Electronic Supplementary Material (ESI) for Chemical Communications.

3-Homoacyl coumarin: an all carbon 1,3-dipole for the enantioselective concerted (3+2) cycloaddition

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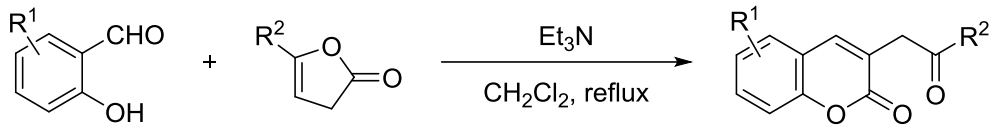
1. General aspects and materials

All reactions were carried out in oven-dried glassware with magnetic stirring. Unless otherwise stated, all reagents were used as purchased from commercial suppliers without further purification. Analytical thin layer chromatography (TLC) was performed on pre-coated, alumina-backed silica gel plates (Merck 60 F_{254}, 0.2 mm thickness) which were developed using UV irradiation at 254 nm. Flash column chromatography was performed using silica gel (Silicycle SiliaFlash P60, 230-400 mesh). Melting points were measured on a Fargo melting point apparatus and are uncorrected. IR spectra were recorded on a Perkin Elmer 500 spectrometer and only selected peaks are mentioned. ^{1}H NMR spectra were recorded on either a Bruker AV-400 spectrometer or a Bruker AV-III HD-400 spectrometer. Chemical shifts are reported in δ ppm referenced to an internal TMS standard (δ = 0.0 ppm) for ^{1}H NMR and chloroform-d (δ = 77.00 ppm) for ^{13}C NMR. The following abbreviations (or combinations thereof) were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublet, td = triplet of doublet, qd = quartet of doublet, br = broad, p = pseudo. High resolution mass spectra were recorded on JEOL MStation JMS-700 (2) using EI (Magnetic sector analyzer) or on Waters Xevo G2-S Tof using ESI (TOF analyzer). The X-ray diffraction measurements were carried out at 200 K on a Bruker KAPPA APEX II CCD area detector system equipped with a graphite monochromator and either a Mo-Kα fine-focus sealed tube (k = 0.71073 Å). Optical rotations were measured in CH_2Cl_2 on a Polari meter with a 50 mm cell (c given in g/100 mL) operating at λ = 589 nm, corresponding to the sodium D line, at the indicated temperatures.

The known 2-arylidene indandione substrates **2a**, **2b**, **2c**, **2e**, **2f**, **2g**, **2i**, **2j**, **2l**, **2m** and **2n** were synthesized following the procedure reported earlier by our group[1] whereas **2d** and **2k** were synthesized following the procedures reported by Ren[2] and Huang[3] groups respectively. Coumarin derivatives were prepared following the procedure described in this supporting information.

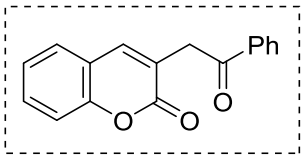
2. Experimental procedures and characterization data for new compounds

a. General procedure-A for the preparation of 3-homoacylcoumarin derivatives 1



A 100 mL round-bottomed flask equipped with a magnetic stir bar was charged with lactone derivative (1.0 mmol), substituted salicylaldehyde (1.1 equiv.), Et_3N (1.0 mL) and CH_2Cl_2 (30.0 mL) and the resulting mixture was refluxed for 1 h. Afterwards, the reaction mixture was diluted with CH_2Cl_2 and washed with 1 M $HCl_{(aq)}$ and then brine. The organic layer was then dried over anhydrous $MgSO_4$ and concentrated under reduced pressure to give a crude residue which was subjected to flash column chromatography over silica gel with EtOAc/hexanes (1/2) as eluent to yield the desired product **1**.

3-(2-oxo-2-phenylethyl)-2*H*-chromen-2-one (1a)[4]



Following the general procedure-A, **1a** was obtained as a white solid (72% yield, 190.3 mg).

R_f 0.36 (EtOAc/hexanes = 1/5); mp.: 169.9-171.4 °C.

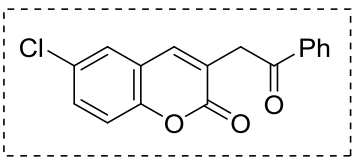
^{1}H NMR (400 MHz, $CDCl_3$) δ/ppm: 8.04 (d, J = 7.9 Hz, 2H), 7.67 (s, 1H), 7.58 (pt, J = 7.5 Hz, 1H), 3.40-3.52 (m, 4H), 7.31 (d, J = 8.3 Hz, 1H), 7.25 (pt, J = 7.6 Hz, 1H), 4.24 (s, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ/ppm: 195.9, 161.4, 153.4, 141.9, 136.3, 133.4, 131.1, 128.7, 128.3, 127.5, 124.4, 123.1, 119.2, 116.5, 39.5.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 1717, 1687, 1605, 1338, 1213, 1072, 757.

HRMS (ESI) calcd for $C_{17}H_{13}O_3$, $[M+H]^+$ 265.0859, found: 265.0867.

6-chloro-3-(2-oxo-2-phenylethyl)-2*H*-chromen-2-one (1b)



Following the general procedure-A, **1b** was obtained as a yellow solid (62% yield, 185.2 mg).

R_f 0.44 (EtOAc/hexanes = 1/5); mp.: 197.0-197.8 °C.

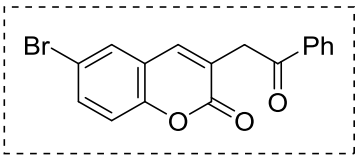
^{1}H NMR (400 MHz, $CDCl_3$) δ/ppm: 8.05 (d, J = 7.4 Hz, 2H), 7.57-7.65 (m, 2H), 7.50 (pt, J = 7.8 Hz, 2H), 7.42-7.47 (m, 2H), 7.27-7.30 (m 1H), 4.26, (s, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ/ppm: 195.6, 160.9, 151.9, 140.7, 136.2, 133.7, 131.2, 129.7, 128.8, 128.4, 126.8, 124.5, 120.3, 118.0, 39.5.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 1719, 1682, 1607, 1587, 755.

HRMS (ESI) calcd for $C_{17}H_{12}O_3{}^{35}Cl$, $[M+H]^+$ 299.0469, found: 299.0476; calcd for $C_{17}H_{12}O_3{}^{37}Cl$, $[M+H]^+$ 301.0440, found: 301.0452.

6-bromo-3-(2-oxo-2-phenylethyl)-2*H*-chromen-2-one (1c)



Following the general procedure-A, **1c** was obtained as a white solid (58% yield, 199.0 mg).

R_f 0.44 (EtOAc/hexanes = 1/5); mp.: 216.5-217.8 °C.

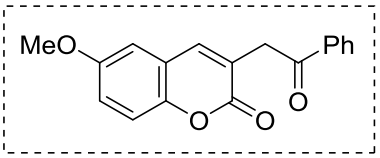
^{1}H NMR (400 MHz, $CDCl_3$) δ/ppm: 8.05 (d, J = 7.5 Hz, 2H,), 7.56-7.65 (m, 4H), 7.50 (pt, J =7.7 Hz, 2H), 7.23 (d, J = 8.5 Hz, 1H), 4.26 (s 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ/ppm: 195.6, 160.8, 152.3, 140.6, 136.2, 134.0, 133.7, 129.8, 128.8, 128.4, 124.5, 120.8, 118.3, 117.0, 39.5.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 1728, 1676, 1476, 1329, 1217, 1068, 821, 757.

HRMS (ESI) calcd for $C_{17}H_{12}O_3{}^{79}Br$, $[M+H]^+$ 342.9964, found: 342.9970; calcd for $C_{17}H_{12}O_3{}^{81}Br$, $[M+H]^+$ 344.9944, found: 344.9951.

6-methoxy-3-(2-oxo-2-phenylethyl)-2*H*-chromen-2-one (1d)



Following the general procedure-A, **1d** was obtained as an orange solid (56% yield, 164.8 mg).

R_f 0.21 (EtOAc/hexanes = 1/5); mp.: 174.5-175.5 °C.

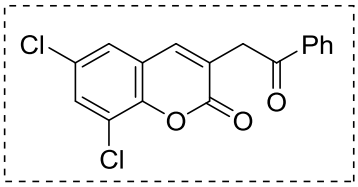
^{1}H NMR (400 MHz, $CDCl_3$) δ/ppm: 7.96-8.01 (m, 2H), 7.57 (s, 1H), 7.53 (tt, J = 7.5, 1.3 Hz, 1H), 7.42 (pt, J = 8.4 Hz, 2H), 7.19 (d, J = 1.2 Hz, 1H), 7.01 (dd, J = 9.2, 3.1 Hz, 1H), 6.83 (d, J = 3.1 Hz, 1H), 4.18 (s, 2H), 3.77 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ/ppm: 196.0, 161.6, 156.1, 147.9, 141.8, 136.3, 133.5, 128.7, 128.4, 123.4, 119.6, 118.9, 117.5, 109.7, 55.8, 39.4.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 1684, 1578, 1496, 1284.

HRMS (ESI) calcd for $C_{18}H_{14}O_4Na$, $[M+Na]^+$ 317.0784, found: 317.0790.

6,8-dichloro-3-(2-oxo-2-phenylethyl)-2*H*-chromen-2-one (1e)



Following the general procedure-A, **1e** was obtained as a pale yellow solid (49% yield, 163.2 mg).

R_f 0.39 (EtOAc/hexanes = 1/5); mp.: 210.3-211.7 °C.

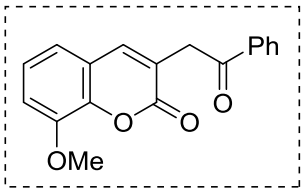
^{1}H NMR (400 MHz, $CDCl_3$) δ/ppm: 8.02-8.06 (m, 2H), 7.59-7.66 (m, 2H), 7.56 (d, J = 2.4 Hz, 1H), 7.51 (t, J =7.7 Hz, 2H), 7.37 (d, J = 2.3 Hz, 1H), 4.28 (d, J = 0.9 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ/ppm: 195.2, 159.9, 147.9, 140.4, 136.1, 133.8, 131.2, 129.6, 128.8, 128.4, 125.40, 125.37, 122.5, 121.1, 39.4.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 1735, 1677, 1595, 1447, 759.

HRMS (ESI) calcd for $C_{17}H_{11}O_3{}^{35}Cl_2$, $[M+H]^+$ 333.0080, found: 333.0085; calcd for $C_{17}H_{11}O_3{}^{35}Cl^{37}Cl$, $[M+H]^+$ 335.0050, found: 335.0059.

8-methoxy-3-(2-oxo-2-phenylethyl)-2*H*-chromen-2-one (1f)



Following the general procedure-A, **1f** was obtained as a pale yellow solid (58% yield, 170.7 mg).

R_f 0.17 (EtOAc/hexanes = 1/5); mp.: 185.0-186.0 °C.

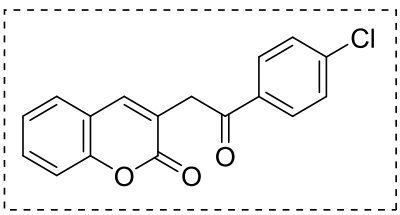
^{1}H NMR (400 MHz, $CDCl_3$) δ/ppm: 8.05 (dd, J = 8.3, 1.3 Hz, 2H,), 7.67 (s, 1H), 7.59 (pt, J = 7.4 Hz, 1H), 7.49 (pt, J = 7.6 Hz, 2H), 7.20 (pt, J = 8.0 Hz, 1H), 7.05 (td, J = 8.0, 1.3 Hz, 2H), 4.27 (s, 2H), 3.97 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ/ppm: 195.9, 161.0, 147.1, 143.2, 142.1, 136.3, 133.5, 128.7, 128.5, 124.3, 123.3, 119.9, 119.1, 113.3, 56.3, 39.4.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 1708, 1680, 1484, 1281.

HRMS (ESI) calcd for $C_{18}H_{15}O_4$, $[M+H]^+$ 295.0965, found: 295.0970.

3-(2-(4-chlorophenyl)-2-oxoethyl)-2*H*-chromen-2-one (1g)



Following the general procedure-A, **1g** was obtained as a yellow solid (54% yield, 161.3 mg).

R_f 0.32 (EtOAc/hexanes = 1/5); mp.: 170.7-171.5 °C.

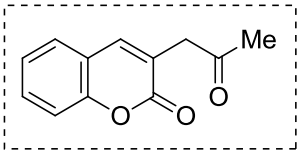
^{1}H NMR (400 MHz, $CDCl_3$) δ/ppm: 8.00 (d, J = 8.4 Hz, 2H,), 7.69 (s, 1H), 7.51 (pt, J = 8.2 Hz, 1H), 7.44-7.49 (m, 3H), 7.34 (d, J = 8.4 Hz, 1H), 7.28 (pt, J = 7.5 Hz, 1H), 4.22 (s, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ/ppm: 194.8, 161.5, 153.5, 142.1, 140.1, 134.6, 131.4, 129.9, 129.1, 127.6, 124.5, 122.8, 119.2, 116.6, 39.5.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 1715, 1693, 1399, 756.

HRMS (ESI) calcd for $C_{17}H_{12}O_3{}^{35}Cl$, $[M+H]^+$ 299.0469, found: 299.0476; calcd for $C_{17}H_{12}O_3{}^{37}Cl$, $[M+H]^+$ 301.0440, found: 301.0452.

3-(2-oxopropyl)-2*H*-chromen-2-one (1h)[5]



Following the general procedure-A, **1g** was obtained as an off-white solid (74% yield, 150.3 mg).

R_f 0.11 (EtOAc/hexanes = 1/5); mp.: 100.7-102.3 °C.

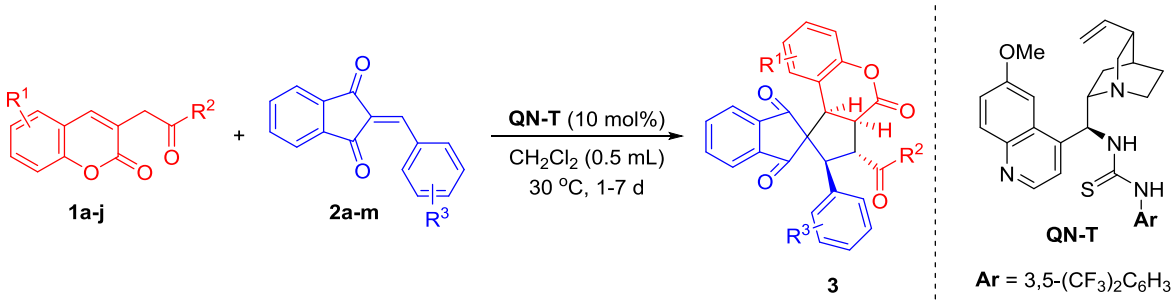
^{1}H NMR (400 MHz, $CDCl_3$) δ/ppm: 7.61 (s, 1H), 7.51 (ddd, J = 8.7, 7.0, 1.7 Hz, 1H), 7.46 (dd, J =7.9, 1.8 Hz, 1H), 7.34 (d, J = 8.2 Hz, 1H), 7.27 (td, J = 7.9, 1.3 Hz, 1H), 3.69 (s, 2H), 2.32 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ/ppm: 204.1, 161.5, 153.5, 141.9, 131.2, 127.5, 124.4, 122.9, 119.1, 116.6, 44.4, 30.2.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 1717, 1704, 1632, 1612, 1329, 755.

HRMS (ESI) calcd for $C_{12}H_{11}O_3$, $[M+H]^+$ 203.0703, found: 203.0707.

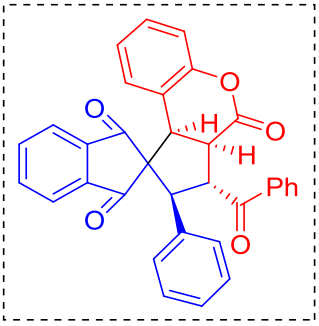
b. General procedure B for the preparation of cycloaddition products 3



A glass vial equipped with a magnetic stir bar was charged **1** (0.1 mmol), **2** (1.1 equiv.), **QN-T** (10 mol%) and CH_2Cl_2 (0.5 mL) and stirred at 30 °C for 1-7 days. After the completion of reaction, the reaction mixture was quenched with of 0.1 M $HCl_{(aq)}$ and the aqueous layer was extracted with CH_2Cl_2. Combined organic layers were dried over anhydrous $MgSO_4$ and concentrated in vacuo. The crude

residue was subjected to flash column chromatography over silica gel with EtOAc/hexanes to obtain pure **3**.

(2*S*,3*R*,3a*R*,9b*R*)-3-benzoyl-2-phenyl-3,3a-dihydro-4*H*-spiro[cyclopenta[*c*]chromene-1,2'-indene]-1',3',4(9b*H*)-trione (3aa)



Following the general procedure-B, **3aa** was obtained as a white solid (89% yield, 44.3 mg).

R_f 0.18 (EtOAc/hexanes = 1/5); mp.: 201.6-202.2 °C; $[\alpha]_D^{28}$ = 233.0 (c = 0.5 in CH_2Cl_2).

HPLC: 96% ee, Chiralpak IB column, *n*-hexane/*i*-PrOH = 70:30, flow rate = 1.00 mL/min, λ= 248 nm, t_{minor} = 9.67 min, t_{major} = 16.14 min.

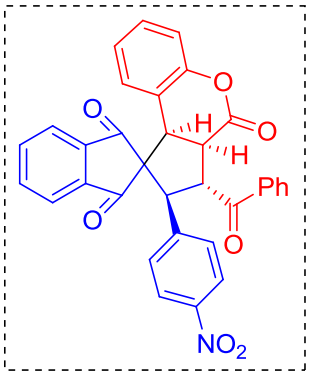
^{1}H NMR (400 MHz, $CDCl_3$) *δ*/ppm: 8.06 (d, *J* = 8.4 Hz, 2H), 7.82 (d, *J* = 7.4 Hz, 1H), 7.56-7.65 (m, 1H), 7.48-7.55 (m, 2H), 7.44 (pt, *J* = 7.5 Hz, 1H), 7.31 (pt, *J* = 7.9 Hz, 2H), 7.05-7.13 (m, 3H), 6.87-7.00 (m, 4H), 6.84 (dd, *J* = 7.9, 1.3 Hz, 1H), 6.76 (pt, *J* = 7.8 Hz, 1H), 5.51 (dd, *J* = 10.6, 3.9 Hz, 1H), 4.41 (d, *J* = 10.6 Hz, 1H), 4.26 (d, *J* = 10.6 Hz, 1H), 3.89 (dd, *J* = 10.6, 3.9 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) *δ*/ppm: 201.3, 200.5, 199.9, 168.1, 150.9, 142.4, 142.1, 135.9, 135.8, 135.7, 134.3, 133.4, 129.4, 129.1, 128.3, 128.2, 127.8, 127.5, 124.1, 122.8, 122.7, 117.3, 117.1, 71.7, 58.1, 53.0, 46.5, 44.6.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3063, 2923, 1763, 1702, 1597, 1264, 1226.

HRMS (ESI) calcd for $C_{33}H_{23}O_5$, $[M+H]^+$ 499.1540, found: 499.1550.

(2*S*,3*R*,3a*R*,9b*R*)-3-benzoyl-2-(4-nitrophenyl)-3,3a-dihydro-4*H*-spiro[cyclopenta[*c*]chromene-1,2'-indene]-1',3',4(9b*H*)-trione (3ab)



Following the general procedure-B, **3ab** was obtained as a pale yellow solid (84% yield, 45.6 mg).

R_f 0.13 (EtOAc/hexanes = 1/5); mp.: 148.4-149.0 °C; $[\alpha]_D^{26}$ = 154.3 (c = 0.5 in CH_2Cl_2).

HPLC: 92% ee, Chiralpak IB column, *n*-hexane/*i*-PrOH = 70:30, flow rate = 1.00 mL/min, λ= 248 nm, t_{minor} = 17.99 min, t_{major} = 26.66 min.

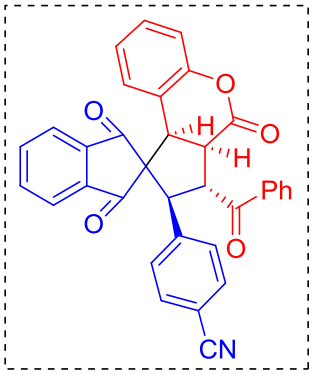
^{1}H NMR (400 MHz, $CDCl_3$) *δ*/ppm: 8.14 (d, *J* = 7.8 Hz, 2H), 7.80-7.93 (m, 3H), 7.70 (pt, *J* = 7.5 Hz, 1H), 7.61 (pt, *J* = 7.5 Hz, 1H), 7.47-7.58 (m, 2H), 7.40 (pt, *J* = 7.6 Hz, 2H), 7.33 (d, *J* = 8.7 Hz, 2H), 7.12 (ddd, *J* = 8.4, 6.5, 1.9 Hz, 1H), 7.00 (d, *J* = 8.0 Hz, 1H), 6.72-6.84 (m, 2H), 5.53 (dd, *J* = 10.1, 3.6 Hz, 1H), 4.51 (d, *J* = 10.1 Hz, 1H), 4.39 (d, *J* = 10.1 Hz, 1H), 3.85 (dd, *J* = 10.1, 3.6 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) *δ*/ppm: 200.8, 199.7, 199.4, 167.7, 150.9, 147.3, 142.33, 142.30, 141.8, 136.40, 136.36, 135.7, 133.9, 129.8, 129.5, 129.1, 128.7, 127.6, 124.3, 123.6, 123.1, 123.0, 117.5, 116.4, 71.2, 56.2, 53.0, 47.4, 44.9.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3068, 2928, 1761, 1704, 1595, 1522, 1350, 1264, 1163.

HRMS (ESI) calcd for $C_{33}H_{22}O_7N$, $[M+H]^+$ 544.1391, found: 544.1392.

4-((2*S*,3*R*,3a*R*,9b*R*)-3-benzoyl-1',3',4-trioxo-1',3,3a,3',4,9b-hexahydro-4*H*-spiro[cyclopenta[*c*]chromene-1,2'-inden]-2-yl)benzonitrile (3ac)



Following the general procedure-B, **3ac** was obtained as a pale yellow solid (90% yield, 47.1 mg).

R_f 0.10 (EtOAc/hexanes = 1/5); mp.: 191.4-192.2 °C; $[\alpha]_D^{25}$ = 127.0 (c = 0.5 in CH_2Cl_2).

HPLC: 94% ee, Chiralpak IB column, *n*-hexane/*i*-PrOH = 70:30, flow rate = 1.00 mL/min, λ= 248 nm, t_{minor} = 17.45 min, t_{major} = 25.03 min.

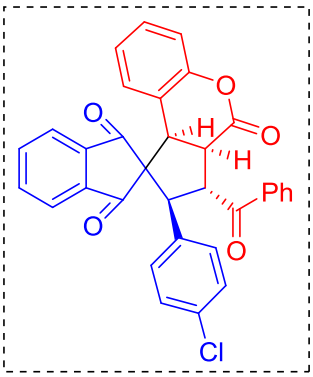
^{1}H NMR (400 MHz, $CDCl_3$) *δ*/ppm: 8.11 (dd, *J* = 8.8, 1.3 Hz, 2H), 7.86 (d, *J* = 7.5 Hz, 1H), 7.70 (td, *J* = 7.4, 1.3 Hz, 1H), 7.61 (td, *J* = 7.5, 1.3 Hz, 1H), 7.50-7.57 (m, 2H), 7.40 (pt, *J* = 7.9 Hz, 2H), 7.30 (d, *J* = 8.4 Hz, 2H), 7.24 (d, *J* = 8.4 Hz, 2H), 7.12 (ddd, *J* = 8.4, 6.2, 2.2 Hz, 1H), 7.00 (d, *J* = 8.4, 1H), 6.74-6.83 (m, 2H), 5.48 (dd, *J* = 10.1, 3.9 Hz, 1H), 4.42 (d, *J* = 10.1, 1H), 4.37 (d, *J* = 10.1, 1H), 3.84 (dd, *J* = 10.1, 3.9 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) *δ*/ppm: 200.9, 199.7, 199.5, 167.8, 150.9, 142.4, 141.9, 140.3, 136.4, 136.3, 135.8, 133.9, 132.3, 129.8, 129.3, 129.2, 128.7, 127.6, 124.4, 123.1, 123.0, 118.0, 117.6, 116.5, 111.9, 71.3, 56.7, 52.9, 47.3, 44.9.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3063, 2923, 2233, 1763, 1702, 1595, 1237, 1163.

HRMS (ESI) calcd for $C_{34}H_{22}O_5N$, $[M+H]^+$ 524.1492, found: 524.1497.

(2*S*,3*R*,3a*R*,9b*R*)-3-benzoyl-2-(4-chlorophenyl)-3,3a-dihydro-4*H*-spiro[cyclopenta[*c*]chromene-1,2'-indene]-1',3',4(9b*H*)-trione (3ad)



Following the general procedure-B, **3ad** was obtained as a white solid (88% yield, 46.9 mg).

R_f 0.27 (EtOAc/hexanes = 1/5); mp.: 159.2-160.4 °C; $[\alpha]_D^{27}$ = 166.3 (c = 0.5 in CH_2Cl_2).

HPLC: 94% ee, Chiralpak IB column, *n*-hexane/*i*-PrOH = 70:30, flow rate = 1.00 mL/min, λ= 248 nm, t_{minor} = 9.70 min, t_{major} = 14.42 min.

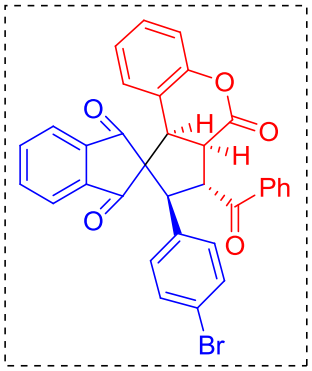
^{1}H NMR (400 MHz, $CDCl_3$) δ/ppm: 8.09 (d, *J* = 8.0 Hz, 2H), 7.84 (d, *J* = 7.6 Hz, 1H), 7.67 (pt, *J* = 7.2 Hz, 1H), 7.59 (pt, *J* = 7.7 Hz, 1H), 7.55 (d, *J* = 7.7 Hz, 1H), 7.50 (pt, *J* = 7.7 Hz, 1H), 7.37 (pt, *J* = 7.70 Hz, 2H), 7.11 (pt, *J* = 7.7 Hz, 1H), 7.04 (d, *J* = 8.1 Hz, 2H), 6.90-7.01 (m, 3H), 6.72-6.84 (m, 2H), 5.45 (dd, *J* = 10.6, 3.8 Hz, 1H), 3.47 (d, *J* = 10.6 Hz, 1H), 4.28 (d, *J* = 10.6 Hz, 1H), 3.85 (dd, *J* = 10.6, 3.8 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ/ppm: 201.2, 200.3, 199.8, 168.0, 151.0, 142.5, 142.1, 136.1, 136.0, 135.9, 133.72, 133.70, 133.1, 129.7, 129.6, 129.1, 128.6, 128.5, 127.6, 124.2, 123.0, 122.9, 117.5, 116.8, 71.4, 56.9, 53.1, 47.0, 44.8.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3063, 2923, 1763, 1702, 1596, 1268, 1165.

HRMS (ESI) calcd for $C_{33}H_{22}O_5{}^{35}Cl$, $[M+H]^+$ 533.1150, found: 533.1158; calcd for $C_{33}H_{22}O_5{}^{37}Cl$, $[M+H]^+$ 535.1121, found: 535.1147.

(2*S*,3*R*,3a*R*,9b*R*)-3-benzoyl-2-(4-bromophenyl)-3,3a-dihydro-4*H*-spiro[cyclopenta[*c*]chromene-1,2'-indene]-1',3',4(9b*H*)-trione (3ae)



Following the general procedure-B, **3ae** was obtained as a white solid (85% yield, 49.1 mg).

R_f 0.27 (EtOAc/hexanes = 1/5); mp.: 228.8-230.6 °C; $[\alpha]_D^{27}$ = 172.6 (c = 0.5 in CH_2Cl_2).

HPLC: 93% ee, Chiralpak IB column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.00 mL/min, λ= 248 nm, t_{minor} = 24.50 min, t_{major} = 35.47 min.

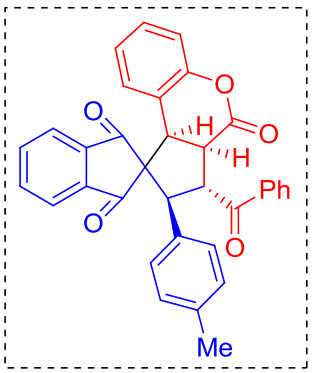
^{1}H NMR (400 MHz, $CDCl_3$) *δ*/ppm: 8.09 (d, *J* = 8.3 Hz, 2H), 7.85 (d, *J* = 8.0 Hz, 1H), 7.68 (pt, *J* = 7.3 Hz, 1H), 7.60 (pt, *J* = 7.5 Hz, 1H), 7.55 (d, *J* = 7.5 Hz, 1H), 7.51 (pt, *J* = 8.0 Hz, 1H), 7.38 (pt, *J* = 8.0 Hz, 2H), 7.07-7.16 (m, 3H), 6.94-7.03 (m, 3H), 6.73-6.83 (m, 2H), 5.44 (dd, *J* = 10.6, 4.0 Hz, 1H), 4.37 (d, *J* = 10.6 Hz, 1H), 4.28 (d, *J* = 10.6 Hz, 1H), 3.84 (dd, *J* = 10.6, 4.0 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) *δ*/ppm: 201.2, 200.2, 199.8, 167.9, 150.9, 142.4, 142.0, 136.1, 136.0, 135.9, 133.7, 133.6, 131.6, 130.0, 129.5, 129.1, 128.5, 127.6, 124.2, 123.0, 122.9, 121.9, 117.4, 116.8, 71.3, 56.9, 53.0, 47.0, 44.7.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3068, 2923, 1763, 1704, 1594, 1237, 1170, 1102.

HRMS (ESI) calcd for $C_{33}H_{21}O_5{}^{79}BrNa$, $[M+Na]^+$ 599.0465, found: 599.0470; calcd for $C_{33}H_{21}O_5{}^{81}BrNa$, $[M+Na]^+$ 601.0444, found: 601.0446.

(2*S*,3*R*,3a*R*,9b*R*)-3-benzoyl-2-(*p*-tolyl)-3,3a-dihydro-4*H*-spiro[cyclopenta[*c*]chromene-1,2'-indene]-1',3',4(9b*H*)-trione (3af)



Following the general procedure-B, **3af** was obtained as a white solid (92% yield, 47.1 mg).

R_f 0.20 (EtOAc/hexanes = 1/5); mp.: 167.9-168.3 °C; $[\alpha]_D^{26}$ = 183.7 (c = 0.5 in CH_2Cl_2).

HPLC: 93% ee, Chiralpak IB column, *n*-hexane/*i*-PrOH = 85:15, flow rate = 1.00 mL/min, λ= 248 nm, t_{minor} = 17.74 min, t_{major} = 28.74 min.

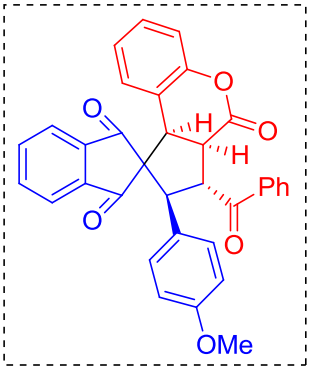
^{1}H NMR (400 MHz, $CDCl_3$) *δ*/ppm: 8.08 (d, *J* = 8.3 Hz, 2H), 7.82 (d, *J* = 7.8 Hz, 1H), 7.62 (ddd, *J* = 8.3, 5.8, 2.6 Hz, 1H), 7.50-7.58 (m, 2H), 7.47 (pt, *J* = 7.3 Hz, 1H), 7.34 (pt, *J* = 7.9 Hz, 2H), 7.10 (td, *J* = 8.4, 1.8 Hz, 1H), 6.99 (d, *J* = 8.4 Hz, 1H), 6.96 (d, *J* = 8.2 Hz, 2H), 6.83 (dd, *J* = 7.9, 1.4 Hz, 1H), 6.72-6.80 (m, 3H), 5.48 (dd, *J* = 10.6, 4.0 Hz, 1H), 4.40 (d, *J* = 10.6 Hz, 1H), 4.24 (d, *J* = 10.6 Hz, 1H), 3.85 (dd, *J* = 10.6, 4.0 Hz, 1H), 2.03 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) *δ*/ppm: 201.5, 200.8, 200.1, 168.2, 151.0, 142.6, 142.3, 137.5, 136.1, 135.8, 135.7, 133.4, 131.2, 129.4, 129.2, 129.1, 128.4, 128.2, 127.6, 124.2, 122.9, 122.8, 117.4, 117.2, 71.7, 57.9, 53.1, 46.7, 44.8, 20.7.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3068, 2927, 1761, 1704, 1595, 1264, 1163.

HRMS (ESI) calcd for $C_{34}H_{25}O_5$, $[M+H]^+$ 513.1697, found: 513.1704.

(2*S*,3*R*,3a*R*,9b*R*)-3-benzoyl-2-(4-methoxyphenyl)-3,3a-dihydro-4*H*-spiro[cyclopenta[*c*]chromene-1,2'-indene]-1',3',4(9b*H*)-trione (3ag)



Following the general procedure-B, **3ag** was obtained as a pale yellow solid (84% yield, 44.4 mg).

R_f 0.13 (EtOAc/hexanes = 1/5); mp.: 205.1-207.1 °C; $[\alpha]_D^{26}$ = 174.4 (c = 0.5 in CH_2Cl_2).

HPLC: 91% ee, Chiralpak IB column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.00 mL/min, λ= 248 nm, t_{minor} = 33.88 min, t_{major} = 45.19 min.

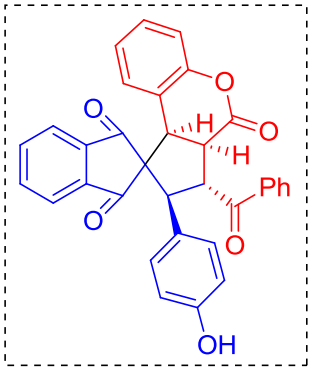
^{1}H NMR (400 MHz, $CDCl_3$) *δ*/ppm: 8.06 (dd, *J* = 8.3, 1.3 Hz, 2H), 7.82 (d, *J* = 7.5 Hz, 1H), 7.64 (ddd, *J* = 8.0, 6.2, 1.8 Hz, 1H), 7.51-7.59 (m, 2H), 7.48 (pt, *J* = 7.3 Hz, 1H), 7.35 (pt, *J* = 7.7 Hz, 2H), 7.11 (ddd, *J* = 8.6, 7.0, 1.6 Hz, 1H), 6.96-7.03 (m, 3H), 6.83 (dd, *J* = 7.6, 1.8 Hz, 1H), 6.77 (td, *J* = 7.4, 1.3 1H), 6.48 (d, *J* = 8.6 Hz, 2H), 5.45 (dd, *J* = 10.5, 4.0 Hz, 1H), 4.39 (d, *J* = 10.5 Hz, 1H), 4.21 (d, *J* = 10.5 Hz, 1H), 3.87 (dd, *J* = 10.5, 4.0 Hz, 1H), 3.56 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) *δ*/ppm: 201.6, 200.8, 200.2, 168.2, 159.0, 151.0, 142.7, 142.3, 136.1, 135.9, 135.8, 133.5, 129.5, 129.4, 129.2, 128.4, 127.6, 126.3, 124.2, 122.9, 122.8, 117.5, 117.2, 113.8, 71.8, 57.7, 55.0, 53.4, 46.6, 44.7.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3068, 2928, 1761, 1702, 1595, 1255, 1165.

HRMS (ESI) calcd for $C_{34}H_{25}O_6$ $[M+H]^+$ 529.1646, found: 529.1655.

(2*S*,3*R*,3a*R*,9b*R*)-3-benzoyl-2-(4-hydroxyphenyl)-3,3a-dihydro-4*H*-spiro[cyclopenta[*c*]chromene-1,2'-indene]-1',3',4(9b*H*)-trione (3ah)



Following the general procedure-B, **3ah** was obtained as an off-white solid (70% yield, 36.0 mg).

R_f 0.30 (EtOAc/hexanes = 1/1); mp.: 127.1-128.5 °C; $[\alpha]_D^{21}$ = 211.6 (c = 0.5 in CH_2Cl_2).

HPLC: 69% ee, Chiralpak IB column, *n*-hexane/*i*-PrOH = 70:30, flow rate = 1.00 mL/min, λ= 248 nm, t_{minor} = 10.10 min, t_{major} = 17.74 min.

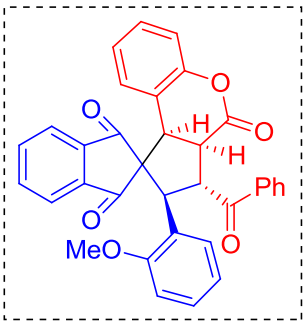
^{1}H NMR (400 MHz, $CDCl_3$) *δ*/ppm: 8.03 (d, *J* = 8.3 Hz, 2H), 7.82 (d, *J* = 8.1 Hz, 1H), 7.64 (pt, *J* = 8.1 Hz, 1H), 7.52-7.61 (m, 2H), 7.45 (pt, *J* = 7.7 Hz, 1H), 7.31 (pt, *J* = 8.1 Hz, 2H), 7.11 (pt, *J* = 8.3 Hz, 1H), 6.99 (d, *J* = 8.3 Hz, 1H), 6.95 (d, *J* = 8.3 Hz, 2H), 6.83 (d, *J* = 7.7 Hz, 1H), 6.78 (pt, *J* = 7.7 Hz, 1H), 6.46 (d, *J* = 8.4 Hz, 2H), 5.42 (dd, *J* = 10.6, 4.0 Hz, 1H), 5.02 (s, 1H), 4.39 (d, *J* = 10.6 Hz, 1H), 4.19 (d, *J* = 10.6 Hz, 1H), 3.86 (dd, *J* = 10.6, 4.0 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) *δ*/ppm: 200.8, 200.1, 200.4, 168.7, 155.6, 150.9, 142.6, 142.3, 136.0, 135.94, 135.91, 133.6, 129.6, 129.5, 129.2, 128.4, 127.6, 126.0, 124.4, 123.0, 122.9, 117.4, 117.2, 115.5, 71.8, 57.8, 53.4, 46.5, 44.7.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3064, 2923, 1735, 1702, 1594, 1266, 1235, 1163, 762.

HRMS (ESI) calcd for $C_{33}H_{23}O_6$, $[M+H]^+$ 515.1489, found: 515.1497.

(2*S*,3*R*,3a*R*,9b*R*)-3-benzoyl-2-(2-methoxyphenyl)-3,3a-dihydro-4*H*-spiro[cyclopenta[*c*]chromene-1,2'-indene]-1',3',4(9b*H*)-trione (3ai)



Following the general procedure-B, **3ai** was obtained as a pale yellow solid (93% yield, 49.1 mg).

R_f 0.10 (EtOAc/hexanes = 1/5); mp.: 78.7-80.4 °C; $[\alpha]_D^{26}$ = 197.9 (c = 0.5 in CH_2Cl_2).

HPLC: 88% ee, Chiralpak IB column, *n*-hexane/*i*-PrOH = 70:30, flow rate = 1.00 mL/min, λ= 248 nm, t_{minor} = 10.76 min, t_{major} = 18.93 min.

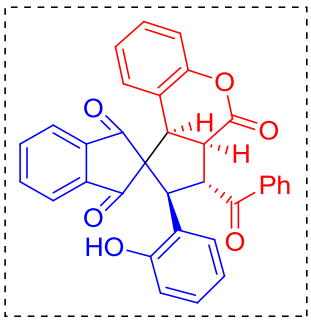
^{1}H NMR (400 MHz, $CDCl_3$) *δ*/ppm: 8.03 (dd, *J* = 8.4, 1.3 Hz, 2H), 7.85 (d, *J* = 7.8 Hz, 1H), 7.63 (td, *J* = 7.5, 1.3 Hz, 1H), 7.43-7.52 (m, 2H), 7.42 (dd, *J* = 7.9, 1.3 Hz, 1H), 7.38 (d, *J* = 7.8 Hz, 1H), 7.32 (pt, *J* = 7.8 Hz, 2H), 7.11 (ddd, *J* = 8.6, 7.1, 1.5 Hz, 1H), 6.99 (d, *J* = 8.3 Hz, 1H), 6.87-6.94 (m, 2H), 6.73-6.83 (m, 2H), 6.27 (d, *J* = 8.2 Hz, 1H), 5.50 (dd, *J* = 8..8, 3.1 Hz, 1H), 4.77 (d, *J* = 8.8 Hz, 1H), 4.50 (d, *J* = 10.5 Hz, 1H), 3.90 (dd, *J* = 10.5, 3.1 Hz, 1H), 3.34 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) *δ*/ppm: 200.7, 199.8, 199.0, 168.2, 156.6, 151.3, 142.8, 141.5, 135.7, 135.3, 133.3, 129.2, 129.0, 128.9, 128.6, 128.3, 127.7, 124.1, 123.0, 122.5, 122.2, 120.5, 117.9, 117.2, 109.6, 71.1, 54.4, 52.1, 50.0, 45.7, 44.9.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3062, 2932, 1761, 1704, 1597, 1250.

HRMS (ESI) calcd for $C_{34}H_{25}O_6$ $[M+H]^+$ 529.1646, found: 529.1657.

(2*S*,3*R*,3a*R*,9b*R*)-3-benzoyl-2-(2-hydroxyphenyl)-3,3a-dihydro-4*H*-spiro[cyclopenta[*c*]chromene-1,2'-indene]-1',3',4(9b*H*)-trione (3aj)



Following the general procedure-B, **3aj** was obtained as a white solid (42% yield, 21.6 mg).

R_f 0.30 (EtOAc/hexanes = 1/1); mp.: 266.2-267.4 °C (decomposition).

HPLC: 25% ee, Chiralpak IB column, *n*-hexane/*i*-PrOH = 70:30, flow rate = 1.00 mL/min, λ= 248 nm, t_{minor} = 9.50 min, t_{major} = 18.58 min.

^{1}H NMR (400 MHz, $CDCl_3$) *δ*/ppm: 8.07 (d, *J* = 8.4 Hz, 2H), 7.87 (d, *J* = 7.9 Hz, 1H), 7.66 (pt, *J* = 7.4 Hz, 1H), 7.56 (pt, *J* = 7.9 Hz, 1H), 7.44-7.54 (m, 2H), 7.34 (pt, *J* = 7.7 Hz, 3H), 7.12 (ddd, *J* = 8.6, 7.0, 1.5 Hz, 1H), 7.01 (d, *J* = 7.9 Hz, 1H), 6.75-6.88 (m, 3H), 6.71 (pt, *J* = 7.9 Hz, 1H), 6.39 (d, *J* = 8.0 Hz, 1H), 5.50 (dd, *J* = 9.6, 3.5 Hz, 1H), 5.19 (brs, 1H), 4.79 (d, *J* = 9.6 Hz, 1H), 4.43 (d, *J* = 10.6 Hz, 1H), 3.90 (dd, *J* = 10.6, 3.5 Hz, 1H).

^{1}H NMR (400 MHz, Acetone-d^6) *δ*/ppm: 8.74 (brs, 1H), 8.06 (d, *J* = 8.4 Hz, 2H), 7.88 (d, *J* = 7.9 Hz, 1H), 7.78 (pt, *J* = 8.0 Hz, 1H), 7.67 (ddd, *J* = 8.0, 7.1, 0.9 Hz, 1H), 7.56 (pt, *J* = 8.4 Hz, 1H), 7.38-7.51 (m, 3H), 7.32 (d, *J* = 8.0 Hz, 1H), 7.17 (ddd, *J* = 8.4, 6.7, 1.3 Hz, 1H), 6.98 (d, *J* = 8.4 Hz, 1H), 6.81-

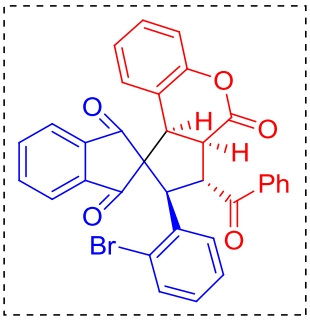
6.92 (m, 2H), 6.75 (ddd, *J* = 8.0, 6.8, 1.1 Hz, 1H), 6.63 (pt, *J* = 8.1 Hz, 1H), 6.38 (d, *J* = 8.1 Hz, 1H), 5.49 (dd, *J* = 9.7, 3.5 Hz, 1H), 4.82 (d, *J* = 9.7 Hz, 1H), 4.49 (d, *J* = 10.6 Hz, 1H), 4.09 (dd, *J* = 10.6, 3.5 Hz, 1H),

^{13}C NMR (100 MHz, Acetone-d^6) *δ*/ppm: 202.2, 201.3, 199.9, 168.9, 156.1, 156.0, 152.4, 144.0, 142.9, 137.3, 136.9, 136.7, 134.4, 130.2, 129.8, 129.42, 129.38, 128.5, 124.9, 123.6, 123.2, 122.05, 122.00, 120.2, 119.4, 117.9, 115.7, 115.6, 72.2, 53.6, 51.2, 47.0, 45.7.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3415, 1741, 1702, 1595, 1237, 1165.

HRMS (ESI) calcd for $C_{33}H_{23}O_6$, $[M+H]^+$ 515.1489, found: 515.1496.

(2*R*,3*R*,3a*R*,9b*R*)-3-benzoyl-2-(2-bromophenyl)-3,3a-dihydro-4*H*-spiro[cyclopenta[*c*]chromene-1,2'-indene]-1',3',4(9b*H*)-trione (3ak)



Following the general procedure-B, **3ak** was obtained as a white solid (88% yield, 50.8 mg).

R_f 0.15 (EtOAc/hexanes = 1/5); mp.: 247.3-248.7 °C; $[\alpha]_D^{25}$ = 205.84 (c = 0.25 in CH_2Cl_2).

HPLC: 91% ee, Chiralpak IB column, *n*-hexane/*i*-PrOH = 70:30, flow rate = 1.00 mL/min, λ= 248 nm, t_{minor} = 10.98 min, t_{major} = 21.30 min.

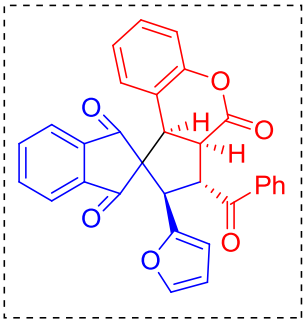
^{1}H NMR (400 MHz, $CDCl_3$) *δ*/ppm: 7.86-7.95 (m, 3H), 7.66 (td, *J* = 7.5, 0.9 Hz, 1H), 7.52-7.59 (m, 2H), 7.42-7.51 (m, 2H), 7.29 (pt, *J* = 7.9 Hz, 2H), 7.12-7.18 (m, 2H), 7.10 (dd, *J* = 7.9, 1.3 Hz, 1H), 7.03 (d, *J* = 8.4 Hz, 1H), 6.88 (d, *J* = 7.9, 1.7 Hz, 1H), 6.81 (td, *J* = 7.7, 1.1 Hz, 2H), 5.31 (dd, *J* = 9.3, 3.1 Hz, 1H), 4.98 (d, *J* = 9.3 Hz, 1H), 4.48 (d, *J* = 10.6 Hz, 1H), 4.00 (dd, *J* = 10. 6, 3.1 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) *δ*/ppm: 201.0, 199.0, 198.8, 168.2, 151.4, 142.7, 142.0, 135.9, 135.8, 135.6, 134.9, 133.4, 132.9, 130.3, 129.5, 129.1, 129.0, 128.3, 127.8, 127.6, 125.6, 124.3, 123.2, 122.8, 117.3, 71.4, 55.4, 54.7, 46.3, 44.5.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3064, 2923, 1761, 1704, 1597, 1237, 1163, 1009.

HRMS (ESI) calcd for $C_{33}H_{22}O_5{}^{79}Br$, $[M+H]^+$ 577.0645, found: 577.0657; calcd for $C_{33}H_{22}O_5{}^{81}Br$, $[M+H]^+$ 579.0625, found: 579.0643.

(2*R*,3*R*,3a*R*,9b*R*)-3-benzoyl-2-(furan-2-yl)-3,3a-dihydro-4*H*-spiro[cyclopenta[*c*]chromene-1,2'-indene]-1',3',4(9b*H*)-trione (3al)



Following the general procedure-B, **3al** was obtained as a pale yellow solid (73% yield, 35.7 mg).

R_f 0.13 (EtOAc/hexanes = 1/5); mp.: 216.7-218.2 °C; $[\alpha]_D^{25}$ = 101.0 (c = 0.5 in CH_2Cl_2).

HPLC: 89% ee, Chiralpak IB column, *n*-hexane/*i*-PrOH = 70:30, flow rate = 1.00 mL/min, λ= 248 nm, t_{minor} = 11.94 min, t_{major} = 18.63 min.

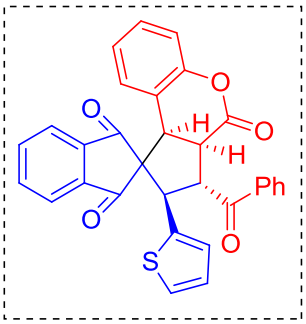
^{1}H NMR (400 MHz, $CDCl_3$) *δ*/ppm: 8.12 (dd, *J* = 7.5, 0.9 Hz, 2H), 7.92 (d, *J* = 7.5 Hz, 1H), 7.69-7.77 (m, 1H), 7.63-7.68 (m, 2H), 7.54 (pt, *J* = 7.5 Hz, 1H), 7.42 (pt, *J* = 7.5 Hz, 2H), 7.12 (td, *J* = 7.7, 1.3 Hz, 1H), 6.99 (d, *J* = 8.2 Hz, 1H), 6.92 (d, *J* = 1.6 Hz, 1H), 6.85 (dd, *J* = 7.6, 1.6 Hz, 1H), 6.79 (pt, *J* = 7.9 Hz, 1H), 5.85-5.88 (m, 1H), 5.83 (d, *J* = 3.1 Hz, 1H), 5.45 (dd, *J* = 10.6, 4.3 Hz, 1H), 4.36 (d, *J* = 10.9 Hz, 1H), 4.31 (d, *J* = 10.6 Hz, 1H), 3.88 (dd, *J* = 10.9, 4.3 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) *δ*/ppm: 200.3, 200.1, 199.5, 167.9, 151.0, 148.4, 142.4, 142.3, 142.0, 136.0, 135.9, 135.8, 133.7, 129.5, 129.1, 128.6, 127.6, 124.3, 123.1, 123.0, 117.5, 116.8, 110.1, 108.7, 69.7, 52.0, 51.1, 46.0, 44.1.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 1759, 1704, 1237, 1163.

HRMS (ESI) calcd for $C_{31}H_{21}O_6$, $[M+H]^+$ 489.1333, found: 489.1339.

(2*R*,3*S*,3a*R*,9b*R*)-3-benzoyl-2-(thiophen-2-yl)-3,3a-dihydro-4*H*-spiro[cyclopenta[*c*]chromene-1,2'-indene]-1',3',4(9b*H*)-trione (3am)



Following the general procedure B, **3am** was obtained as a pale yellow solid (78% yield, 39.3 mg).

R_f 0.15 (EtOAc/hexanes = 1/5); mp.: 200.6-201.4 °C; $[\alpha]_D^{25}$ = 203.8 (c = 0.5 in CH_2Cl_2).

HPLC: 88% ee, Chiralpak IB column, *n*-hexane/*i*-PrOH = 90:10, flow rate = 1.00 mL/min, λ= 248 nm, t_{minor} = 34.84 min, t_{major} = 56.14 min.

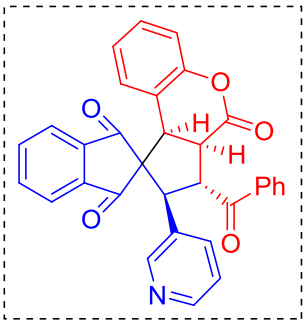
^{1}H NMR (400 MHz, $CDCl_3$) δ/ppm: 8.17 (dd, *J* = 8.3, 1.3 Hz, 2H), 7.89 (d, *J* = 7.5 Hz, 1H), 7.65-7.73 (m, 1H), 7.60-7.64 (m, 2H), 7.53 (tt, *J* = 7.5, 1.3 Hz, 1H), 7.41 (pt, *J* = 7.9 Hz, 2H), 7.11 (ddd, *J* = 8.8, 7.0, 1.8 Hz, 1H), 6.99 (d, *J* = 8.4 Hz, 1H), 6.81-6.86 (m, 2H), 6.78 (td, *J* = 7.4, 1.2 Hz, 1H), 6.72 (d, *J* = 3.6 Hz, 1H), 6.57 (dd, *J* = 5.3, 3.5 Hz, 1H), 5.44 (dd, *J* = 10.6, 4.4 Hz, 1H), 4.59 (d, *J* = 10.6 Hz, 1H), 4.38 (d, *J* = 10.6 Hz, 1H), 3.81 (dd, *J* = 10.80, 4.4 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ/ppm: 201.3, 200.5, 199.7, 167.9, 150.9, 142.9, 142.4, 137.0, 136.1, 136.0, 135.9, 133.7, 129.6, 129.3, 128.5, 127.6, 126.9, 126.7, 124.9, 124.3, 123.1, 123.0, 117.6, 116.9, 71.2, 54.9, 52.8, 46.6, 44.6.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3063, 2923, 1763, 1702, 1595, 1264, 1163.

HRMS (ESI) calcd for $C_{31}H_{21}O_5S$, $[M+H]^+$ 505.1104, found: 505.1113.

(2*S*,3*R*,3a*R*,9b*R*)-3-benzoyl-2-(pyridin-3-yl)-3,3a-dihydro-4*H*-spiro[cyclopenta[*c*]chromene-1,2'-indene]-1',3',4(9b*H*)-trione (3an)



Following the general procedure-B, **3an** was obtained as a pale yellow solid (82% yield, 40.9 mg).

R_f 0.2 (EtOAc/hexanes = 1/1); mp.: 191.1-192.4 °C; $[\alpha]_D^{25}$ = 170.0 (c = 0.5 in CH_2Cl_2).

HPLC: 77% ee, Chiralpak IB column, *n*-hexane/*i*-PrOH = 70:30, flow rate = 1.00 mL/min, λ= 248 nm, t_{minor} = 15.60 min, t_{major} = 24.58 min.

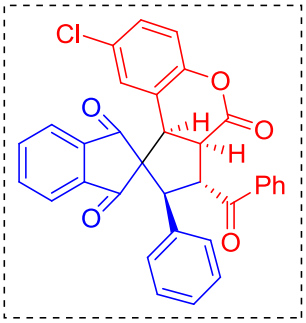
^{1}H NMR (400 MHz, $CDCl_3$) δ/ppm: 8.26 (d, *J* = 2.1 Hz, 1H), 8.21 (dd, *J* = 4.8, 1.4 Hz, 1H), 8.10 (dd, *J* = 8.4, 1.4 Hz, 2H), 7.86 (d, *J* = 7.5 Hz, 1H), 7.68 (td, *J* = 7.7, 1.3 Hz, 1H), 7.53-7.63 (m, 3H), 7.51 (tt, *J* = 7.5, 1.3 Hz, 1H), 7.39 (pt, *J* = 7.8 Hz, 2H), 7.12 (ddd, *J* = 8.4, 6.6, 1.8 Hz, 1H), 6.94-7.04 (m, 2H), 6.74-6.84 (m, 2H), 5.48 (dd, *J* = 10.6, 3.5 Hz, 1H), 4.39 (d, *J* = 10.6 Hz, 1H), 4.35 (d, *J* = 10.6 Hz, 1H), 3.86 (dd, *J* = 10.6, 3.5 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ/ppm: 201.1, 199.9, 199.5, 167.9, 151.0, 149.9, 149.4, 142.5, 142.0, 136.2, 135.8, 135.7, 133.8, 130.6, 129.7, 129.2, 128.7, 127.6, 124.4, 123.3, 123.2, 123.1, 117.6, 116.7, 71.3, 54.4, 53.0, 47.2, 44.8.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 3059, 1759, 1704, 1595, 1266, 1165.

HRMS (ESI) calcd for $C_{32}H_{22}O_5N$, $[M+H]^+$ 500.1492, found: 500.1500.

(2*S*,3*R*,3a*R*,9b*R*)-3-benzoyl-8-chloro-2-phenyl-3,3a-dihydro-4*H*-spiro[cyclopenta[*c*]chromene-1,2'-indene]-1',3',4(9b*H*)-trione (3ba)



Following the general procedure-B, **3ba** was obtained as a white solid (90% yield, 48.0 mg).

R_f 0.25 (EtOAc/hexanes = 1/5); mp.: 250.6-251.3 °C; $[\alpha]_D^{25}$ = 158.13 (c = 0.25 in CH_2Cl_2).

HPLC: 95% ee, Chiralpak IB column, *n*-hexane/*i*-PrOH = 70:30, flow rate = 1.00 mL/min, λ= 248 nm, t_{minor} = 10.23 min, t_{major} = 20.32 min.

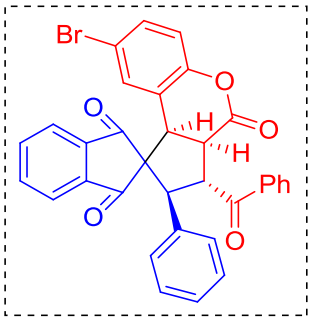
^{1}H NMR (400 MHz, $CDCl_3$) *δ*/ppm: 8.04 (d, *J* = 7.9 Hz, 2H), 7.84 (d, *J* = 7.9 Hz, 1H), 7.65 (td, *J* =7.2, 1.2 Hz, 1H), 7.52-7.61 (m, 2H), 7.47 (pt, *J* =7.5 Hz 1H), 7.33 (pt, *J* = 7.5 Hz, 2H), 7.01-7.12 (m, 3H), 6.88-7.00 (m, 4H), 6.84 (d, *J* =2.5 Hz, 1H), 5.47 (dd, *J* = 10.6, 3.9 Hz, 1H), 4.37 (d, *J* = 10.6 Hz, 1H), 4.21 (d, *J* = 10.6 Hz, 1H), 3.88 (dd, *J* = 10.6, 3.9 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) *δ*/ppm: 201.2, 200.4, 199.6, 167.6, 149.7, 142.5, 142.2, 136.05, 136.00, 135.9, 134.0, 133.6, 129.6, 129.24, 129.19, 128.5, 128.4, 128.3, 128.0, 127.4, 123.0, 119.0, 118.9, 71.8, 58.5, 52.9, 50.0, 44.4.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 1768, 1737, 1704, 1487, 702.

HRMS (ESI) calcd for $C_{33}H_{22}O_5{}^{35}Cl$, $[M+H]^+$ 533.1150, found: 533.1155; calcd for $C_{33}H_{22}O_5{}^{37}Cl$, $[M+H]^+$ 535.1121, found: 535.1147.

(2*S*,3*R*,3a*R*,9b*R*)-3-benzoyl-8-bromo-2-phenyl-3,3a-dihydro-4*H*-spiro[cyclopenta[*c*]chromene-1,2'-indene]-1',3',4(9b*H*)-trione (3ca)



Following the general procedure-B, **3ca** was obtained as a white solid (85% yield, 49.1 mg).

R_f 0.25 (EtOAc/hexanes = 1/5); mp.: 243.5-244.2 °C; $[\alpha]_D^{25}$ = 142.44 (c = 0.25 in CH_2Cl_2).

HPLC: 93% ee, Chiralpak IB column, *n*-hexane/*i*-PrOH = 70:30, flow rate = 1.00 mL/min, λ= 248 nm, t_{minor} = 11.78 min, t_{major} = 22.21 min.

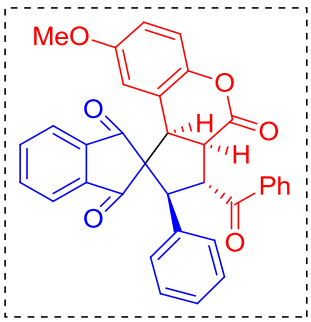
^{1}H NMR (400 MHz, $CDCl_3$) δ/ppm: 8.03 (dd, *J* = 8.3, 1.3 Hz, 2H), 7.84 (dt, *J* = 7.5, 0.9 Hz, 1H), 7.65 (td, *J* =7.2, 1.3 Hz, 2H), 7.58 (td, *J* = 7.2, 1.3 Hz, 1H), 7.52-7.56 (m 1H), 7.47 (tt, *J* = 7.5, 1.7 Hz, 1H), 7.33 (pt, *J* = 7.9 Hz, 1H), 7.23 (dd, *J* = 8.8, 2.2 Hz, 1H), 7.05 (dd, *J* = 8.3, 1.7 Hz, 2H), 6.91-7.00 (m, 4H), 6.89 (d, *J* =8.7 Hz, 1H), 5.46 (dd, *J* = 10.6, 4.0 Hz, 1H), 4.36 (d, *J* = 10.6 Hz, 1H), 4.21 (d, *J* = 10.6 Hz, 1H), 3.87 (dd, *J* = 10.6, 4.0 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ/ppm: 201.2, 200.4, 199.6, 167.6, 150.3, 142.5, 142.2, 136.1, 136.0, 135.9, 134.1, 133.6, 132.6, 130.3, 129.2, 128.50, 128.46, 128.3, 128.0, 123.0, 119.5, 119.3, 116.7, 71.8, 58.5, 53.0, 46.0, 44.4.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 1768, 1702, 1678, 1483, 702.

HRMS (ESI) calcd for $C_{33}H_{22}O_5{}^{79}Br$, $[M+H]^+$ 577.0645, found: 577.0651; calcd for $C_{33}H_{22}O_5{}^{81}Br$, $[M+H]^+$ 579.0625, found: 579.0638.

(2*S*,3*R*,3a*R*,9b*R*)-3-benzoyl-8-methoxy-2-phenyl-3,3a-dihydro-4*H*-spiro[cyclopenta[*c*]chromene-1,2'-indene]-1',3',4(9b*H*)-trione (3da)



Following the general procedure-B, **3da** was obtained as a white solid (92% yield, 48.6 mg).

R_f 0.13 (EtOAc/hexanes = 1/5); mp.: 246.5-247.4 °C; $[\alpha]_D^{25}$ = 161.06 (c = 0.25 in CH_2Cl_2).

HPLC: 91% ee, Chiralpak IB column, *n*-hexane/*i*-PrOH = 70:30, flow rate = 1.00 mL/min, λ= 248 nm, t_{minor} = 12.65 min, t_{major} = 20.90 min.

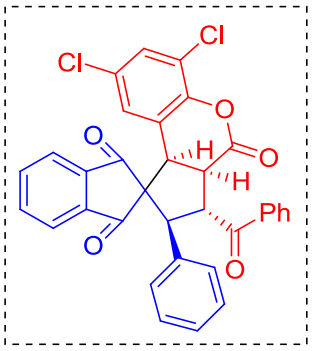
^{1}H NMR (400 MHz, $CDCl_3$) δ/ppm: 8.07 (dd, *J* = 8.4, 1.4 Hz, 2H), 7.82 (d, *J* = 7.9 Hz, 1H), 7.64 (td, *J* =7.0, 2.2 Hz, 1H), 7.52-7.60 (m, 2H), 7.47 (pt, *J* = 7.3 Hz, 1H), 7.34 (pt, *J* = 7.8 Hz, 2H), 7.08 (dd, *J* = 8.2, 1.4 Hz, 2H), 6.88-7.00 (m, 4H), 6.65 (dd, *J* =8.8, 3.0 Hz, 1H), 6.32 (d, *J* = 3.0 Hz, 1H), 5.49 (dd, *J* = 10.6, 4.0 Hz, 1H), 4.36 (d, *J* = 10.6 Hz, 1H), 4.25 (d, *J* = 10.6 Hz, 1H), 3.84 (dd, *J* = 10.8, 4.0 Hz, 1H), 3.52 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ/ppm: 201.2, 200.7, 200.1, 168.3, 155.7, 145.0, 142.6, 142.3, 136.1, 135.9, 135.8, 134.4, 133.5, 129.2, 128.44, 128.37, 127.8, 123.0, 122.7, 118.4, 117.8, 115.5, 111.7, 71.8, 58.2, 55.5, 53.1, 47.0, 44.6.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 1752, 1703, 1499, 1173.

HRMS (ESI) calcd for $C_{34}H_{25}O_6$, $[M+H]^+$ 529.1646, found: 529.1652.

(2*S*,3*R*,3a*R*,9b*R*)-3-benzoyl-6,8-dichloro-2-phenyl-3,3a-dihydro-4*H*-spiro[cyclopenta[*c*]chromene-1,2'-indene]-1',3',4(9b*H*)-trione (3ea)



Following the general procedure-B, **3ea** was obtained as a white solid (82% yield, 46.5 mg).

R_f 0.19 (EtOAc/hexanes = 1/5); mp.: 245.7-246.6 °C; $[\alpha]_D^{25}$ = 19.32 (c = 0.25 in CH_2Cl_2).

HPLC: 95% ee, Chiralpak IB column, *n*-hexane/*i*-PrOH = 70:30, flow rate = 1.00 mL/min, λ= 248 nm, t_{minor} = 9.82 min, t_{major} = 12.50 min.

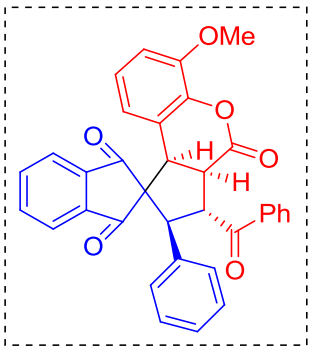
^{1}H NMR (400 MHz, $CDCl_3$) *δ*/ppm: 8.02 (dd, *J* = 8.4, 1.3 Hz, 2H), 7.84 (d, *J* = 7.5 Hz, 1H), 7.67 (td, *J* = 7.3, 1.3 Hz, 1H), 7.60 (td, *J* = 7.3, 1.3 Hz, 2H), 7.57 (d, *J* = 7.2 Hz, 1H), 7.48 (tt, *J* = 7.3, 1.3 Hz, 1H), 7.33 (pt, *J* = 8.0 Hz, 1H), 7.21 (d, *J* = 2.6 Hz, 1H), 7.04 (dd, *J* = 8.4, 1.3 Hz, 2H), 6.89-6.99 (m, 3H), 6.76 (d, *J* = 2.6 Hz, 1H), 5.45 (dd, *J* = 10.6, 3.9 Hz, 1H), 4.37 (d, *J* = 10.6 Hz, 1H), 4.18 (d, *J* = 10.1 Hz, 1H), 3.90 (dd, *J* = 10.6, 3.9 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) *δ*/ppm: 200.9, 200.0, 199.4, 166.5, 146.1, 142.4, 142.1, 136.2, 136.1, 135.7, 133.9, 133.7, 130.2, 129.2, 129.0, 128.55, 128.49, 128.3, 128.1, 125.8, 123.3, 123.2, 123.0, 120.4, 71.8, 58.6, 52.9, 45.9, 44.3.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 1777, 1703, 1682, 1456, 702.

HRMS (ESI) calcd for $C_{33}H_{21}O_5{}^{35}Cl_2$, $[M+H]^+$ 567.0761, found: 567.0765; calcd for $C_{33}H_{21}O_5{}^{35}Cl^{37}Cl$, $[M+H]^+$ 569.0731, found: 569.0749.

(2*S*,3*R*,3a*R*,9b*R*)-3-benzoyl-6-methoxy-2-phenyl-3,3a-dihydro-4*H*-spiro[cyclopenta[*c*]chromene-1,2'-indene]-1',3',4(9b*H*)-trione (3fa)



Following the general procedure-B, **3fa** was obtained as a white solid (86% yield, 45.4 mg).

R_f 0.13 (EtOAc/hexanes = 1/5); mp.: 243.4-244.1 °C; $[\alpha]_D^{25}$ = 292.44 (c = 0.25 in CH_2Cl_2).

HPLC: 92% ee, Chiralpak IB column, *n*-hexane/*i*-PrOH = 70:30, flow rate = 1.00 mL/min, λ= 248 nm, t_{minor} = 19.61 min, t_{major} = 30.82 min.

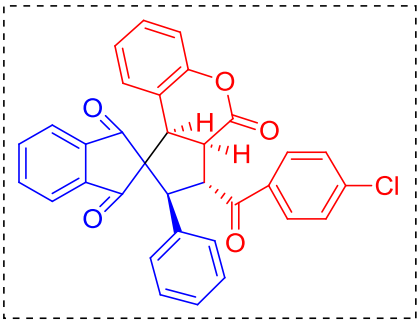
^{1}H NMR (400 MHz, $CDCl_3$) *δ*/ppm: 8.05 (d, *J* = 7.9 Hz, 2H), 7.81 (d, *J* = 7.5 Hz, 1H), 7.58-7.66 (m, 1H), 7.51-7.58 (m, 2H), 7.46 (pt, *J* = 7.5 Hz, 1H), 7.32 (pt, *J* = 7.9 Hz, 2H), 7.07 (dd, *J* = 8.3, 1.5 Hz, 2H), 6.88-6.99 (m, 3H), 6.64-6.75 (m, 2H), 6.40 (dd, *J* = 7.3, 1.7 Hz, 1H), 5.49 (dd, *J* = 10.6, 3.9 Hz, 1H), 4.39 (d, *J* = 10.6 Hz, 1H), 4.23 (d, *J* = 10.6 Hz, 1H), 3.88 (dd, *J* = 10.6, 3.9 Hz, 1H), 3.82 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) *δ*/ppm: 201.2, 200.6, 200.1, 167.5, 147.6, 142.6, 142.3, 140.6, 136.0, 135.8, 135.7, 134.4, 133.5, 129.2, 128.4, 127.8, 124.1, 123.0, 122.8, 118.8, 118.0, 111.7, 71.7, 58.3, 55.9, 53.2, 46.8, 44.5.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 1759, 1703, 1484, 1161.

HRMS (ESI) calcd for $C_{34}H_{25}O_6$, $[M+H]^+$ 529.1646, found: 529.1650.

(2*S*,3*R*,3a*R*,9b*R*)-3-(4-chlorobenzoyl)-2-phenyl-3,3a-dihydro-4*H*-spiro[cyclopenta[*c*]chromene-1,2'-indene]-1',3',4(9b*H*)-trione (3ga)



Following the general procedure-B, **3ga** was obtained as a white solid (87% yield, 46.4 mg).

R_f 0.17 (EtOAc/hexanes = 1/5); mp.: 255.3-255.9 °C; $[\alpha]_D^{25}$ = 84.16 (c = 0.25 in CH_2Cl_2).

HPLC: 90% ee, Chiralpak IB column, *n*-hexane/*i*-PrOH = 70:30, flow rate = 1.00 mL/min, λ= 248 nm, t_{minor} = 9.38 min, t_{major} = 20.19 min.

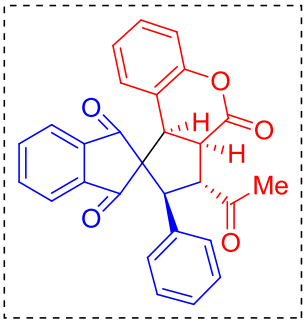
^{1}H NMR (400 MHz, $CDCl_3$) *δ*/ppm: 8.01 (d, *J* = 8.7 Hz, 2H), 7.82 (d, *J* = 7.5 Hz, 1H), 7.58-7.66 (m, 1H), 7.48-7.57 (m, 2H), 7.26-7.33 (m, 2H), 7.02-7.14 (m, 3H), 6.88-7.02 (m, 4H), 6.84 (d, *J* = 7.2 Hz, 1H), 6.77 (pt, *J* = 7.4 Hz, 1H), 5.44 (dd, *J* = 10.6, 3.9 Hz, 1H), 4.41 (d, *J* = 10.6 Hz, 1H), 4.24 (d, *J* = 10.6 Hz, 1H), 3.87 (dd, *J* = 10.6, 3.9 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) *δ*/ppm: 201.4, 199.8, 199.5, 168.2, 150.9, 142.5, 142.2, 140.1, 135.9, 135.8, 134.4, 134.1, 130.6, 129.5, 128.7, 128.5, 128.4, 128.2, 128.0, 127.6, 124.2, 122.9, 122.8, 117.4, 117.0, 71.6, 58.1, 53.1, 46.6, 44.7.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 1764, 1703, 1458, 762.

HRMS (ESI) calcd for $C_{33}H_{22}O_5{}^{35}Cl$, $[M+H]^+$ 533.1150, found: 533.1156; calcd for $C_{33}H_{22}O_5{}^{37}Cl$, $[M+H]^+$ 535.1121, found: 535.1142.

(2*S*,3*R*,3a*R*,9b*R*)-3-acetyl-2-phenyl-3,3a-dihydro-4*H*-spiro[cyclopenta[*c*]chromene-1,2'-indene]-1',3',4(9b*H*)-trione (3ha)



Following the general procedure-B, **3ha** was obtained as a white solid (95% yield, 41.5 mg).

R_f 0.09 (EtOAc/hexanes = 1/5); mp.: 220.7-221.2 °C; $[\alpha]_D^{28}$ = 236.6 (c = 0.25 in CH_2Cl_2).

HPLC: 93% ee, Chiralpak IB column, *n*-hexane/*i*-PrOH = 85:15, flow rate = 1.00 mL/min, λ= 248 nm, t_{minor} = 17.72 min, t_{major} = 20.08 min.

^{1}H NMR (400 MHz, $CDCl_3$) *δ*/ppm: 7.81 (d, *J* = 7.5 Hz, 1H), 7.63 (pt, *J* = 7.3, 1.5 Hz, 1H), 7.48-7.58 (m, 2H), 7.15 (d, *J* = 7.5 Hz, 2H), 7.02-7.11 (m, 4H), 6.99 (d, *J* = 8.4 Hz, 1H), 6.72-6.82 (m, 2H), 4.65 (d, *J* = 11.1, 4.3 Hz, 1H), 4.23 (d, *J* = 11.1 Hz, 1H), 3.99 (dd, *J* = 11.1, 4.3 Hz, 1H), 3.92 (d, *J* = 11.1 Hz, 1H), 2.09 (s, 3H).

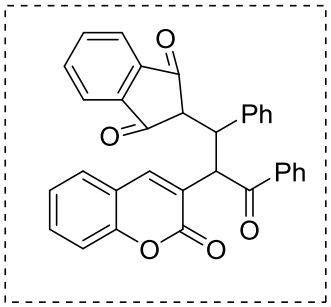
^{13}C NMR (100 MHz, $CDCl_3$) *δ*/ppm: 206.7, 201.1, 200.0, 168.5, 150.9, 142.6, 142.2, 135.9, 135.8, 134.4, 129.5, 128.8, 128.2, 128.0, 127.6, 124.2, 122.9, 122.8, 117.5, 116.9, 71.6, 58.6, 56.7, 45.8, 41.9, 30.3.

IR (KBr) $\tilde{\nu}$ (cm^{-1}): 1764, 1702, 1458, 1356.

HRMS (ESI) calcd for $C_{28}H_{21}O_5$, $[M+H]^+$ 437.1384, found: 437.1387.

c. Typical procedure for the synthesis of intermediate *rac*-4aa

2-(3-oxo-2-(2-oxo-2*H*-chromen-3-yl)-1,3-diphenylpropyl)-1*H*-indene-1,3(2*H*)-dione (*rac*-4aa)



To a glass vial equipped with a magnetic stir bar were charged **1a** (1.0 mmol, 264.3 mg), **2a** (1.1 equiv., 257.7 mg), DABCO (0.2 equiv., 22.6 mg) and CH_2Cl_2 (5.0 mL) then stirred at 30 °C for 21 h. Reaction was quenched by the addition of 0.1 M $HCl_{(aq)}$ and the aqueous layer was extracted with CH_2Cl_2. The combined organic layers were dried over anhydrous $MgSO_4$. The filtrate was concentrated in vacuo, then purified by flash column chromatography over silica gel with EtOAc/hexanes/CH_2Cl_2 (1/8/1) as eluent to give the pure compound **4aa** in 17% yield (84.7 mg) as a white solid.

R_f 0.28 (CH_2Cl_2/hexanes = 1/1); mp.: 190.4-191.2 °C.

^{1}H NMR (400 MHz, $CDCl_3$): δ/ppm, 8.35 (d, J = 8.6 Hz, 2H), 7.85 (d, J = 7.4 Hz, 1H), 7.80 (s, 1H), 7.55-7.75 (m, 4H), 7.51 (t, J = 7.5 Hz, 2H), 7.37 (t, J = 8.9 Hz, 2H), 7.13-7.24 (m, 3H), 7.09 (d, J = 8.2 Hz, 1H), 6.97 (t, J = 7.3 Hz, 2H), 6.88-6.94 (m, 1H), 6.64 (d, J = 12.0 Hz, 1H), 4.49 (dd, J = 11.9, 4.0 Hz, 1H). 3.51 (d, J = 4.2 Hz, 1H).

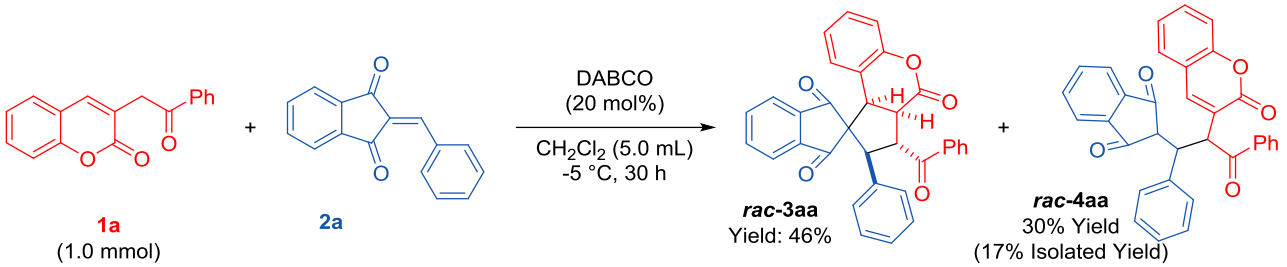
^{13}C NMR (100 MHz, $CDCl_3$): δ/ppm, 200.1, 199.1, 198.8, 160.6, 153.0, 142.6, 142.3, 141.1, 136.4, 136.1, 135.4, 135.3, 134.0, 131.5, 129.3,129.2, 128.9, 128.2, 127.8, 127.3, 125.5, 124.3, 122.9, 122.8, 119.0, 116.3 55.9, 48.2, 45.1.

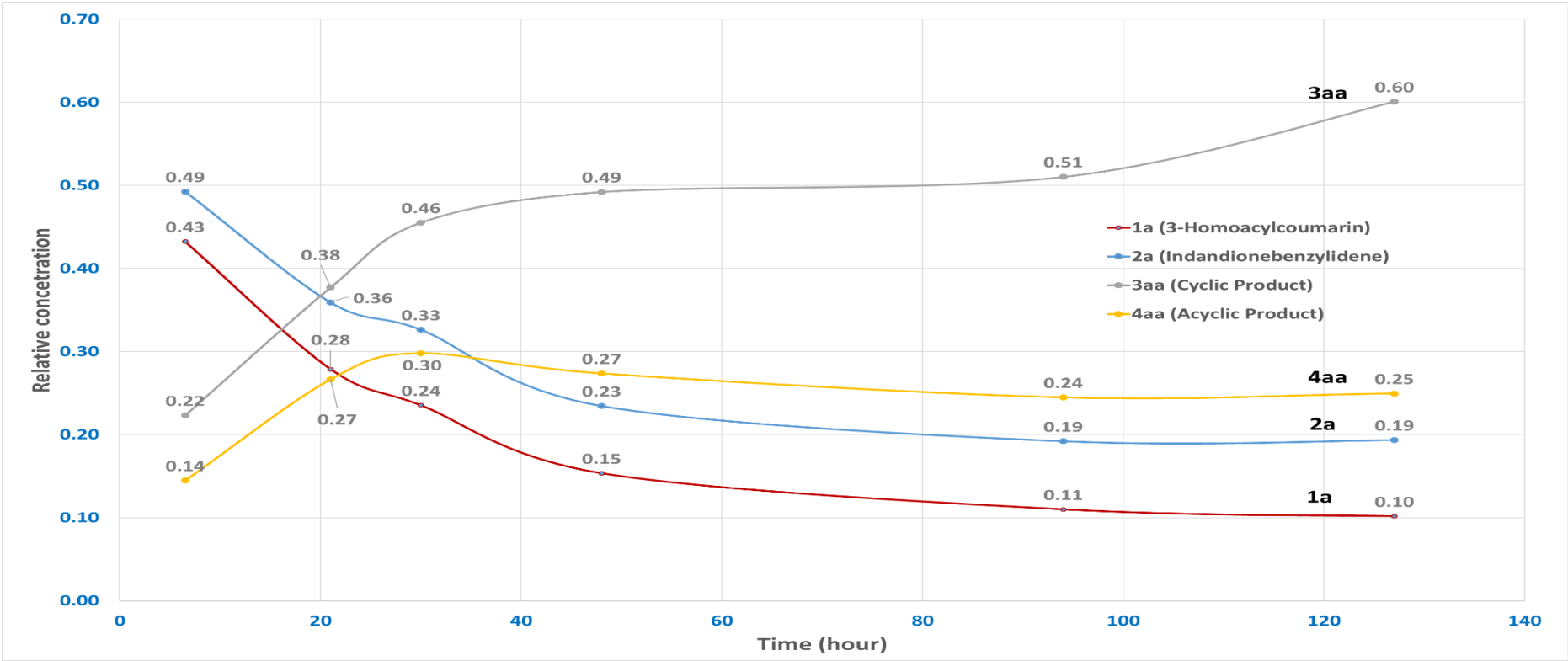
IR (KBr) 2927, 2857, 2360, 1706, 1524, 1259, 1169 cm^{-1}.

HRMS (ESI) for $C_{33}H_{22}O_5Na$ $[M + Na]^+$ (521.1359), found 521.1370.

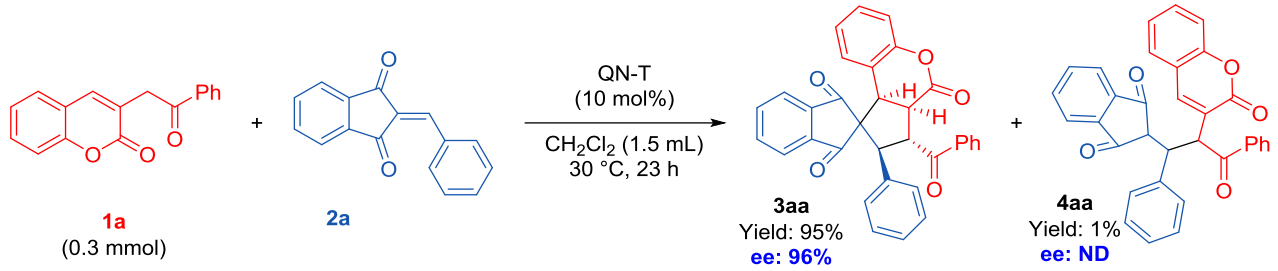
Reaction progress monitoring: Time vs Consumption plots

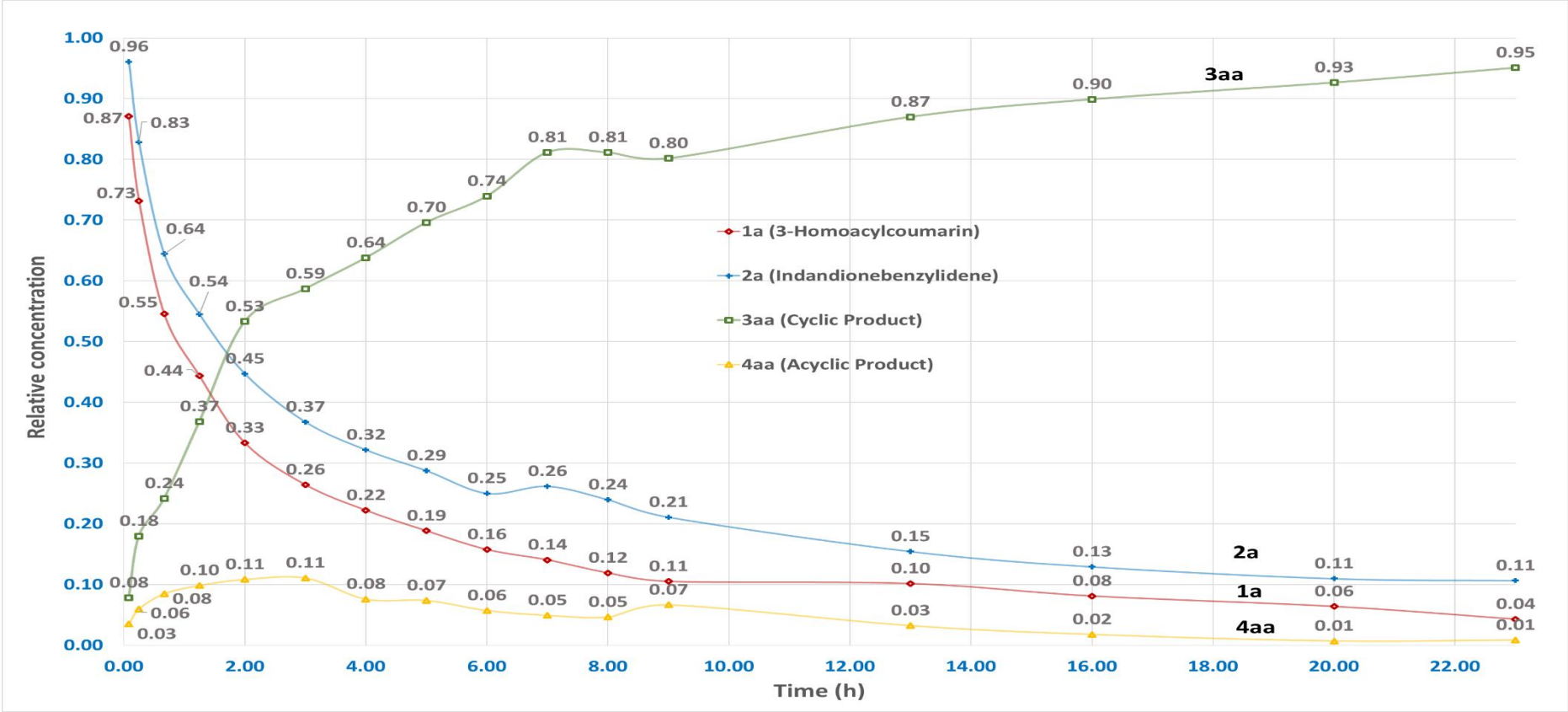
a. Time vs Consumption plot for the reaction of 1a and 2a at -5 °C under racemic conditions



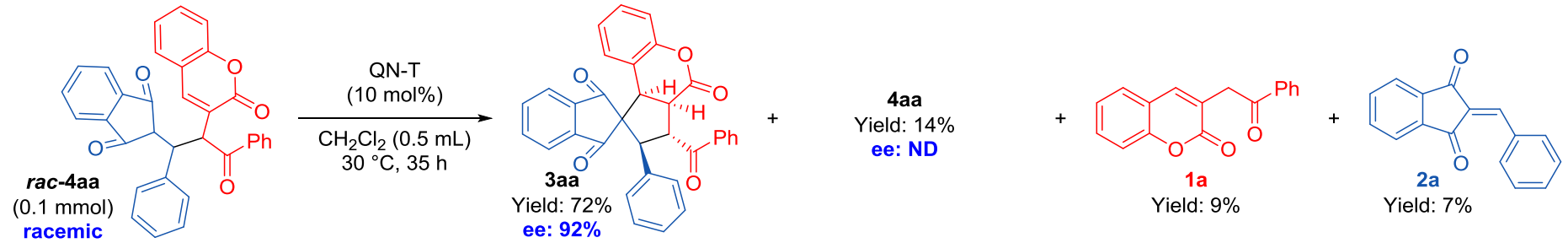


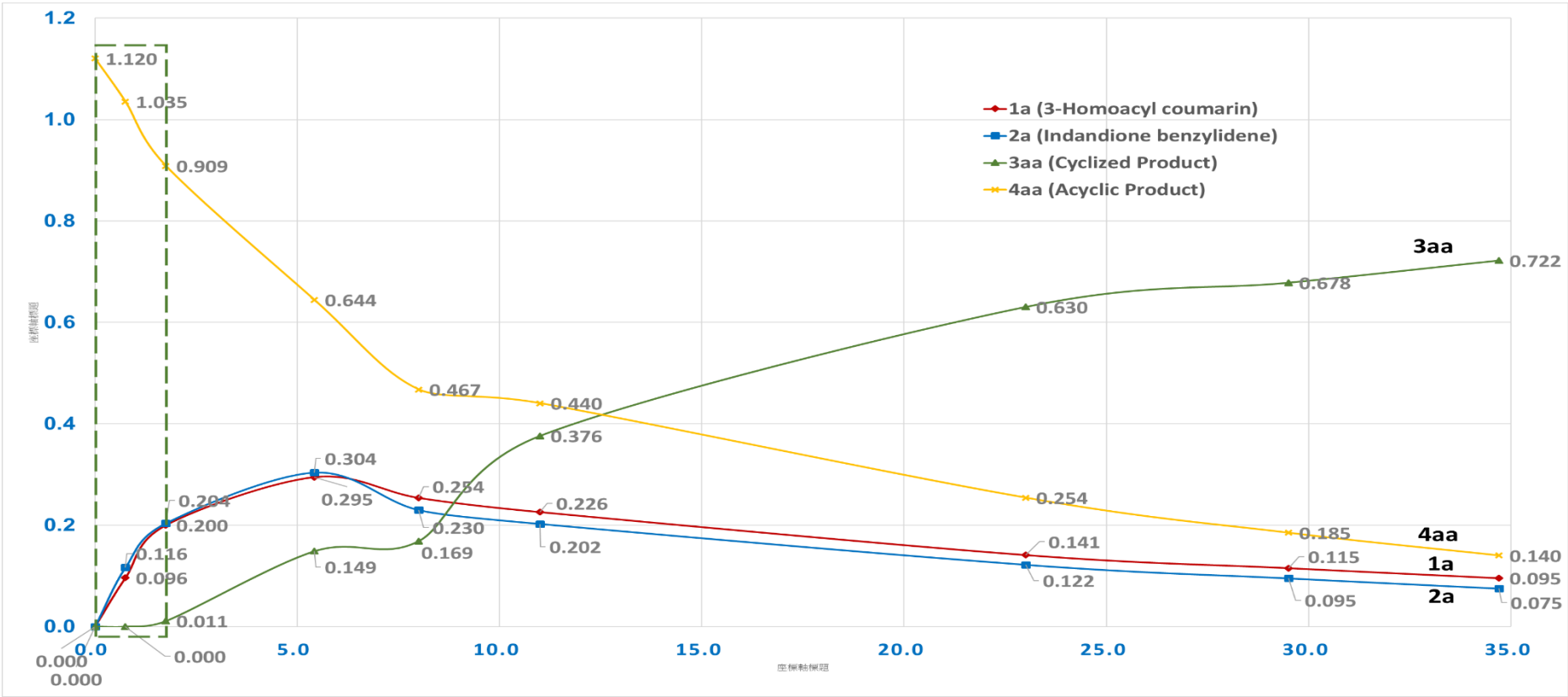
b. Time vs consumption plot for the reaction of 1a and 2a under optimized conditions (30 °C)



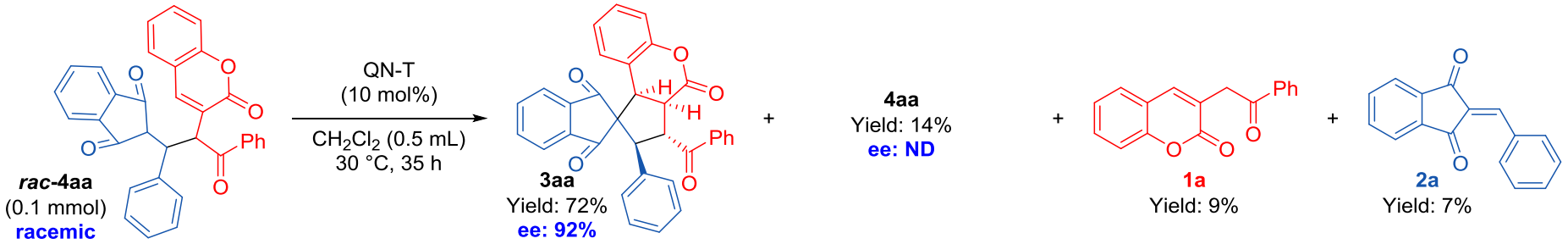


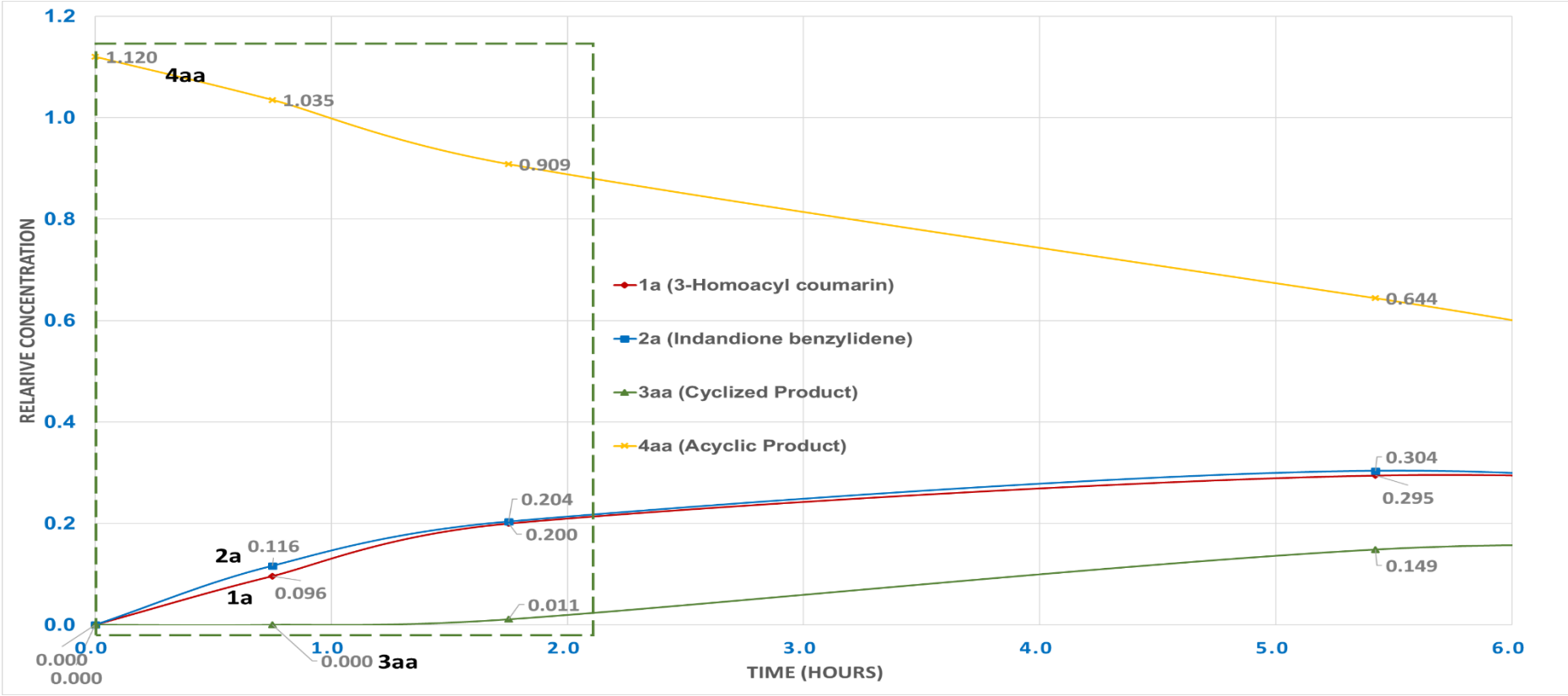
c. Time vs Consumption plot for the reaction of 4aa **with** QN-T **under optimized conditions (30 °C) [t=0 to 35 h]**





c. Time vs Consumption plot for the reaction of 4aa with QN-T under optimized conditions (30 °C) [t=0 to 6 h]

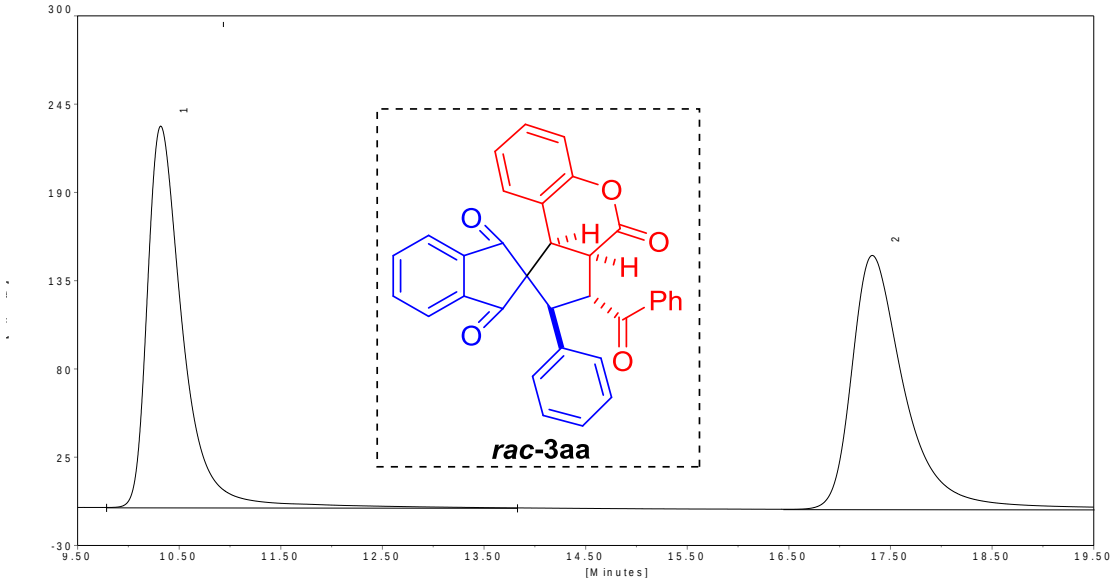




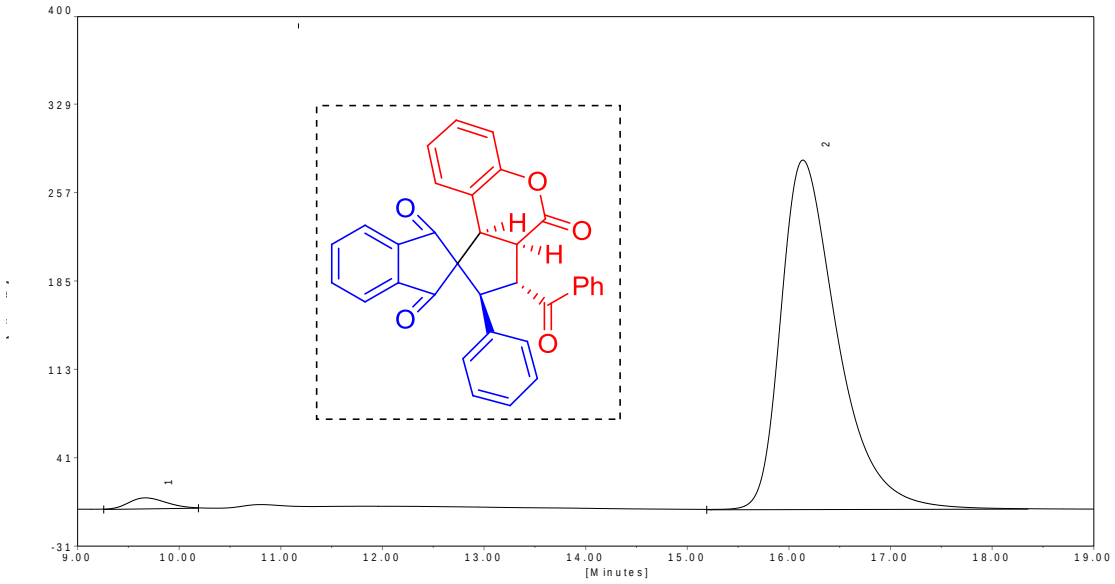
HPLC CHROMATOGRAMS FOR PRODUCTS:

HPLC chromatogram for **3aa**

Column: Chiralpak IB	Flow rate: 1.0 mL/min
Solvent: n-Hexane:IPA=70:30	Detector: UV 248 nm



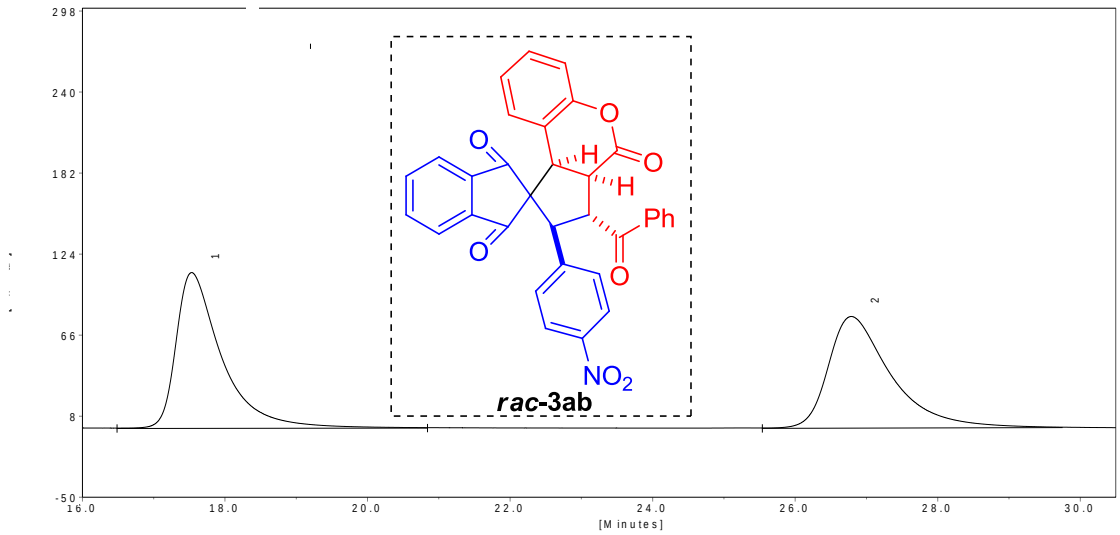
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
10.32	237.68	5803.63	49.9530
17.32	158.20	5814.56	50.0470



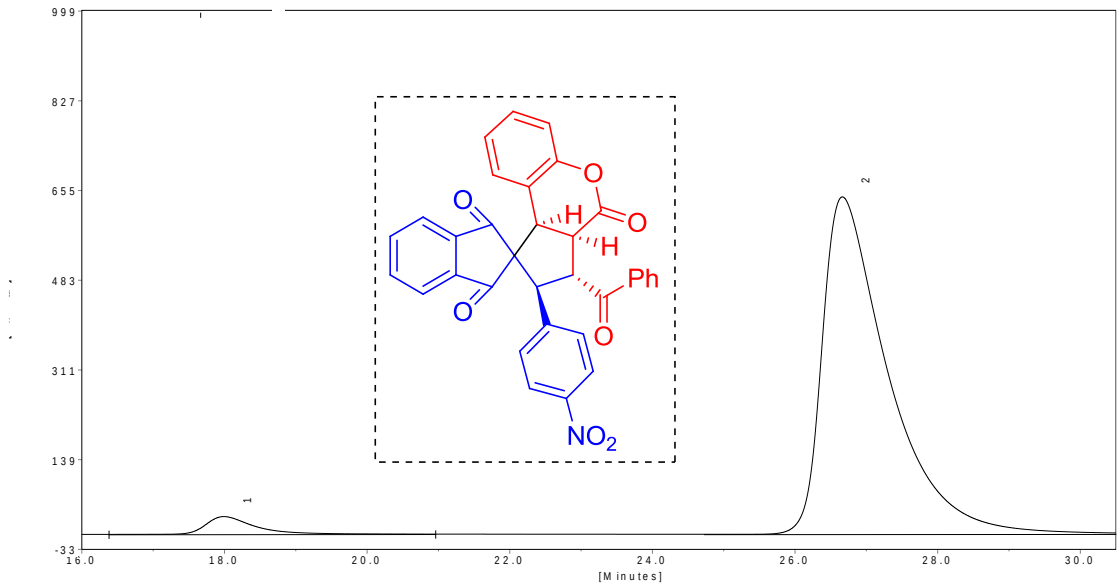
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
9.67	8.69	216.95	1.9404
16.14	284.34	10963.73	98.0596

HPLC chromatogram for **3ab**

Column: Chiralpak IB	Flow rate: 1.0 mL/min
Solvent: n-Hexane:IPA=70:30	Detector: UV 248 nm



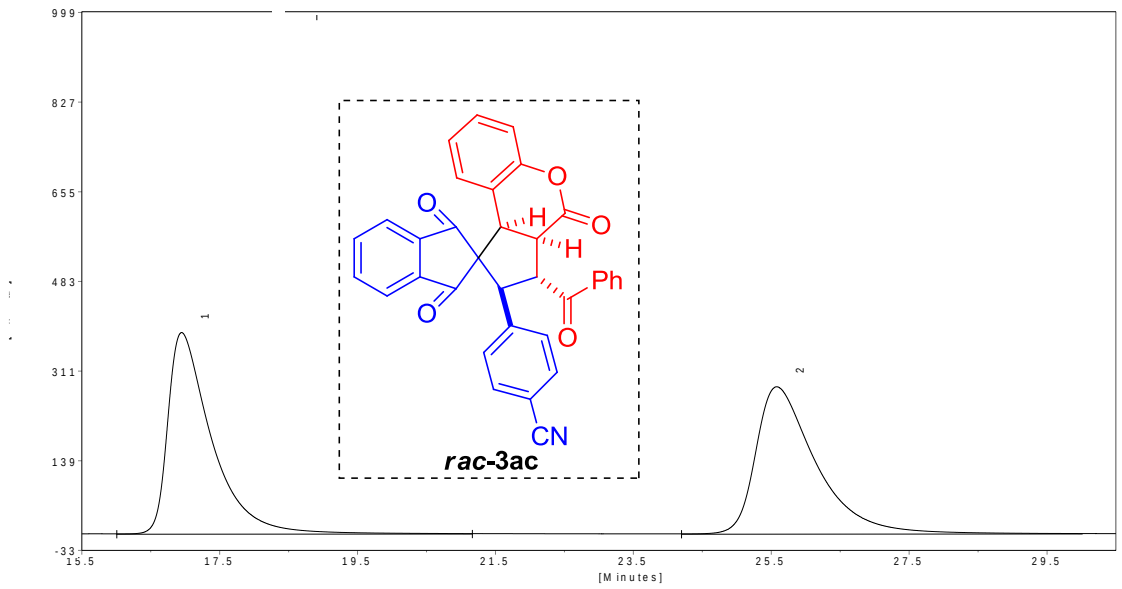
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
17.53	111.23	4923.25	50.4649
26.79	79.62	4832.55	49.5351



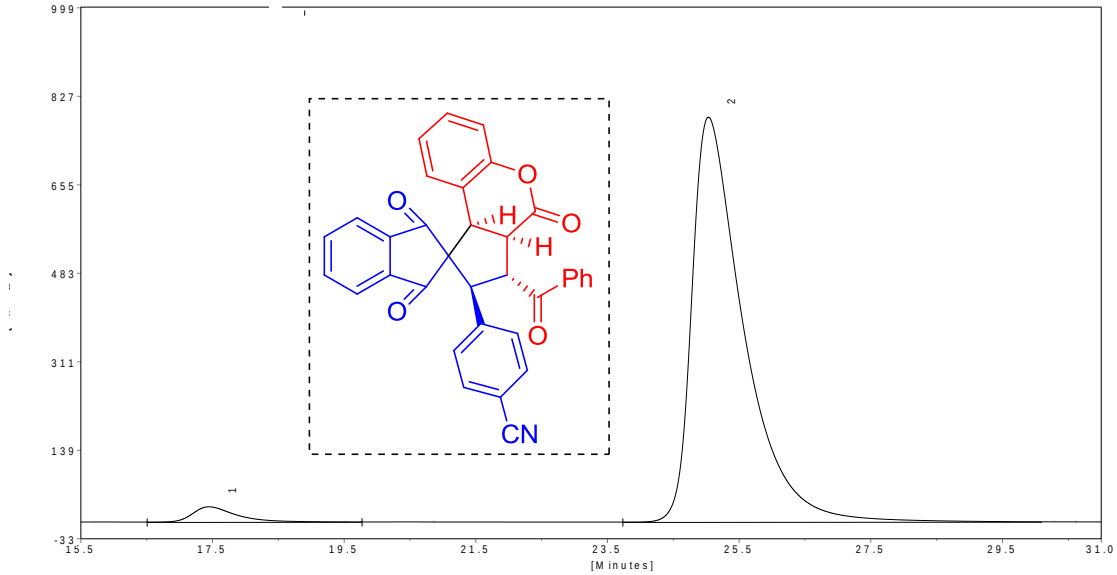
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
17.99	33.92	1623.02	3.7536
26.66	646.75	41615.74	96.2464

HPLC chromatogram for **3ac**

Column: Chiralpak IB	Flow rate: 1.0 mL/min
Solvent: n-Hexane:IPA=70:30	Detector: UV 248 nm



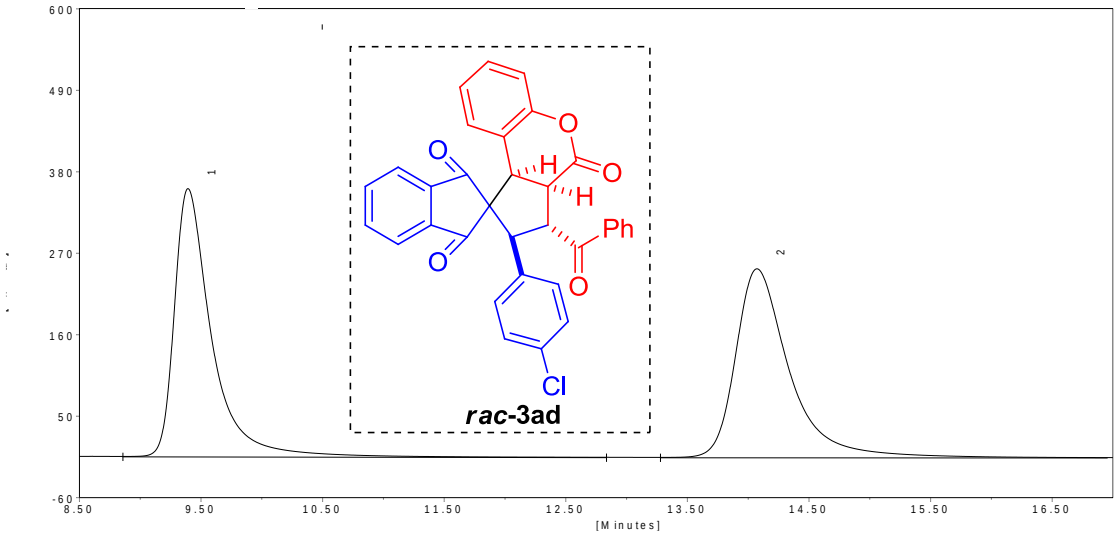
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
16.95	386.02	17020.90	49.8601
25.58	281.98	17116.44	50.1399



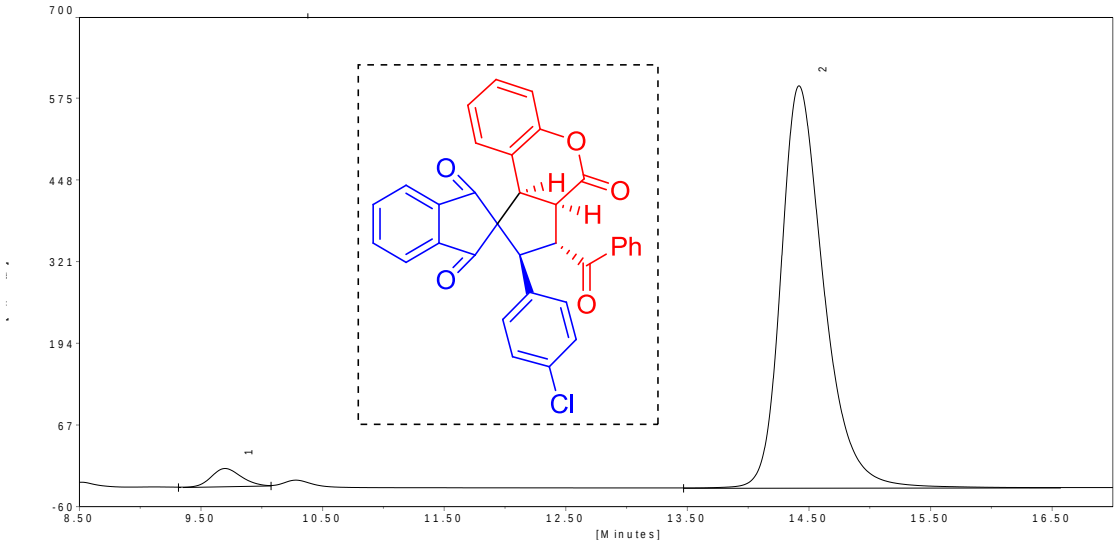
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
17.45	29.58	1353.92	3.0540
25.03	786.97	42978.80	96.9460

HPLC chromatogram for **3ad**

Column: Chiralpak IB	Flow rate: 1.0 mL/min
Solvent: n-Hexane:IPA=70:30	Detector: UV 248 nm



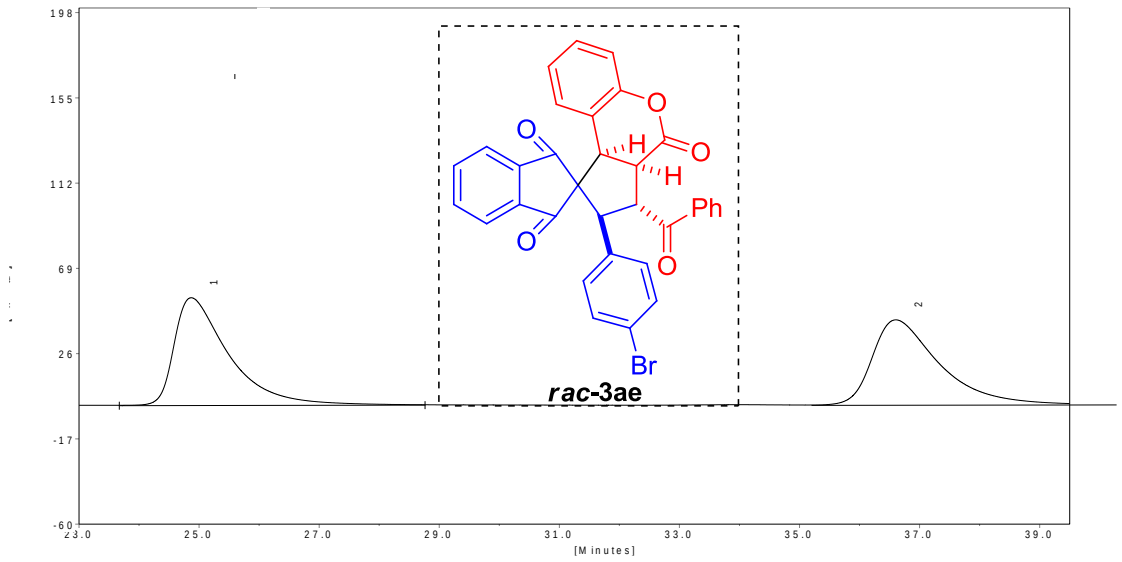
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
9.39	361.74	7711.92	49.6194
14.07	254.70	7830.23	50.3806



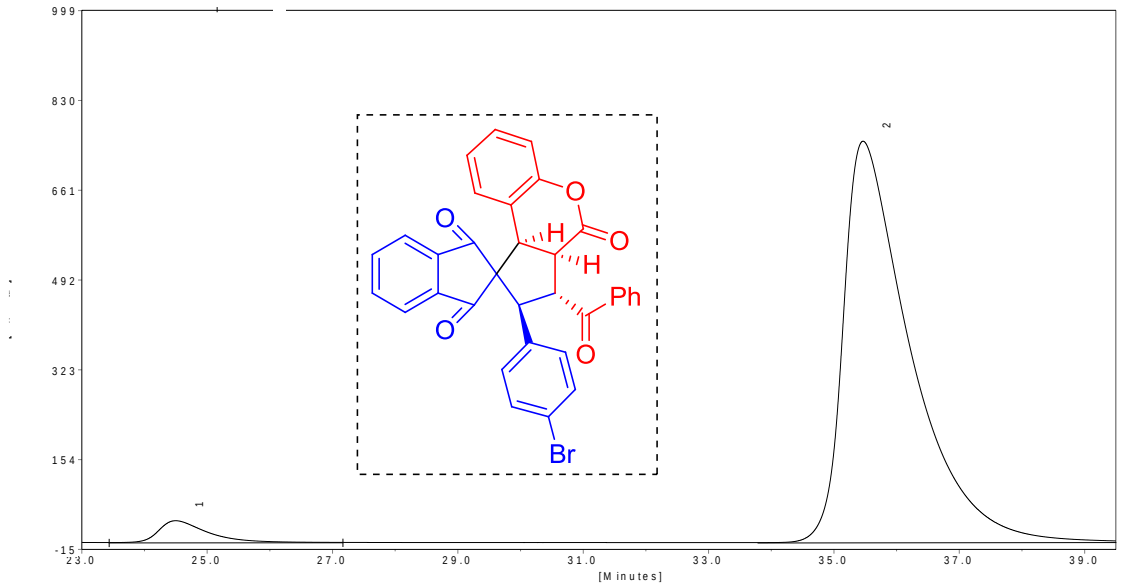
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
9.70	27.73	461.04	2.9333
14.42	624.87	15256.07	97.0667

HPLC chromatogram for **3ae**

Column: Chiralpak IB	Flow rate: 1.0 mL/min
Solvent: n-Hexane:IPA=90:10	Detector: UV 248 nm



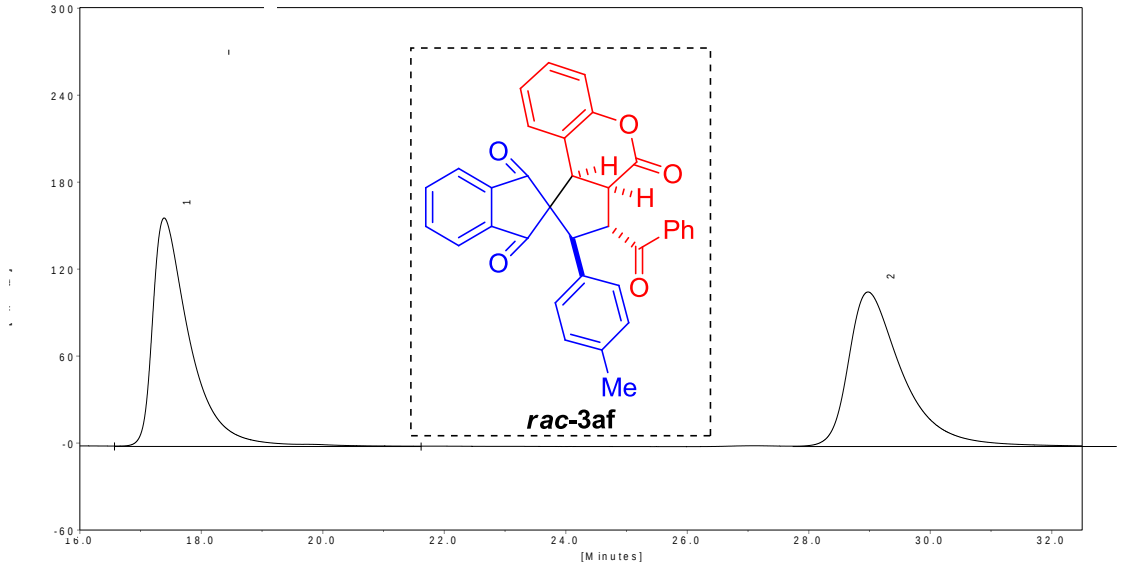
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
24.87	54.18	3404.49	50.5324
36.61	42.89	3332.76	49.4676



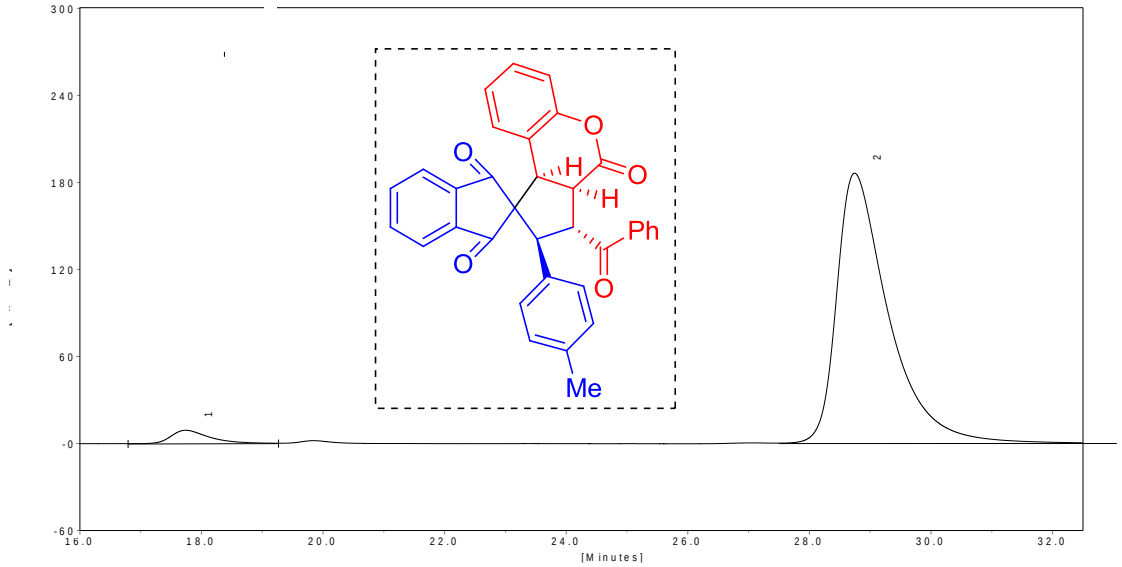
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
24.50	41.03	2187.21	3.7382
35.47	755.31	56322.84	96.2618

HPLC chromatogram for **3af**

Column: Chiralpak IB	Flow rate: 1.0 mL/min
Solvent: n-Hexane:IPA = 85:15	Detector: UV 248 nm



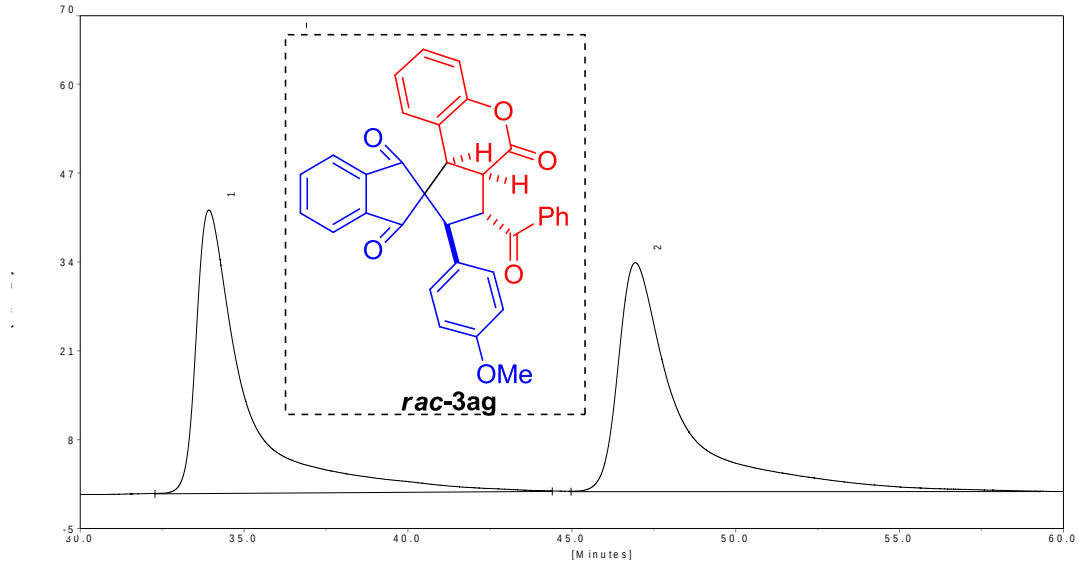
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
17.39	157.36	6625.99	50.3523
28.97	106.39	6533.26	49.6477



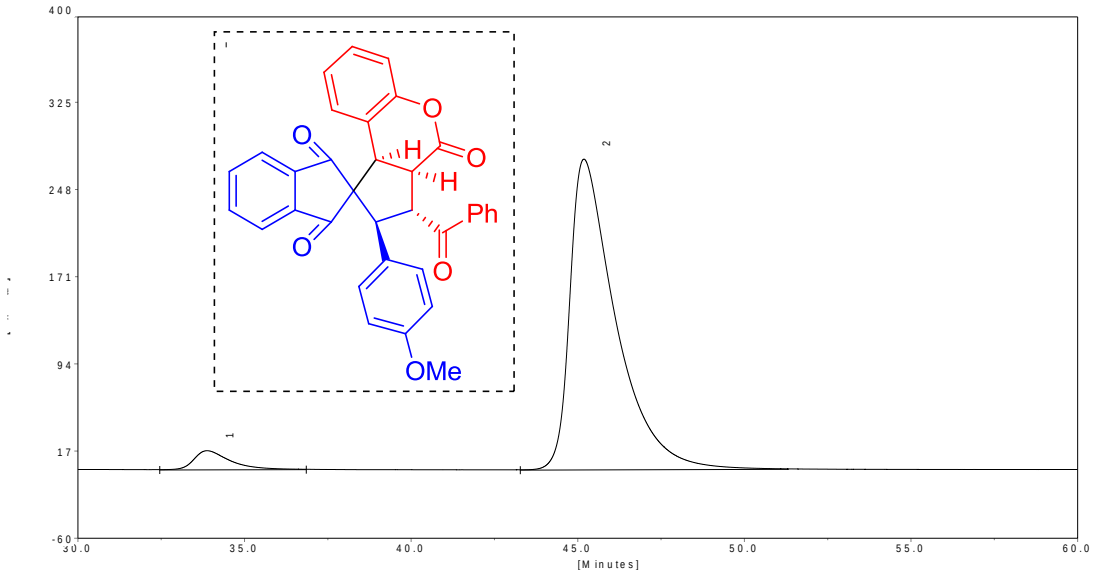
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
17.74	9.13	391.39	3.4375
28.74	186.13	10994.50	96.5625

HPLC chromatogram for **3ag**

Column: Chiralpak IB	Flow rate: 1.0 mL/min
Solvent: n-Hexane:IPA = 90:10	Detector: UV 248 nm



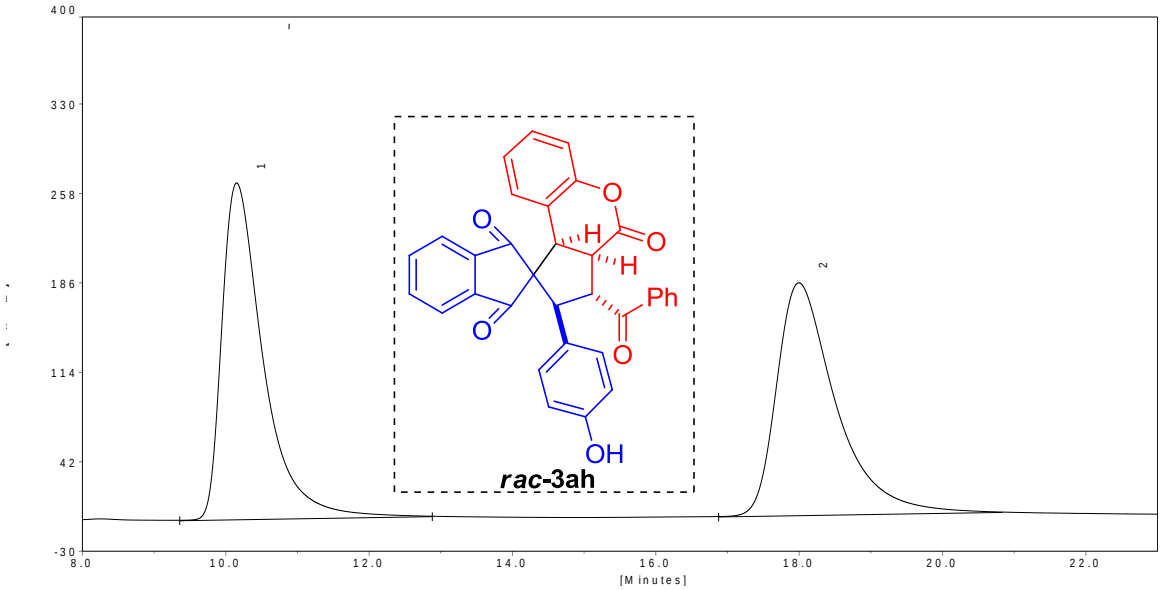
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
33.93	41.42	4318.48	49.8986
46.94	33.45	4336.02	50.1014



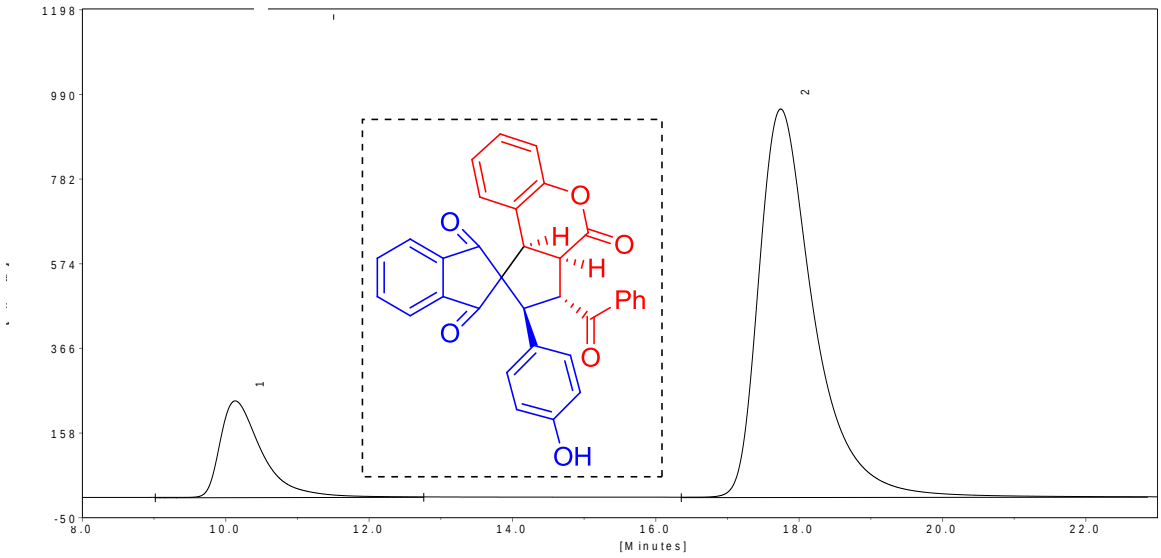
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
33.88	16.60	1224.25	4.4721
45.19	274.18	26150.81	95.5279

HPLC chromatogram for **3ah**

Column: Chiralpak IB	Flow rate: 1.0 mL/min
Solvent: n-Hexane:IPA=70:30	Detector: UV 248 nm



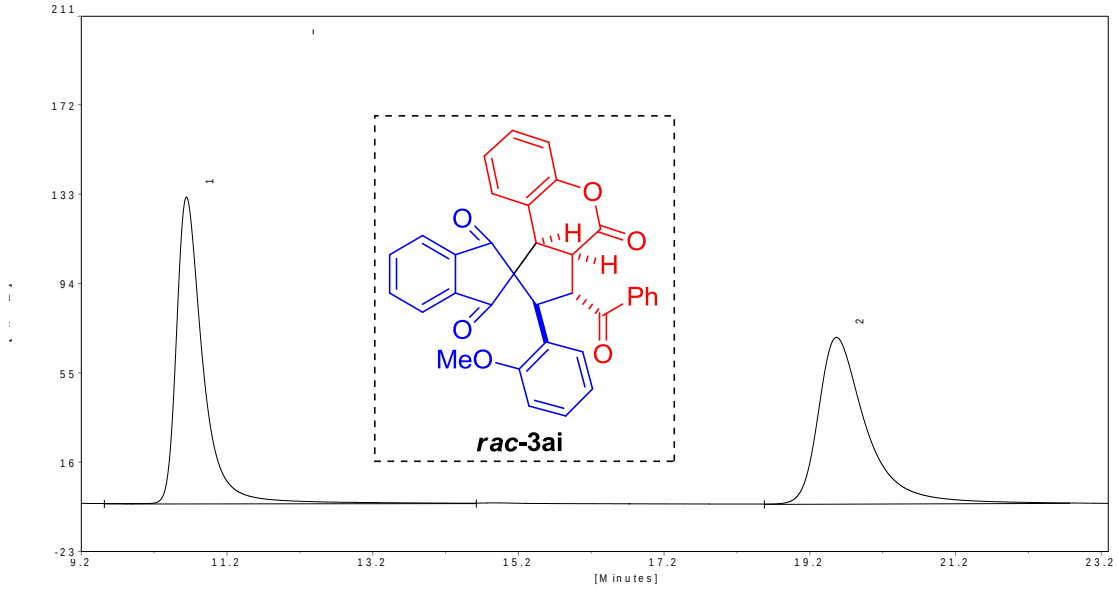
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
10.15	270.79	10846.58	50.3131
18.00	187.13	10711.58	49.6869



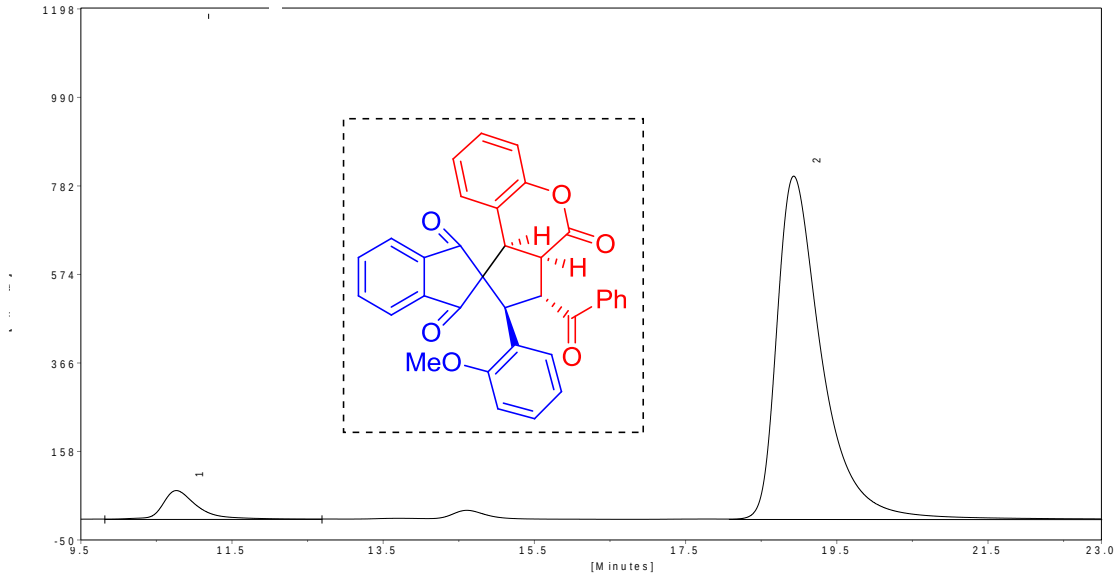
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
10.13	236.80	9486.88	15.7100
17.74	953.63	50900.65	84.2900

HPLC chromatogram for **3ai**

Column: Chiralpak IB	Flow rate: 1.0 ml/min
Solvent: n-Hexane:IPA = 70:30	Detector: UV 248 nm



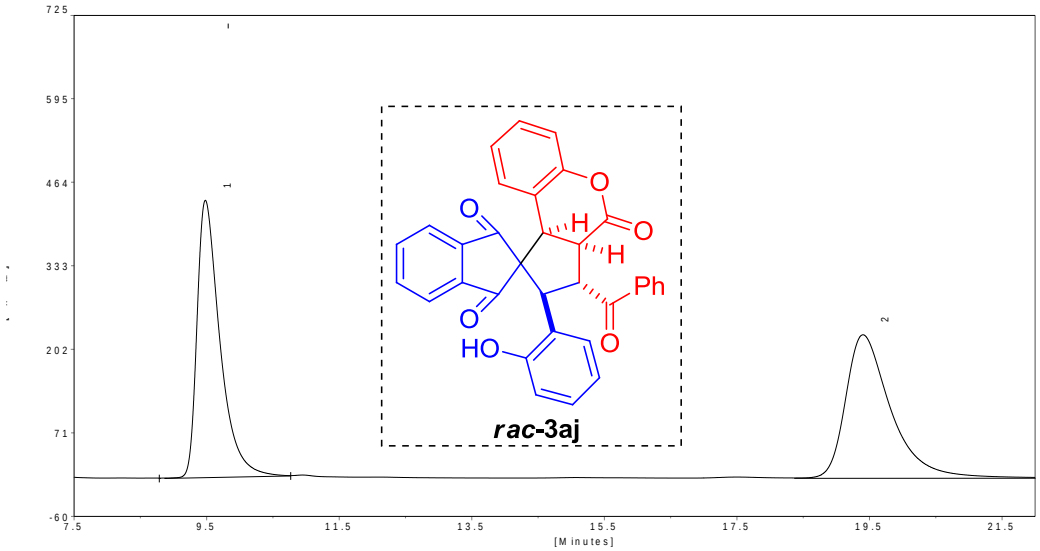
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
10.69	133.74	3378.36	50.5450
19.61	72.62	3305.51	49.4550



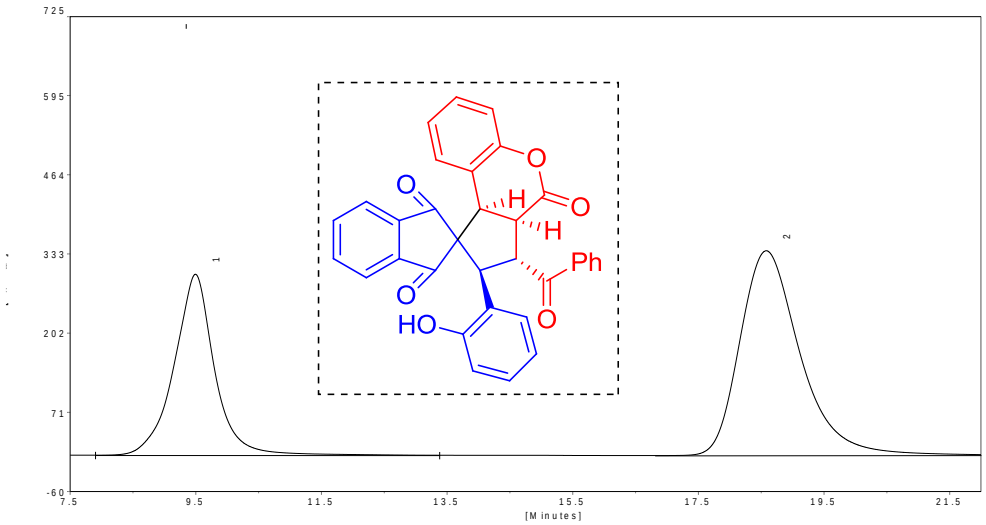
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
10.76	67.01	2092.05	5.9078
18.93	806.05	33319.35	94.0922

HPLC chromatogram for **3aj**

Column: Chiralpak IB	Flow rate: 1.0 ml/min
Solvent: n-Hexane:IPA = 70:30	Detector: UV 248 nm



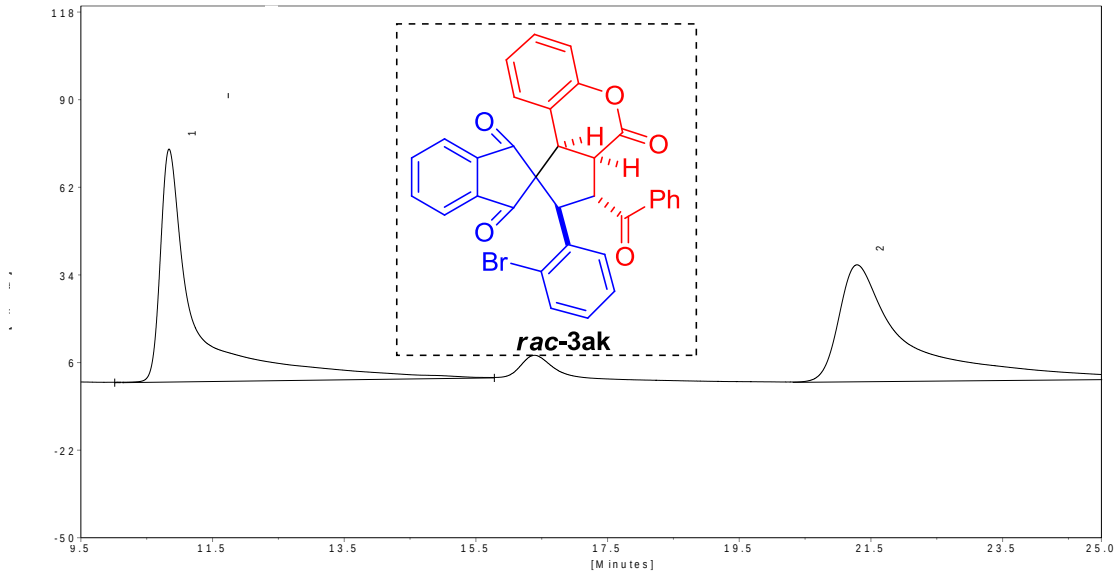
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
9.48	434.23	10409.67	49.5206
19.40	224.49	10611.22	50.4794



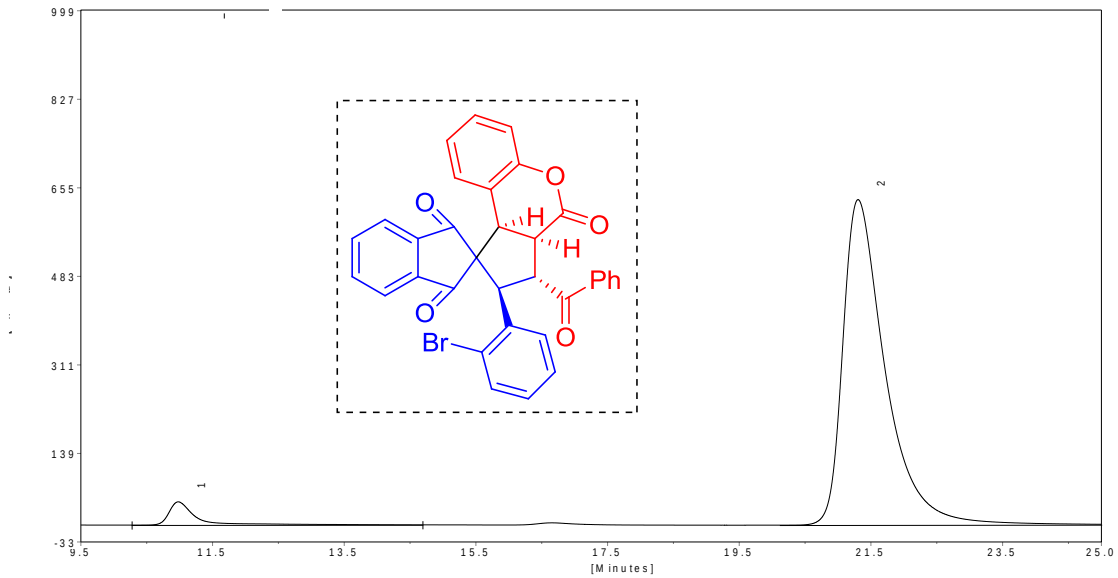
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
9.50	299.29	13412.47	37.2615
18.58	338.61	22583.07	62.7385

HPLC chromatogram for **3ak**

Column: Chiralpak IB	Flow rate: 1.0 ml/min
Solvent: n-Hexane:IPA = 70:30	Detector: UV 248 nm



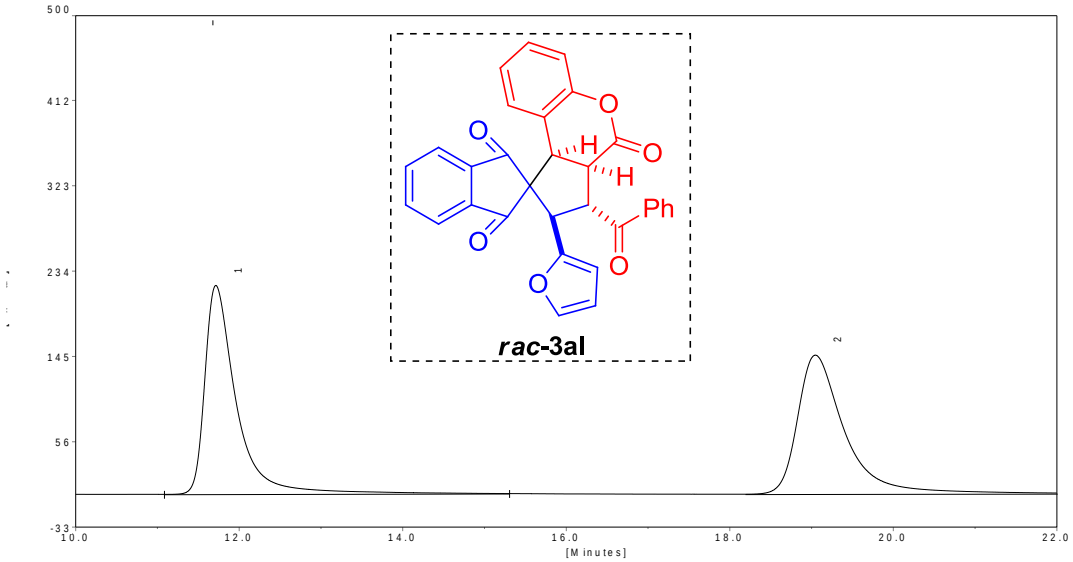
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
10.84	74.31	2740.08	49.5064
21.29	37.34	2794.71	50.4936



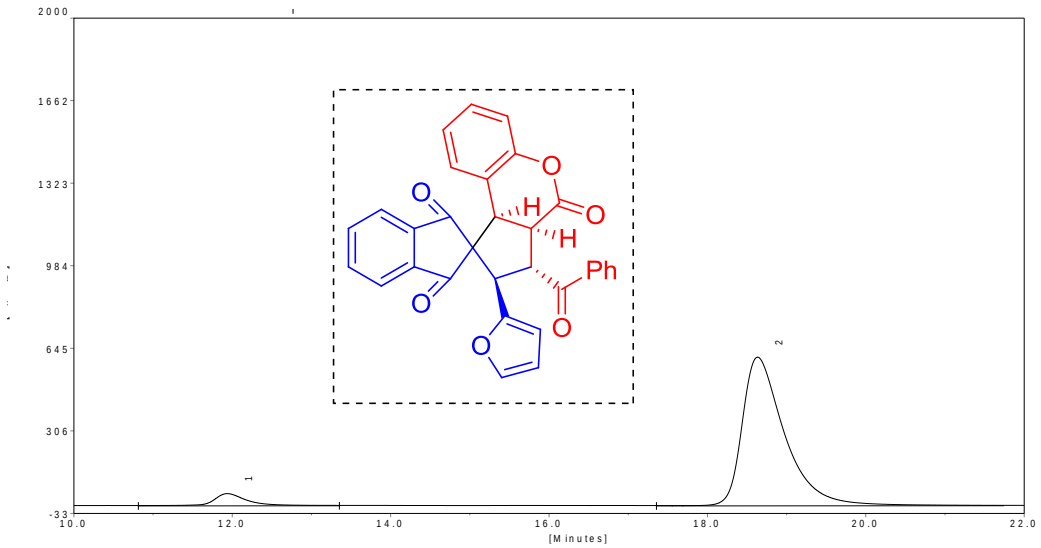
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
10.98	45.14	1321.93	4.4504
21.30	632.29	28381.84	95.5496

HPLC chromatogram for **3al**

Column: Chiralpak IB	Flow rate: 1.0 mL/min
Solvent: n-Hexane:IPA=70:30	Detector: UV 248 nm



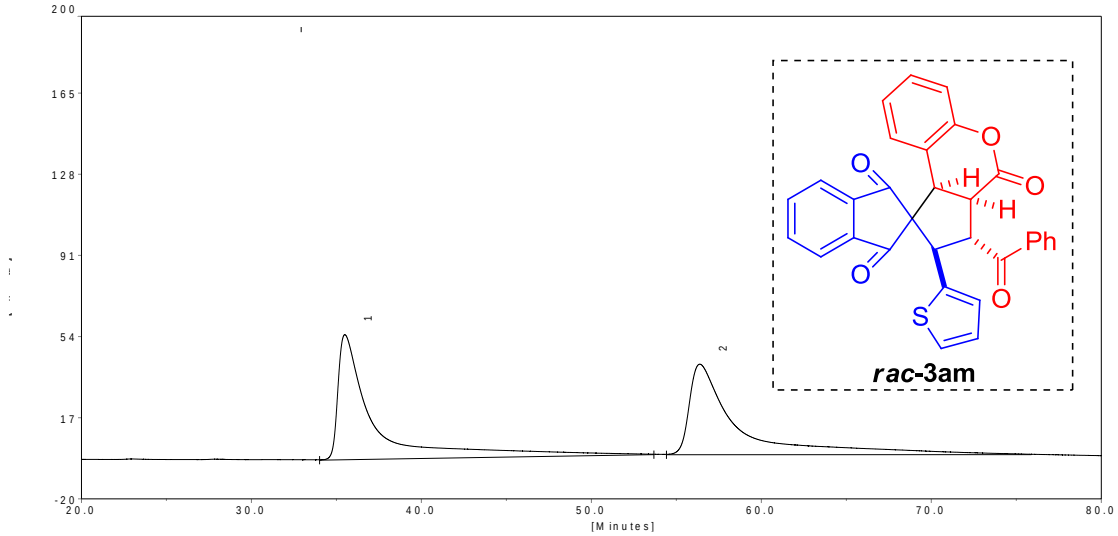
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
11.71	217.76	5880.58	49.9580
19.05	144.98	5890.46	50.0420



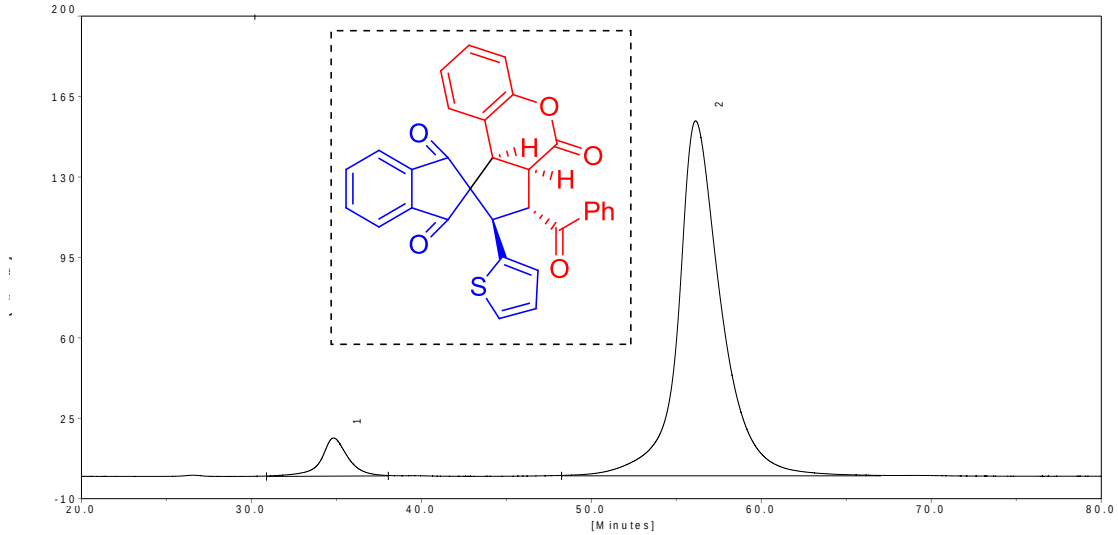
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
11.94	48.69	1338.07	5.6010
18.63	608.98	22551.67	94.3990

HPLC chromatogram for **3am**

Column: Chiralpak IB	Flow rate: 1.0 mL/min
Solvent: n-Hexane:IPA = 90:10	Detector: UV 248 nm



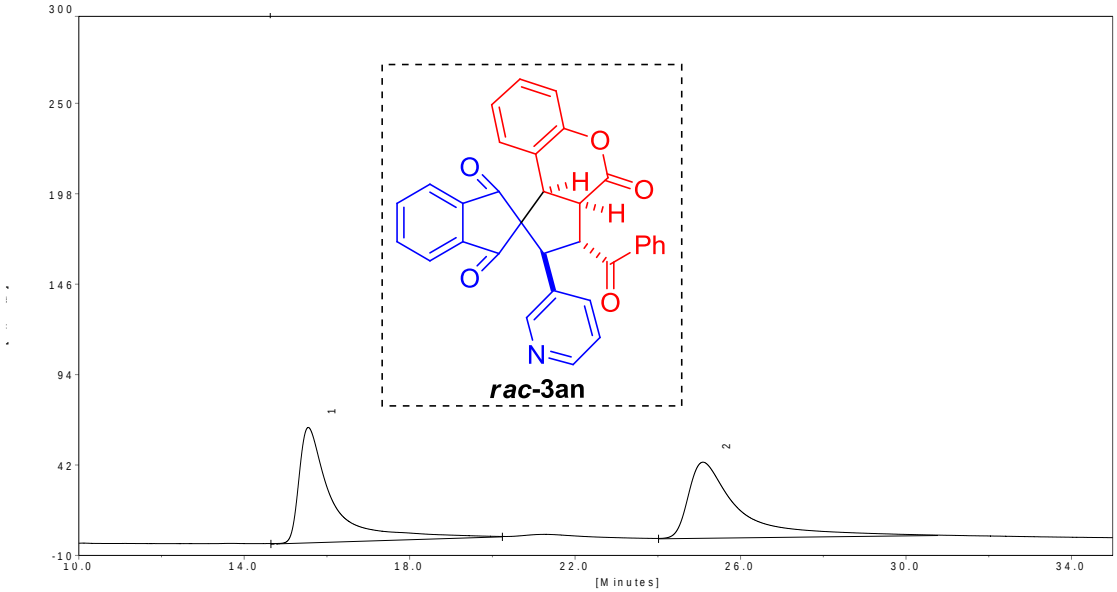
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
35.50	56.87	8242.34	50.7876
56.38	41.13	7986.69	49.2124



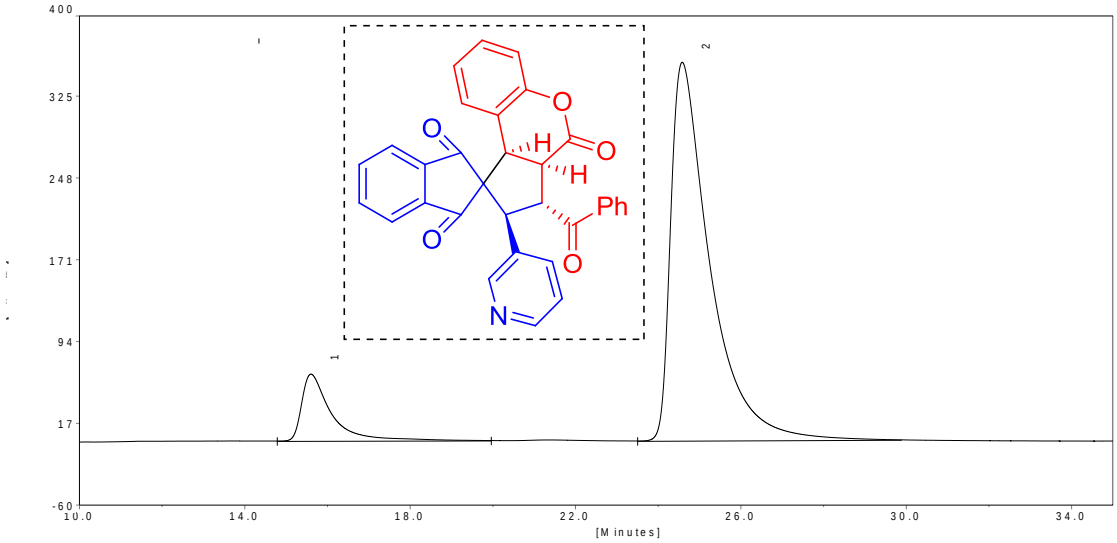
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
34.84	16.46	1774.73	6.0954
56.14	154.30	27340.93	93.9046

HPLC chromatogram for **3an**

Column: Chiralpak IB	Flow rate: 1.0 mL/min
Solvent: n-Hexane:IPA=70:30	Detector: UV 248 nm



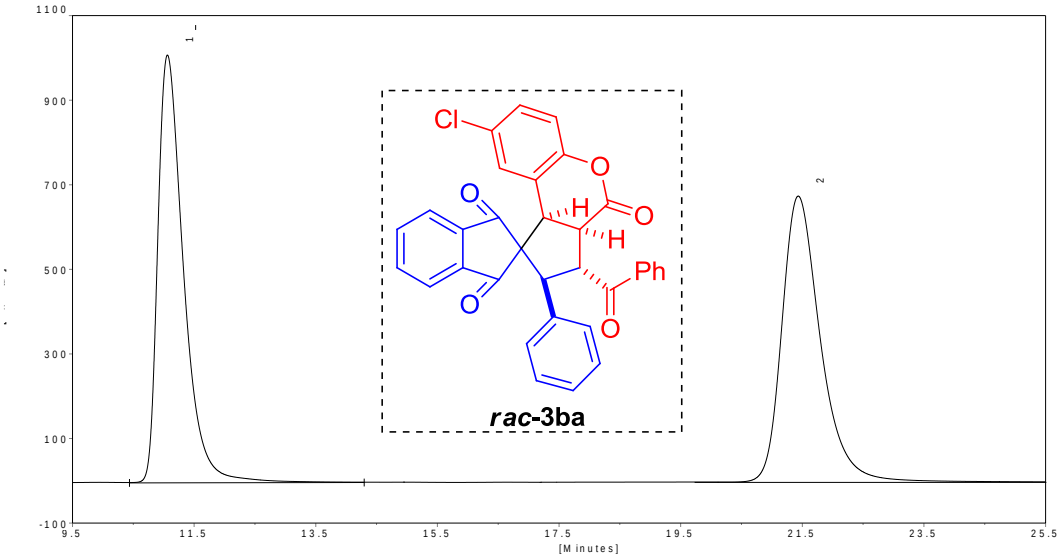
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
15.55	66.11	3700.67	50.4079
25.09	43.55	3640.79	49.5921



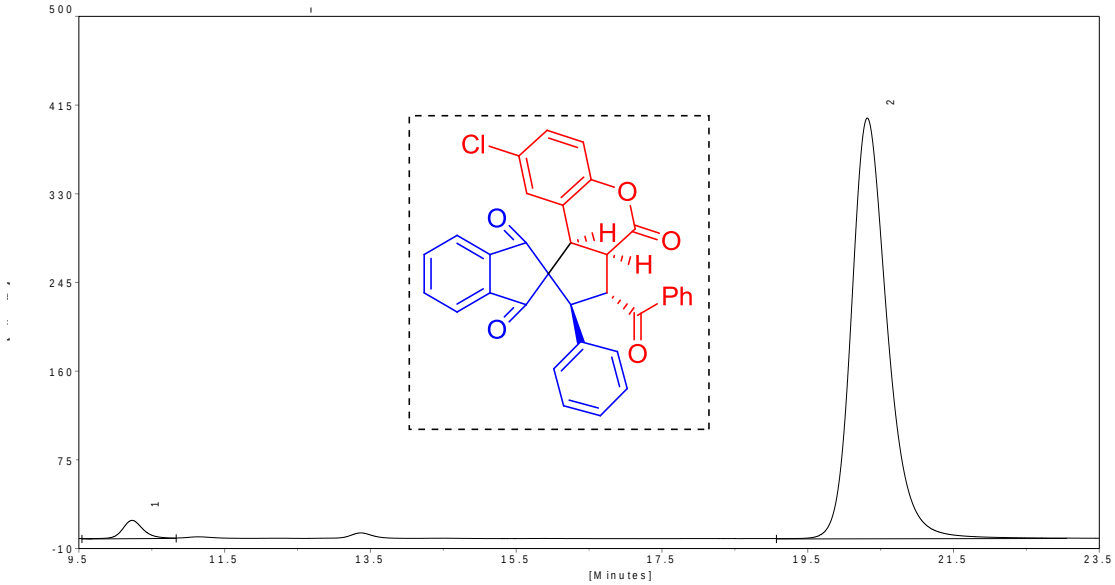
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
15.60	62.92	3128.73	11.7275
24.58	356.30	23549.91	88.2725

HPLC chromatogram for **3ba**

Column: Chiralpak IB	Flow rate: 1.0 mL/min
Solvent: n-Hexane:IPA=70:30	Detector: UV 248 nm



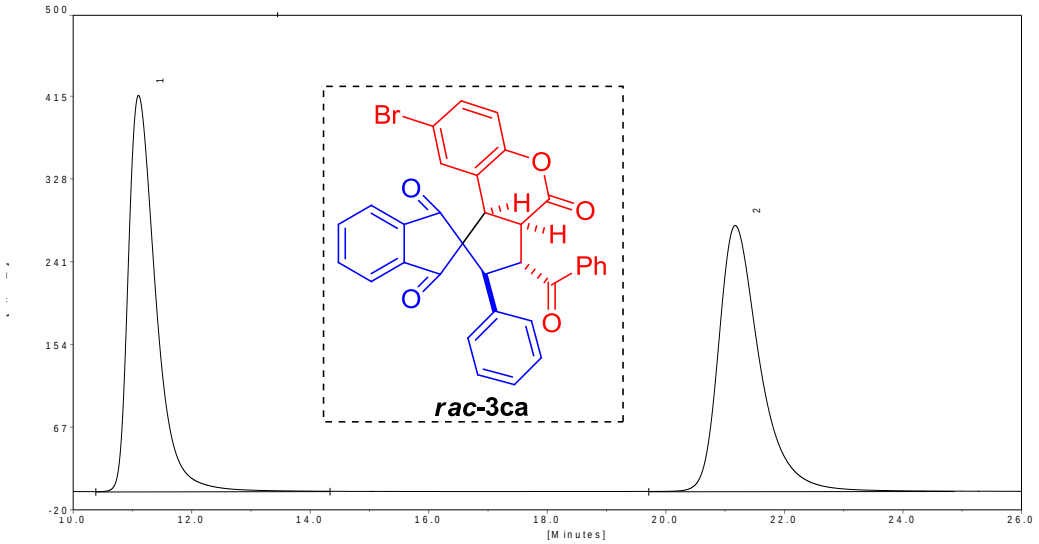
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
11.06	1010.80	29698.60	49.9403
21.43	676.30	29769.63	50.0597



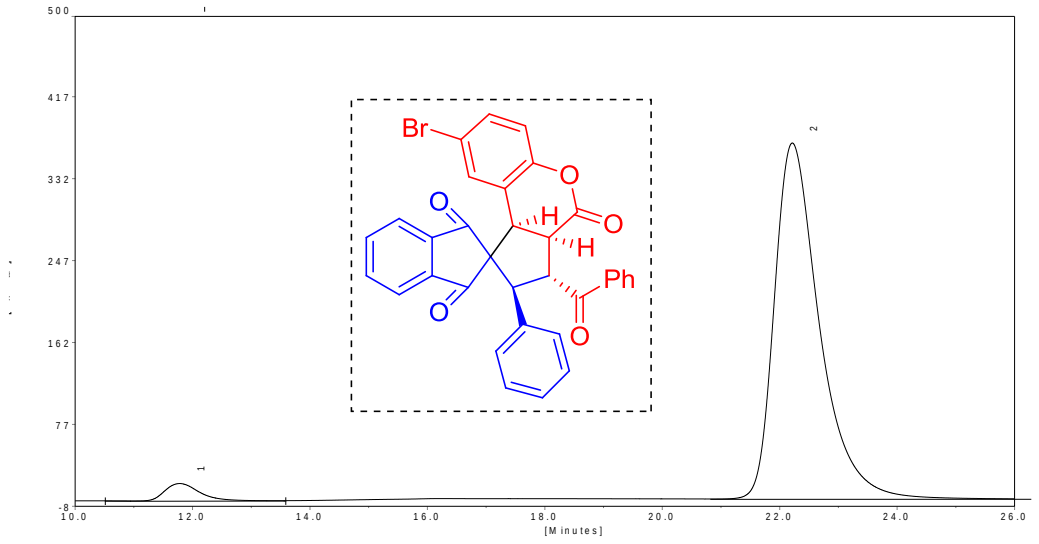
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
10.23	17.16	325.51	2.3080
20.32	402.92	13777.90	97.6920

HPLC chromatogram for **3ca**

Column: Chiralpak IB	Flow rate: 1.0 mL/min
Solvent: n-Hexane:IPA=70:30	Detector: UV 248 nm



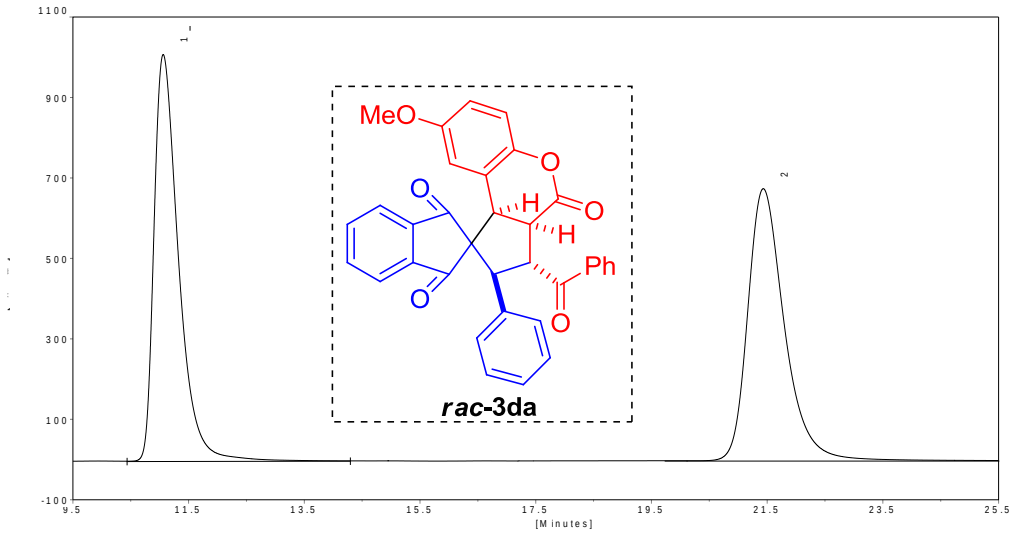
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
11.10	515.92	13471.65	49.9603
21.17	379.06	13493.02	50.0396



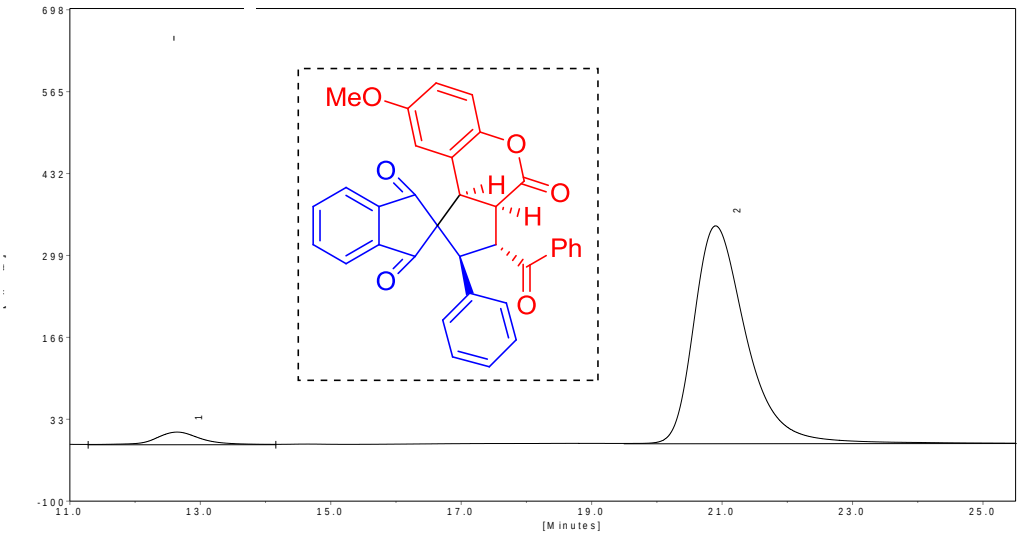
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
11.78	17.87	717.61	3.5224
22.21	369.37	19654.83	96.4776

HPLC chromatogram for **3da**

Column: Chiralpak IB	Flow rate: 1.0 mL/min
Solvent: n-Hexane:IPA=70:30	Detector: UV 248 nm



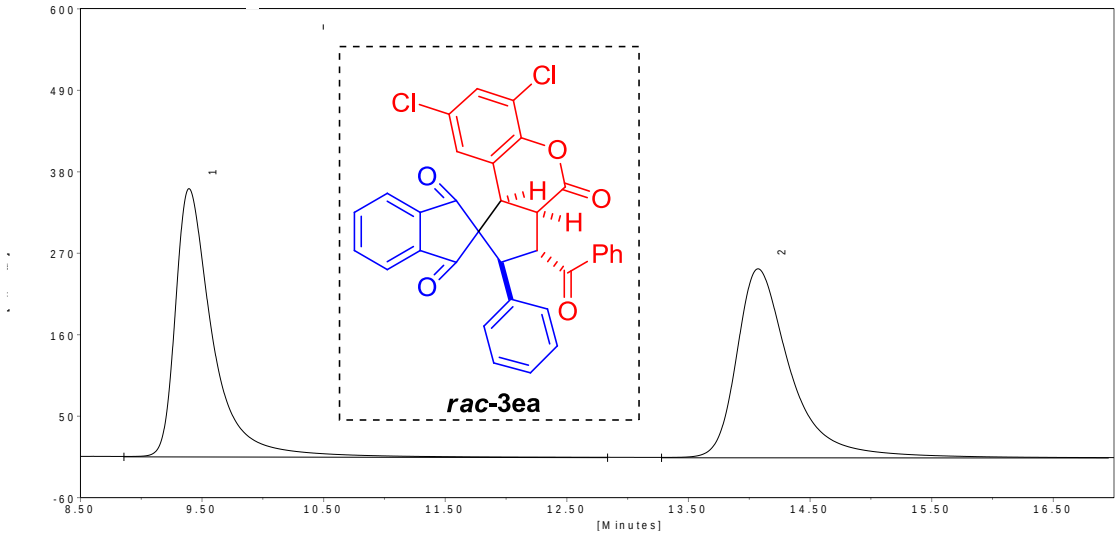
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
11.06	1010.80	29698.60	49.9403
21.43	676.30	29769.63	50.0597



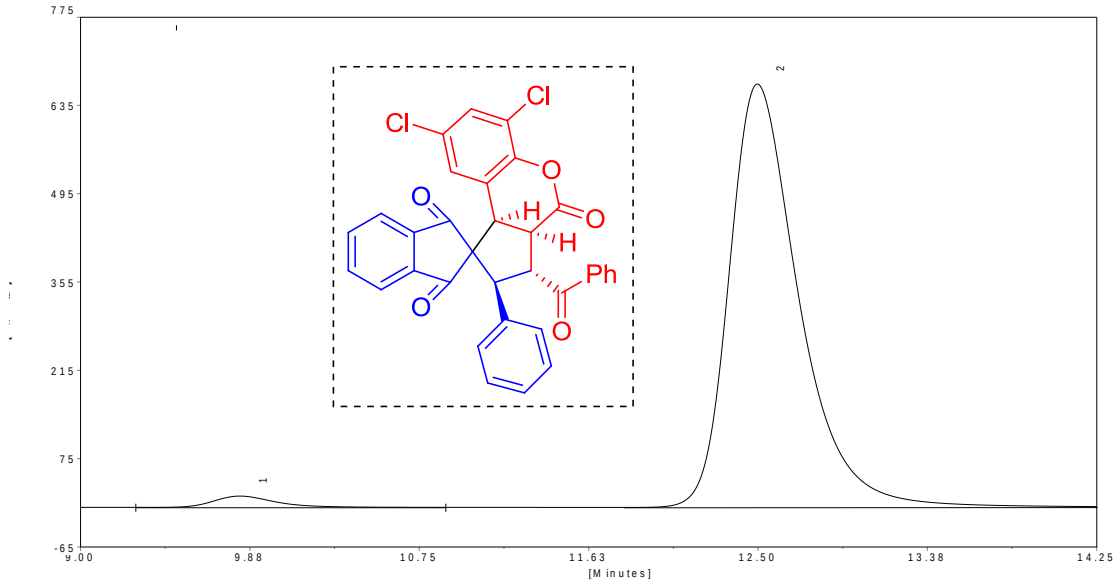
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
12.65	19.99	907.50	4.4237
20.90	353.21	19607.16	95.5763

HPLC chromatogram for **3ea**

Column: Chiralpak IB	Flow rate: 1.0 mL/min
Solvent: n-Hexane:IPA=70:30	Detector: UV 248 nm



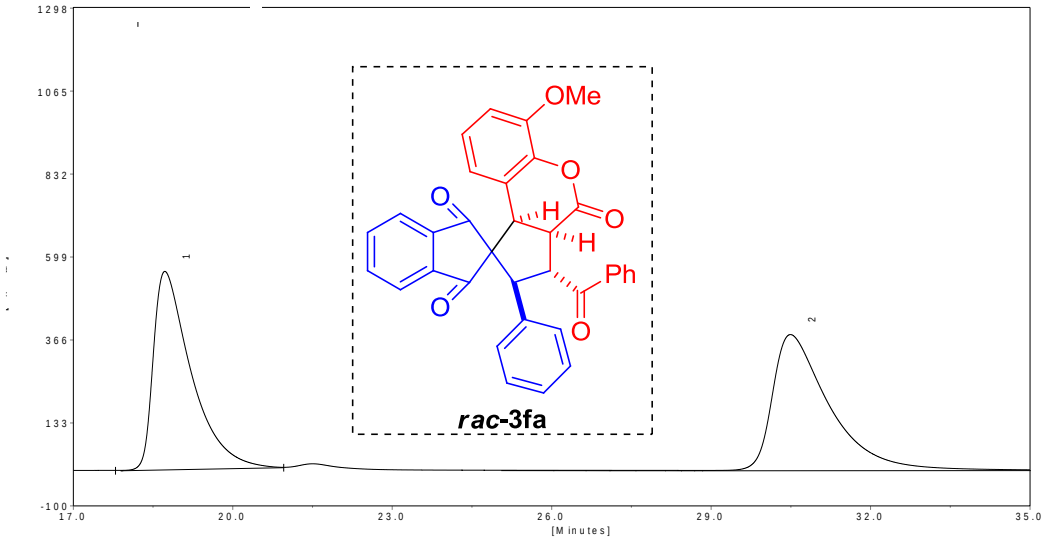
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
9.39	361.74	7711.92	49.6194
12.07	254.70	7830.23	50.3806



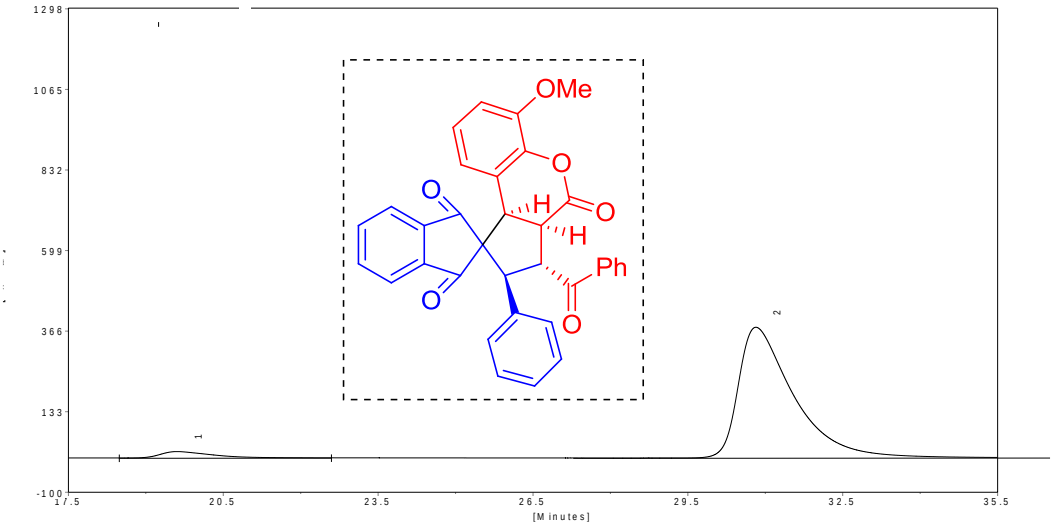
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
9.82	17.86	396.89	2.3003
12.50	670.94	16856.60	97.6997

HPLC chromatogram for **3fa**

Column: Chiralpak IB	Flow rate: 1.0 mL/min
Solvent: n-Hexane:IPA=70:30	Detector: UV 248 nm



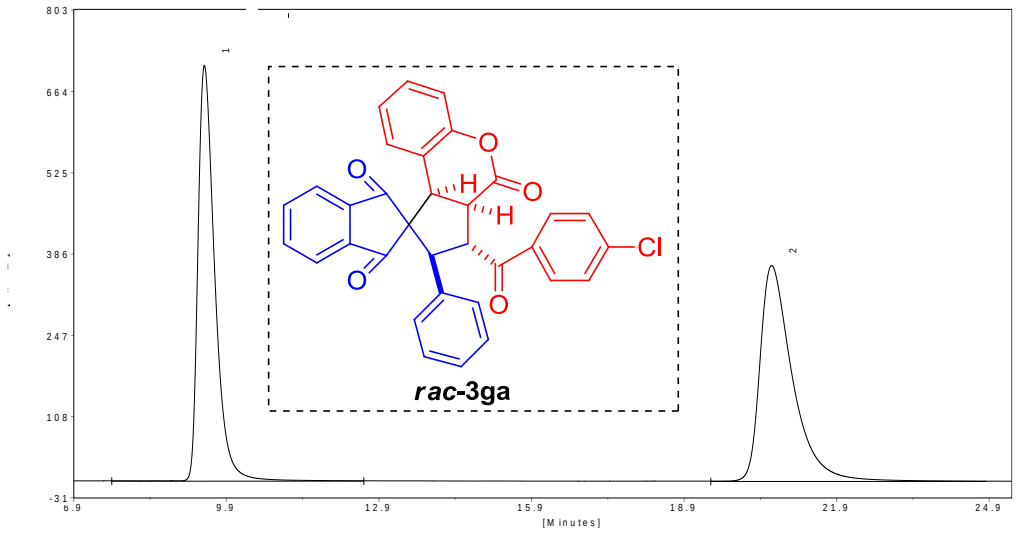
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
18.72	556.39	27417.52	49.2874
30.49	381.02	28210.37	50.7126



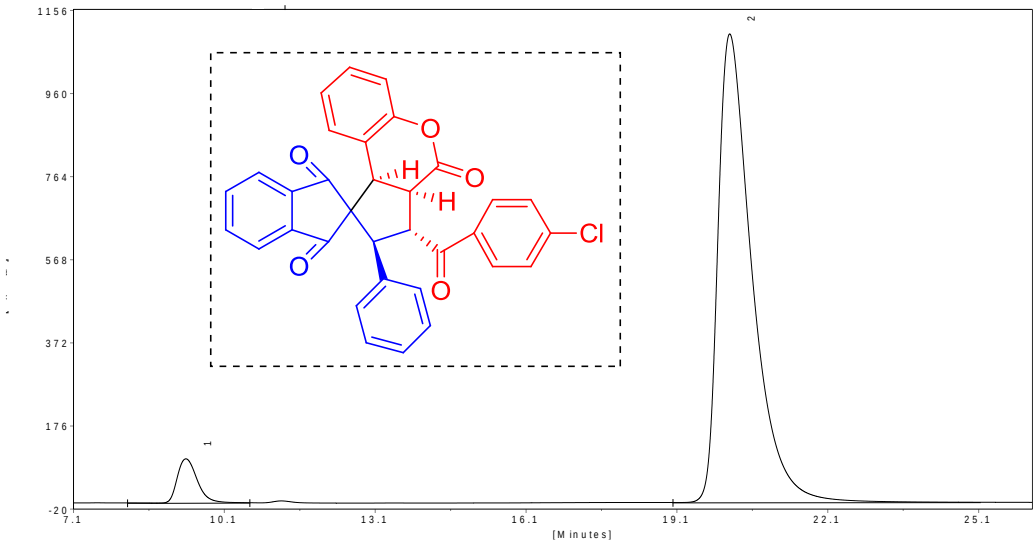
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
19.61	18.27	1237.45	4.2195
30.82	377.73	28089.83	95.7805

HPLC chromatogram for **3ga**

Column: Chiralpak IB	Flow rate: 1.0 mL/min
Solvent: n-Hexane:IPA=70:30	Detector: UV 248 nm



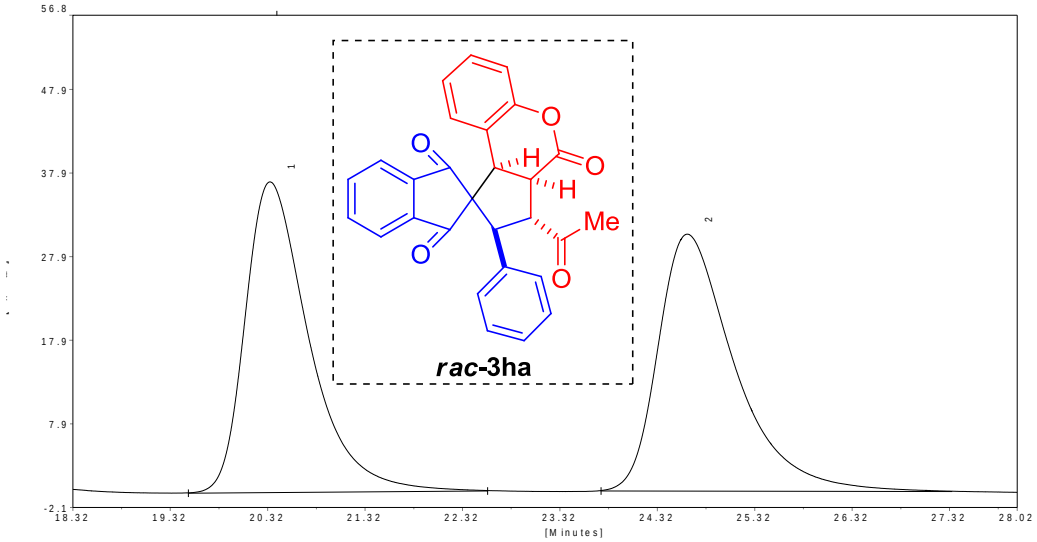
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
9.42	710.35	16269.16	49.9971
20.57	368.53	16271.05	50.0029



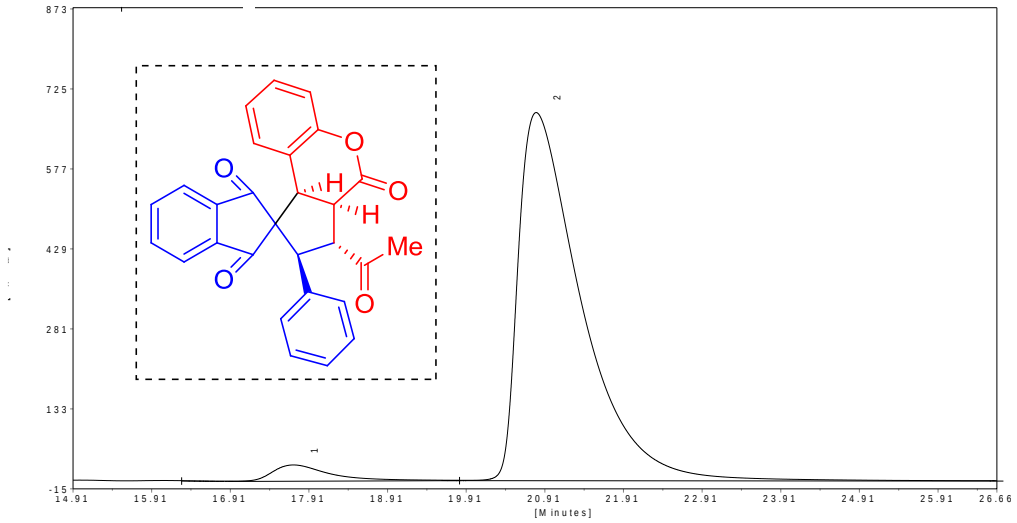
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
9.38	103.75	2823.92	5.1516
20.19	1104.94	51991.96	94.8484

HPLC chromatogram for **3ha**

Column: Chiralpak IB	Flow rate: 1.0 mL/min
Solvent: n-Hexane:IPA = 85:15	Detector: UV 248 nm



Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
20.35	37.12	1685.91	50.2069
24.63	30.69	1672.01	49.7931



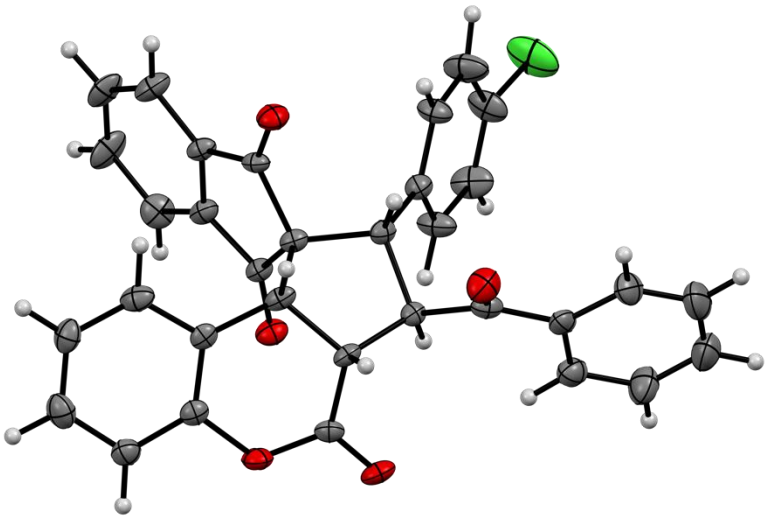
Ret. time (min)	Height (mv)	Area (mv.sec)	Rel. area (%)
17.72	28.80	1337.34	3.4283
20.80	680.44	37671.40	96.5717

X-ray crystallographic data for 3ad

3ad (CCDC no. 1484301): The crystal of **3ad** for X-ray analysis was grown form a dichloromethane solution.

The asymmetric unit has two independent molecules of **3ad** (with the same absolute configuration) as well as a half-occupancy dichloromethane molecule of solvation (with disordered chlorine atoms).

In order to clearly demonstrate the absolute configuration of **3ad**, one of the **3ad** molecule and the half-occupancy dichloromethane molecule was hidden from the ORTEP drawings.



The thermal ellipsoids are drawn at 30 % probability level.

Empirical formula	C66.50 H43 Cl3 O10 [(C33 H21 Cl O5)·0.25(C1 H2 Cl2)]	
Formula weight	1108.36 [(532.98)+0.25(84.8) = 554.18]	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21	
Unit cell dimensions	a = 13.2268(8) Å	α= 90°.
	b = 6.9147(4) Å	β= 99.015(3)°.
	c = 33.035(2) Å	γ = 90°.
Volume	2984.0(3) $Å^3$	
Z	2	
Density (calculated)	1.234 Mg/m^3	
Absorption coefficient	0.211 mm^{-1}	
F(000)	1146	
Crystal size	0.610 x 0.380 x 0.100 mm^3	
Theta range for data collection	1.248 to 25.003°.	
Index ranges	-15<=h<=15, -8<=k<=7, -38<=l<=39	

Reflections collected	20104
Independent reflections	8423 [R(int) = 0.0405]
Completeness to theta = 25.003°	97.9 %
Absorption correction	Empirical
Max. and min. transmission	0.7452 and 0.6768
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	8423 / 4 / 730
Goodness-of-fit on F^2	1.212
Final R indices [I>2sigma(I)]	R1 = 0.0744, wR2 = 0.2113
R indices (all data)	R1 = 0.0992, wR2 = 0.2324
Absolute structure parameter	0.12(14)
Extinction coefficient	n/a
Largest diff. peak and hole	0.643 and -0.371 e.Å^{-3}

***A PLATON_SQUEEZE procedure was used to deal with the electron density disorder of disorder solvation dichloromethane.**

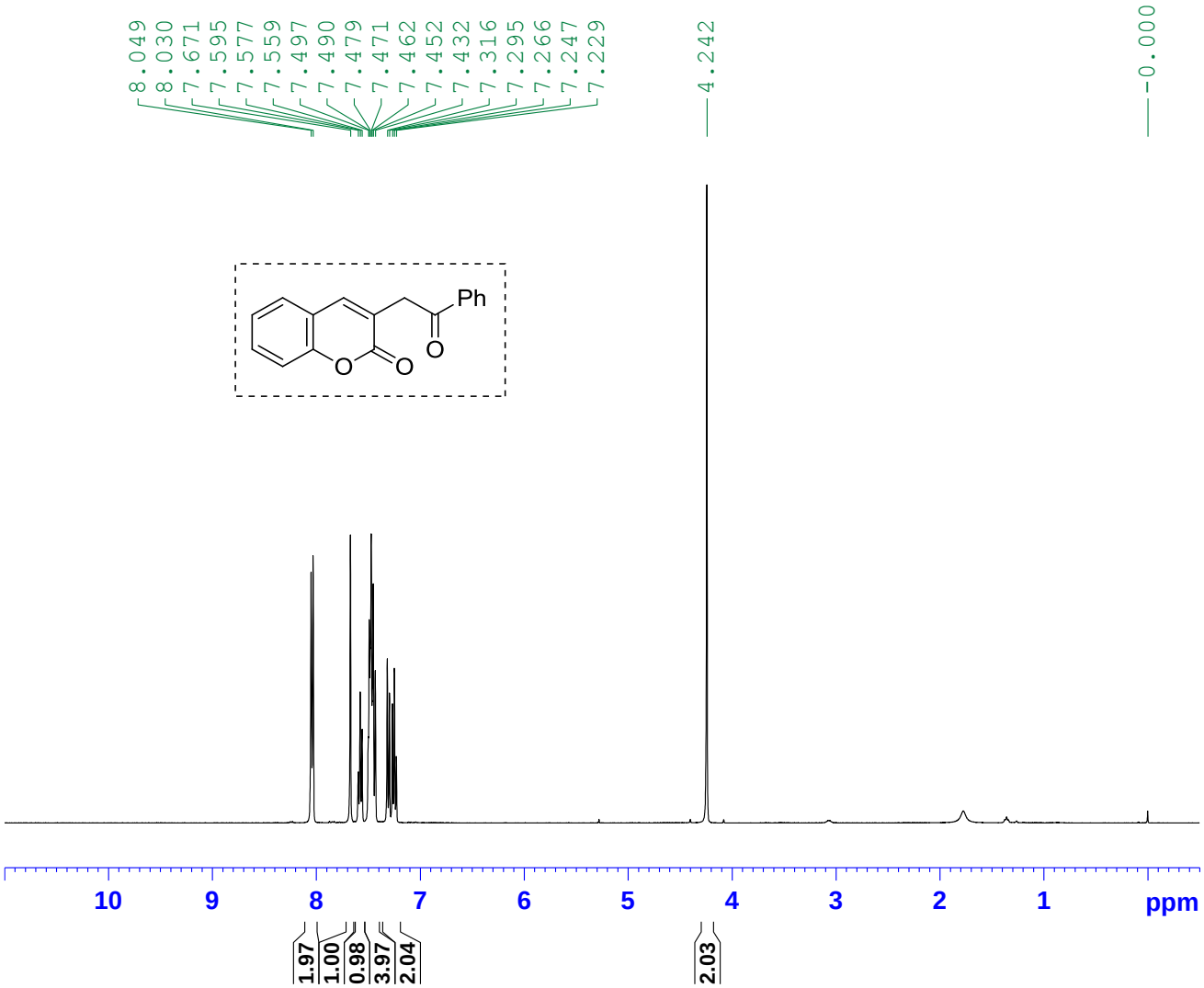
In the structure of **3ad**, a removal of the CH_2Cl_2 atoms from the refined structure followed by a treatment of the crystallographic data with the PLATON_SQUEEZE procedure.

The PLATON_SQUEEZE results suggested a cavity electron population of 140 e^- per formula unit in **3ad**, agreeing with 140 e^- for 3.33 CH_2Cl_2 molecules. For the details of SQUEEZE results, please find the CIF file which the corresponding information was appended to the bottom of the text.

```
# SQUEEZE RESULTS (Version = 160117)
# Note: Data are Listed for all Voids in the P1 Unit Cell
# i.e. Centre of Gravity, Solvent Accessible Volume,
# Recovered number of Electrons in the Void and
# Details about the Squeezed Material
loop_
 _platon_squeeze_void_nr
 _platon_squeeze_void_average_x
 _platon_squeeze_void_average_y
 _platon_squeeze_void_average_z
 _platon_squeeze_void_volume
 _platon_squeeze_void_count_electrons
 _platon_squeeze_void_content
  1  0.956 -0.107  0.167       151        72 ' '
  2  1.044  0.045  0.833       152        68 ' '
 _platon_squeeze_void_probe_radius                1.20
 _platon_squeeze_details
```

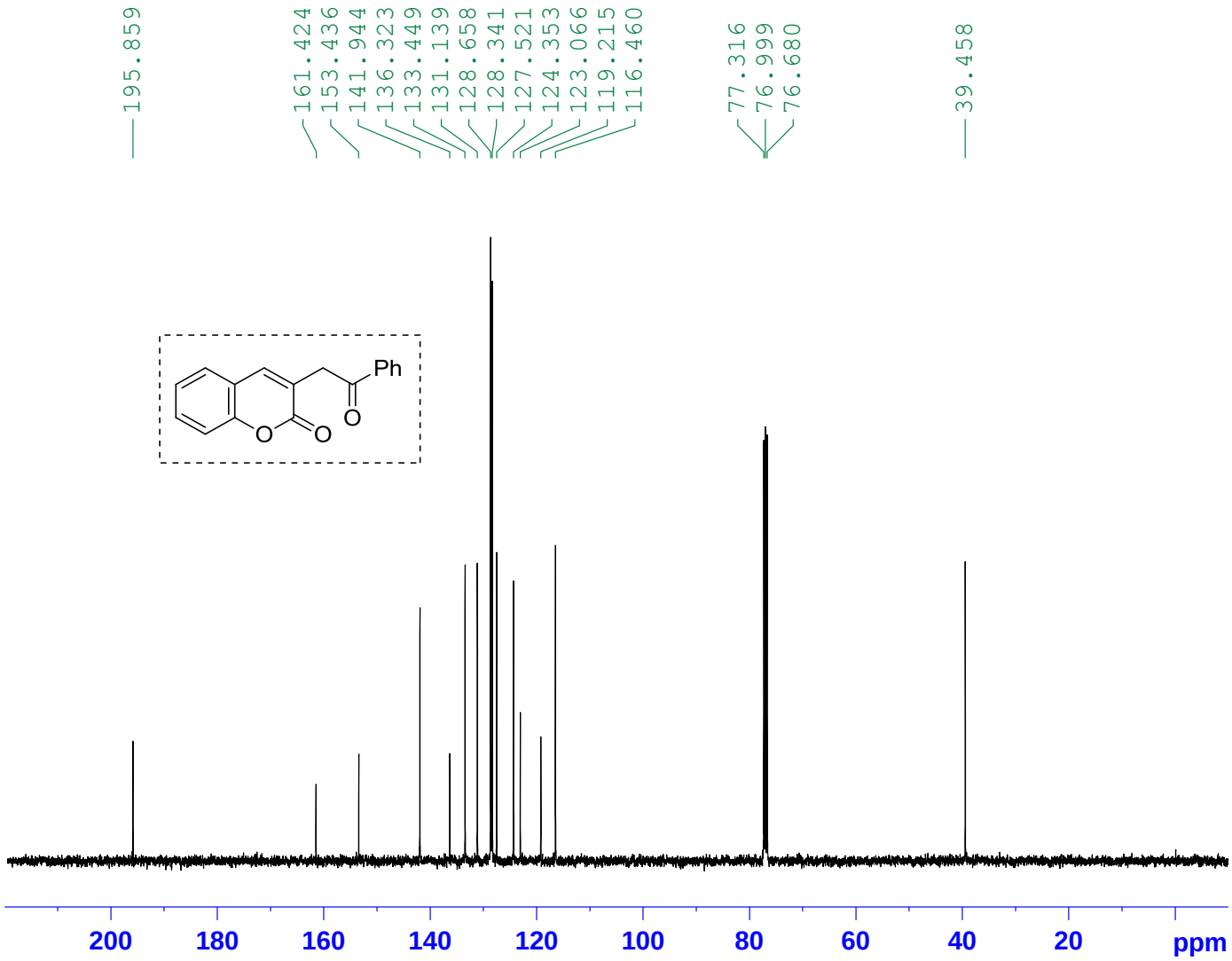
^{1}H and ^{13}C NMR spectra of all new compounds

^{1}H NMR spectrum of **1a** ($CDCl_3$)



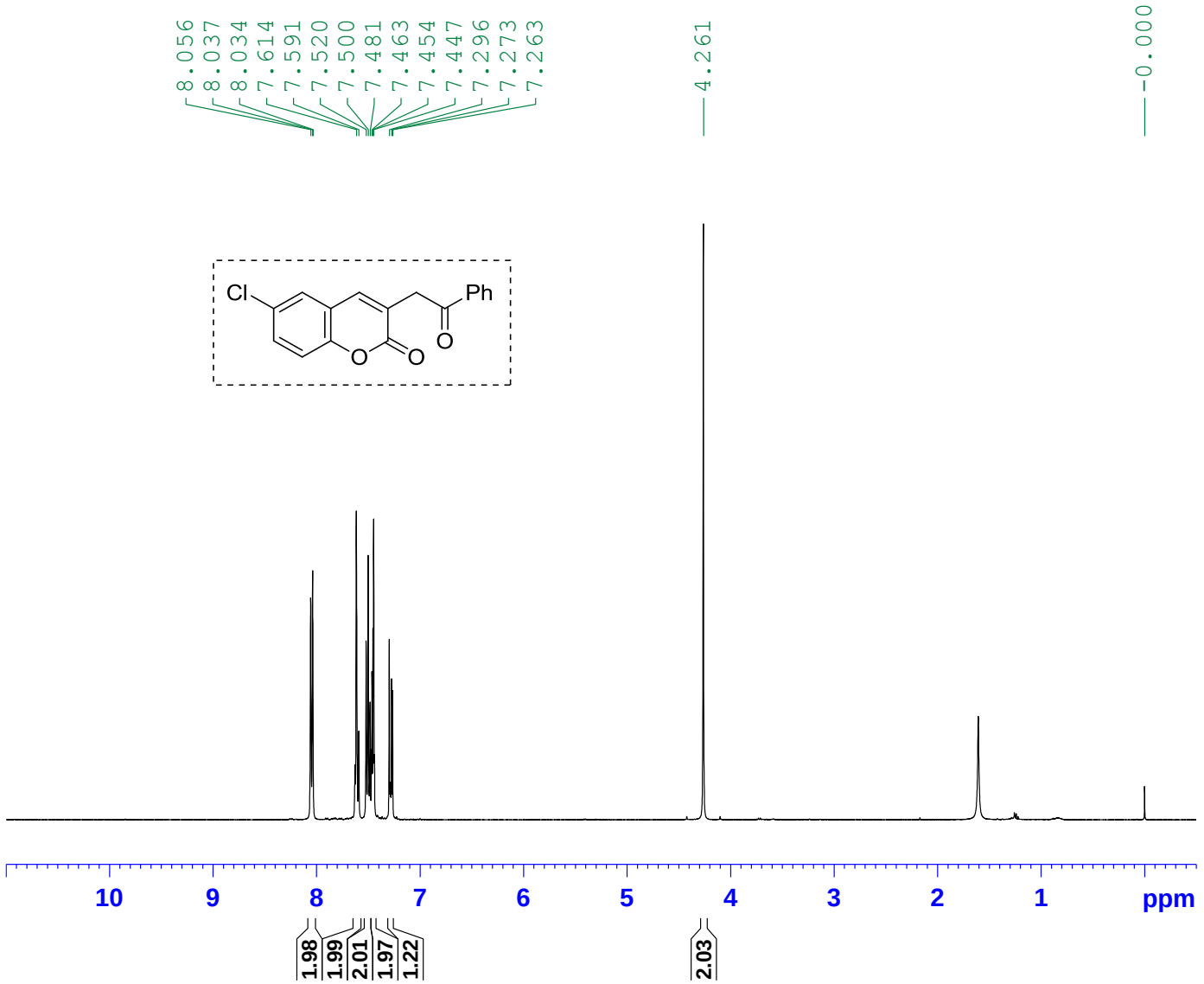

```
Current Data Parameters
NAME                  1a
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160623
Time               14.05
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ             2.2719147 sec
RG                 63.58
DW                69.333 usec
DE                10.50 usec
TE                304.7 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 12.90 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                 16384
SF           400.1300075 MHz
WDW                   EM
SSB       0
LB        0 Hz
GB        0
PC                  1.00
```

^{13}C NMR spectrum of **1a** ($CDCl_3$)



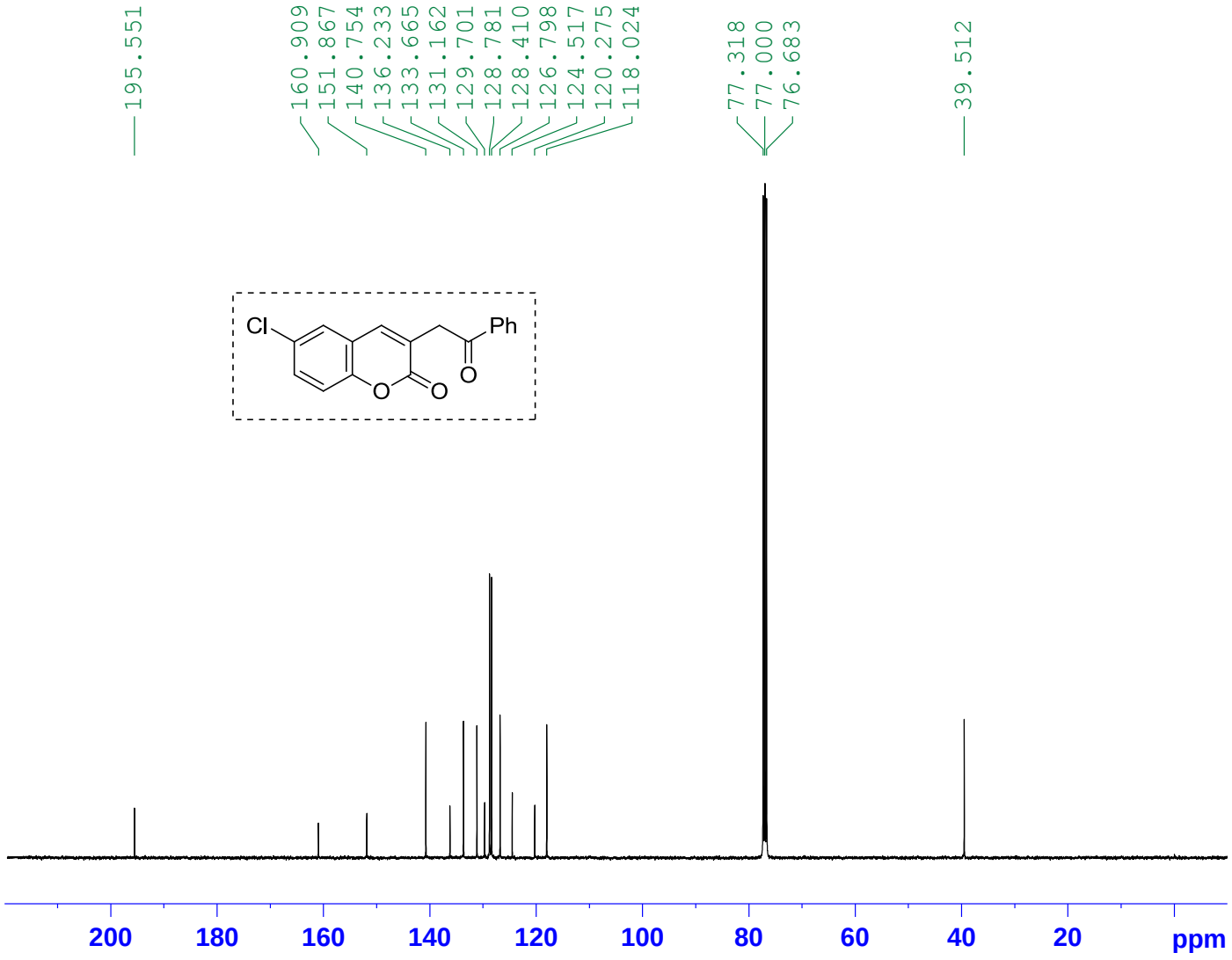

```
Current Data Parameters
NAME                   1a
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160623
Time               14.06
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  100
DS                    0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                304.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        47.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        15.00000000 W
PLW12        0.33750001 W
PLW13        0.27338001 W

F2 - Processing parameters
SI                 32768
SF          100.6127773 MHz
WDW                EM
SSB       0
LB                2.00 Hz
GB        0
PC                1.00
```

^{1}H NMR spectrum of **1b** ($CDCl_3$)



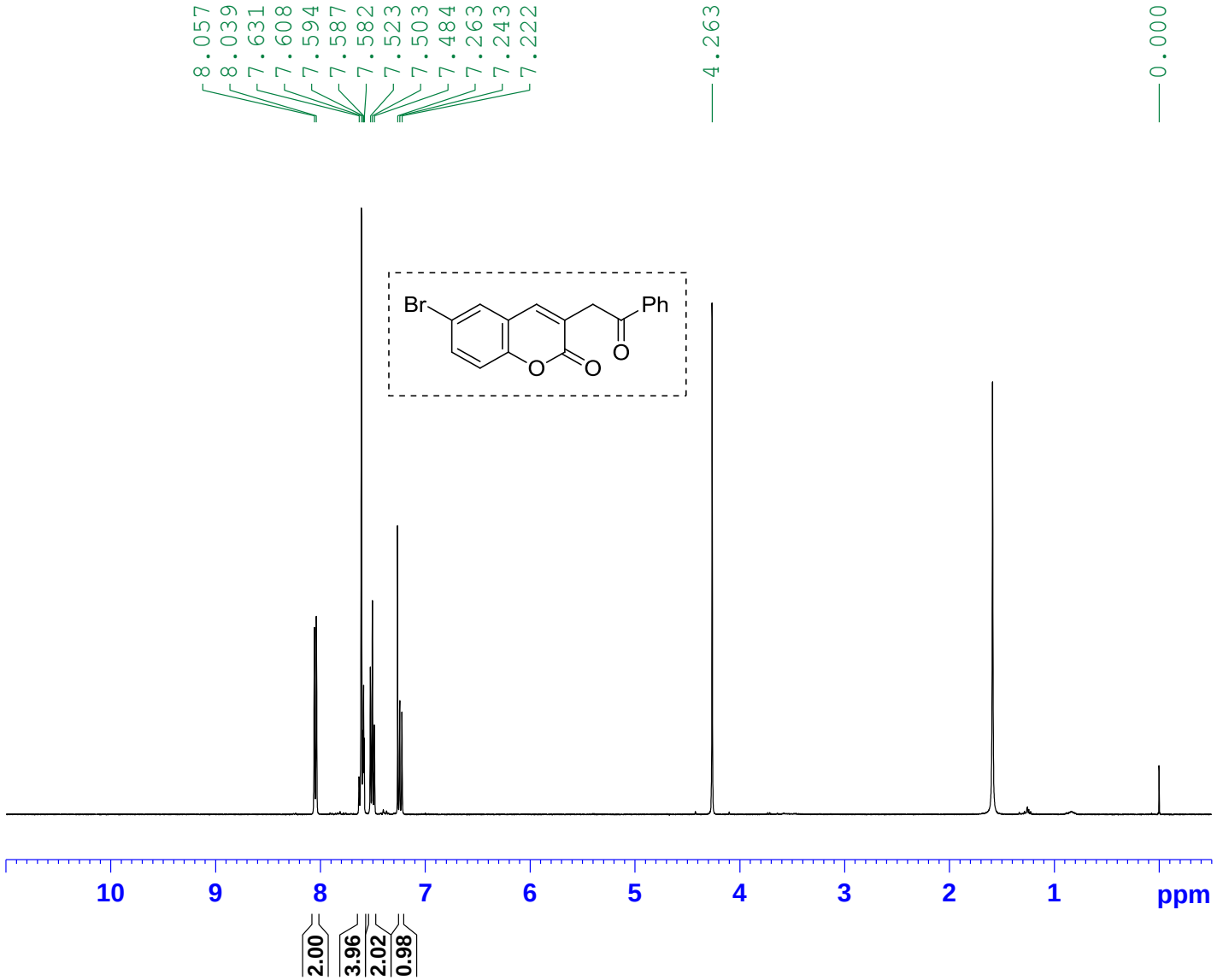

```
Current Data Parameters
NAME                  1b
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170201
Time              17.07
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   32
DS                    0
SWH            7211.539 Hz
FIDRES         0.220079 Hz
AQ             2.2719147 sec
RG                128.9
DW               69.333 usec
DE                10.50 usec
TE                297.3 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                 1H
P1                12.90 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                16384
SF           400.1300087 MHz
WDW                  EM
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```

^{13}C NMR spectrum of **1b** ($CDCl_3$)



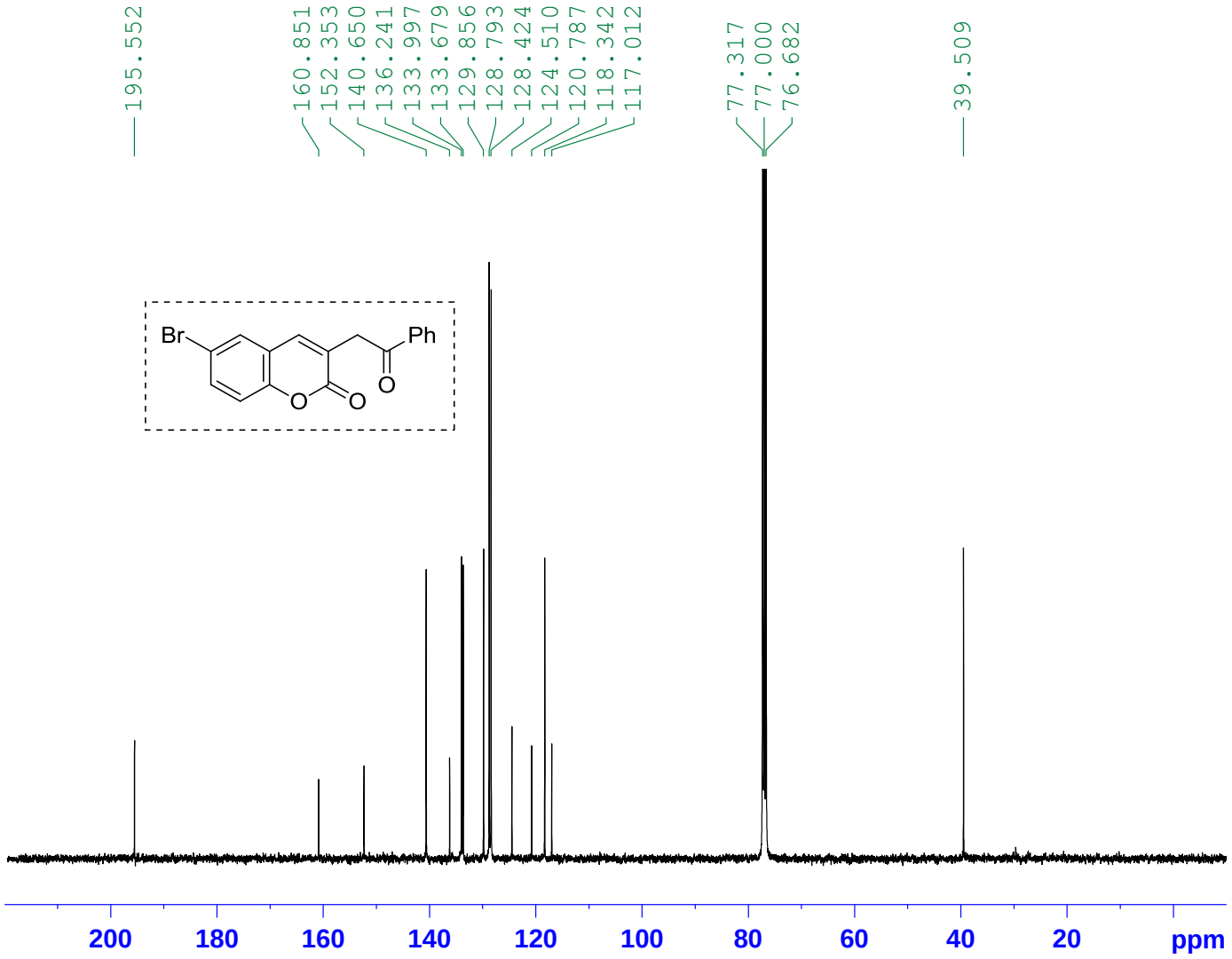

```
Current Data Parameters
NAME                   1b
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20170201
Time               17.20
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                 5000
DS                    0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            0.6815744 sec
RG               198.09
DW               20.800 usec
DE                6.50 usec
TE                298.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        47.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        15.00000000 W
PLW12        0.33750001 W
PLW13        0.27338001 W

F2 - Processing parameters
SI                32768
SF          100.6127716 MHz
WDW                 EM
SSB       0
LB                2.00 Hz
GB        0
PC                1.00
```

^{1}H NMR spectrum of **1c** ($CDCl_3$)



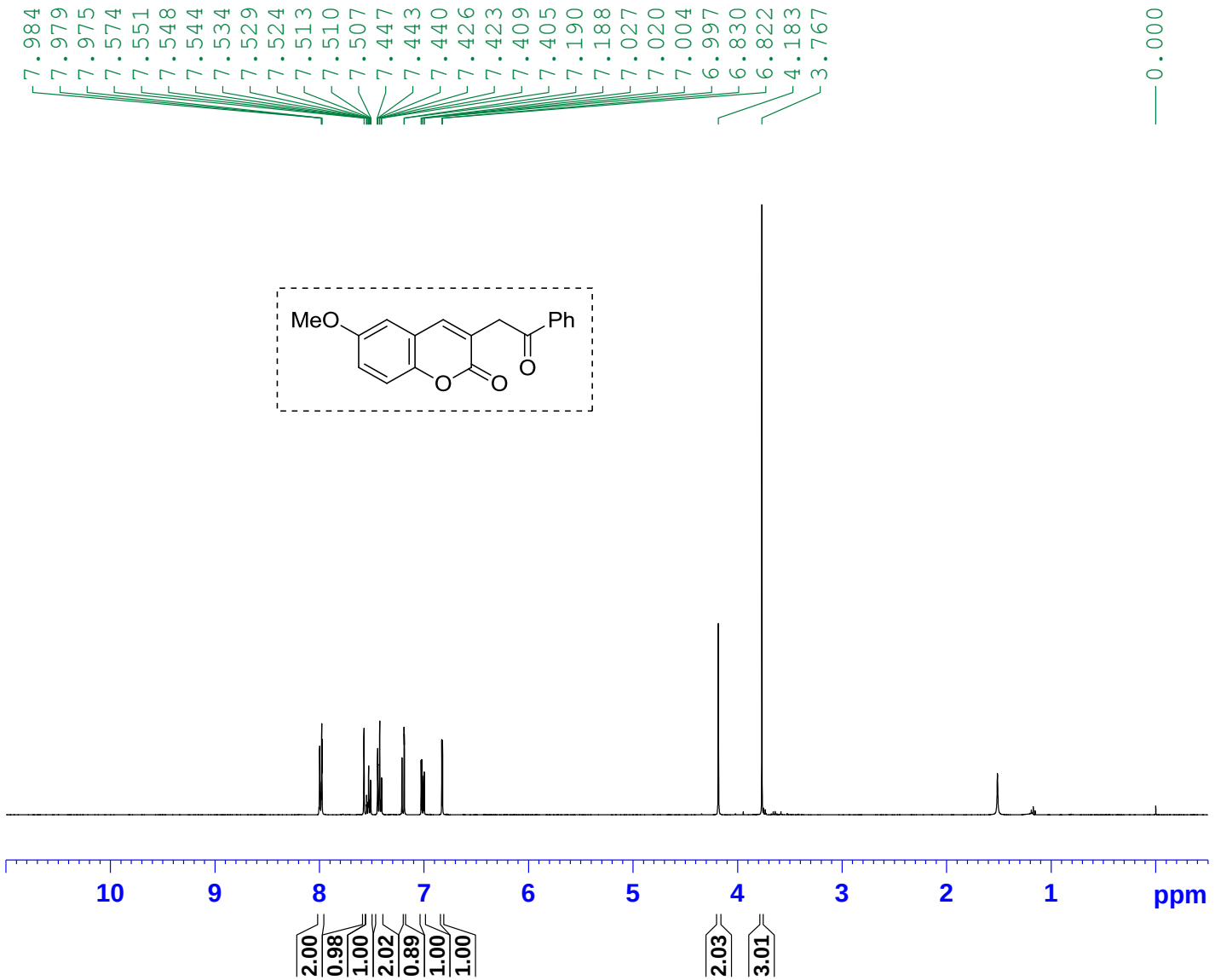

```
Current Data Parameters
NAME                   1c
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160709
Time               11.14
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ             2.2719147 sec
RG                198.09
DW                69.333 usec
DE                10.50 usec
TE                297.5 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 12.90 usec
PLW1        15.00000000 W

F2 - Processing parameters
SI                 16384
SF           400.1300090 MHz
WDW                   EM
SSB       0
LB        0 Hz
GB        0
PC                  1.00
```

^{13}C NMR spectrum of **1c** ($CDCl_3$)



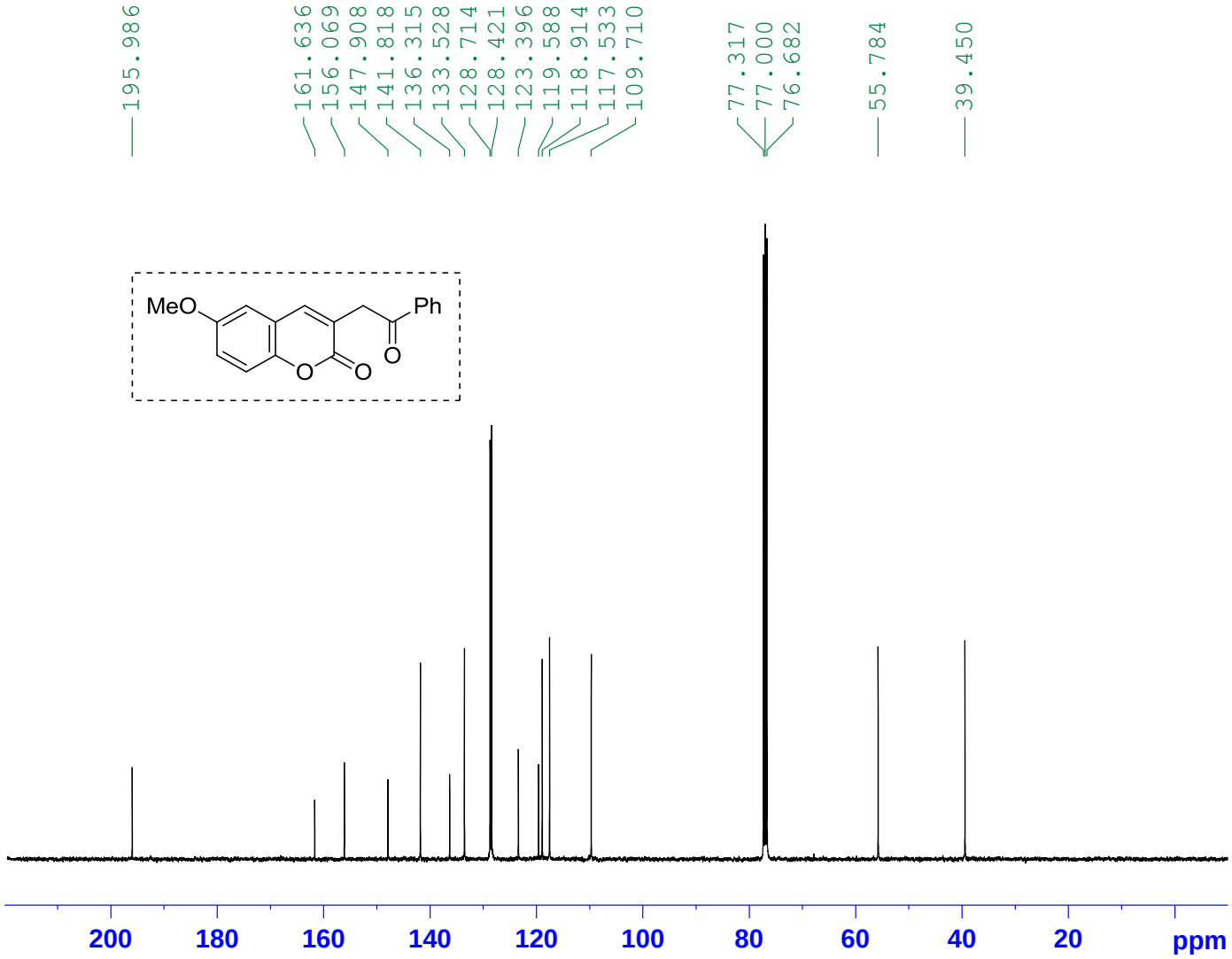

```
Current Data Parameters
NAME                   1c
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20170313
Time               22.10
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                13178
DS                    0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                298.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        47.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        15.00000000 W
PLW12        0.33750001 W
PLW13        0.27338001 W

F2 - Processing parameters
SI                32768
SF          100.6127705 MHz
WDW                EM
SSB       0
LB                2.00 Hz
GB        0
PC                1.00
```

^{1}H NMR spectrum of **1d** ($CDCl_3$)



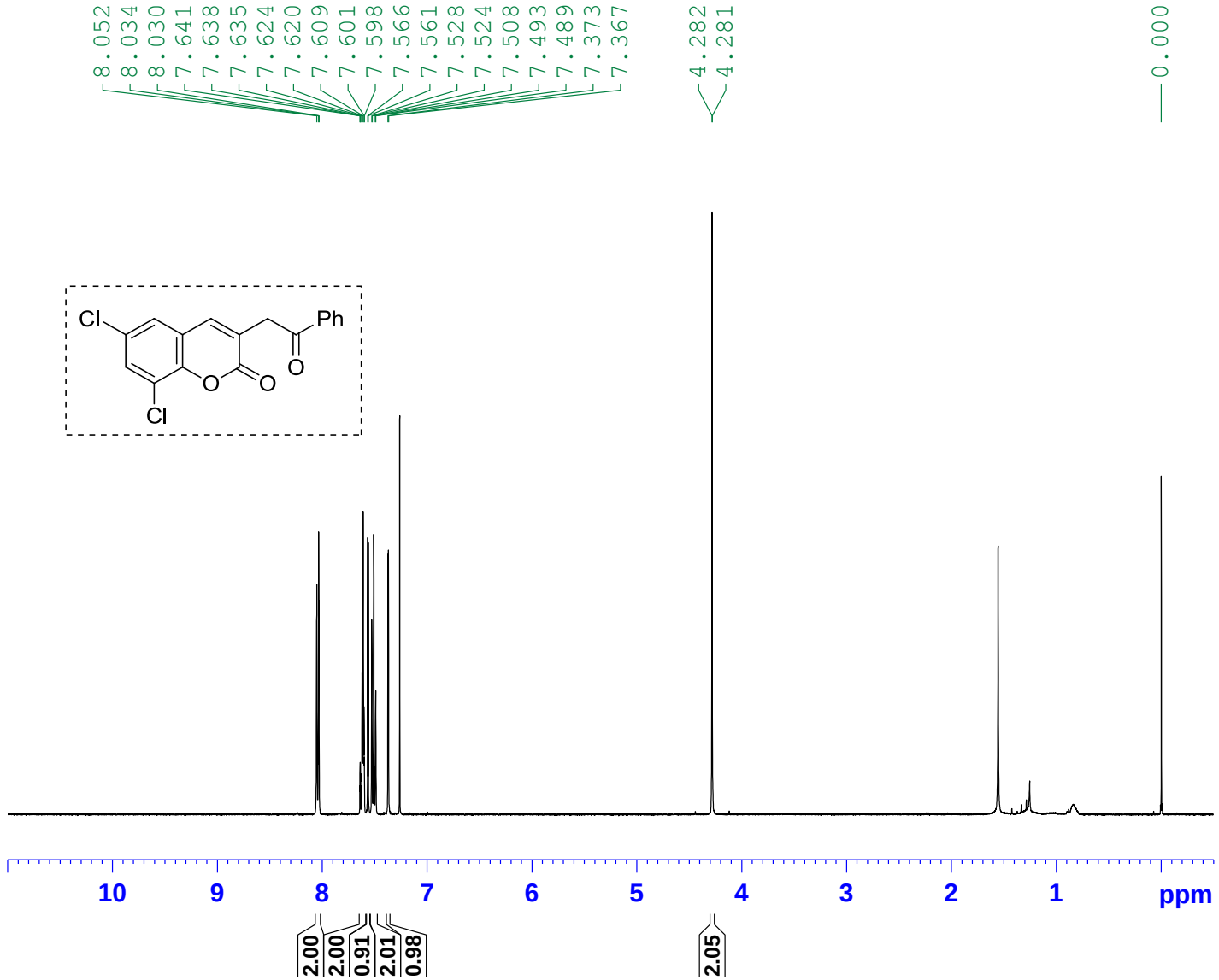

```
Current Data Parameters
NAME                   1d
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_           20170522
Time               11.36
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ             2.2719147 sec
RG                198.09
DW                69.333 usec
DE                10.50 usec
TE                297.9 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 12.90 usec
PLW1        15.00000000 W

F2 - Processing parameters
SI                 16384
SF           400.1300377 MHz
WDW                   EM
SSB       0
LB        0 Hz
GB        0
PC                  1.00
```

^{13}C NMR spectrum of **1d** ($CDCl_3$)



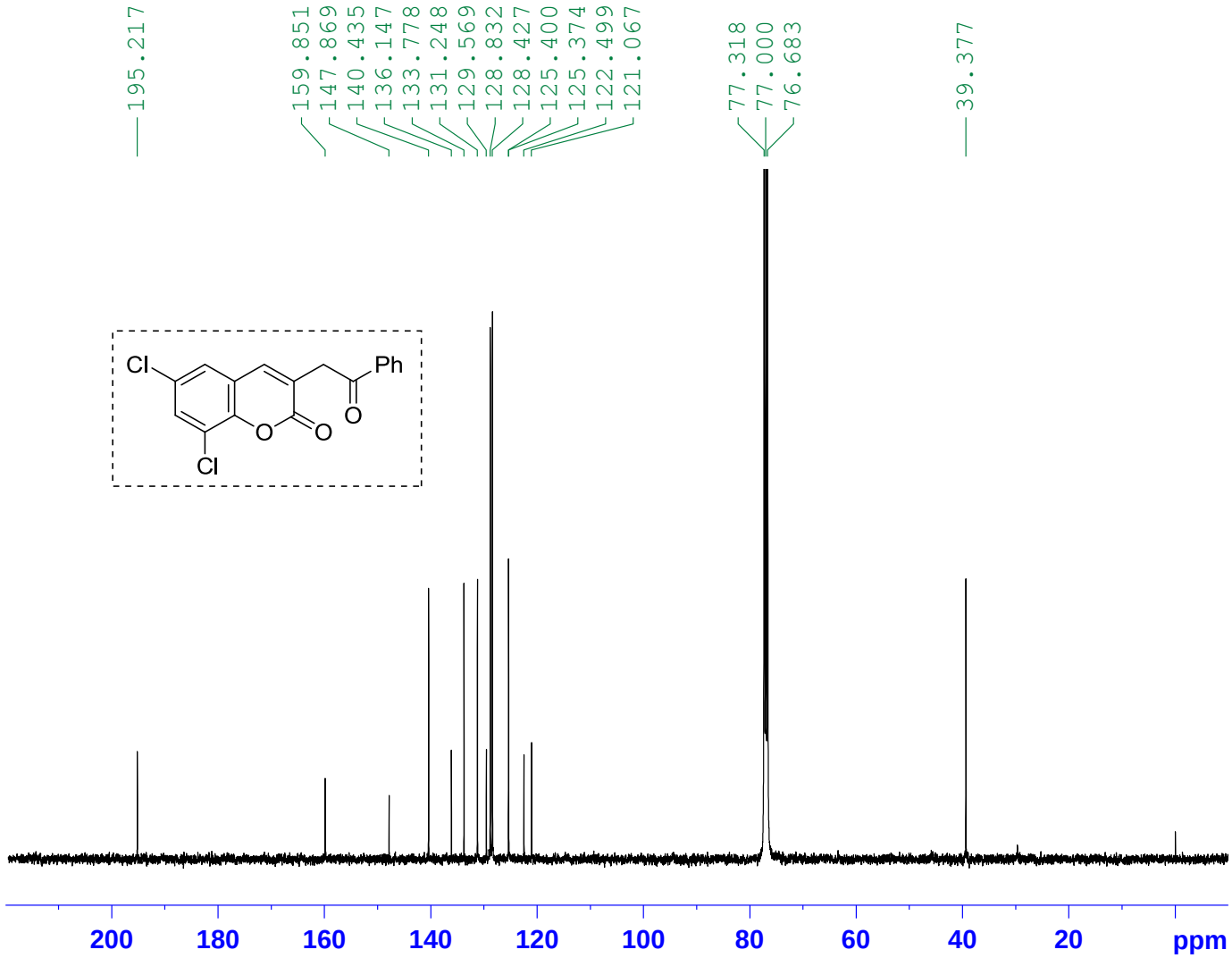

```
Current Data Parameters
NAME                   1d
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20170228
Time               17.21
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                 1823
DS                    0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                298.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        47.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        15.00000000 W
PLW12        0.33750001 W
PLW13        0.27338001 W

F2 - Processing parameters
SI                 32768
SF          100.6127736 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.00
```

^{1}H NMR spectrum of **1e** ($CDCl_3$)



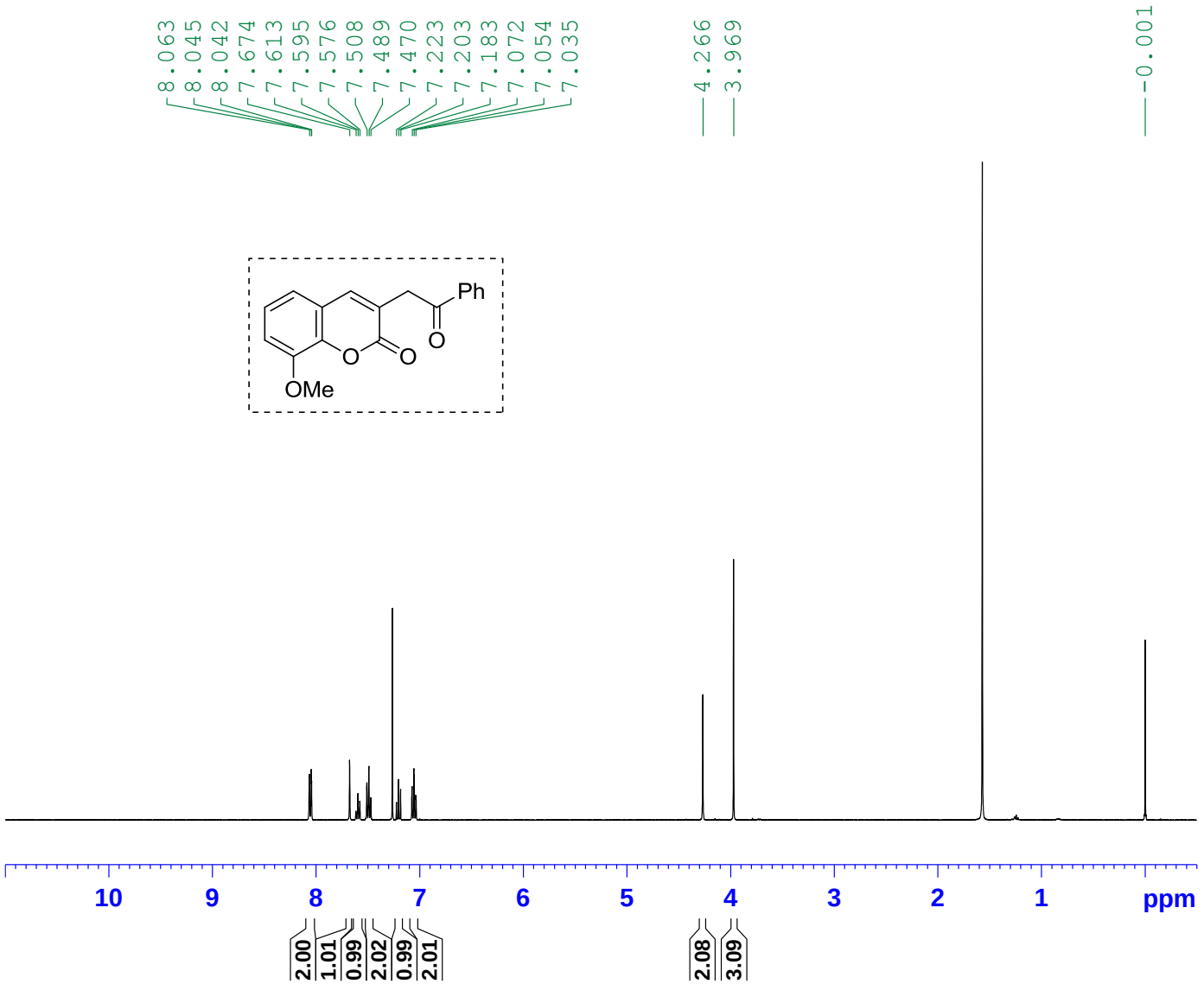

```
Current Data Parameters
NAME                   1e
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_           20170310
Time              21.37
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            7211.539 Hz
FIDRES        0.220079 Hz
AQ            2.2719147 sec
RG               198.09
DW               69.333 usec
DE                10.50 usec
TE                297.5 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                1H
P1                12.90 usec
PLW1        15.00000000 W

F2 - Processing parameters
SI                16384
SF          400.1300095 MHz
WDW                 EM
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```

^{13}C NMR spectrum of **1e** ($CDCl_3$)



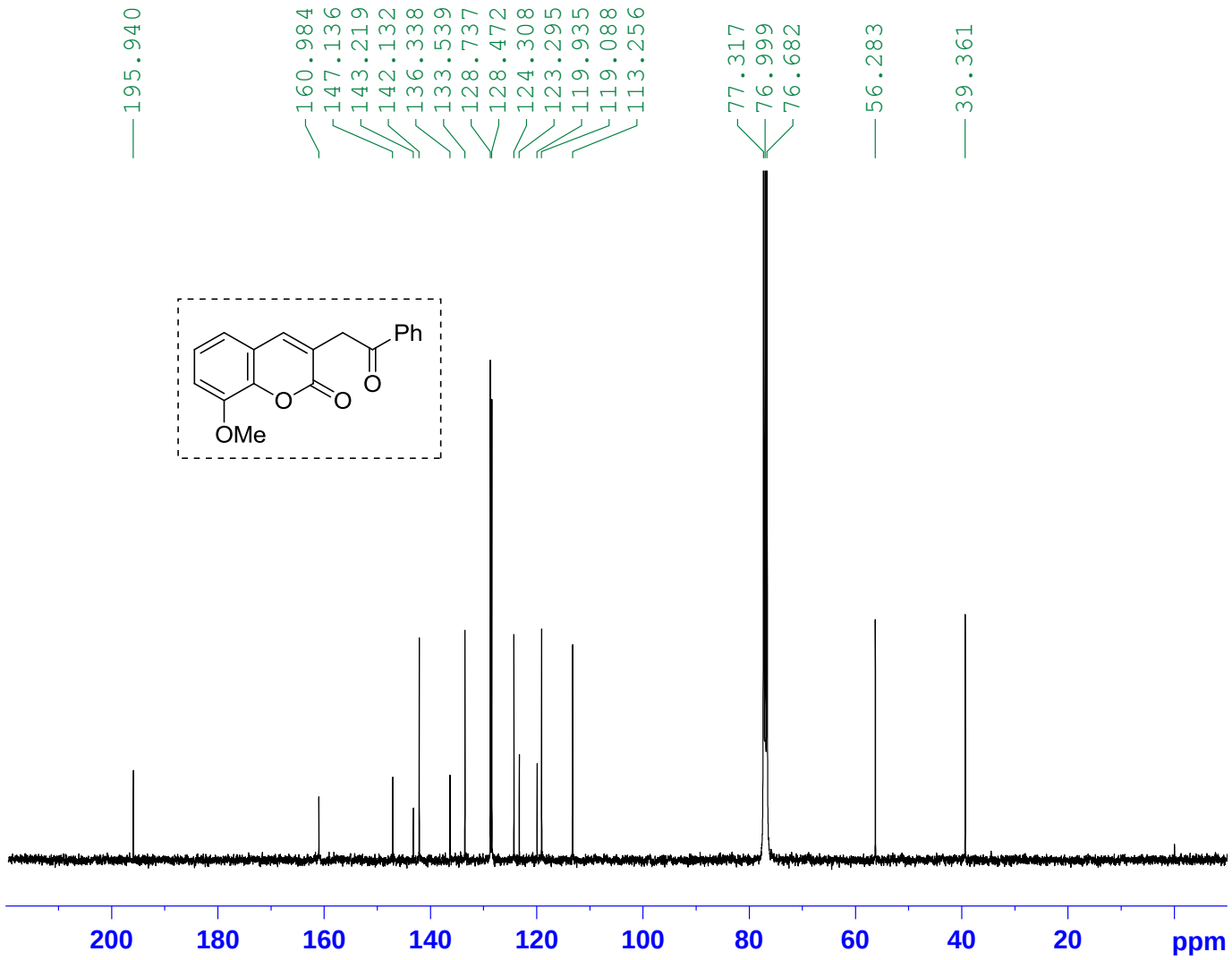

```
Current Data Parameters
NAME                   1e
EXPNO                   6
PROCNO                  1

F2 - Acquisition Parameters
Date_           20170310
Time               21.57
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                15391
DS                    0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                298.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        47.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        15.00000000 W
PLW12        0.33750001 W
PLW13        0.27338001 W

F2 - Processing parameters
SI                32768
SF          100.6127699 MHz
WDW                  EM
SSB       0
LB                2.00 Hz
GB        0
PC                1.00
```

^{1}H NMR spectrum of **1f** ($CDCl_3$)



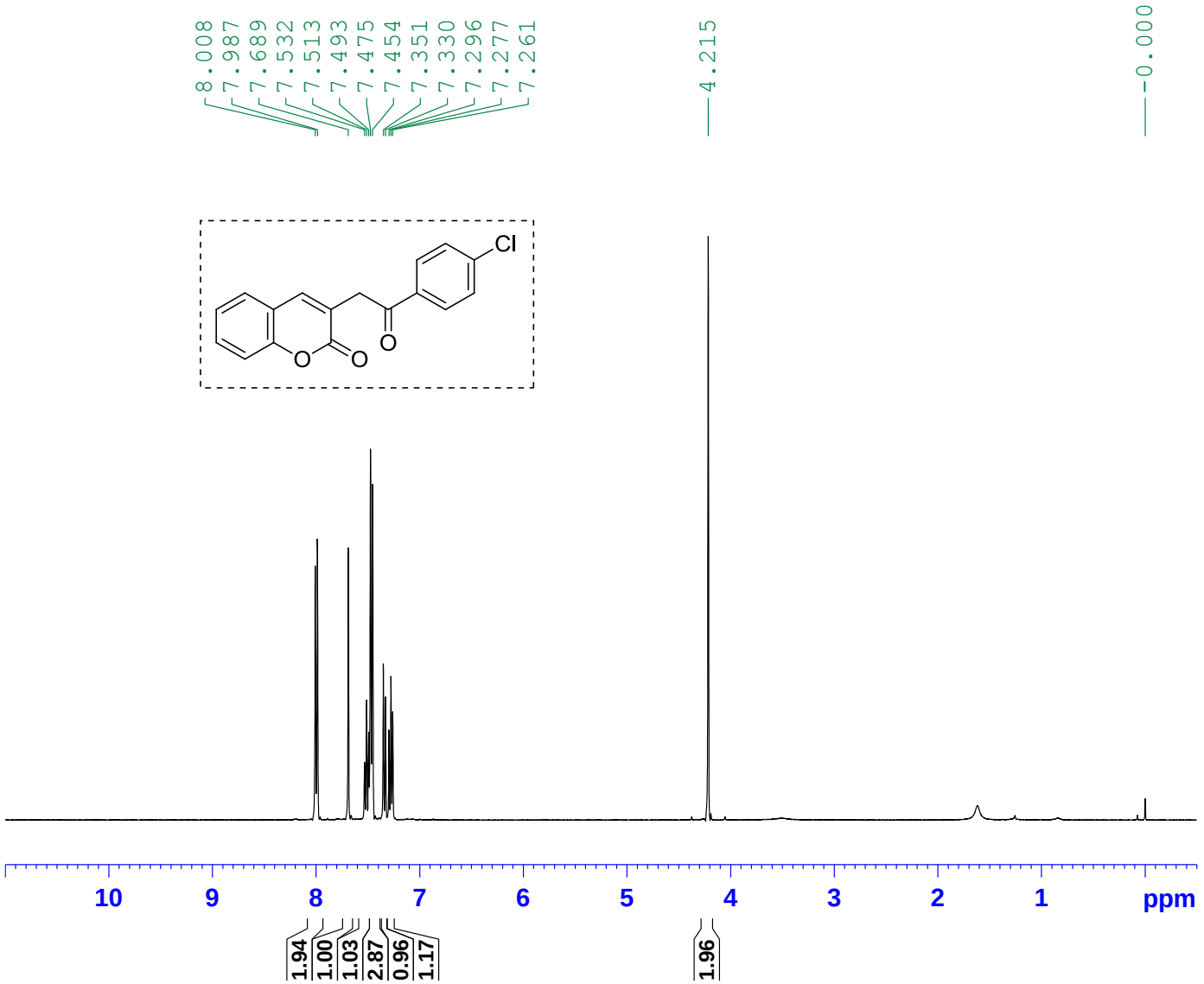

```
Current Data Parameters
NAME                  1f
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160603
Time              19.05
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            7211.539 Hz
FIDRES         0.220079 Hz
AQ             2.2719147 sec
RG                198.09
DW                69.333 usec
DE                10.50 usec
TE                298.6 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                 1H
P1                12.90 usec
PLW1        15.00000000 W

F2 - Processing parameters
SI                16384
SF           400.1300094 MHz
WDW                  EM
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```

^{13}C NMR spectrum of **1f** ($CDCl_3$)



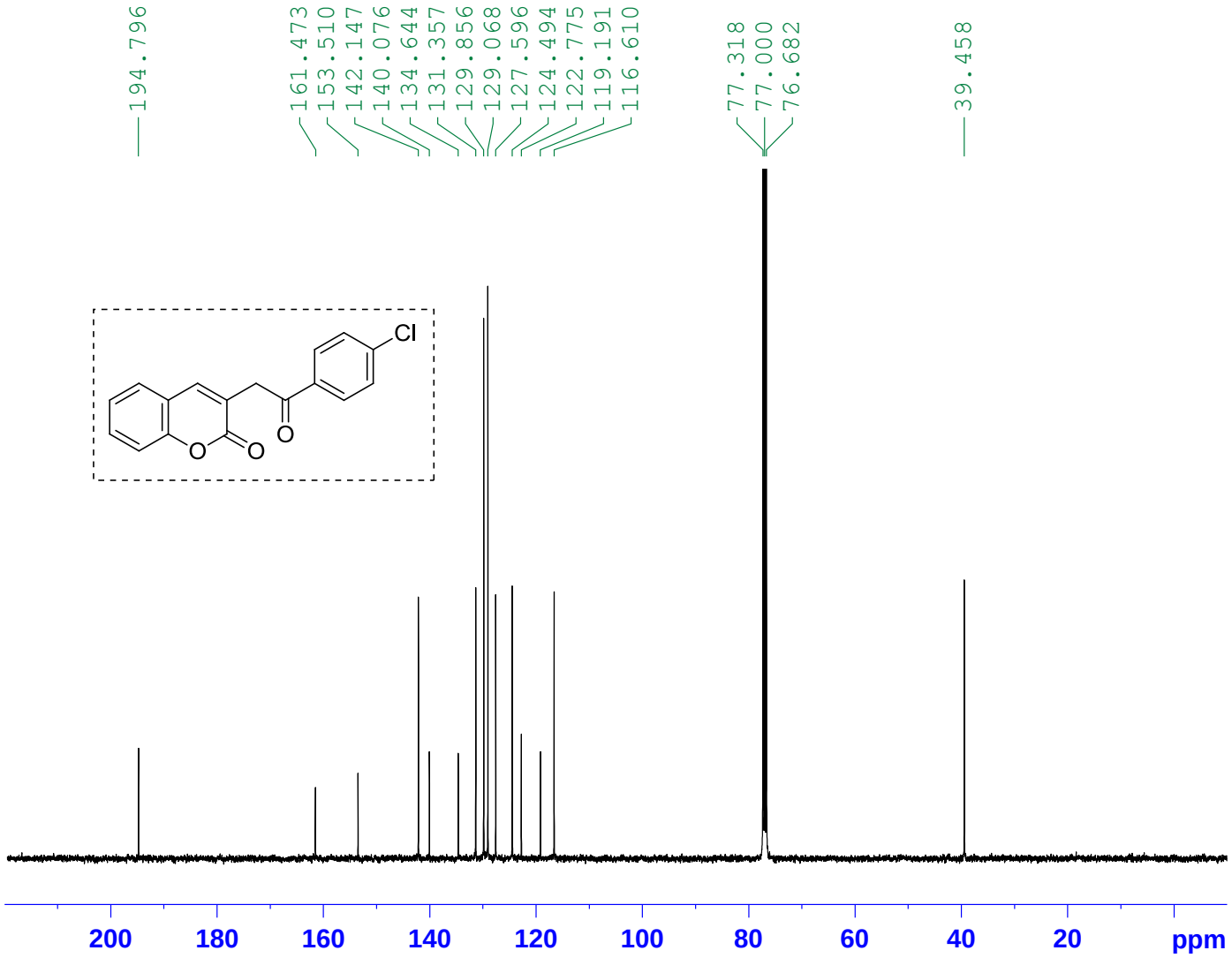

```
Current Data Parameters
NAME                  1f
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170322
Time              22.08
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                32768
SOLVENT           CDCl3
NS                15020
DS                    0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            0.6815744 sec
RG               198.09
DW               20.800 usec
DE                6.50 usec
TE                298.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        47.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        15.00000000 W
PLW12        0.33750001 W
PLW13        0.27338001 W

F2 - Processing parameters
SI                32768
SF          100.6127702 MHz
WDW                EM
SSB       0
LB                2.00 Hz
GB        0
PC                1.00
```

^{1}H NMR spectrum of **1g** ($CDCl_3$)



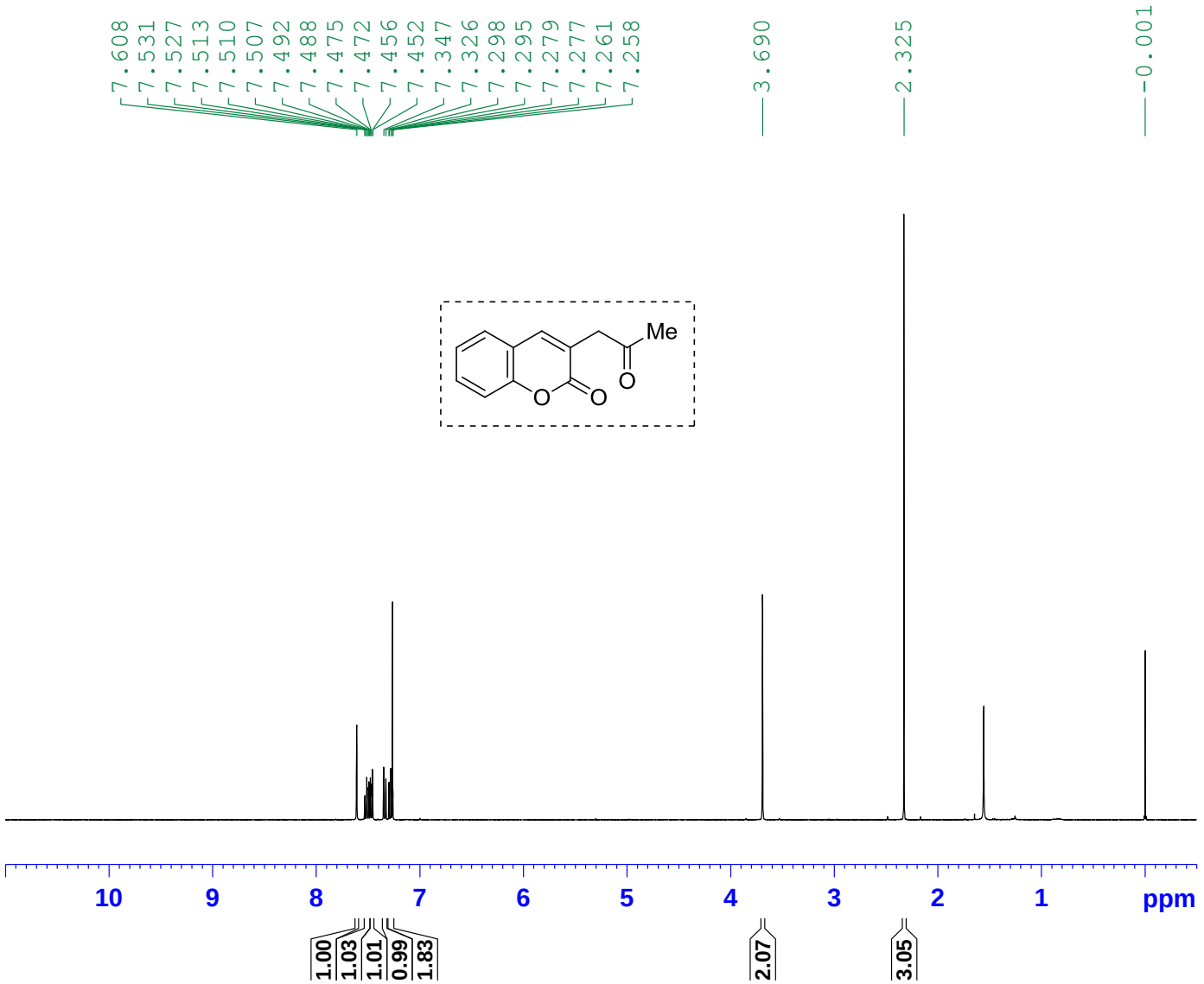

```
Current Data Parameters
NAME                  1g
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170525
Time              20.53
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   32
DS                    0
SWH            7246.377 Hz
FIDRES        0.221142 Hz
AQ            2.2609921 sec
RG                228.1
DW               69.000 usec
DE                6.50 usec
TE                298.6 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.40 usec
PL1                1.80 dB
SFO1        400.1324008 MHz

F2 - Processing parameters
SI                16384
SF          400.1300086 MHz
WDW                  EM
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```

^{13}C NMR spectrum of **1g** ($CDCl_3$)



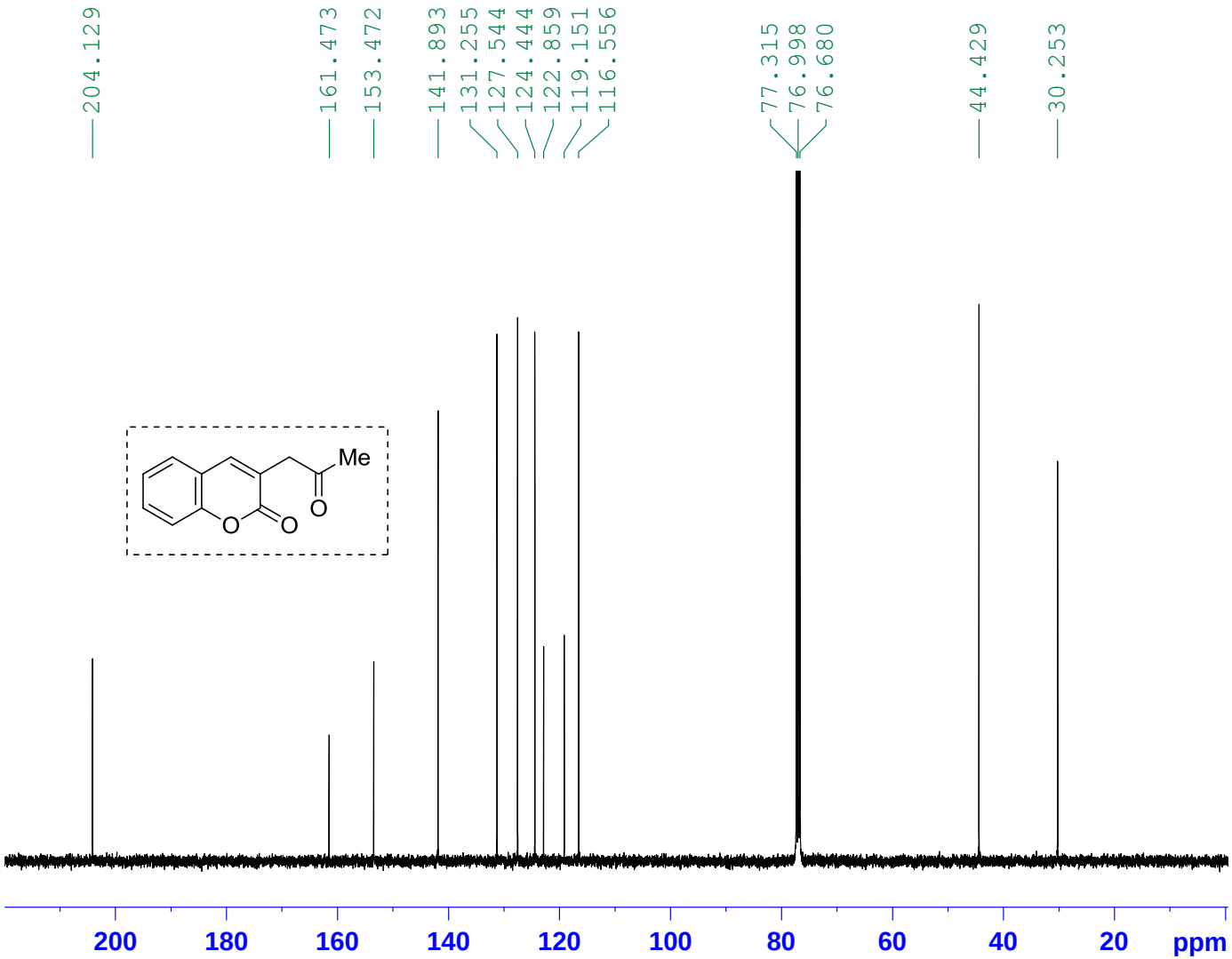

```
Current Data Parameters
NAME                   1g
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20170120
Time               18.38
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                 1839
DS                    0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            0.6815744 sec
RG               198.09
DW               20.800 usec
DE                6.50 usec
TE               298.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        47.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        15.00000000 W
PLW12        0.33750001 W
PLW13        0.27338001 W

F2 - Processing parameters
SI                32768
SF          100.6127722 MHz
WDW                EM
SSB       0
LB                2.00 Hz
GB        0
PC                1.00
```

^{1}H NMR spectrum of **1h** ($CDCl_3$)



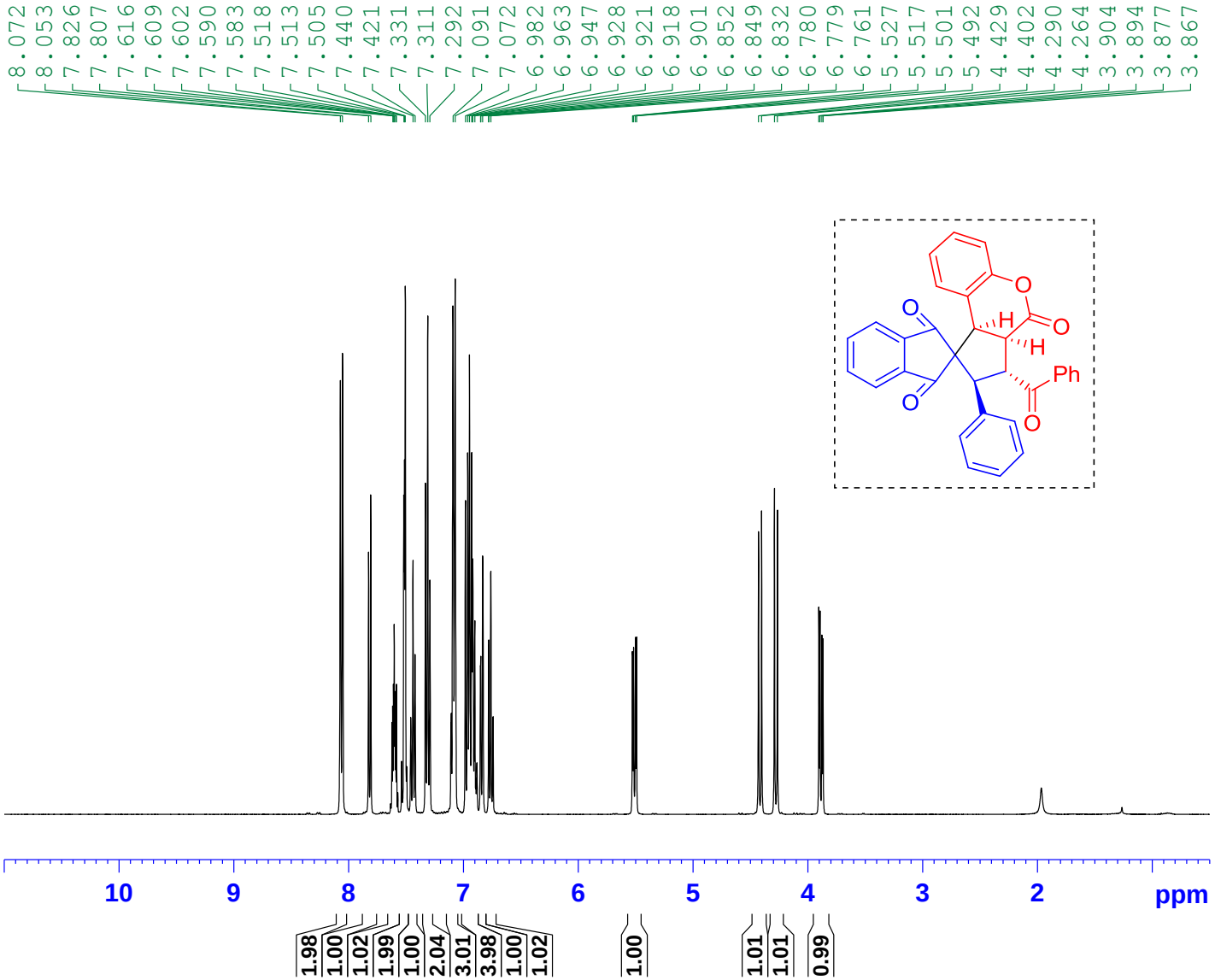

```
Current Data Parameters
NAME                  1h
EXPNO                101
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170315
Time               12.30
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                198.09
DW                69.333 usec
DE                10.50 usec
TE                297.5 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 12.90 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                 16384
SF           400.1300094 MHz
WDW                   EM
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```

^{13}C NMR spectrum of **1h** ($CDCl_3$)



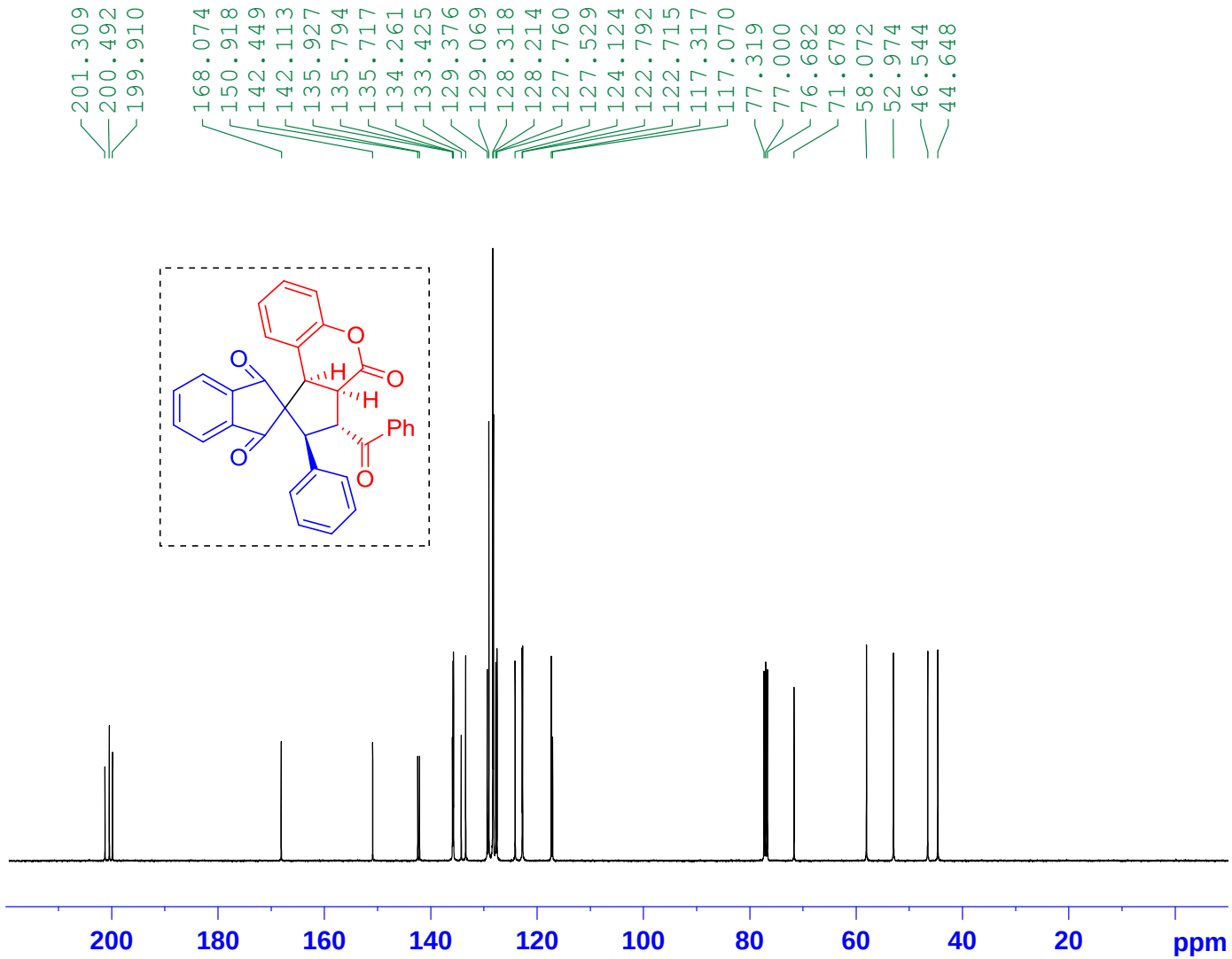

```
Current Data Parameters
NAME                   1h
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20170527
Time               21.13
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                 1107
DS                    0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6815744 sec
RG                5792.6
DW                20.800 usec
DE                 6.50 usec
TE                298.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.50 usec
PL1                7.00 dB
SFO1        100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               -0.60 dB
PL12              15.00 dB
PL13              18.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127731 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.00
```

^{1}H NMR spectrum of **3aa** ($CDCl_3$)



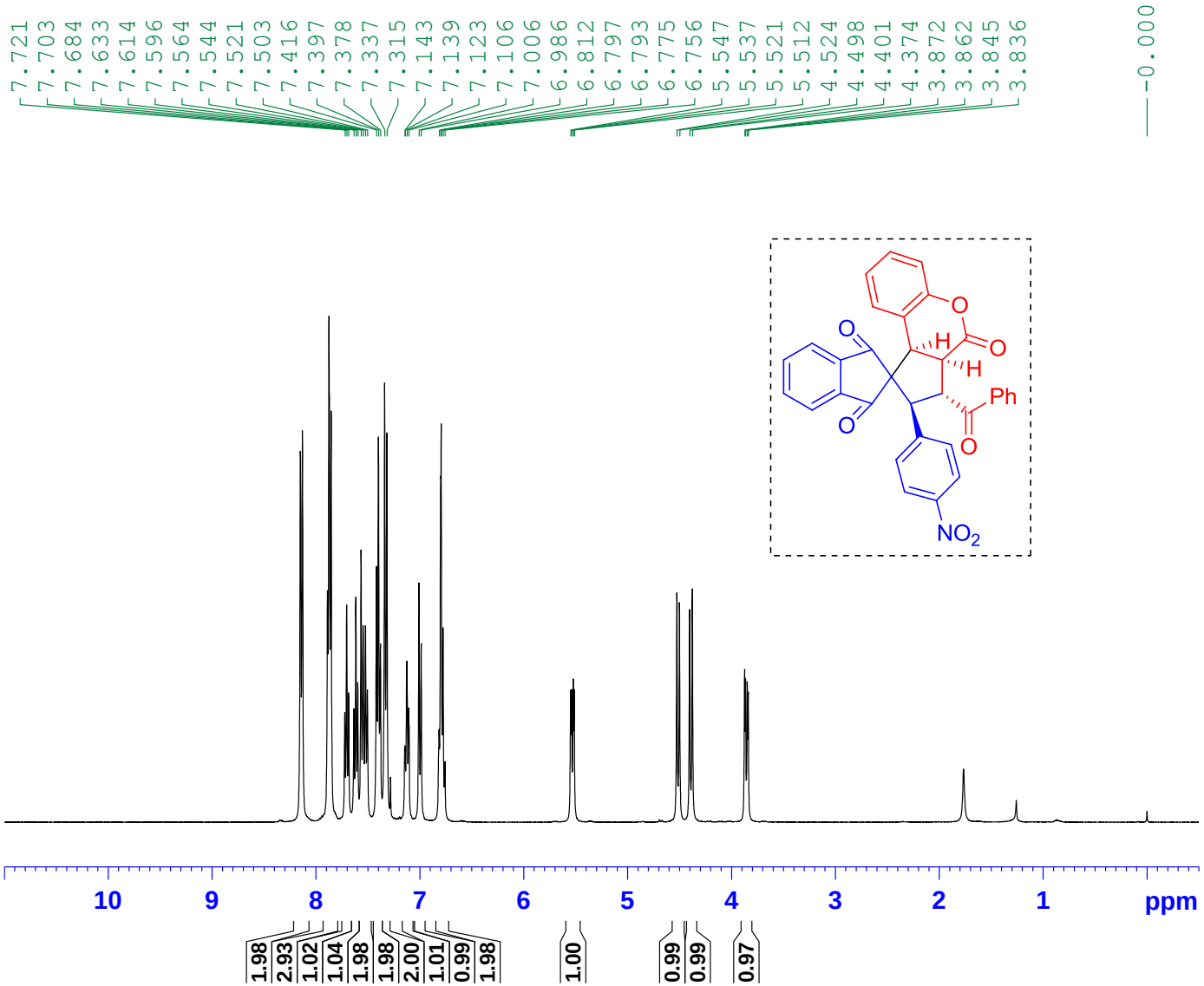

```
Current Data Parameters
NAME                  3aa
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160627
Time                17.04
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              7211.539 Hz
FIDRES           0.220079 Hz
AQ               2.2719147 sec
RG                  31.16
DW                 69.333 usec
DE                 10.50 usec
TE                 298.3 K
D1              2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                   1H
P1                  12.90 usec
PLW1          15.00000000 W

F2 - Processing parameters
SI                  16384
SF            400.1299986 MHz
WDW                    EM
SSB       0
LB        0 Hz
GB        0
PC                   1.00
```

^{13}C NMR spectrum of **3aa** ($CDCl_3$)



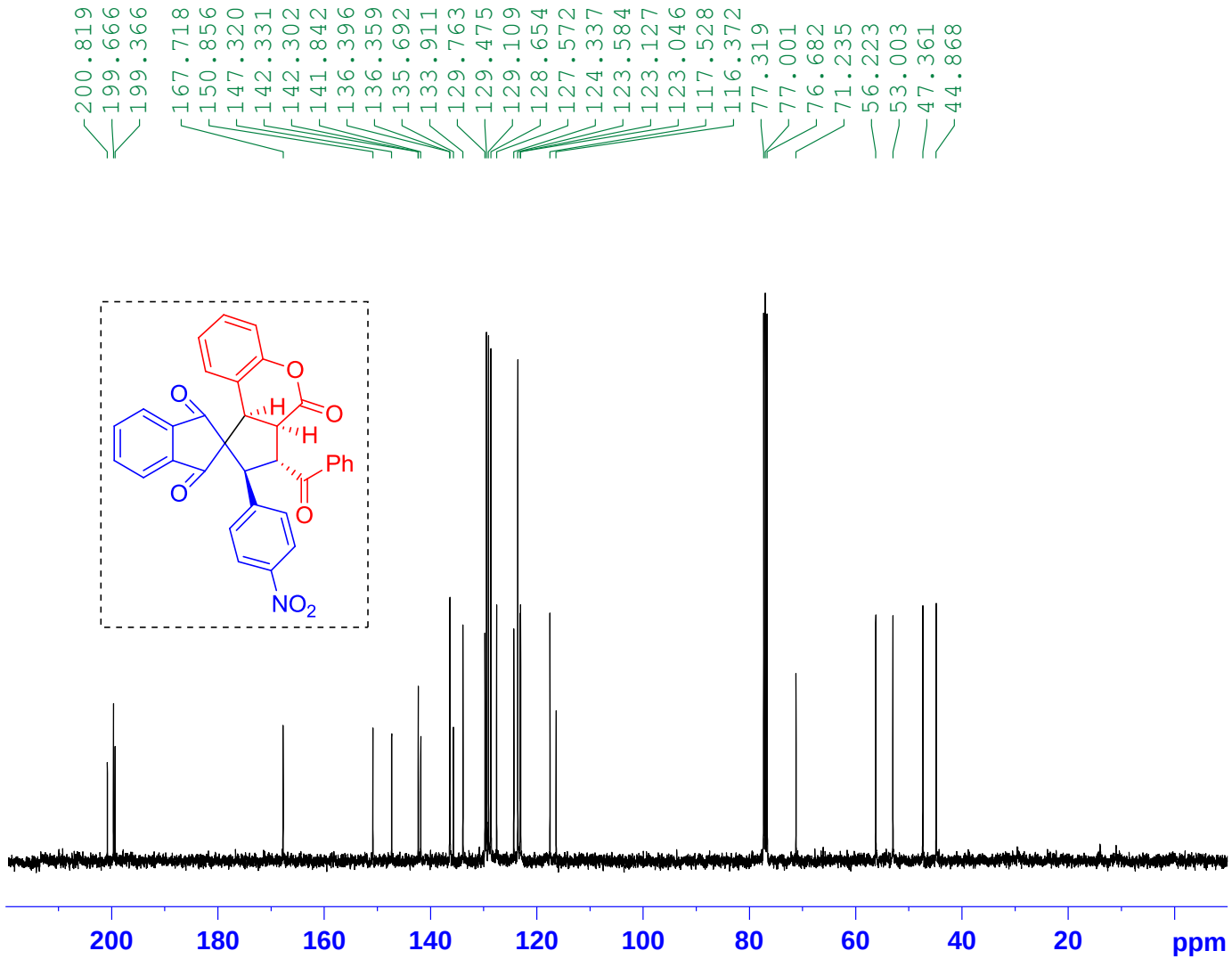

```
Current Data Parameters
NAME                   3aa
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_             20160627
Time                 17.05
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                   32768
SOLVENT              CDCl3
NS                    1378
DS                       0
SWH              24038.461 Hz
FIDRES            0.733596 Hz
AQ                0.6815744 sec
RG                   198.09
DW                   20.800 usec
DE                    6.50 usec
TE                   298.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          100.6228298 MHz
NUC1                   13C
P1                   10.00 usec
PLW1          47.50000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2                90.00 usec
PLW2          15.00000000 W
PLW12          0.33750001 W
PLW13          0.27338001 W

F2 - Processing parameters
SI                   32768
SF            100.6127839 MHz
WDW                     EM
SSB       0
LB                    2.00 Hz
GB        0
PC                    1.00
```

^{1}H NMR spectrum of **3ab** ($CDCl_3$)



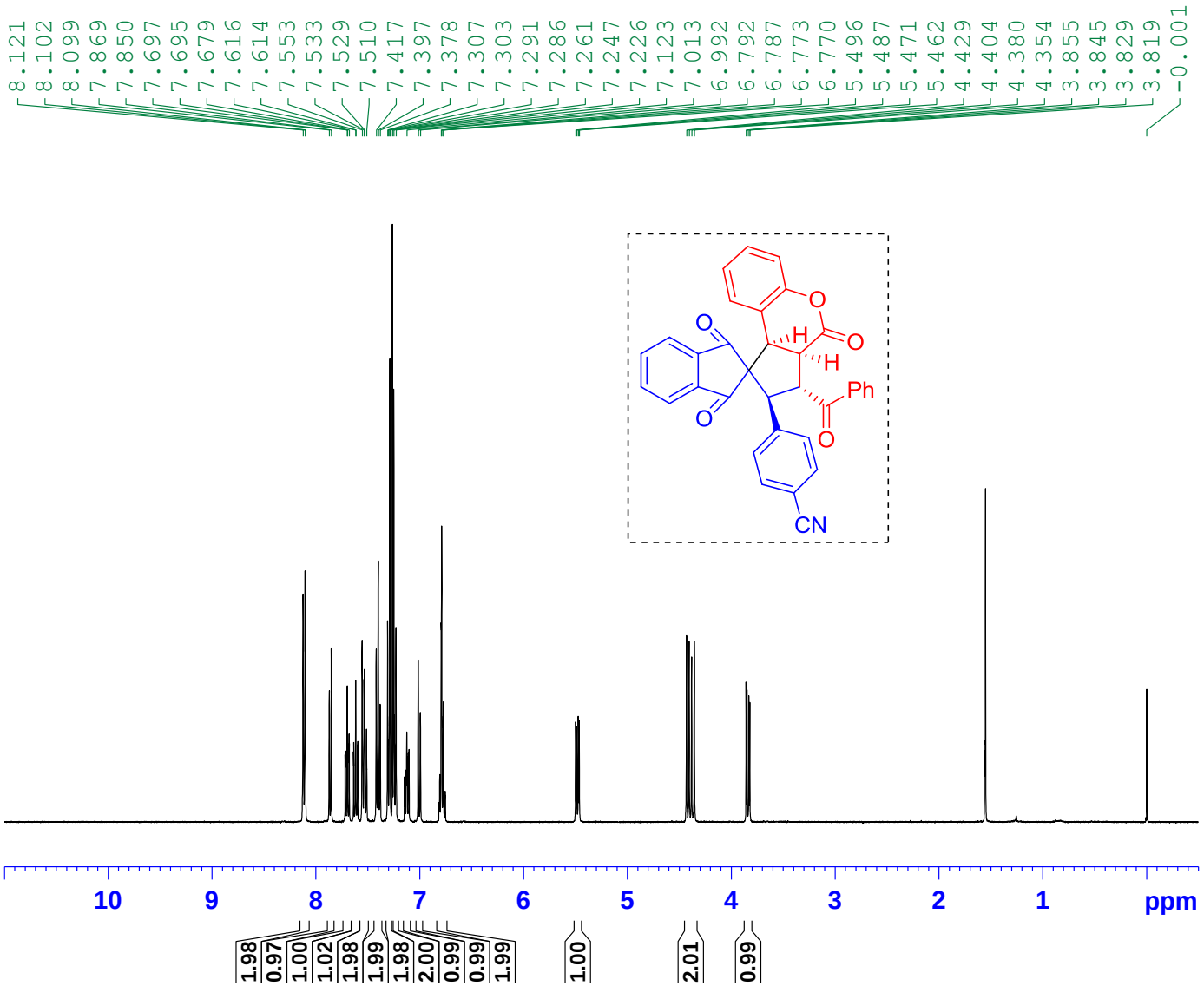

```
Current Data Parameters
NAME                  3ab
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170531
Time                21.30
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     32
DS                      0
SWH              7211.539 Hz
FIDRES           0.220079 Hz
AQ               2.2719147 sec
RG                  57.42
DW                69.333 usec
DE                10.50 usec
TE                298.2 K
D1           2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 12.90 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                  16384
SF           400.1300019 MHz
WDW                    EM
SSB       0
LB        0 Hz
GB        0
PC                  1.00
```

^{13}C NMR spectrum of **3ab** ($CDCl_3$)



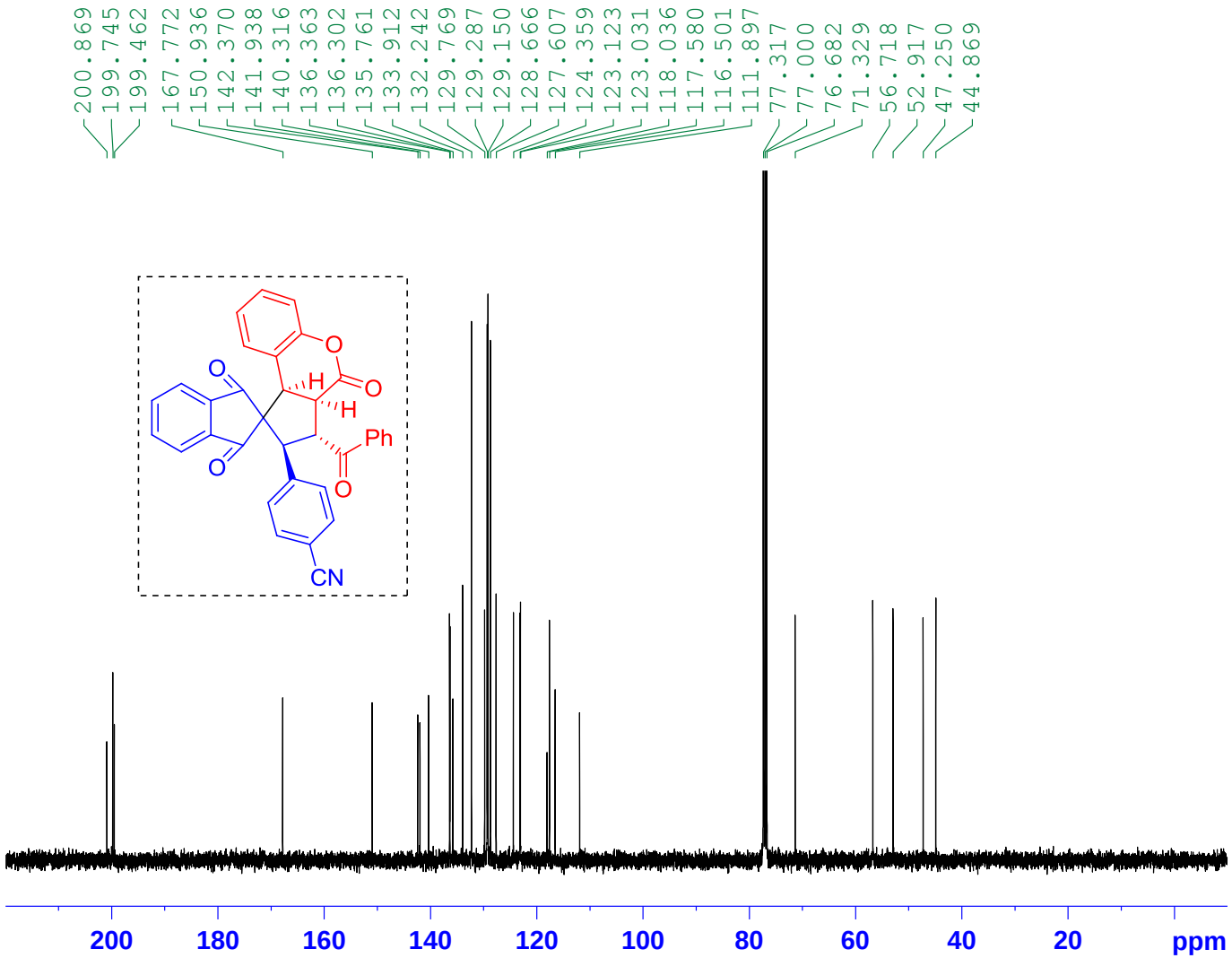

```
Current Data Parameters
NAME                  3ab
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160531
Time               17.16
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  118
DS                    0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                298.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        47.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        15.00000000 W
PLW12        0.33750001 W
PLW13        0.27338001 W

F2 - Processing parameters
SI                32768
SF          100.6127773 MHz
WDW                 EM
SSB       0
LB                2.00 Hz
GB        0
PC                1.00
```

^{1}H NMR spectrum of **3ac** ($CDCl_3$)



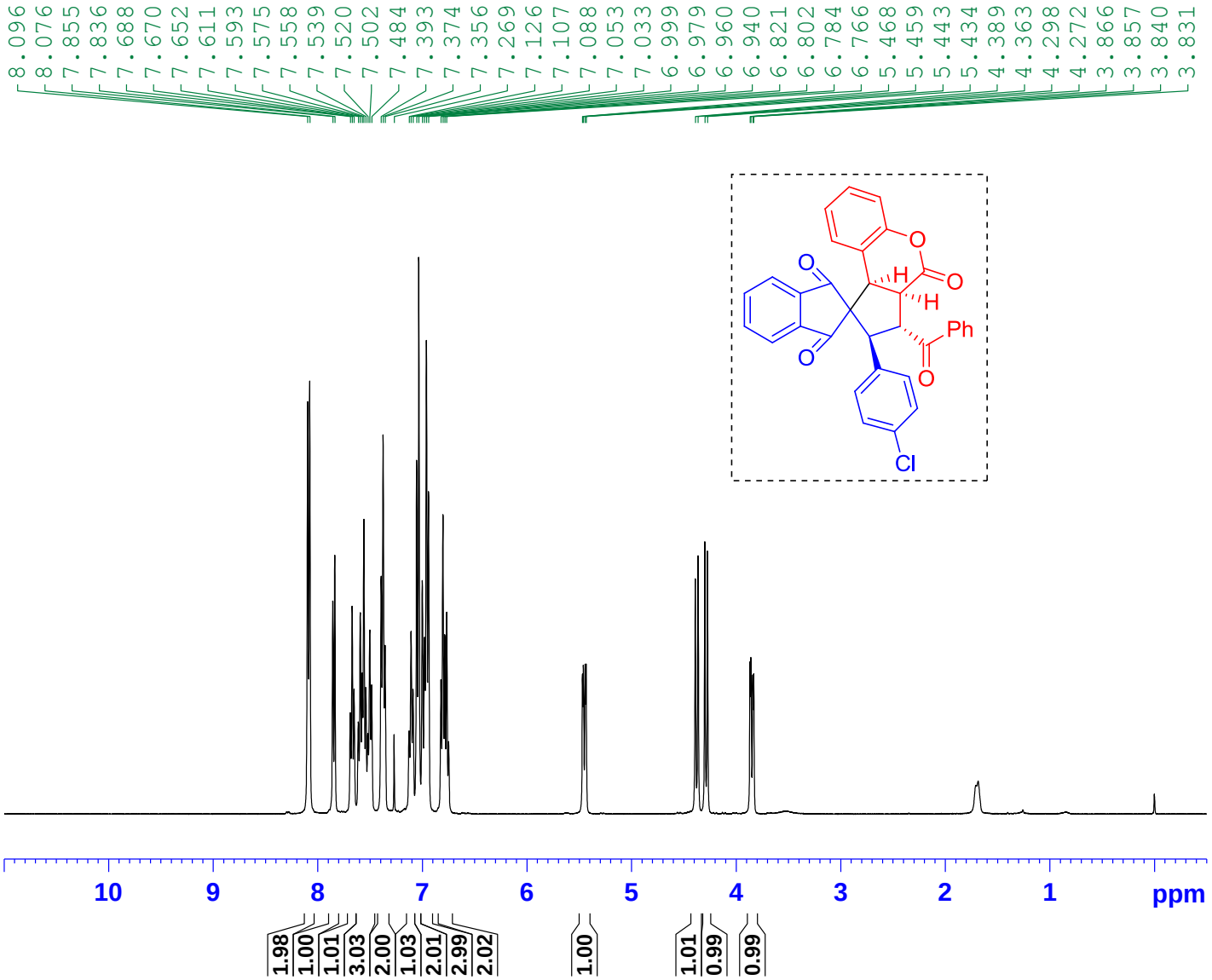

```
Current Data Parameters
NAME                  3ac
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170505
Time                15.35
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     32
DS                      0
SWH              7211.539 Hz
FIDRES           0.220079 Hz
AQ               2.2719147 sec
RG                 198.09
DW                 69.333 usec
DE                 10.50 usec
TE                 298.9 K
D1            2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 12.90 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                  16384
SF           400.1300099 MHz
WDW                    EM
SSB       0
LB        0 Hz
GB        0
PC                  1.00
```

^{13}C NMR spectrum of **3ac** ($CDCl_3$)



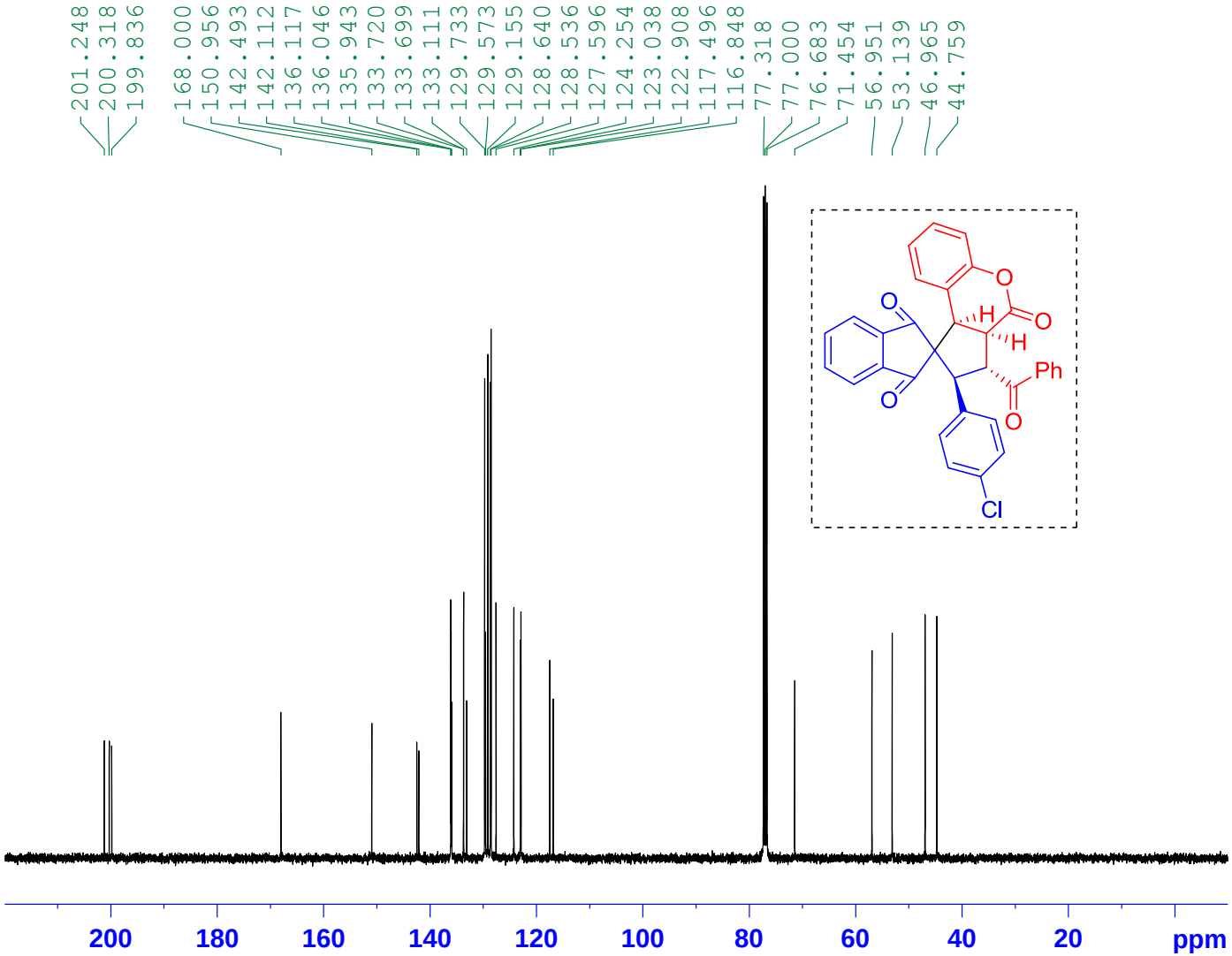

```
Current Data Parameters
NAME                   3ac
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_             20170507
Time                 18.22
INSTRUM              spect
PROBHD    5 mm BBO BB-1H
PULPROG            zgpg30
TD                   32768
SOLVENT              CDCl3
NS                     912
DS                       0
SWH              24038.461 Hz
FIDRES            0.733596 Hz
AQ                0.6815744 sec
RG                   5792.6
DW                  20.800 usec
DE                   6.50 usec
TE                  299.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.00 usec
PL1                   7.00 dB
SFO1          100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                   1H
PCPD2                90.00 usec
PL2                   1.80 dB
PL12                 17.00 dB
PL13                 20.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF            100.6127728 MHz
WDW                     EM
SSB          0
LB                    1.00 Hz
GB           0
PC                    1.00
```

^{1}H NMR spectrum of **3ad** ($CDCl_3$)



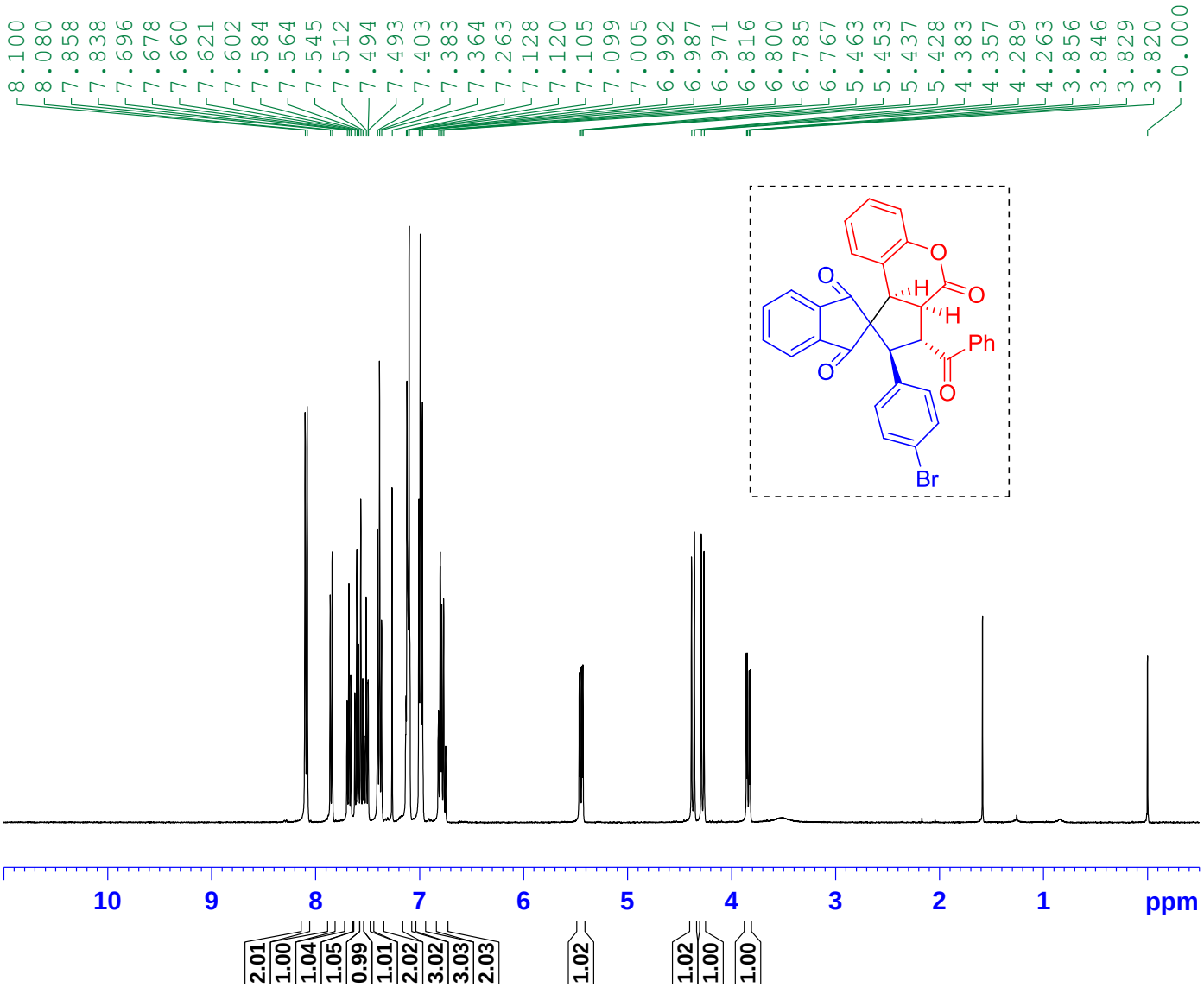

```
Current Data Parameters
NAME                   3ad
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20170527
Time                 22.57
INSTRUM              spect
PROBHD   5 mm BBO BB-1H
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      32
DS                       0
SWH               7246.377 Hz
FIDRES            0.221142 Hz
AQ               2.2609921 sec
RG                     114
DW                  69.000 usec
DE                   6.50 usec
TE                  298.6 K
D1             2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   14.40 usec
PL1                   1.80 dB
SFO1          400.1324008 MHz

F2 - Processing parameters
SI                   16384
SF            400.1300060 MHz
WDW                     EM
SSB       0
LB        0 Hz
GB        0
PC                    1.00
```

^{13}C NMR spectrum of **3ad** ($CDCl_3$)



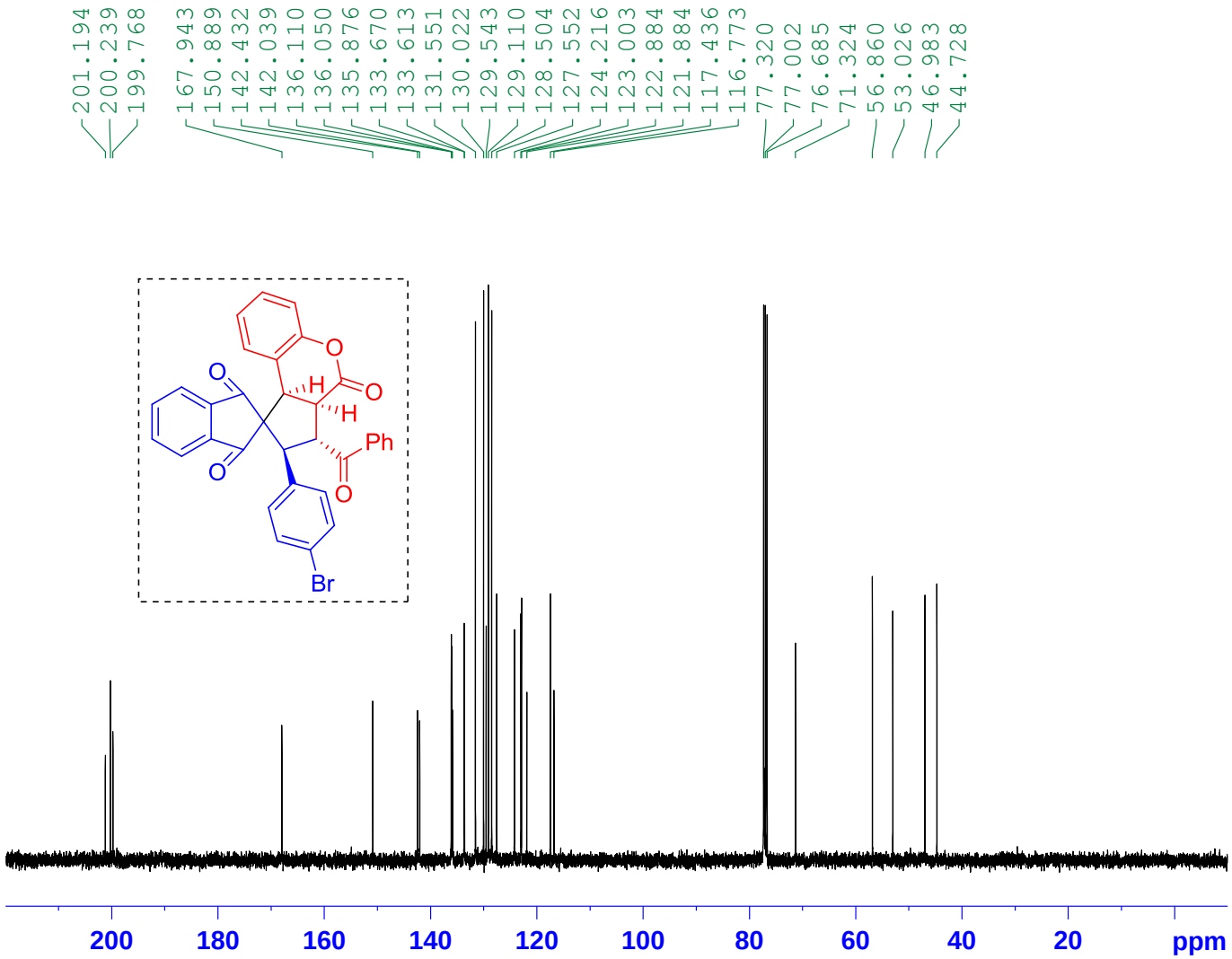

```
Current Data Parameters
NAME                   3ad
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20170527
Time                 23.02
INSTRUM              spect
PROBHD    5 mm BBO BB-1H
PULPROG             zgpg30
TD                   32768
SOLVENT              CDCl3
NS                    1305
DS                       0
SWH              24038.461 Hz
FIDRES            0.733596 Hz
AQ                0.6815744 sec
RG                   5792.6
DW                  20.800 usec
DE                   6.50 usec
TE                  299.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.00 usec
PL1                   7.00 dB
SFO1          100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                   1H
PCPD2                90.00 usec
PL2                   1.80 dB
PL12                 17.00 dB
PL13                 20.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF            100.6127746 MHz
WDW                     EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.00
```

^{1}H NMR spectrum of **3ae** ($CDCl_3$)



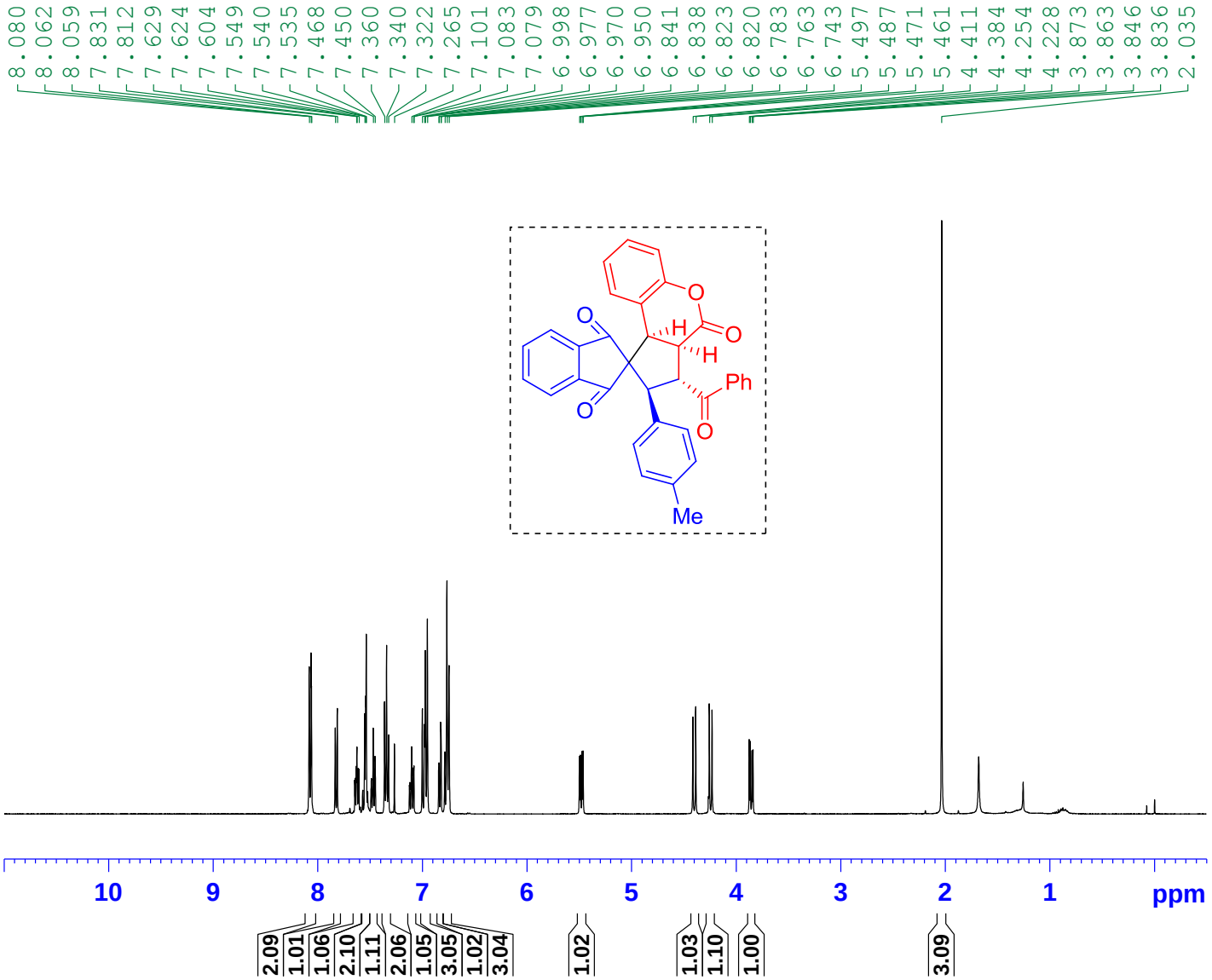

```
Current Data Parameters
NAME                  3ae
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_           20180118
Time               20.44
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH             7246.377 Hz
FIDRES          0.221142 Hz
AQ             2.2609921 sec
RG                   181
DW                69.000 usec
DE                 6.50 usec
TE                298.6 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.00 usec
PL1       0 dB
SFO1         400.1324008 MHz

F2 - Processing parameters
SI                 16384
SF           400.1300084 MHz
WDW                   EM
SSB       0
LB        0 Hz
GB        0
PC                  1.00
```

^{13}C NMR spectrum of **3ae** ($CDCl_3$)



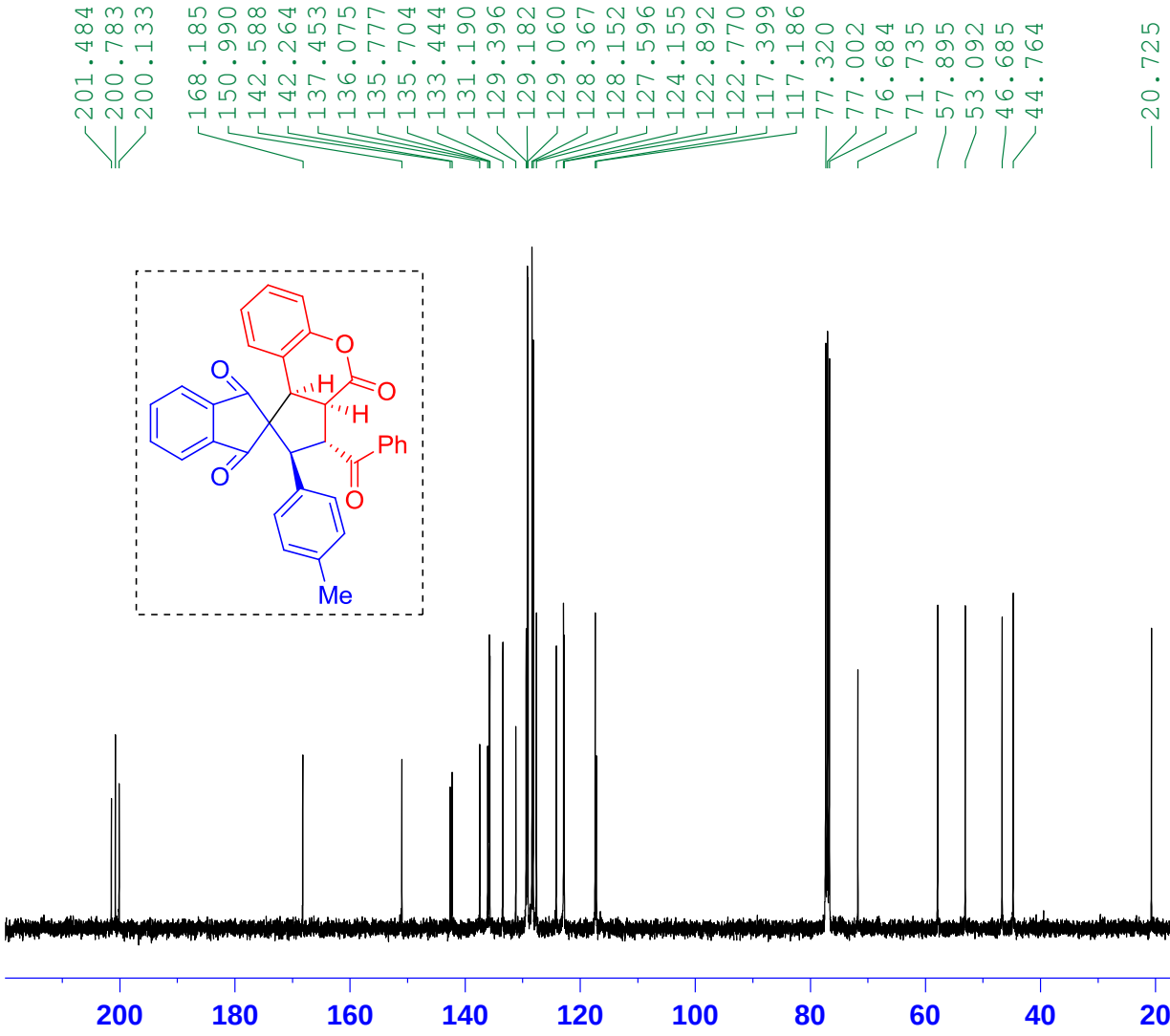

```
Current Data Parameters
NAME                   3ae
EXPNO                    6
PROCNO                   1

F2 - Acquisition Parameters
Date_             20160607
Time                 18.25
INSTRUM              spect
PROBHD    5 mm BBO BB-1H
PULPROG            zgpg30
TD                   32768
SOLVENT              CDCl3
NS                     156
DS                       0
SWH              24038.461 Hz
FIDRES            0.733596 Hz
AQ                0.6815744 sec
RG                   5792.6
DW                  20.800 usec
DE                   6.50 usec
TE                  299.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                   9.00 usec
PL1                  7.00 dB
SFO1          100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                  1.80 dB
PL12                17.00 dB
PL13                20.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF            100.6127789 MHz
WDW                     EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.00
```

^{1}H NMR spectrum of **3af** ($CDCl_3$)



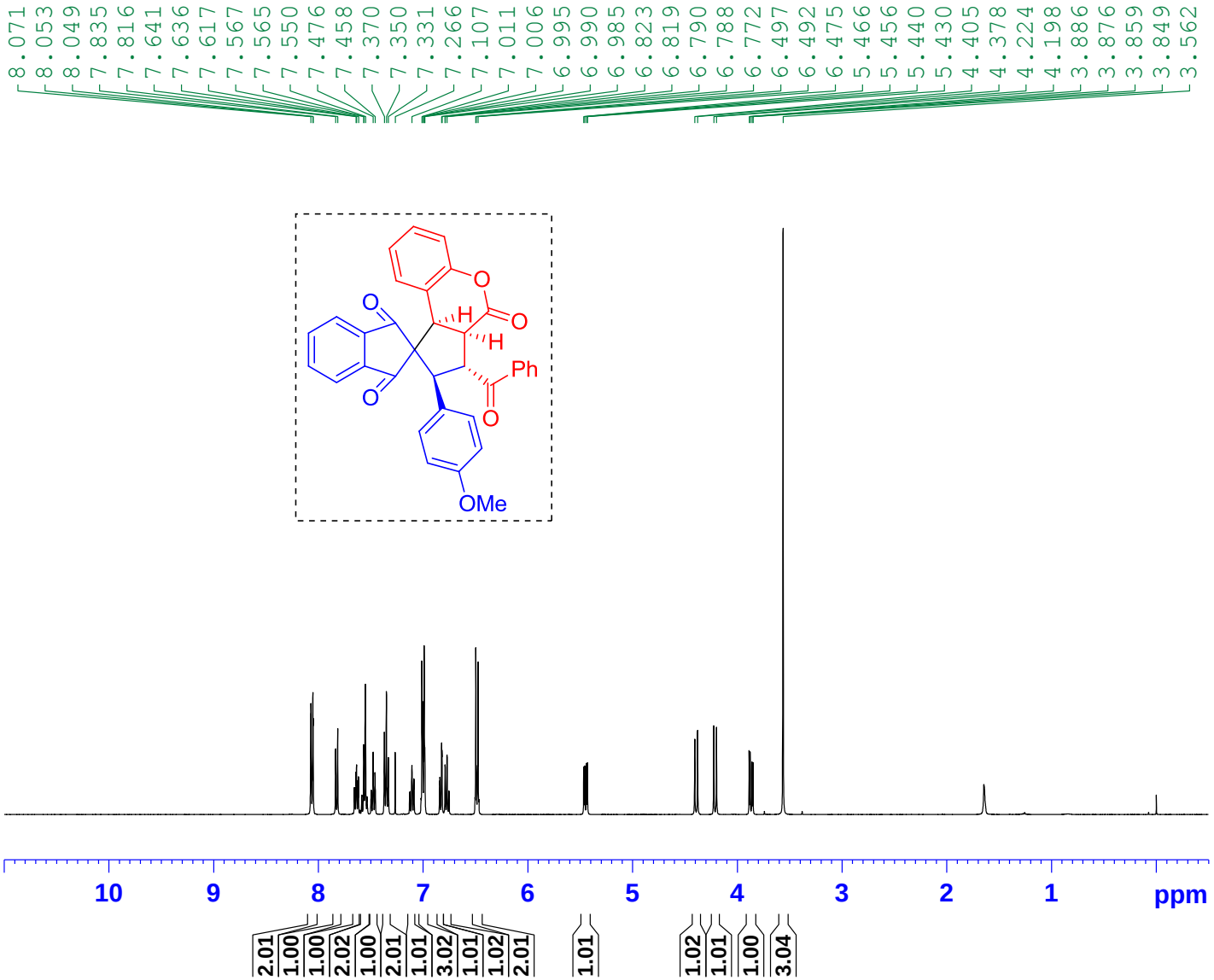

```
Current Data Parameters
NAME                   3af
EXPNO                    6
PROCNO                   1

F2 - Acquisition Parameters
Date_             20160627
Time                 16.28
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      16
DS                       0
SWH               7211.539 Hz
FIDRES           0.220079 Hz
AQ               2.2719147 sec
RG                   78.51
DW                  69.333 usec
DE                  10.50 usec
TE                  298.3 K
D1              2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                    1H
P1                   12.90 usec
PLW1          15.00000000 W

F2 - Processing parameters
SI                   16384
SF            400.1300077 MHz
WDW                     EM
SSB       0
LB        0 Hz
GB        0
PC                    1.00
```

^{13}C NMR spectrum of **3af** ($CDCl_3$)



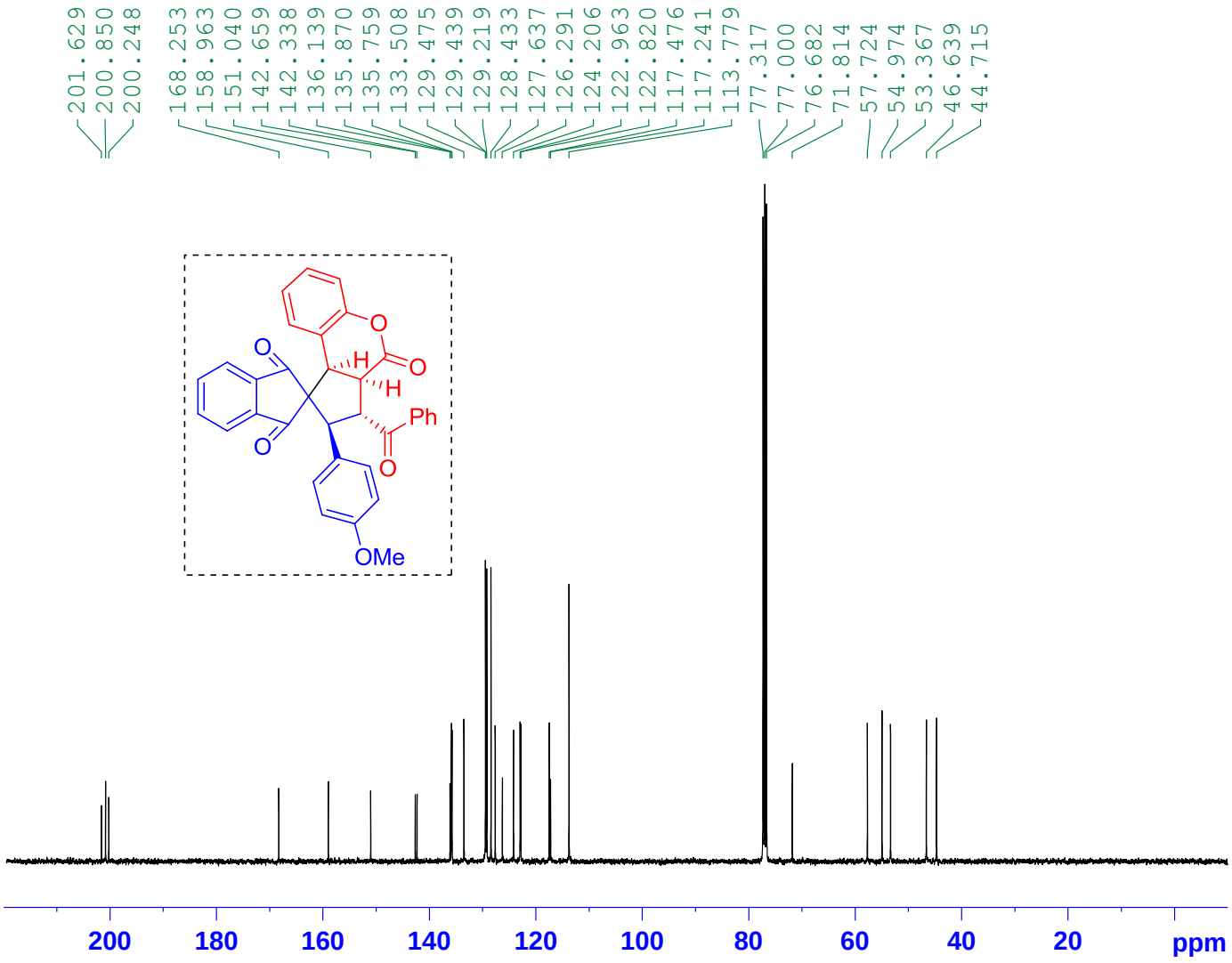

```
Current Data Parameters
NAME                  3af
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160607
Time               18.41
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  500
DS                    0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            0.6815744 sec
RG               5792.6
DW                20.800 usec
DE                 6.50 usec
TE                299.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.00 usec
PL1                 7.00 dB
SFO1        100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2              90.00 usec
PL2                 1.80 dB
PL12               17.00 dB
PL13               20.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127767 MHz
WDW                  EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.00
```

^{1}H NMR spectrum of **3ag** ($CDCl_3$)



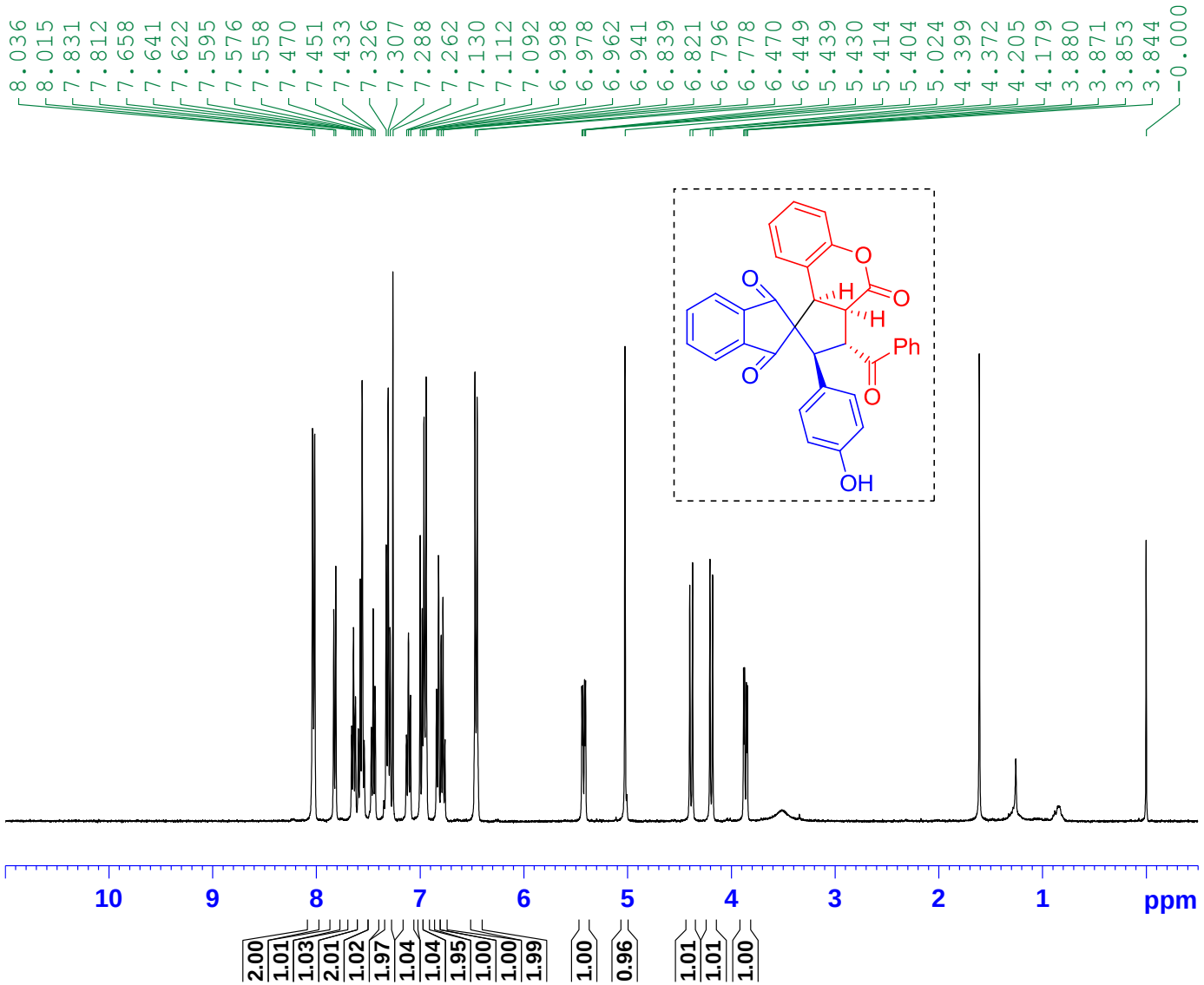

```
Current Data Parameters
NAME                   3ag
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20170527
Time                 20.58
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      32
DS                       0
SWH               7211.539 Hz
FIDRES            0.220079 Hz
AQ               2.2719147 sec
RG                   89.08
DW                  69.333 usec
DE                  10.50 usec
TE                  297.6 K
D1             2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                    1H
P1                   12.90 usec
PLW1          15.00000000 W

F2 - Processing parameters
SI                   16384
SF            400.1300074 MHz
WDW                     EM
SSB       0
LB        0 Hz
GB        0
PC                    1.00
```

^{13}C NMR spectrum of **3ag** ($CDCl_3$)



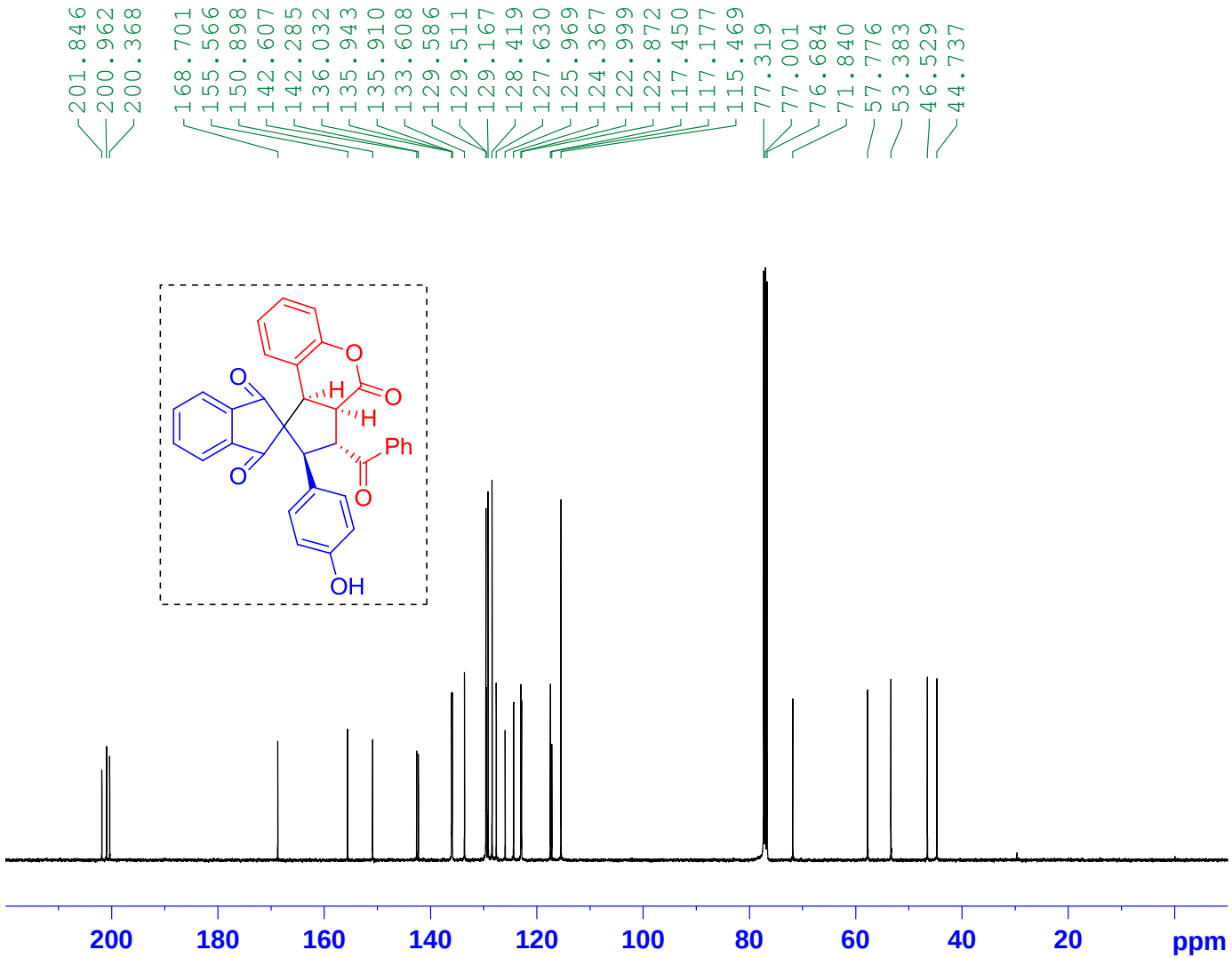

```
Current Data Parameters
NAME                  3ag
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170527
Time                21.02
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                  1479
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1         47.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         15.00000000 W
PLW12         0.33750001 W
PLW13         0.27338001 W

F2 - Processing parameters
SI                 32768
SF           100.6127721 MHz
WDW                  EM
SSB       0
LB                2.00 Hz
GB        0
PC                1.00
```

^{1}H NMR spectrum of **3ah** ($CDCl_3$)



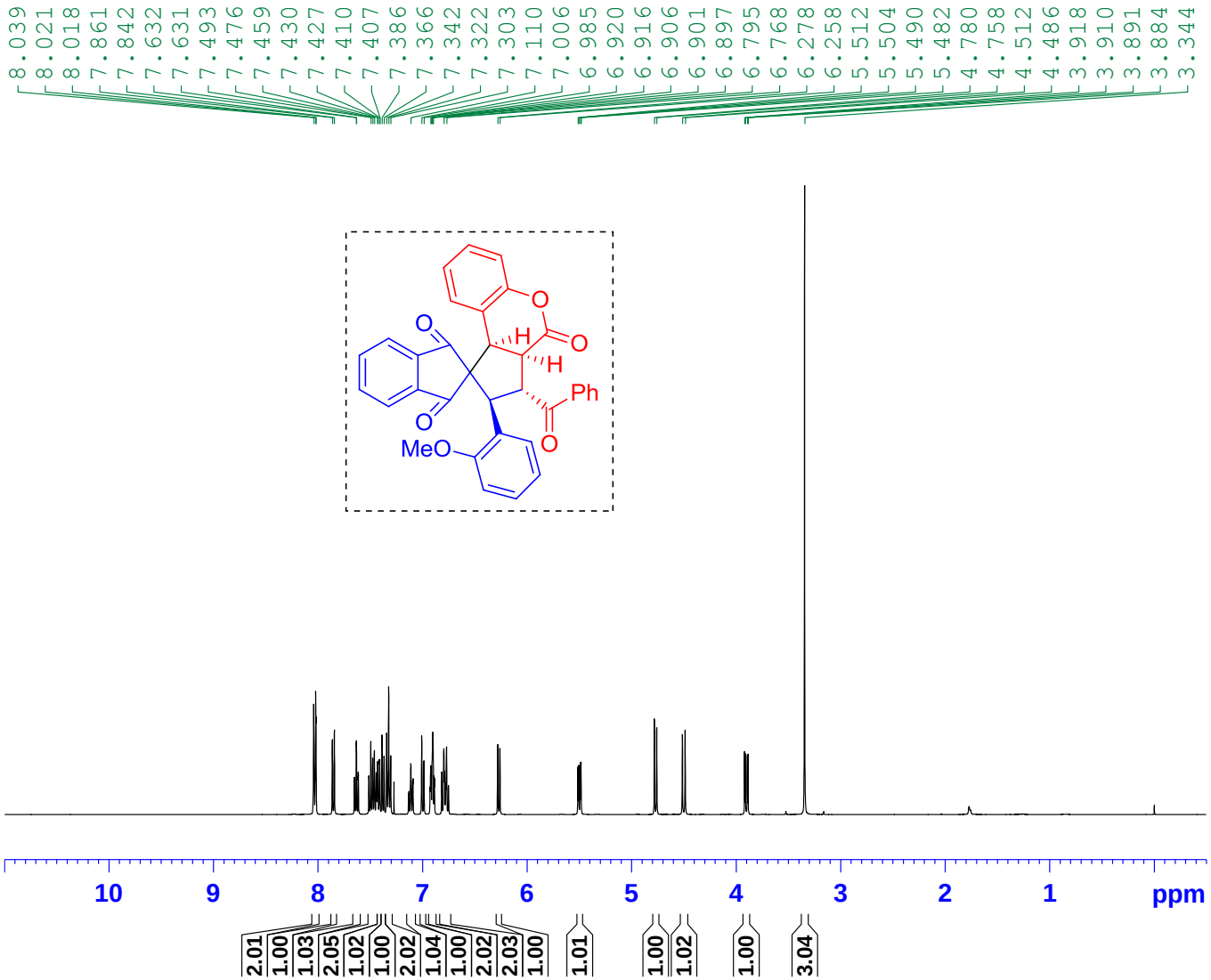

```
Current Data Parameters
NAME                  3ah
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_           20180119
Time              20.34
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG           zg30
TD               32768
SOLVENT           CDCl3
NS                  32
DS                   0
SWH            7246.377 Hz
FIDRES         0.221142 Hz
AQ             2.2609921 sec
RG                228.1
DW               69.000 usec
DE                6.50 usec
TE                298.4 K
D1           2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                 1H
P1                15.00 usec
PL1       0 dB
SFO1         400.1324008 MHz

F2 - Processing parameters
SI                16384
SF           400.1300091 MHz
WDW                  EM
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```

^{13}C NMR spectrum of **3ah** ($CDCl_3$)



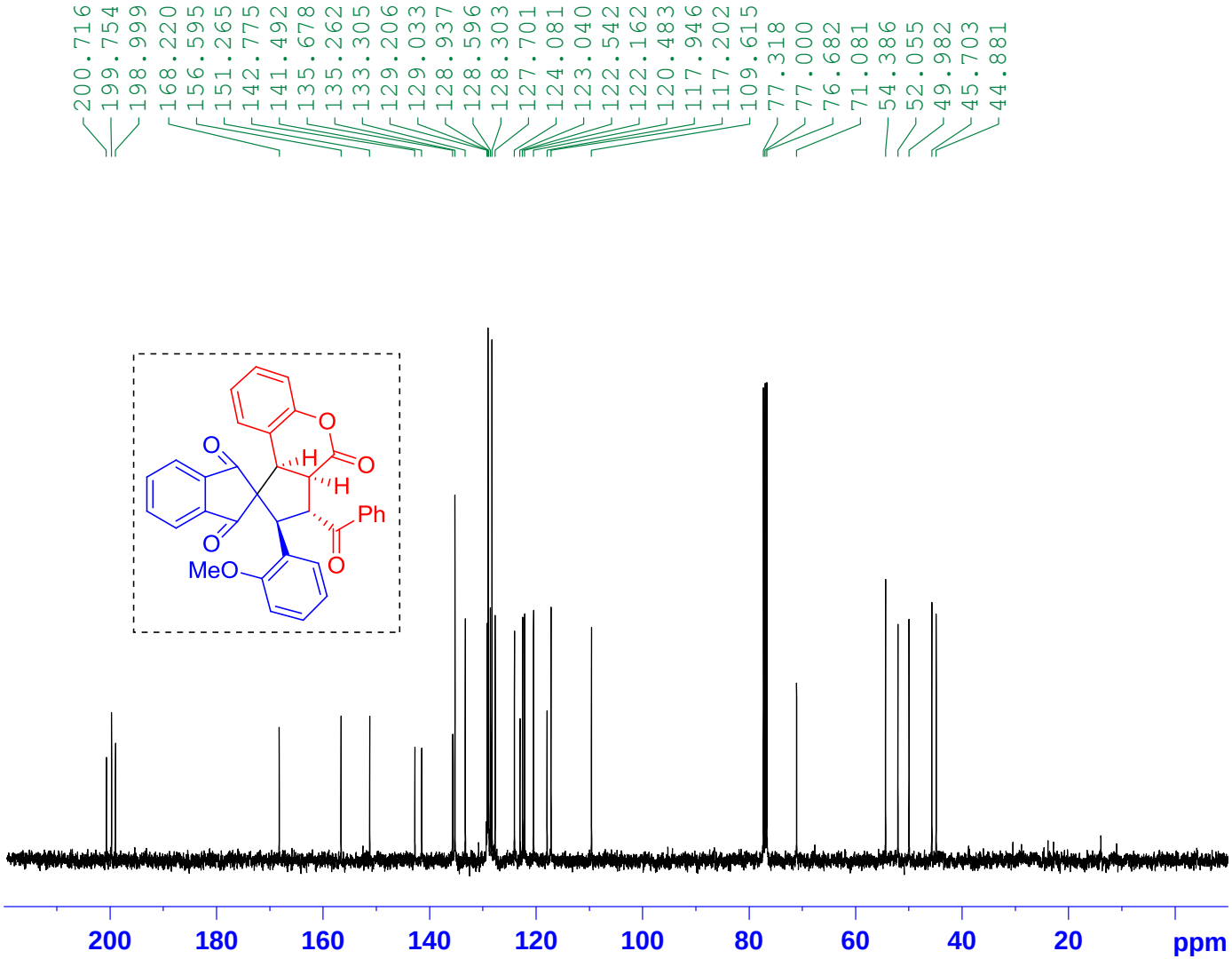

```
Current Data Parameters
NAME                   3ah
EXPNO                    5
PROCNO                   1

F2 - Acquisition Parameters
Date_           20180120
Time                 0.52
INSTRUM             spect
PROBHD    5 mm BBO BB-1H
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                 12019
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                  4096
DW                20.800 usec
DE                 6.50 usec
TE                298.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.45 usec
PL1                 7.00 dB
SFO1         100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2       0 dB
PL12               15.00 dB
PL13               20.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127731 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.00
```

^{1}H NMR spectrum of **3ai** ($CDCl_3$)



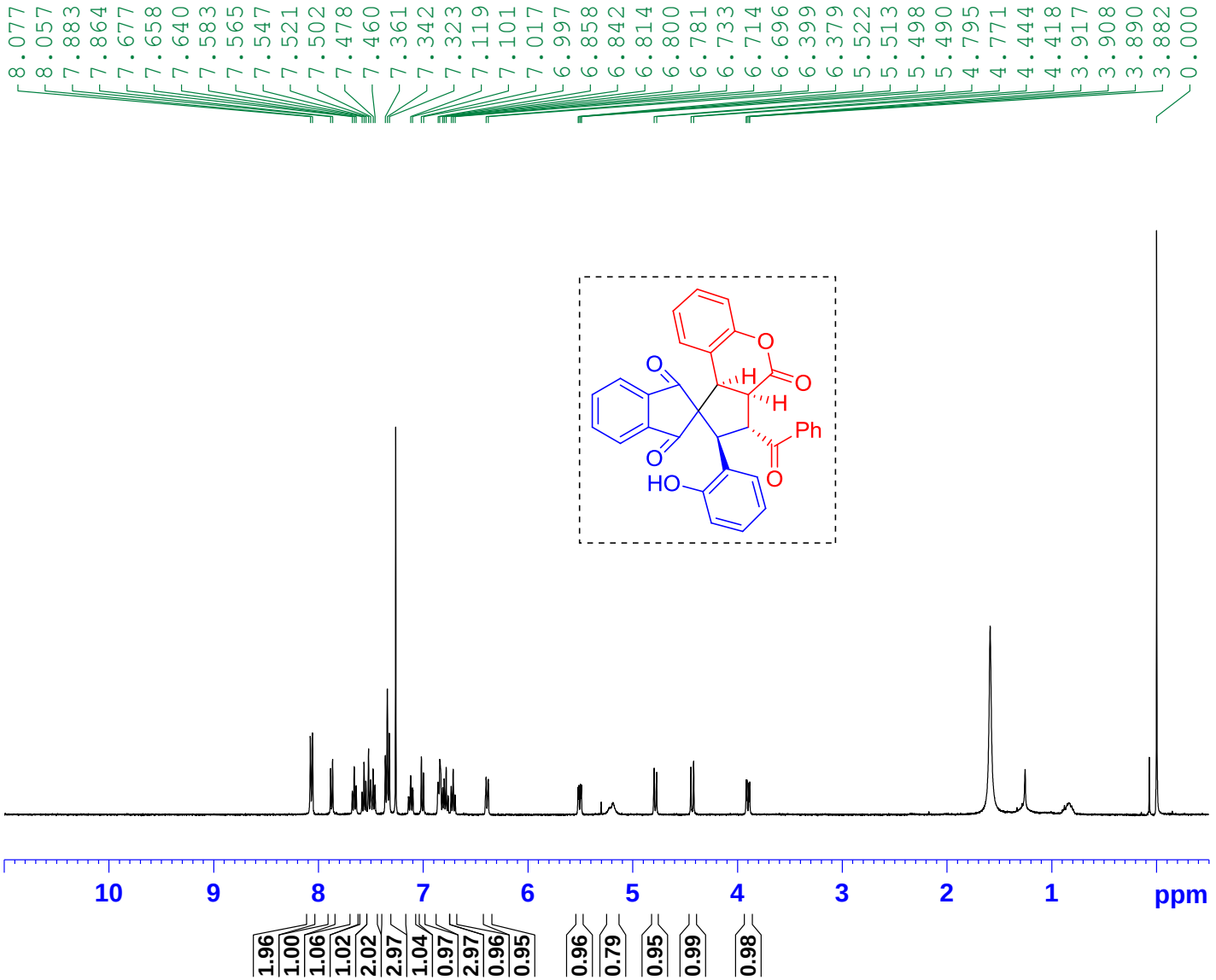

```
Current Data Parameters
NAME                   3ai
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_           20170524
Time               15.32
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719147 sec
RG                 57.42
DW                69.333 usec
DE                10.50 usec
TE                298.2 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 12.90 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                 16384
SF           400.1300055 MHz
WDW                   EM
SSB       0
LB        0 Hz
GB        0
PC                  1.00
```

^{13}C NMR spectrum of **3ai** ($CDCl_3$)



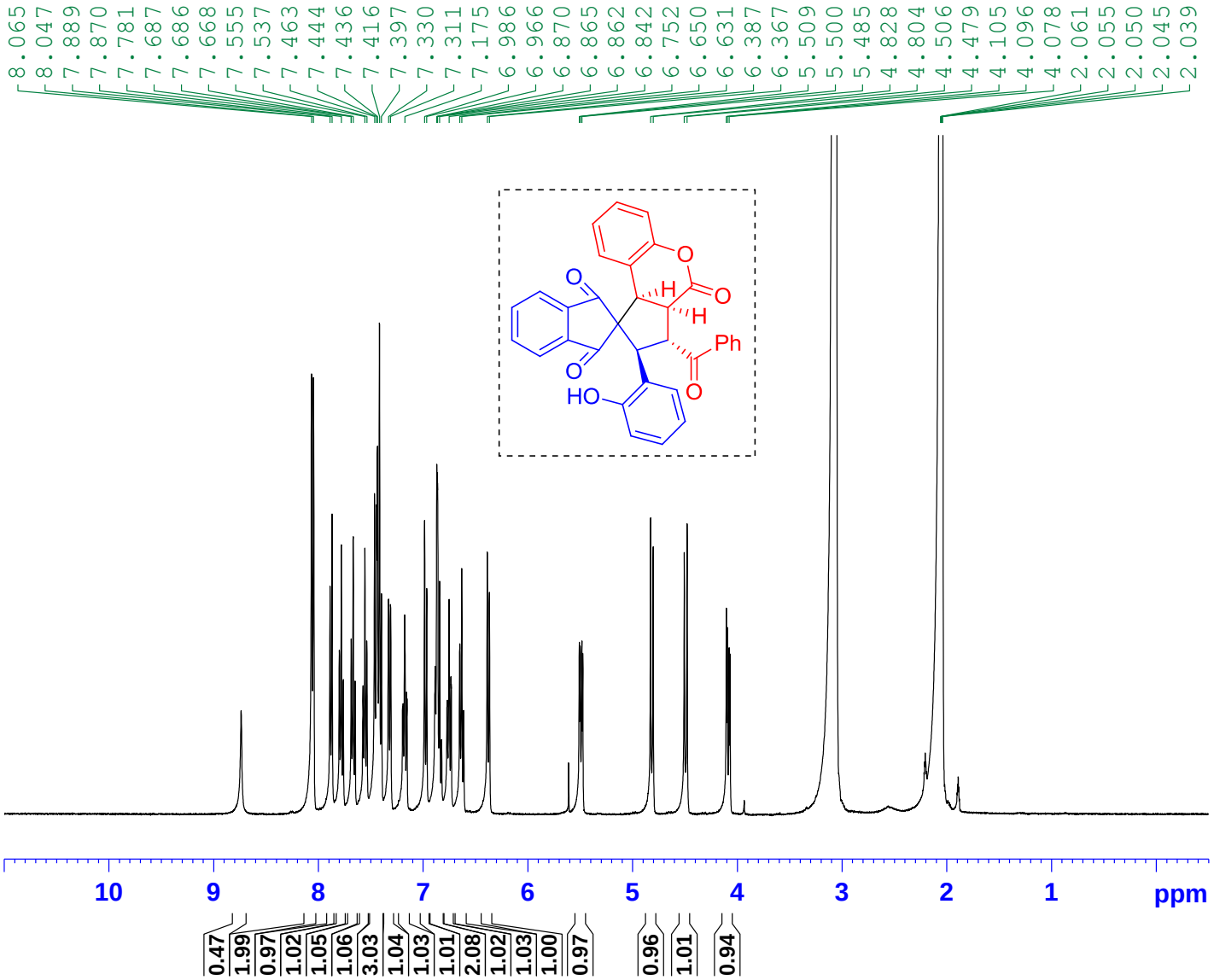

```
Current Data Parameters
NAME                  3ai
EXPNO                   8
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160606
Time                20.28
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 32768
SOLVENT             CDCl3
NS                     50
DS                      0
SWH             24038.461 Hz
FIDRES           0.733596 Hz
AQ               0.6815744 sec
RG                 198.09
DW                 20.800 usec
DE                  6.50 usec
TE                 299.3 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
PLW1         47.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         15.00000000 W
PLW12         0.33750001 W
PLW13         0.27338001 W

F2 - Processing parameters
SI                 32768
SF           100.6127806 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.00
```

^{1}H NMR spectrum of **3aj** ($CDCl_3$)



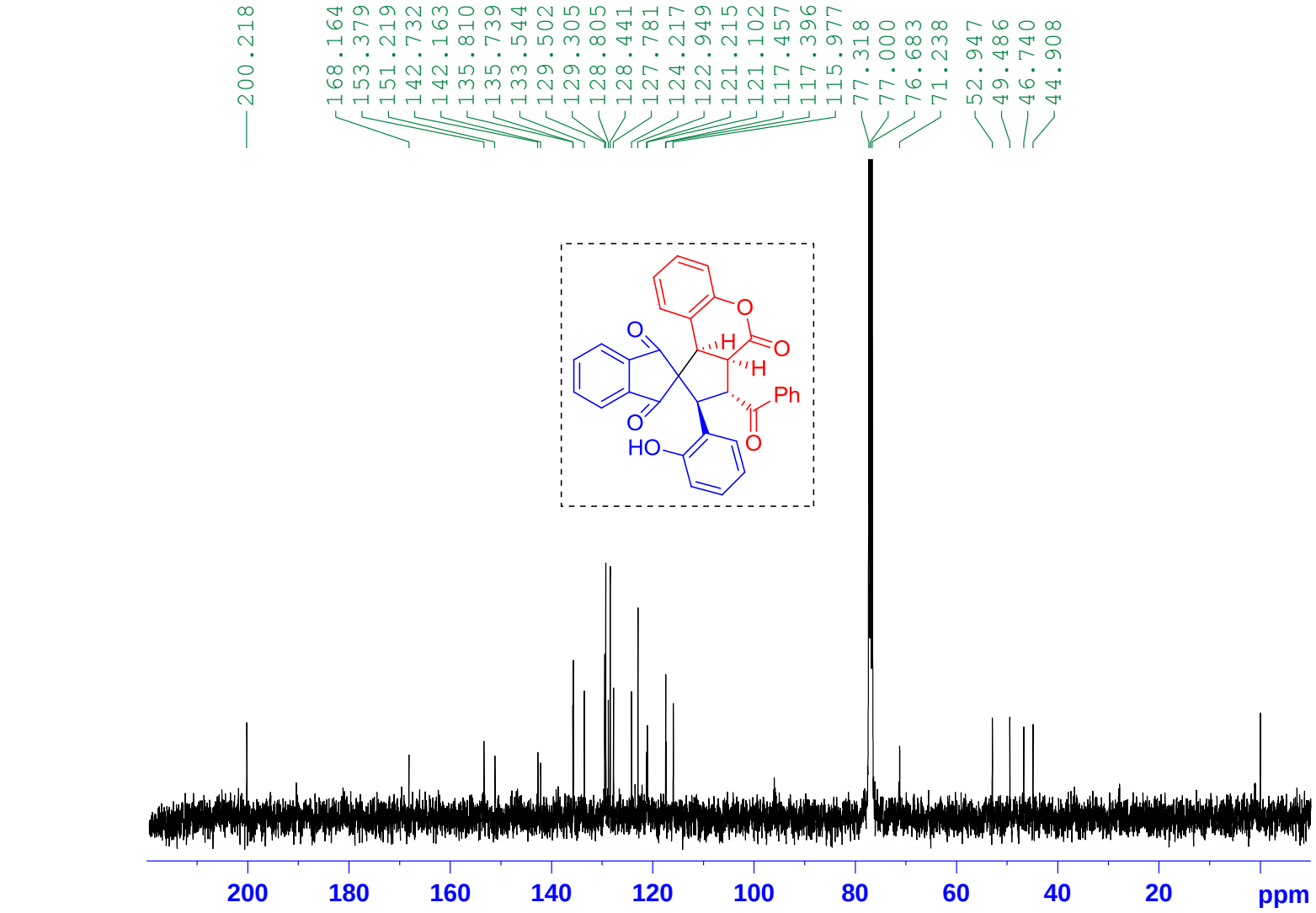

```
Current Data Parameters
NAME                  3aj
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160530
Time                17.40
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              7211.539 Hz
FIDRES           0.220079 Hz
AQ               2.2719147 sec
RG                 198.09
DW                 69.333 usec
DE                 10.50 usec
TE                 298.2 K
D1              2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                   1H
P1                  12.90 usec
PLW1          15.00000000 W

F2 - Processing parameters
SI                  16384
SF            400.1300092 MHz
WDW                    EM
SSB       0
LB        0 Hz
GB        0
PC                   1.00
```

^{1}H NMR spectrum of **3aj** (Acetone-d^{6})



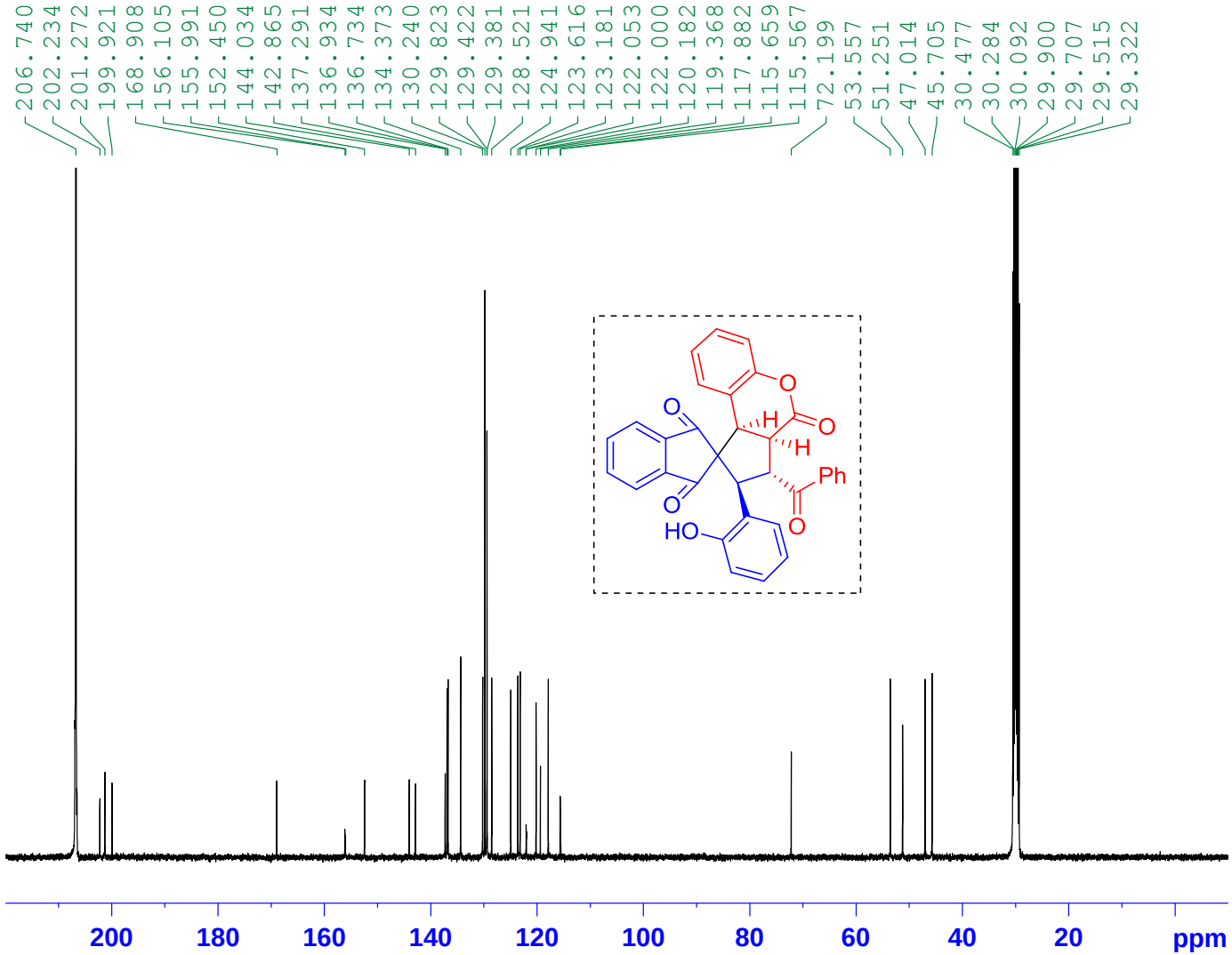

```
Current Data Parameters
NAME                  439
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20171227
Time                15.32
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG              zg30
TD                  32768
SOLVENT           Acetone
NS                     32
DS                      0
SWH              7246.377 Hz
FIDRES           0.221142 Hz
AQ              2.2609921 sec
RG                    114
DW                 69.000 usec
DE                  6.50 usec
TE                 298.3 K
D1            2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 15.00 usec
PL1       0 dB
SFO1          400.1324008 MHz

F2 - Processing parameters
SI                  16384
SF            400.1300065 MHz
WDW                    EM
SSB       0
LB        0 Hz
GB        0
PC                  1.00
```

^{13}C NMR spectrum of **3aj** ($CDCl_3$)



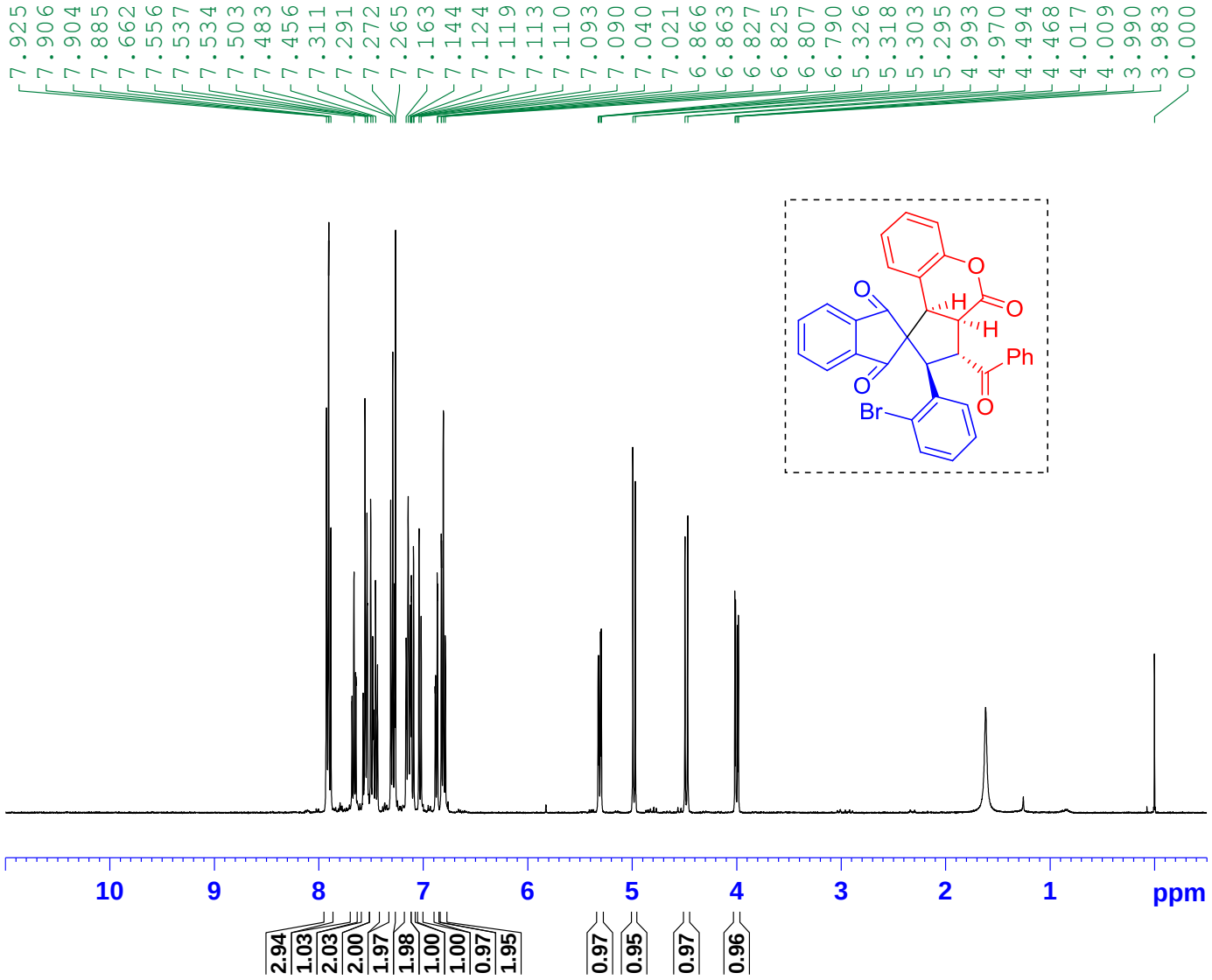

```
Current Data Parameters
NAME                   3aj
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160530
Time              18.54
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                 1580
DS                    0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            0.6815744 sec
RG               198.09
DW               20.800 usec
DE                6.50 usec
TE                299.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        47.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        15.00000000 W
PLW12        0.33750001 W
PLW13        0.27338001 W

F2 - Processing parameters
SI                32768
SF          100.6127693 MHz
WDW                EM
SSB       0
LB                2.00 Hz
GB        0
PC                1.00
```

^{13}C NMR spectrum of **3aj** (Acetone-d^{6})



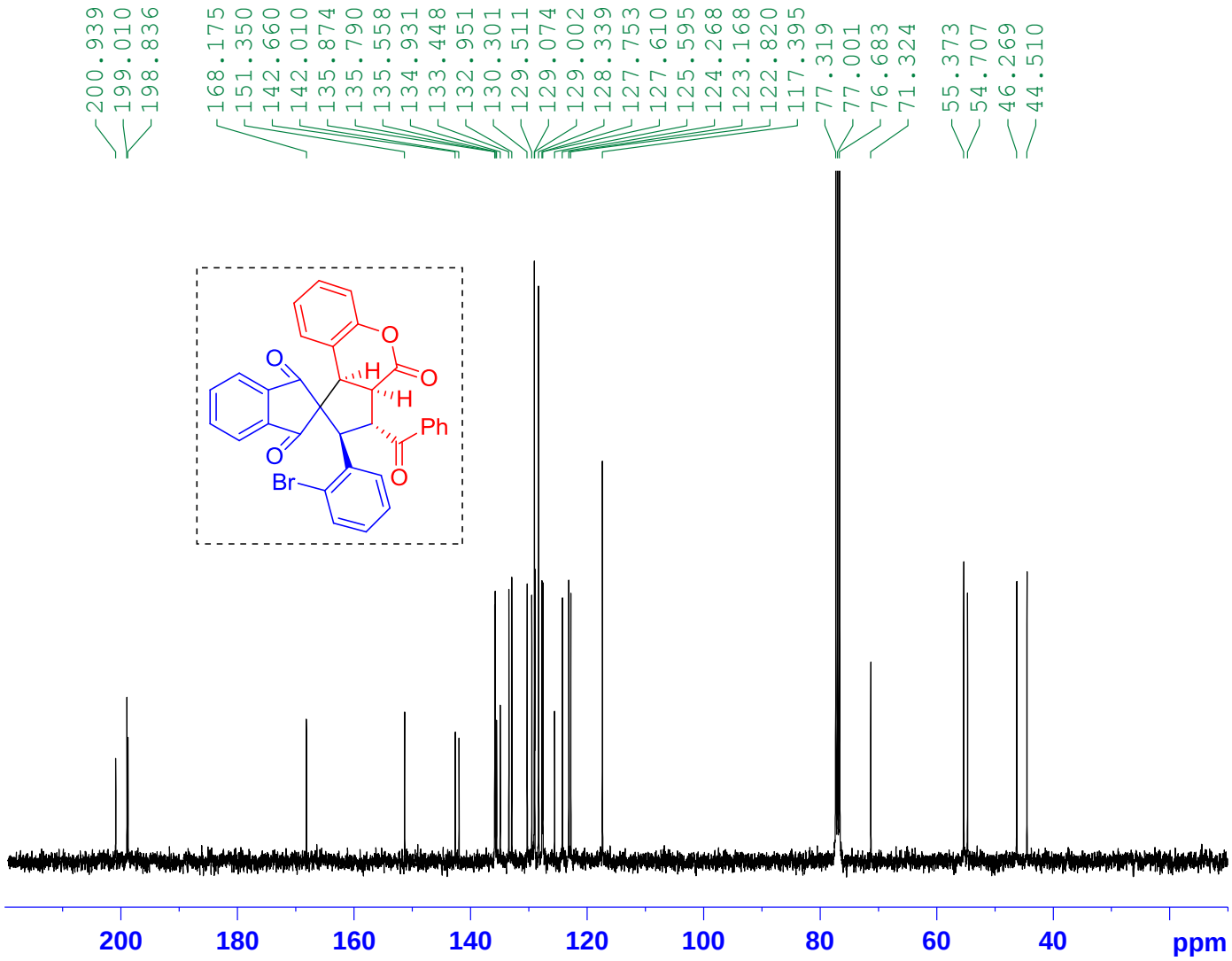

```
Current Data Parameters
NAME                  439
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20171228
Time                22.01
INSTRUM             spect
PROBHD    5 mm BBO BB-1H
PULPROG            zgpg30
TD                  32768
SOLVENT           Acetone
NS                  14547
DS                      0
SWH             24038.461 Hz
FIDRES           0.733596 Hz
AQ               0.6815744 sec
RG                   4096
DW                 20.800 usec
DE                  6.50 usec
TE                 298.5 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.45 usec
PL1                 7.00 dB
SFO1          100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2       0 dB
PL12               15.00 dB
PL13               20.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF            100.6126750 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.00
```

^{1}H NMR spectrum of **3ak** ($CDCl_3$)



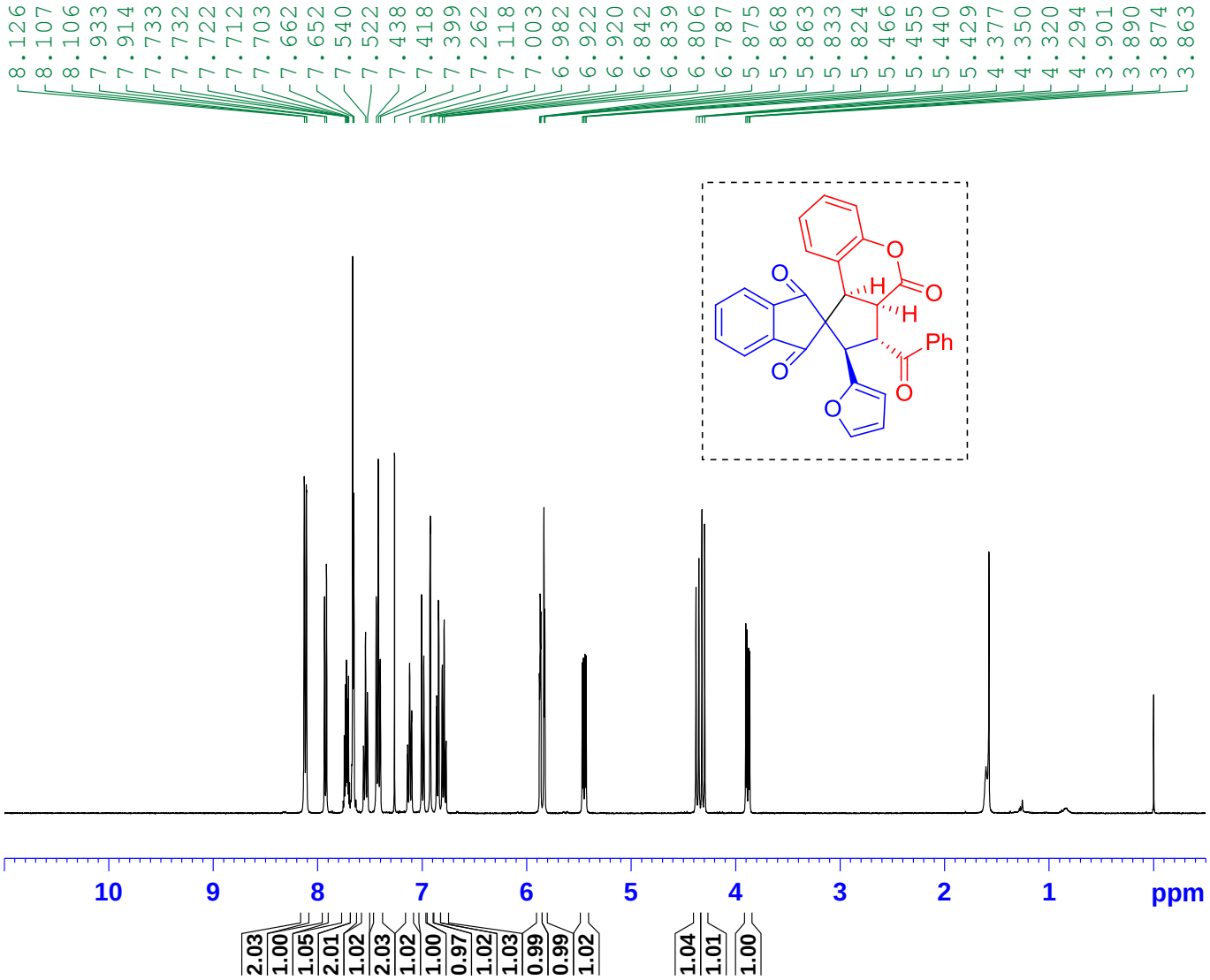

```
Current Data Parameters
NAME                   3ak
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20160418
Time                 17.02
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      16
DS                       0
SWH               7211.539 Hz
FIDRES            0.220079 Hz
AQ               2.2719147 sec
RG                   128.9
DW                  69.333 usec
DE                  10.50 usec
TE                  297.6 K
D1              2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                    1H
P1                   12.90 usec
PLW1          15.00000000 W

F2 - Processing parameters
SI                   16384
SF            400.1300081 MHz
WDW                     EM
SSB       0
LB        0 Hz
GB        0
PC                    1.00
```

^{13}C NMR spectrum of **3ak** ($CDCl_3$)



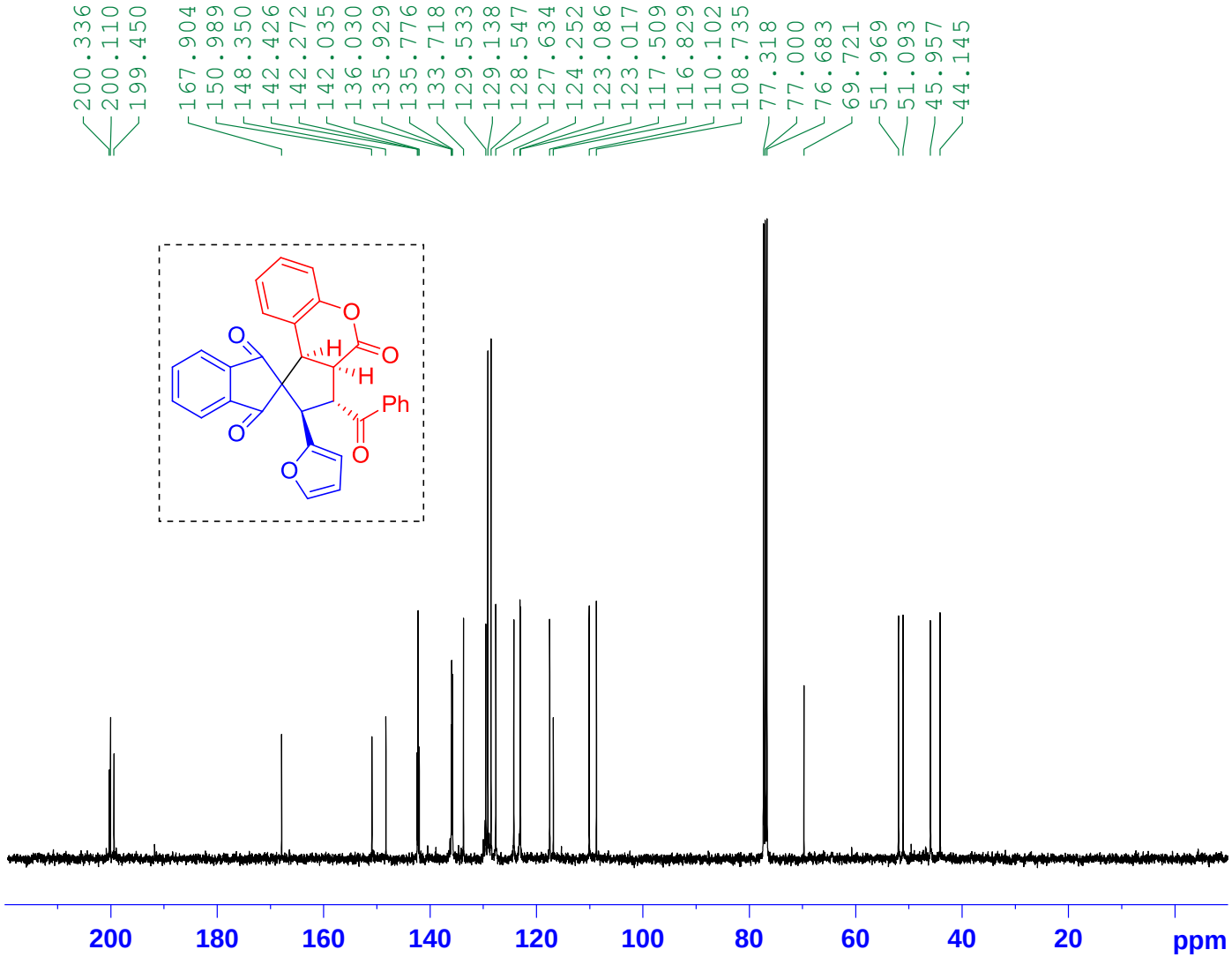

```
Current Data Parameters
NAME                   3ak
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_           20170524
Time                15.56
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                  32768
SOLVENT             CDCl3
NS                    207
DS                      0
SWH             24038.461 Hz
FIDRES           0.733596 Hz
AQ               0.6815744 sec
RG                 198.09
DW                 20.800 usec
DE                  6.50 usec
TE                 299.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
PLW1          47.50000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          15.00000000 W
PLW12          0.33750001 W
PLW13          0.27338001 W

F2 - Processing parameters
SI                  32768
SF            100.6127737 MHz
WDW                    EM
SSB       0
LB                  2.00 Hz
GB        0
PC                  1.00
```

^{1}H NMR spectrum of **3al** ($CDCl_3$)



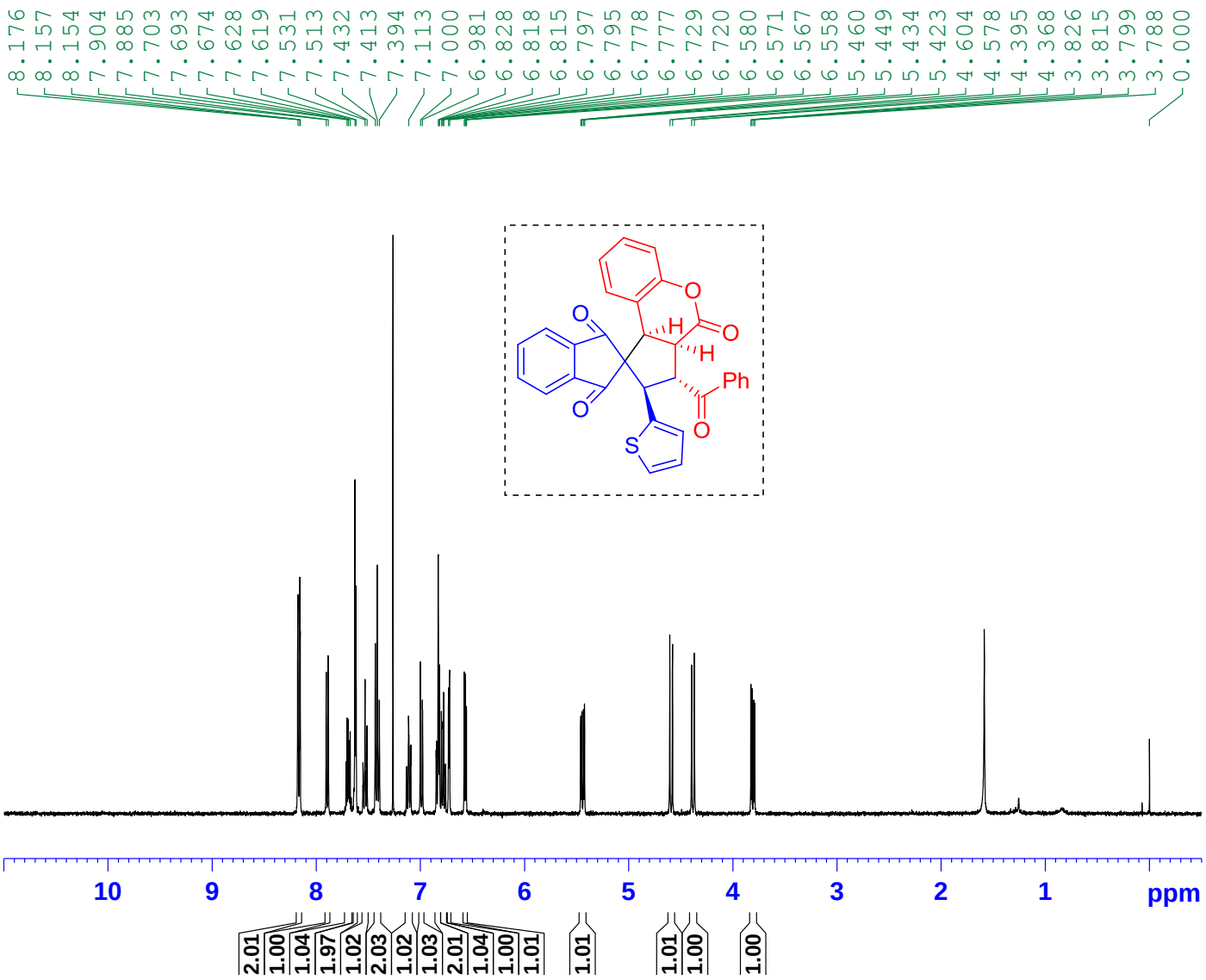

```
Current Data Parameters
NAME                   3al
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_           20170525
Time              21.06
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   32
DS                    0
SWH            7211.539 Hz
FIDRES         0.220079 Hz
AQ            2.2719147 sec
RG                128.9
DW               69.333 usec
DE                10.50 usec
TE                297.6 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                 1H
P1                12.90 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                16384
SF           400.1300090 MHz
WDW                  EM
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```

^{13}C NMR spectrum of **3al** ($CDCl_3$)



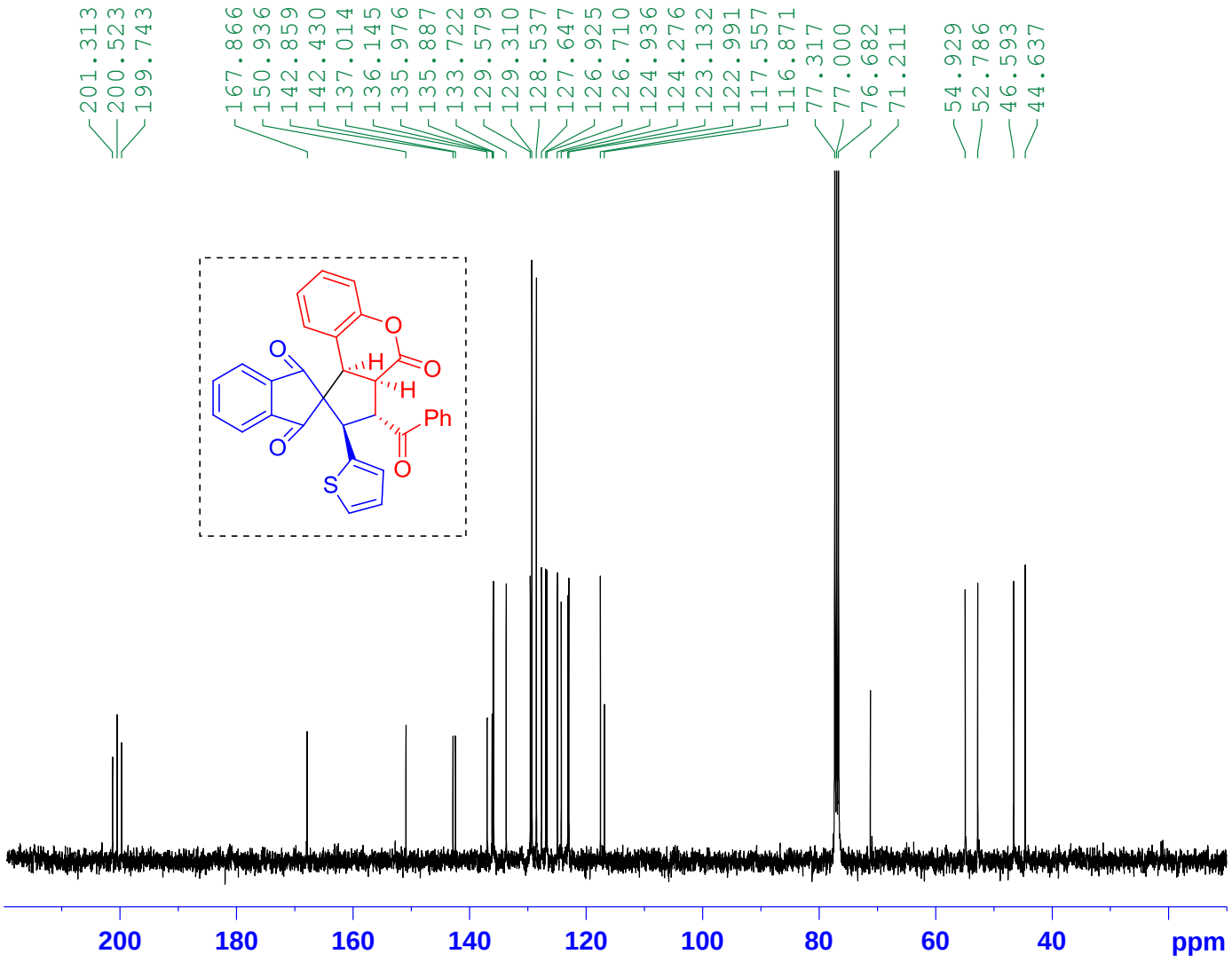

```
Current Data Parameters
NAME                  3al
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160328
Time               14.00
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                  554
DS                    0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        47.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        15.00000000 W
PLW12        0.33750001 W
PLW13        0.27338001 W

F2 - Processing parameters
SI                32768
SF          100.6127739 MHz
WDW                 EM
SSB       0
LB                2.00 Hz
GB        0
PC                1.00
```

^{1}H NMR spectrum of **3am** ($CDCl_3$)



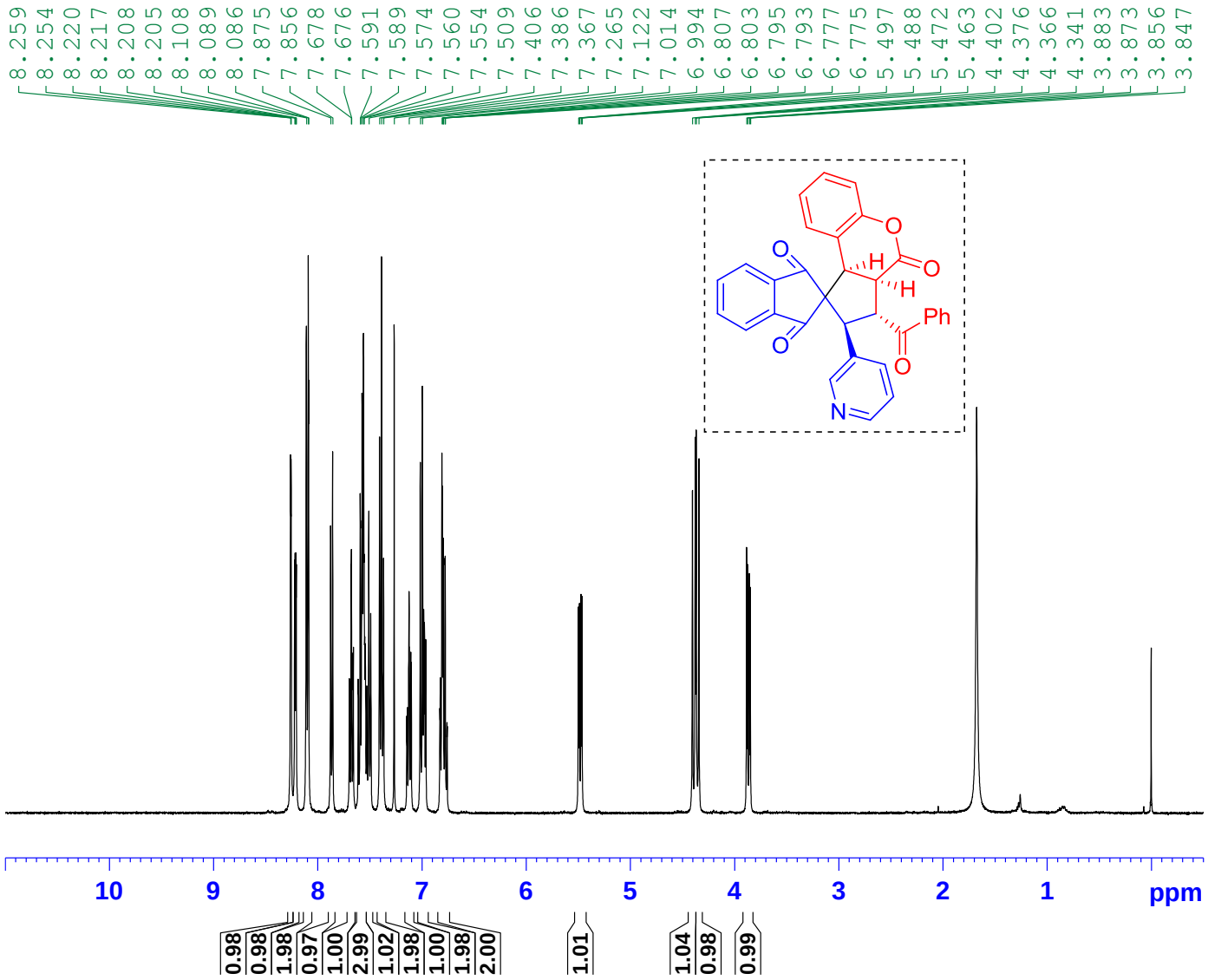

```
Current Data Parameters
NAME                  3am
EXPNO                   6
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160315
Time              13.48
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    1
DS                    0
SWH            7211.539 Hz
FIDRES         0.220079 Hz
AQ             2.2719147 sec
RG                198.09
DW                69.333 usec
DE                10.50 usec
TE                297.1 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                1H
P1                12.90 usec
PLW1        15.00000000 W

F2 - Processing parameters
SI                16384
SF           400.1300088 MHz
WDW                EM
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```

^{13}C NMR spectrum of **3am** ($CDCl_3$)



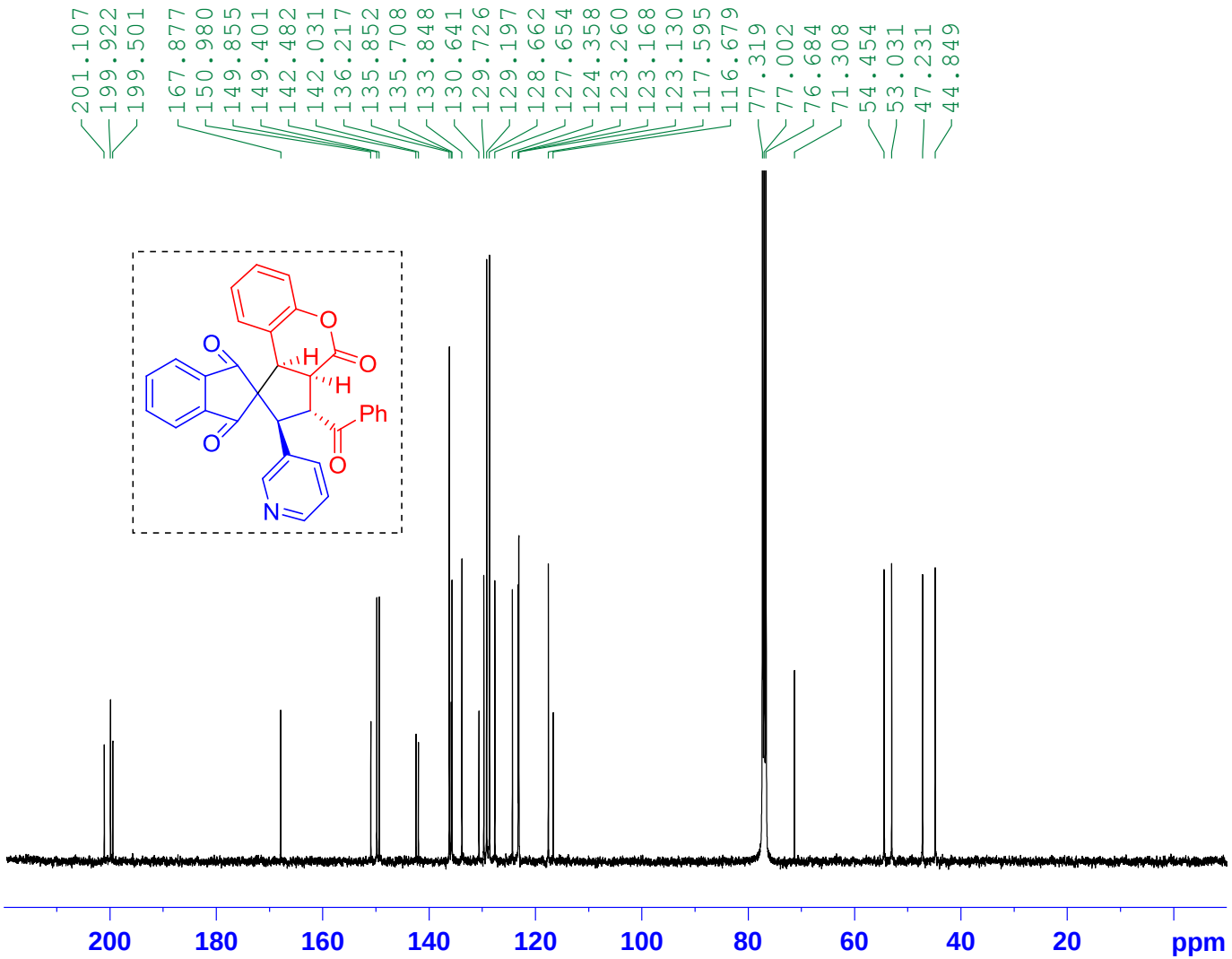

```
Current Data Parameters
NAME                  3am
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160328
Time                13.24
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                  32768
SOLVENT             CDCl3
NS                    555
DS                      0
SWH             24038.461 Hz
FIDRES           0.733596 Hz
AQ               0.6815744 sec
RG                 198.09
DW                 20.800 usec
DE                  6.50 usec
TE                 298.1 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                 10.00 usec
PLW1         47.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         15.00000000 W
PLW12         0.33750001 W
PLW13         0.27338001 W

F2 - Processing parameters
SI                  32768
SF           100.6127718 MHz
WDW                    EM
SSB       0
LB                  2.00 Hz
GB        0
PC                  1.00
```

^{1}H NMR spectrum of **3an** ($CDCl_3$)



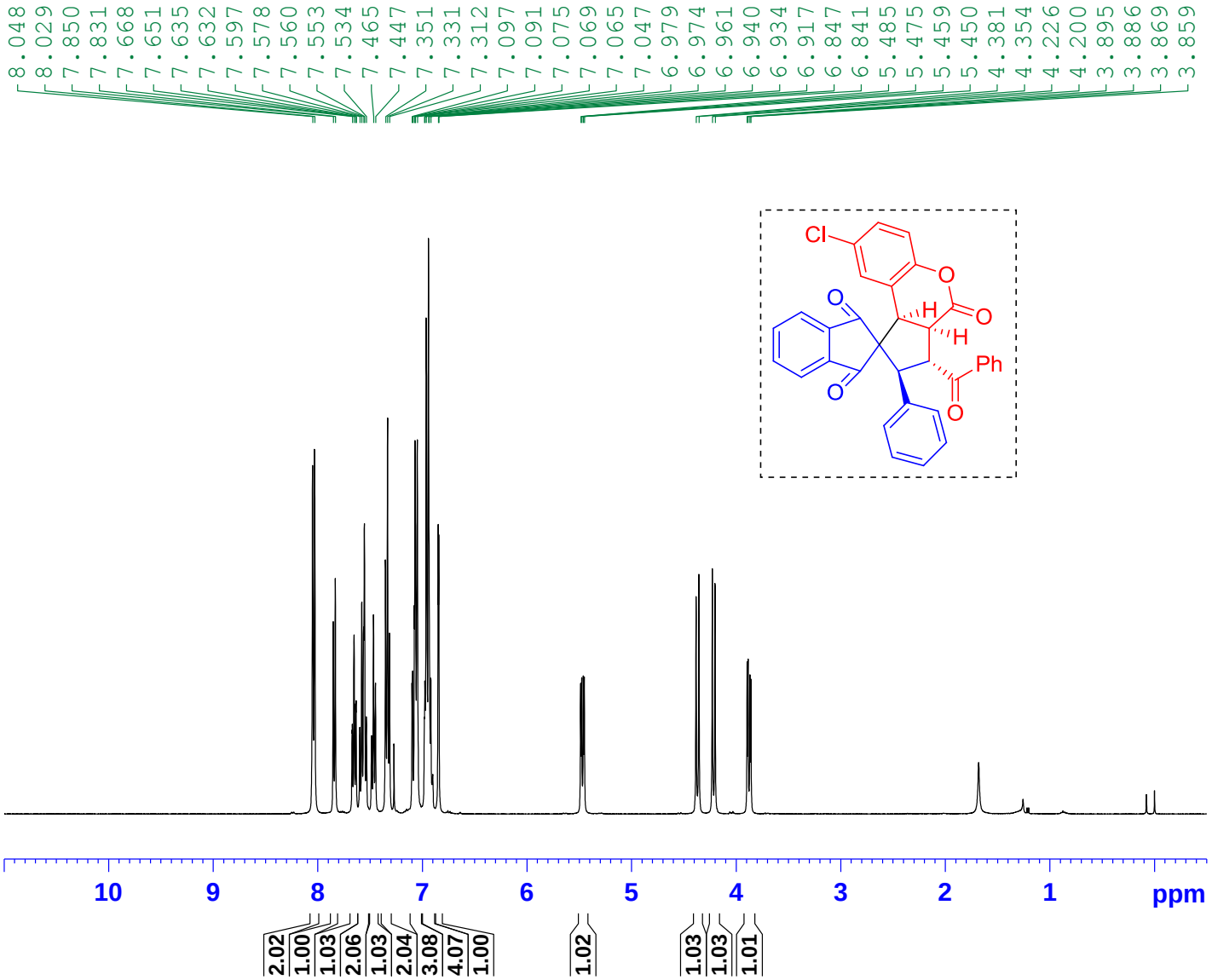

```
Current Data Parameters
NAME                      3an
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20170530
Time                  23.52
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       32
DS                        0
SWH                7211.539 Hz
FIDRES             0.220079 Hz
AQ                2.2719147 sec
RG                   198.09
DW                   69.333 usec
DE                   10.50 usec
TE                   298.6 K
D1              2.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           400.1324008 MHz
NUC1                     1H
P1                   12.90 usec
PLW1           15.00000000 W

F2 - Processing parameters
SI                    16384
SF             400.1300080 MHz
WDW                      EM
SSB       0
LB        0 Hz
GB        0
PC                     1.00
```

^{13}C NMR spectrum of **3an** ($CDCl_3$)



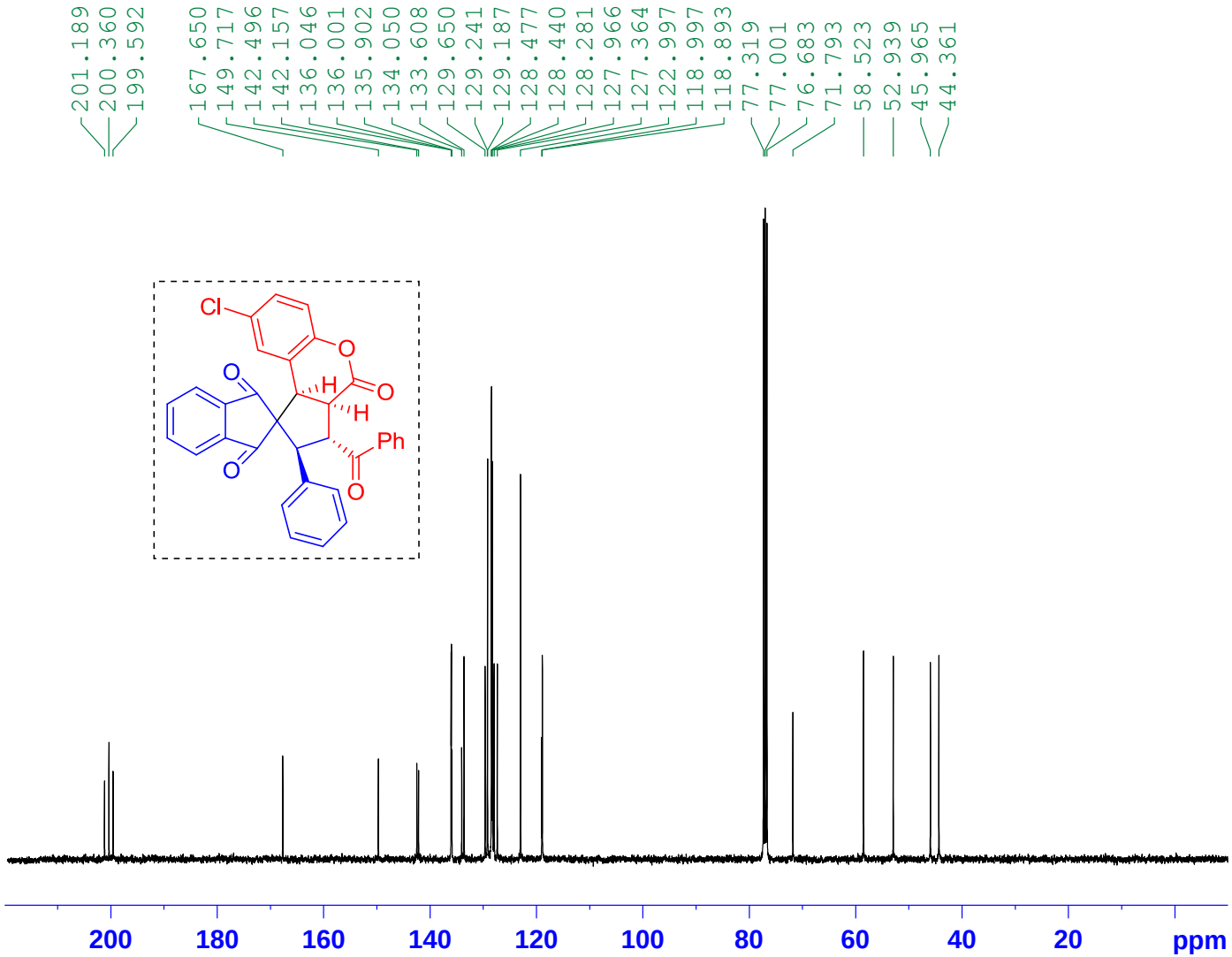

```
Current Data Parameters
NAME                  3an
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20170530
Time              23.55
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                13083
DS                    0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            0.6815744 sec
RG               198.09
DW               20.800 usec
DE                6.50 usec
TE                298.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        47.50000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        15.00000000 W
PLW12        0.33750001 W
PLW13        0.27338001 W

F2 - Processing parameters
SI                32768
SF          100.6127703 MHz
WDW                 EM
SSB       0
LB                2.00 Hz
GB        0
PC                1.00
```

^{1}H NMR spectrum of **3ba** ($CDCl_3$)



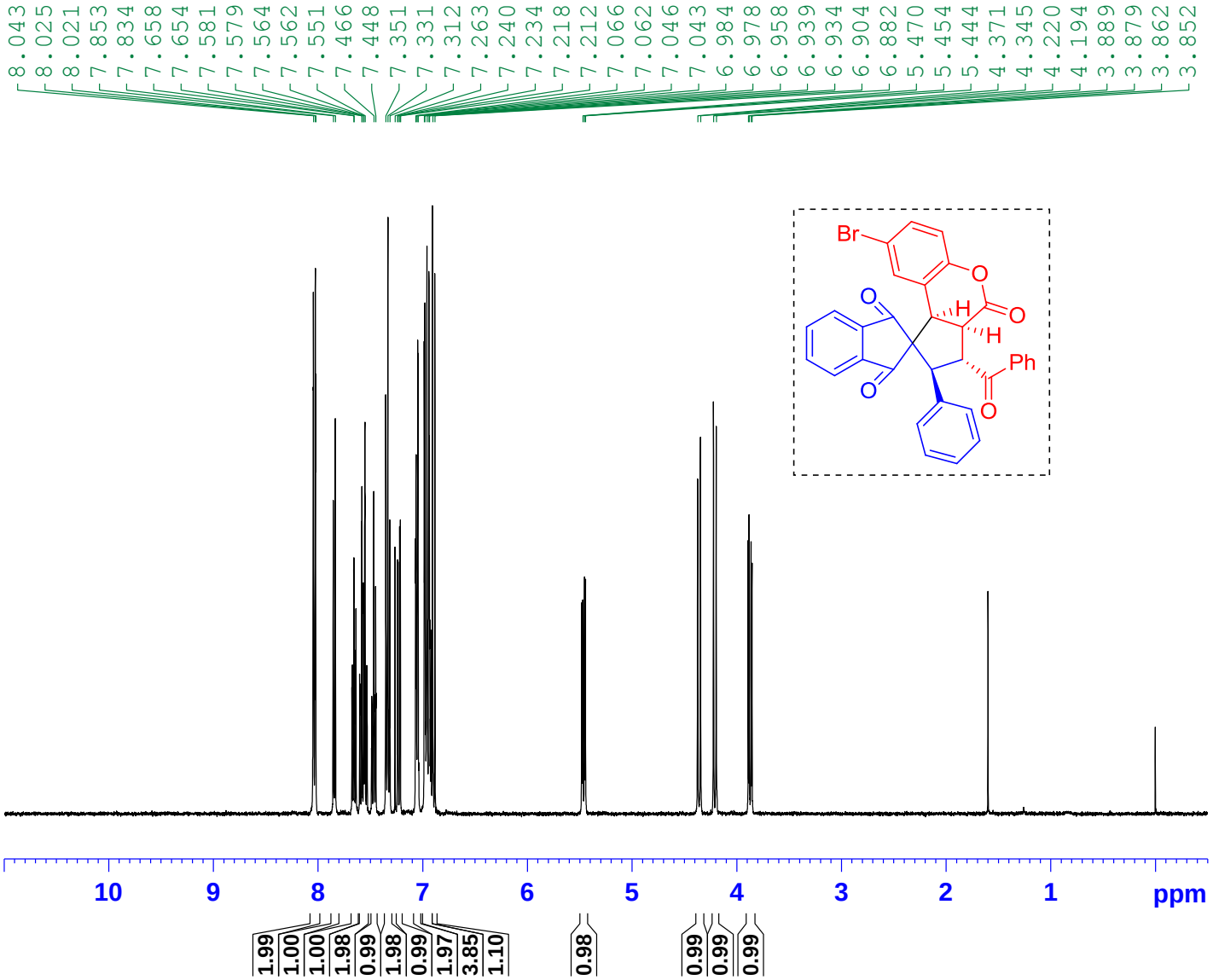

```
Current Data Parameters
NAME                  3ba
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170518
Time                10.32
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     32
DS                      0
SWH              7211.539 Hz
FIDRES           0.220079 Hz
AQ               2.2719147 sec
RG                  71.42
DW                 69.333 usec
DE                 10.50 usec
TE                 298.1 K
D1              2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                    1H
P1                  12.90 usec
PLW1          15.00000000 W

F2 - Processing parameters
SI                  16384
SF            400.1300064 MHz
WDW                    EM
SSB       0
LB        0 Hz
GB        0
PC                   1.00
```

^{13}C NMR spectrum of **3ba** ($CDCl_3$)



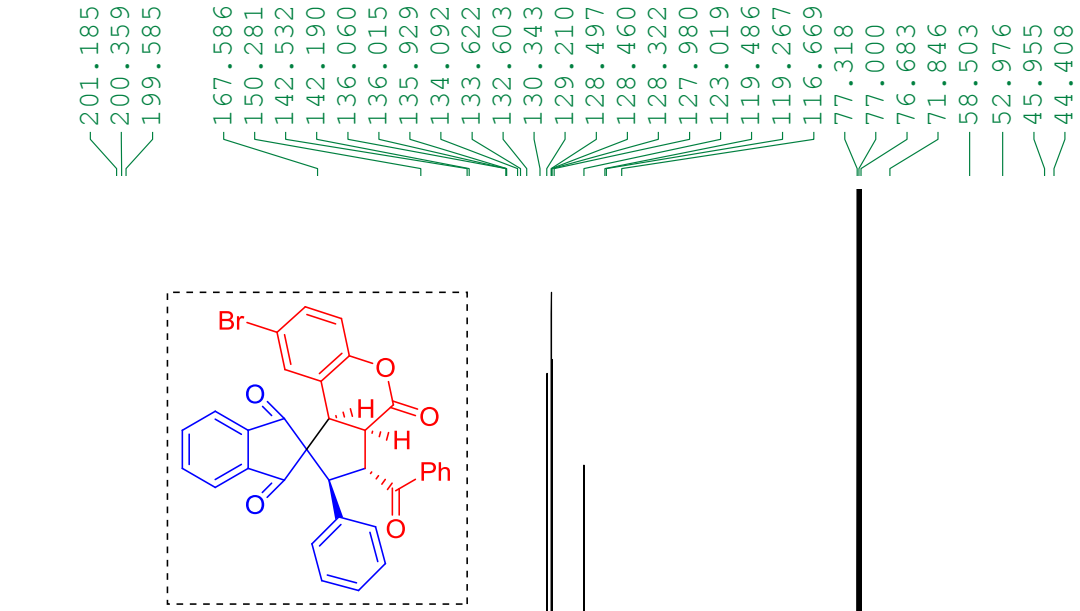

```
Current Data Parameters
NAME                   3ba
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_           20170518
Time               10.39
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   503
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                298.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                 13C
P1                10.00 usec
PLW1         47.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2         15.00000000 W
PLW12         0.33750001 W
PLW13         0.27338001 W

F2 - Processing parameters
SI                 32768
SF           100.6127737 MHz
WDW                  EM
SSB       0
LB                2.00 Hz
GB        0
PC                1.00
```

^{1}H NMR spectrum of **3ca** ($CDCl_3$)



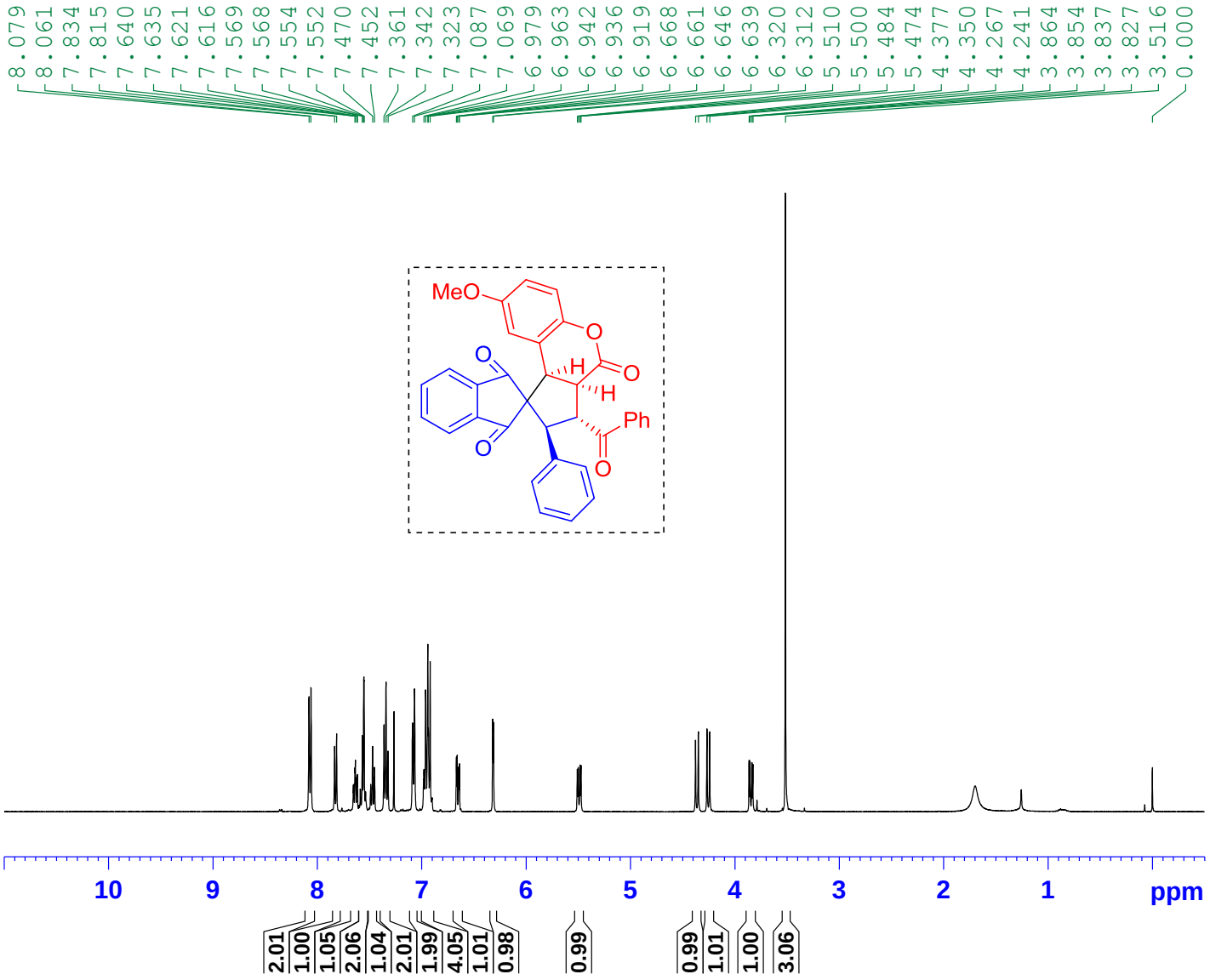

```
Current Data Parameters
NAME                   3ca
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_           20170416
Time                17.53
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                      1
DS                      0
SWH              7211.539 Hz
FIDRES           0.220079 Hz
AQ               2.2719147 sec
RG                 113.31
DW                 69.333 usec
DE                 10.50 usec
TE                 301.1 K
D1              2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                    1H
P1                  12.90 usec
PLW1          15.00000000 W

F2 - Processing parameters
SI                  16384
SF            400.1300089 MHz
WDW                    EM
SSB       0
LB        0 Hz
GB        0
PC                   1.00
```

^{13}C NMR spectrum of **3ca** ($CDCl_3$)



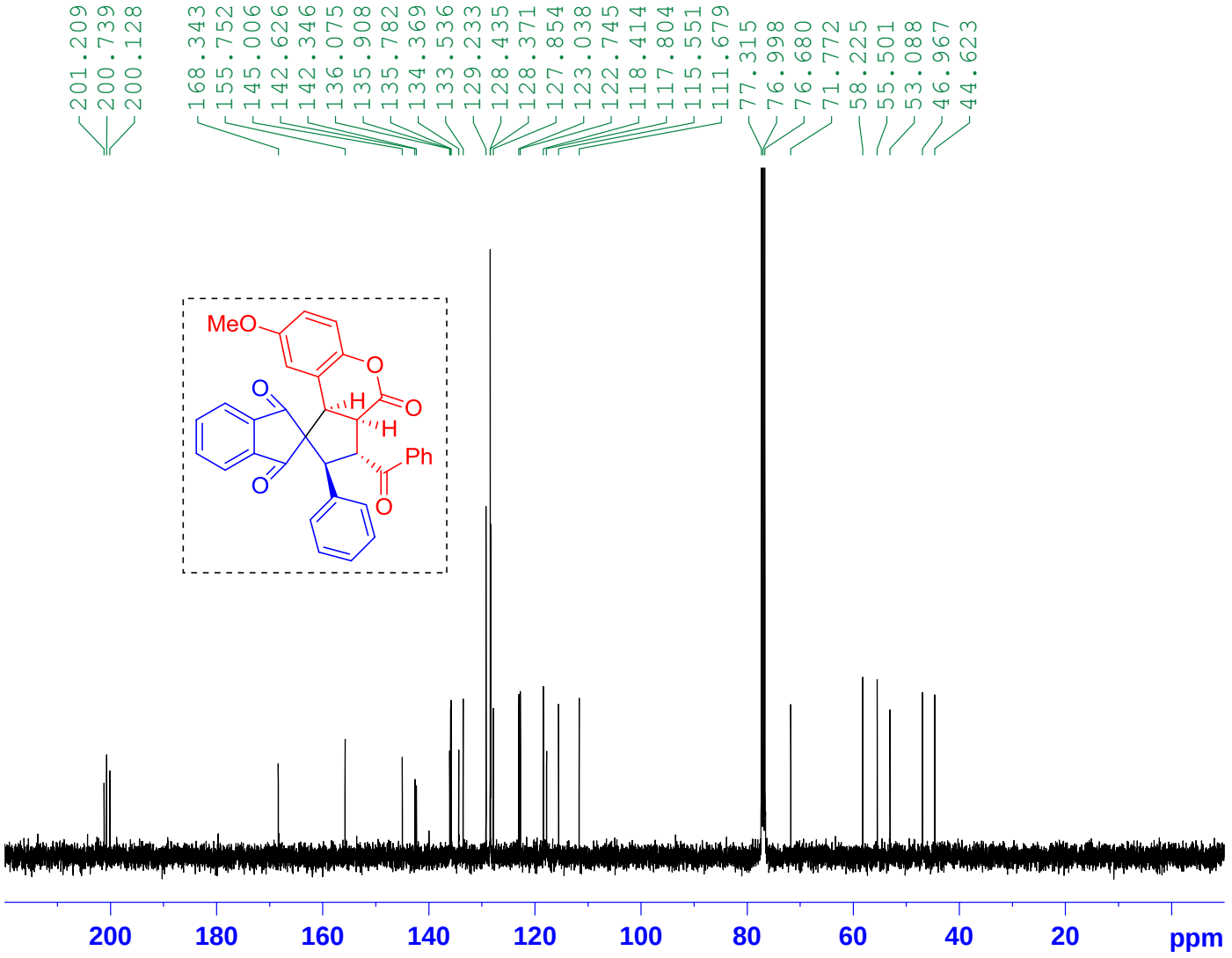

```
Current Data Parameters
NAME                  3ca
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170416
Time                17.55
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                  4155
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                301.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1         47.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         15.00000000 W
PLW12         0.33750001 W
PLW13         0.27338001 W

F2 - Processing parameters
SI                 32768
SF           100.6127714 MHz
WDW                  EM
SSB       0
LB                2.00 Hz
GB        0
PC                1.00
```

^{1}H NMR spectrum of **3da** ($CDCl_3$)



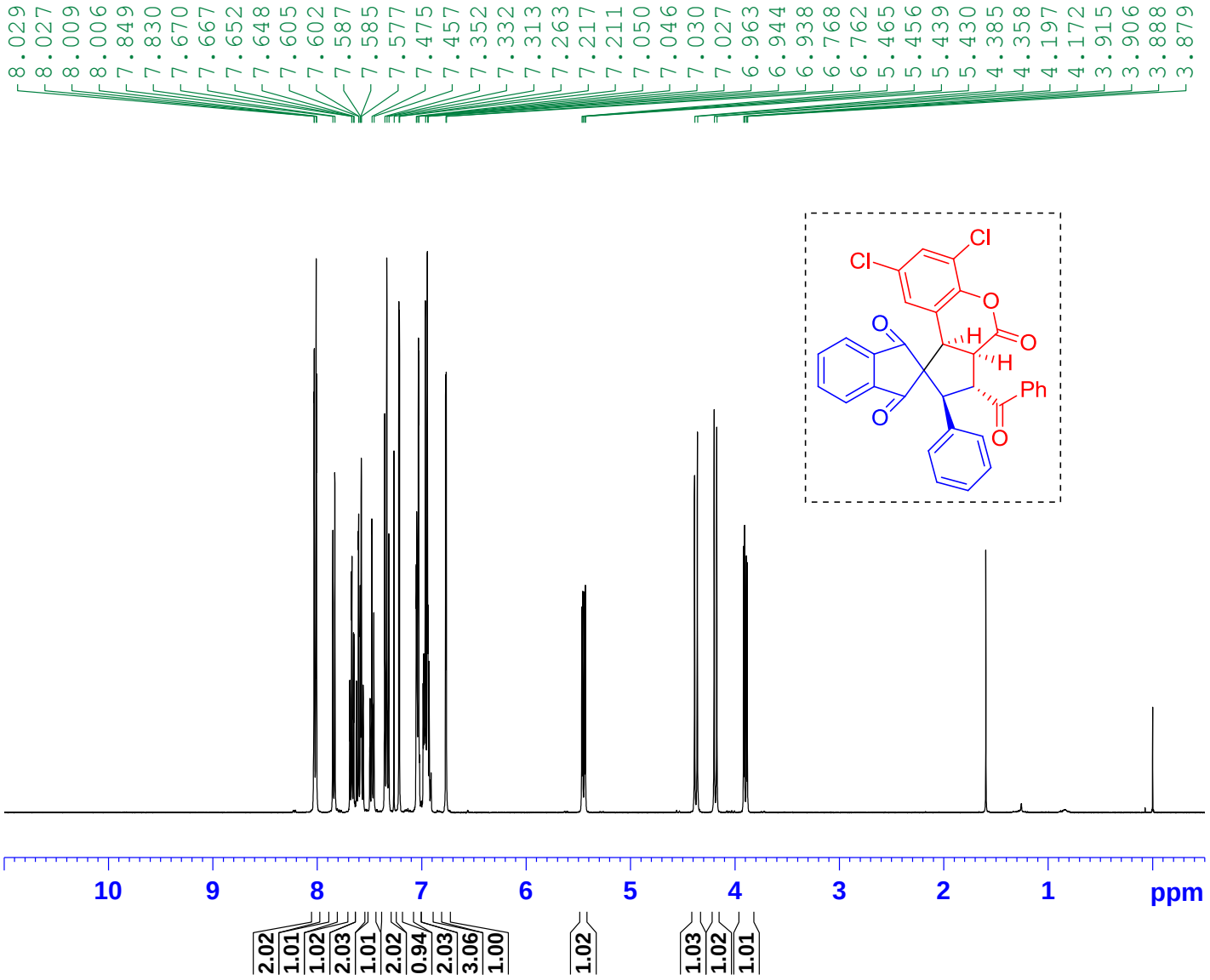

```
Current Data Parameters
NAME                   3da
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160606
Time               11.01
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             7246.377 Hz
FIDRES          0.221142 Hz
AQ             2.2609921 sec
RG                 161.3
DW                69.000 usec
DE                 6.50 usec
TE                297.8 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.40 usec
PL1                1.80 dB
SFO1        400.1324008 MHz

F2 - Processing parameters
SI                 16384
SF          400.1300070 MHz
WDW                   EM
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```

^{13}C NMR spectrum of **3da** ($CDCl_3$)



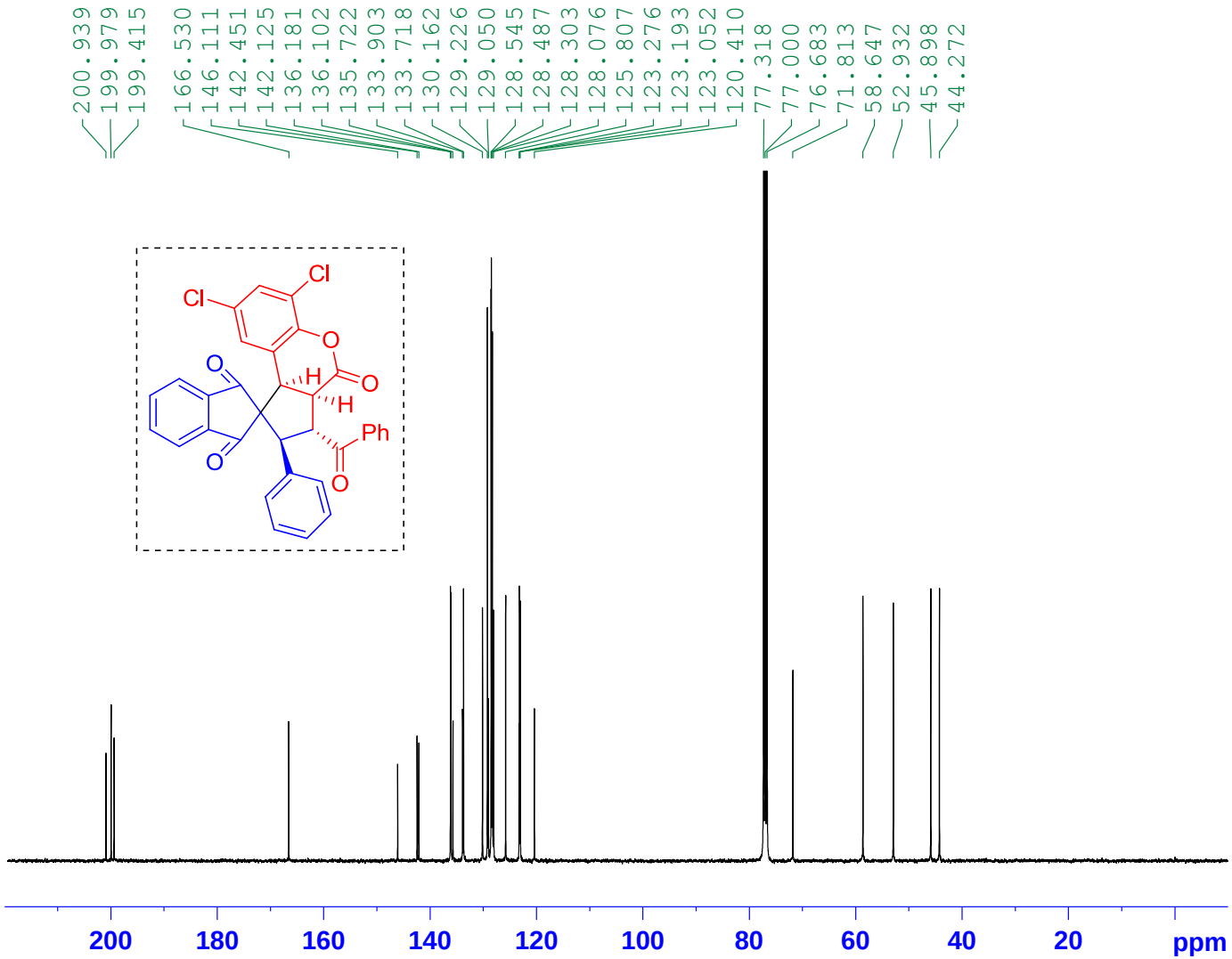

```
Current Data Parameters
NAME                   3da
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160606
Time               11.06
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   492
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ             0.6815744 sec
RG                5792.6
DW                20.800 usec
DE                 6.50 usec
TE                298.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.00 usec
PL1                 7.00 dB
SFO1        100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2              90.00 usec
PL2                 1.80 dB
PL12               17.00 dB
PL13               20.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127716 MHz
WDW                  EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.00
```

^{1}H NMR spectrum of **3ea** ($CDCl_3$)



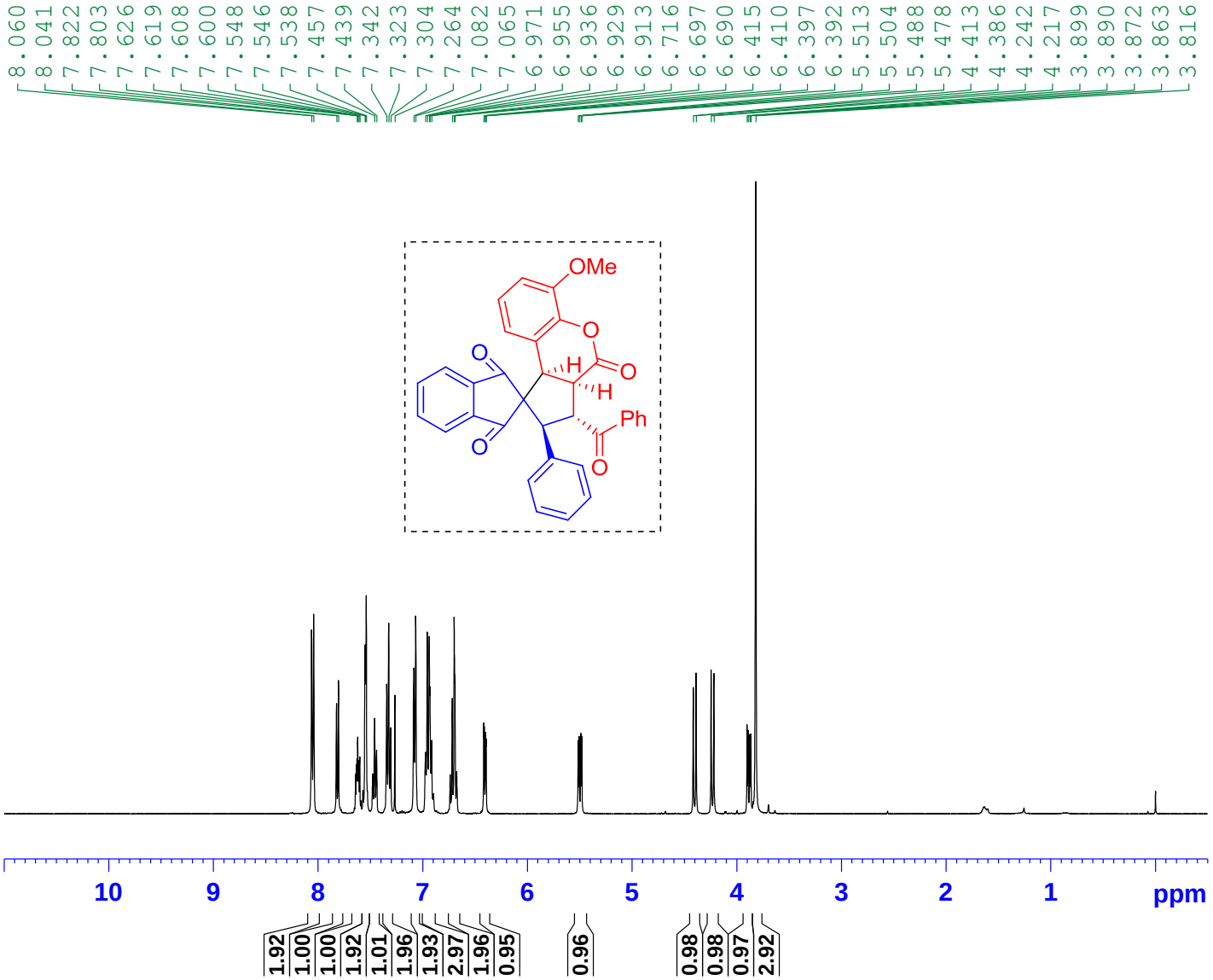

```
Current Data Parameters
NAME                   3ea
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20170422
Time                 11.35
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      32
DS                       0
SWH               7211.539 Hz
FIDRES            0.220079 Hz
AQ               2.2719147 sec
RG                  113.31
DW                  69.333 usec
DE                  10.50 usec
TE                  296.8 K
D1             2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          400.1324008 MHz
NUC1                    1H
P1                   12.90 usec
PLW1          15.00000000 W

F2 - Processing parameters
SI                   16384
SF            400.1300085 MHz
WDW                     EM
SSB       0
LB        0 Hz
GB        0
PC                    1.00
```

^{13}C NMR spectrum of **3ea** ($CDCl_3$)



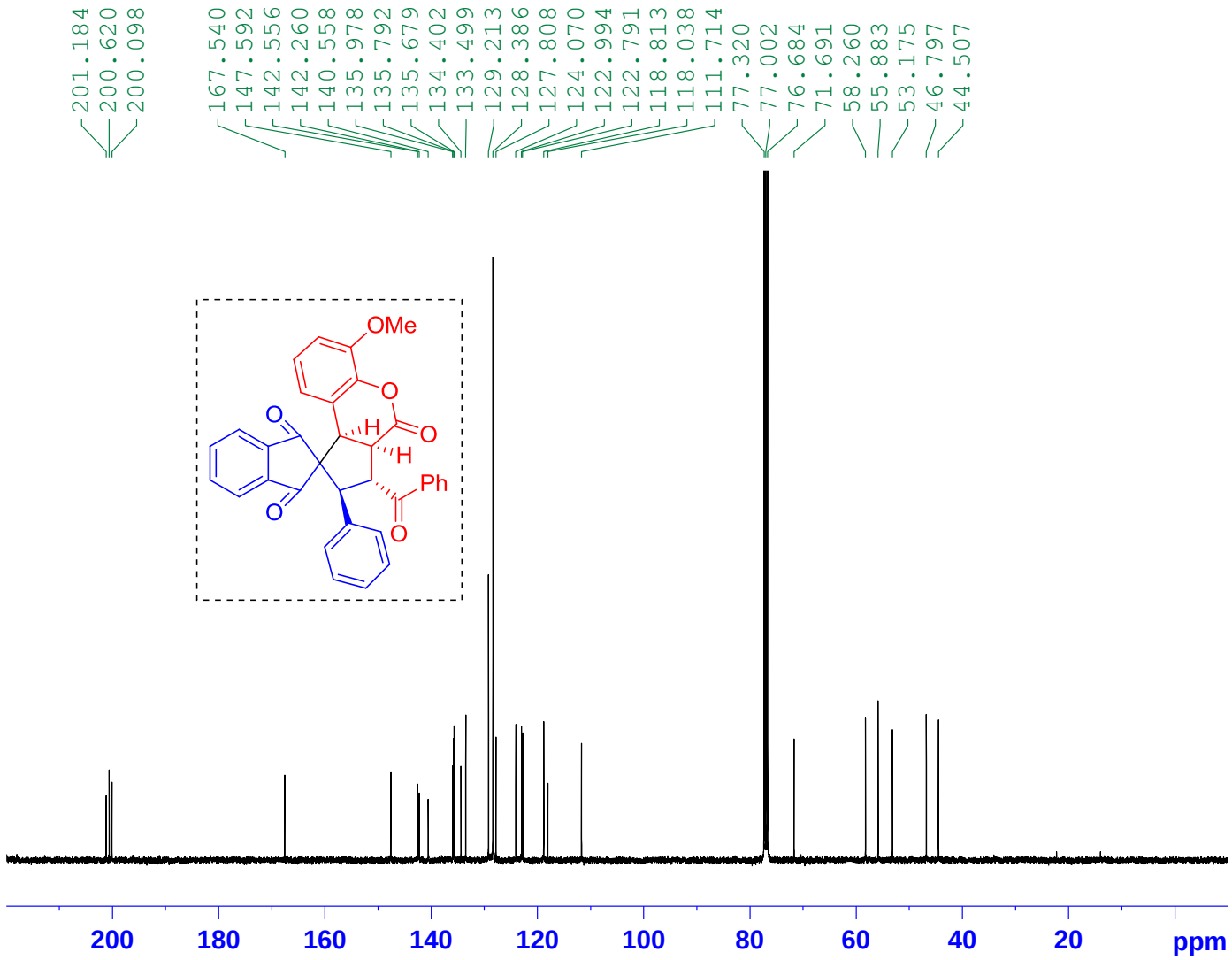

```
Current Data Parameters
NAME                  3ea
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170423
Time                19.12
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                 18981
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                296.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
PLW1         47.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         15.00000000 W
PLW12         0.33750001 W
PLW13         0.27338001 W

F2 - Processing parameters
SI                 32768
SF           100.6127718 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.00
```

^{1}H NMR spectrum of **3fa** ($CDCl_3$)



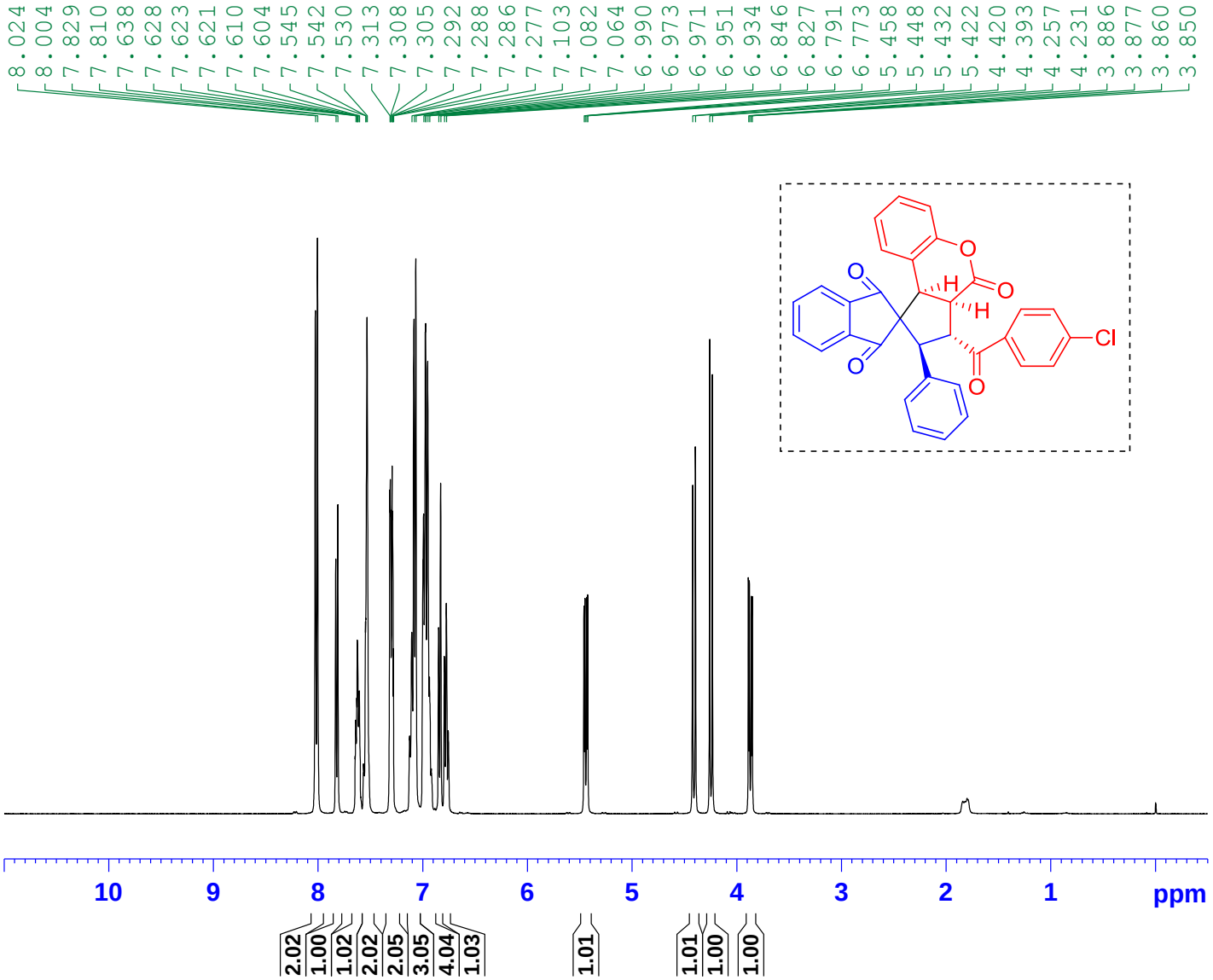

```
Current Data Parameters
NAME                   3fa
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20170502
Time                21.00
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     32
DS                      0
SWH              7211.539 Hz
FIDRES           0.220079 Hz
AQ               2.2719147 sec
RG                 113.31
DW                 69.333 usec
DE                 10.50 usec
TE                 296.7 K
D1            2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         400.1324008 MHz
NUC1                   1H
P1                 12.90 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                  16384
SF           400.1300081 MHz
WDW                    EM
SSB       0
LB        0 Hz
GB        0
PC                   1.00
```

^{13}C NMR spectrum of **3fa** ($CDCl_3$)



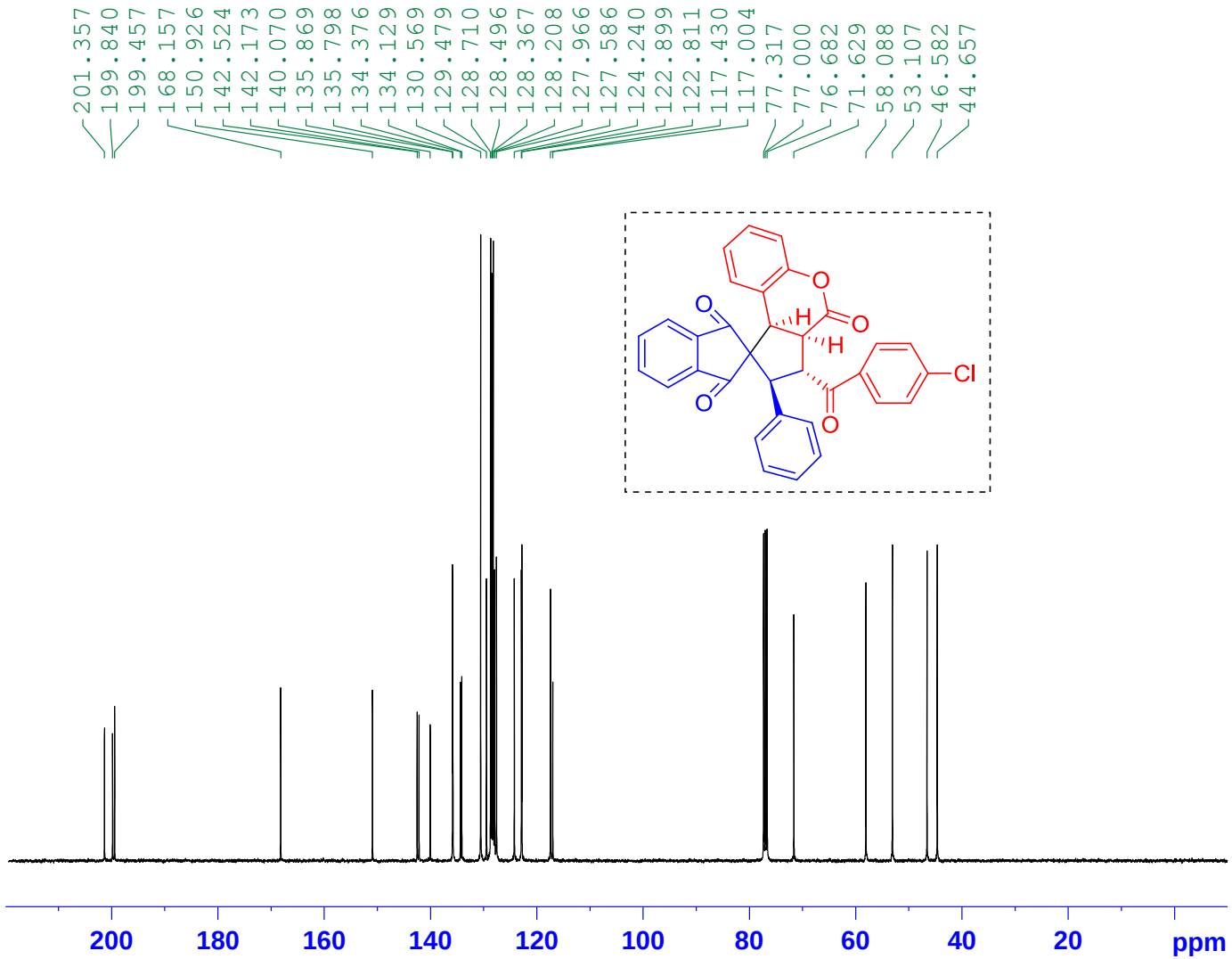

```
Current Data Parameters
NAME                   3fa
EXPNO                    6
PROCNO                   1

F2 - Acquisition Parameters
Date_           20170501
Time               18.07
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                  1322
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                 5792.6
DW                20.800 usec
DE                 6.50 usec
TE                296.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.00 usec
PL1                  7.00 dB
SFO1        100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                 1.80 dB
PL12               17.00 dB
PL13               20.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127740 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.00
```

^{1}H NMR spectrum of **3ga** ($CDCl_3$)



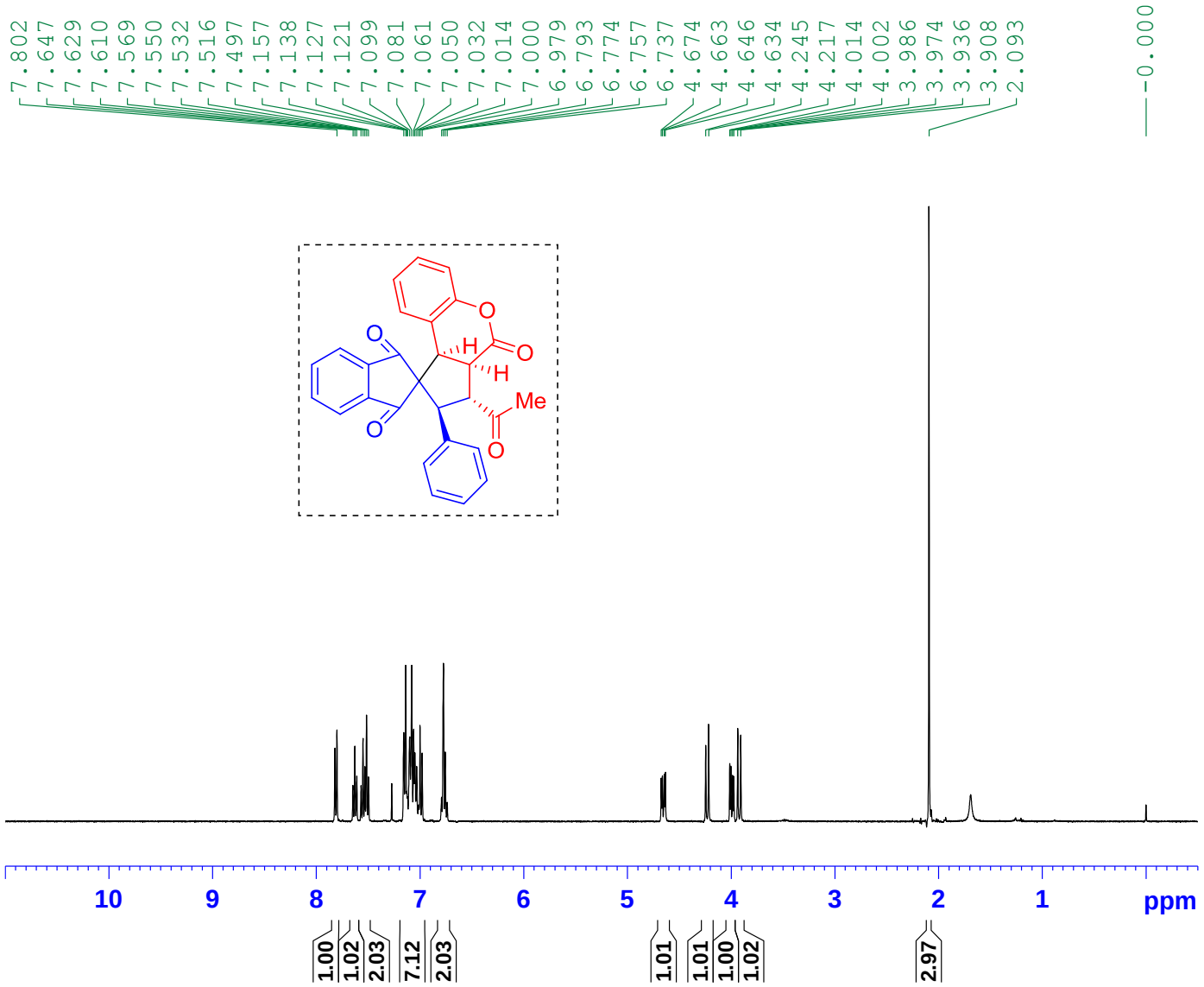

```
Current Data Parameters
NAME                   3ga
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20170530
Time                 22.12
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      32
DS                       0
SWH                7211.539 Hz
FIDRES            0.220079 Hz
AQ                2.2719147 sec
RG                    51.8
DW                  69.333 usec
DE                   10.50 usec
TE                   298.2 K
D1              2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           400.1324008 MHz
NUC1                   1H
P1                  12.90 usec
PLW1           15.00000000 W

F2 - Processing parameters
SI                   16384
SF             400.1300028 MHz
WDW                    EM
SSB       0
LB        0 Hz
GB        0
PC                    1.00
```

^{13}C NMR spectrum of **3ga** ($CDCl_3$)



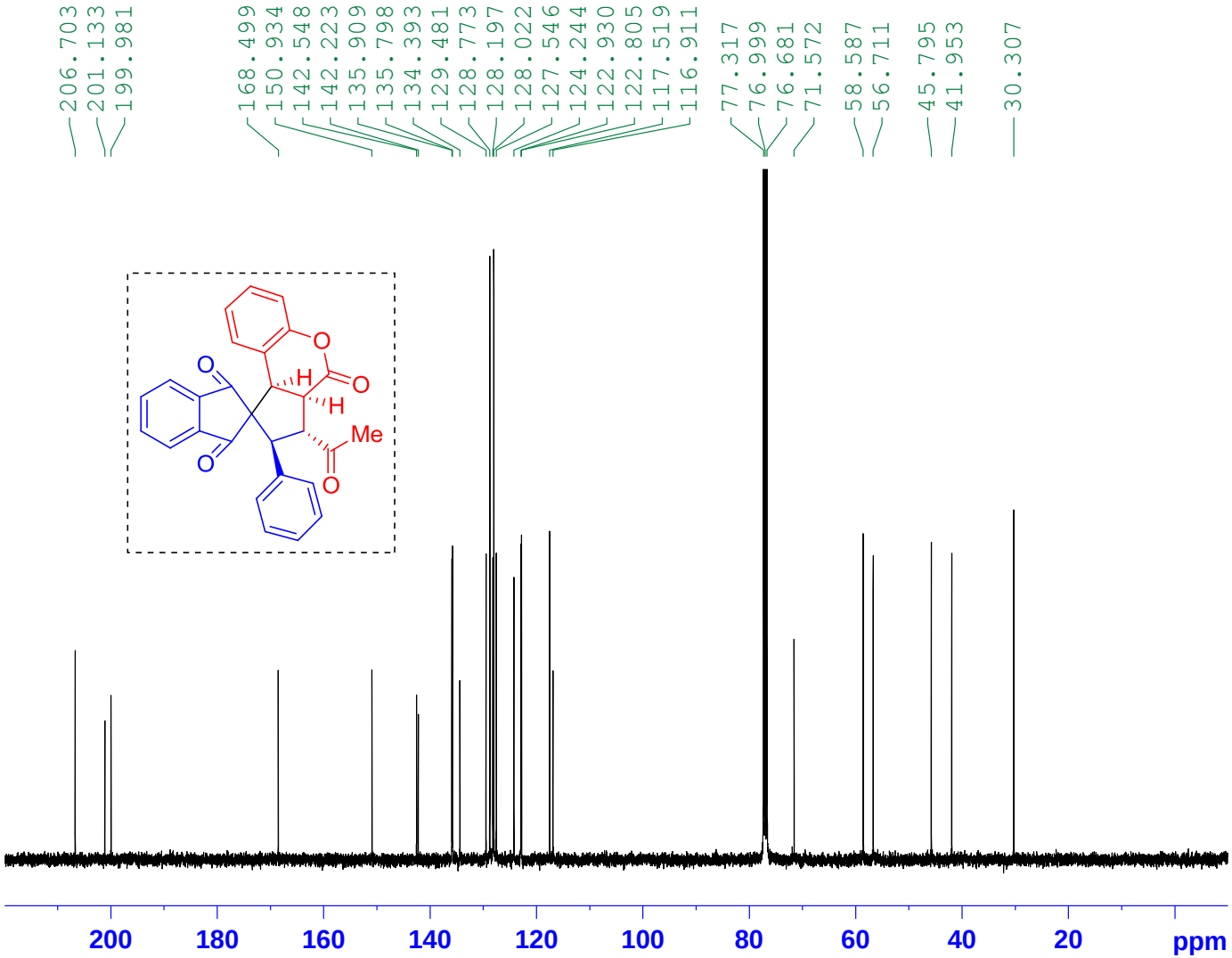

```
Current Data Parameters
NAME                   3ga
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_           20170530
Time                22.15
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 32768
SOLVENT            CDCl3
NS                  2026
DS                     0
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6815744 sec
RG                198.09
DW                20.800 usec
DE                 6.50 usec
TE                298.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1                 13C
P1                10.00 usec
PLW1         47.50000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2         15.00000000 W
PLW12         0.33750001 W
PLW13         0.27338001 W

F2 - Processing parameters
SI                 32768
SF           100.6127779 MHz
WDW                  EM
SSB       0
LB                2.00 Hz
GB        0
PC                1.00
```

^{1}H NMR spectrum of **3ha** ($CDCl_3$)



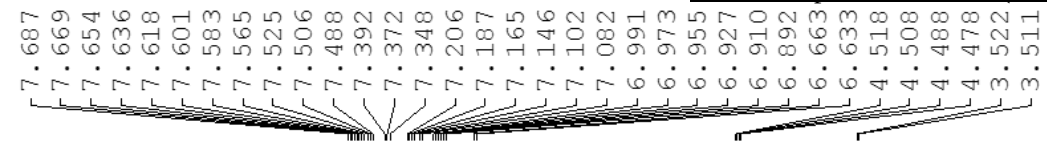

```
Current Data Parameters
NAME                  3ha
EXPNO                  33
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170427
Time                18.35
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                      1
DS                      0
SWH              7246.377 Hz
FIDRES           0.221142 Hz
AQ               2.2609921 sec
RG                    114
DW                 69.000 usec
DE                  6.50 usec
TE                 298.6 K
D1            2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.40 usec
PL1                 1.80 dB
SFO1         400.1324008 MHz

F2 - Processing parameters
SI                  16384
SF           400.1300042 MHz
WDW                    EM
SSB       0
LB        0 Hz
GB        0
PC                  1.00
```

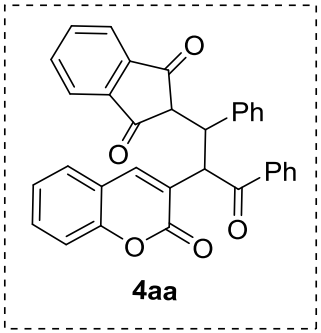
^{13}C NMR spectrum of **3ha** ($CDCl_3$)




```
Current Data Parameters
NAME                  3ha
EXPNO                  32
PROCNO                  1

F2 - Acquisition Parameters
Date_           20170426
Time               18.48
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                32768
SOLVENT           CDCl3
NS                 1327
DS                    0
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             0.6815744 sec
RG                 4096
DW                20.800 usec
DE                 6.50 usec
TE                298.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.00 usec
PL1                7.00 dB
SFO1        100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                1H
PCPD2             90.00 usec
PL2                1.80 dB
PL12              17.00 dB
PL13              20.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127730 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.00
```

^{1}H NMR spectrum of **4aa** ($CDCl_3$)

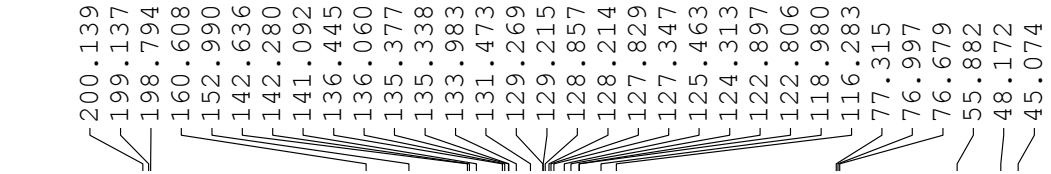


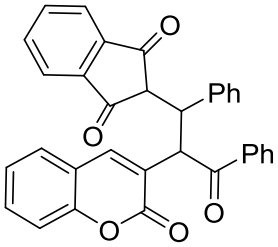


4aa

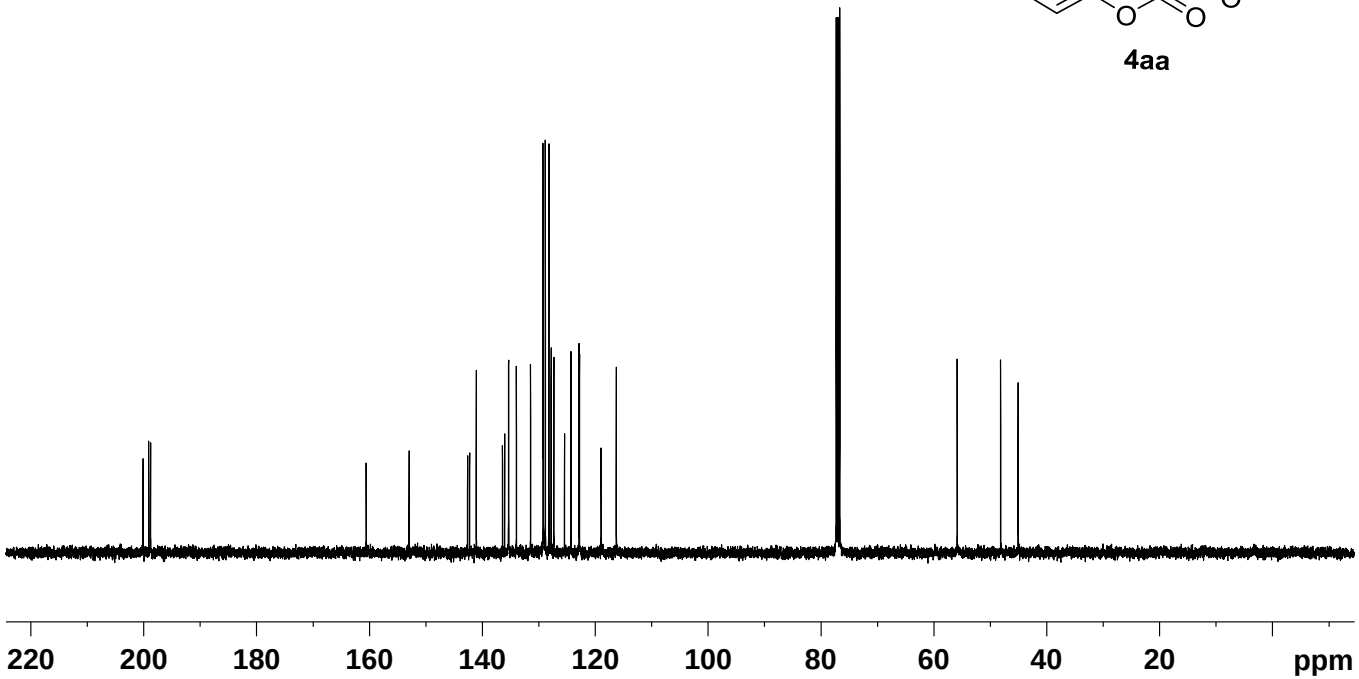

```
Current Data Parameters
NAME          KCinter Ph
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170614
Time               19.17
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     0
SWH             7246.377 Hz
FIDRES          0.221142 Hz
AQ              2.2609921 sec
RG                   114
DW                69.000 usec
DE                 6.50 usec
TE                297.7 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 16.80 usec
PL1                 1.80 dB
SFO1         400.1324008 MHz

F2 - Processing parameters
SI                 16384
SF           400.1300069 MHz
WDW                   EM
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```

^{13}C NMR spectrum of **4aa** ($CDCl_3$)





4aa




```
Current Data Parameters
NAME          KCinter Ph
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170614
Time               19.22
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                 32768
SOLVENT            CDCl3
NS                   653
DS                     0
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6815744 sec
RG               5792.6
DW                20.800 usec
DE                 6.50 usec
TE                298.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.45 usec
PL1                7.00 dB
SFO1        100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2                1.80 dB
PL12               15.68 dB
PL13               20.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127738 MHz
WDW                  EM
SSB      0
LB                1.00 Hz
GB       0
PC                1.00
```

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