Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Bifunctional N-heterocyclic carbene catalyzed [3+4] annulation of enals with azadienes: Stereoselective synthesis of benzofuroazepinones

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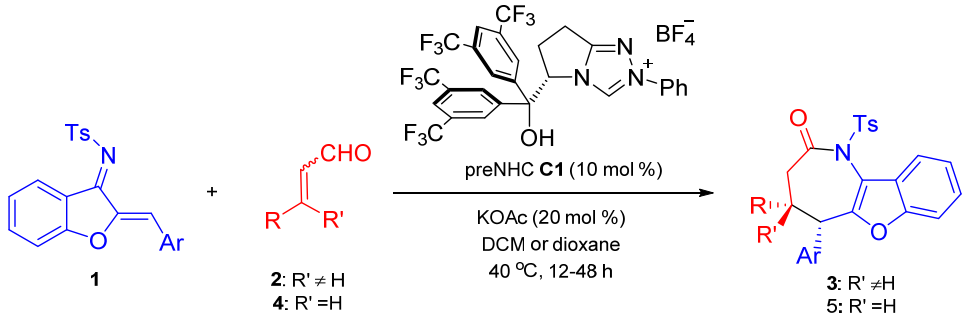
Part I Experimental part

1. General information.

Unless otherwise indicated, all reactions were carried out under N_2 atmosphere with magnetic stirring. Anhydrous THF, ether, 1,4-dioxane and toluene were distilled from sodium and benzophenone. Anhydrous CH_2Cl_2 and acetonitrile were distilled from CaH_2. Chiral triazolium salts **A-C**,[1] enals **2** and 4,[2] and aurone-derived imines[3] were prepared according to literatures. Column chromatography was performed on silica gel 200~300 mesh. All ^{1}H NMR (300, 400 and 500 MHz), ^{13}C NMR (75, 100 and 125 MHz) and ^{19}F NMR (376 MHz) spectra were recorded on a Bruker Avance 300, Avance 400 and Avance 500 spectrometer in $CDCl_3$, with tetramethylsilane as an internal standard and reported in parts per million (ppm, δ). ^{1}H NMR spectroscopy splitting patterns were designated as singlet (s), doublet (d), triplet (t), quartet (q). Splitting patterns that could not be interpreted or easily visualized were designated as multiplet (m) or broad (br). Infrared spectra were recorded on a JASCO FT/IR-480 spectrophotometer and reported as wave number (cm^{-1}). Optical rotations were measured on Perkin Elmer/Model-343 digital polarimeter operating at the sodium D line with a 100 mm path cell and were reported as follows: $[\alpha]_D^T$ (concentration (g/100 mL), solvent).

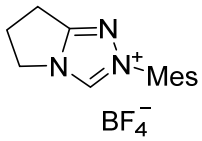
2. NHC-catalyzed [3 + 4] annulation of enals with aurone-derived imines.



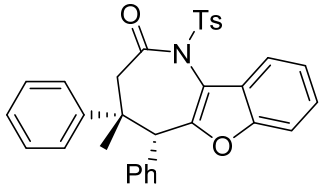
General Procedure An oven-dried 25 mL Schlenk tube was charged with

aurone-derived azadienes **1** (0.1 mmol), enals **2** or **4** (0.2 mmol, 2.0 equiv), NHC precursor **C1** KOAc (1.96 mg, 0.02 mmol) and freshly distilled dichloromethane (for enals **2**) or 1,4-dioxane (for enals 4) (2 mL) was added. The reaction mixture was stirred at 40 °C until the full consumption of the aurone-derived azadienes (typically 12-48 h). The reaction mixture was concentrated under reduced pressure and the residue was purified by column chromatography on silica gel (petroleum ether/EtOAc as the eluent, typically 15:1 to 6:1) to furnish the desired cycloadducts **3**/**5**.

Racemic samples for the chiral phase HPLC analysis were prepared using triazolium D as the pre-NHC under the same conditions.



D

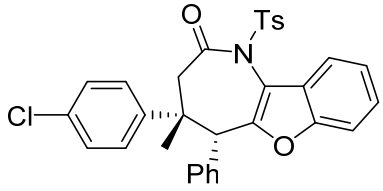


3a

(4*R*,5*R*)-4-methyl-4,5-diphenyl-1-tosyl-1,3,4,5-tetrahydro-2*H*-benzofuro[3,2-*b*]azepin-2-one

50.7 mg, >20:1 dr, 97% yield. White solid, m.p. 240-242 °C. R_f = 0.3 (petroleum ether/ethyl acetate 5:1). $[\alpha]_D^{23}$ -57.3 (*c* 1.18, $CHCl_3$). HPLC analysis: 99% ee [Daicel CHIRALPAK IC column, 20 °C, 254 nm, hexane/*i*-PrOH = 70:30, 1.0 mL/min, 26.3 min (minor), 34.7 min (major)]. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98-7.95 (m, 3H), 7.49 (d, *J* = 8.1 Hz, 1H), 7.43-7.38 (m, 4H), 7.22-7.18 (m, 2H), 7.13 (t, *J* = 7.7 Hz, 2H), 7.06 (t, *J* = 7.7 Hz, 2H), 6.55 (d, *J* = 7.7 Hz, 2H), 6.40 (d, *J* = 7.5 Hz, 2H), 3.45 (s, 1H), 3.29 (d, *J* = 12.3 Hz, 1H), 2.56 (s, 3H), 2.34 (d, *J* = 12.3 Hz, 1H), 1.31 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.6, 153.7, 153.2, 145.6, 141.1, 135.3, 132.4, 131.2, 129.6, 129.1, 128.0, 127.8, 127.6, 127.4, 126.8, 125.04, 124.96, 124.1, 121.2, 117.2,

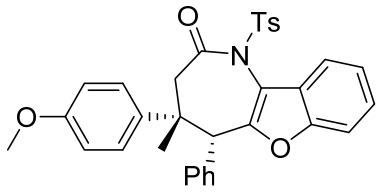
111.9, 56.1, 52.7, 49.3, 25.3, 21.8. IR (KBr) 2920, 1714, 1634, 1162, 753. HRMS (ESI) calcd for $C_{32}H_{27}NO_4SNa$ $[M+Na]^+$ 544.1553, found 544.1552.



3b

(4*R*,5*R*)-4-(4-chlorophenyl)-4-methyl-5-phenyl-1-tosyl-1,3,4,5-tetrahydro-2*H*-benzofuro[3,2-*b*]azepin-2-one

46.3 mg, >20:1 dr, 83% yield. White solid, m.p. 243-244 ^oC. R_f = 0.3 (petroleum ether/ethyl acetate 5:1). $[\alpha]_D^{23}$ -72.4 (*c* 0.36, $CHCl_3$). HPLC analysis: 99% ee [Daicel CHIRALPAK IC column, 20 ^oC, 254 nm, hexane/*i*-PrOH = 70:30, 1.0 mL/min, 33.7 min (minor), 39.1 min (major)].^1H NMR (500 MHz, $CDCl_3$) δ 7.96-7.95 (m, 3H), 7.50 (d, J = 8.1 Hz, 1H), 7.45-7.38 (m, 4H), 7.22 (t, J = 7.4 Hz, 1H), 7.12-7.10 (m, 4H), 6.48-6.43 (m, 4H), 3.43 (s, 1H), 3.21 (d, J = 12.3 Hz, 1H), 2.56 (s, 3H), 2.32 (d, J = 12.3 Hz, 1H), 1.29 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.4, 153.8, 153.0, 145.7, 139.9, 135.3, 133.6, 132.2, 131.3, 129.8, 129.2, 128.4, 128.2, 128.1, 127.7, 125.3, 125.0, 124.4, 121.4, 117.5, 112.1, 55.9, 52.7, 49.3, 25.4, 22.0. IR (KBr) 2920, 1714, 1647, 1162, 750. HRMS (ESI) calcd for $C_{32}H_{26}NO_4ClSNa$ $[M+Na]^+$ 578.1163, found 578.1169.

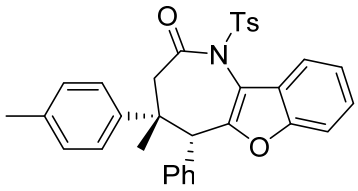


3c

(4*R*,5*R*)-4-(4-methoxyphenyl)-4-methyl-5-phenyl-1-tosyl-1,3,4,5-tetrahydro-2*H*-benzofuro[3,2-*b*]azepin-2-one

51.4 mg, >20:1 dr, 96% yield. White solid, m.p. 108-110 ^oC. R_f = 0.2 (petroleum ether/ethyl acetate 5:1). $[\alpha]_D^{23}$ -88.0 (*c* 0.10, $CHCl_3$). HPLC analysis: 99% ee [Daicel CHIRALPAK IC column, 20 ^oC, 254 nm, hexane/*i*-PrOH = 60:40, 1.0 mL/min, 26.9

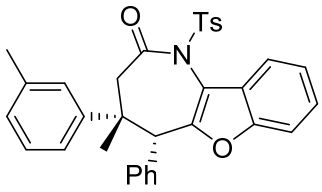
min (minor), 44.2 min (major)]. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.96-7.95 (m, 3H), 7.49 (d, J = 8.1 Hz, 1H), 7.44-7.36 (m, 4H), 7.20 (t, J = 7.4 Hz, 1H), 7.09 (t, J = 7.6 Hz, 2H), 6.66 (d, J = 8.8 Hz, 2H), 6.46-6.41 (m, 4H), 3.76 (s, 3H), 3.41 (s, 1H), 3.23 (d, J = 12.3 Hz, 1H), 2.55 (s, 3H), 2.31 (d, J = 12.3 Hz, 1H), 1.28 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.6, 158.8, 153.6, 153.3, 145.5, 135.3, 133.2, 132.5, 131.3, 129.6, 129.1, 128.0, 127.7, 127.4, 125.0, 124.9, 124.1, 121.2, 117.1, 113.2, 111.9, 55.7, 55.3, 52.8, 49.4, 25.5, 21.8. IR (KBr) 2918, 1714, 1648, 1162, 753. HRMS (ESI) calcd for $C_{33}H_{29}NO_5SNa$ $[M+Na]^+$ 574.1659, found 574.1664.



3d

(4*R*,5*R*)-4-methyl-5-phenyl-4-(*p*-tolyl)-1-tosyl-1,3,4,5-tetrahydro-2*H*-benzofuro[3,2-*b*]azepin-2-one

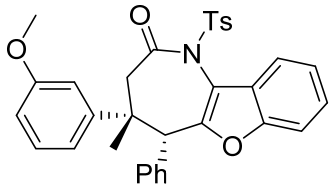
38.6 mg, >20:1 dr, 72% yield. White solid, m.p. 226-227 ^{o}C. R_f = 0.3 (petroleum ether/ethyl acetate 5:1). $[\alpha]_D^{23}$ -79.3 (*c* 0.30, $CHCl_3$). HPLC analysis: 99% ee [Daicel CHIRALPAK IC column, 20 ^{o}C, 254 nm, hexane/*i*-PrOH = 70:30, 1.0 mL/min, 24.0 min (minor), 36.9 min (major)]. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98-7.95 (m, 3H), 7.49 (d, J = 8.2 Hz, 1H), 7.45-7.36 (m, 4H), 7.20 (t, J = 7.5 Hz, 1H), 7.08 (t, J = 7.5 Hz, 2H), 6.94 (d, J = 7.9 Hz, 2H), 6.42 (t, J = 7.9 Hz, 4H), 3.43 (s, 1H), 3.26 (d, J = 12.3 Hz, 1H), 2.56 (s, 3H), 2.32-2.29 (m, 4H), 1.29 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.6, 153.6, 153.3, 145.5, 138.1, 137.2, 135.3, 132.5, 131.3, 129.6, 129.1, 128.7, 127.7, 127.3, 126.7, 125.1, 124.9, 124.1, 121.2, 117.1, 111.9, 55.9, 52.7, 49.4, 25.3, 21.8, 20.9. IR (KBr) 2921, 1713, 1610, 1162, 753. HRMS (ESI) calcd for $C_{33}H_{29}NO_4SNa$ $[M+Na]^+$ 558.1710, found 558.1711.



3e

(4*R*,5*R*)-4-methyl-5-phenyl-4-(*m*-tolyl)-1-tosyl-1,3,4,5-tetrahydro-2*H*-benzofuro[3,2-*b*]azepin-2-one

42.1 mg, >20:1 dr, 79% yield. White solid, m.p. 205-207 ^{o}C. R_f = 0.3 (petroleum ether/ethyl acetate 5:1). $[\alpha]_D^{23}$ -33.0 (*c* 0.74, $CHCl_3$). HPLC analysis: 99% ee [Daicel CHIRALPAK IC column, 20 ^{o}C, 254 nm, hexane/*i*-PrOH = 70:30, 1.0 mL/min, 20.0 min (minor), 33.5 min (major)]. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99-7.96 (m, 3H), 7.49 (d, *J* = 8.2 Hz, 1H), 7.45-7.37 (m, 4H), 7.20 (t, *J* = 7.4 Hz, 1H), 7.08-7.01 (m, 4H), 6.37 (t, *J* = 8.6 Hz, 3H), 6.28 (s, 1H), 3.43 (s, 1H), 3.27 (d, *J* = 12.3 Hz, 1H), 2.56 (s, 3H), 2.33 (d, *J* = 12.3 Hz, 1H), 2.13 (s, 3H), 1.29 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.7, 153.7, 153.4, 145.5, 141.0, 137.5, 135.3, 132.4, 131.2, 129.6, 129.1, 128.2, 127.82, 127.77, 127.3, 125.1, 125.0, 124.1, 123.9, 121.2, 117.1, 111.8, 56.2, 52.8, 49.3, 25.2, 21.8, 21.5. IR (KBr) 2919, 1714, 1606, 1181, 743. HRMS (ESI) calcd for $C_{33}H_{29}NO_4SNa$ $[M+Na]^+$ 558.1710, found 558.1708.

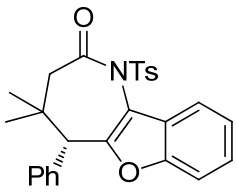


3f

(4*R*,5*R*)-4-(3-methoxyphenyl)-4-methyl-5-phenyl-1-tosyl-1,3,4,5-tetrahydro-2*H*-benzofuro[3,2-*b*]azepin-2-one

44.8 mg, >20:1 dr, 84% yield. White solid, m.p. 232-233 ^{o}C. R_f = 0.2 (petroleum ether/ethyl acetate 5:1). $[\alpha]_D^{23}$ -86.7 (*c* 0.29, $CHCl_3$). HPLC analysis: 99% ee [Daicel CHIRALPAK IC column, 20 ^{o}C, 254 nm, hexane/*i*-PrOH = 60:40, 1.0 mL/min, 18.8 min (minor), 33.4 min (major)]. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98-7.95 (m, 3H), 7.49 (d, *J* = 8.2 Hz, 1H), 7.44-7.37 (m, 4H), 7.21 (t, *J* = 7.4 Hz, 1H), 7.10-7.03 (m, 3H), 6.77 (dd, *J* = 8.2, 2.4 Hz, 1H), 6.45 (d, *J* = 7.4 Hz, 2H), 6.17 (d, *J* = 7.8 Hz, 1H),

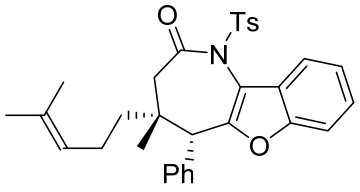
6.08 (s, 1H), 3.53 (s, 3H), 3.44 (s, 1H), 3.25 (d, J = 12.3 Hz, 1H), 2.56 (s, 3H), 2.34 (d, J = 12.3 Hz, 1H), 1.29 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.5, 159.2, 153.6, 153.2, 145.6, 142.8, 135.3, 132.3, 131.2, 129.6, 129.1, 128.9, 127.8, 127.4, 125.01, 124.99, 124.1, 121.2, 119.3, 117.2, 113.1, 112.9, 111.8, 56.1, 55.1, 52.7, 49.4, 25.3, 21.8. IR (KBr) 2917, 1714, 1654, 1181, 743. HRMS (ESI) calcd for $C_{33}H_{29}NO_5SNa$ $[M+Na]^+$ 574.1659, found 574.1663.



3g

(*R*)-4,4-dimethyl-5-phenyl-1-tosyl-4,5-dihydro-1*H*-benzofuro[3,2-*b*]azepin-2(3*H*)-one

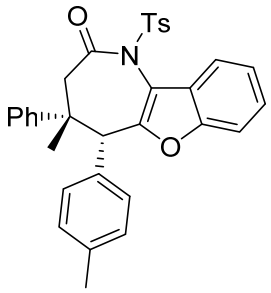
Total yield: 29 mg, (0.1 mmol scale) 57%. Analytical data for **3d**: yellow oil; $[\alpha]_D^{25}$ -20.2 (*c* 1.0, $CHCl_3$); 92% ee as determined by HPLC (IB, 90:10 hexanes/*i*-PrOH, 1.0 ml/min), $t_{r\,min}$ = 8.9 min, $t_{r\,maj}$ = 9.4 min; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 (d, J = 8.1 Hz, 2H), 7.87 (d, J = 7.4 Hz, 1H), 7.52 (d, J = 7.9 Hz, 1H), 7.37 (d, J = 6.6 Hz, 7H), 7.24 – 7.23 (m, 2H), 3.21 (s, 1H), 2.54 (s, 3H), 2.31 (d, J = 12.1 Hz, 1H), 2.13 (d, J = 12.0 Hz, 1H), 0.84 (s, 5H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.1, 154.0, 154.0, 145.5, 135.4, 133.2, 131.5, 129.7, 129.2, 128.1, 128.0, 124.9, 124.9, 124.1, 121.2, 117.0, 111.8, 51.2, 50.3, 49.8, 27.4, 25.4, 21.9. IR (KBr) v 3446, 2927, 1712, 1386, 1087. cm^{-1} HRMS (ESI) calcd for $C_{27}H_{25}NNaO_4S$ $[M+Na]^+$ 482.1402, found 482.1407.



3h

(4*R*,5*S*)-4-methyl-4-(4-methylpent-3-en-1-yl)-5-phenyl-1-tosyl-1,3,4,5-tetrahydro-2*H*-benzofuro[3,2-*b*]azepin-2-one

32.0 mg, >20:1 dr, 61% yield. White solid, m.p. 58-60 ^{o}C. R_f = 0.4 (petroleum ether/ethyl acetate 5:1). $[\alpha]_D^{23}$ -21.1 (*c* 0.83, $CHCl_3$). HPLC analysis: 99% ee [Daicel CHIRALPAK IC column, 20 ^{o}C, 254 nm, hexane/*i*-PrOH = 70:30, 1.0 mL/min, 21.1 min (minor), 31.5 min (major)].^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85-7.81 (m, 3H), 7.45 (d, *J* = 7.4 Hz, 1H), 7.32-7.27 (m, 7H), 7.16 (s, 1H), 7.15-7.14 (m, 2H), 4.78 (t, *J* = 7.1 Hz, 1H), 3.15 (s, 1H), 2.46 (s, 3H), 2.20 (d, *J* = 11.9 Hz, 1H), 2.14 (d, *J* = 11.9 Hz, 1H), 1.59-1.55 (m, 1H), 1.52 (s, 3H), 1.35 (s, 3H), 1.24-1.19 (m, 1H), 1.16-1.12 (m, 1H), 0.78 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 172.7, 153.9, 153.7, 145.4, 135.2, 132.7, 132.0, 131.6, 129.5, 129.0, 127.9, 127.8, 124.7, 124.0, 123.6, 121.1, 117.0, 111.8, 52.5, 52.2, 47.6, 37.3, 25.6, 23.8, 22.0, 21.8, 17.7. IR (KBr) 2924, 1713, 1384, 1172, 747. HRMS (ESI) calcd for $C_{32}H_{33}NO_4SNa$ $[M+Na]^+$ 550.2023, found 550.2024.

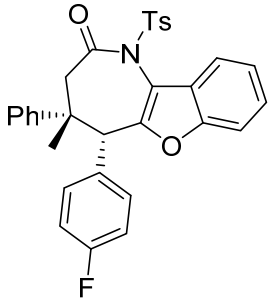


3i

(4*R*,5*R*)-4-methyl-5-phenyl-4-(*p*-tolyl)-1-tosyl-4,5-dihydro-1*H*-benzofuro[3,2-*b*]azepin-2(3*H*)-one

34.0 mg, >20:1 dr, 64% yield. White solid, m.p. 235-237 ^{o}C. R_f = 0.3 (petroleum ether/ethyl acetate 5:1). $[\alpha]_D^{25}$ -75.5 (*c* 1.0, $CHCl_3$). HPLC analysis: 97% ee [Daicel CHIRALPAK IC, 20 ^{o}C, 210 nm, hexanes/*i*-PrOH = 70:30, 1.0 mL/min, 28.4 min (minor), 42.2 min (major)]. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.90-7.88 (m, 3H), 7.41 (d, *J* = 8.1 Hz, 1H), 7.37-7.28 (m, 4H), 7.17-7.13 (m, 1H), 7.06 (t, *J* = 7.6 Hz, 2H), 6.80 (d, *J* = 7.8 Hz, 2H), 6.48 (d, *J* = 7.7 Hz, 2H), 6.18 (d, *J* = 8.0 Hz, 2H), 3.32 (s, 1H), 3.20 (d, *J* = 12.3 Hz, 1H), 2.48 (s, 3H), 2.24 (d, *J* = 12.3 Hz, 1H), 2.18 (s, 3H), 1.23 (s, 3H).^{13}C NMR (125 MHz, $CDCl_3$) δ 171.8, 153.7, 153.6, 145.6, 141.4, 137.6, 135.4, 131.2, 129.7, 129.3, 129.2, 128.2, 128.1, 127.6, 127.0, 125.2, 125.0, 124.2, 121.3, 117.2, 112.0,

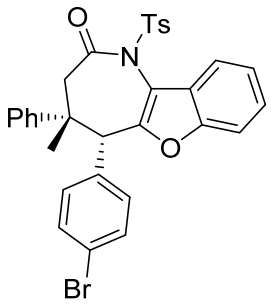
56.2, 52.5, 49.4, 25.4, 21.9, 21.2. IR (KBr) 3335, 2921, 1712, 1384, 1170. HRMS (ESI) calcd for $C_{33}H_{29}NSO_4Na$ $[M+Na]^+$ 558.1710, found 558.1713.



3j

(4*R*,5*R*)-4-(4-fluorophenyl)-4-methyl-5-phenyl-1-tosyl-4,5-dihydro-1*H*-benzofuro [3,2-*b*]azepin-2(3*H*)-one

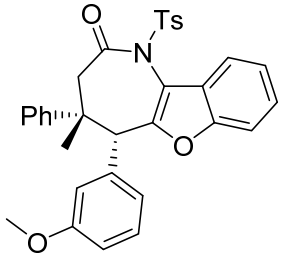
40.0 mg, >20:1 dr, 74% yield. White solid, m.p. 224-226 ^oC. R_f = 0.3 (petroleum ether/ethyl acetate 5:1). $[\alpha]_D^{25}$ -34.3 (*c* 1.0, $CHCl_3$). HPLC analysis: 99% ee [Daicel CHIRALPAK IC column, 20 ^oC, 254 nm, hexane/*i*-PrOH = 70:30, 1.0 mL/min, 27.7 min (major), 35.6 min (minor)]. ^1H NMR (400 MHz, $CDCl_3$) δ 7.90-7.88 (m, 3H), 7.44-7.31 (m, 4H), 7.19-7.14 (m, 2H), 7.07 (t, J = 7.6 Hz, 2H), 6.69 (t, J = 8.6 Hz, 2H), 6.47 (d, J = 7.7 Hz, 2H), 6.31-6.28 (m, 2H), 3.40 (s, 1H), 3.21 (d, J = 12.3 Hz, 1H), 2.48 (s, 3H), 2.27 (d, J = 12.3 Hz, 1H), 1.24 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.6, 162. 4 (J_{CF} = 247.1 Hz), 153.7, 153.0, 145.7, 141.0, 135.5, 132.8 (J_{CF} = 8.0 Hz), 129.7, 129.6, 129.3, 128.2, 127.8, 126.9, 125.2, 125.1, 124.3, 121.3, 117.3, 114.4 (J_{CF} = 121.1 Hz), 112.0, 56.1, 52.0, 49.2, 25.3, 22.0. ^{19}F NMR (376 MHz, $CDCl_3$) -114.3 (Ar-F). IR (KBr) 3407, 2924, 1712, 1384, 1170. HRMS (ESI) calcd for $C_{32}H_{26}NFSO_4Na$ $[M+Na]^+$ 562.1459, found 562.1462.



3k

(4*R*,5*R*)-4-(4-bromophenyl)-4-methyl-5-phenyl-1-tosyl-4,5-dihydro-1*H*-benzofuro [3,2-*b*]azepin-2(3*H*)-one

30.0 mg, >20:1 dr, 60% yield. White solid, m.p. 260-263 ^{o}C. R_f = 0.3 (petroleum ether/ethyl acetate 5:1). $[\alpha]_D^{25}$ -79.4 (*c* 1.15, $CHCl_3$). HPLC analysis: 99% ee [Daicel CHIRALPAK IC column, 20 ^{o}C, 210 nm, hexane/*i*-PrOH = 70:30, 1.0 mL/min, 26.5 min (minor), 38.6 min (major)]. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91-7.88 (m, 3H), 7.43-7.32 (m, 5H), 7.17-7.06 (m, 5H), 6.47 (d, *J* = 7.7 Hz, 2H), 6.17 (d, *J* = 8.6 Hz, 2H), 3.34 (s, 1H), 3.22 (d, *J* = 12.3 Hz, 1H), 2.48 (s, 3H), 2.27 (d, *J* = 12.3 Hz, 1H), 1.24 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.5, 153.8, 152.6, 145.7, 140.8, 135.4, 132.8, 131.5, 130.7, 129.7, 129.2, 128.3, 127.9, 126.9, 125.3, 125.1, 124.4, 122.3, 121.4, 117.4, 112.0, 56.1, 52.3, 49.3, 25.3, 22.0. IR (KBr) 3357, 2922, 1712, 1384, 1170; HRMS (ESI) calcd for $C_{32}H_{26}NNaBrO_4S$ $[M+Na]^+$ 622.0658, found 622.0668.

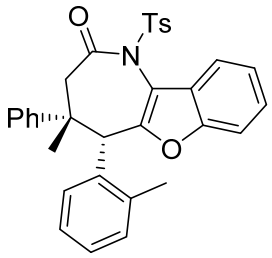


3l

(4*R*,5*R*)-4-(3-methoxyphenyl)-4-methyl-5-phenyl-1-tosyl-4,5-dihydro-1*H*-benzofuro[3,2-*b*]azepin-2(3*H*)-one

38.0 mg, >20:1 dr, 69% yield. White solid, m.p. 90-92 ^{o}C. R_f = 0.3 (petroleum ether/ethyl acetate 5:1). $[\alpha]_D^{25}$ -41.5 (*c* 1.0, $CHCl_3$). HPLC analysis: 99% ee [Daicel CHIRALPAK IC column, 20 ^{o}C, 254 nm, hexanes/*i*-PrOH = 70:30, 1.0 mL/min, 32.2 min (minor), 41.7 min (major)]. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.91-7.89 (m, 3H),

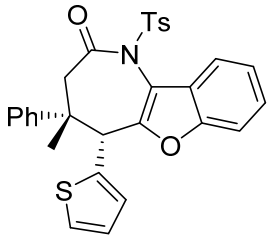
7.42-7.30 (m, 5H), 7.19-7.14 (m, 1H), 7.08 (t, J = 7.6 Hz, 2H), 6.93 (t, J = 7.9 Hz, 1H), 6.68-6.66 (m, 1H), 6.52 (d, J = 7.7 Hz, 2H), 5.93 (d, J = 7.6 Hz, 1H), 5.86 (s, 1H), 3.45 (s, 3H), 3.35 (s, 1H), 3.21 (d, J = 12.3 Hz, 1H), 2.48 (s, 3H), 2.27 (d, J = 12.3 Hz, 1H), 1.25 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.7, 158.6, 153.8, 153.2, 145.8, 141.5, 135.4, 133.8, 129.8, 129.2, 128.3, 128.2, 127.6, 127.1, 125.2, 125.1, 124.3, 123.8, 121.3, 117.4, 116.9, 113.8, 112.0, 56.2, 55.1, 52.7, 49.5, 25.4, 21.9. IR (KBr) 3364, 2923, 1712, 1384, 1171. HRMS (ESI) calcd for $C_{33}H_{29}NNaO_5S$ $[M+Na]^+$ 574.1659, found 574.1658.



3m

(4*R*,5*R*)-4-methyl-5-phenyl-4-(*o*-tolyl)-1-tosyl-4,5-dihydro-1*H*-benzofuro[3,2-*b*]azepin-2(3*H*)-one

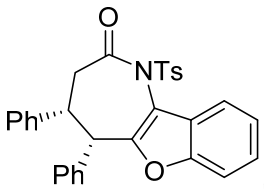
35.0 mg, >20:1 dr, 65% yield. White solid, m.p. 220-221 ^{o}C. R_f = 0.3 (petroleum ether/ethyl acetate 5:1). $[\alpha]_D^{25}$ -26.0 (*c* 1.0, $CHCl_3$). HPLC analysis: 99% [Daicel CHIRALPAK IC column, 20 ^{o}C, 254 nm, hexane/*i*-PrOH = 70:30, 1.0 mL/min, 22.2 min (minor), 27.3 min (major)]. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.00 (d, J = 8.2 Hz, 2H), 7.93 (d, J = 7.8 Hz, 1H), 7.41-7.29 (m, 5H), 7.17 (d, J = 7.3 Hz, 1H), 7.08 (t, J = 7.7 Hz, 2H), 7.07-6.98 (m, 2H), 6.70-6.66 (m, 1H), 6.51 (d, J = 7.8 Hz, 2H), 6.12 (d, J = 7.9 Hz, 1H), 4.28 (s, 1H), 3.26 (d, J = 12.3 Hz, 1H), 2.42 (s, 3H), 2.32 (d, J = 12.3 Hz, 1H), 1.75 (s, 3H), 1.37 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.7, 153.7, 153.4, 145.7, 141.8, 137.2, 135.6, 131.8, 130.9, 130.2, 129.8, 129.6, 128.7, 128.2, 127.7, 127.6, 127.2, 126.7, 125.04, 125.00, 124.8, 124.1, 121.6, 117.1, 112.1, 57.9, 49.9, 45.9, 25.4, 21.9, 19.9. IR (KBr) 3407, 1608, 1384, 1161. HRMS (ESI) calcd for $C_{33}H_{29}NNaO_4S$ $[M+Na]^+$ 558.1710, found 558.1715.



3n

(4*R*,5*R*)-4-methyl-4-phenyl-5-(thiophen-2-yl)-1-tosyl-1,3,4,5-tetrahydro-2*H*-benzofuro[3,2-*b*]azepin-2-one

22.9 mg, >20:1 dr, 43% yield. White solid, m.p. 237-239 ^{o}C. R_f = 0.3 (petroleum ether/ethyl acetate 5:1). $[\alpha]_D^{23}$ -73.8 (*c* 0.26, $CHCl_3$). HPLC analysis: 97% ee [Daicel CHIRALPAK IA column, 20 ^{o}C, 254 nm, hexane/*i*-PrOH = 70:30, 1.0 mL/min, 27.9 min (minor), 37.3 min (major)]. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.92-7.88 (m, 3H), 7.45 (d, *J* = 8.0 Hz, 1H), 7.39-7.32 (m, 4H), 7.16 (d, *J* = 7.3 Hz, 1H), 7.10 (t, *J* = 7.6 Hz, 2H), 7.03 (d, *J* = 5.1 Hz, 1H), 6.73-6.71 (m, 1H), 6.57 (d, *J* = 7.8, 2H), 6.06 (d, *J* = 3.3 Hz, 1H), 3.77 (s, 1H), 3.13 (d, *J* = 12.4 Hz, 1H), 2.48 (s, 3H), 2.27 (d, *J* = 12.4 Hz, 1H), 1.30 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.2, 153.6, 152.0, 145.7, 141.3, 135.3, 133.2, 129.8, 128.9, 128.2, 127.7, 126.9, 126.1, 125.4, 125.19, 125.15, 124.2, 121.2, 117.1, 112.0, 55.9, 49.7, 47.7, 25.4, 21.9. IR (KBr) 2921, 1714, 1609, 1169, 751. HRMS (ESI) calcd for $C_{30}H_{25}NO_4SNa$ $[M+Na]^+$ 550.1117, found 550.1118.

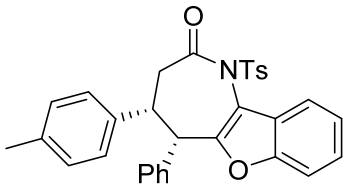


5a

(4*R*,5*S*)-4,5-diphenyl-1-tosyl-4,5-dihydro-1*H*-benzofuro[3,2-*b*]azepin-2(3*H*)-one

40.0 mg, 9:1 dr, 79% yield. Yellow solid, m.p. 185-187 °C. R_f = 0.3 (petroleum ether/ethyl acetate 3:1). $[\alpha]_D^{25}$ -21.8 (*c* 1.0, $CHCl_3$). HPLC analysis: 98% ee [Daicel CHIRALPAK IA column, 20 ^{o}C, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 10.3 min (major), 13.9 min (minor)]. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98-7.96 (m, 3H), 7.55 (d, *J* = 8.1 Hz, 1H), 7.46-7.37 (m, 4H), 7.19-7.15 (m, 2H), 7.11-7.07 (m, 4H), 6.68 (d, *J* = 7.7 Hz, 2H), 6.45 (d, *J* = 7.7 Hz, 2H), 3.96 (d, *J* = 6.8 Hz, 1H), 3.80-3.75 (m, 1H), 2.87 (t, *J* = 12.0 Hz, 1H), 2.67 (dd, *J* = 12.5, 5.9 Hz, 1H), 2.52 (s, 3H). ^{13}C

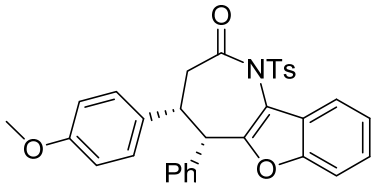
NMR (100 MHz, $CDCl_3$) δ 172.6, 154.0, 152.4, 145.8, 138.4, 135.2, 134.2, 130.1, 129.8, 129.2, 128.5, 128.3, 127.93, 127.87, 127.6, 125.1, 125.0, 124.3, 121.3, 117.6, 112.0, 56.7, 46.0, 43.1, 21.9. IR (KBr) 3501, 1713, 1386, 1175, 1087. HRMS (ESI) calcd for $C_{31}H_{25}NNaO_4S$ $[M+Na]^+$ 530.1408, found 530.1405.



5b

(4*R*,5*S*)-5-phenyl-4-(*p*-tolyl)-1-tosyl-4,5-dihydro-1*H*-benzofuro[3,2-*b*]azepin-2(3*H*)-one

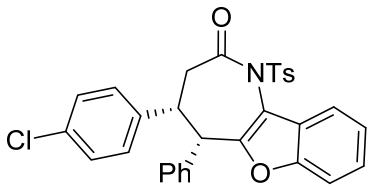
38.0 mg, 13:1 dr, 73% yield. Yellow oil. R_f = 0.2 (petroleum ether/ethyl acetate 10:1). $[\alpha]_D^{25}$ -20.6 (*c* 1.0, $CHCl_3$). HPLC analysis: 78% ee [Daicel CHIRALPAK IA column, 20 ^oC, 220 nm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 9.6 min (major), 13.1 min (minor)]. ^1H NMR (500 MHz, $CDCl_3$) δ 7.97-7.95 (m, 3H), 7.54 (d, *J* = 8.1 Hz, 1H), 7.44-7.37 (m, 4H), 7.18 (t, *J* = 7.3 Hz, 1H), 7.10 (t, *J* = 7.5 Hz, 2H), 6.89 (d, *J* = 7.8 Hz, 2H), 6.69 (d, *J* = 7.6 Hz, 2H), 6.33 (d, *J* = 7.9 Hz, 2H), 3.92 (d, *J* = 6.7 Hz, 1H), 3.77-3.72 (m, 1H), 2.84 (t, *J* = 11.9 Hz, 1H), 2.66-2.62 (m, 1H), 2.51 (s, 3H), 2.25 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.7, 154.0, 152.5, 145.7, 137.6, 135.3, 135.2, 134.3, 130.2, 129.8, 129.2, 129.1, 128.2, 127.9, 127.6, 125.1, 124.3, 121.3, 117.6, 112.0, 56.4, 46.1, 43.3, 21.9, 21.2. IR (KBr) 3419, 2922, 1715, 1372, 1174. HRMS (ESI) calcd for $C_{32}H_{27}NNaO_4S$ $[M+Na]^+$ 544.1564, found 544.1563.



5c

(4*R*,5*S*)-4-(4-methoxyphenyl)-5-phenyl-1-tosyl-4,5-dihydro-1H-benzofuro[3,2-*b*]azepin-2(3*H*)-one

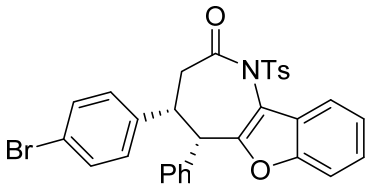
36.0 mg, 13:1 dr, 67% yield. Yellow solid, m.p. 185-187 °C. R_f = 0.2 (petroleum ether/ethyl acetate 10:1). $[\alpha]_D^{25}$ -3.8 (*c* 1.0, $CHCl_3$). HPLC analysis: 99% ee [Daicel CHIRALPAK IA column, 20 °C, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 12.0 min (major), 16.4 min (minor)]. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.97-7.95 (m, 3H), 7.54 (d, *J* = 8.0 Hz, 1H), 7.46-7.37 (m, 4H), 7.18 (t, *J* = 7.3 Hz, 1H), 7.11 (t, *J* = 7.5 Hz, 2H), 6.69 (d, *J* = 7.6 Hz, 2H), 6.62 (d, *J* = 8.4 Hz, 2H), 6.36 (d, *J* = 8.4 Hz, 2H), 3.90 (d, *J* = 6.6 Hz, 1H), 3.76-3.72 (m, 4H), 2.81 (t, *J* = 11.9 Hz, 1H), 2.63 (dd, *J* = 12.4, 6.0 Hz, 1H), 2.52 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 172.7, 159.2, 154.0, 152.5, 145.8, 135.2, 134.3, 130.4, 130.2, 129.8, 129.3, 129.2, 128.0, 127.6, 125.1, 125.0, 124.3, 121.3, 117.5, 113.8, 112.0, 56.1, 55.3, 46.2, 43.3, 21.9. IR (KBr) 3390, 2918, 1747, 1385, 1161. HRMS (ESI) calcd for $C_{32}H_{27}NNaO_5S$ $[M+Na]^+$ 560.1513, found 560.1509.



5d

(4*R*,5*S*)-4-(4-chlorophenyl)-5-phenyl-1-tosyl-4,5-dihydro-1*H*-benzofuro[3,2-*b*]azepin-2(3*H*)-one

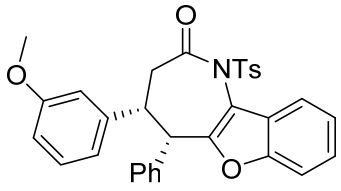
50.4 mg, 9:1 dr, 93% yield. Yellow oil. R_f = 0.2 (petroleum ether/ethyl acetate 10:1). $[\alpha]_D^{25}$ -3.7 (*c* 1.0, $CHCl_3$). HPLC analysis: 99% ee [Daicel CHIRALPAK IA column, 20 °C, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 12.3 min (major), 16.3 min (minor)]. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.4 Hz, 3H), 7.56-7.54 (m, 1H), 7.45-7.36 (m, 4H), 7.19-7.11 (m, 3H), 7.05 (d, *J* = 8.4 Hz, 2H), 6.72 (d, *J* = 7.2 Hz, 2H), 6.38 (d, *J* = 8.4 Hz, 2H), 3.94 (t, *J* = 6.7 Hz, 1H), 3.77-3.74 (m, 1H), 2.80 (t, *J* = 11.9 Hz, 1H), 2.63 (dd, *J* = 12.4, 6.0 Hz, 1H), 2.51 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.2, 154.0, 152.0, 145.8, 136.9, 135.1, 133.9, 133.7, 130.0, 129.8, 129.6, 129.2, 128.6, 128.1, 127.8, 125.3, 124.9, 124.4, 121.4, 117.8, 112.0, 56.1, 45.8, 43.0, 21.9. IR (KBr) 3419, 2924, 1713, 1385, 1174. HRMS (ESI) calcd for $C_{31}H_{24}ClNNaO_4S$ $[M+Na]^+$ 564.1018, found 564.1017.



5e

(4*R*,5*S*)-4-(4-bromophenyl)-5-phenyl-1-tosyl-4,5-dihydro-1*H*-benzofuro[3,2-*b*]azepin-2(3*H*)-one

44.0 mg, 8:1 dr, 75% yield. Yellow solid, m.p. 149-151°C. R_f = 0.2 (petroleum ether/ethyl acetate 10:1). $[\alpha]_D^{25}$ +13.6 (*c* 1.0, $CHCl_3$). HPLC analysis: 99% ee [Daicel CHIRALPAK IA column, 20 °C, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 12.9 min (major), 16.8 min (minor)]. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.96-7.95 (m, 3H), 7.55 (d, *J* = 8.0 Hz, 1H), 7.46-7.36 (m, 4H), 7.25-7.18 (m, 3H), 7.13 (t, *J* = 7.4 Hz, 2H), 6.71 (d, *J* = 7.5 Hz, 2H), 6.32 (d, *J* = 8.3 Hz, 2H), 3.94 (d, *J* = 6.5 Hz, 1H), 3.75-3.73 (m, 1H), 2.79 (t, *J* = 12.2 Hz, 1H), 2.64 (dd, *J* = 12.2, 5.9 Hz, 1H), 2.51 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 172.1, 153.9, 151.9, 145.7, 137.3, 135.0, 133.8, 131.5, 129.9, 129.8, 129.7, 129.1, 128.0, 127.7, 125.2, 124.8, 124.3, 121.7, 121.3, 117.7, 111.9, 56.0, 45.6, 42.8, 21.8. IR (KBr) 3446, 2924, 1715, 1386, 1137. HRMS (ESI) calcd for $C_{31}H_{24}BrNNaO_4S$ $[M+Na]^+$ 608.0513, found 608.0511.

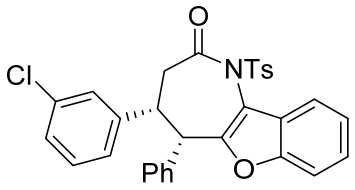


5f

(4*R*,5*S*)-4-(3-bromophenyl)-5-phenyl-1-tosyl-4,5-dihydro-1*H*-benzofuro[3,2-*b*]azepin-2(3*H*)-one

43.0 mg, 13:1 dr, 77% yield. Yellow oil. $[\alpha]_D^{25}$ -18.3 (*c* 1.0, $CHCl_3$). R_f = 0.2 (petroleum ether/ethyl acetate 10:1). HPLC analysis: >99% ee [Daicel CHIRALPAK IA column, 20 °C, 210 nm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 11.4 min (major), 17.4 min (minor)]. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98-7.96 (m, 3H), 7.54 (d, *J* = 7.7 Hz, 1H), 7.45-7.37 (m, 4H), 7.18-7.10 (m, 3H), 7.00 (t, *J* = 7.9 Hz, 1H), 6.74-6.69 (m,

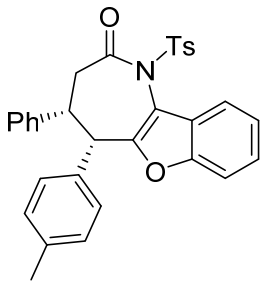
3H), 6.07 (d, *J* = 7.7 Hz, 1H), 5.96 (s, 1H), 3.95 (d, *J* = 6.7 Hz, 1H), 3.76-3.73 (m, 1H), 3.53 (s, 3H), 2.83 (t, *J* = 11.9 Hz, 1H), 2.66 (dd, *J* = 12.4, 6.0 Hz, 1H), 2.51 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.6, 159.5, 154.0, 152.4, 145.8, 139.9, 135.2, 134.2, 130.1, 129.8, 129.4, 129.2, 128.0, 127.6, 125.2, 125.0, 124.3, 121.4, 120.7, 117.7, 113.7, 111.9, 56.6, 55.2, 45.9, 43.2, 21.9. IR (KBr) 3446, 2923, 1716, 1366, 1175. HRMS (ESI) calcd for $C_{32}H_{27}NNaO_5S$ $[M+Na]^+$ 560.1513, found 560.1514.



5g

(4*R*,5*S*)-4-(3-chlorophenyl)-5-phenyl-1-tosyl-4,5-dihydro-1*H*-benzofuro[3,2-*b*]azepin-2(3*H*)-one

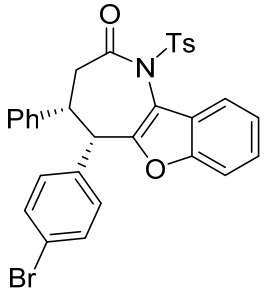
43.0 mg, 9:1 dr, 80% yield. Yellow oil. R_f = 0.2 (petroleum ether/ethyl acetate 10:1). $[\alpha]_D^{25}$ +140.8 (*c* 1.0, $CHCl_3$). HPLC analysis: 98% ee [Daicel CHIRALPAK IB column, 20 ^{o}C, 210 nm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 11.0 min (major), 12.3 min (minor)]. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.97-7.96 (m, 3H), 7.57 (d, *J* = 8.1 Hz, 1H), 7.47-7.37 (m, 4H), 7.22-7.19 (m, 1H), 7.15-7.12 (m, 3H), 7.02 (t, *J* = 7.7 Hz, 1H), 6.72 (d, *J* = 7.8 Hz, 2H), 6.43 (s, 1H), 6.35 (d, *J* = 7.6 Hz, 1H), 3.96 (d, *J* = 6.6 Hz, 1H), 3.76-3.71 (m, 1H), 2.80 (t, *J* = 11.8 Hz, 1H), 2.65 (dd, *J* = 12.3, 5.6 Hz, 1H), 2.52 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 172.2, 154.1, 151.9, 145.9, 140.5, 135.1, 134.3, 133.8, 130.0, 129.8, 129.7, 129.2, 128.5, 128.2, 128.0, 127.9, 126.4, 125.4, 124.9, 124.4, 121.3, 117.9, 112.1, 56.1, 45.8, 42.9, 21.9. IR (KBr) 3421, 2924, 1715, 1385, 1175. HRMS (ESI) calcd for $C_{31}H_{24}ClNNaO_4S$ $[M+Na]^+$ 564.1007, found 564.1009.



5h

(4*R*,5*S*)-4-phenyl-5-(*p*-tolyl)-1-tosyl-4,5-dihydro-1*H*-benzofuro[3,2-*b*]azepin-2(3*H*)-one

39.0 mg, 12:1 dr, 75% yield. Yellow solid, m.p. 112-114°C; R_f = 0.2 (petroleum ether/ethyl acetate 10:1). $[\alpha]_D^{25}$ -32.2 (*c* 1.0, $CHCl_3$). HPLC analysis: 99% ee [Daicel CHIRALPAK IA column, 20 °C, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 10.1 min (major), 13.2 min (minor)]. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97-7.94 (m, 3H), 7.54 (d, *J* = 7.9 Hz, 1H), 7.46-7.37 (m, 4H), 7.17 (t, *J* = 7.4 Hz, 1H), 7.09 (t, *J* = 7.4 Hz, 2H), 6.90 (d, *J* = 7.9 Hz, 2H), 6.53 (d, *J* = 8.0 Hz, 2H), 6.47 (d, *J* = 7.4 Hz, 2H), 3.90 (d, *J* = 6.7 Hz, 1H), 3.79-3.74 (m, 1H), 2.86 (t, *J* = 11.9 Hz, 1H), 2.66 (dd, *J* = 12.4, 6.0 Hz, 1H), 2.52 (s, 3H), 2.25 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 172.7, 154.0, 152.6, 145.7, 138.5, 137.3, 135.1, 131.0, 130.0, 129.8, 129.2, 128.7, 128.5, 128.3, 127.8, 125.1, 124.2, 121.3, 117.4, 112.0, 56.7, 45.7, 43.2, 21.9, 21.1. IR (KBr) 3445, 2921, 1715, 1373, 1187. HRMS (ESI) calcd for $C_{32}H_{27}NNaO_4S$ $[M+Na]^+$ 544.1553, found 544.1551.

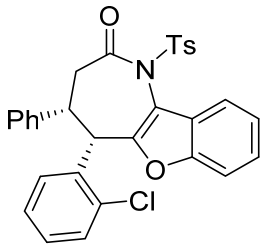


5i

(4*R*,5*S*)-5-(4-bromophenyl)-4-phenyl-1-tosyl-4,5-dihydro-1*H*-benzofuro[3,2-*b*]azepin-2(3*H*)-one

46.3 mg, 8:1 dr, 79% yield. Yellow solid, m.p. 145-150 °C. R_f = 0.2 (petroleum

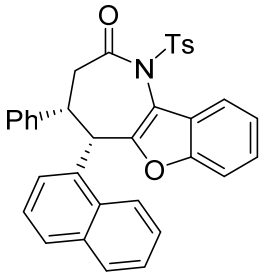
ether/ethyl acetate 10:1). $[\alpha]_D^{25}$ -15.9 (*c* 1.0, $CHCl_3$). HPLC analysis: >99% ee [Daicel CHIRALPAK IA column, 20 °C, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 12.5 min (major), 16.8 min (minor)]. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.97-7.95 (m, 3H), 7.54 (d, *J* = 8.0 Hz, 1H), 7.47-7.37 (m, 4H), 7.23-7.17 (m, 3H), 7.11 (t, *J* = 7.5 Hz, 2H), 6.55 (d, *J* = 8.2 Hz, 2H), 6.45 (d, *J* = 7.6 Hz, 2H), 3.91 (d, J = 6.7 Hz, 1H), 3.78-3.73 (m, 1H), 2.85 (t, *J* = 12.0 Hz, 1H), 2.67 (dd, *J* = 12.5, 6.0 Hz, 1H), 2.52 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 172.3, 153.9, 151.5, 145.7, 137.8, 135.0, 133.0, 131.6, 131.0, 129.7, 129.1, 128.5, 128.1, 128.0, 125.2, 124.8, 124.3, 121.8, 121.3, 117.6, 111.9, 56.4, 45.3, 42.9, 21.8. IR (KBr) 3419, 2922, 1715, 1385, 1174. HRMS (ESI) calcd for $C_{31}H_{24}BrNNaO_4S$ $[M+Na]^+$ 608.0502, found 608.0500.



5j

(4*R*,5*S*)-5-(2-chlorophenyl)-4-phenyl-1-tosyl-4,5-dihydro-1*H*-benzofuro[3,2-*b*]azepin-2(3*H*)-one

38.0 mg, 10:1 dr, 70% yield. Yellow oil. R_f = 0.2 (petroleum ether/ethyl acetate 10:1). $[\alpha]_D^{25}$ +36.4 (*c* 1.0, $CHCl_3$). HPLC analysis: 99% ee [Daicel CHIRALPAK IA column, 20 °C, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 10.5 min (major), 16.6 min (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.05-8.00 (m, 3H), 7.62-7.59 (m, 1H), 7.49-7.45 (m, 2H), 7.37 (d, *J* = 8.2 Hz, 2H), 7.31-7.28 (m, 1H), 7.19-7.04 (m, 5H), 6.93-6.90 (m, 1H), 6.44 (d, *J* = 7.2 Hz, 2H), 4.93 (d, *J* = 6.8 Hz, 1H), 3.91-3.89 (m, 1H), 2.92 (t, *J* = 12.1 Hz, 1H), 2.71 (dd, *J* = 12.4, 6.0 Hz, 1H), 2.50 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.8, 153.9, 151.5, 145.8, 138.2, 135.1, 133.7, 132.2, 132.0, 129.9, 129.24, 129.18, 128.6, 128.4, 128.0 127.9, 126.1, 125.2, 124.9, 124.4, 121.6, 118.2, 111.9, 54.6, 42.6, 41.0, 21.9. IR (KBr) 3420, 2923, 1713, 1387, 1174. HRMS (ESI) calcd for $C_{31}H_{24}ClNNaO_4S$ $[M+Na]^+$ 564.1007, found 564.1008.

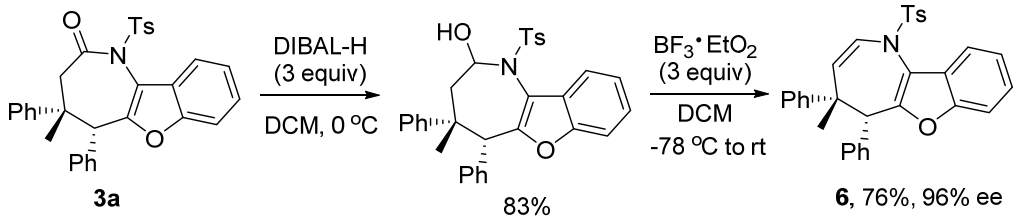


5k

(4*R*,5*S*)-5-(naphthalen-1-yl)-4-phenyl-1-tosyl-4,5-dihydro-1*H*-benzofuro[3,2-*b*]azepin-2(3*H*)-one

36.7 mg, 12:1 dr, 66% yield. Yellow oil. R_f = 0.2 (petroleum ether/ethyl acetate 10:1). $[\alpha]_D^{25}$ +44.9 (*c* 1.0, $CHCl_3$). HPLC analysis: >99% ee [Daicel CHIRALPAK IA column, 20 °C, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 11.3 min (major), 20.6 min (minor)]. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.04-7.99 (m, 3H), 7.82 (d, *J* = 8.1 Hz, 1H), 7.66-7.62 (m, 2H), 7.48-7.44 (m, 3H), 7.36 (d, *J* = 7.3 Hz, 1H), 7.28-7.24 (m, 3H), 7.13-7.04 (m, 3H), 6.97 (t, *J* = 7.6 Hz, 2H), 6.23 (d, *J* = 7.6 Hz, 2H), 5.07 (d, *J* = 6.5 Hz, 1H), 3.94-3.90 (m, 1H), 2.99 (t, *J* = 12.0 Hz, 1H), 2.74 (dd, *J* = 12.4, 6.0 Hz, 1H), 2.35 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 172.7, 153.9, 152.5, 145.7, 138.1, 135.1, 133.7, 130.8, 129.9, 129.32, 129.29, 129.1, 128.7, 128.1, 127.9, 127.8, 127.6, 126.3, 125.2, 125.1, 124.8, 124.7, 124.3, 121.5, 121.3, 118.3, 111.9, 55.8, 42.5, 39.4, 21.9. IR (KBr) 3446, 2922, 1714, 1384, 1175. HRMS (ESI) calcd for $C_{35}H_{27}NNaO_4S$ $[M+Na]^+$ 580.1553, found 580.1552.

3. Synthesis of dihydroazepine 6 (Scheme 4)



To a stirred solution of compound **3a** (52.1 mg, 0.1 mmol) in CH_2Cl_2 (2.0 mL) at 0 °C under N_2 was added dropwisely DIBAL-H (1.5 M in toluene, 0.2 ml, 0.3 mmol).[4] The

mixture was stirred at 0 °C for about 30 min (TLC). When the reaction was completed, it was quenched with brine and extracted with Et_2O. The organic layer were dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified *via* SiO_2 flash chromatography (petroleum ether/EtOAc 5:1 as the eluent) to afford the hemiaminal **3a'** (43 mg, 83% yield).

To a stirred solution of the hemiaminal **3a'** (26 mg, 0.05 mmol) in CH_2Cl_2 (2.0 mL) at -78 °C under N_2 was added $BF_3.Et_2O$ (19 μL, 0.15 mmol). The mixture was allowed to warm to room temperature and stirred for 2 h (TLC). The reaction was quenched with aqueous $NaHCO_3$ (aq.) and extracted with CH_2Cl_2. The combined organic layers were dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified via SiO_2 flash chromatography (petroleum ether/EtOAc 10:1 as the eluent) to afford dihydroazepine **6**.

(4*R*,5*R*)-4-methyl-4,5-diphenyl-1-tosyl-4,5-dihydro-1*H*-benzofuro[3,2-*b*]azepine

20 mg, 75% yield. White solid. m.p. 95-97°C R_f = 0.3 (petroleum ether/ethyl acetate 10:1). $[\alpha]_D^{25}$ -47.2 (*c* 1.0, $CHCl_3$). HPLC analysis: 96% ee [Daicel CHIRALPAK IB column, 20 ^oC, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL/min, 5.9 min (major), 7.0 min (minor)]. ^1H NMR (500 MHz, $CDCl_3$) δ 7.94 (d, J = 7.8 Hz, 1H), 7.69 (d, J = 8.2 Hz, 2H), 7.40 – 7.29 (m, 3H), 7.25 – 7.15 (m, 3H), 7.15 – 7.10 (m, 3H), 6.99 (t, J = 7.8 Hz, 2H), 6.89 (d, J = 9.8 Hz, 1H), 6.62 (d, J = 7.5 Hz, 2H), 6.33 (d, J = 7.4 Hz, 2H), 5.12 (d, J = 9.8 Hz, 1H), 3.18 (s, 1H), 2.58 (s, 3H), 1.04 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 152.8, 152.1, 144.3, 140.4, 134.4, 134.0, 131.5, 129.8, 128.1, 128.0, 127.9, 127.6, 127.5, 127.2, 127.1, 126.0, 124.1, 123.4, 123.3, 120.8, 118.3, 111.4, 52.6, 45.3, 27.6, 21.7. IR (KBr) 3460, 2929, 1723, 1387, 1170, 598. HRMS (ESI) calcd for $C_{32}H_{26}NO_3S$ $[M-H]^-$ 504.1639, found 504.1648.

4. References

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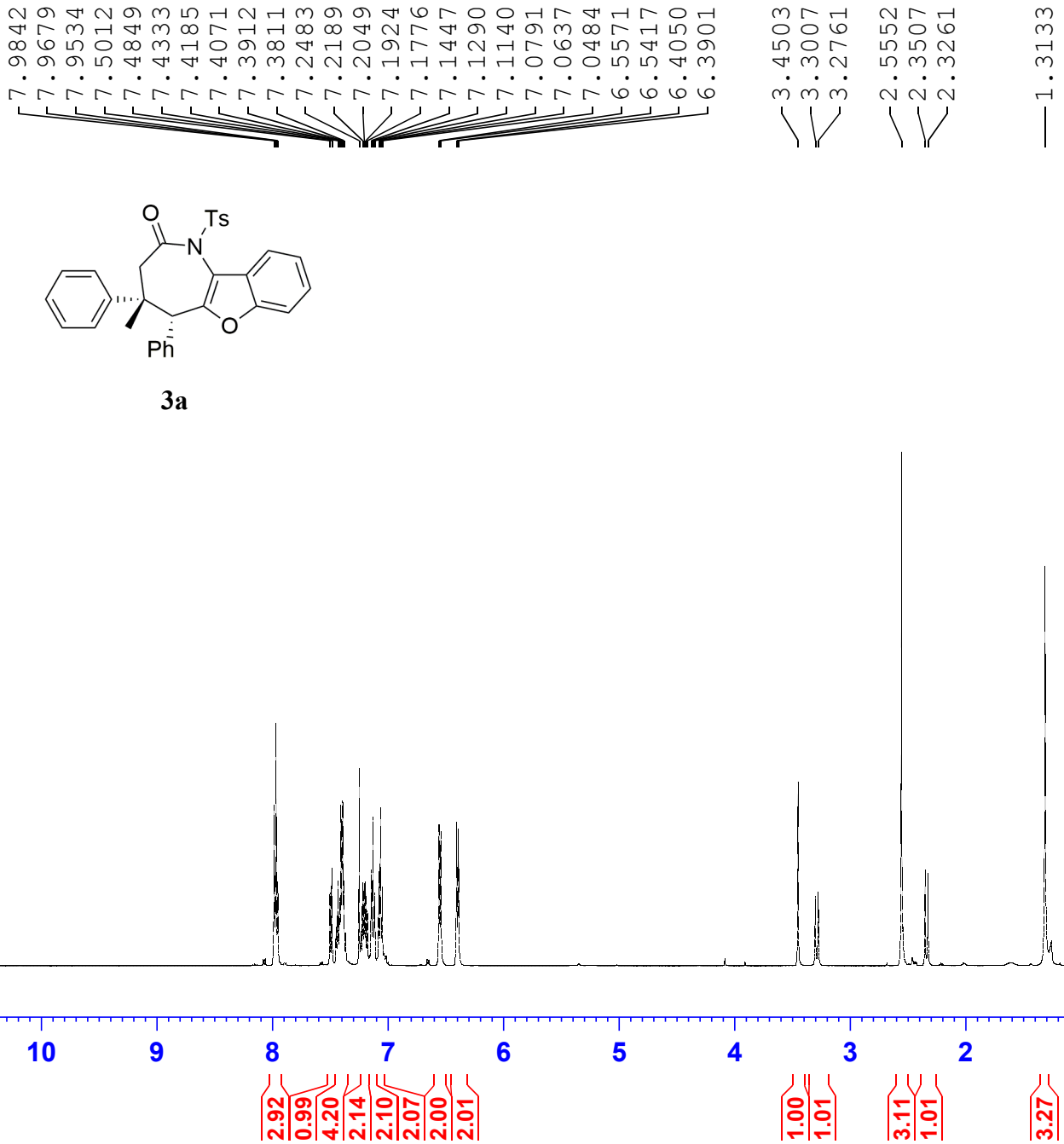
2 R. Zetzsche, *Chem. Ber.*, 1923, **56**, 1483.

3 Z.-Q. Rong, W. Min, C. C. H. Eugene and Z. Yu, *Chem. Eur. J.*, 2016, **22**, 9483.

4 S. Chen, L. Hao, Y. Zhang, B. Tiwari and Y. R. Chi, *Org. Lett.*, 2013, **15**, 5822.



807B

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PROCNO                   1

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RG                   31.72
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DE                   6.50 usec
TE                  298.1 K
D1              1.00000000 sec
TD0                      1
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NUC1                   1H
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PLW1          20.00000000 W

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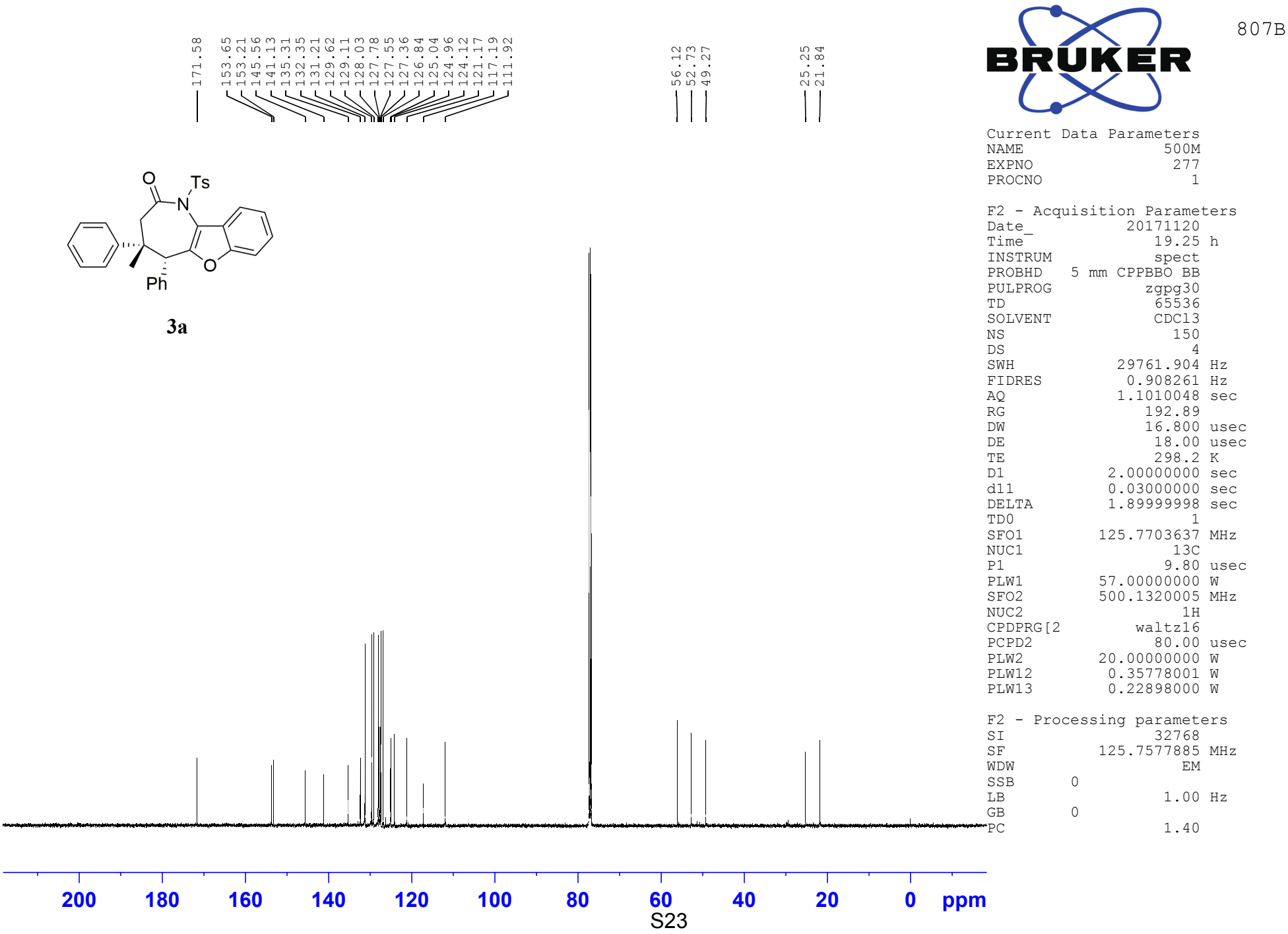

807B

Current Data Parameters
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SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
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NUC1 13C
P1 9.80 usec
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NUC2 1H
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PLW2 20.00000000 W
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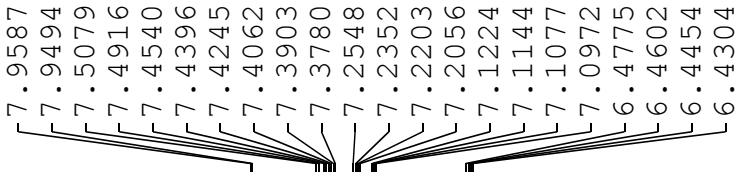
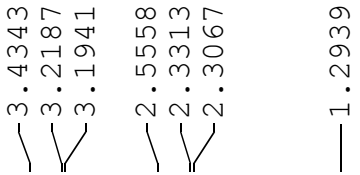
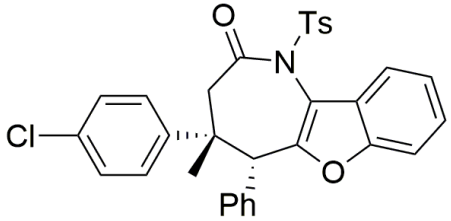
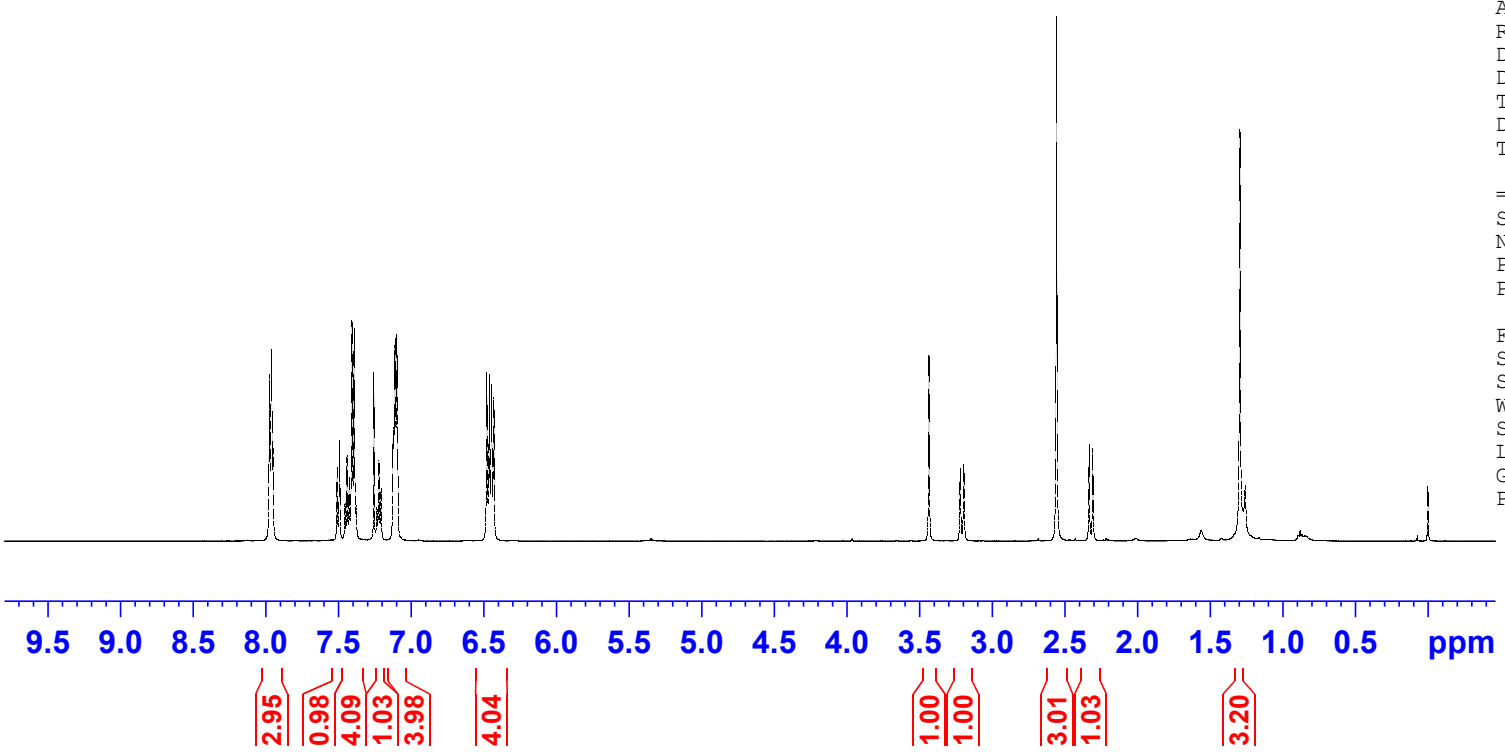






3b



836B

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PROCNO                      1

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TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
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FIDRES           0.152588 Hz
AQ               3.2767999 sec
RG                    31.72
DW                  50.000 usec
DE                   6.50 usec
TE                   298.1 K
D1              1.00000000 sec
TD0                       1

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NUC1                     1H
P1                    11.50 usec
PLW1           20.00000000 W

F2 - Processing parameters
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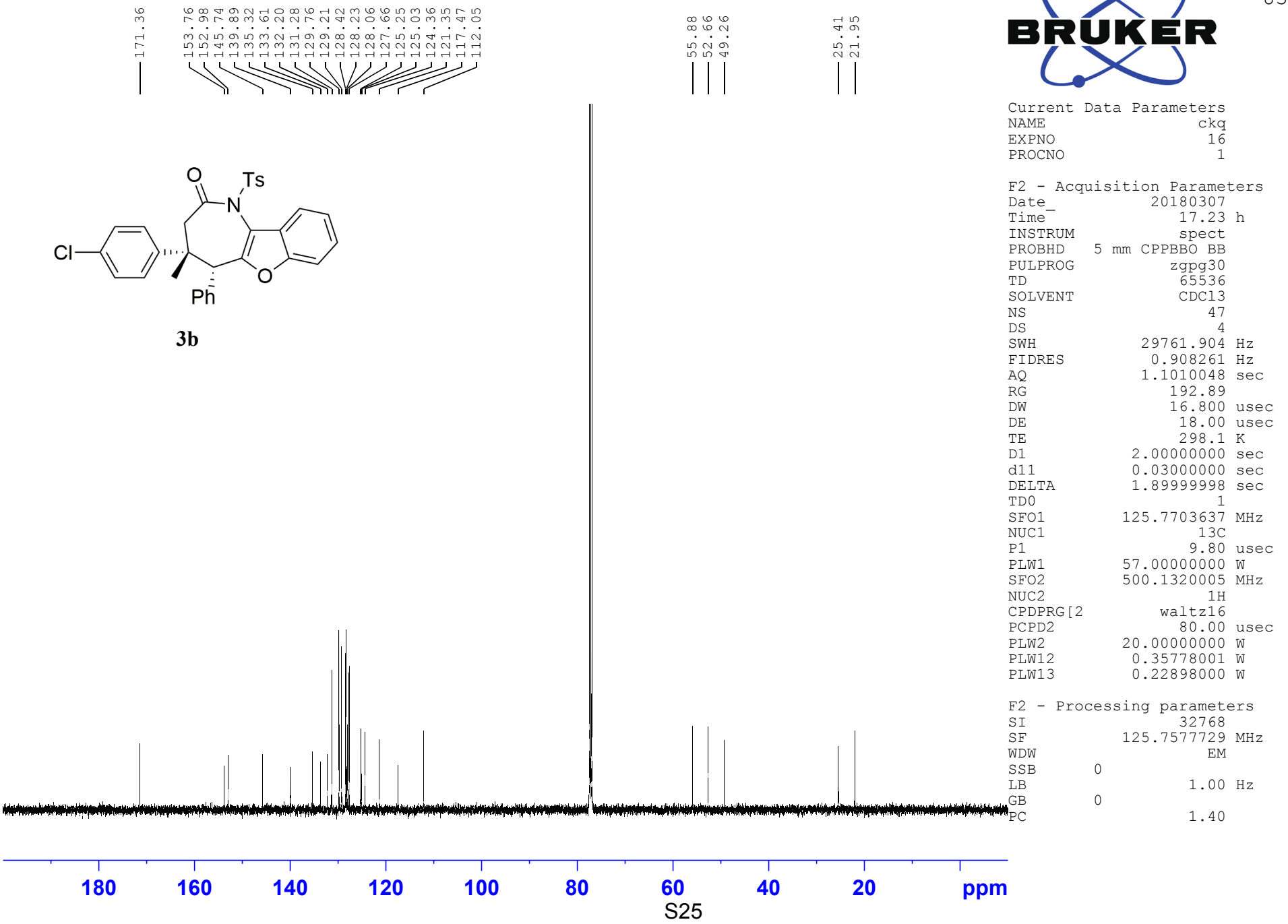

836B

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PROCNO 1

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FIDRES 0.908261 Hz
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RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.1 K
D1 2.00000000 sec
d11 0.03000000 sec
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NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
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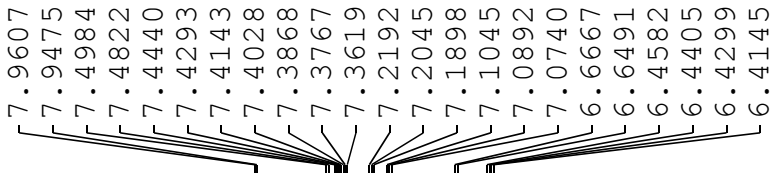
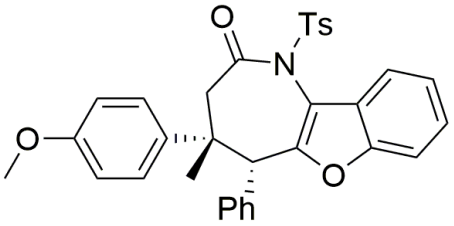
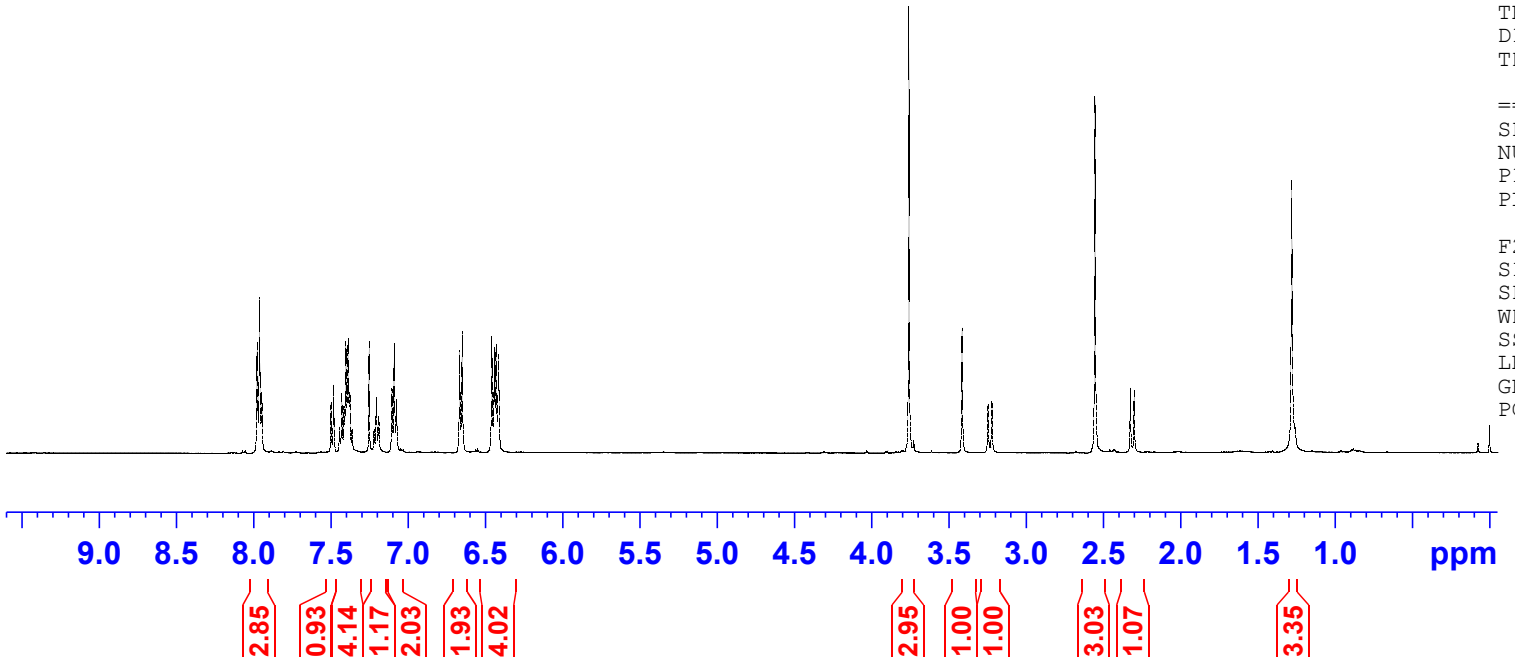




3c



837C

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EXPNO                       3
PROCNO                      1

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PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
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FIDRES            0.152588 Hz
AQ               3.2767999 sec
RG                    31.72
DW                  50.000 usec
DE                   6.50 usec
TE                  298.2 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
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NUC1                     1H
P1                   11.50 usec
PLW1           20.00000000 W

F2 - Processing parameters
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WDW                      EM
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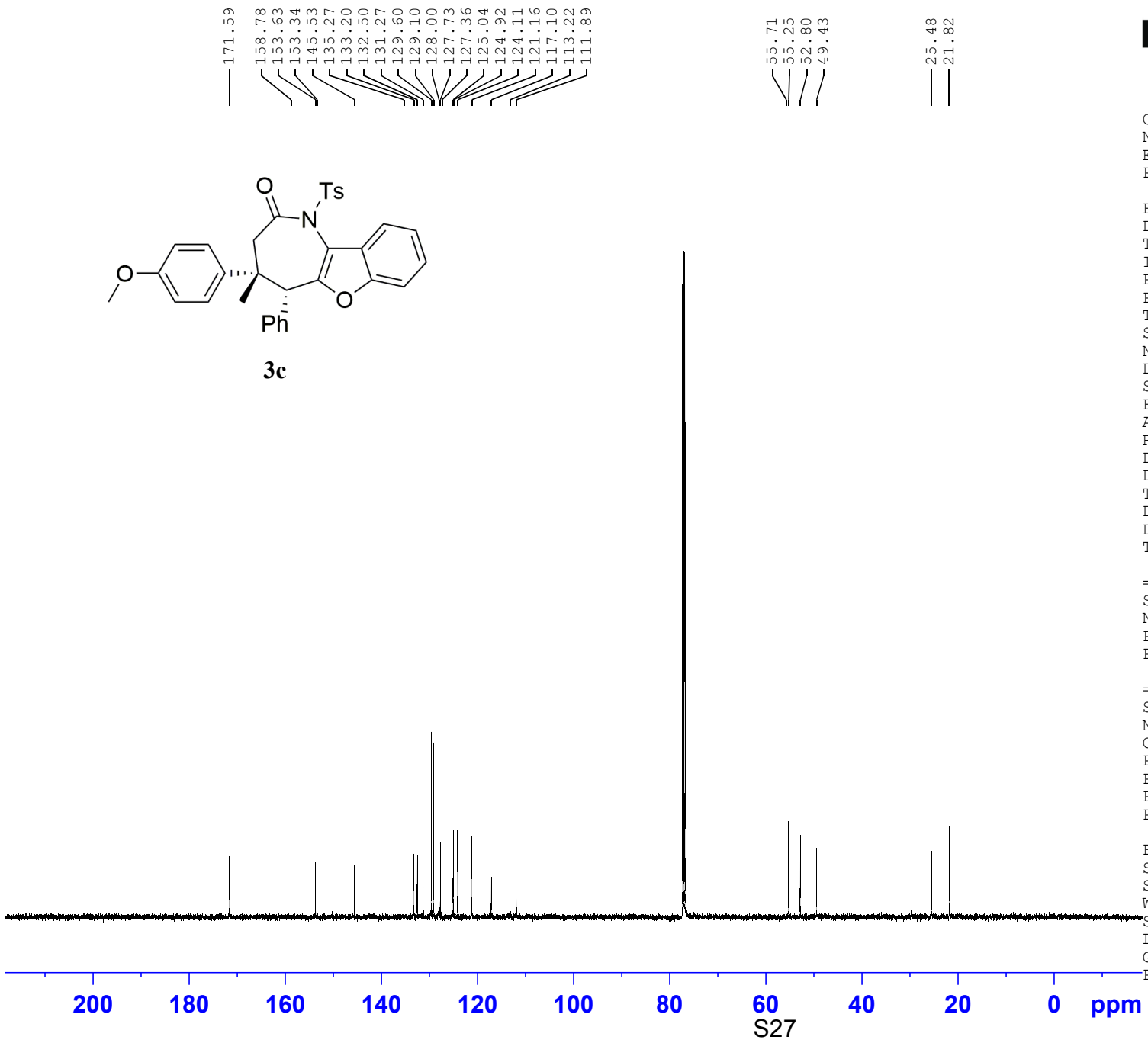

837C

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PROCNO 1

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TD 65536
SOLVENT CDCl3
NS 50
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
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NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W

======== CHANNEL f2 ========
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PLW12 0.35778001 W
PLW13 0.22898000 W

F2 - Processing parameters
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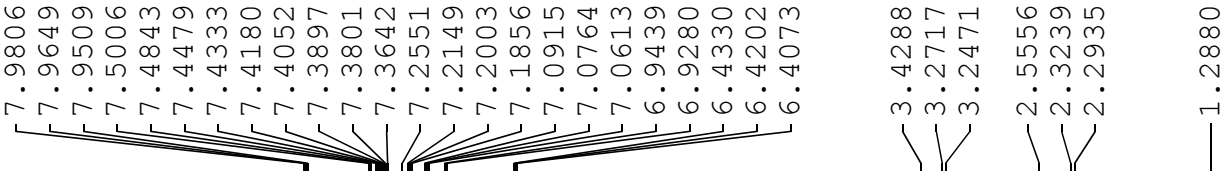
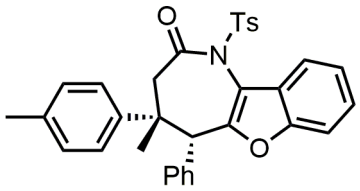
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

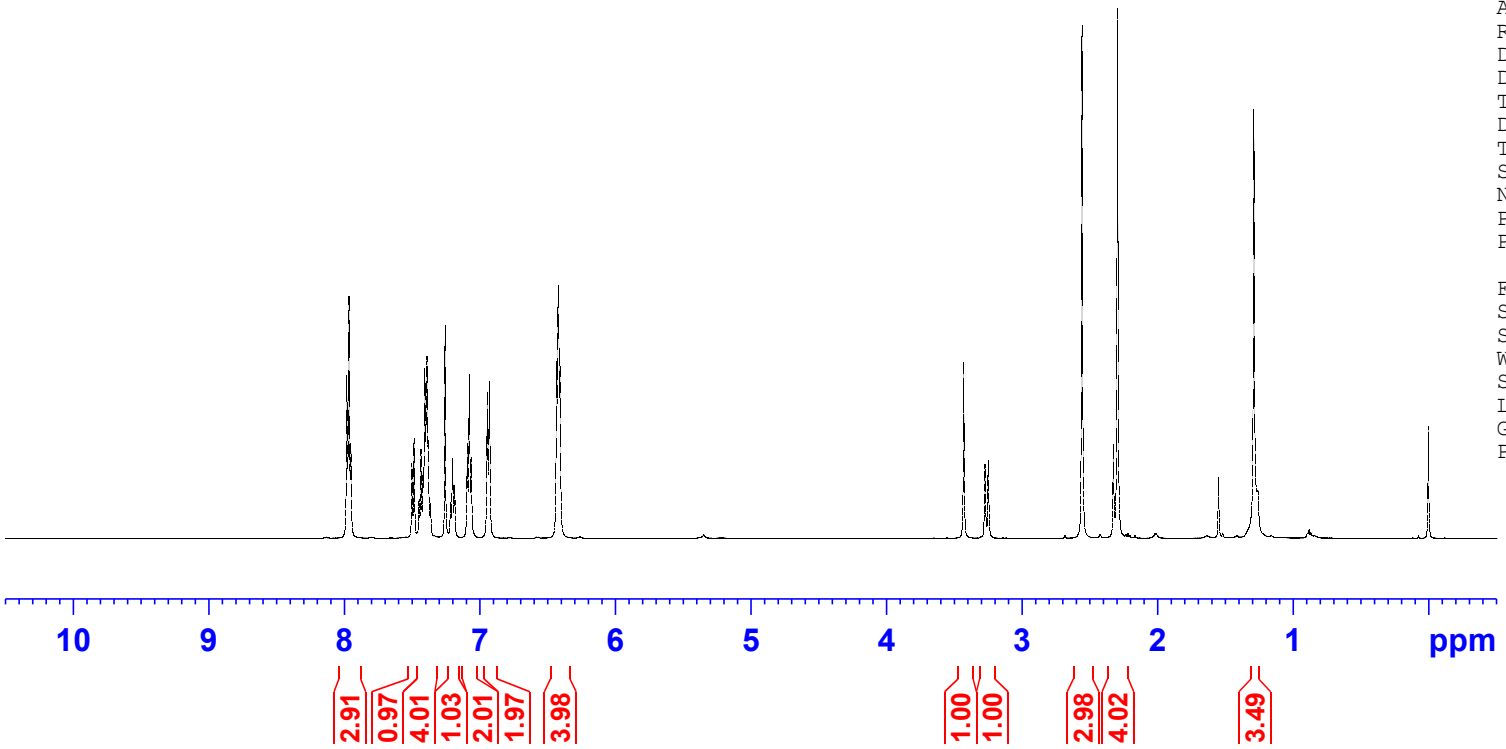


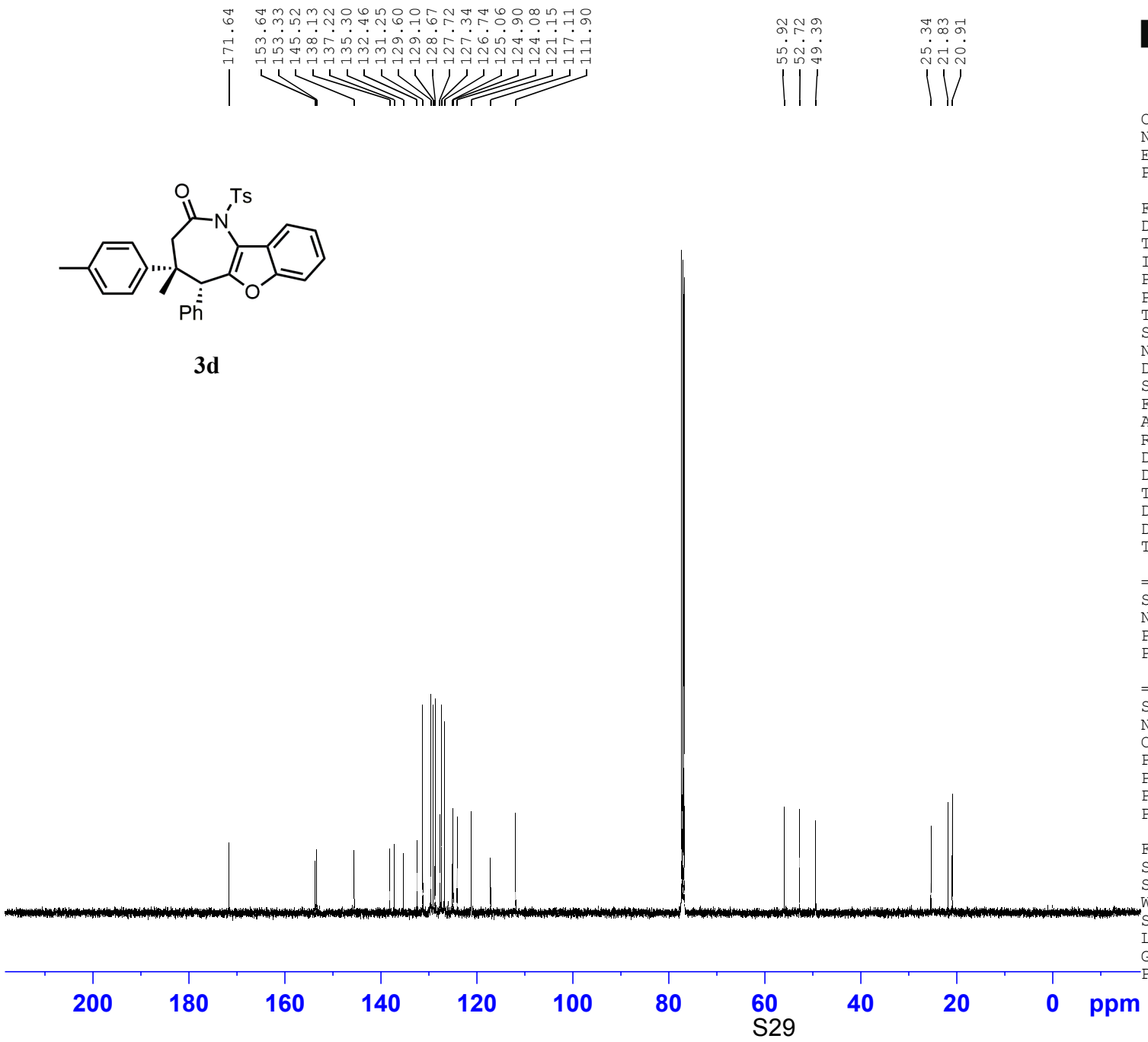


3d

836AA

```
Current Data Parameters
NAME                  500 M
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20180115
Time                 15.16 h
INSTRUM                spect
PROBHD    5 mm CPPBBO BB
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH              10000.000 Hz
FIDRES            0.305176 Hz
AQ                3.2767999 sec
RG                    55.37
DW                   50.000 usec
DE                    6.50 usec
TE                   298.1 K
D1              1.00000000 sec
TD0                       1
SFO1           500.1330885 MHz
NUC1                    1H
P1                   11.50 usec
PLW1           20.00000000 W

F2 - Processing parameters
SI                    65536
SF             500.1300143 MHz
WDW                      EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```





3d

836AA

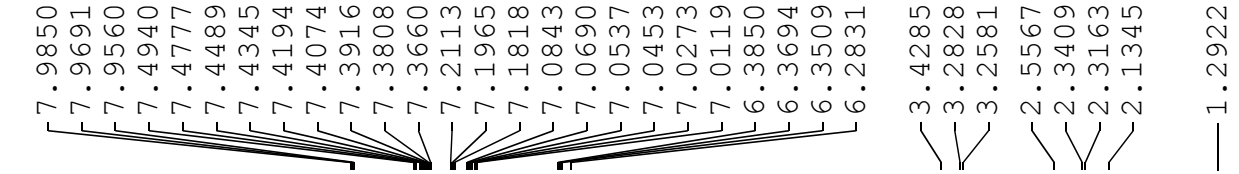
Current Data Parameters
NAME 500 M-1
EXPNO 8
PROCNO 1

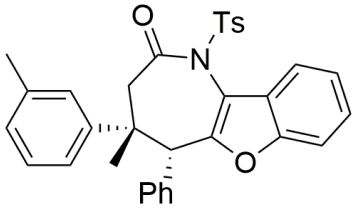
F2 - Acquisition Parameters
Date_ 20171215
Time 20.53
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 55
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W

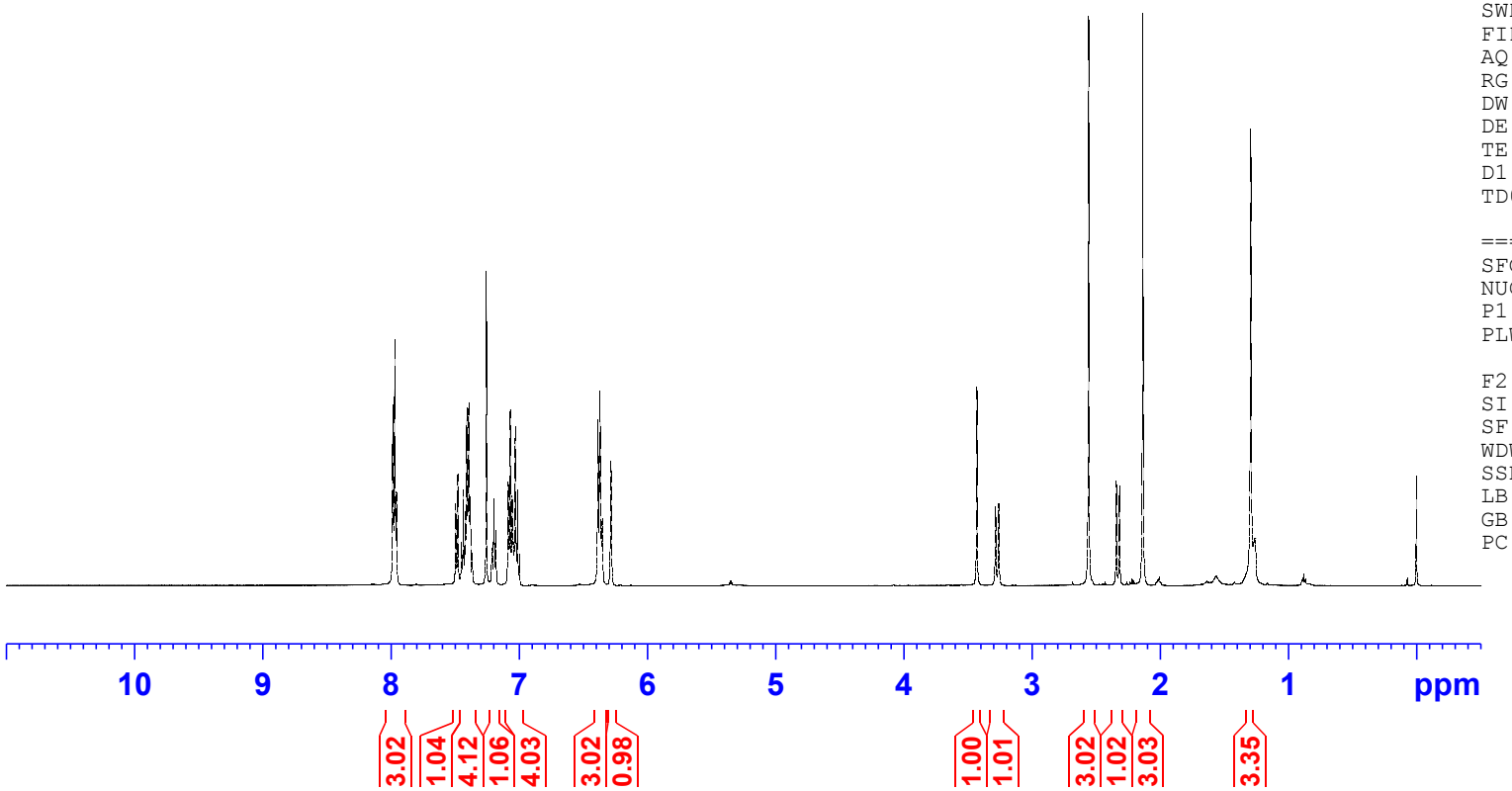
======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W

F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



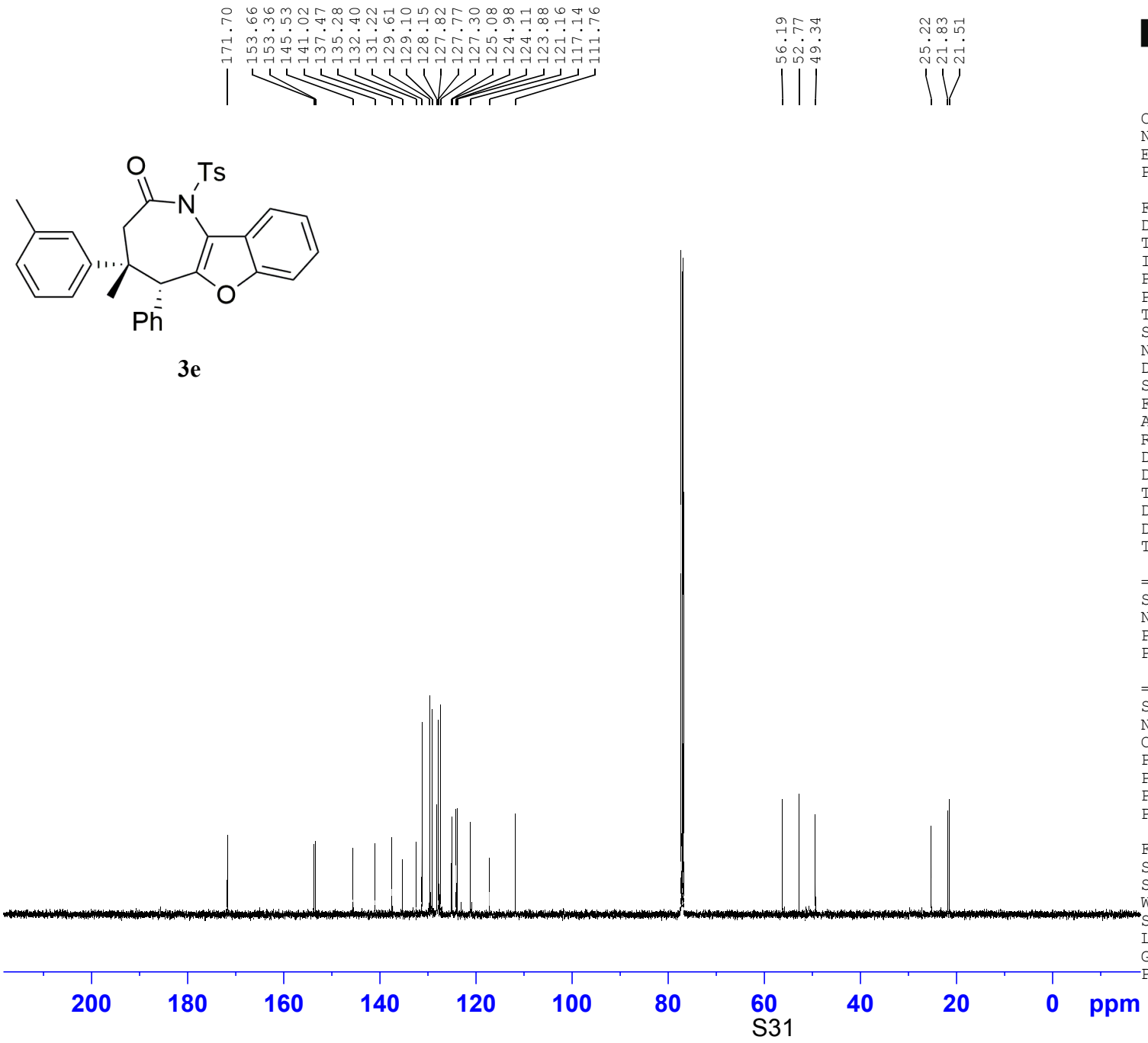


3e



838A

```
Current Data Parameters
NAME                  500 M-1
EXPNO                      22
PROCNO                      1

F2 - Acquisition Parameters
Date_               20171228
Time                   16.21
INSTRUM                spect
PROBHD    5 mm CPPBBO BB
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH              10000.000 Hz
FIDRES            0.152588 Hz
AQ                3.2767999 sec
RG                    31.72
DW                   50.000 usec
DE                    6.50 usec
TE                   298.1 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           500.1330885 MHz
NUC1                     1H
P1                    11.50 usec
PLW1           20.00000000 W

F2 - Processing parameters
SI                    65536
SF             500.1300146 MHz
WDW                      EM
SSB          0
LB                    0.30 Hz
GB           0
PC                    1.00
```



3e

838A

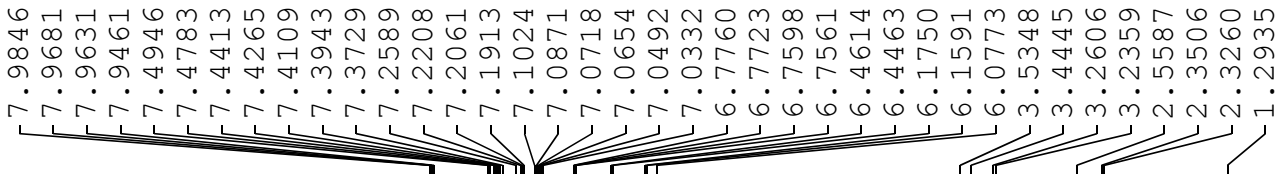
Current Data Parameters
NAME 500 M-1
EXPNO 10
PROCNO 1

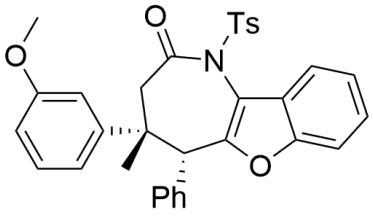
F2 - Acquisition Parameters
Date_ 20171215
Time 21.01
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 40
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W

======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W

F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





3f

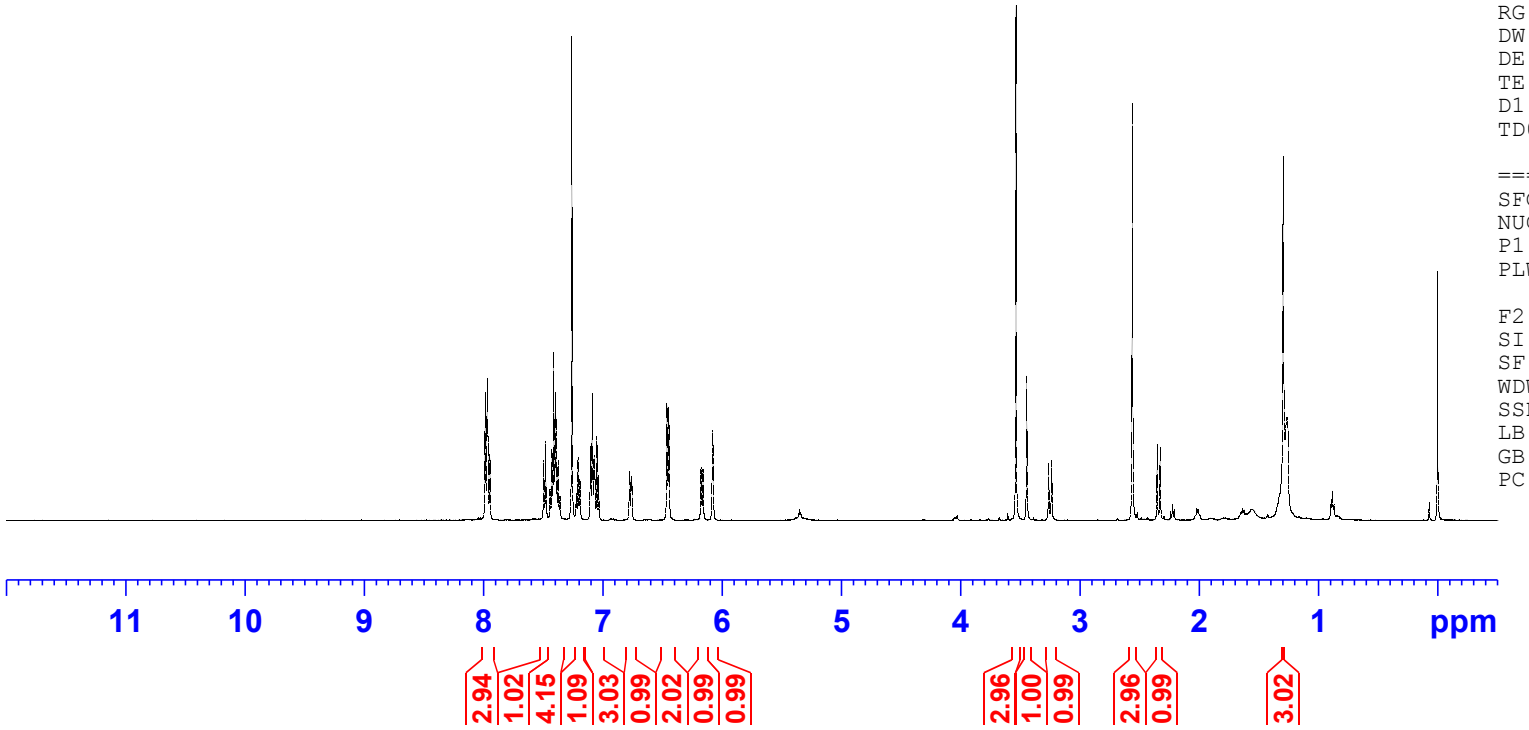
838C

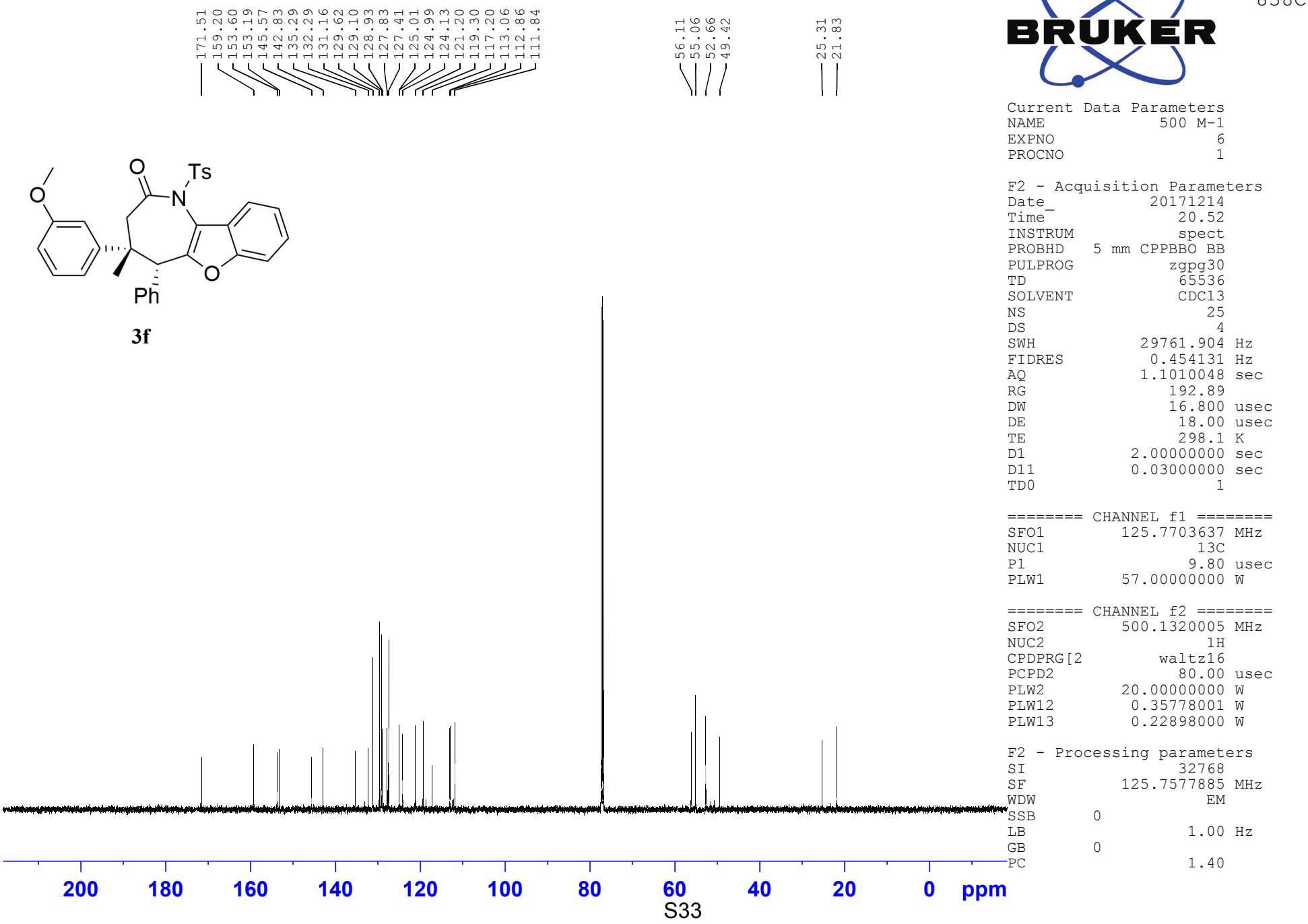
Current Data Parameters
NAME 500 M-1
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171220
Time 21.00
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 10
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 62.06
DW 50.000 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 11.50 usec
PLW1 20.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300124 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





3f

838C

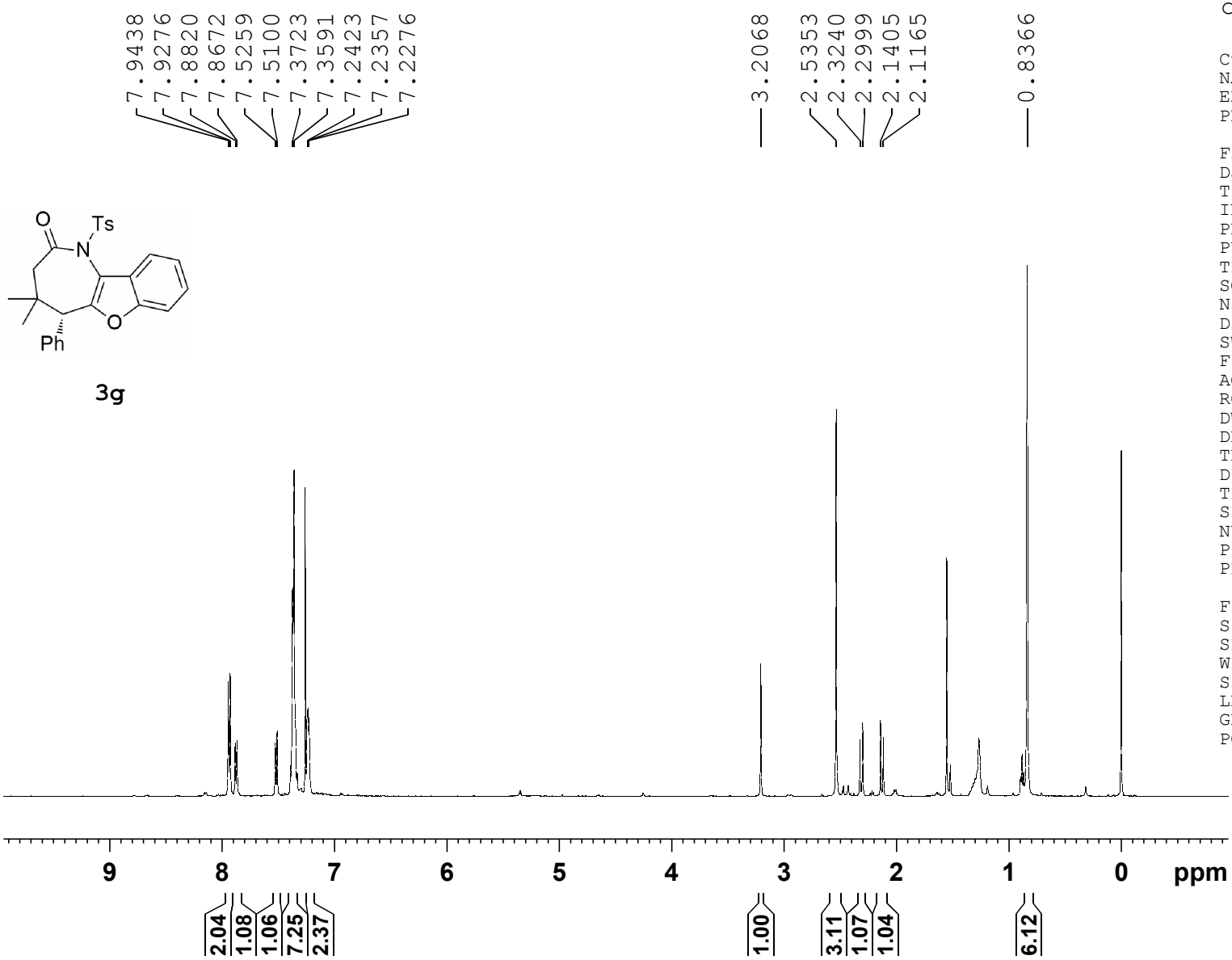
Current Data Parameters
NAME 500 M-1
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171214
Time 20.52
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 25
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W

======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W

F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



ckq-526

Current Data Parameters

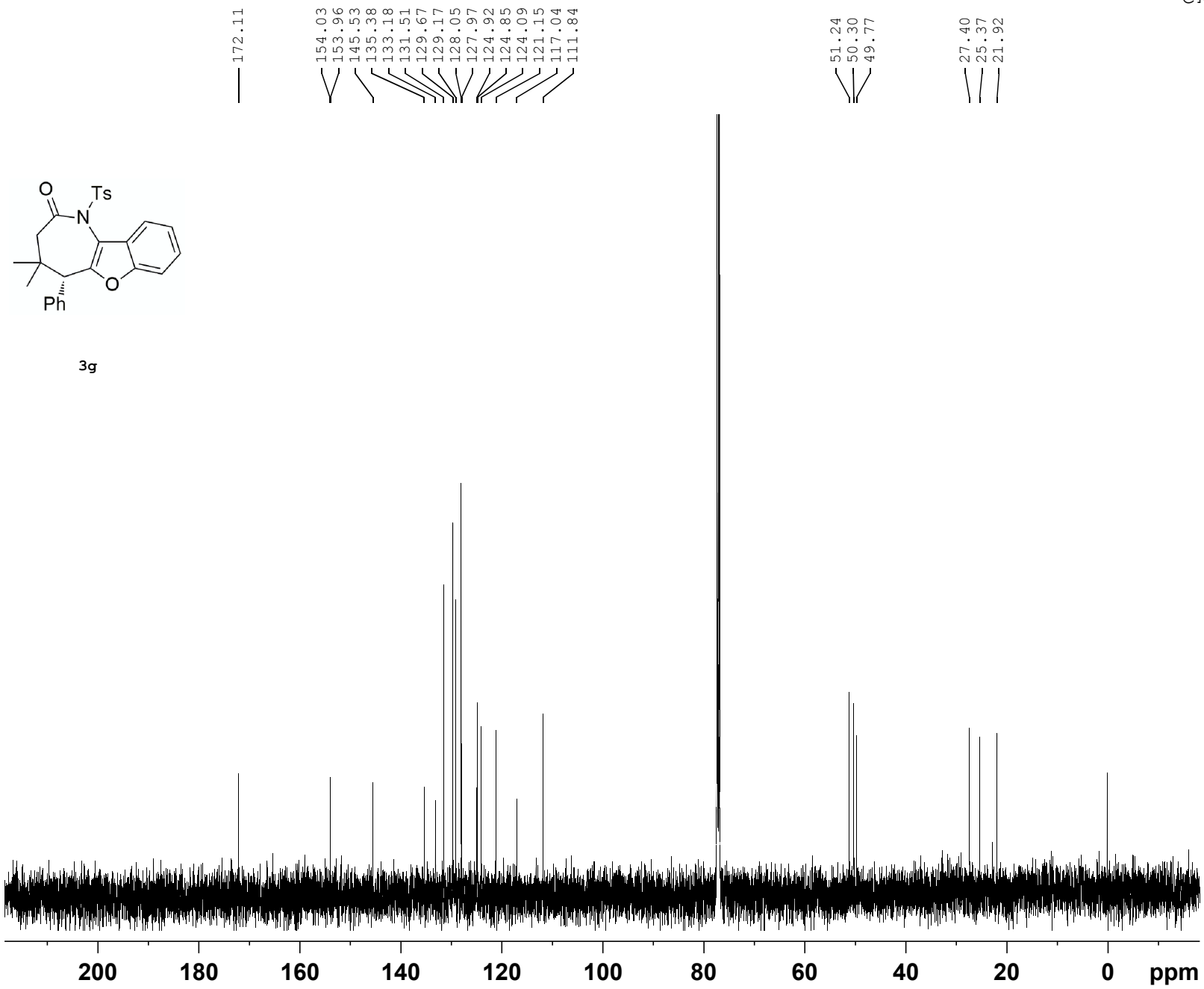
NAME	ckq
EXPNO	60
PROCNO	1

F2 - Acquisition Parameters

Date_	20160908
Time	8.21
INSTRUM	spect
PROBHD	5 mm CPPBBO BB
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2767999 sec
RG	55.37
DW	50.000 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
SFO1	500.1330885 MHz
NUC1	1H
P1	10.60 usec
PLW1	20.00000000 W

F2 - Processing parameters

SI	65536
SF	500.1300126 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



ckq-526

Current Data Parameters

NAME	ckq
EXPNO	61
PROCNO	1

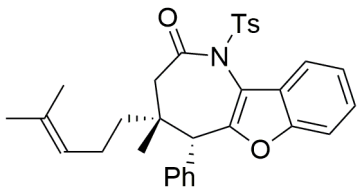
F2 - Acquisition Parameters

Date_	20160908
Time	8.24
INSTRUM	spect
PROBHD	5 mm CPPBBO BB
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	58
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010048 sec
RG	192.89
DW	16.800 usec
DE	18.00 usec
TE	298.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
SFO1	125.7703637 MHz
NUC1	13C
P1	9.80 usec
PLW1	57.00000000 W
SFO2	500.1320005 MHz
NUC2	1H
CPDPRG[2	waltz16
PCPD2	80.00 usec
PLW2	20.00000000 W
PLW12	0.35778001 W
PLW13	0.22898000 W

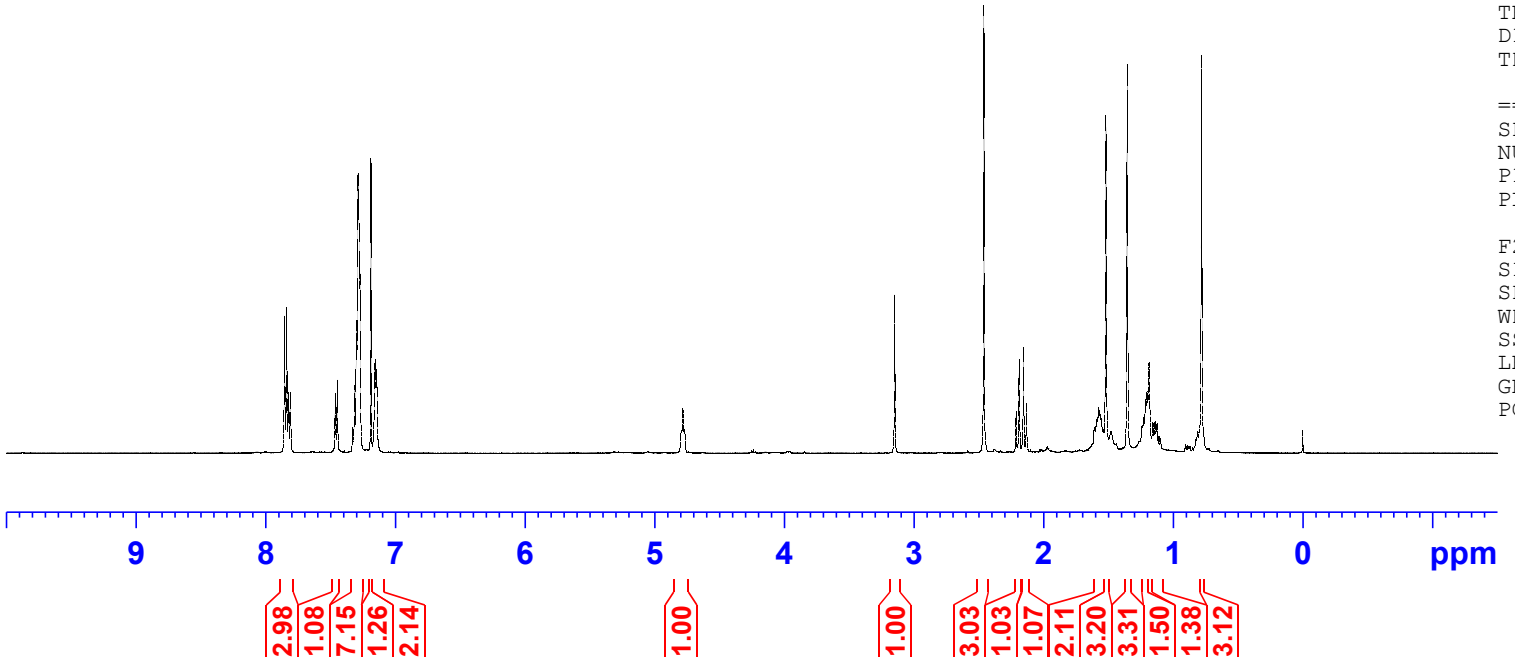
F2 - Processing parameters

SI	32768
SF	125.7577713 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

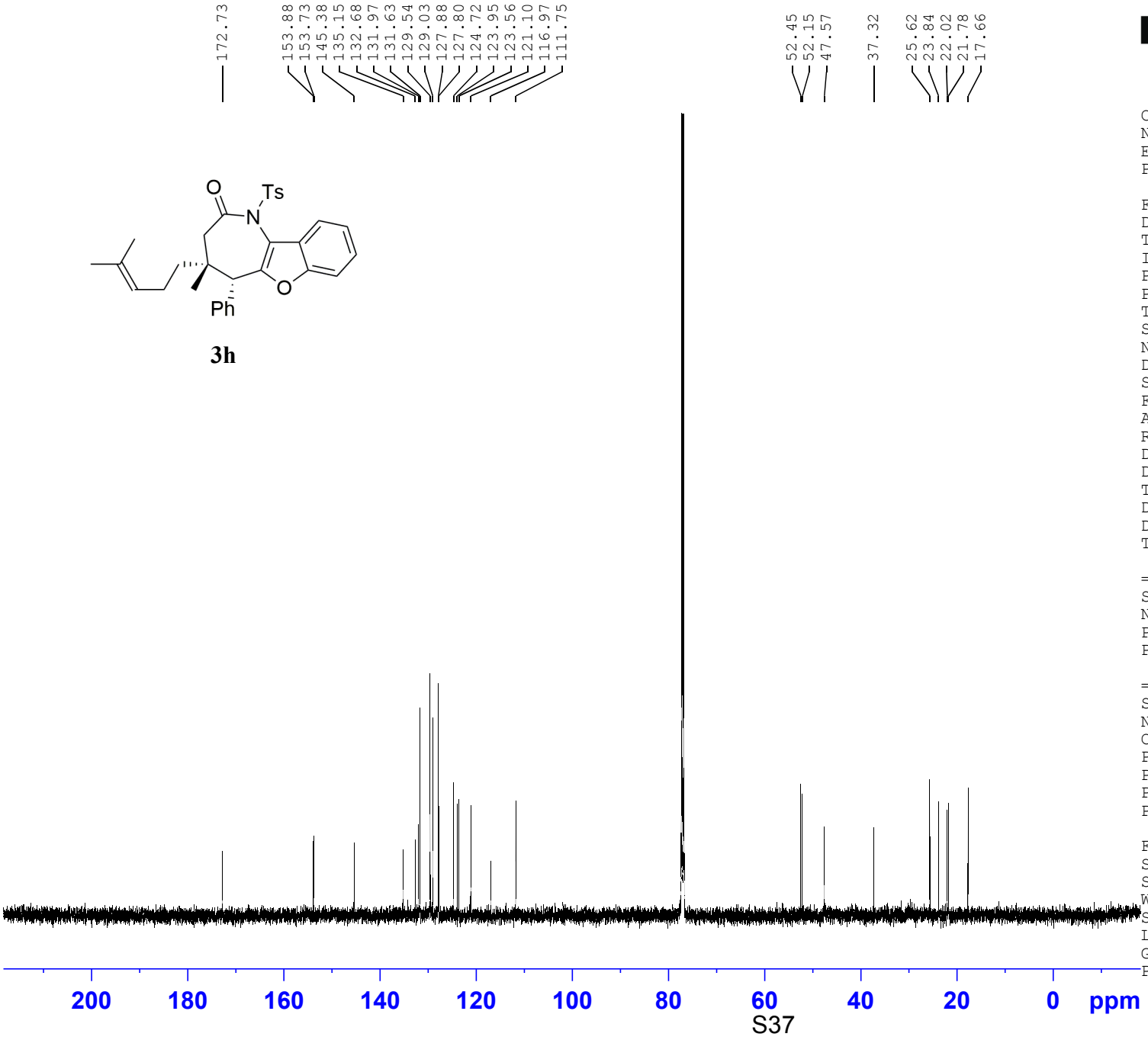
7.8542 7.8378 7.8248 7.8224 7.8083 7.4626 7.4477 7.3152 7.3133 7.3006 7.2976 7.2876 7.2819 7.2776 7.2717 7.1595 7.1521 7.1446 4.7944 4.7812 4.7680 3.1477 2.4596 2.2108 2.1871 2.1541 2.1306 1.5853 1.5737 1.5627 1.5527 1.5183 1.3531 1.2382 1.2276 1.2118 1.2005 1.1854 1.1576 1.1458 1.1353 1.1237 0.7808



3h




```
Current Data Parameters
NAME                  ckq
EXPNO                  37
PROCNO                  1

F2 - Acquisition Parameters
Date_            20180104
Time                 8.36
INSTRUM             spect
PROBHD   5 mm CPPBBO BB
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ             3.2767999 sec
RG                  31.72
DW                50.000 usec
DE                 6.50 usec
TE                298.1 K
D1           1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                   1H
P1                 11.50 usec
PLW1        20.00000000 W

F2 - Processing parameters
SI                  65536
SF           500.1300482 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



3h

845A

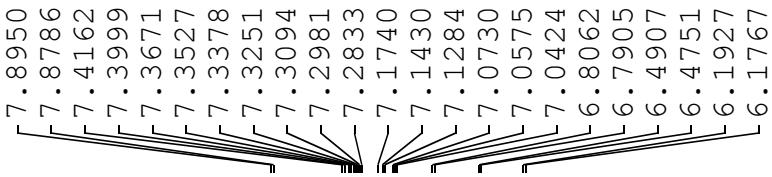
Current Data Parameters
NAME ckq
EXPNO 38
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180104
Time 8.40
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 148
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

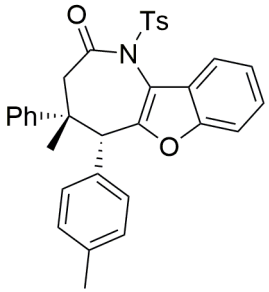
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W

======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W

F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

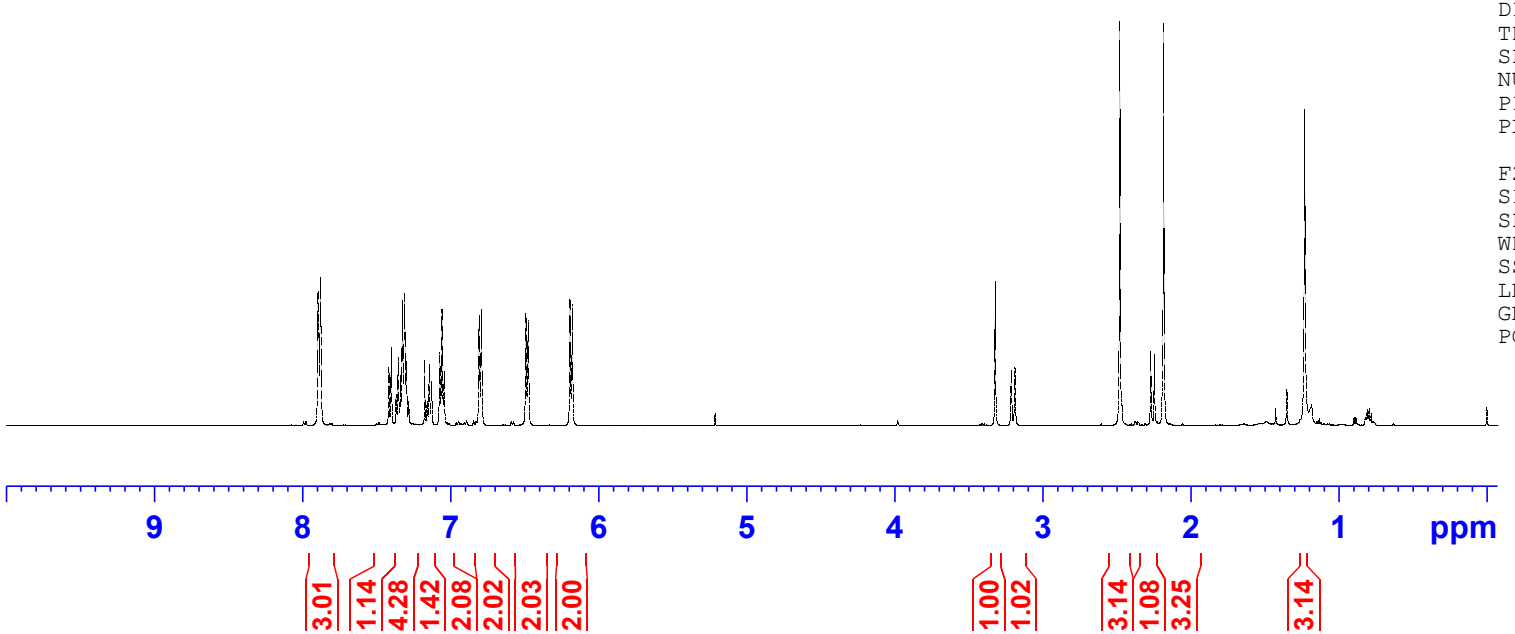


ckq-757

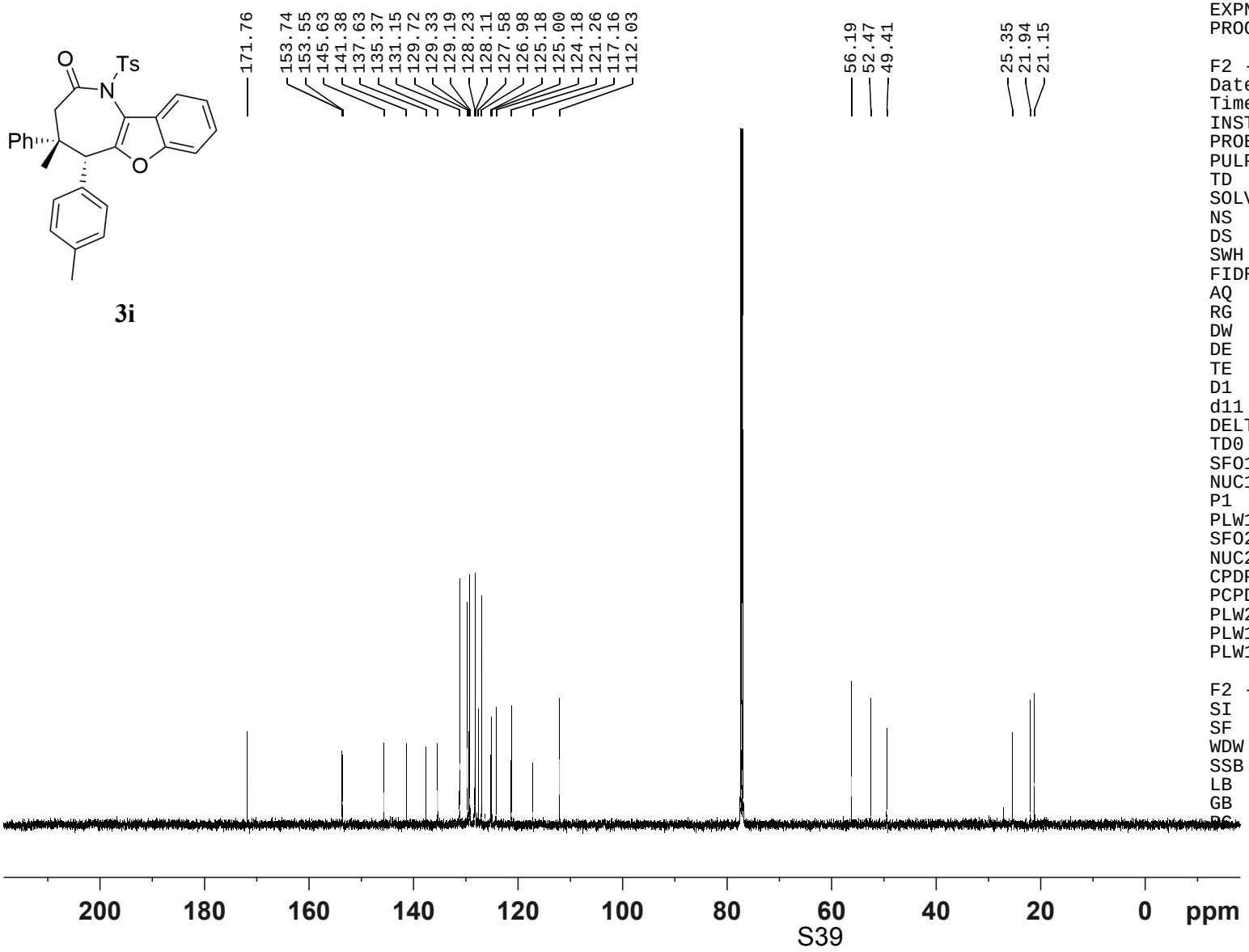


3i

```
Current Data Parameters
NAME                  CKQ
EXPNO                 102
PROCNO                  1

F2 - Acquisition Parameters
Date_            20171209
Time                16.17 h
INSTRUM              spect
PROBHD   5 mm CPPBBO BB
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                  31.72
DW                 50.000 usec
DE                  6.50 usec
TE                 298.1 K
D1            1.00000000 sec
TD0                     1
SFO1         500.1330885 MHz
NUC1                   1H
P1                 11.50 usec
PLW1         20.00000000 W

F2 - Processing parameters
SI                  65536
SF           500.1300551 MHz
WDW                    EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

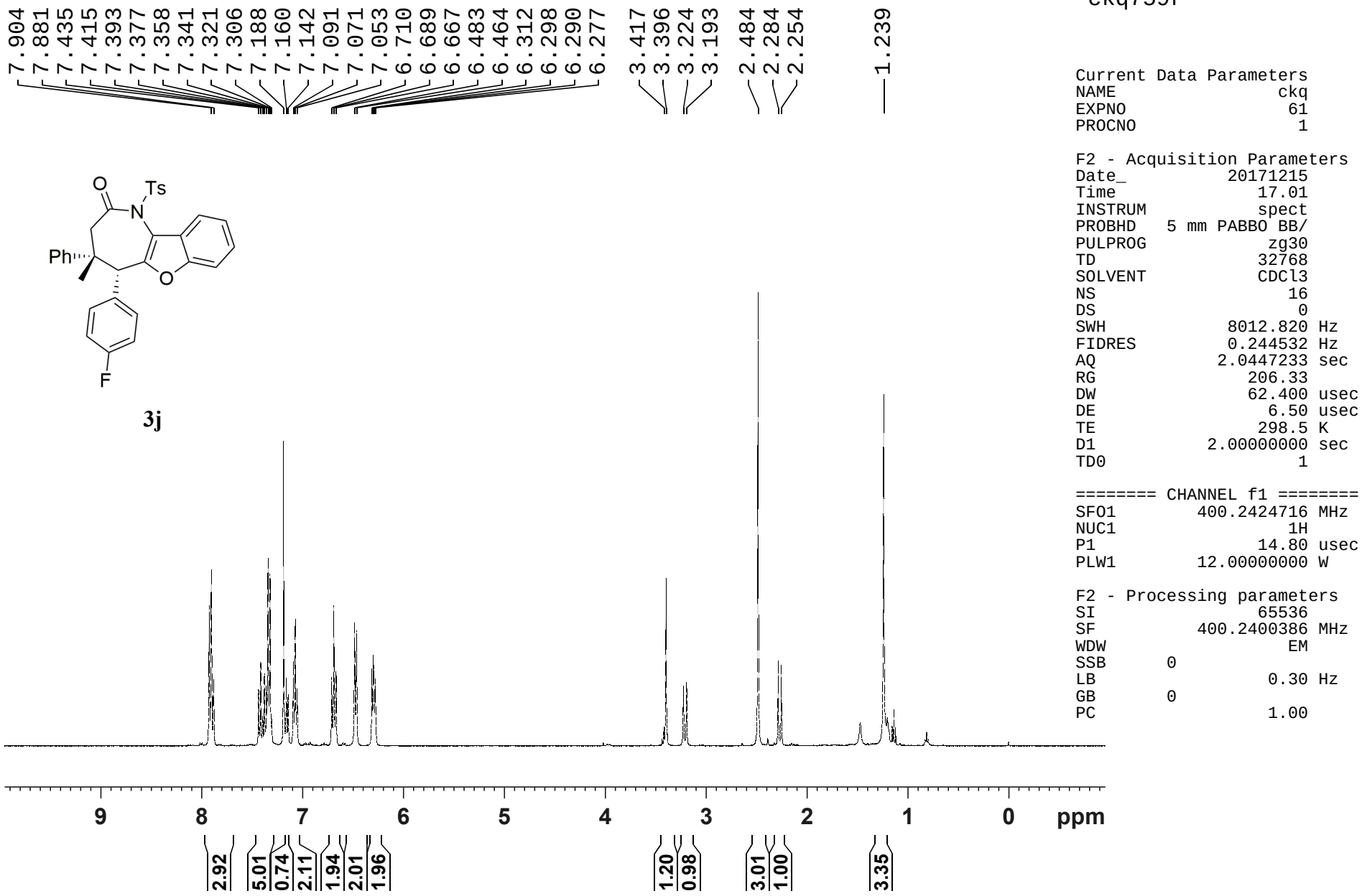


ckq-757



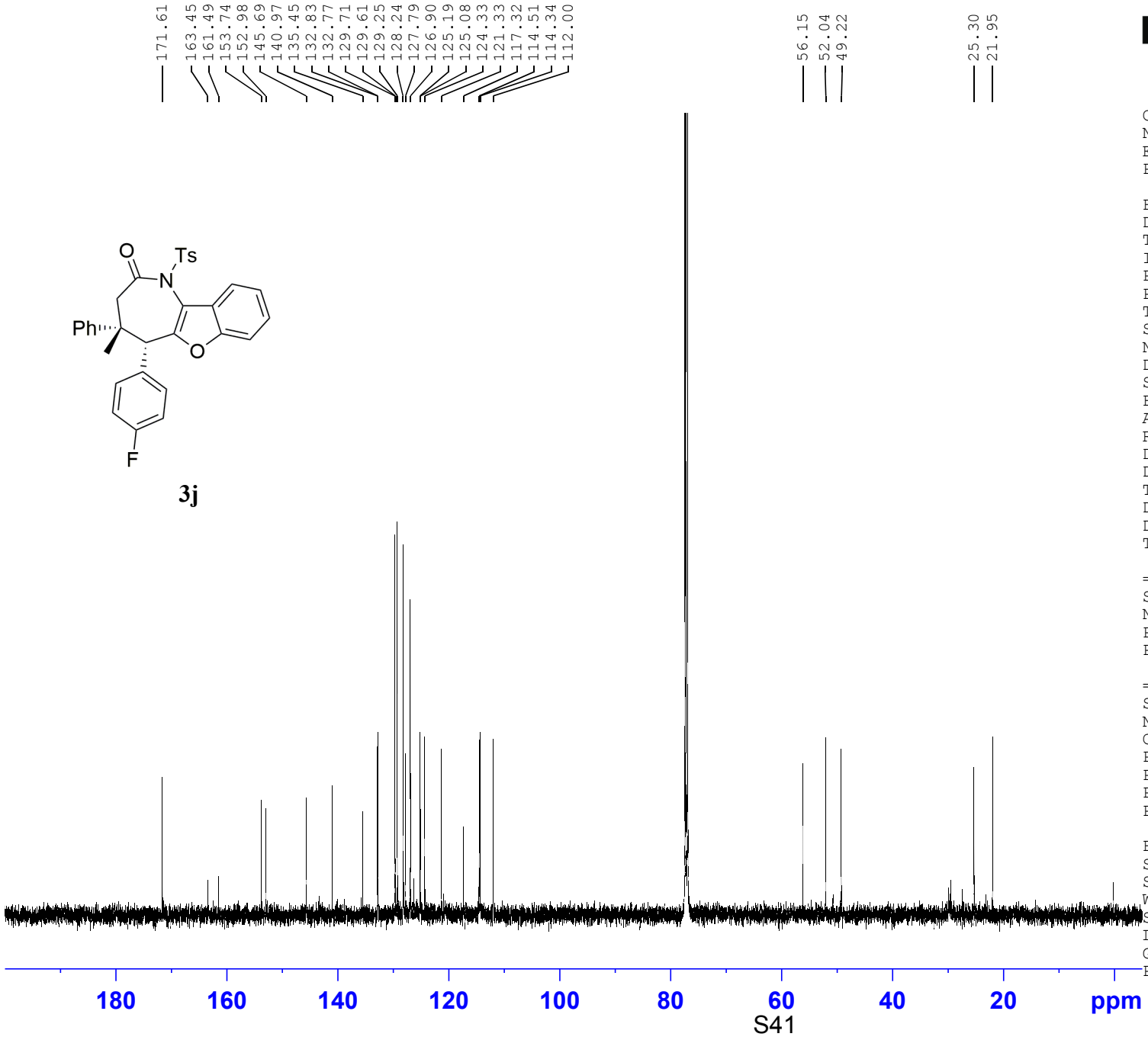
3i

```
Current Data Parameters
NAME                  ckq
EXPNO                 103
PROCNO                  1

F2 - Acquisition Parameters
Date_            20171209
Time                16.19 h
INSTRUM             spect
PROBHD   5 mm CPPBBO BB
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    30
DS                     4
SWH             29761.904 Hz
FIDRES           0.908261 Hz
AQ              1.1010048 sec
RG                 192.89
DW                 16.800 usec
DE                 18.00 usec
TE                 298.1 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                    1
SFO1          125.7703637 MHz
NUC1                  13C
P1                  9.80 usec
PLW1           57.00000000 W
SFO2          500.1320005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2           20.00000000 W
PLW12           0.35778001 W
PLW13           0.22898000 W

F2 - Processing parameters
SI                 32768
SF            125.7577752 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



ckq759r

```
Current Data Parameters
NAME                  ckq
EXPNO                  61
PROCNO                  1

F2 - Acquisition Parameters
Date_            20171215
Time                17.01
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              8012.820 Hz
FIDRES           0.244532 Hz
AQ              2.0447233 sec
RG                 206.33
DW                 62.400 usec
DE                  6.50 usec
TE                 298.5 K
D1            2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         400.2424716 MHz
NUC1                   1H
P1                  14.80 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                  65536
SF           400.2400386 MHz
WDW                    EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```



ckq-759

Current Data Parameters
NAME CKQ
EXPNO 99
PROCNO 1

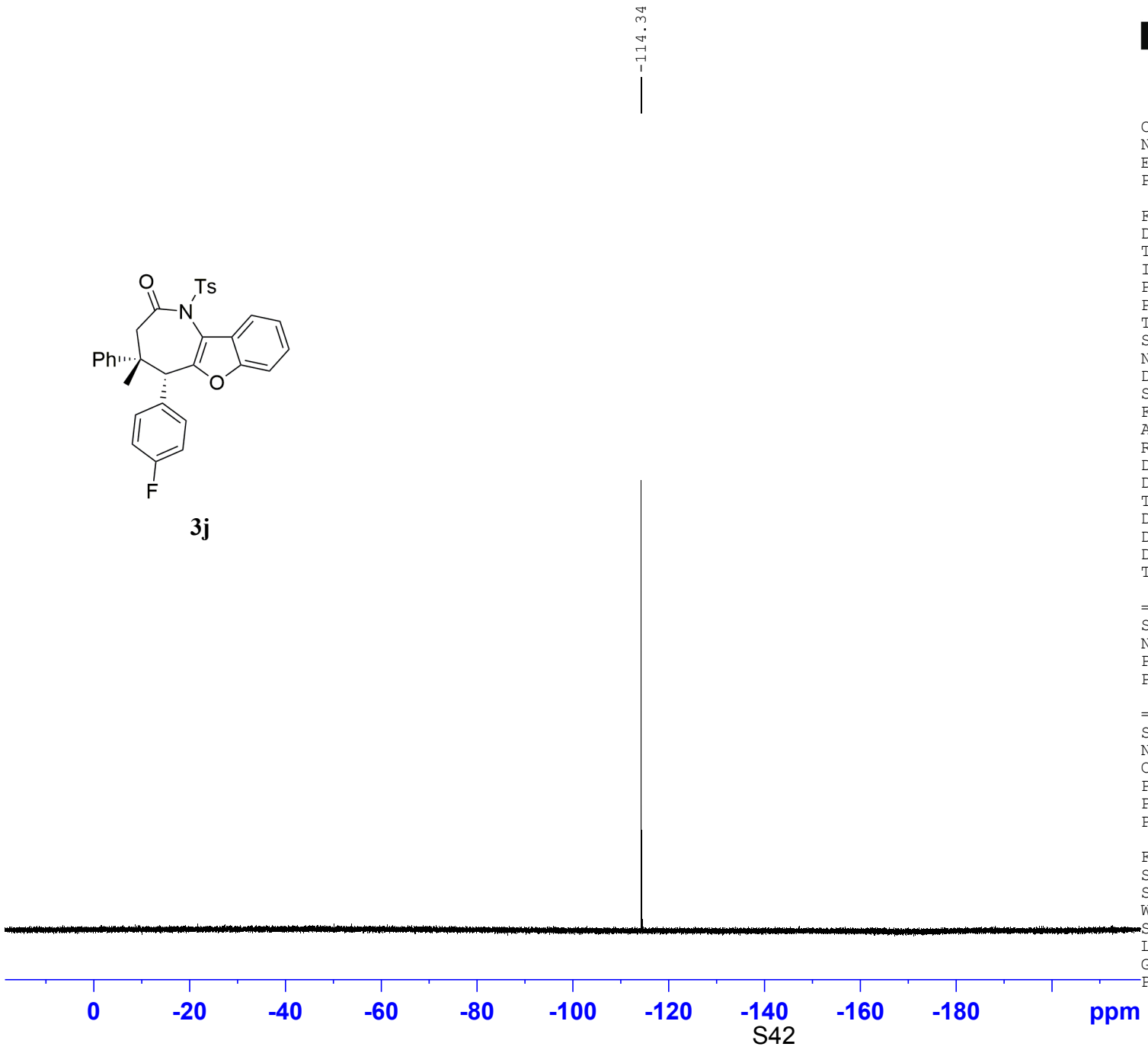
F2 - Acquisition Parameters
Date_ 20180607
Time 21.44
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 98
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W

======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W

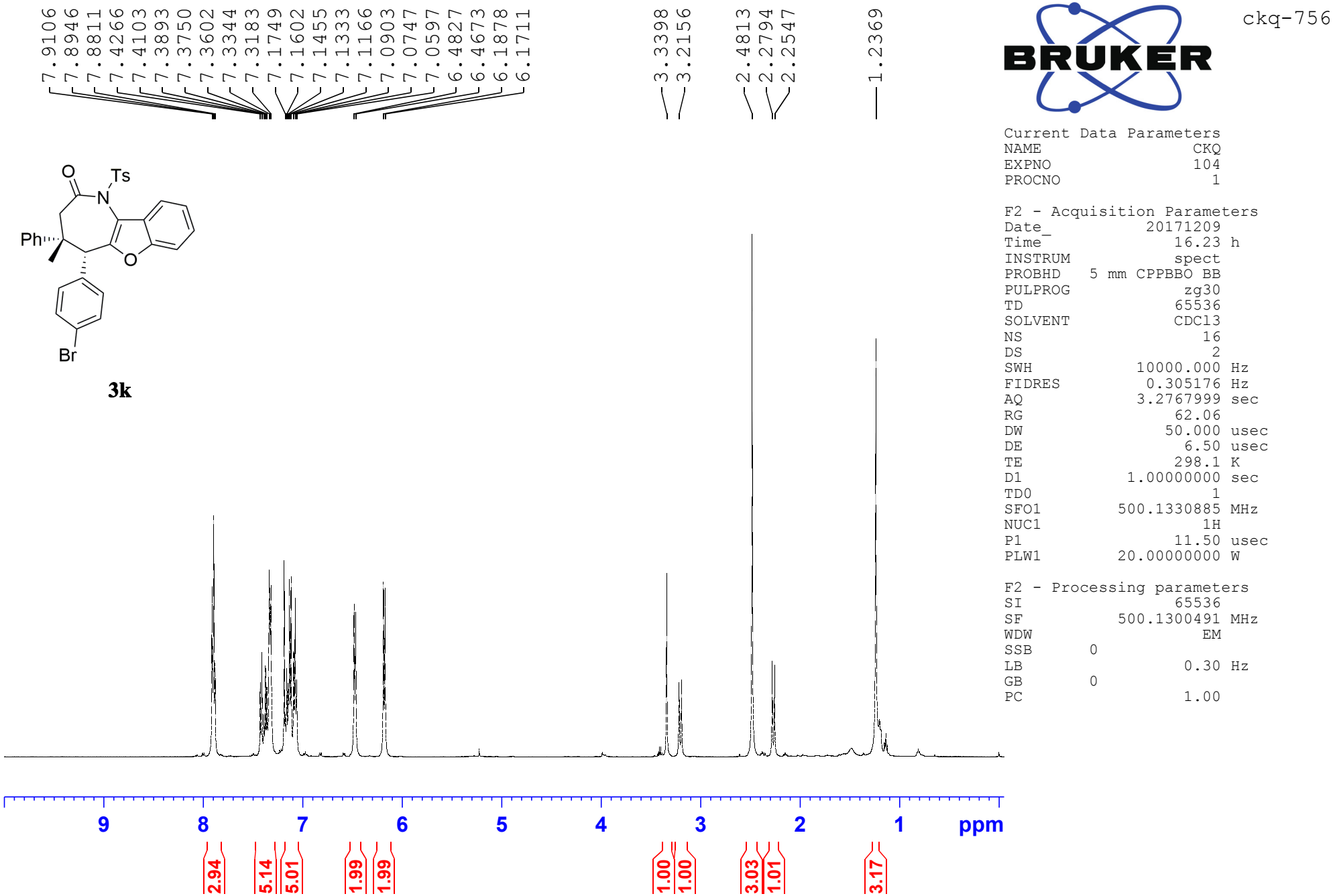
F2 - Processing parameters
SI 32768
SF 125.7577729 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

3j



CKQ-759 4-F

```
Current Data Parameters
NAME                  19F
EXPNO                   7
PROCNO                  1

F2 - Acquisition Parameters
Date_            20180105
Time                23.29
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG       zgfhigqn.2
TD                 131072
SOLVENT             CDCl3
NS                     16
DS                      4
SWH            89285.711 Hz
FIDRES          0.681196 Hz
AQ             0.7340032 sec
RG                206.33
DW                 5.600 usec
DE                 6.50 usec
TE                298.1 K
D1            1.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         376.5642094 MHz
NUC1                19F
P1                14.50 usec
PLW1         17.98900032 W

======== CHANNEL f2 ========
SFO2         400.2416010 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         12.00000000 W
PLW12         0.34680000 W

F2 - Processing parameters
SI                 65536
SF           376.6018696 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



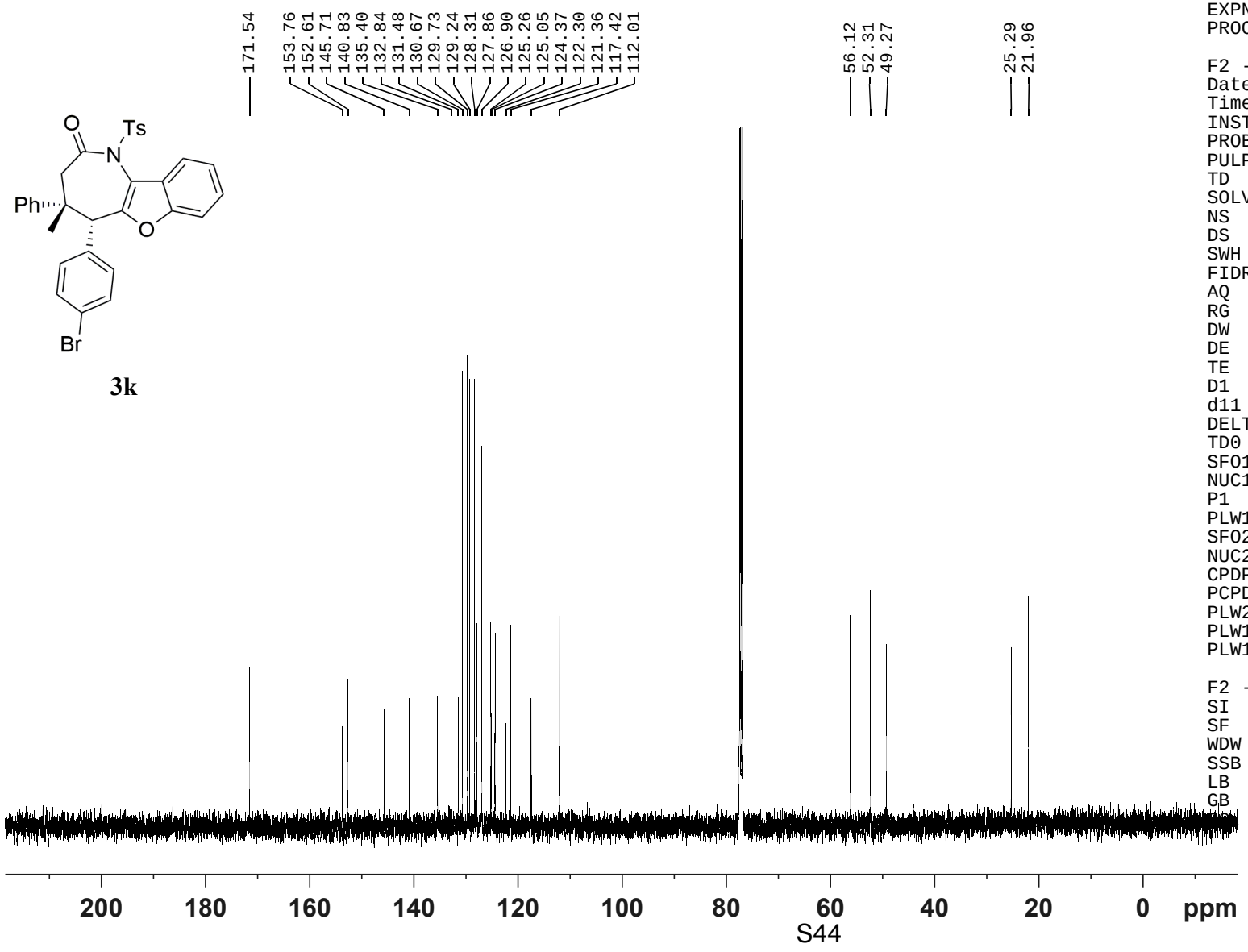
ckq-756

Current Data Parameters
NAME CKQ
EXPNO 104
PROCNO 1

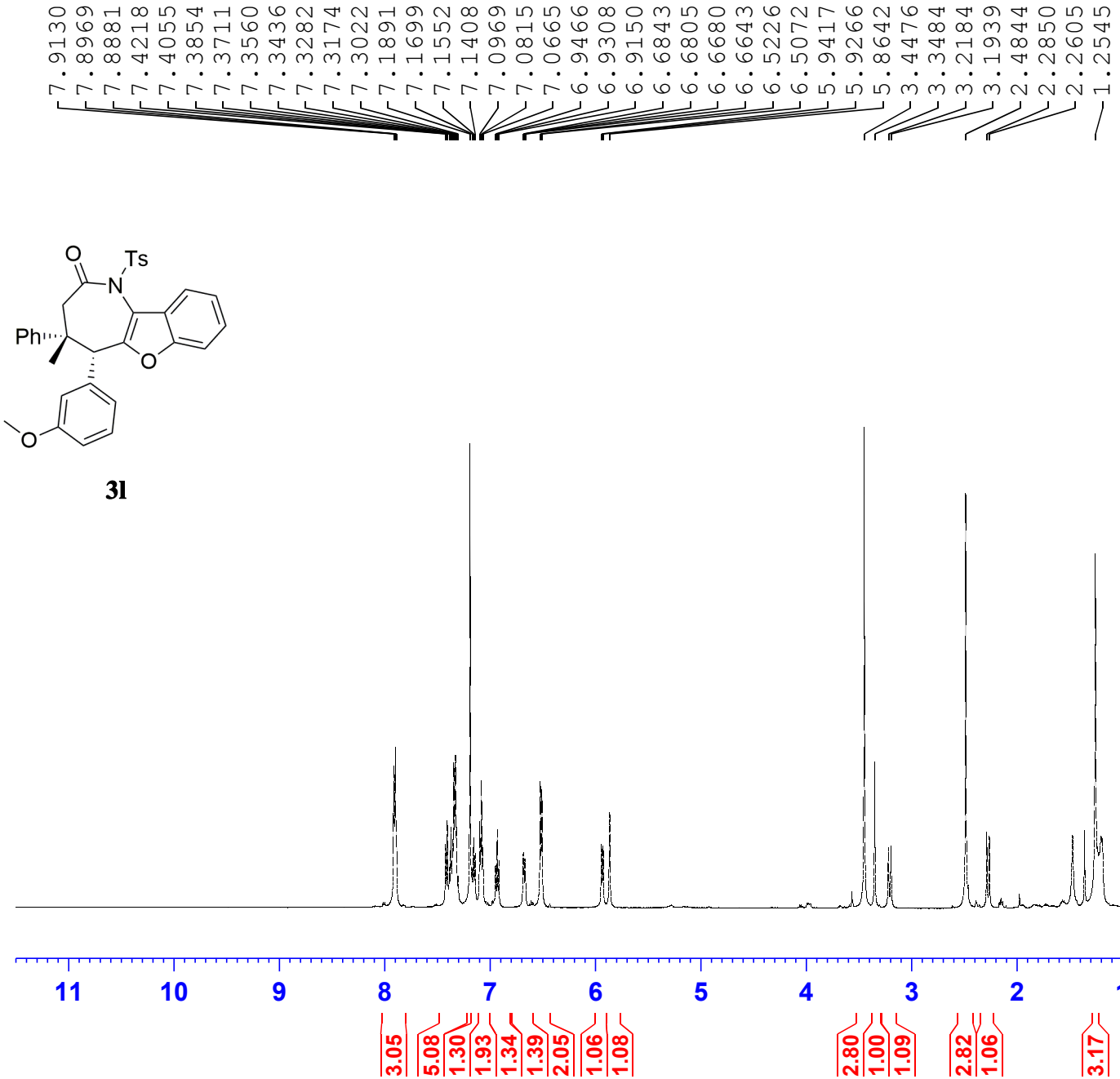
F2 - Acquisition Parameters
Date_ 20171209
Time 16.23 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 62.06
DW 50.000 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 11.50 usec
PLW1 20.00000000 W

F2 - Processing parameters
SI 65536
SF 500.1300491 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

ckq-756




```
Current Data Parameters
NAME                  ckq
EXPNO                 105
PROCNO                  1

F2 - Acquisition Parameters
Date_            20171209
Time                16.26 h
INSTRUM             spect
PROBHD   5 mm CPPBBO BB
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   101
DS                     4
SWH            29761.904 Hz
FIDRES          0.908261 Hz
AQ             1.1010048 sec
RG                192.89
DW                16.800 usec
DE                18.00 usec
TE                298.1 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1
SFO1         125.7703637 MHz
NUC1                 13C
P1                  9.80 usec
PLW1          57.00000000 W
SFO2         500.1320005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2               80.00 usec
PLW2          20.00000000 W
PLW12          0.35778001 W
PLW13          0.22898000 W

F2 - Processing parameters
SI                  32768
SF           125.7577727 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
                    1.40
```



3l

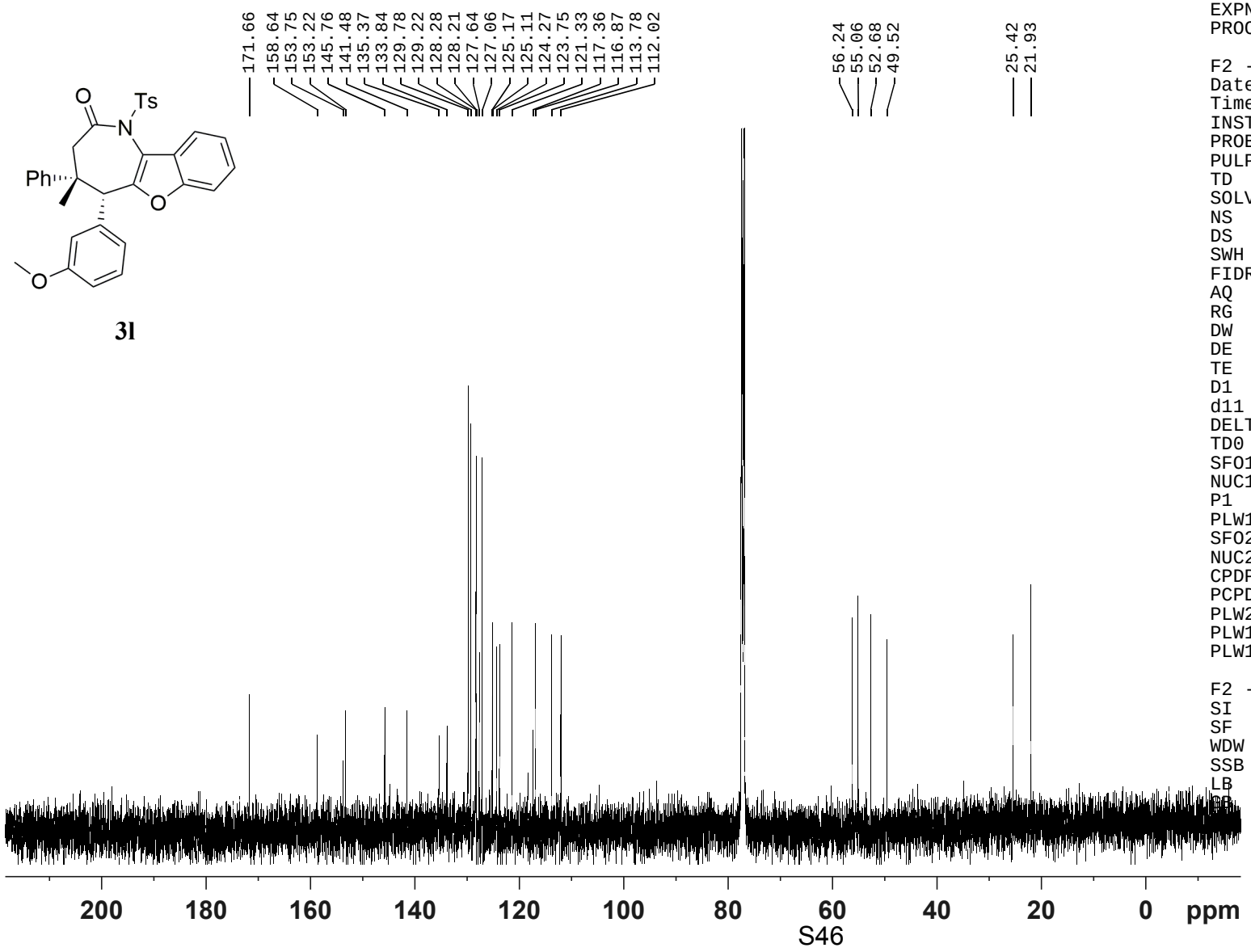
ckq-761

Current Data Parameters
NAME CKQ
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20171216
Time 14.39 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 31.72
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 11.50 usec
PLW1 20.00000000 W

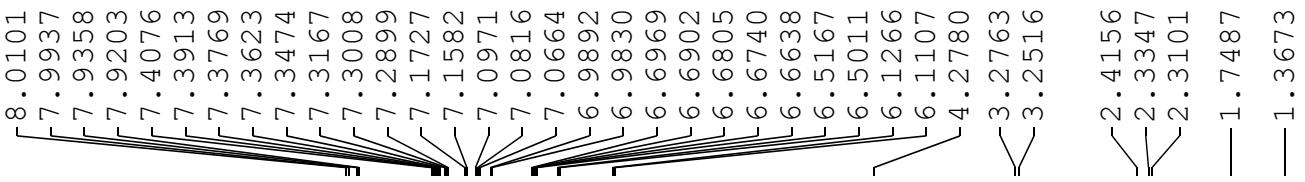
F2 - Processing parameters
SI 65536
SF 500.1300476 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

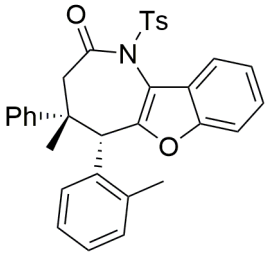
ckq-761r



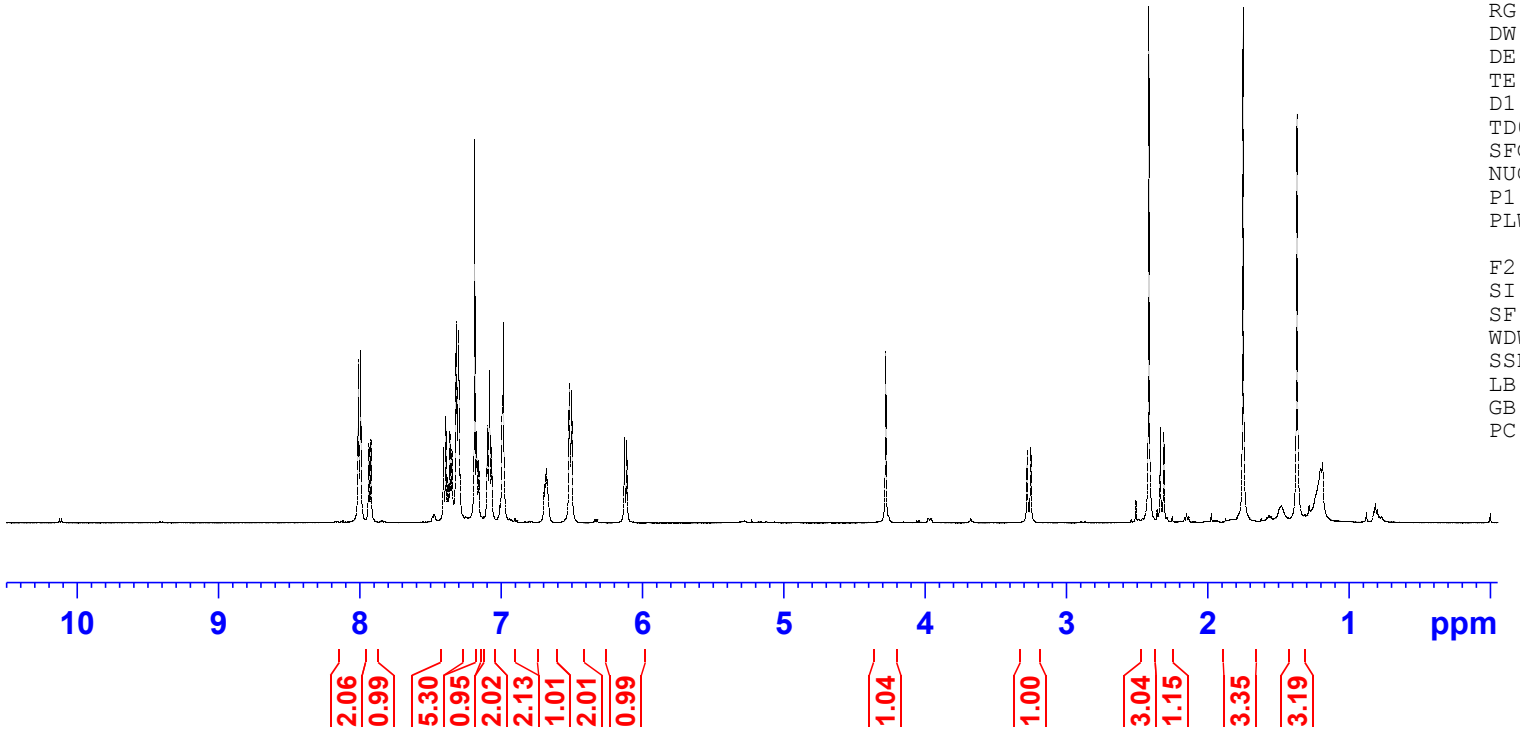
3l

```
Current Data Parameters
NAME                  ckq
EXPNO                  21
PROCNO                  1

F2 - Acquisition Parameters
Date_            20171216
Time                14.41 h
INSTRUM             spect
PROBHD   5 mm CPPBBO BB
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    200
DS                      4
SWH             29761.904 Hz
FIDRES           0.908261 Hz
AQ              1.1010048 sec
RG                 192.89
DW                 16.800 usec
DE                 18.00 usec
TE                 298.1 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                     1
SFO1          125.7703637 MHz
NUC1                  13C
P1                  9.80 usec
PLW1           57.00000000 W
SFO2          500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2           20.00000000 W
PLW12           0.35778001 W
PLW13           0.22898000 W

F2 - Processing parameters
SI                  32768
SF            125.7577716 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
                    1.40
```





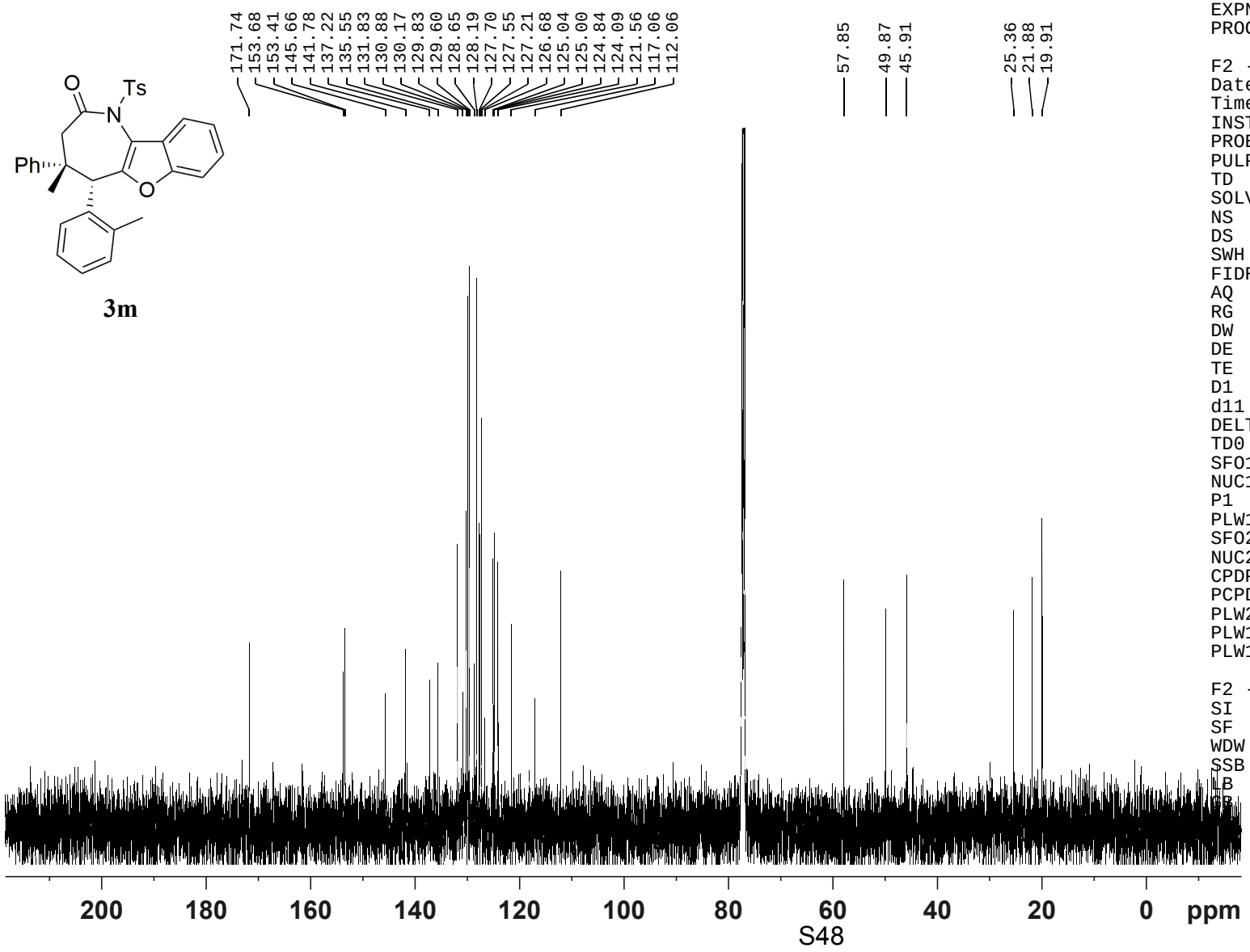
3m



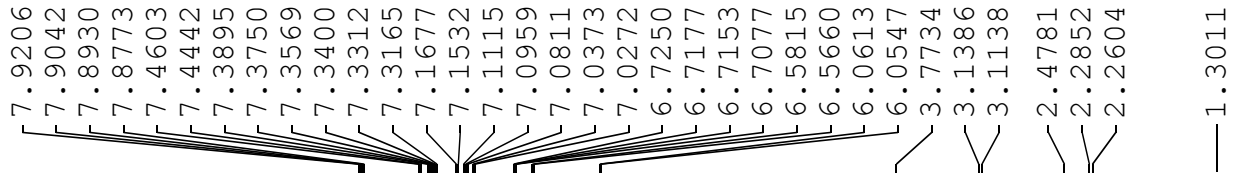
ckq-760

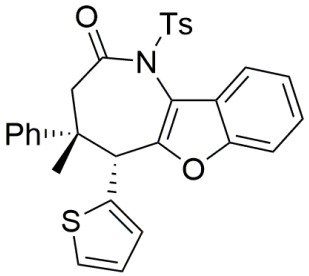
```
Current Data Parameters
NAME                  CKQ
EXPNO                  19
PROCNO                  1

F2 - Acquisition Parameters
Date_            20171216
Time                14.36 h
INSTRUM              spect
PROBHD   5 mm CPPBBO BB
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                  31.72
DW                 50.000 usec
DE                  6.50 usec
TE                 298.2 K
D1            1.00000000 sec
TD0                     1
SFO1         500.1330885 MHz
NUC1                   1H
P1                 11.50 usec
PLW1         20.00000000 W

F2 - Processing parameters
SI                  65536
SF           500.1300489 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

ckq-760




```
Current Data Parameters
NAME                  ckq
EXPNO                  18
PROCNO                  1

F2 - Acquisition Parameters
Date_            20171215
Time                8.43 h
INSTRUM             spect
PROBHD   5 mm CPPBBO BB
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    141
DS                      4
SWH             29761.904 Hz
FIDRES           0.908261 Hz
AQ              1.1010048 sec
RG                 192.89
DW                 16.800 usec
DE                 18.00 usec
TE                 298.1 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                     1
SFO1          125.7703637 MHz
NUC1                  13C
P1                  9.80 usec
PLW1           57.00000000 W
SFO2          500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2           20.00000000 W
PLW12           0.35778001 W
PLW13           0.22898000 W

F2 - Processing parameters
SI                  32768
SF            125.7577717 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
                    1.40
```

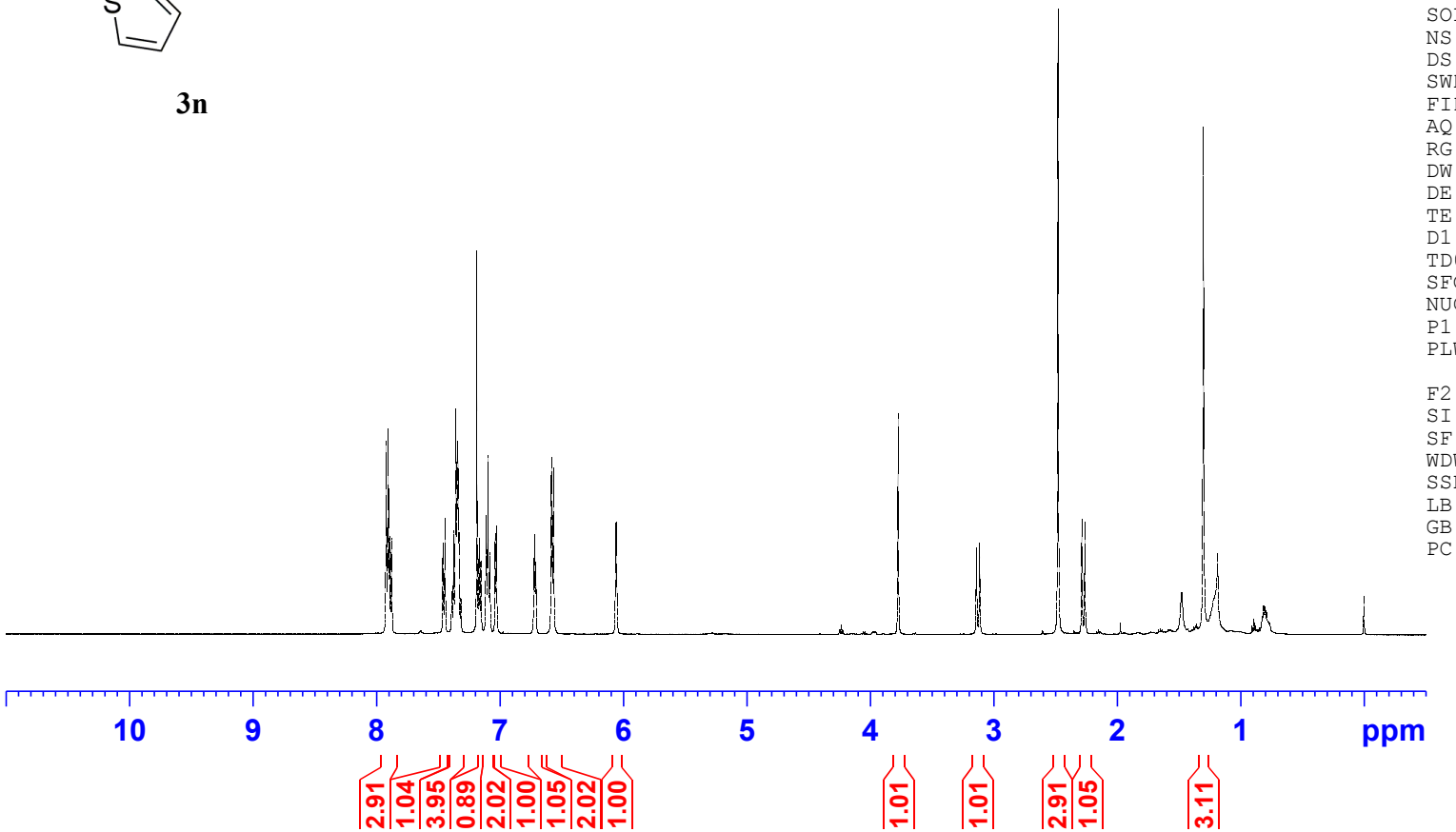


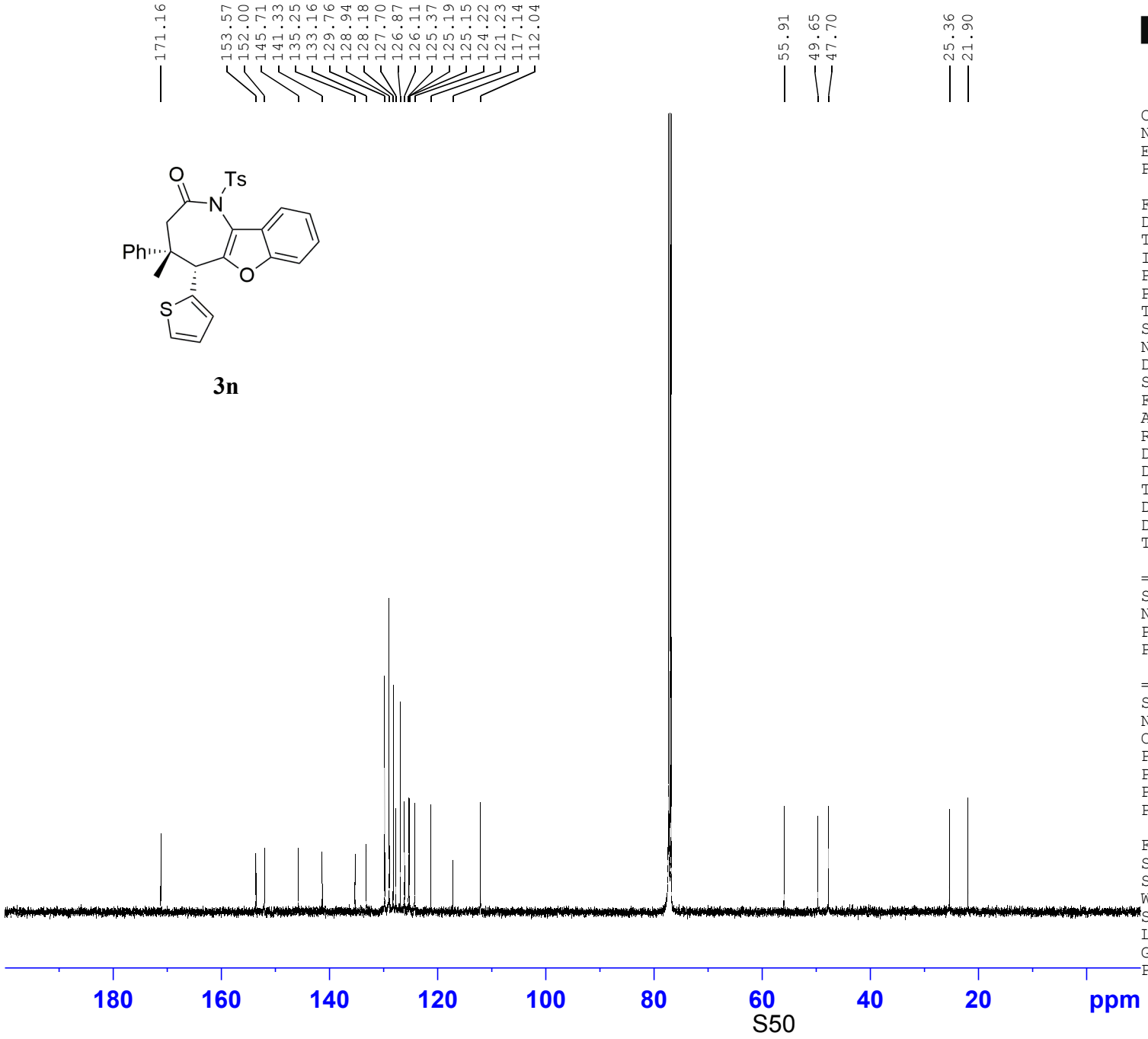


3n

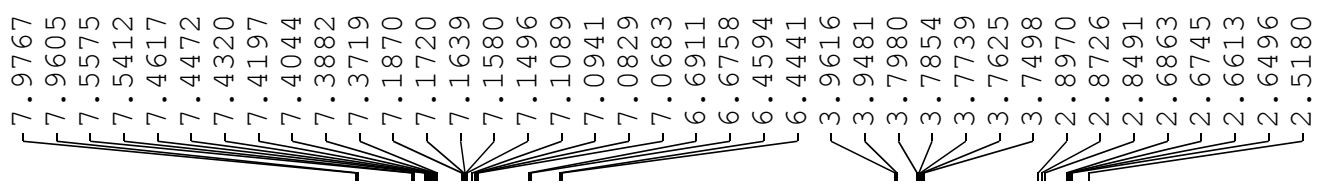
841E

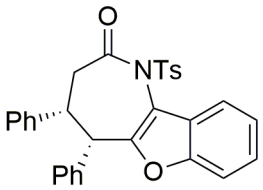
```
Current Data Parameters
NAME                  ckq
EXPNO                  17
PROCNO                  1

F2 - Acquisition Parameters
Date_           20180307
Time                17.26 h
INSTRUM             spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                 31.72
DW                50.000 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1
SFO1         500.1330885 MHz
NUC1                  1H
P1                 11.50 usec
PLW1         20.00000000 W

F2 - Processing parameters
SI                 65536
SF           500.1300482 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



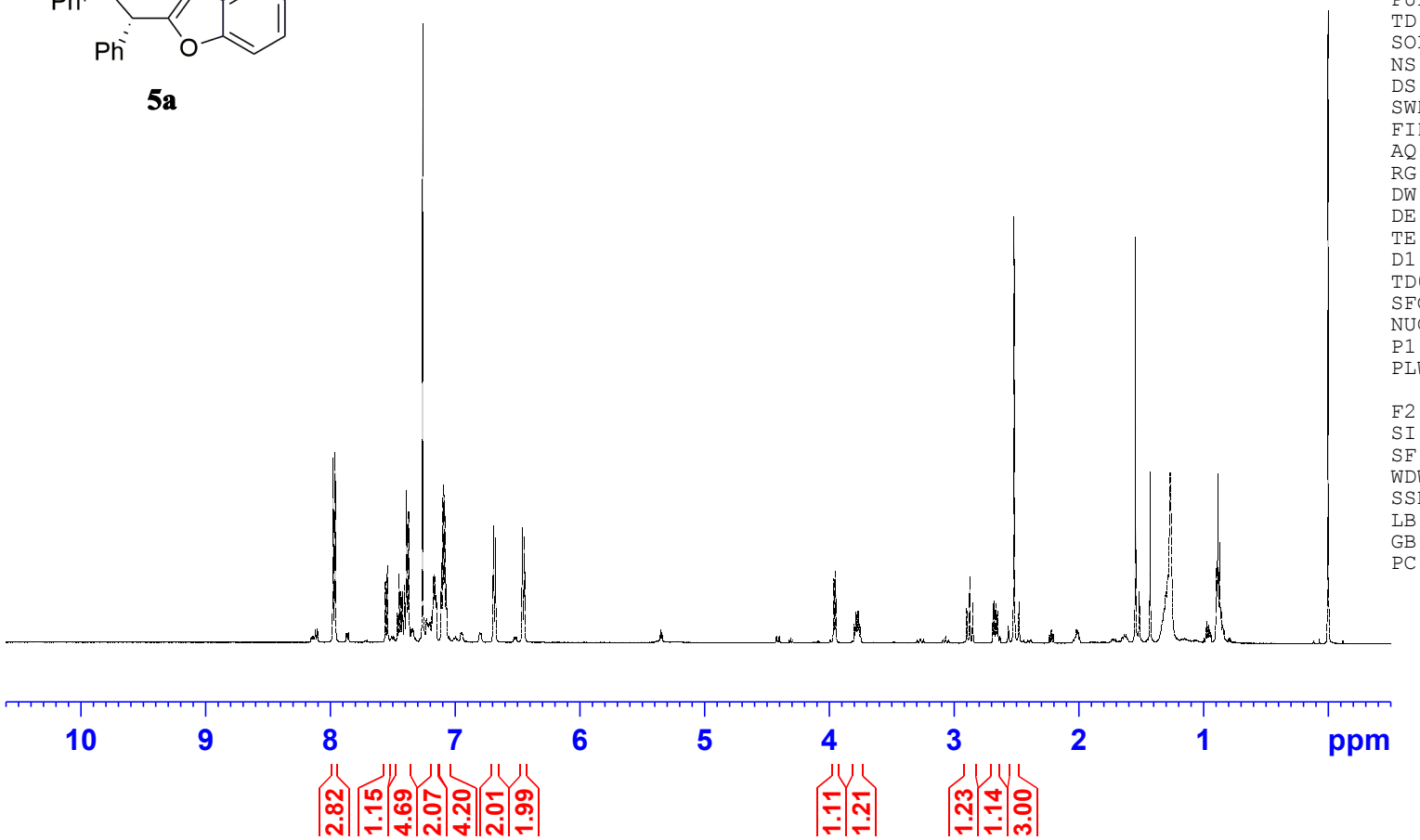



```
Current Data Parameters
NAME                  ckq
EXPNO                  40
PROCNO                  1

F2 - Acquisition Parameters
Date_            20180105
Time                15.31
INSTRUM             spect
PROBHD   5 mm CPPBBO BB
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    112
DS                      4
SWH            29761.904 Hz
FIDRES          0.454131 Hz
AQ             1.1010048 sec
RG                 192.89
DW                 16.800 usec
DE                 18.00 usec
TE                 298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                  13C
P1                  9.80 usec
PLW1         57.00000000 W

======== CHANNEL f2 ========
SFO2         500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2         20.00000000 W
PLW12         0.35778001 W
PLW13         0.22898000 W

F2 - Processing parameters
SI                  32768
SF           125.7577885 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```





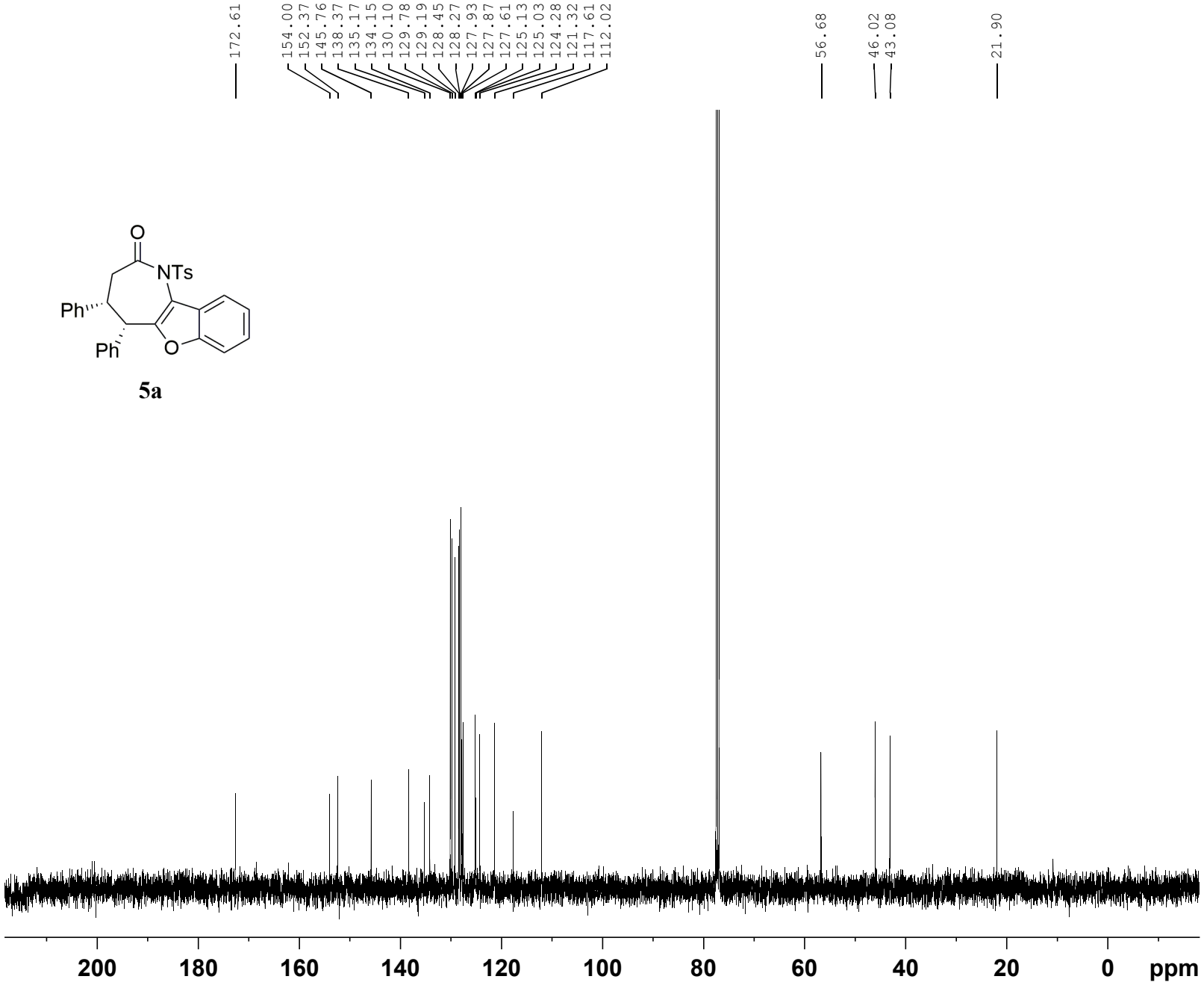
5a



ckq-539

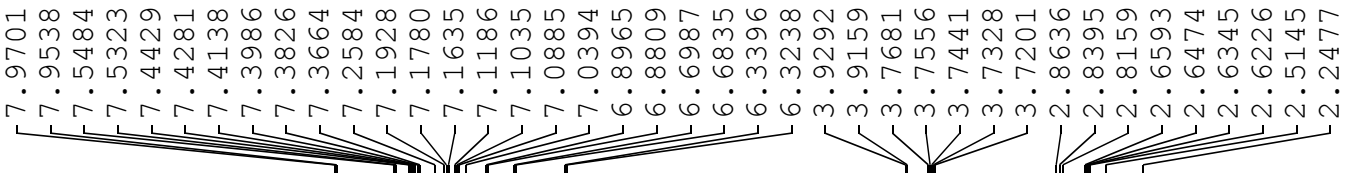
```
Current Data Parameters
NAME                  CKQ
EXPNO                  62
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160922
Time                8.19 h
INSTRUM             spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                 31.72
DW                50.000 usec
DE                 6.50 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1
SFO1        500.1330885 MHz
NUC1                  1H
P1                10.60 usec
PLW1        20.00000000 W

F2 - Processing parameters
SI                 65536
SF          500.1300131 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

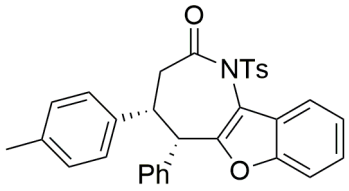
ckq-539



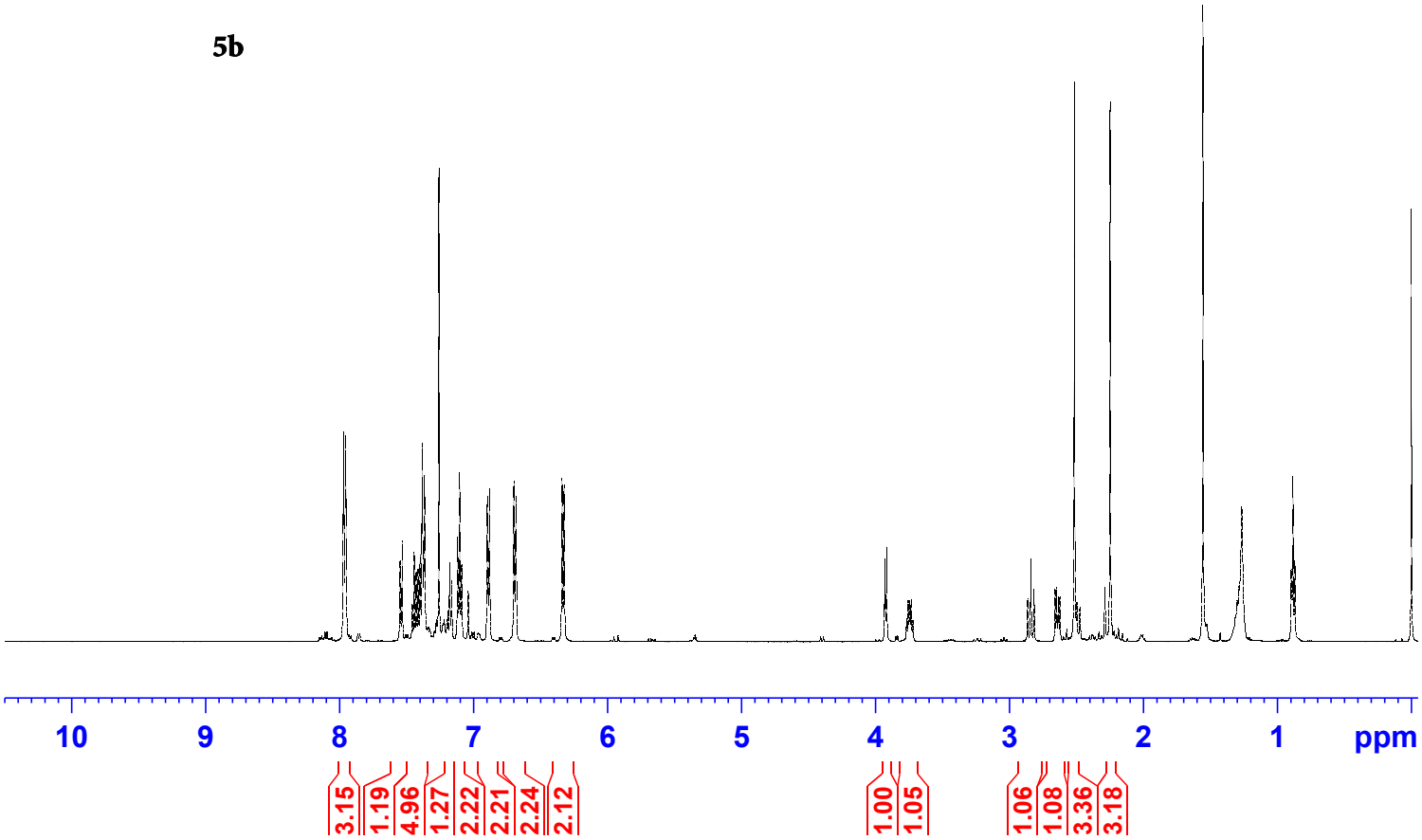
5a

```
Current Data Parameters
NAME                  ckq
EXPNO                  54
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160922
Time                21.00
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     43
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631488 sec
RG                 206.33
DW                 20.800 usec
DE                  6.50 usec
TE                 299.2 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                     1
SFO1          100.6504916 MHz
NUC1                  13C
P1                 10.00 usec
PLW1           54.00000000 W
SFO2          400.2416010 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2           12.00000000 W
PLW12           0.34680000 W
PLW13           0.28090999 W

F2 - Processing parameters
SI                  32768
SF            100.6404168 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



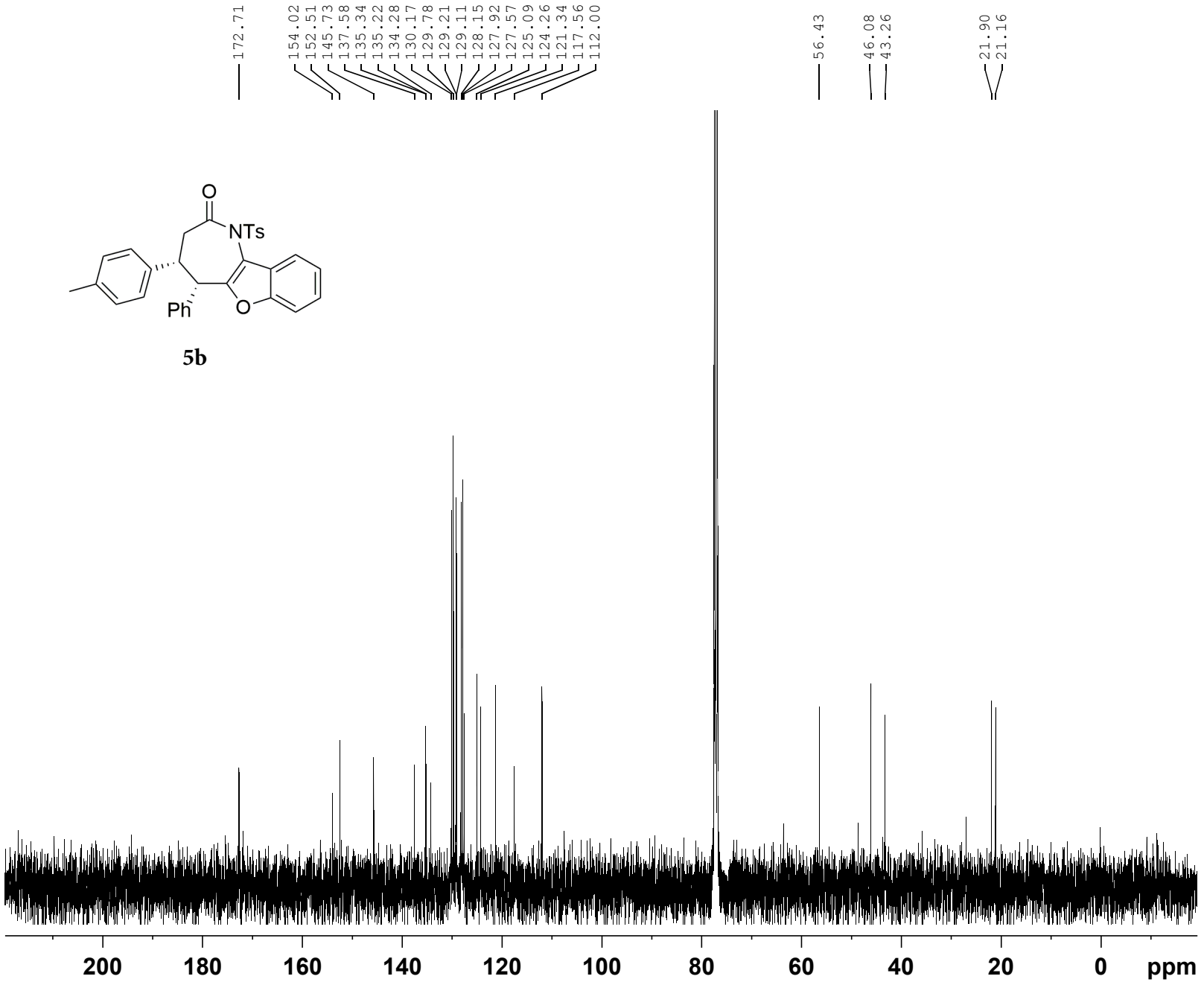


5b



ckq-540

```
Current Data Parameters
NAME                  CKQ
EXPNO                  65
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160924
Time                10.52 h
INSTRUM              spect
PROBHD   5 mm CPPBBO BB
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                  62.06
DW                50.000 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                     1
SFO1         500.1330885 MHz
NUC1                   1H
P1                10.60 usec
PLW1        20.00000000 W

F2 - Processing parameters
SI                  65536
SF           500.1300131 MHz
WDW                    EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



5b

ckq-540

Current Data Parameters

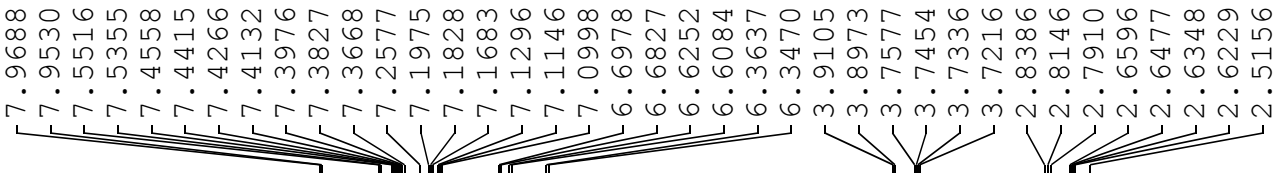
NAME	ckq
EXPNO	65
PROCNO	1

F2 - Acquisition Parameters

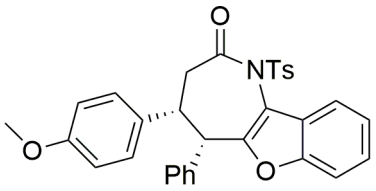
Date_	20160926
Time	14.36
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	272
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631488 sec
RG	206.33
DW	20.800 usec
DE	6.50 usec
TE	301.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
SFO1	100.6504916 MHz
NUC1	13C
P1	10.00 usec
PLW1	54.00000000 W
SFO2	400.2416010 MHz
NUC2	1H
CPDPRG[2	waltz16
PCPD2	90.00 usec
PLW2	12.00000000 W
PLW12	0.34680000 W
PLW13	0.28090999 W

F2 - Processing parameters

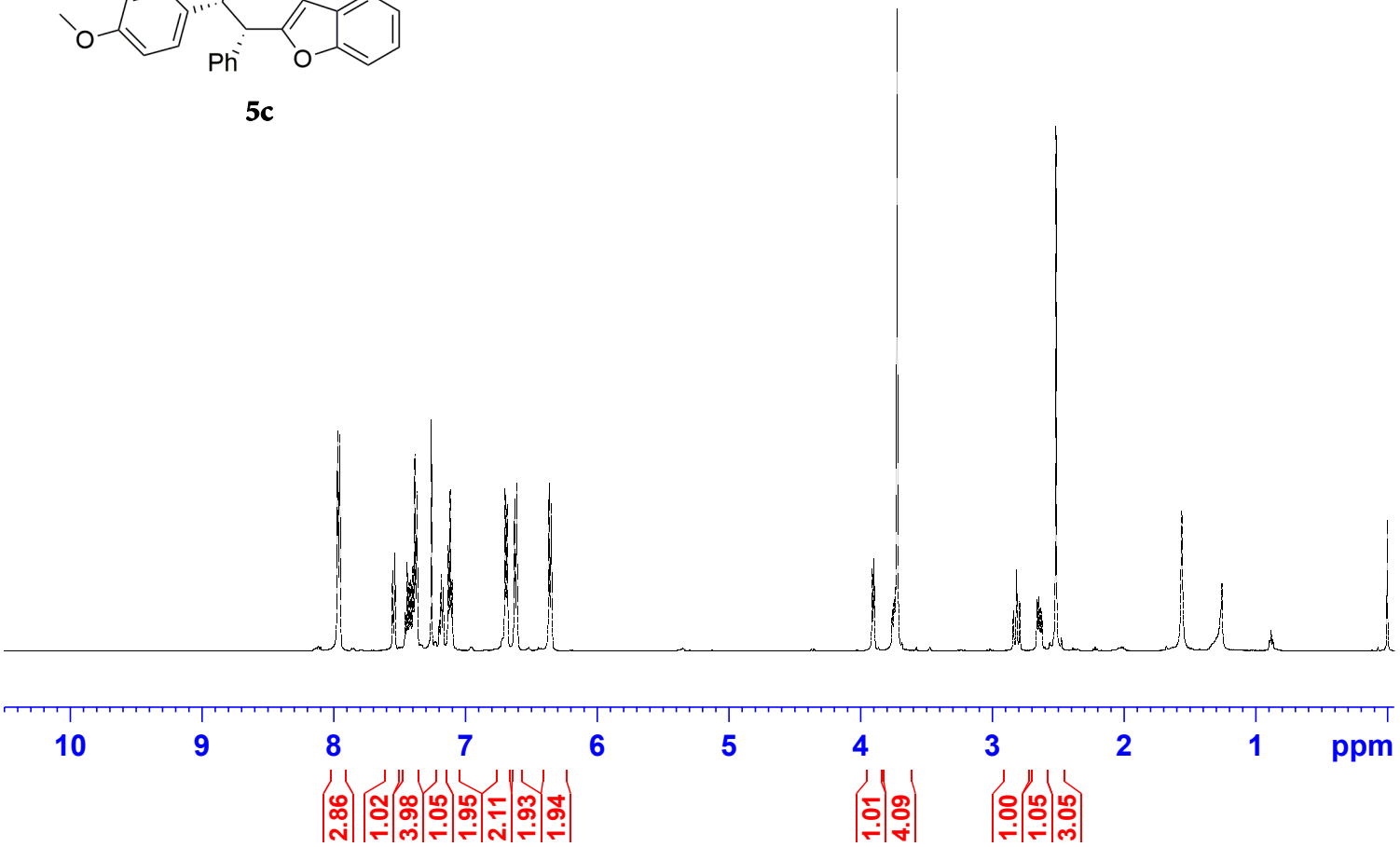
SI	32768
SF	100.6404139 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



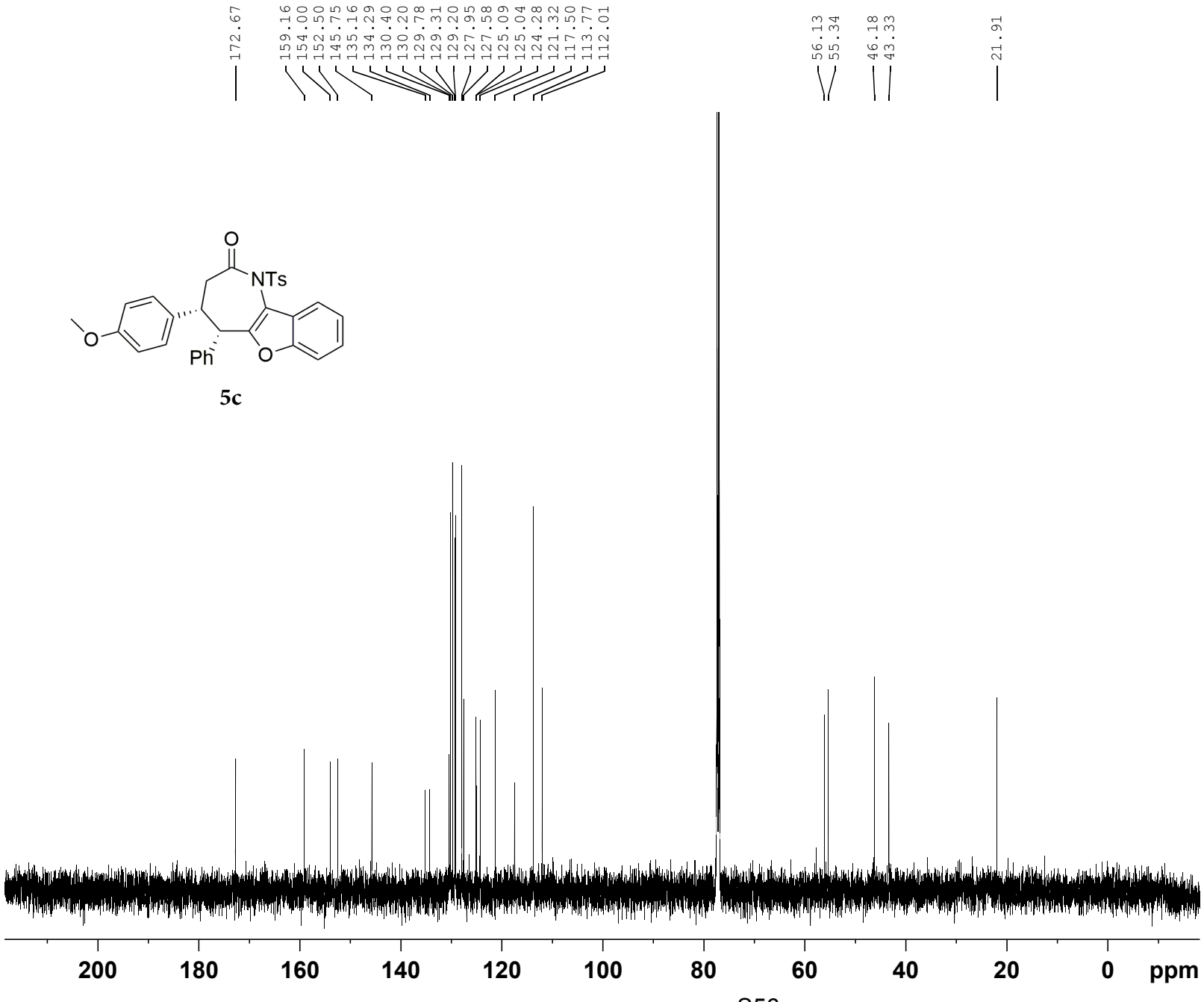
ckq-541



5c




```
Current Data Parameters
NAME                  CKQ
EXPNO                  68
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160929
Time                10.44 h
INSTRUM             spect
PROBHD    5 mm CPPBBO BB
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                  69.95
DW                 50.000 usec
DE                  6.50 usec
TE                 298.0 K
D1            1.00000000 sec
TD0                     1
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.60 usec
PLW1         20.00000000 W

F2 - Processing parameters
SI                  65536
SF           500.1300132 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



ckq-541

Current Data Parameters

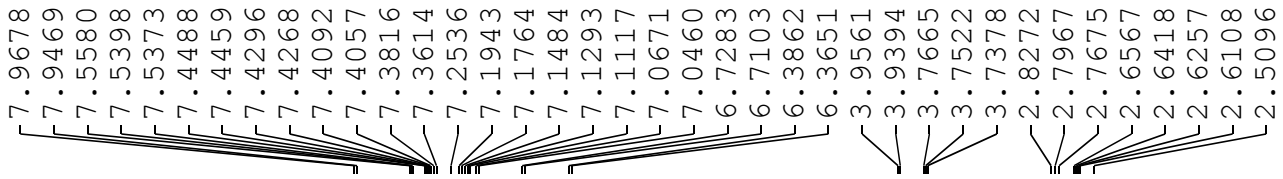
NAME	ckq
EXPNO	69
PROCNO	1

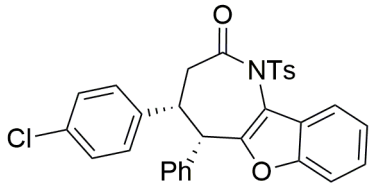
F2 - Acquisition Parameters

Date_	20160929
Time	10.48
INSTRUM	spect
PROBHD	5 mm CPPBBO BB
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	41
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010048 sec
RG	192.89
DW	16.800 usec
DE	18.00 usec
TE	298.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
SFO1	125.7703637 MHz
NUC1	13C
P1	9.80 usec
PLW1	57.00000000 W
SFO2	500.1320005 MHz
NUC2	1H
CPDPRG[2	waltz16
PCPD2	80.00 usec
PLW2	20.00000000 W
PLW12	0.35778001 W
PLW13	0.22898000 W

F2 - Processing parameters

SI	32768
SF	125.7577720 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

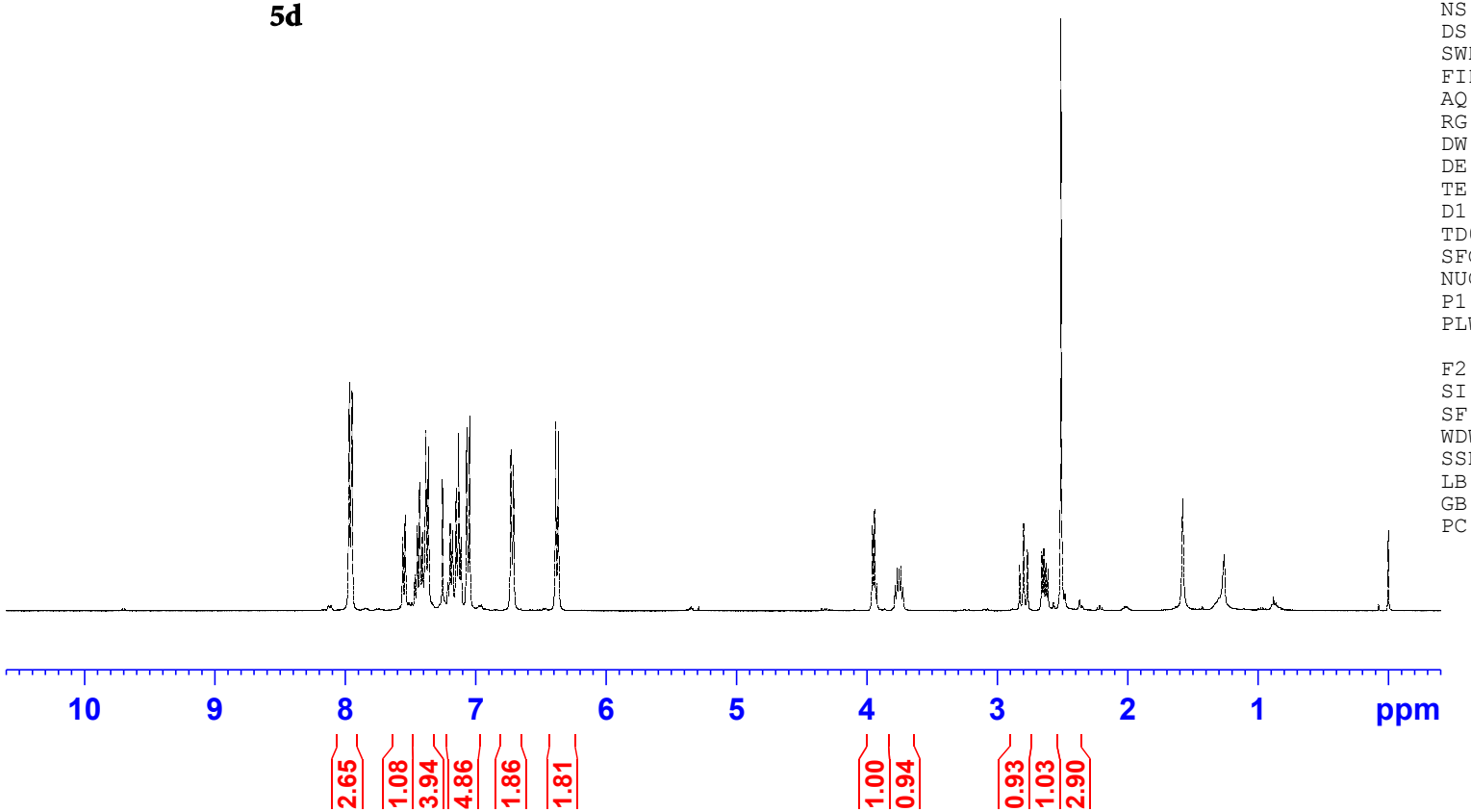


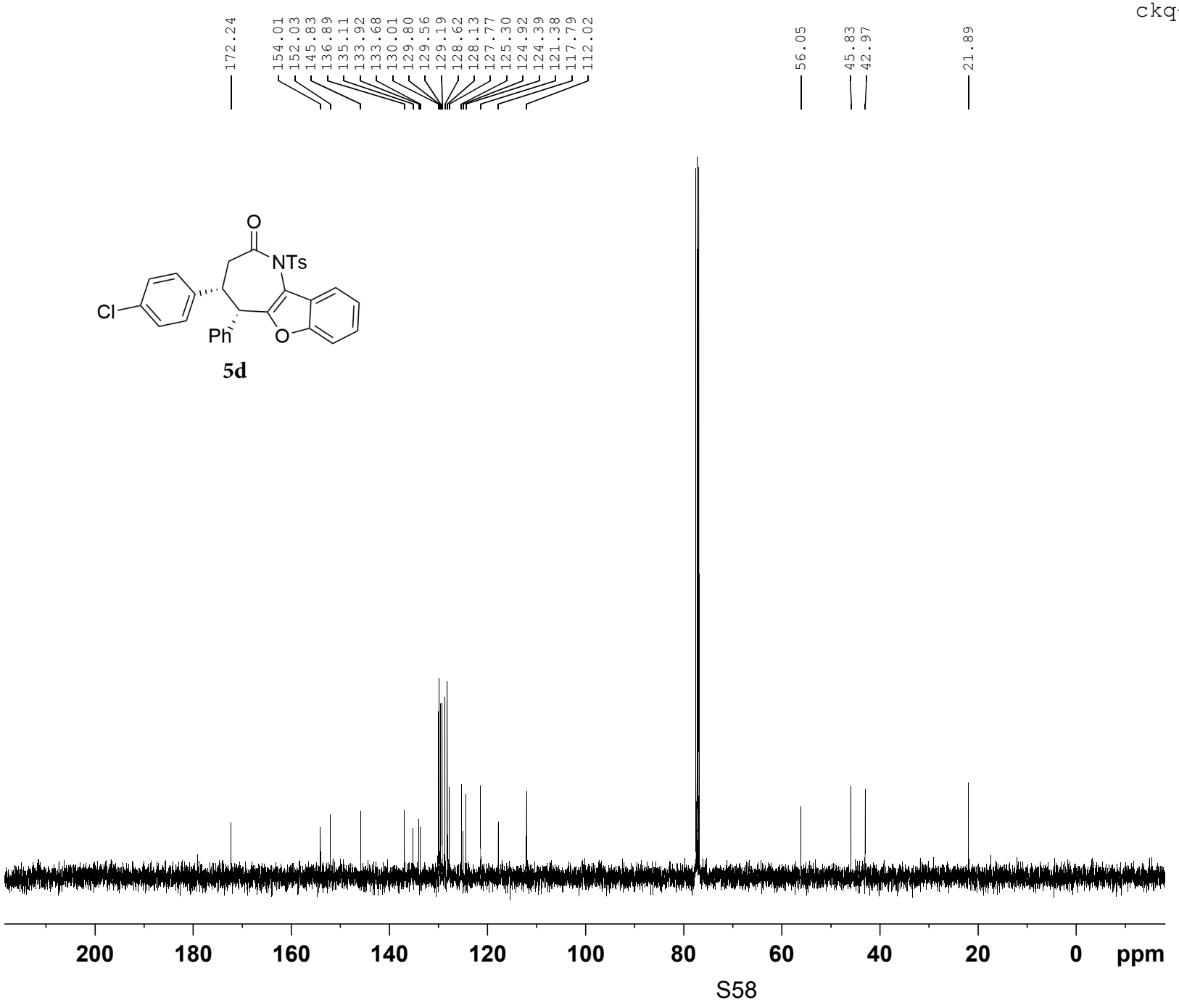


5d

ckq-545

```
Current Data Parameters
NAME                  ckq
EXPNO                  61
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160926
Time                14.22 h
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH              8012.820 Hz
FIDRES           0.489064 Hz
AQ              2.0447233 sec
RG                 125.02
DW                 62.400 usec
DE                  6.50 usec
TE                 300.5 K
D1             2.00000000 sec
TD0                    1
SFO1         400.2424716 MHz
NUC1                  1H
P1                 14.80 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.2400122 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```





5d

ckq-545

Current Data Parameters

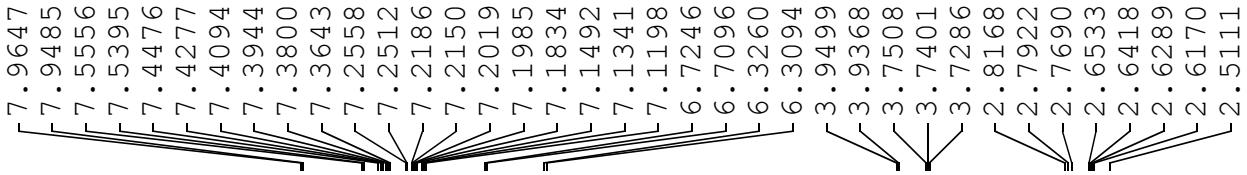
NAME	ckq
EXPNO	63
PROCNO	1

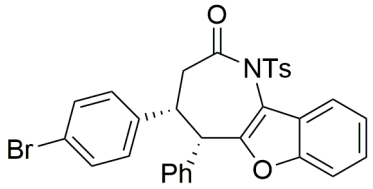
F2 - Acquisition Parameters

Date_	20160926
Time	14.24
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	40
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631488 sec
RG	206.33
DW	20.800 usec
DE	6.50 usec
TE	300.9 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
SFO1	100.6504916 MHz
NUC1	13C
P1	10.00 usec
PLW1	54.00000000 W
SFO2	400.2416010 MHz
NUC2	1H
CPDPRG[2	waltz16
PCPD2	90.00 usec
PLW2	12.00000000 W
PLW12	0.34680000 W
PLW13	0.28090999 W

F2 - Processing parameters

SI	32768	
SF	100.6404160	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

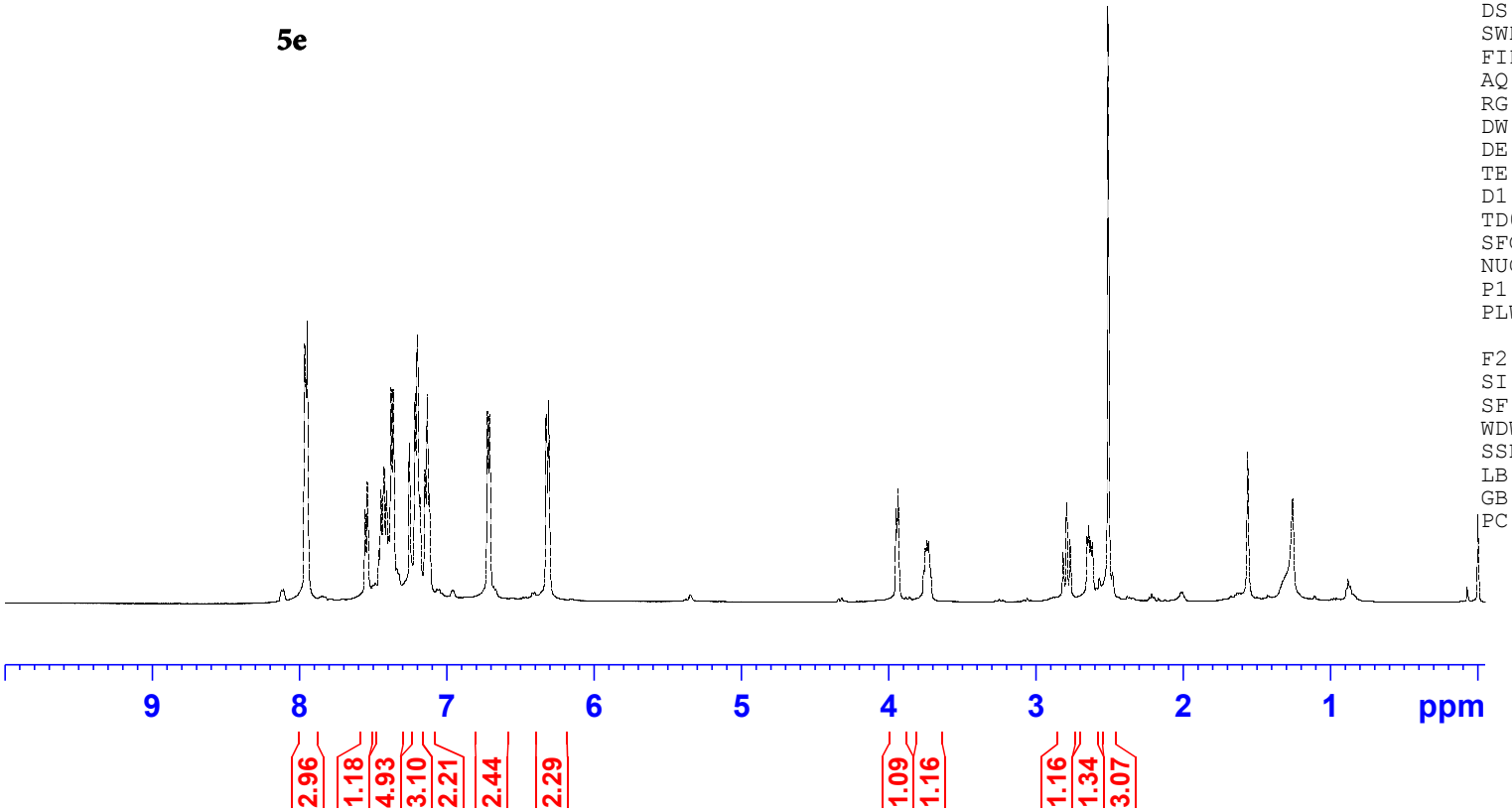


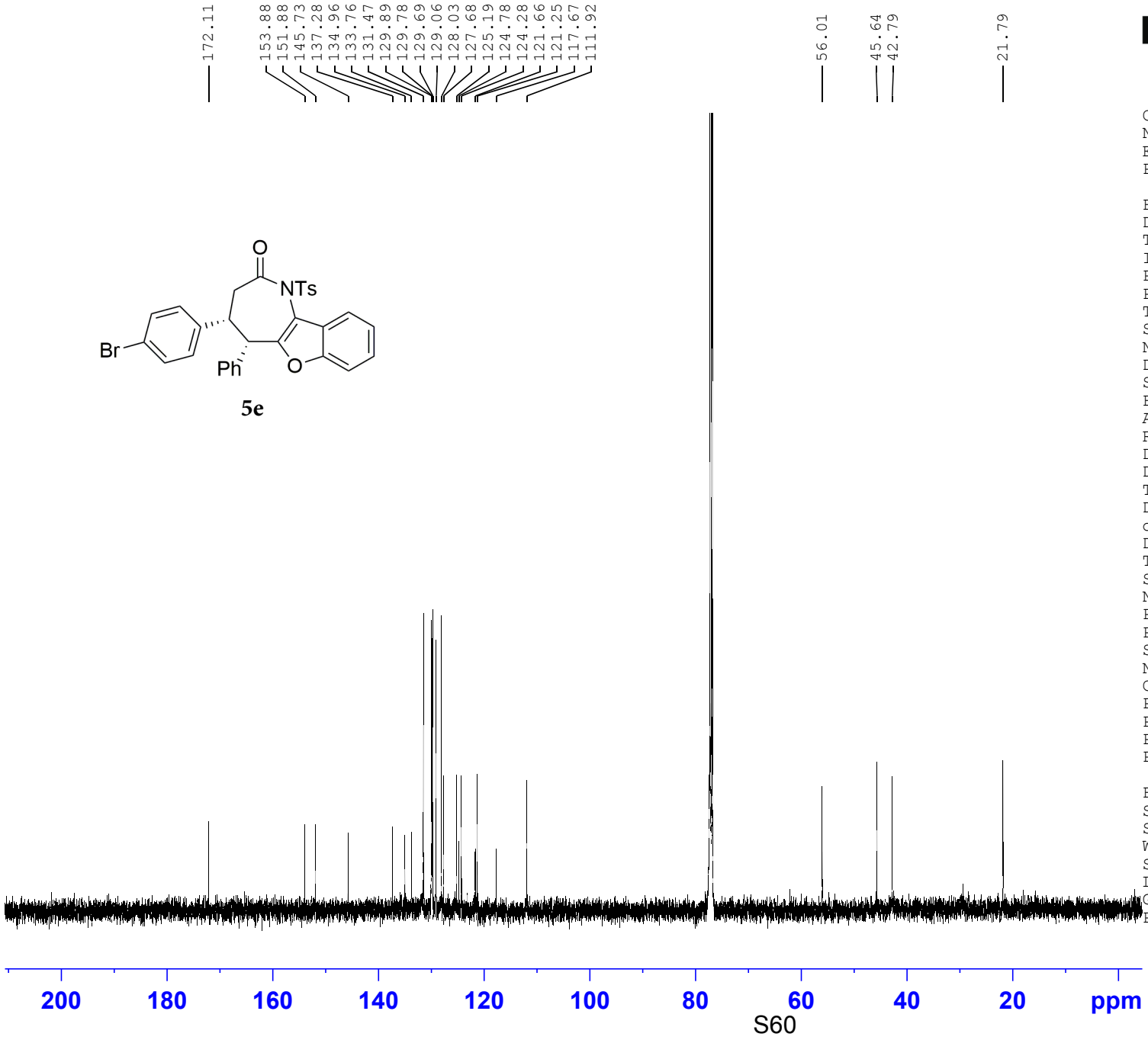


5e

ckq-549

```
Current Data Parameters
NAME                  CKQ
EXPNO                  72
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160929
Time                10.55 h
INSTRUM              spect
PROBHD   5 mm CPPBBO BB
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                 62.06
DW                50.000 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                     1
SFO1         500.1330885 MHz
NUC1                   1H
P1                10.60 usec
PLW1         20.00000000 W

F2 - Processing parameters
SI                  65536
SF           500.1300149 MHz
WDW                    EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```





5e

Current Data Parameters

NAME	CKQ
EXPNO	73
PROCNO	1

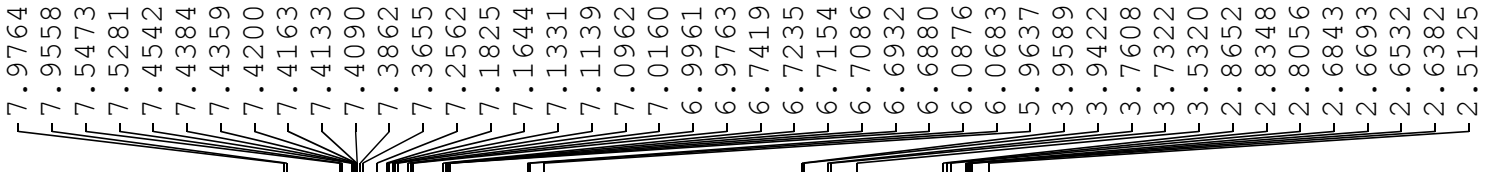
F2 - Acquisition Parameters

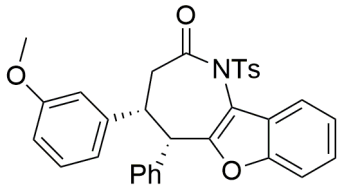
Date_	20160929
Time	10.57 h
INSTRUM	spect
PROBHD	5 mm CPPBBO BB
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	43
DS	4
SWH	29761.904 Hz
FIDRES	0.908261 Hz
AQ	1.1010048 sec
RG	192.89
DW	16.800 usec
DE	18.00 usec
TE	298.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
SFO1	125.7703637 MHz
NUC1	13C
P1	9.80 usec
PLW1	57.00000000 W
SFO2	500.1320005 MHz
NUC2	1H
CPDPRG[2	waltz16
PCPD2	80.00 usec
PLW2	20.00000000 W
PLW12	0.35778001 W
PLW13	0.22898000 W

F2 - Processing parameters

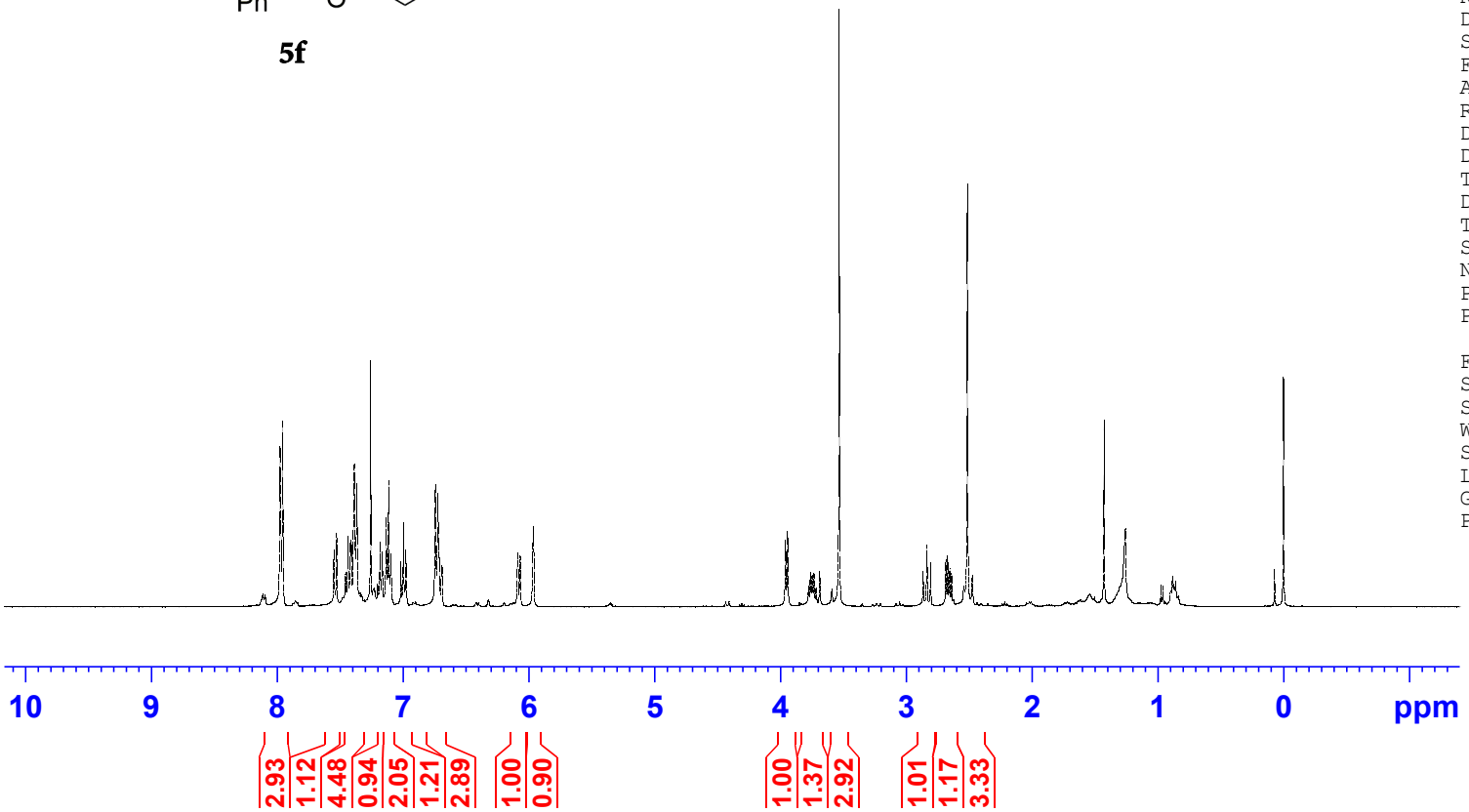
SI	32768
SF	125.7577885 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

ckq-577





5f

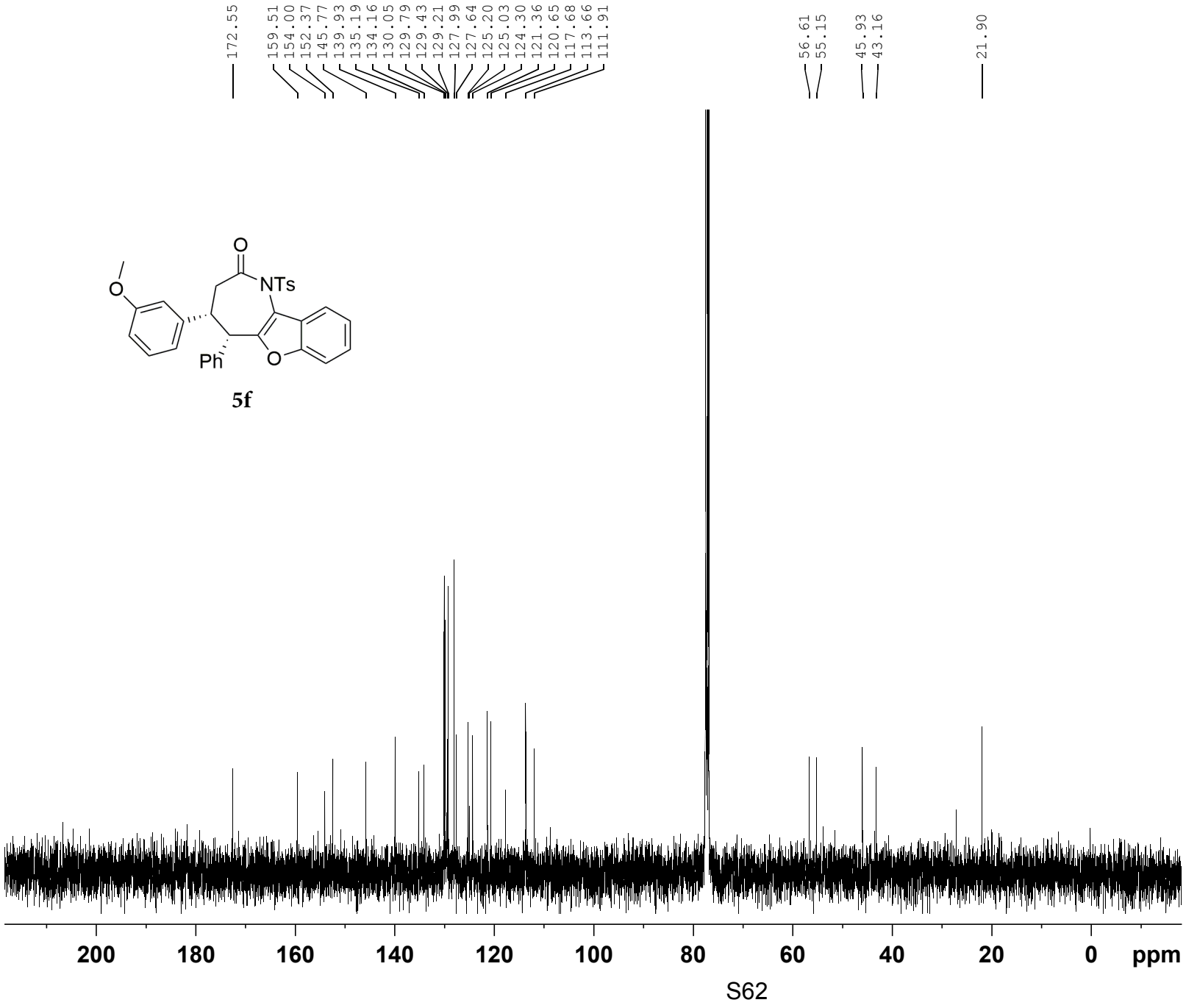


Current Data Parameters
NAME ckq
EXPNO 73
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161114
Time 14.41 h
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 206.33
DW 62.400 usec
DE 6.50 usec
TE 299.5 K
D1 2.00000000 sec
TD0 1
SFO1 400.2424716 MHz
NUC1 1H
P1 14.80 usec
PLW1 12.00000000 W

F2 - Processing parameters
SI 65536
SF 400.2400112 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

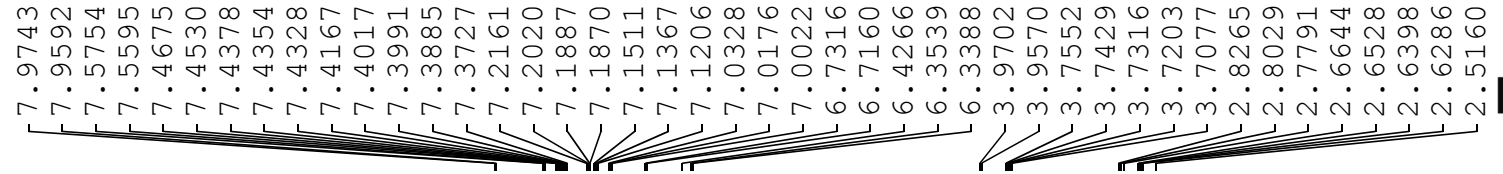
ckq-577



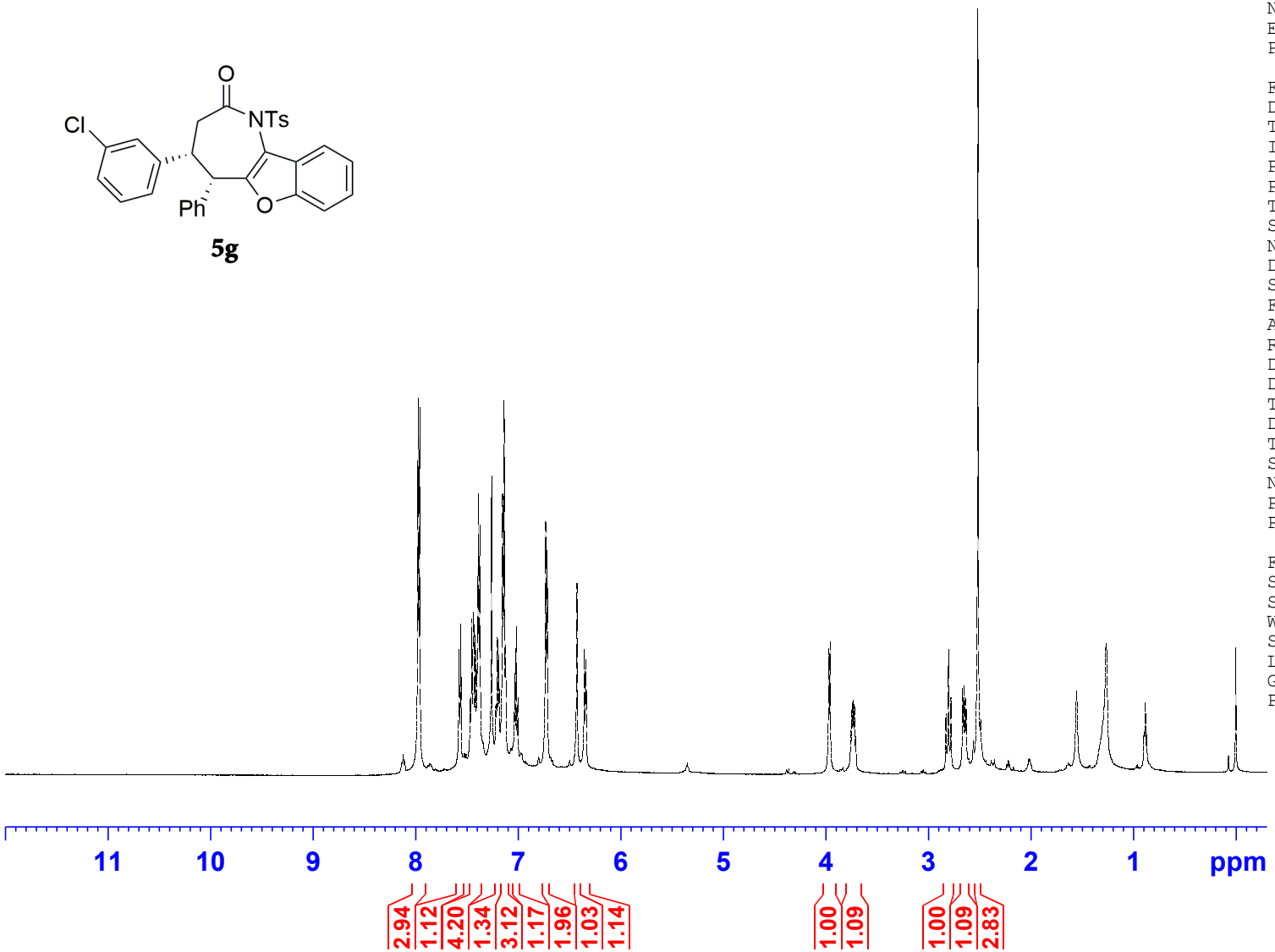
5f

```
Current Data Parameters
NAME                  ckq
EXPNO                  74
PROCNO                  1

F2 - Acquisition Parameters
Date_            20161114
Time                14.43
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   369
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                 206.33
DW                 20.800 usec
DE                  6.50 usec
TE                 300.2 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1
SFO1          100.6504916 MHz
NUC1                  13C
P1                 10.00 usec
PLW1          54.00000000 W
SFO2          400.2416010 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          12.00000000 W
PLW12          0.34680000 W
PLW13          0.28090999 W

F2 - Processing parameters
SI                 32768
SF            100.6404146 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

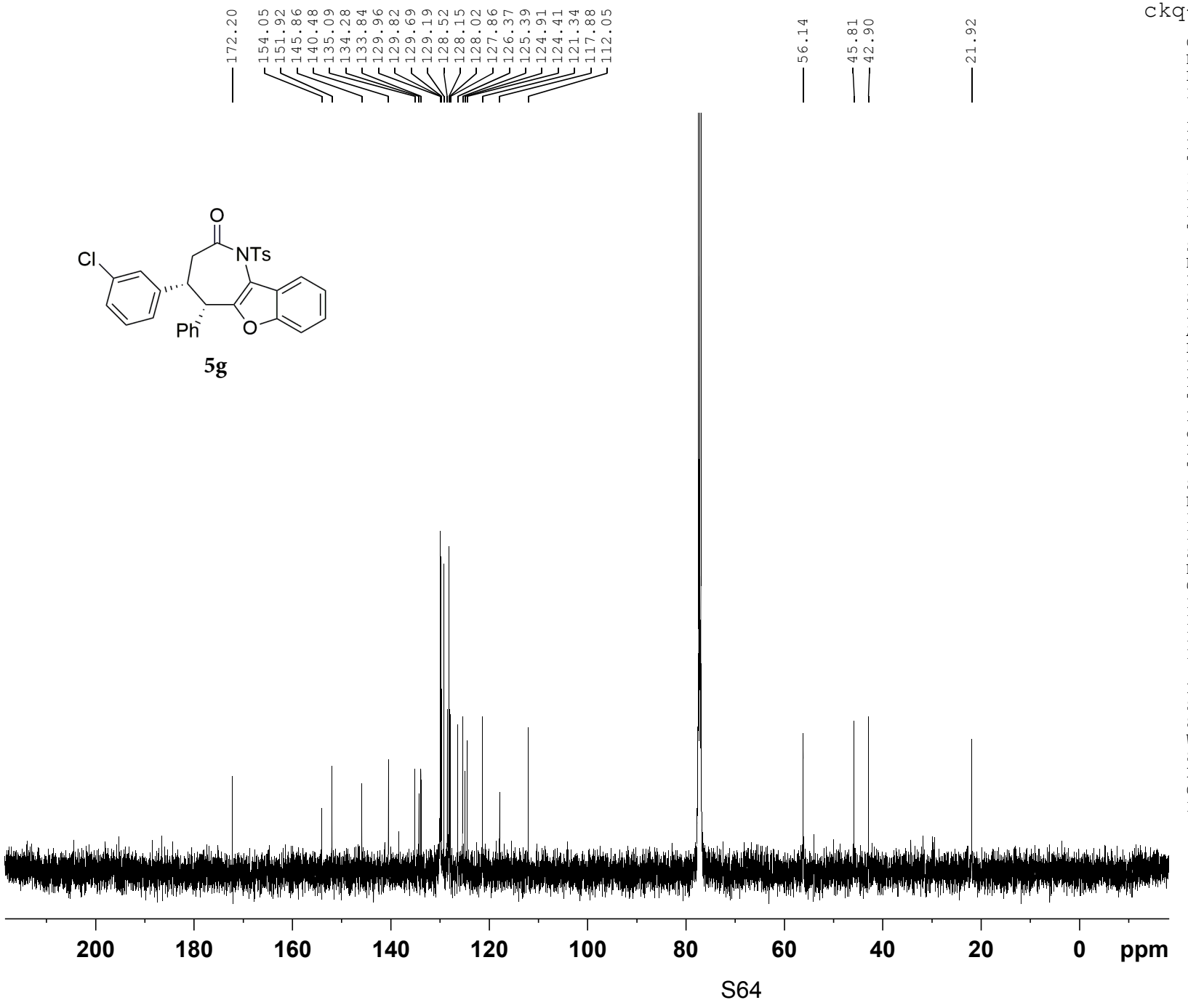
ckq-579



5g




```
Current Data Parameters
NAME                  CKQ
EXPNO                  80
PROCNO                  1

F2 - Acquisition Parameters
Date_            20161114
Time                14.40 h
INSTRUM              spect
PROBHD   5 mm CPPBBO BB
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                  55.37
DW                 50.000 usec
DE                  6.50 usec
TE                 298.0 K
D1            1.00000000 sec
TD0                     1
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.60 usec
PLW1         20.00000000 W

F2 - Processing parameters
SI                  65536
SF           500.1300132 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



ckq-579

Current Data Parameters

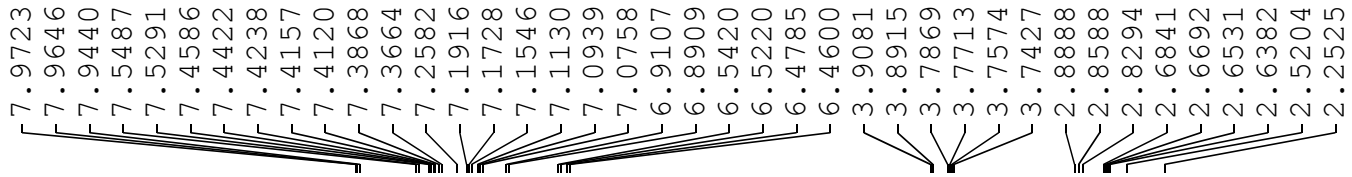
NAME	ckq
EXPNO	81
PROCNO	1

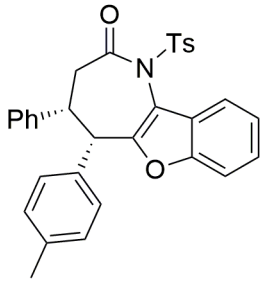
F2 - Acquisition Parameters

Date_	20161114
Time	14.42
INSTRUM	spect
PROBHD	5 mm CPPBBO BB
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	43
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010048 sec
RG	192.89
DW	16.800 usec
DE	18.00 usec
TE	298.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
SFO1	125.7703637 MHz
NUC1	13C
P1	9.80 usec
PLW1	57.00000000 W
SFO2	500.1320005 MHz
NUC2	1H
CPDPRG[2	waltz16
PCPD2	80.00 usec
PLW2	20.00000000 W
PLW12	0.35778001 W
PLW13	0.22898000 W

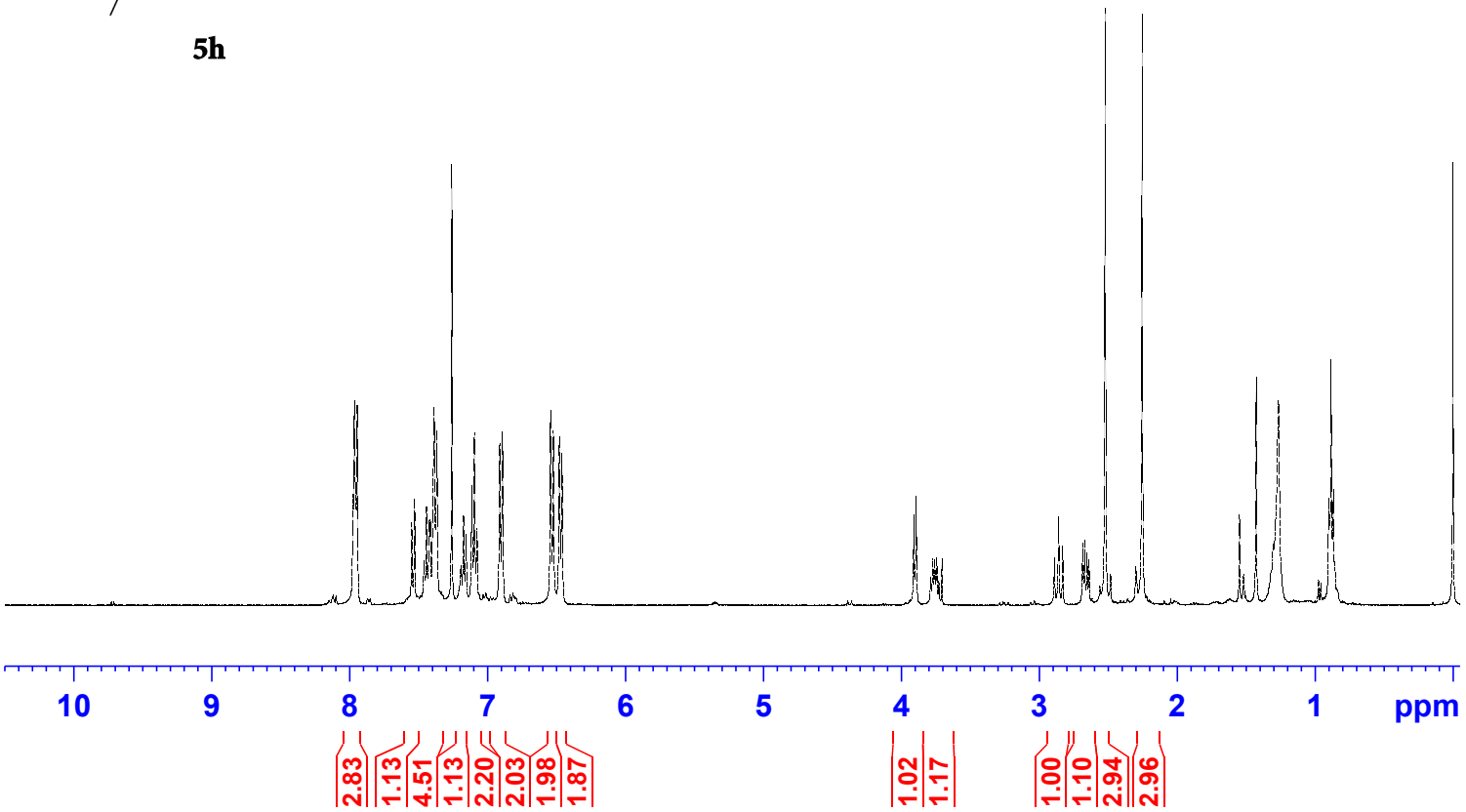
F2 - Processing parameters

SI	32768
SF	125.7577729 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



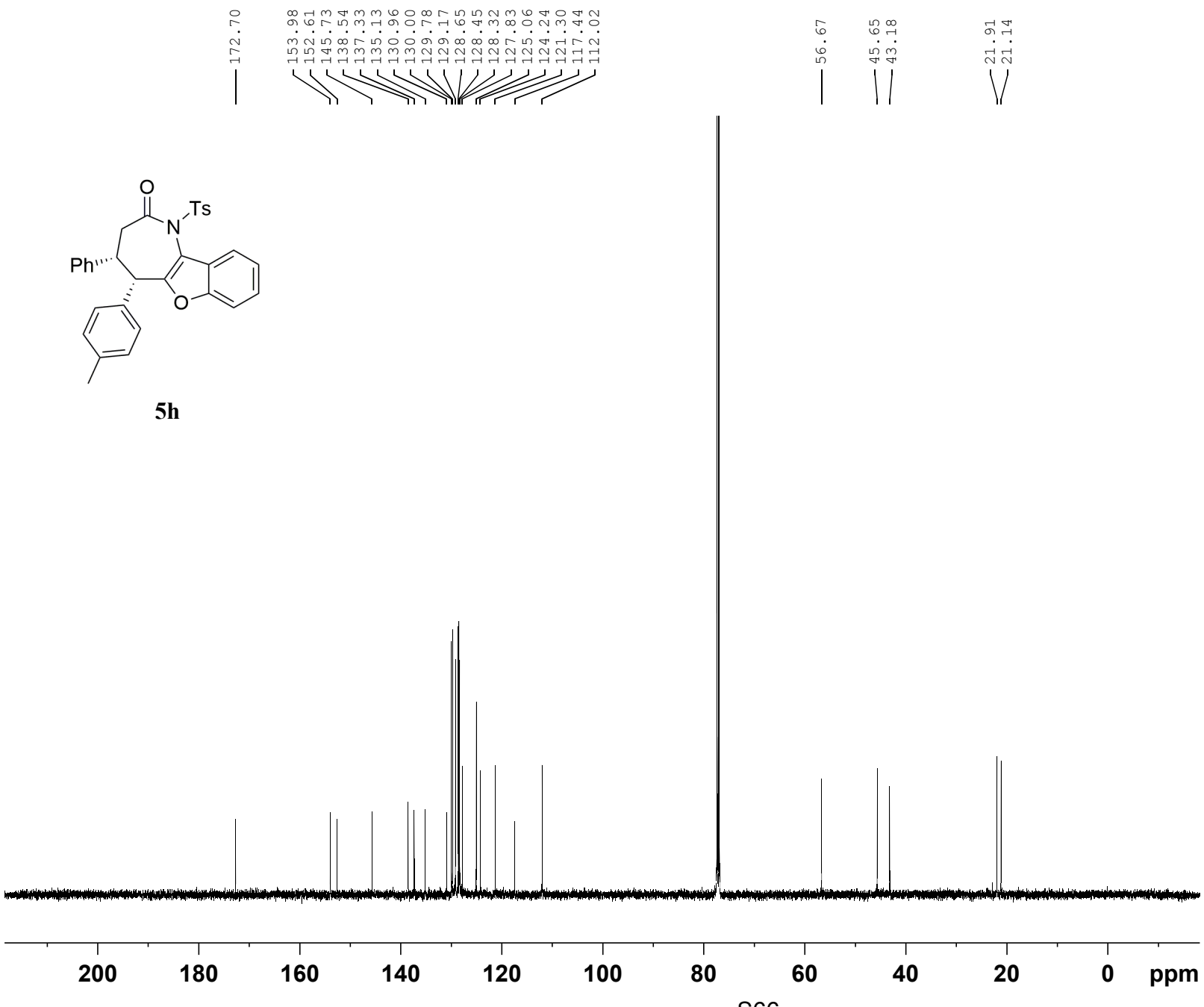


5h



ckq-634

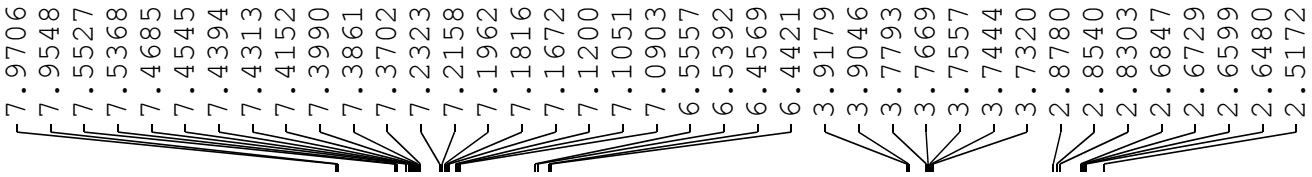
```
Current Data Parameters
NAME                  CKQ
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20171212
Time                17.05 h
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH              8012.820 Hz
FIDRES           0.489064 Hz
AQ              2.0447233 sec
RG                 206.33
DW                 62.400 usec
DE                  6.50 usec
TE                 298.4 K
D1             2.00000000 sec
TD0                    1
SFO1          400.2424716 MHz
NUC1                  1H
P1                 14.80 usec
PLW1          12.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.2400106 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

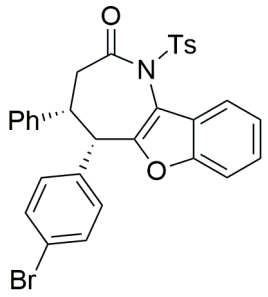


5h

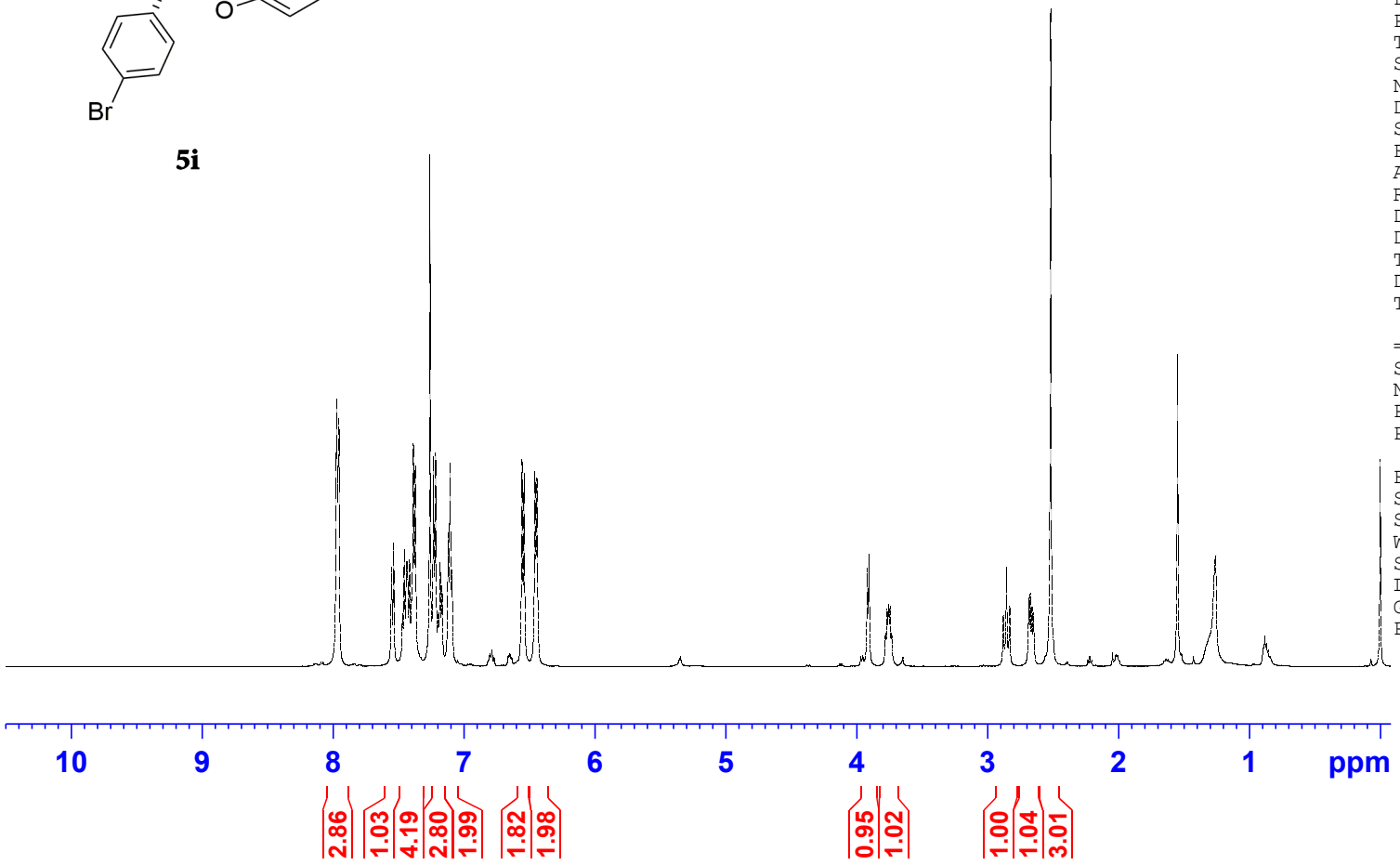
ckq-634

```
Current Data Parameters
NAME                  ckq
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170213
Time                15.24
INSTRUM             spect
PROBHD   5 mm CPPBBO BB
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     68
DS                      4
SWH             29761.904 Hz
FIDRES          0.454131 Hz
AQ              1.1010048 sec
RG                 192.89
DW                 16.800 usec
DE                 18.00 usec
TE                 298.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                  13C
P1                  9.80 usec
PLW1         57.00000000 W

======== CHANNEL f2 ========
SFO2         500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2               80.00 usec
PLW2         20.00000000 W
PLW12         0.35778001 W
PLW13         0.22898000 W

F2 - Processing parameters
SI                  32768
SF           125.7577748 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



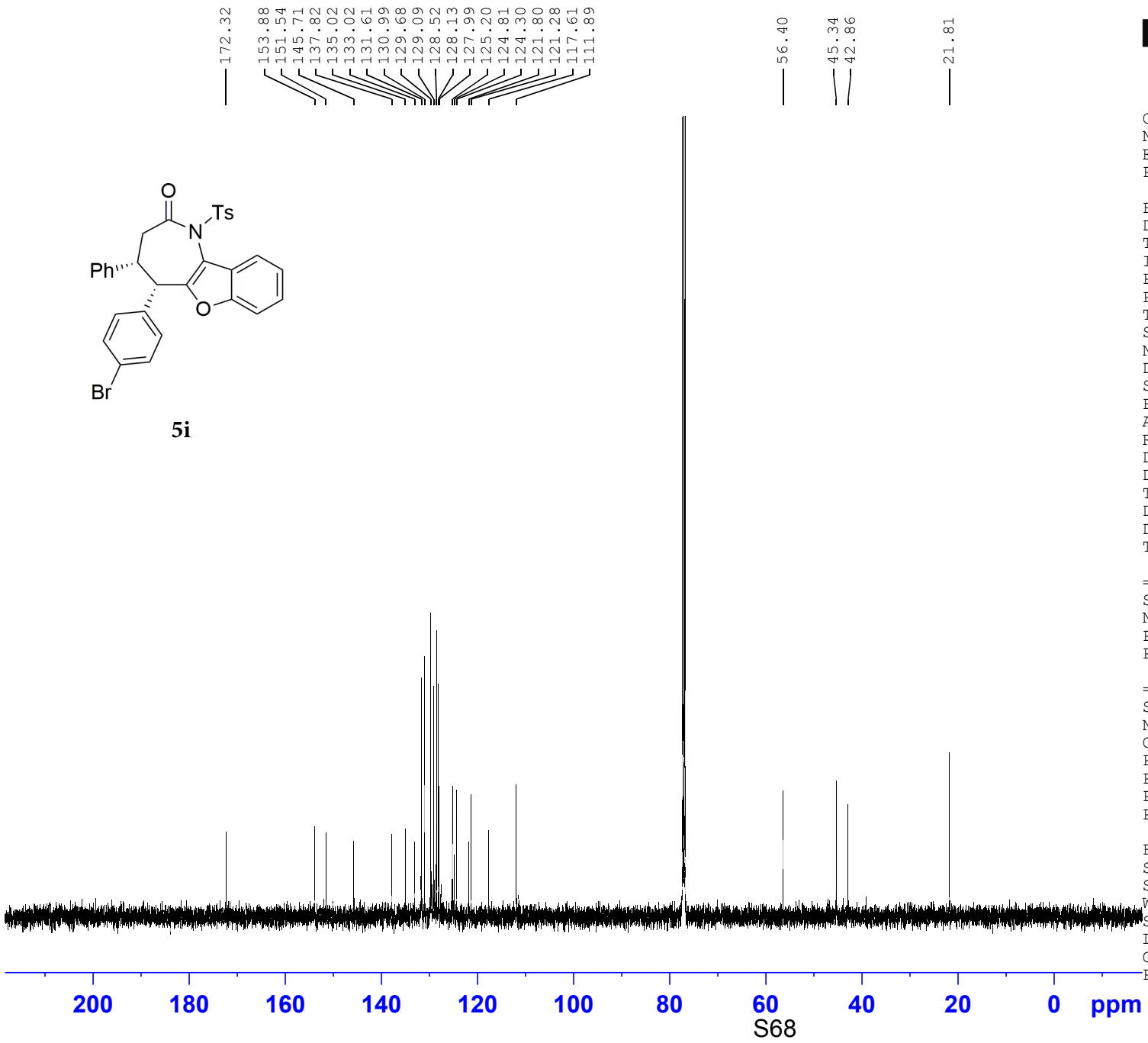


5i



Z63

```
Current Data Parameters
NAME                  500 M
EXPNO                    57
PROCNO                    1

F2 - Acquisition Parameters
Date_              20180531
Time                  21.19
INSTRUM               spect
PROBHD    5 mm CPPBBO BB
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       32
DS                        2
SWH             10000.000 Hz
FIDRES              0.152588 Hz
AQ                3.2767999 sec
RG                    55.37
DW                  50.000 usec
DE                   6.50 usec
TE                  298.2 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1          500.1330885 MHz
NUC1                     1H
P1                   11.50 usec
PLW1          20.00000000 W

F2 - Processing parameters
SI                    65536
SF            500.1300131 MHz
WDW                      EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```



Z63

Current Data Parameters
NAME 500 M
EXPNO 42
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180528
Time 20.05
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 125
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

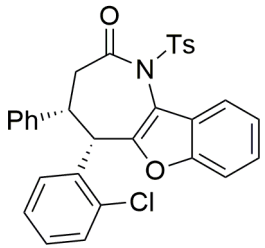
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W

======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W

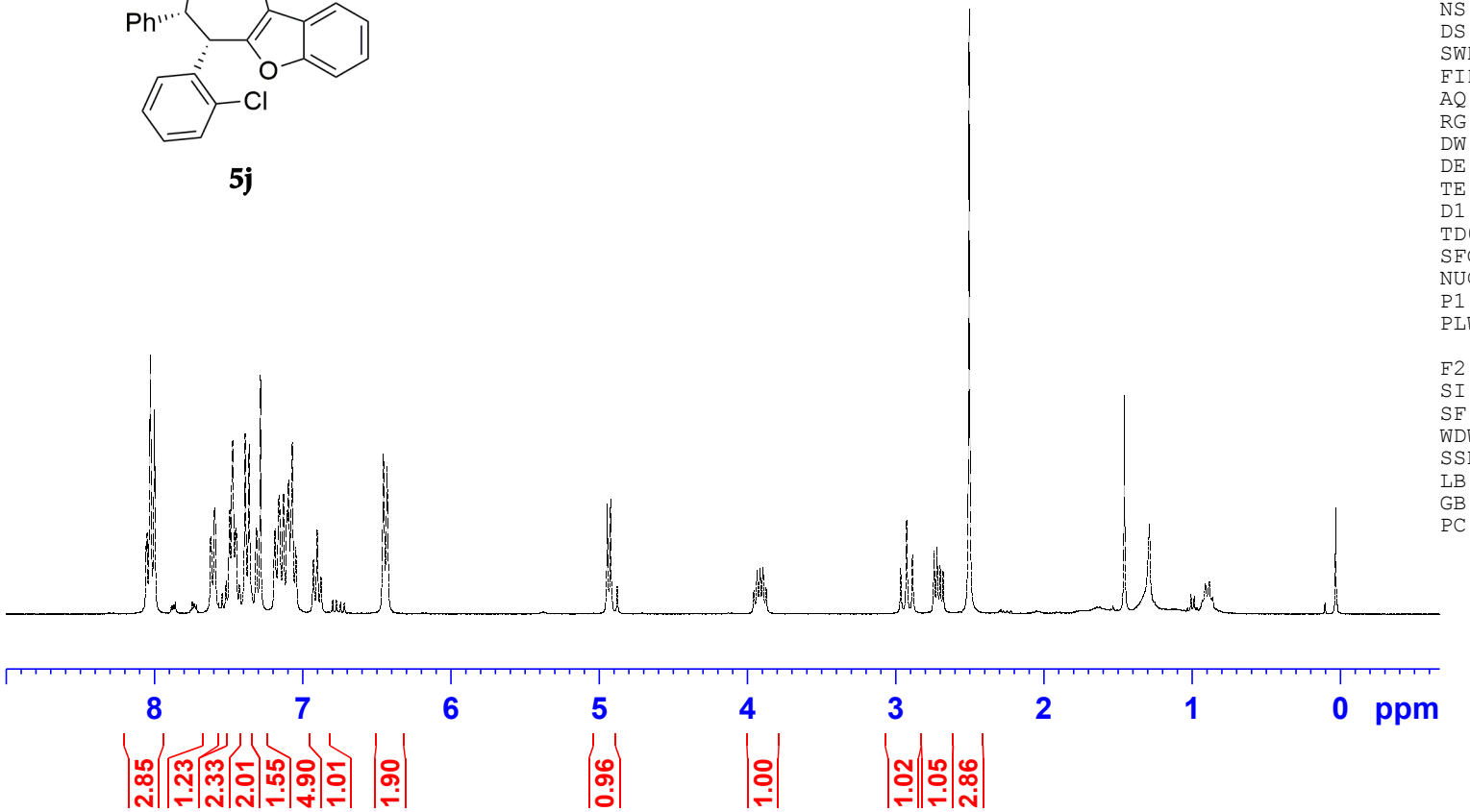
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

ckq-637

8.0546 8.0489 8.0270 7.9995 7.6196 7.5954 7.5903 7.4951 7.4905 7.4770 7.4716 7.4513 7.4459 7.3877 7.3606 7.3107 7.2845 7.1904 7.1860 7.1595 7.1535 7.1287 7.1072 7.1024 7.0942 7.0816 7.0688 7.0453 6.9284 6.9251 6.9019 6.4547 6.4307 4.9443 4.9215 3.9137 3.8939 2.9648 2.9243 2.8844 2.7397 2.7197 2.6985 2.5025



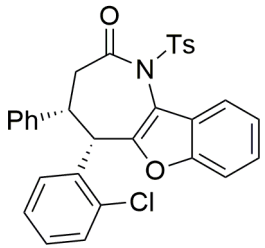
5j



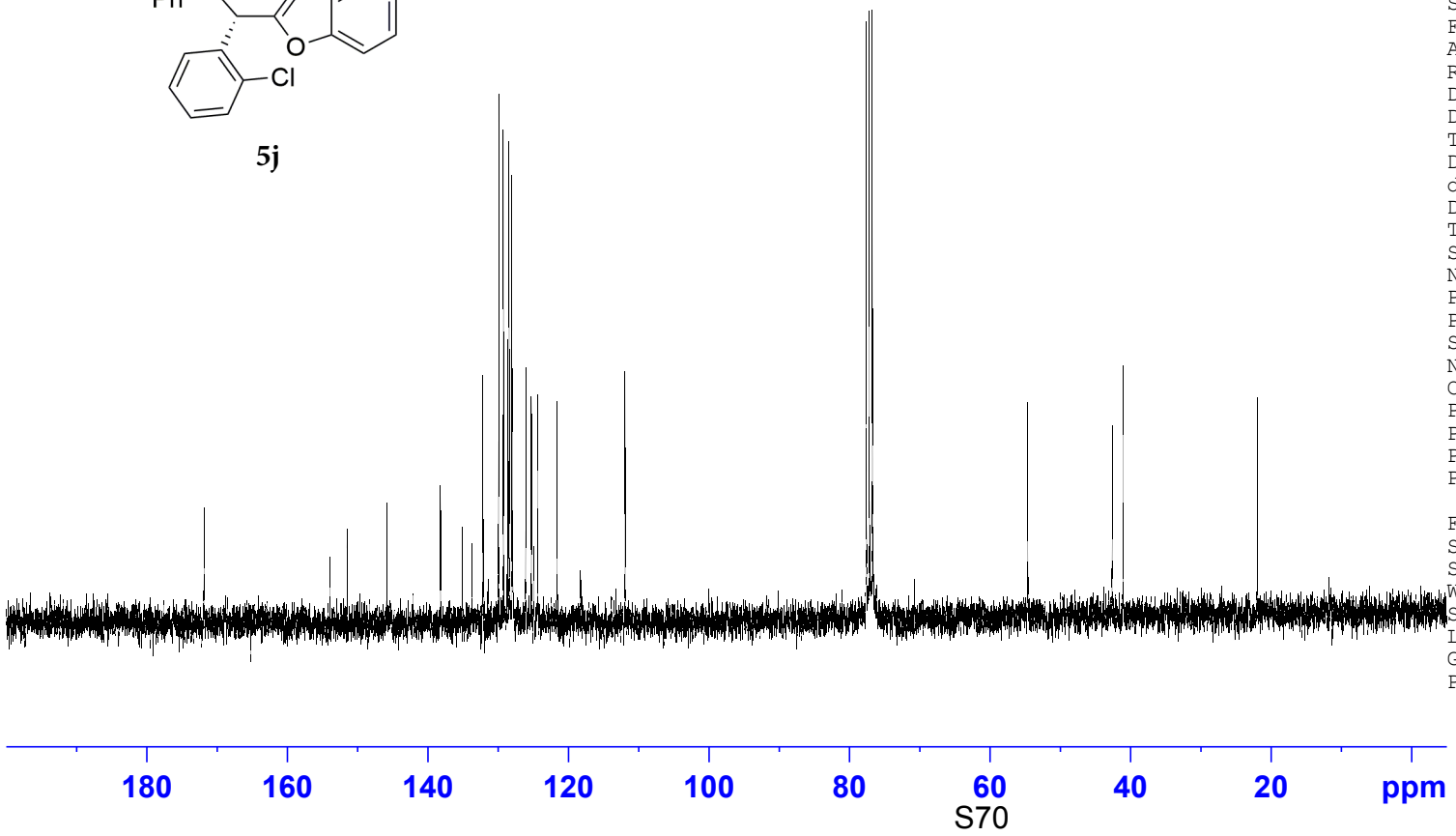

```
Current Data Parameters
NAME                  CKQ
EXPNO                  28
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170211
Time                16.24 h
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH              6009.615 Hz
FIDRES           0.183399 Hz
AQ               5.4525952 sec
RG                 209.09
DW                 83.200 usec
DE                   6.50 usec
TE                  298.2 K
D1            1.00000000 sec
TD0                    1
SFO1          300.1318534 MHz
NUC1                  1H
P1                  8.00 usec
PLW1         18.00000000 W

F2 - Processing parameters
SI                 65536
SF           300.1300000 MHz
WDW                   EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

ckq-637

171.84
153.93
151.50
145.82
138.18
135.11
133.67
132.22
132.00
129.91
129.24
129.18
128.63
128.42
128.02
127.89
126.06
125.24
124.92
124.37
121.59
118.24
111.91
54.60
42.58
41.00
21.91

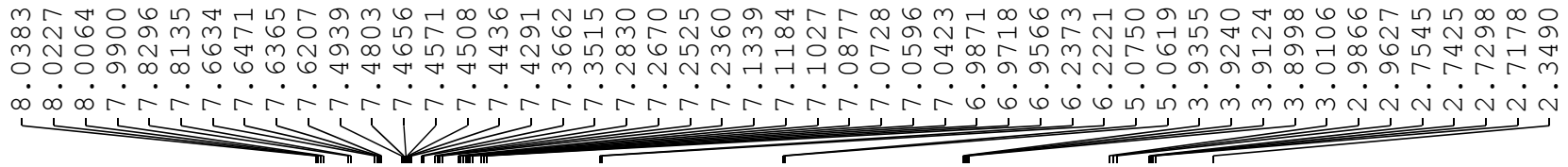


5j

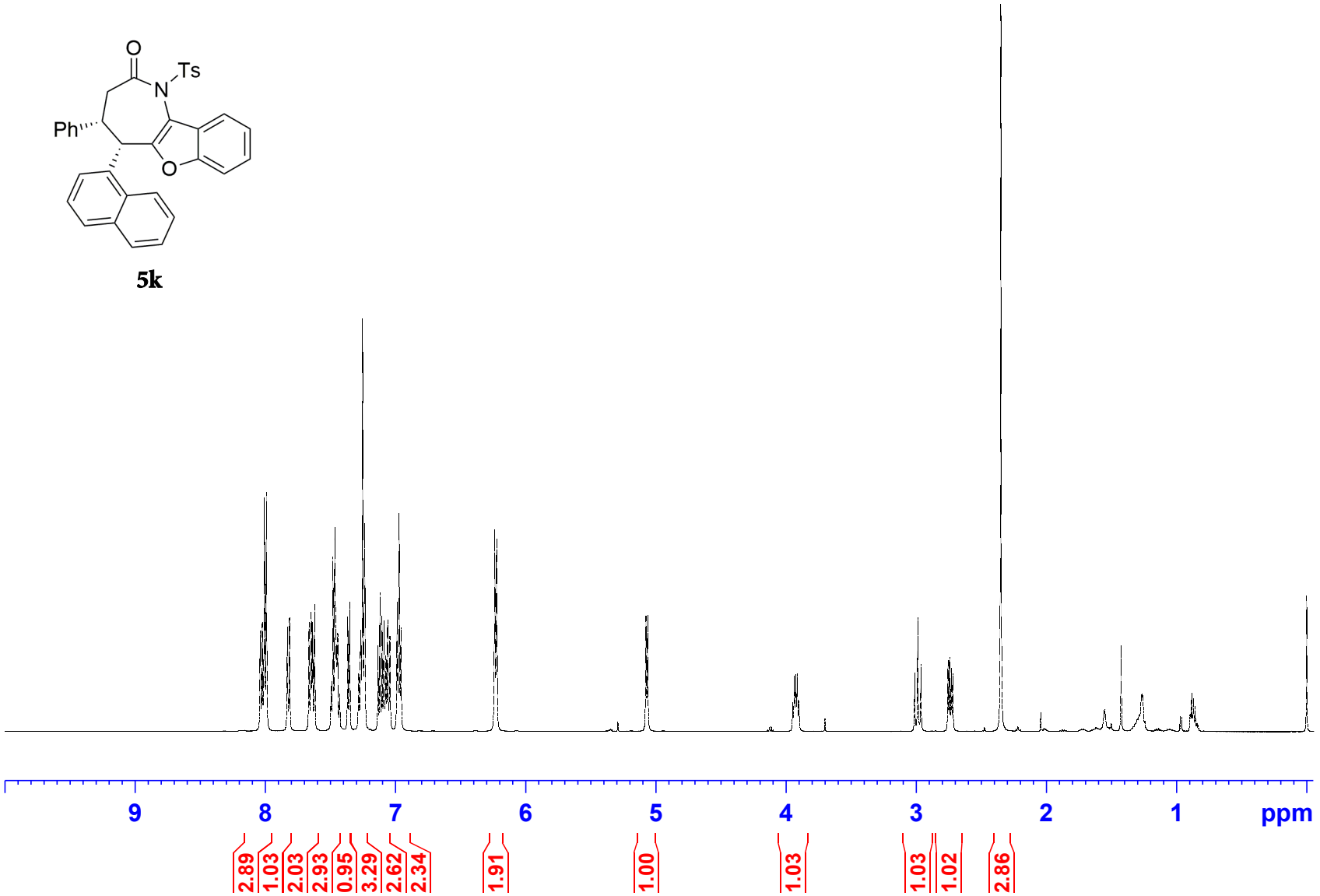



```
Current Data Parameters
NAME                   CKQ
EXPNO                   29
PROCNO                   1

F2 - Acquisition Parameters
Date_             20170211
Time                 16.27 h
INSTRUM              spect
PROBHD    5 mm DUL 13C-1
PULPROG            zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     174
DS                       4
SWH              18028.846 Hz
FIDRES            0.550197 Hz
AQ                1.8175317 sec
RG                   209.09
DW                  27.733 usec
DE                   6.50 usec
TE                  298.2 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                      1
SFO1            75.4752949 MHz
NUC1                   13C
P1                  11.00 usec
PLW1          195.00000000 W
SFO2           300.1312005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2           14.00000000 W
PLW12           0.17284000 W
PLW13           0.14000000 W

F2 - Processing parameters
SI                   32768
SF              75.4677393 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

ckq-635



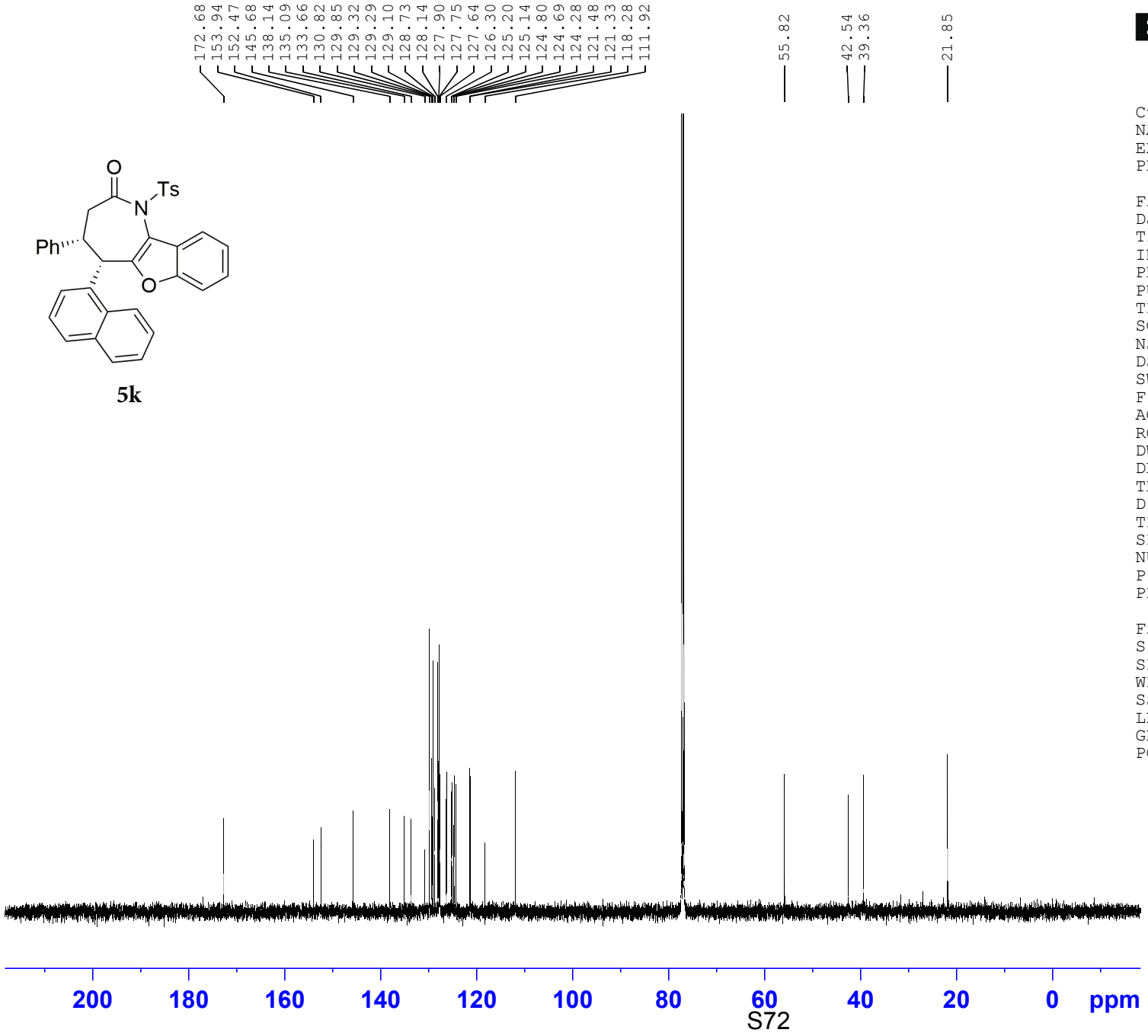
5k



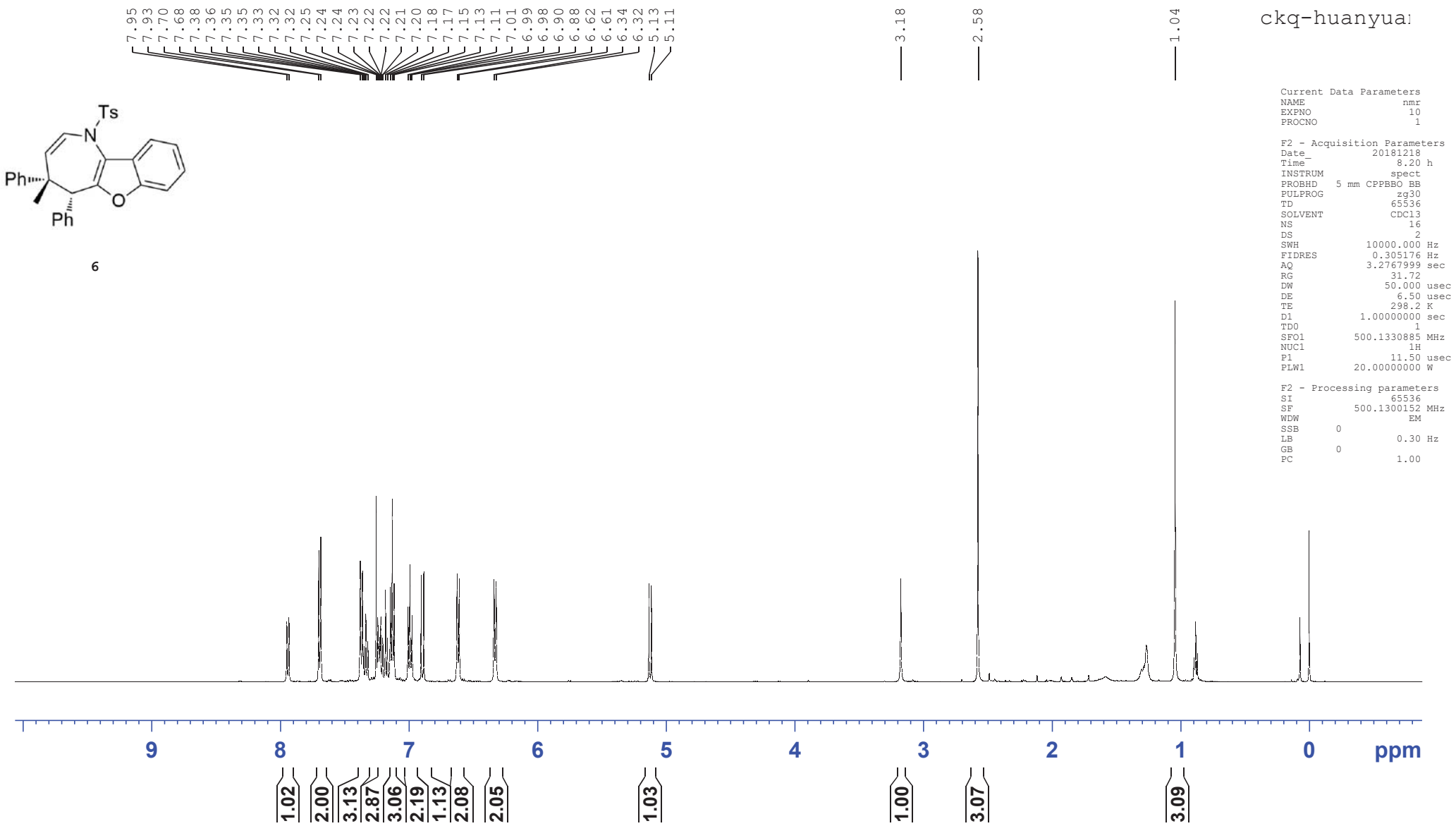

```
Current Data Parameters
NAME                  CKQ
EXPNO                  12
PROCNO                  1

F2 - Acquisition Parameters
Date_           20170213
Time                15.35 h
INSTRUM             spect
PROBHD   5 mm CPPBBO BB
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                 55.37
DW                50.000 usec
DE                 6.50 usec
TE                298.0 K
D1            1.00000000 sec
TD0                     1
SFO1         500.1330885 MHz
NUC1                   1H
P1                 10.60 usec
PLW1         20.00000000 W

F2 - Processing parameters
SI                  65536
SF           500.1300154 MHz
WDW                    EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

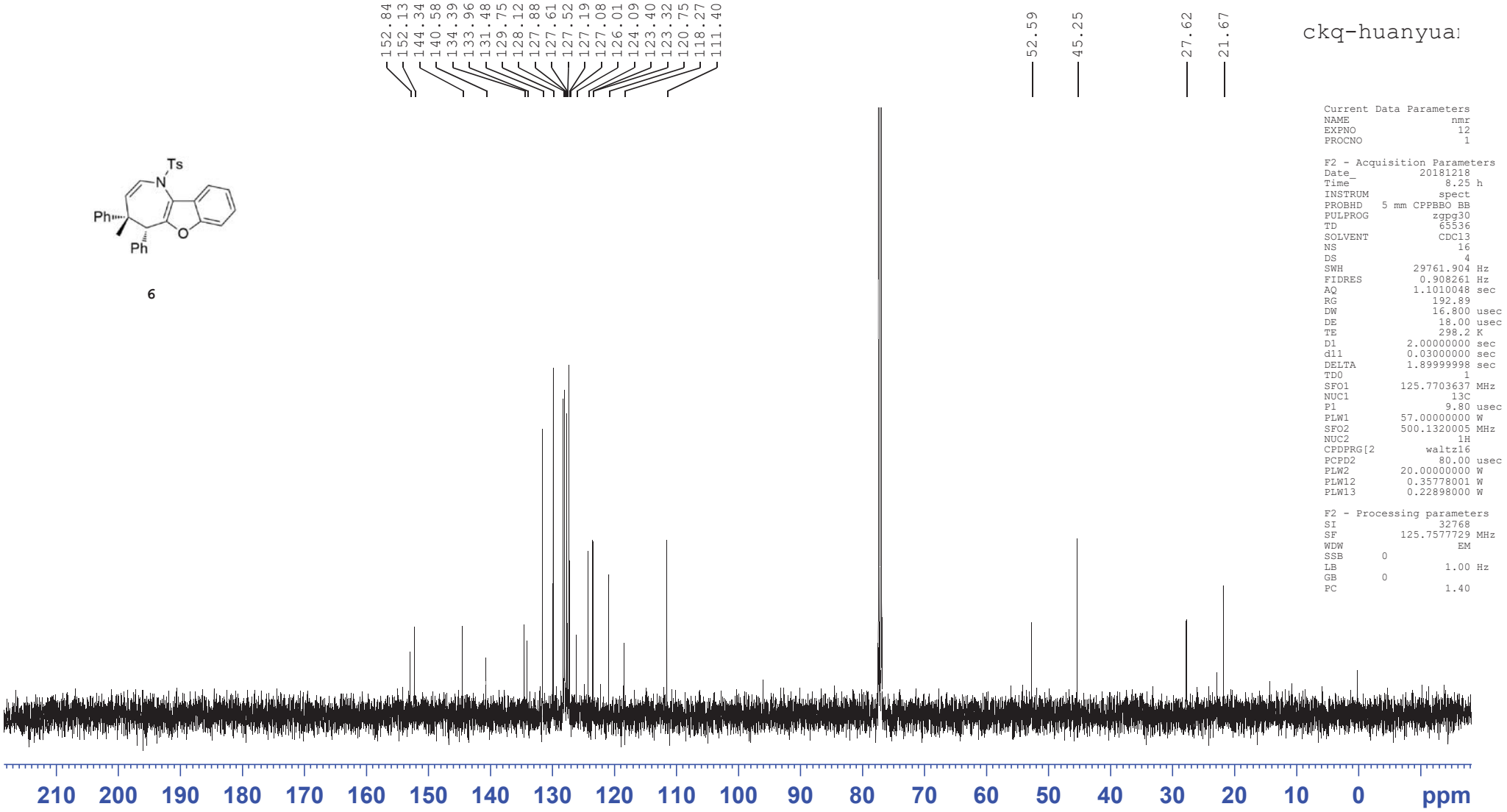
ckq-635

5k



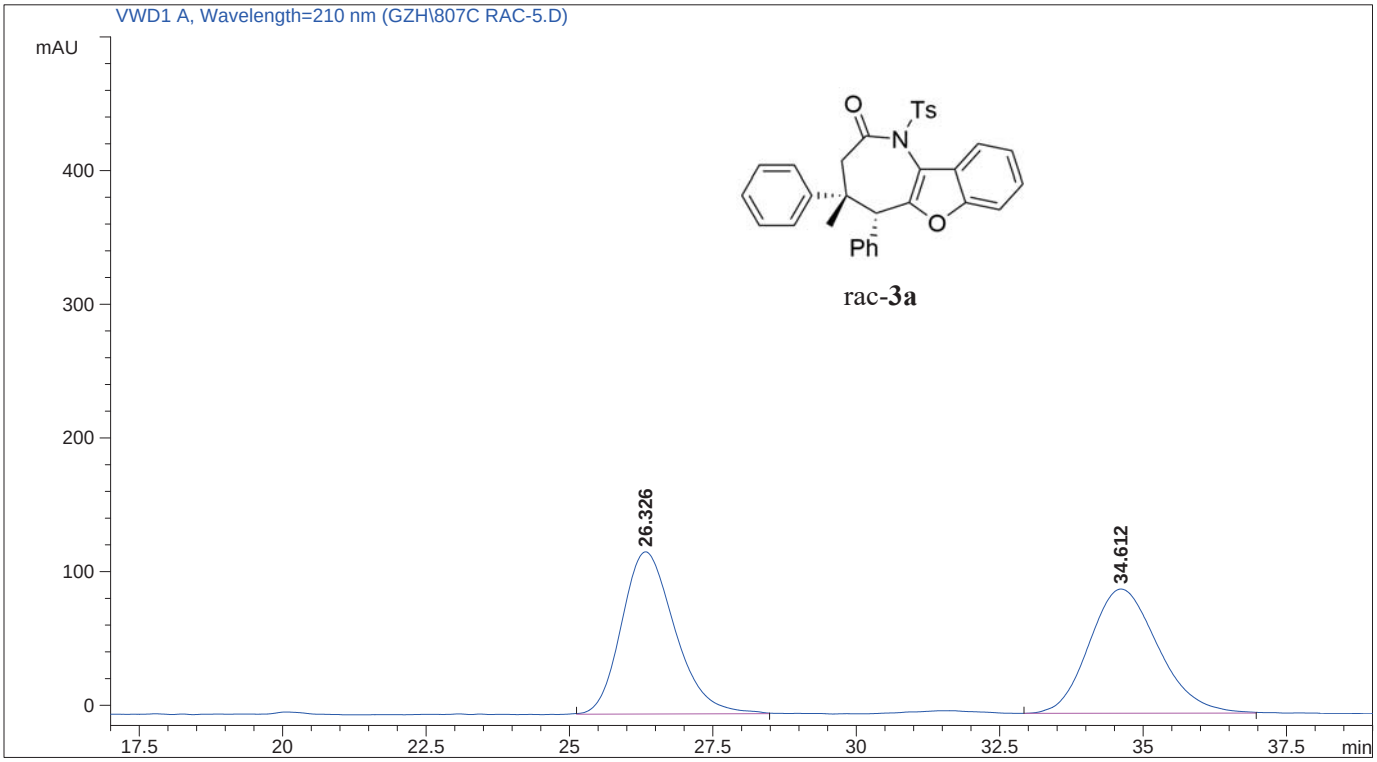

```
Current Data Parameters
NAME                  CKQ
EXPNO                  13
PROCNO                  1

F2 - Acquisition Parameters
Date_            20171214
Time                10.01 h
INSTRUM             spect
PROBHD   5 mm CPPBBO BB
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH           10000.000 Hz
FIDRES         0.305176 Hz
AQ             3.2767999 sec
RG                  31.72
DW                 50.000 usec
DE                  6.50 usec
TE                 298.1 K
D1            1.00000000 sec
TD0                     1
SFO1         500.1330885 MHz
NUC1                   1H
P1                 11.50 usec
PLW1         20.00000000 W

F2 - Processing parameters
SI                  32768
SF           125.7577885 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```


7.95
7.93
7.70
7.68
7.38
7.36
7.35
7.35
7.33
7.32
7.32
7.25
7.24
7.24
7.23
7.22
7.22
7.21
7.20
7.18
7.17
7.15
7.13
7.11
7.01
6.99
6.98
6.90
6.88
6.62
6.61
6.34
6.32
5.13
5.11
3.18
2.58
1.04
ckq-huanyua
Ts
N
Ph
Ph
O
6
Current Data Parameters
NAME nmr
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20181218
Time 8.20 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 31.72
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 11.50 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300152 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
1.02
2.00
3.13
2.87
3.06
2.19
1.13
2.08
2.05
1.03
1.00
3.07
3.09


152.84
152.13
144.34
140.58
134.39
133.96
131.48
129.75
128.12
127.88
127.61
127.52
127.19
127.08
126.01
124.09
123.40
123.32
120.75
118.27
111.40
52.59
45.25
27.62
21.67
ckq-huanyua
Ts
N
Ph
Ph
O
6
Current Data Parameters
NAME nmr
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20181218
Time 8.25 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577729 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Data File C:\CHEM32\1\DATA\GZH\807C RAC-5.D
Sample Name: 807C rac

```
==========================================================================
Acq. Operator   : GZH
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 2017-11-21 10:02:02
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2017-11-21 9:55:41  by GZH
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2018-3-7 15:21:35  by LZ
                  (modified after loading)
Sample Info     : IC H/I  70:30  1 mL/min, 254 nm
```

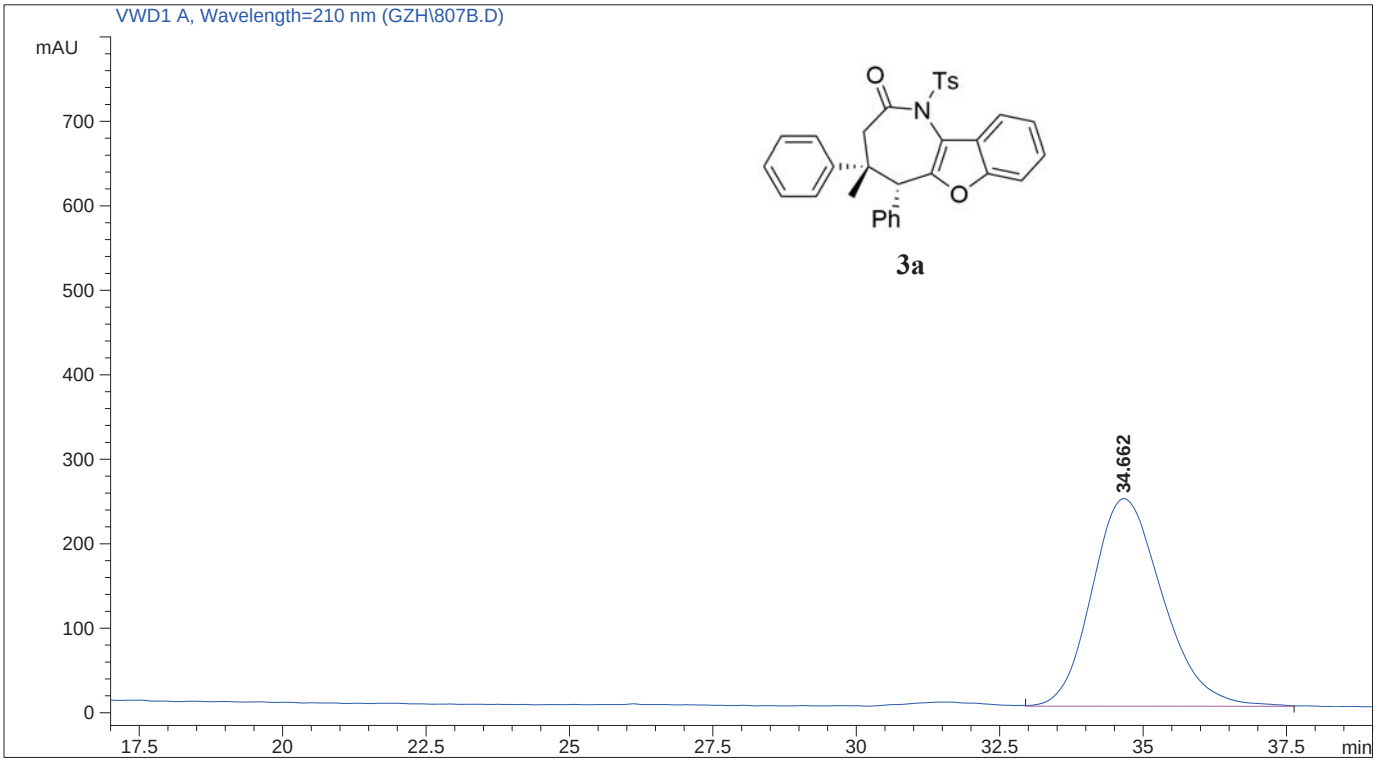


rac-**3a**

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=210 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  26.326 BB    0.9991 7848.02246  121.34949  50.2794
   2  34.612 BB    1.3124 7760.80518   92.89912  49.7206

Totals :                  1.56088e4   214.24861

==========================================================================
                          *** End of Report ***
```

Data File C:\CHEM32\1\DATA\GZH\807B.D
Sample Name: 807B

```
    ======================================================================
    Acq. Operator   : GZH
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2017-11-21 10:43:21
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2017-11-21 9:55:41  by GZH
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2018-3-7 15:23:36  by LZ
                      (modified after loading)
    Sample Info     : IC H/I  70:30  1 mL/min, 254 nm
```

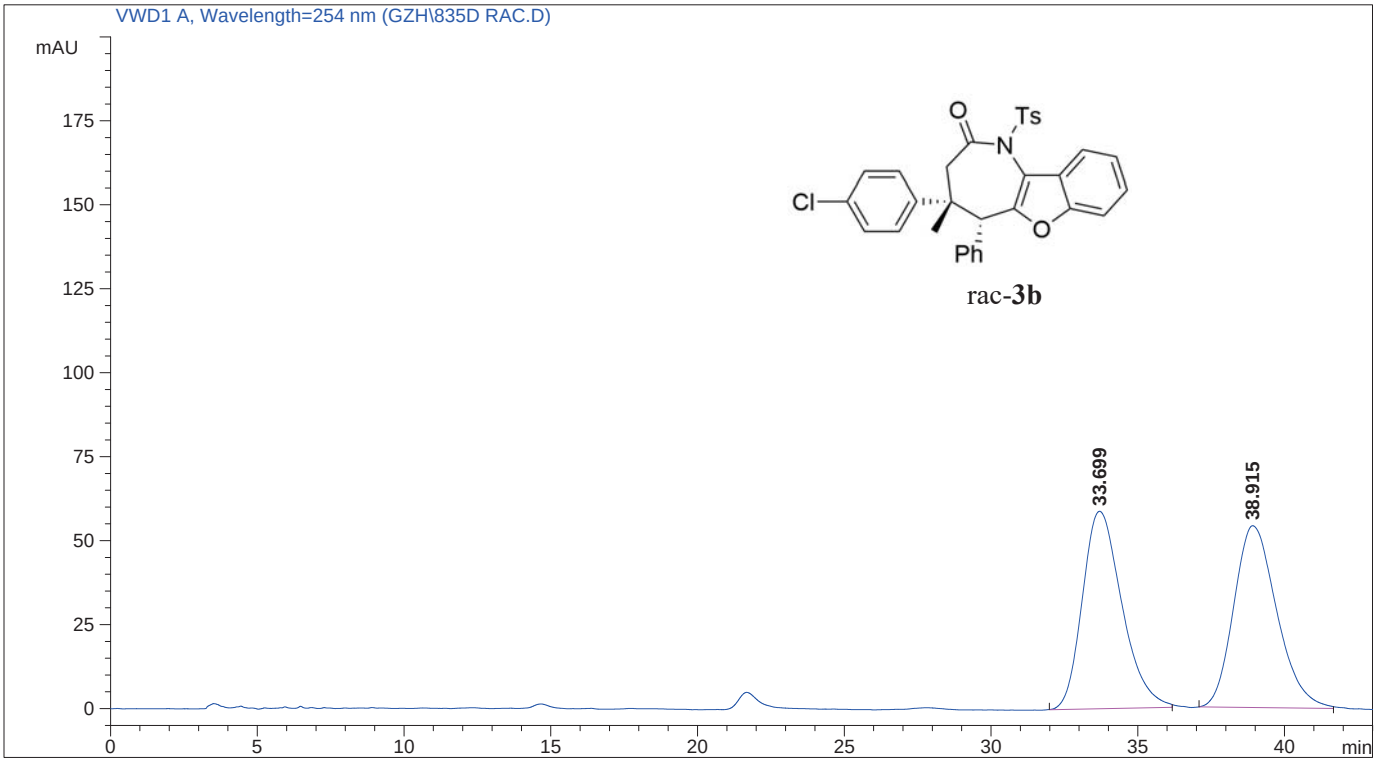


3a

```
    ======================================================================
                            Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  34.662 VB    1.2989 2.11020e4   245.73792 100.0000

    Totals :                  2.11020e4   245.73792


    ======================================================================
                            *** End of Report ***
```

Data File C:\CHEM32\1\DATA\GZH\835D RAC.D
Sample Name: 835D rac

```
    ========================================================================
    Acq. Operator   : gzh
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2017-12-20 16:46:36
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2017-12-20 14:52:44  by ZMB
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2018-2-28 12:39:12  by lz
                      (modified after loading)
    Sample Info     : IC H/I 70:30 1 mL/min, 254nm
```

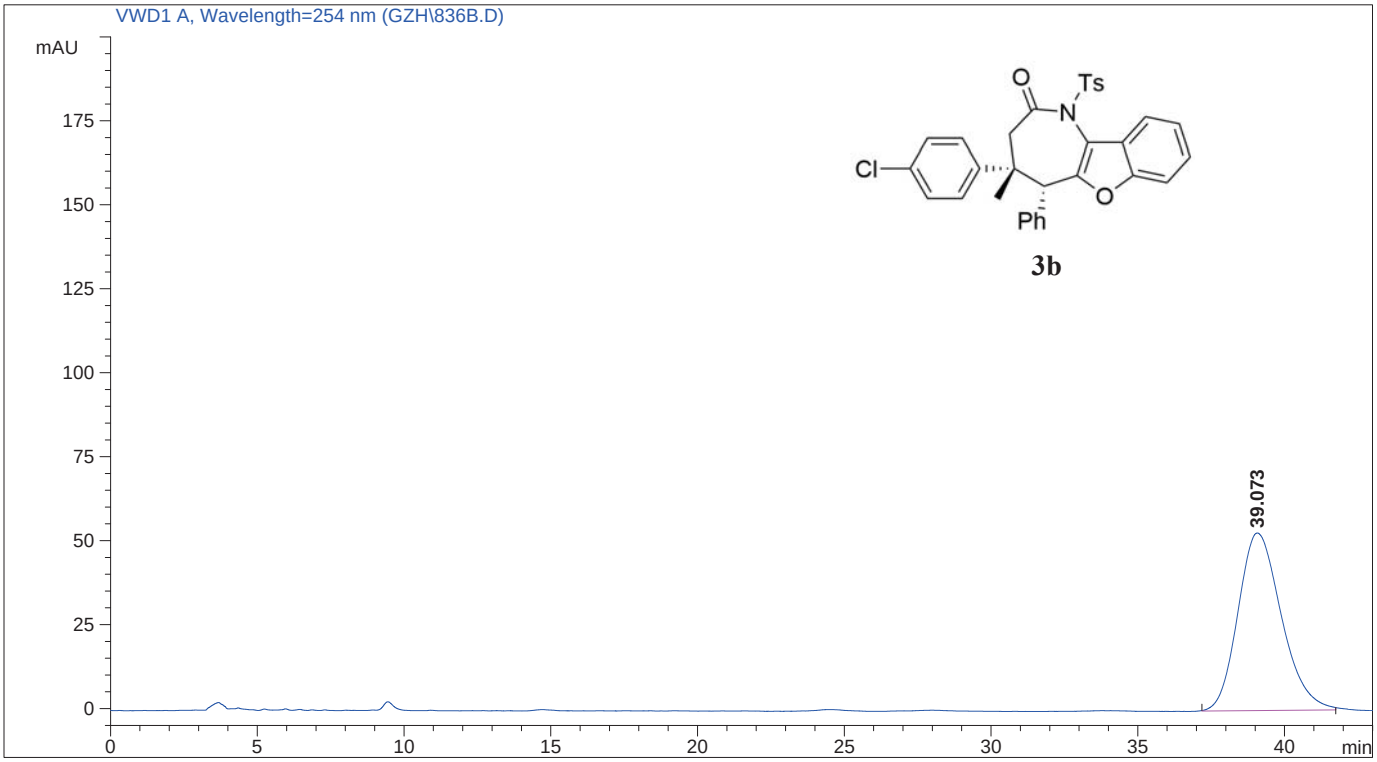


rac-**3b**

```
    ========================================================================
                              Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  33.699 BB    1.4479 5505.96533   58.79286  50.0233
       2  38.915 BB    1.5759 5500.83105   54.12525  49.9767

    Totals :                  1.10068e4   112.91811


    ========================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\GZH\836B.D
Sample Name: 836B 4-Cl

```
    ========================================================================
    Acq. Operator   : gzh
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2017-12-20 17:33:07
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2017-12-20 17:34:08  by gzh
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2018-2-28 12:40:11  by lz
                      (modified after loading)
    Sample Info     : IC H/I 70:30 1 mL/min, 254nm
```

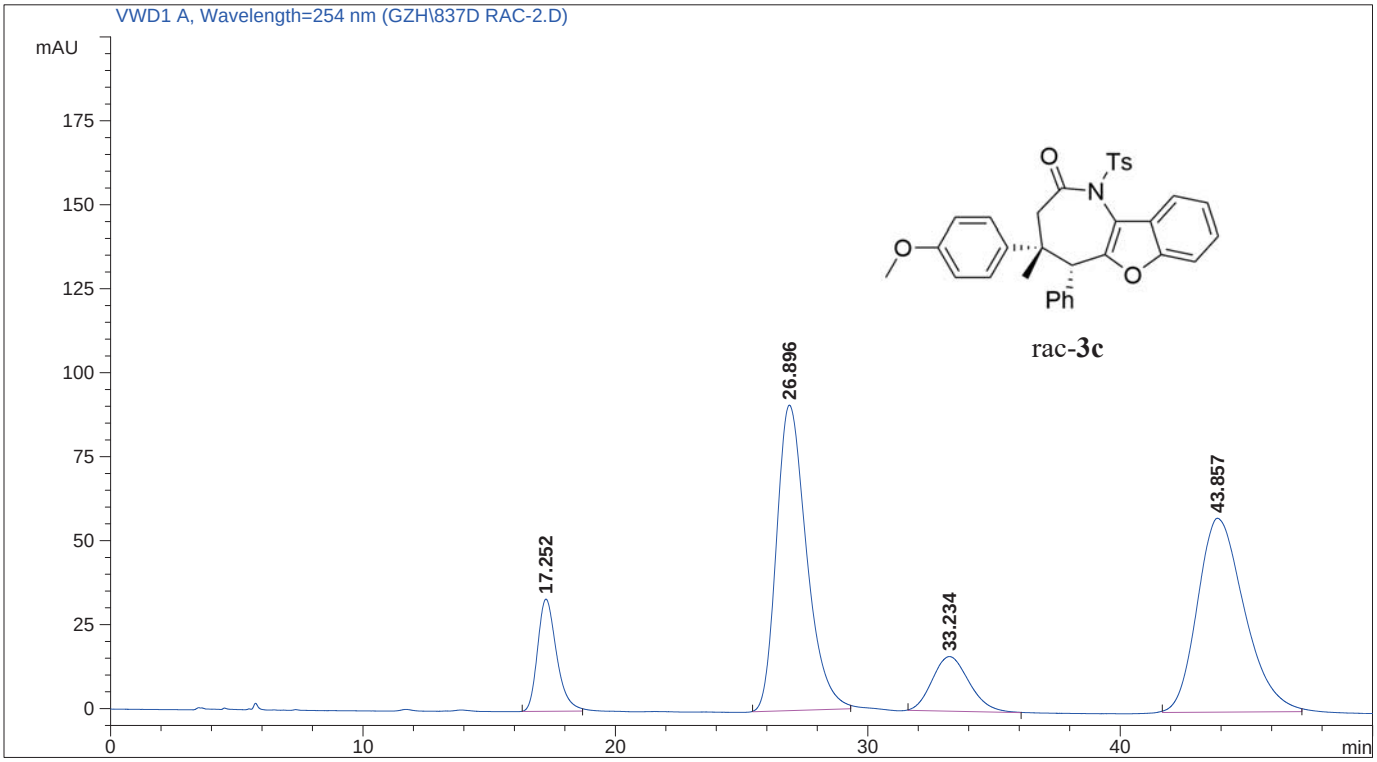


3b

```
    ========================================================================
                            Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  39.073 BB    1.5887 5431.23438   52.87089 100.0000

    Totals :                  5431.23438   52.87089


    ========================================================================
                            *** End of Report ***
```

Data File C:\CHEM32\1\DATA\GZH\837D RAC-2.D
Sample Name: 837D rac

```
    ========================================================================
    Acq. Operator    : gzh
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date   : 2017-12-21 9:12:03
    Acq. Method      : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2017-12-21 8:48:29  by gzh
                       (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2018-2-28 11:11:27  by lz
                       (modified after loading)
    Sample Info      : IC H/I 60:40 1 mL/min, 254nm
```

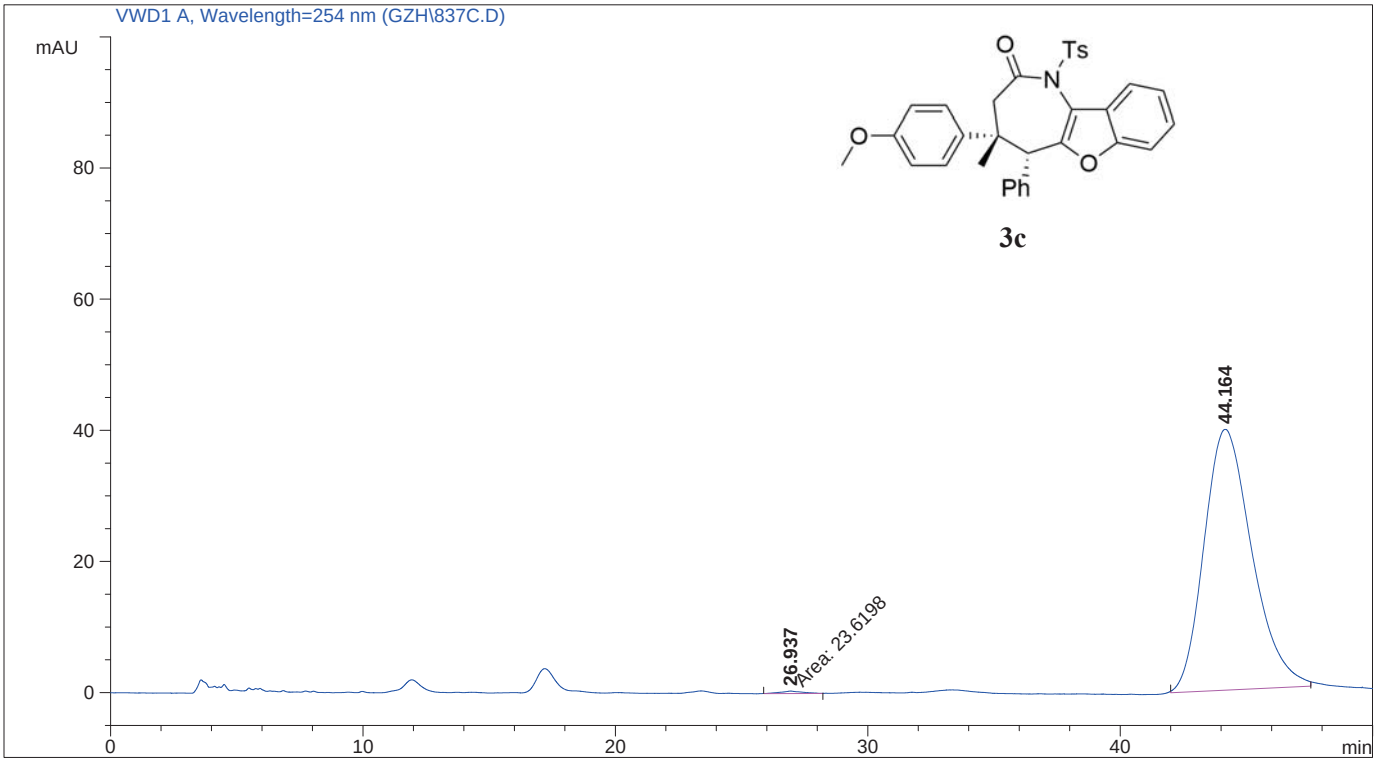


rac-**3c**

```
    ========================================================================
                              Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  17.252 BB    0.8013 1744.57556   33.41561   9.4867
       2  26.896 BB    1.2560 7521.49316   90.95061  40.9004
       3  33.234 BB    1.6101 1651.76941   16.26240   8.9820
       4  43.857 BB    1.9816 7471.91943   57.73475  40.6309

    Totals :                  1.83898e4   198.36338


    ========================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\GZH\837C.D
Sample Name: 837C

```
    ========================================================================
    Acq. Operator    : gzh
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date   : 2017-12-21 10:06:49
    Acq. Method      : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2017-12-21 8:48:29  by gzh
                       (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2018-2-28 11:13:18  by lz
                       (modified after loading)
    Sample Info      : IC H/I 60:40 1 mL/min, 254nm
```

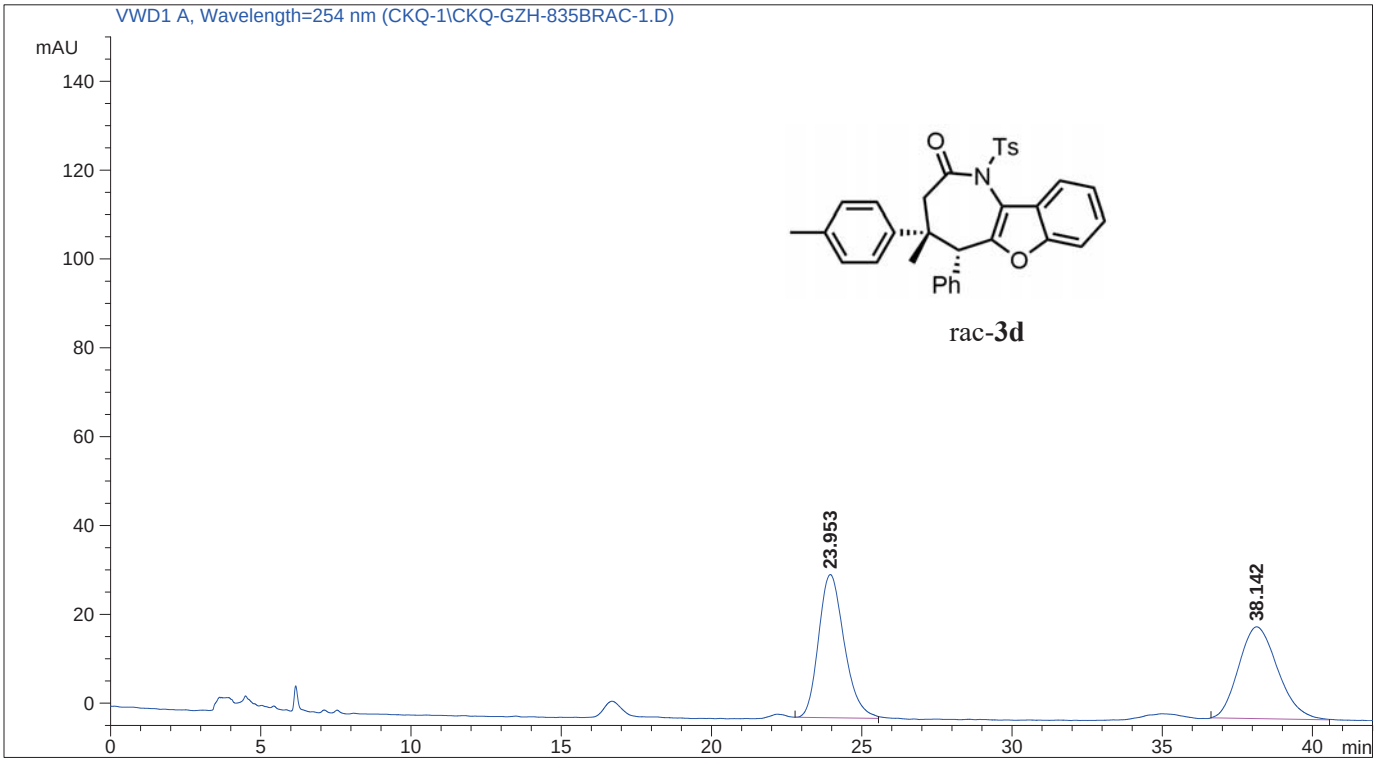


3c

```
    ========================================================================
                              Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  26.937 MM    1.0329   23.61984 3.81139e-1   0.4478
       2  44.164 BB    2.0469 5251.01221   39.77457  99.5522

    Totals :                  5274.63205   40.15571


    ========================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\CKQ-GZH-835BRAC-1.D
Sample Name: CKQ-GZH-835BRAC-1

```
    ======================================================================
    Acq. Operator   : ckq
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2017-12-29 10:50:56
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2017-12-29 10:40:17  by ckq
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2018-2-28 11:44:38  by lz
                      (modified after loading)
    Sample Info     : ic-70:30 1.0 mL/min, 254nm
```

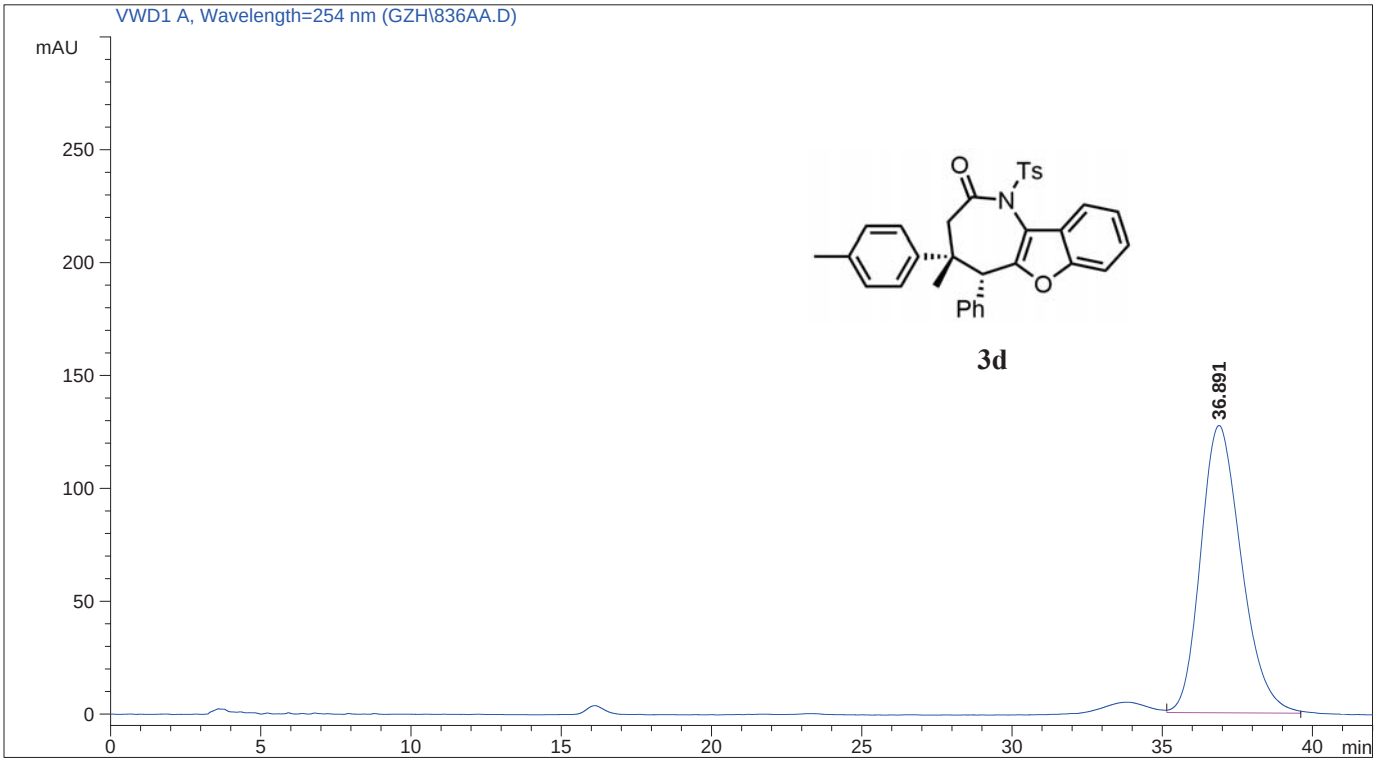


rac-**3d**

```
    ======================================================================
                               Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  23.953 BB    0.9246 1910.47778   32.22716  50.5944
       2  38.142 BB    1.3843 1865.58752   20.68359  49.4056

    Totals :                  3776.06531   52.91075


    ======================================================================
                               *** End of Report ***
```

Data File C:\CHEM32\1\DATA\GZH\836AA.D
Sample Name: 836AA

```
    ======================================================================
    Acq. Operator   : gzh
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2017-12-20 16:01:16
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2017-12-20 14:52:44  by ZMB
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2018-2-28 11:45:22  by lz
                      (modified after loading)
    Sample Info     : IC H/I 70:30 1 mL/min, 254nm
```

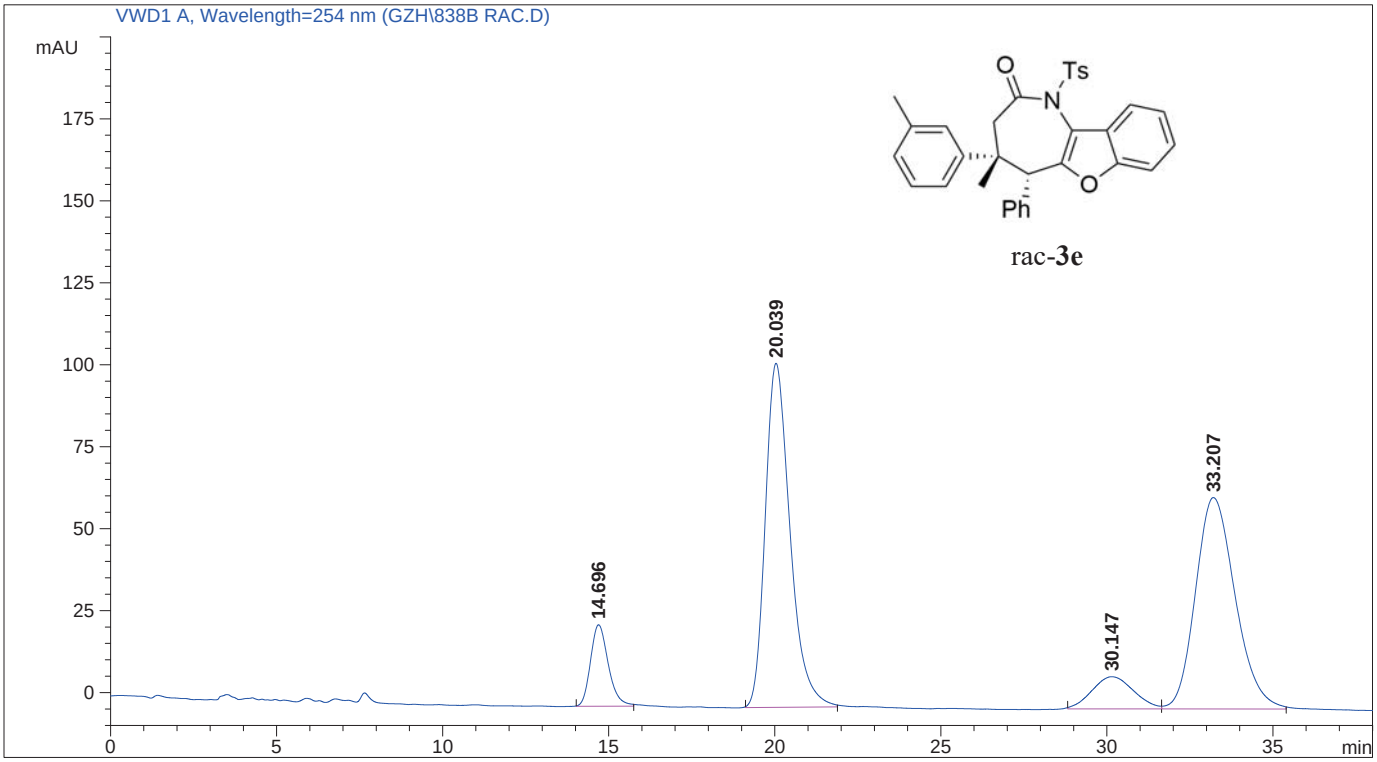


3d

```
    ======================================================================
                            Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  36.891 BB    1.4583 1.19700e4   127.27282 100.0000

    Totals :                  1.19700e4   127.27282


    ======================================================================
                            *** End of Report ***
```

Data File C:\CHEM32\1\DATA\GZH\838B RAC.D
Sample Name: 838B RAC

```
==========================================================================
Acq. Operator   : GZH
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 2018-2-28 14:36:21
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2018-2-28 14:33:34  by GZH
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2018-2-28 15:54:25  by GZH
                  (modified after loading)
Sample Info     : IC H/I =70/30 1.0 mL/min, 254 nm
```

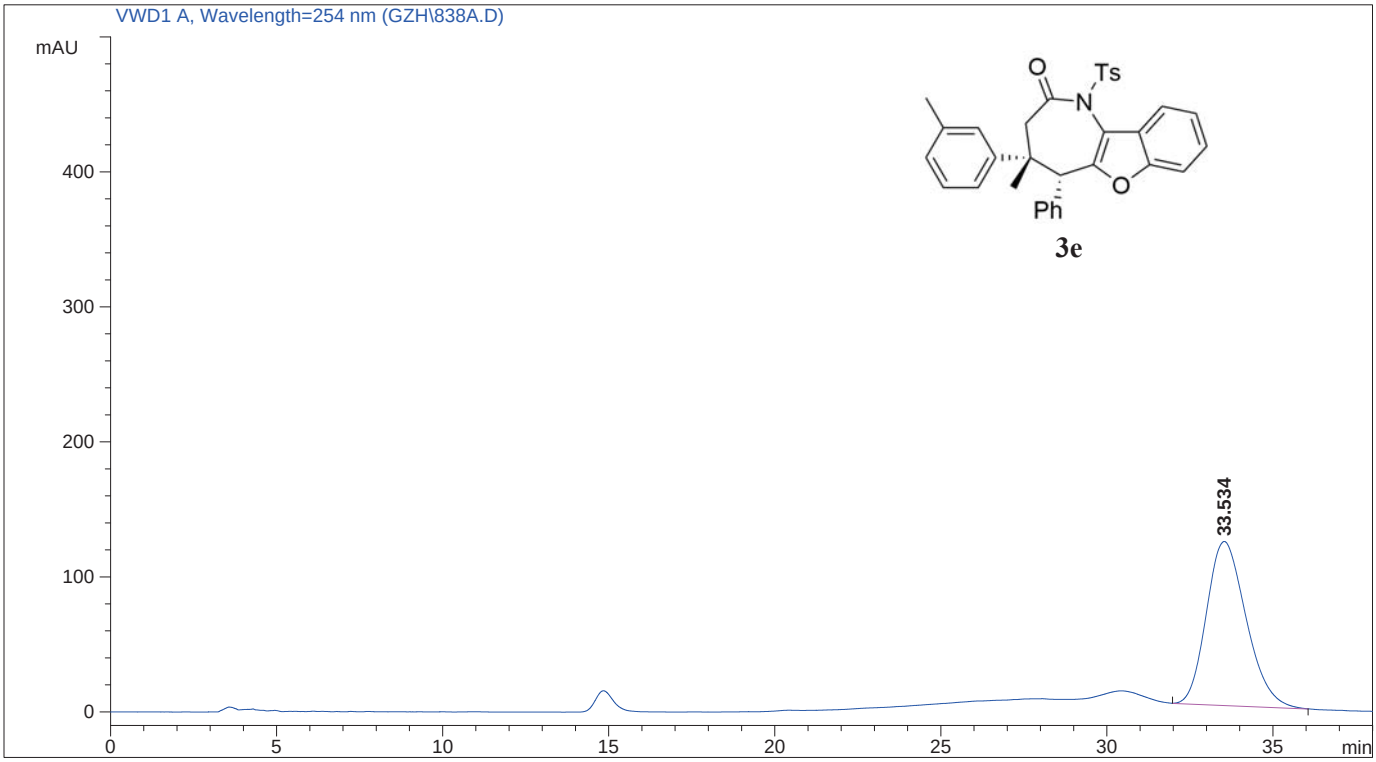


rac-**3e**

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  14.696 BB    0.5746  926.71936   24.86389   7.3757
   2  20.039 BB    0.7957 5427.34717  104.91295  43.1960
   3  30.147 BV    1.3874  853.22473    9.81036   6.7908
   4  33.207 VB    1.2840 5357.17041   64.46886  42.6375

Totals :                  1.25645e4   204.05607


==========================================================================
                          *** End of Report ***
```

Data File C:\CHEM32\1\DATA\GZH\838A.D
Sample Name: 838A

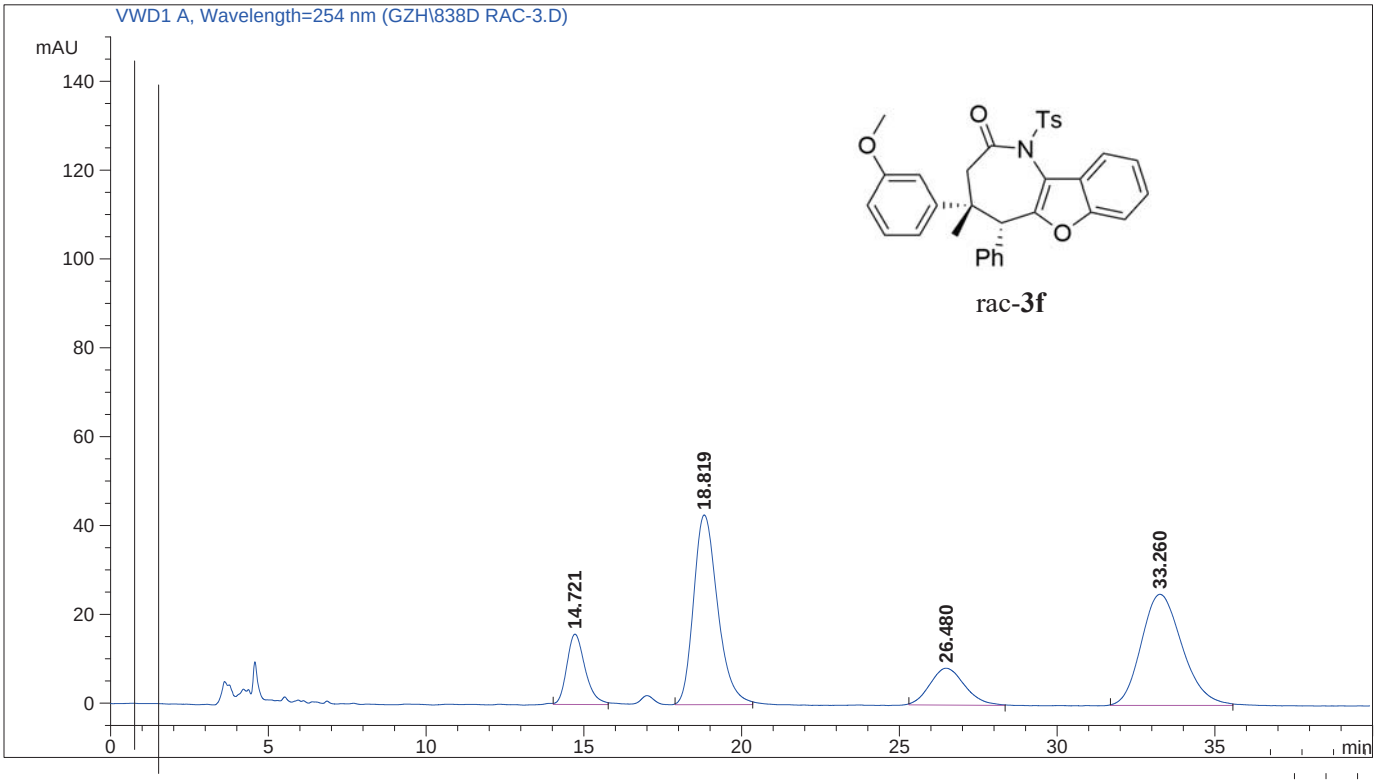
```
    ======================================================================
    Acq. Operator   : GZH
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2018-2-28 15:17:49
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2018-2-28 15:54:25  by GZH
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2018-2-28 16:00:49  by GZH
                      (modified after loading)
    Sample Info     : IC H/I =70/30 1.0 mL/min, 254 nm
```




```
    ======================================================================
                            Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  33.534 VB    1.2915 1.01216e4   121.59506 100.0000

    Totals :                  1.01216e4   121.59506

    ======================================================================
                            *** End of Report ***
```

Data File C:\CHEM32\1\DATA\GZH\838D RAC-3.D
Sample Name: 838D rac-3

```
    ========================================================================
    Acq. Operator    : gzh
    Acq. Instrument  : Instrument 1                    Location : Vial 1
    Injection Date   : 2017-12-21 16:09:33
    Acq. Method      : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2017-12-21 16:08:55  by gzh
                       (modified after loading)
    Analysis Method  : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2018-2-28 12:41:50  by lz
                       (modified after loading)
    Sample Info      : IC H/I 60:40 1 mL/min, 254nm
```

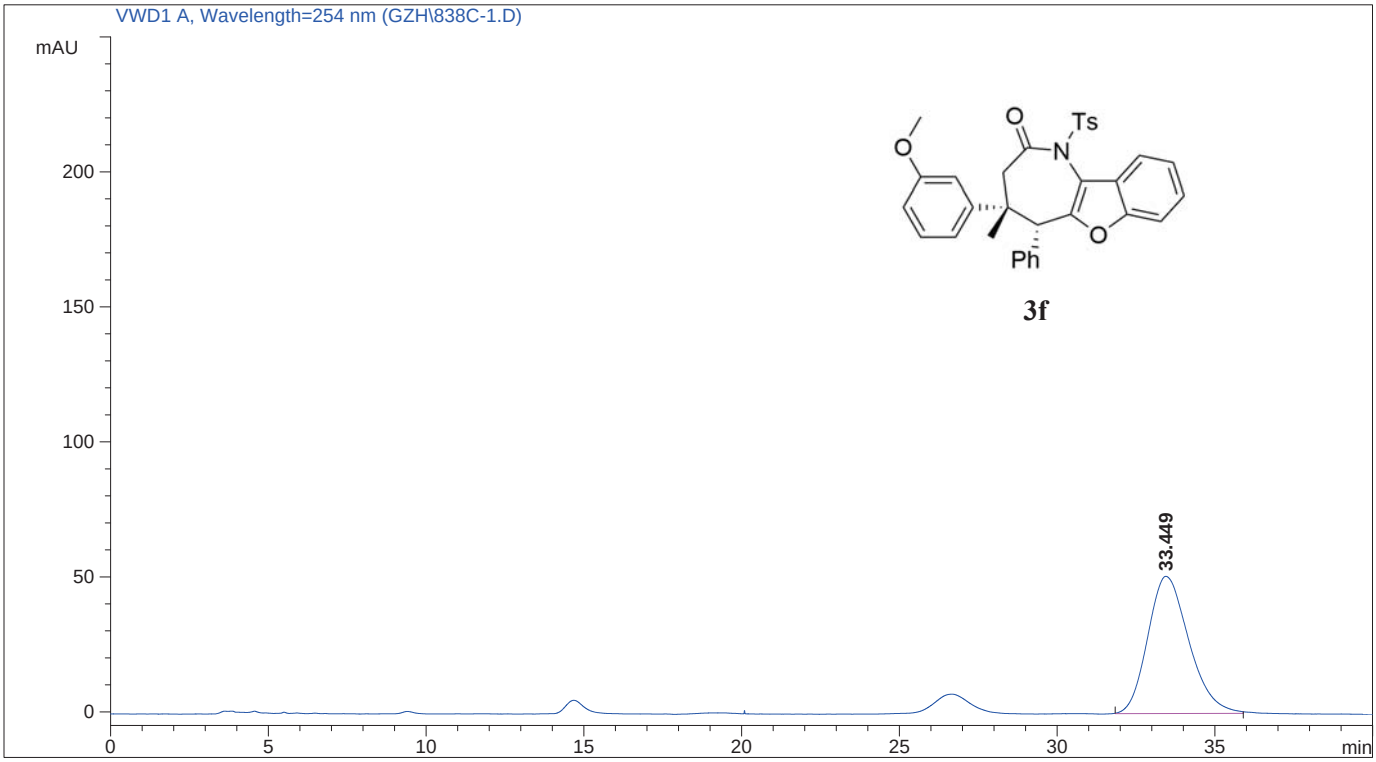


rac-**3f**

```
    ========================================================================
                               Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  14.721 BB    0.6144  636.57422   15.84060  10.9928
       2  18.819 BB    0.8109 2264.49072   42.69736  39.1049
       3  26.480 BB    1.1512  636.88281    8.30277  10.9982
       4  33.260 BB    1.3885 2252.86597   25.01568  38.9041

    Totals :                  5790.81372   91.85640


    ========================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\GZH\838C-1.D
Sample Name: 838C-1

```
    ======================================================================
    Acq. Operator   : gzh
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2017-12-21 12:55:38
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2017-12-21 12:56:18  by gzh
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2018-2-28 12:44:07  by lz
                      (modified after loading)
    Sample Info     : IC H/I 60:40 1 mL/min, 254nm
```

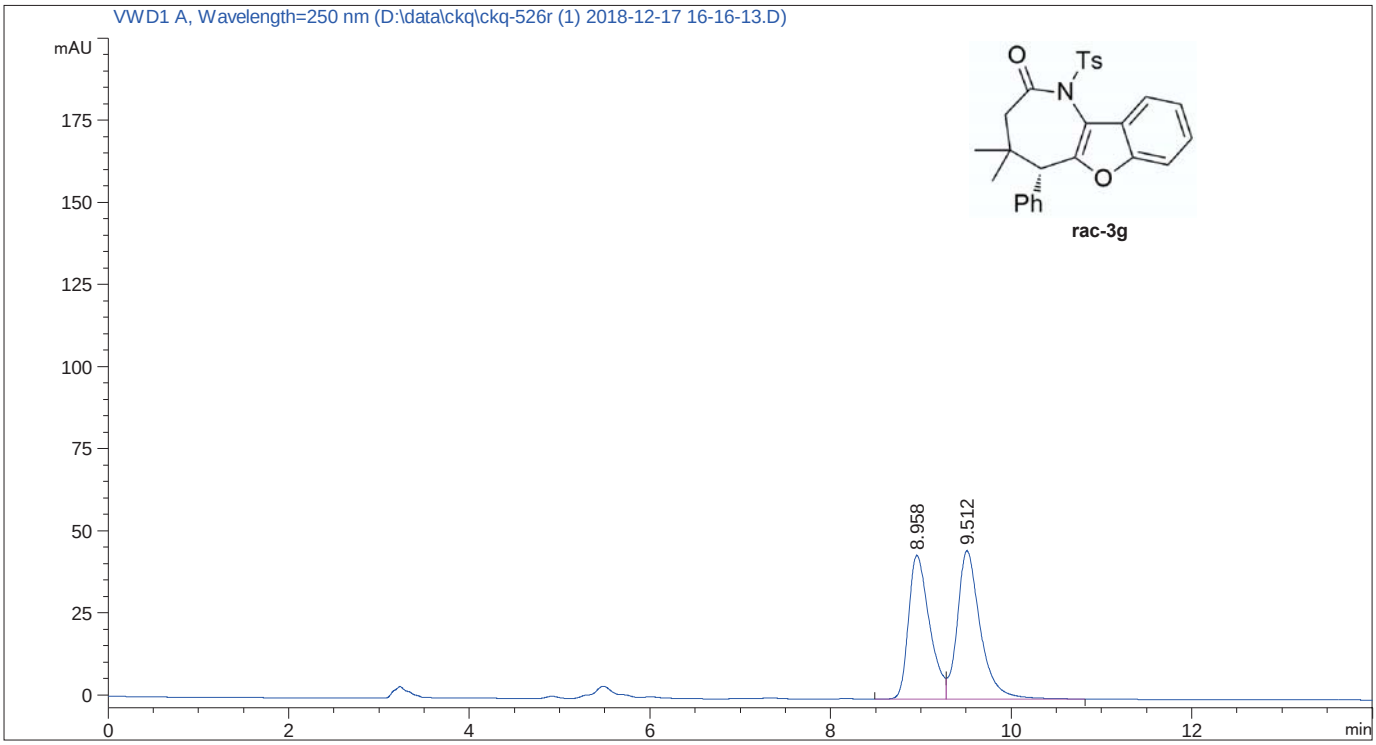


3f

```
    ======================================================================
                            Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  33.449 BB    1.4250 4609.53369   50.82317 100.0000

    Totals :                  4609.53369   50.82317

    ======================================================================
                            *** End of Report ***
```

Data File D:\data\ckq\ckq-526r (1) 2018-12-17 16-16-13.D
Sample Name: ckq-526r

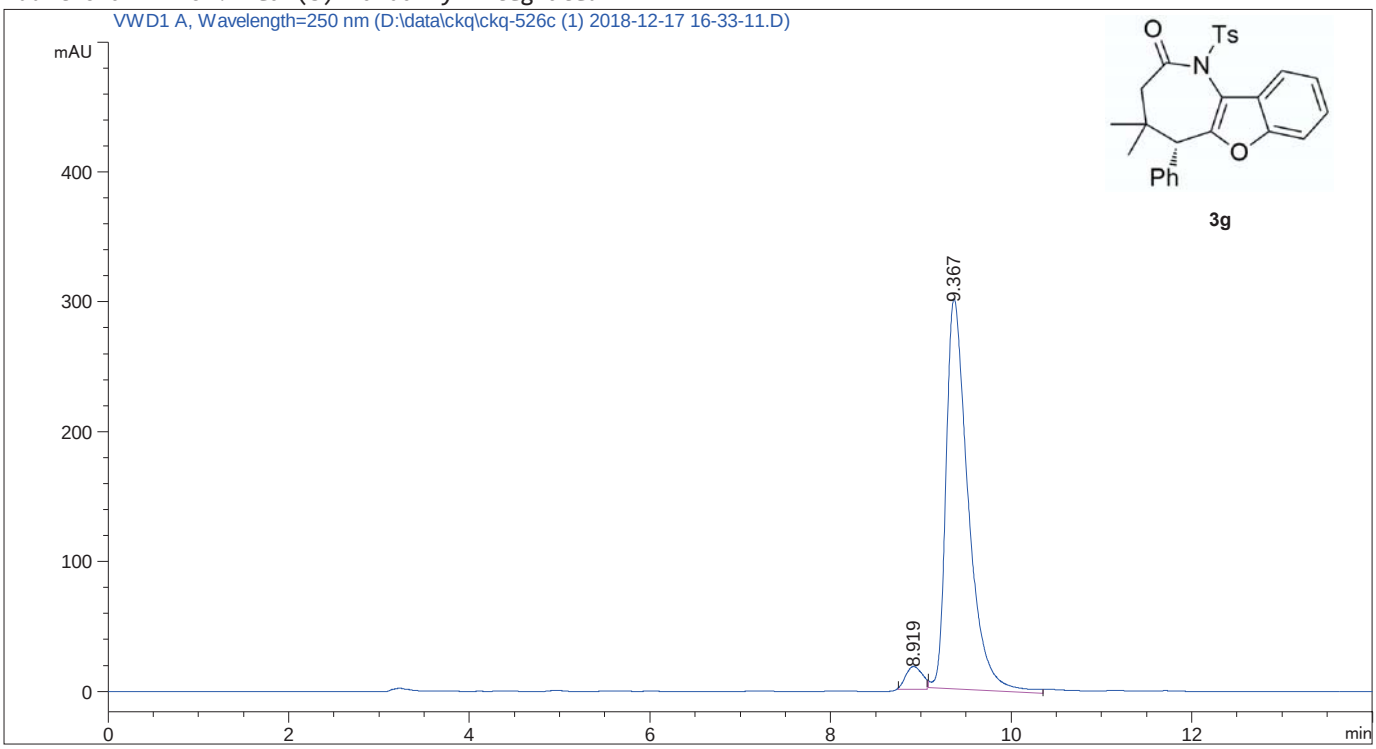
```
======================================================================
Acq. Operator   : ckq
Sample Operator : ckq
Acq. Instrument : HPLC                          Location :   P1-D-06
Injection Date  : 12/17/2018 4:16:54 PM              Inj :   1
                                                Inj Volume : 5.000 µl
Different Inj Volume from Sample Entry!  Actual Inj Volume : 10.000 µl
Acq. Method     : C:\Users\Public\Documents\ChemStation\1\Methods
Last changed    \DEF_LC.M : 12/17/2018 10:36:48 AM by ckq
                  (modified after loading)
Analysis Method : C:\Users\Public\Documents\ChemStation\1\Methods\DEF_LC.M
Last changed    : 12/18/2018 11:26:29 by  ckq
          (modified after loading)
Sample Info     : b, H/I=95/5  nm,1 mL/min254nm
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=250 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.958 BV    0.2450  708.91547   43.76809  49.0312
   2   9.512 VB    0.2633  709.91370   45.15989  50.9688
```

Data File D:\data\ckq\ckq-526c (1) 2018-12-17 16-33-11.D
Sample Name: ckq-526c

```
======================================================================
Acq. Operator   : ckq
Sample Operator : ckq
Acq. Instrument : HPLC                          Location :   P1-D-07
Injection Date  : 12/17/2018 4:33:51 PM              Inj :   1


Acq. Method     : C:\Users\Public\Documents\ChemStation\1\Methods
Last changed    \DEF_LC.M : 12/17/2018 10:36:48 AM by ckq
                  (modified after loading)
Analysis Method : C:\Users\Public\Documents\ChemStation\1\Methods\DEF_LC.M
Last changed    : 12/18/2018 11:25:44 AM
        by ckq
Sample Info(modified after loading)
        : Ib, H/I=95/5  nm,1 mL/min254nm
Additional Info : Peak(s) manually integrated
```

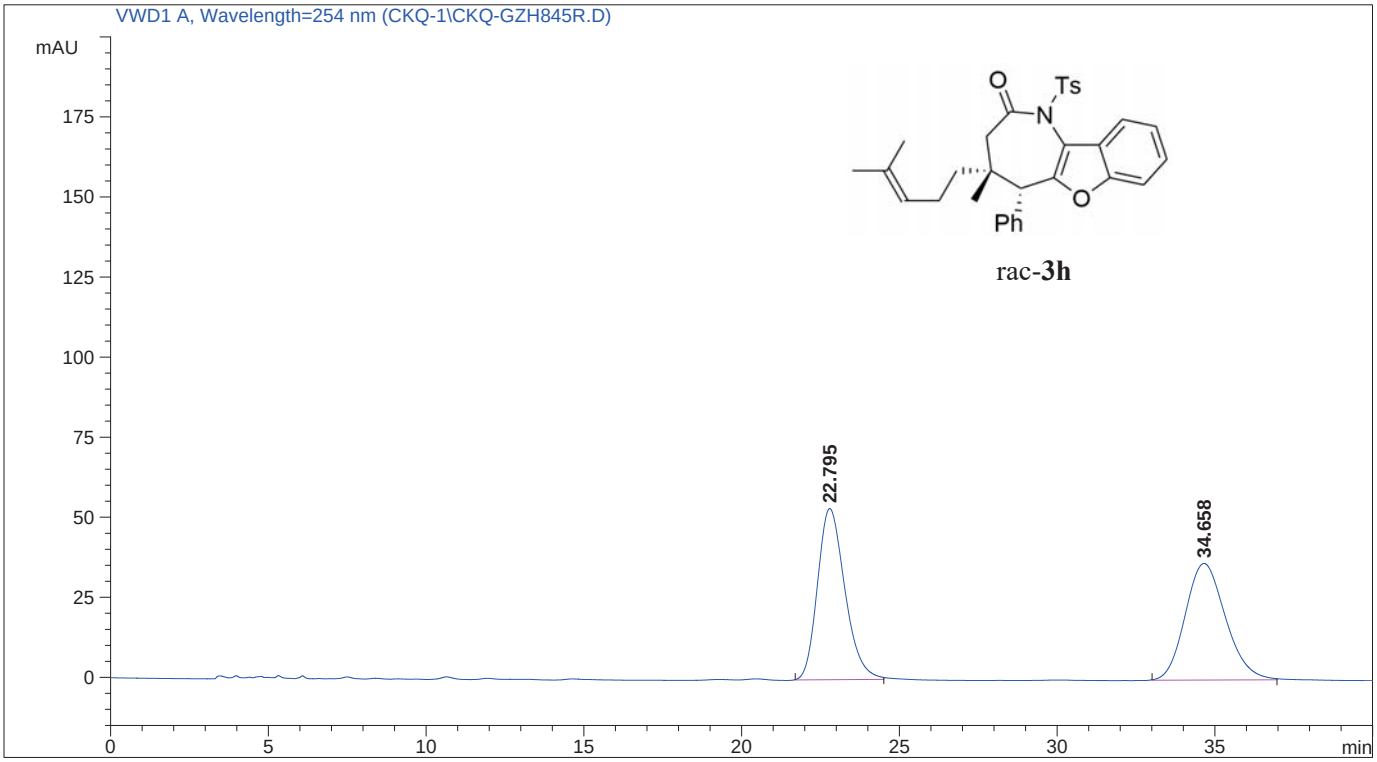



```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=250 nm
```

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.919	MM	0.2119	220.97455	17.37925	4.1411
2	9.367	MM	0.2842	5115.20898	299.93854	95.8589

Data File C:\CHEM32\1\DATA\CKQ-1\CKQ-GZH845R.D
Sample Name: CKQ-gzh-845r

```
    ======================================================================
    Acq. Operator   : ckq
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2018-1-3 10:11:35
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2018-1-3 10:02:55  by ckq
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2018-2-28 11:50:41  by lz
                      (modified after loading)
    Sample Info     : ic-70:30 1 mL/min, 254nm
```

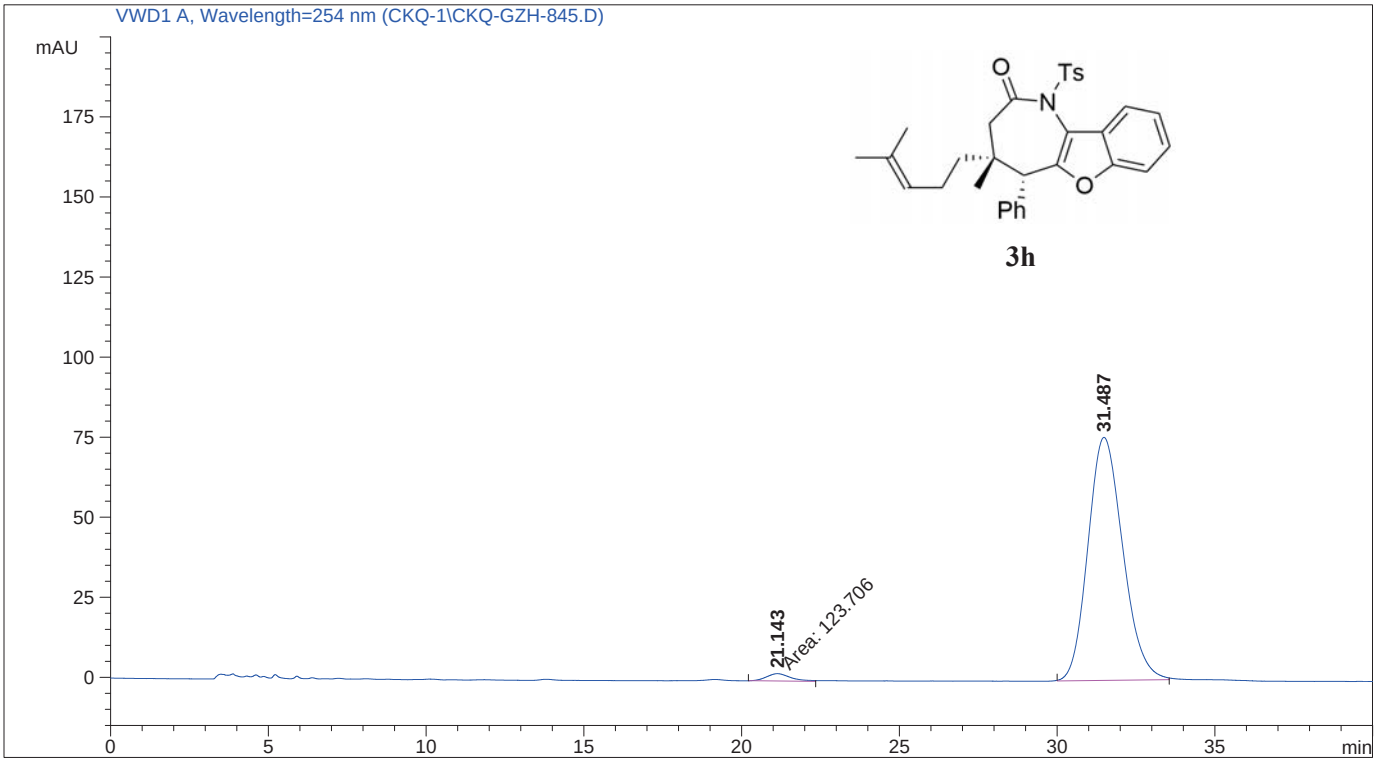


rac-**3h**

```
    ======================================================================
                               Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  22.795 BB    0.9317 3226.53101   53.42978  49.9620
       2  34.658 BB    1.3615 3231.43408   36.41167  50.0380

    Totals :                  6457.96509   89.84145


    ======================================================================
                               *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\CKQ-GZH-845.D
Sample Name: ckq-gzh-845

```
    ======================================================================
    Acq. Operator    : ckq
    Acq. Instrument  : Instrument 1                    Location : Vial 1
    Injection Date   : 2018-1-4 8:46:18
    Acq. Method      : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2018-1-4 8:34:27  by zzf
                       (modified after loading)
    Analysis Method  : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2018-2-28 11:52:06  by lz
                       (modified after loading)
    Sample Info      : ic-70:30 1 mL/min, 254nm
```

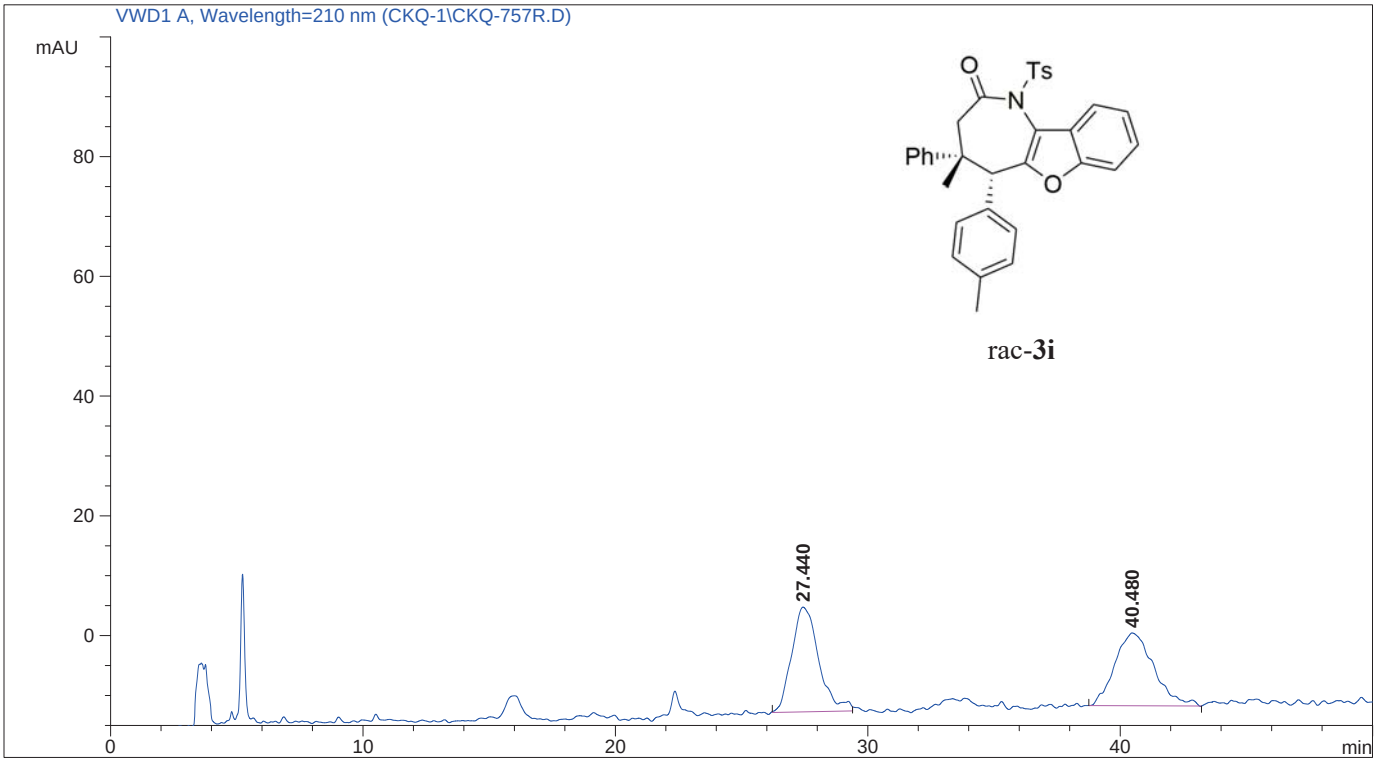


3h

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  21.143 MM    0.8947  123.70618    2.30439   2.0768
       2  31.487 BB    1.1937 5832.80225   75.88766  97.9232

    Totals :                  5956.50843   78.19205


    ======================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\CKQ-757R.D
Sample Name: CKQ-757r

```
    ========================================================================
    Acq. Operator    : CKQ
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date   : 2017-11-29 17:01:09
    Acq. Method      : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2017-11-29 14:26:04  by CKQ
                       (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2018-3-13 18:40:07  by LZ
                       (modified after loading)
    Sample Info      : IcH/I  70:30  1 mL/min, 254 nm
```

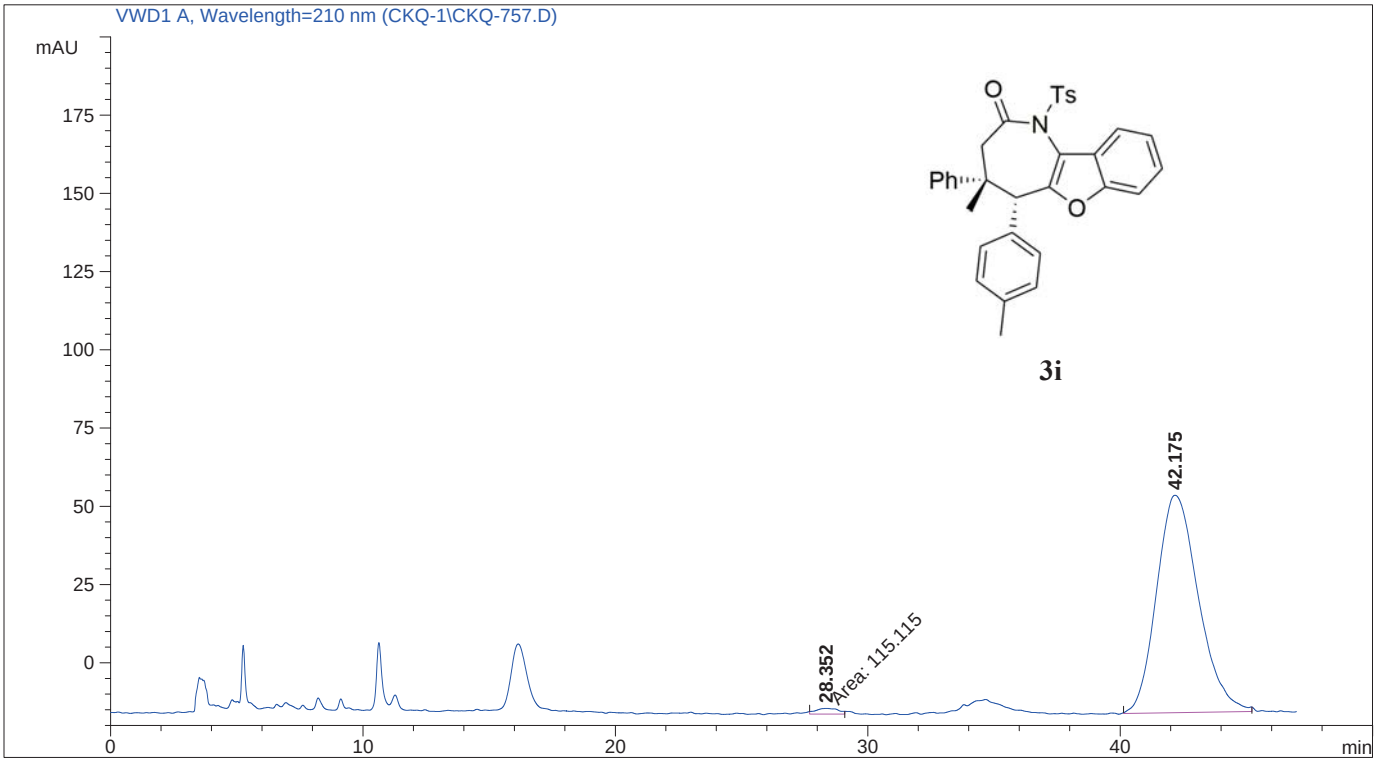


rac-**3i**

```
    ========================================================================
                              Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  27.440 BB    1.1105 1307.81189   17.50349  50.0572
       2  40.480 BB    1.4092 1304.82056   12.15891  49.9428

    Totals :                  2612.63245   29.66240


    ========================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\CKQ-757.D
Sample Name: CKQ-757

```
    ======================================================================
    Acq. Operator    : CKQ
    Acq. Instrument  : Instrument 1                    Location : Vial 1
    Injection Date   : 2017-11-29 16:13:26
    Acq. Method      : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2017-11-29 14:26:04  by CKQ
                       (modified after loading)
    Analysis Method  : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2018-3-13 18:38:49  by LZ
                       (modified after loading)
    Sample Info      : IcH/I  70:30  1 mL/min, 254 nm
```

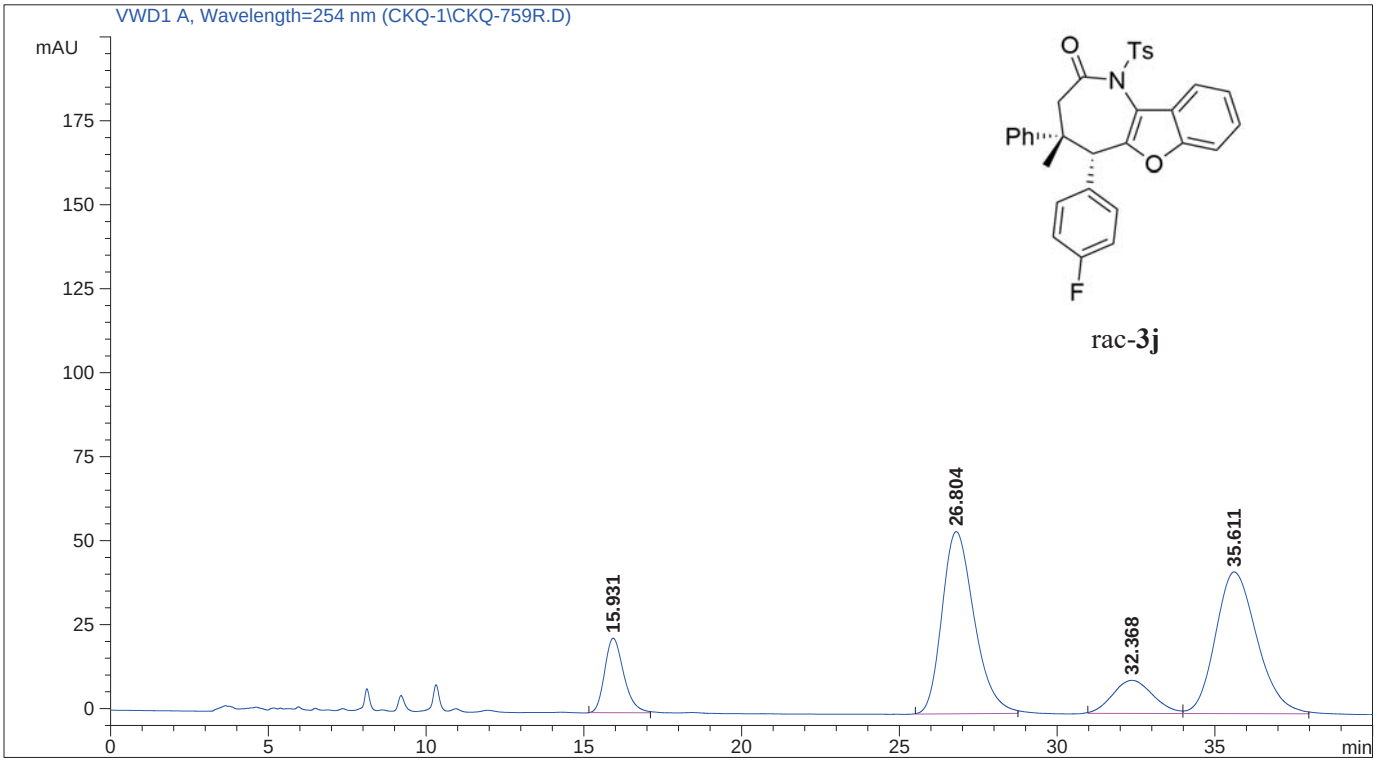


3i

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  28.352 MM    1.0566  115.11494    1.81573   1.4173
       2  42.175 BB    1.7409 8006.81445   69.45690  98.5827

    Totals :                  8121.92939   71.27263


    ======================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\CKQ-759R.D
Sample Name: CKQ-759R

```
    ========================================================================
    Acq. Operator    : ckq
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date   : 2017-12-22 9:04:42
    Acq. Method      : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2017-12-22 8:47:38  by gzh
                       (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2018-6-12 8:48:43  by LZ
                       (modified after loading)
    Sample Info      : IC H/I 70:30 1 mL/min, 254nm
```

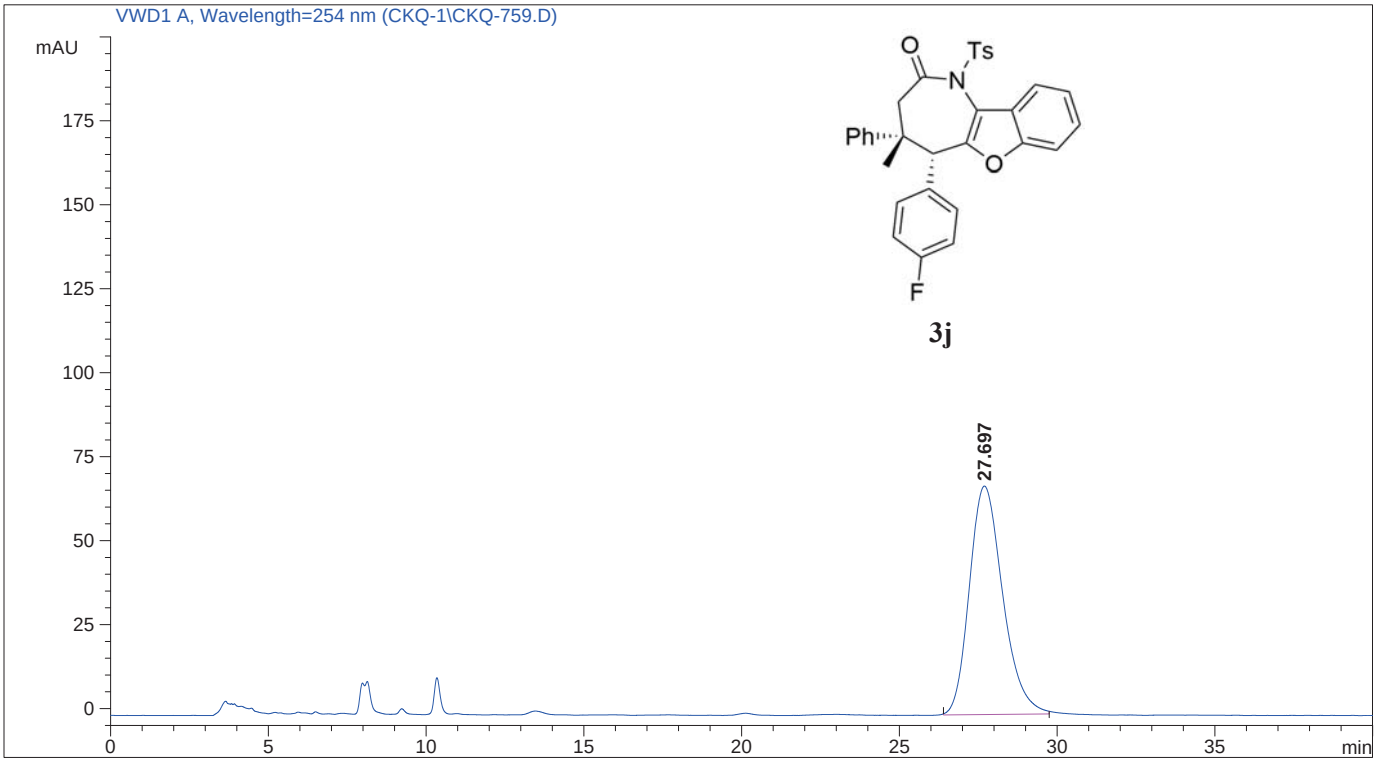


rac-**3j**

```
    ========================================================================
                              Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  15.931 BB    0.6474  932.00079   22.18374   9.7906
       2  26.804 BB    1.0916 3857.43115   54.25503  40.5219
       3  32.368 BV    1.3638  878.61279    9.87854   9.2297
       4  35.611 VB    1.4199 3851.33423   42.20288  40.4578

    Totals :                  9519.37897  128.52019


    ========================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\CKQ-759.D
Sample Name: CKQ-759

```
    ========================================================================
    Acq. Operator    : ckq
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date   : 2017-12-22 10:01:06
    Acq. Method      : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2017-12-22 8:47:38  by gzh
                       (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2018-6-12 8:48:08  by LZ
                       (modified after loading)
    Sample Info      : IC H/I 70:30 1 mL/min, 254nm
```

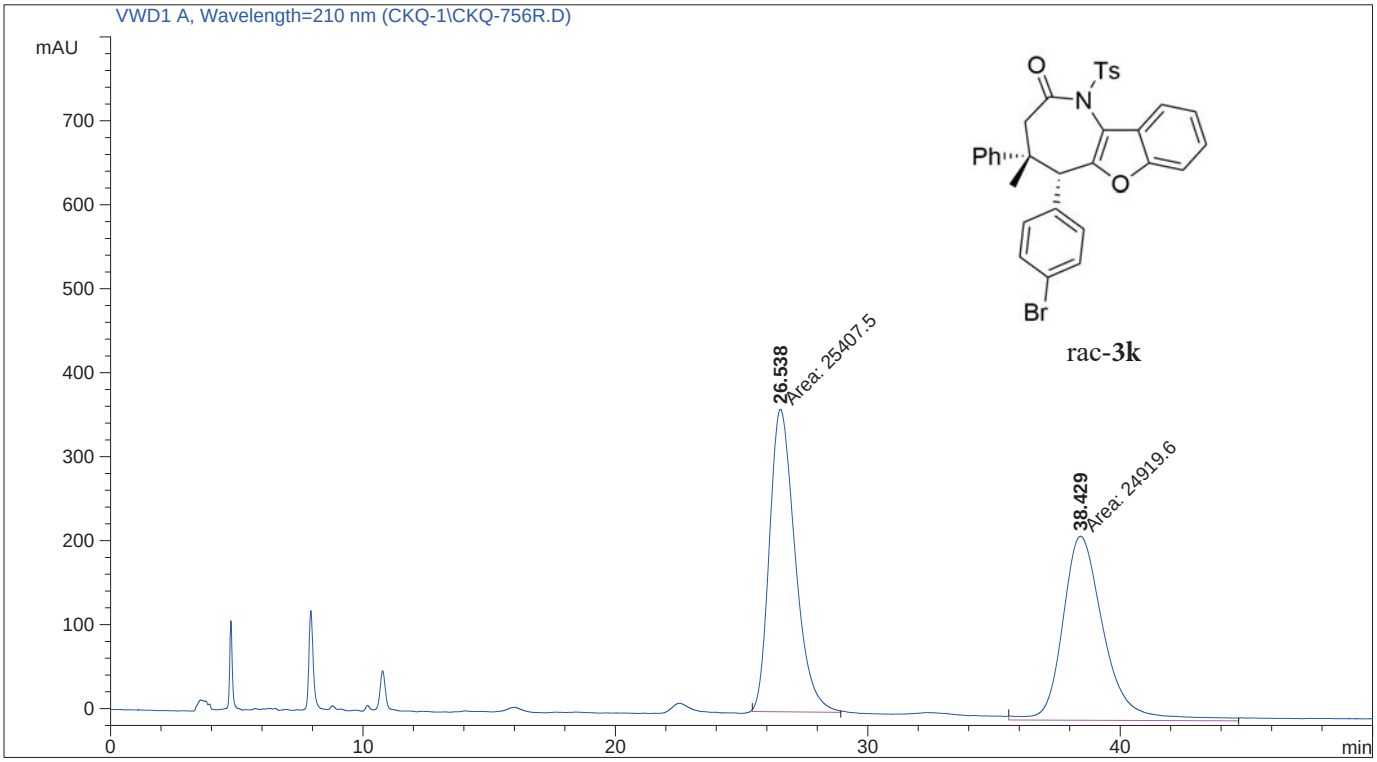


3j

```
    ========================================================================
                            Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  27.697 BB    1.1315 5003.79736   68.05922 100.0000

    Totals :                  5003.79736   68.05922

    ========================================================================
                            *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\CKQ-756R.D
Sample Name: CKQ-756r

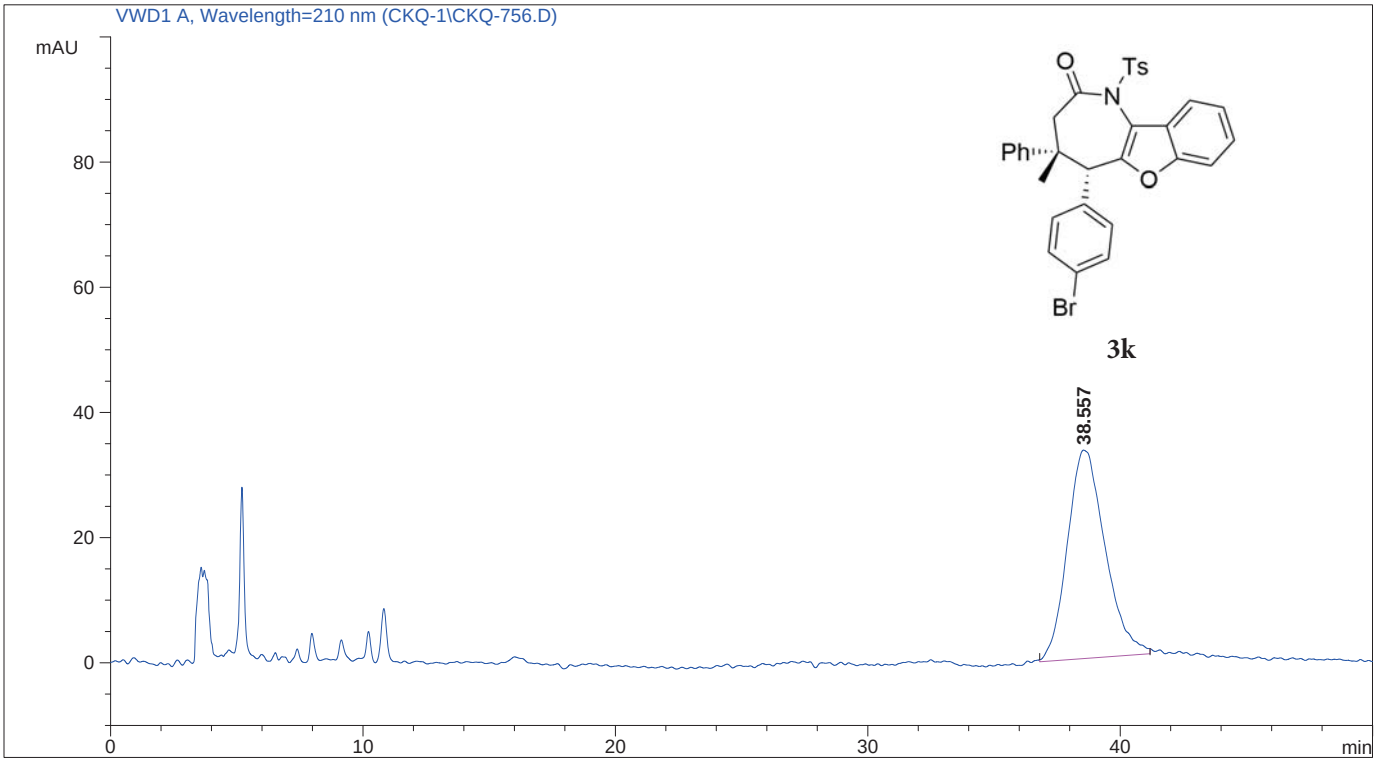
```
    ======================================================================
    Acq. Operator   : CKQ
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2017-11-29 14:33:36
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2017-11-29 14:26:04  by CKQ
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2018-3-13 17:30:51  by LZ
                      (modified after loading)
    Sample Info     : IcH/I  70:30  1 mL/min, 254 nm
```




```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  26.538 MM    1.1753 2.54075e4   360.28333  50.4847
       2  38.429 MM    1.8947 2.49196e4   219.20120  49.5153

    Totals :                  5.03271e4   579.48453


    ======================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\CKQ-756.D
Sample Name: CKQ-756

```
    ======================================================================
    Acq. Operator    : CKQ
    Acq. Instrument  : Instrument 1                    Location : Vial 1
    Injection Date   : 2017-11-29 11:02:35
    Acq. Method      : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2017-11-29 9:40:20  by CKQ
                       (modified after loading)
    Analysis Method  : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2018-3-13 17:32:21  by LZ
                       (modified after loading)
    Sample Info      : IcH/I  70:30  1 mL/min, 254 nm
```

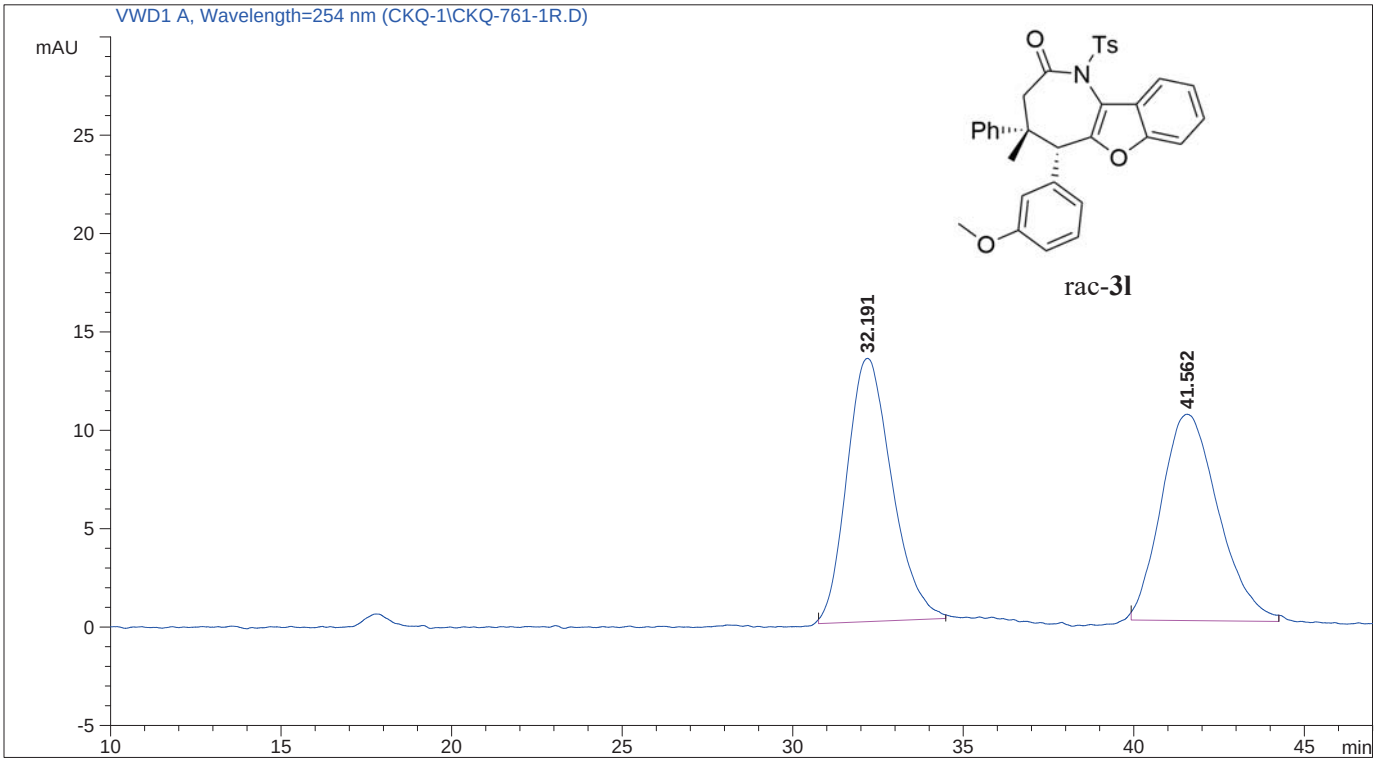


3k

```
    ======================================================================
                            Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  38.557 BB    1.4452 3459.05957   33.28918 100.0000

    Totals :                  3459.05957   33.28918

    ======================================================================
                            *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\CKQ-761-1R.D
Sample Name: CKQ-761-1r

```
    ========================================================================
    Acq. Operator    : ckq
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date   : 2018-1-2 19:27:28
    Acq. Method      : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2018-1-2 19:25:39  by ckq
                       (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2018-3-13 17:44:35  by LZ
                       (modified after loading)
    Sample Info      : ic-70:30 1.1 mL/min, 254nm
```

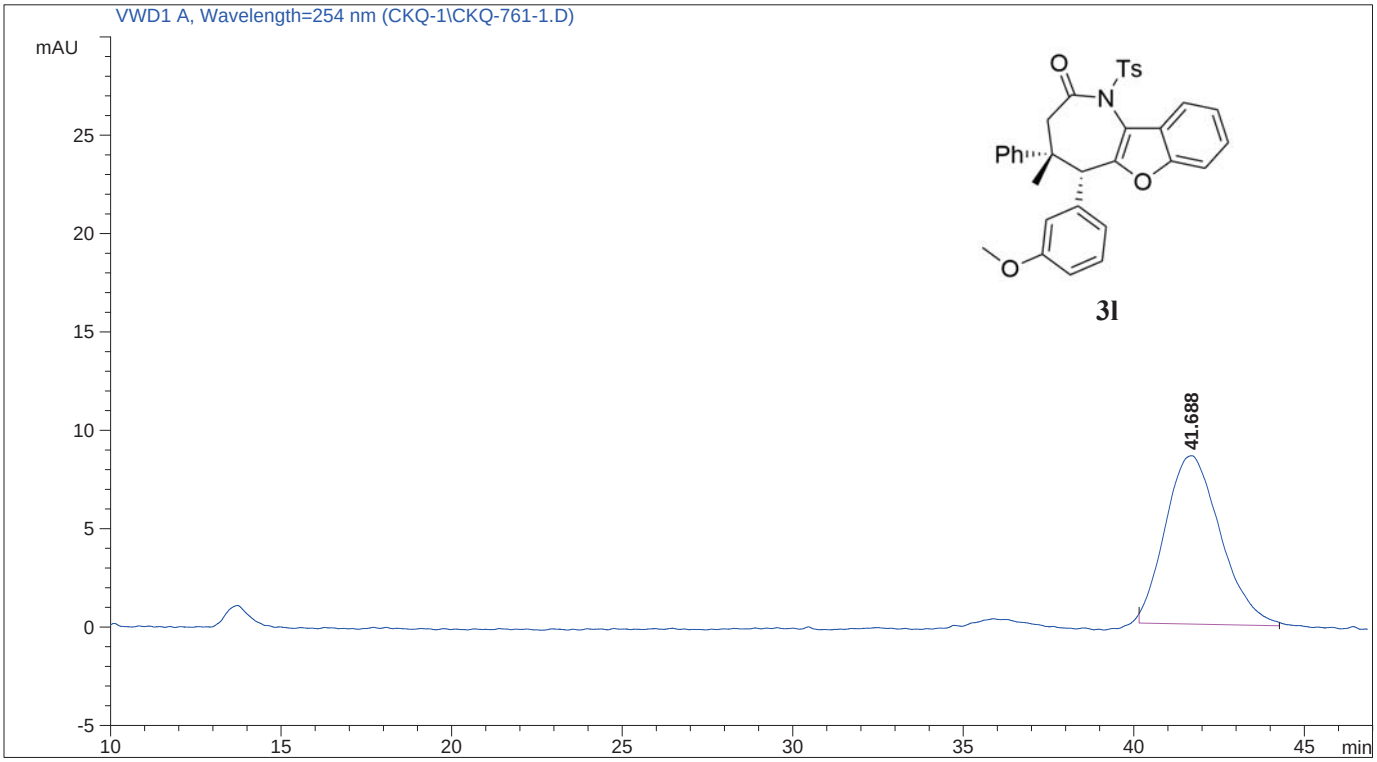


rac-**3l**

```
    ========================================================================
                              Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  32.191 BB    1.4110 1224.71289   13.38583  50.0550
       2  41.562 BB    1.7382 1222.02332   10.48544  49.9450

    Totals :                  2446.73621   23.87128


    ========================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\CKQ-761-1.D
Sample Name: CKQ-761-1

```
    ========================================================================
    Acq. Operator    : ckq
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date   : 2018-1-2 18:38:43
    Acq. Method      : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2018-1-2 19:24:41  by ckq
                       (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2018-3-13 17:44:35  by LZ
                       (modified after loading)
    Sample Info      : ic-70:30 1.1 mL/min, 254nm
```

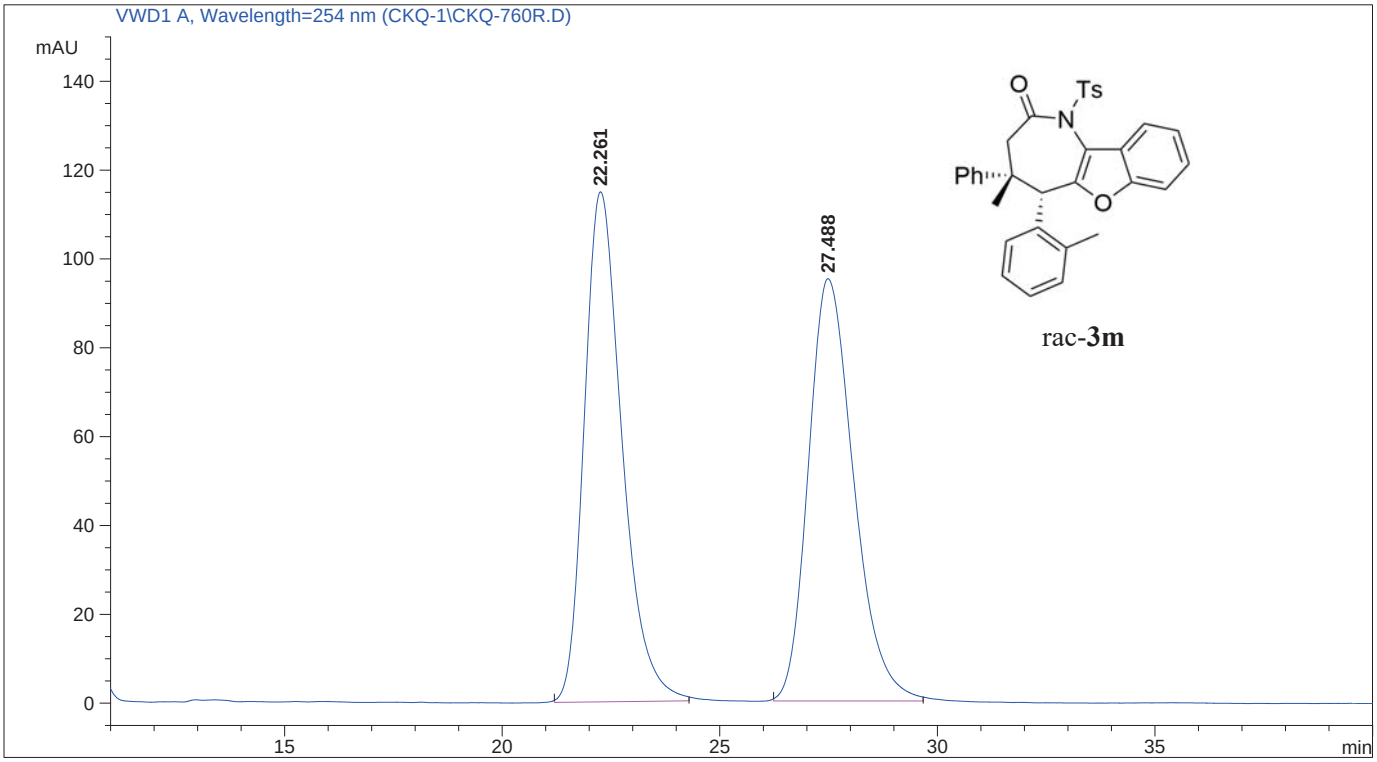


3l

```
    ========================================================================
                            Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  41.688 BB    1.6660  960.92560    8.55274 100.0000

    Totals :                   960.92560    8.55274

    ========================================================================
                            *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\CKQ-760R.D
Sample Name: CKQ-760R

```
    ======================================================================
    Acq. Operator   : ckq
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2017-12-22 10:43:03
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2017-12-22 8:47:38  by gzh
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2018-3-13 18:33:47  by LZ
                      (modified after loading)
    Sample Info     : IC H/I 70:30 1 mL/min, 254nm
```

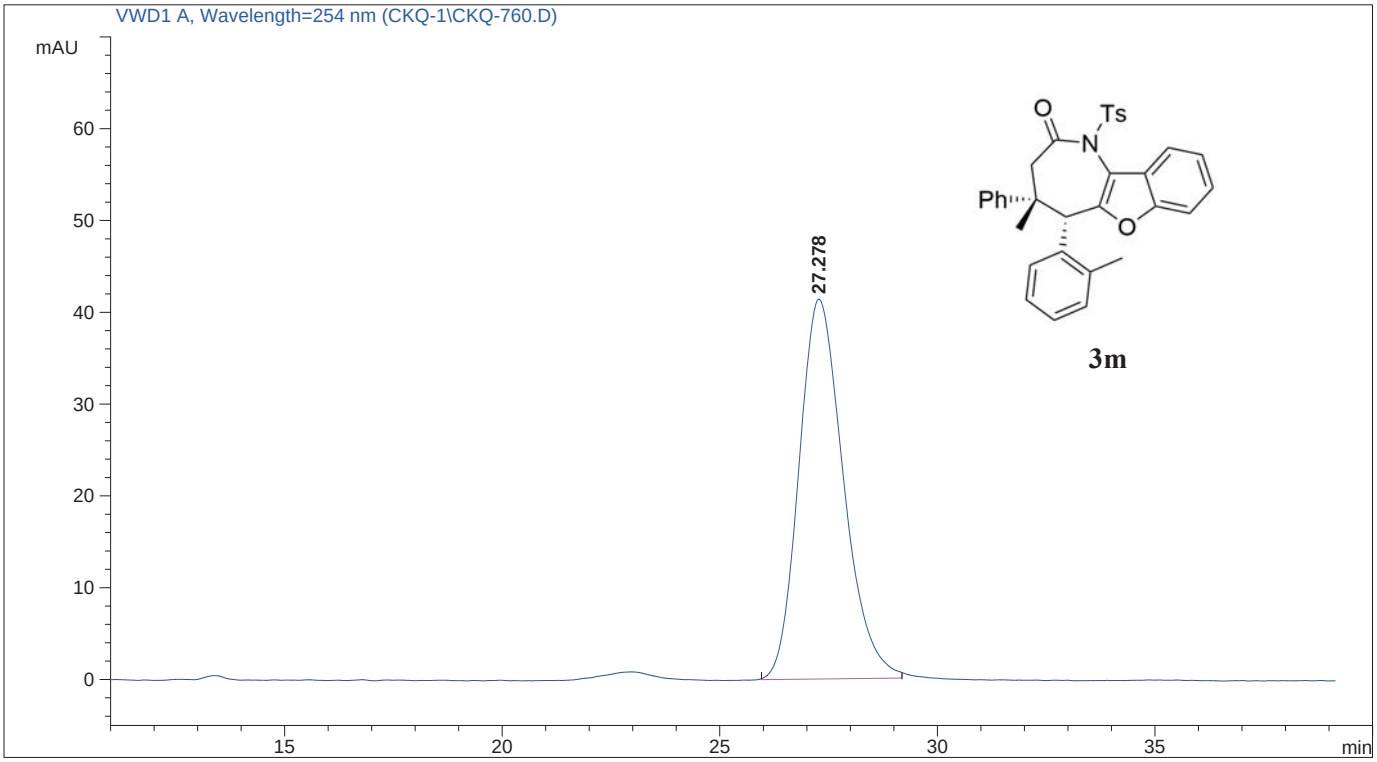


rac-**3m**

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  22.261 BB    0.9114 6906.41162  114.82349  50.1114
       2  27.488 BB    1.1059 6875.70801   95.05863  49.8886

    Totals :                  1.37821e4   209.88213


    ======================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\CKQ-760.D
Sample Name: CKQ-760

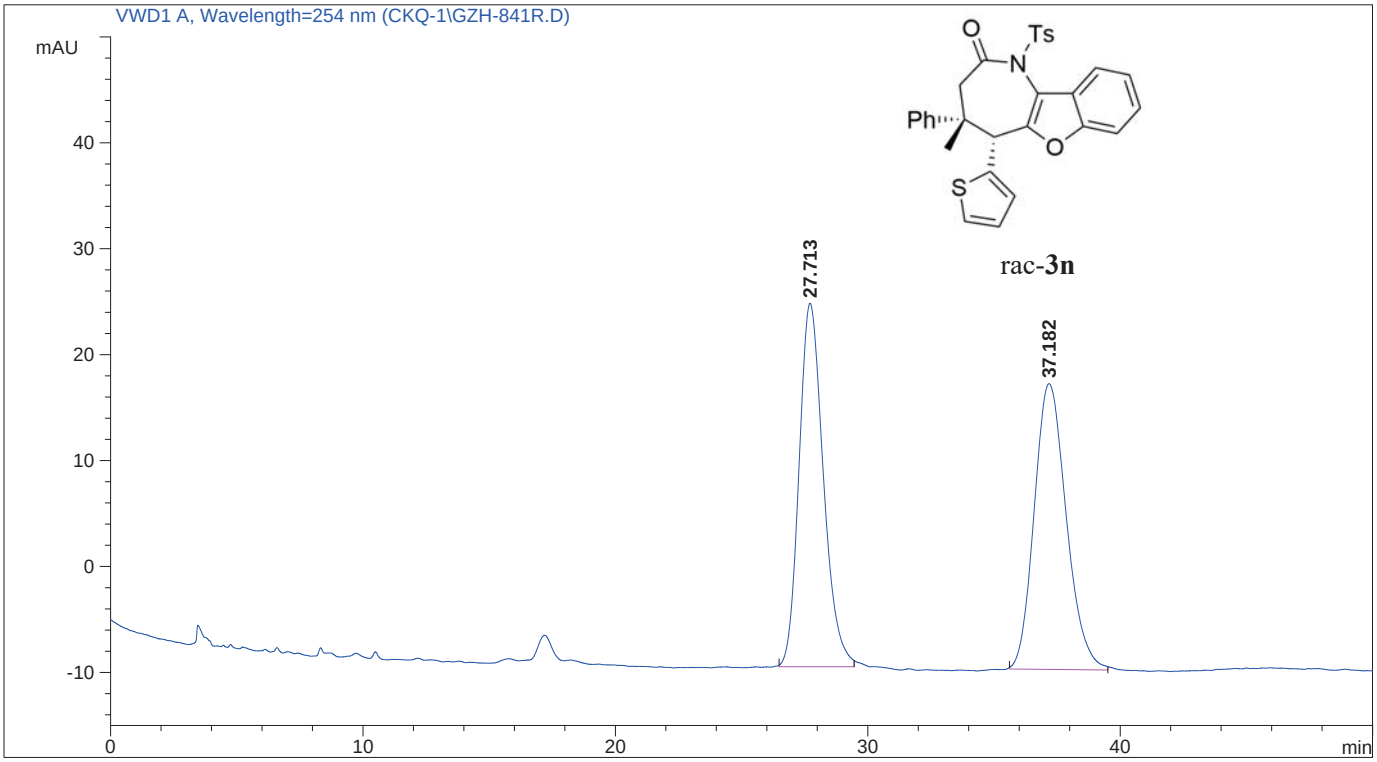
```
    ========================================================================
    Acq. Operator   : ckq
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2017-12-22 12:03:13
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2017-12-22 8:47:38  by gzh
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2018-3-13 18:33:06  by LZ
                      (modified after loading)
    Sample Info     : IC H/I 70:30 1 mL/min, 254nm
```




```
    ========================================================================
                            Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  27.278 BB    1.1141 2939.07104   41.37601 100.0000

    Totals :                  2939.07104   41.37601

    ========================================================================
                            *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\GZH-841R.D
Sample Name: ckq-gzh-841r

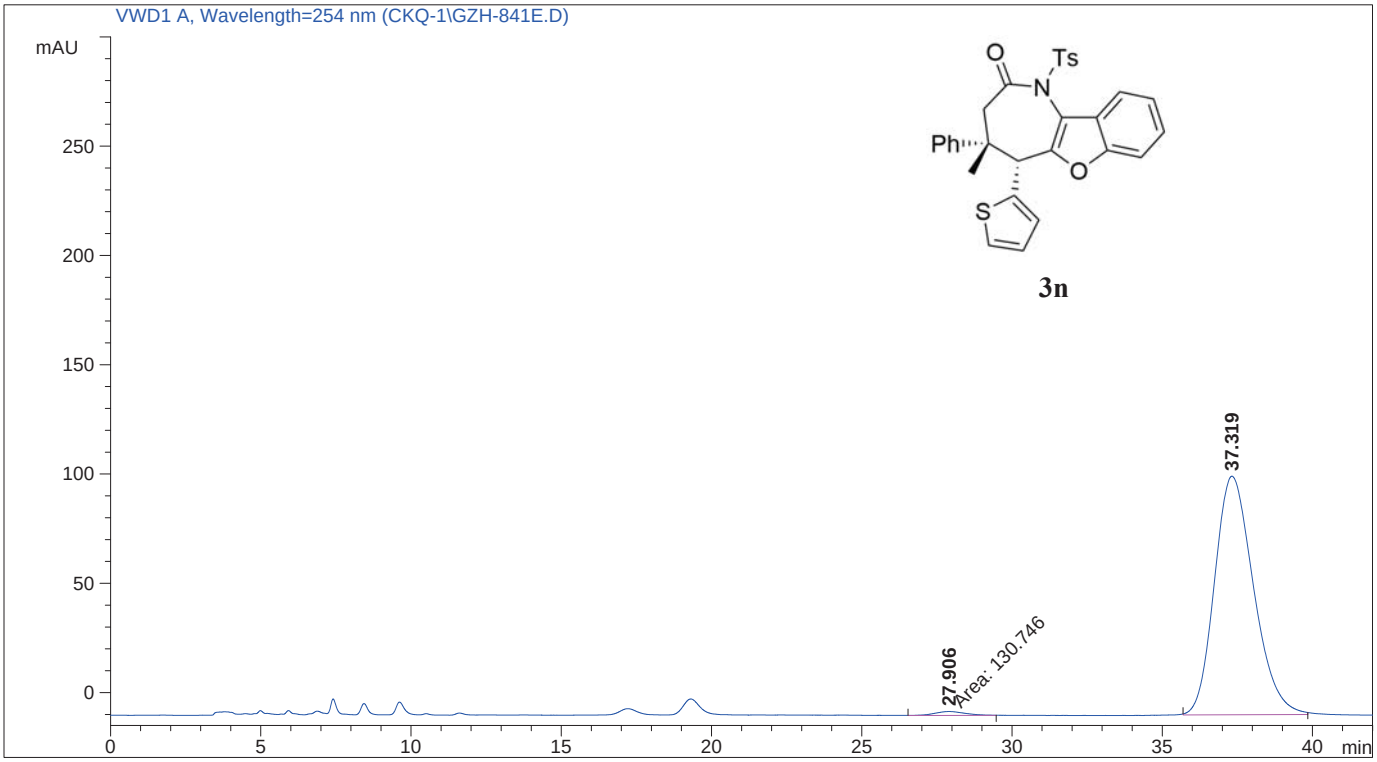
```
    ======================================================================
    Acq. Operator   : ckq
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2017-12-28 17:49:23
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2017-12-28 17:42:38  by ckq
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2017-12-28 21:22:25  by ckq
                      (modified after loading)
    Sample Info     : ia-70:30 1 mL/min, 254nm
```




```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  27.713 BB    1.0284 2287.78442   34.30684  49.0019
       2  37.182 BB    1.3792 2380.98560   26.97445  50.9981

    Totals :                  4668.77002   61.28129


    ======================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\GZH-841E.D
Sample Name: ckq-gzh-841e

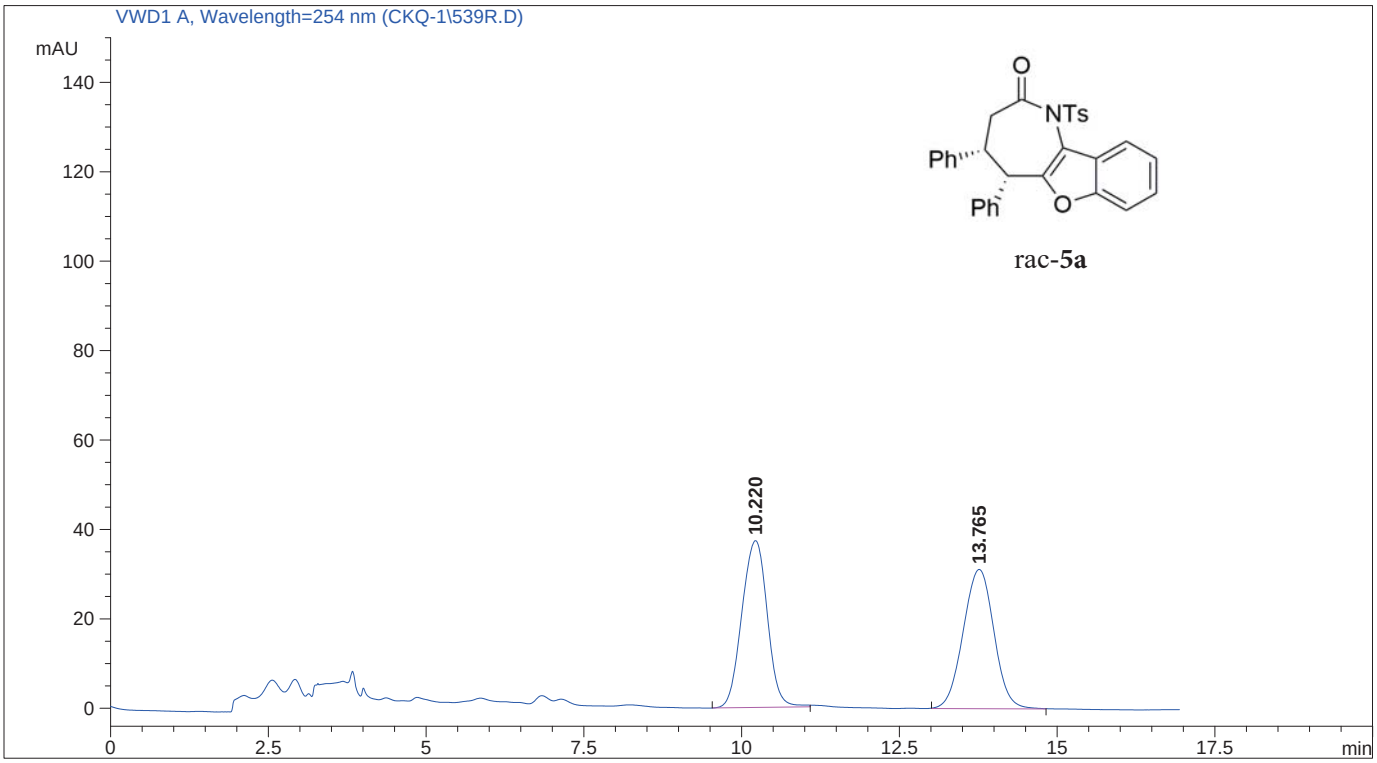
```
    ======================================================================
    Acq. Operator   : ckq
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2017-12-28 19:21:39
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2017-12-28 17:42:38  by ckq
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2018-2-28 11:48:45  by lz
                      (modified after loading)
    Sample Info     : ia-70:30 1 mL/min, 254nm
```




```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  27.906 MM    1.2099  130.74565    1.80108   1.3097
       2  37.319 BB    1.3909 9851.77734  109.14509  98.6903

    Totals :                  9982.52299  110.94616


    ======================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\539R.D
Sample Name: CKQ-539R

```
    ========================================================================
    Acq. Operator   : CKQ
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2016-9-20 18:36:50
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2016-9-20 18:34:18  by XF2016
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2016-9-20 19:24:31  by CKQ
                      (modified after loading)
    Sample Info     : IA-H H/I=90:10
                       1ml/min, 254nm
```

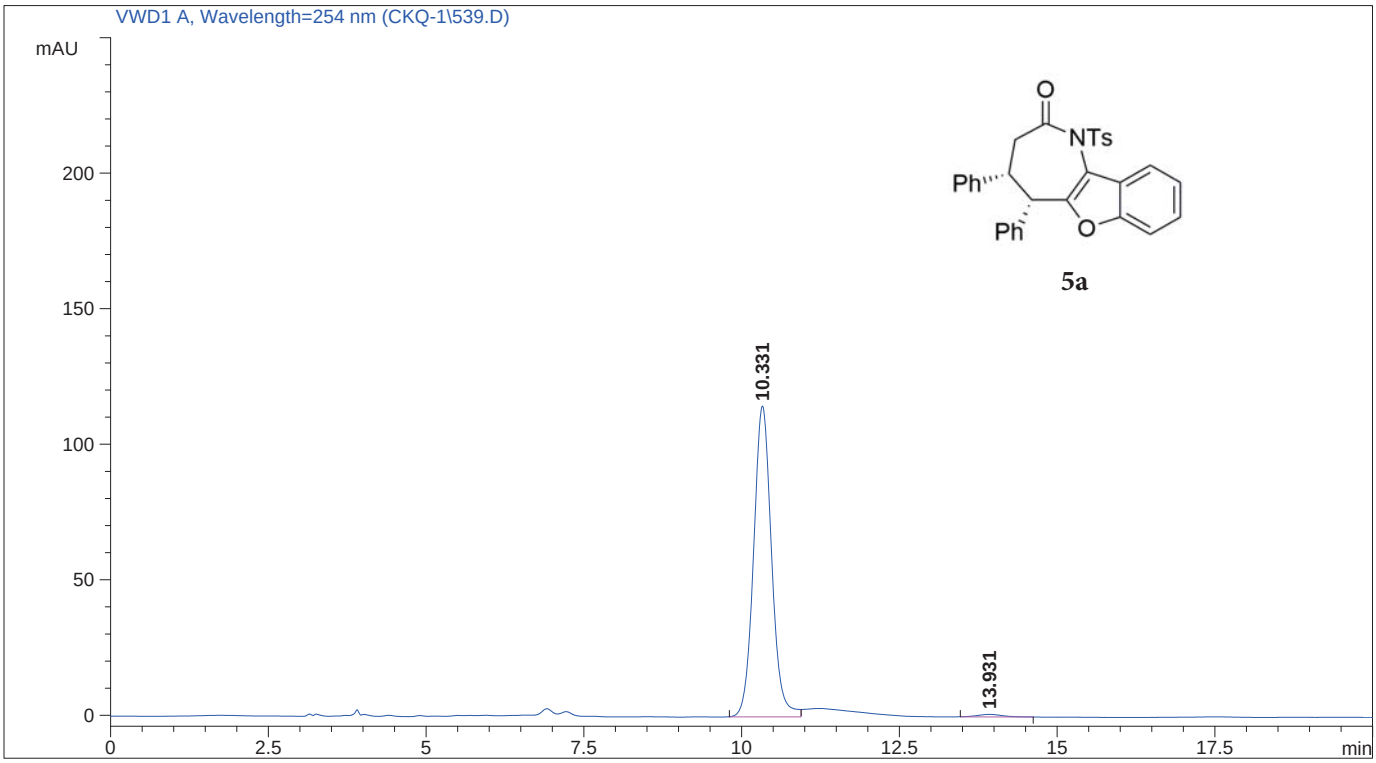


rac-**5a**

```
    ========================================================================
                            Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  10.220 BB    0.4499 1047.87097   37.31623  49.8059
       2  13.765 BB    0.5352 1056.03870   31.15582  50.1941

    Totals :                  2103.90967   68.47205


    ========================================================================
                            *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\539.D
Sample Name: CKQ-539

```
    ======================================================================
    Acq. Operator   : CKQ
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2016-9-20 18:55:00
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2016-9-20 18:34:18  by XF2016
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2016-9-20 19:25:39  by CKQ
                      (modified after loading)
    Sample Info     : IA-H H/I=90:10
                       1ml/min, 254nm
```

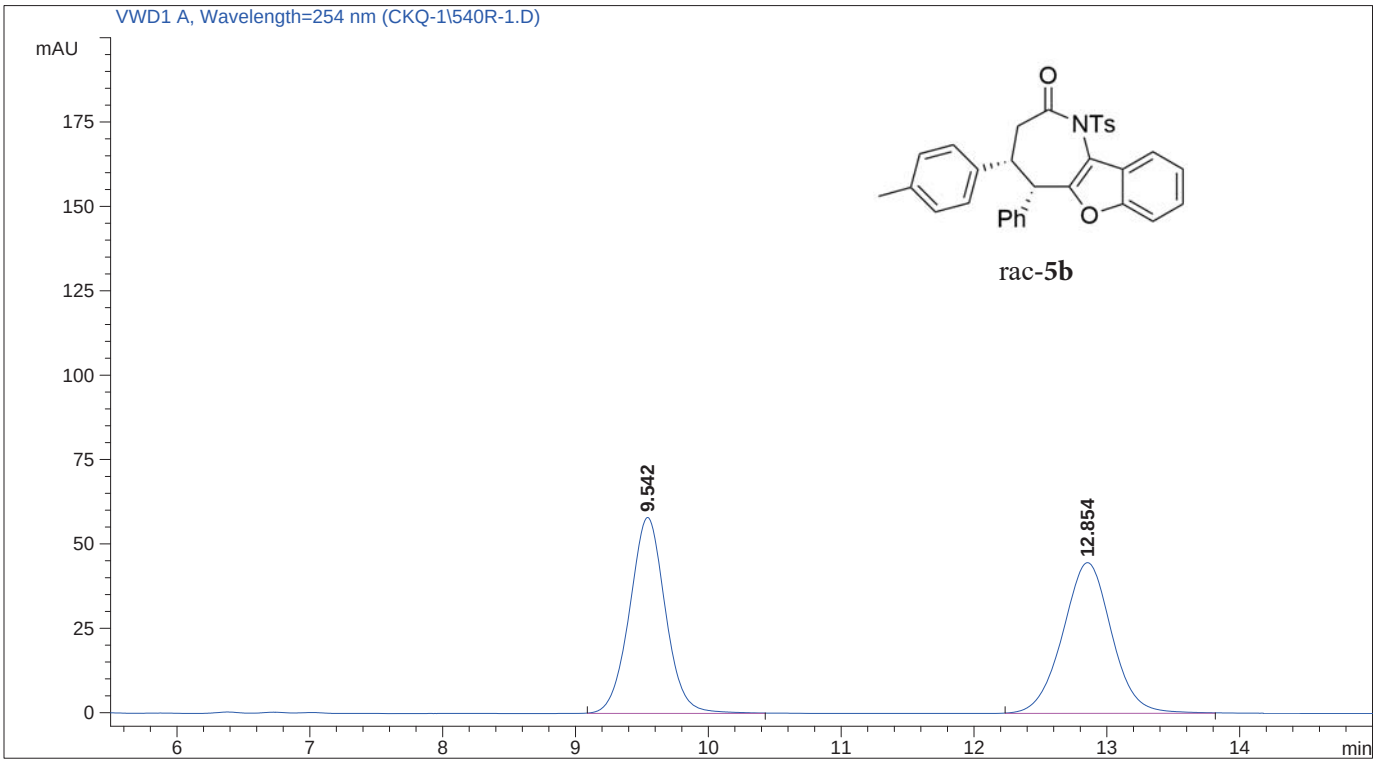


5a

```
    ======================================================================
                            Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  10.331 BV    0.3039 2270.85010  114.65051  98.8195
       2  13.931 BB    0.4362   27.12804 9.46730e-1   1.1805

    Totals :                  2297.97814  115.59724


    ======================================================================
                            *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\540R-1.D
Sample Name: CKQ-540r

```
    ========================================================================
    Acq. Operator   : CKQ
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2016-9-22 17:00:33
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2016-9-22 15:46:17  by CKQ
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2016-9-23 15:19:42  by CKQ
                      (modified after loading)
    Sample Info     : IA-H H/I=90:10
                       1ml/min, 254nm
```

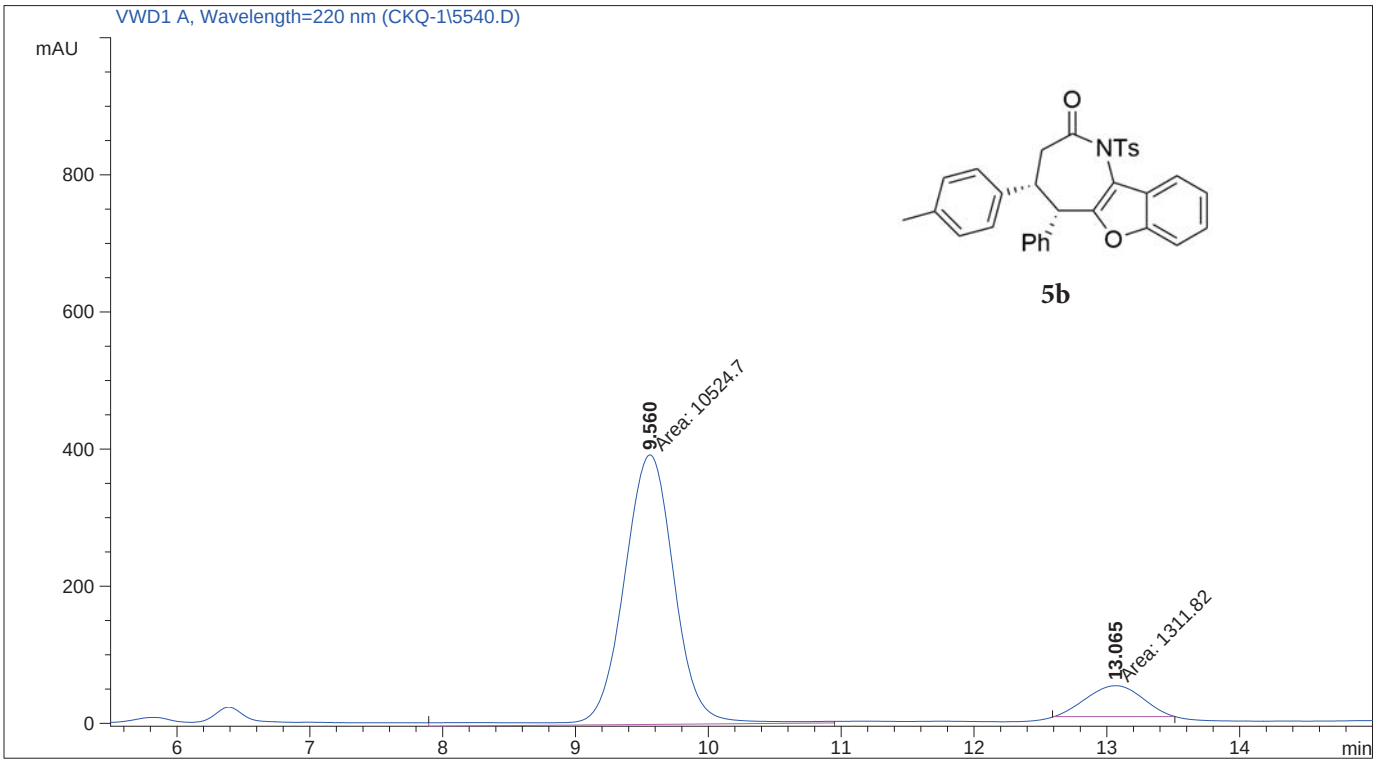


rac-**5b**

```
    ========================================================================
                            Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   9.542 BB    0.2857 1088.46753   58.05257  48.7779
       2  12.854 BB    0.3890 1143.00940   44.64327  51.2221

    Totals :                  2231.47693  102.69584


    ========================================================================
                            *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\5540.D
Sample Name: ckq-540

```
    ========================================================================
    Acq. Operator   : CKQ
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2016-9-23 15:01:38
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2016-9-23 14:58:53  by CKQ
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2016-9-23 15:18:07  by CKQ
                      (modified after loading)
    Sample Info     : ia H/I=90:10
                      0.5ml/min, 220nm
```

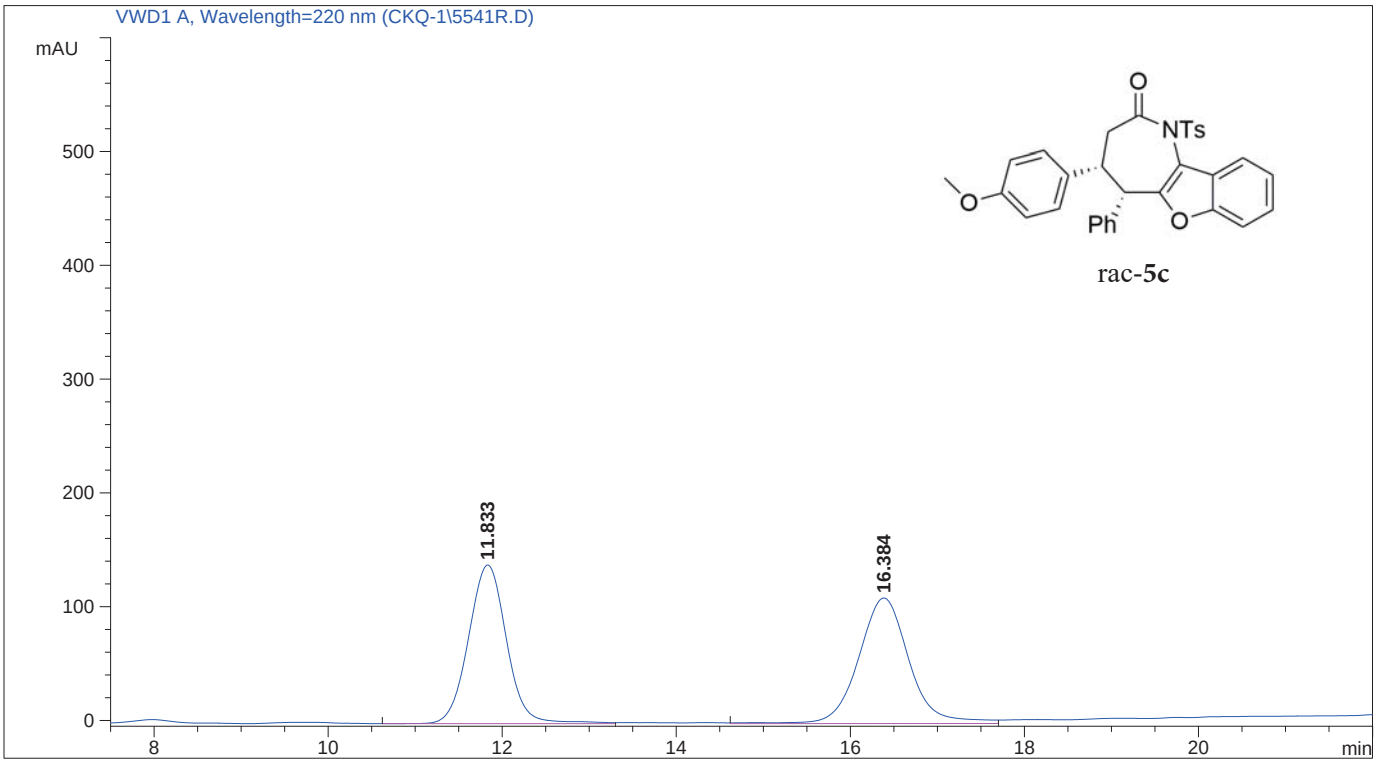


5b

```
    ========================================================================
                            Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   9.560 MM    0.4462 1.05247e4   393.15311  88.9172
       2  13.065 MM    0.4859 1311.82068   44.99678  11.0828

    Totals :                  1.18365e4   438.14989


    ========================================================================
                            *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\5541R.D
Sample Name: ckq-541r

```
    ======================================================================
    Acq. Operator   : CKQ
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2016-9-23 15:31:46
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2016-9-23 15:19:42  by CKQ
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2016-9-23 19:41:30  by ckq
                      (modified after loading)
    Sample Info     : ia H/I=90:10
                      0.5ml/min, 220nm
```

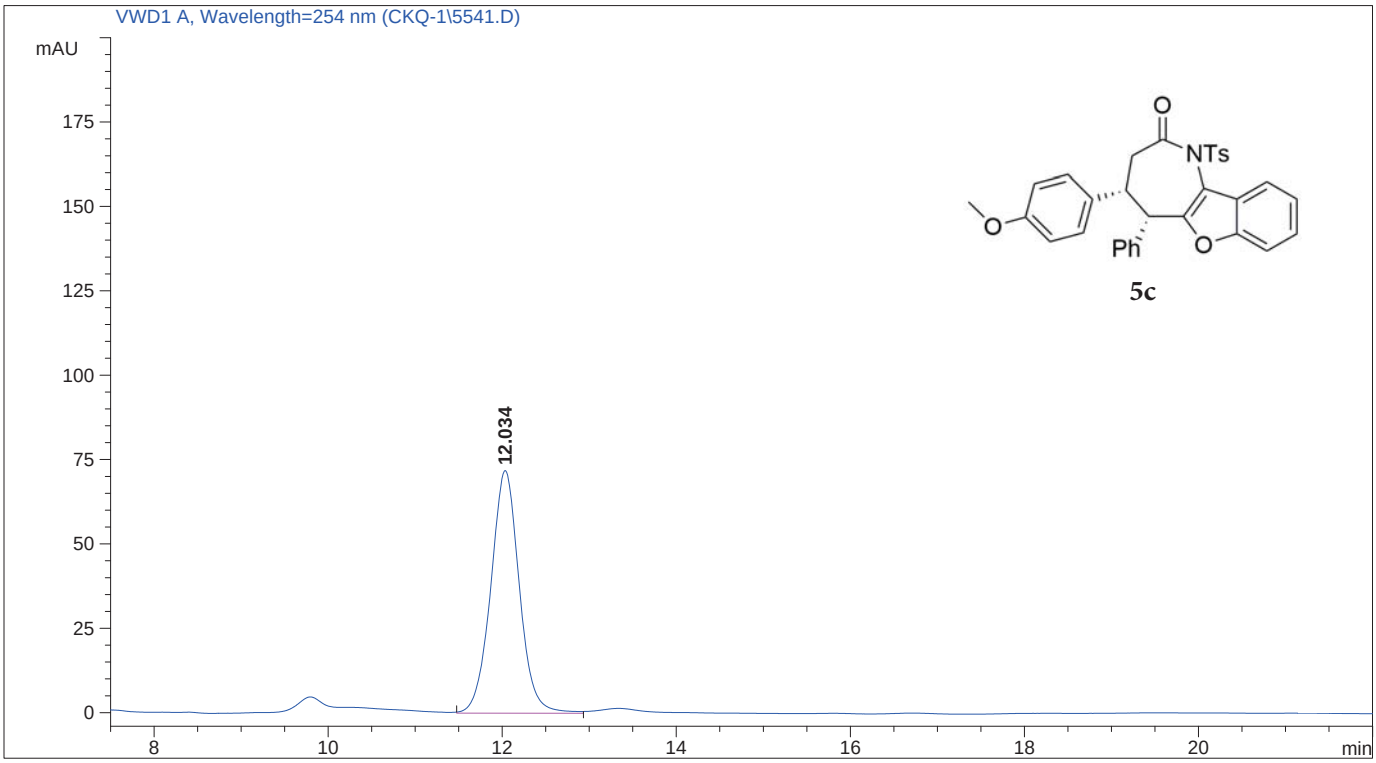


rac-**5c**

```
    ======================================================================
                            Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  11.833 VV    0.4714 4238.75244  139.34964  48.7600
       2  16.384 VV    0.6196 4454.33447  110.32248  51.2400

    Totals :                  8693.08691  249.67212


    ======================================================================
                            *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\5541.D
Sample Name: ckq-1

```
==========================================================================
Acq. Operator   : ckq
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 2016-9-23 19:15:00
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2016-9-23 19:12:54  by XF2016
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2016-9-23 19:40:18  by ckq
                  (modified after loading)
Sample Info     : IA H/I=90:1
                  1ml/min, 254nm
```

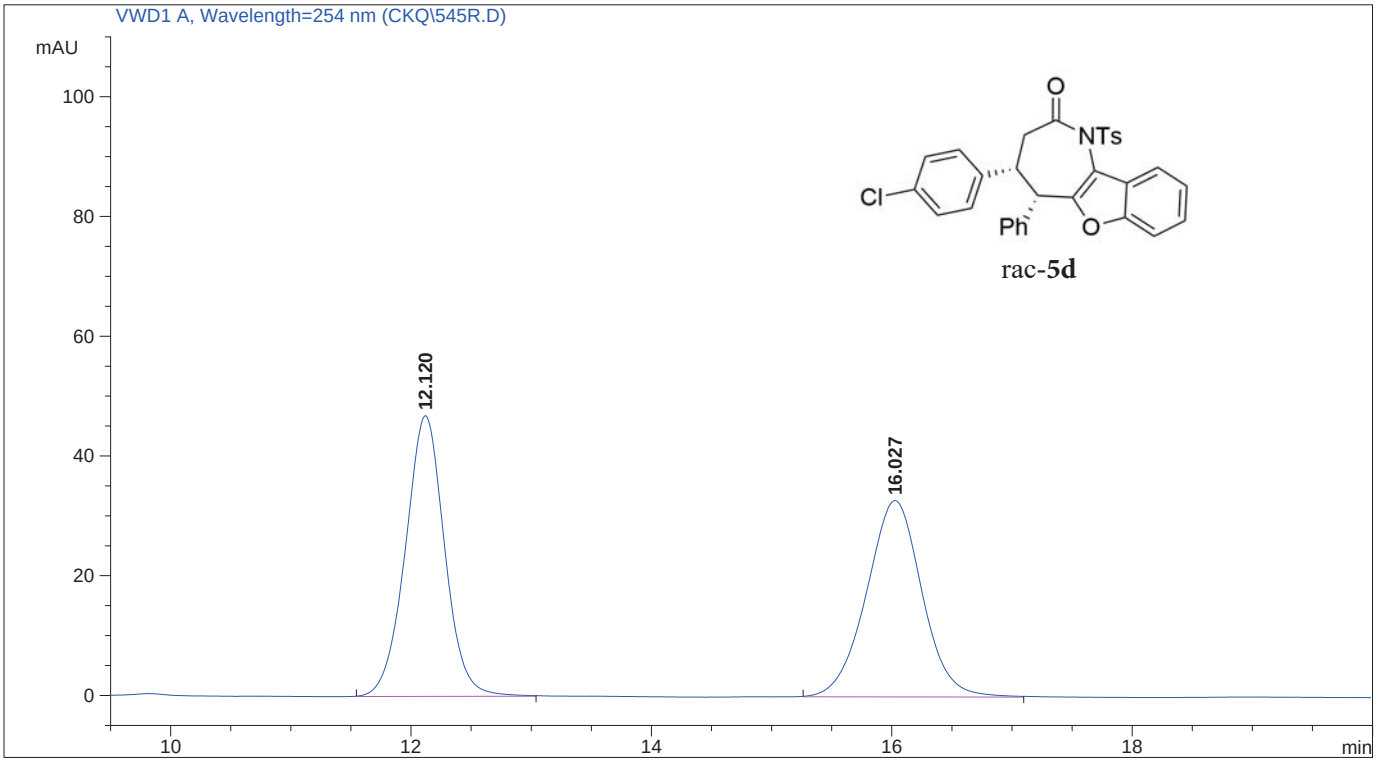


5c

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  12.034 BB    0.3469 1634.67871   71.85631 100.0000

Totals :                  1634.67871   71.85631


==========================================================================
                        *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ\545R.D
Sample Name: ckq-545r

```
    ========================================================================
    Acq. Operator    : ckq
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date   : 2016-9-26 8:54:54
    Acq. Method      : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2016-9-26 8:51:19  by ckq
                       (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2016-9-26 9:36:25  by ckq
                       (modified after loading)
    Sample Info      : IA H/I=90:10 1.0 ml/min, 254nm
```

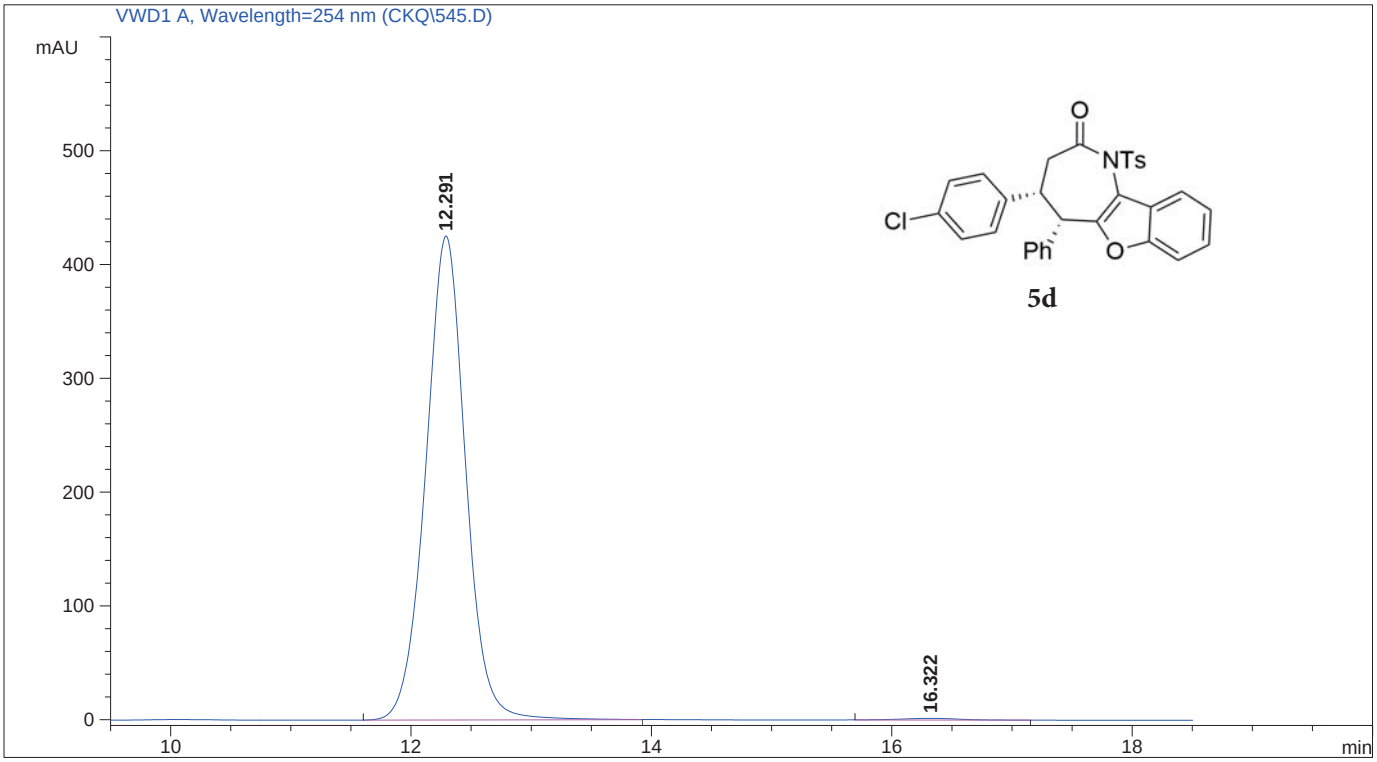


rac-**5d**

```
    ========================================================================
                              Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  12.120 BB    0.3465 1064.73584   46.87847  50.3007
       2  16.027 BB    0.4906 1052.00781   32.80226  49.6993

    Totals :                  2116.74365   79.68073


    ========================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ\545.D
Sample Name: ckq-545

```
    ========================================================================
    Acq. Operator    : ckq
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date   : 2016-9-26 9:16:09
    Acq. Method      : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2016-9-26 8:51:19  by ckq
                       (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2016-9-26 9:37:26  by ckq
                       (modified after loading)
    Sample Info      : IA H/I=90:10 1.0 ml/min, 254nm
```

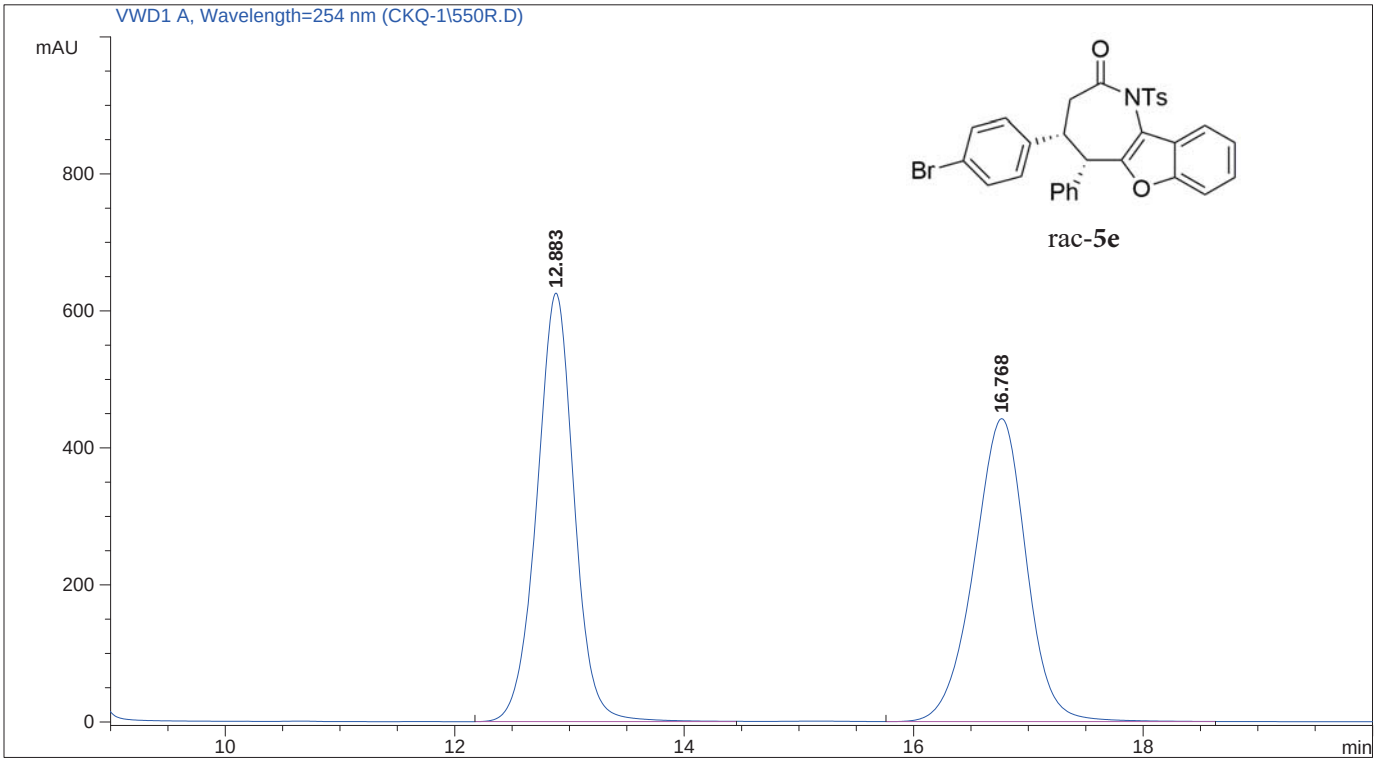


5d

```
    ========================================================================
                              Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  12.291 BB    0.3503 9795.81152  425.24097  99.4756
       2  16.322 BB    0.5180   51.63790    1.52257   0.5244

    Totals :                  9847.44942  426.76354


    ========================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\550R.D
Sample Name: CKQ-550R

```
    ========================================================================
    Acq. Operator   : CKQ
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2016-9-27 20:13:24
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2016-9-27 19:08:48  by XF2016
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2016-9-27 21:07:06  by CKQ
                      (modified after loading)
    Sample Info     : IA-H H/I=90:10 1ml/min, 254nm
```

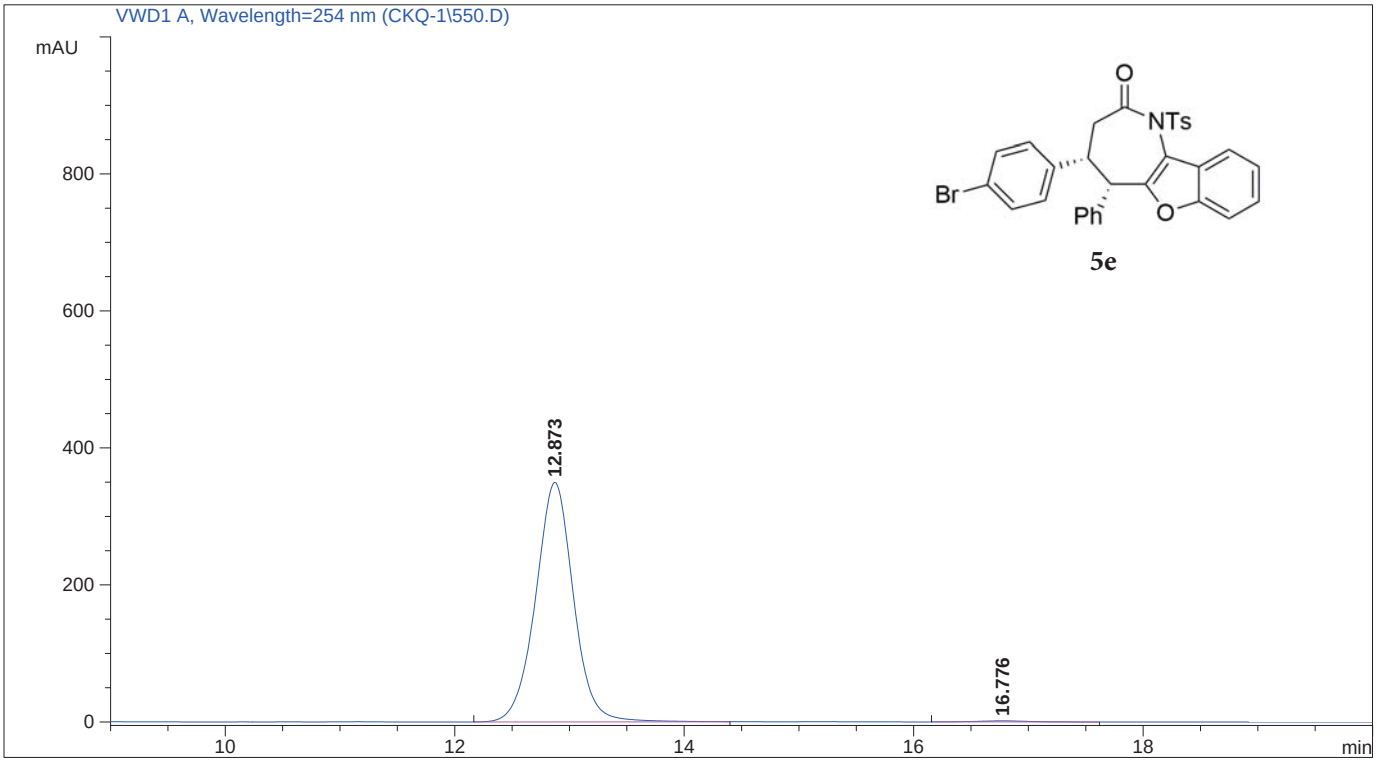


rac-**5e**

```
    ========================================================================
                              Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  12.883 BB    0.3419 1.41131e4   625.42303  50.2276
       2  16.768 VB    0.4826 1.39852e4   442.18457  49.7724

    Totals :                  2.80983e4  1067.60760


    ========================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\550.D
Sample Name: CKQ-550

```
    ======================================================================
    Acq. Operator   : CKQ
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2016-9-27 20:35:52
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2016-9-27 20:35:57  by CKQ
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2016-9-27 21:07:06  by CKQ
                      (modified after loading)
    Sample Info     : IA-H H/I=90:10 1ml/min, 254nm
```

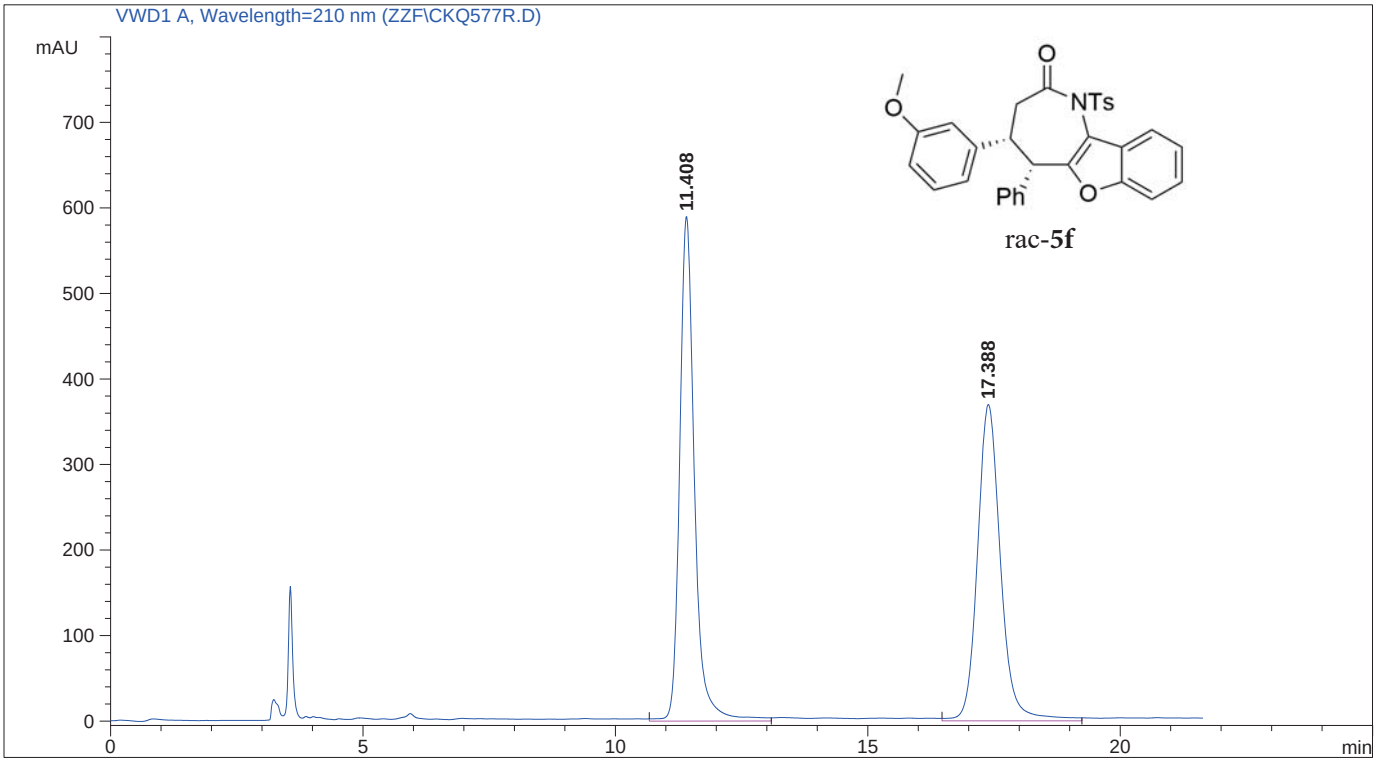


5e

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  12.873 BB    0.3495 8027.02100  349.47656  99.4002
       2  16.776 BB    0.4532   48.43775    1.58227   0.5998

    Totals :                  8075.45874  351.05883


    ======================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\ZZF\CKQ577R.D
Sample Name: ckq577r

```
    ========================================================================
    Acq. Operator   : ckq
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2016-11-12 9:35:04
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2016-11-12 9:31:22  by ckq
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2016-11-12 10:35:53  by ckq
                      (modified after loading)
    Sample Info     : Ia-H/ I =90/10 1ml/min, 210nm
```

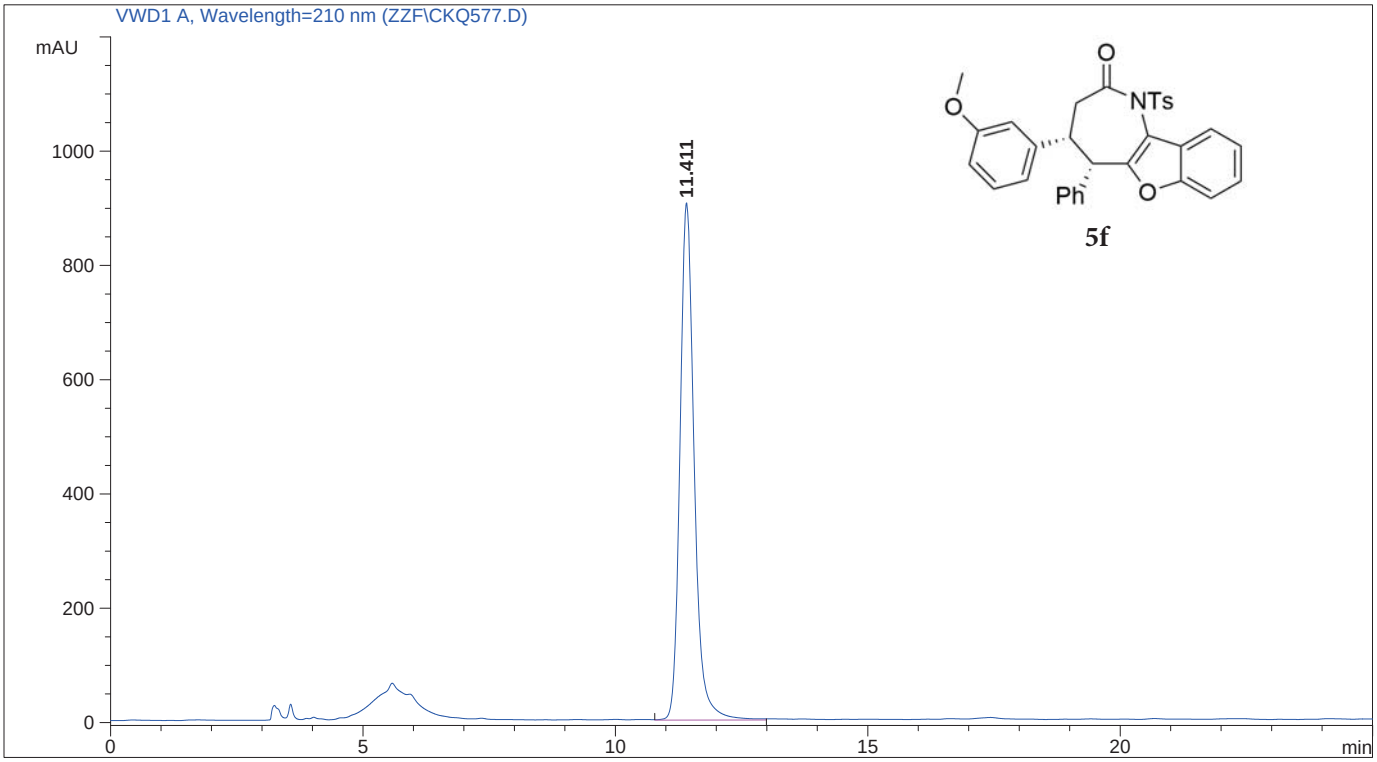


rac-**5f**

```
    ========================================================================
                            Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  11.408 BV    0.3110 1.20429e4   589.78009  50.4924
       2  17.388 VB    0.4861 1.18080e4   369.83765  49.5076

    Totals :                  2.38509e4   959.61774
    ========================================================================
                            Summed Peaks Report
    ========================================================================
    Signal 1: VWD1 A, Wavelength=210 nm

                                    *** End of Report ***
```

Data File C:\CHEM32\1\DATA\ZZF\CKQ577.D
Sample Name: ckq577

```
    ========================================================================
    Acq. Operator   : ckq
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2016-11-12 10:02:29
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2016-11-12 9:31:22  by ckq
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2016-11-12 10:35:02  by ckq
                      (modified after loading)
    Sample Info     : Ia-H/ I =90/10 1ml/min, 210nm
```

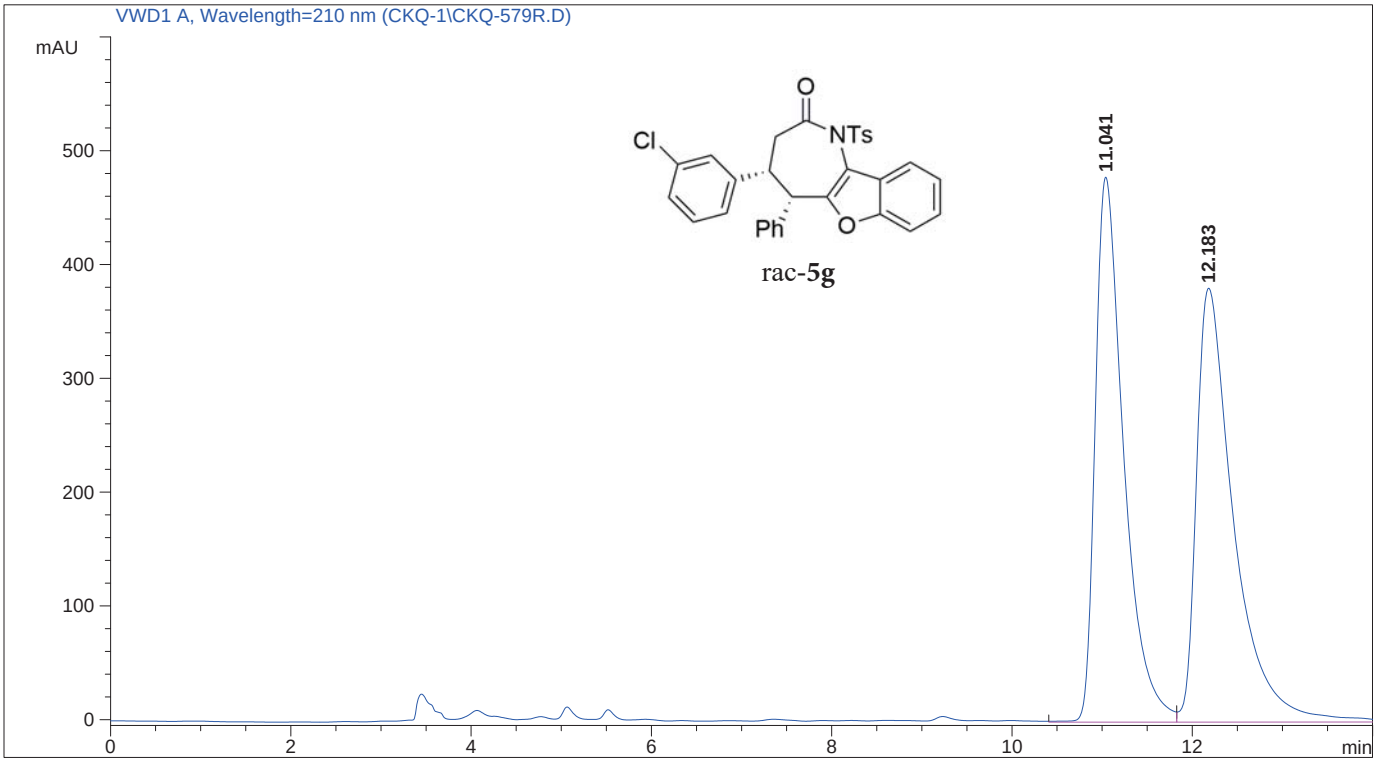


5f

```
    ========================================================================
                              Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  11.411 VB    0.3018 1.77680e4   905.08759 100.0000

    Totals :                  1.77680e4   905.08759

    ========================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\CKQ-579R.D
Sample Name: ckq-579r

```
    ========================================================================
    Acq. Operator   : ckq
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2016-11-8 19:24:25
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2016-11-8 19:19:34  by ckq
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2016-11-8 19:59:46  by ckq
                      (modified after loading)
    Sample Info     : Ib-H/ I =90/10 1ml/min, 210nm
```

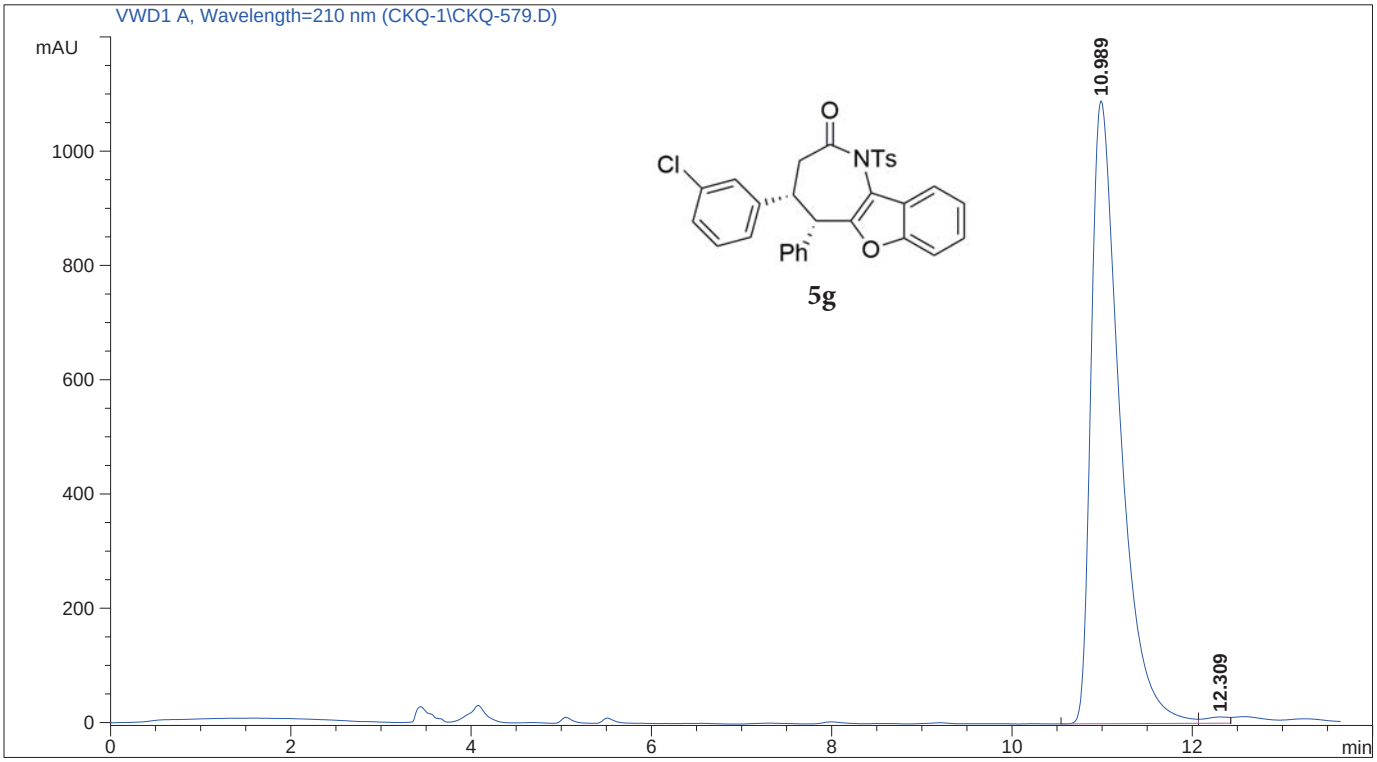


rac-**5g**

```
    ========================================================================
                              Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  11.041 VV    0.3314 1.04914e4   478.70703  48.9134
       2  12.183 VV    0.4256 1.09576e4   381.25790  51.0866

    Totals :                  2.14490e4   859.96494


    ========================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\CKQ-579.D
Sample Name: ckq-579

```
    ======================================================================
    Acq. Operator   : ckq
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2016-11-8 19:45:12
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2016-11-8 19:19:34  by ckq
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2016-11-8 20:01:58  by ckq
                      (modified after loading)
    Sample Info     : Ib-H/ I =90/10 1ml/min, 210nm
```

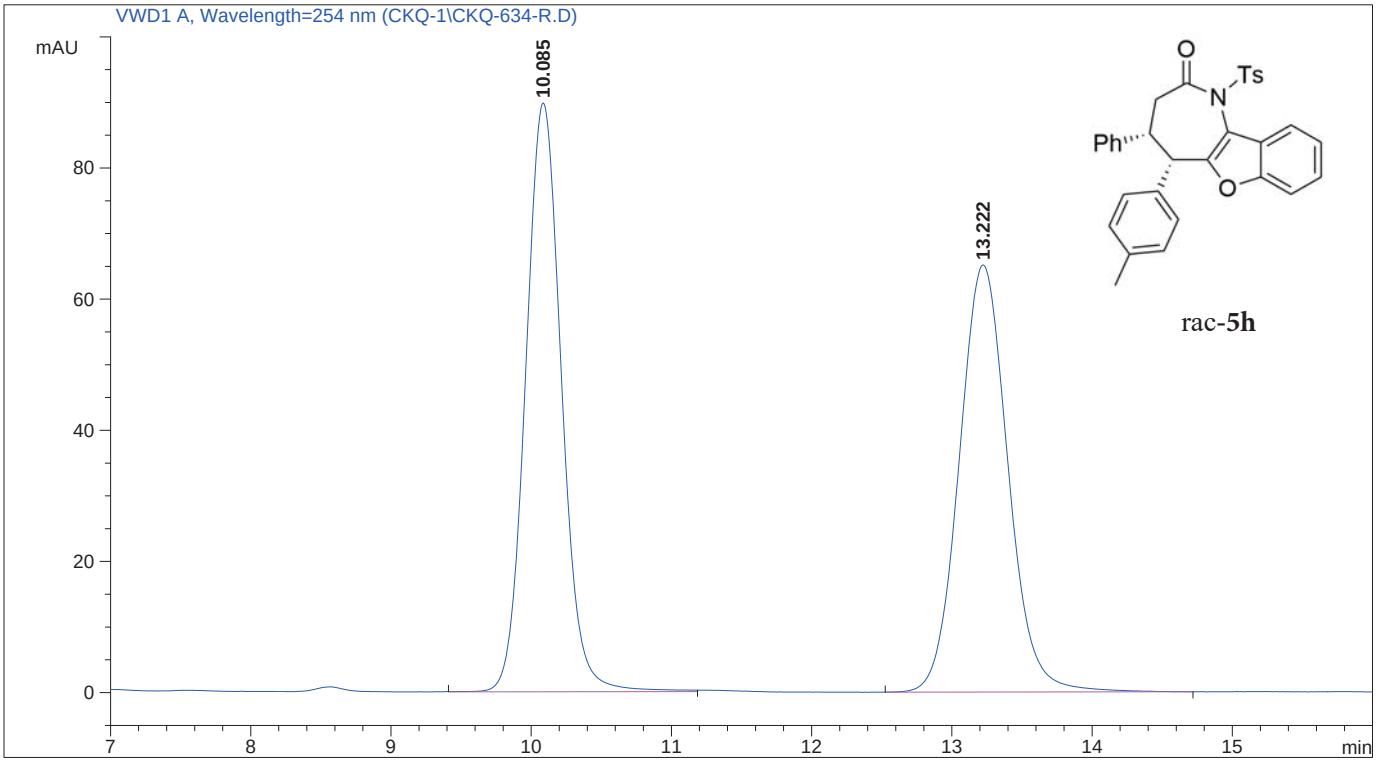


5g

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  10.989 VV    0.3330 2.42963e4  1089.25500  99.1671
       2  12.309 VV    0.2708  204.07094   10.90391   0.8329

    Totals :                  2.45003e4  1100.15891


    ======================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\CKQ-634-R.D
Sample Name: ckq-634-r

```
    ======================================================================
    Acq. Operator   : ckq
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2017-2-8 19:00:55
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2017-2-8 18:24:31  by yl
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2017-2-8 19:28:19  by ckq
                      (modified after loading)
    Sample Info     : IA H/ I =90:10  1ml/min, 254nm
```

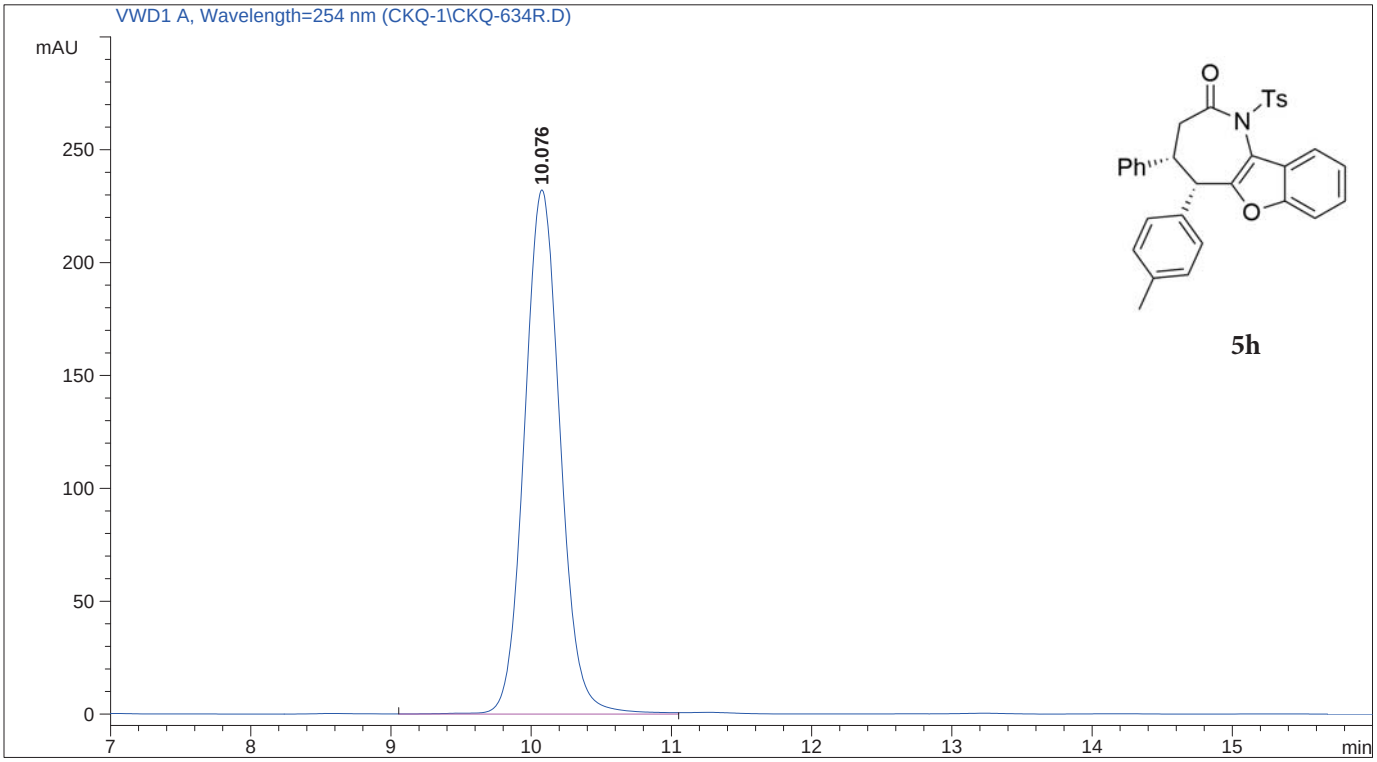


rac-**5h**

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  10.085 BB    0.2769 1593.04297   89.76314  50.3368
       2  13.222 BB    0.3748 1571.72437   65.11993  49.6632

    Totals :                  3164.76733  154.88306


    ======================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\CKQ-634R.D
Sample Name: ckq-634r

```
    ======================================================================
    Acq. Operator   : ckq
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2017-2-8 18:34:25
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2017-2-8 18:24:31  by yl
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2017-2-8 19:29:16  by ckq
                      (modified after loading)
    Sample Info     : IA H/ I =90:10  1ml/min, 254nm
```

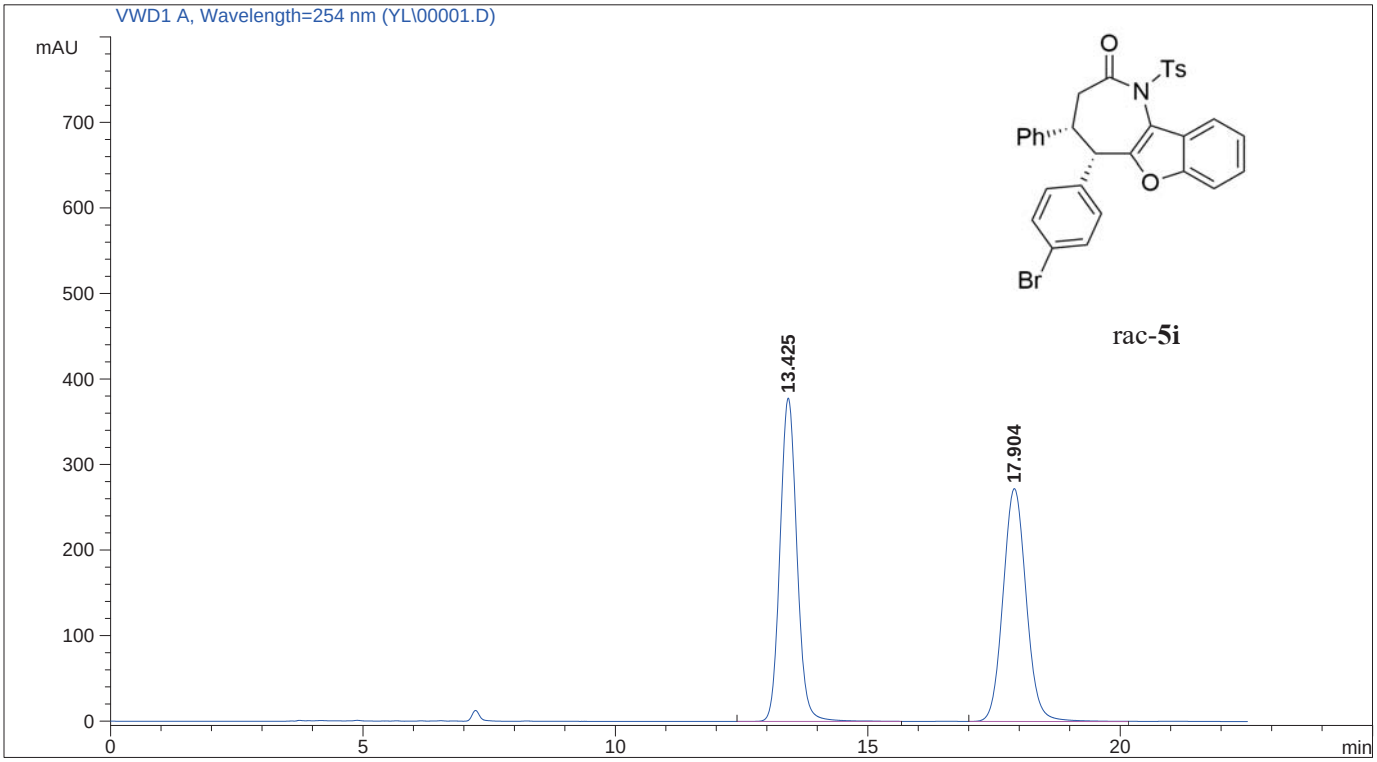


5h

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  10.076 VV    0.2770 4120.45947  232.15909 100.0000

    Totals :                  4120.45947  232.15909

    ======================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\YL\00001.D
Sample Name: Z-63B

```
    ======================================================================
    Acq. Operator   : yl
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2017-2-8 16:01:17
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2017-2-8 15:58:07  by yl
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2017-2-8 16:34:36  by yl
                      (modified after loading)
    Sample Info     : IA H/ I =90:10  1ml/min, 254nm
```

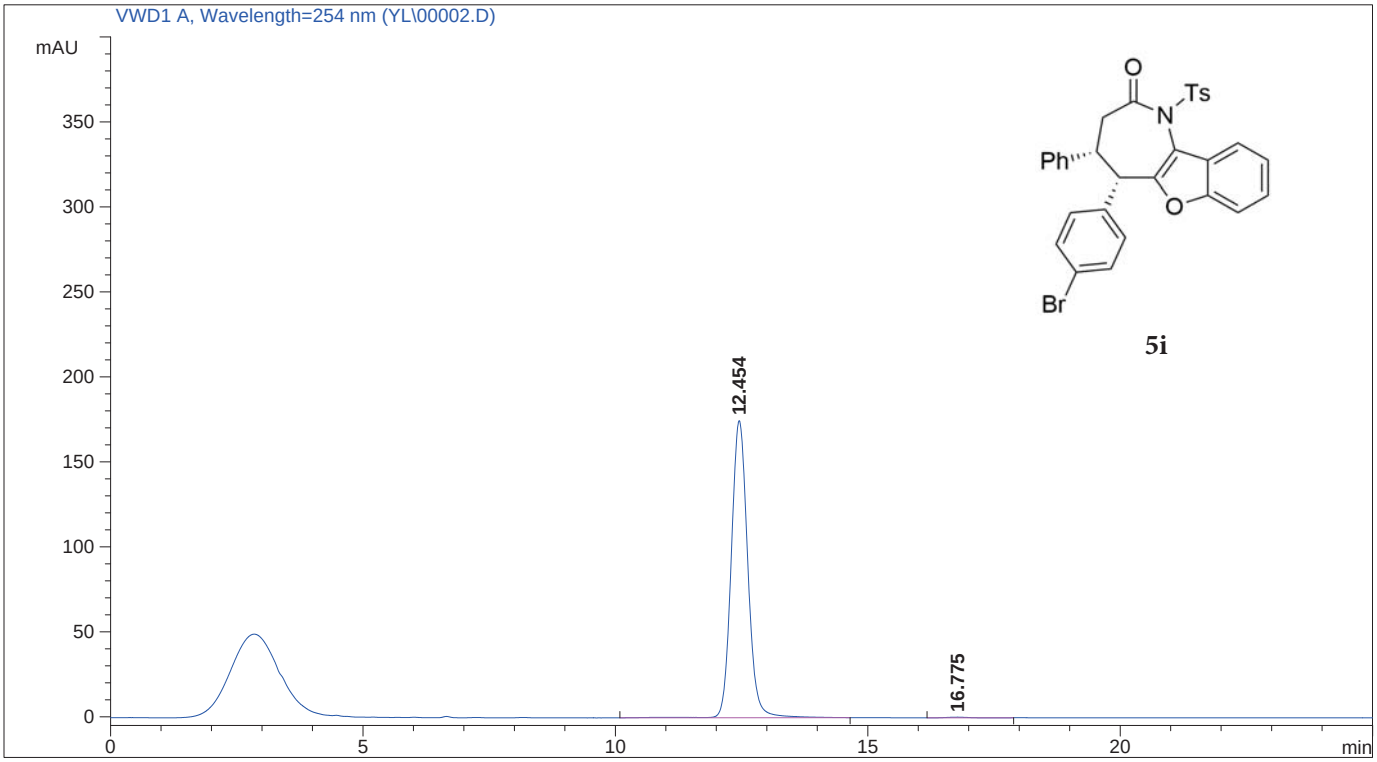


rac-**5i**

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  13.425 BB    0.3647 8800.05859  378.08847  50.3342
       2  17.904 VB    0.4945 8683.20215  272.19330  49.6658

    Totals :                  1.74833e4   650.28177


    ======================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\YL\00002.D
Sample Name: Z-63A

```
==========================================================================
Acq. Operator   : yl
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 2017-2-8 16:33:13
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2017-2-8 16:34:36  by yl
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2017-2-8 17:06:34  by yl
                  (modified after loading)
Sample Info     : IA H/ I =90:10  1ml/min, 254nm
```

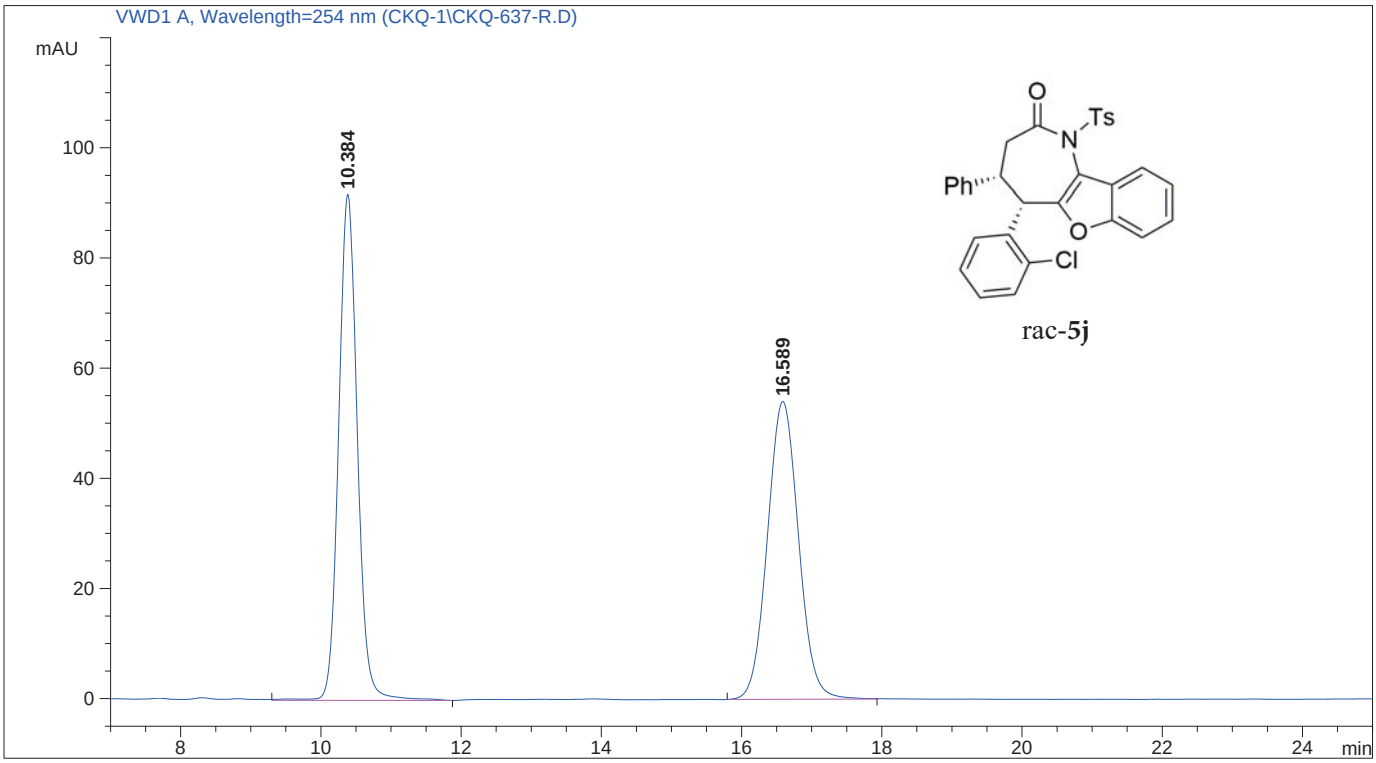


5i

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  12.454 BB    0.3528 3975.26172  174.74666  99.7049
   2  16.775 BB    0.4105   11.76735 3.96294e-1   0.2951

Totals :                  3987.02906  175.14295


==========================================================================
                          *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\CKQ-637-R.D
Sample Name: ckq-637r

```
    ========================================================================
    Acq. Operator   : ckq
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2017-2-13 15:24:11
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2017-2-13 14:52:40  by yl
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2017-2-13 15:59:29  by ckq
                      (modified after loading)
    Sample Info     :
                      IA H/ I =90:10  1ml/min, 254nm
```

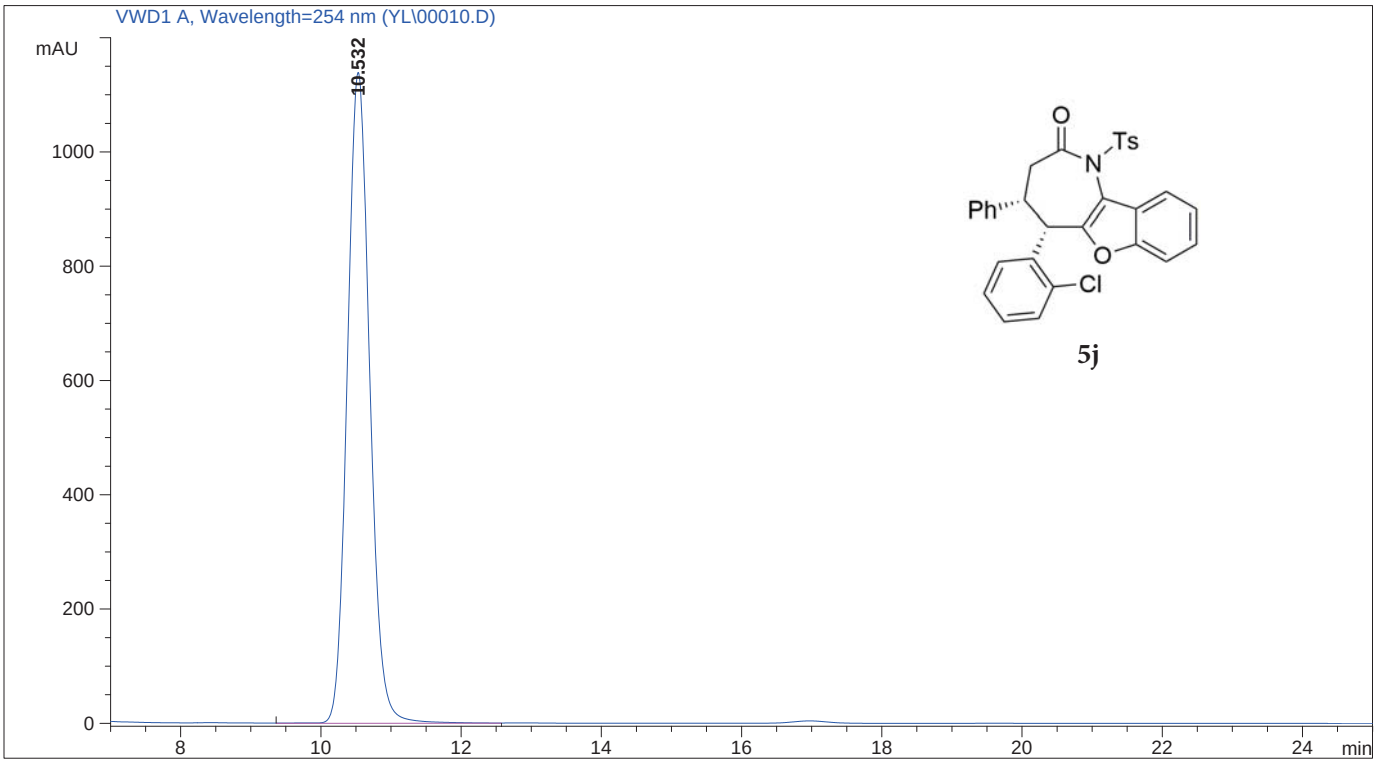


rac-**5j**

```
    ========================================================================
                              Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  10.384 VV    0.2867 1706.42175   91.83783  50.6101
       2  16.589 BB    0.4817 1665.27966   54.06171  49.3899

    Totals :                  3371.70142  145.89954


    ========================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\YL\00010.D
Sample Name: ckq-637chrial

```
    ========================================================================
    Acq. Operator   : yl
    Acq. Instrument : Instrument 1                    Location : Vial 1
    Injection Date  : 2017-2-10 18:54:10
    Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2017-2-10 18:53:33  by yl
                      (modified after loading)
    Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed    : 2017-2-13 15:57:30  by ckq
                      (modified after loading)
    Sample Info     :
                      IA H/ I =90:10  1ml/min, 254nm
```

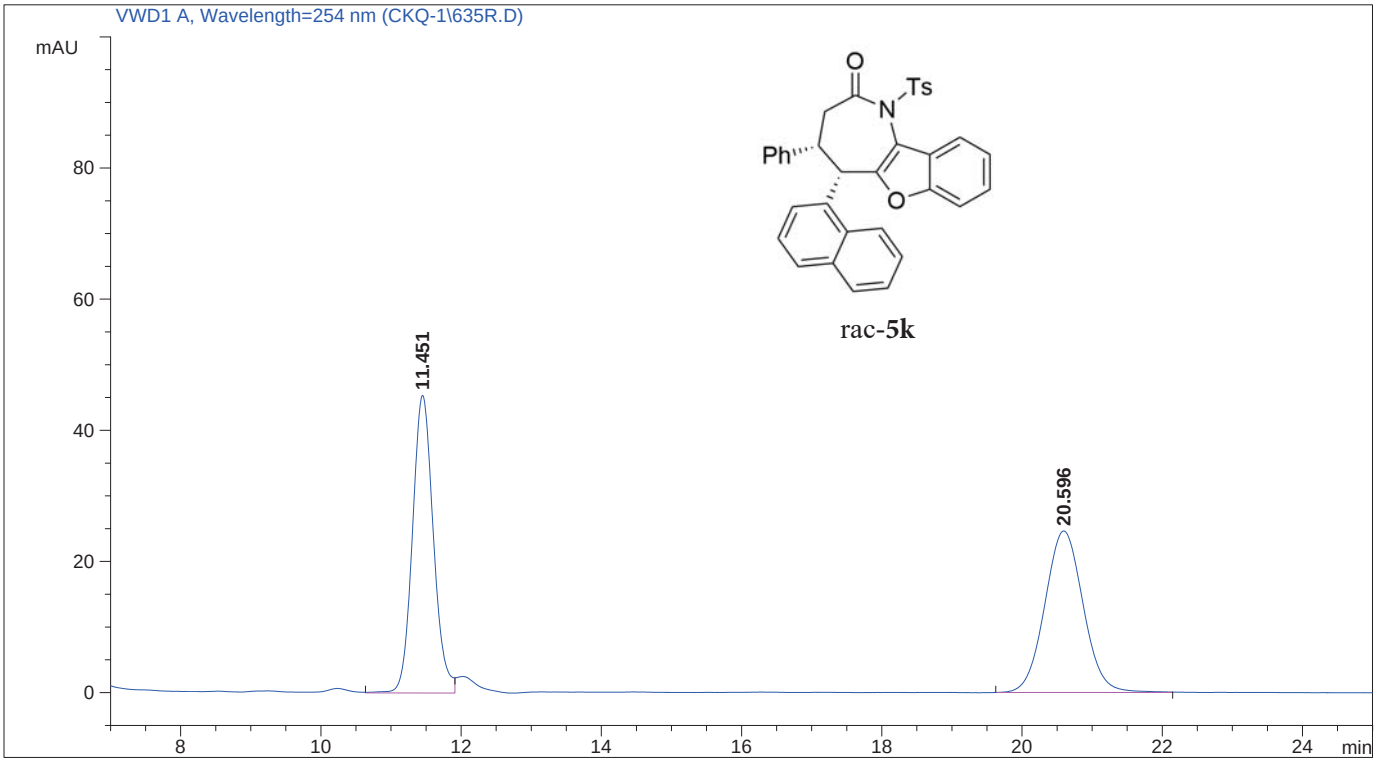


5j

```
    ========================================================================
                            Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  10.532 VB    0.3468 2.53277e4  1138.85254 100.0000

    Totals :                  2.53277e4  1138.85254


    ========================================================================
                            *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\635R.D
Sample Name: ckq635r

```
    ========================================================================
    Acq. Operator    : ckq
    Acq. Instrument  : Instrument 1                    Location : Vial 1
    Injection Date   : 2017-2-9 16:09:11
    Acq. Method      : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2017-2-9 16:04:26  by yl
                       (modified after loading)
    Analysis Method  : C:\CHEM32\1\METHODS\JWQ20121205.M
    Last changed     : 2017-2-9 17:24:13  by ckq
                       (modified after loading)
    Sample Info      : IA H/ I =90:10  1ml/min, 254nm
```

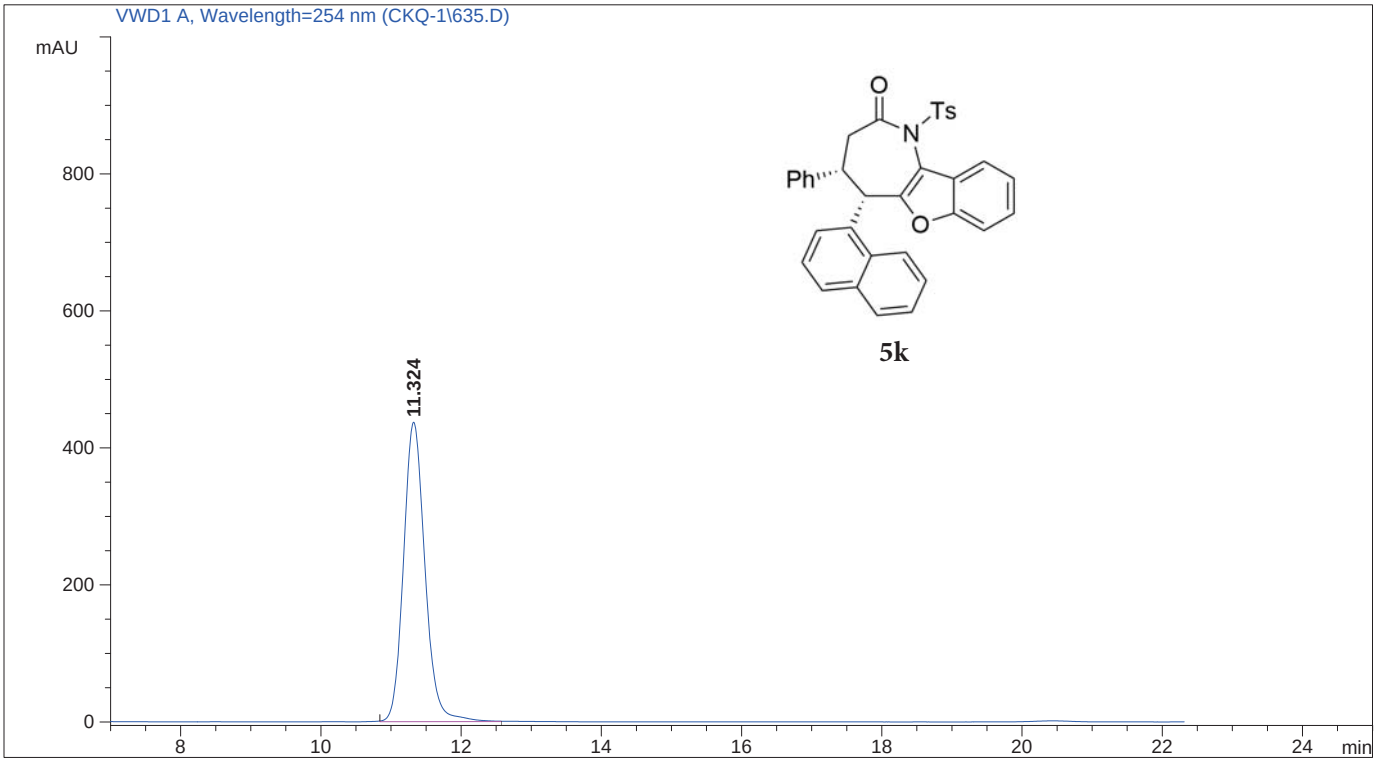


rac-**5k**

```
    ========================================================================
                              Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  11.451 VV    0.3205  940.79059   45.37791  50.1235
       2  20.596 BB    0.5949  936.15454   24.62747  49.8765

    Totals :                  1876.94513   70.00538


    ========================================================================
                              *** End of Report ***
```

Data File C:\CHEM32\1\DATA\CKQ-1\635.D
Sample Name: ckq635

```
==========================================================================
Acq. Operator   : ckq
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 2017-2-9 16:37:13
Acq. Method     : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2017-2-9 16:04:26  by yl
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\JWQ20121205.M
Last changed    : 2018-5-23 14:13:25  by ckq
                  (modified after loading)
Sample Info     : IA H/ I =90:10  1ml/min, 254nm
```

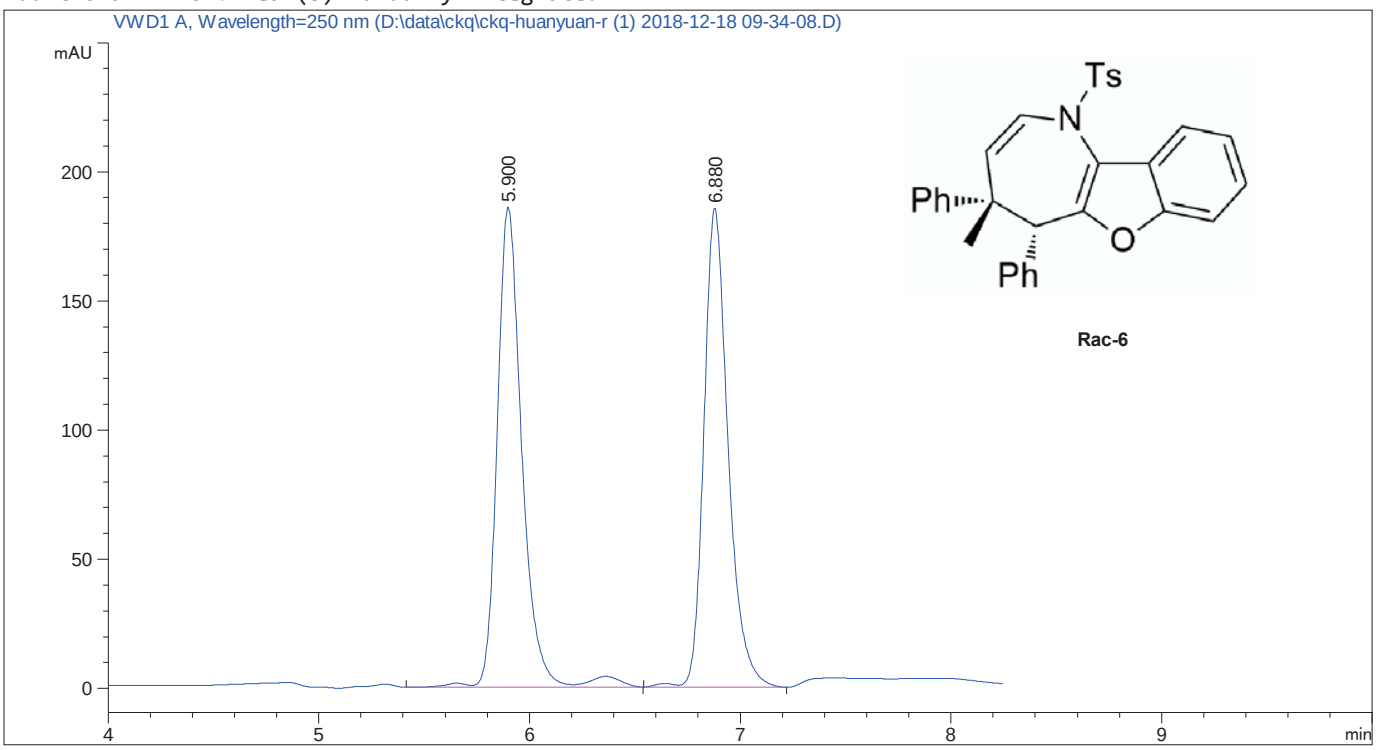


5k

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s    [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  11.324 BB    0.3295 9280.62207  436.65036 100.0000

Totals :                  9280.62207  436.65036

==========================================================================
                        *** End of Report ***
```

Data File D:\data\ckq\ckq-huanyuan-r (1) 2018-12-18 09-34-08.D
Sample Name: ckq-huanyuan-r

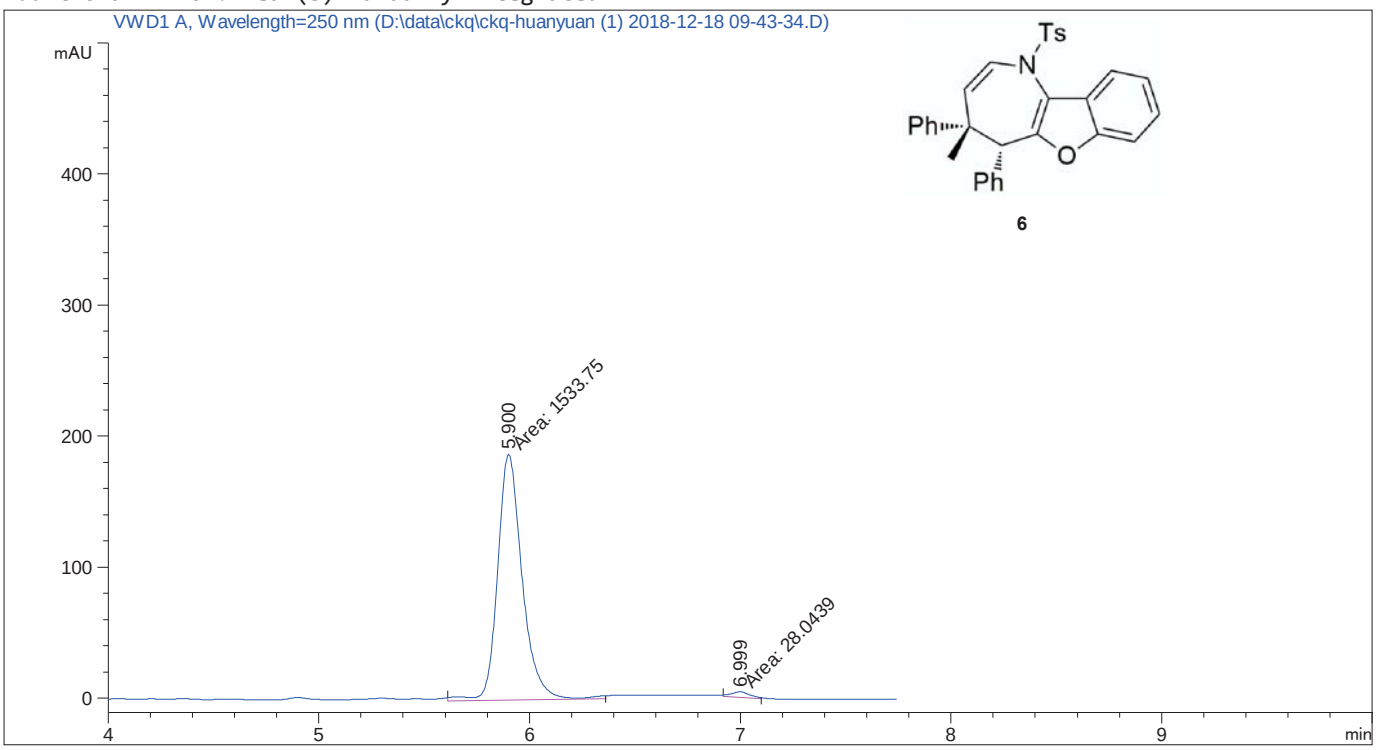
```
======================================================================
Acq. Operator   : ckq
Sample Operator : ckq
Acq. Instrument : HPLC                          Location :   P1-D-03
Injection Date  : 12/18/2018 9:34:48 AM              Inj :   1
                                                Inj Volume : 10.000 µl
Acq. Method     : C:\Users\Public\Documents\ChemStation\1\Methods
Last changed \DEF_LC.M     : 12/18/2018 9:42:13 AM by ckq
                  (modified after loading)
Analysis Method : C:\Users\Public\Documents\ChemStation\1\Methods\DEF_LC.M
Last changed    : 12/18/2018 11:27:42 AM by ckq
                  (modified after loading)
Sample Info     : IB, H/I = 90/10, 1.0 mL/min   254nm

Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=250 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.900 VV R  0.1218 1536.04810  185.83311  50.8773
   2   6.880 VB R  0.1213 1483.07324  185.40144  49.1227
```

Data File D:\data\ckq\ckq-huanyuan (1) 2018-12-18 09-43-34.D
Sample Name: ckq-huanyuan

```
======================================================================
Acq. Operator   : ckq
Sample Operator : ckq
Acq. Instrument : HPLC                          Location :   P1-D-03
Injection Date  : 12/18/2018 9:44:14 AM              Inj :   1
                                                Inj Volume : 10.000 µl
Acq. Method      : C:\Users\Public\Documents\ChemStation\1\Methods
Last changed \DEF_LC.M     : 12/18/2018 9:51:21 AM by ckq
                  (modified after loading)
Analysis Method : C:\Users\Public\Documents\ChemStation\1\Methods\DEF_LC.M
Last changed    : 12/18/2018 11:28:56 AM by ckq
                  (modified after loading)
Sample Info     : IB, H/I = 90/10, 1.0 mL/min   254nm

Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=250 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.900 MM    0.1364 1533.75183  187.45512  98.2044
   2   6.999 MM    0.1036   28.04395    4.51171   1.7956
```