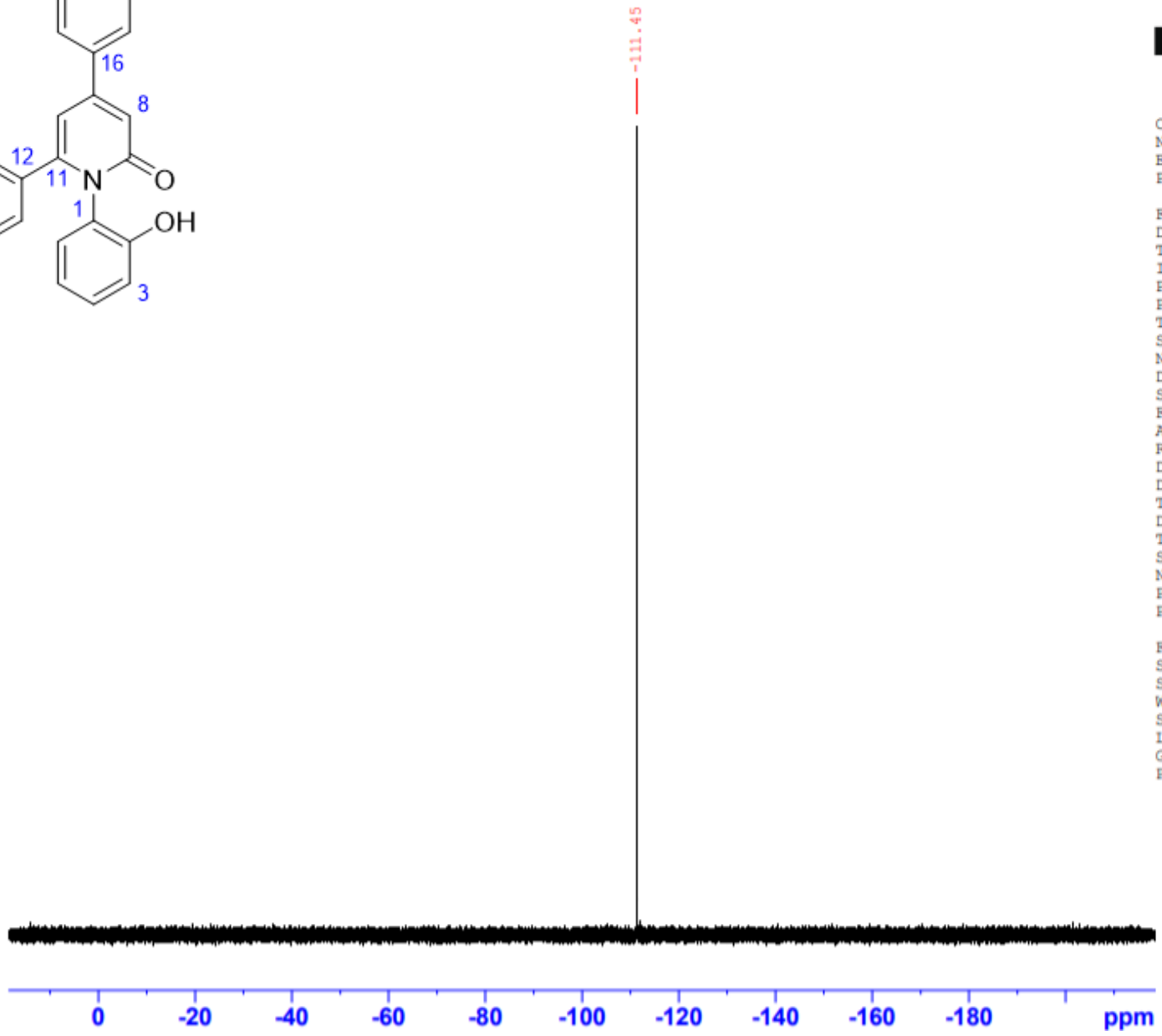
```
Current Data Parameters
NAME        JS-4-280 col
EXPNO                20
PROCNO                1

F2 - Acquisition Parameters
Date_          20210318
Time               23.13 h
INSTRUM       AVIII 400
PROBHD   Z108618_0146 (
PULPROG          zgpg30
TD                96150
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           24038.461 Hz
FIDRES        0.500020 Hz
AQ            1.9999200 sec
RG                 2050
DW               20.800 usec
DE                6.50 usec
TE               300.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1        100.6178003 MHz
NUC1                13C
P0                3.00 usec
P1                9.00 usec
PLW1         96.68000031 W
SFO2        400.1116004 MHz
NUC2                1H
CPDPRG[2        waltz64
PCPD2            90.00 usec
PLW2         17.29199982 W
PLW12         0.48032999 W
PLW13         0.24160001 W

F2 - Processing parameters
SI                131072
SF          100.6077405 MHz
WDW                 EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

3c - ^{19}F NMR, 376 MHz, $CDCl_3$



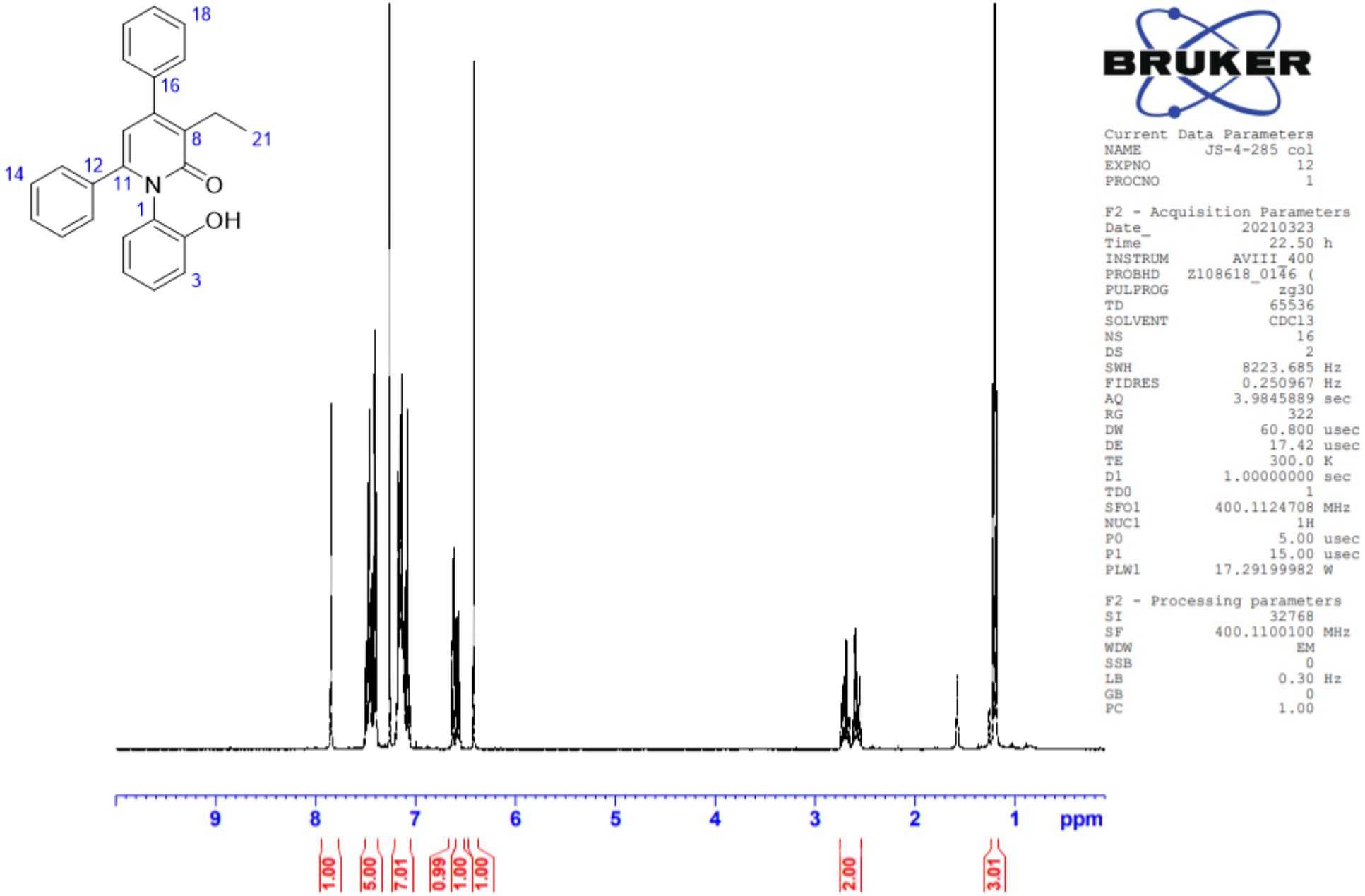



```
Current Data Parameters
NAME        JS-4-280 col
EXPNO                47
PROCNO                1

F2 - Acquisition Parameters
Date_          20210505
Time               13.55 h
INSTRUM       AVIII_400
PROBHD   Z108618_0146 (
PULPROG              zg
TD               261992
SOLVENT            CDCl3
NS                    16
DS                     4
SWH            89285.711 Hz
FIDRES         0.681591 Hz
AQ             1.4671552 sec
RG                   575
DW                 5.600 usec
DE                 7.11 usec
TE                 300.0 K
D1            1.00000000 sec
TD0                    1
SFO1         376.4418995 MHz
NUC1                 19F
P1                11.80 usec
PLW1         32.96500015 W

F2 - Processing parameters
SI                262144
SF         376.4795470 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

3d - ^{1}H NMR, 400 MHz, $CDCl_3$

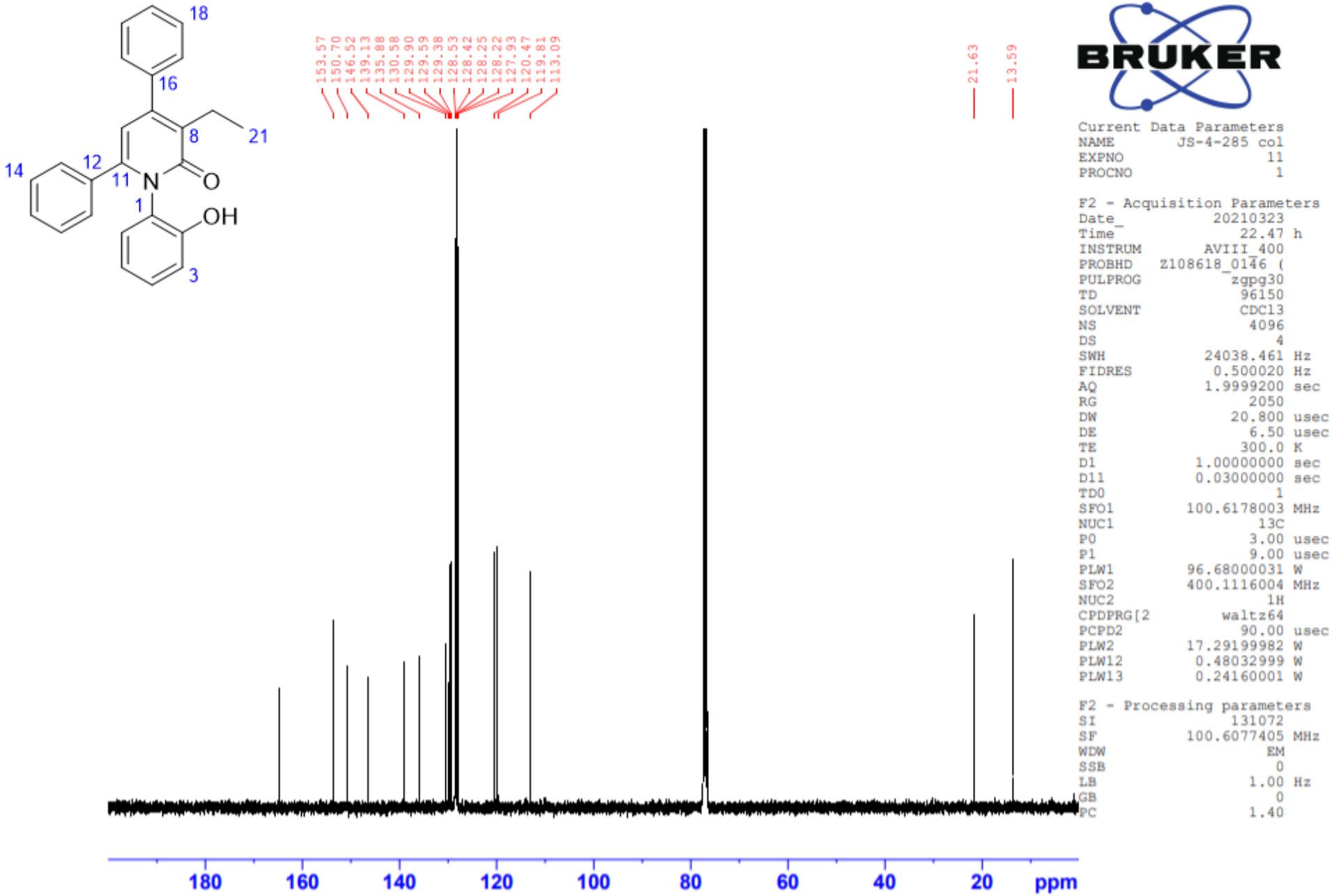



```
Current Data Parameters
NAME        JS-4-285 col
EXPNO                12
PROCNO                1

F2 - Acquisition Parameters
Date_          20210323
Time             22.50 h
INSTRUM       AVIII 400
PROBHD    Z108618_0146 (
PULPROG          zg30
TD              65536
SOLVENT         CDCl3
NS                 16
DS                  2
SWH          8223.685 Hz
FIDRES       0.250967 Hz
AQ           3.9845889 sec
RG                322
DW             60.800 usec
DE             17.42 usec
TE             300.0 K
D1          1.00000000 sec
TD0                 1
SFO1       400.1124708 MHz
NUC1               1H
P0              5.00 usec
P1             15.00 usec
PLW1       17.29199982 W

F2 - Processing parameters
SI              32768
SF         400.1100100 MHz
WDW               EM
SSB                0
LB              0.30 Hz
GB                 0
PC              1.00
```

3d - ^{13}C NMR, 101 MHz, $CDCl_3$



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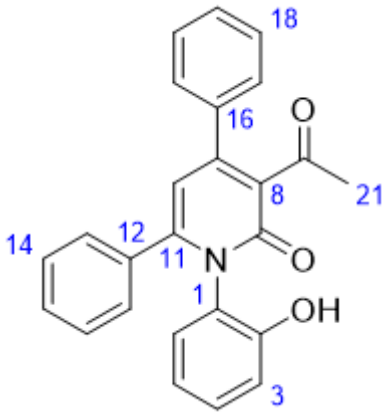
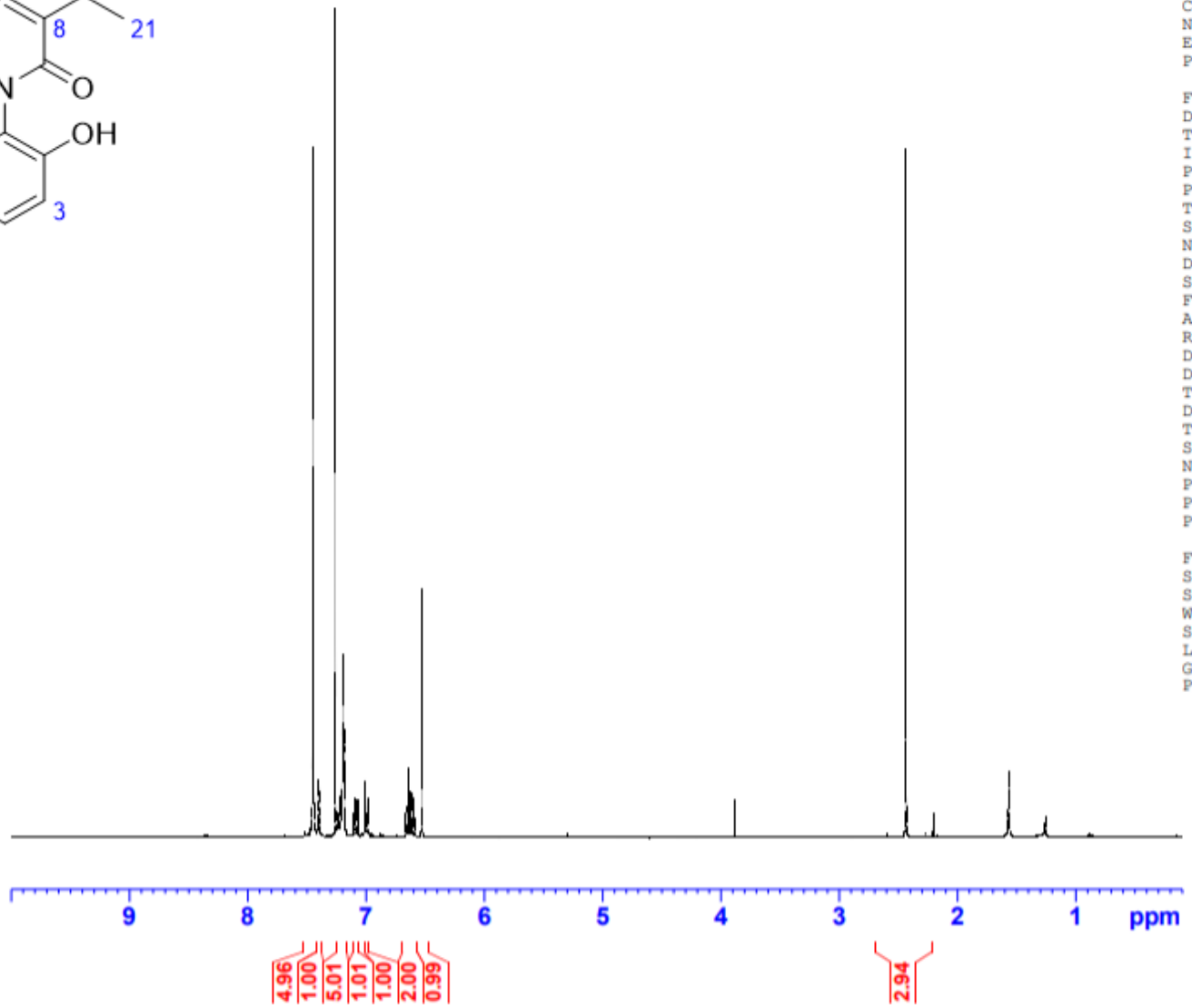
```
Current Data Parameters
NAME          JS-4-285 col
EXPNO                   11
PROCNO                   1

F2 - Acquisition Parameters
Date_              20210323
Time                 22.47 h
INSTRUM          AVIII_400
PROBHD    Z108618_0146 (
PULPROG             zgpg30
TD                   96150
SOLVENT              CDCl3
NS                    4096
DS                       4
SWH              24038.461 Hz
FIDRES           0.500020 Hz
AQ               1.9999200 sec
RG                    2050
DW                  20.800 usec
DE                   6.50 usec
TE                  300.0 K
D1               1.00000000 sec
D11              0.03000000 sec
TD0                      1
SFO1           100.6178003 MHz
NUC1                   13C
P0                   3.00 usec
P1                   9.00 usec
PLW1           96.68000031 W
SFO2           400.1116004 MHz
NUC2                   1H
CPDPRG[2           waltz64
PCPD2                90.00 usec
PLW2           17.29199982 W
PLW12           0.48032999 W
PLW13           0.24160001 W

F2 - Processing parameters
SI                   131072
SF             100.6077405 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

3e - ^{1}H NMR, 400 MHz, $CDCl_3$



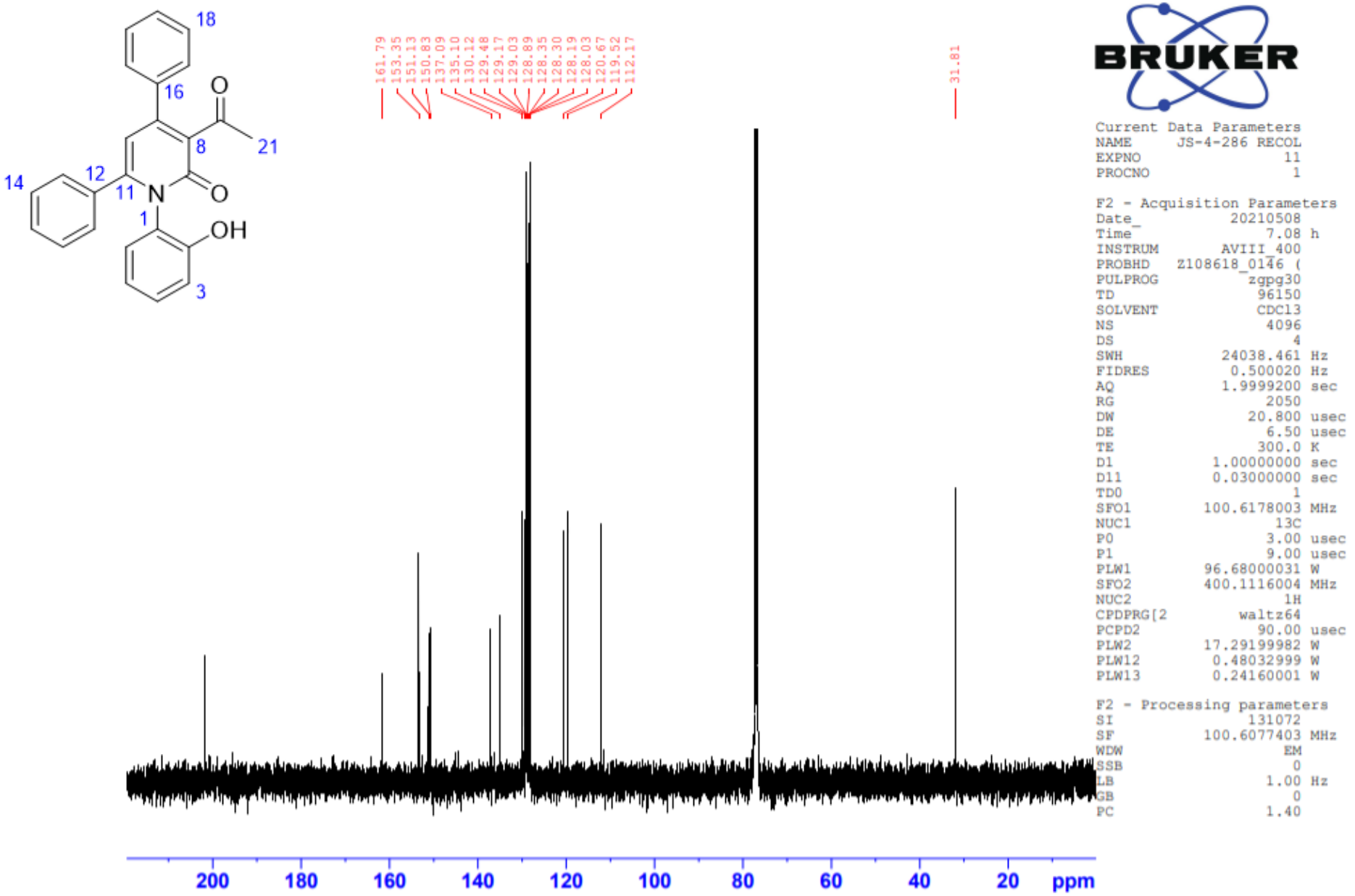



```
Current Data Parameters
NAME      JS-4-286 RECOL
EXPNO               10
PROCNO               1

F2 - Acquisition Parameters
Date_          20210507
Time             17.10 h
INSTRUM       AVIII_400
PROBHD   Z108618_0146 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.250967 Hz
AQ             3.9845889 sec
RG                  256
DW               60.800 usec
DE               17.42 usec
TE               300.0 K
D1            1.00000000 sec
TD0                   1
SFO1         400.1124708 MHz
NUC1                 1H
P0                5.00 usec
P1               15.00 usec
PLW1         17.29199982 W

F2 - Processing parameters
SI                32768
SF           400.1100095 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                 1.00
```

3e - ^{13}C NMR, 101 MHz, $CDCl_3$



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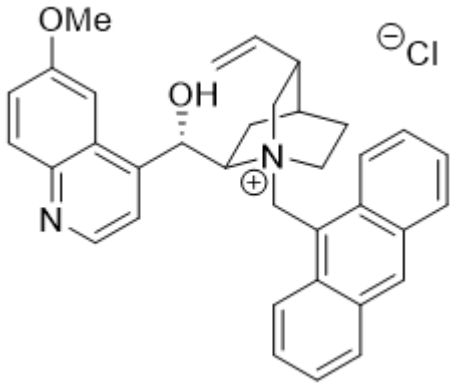
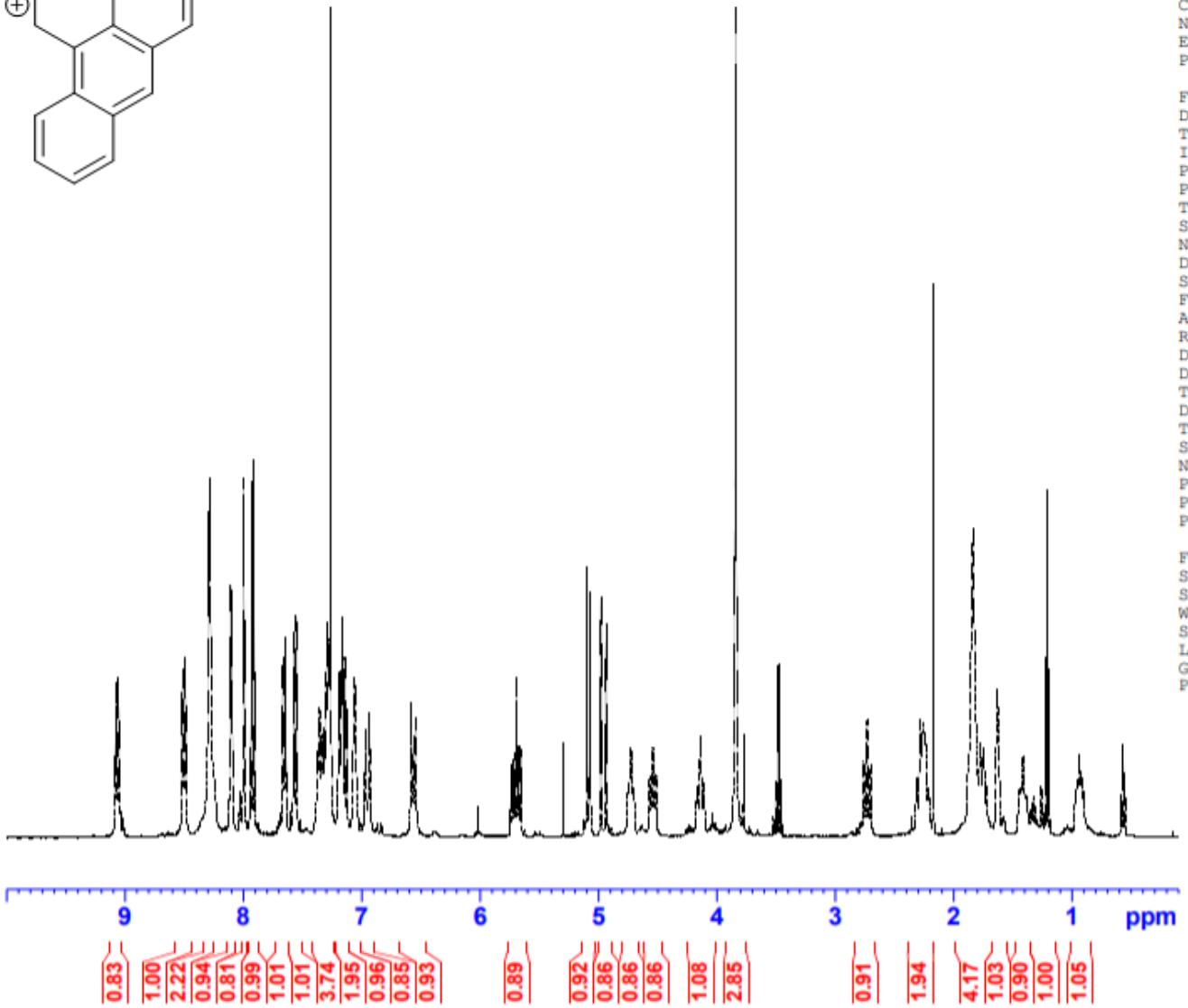
```
Current Data Parameters
NAME      JS-4-286 RECOL
EXPNO                11
PROCNO                1

F2 - Acquisition Parameters
Date_          20210508
Time               7.08 h
INSTRUM       AVIII_400
PROBHD    Z108618_0146 (
PULPROG          zgpg30
TD                96150
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           24038.461 Hz
FIDRES         0.500020 Hz
AQ             1.9999200 sec
RG                 2050
DW               20.800 usec
DE                6.50 usec
TE                300.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1        100.6178003 MHz
NUC1                13C
P0                3.00 usec
P1                9.00 usec
PLW1         96.68000031 W
SFO2        400.1116004 MHz
NUC2                1H
CPDPRG[2        waltz64
PCPD2            90.00 usec
PLW2         17.29199982 W
PLW12         0.48032999 W
PLW13         0.24160001 W

F2 - Processing parameters
SI                131072
SF          100.6077403 MHz
WDW                 EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

QD-1 - ^{1}H NMR, 400 MHz, $CDCl_3$



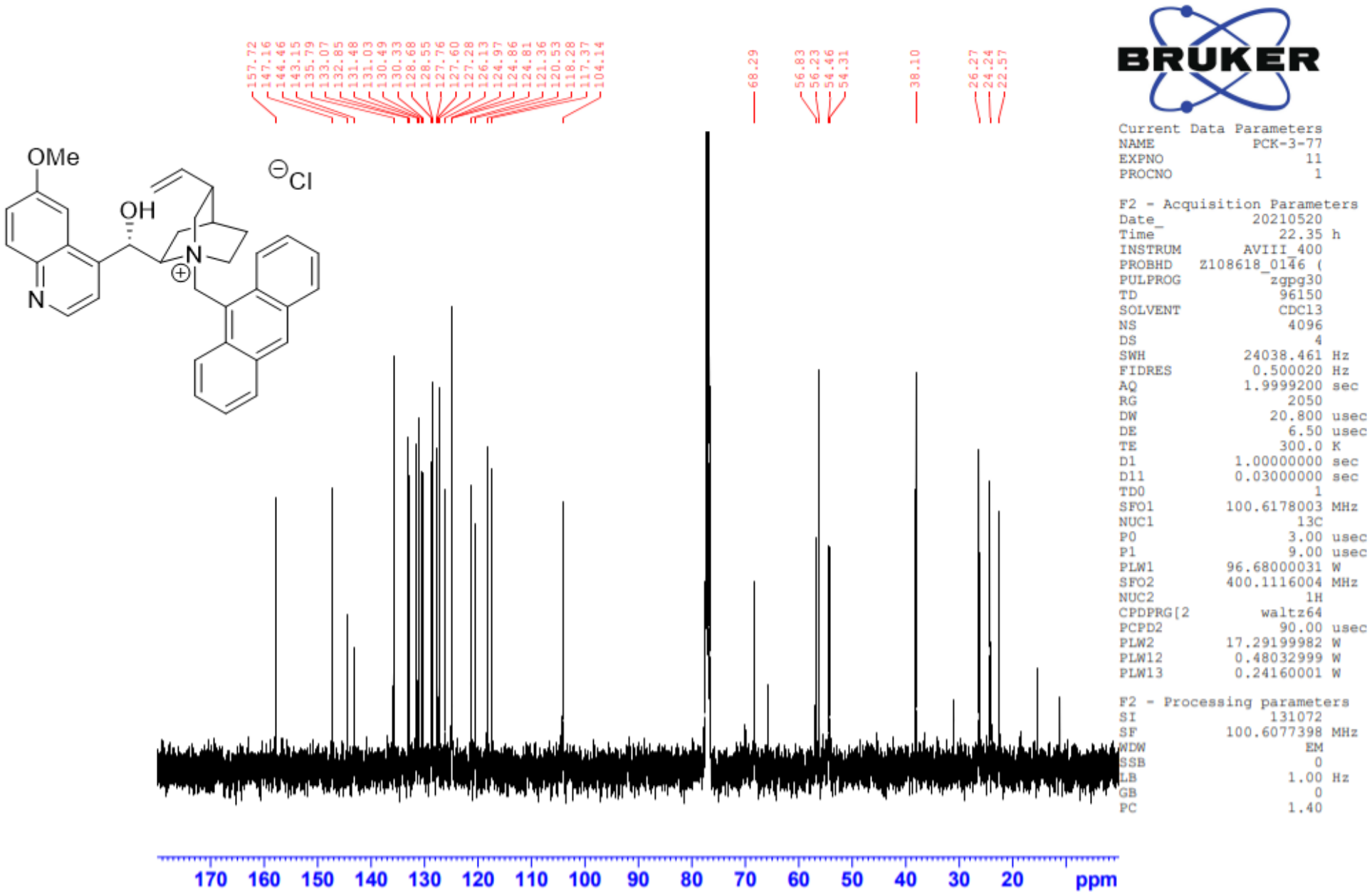



```
Current Data Parameters
NAME                 PCK-3-77
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_               20210520
Time                  17.06 h
INSTRUM             AVIII_400
PROBHD    Z108618_0146 (
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                        128
DS                          2
SWH                  8223.685 Hz
FIDRES               0.250967 Hz
AQ                   3.9845889 sec
RG                        256
DW                     60.800 usec
DE                     17.42 usec
TE                     300.0 K
D1                1.00000000 sec
TD0                         1
SFO1             400.1124708 MHz
NUC1                       1H
P0                     5.00 usec
P1                    15.00 usec
PLW1             17.29199982 W

F2 - Processing parameters
SI                      32768
SF               400.1100067 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                     1.00
```

QD-1 - ^{13}C NMR, 101 MHz, $CDCl_3$

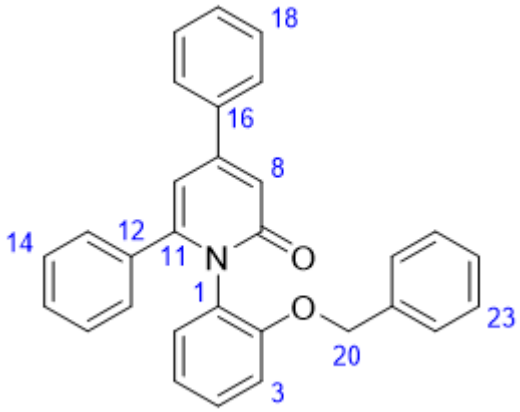


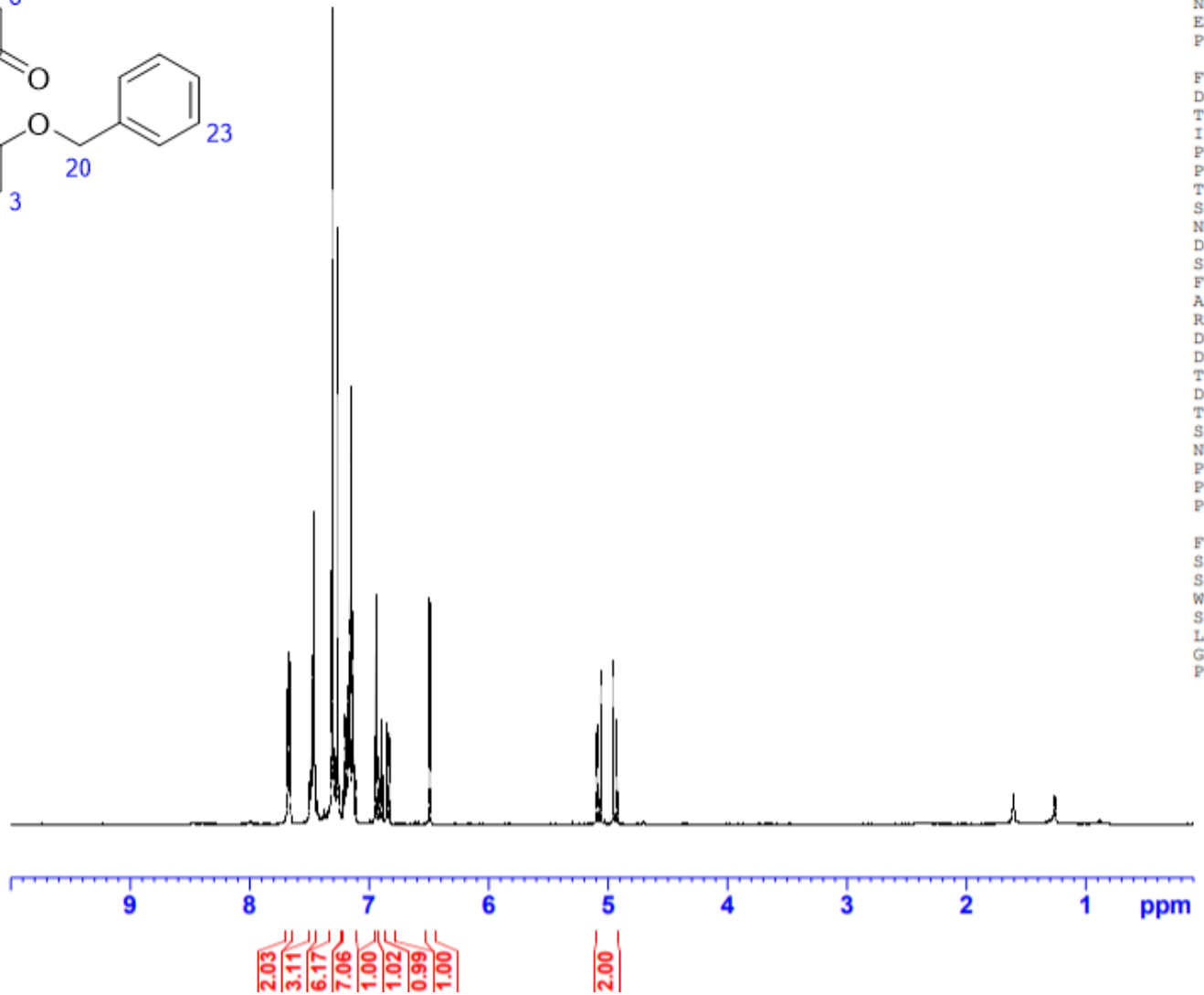

```
Current Data Parameters
NAME                  PCK-3-77
EXPNO                       11
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20210520
Time                     22.35 h
INSTRUM               AVIII_400
PROBHD    Z108618_0146 (
PULPROG                 zgpg30
TD                       96150
SOLVENT                  CDCl3
NS                        4096
DS                           4
SWH                  24038.461 Hz
FIDRES                0.500020 Hz
AQ                    1.9999200 sec
RG                        2050
DW                      20.800 usec
DE                       6.50 usec
TE                      300.0 K
D1                    1.00000000 sec
D11                   0.03000000 sec
TD0                          1
SFO1                100.6178003 MHz
NUC1                        13C
P0                       3.00 usec
P1                       9.00 usec
PLW1                96.68000031 W
SFO2                400.1116004 MHz
NUC2                        1H
CPDPRG[2                waltz64
PCPD2                   90.00 usec
PLW2                17.29199982 W
PLW12                0.48032999 W
PLW13                0.24160001 W

F2 - Processing parameters
SI                       131072
SF                  100.6077398 MHz
WDW                         EM
SSB                          0
LB                       1.00 Hz
GB                           0
PC                       1.40
```

4a - ^{1}H NMR, 400 MHz, $CDCl_3$



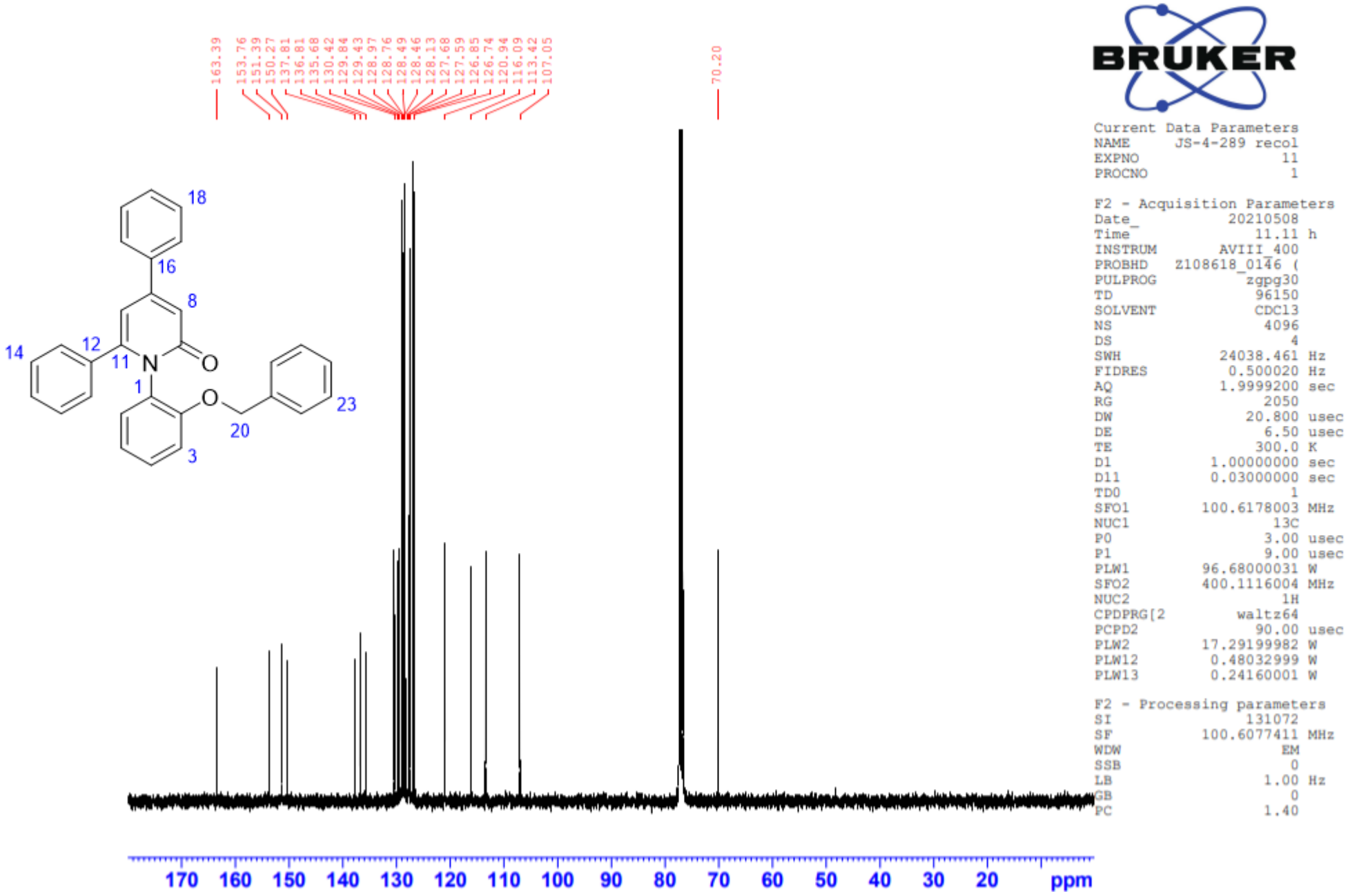



```
Current Data Parameters
NAME      JS-4-289 recol
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210507
Time              17.16 h
INSTRUM        AVIII_400
PROBHD   Z108618_0146 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ              3.9845889 sec
RG                   256
DW                60.800 usec
DE                17.42 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1
SFO1         400.1124708 MHz
NUC1                  1H
P0                 5.00 usec
P1                15.00 usec
PLW1         17.29199982 W

F2 - Processing parameters
SI                 32768
SF           400.1100095 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

4a - ^{13}C NMR, 101 MHz, $CDCl_3$

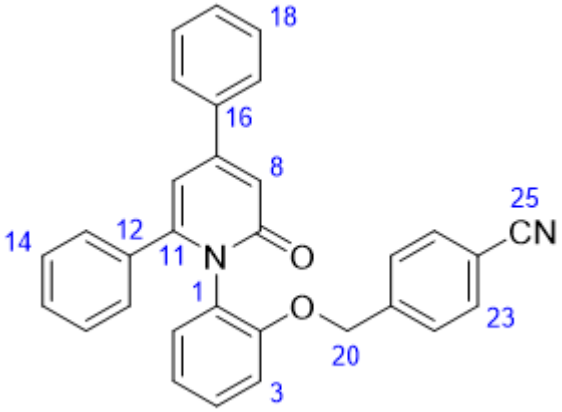


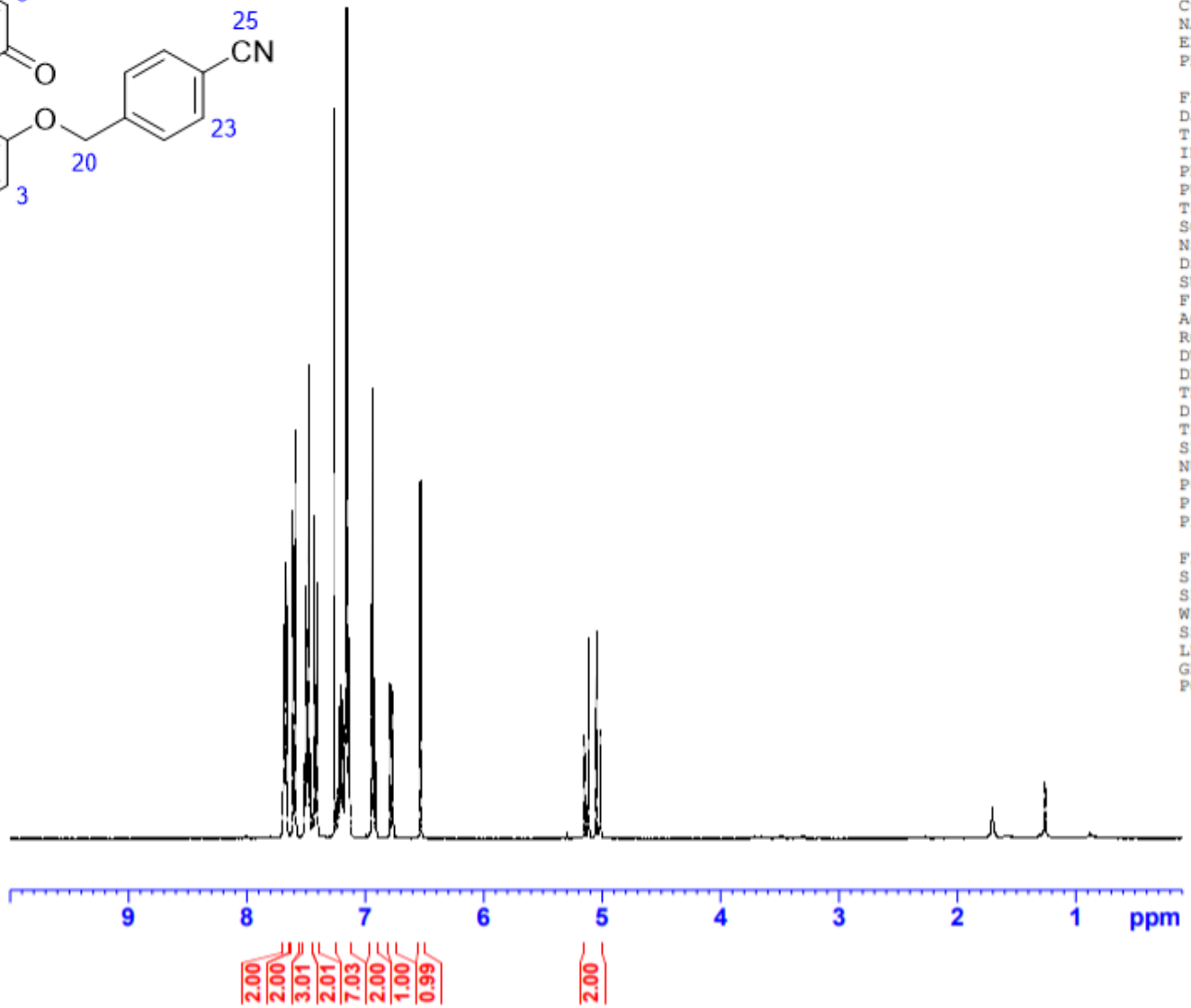

```
Current Data Parameters
NAME      JS-4-289 recol
EXPNO                11
PROCNO                1

F2 - Acquisition Parameters
Date_          20210508
Time              11.11 h
INSTRUM        AVIII_400
PROBHD   Z108618_0146 (
PULPROG          zgpg30
TD                96150
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           24038.461 Hz
FIDRES         0.500020 Hz
AQ             1.9999200 sec
RG                 2050
DW               20.800 usec
DE                6.50 usec
TE               300.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         100.6178003 MHz
NUC1                13C
P0                3.00 usec
P1                9.00 usec
PLW1         96.68000031 W
SFO2         400.1116004 MHz
NUC2                1H
CPDPRG[2         waltz64
PCPD2             90.00 usec
PLW2         17.29199982 W
PLW12         0.48032999 W
PLW13         0.24160001 W

F2 - Processing parameters
SI                131072
SF         100.6077411 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

4b - ^{1}H NMR, 400 MHz, $CDCl_3$



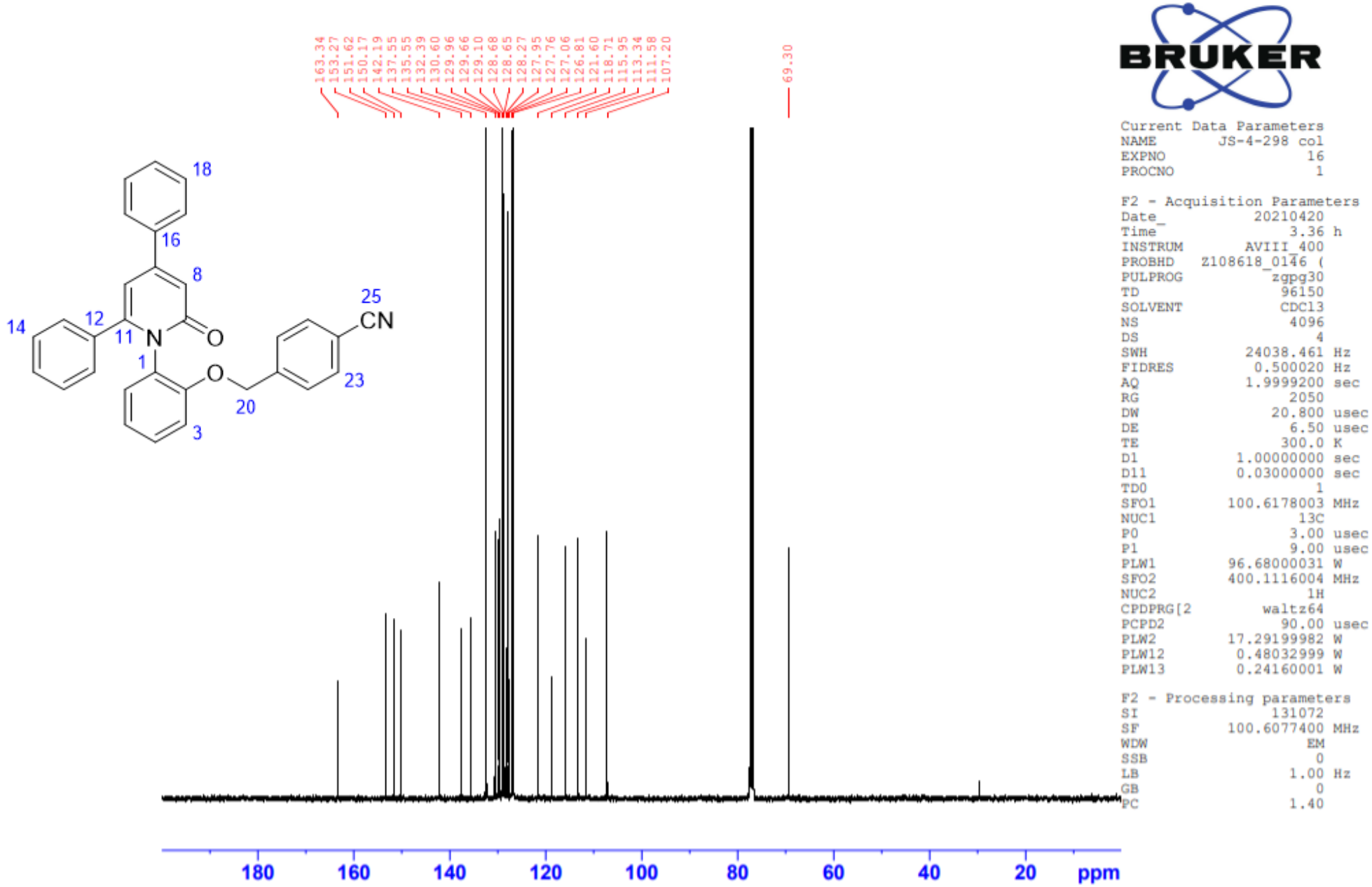



```
Current Data Parameters
NAME        JS-4-298 col
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20210413
Time              15.47 h
INSTRUM        AVIII_400
PROBHD   Z108618_0146 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ              3.9845889 sec
RG                   144
DW                60.800 usec
DE                17.42 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1124708 MHz
NUC1                  1H
P0                 5.00 usec
P1                15.00 usec
PLW1          17.29199982 W

F2 - Processing parameters
SI                 32768
SF            400.1100099 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

4b - ^{13}C NMR, 101 MHz, $CDCl_3$



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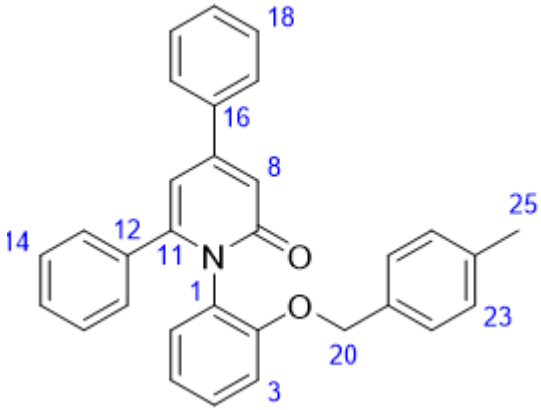
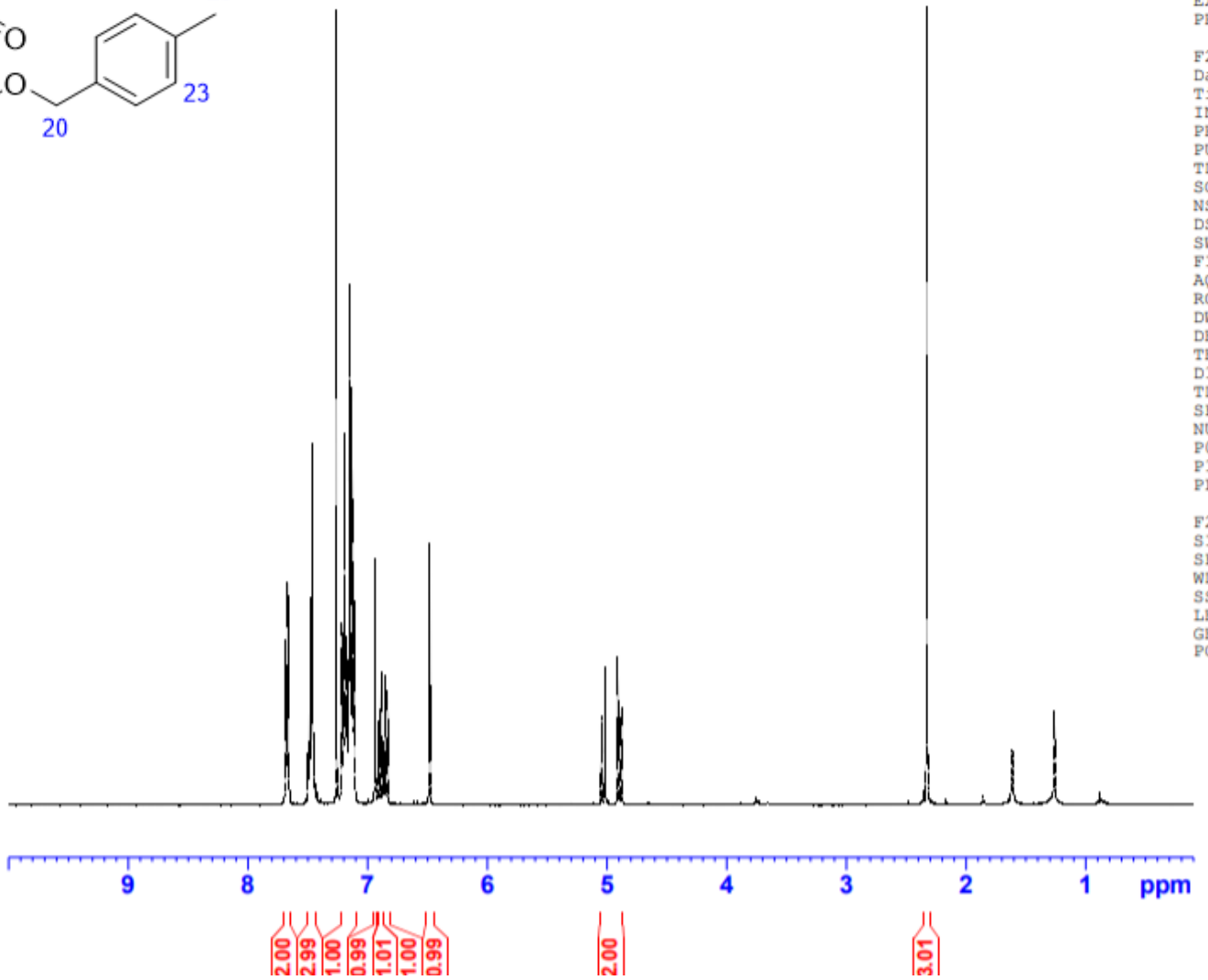
```
Current Data Parameters
NAME        JS-4-298 col
EXPNO                 16
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210420
Time               3.36 h
INSTRUM       AVIII_400
PROBHD   Z108618_0146 (
PULPROG          zgpg30
TD                96150
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           24038.461 Hz
FIDRES         0.500020 Hz
AQ             1.9999200 sec
RG                 2050
DW                20.800 usec
DE                 6.50 usec
TE                300.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1        100.6178003 MHz
NUC1                13C
P0                 3.00 usec
P1                 9.00 usec
PLW1         96.68000031 W
SFO2        400.1116004 MHz
NUC2                1H
CPDPRG[2        waltz64
PCPD2             90.00 usec
PLW2         17.29199982 W
PLW12         0.48032999 W
PLW13         0.24160001 W

F2 - Processing parameters
SI                131072
SF          100.6077400 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

4c - ^{1}H NMR, 400 MHz, $CDCl_3$



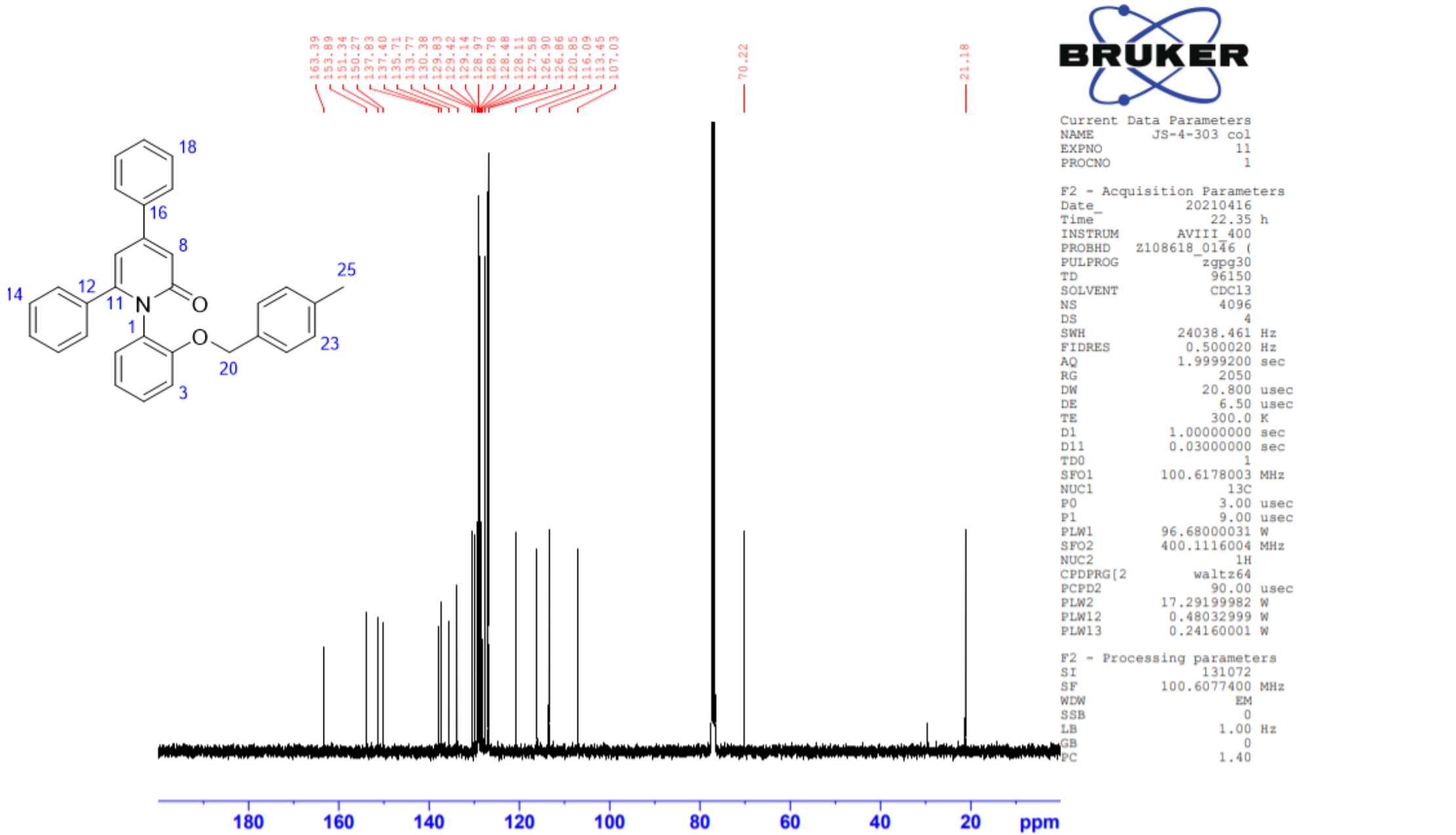



```
Current Data Parameters
NAME        JS-4-303 col
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20210416
Time             10.29 h
INSTRUM        AVIII 400
PROBHD   Z108618_0146 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           8223.685 Hz
FIDRES        0.250967 Hz
AQ            3.9845889 sec
RG                   256
DW                60.800 usec
DE                17.42 usec
TE                300.0 K
D1           1.00000000 sec
TD0                    1
SFO1        400.1124708 MHz
NUC1                  1H
P0                 5.00 usec
P1                15.00 usec
PLW1        17.29199982 W

F2 - Processing parameters
SI                 32768
SF          400.1100100 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

4c - ^{13}C NMR, 101 MHz, $CDCl_3$



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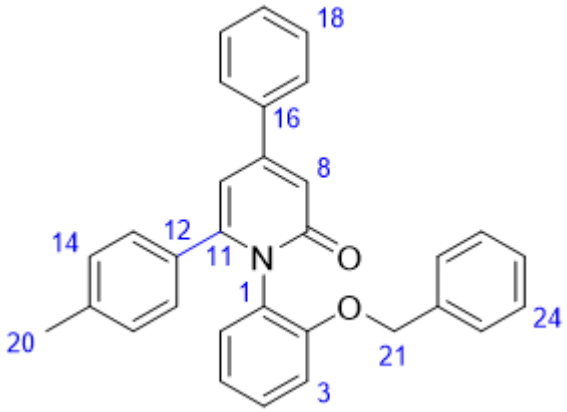
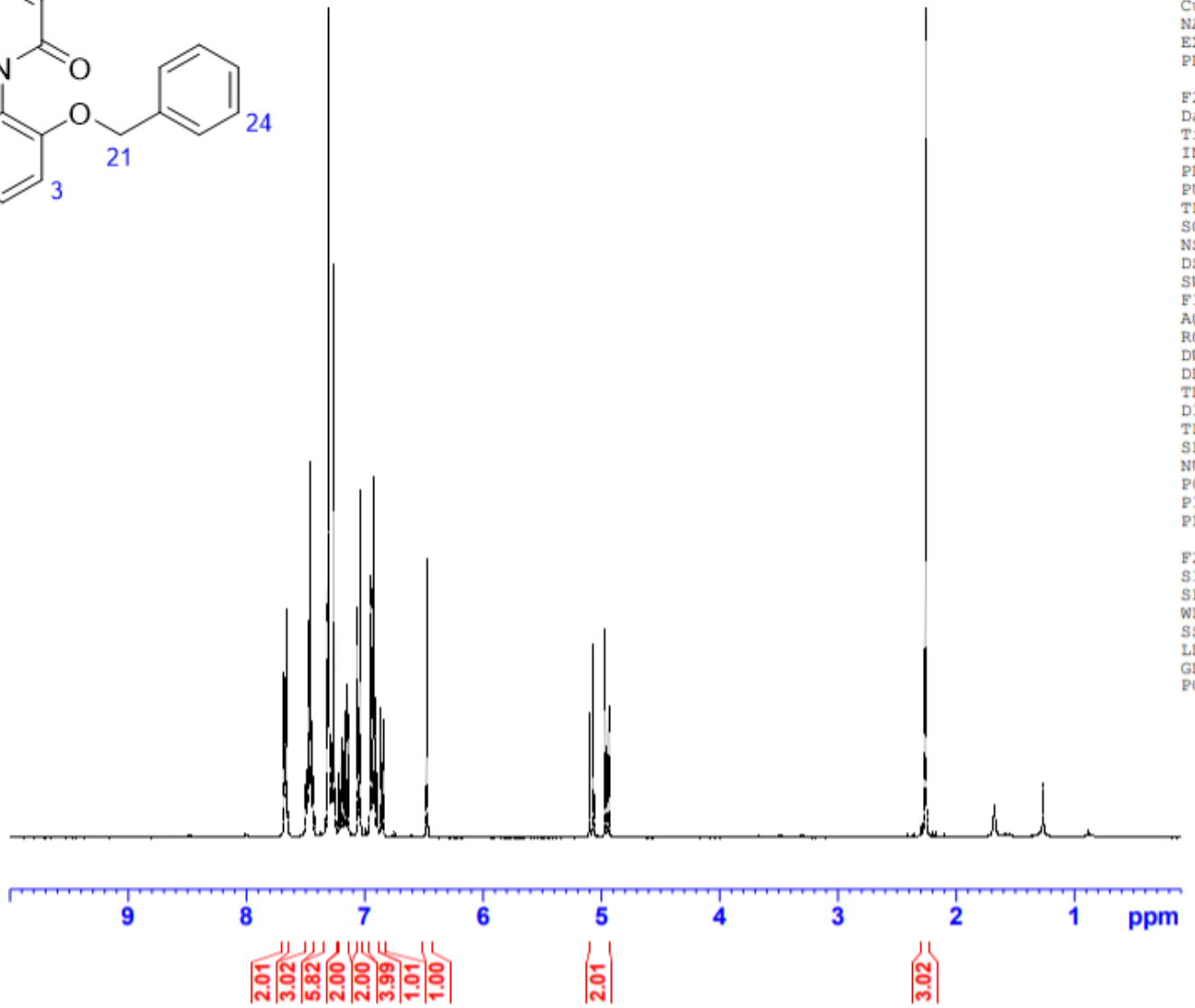
```
Current Data Parameters
NAME        JS-4-303 col
EXPNO                11
PROCNO                1

F2 - Acquisition Parameters
Date_          20210416
Time             22.35 h
INSTRUM       AVIII_400
PROBHD   Z108618_0146 (
PULPROG          zgpg30
TD                96150
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           24038.461 Hz
FIDRES         0.500020 Hz
AQ             1.9999200 sec
RG                 2050
DW               20.800 usec
DE                6.50 usec
TE               300.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         100.6178003 MHz
NUC1                13C
P0                3.00 usec
P1                9.00 usec
PLW1         96.68000031 W
SFO2         400.1116004 MHz
NUC2                1H
CPDPRG[2         waltz64
PCPD2             90.00 usec
PLW2         17.29199982 W
PLW12         0.48032999 W
PLW13         0.24160001 W

F2 - Processing parameters
SI                131072
SF           100.6077400 MHz
WDW                 EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

4d - ^{1}H NMR, 400 MHz, $CDCl_3$



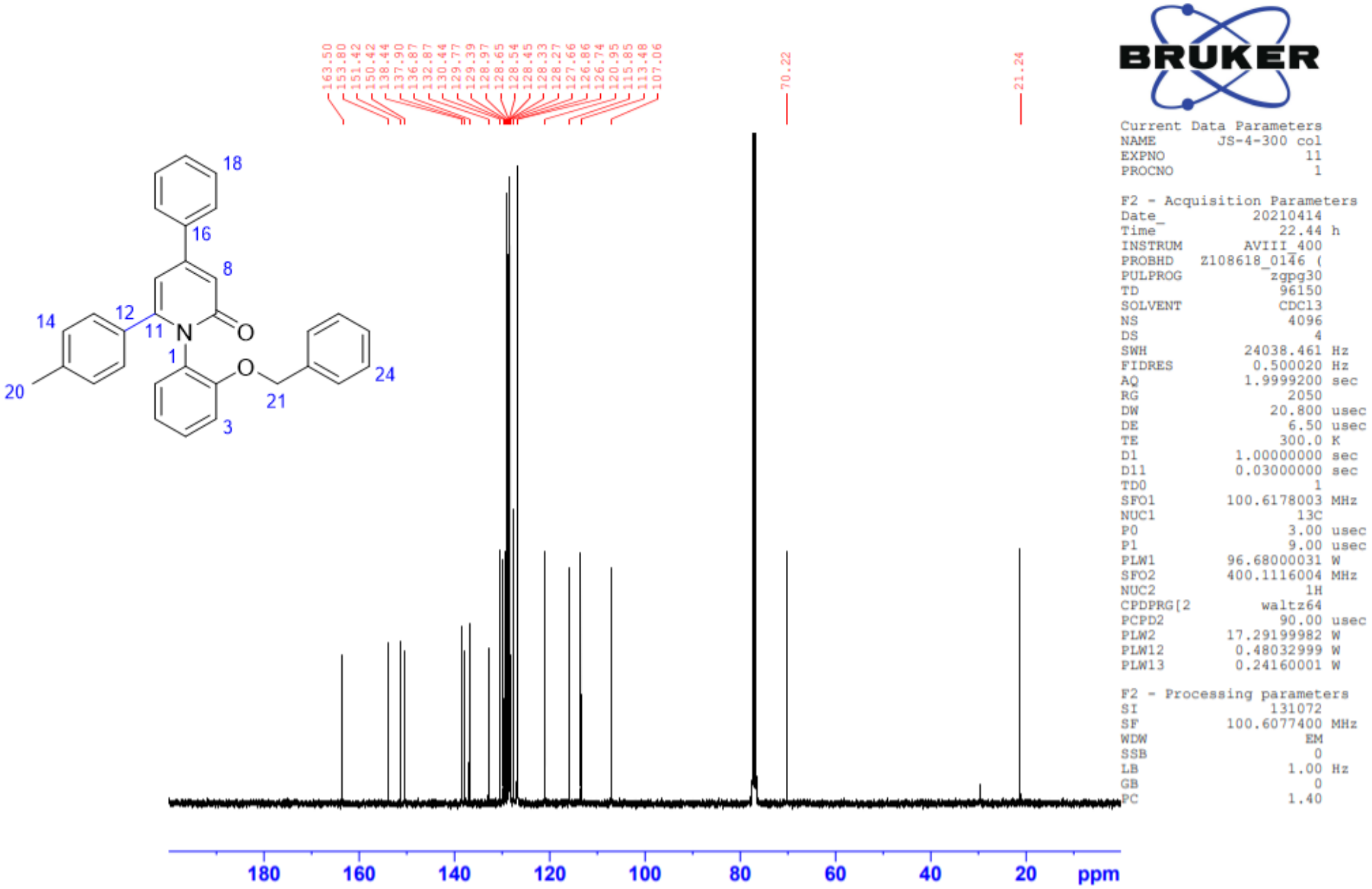



```
Current Data Parameters
NAME       JS-4-300 col
EXPNO               10
PROCNO               1

F2 - Acquisition Parameters
Date_          20210414
Time              15.25 h
INSTRUM       AVIII_400
PROBHD   Z108618_0146 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.250967 Hz
AQ             3.9845889 sec
RG                  161
DW                60.800 usec
DE                17.42 usec
TE                300.0 K
D1           1.00000000 sec
TD0                  1
SFO1        400.1124708 MHz
NUC1                1H
P0                5.00 usec
P1               15.00 usec
PLW1        17.29199982 W

F2 - Processing parameters
SI                32768
SF          400.1100098 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```

4d - ^{13}C NMR, 101 MHz, $CDCl_3$

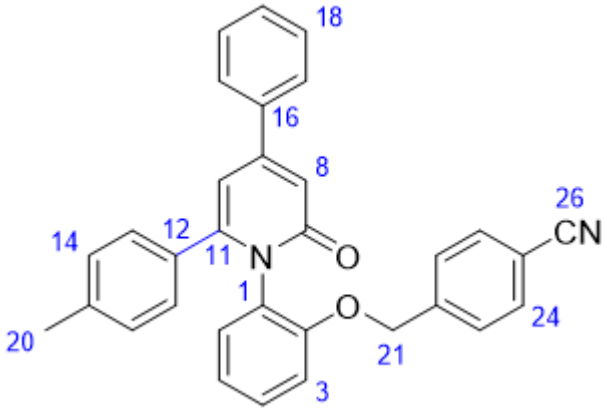


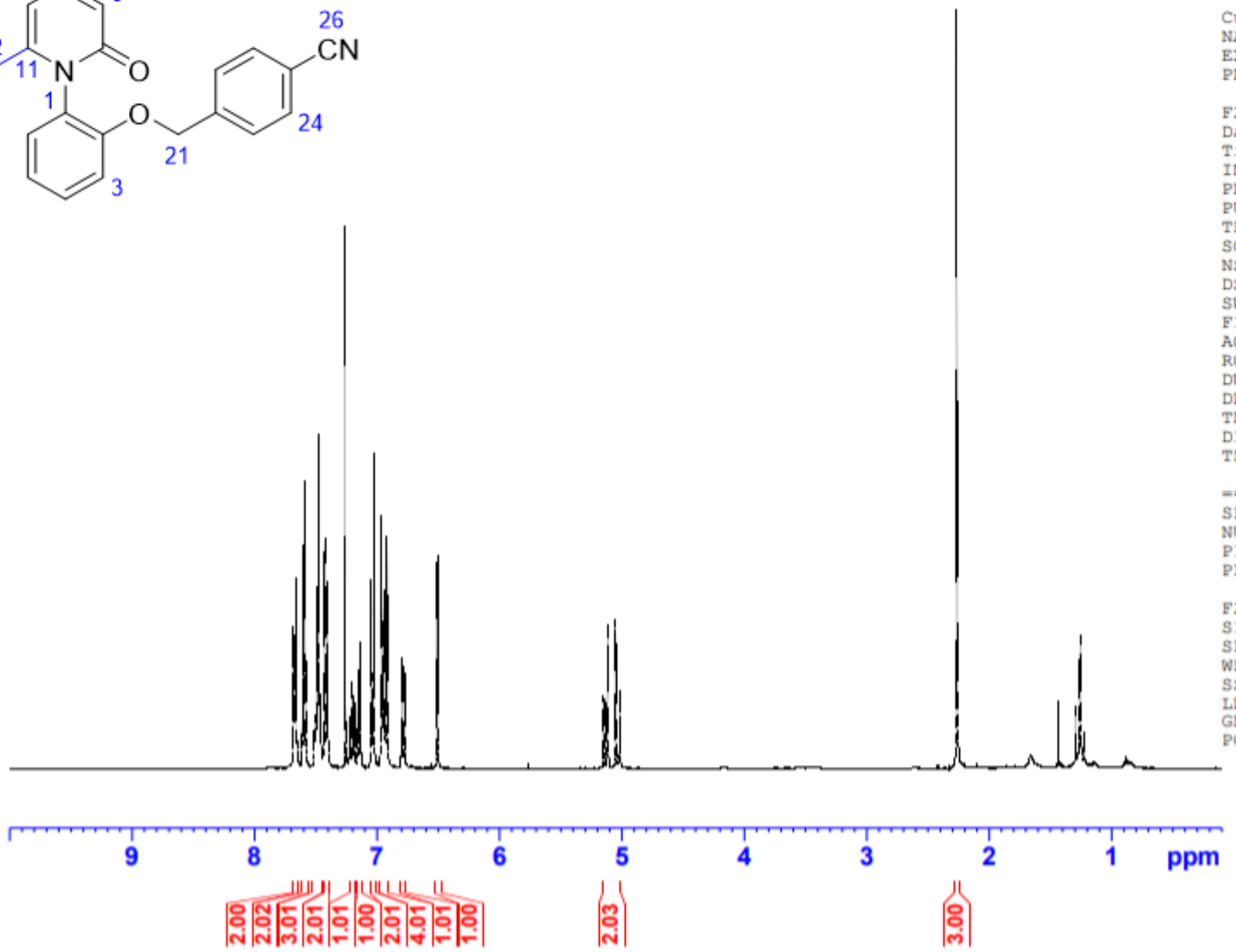

```
Current Data Parameters
NAME                  JS-4-300 col
EXPNO                          11
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20210414
Time                        22.44 h
INSTRUM                  AVIII_400
PROBHD    Z108618_0146 (
PULPROG                    zgpg30
TD                          96150
SOLVENT                     CDCl3
NS                           4096
DS                              4
SWH                     24038.461 Hz
FIDRES                   0.500020 Hz
AQ                       1.9999200 sec
RG                           2050
DW                         20.800 usec
DE                          6.50 usec
TE                         300.0 K
D1                       1.00000000 sec
D11                      0.03000000 sec
TD0                             1
SFO1                  100.6178003 MHz
NUC1                          13C
P0                          3.00 usec
P1                          9.00 usec
PLW1                 96.68000031 W
SFO2                  400.1116004 MHz
NUC2                          1H
CPDPRG[2                 waltz64
PCPD2                      90.00 usec
PLW2                 17.29199982 W
PLW12                 0.48032999 W
PLW13                 0.24160001 W

F2 - Processing parameters
SI                          131072
SF                    100.6077400 MHz
WDW                            EM
SSB                             0
LB                           1.00 Hz
GB                              0
PC                           1.40
```

4e - ^{1}H NMR, 400 MHz, $CDCl_3$





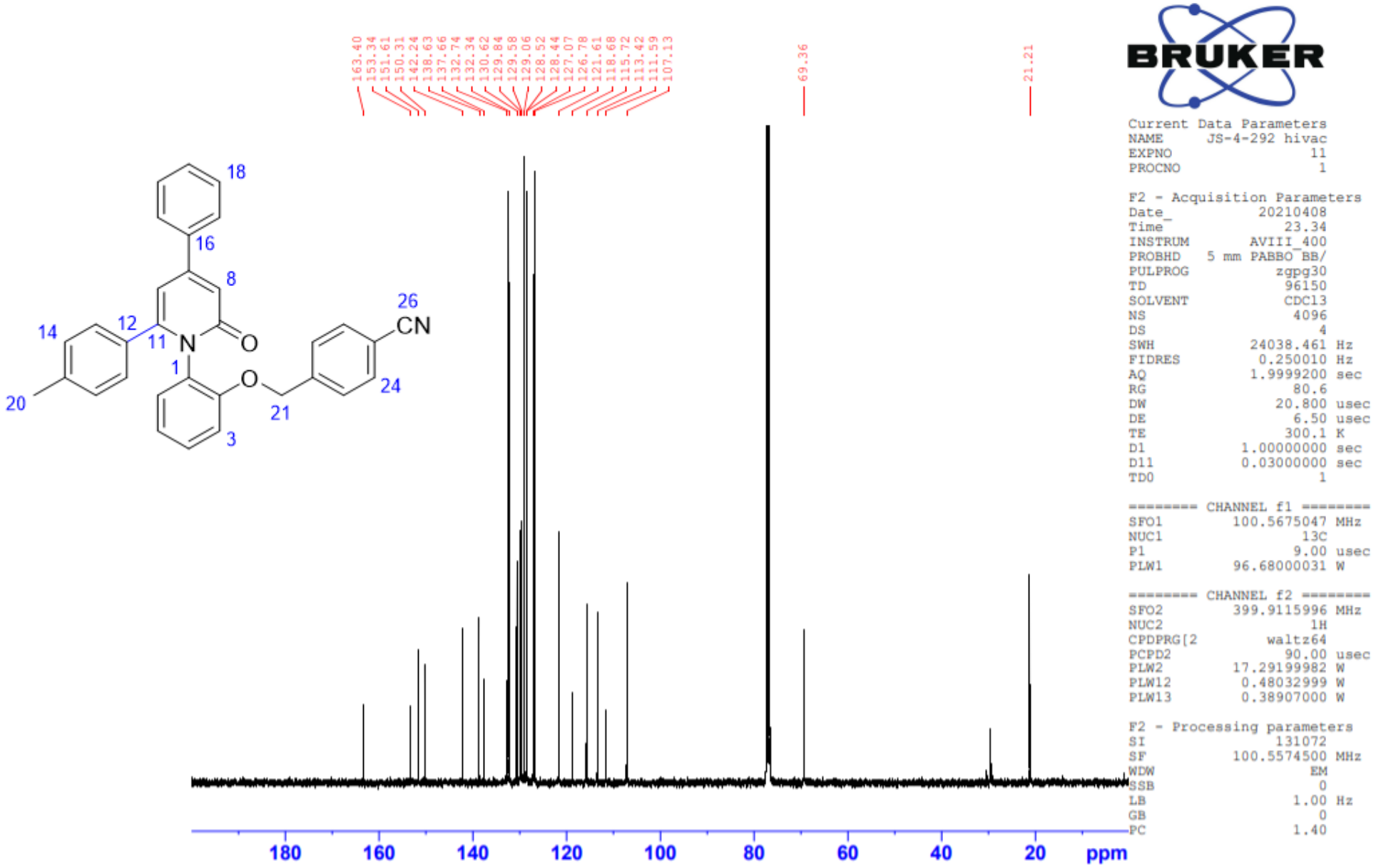

```
Current Data Parameters
NAME      JS-4-292 hivac
EXPNO                 12
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210408
Time              23.38
INSTRUM        AVIII_400
PROBHD    5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ             3.9845889 sec
RG                80.6
DW               60.800 usec
DE                6.50 usec
TE                295.9 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         399.9124696 MHz
NUC1                 1H
P1                15.00 usec
PLW1         17.29199982 W

F2 - Processing parameters
SI                32768
SF           399.9100098 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

4e - ^{13}C NMR, 101 MHz, $CDCl_3$



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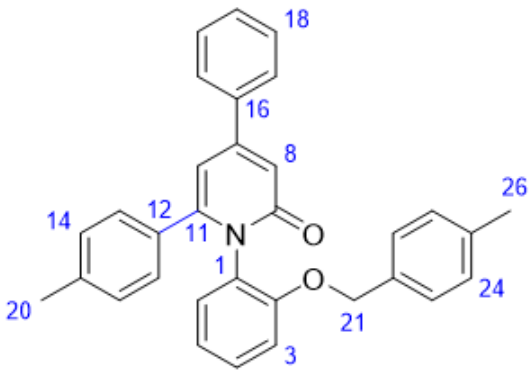
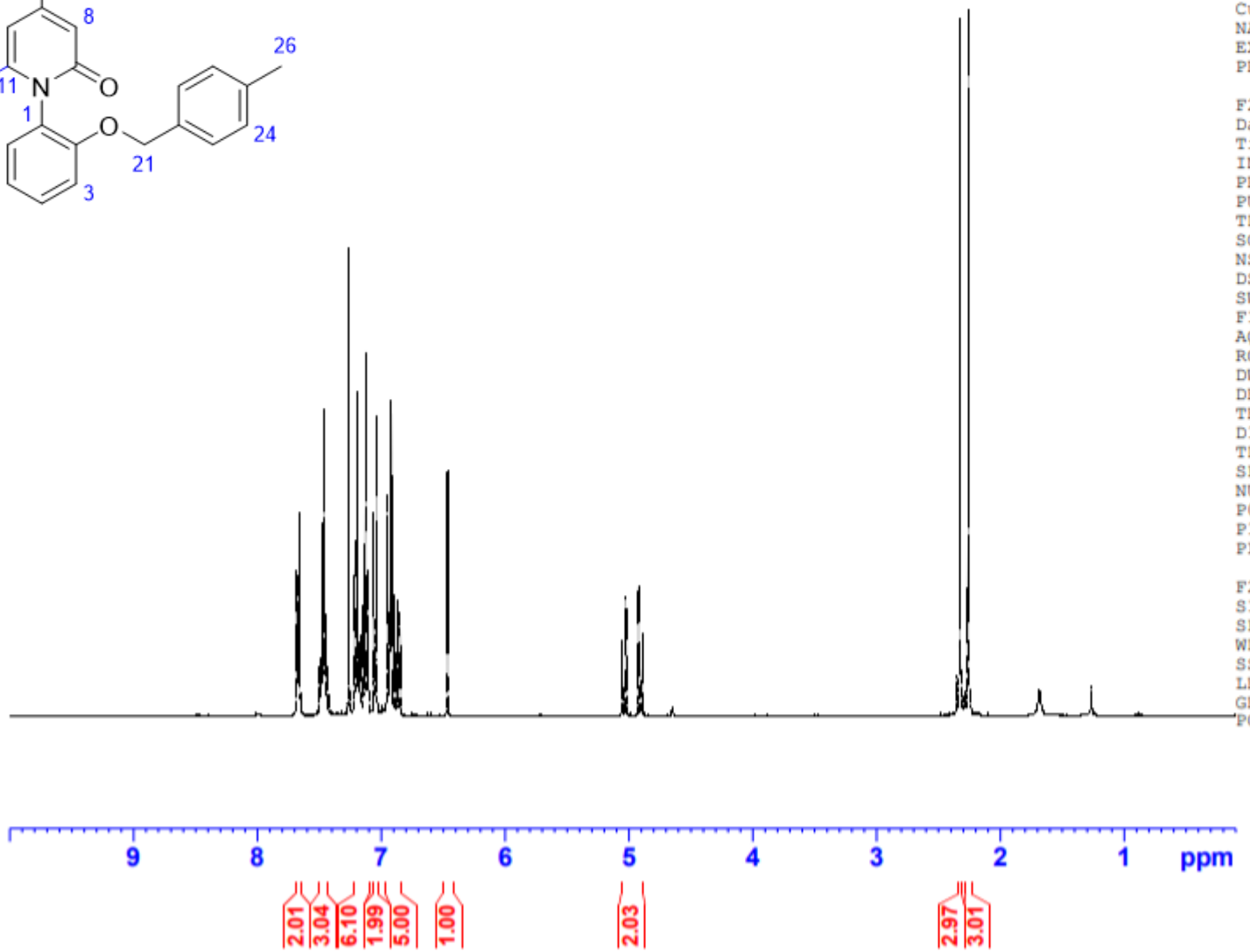
```
Current Data Parameters
NAME      JS-4-292 hivac
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210408
Time               23.34
INSTRUM         AVIII_400
PROBHD    5 mm PABBO BB/
PULPROG          zgpg30
TD                96150
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           24038.461 Hz
FIDRES         0.250010 Hz
AQ             1.9999200 sec
RG                 80.6
DW                20.800 usec
DE                 6.50 usec
TE                300.1 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.5675047 MHz
NUC1                 13C
P1                  9.00 usec
PLW1         96.68000031 W

======== CHANNEL f2 ========
SFO2         399.9115996 MHz
NUC2                 1H
CPDPRG[2          waltz64
PCPD2               90.00 usec
PLW2         17.29199982 W
PLW12         0.48032999 W
PLW13         0.38907000 W

F2 - Processing parameters
SI                 131072
SF           100.5574500 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

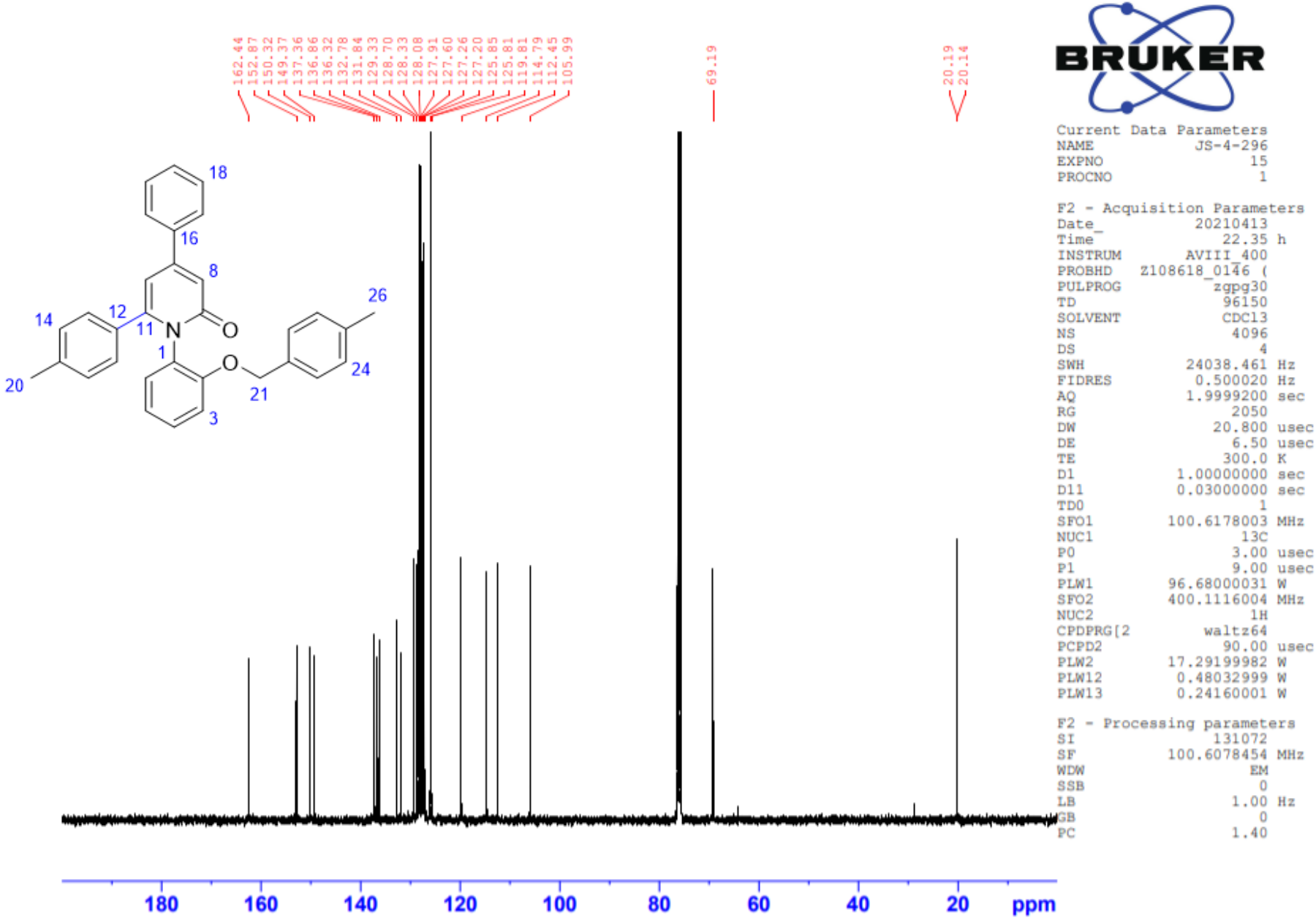
4f - ^{1}H NMR, 400 MHz, $CDCl_3$





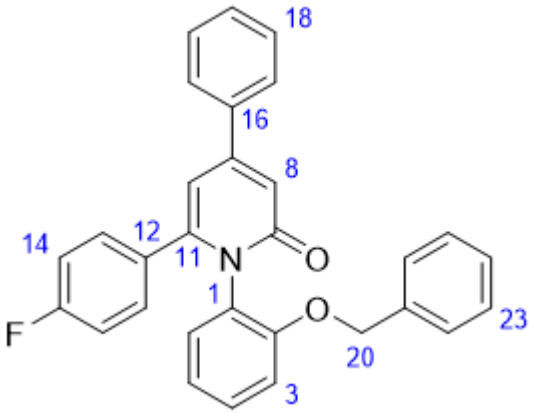

```
Current Data Parameters
NAME                  JS-4-296
EXPNO                       14
PROCNO                       1

F2 - Acquisition Parameters
Date_                  20210413
Time                      13.15 h
INSTRUM                AVIII_400
PROBHD    Z108618_0146 (
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                           16
DS                            2
SWH                    8223.685 Hz
FIDRES                 0.250967 Hz
AQ                     3.9845889 sec
RG                          181
DW                       60.800 usec
DE                       17.42 usec
TE                       300.0 K
D1                    1.00000000 sec
TD0                           1
SFO1                400.1124708 MHz
NUC1                        1H
P0                        5.00 usec
P1                       15.00 usec
PLW1                17.29199982 W

F2 - Processing parameters
SI                        32768
SF                  400.1100099 MHz
WDW                          EM
SSB                           0
LB                       0.30 Hz
GB                            0
PC                        1.00
```

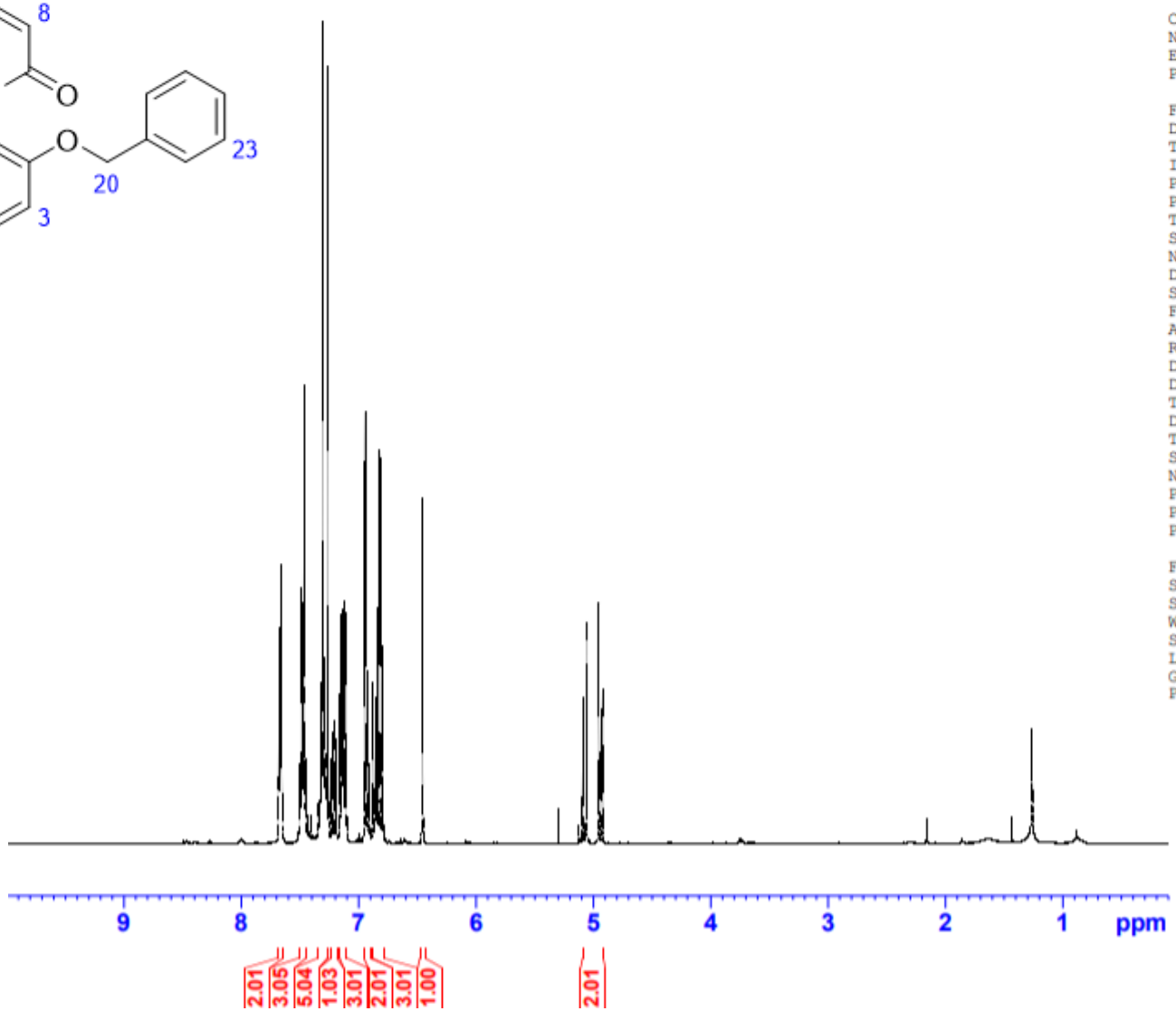
4f - ^{13}C NMR, 101 MHz, $CDCl_3$




```
Current Data Parameters
NAME                  JS-4-296
EXPNO                       15
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20210413
Time                     22.35 h
INSTRUM               AVIII 400
PROBHD    Z108618_0146 (
PULPROG                zgpg30
TD                      96150
SOLVENT                 CDCl3
NS                       4096
DS                          4
SWH                 24038.461 Hz
FIDRES                0.500020 Hz
AQ                    1.9999200 sec
RG                       2050
DW                     20.800 usec
DE                      6.50 usec
TE                      300.0 K
D1                  1.00000000 sec
D11                 0.03000000 sec
TD0                          1
SFO1               100.6178003 MHz
NUC1                       13C
P0                      3.00 usec
P1                      9.00 usec
PLW1               96.68000031 W
SFO2               400.1116004 MHz
NUC2                       1H
CPDPRG[2                waltz64
PCPD2                   90.00 usec
PLW2               17.29199982 W
PLW12               0.48032999 W
PLW13               0.24160001 W

F2 - Processing parameters
SI                       131072
SF                 100.6078454 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                      1.40
```

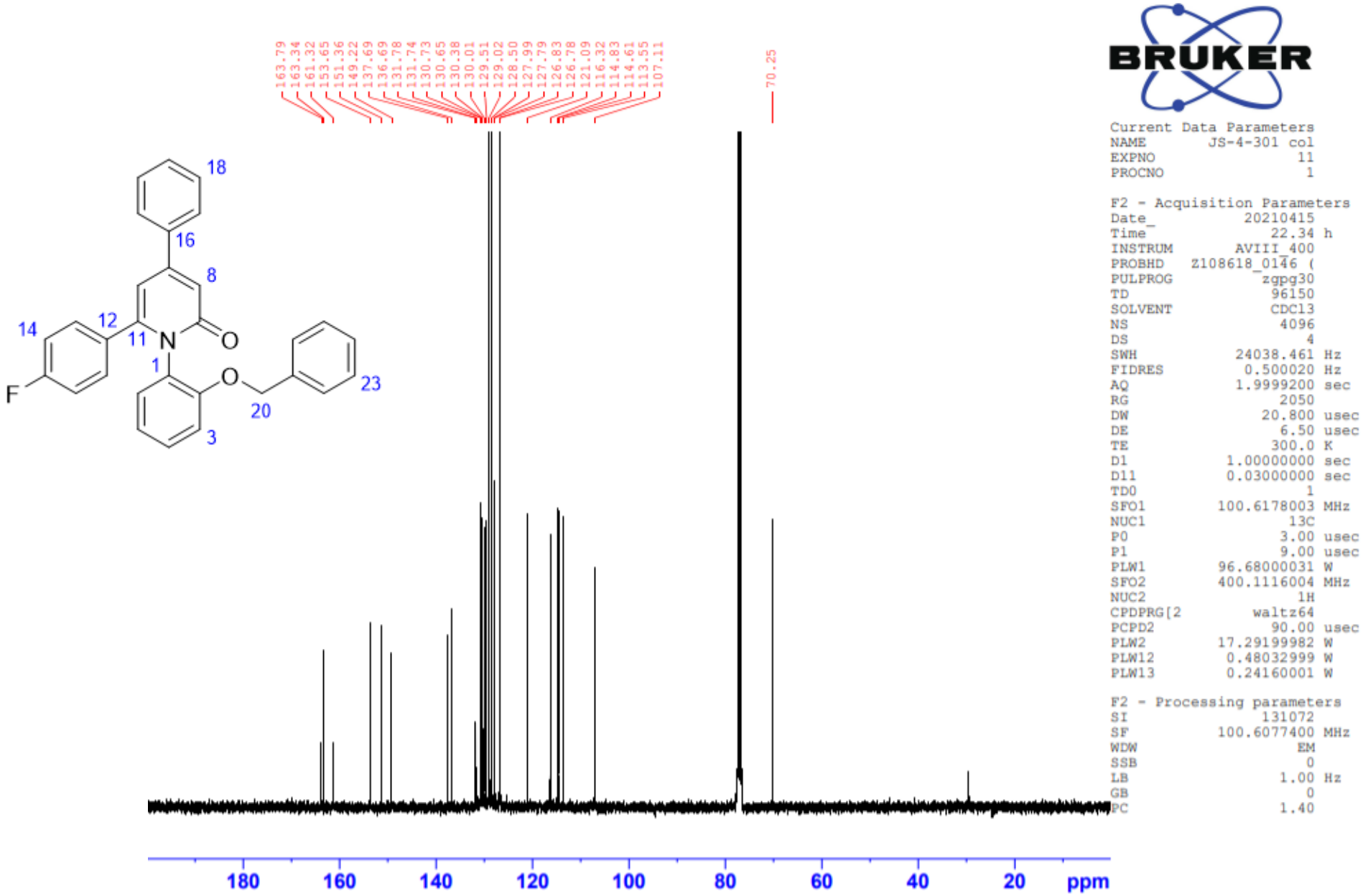
4g - ^{1}H NMR, 400 MHz, $CDCl_3$






```
Current Data Parameters
NAME       JS-4-301 col
EXPNO               10
PROCNO               1

F2 - Acquisition Parameters
Date_          20210415
Time             14.07 h
INSTRUM       AVIII_400
PROBHD   Z108618_0146 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ              3.9845889 sec
RG                   256
DW                60.800 usec
DE                17.42 usec
TE                300.0 K
D1           1.00000000 sec
TD0                    1
SFO1        400.1124708 MHz
NUC1                  1H
P0                 5.00 usec
P1                15.00 usec
PLW1        17.29199982 W

F2 - Processing parameters
SI                 32768
SF          400.1100097 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

4g - ^{13}C NMR, 101 MHz, $CDCl_3$



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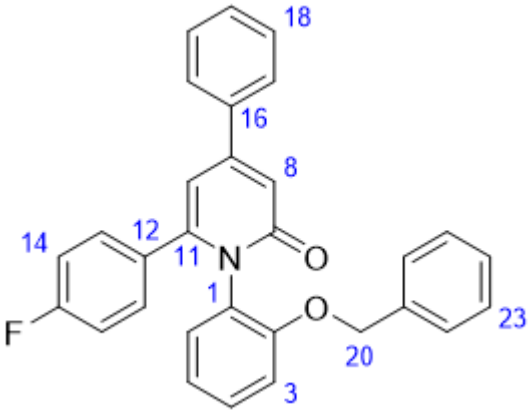
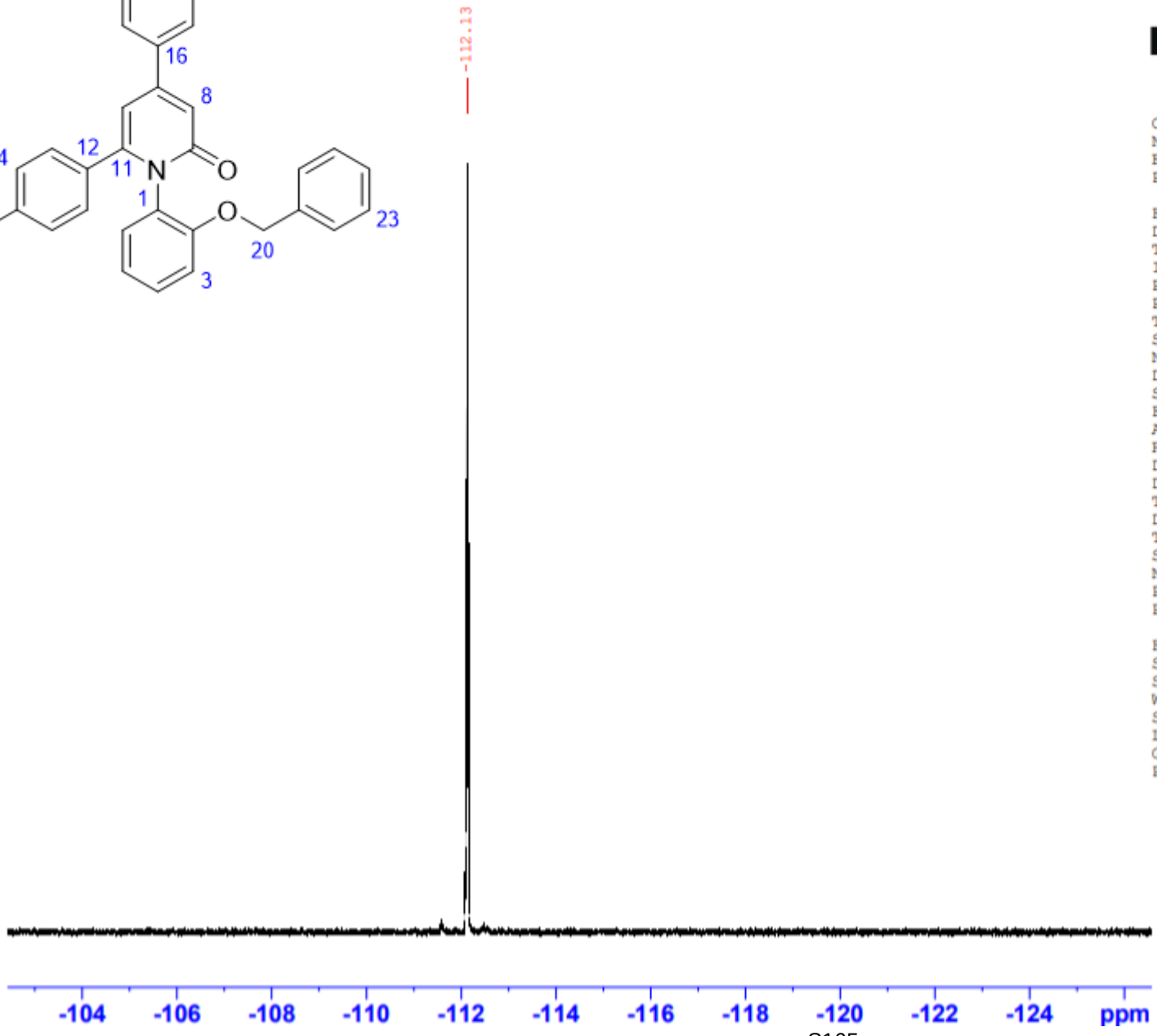
```
Current Data Parameters
NAME        JS-4-301 col
EXPNO                11
PROCNO                1

F2 - Acquisition Parameters
Date_          20210415
Time             22.34 h
INSTRUM       AVIII_400
PROBHD   Z108618_0146 (
PULPROG          zgpg30
TD                96150
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           24038.461 Hz
FIDRES         0.500020 Hz
AQ             1.9999200 sec
RG                 2050
DW               20.800 usec
DE                6.50 usec
TE               300.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         100.6178003 MHz
NUC1                13C
P0                3.00 usec
P1                9.00 usec
PLW1         96.68000031 W
SFO2         400.1116004 MHz
NUC2                1H
CPDPRG[2         waltz64
PCPD2             90.00 usec
PLW2         17.29199982 W
PLW12         0.48032999 W
PLW13         0.24160001 W

F2 - Processing parameters
SI                131072
SF           100.6077400 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

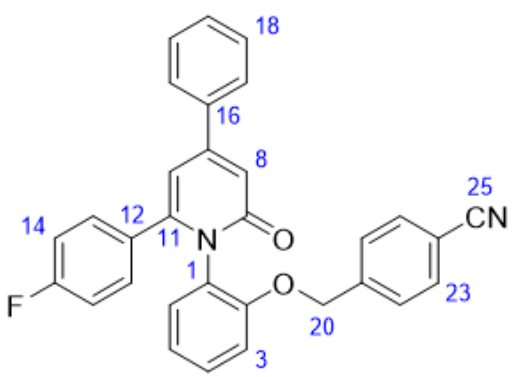
4g - ^{19}F NMR, 376 MHz, $CDCl_3$

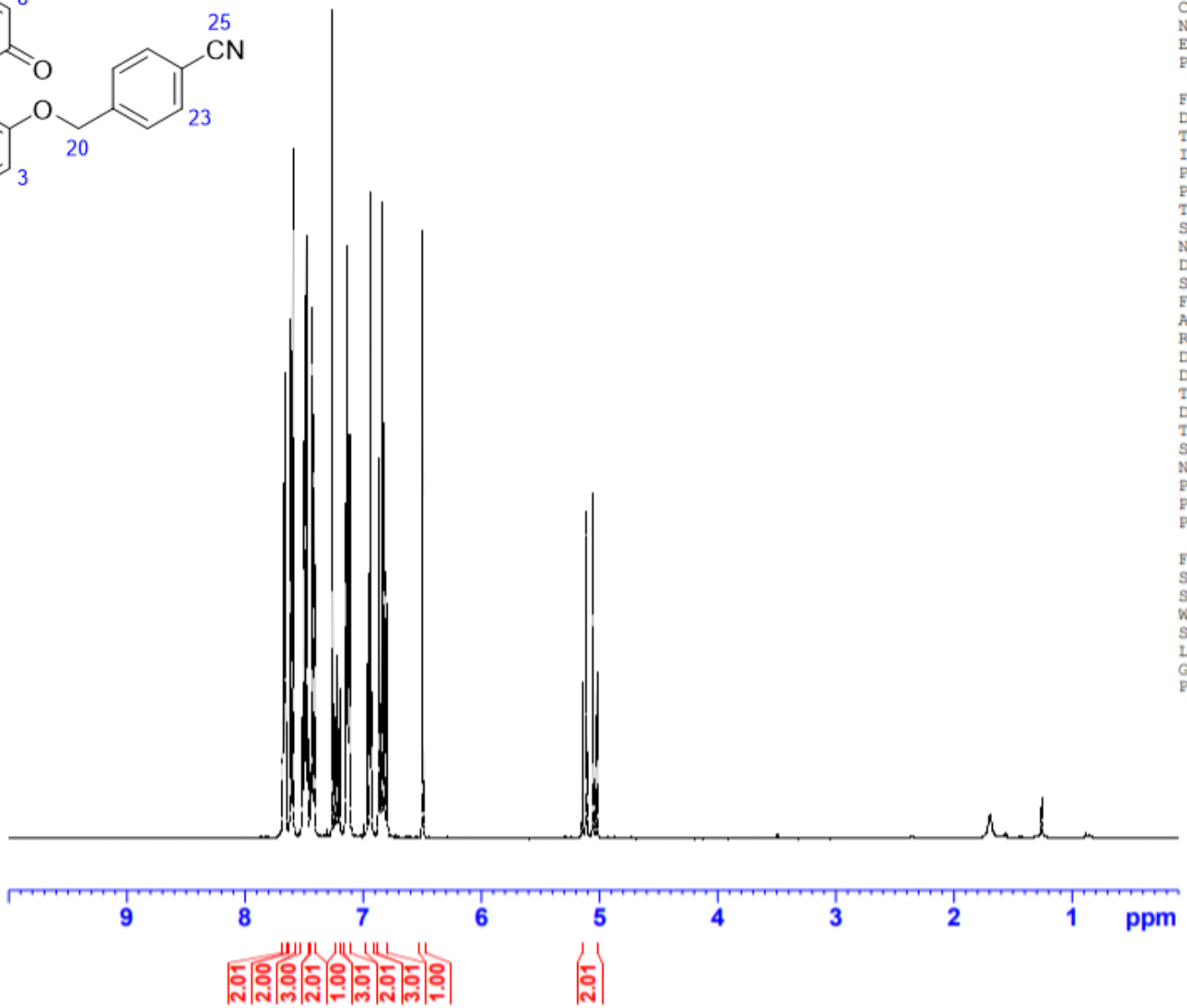





```
Current Data Parameters
NAME        JS-4-301 col
EXPNO                25
PROCNO                1

F2 - Acquisition Parameters
Date_          20210505
Time              14.13 h
INSTRUM        AVIII_400
PROBHD   Z108618_0146 (
PULPROG               zg
TD                261992
SOLVENT             CDCl3
NS                    16
DS                     4
SWH           89285.711 Hz
FIDRES         0.681591 Hz
AQ            1.4671552 sec
RG                   575
DW                 5.600 usec
DE                 7.11 usec
TE                300.0 K
D1           1.00000000 sec
TD0                    1
SFO1        376.4418995 MHz
NUC1                 19F
P1                11.80 usec
PLW1         32.96500015 W

F2 - Processing parameters
SI                262144
SF          376.4795470 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

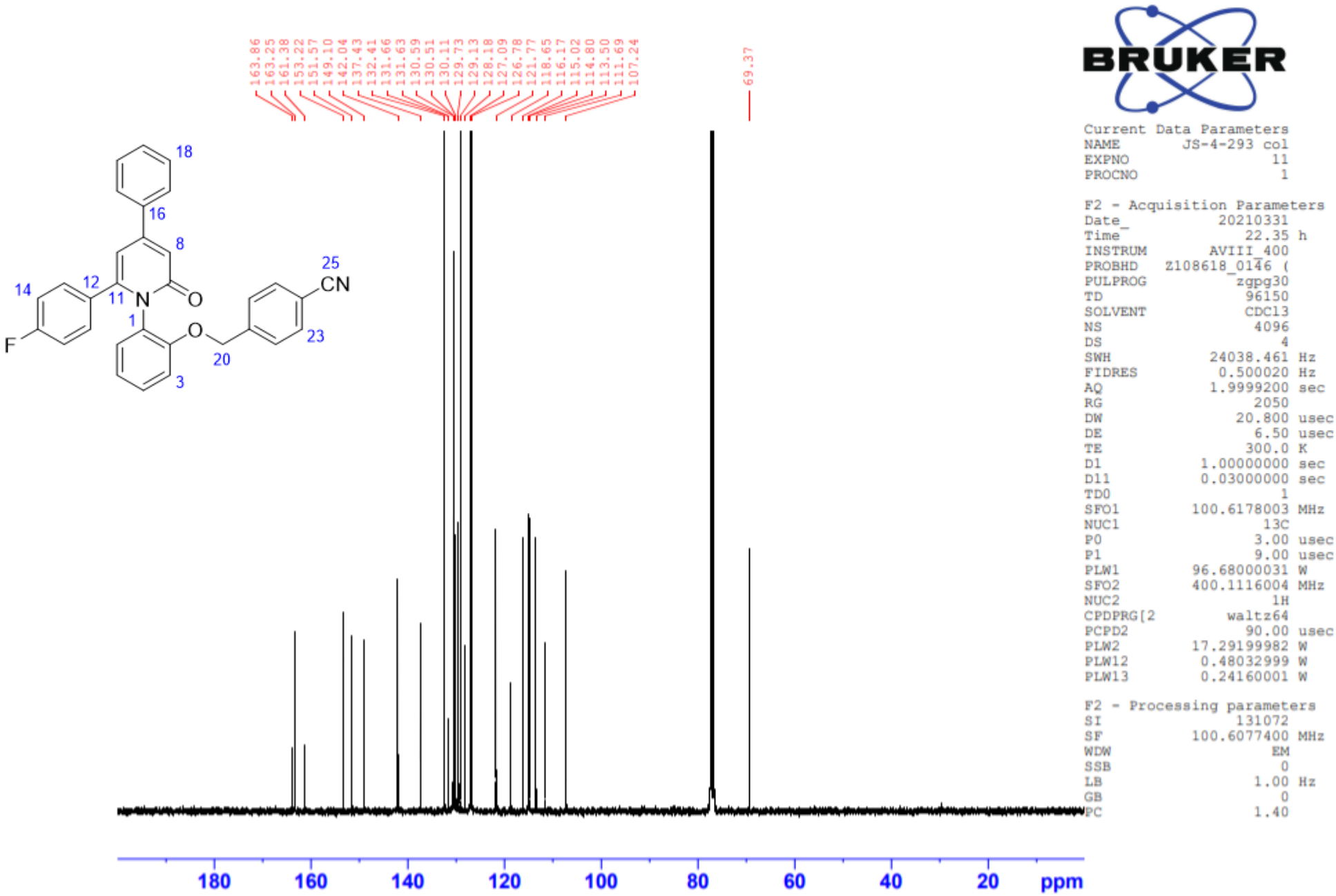
4h - ^{1}H NMR, 400 MHz, $CDCl_3$






```
Current Data Parameters
NAME        JS-4-293 col
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20210331
Time              15.06 h
INSTRUM        AVIII_400
PROBHD   Z108618_0146 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ              3.9845889 sec
RG                   161
DW                60.800 usec
DE                17.42 usec
TE                300.0 K
D1           1.00000000 sec
TD0                    1
SFO1         400.1124708 MHz
NUC1                  1H
P0                 5.00 usec
P1                15.00 usec
PLW1         17.29199982 W

F2 - Processing parameters
SI                 32768
SF          400.1100104 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

4h - ^{13}C NMR, 101 MHz, $CDCl_3$

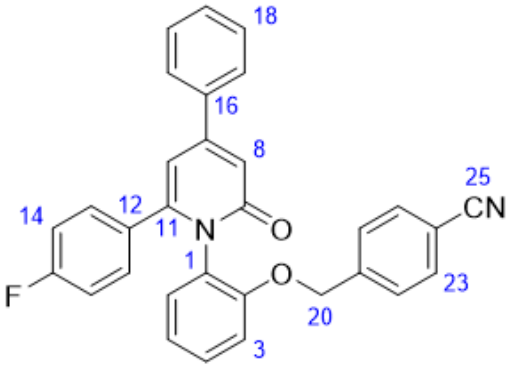


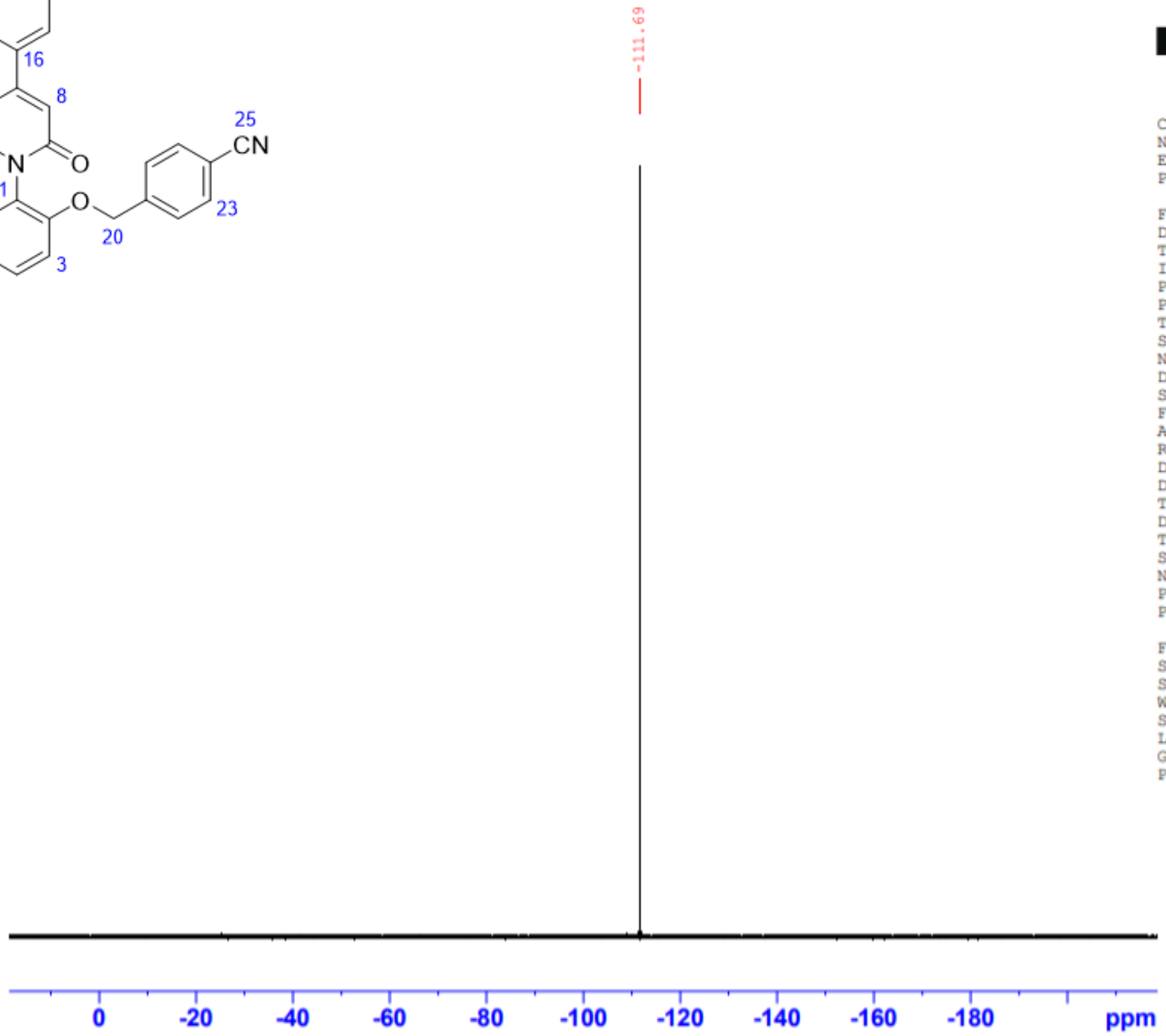
Current Data Parameters
NAME JS-4-293 col
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210331
Time 22.35 h
INSTRUM AVIII_400
PROBHD Z108618_0146 (
PULPROG zgpg30
TD 96150
SOLVENT CDCl3
NS 4096
DS 4
SWH 24038.461 Hz
FIDRES 0.500020 Hz
AQ 1.9999200 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6178003 MHz
NUC1 13C
P0 3.00 usec
P1 9.00 usec
PLW1 96.68000031 W
SFO2 400.1116004 MHz
NUC2 1H
CPDPRG[2 waltz64
PCPD2 90.00 usec
PLW2 17.29199982 W
PLW12 0.48032999 W
PLW13 0.24160001 W

F2 - Processing parameters
SI 131072
SF 100.6077400 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

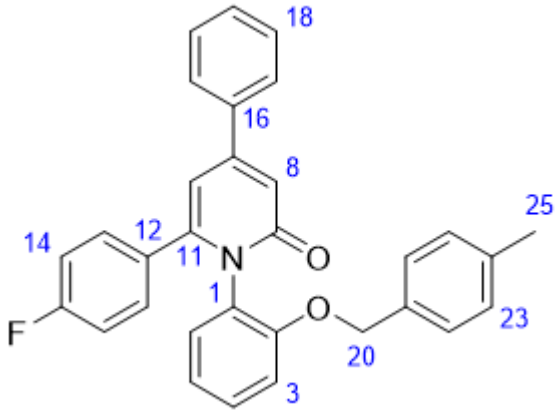
4h - ^{19}F NMR, 376 MHz, $CDCl_3$

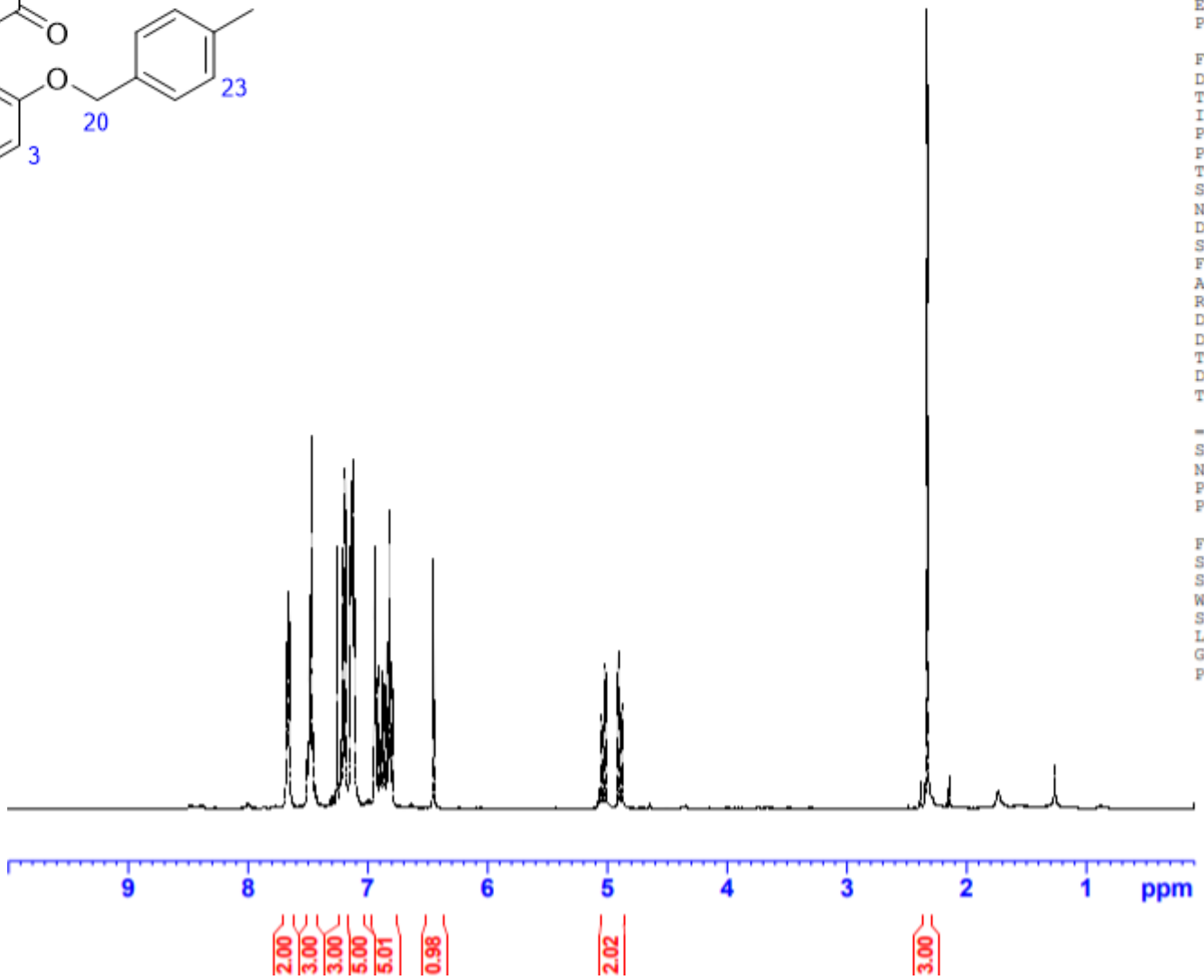





```
Current Data Parameters
NAME        JS-4-293 col
EXPNO                18
PROCNO                1

F2 - Acquisition Parameters
Date_          20210505
Time             14.07 h
INSTRUM       AVIII_400
PROBHD   Z108618_0146 (
PULPROG              zg
TD               261992
SOLVENT            CDCl3
NS                    16
DS                     4
SWH           89285.711 Hz
FIDRES         0.681591 Hz
AQ             1.4671552 sec
RG                   645
DW                 5.600 usec
DE                 7.11 usec
TE                 300.0 K
D1            1.00000000 sec
TD0                    1
SFO1        376.4418995 MHz
NUC1                 19F
P1                11.80 usec
PLW1        32.96500015 W

F2 - Processing parameters
SI                262144
SF          376.4795470 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

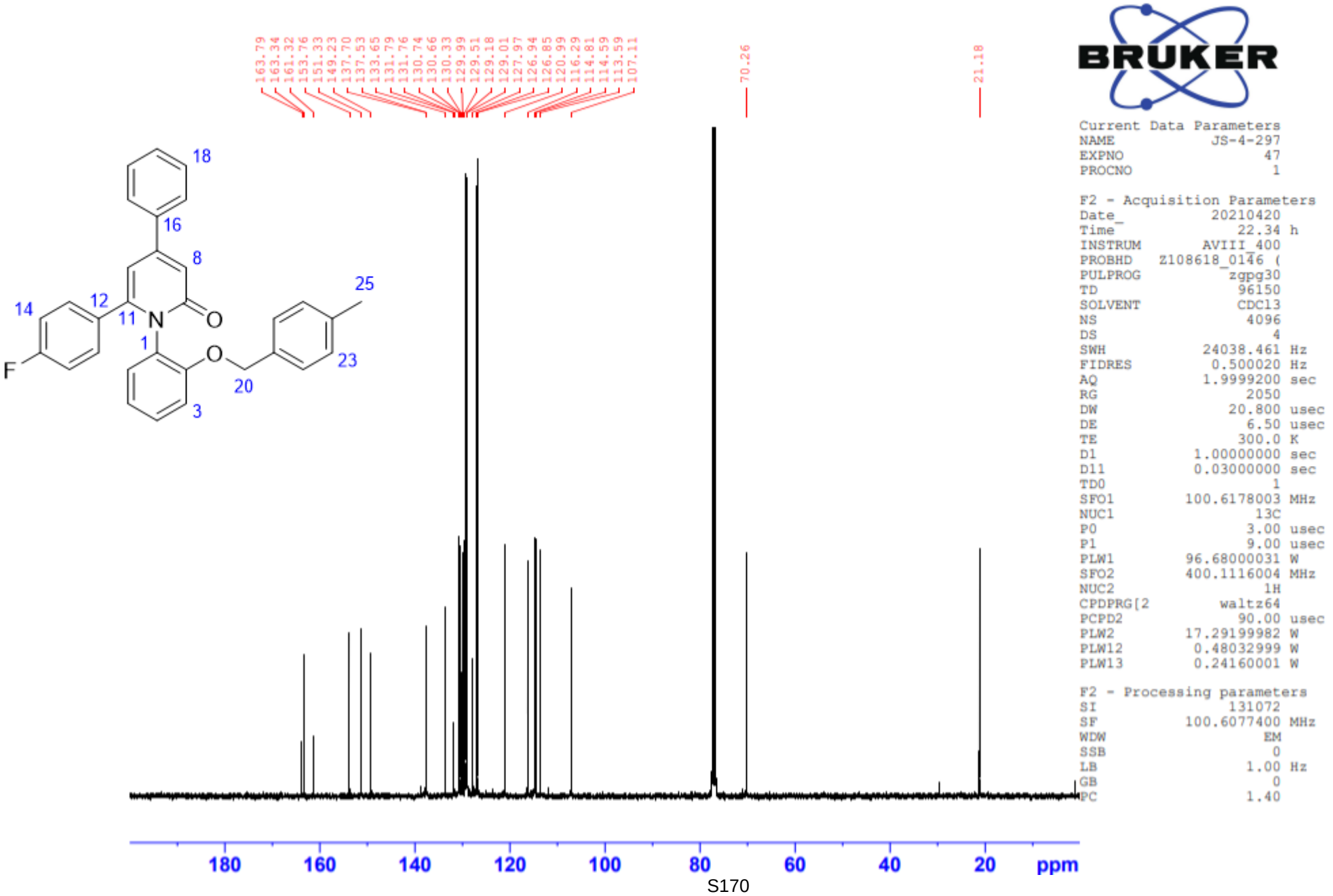
4i - ^{1}H NMR, 400 MHz, $CDCl_3$






```
Current Data Parameters
NAME                  JS-4-297
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20210411
Time                     18.10
INSTRUM               AVIII_400
PROBHD   5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                  8223.685 Hz
FIDRES               0.125483 Hz
AQ                   3.9845889 sec
RG                         57
DW                     60.800 usec
DE                      6.50 usec
TE                     294.9 K
D1                  1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               399.9124696 MHz
NUC1                       1H
P1                      15.00 usec
PLW1               17.29199982 W

F2 - Processing parameters
SI                      32768
SF                 399.9100096 MHz
WDW                        EM
SSB                         0
LB                      0.30 Hz
GB                          0
PC                      1.00
```

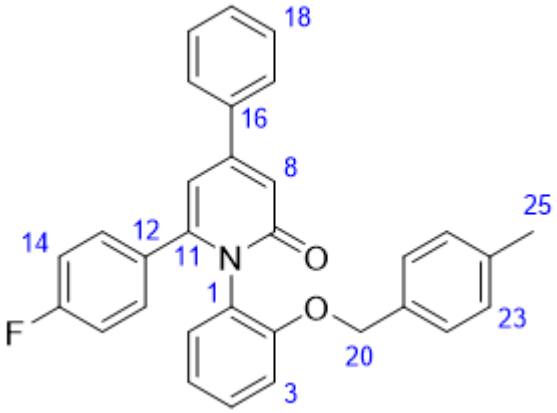
4i - ^{13}C NMR, 101 MHz, $CDCl_3$

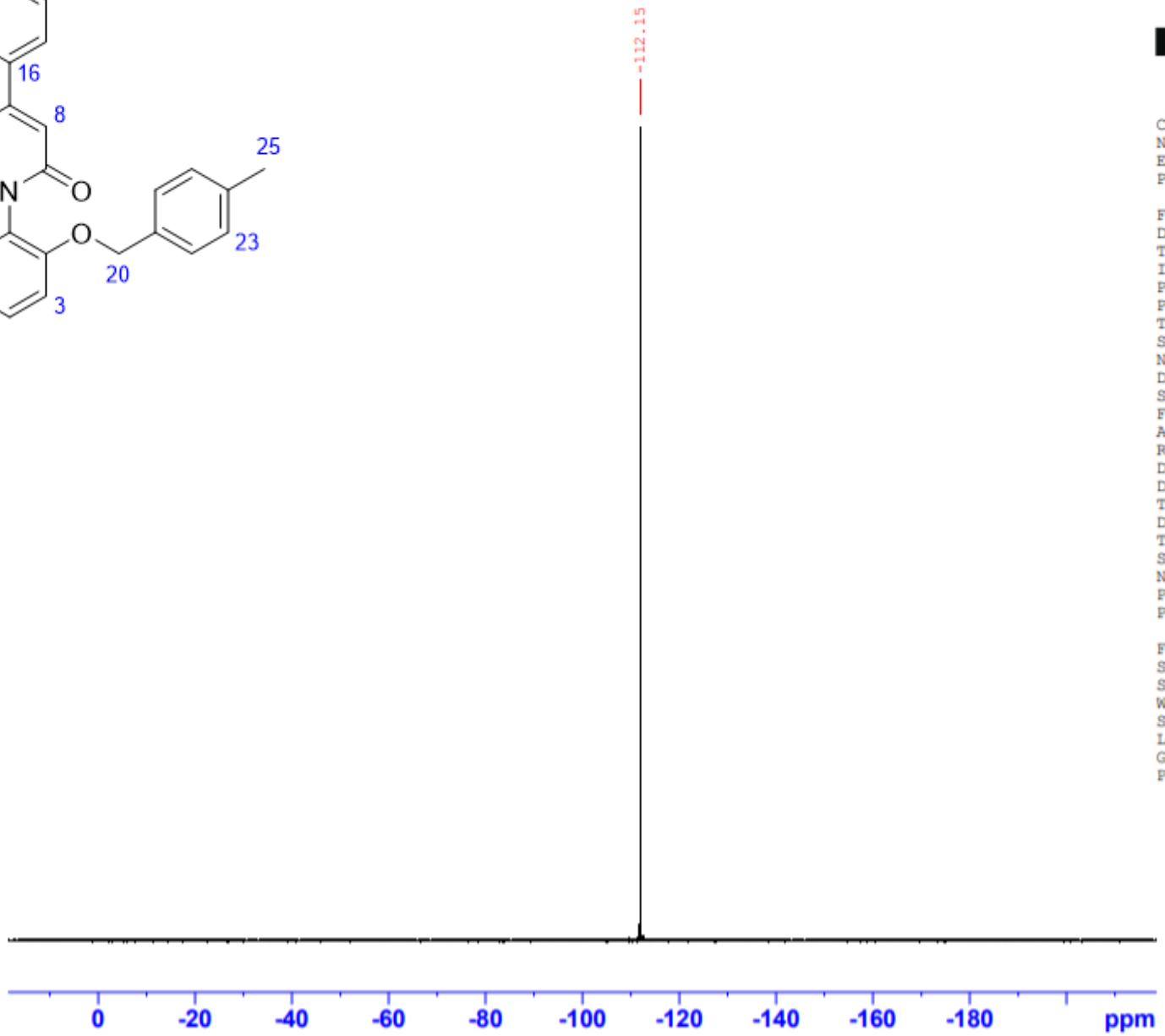


BRUKER

```
Current Data Parameters
NAME                  JS-4-297
EXPNO                       47
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20210420
Time                     22.34 h
INSTRUM               AVIII_400
PROBHD    Z108618_0146 (
PULPROG                zgpg30
TD                      96150
SOLVENT                 CDCl3
NS                       4096
DS                          4
SWH                  24038.461 Hz
FIDRES                0.500020 Hz
AQ                    1.9999200 sec
RG                       2050
DW                      20.800 usec
DE                       6.50 usec
TE                      300.0 K
D1                    1.00000000 sec
D11                   0.03000000 sec
TD0                          1
SFO1                100.6178003 MHz
NUC1                        13C
P0                       3.00 usec
P1                       9.00 usec
PLW1                96.68000031 W
SFO2                400.1116004 MHz
NUC2                        1H
CPDPRG[2                waltz64
PCPD2                   90.00 usec
PLW2                17.29199982 W
PLW12                0.48032999 W
PLW13                0.24160001 W

F2 - Processing parameters
SI                        131072
SF                  100.6077400 MHz
WDW                         EM
SSB                          0
LB                       1.00 Hz
GB                           0
PC                        1.40
```

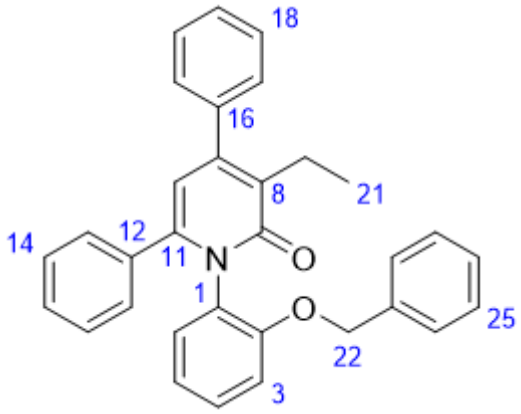
4i - ^{19}F NMR, 376 MHz, $CDCl_3$

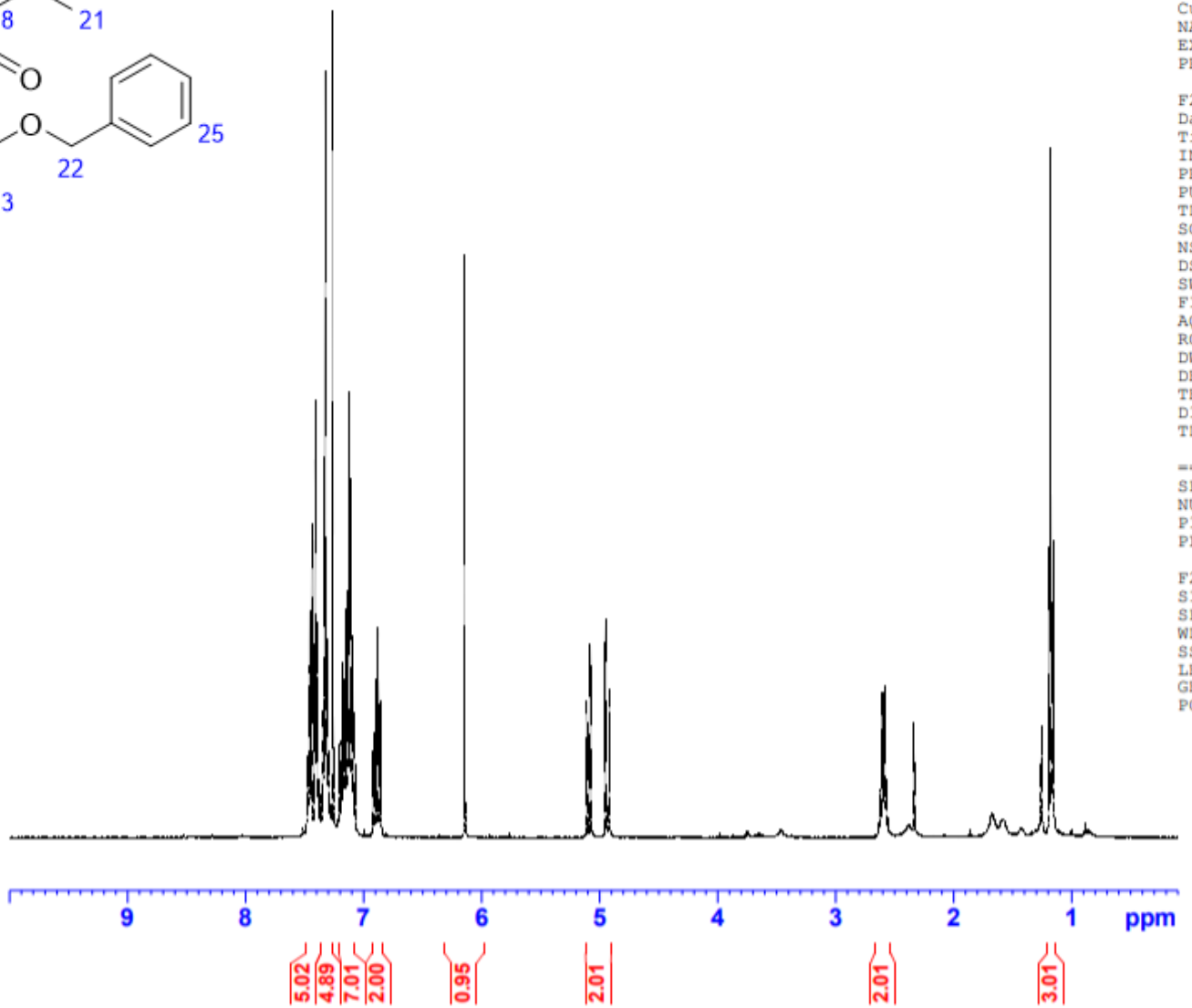





```
Current Data Parameters
NAME                  JS-4-297
EXPNO                       14
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20210420
Time                     23.06 h
INSTRUM               AVIII_400
PROBHD    Z108618_0146 (
PULPROG                     zg
TD                      261992
SOLVENT                  CDCl3
NS                          16
DS                           4
SWH                  89285.711 Hz
FIDRES                0.681591 Hz
AQ                    1.4671552 sec
RG                          575
DW                        5.600 usec
DE                        7.11 usec
TE                        300.0 K
D1                  1.00000000 sec
TD0                          1
SFO1                376.4418995 MHz
NUC1                        19F
P1                       11.80 usec
PLW1                32.96500015 W

F2 - Processing parameters
SI                      262144
SF                  376.4795470 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```

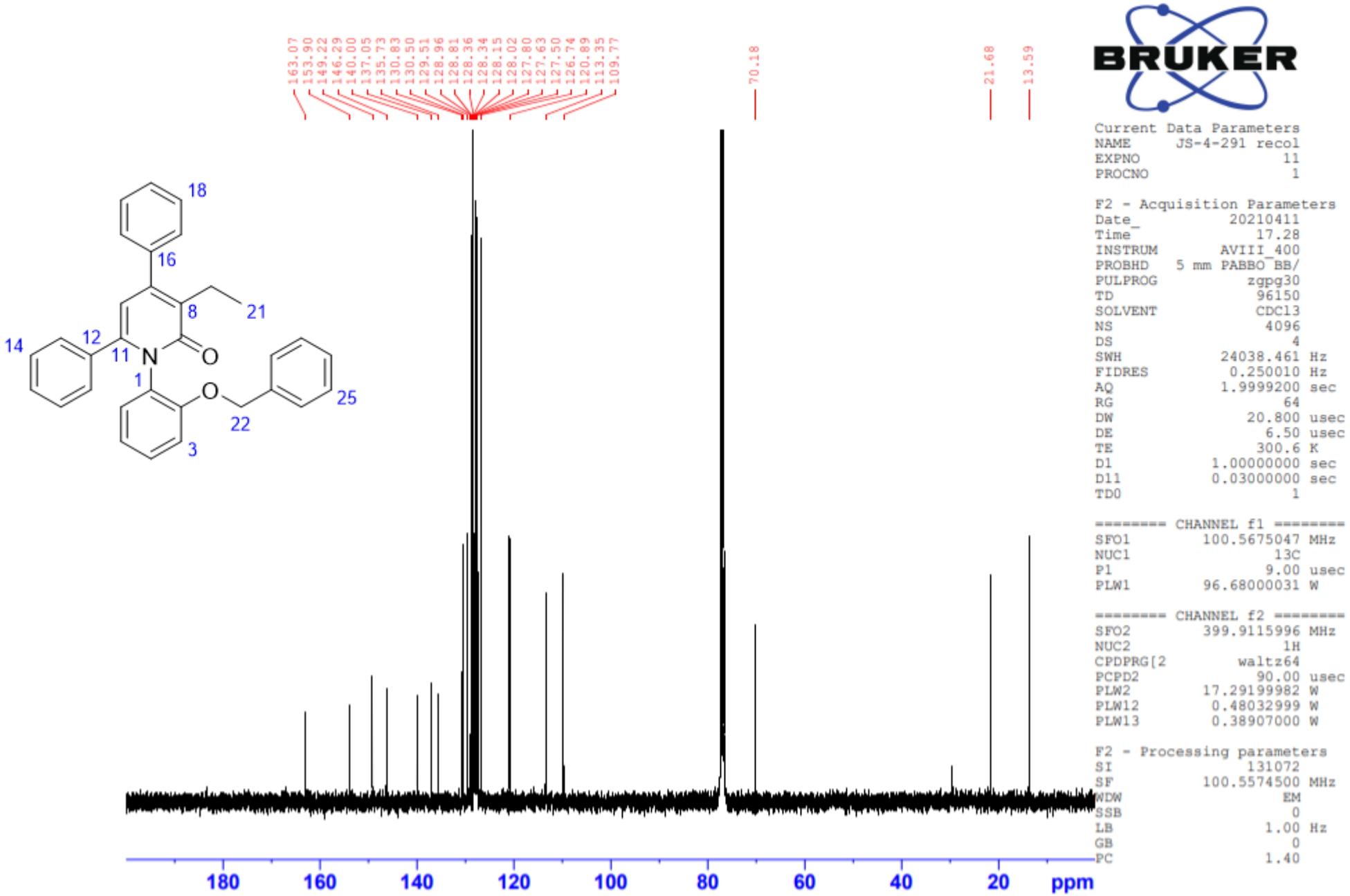
4j - ^{1}H NMR, 400 MHz, $CDCl_3$





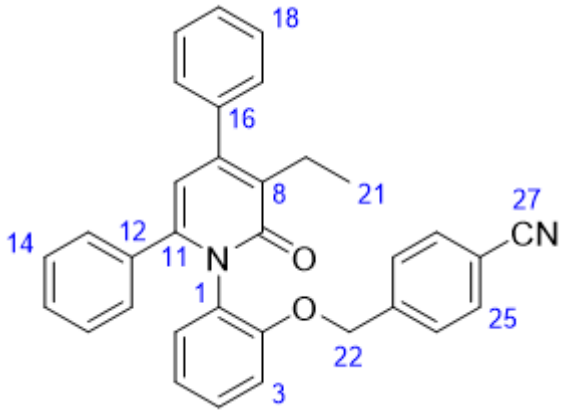

```
Current Data Parameters
NAME      JS-4-291 recol
EXPNO               10
PROCNO               1

F2 - Acquisition Parameters
Date_          20210409
Time_             13.12
INSTRUM       AVIII_400
PROBHD   5 mm PABBO_BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ             3.9845889 sec
RG                 114
DW                60.800 usec
DE                 6.50 usec
TE                294.8 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         399.9124696 MHz
NUC1                 1H
P1                15.00 usec
PLW1        17.29199982 W

F2 - Processing parameters
SI                32768
SF           399.9100097 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                1.00
```

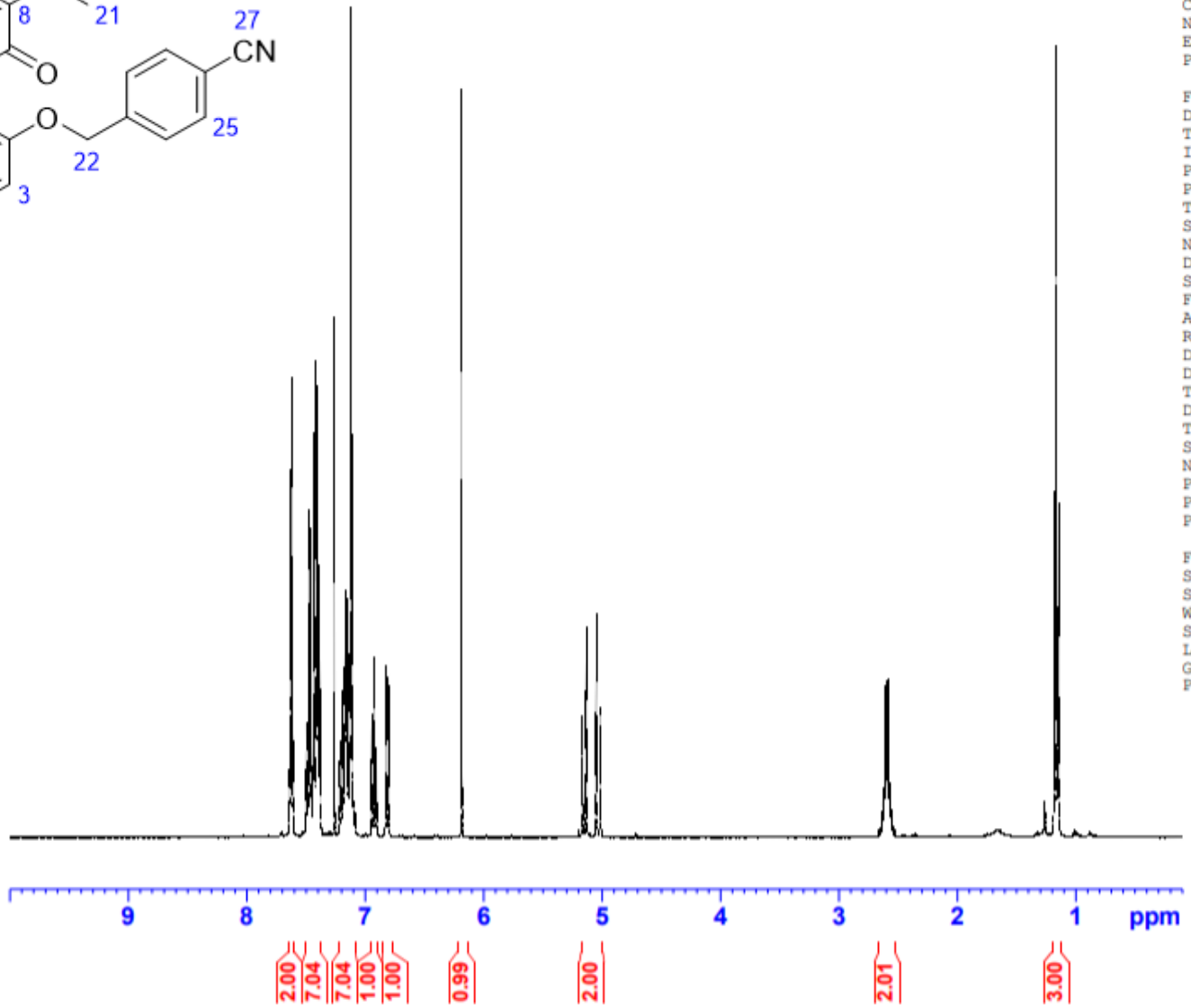
4j - ^{13}C NMR, 101 MHz, $CDCl_3$




```
Current Data Parameters
NAME      JS-4-291 recol
EXPNO                11
PROCNO                1

F2 - Acquisition Parameters
Date_          20210411
Time               17.28
INSTRUM        AVIII_400
PROBHD   5 mm PABBO_BB/
PULPROG          zgpg30
TD                96150
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           24038.461 Hz
FIDRES         0.250010 Hz
AQ             1.9999200 sec
RG                   64
DW                20.800 usec
DE                 6.50 usec
TE                300.6 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.5675047 MHz
NUC1                 13C
P1                  9.00 usec
PLW1         96.68000031 W

======== CHANNEL f2 ========
SFO2         399.9115996 MHz
NUC2                 1H
CPDPRG[2          waltz64
PCPD2               90.00 usec
PLW2         17.29199982 W
PLW12         0.48032999 W
PLW13         0.38907000 W

F2 - Processing parameters
SI                 131072
SF           100.5574500 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

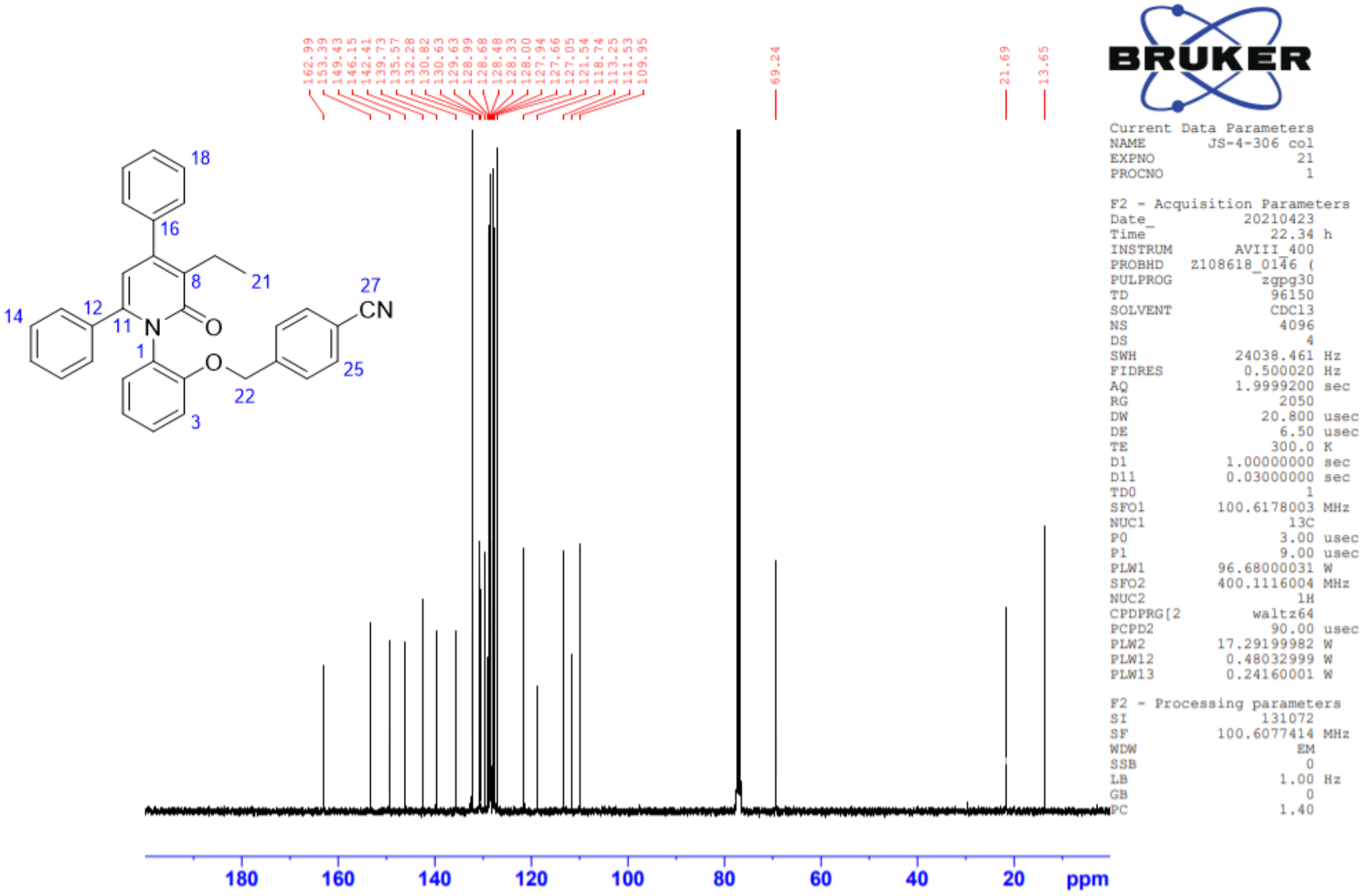
4k - ^{1}H NMR, 400 MHz, $CDCl_3$





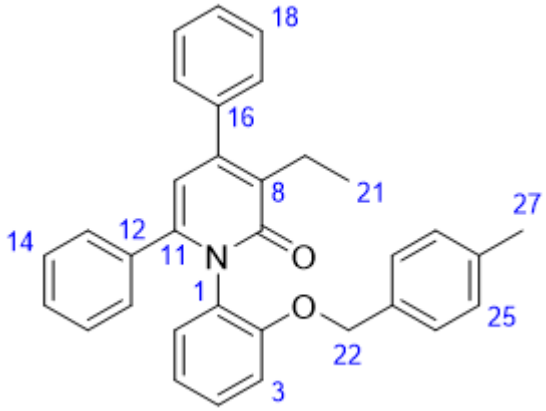

```
Current Data Parameters
NAME        JS-4-306 col
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20210422
Time              17.26 h
INSTRUM       AVIII_400
PROBHD   Z108618_0146 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ              3.9845889 sec
RG                   161
DW                60.800 usec
DE                17.42 usec
TE                300.0 K
D1           1.00000000 sec
TD0                    1
SFO1        400.1124708 MHz
NUC1                  1H
P0                 5.00 usec
P1                15.00 usec
PLW1        17.29199982 W

F2 - Processing parameters
SI                 32768
SF          400.1100100 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.00
```

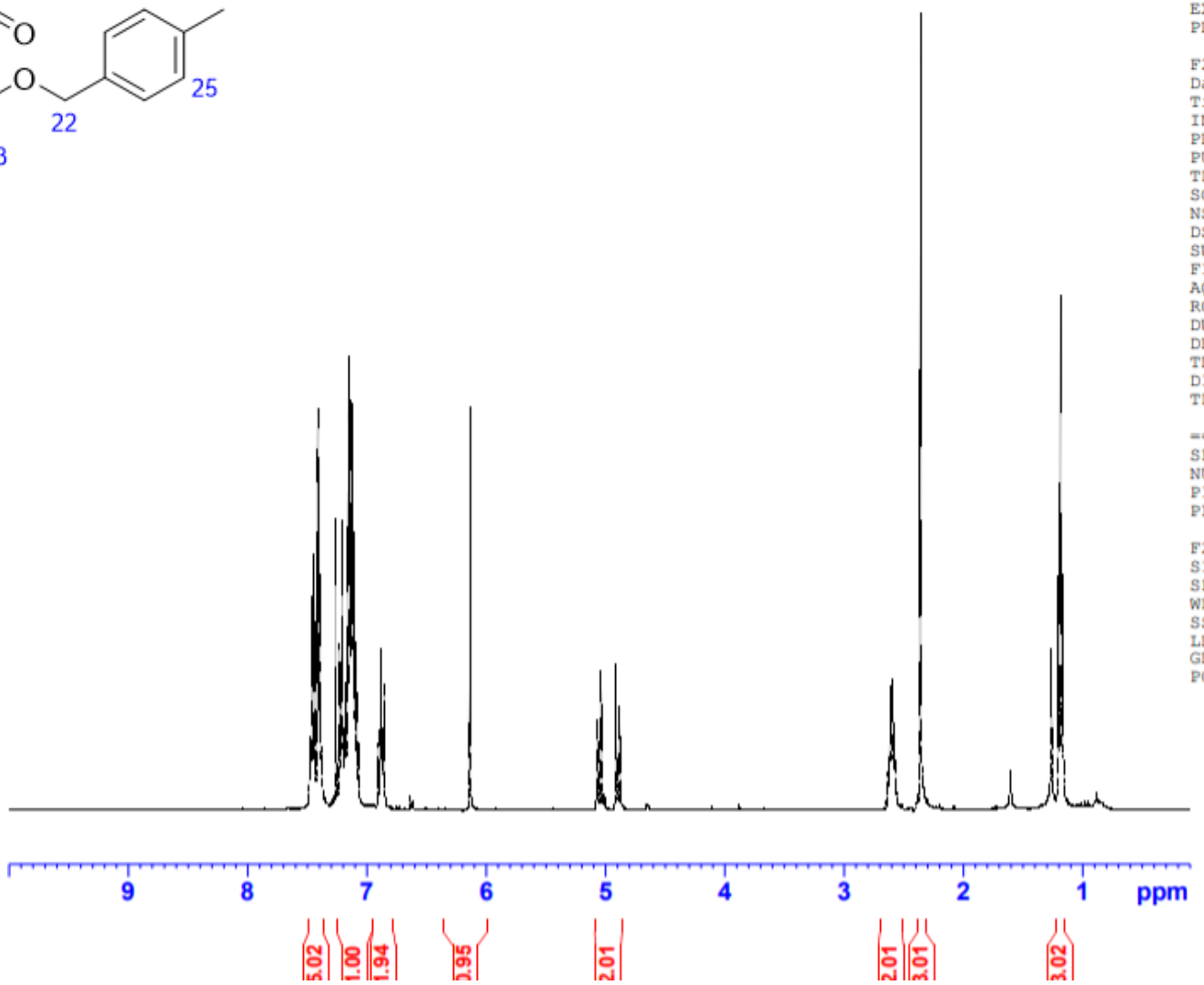
4k - ^{13}C NMR, 101 MHz, $CDCl_3$




```
Current Data Parameters
NAME        JS-4-306 col
EXPNO                21
PROCNO                1

F2 - Acquisition Parameters
Date_          20210423
Time              22.34 h
INSTRUM       AVIII_400
PROBHD   Z108618_0146 (
PULPROG          zgpg30
TD                96150
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           24038.461 Hz
FIDRES         0.500020 Hz
AQ             1.9999200 sec
RG                 2050
DW               20.800 usec
DE                6.50 usec
TE               300.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         100.6178003 MHz
NUC1                13C
P0                3.00 usec
P1                9.00 usec
PLW1         96.68000031 W
SFO2         400.1116004 MHz
NUC2                1H
CPDPRG[2         waltz64
PCPD2            90.00 usec
PLW2         17.29199982 W
PLW12         0.48032999 W
PLW13         0.24160001 W

F2 - Processing parameters
SI                131072
SF           100.6077414 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

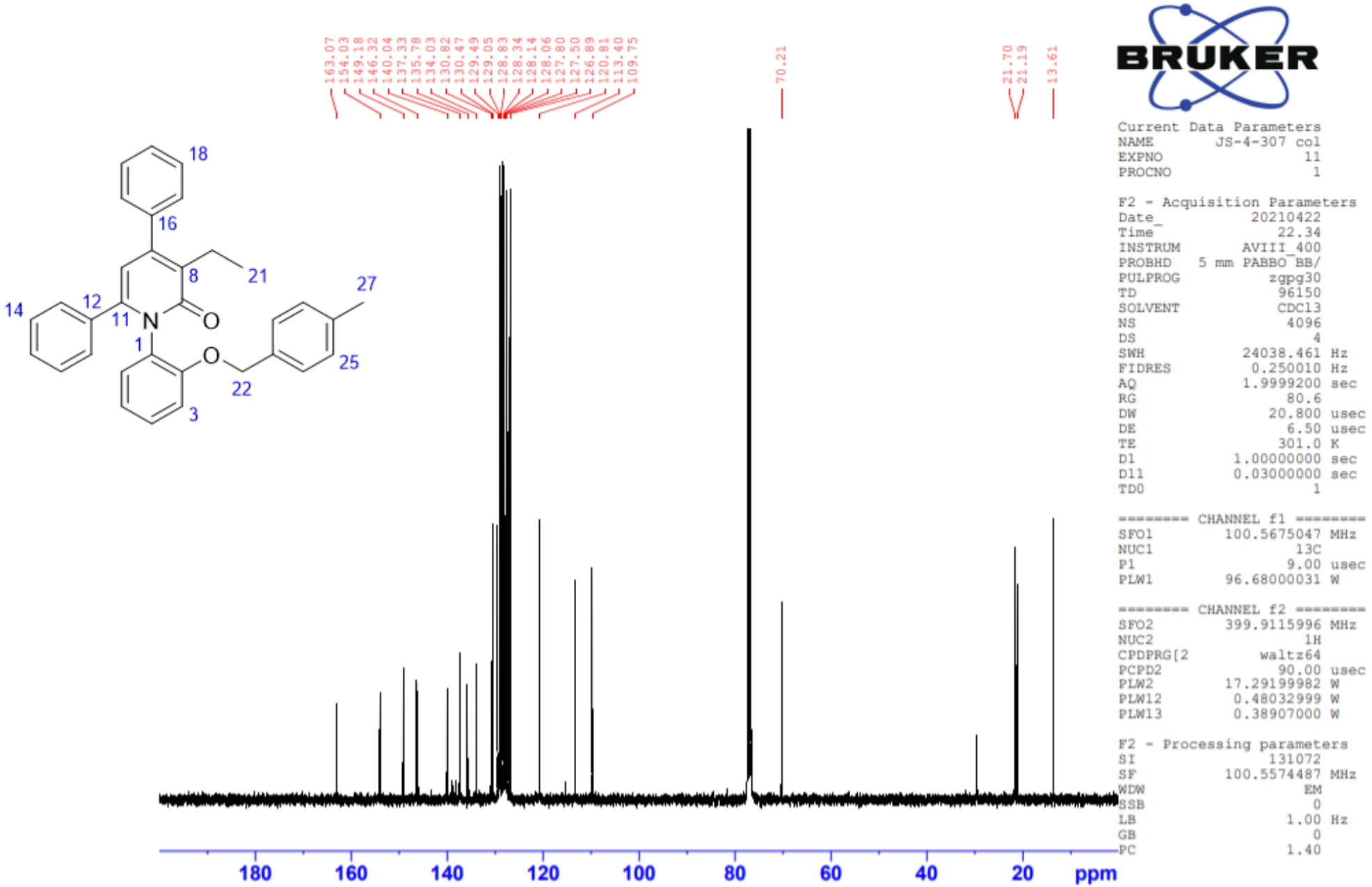
4l - ^{1}H NMR, 400 MHz, $CDCl_3$



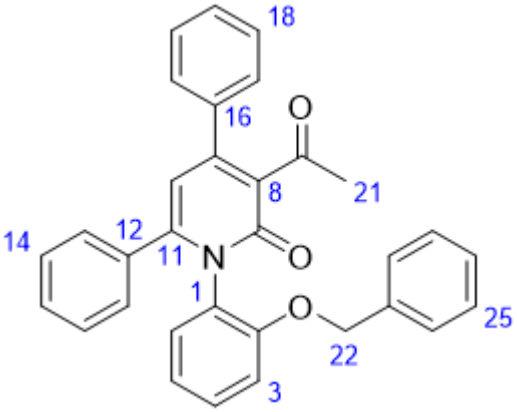


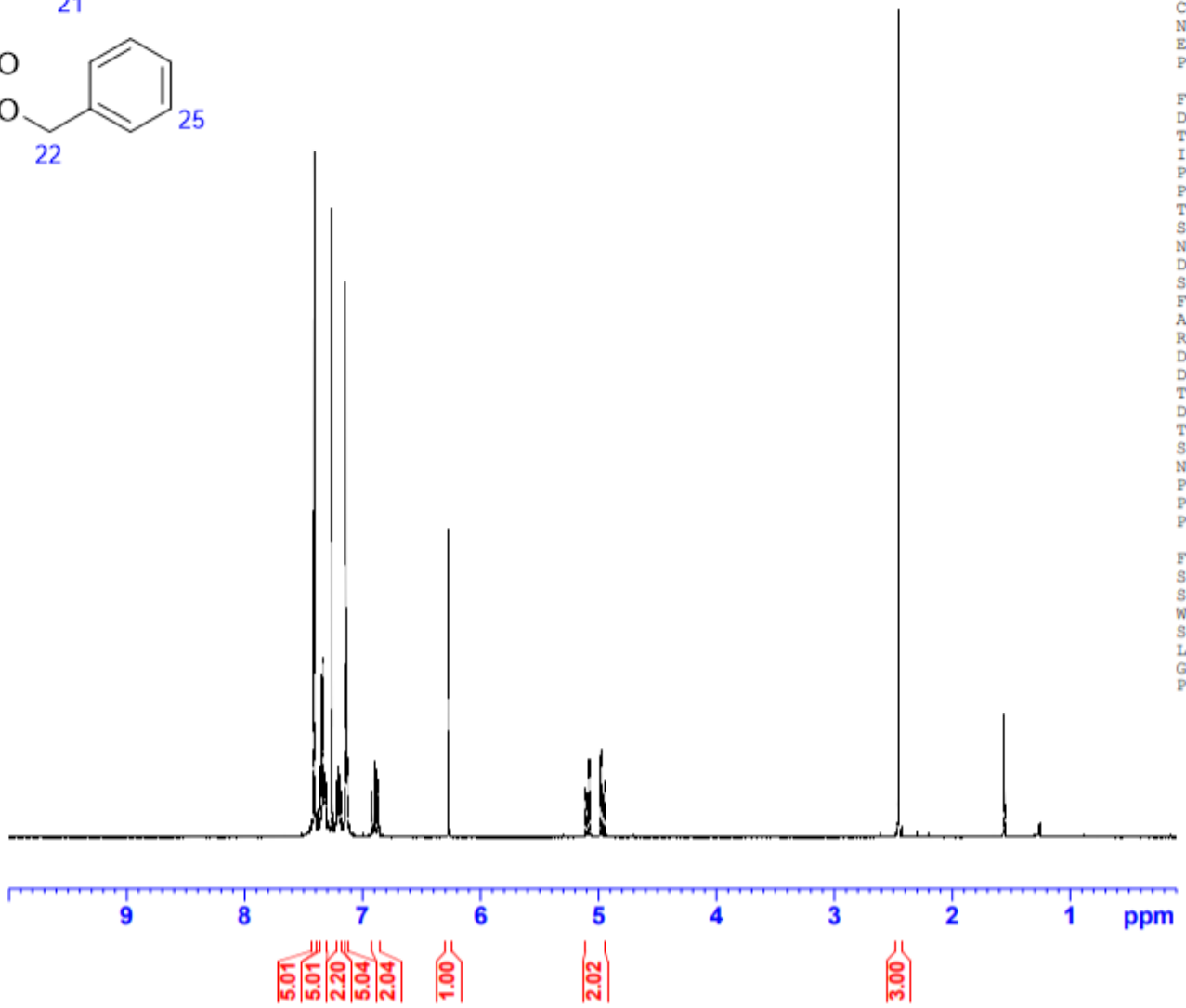

```
Current Data Parameters
NAME        JS-4-307 col
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20210422
Time_             17.25
INSTRUM       AVIII_400
PROBHD   5 mm PABBO_BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ             3.9845889 sec
RG                80.6
DW              60.800 usec
DE               6.50 usec
TE              296.0 K
D1          1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        399.9124696 MHz
NUC1                1H
P1               15.00 usec
PLW1        17.29199982 W

F2 - Processing parameters
SI                32768
SF          399.9100098 MHz
WDW                  EM
SSB                   0
LB               0.30 Hz
GB                    0
PC                 1.00
```

4l - ^{13}C NMR, 101 MHz, $CDCl_3$



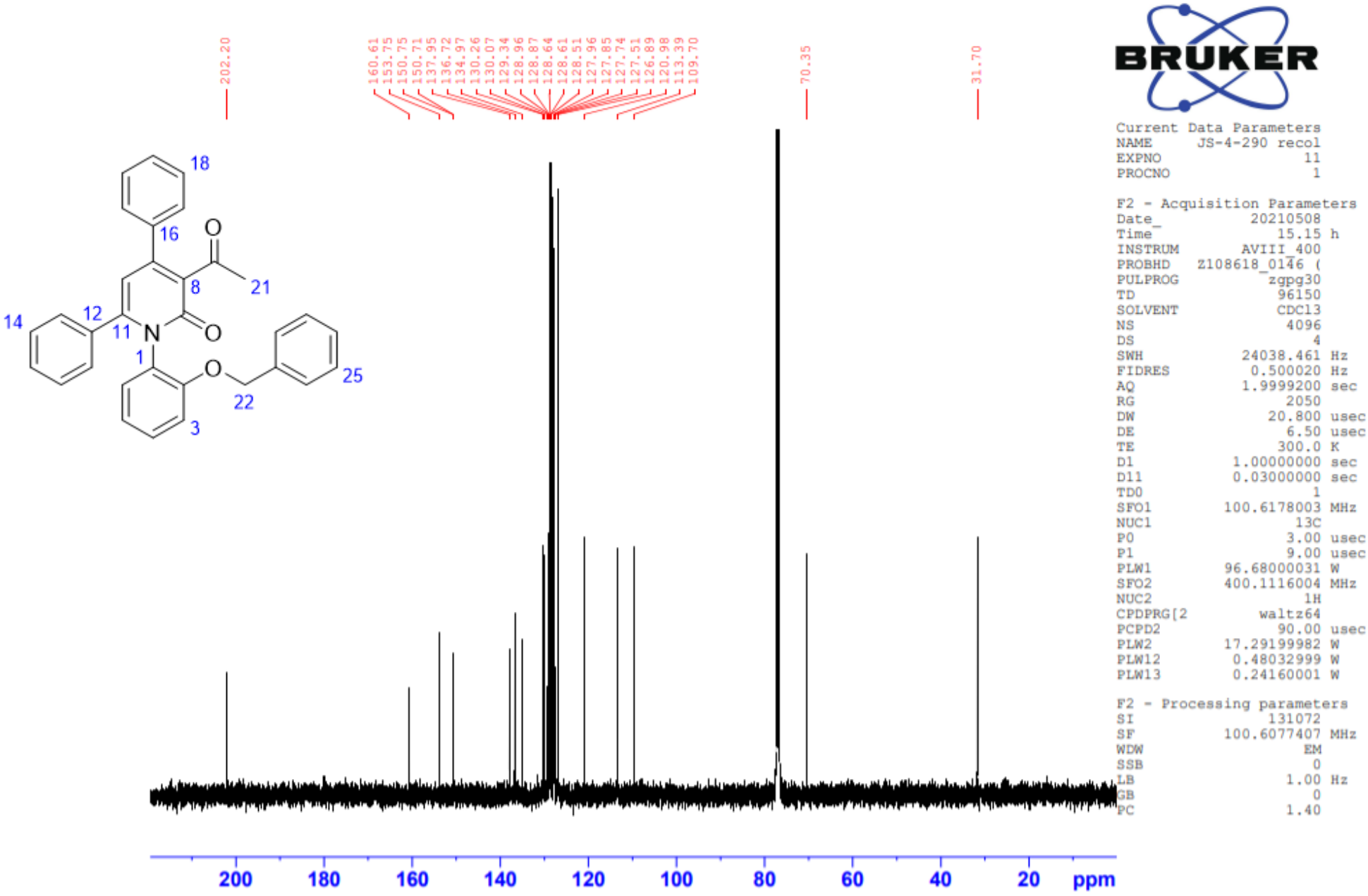
4m - ^{1}H NMR, 400 MHz, $CDCl_3$





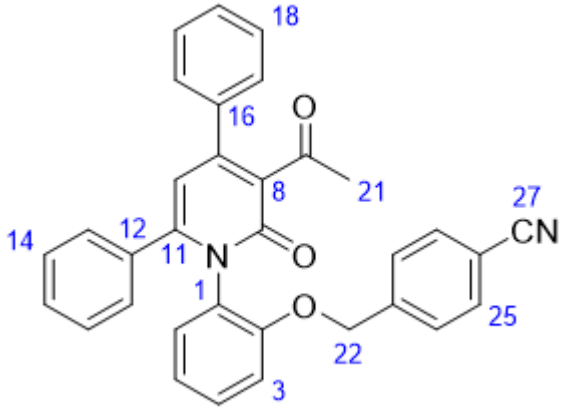

```
Current Data Parameters
NAME      JS-4-290 recol
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20210507
Time              17.23 h
INSTRUM        AVIII_400
PROBHD   Z108618_0146 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ              3.9845889 sec
RG                   256
DW                60.800 usec
DE                17.42 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1
SFO1         400.1124708 MHz
NUC1                  1H
P0                 5.00 usec
P1                15.00 usec
PLW1         17.29199982 W

F2 - Processing parameters
SI                 32768
SF           400.1100095 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

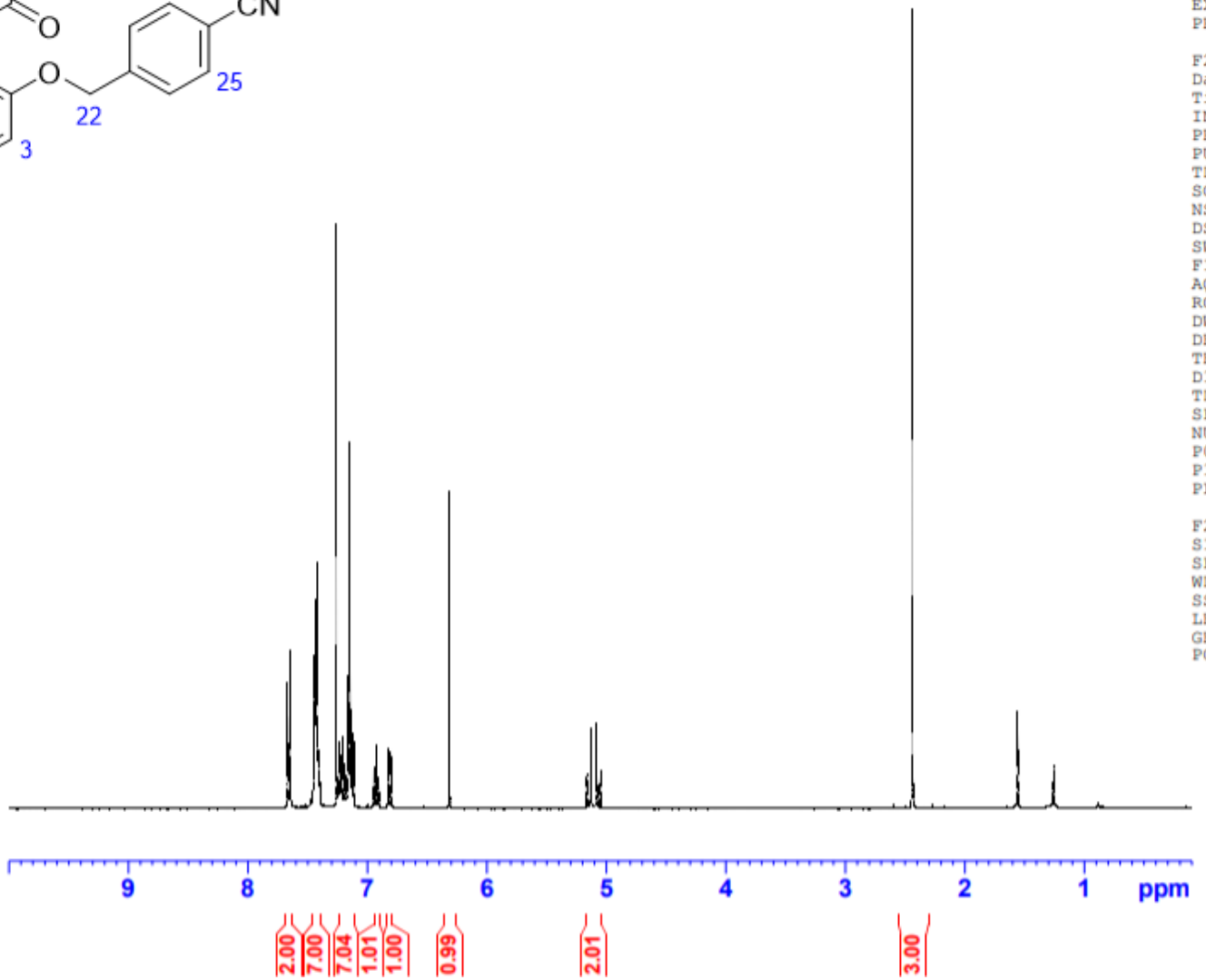
4m - ^{13}C NMR, 101 MHz, $CDCl_3$




```
Current Data Parameters
NAME      JS-4-290 recol
EXPNO                11
PROCNO                1

F2 - Acquisition Parameters
Date_          20210508
Time              15.15 h
INSTRUM       AVIII_400
PROBHD   Z108618_0146 (
PULPROG          zgpg30
TD                96150
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           24038.461 Hz
FIDRES         0.500020 Hz
AQ             1.9999200 sec
RG                 2050
DW               20.800 usec
DE                6.50 usec
TE               300.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1        100.6178003 MHz
NUC1               13C
P0                3.00 usec
P1                9.00 usec
PLW1         96.68000031 W
SFO2        400.1116004 MHz
NUC2               1H
CPDPRG[2        waltz64
PCPD2            90.00 usec
PLW2         17.29199982 W
PLW12         0.48032999 W
PLW13         0.24160001 W

F2 - Processing parameters
SI                131072
SF          100.6077407 MHz
WDW                 EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

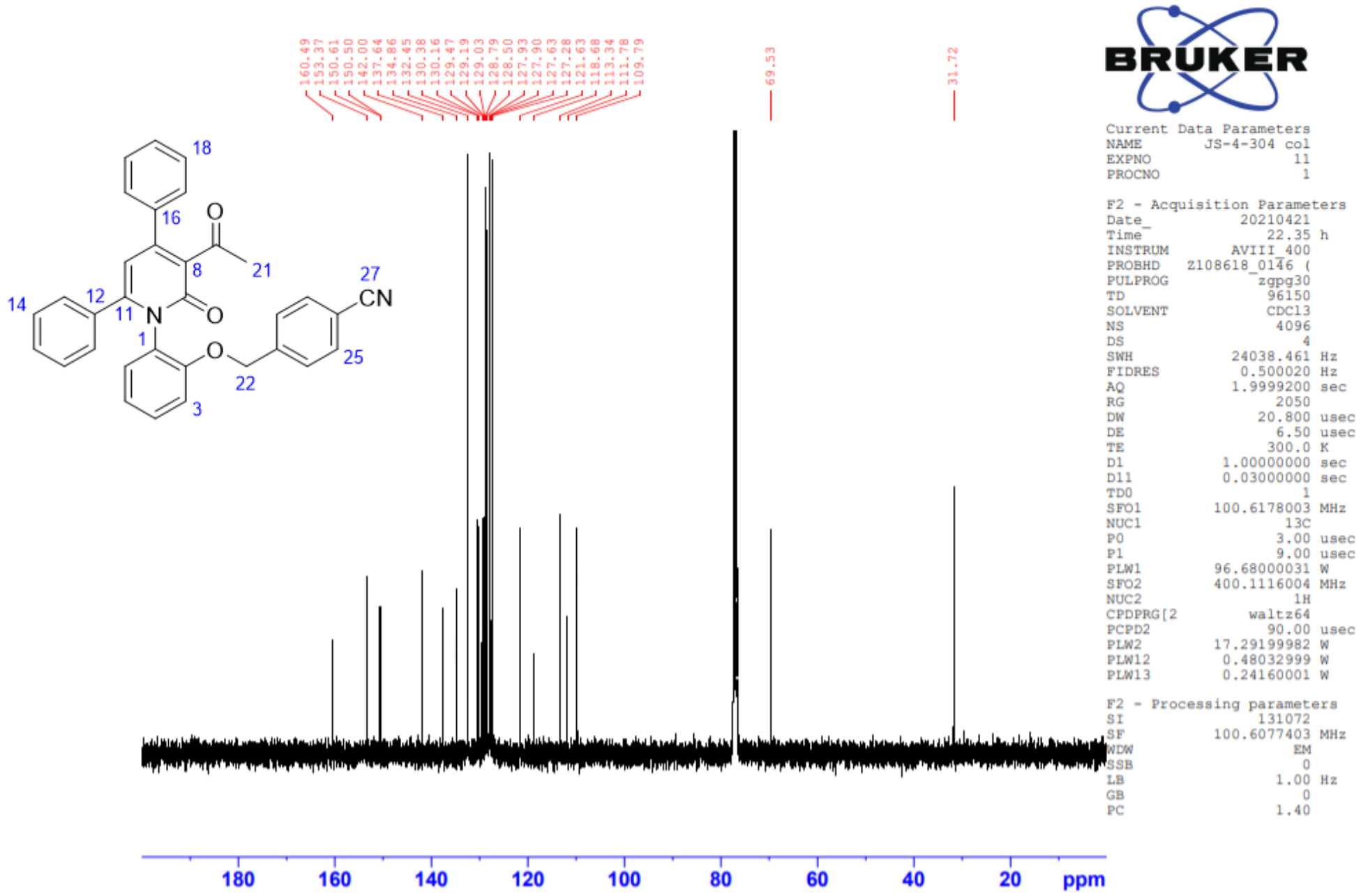
4n - ^{1}H NMR, 400 MHz, $CDCl_3$





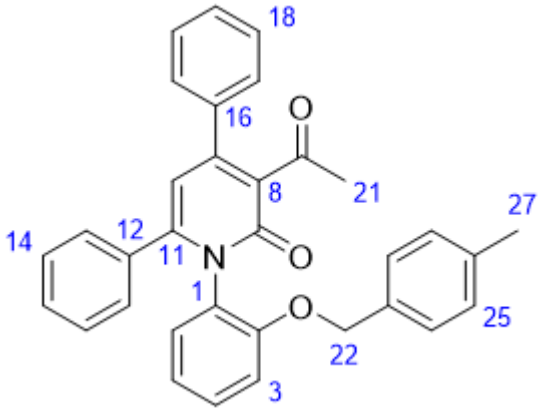

```
Current Data Parameters
NAME        JS-4-304 col
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20210421
Time             16.56 h
INSTRUM        AVIII_400
PROBHD   Z108618_0146 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           8223.685 Hz
FIDRES        0.250967 Hz
AQ            3.9845889 sec
RG                   256
DW                60.800 usec
DE                17.42 usec
TE                300.0 K
D1           1.00000000 sec
TD0                    1
SFO1        400.1124708 MHz
NUC1                  1H
P0                 5.00 usec
P1                15.00 usec
PLW1        17.29199982 W

F2 - Processing parameters
SI                 32768
SF          400.1100100 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

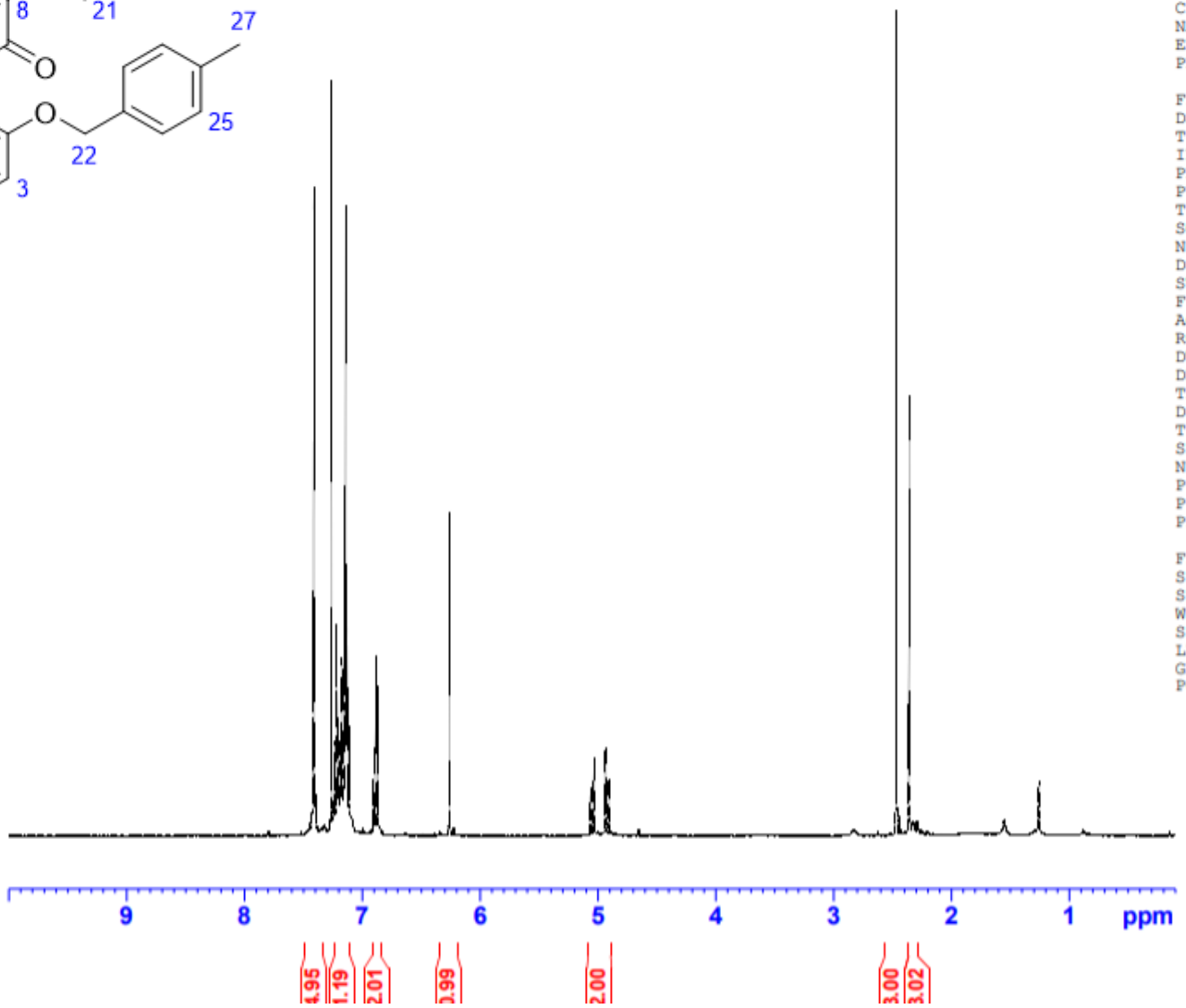
4n - ^{13}C NMR, 101 MHz, $CDCl_3$




```
Current Data Parameters
NAME        JS-4-304 col
EXPNO                11
PROCNO                1

F2 - Acquisition Parameters
Date_          20210421
Time             22.35 h
INSTRUM       AVIII_400
PROBHD   Z108618_0146 (
PULPROG          zgpg30
TD                96150
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           24038.461 Hz
FIDRES         0.500020 Hz
AQ             1.9999200 sec
RG                 2050
DW               20.800 usec
DE                6.50 usec
TE               300.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1        100.6178003 MHz
NUC1               13C
P0                3.00 usec
P1                9.00 usec
PLW1         96.68000031 W
SFO2        400.1116004 MHz
NUC2               1H
CPDPRG[2       waltz64
PCPD2            90.00 usec
PLW2         17.29199982 W
PLW12         0.48032999 W
PLW13         0.24160001 W

F2 - Processing parameters
SI                131072
SF          100.6077403 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

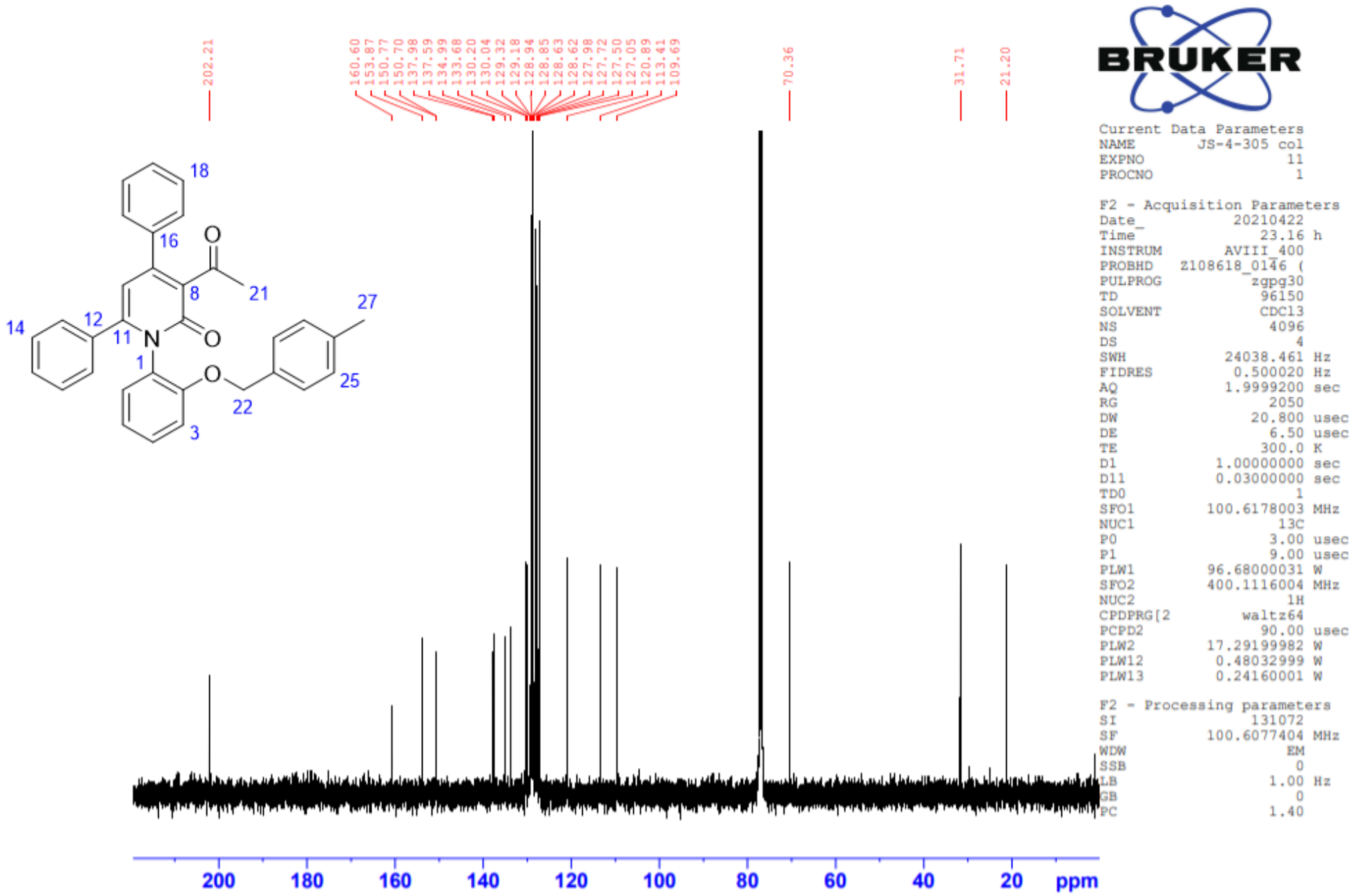
4o - ^{1}H NMR, 400 MHz, $CDCl_3$






```
Current Data Parameters
NAME        JS-4-305 col
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20210422
Time              11.38 h
INSTRUM       AVIII_400
PROBHD   Z108618_0146 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ              3.9845889 sec
RG                   256
DW                60.800 usec
DE                17.42 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1
SFO1         400.1124708 MHz
NUC1                  1H
P0                 5.00 usec
P1                15.00 usec
PLW1         17.29199982 W

F2 - Processing parameters
SI                 32768
SF           400.1100100 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```

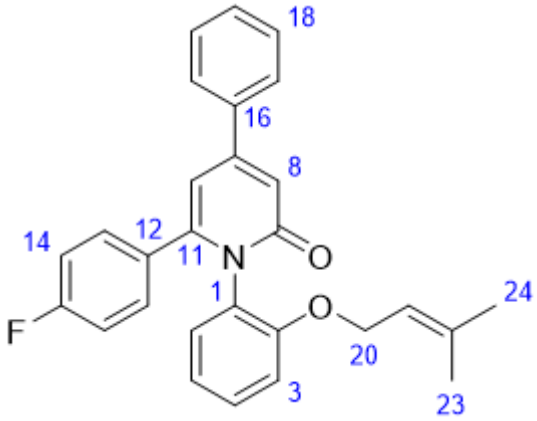
4o - ^{13}C NMR, 101 MHz, $CDCl_3$

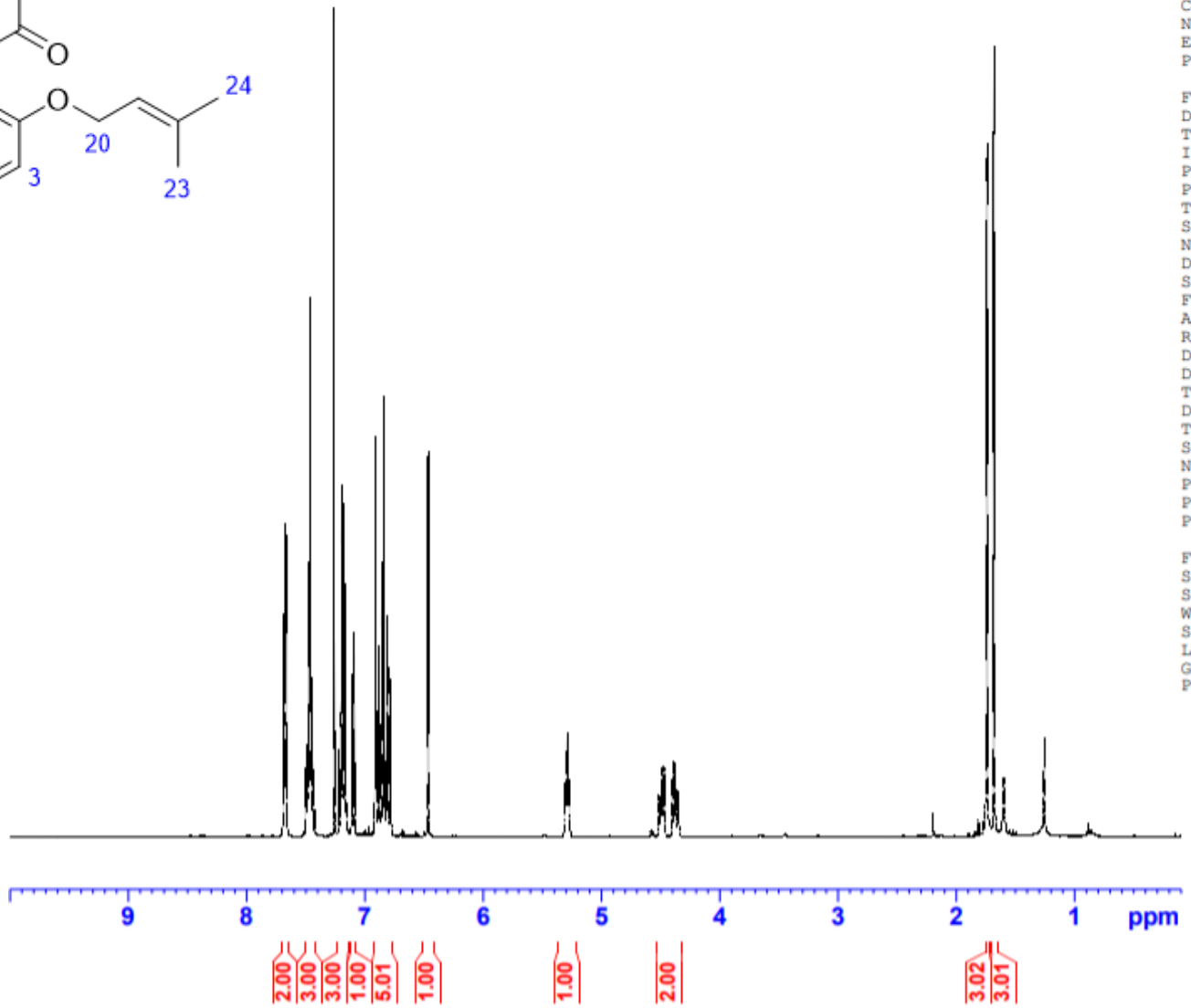


BRUKER

```
Current Data Parameters
NAME        JS-4-305 col
EXPNO                11
PROCNO                1

F2 - Acquisition Parameters
Date_          20210422
Time              23.16 h
INSTRUM       AVIII_400
PROBHD   Z108618_0146 (
PULPROG          zgpg30
TD                96150
SOLVENT           CDCl3
NS                 4096
DS                    4
SWH           24038.461 Hz
FIDRES         0.500020 Hz
AQ             1.9999200 sec
RG                 2050
DW                20.800 usec
DE                 6.50 usec
TE                300.0 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         100.6178003 MHz
NUC1                13C
P0                 3.00 usec
P1                 9.00 usec
PLW1         96.68000031 W
SFO2         400.1116004 MHz
NUC2                1H
CPDPRG[2         waltz64
PCPD2             90.00 usec
PLW2         17.29199982 W
PLW12         0.48032999 W
PLW13         0.24160001 W

F2 - Processing parameters
SI                131072
SF           100.6077404 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

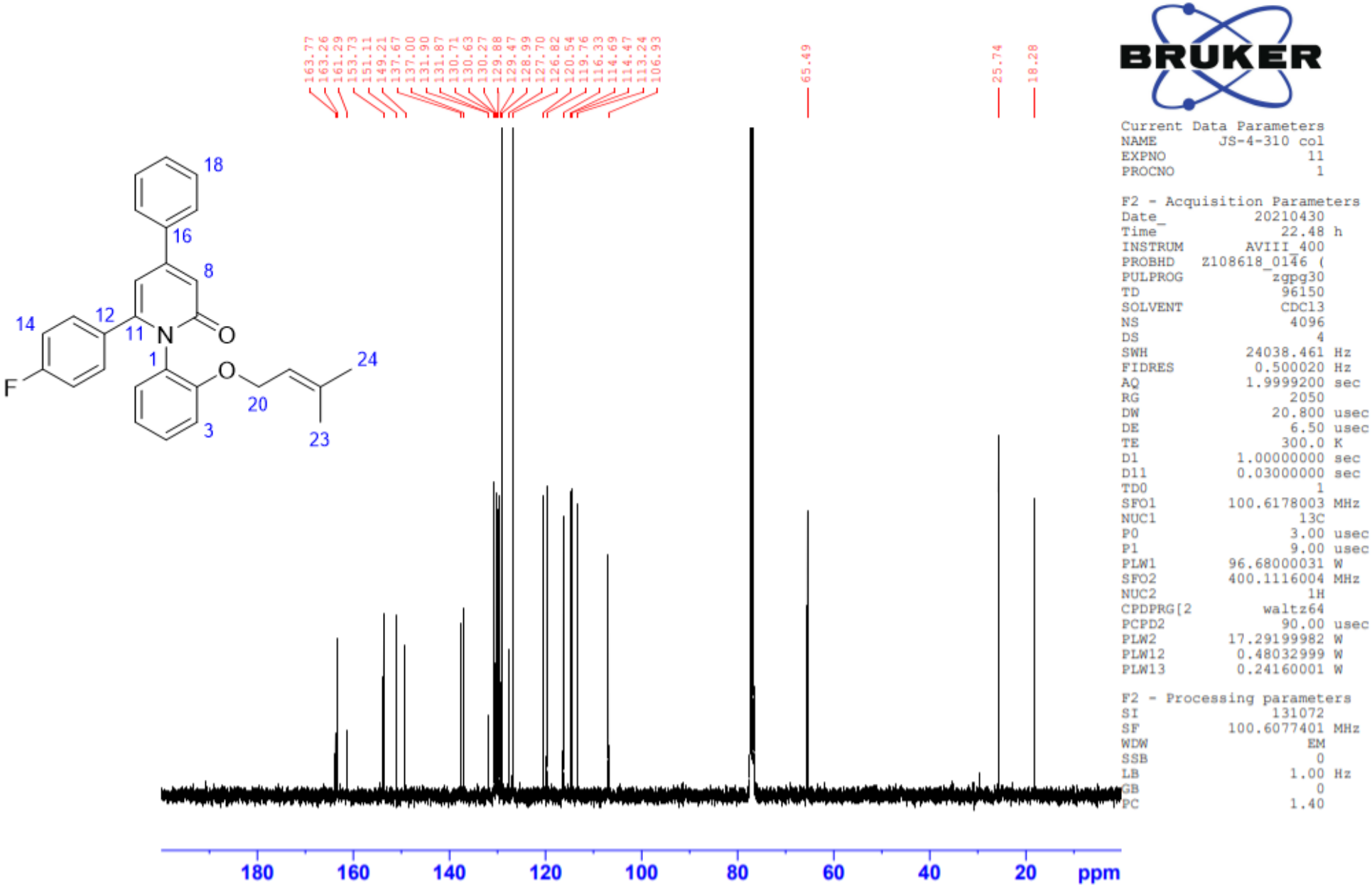
4p - ^{1}H NMR, 400 MHz, $CDCl_3$





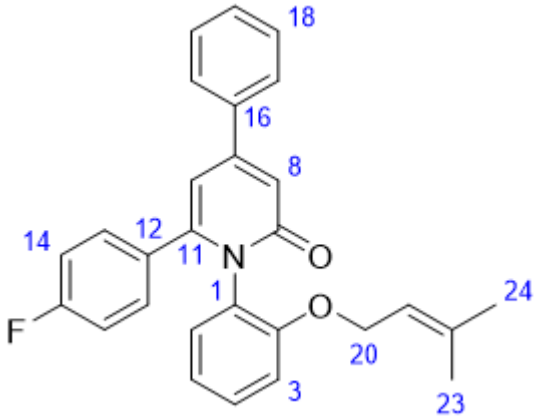

```
Current Data Parameters
NAME        JS-4-310 col
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20210430
Time              14.54 h
INSTRUM        AVIII_400
PROBHD   Z108618_0146 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ             3.9845889 sec
RG                   256
DW                60.800 usec
DE                17.42 usec
TE                300.0 K
D1           1.00000000 sec
TD0                    1
SFO1         400.1124708 MHz
NUC1                  1H
P0                 5.00 usec
P1                15.00 usec
PLW1         17.29199982 W

F2 - Processing parameters
SI                 32768
SF           400.1100102 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

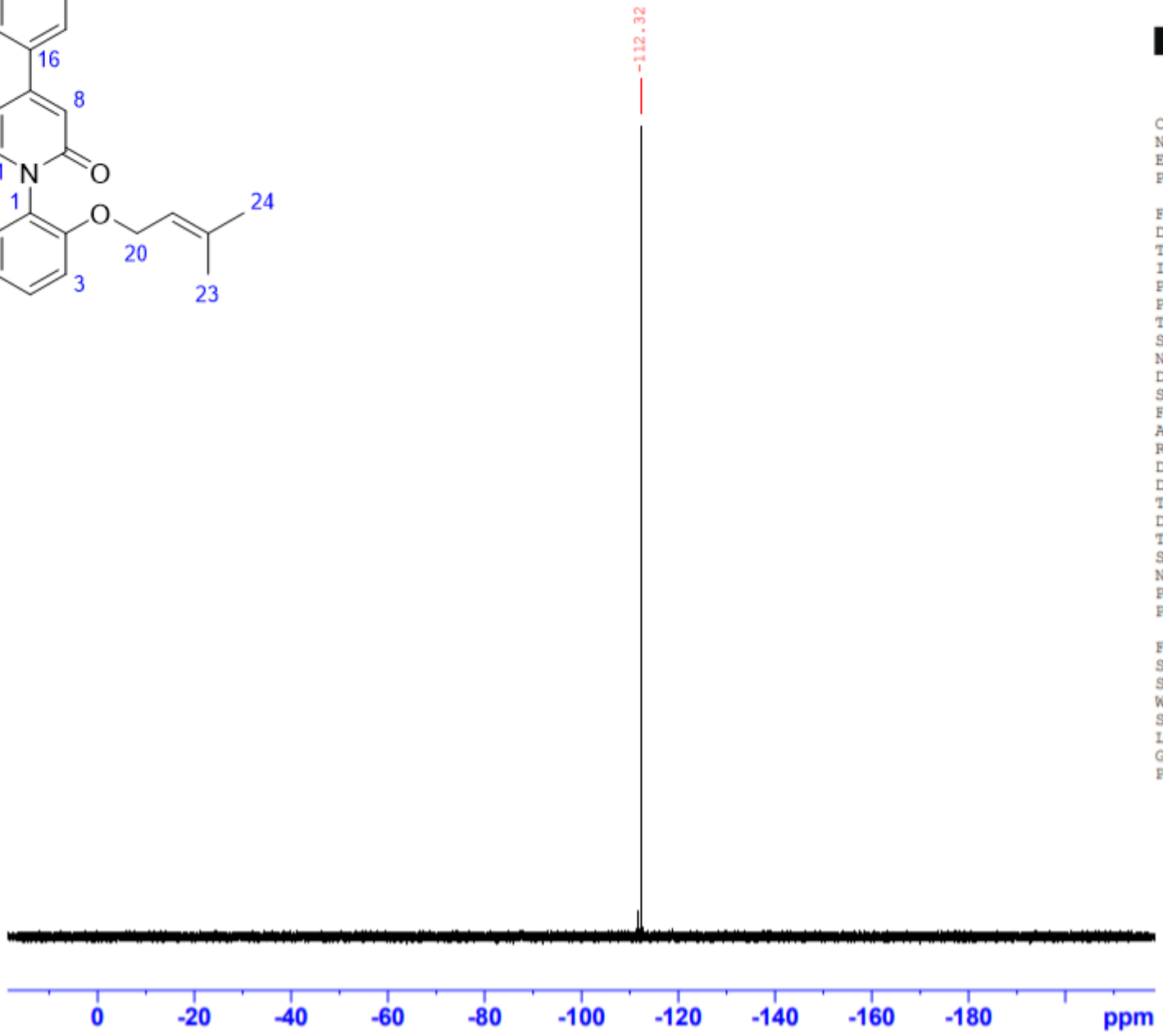
4p - ^{13}C NMR, 101 MHz, $CDCl_3$




```
Current Data Parameters
NAME                  JS-4-310 col
EXPNO                          11
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20210430
Time                        22.48 h
INSTRUM                  AVIII_400
PROBHD           Z108618_0146 (
PULPROG                   zgpg30
TD                         96150
SOLVENT                    CDCl3
NS                          4096
DS                             4
SWH                    24038.461 Hz
FIDRES                  0.500020 Hz
AQ                      1.9999200 sec
RG                          2050
DW                        20.800 usec
DE                         6.50 usec
TE                        300.0 K
D1                     1.00000000 sec
D11                    0.03000000 sec
TD0                            1
SFO1                 100.6178003 MHz
NUC1                         13C
P0                         3.00 usec
P1                         9.00 usec
PLW1                96.68000031 W
SFO2                400.1116004 MHz
NUC2                         1H
CPDPRG[2                waltz64
PCPD2                     90.00 usec
PLW2                17.29199982 W
PLW12                0.48032999 W
PLW13                0.24160001 W

F2 - Processing parameters
SI                          131072
SF                   100.6077401 MHz
WDW                            EM
SSB                             0
LB                          1.00 Hz
GB                              0
PC                           1.40
```

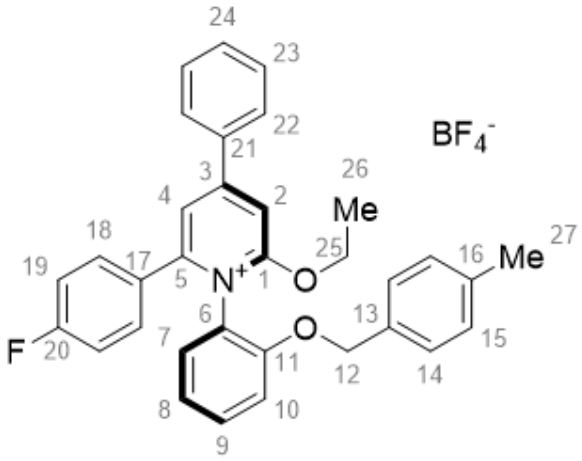
4p - ^{19}F NMR, 376 MHz, $CDCl_3$

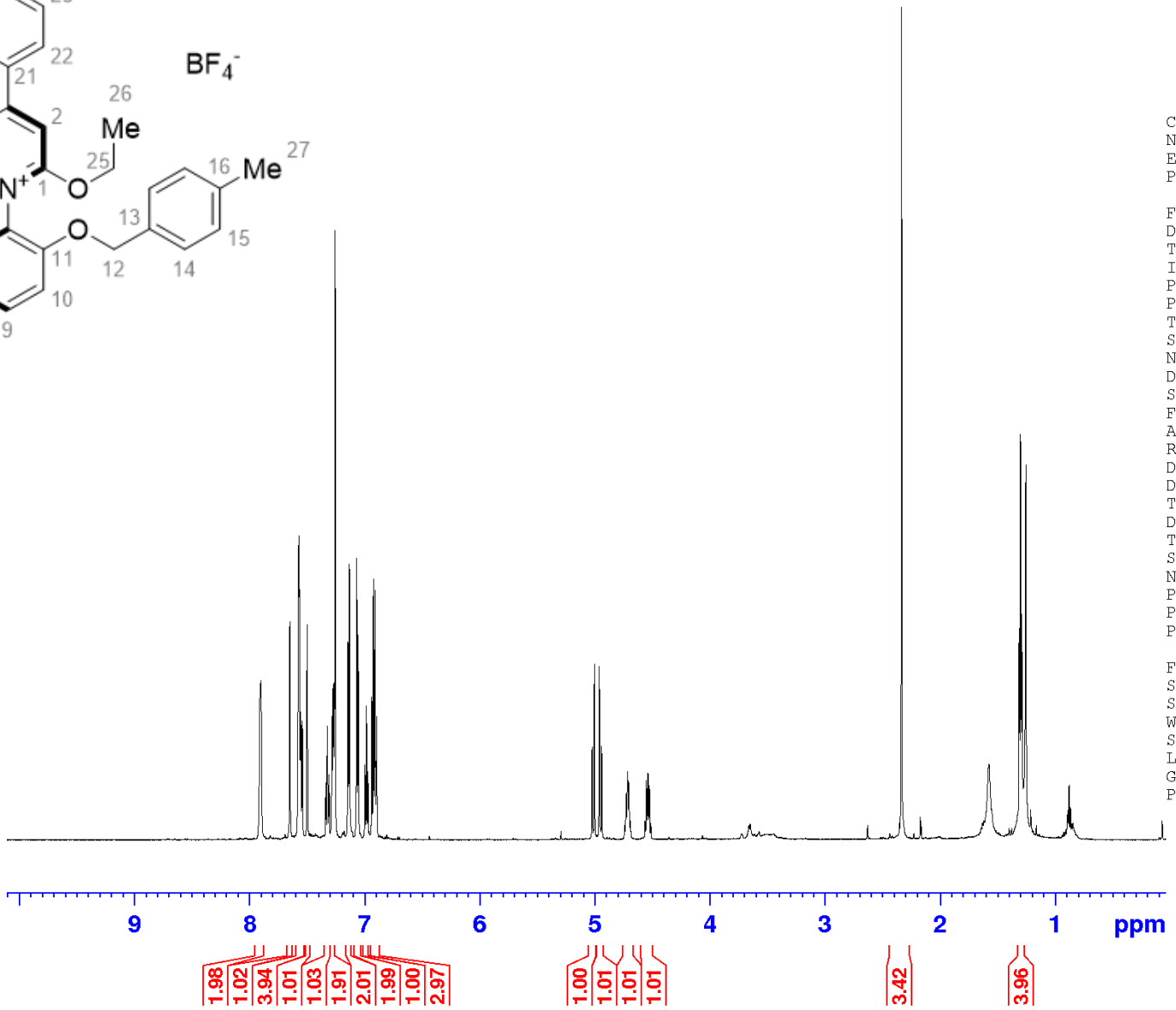





```
Current Data Parameters
NAME        JS-4-310 col
EXPNO                19
PROCNO                1

F2 - Acquisition Parameters
Date_          20210505
Time             14.01 h
INSTRUM       AVIII_400
PROBHD   Z108618_0146 (
PULPROG              zg
TD               261992
SOLVENT            CDCl3
NS                   16
DS                    4
SWH           89285.711 Hz
FIDRES         0.681591 Hz
AQ             1.4671552 sec
RG                 575
DW                5.600 usec
DE                7.11 usec
TE                300.0 K
D1            1.00000000 sec
TD0                   1
SFO1        376.4418995 MHz
NUC1                19F
P1                11.80 usec
PLW1        32.96500015 W

F2 - Processing parameters
SI               262144
SF          376.4795470 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

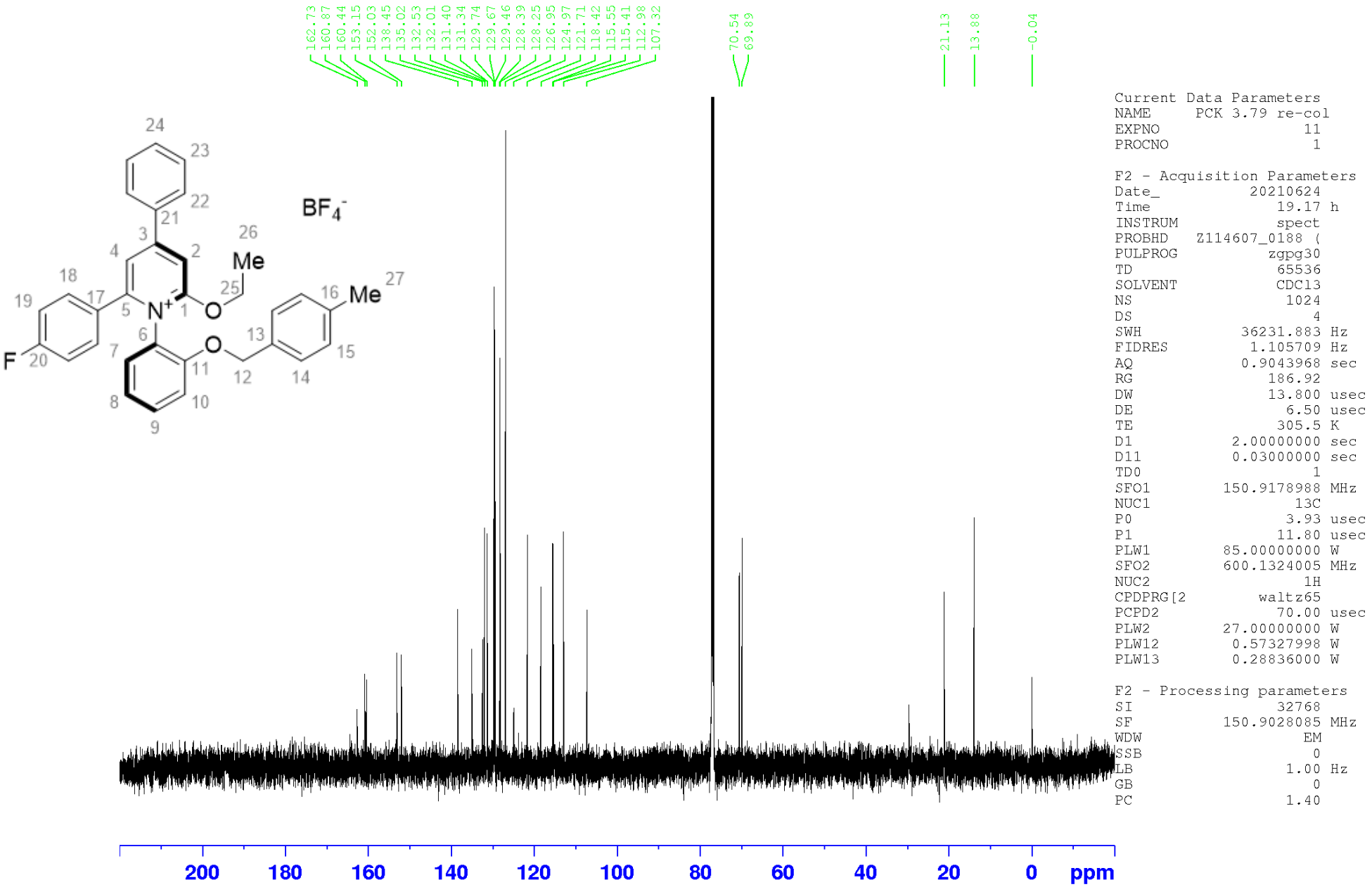
7 - ^{1}H NMR, 600 MHz, $CDCl_3$





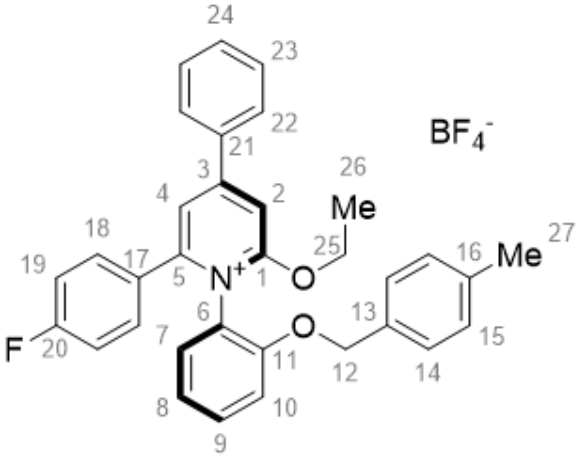

```
Current Data Parameters
NAME      PCK 3.79 re-col
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210624
Time              18.25 h
INSTRUM             spect
PROBHD    Z114607_0188 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           12019.230 Hz
FIDRES         0.366798 Hz
AQ             2.7262976 sec
RG                105.21
DW                41.600 usec
DE                12.10 usec
TE                307.4 K
D1            1.00000000 sec
TD0                    1
SFO1         600.1337058 MHz
NUC1                  1H
P0                  3.33 usec
P1                 10.00 usec
PLW1         26.60000038 W

F2 - Processing parameters
SI                 65536
SF           600.1300153 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

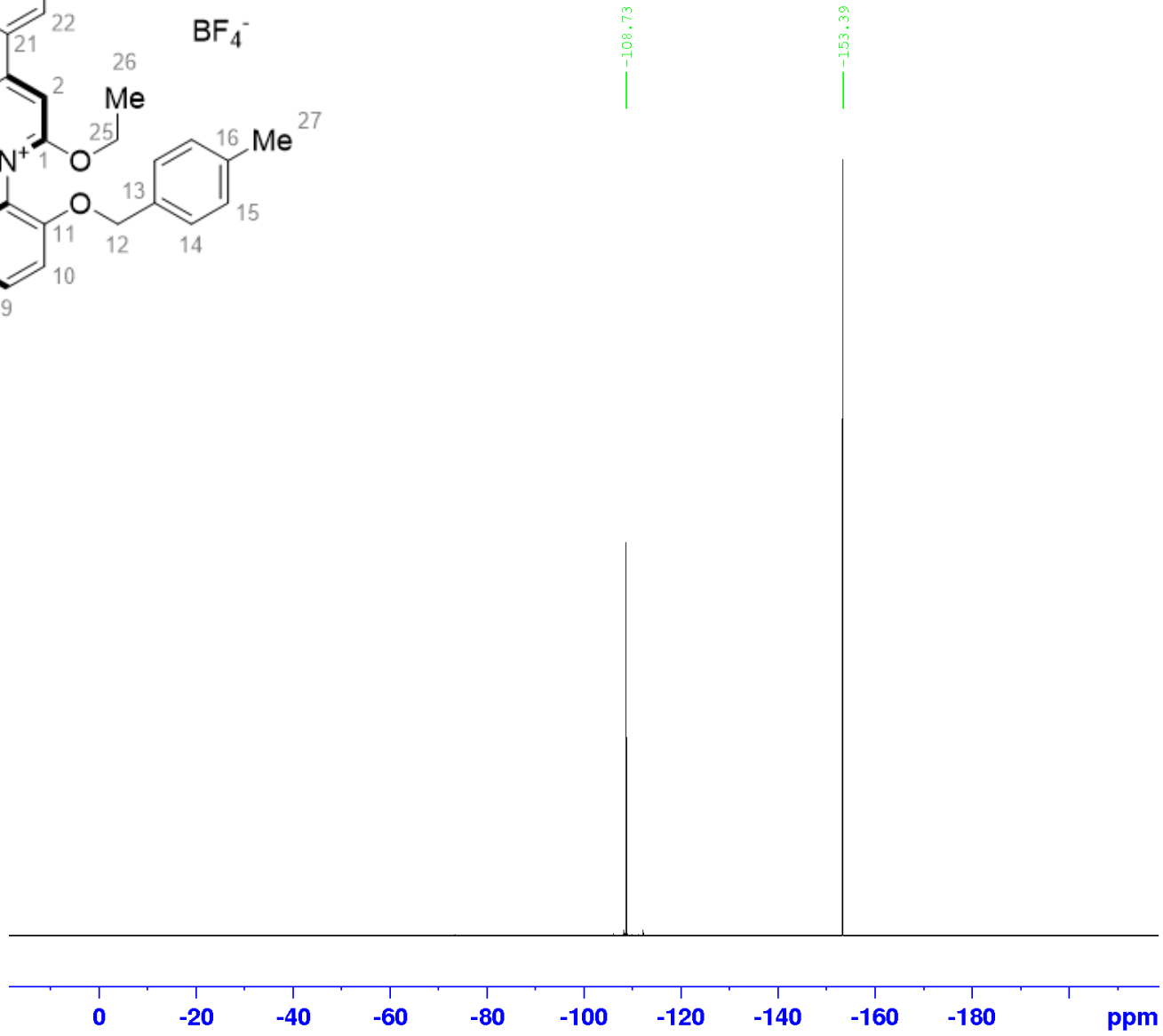
7 - ^{13}C NMR, 151 MHz, $CDCl_3$




```
Current Data Parameters
NAME      PCK 3.79 re-col
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210624
Time              19.17 h
INSTRUM             spect
PROBHD   Z114607_0188 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           36231.883 Hz
FIDRES         1.105709 Hz
AQ             0.9043968 sec
RG                186.92
DW                13.800 usec
DE                 6.50 usec
TE                305.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        150.9178988 MHz
NUC1                13C
P0                 3.93 usec
P1                11.80 usec
PLW1        85.00000000 W
SFO2        600.1324005 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             70.00 usec
PLW2        27.00000000 W
PLW12        0.57327998 W
PLW13        0.28836000 W

F2 - Processing parameters
SI                32768
SF          150.9028085 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

7 - ^{19}F NMR, 564 MHz, $CDCl_3$






```
Current Data Parameters
NAME            PCK 3.79
EXPNO                 18
PROCNO                 1

F2 - Acquisition Parameters
Date_           20210612
Time                4.14 h
INSTRUM             spect
PROBHD   Z114607_0188 (
PULPROG       zgfhigqn.b
TD                261896
SOLVENT             CDCl3
NS                    16
DS                     4
SWH           133928.578 Hz
FIDRES          1.022761 Hz
AQ              0.9777451 sec
RG                186.92
DW                 3.733 usec
DE                 6.70 usec
TE                302.0 K
D1             4.00000000 sec
D11            0.03000000 sec
D12            0.00002000 sec
TD0                    1
SFO1         564.6299217 MHz
NUC1                  19F
P1                12.00 usec
PLW1         49.00000000 W
SFO2         600.1324005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2             70.00 usec
PLW2         27.00000000 W
PLW12         0.57327998 W

F2 - Processing parameters
SI                262144
SF           564.6863882 MHz
WDW                   EM
SSB                    0
LB                  0.50 Hz
GB                     0
PC                  2.00
```

9 References

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