Electronic Supplementary Material (ESI) for RSC Advances.

Synthesis of 4-(1*H*-isochromen-1-yl)isoquinolines through silver-catalysed homodimerization of ortho-alkynylaryl-aldehydes and subsequent condensation of 1,5-dicarbonyl motif with NH_3

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General information

All reactions were conducted in clean glassware with magnetic stirring. Commercially available reagents were used as received without further purification. Solvents were treated prior to use according to the standard methods unless otherwise noted. Dry THF was purchased from *Energy Chemical* and used as received. For chromatographic purification, technical-grade solvents were used. Purified compounds were further dried on high vacuum. NMR-spectra were measured in the given solvent at RT on *Bruker Ascend*^{TM} *500M* (500.1 MHz, ^{1}H; 125.8 MHz, ^{13}C) instrument operating at the denoted spectrometer frequency given in mega Hertz (MHz) for the specified nucleus. Chemical shifts are given in parts per million (ppm) relative to tetramethylsilane (TMS) as an external standard for ^{1}H- and ^{13}C-NMR spectra and calibrated against the solvent residual peak. Multiplicities are reported as follows: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, or as combination of them. Coupling constants *J* are given in Hertz (Hz). High resolution mass spectra were obtained with a Micromass GCT-TOF mass spectrometer.

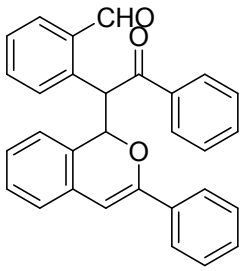
All the substrates were prepared according to the literature procedures.[1]

1 Z. Cao, H. Zhu, X. Meng, L. Tian, X. Sun, G. Chen and J. You, *Chem. Eur. J.*, 2016, **22**, 9125.

Representative procedure for the synthesis of 4-(1*H*-isochromen-1-yl) isoquinolines.

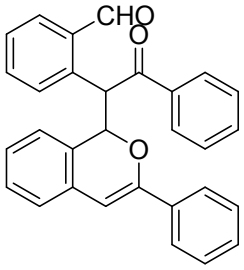
To a stirred solution of ortho-alkynylbenzaldehyde **1a** (82.5 mg, 0.4 mmol, 1.0 equiv) in THF (1.6 mL) was added $AgBF_4$ (7.8 mg, 0.04 mmol, 0.1 equiv) at room temperature. The reaction proceeded for about 8 hours. The mixture was filtered by a short silica gel column and volatiles were then evaporated. The resulting crude 1,5-dicarbonyl **2** was dissolved into methanol (1.6 mL) followed by injecting the solution of NH_3 in methanol (0.15 mL, 2.0 mol/L, 1.5 equiv). The mixture was then stirred at room temperature for about 12 hours. After the evaporation of solvent, the residue was then suffered to column chromatography to afford the desired isoquinolines **3a**.

Characterization for all new compounds



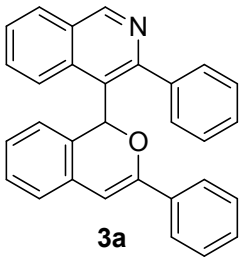
one isomer of **2** (High polarity)

48% yield, White solid, m.p. 109-110 ℃. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 9.58 (s, 1H), 7.99 (d, *J* = 7.3 Hz, 2H), 7.79 (d, *J* = 7.8 Hz, 1H), 7.58-7.51 (m, 3H), 7.49 (dd, *J* = 7.6, 1.3 Hz, 1H), 7.44-7.34 (m, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 7.23-7.17 (m, 3H), 7.10 (ddd, *J* = 7.4, 7.1, 1.4 Hz, 1H), 7.06 (d, *J* = 6.7 Hz, 1H), 7.04 (d, *J* = 9.8 Hz, 1H), 6.67 (ddd, *J* = 7.4, 7.4, 1.4 Hz, 1H), 6.59 (s, 1H), 6.19 (d, *J* = 9.7 Hz, 1H), 5.93 (d, *J* = 7.5 Hz, 1H); **^{13}C NMR** (125.8 MHz, $CDCl_3$): δ 198.2, 191.9, 150.7, 136.9, 135.5, 134.4, 134.0, 133.5, 133.2, 131.3, 129.3, 128.7, 128.64, 128.55, 128.2, 128.1, 128.0, 127.0, 125.4, 125.2, 125.1, 123.7, 100.5, 79.9, 46.1 ppm. **HRMS** (ESI) calcd. For $C_{30}H_{23}O_3$ $(M + H)^+$: 431.1642, Found: 431.1648.



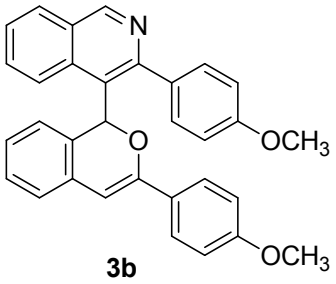
the other isomer of **2** (Low polarity)

45% yield, White solid, m.p. 107-109 ℃. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 9.65 (s, 1H), 8.01 (d, *J* = 7.9 Hz, 1H), 7.86 (dd, *J* = 8.2, 1.0 Hz, 2H), 7.63 (ddd, *J* = 7.8, 7.3, 1.6 Hz, 1H), 7.50 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.45 (ddd, *J* = 7.6, 7.3, 1.1 Hz, 1H), 7.39 (dddd, *J* = 7.4, 7.4, 1.2, 1.2 Hz, 1H), 7.29-7.24 (m, 2H), 7.24-7.12 (m, 6H), 7.10-7.06 (m, 2H), 7.04 (ddd, *J* = 7.4, 7.4, 1.5 Hz, 1H), 7.00 (d, *J* = 9.8 Hz, 1H), 6.00 (s, 1H), 6.30 (d, *J* = 9.8 Hz, 1H); **^{13}C NMR** (125.8 MHz, $CDCl_3$): δ 198.6, 192.9, 150.7, 136.9, 136.8, 135.1, 134.7, 133.9, 133.5, 133.2, 130.5, 129.8, 129.6, 128.7, 128.6, 128.41, 128.37, 127.9, 127.7, 126.5, 126.0, 124.5, 124.4, 101.0, 79.8, 49.1 ppm. **HRMS** (ESI) calcd. For $C_{30}H_{23}O_3$ $(M + H)^+$: 431.1642, Found: 431.1645.



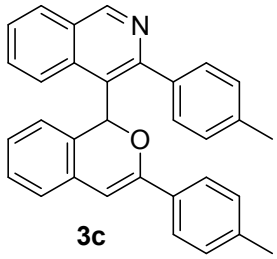
3a

89% yield, White solid, m.p. 181-182 ℃. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 9.42 (s, 1H), 8.25 (d, *J* = 8.6 Hz, 1H), 8.07 (d, *J* = 8.1 Hz, 1H), 7.71-7.65 (m, 4H), 7.57 (ddd, *J* = 8.0, 7.0, 0.8 Hz, 1H), 7.50 (ddd, *J* = 7.2, 7.2, 1.2 Hz, 1H), 7.45-7.36 (m, 3H), 7.36-7.27 (m, 3H), 7.24 (dd, *J* = 7.6, 7.4 Hz, 1H), 7.19 (d, *J* = 6.7 Hz, 1H), 6.98 (ddd, *J* = 7.5, 7.5, 1.1 Hz, 1H), 6.88 (s, 1H), 6.57 (s, 1H), 6.51 (d, *J* = 7.6 Hz, 1H); **^{13}C NMR** (125.8 MHz, $CDCl_3$): δ 154.1, 153.6, 153.2, 140.2, 135.3, 134.0, 131.9, 131.3, 129.9, 129.2, 128.9, 128.7, 128.4, 128.3, 128.23, 128.20, 128.16, 127.7, 126.8, 126.7, 125.0, 124.9, 124.5, 123.8, 101.2, 78.4 ppm. **HRMS** (ESI) calcd. For $C_{30}H_{22}NO$ $(M + H)^+$: 412.1696, Found: 412.1690.



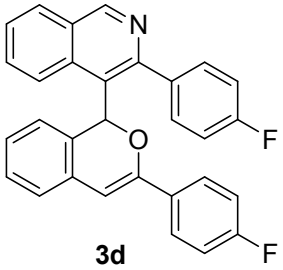
3b

62% yield, White solid, m.p. 91-93 ℃. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 9.39 (s, 1H), 8.21 (d, *J* = 8.6 Hz, 1H), 8.04 (d, *J* = 8.0 Hz, 1H), 7.62 (d, *J* = 8.9 Hz, 2H), 7.60 (d, *J* = 8.5 Hz, 2H), 7.54 (dd, *J* = 7.5, 7.3 Hz, 1H), 7.47 (dd, *J* = 8.3, 7.1 Hz, 1H), 7.21 (dd, *J* = 7.5, 7.4 Hz, 1H), 7.16 (d, *J* = 7.2 Hz, 1H), 6.97-6.92 (m, 3H), 6.87-6.83 (m, 3H), 6.48 (d, *J* = 7.5 Hz, 1H), 6.45 (s, 1H), 3.81 (s, 3H), 3.80 (s, 3H); **^{13}C NMR** (125.8 MHz, $CDCl_3$): δ 160.3, 159.7, 153.8, 153.7, 153.1, 135.4, 132.7, 132.3, 131.1, 130.6, 129.7, 128.5, 128.14, 128.10, 127.7, 126.6, .126.3, 124.9, 124.1, 123.4, 113.9, 113.7, 99.6, 78.5, 55.3(2C) ppm. **HRMS** (ESI) calcd. For $C_{32}H_{26}NO_3$ $(M + H)^+$: 472.1907, Found: 472.1904.



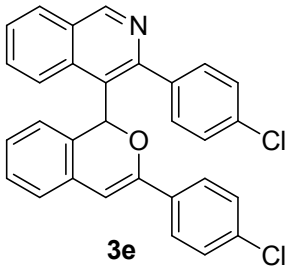
3c

91% yield, White solid, m.p. 90-92 ℃. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 9.41 (s, 1H), 8.23 (d, *J* = 8.6 Hz, 1H), 8.06 (d, *J* = 8.0 Hz, 1H), 7.59-7.52 (m, *5*H), 7.49 (dd, *J* = 7.9, 7.7 Hz, 1H), 7.24-7.19 (m, 3H), 7.16 (d, *J* = 7.4 Hz, 1H), 7.13 (d, *J* = 8.1 Hz, 2H), 6.95 (dd, *J* = 7.4, 7.3 Hz, 1H), 6.85 (s, 1H), 6.50 (s, 1H), 6.46 (d, *J* = 7.6 Hz, 1H), 2.36 (s, 3H), 2.34 (s, 3H); **^{13}C NMR** (125.8 MHz, $CDCl_3$): δ 154.2, 153.8, 153.1, 139.0, 138.1, 137.3, 135.3, 132.1, 131.3, 131.2, 129.8, 129.12, 129.08, 129.0, 128.6, 128.1, 127.7, 126.7, 126.5, 125.0, 124.9, 124.4, 123.6, 100.4, 78.4, 21.3, 21.2 ppm. **HRMS** (ESI) calcd. For $C_{32}H_{26}NO$ $(M + H)^+$: 440.2009, Found: 440.2006.



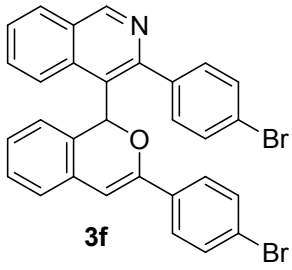
3d

76% yield, White solid, m.p. 141-143 ℃. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 9.40 (s, 1H), 8.21 (d, *J* = 8.5 Hz, 1H), 8.07 (d, *J* = 8.1 Hz, 1H), 7.68-7.60 (m, 4H), 7.58 (dd, *J* = 7.4, 7.3 Hz, 1H), 7.51 (dd, *J* = 7.8, 7.3 Hz, 1H), 7.25 (dd, *J* = 7.5, 7.5 Hz, 1H), 7.19 (d, *J* = 7.5 Hz, 1H), 7.11 (dd, *J* = 8.6, 8.4 Hz, 2H), 7.04-6.96 (m, 3H), 6.80 (s, 1H), 6.52-6.47 (m, 2H); **^{13}C NMR** (125.8 MHz, $CDCl_3$): 163.2 (d, *J* = 249.0 Hz), 162.9 (d, *J* = 248.2 Hz), 153.3, 153.1, 152.8, 136.2, 135.2, 131.8, 131.0 (d, *J* = 8.3 Hz), 130.9, 130.1, 130.04, 130.01, 128.7, 128.4, 128.2, 127.6, 126.9 (d, *J* = 12.8 Hz), 126.8 (d, *J* = 12.6 Hz), 124.8, 124.4, 123.8, 115.4 (d, *J* = 21.6 Hz), 115.3 (d, *J* = 21.7 Hz), 100.9, 78.5. **HRMS** (ESI) calcd. For $C_{30}H_{20}F_2NO$ $(M + H)^+$: 448.1507, Found: 448.1508.



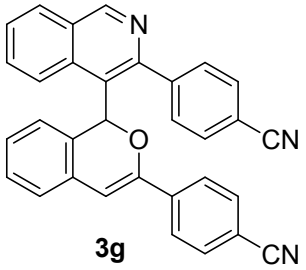
3e

63% yield, White solid, m.p. 117-118 ℃. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 9.40 (s, 1H), 8.21 (d, *J* = 8.6 Hz, 1H), 8.07 (d, *J* = 8.1 Hz, 1H), 7.61-7.55 (m, 5H), 7.51 (dd, *J* = 7.8, 7.5 Hz, 1H), 7.39 (d, *J* = 8.3 Hz, 2H), 7.28 (d, *J* = 8.7 Hz, 2H), 7.24 (d, *J* = 7.5 Hz, 1H), 7.19 (d, *J* = 7.3 Hz, 1H), 6.99 (ddd, *J* = 7.5, 7.5, 1.0 Hz, 1H), 6.79 (s, 1H), 6.55 (s, 1H), 6.49 (d, *J* = 7.6 Hz, 1H); **^{13}C NMR** (125.8 MHz, $CDCl_3$): δ 153.4, 152.9, 152.6, 138.5, 135.1, 134.7, 134.5, 132.3, 131.6, 130.9, 130.6, 130.1, 128.7, 128.6, 128.5, 128.4, 128.2, 127.6, 127.08, 127.05, 126.2, 124.8, 124.4, 124.0, 101.6, 78.4 ppm. **HRMS** (ESI) calcd. For $C_{30}H_{20}Cl_2NO$ $(M + H)^+$: 480.0916, Found: 480.0918.



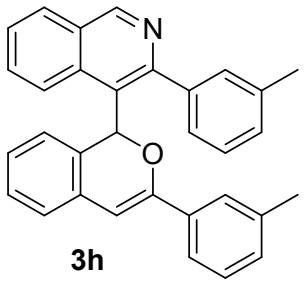
3f

53% yield, White solid, m.p. 121-123 ℃. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 9.40 (s, 1H), 8.19 (d, *J* = 8.6 Hz, 1H), 8.07 (d, *J* = 8.1 HZ, 1H), 7.61-7.49 (m, 8H), 7.44 (d, *J* = 8.6 Hz, 2H), 7.24 (d, *J* = 7.5 Hz, 1H), 7.19 (d, *J* = 7.2 Hz, 1H), 6.99 (dd, *J* = 7.5, 7.5 Hz, 1H), 6.76 (s, 1H), 6.55 (s, 1H), 6.46 (d, *J* = 7.6 Hz, 1H); **^{13}C NMR** (125.8 MHz, $CDCl_3$): δ 153.4, 152.8, 152.6, 138.9, 135.2, 132.8, 131.6, 131.54, 131.47, 130.90, 130.87, 130.2, 128.7, 128.5, 128.3, 127.6, 127.2, 127.1, 126.5, 124.8, 124.5, 124.0, 123.0, 122.9, 101.7, 78.4 ppm. **HRMS** (ESI) calcd. For $C_{30}H_{20}Br_2NO$ $(M + H)^+$: 567.9906, Found: 567.9911.



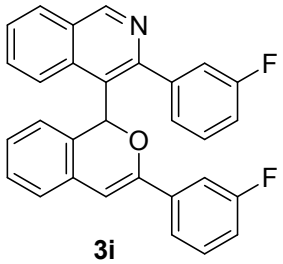
3g

31% yield, White solid, m.p. 111-113 ℃. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 9.43 (s, 1H), 8.16 (d, *J* = 9.1 Hz, 1H), 8.11 (d, *J* = 8.2 Hz, 1H), 7.78-7.68 (m, 6H), 7.67-7.62 (m, 2H), 7.61-7.52 (m, 3H), 7.27 (dd, *J* = 7.5, 7.5 Hz, 1H), 7.05 (dd, *J* = 7.5, 7.5 Hz, 1H), 6.73-6.67 (m, 2H), 6.50 (d, *J* = 7.6 Hz, 1H); **^{13}C NMR** (125.8 MHz, $CDCl_3$): δ 153.8, 151.9, 151.4, 137.9, 135.0, 132.3, 132.2, 130.9, 130.8, 130.6, 130.5, 130.1, 128.8, 128.5, 128.0, 127.7, 127.5, 127.0, 125.1, 124.8, 124.7, 118.6, 118.5, 112.1, 104.3, 78.4 ppm. **HRMS** (ESI) calcd. For $C_{32}H_{20}N_3O$ $(M + H)^+$: 462.1601, Found: 462.1598.



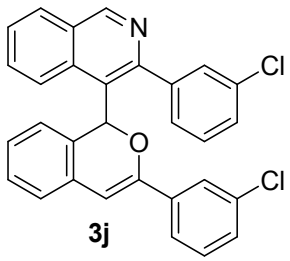
3h

76% yield, White solid, m.p. 73-74 ℃. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 9.42 (s, 1H), 8.25 (d, *J* = 8.6 Hz, 1H), 8.06 (d, *J* = 8.1 Hz, 1H), 7.59-7.54 (m, 2H), 7.54-7.47 (m, 3H), 7.43 (d, *J* = 7.5 Hz, 1H), 7.29 (dd, *J* =7.7, 7.6 Hz, 1H), 7.25-7.19 (m, *3* H), 7.17 (d, *J* = 7.3 Hz, 1H), 7.14 (d, *J* = 7.5 Hz, 1H), 6.96 (dd, *J* = 7.5, 7.4 Hz, 1H), 6.87 (s, 1H), 6.55 (s, 1H), 6.47 (d, *J* = 7.5 Hz, 1H), 2.38 (s , *3*H), 2.35(s, 3H). **^{13}C NMR** (125.8 MHz, $CDCl_3$): δ 154.4, 153.8, 153.2, 140.1, 138.1, 137.9, 135.3, 134.0, 131.9, 131.4, 130.1, 129.8, 129.7, 129.0, 128.7, 128.2, 128.13, 128.12, 128.1, 127.7, 126.7, 126.6, 126.1, 125.7, 124.9, 124.4, 123.7, 122.3, 101.1, 78.5, 21.4 (2C) ppm. **HRMS** (ESI) calcd. For $C_{32}H_{26}NO$ $(M + H)^+$: 440.2009, Found: 440.2008.



3i

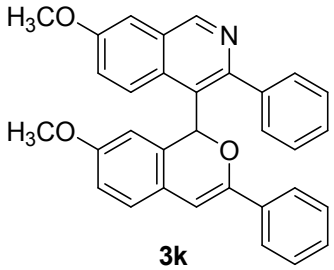
70% yield, White solid, m.p. 101-103 ℃. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 9.39 (s, 1H), 8.20 (d, *J* = 8.6 Hz, 1H), 8.07 (d, *J* = 8.1 Hz, 1H), 7.58 (dd, *J* = 7.8, 7.2 Hz, 1H), 7.51 (dd, *J* = 7.8, 7.4 Hz, 1H), 7.45 (d, *J* = 7.9 Hz, 1H), 7.41-7.31 (m, 3H), 7.31-7.21 (m, 3H), 7.19 (d, *J* = 7.3 Hz, 1H), 7.08 (dd, *J* = 8.4, 7.4 Hz, 1H), 7.02-6.95 (m, 2H), 6.81 (s, 1H), 6.56 (s, 1H), 6.47 (d, *J* = 7.6 Hz, 1H); **^{13}C NMR** (125.8 MHz, $CDCl_3$): δ 162.9 (d, *J* = 245.4 Hz), 162.6 (d, *J* = 247.1 Hz), 153.5, 152.8 (d, *J* = 1.5 Hz), 152.3 (d, *J* = 2.3 Hz), 142.2 (d, *J* = 7.3 Hz), 136.2 (d, *J* = 7.7 Hz), 135.1, 131.4, 131.1, 130.1, 129.9 (d, *J* = 8.3 Hz), 129.8 (d, *J* = 9.5 Hz), 128.8, 128.4 (d, *J* = 21.0 Hz), 127.6, 127.24, 127.16, 124.84 (d, *J* = 2.6 Hz), 124.78, 124.69, 124.6, 124.1, 120.5 (d, *J* = 2.5 Hz), 116.5 (d, *J* = 22.3 Hz), 115.7 (d, *J* = 21.3 Hz), 115.4 (d, *J* = 21.2 Hz), 111.9 (d, *J* = 23.1 Hz), 102.2, 78.4. **HRMS** (ESI) calcd. For $C_{30}H_{20}F_2NO$ $(M + H)^+$: 448.1507, Found: 448.4506.



3j

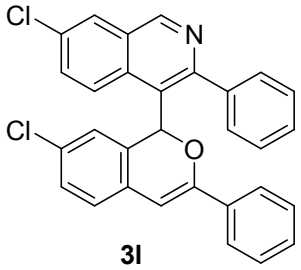
86% yield, White solid, m.p. 72-74 ℃. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 9.41 (s, 1H), 8.19 (d, *J* = 8.6 Hz, 1H), 8.08 (d, *J* = 8.1 Hz, 1H), 7.72 (s, 1H), 7.64 (s, 1H), 7.60 (dd, *J* = 7.8, 7.2 Hz, 1H), 7.56 (d, *J* = 6.5 Hz, 1H), 7.52 (dd, *J* = 7.8, 7.5 Hz, 1H), 7.47 (d, *J* = 7.5 Hz, 1H), 7.38 (d, *J* = 8.0Hz, 1H), 7.32 (dd, *J* = 7.8, 7.7 Hz, 1H), 7.29-7.23 (m, 3H), 7.19 (d, *J* = 7.3 Hz, 1H), 7.00 (dd, *J* = 7.5, 7.5 Hz, 1H), 6.80 (s, 1H), 6.57 (s, 1H), 6.47 (d, *J* = 7.6 Hz, 1H); **^{13}C NMR** (125.8 MHz, $CDCl_3$): δ 153.5, 152.7, 152.2, 141.9, 135.7, 135.1, 134.5, 134.4, 131.3, 131.0, 130.1, 129.7, 129.6, 129.5, 128.8, 128.5, 128.4, 128.3, 127.5, 127.3, 127.2, 127.1, 125.0, 124.8, 124.6, 124.1, 123.1, 102.3,

78.4 ppm. **HRMS** (ESI) calcd. For $C_{30}H_{20}Cl_2NO$ $(M + H)^+$: 480.0916, Found: 480.0914.



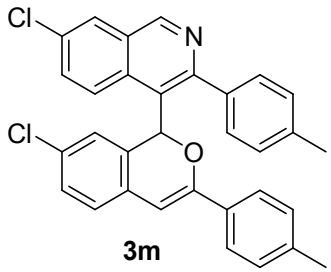
3k

71% yield, White solid, m.p. 102-103 ℃. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 9.32 (s, 1H), 8.15 (d, *J* = 9.4 Hz, 1H), 7.64 (dd, *J* = 6.6, 6.3 Hz, 4H), 7.43-7.35 (m, 3H), 7.33-7.27 (m, 4H), 7.16 (dd, *J* = 9.4, 2.5 Hz, 1H), 7.11 (d, *J* = 8.3 Hz, 1H), 6.78-6.74(m, 2H), 6.51 (s, 1H), 6.05 (d, *J* = 1.9 Hz, 1H), 3.95 (s, 3H), 3.59 (s, 3H); **^{13}C NMR** (125.8 MHz, $CDCl_3$): δ 158.8, 158.0, 151.7, 134.2, 133.3, 130.8, 130.2, 129.3, 129.2, 128.5, 128.4, 128.3, 128.2, 125.0, 124.8, 124.7, 123.1, 112.19, 112.15, 105.4, 100.9, 78.1, 55.5, 55.3 ppm. **HRMS** (ESI) calcd. For $C_{32}H_{26}NO_3$ $(M + H)^+$: 472.1907, Found: 472.1902.



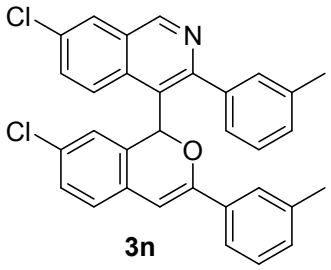
3l

50% yield, White solid, m.p. 115-116 ℃. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 9.36 (s, 1H), 8.14 (d, *J* = 9.2 Hz, 1H), 8.06 (d, *J* = 2.0 Hz, 1H), 7.67-7.61 (m, 4H), 7.49-7.40 (m, 4H), 7.36-7.31 (m, 3H), 7.21 (dd, *J* = 8.1, 1.3 Hz, 1H), 7.11 (d, *J* = 8.2 Hz, 1H), 6.79 (s, 1H), 6.52 (s, 1H), 6.43 (s, 1H); **^{13}C NMR** (125.8 MHz, $CDCl_3$): δ 154.6, 153.9, 152.5, 139.6, 133.4, 133.3, 132.8, 132.6, 132.1, 131.2, 130.5, 129.4, 129.3, 129.14, 129.10, 128.6, 128.4, 127.0, 125.1, 125.0, 124.8, 123.8, 100.4, 77.9 ppm. **HRMS** (ESI) calcd. For $C_{30}H_{20}Cl_2NO$ $(M + H)^+$: 480.0916, Found: 480.0913.



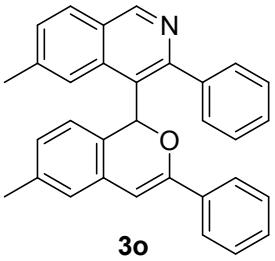
3m

71% yield, White solid, m.p. 128-129 ℃. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 9.32 (s, 1H), 8.12 (d, *J* = 9.2 Hz, 1H), 7.99 (d, *J* = 2.0 Hz, 1H), 7.56-7.50 (m, 4H), 7.40 (dd, *J* = 9.2, 2.2 Hz, 1H), 7.22 (d, *J* = 7.7 Hz, 2H), 7.16 (dd, *J* = 8.1, 1.3 Hz, 1H), 7.10 (d, *J* = 8.1 Hz, 2H), 7.05 (d, *J* = 8.2 Hz, 1H), 6.79 (s, 1H), 6.45 (s, 1H), 6.41 (s, 1H), 2.35 (s, 3H), 2.30 (s, 3H); **^{13}C NMR** (125.8 MHz, $CDCl_3$): δ 154.6, 154.1, 152.4, 139.4, 138.4, 136.7, 133.4, 132.60, 132.57, 131.8, 131.0, 130.7, 130.6, 129.3, 129.2, 129.06, 129.05, 128.4, 126.8, 125.0, 124.9, 124.7, 123.6, 99.6, 77.9, 21.3, 21.2 ppm. **HRMS** (ESI) calcd. For $C_{32}H_{24}Cl_2NO$ $(M + H)^+$: 508.1229, Found: 508.1233.



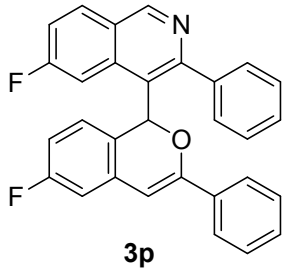
3n

76% yield, White solid, m.p. 108-109 ℃. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 9.32 (s, 1H), 8.13 (d, *J* = 9.2 Hz, 1H), 8.00 (d, *J* = 2.1 Hz, 1H), 7.53 (s, 1H), 7.46 (d, *J* = 6.5 Hz, 2H), 7.41 (d, *J* = 9.2, 2.2 Hz, 1H), 7.38 (d, *J* = 7.6 Hz, 1H), 7.28 (dd, *J* = 7.6, 7.6 Hz, 1H), 7.24-7.18 (m, 2H), 7.16 (d, *J* = 8.2, 0.8 Hz, 1H), 7.11 (d, *J* = 7.6 Hz, 1H), 7.06 (d, *J* = 8.2 Hz, 1H), 6.80 (s, 1H), 6.48 (s, 1H), 6.40 (s, 1H), 2.36 (s, 3H), 2.30 (s, 3H); **^{13}C NMR** (125.8 MHz, $CDCl_3$): δ 154.8, 154.1, 152.4, 139.5, 138.4, 138.1, 133.4, 132.7, 131.9, 131.1, 130.5, 130.08, 130.07, 129.4, 129.3, 129.1, 128.5, 128.31, 128.28, 126.9, 125.9, 125.6, 125.0, 124.7, 123.8, 122.3, 100.3, 77.9, 21.44, 21.42 ppm. **HRMS** (ESI) calcd. For $C_{32}H_{24}Cl_2NO$ $(M + H)^+$: 508.1229, Found: 508.1228.



3o

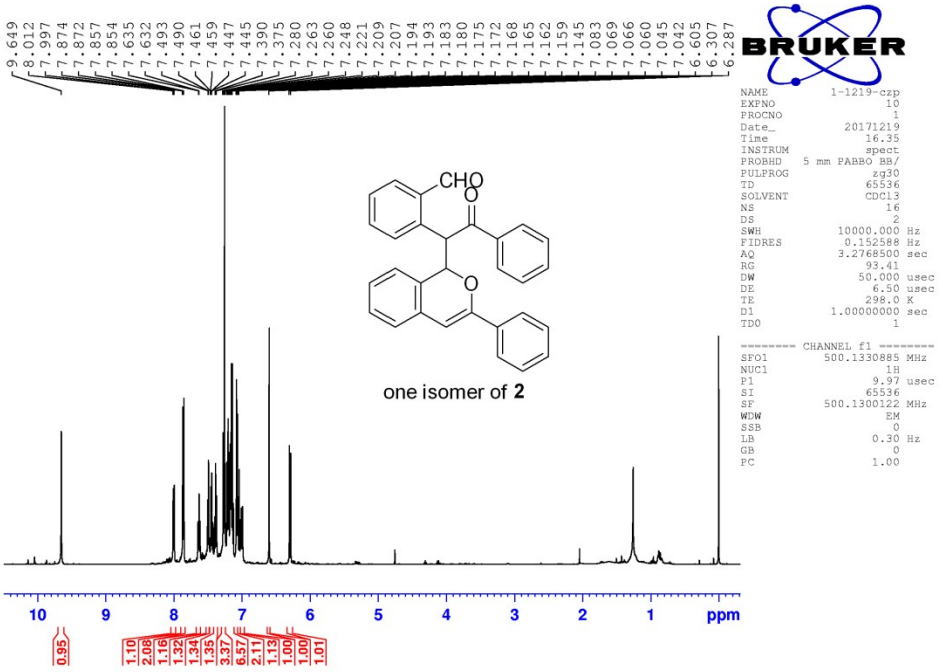
59% yield, White solid, m.p. 80-82 ℃. **^{1}H NMR** (500 MHz, $CDCl_3$): 9.34 (s, 1H), 7.98 (s, 1H), 7.96 (d, *J* = 8.3 Hz, 1H), 7.68 (d, *J* = 6.8 Hz, 2H), 7.62 (d, *J* = 6.8 Hz, 2H), 7.42-7.29 (m, 7H), 6.99 (s, 1H), 6.78 (d, *J* = 7.7 Hz, 1H), 6.77 (s, 1H), 6.50 (s, 1H), 6.37 (d, *J* = 7.7 Hz, 1H), 2.34 (s, 3H), 2.32 (s, 3H); **^{13}C NMR** (125.8 MHz, $CDCl_3$): δ 153.6, 152.4, 140.6, 139.9, 137.8, 135.8, 134.2, 131.7, 130.0, 129.4, 129.2, 128.8, 128.5, 128.4, 128.31, 128.25, 128.1, 127.4, 127.0, 126.8, 125.0, 124.7, 124.5, 101.3, 78.4, 22.7, 21.2 ppm. **HRMS** (ESI) calcd. For $C_{32}H_{26}NO$ $(M + H)^+$: 440.2009, Found: 440.2009.



3p

87% yield, White solid, m.p. 159-160 ℃. **^{1}H NMR** (500 MHz, $CDCl_3$): δ 9.38 (s, 1H), 8.10 (dd, *J* = 8.8, 5.9 Hz, 1H), 7.80 (dd, *J* = 11.2, 2.0 Hz, 1H), 7.70-7.63(m, 2H), 7.62 (d, *J* = 6.3 Hz, 2H), 7.46-7.40 (m, 3H), 7.38-7.32(m, 4H), 6.90 (dd, *J* = 9.2, 2.3 Hz, 1H), 6.78 (s, 1H), 6.68 (ddd, *J* = 8.4, 8.4, 2.3 Hz, 1H), 6.50 (s, 1H), 6.45 (dd, *J* = 7.8, 5.6 Hz, 1H); **^{13}C NMR** (125.8 MHz, $CDCl_3$): δ 163.0 (d, *J* = 245.9 Hz), 162.5 (d, *J* = 252.3 Hz), 154.9, 154.8, 152.8, 139.7, 136.9 (d, *J* = 10.9 Hz), 134.2 (d, *J* = 9.0 Hz), 133.4, 131.2 (d, *J* = 10.1 Hz), 129.4, 129.1, 128.6, 128.5, 128.4, 126.3 (d, *J* = 8.8 Hz), 126.1 (d, *J* = 2.7 Hz), 125.9, 125.2, 123.8 (d, *J* = 5.5 Hz), 117.7 (d, *J* = 26.1 Hz), 113.2 (d, *J* = 22.0 Hz), 111.8 (d, *J* = 23.2 Hz), 110.7 (d, *J* = 22.8 Hz), 100.7 (d, *J* = 1.9 Hz), 78.2 ppm. **HRMS** (ESI) calcd. For $C_{30}H_{20}F_2NO$ $(M + H)^+$: 448.1507, Found: 448.1510.

Copies of NMR spectra



BRUKER

NAME	1-1219-czp
EXPNO	10
PROCNO	1
Date_	20171219
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PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
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FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	93.41
DW	50.000 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
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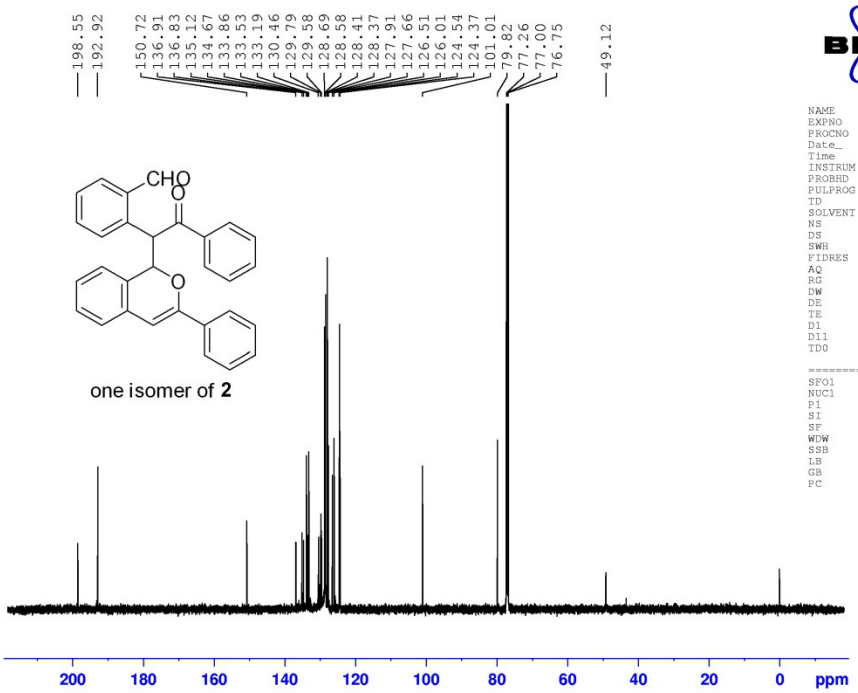
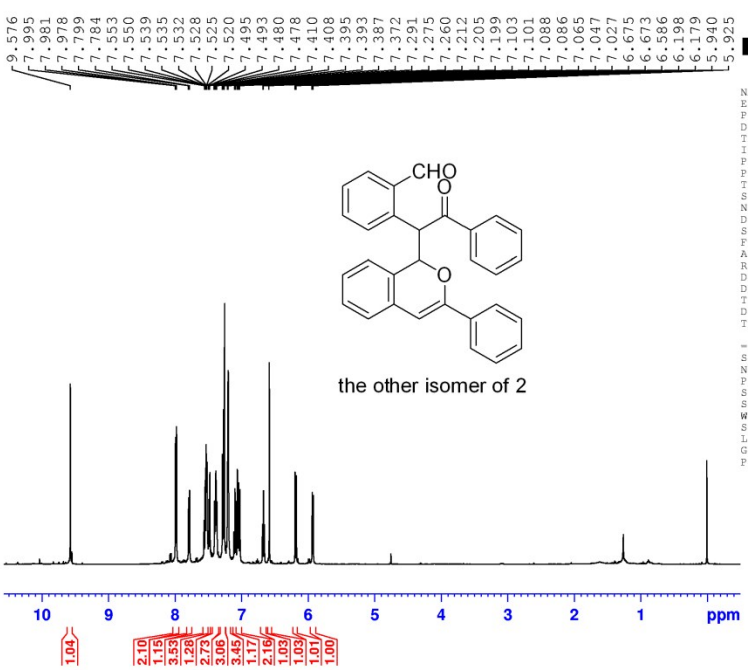
SFO1	500.1330885 MHz
NUC1	1H
P1	9.97 usec
SI	65536
SF	500.1300122 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



BRUKER

NAME	1-1221-czp
EXPNO	20
PROCNO	1
Date_	20171221
Time	17.33
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	192.1
DW	16.800 usec
DE	6.50 usec
TE	298.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	125.7703637 MHz
NUC1	13C
P1	10.03 usec
SI	32768
SF	125.7577916 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



BRUKER

NAME	2-1219-czp
EXPNO	10
PROCNO	1
Date_	20171219
Time	16.42
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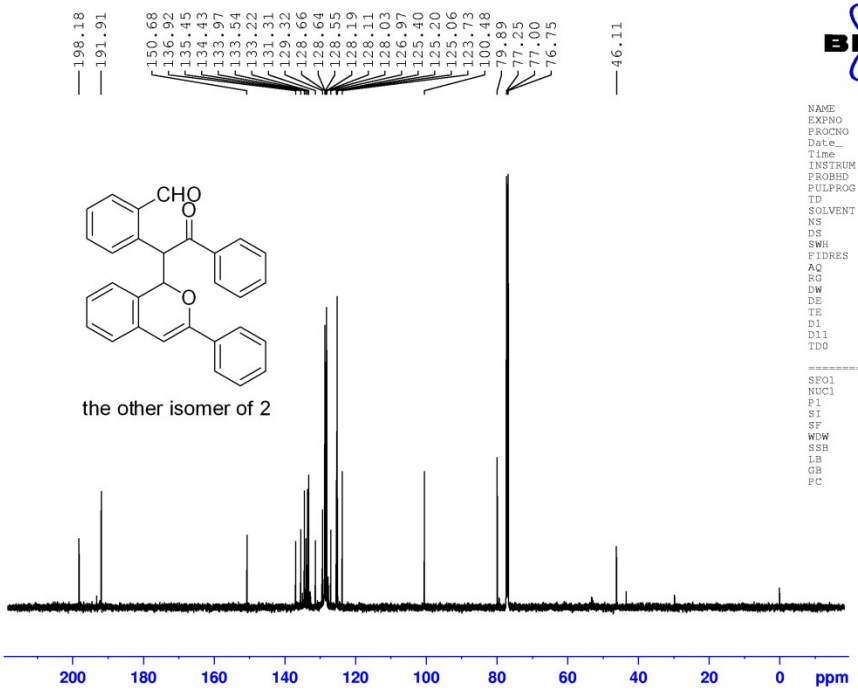
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	78.29
DW	50.000 usec
DE	6.50 usec
TE	297.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	500.1330885 MHz
NUC1	1H
P1	9.97 usec
SI	65536
SF	500.1300119 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



BRUKER

NAME	2-1220-czp
EXPNO	20
PROCNO	1
Date_	20171220
Time	15.35
INSTRUM	spect
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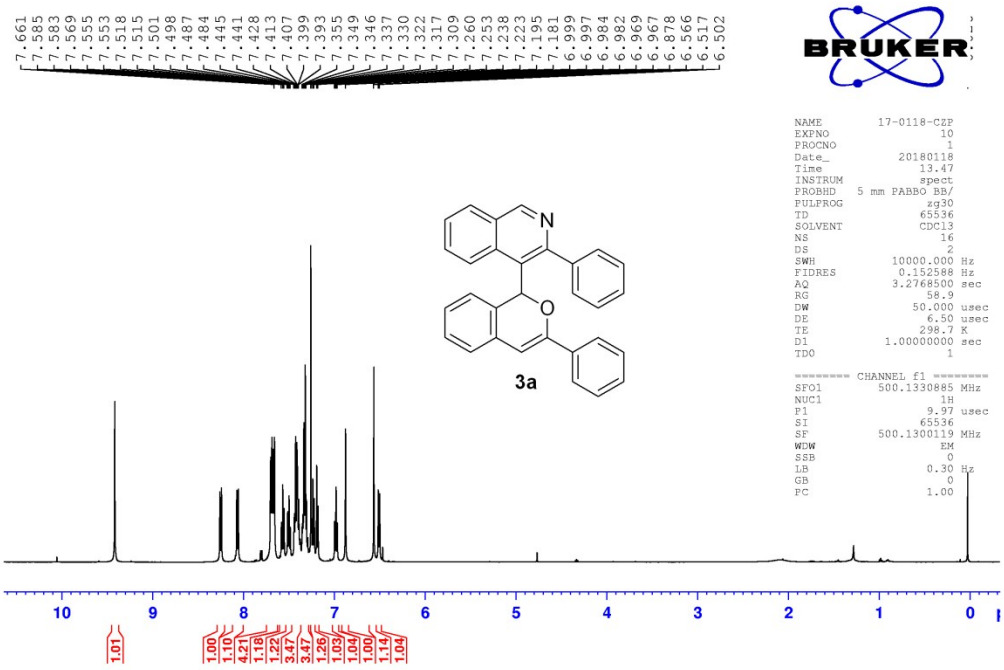
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	500
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	192.1
DW	16.800 usec
DE	6.50 usec
TE	298.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	125.7703637 MHz
NUC1	13C
P1	10.03 usec
SI	32768
SF	125.7577939 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40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7.445
7.441
7.428
7.413
7.407
7.399
7.393
7.355
7.349
7.346
7.337
7.330
7.322
7.317
7.309
7.260
7.253
7.238
7.223
7.195
7.181
6.999
6.997
6.984
6.982
6.969
6.967
6.878
6.566
6.517
6.502
BRUKER
NAME 17-0118-CZP
EXPNO 10
PROCNO 1
Date_ 20180118
Time 13.47
INSTRUM spect
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 58.9
DW 50.000 usec
DE 6.50 usec
TE 298.7 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 500.1330885 MHz
NUC1 1H
P1 9.97 usec
SI 65536
SF 500.1300119 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
O
3a
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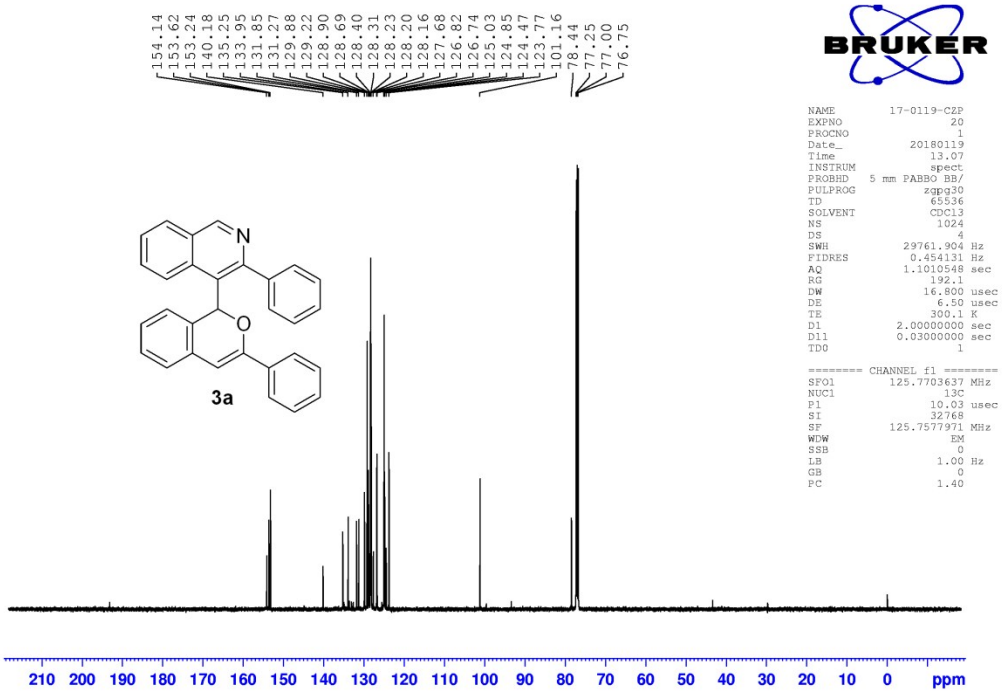
1.01 1.00 1.10 4.21 1.18 1.22 3.47 3.47 1.26 1.05 1.04 1.00 1.14 1.04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129.22
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128.69
128.40
128.31
128.23
128.20
128.16
127.68
126.82
126.74
125.03
124.85
124.47
123.77
101.16
78.44
77.25
77.00
76.75
BRUKER
NAME 17-0119-CZP
EXPNO 20
PROCNO 1
Date_ 20180119
Time 13.07
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
SFO1 125.7703637 MHz
NUC1 13C
P1 10.03 usec
SI 32768
SF 125.7577971 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
O
3a
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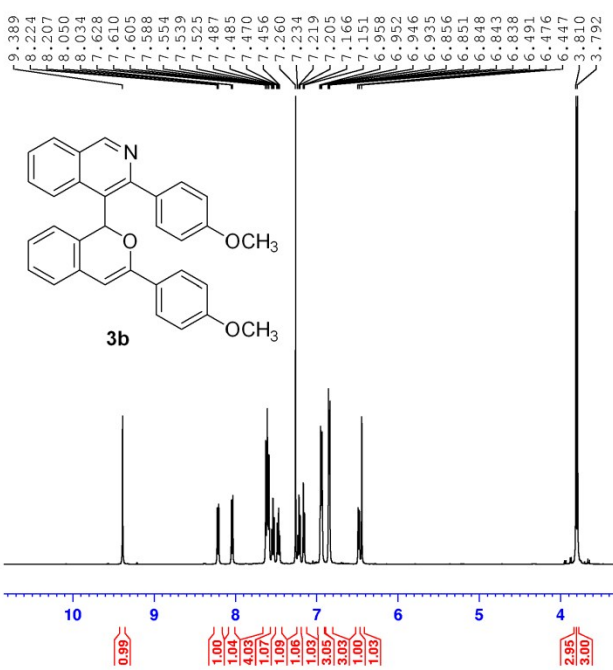

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PROCNO	1
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PULPROG	zg30
TD	65536
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NS	16
DS	2
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FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	87.51
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DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	500.1330885 MHz
NUC1	1H
P1	9.97 usec
SI	65536
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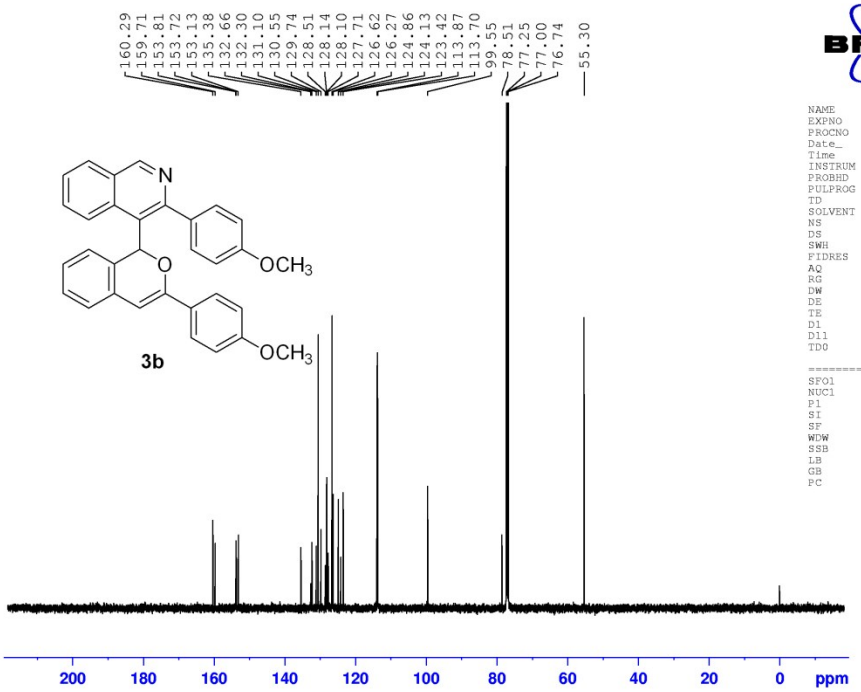
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



NAME	17-0321-C2P
EXPNO	20
PROCNO	1
Date_	20180321
Time	12.22
INSTRUM	spect
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PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	600
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SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	192.1
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	125.7703637 MHz
NUC1	13C
P1	10.03 usec
SI	32768
SF	125.7577943 MHz
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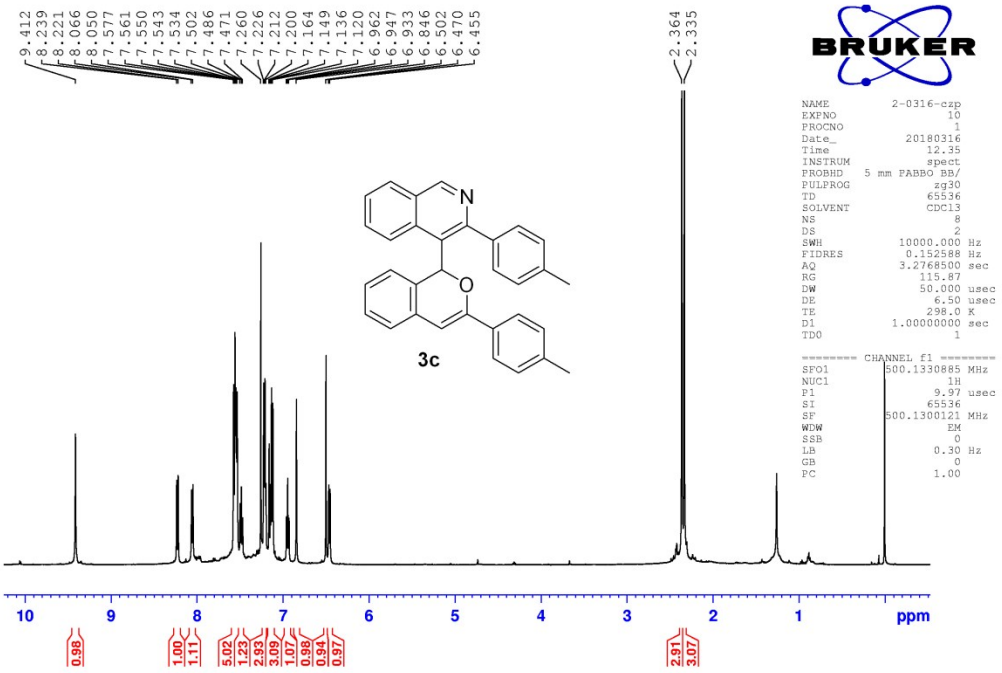
SSB	0
LB	1.00 Hz
GB	0
PC	1.40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7.136
7.120
6.962
6.947
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6.502
6.470
6.455
2.364
2.335
BRUKER
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EXPNO 10
PROCNO 1
Date_ 20180316
Time 12.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
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SOLVENT CDCl3
NS 8
DS 2
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FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 115.87
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
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TD0 1
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SFO1 500.1330885 MHz
NUC1 1H
P1 9.97 usec
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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O
3c
10
9
8
7
6
5
4
3
2
1
ppm
0.98
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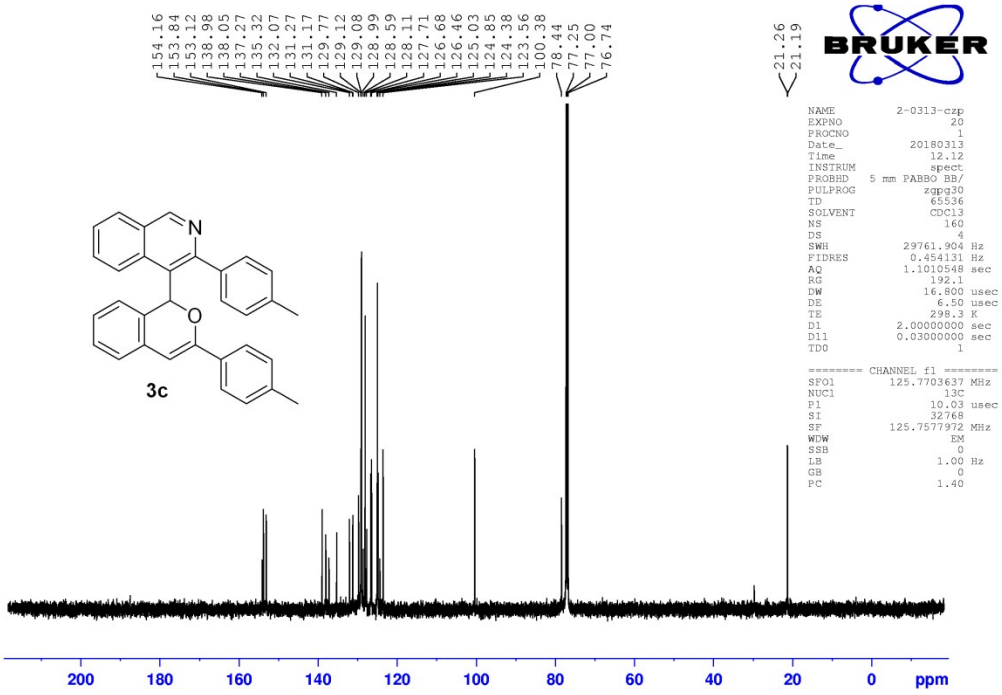
0.98
0.94
0.97
2.91
3.07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137.27
135.32
132.07
131.27
131.17
129.77
129.12
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128.11
127.71
126.68
126.46
125.03
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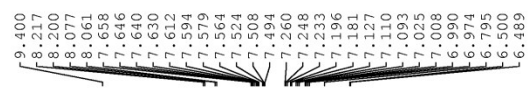
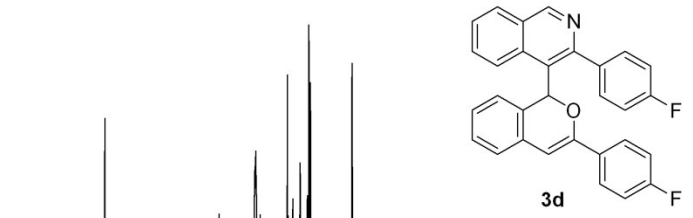
123.56
100.38
78.44
77.25
77.00
76.74
21.26
21.19
BRUKER
NAME 2-0313-czp
EXPNO 20
PROCNO 1
Date_ 20180313
Time 12.12
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 160
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.03 usec
SI 32768
SF 125.7577972 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
O
3c
200
180
160
140
120
100
80
60
40
20
0
ppm

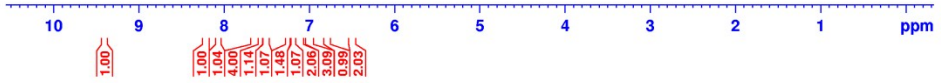


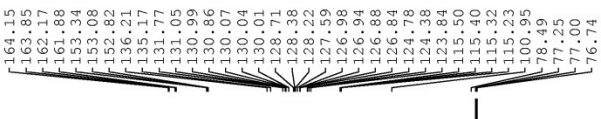


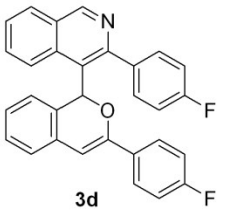
3d

BRUKER

NAME	6-0313-czp
EXPNO	10
PROCNO	1
Date_	20180313
Time	10.37
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	78.29
DW	50.000 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	500.1330885 MHz
NUC1	1H
P1	9.97 usec
SI	65536
SF	500.1300121 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



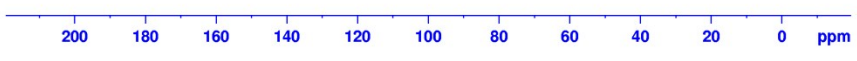


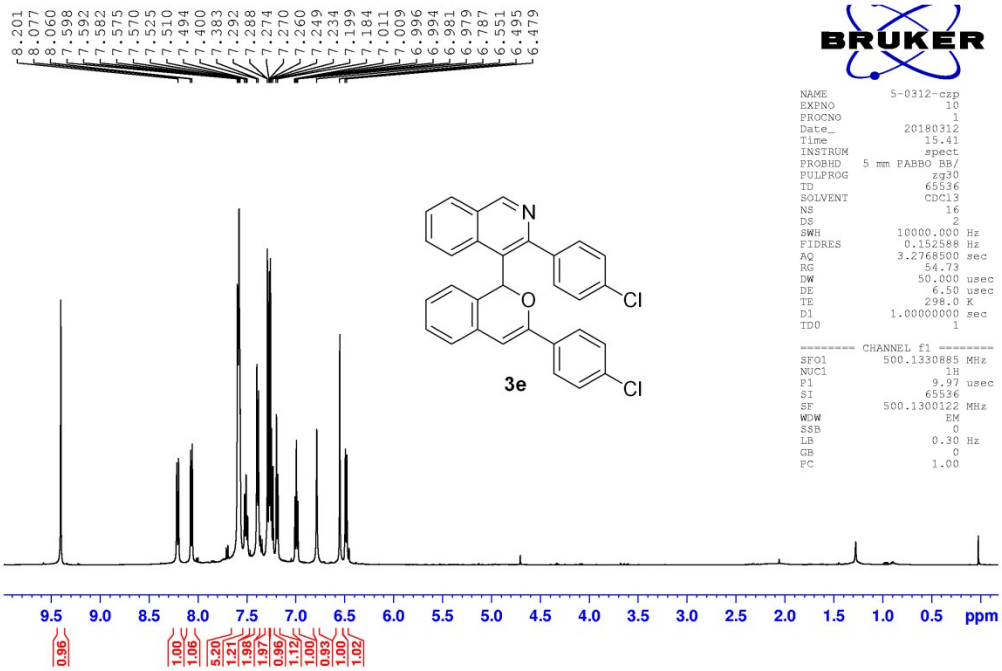


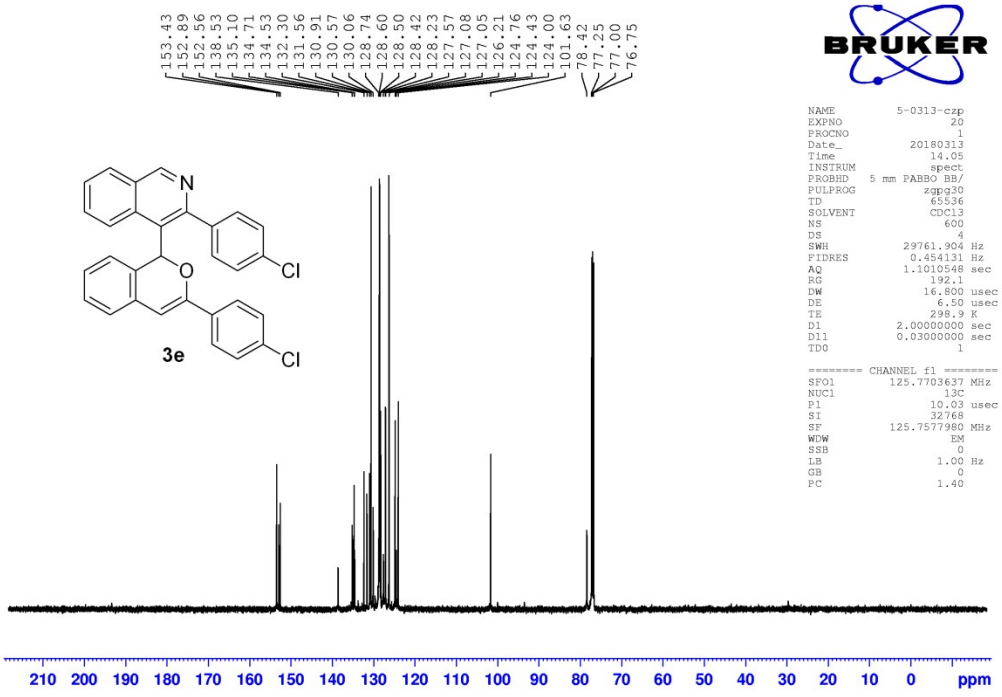
3d

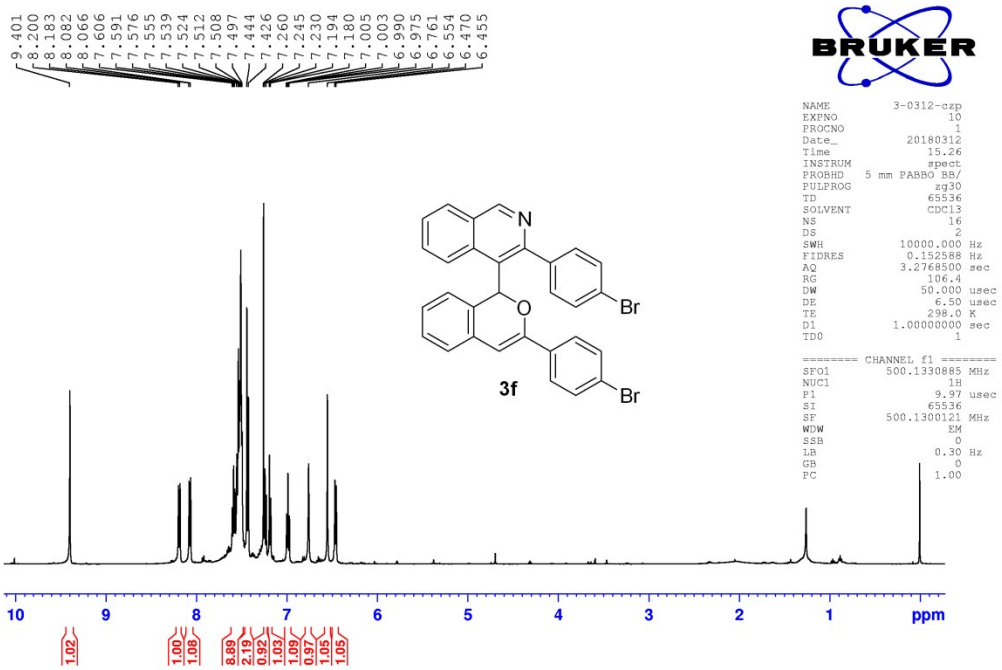
BRUKER

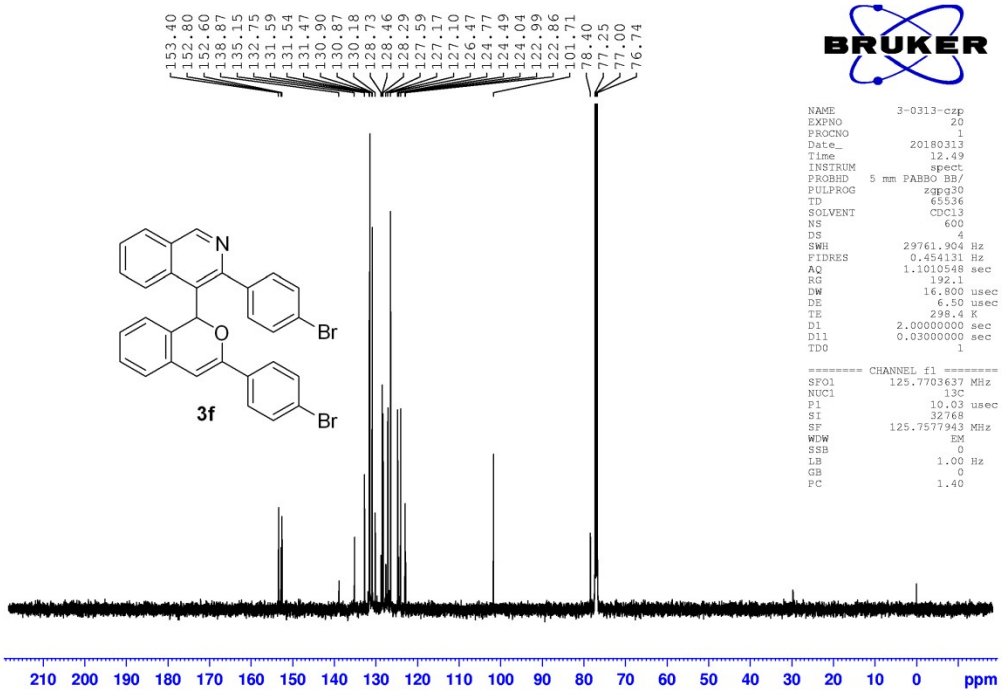
NAME	6-0314-czp
EXPNO	20
PROCNO	1
Date_	20180314
Time	12.20
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	300
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	192.1
DW	16.800 usec
DE	6.50 usec
TE	299.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	125.7703637 MHz
NUC1	13C
P1	10.03 usec
SI	32768
SF	125.7577950 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

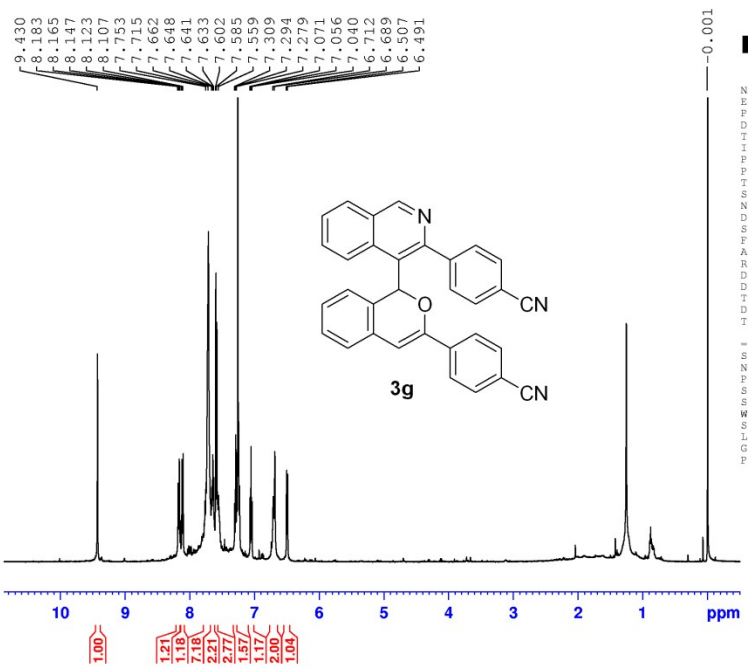



8.201
8.077
8.060
7.598
7.592
7.582
7.575
7.570
7.525
7.510
7.494
7.400
7.383
7.292
7.288
7.274
7.270
7.260
7.249
7.234
7.199
7.184
7.011
7.009
6.996
6.994
6.981
6.979
6.787
6.551
6.495
6.479
N
O
Cl
Cl
3e
BRUKER
NAME 5-0312-czp
EXPNO 10
PROCNO 1
Date_ 20180312
Time 15.41
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 54.73
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.97 usec
SI 65536
SF 500.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.96
1.00
1.06
5.20
1.21
1.98
1.97
0.96
1.12
1.00
0.93
1.00
1.02


153.43
152.89
152.56
138.53
135.10
134.71
134.53
132.30
131.56
130.91
130.57
130.06
128.74
128.60
128.50
128.42
128.23
127.57
127.08
127.05
126.21
124.76
124.43
124.00
101.63
78.42
77.25
77.00
76.75
N
O
Cl
Cl
3e
BRUKER
NAME 5-0313-czp
EXPNO 20
PROCNO 1
Date_ 20180313
Time 14.05
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 600
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 298.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.03 usec
SI 32768
SF 125.7577980 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

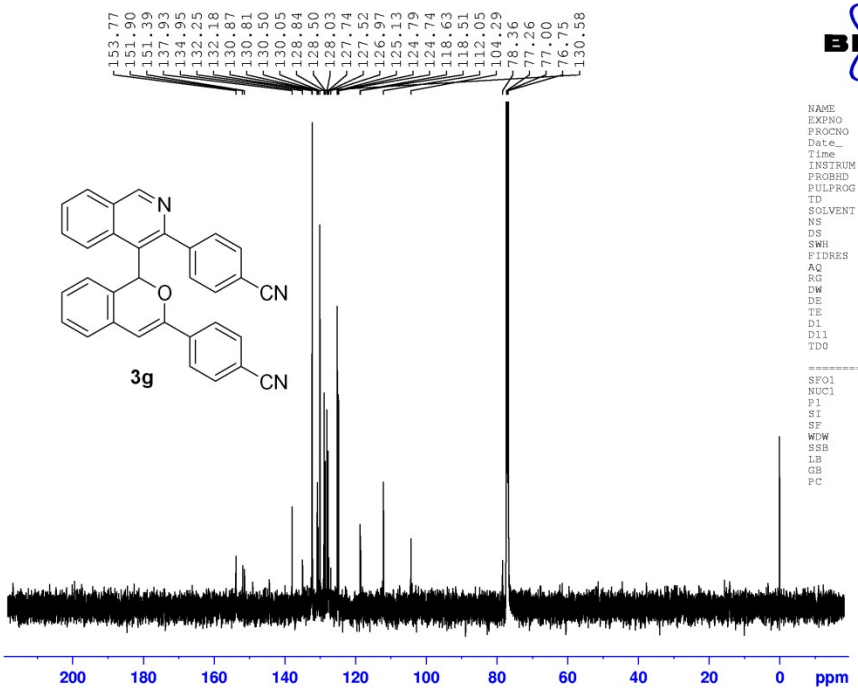

9.401
8.200
8.183
8.082
8.066
7.606
7.591
7.576
7.555
7.539
7.524
7.512
7.508
7.497
7.444
7.426
7.260
7.245
7.230
7.194
7.180
7.005
7.003
6.990
6.975
6.761
6.554
6.470
6.455
BRUKER
NAME 3-0312-czp
EXPNO 10
PROCNO 1
Date_ 20180312
Time 15.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 106.4
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.97 usec
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
O
Br
Br
3f
10
9
8
7
6
5
4
3
2
1
ppm
1.02
1.00
1.08
8.89
2.19
0.92
1.03
1.09
0.97
1.05
1.05


153.40
152.80
152.60
138.87
135.15
132.75
131.59
131.54
131.47
130.90
130.87
130.18
128.73
128.46
128.29
127.59
127.17
127.10
126.47
124.77
124.49
124.04
122.99
122.86
101.71
78.40
77.25
77.00
76.74
BRUKER
NAME 3-0313-czp
EXPNO 20
PROCNO 1
Date_ 20180313
Time 12.49
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 600
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 298.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.03 usec
SI 32768
SF 125.7577943 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
O
Br
Br
3f
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm



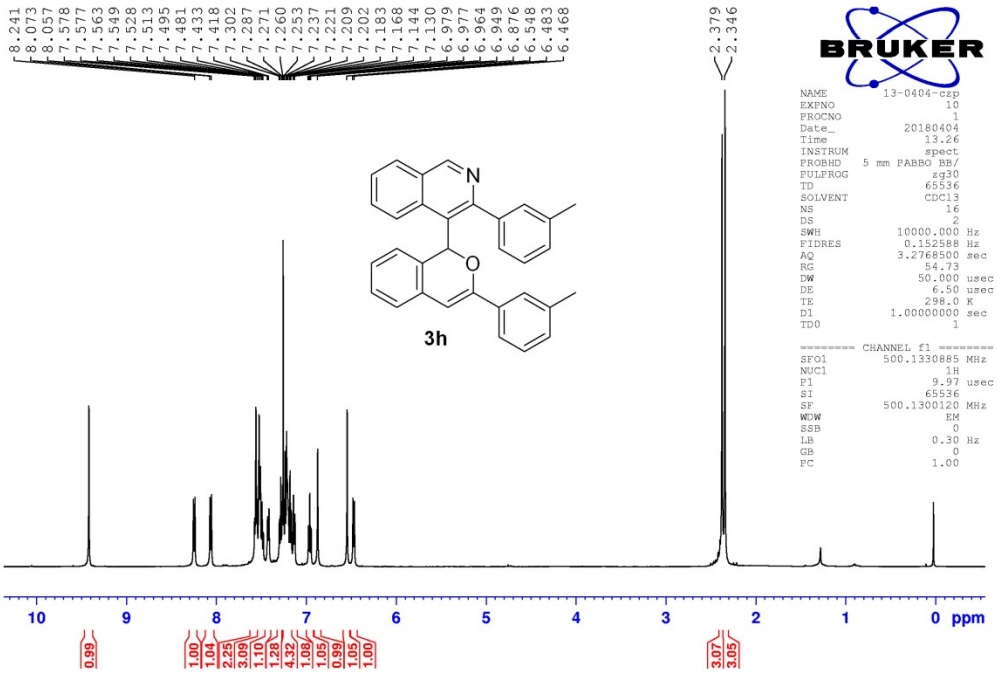
BRUKER

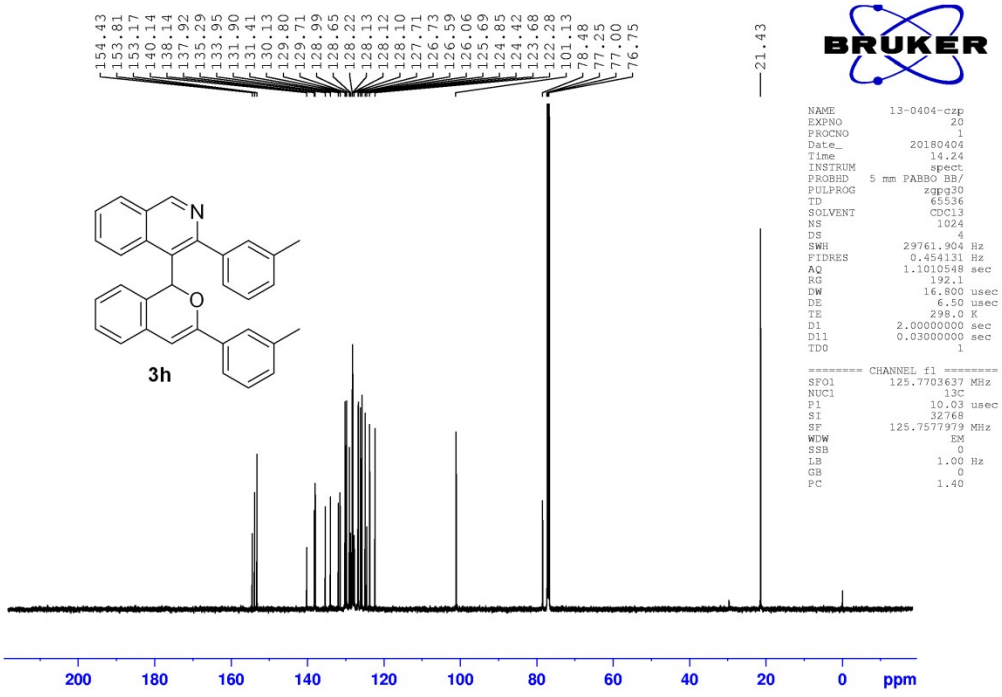
```
NAME                30-0330-CZP
EXPNO                        10
PROCNO                        1
Date_               20180330
Time                     12.01
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                 10000.000 Hz
FIDRES                  0.152588 Hz
AQ                  3.2768500 sec
RG                      130.07
DW                      50.000 usec
DE                       6.50 usec
TE                      298.0 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1             500.1330885 MHz
NUC1                       1H
P1                       9.97 usec
SI                      65536
SF               500.1300124 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```

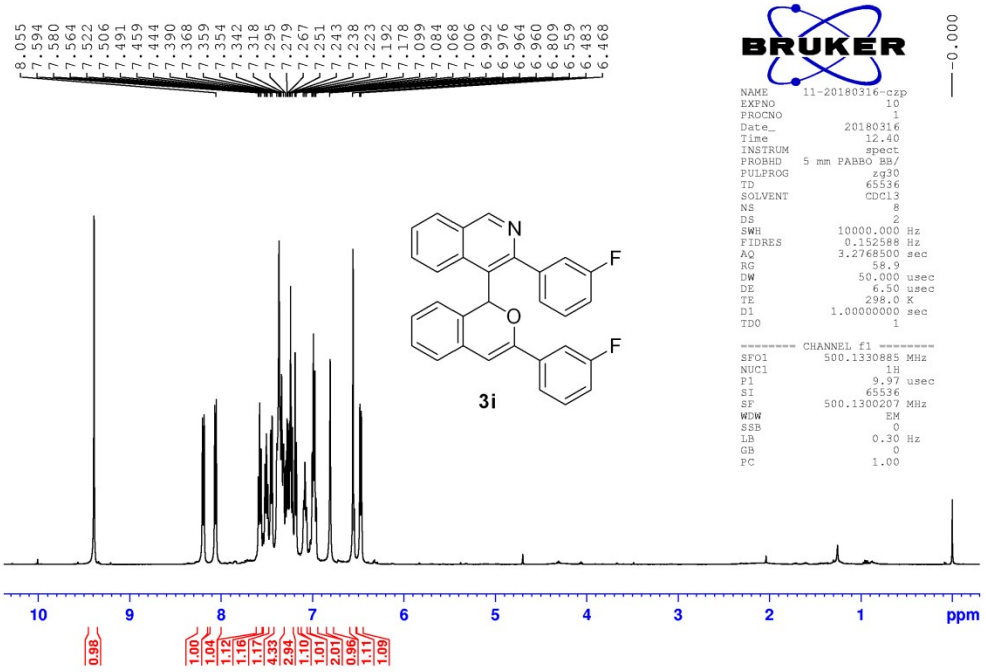


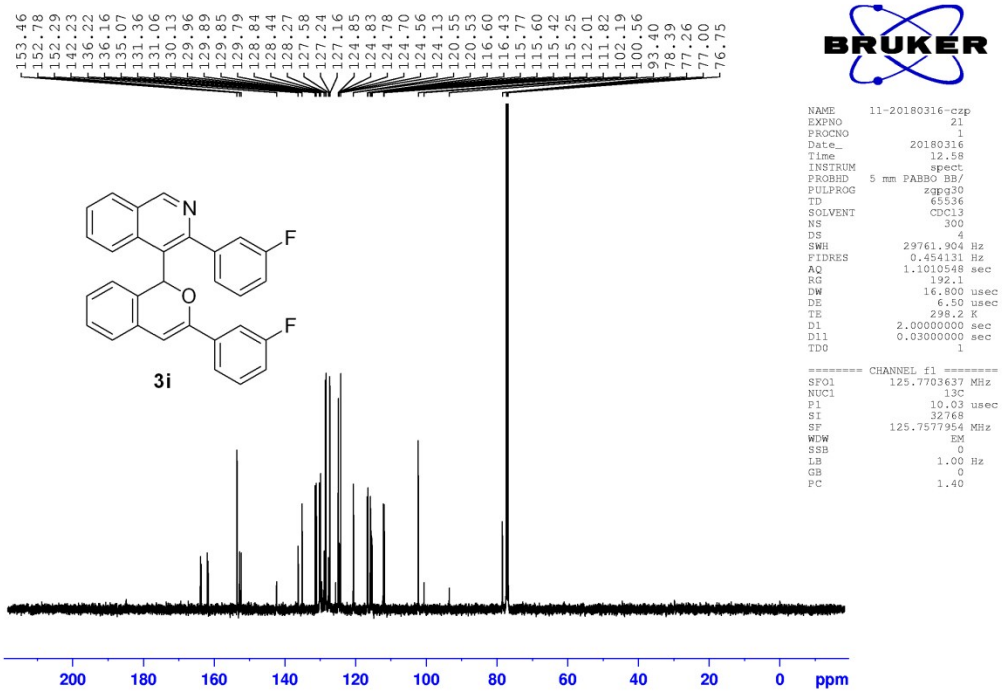
BRUKER

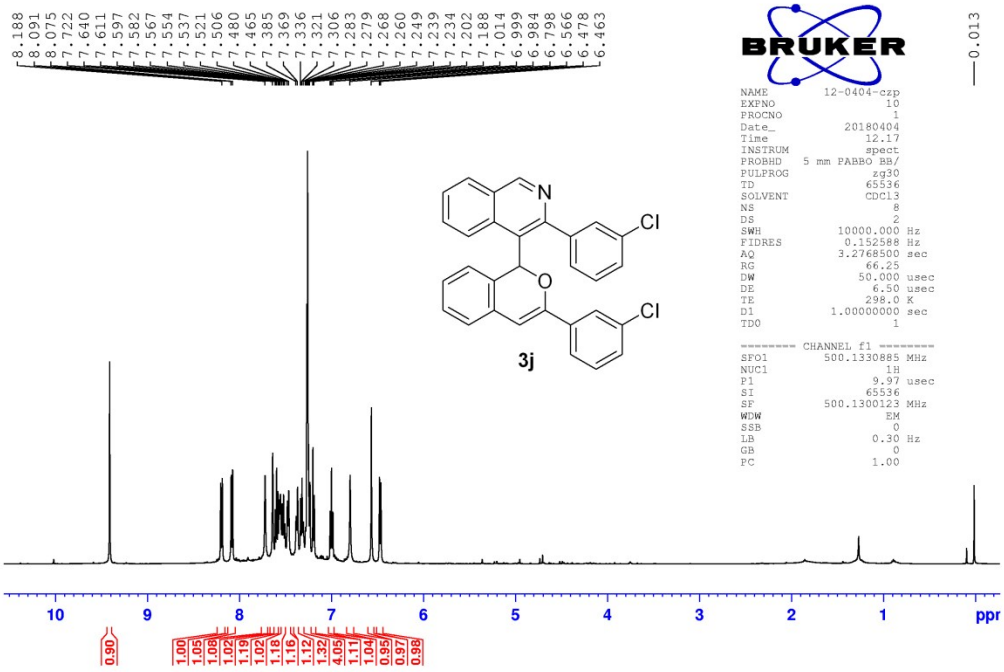
```
NAME                30-0409-czp
EXPNO                        20
PROCNO                        1
Date_               20180409
Time                     15.40
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        2000
DS                           4
SWH                 29761.904 Hz
FIDRES                  0.454131 Hz
AQ                  1.1010548 sec
RG                       192.1
DW                      16.800 usec
DE                       6.50 usec
TE                      298.8 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1            125.7703637 MHz
NUC1                       13C
P1                      10.03 usec
SI                       32768
SF               125.7577916 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```

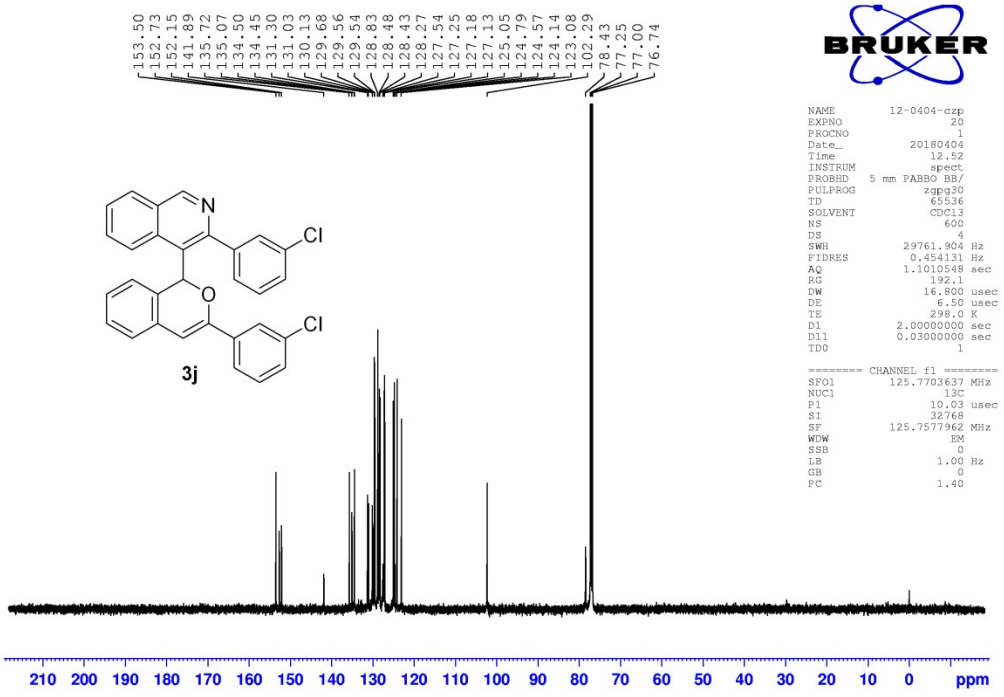

8.241
8.073
8.057
7.578
7.577
7.563
7.549
7.528
7.513
7.495
7.481
7.433
7.418
7.302
7.287
7.271
7.260
7.253
7.237
7.221
7.209
7.202
7.183
7.168
7.144
7.130
6.979
6.977
6.964
6.949
6.876
6.548
6.483
6.468
2.379
2.346
N
O
3h
BRUKER
NAME 13-0404-czp
EXPNO 10
PROCNO 1
Date_ 20180404
Time 13.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 54.73
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.97 usec
SI 65536
SF 500.1300120 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 0 ppm
0.99
1.00 1.04 2.25 3.09 1.10 1.28 4.32 1.08 1.05 0.99 1.05 1.00
3.07 3.05

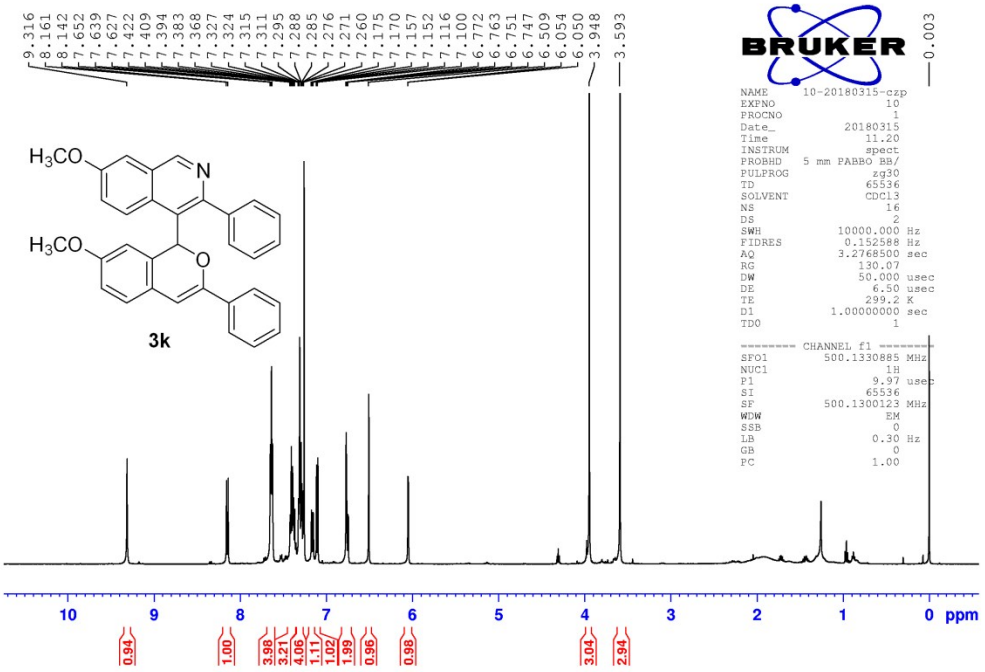

154.43
153.81
153.17
140.14
138.14
137.92
135.29
133.95
131.90
131.41
130.13
129.80
129.71
128.99
128.65
128.22
128.13
128.12
128.10
127.71
126.73
126.59
126.06
125.69
124.85
124.42
123.68
122.28
101.13
78.48
77.25
77.00
76.75
21.43
N
O
3h
BRUKER
NAME 13-0404-czp
EXPNO 20
PROCNO 1
Date_ 20180404
Time 14.24
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.03 usec
SI 32768
SF 125.7577979 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm

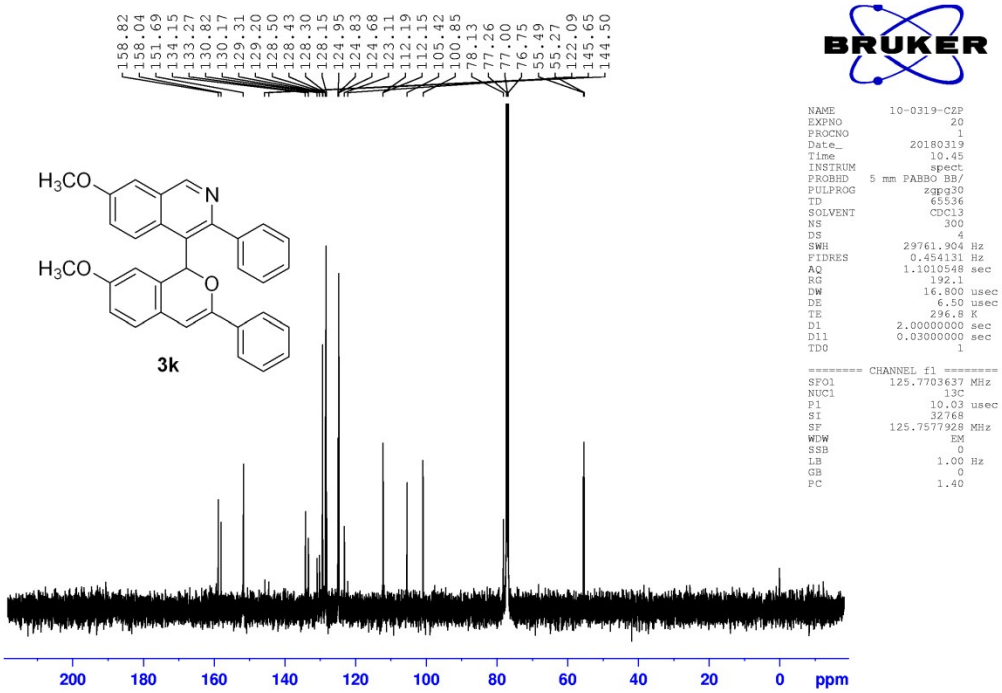

8.055
7.594
7.580
7.564
7.522
7.506
7.491
7.459
7.444
7.390
7.368
7.359
7.354
7.342
7.318
7.295
7.279
7.267
7.251
7.243
7.238
7.223
7.192
7.178
7.099
7.084
7.068
7.006
6.992
6.976
6.964
6.960
6.809
6.559
6.483
6.468
0.000
BRUKER
NAME 11-20180316-czp
EXPNO 10
PROCNO 1
Date_ 20180316
Time 12.40
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 58.9
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.97 usec
SI 65536
SF 500.1300207 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
F
O
F
3i
10
9
8
7
6
5
4
3
2
1
ppm
0.98
1.00
1.04
1.12
1.16
1.17
4.33
2.94
1.10
1.01
2.01
0.96
1.11
1.09

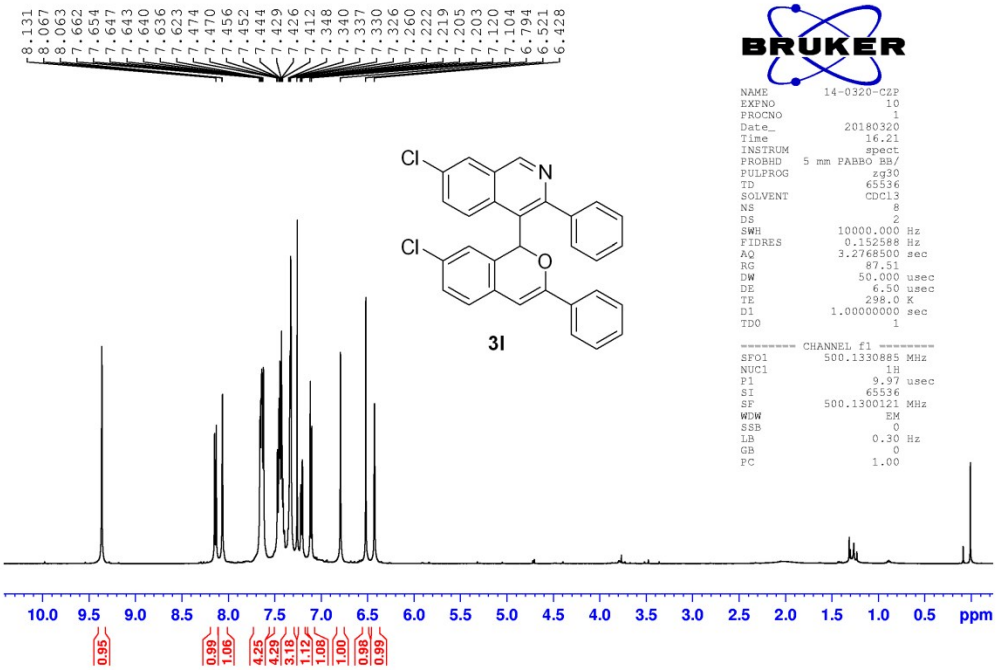

153.46
152.78
152.29
142.23
136.22
136.16
135.07
131.36
131.06
130.13
129.96
129.89
129.85
129.79
128.84
128.44
128.27
127.58
127.24
127.16
124.85
124.83
124.78
124.70
124.56
124.13
120.55
120.53
116.60
116.43
115.77
115.60
115.42
115.25
112.01
111.82
102.19
100.56
93.40
78.39
77.26
77.00
76.75
BRUKER
NAME 11-20180316-czp
EXPNO 21
PROCNO 1
Date_ 20180316
Time 12.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.03 usec
SI 32768
SF 125.7577954 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
F
O
F
3i
200
180
160
140
120
100
80
60
40
20
0
ppm

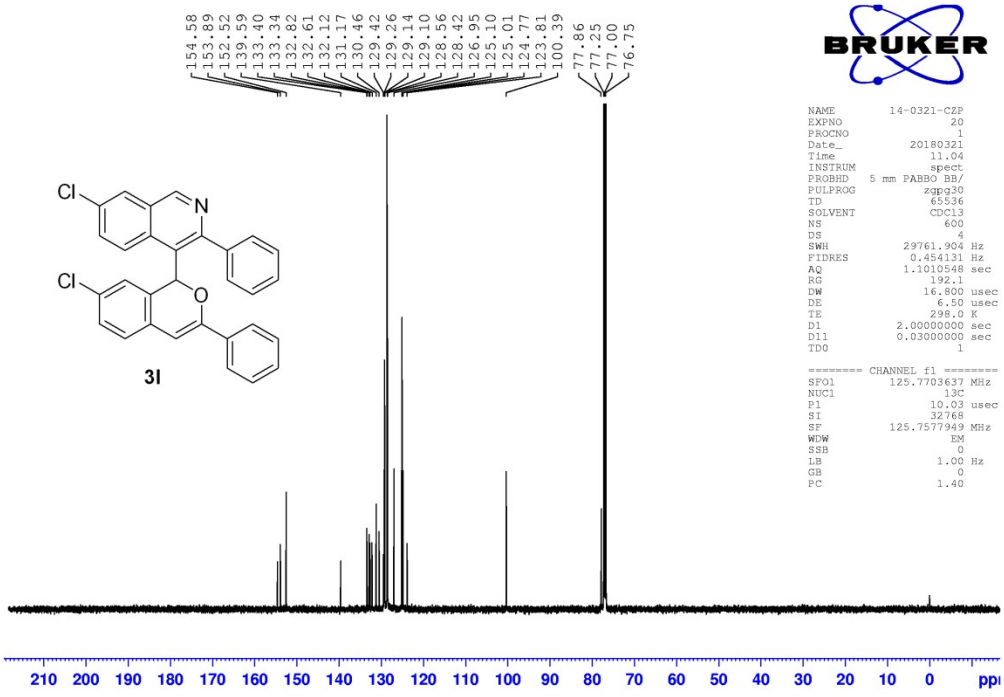

8.188
8.091
8.075
7.722
7.640
7.611
7.597
7.582
7.567
7.554
7.537
7.521
7.506
7.480
7.465
7.385
7.369
7.336
7.321
7.306
7.283
7.279
7.268
7.260
7.249
7.239
7.234
7.202
7.188
7.014
6.999
6.984
6.798
6.566
6.478
6.463
BRUKER
0.013
NAME 12-0404-czp
EXPNO 10
PROCNO 1
Date_ 20180404
Time 12.17
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 66.25
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.97 usec
SI 65536
SF 500.1300123 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
Cl
O
Cl
3j
10
9
8
7
6
5
4
3
2
1
ppm
0.90
1.00
1.05
1.08
1.02
1.19
1.02
1.18
1.16
1.12
1.32
4.05
1.11
1.04
0.95
0.97
0.98

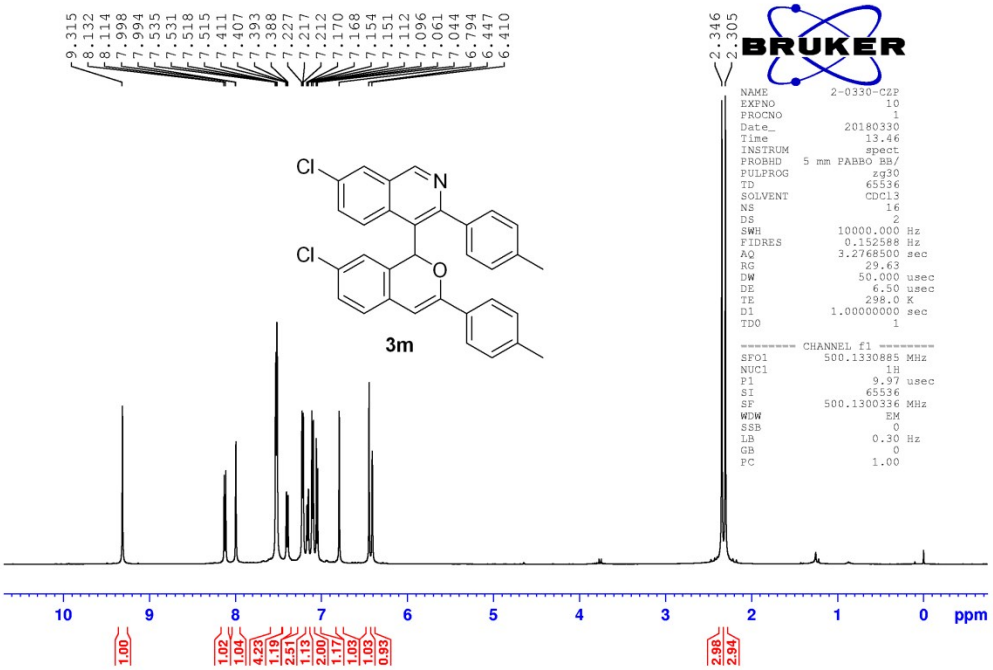

153.50
152.73
152.15
141.89
135.72
135.07
134.50
134.45
131.30
131.03
130.13
129.68
129.56
129.54
128.83
128.48
128.43
128.27
127.54
127.25
127.18
127.13
125.05
124.79
124.57
124.14
123.08
102.29
78.43
77.25
77.00
76.74
BRUKER
NAME 12-0404-czp
EXPNO 20
PROCNO 1
Date_ 20180404
Time 12.52
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 600
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.03 usec
SI 32768
SF 125.7577962 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
Cl
O
Cl
3j
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

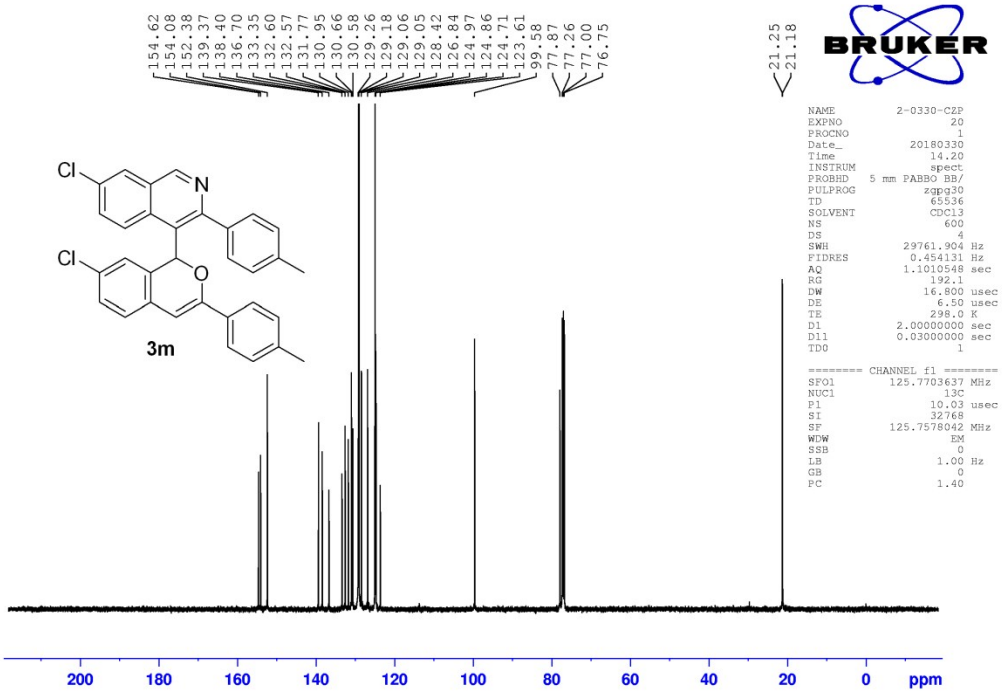

9.316
8.161
8.142
7.652
7.639
7.627
7.422
7.409
7.394
7.383
7.368
7.327
7.324
7.315
7.311
7.295
7.288
7.285
7.276
7.271
7.260
7.175
7.170
7.157
7.152
7.116
7.100
6.772
6.763
6.751
6.747
6.509
6.054
6.050
3.948
3.593
0.003
H3CO
H3CO
N
O
3k
NAME 10-20180315-czp
EXPNO 10
PROCNO 1
Date_ 20180315
Time 11.20
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 130.07
DW 50.000 usec
DE 6.50 usec
TE 299.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.97 usec
SI 65536
SF 500.1300123 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0 ppm
0.94
1.00
3.98
3.21
4.06
1.11
1.02
1.99
0.96
0.98
3.04
2.94

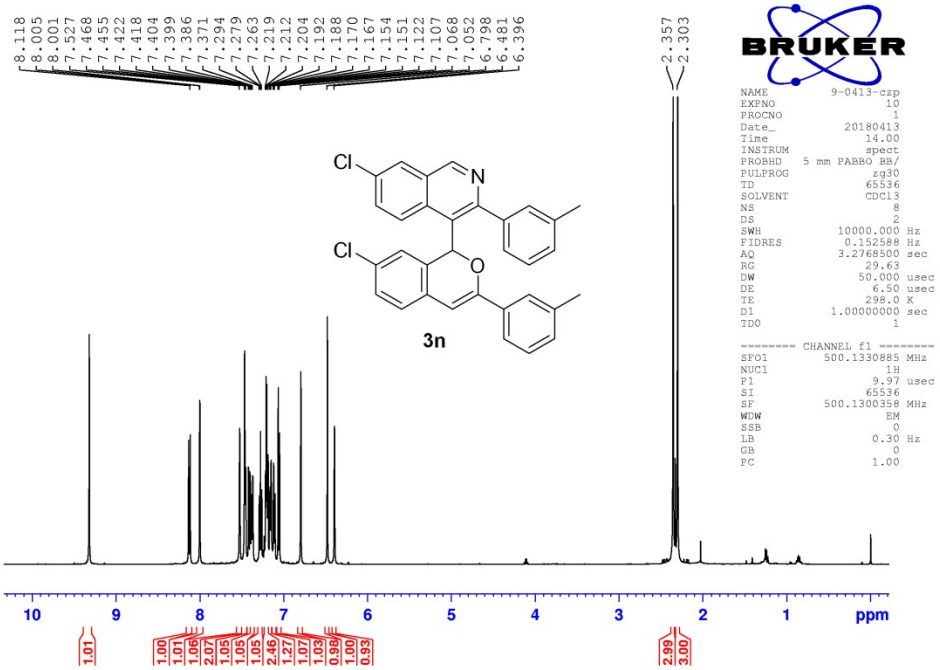

158.82
158.04
151.69
134.15
133.27
130.82
130.17
129.31
129.20
128.50
128.43
128.30
128.15
124.95
124.83
124.68
123.11
112.19
112.15
105.42
100.85
78.13
77.26
77.00
76.75
55.49
55.27
122.09
145.65
144.50
H3CO
H3CO
N
O
3k
NAME 10-0319-CZP
EXPNO 20
PROCNO 1
Date_ 20180319
Time 10.45
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 296.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.03 usec
SI 32768
SF 125.7577928 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0 ppm


8.131
8.067
8.063
7.662
7.654
7.647
7.643
7.640
7.636
7.623
7.474
7.470
7.456
7.452
7.444
7.429
7.426
7.412
7.348
7.340
7.337
7.330
7.326
7.260
7.222
7.219
7.205
7.203
7.120
7.104
6.794
6.521
6.428
Cl
N
Cl
O
3l
BRUKER
NAME 14-0320-CZP
EXPNO 10
PROCNO 1
Date_ 20180320
Time 16.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 87.51
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.97 usec
SI 65536
SF 500.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.95
0.99
1.06
4.25
4.29
3.18
1.12
1.08
1.00
0.98
0.99


154.58
153.89
152.52
139.59
133.40
133.34
132.82
132.61
132.12
131.17
130.46
129.42
129.26
129.14
129.10
128.56
128.42
126.95
125.10
125.01
124.77
123.81
100.39
77.86
77.25
77.00
76.75
Cl
N
Cl
O
3l
BRUKER
NAME 14-0321-CZP
EXPNO 20
PROCNO 1
Date_ 20180321
Time 11.04
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 600
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.03 usec
SI 32768
SF 125.7577949 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppi

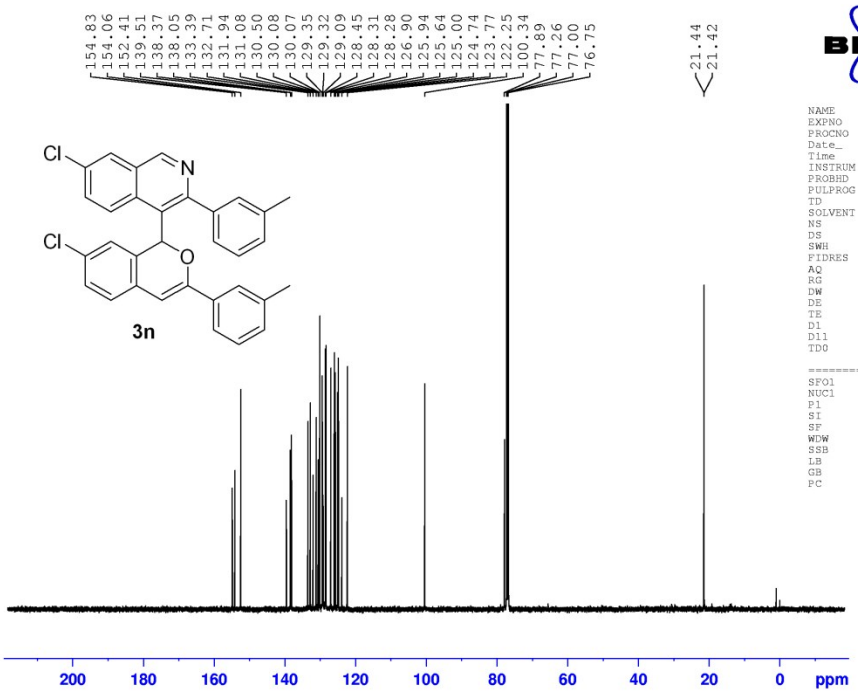

9.315
8.132
8.114
7.998
7.994
7.535
7.531
7.518
7.515
7.411
7.407
7.393
7.388
7.227
7.217
7.212
7.170
7.168
7.154
7.151
7.112
7.096
7.061
7.044
6.794
6.447
6.410
2.346
2.305
Cl
Cl
N
O
3m
BRUKER
NAME 2-0330-CZP
EXPNO 10
PROCNO 1
Date_ 20180330
Time 13.46
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 29.63
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.97 usec
SI 65536
SF 500.1300336 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.02
1.04
4.23
1.19
2.51
1.13
2.00
1.17
1.02
1.03
0.93
2.98
2.94


154.62
154.08
152.38
139.37
138.40
136.70
133.35
132.60
132.57
131.77
130.95
130.66
130.58
129.26
129.18
129.06
129.05
128.42
126.84
124.97
124.86
124.71
123.61
99.58
77.87
77.26
77.00
76.75
21.25
21.18
Cl
Cl
N
O
3m
BRUKER
NAME 2-0330-CZP
EXPNO 20
PROCNO 1
Date_ 20180330
Time 14.20
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 600
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.03 usec
SI 32768
SF 125.7578042 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm



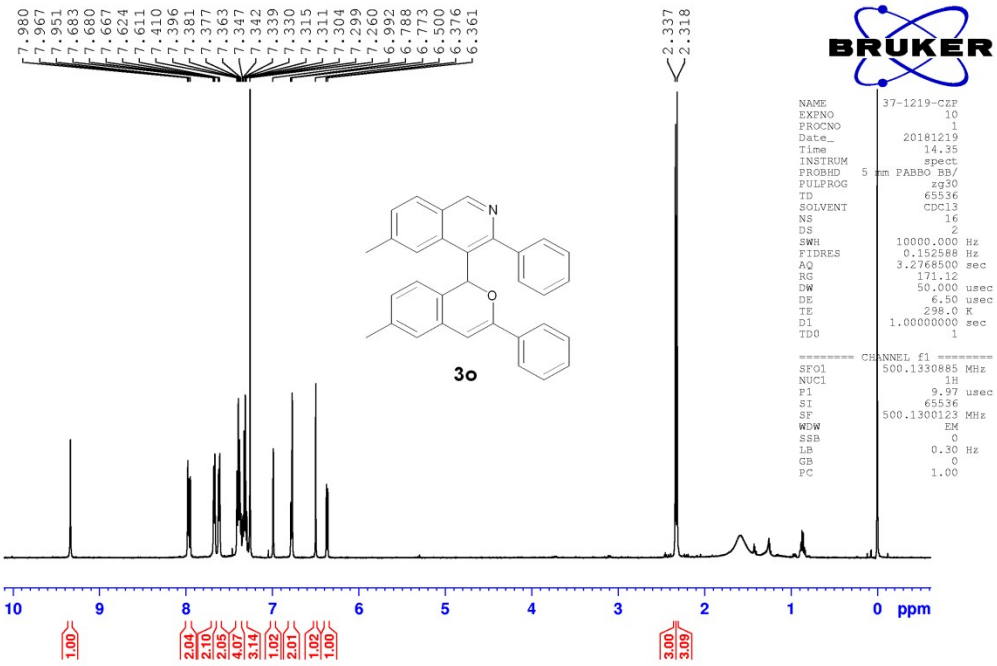
BRUKER

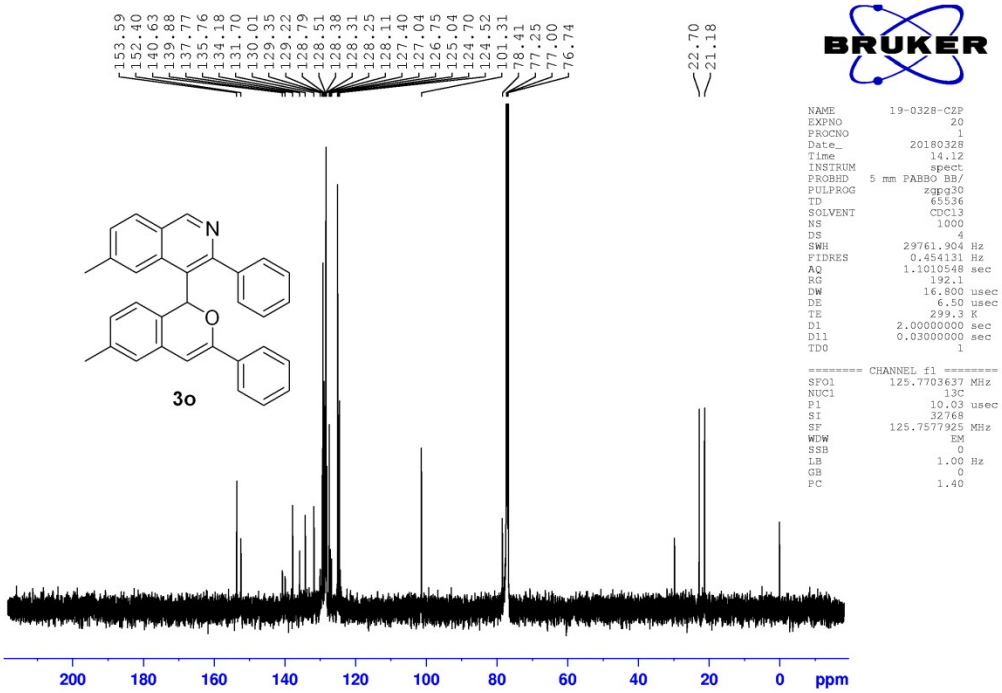
NAME	9-0413-czp
EXPNO	10
PROCNO	1
Date_	20180413
Time	14.00
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	29.63
DW	50.000 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	500.1330885 MHz
NUC1	1H
P1	9.97 usec
SI	65536
SF	500.1300358 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

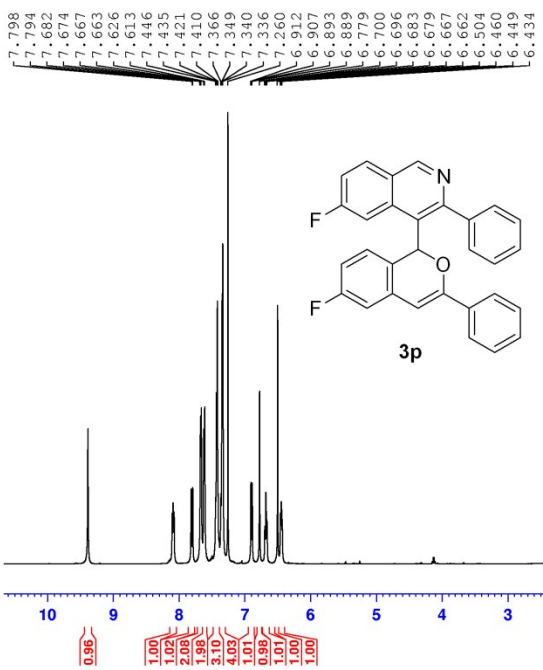


BRUKER

NAME	11-0423-czp
EXPNO	20
PROCNO	1
Date_	20180423
Time	14.40
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	192.1
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	125.7703637 MHz
NUC1	13C
P1	10.03 usec
SI	32768
SF	125.7577982 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

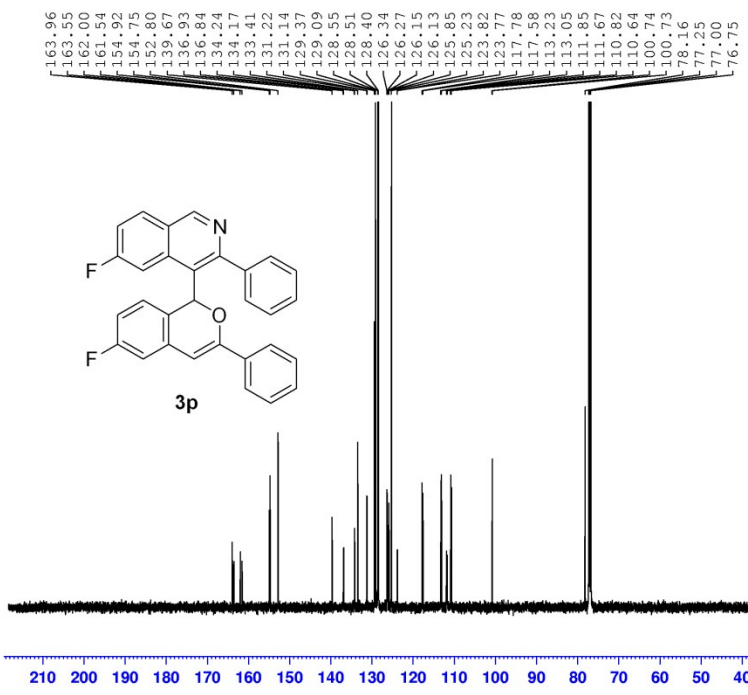

7.980
7.967
7.951
7.683
7.680
7.667
7.624
7.611
7.410
7.396
7.381
7.377
7.363
7.347
7.342
7.339
7.330
7.315
7.311
7.304
7.299
7.260
6.992
6.788
6.773
6.500
6.376
6.361
2.337
2.318
N
O
3o
NAME 37-1219-CZP
EXPNO 10
PROCNO 1
Date_ 20181219
Time 14.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 171.12
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 9.97 usec
SI 65536
SF 500.1300123 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
2.04
2.10
2.05
4.07
3.14
1.02
2.01
1.02
1.00
3.00
3.09


153.59
152.40
140.63
139.88
137.77
135.76
134.18
131.70
130.01
129.35
129.22
128.79
128.51
128.38
128.31
128.25
128.11
127.40
127.04
126.75
125.04
124.70
124.52
101.31
78.41
77.25
77.00
76.74
22.70
21.18
N
O
3o
NAME 19-0328-CZP
EXPNO 20
PROCNO 1
Date_ 20180328
Time 14.12
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1000
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 192.1
DW 16.800 usec
DE 6.50 usec
TE 299.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 10.03 usec
SI 32768
SF 125.7577925 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm



BRUKER

NAME	18-20180321-CZP
EXPNO	10
PROCNO	1
Date_	20180321
Time	12.28
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	78.29
DW	50.000 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	500.1330885 MHz
NUC1	1H
P1	9.97 usec
SI	65536
SF	500.1300122 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



BRUKER

NAME	18-20180321-CZP
EXPNO	21
PROCNO	1
Date_	20180321
Time	13.03
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	600
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	192.1
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	125.7703637 MHz
NUC1	13C
P1	10.03 usec
SI	32768
SF	125.7577953 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40