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Supporting Information

Enantioselective N-Heterocyclic Carbene-Catalyzed Synthesis of Saccharin-derived Dihydropyridinones with *cis*-Selectivity

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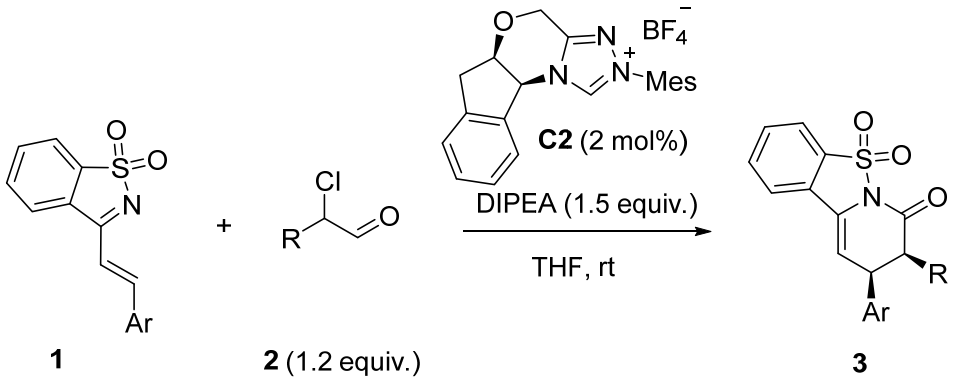
Part I Experimental part

1.1 General Information

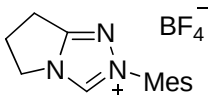
Unless otherwise indicated, all reactions were carried out under an N_2 atmosphere in oven-dried glassware with magnetic stirring. Anhydrous THF and toluene were distilled from sodium and benzophenone, CH_2Cl_2 was distilled from CaH_2. Chiral triazolium salts **B-C**,[1] were synthesized according to our previous reports and the literatures. saccharin-derived 1-azadienes 1 [2] and α-chloroaldehyde **2** [3] were prepared according to literature methods. Column chromatograph was performed on silica gel 200~300 mesh. All ^{1}H NMR (300 MHz), ^{13}C NMR (75 MHz) spectra were recorded on a Bruker-DMX 300 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). 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 are reported as follows: $[\alpha]_D^T$ (concentration (g/100 mL), solvent).

1.2 NHC-catalyzed synthesis of saccharin-derived dihydropyrindinones.

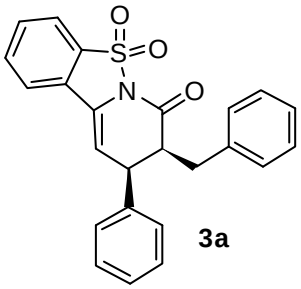


General procedure. To an oven-dried 25 mL Schlenk tube equipped with a stir bar was charged with triazolium salt **C2** (2.6 mg, 0.006 mmol), saccharin-derived 1-azadienes **1** (0.3 mmol). This tube was closed with a septum, evacuated, and back-filled with argon. To this mixture was added freshly distilled THF (3 mL). Then, the α-chloroaldehyde **2** (0.36 mmol) and DIPEA (0.45 mmol) were added. After stirring for 12 hours, the reaction mixture was diluted with diethyl ether and passed through a short silica pad. The solvent was removed under reduced pressure and the residue was purified by chromatography on silica gel (ethyl acetate/petroleum ether, typically 1/8) to give the desired dihydropyridinone **3**.

Racemic samples for the standard of chiral HPLC spectra were prepared using 10 mol% of achiral triazolium salt **S1** as the precatalyst.

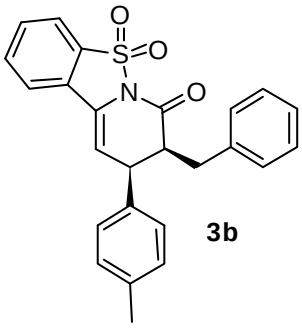


S1



3a

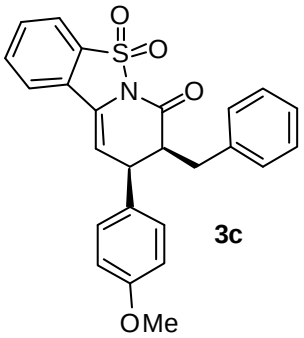
(*8S,9S*)-8-benzyl-9-phenyl-8,9-dihydro-7H-benzo[4,5]isothiazolo[2,3-a]pyridin-7-one 5,5-dioxide (3a). LZQ-660#, Yield: 106 mg (88%), Rf = 0.25 (ethyl acetate/petroleum ether, typically 1/5), white solid, mp 219-221°C, $[\alpha]_D^{20}$ +34.7 (*c* 1.0, CH_2Cl_2), HPLC analysis: >99% ee [Daicel CHIRALPAK IA column, 20 ^oC, 254 nm hexane/*i*-PrOH = 65:35, 1.0 mL/min, 254 nm, 15.7 min (major), 26.6 min (minor)]. ^1H NMR (300 MHz, $CDCl_3$) δ 7.85 (d, *J* = 7.7 Hz, 1H), 7.75 – 7.53 (m, 3H), 7.37 – 7.18 (m, 6H), 7.11 – 7.02 (m, 4H), 6.13 (d, *J* = 6.4 Hz, 1H), 3.74 (t, *J* = 6.8 Hz, 1H), 3.54 – 3.34 (m, 2H), 2.49 – 2.27 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.0, 138.6, 137.6, 134.1, 132.9, 131.0, 129.4, 129.2, 129.1, 128.7, 128.4, 128.2, 126.9, 126.7, 121.7, 121.7, 107.4, 48.3, 42.2, 31.1. IR (KBr) ν 3061.7 cm^{-1}, 1712.7 cm^{-1}, 1341.7 cm^{-1}, 1172.9 cm^{-1}. FTMS (ESI) *m/z*: $[M+Na]^+$ Calc. for $C_{24}H_{19}O_3NNaS^+$, 424.0978, Found 424.0974.



3b

(8S,9S)-8-benzyl-9-(p-tolyl)-8,9-dihydro-7H-benzo[4,5]isothiazolo[2,3-a]pyridin-7-one 5,5-dioxide (3b). LZQ-672#**,** Yield: 120 mg (97%), white solid, Rf = 0.06 (ethyl

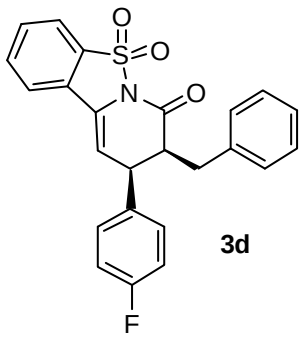
acetate/petroleum ether, typically 1/5), mp 102-103°C, $[\alpha]_D^{20}$ +27.9 (*c* 1.0, CH_2Cl_2), HPLC analysis: 99% ee [Daicel CHIRALPAK IA column, 20 °C, 254 nm hexane/*i*-PrOH = 75:25, 1.0 mL/min, 254 nm, 23.5 min (major), 27.2 min (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.87 (d, *J* = 7.5 Hz, 1H), 7.78 – 7.55 (m, 3H), 7.40 – 7.20 (m, 3H), 7.14 (d, *J* = 8.1 Hz, 2H), 7.10 – 7.04 (m, 2H), 6.98 (d, *J* = 8.1 Hz, 2H), 6.13 (d, *J* = 6.4 Hz, 1H), 3.71 (t, *J* = 6.7 Hz, 1H), 3.54 – 3.34 (m, 2H), 2.40 (dd, *J* = 14.3, 9.6 Hz, 1H), 2.34 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.1, 138.7, 138.0, 134.5, 134.1, 132.9, 131.0, 130.1, 129.2, 129.1, 128.7, 128.3, 127.0, 126.7, 121.9, 121.7, 107.7, 48.4, 41.8, 31.1, 21.2. IR (KBr) ν 3027.7 cm^{-1}, 1713.7 cm^{-1}, 1342.5 cm^{-1}, 1173.5 cm^{-1}. FTMS (ESI) *m/z*: $[M+Na]^+$ Calc. for $C_{25}H_{21}O_3NNaS^+$, 438.1134, Found 438.1131.



3c

(8S,9S)-8-benzyl-9-(4-methoxyphenyl)-8,9-dihydro-7H-benzo[4,5]isothiazolo[2,3-a]pyridin-7-one 5,5-dioxide (3c). LZQ-667#**,** Yield: 115 mg (89%), white solid, Rf = 0.12 (ethyl acetate/petroleum ether, typically 1/5), mp 94-95°C, $[\alpha]_D^{20}$ +30.2 (*c* 1.0, CH_2Cl_2), HPLC analysis: >99% ee [Daicel CHIRALPAK IA column, 20 °C, 254 nm hexane/*i*-PrOH = 65:35, 1.0 mL/min, 254 nm, 21.5 min (major), 30.3 min (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.7 Hz, 1H), 7.75 – 7.56 (m, 3H), 7.31 – 7.22 (m, 3H), 7.10 – 7.04 (m, 2H), 7.00 – 6.98 (m, 2H), 6.85 (d, *J* = 8.9 Hz, 1H), 6.13

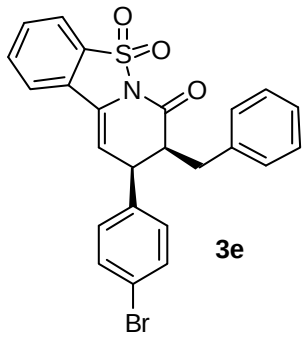
(d, J = 6.4 Hz, 1H), 3.79 (s, 3H), 3.69 (t, J = 6.6 Hz, 1H), 3.52 – 3.34 (m, 2H), 2.38 (dd, J = 14.1, 9.5 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.1, 159.4, 138.7, 134.1, 132.9, 131.0, 129.6, 129.4, 129.1, 128.9, 128.7, 127.0, 126.7, 121.8, 121.7, 114.7, 107.8, 55.4, 48.5, 41.4, 31.1. IR (KBr) ν 3062.6 cm^{-1}, 1715.8 cm^{-1}, 1342.5 cm^{-1}, 1176.5 cm^{-1}. FTMS (ESI) *m/z*: $[M+Na]^+$ Calc. for $C_{25}H_{21}O_4NNaS^+$, 454.1084, Found 454.1077.



3d

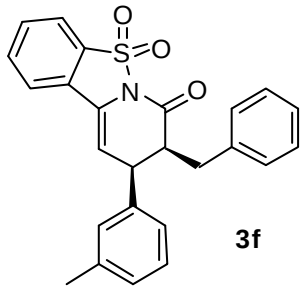
(8S,9S)-8-benzyl-9-(4-fluorophenyl)-8,9-dihydro-7H-benzo[4,5]isothiazolo[2,3-a] pyridin-7-one 5,5-dioxide (3d). LZQ-668#**,** Yield: 112 mg (90%), white solid, Rf = 0.08 (ethyl acetate/petroleum ether, typically 1/5), mp 138-140°C, $[\alpha]_D^{20}$ +29.3 (*c* 1.0, CH_2Cl_2), HPLC analysis: >99 % ee [Daicel CHIRALPAK IA column, 20 ^{o}C, 254 nm hexane/*i*-PrOH = 65:35, 1.0 mL/min, 254 nm, 15.0 min (major), 19.9 min (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.81 (d, J = 7.8 Hz, 1H), 7.70 (d, J = 5.1 Hz, 2H), 7.61 (ddd, J = 8.2, 5.3, 3.2 Hz, 1H), 7.35 – 7.16 (m, 3H), 7.06 – 6.92 (m, 6H), 6.15 (d, J = 6.3 Hz, 1H), 3.74 (t, J = 6.7 Hz, 1H), 3.53 – 3.33 (m, 2H), 2.32 (dd, J = 15.6, 10.8 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.9, 164.0, 160.7, 138.2, 134.2, 133.3, 133.3, 132.7, 131.1, 130.1, 130.0, 129.2, 128.9, 128.7, 126.8, 126.7, 121.8, 121.6, 116.3, 116.0, 107.3, 53.6, 48.0, 41.3, 31.1. IR (KBr) ν 3064.8 cm^{-1}, 1715.3 cm^{-1}, 1343.9 cm^{-1}, 1174.3 cm^{-1}. FTMS (ESI) *m/z*: $[M+Na]^+$ Calc. for $C_{24}H_{18}O_3NFNaS^+$, 442.0884, Found

442.0878.



3e

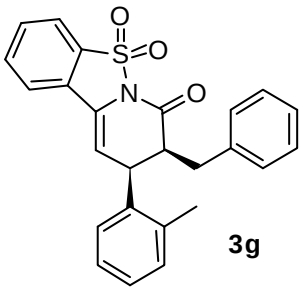
(8S,9S)-8-benzyl-9-(4-bromophenyl)-8,9-dihydro-7H-benzo[4,5]isothiazolo[2,3-a] pyridin-7-one 5,5-dioxide (3e). LZQ-669#**,** Yield: 120 mg (83%), white solid, Rf = 0.10 (ethyl acetate/petroleum ether, typically 1/5), mp 113-114°C, $[\alpha]_D^{20}$ +27.4 (*c* 1.0, CH_2Cl_2), HPLC analysis: >99% ee [Daicel CHIRALPAK IB column, 20 ^oC, 254 nm hexane/*i*-PrOH = 75:25, 1.0 mL/min, 254 nm, 23.1 min (major), 27.6 min (minor)]. ^1H NMR (300 MHz, $CDCl_3$) δ 7.85 (d, *J* = 7.7 Hz, 1H), 7.78 – 7.54 (m, 3H), 7.49 – 7.40 (m, 2H), 7.32 – 7.16 (m, 3H), 7.07 – 7.00 (m, 2H), 6.99 – 6.87 (m, 2H), 6.10 (d, *J* = 6.3 Hz, 1H), 3.71 (t, *J* = 6.7 Hz, 1H), 3.56 – 3.31 (m, 2H), 2.48 – 2.22 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.7, 138.2, 136.6, 134.2, 132.9, 132.5, 131.2, 130.1, 129.5, 129.0, 128.8, 126.8, 126.8, 122.2, 121.8, 121.8, 106.7, 47.9, 41.5, 31.1. IR (KBr) ν 2924.7 cm^{-1}, 1714.6 cm^{-1}, 1341.3 cm^{-1}, 1174.4 cm^{-1}. FTMS (ESI) *m/z*: $[M+Na]^+$ Calc. for $C_{24}H_{18}O_3NBrNaS^+$, 502.0083, Found 502.0076.



3f

(8S,9S)-8-benzyl-9-(m-tolyl)-8,9-dihydro-7H-benzo[4,5]isothiazolo[2,3-a]pyridin-

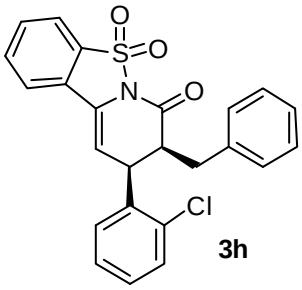
7-one 5,5-dioxide (3f). LZQ-663#**,** Yield: 115 mg (93%), white solid, Rf = 0.17 (ethyl acetate/petroleum ether, typically 1/5), mp 189-190°C, $[\alpha]_D^{20}$ +26.9 (*c* 1.0, CH_2Cl_2), HPLC analysis: >99% ee [Daicel CHIRALPAK IA column, 20 °C, 254 nm hexane/*i*-PrOH = 80:20, 0.5 mL/min, 254 nm, 25.2 min (major), 39.1 min (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.87 (d, *J* = 7.7 Hz, 1H), 7.71 – 7.59 (m, 3H), 7.34 – 7.17 (m, 4H), 7.13 – 7.03 (m, 3H), 6.95 – 6.83 (m, 2H), 6.12 (d, *J* = 6.3 Hz, 1H), 3.71 (t, *J* = 6.6 Hz, 1H), 3.54 – 3.30 (m, 2H), 2.39 (dd, *J* = 13.9, 9.1 Hz, 1H), 2.32 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.1, 139.0, 138.7, 137.5, 134.1, 132.9, 131.0, 129.3, 129.2, 129.2, 128.9, 128.6, 127.0, 126.7, 125.4, 121.7, 121.7, 107.5, 48.3, 42.2, 31.2, 21.6. IR (KBr) ν 3061.4 cm^{-1}, 1715.0 cm^{-1}, 1343.1 cm^{-1}, 1172.8 cm^{-1}. FTMS (ESI) *m/z*: $[M+Na]^+$ Calc. for $C_{25}H_{21}O_3NNaS^+$, 438.1134, Found 438.1131.



3g

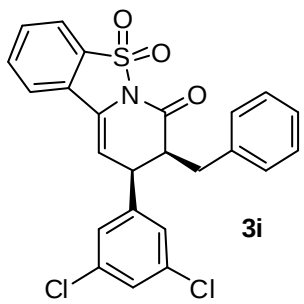
(8S,9S)-8-benzyl-9-(o-tolyl)-8,9-dihydro-7H-benzo[4,5]isothiazolo[2,3-a]pyridin-7-one 5,5-dioxide (3g). LZQ-662#**,** Yield: 120 mg (98%), white solid, Rf = 0.23 (ethyl acetate/petroleum ether, typically 1/5), mp 224-225°C, $[\alpha]_D^{20}$ +29.2 (*c* 1.0, CH_2Cl_2), HPLC analysis: 99% ee [Daicel CHIRALPAK IA column, 20 °C, 254 nm hexane/*i*-PrOH = 80:20, 1.0 mL/min, 254 nm, 17.8 min (major), 20.5 min (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.7 Hz, 1H), 7.75 – 7.50 (m, 3H), 7.28 – 7.10 (m, 7H), 6.85 (dd, *J* = 7.5, 2.0 Hz, 2H), 6.04 (d, *J* = 6.2 Hz, 1H), 3.96 (t, *J* = 6.7

Hz, 1H), 3.61 – 3.32 (m, 2H), 2.54 (dd, J = 13.9, 9.9 Hz, 1H), 1.96 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.6, 138.3, 136.4, 135.9, 134.1, 132.6, 131.1, 130.9, 128.9, 128.8, 128.7, 127.8, 127.5, 127.1, 127.0, 126.7, 121.7, 121.6, 106.2, 47.2, 37.1, 31.4, 19.5. IR (KBr) ν 3063.0 cm^{-1}, 1712.2 cm^{-1}, 1341.1 cm^{-1}, 1173.8 cm^{-1}. FTMS (ESI) *m/z*: $[M+Na]^+$ Calc. for $C_{25}H_{21}O_3NNaS^+$, 438.1134, Found 438.1131.



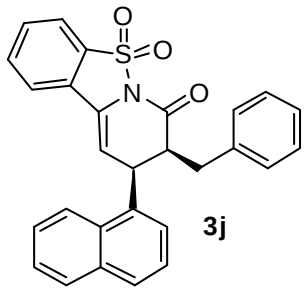
3h

(8S,9S)-8-benzyl-9-(2-chlorophenyl)-8,9-dihydro-7H-benzo[4,5]isothiazolo[2,3-a] pyridin-7-one 5,5-dioxide (3h). LZQ-666**#,** Yield: 133 mg (98%), white solid, Rf = 0.10 (ethyl acetate/petroleum ether, typically 1/5), mp 208-210°C, $[\alpha]_D^{20}$ +23.6 (*c* 1.0, CH_2Cl_2), HPLC analysis: 99% ee [Daicel CHIRALPAK IA column, 20 ^{o}C, 254 nm hexane/*i*-PrOH = 70:30, 1.0 mL/min, 254 nm, 11.3 min (major), 13.6 min (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.85 (d, J = 7.8 Hz, 1H), 7.72 – 7.58 (m, 3H), 7.42 (d, J = 7.0 Hz, 1H), 7.28 – 7.20 (m, 7H), 6.94 (d, J = 7.3 Hz, 2H), 6.12 (d, J = 6.3 Hz, 1H), 4.38 (t, J = 6.6 Hz, 1H), 3.45 (ddd, J = 21.1, 13.4, 5.1 Hz, 2H), 2.58 (dd, J = 13.9, 8.9 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.2, 138.2, 135.6, 134.2, 133.9, 132.7, 131.1, 130.3, 129.5, 129.2, 129.0, 128.7, 128.6, 128.2, 126.8, 126.7, 121.6, 121.7, 105.2, 47.3, 38.1, 31.4. IR (KBr) ν 3063.9 cm^{-1}, 1713.1 cm^{-1}, 1343.5 cm^{-1}, 1173.6 cm^{-1}. FTMS (ESI) *m/z*: $[M+Na]^+$ Calc. for $C_{15}H_{26}O_4N_2Cl_3NaS^+$, 458.0571, Found 454.0578.



3i

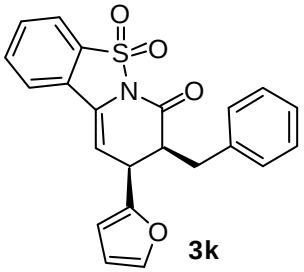
(8S,9S)-8-benzyl-9-(3,5-dichlorophenyl)-8,9-dihydro-7H-benzo[4,5]isothiazolo[2,3-a]pyridin-7-one 5,5-dioxide (3i). LZQ-675#**,** Yield: 140 mg (99%), white solid, Rf = 0.18 (ethyl acetate/petroleum ether, typically 1/5), mp 167-168 °C, $[\alpha]_D^{20}$ +29.3 (*c* 1.0, CH_2Cl_2), HPLC analysis: 99% ee [Daicel CHIRALPAK IA column, 20 ^oC, 254 nm hexane/*i*-PrOH = 80:20, 1.0 mL/min, 254 nm, 18.4 min (major), 20.7 min (minor)]. ^1H NMR (300 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.7 Hz, 1H), 7.73 – 7.59 (m, 3H), 7.43 (d, *J* = 2.2 Hz, 1H), 7.31 – 7.18 (m, 6H), 6.94 (dd, *J* = 7.5, 1.8 Hz, 2H), 6.07 (d, *J* = 6.2 Hz, 1H), 4.32 (t, *J* = 6.7 Hz, 1H), 3.59 – 3.32 (m, 2H), 2.54 (dd, *J* = 14.0, 9.1 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.0, 138.0, 134. 7, 134.4, 131.3, 130.1, 129.8, 129.4, 128.9, 128.8, 128.5, 126.9, 126.7, 121. 8, 104.6, 47.1, 37.6, 31.4. IR (KBr) ν 3064.9 cm^{-1}, 1714.1 cm^{-1}, 1345.8 cm^{-1}, 1176.0 cm^{-1}. FTMS (ESI) *m/z*: $[M+Na]^+$ Calc. for $C_{24}H_{17}O_3NCl_2NaS^+$, 492.0198, Found 492.0192.



3j

(8S,9S)-8-benzyl-9-(naphthalen-1-yl)-8,9-dihydro-7H-benzo[4,5]isothiazolo[2,3-a]pyridin-7-one 5,5-dioxide (3j). LZQ-673#**,** Yield: 126 mg (93%), white solid, Rf =

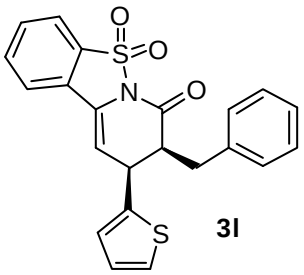
0.12 (ethyl acetate/petroleum ether, typically 1/5), mp 204-205 °C, $[\alpha]_D^{20}$ +30.4 (*c* 1.0, CH_2Cl_2), HPLC analysis: >99% ee [Daicel CHIRALPAK IB column, 20 °C, 254 nm hexane/*i*-PrOH = 60:40, 1.0 mL/min, 254 nm, 16.6 min (major), 29.9 min (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.86 – 7.66 (m, 4H), 7.59 – 7.47 (m, 3H), 7.46 – 7.37 (m, 3H), 7.08 – 6.98 (m, 3H), 6.70 (dd, *J* = 7.5, 2.1 Hz, 2H), 6.13 (d, *J* = 5.9 Hz, 1H), 4.65 (t, *J* = 6.4 Hz, 1H), 3.52 – 3.31 (m, 2H), 2.63 – 2.45 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.5, 138.4, 134.2, 134.1, 133.9, 132.7, 131.2, 131.0, 129.4, 129.3, 129.1, 128.6, 128.6, 126.9, 126.8, 126.6, 126.1, 126.1, 125.0, 122.4, 121.7, 121.7, 106.7, 48.2, 36.4, 31.5. IR (KBr) ν 3061.2 cm^{-1}, 1712.6 cm^{-1}, 1343.1 cm^{-1}, 1174.2 cm^{-1}. FTMS (ESI) *m/z*: $[M+Na]^+$ Calc. for $C_{28}H_{21}O_3NNaS^+$, 474.1134, Found 474.1130.



3k

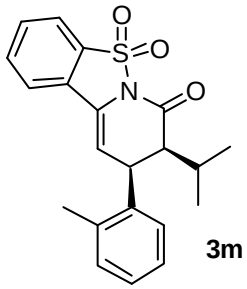
(8S,9S)-8-benzyl-9-(thiophen-2-yl)-8,9-dihydro-7H-benzo[4,5]isothiazolo[2,3-a]pyridin-7-one 5,5-dioxide (3k). LZQ-676#**,** Yield: 100 mg (86%), white solid, Rf = 0.06 (ethyl acetate/petroleum ether, typically 1/5), mp 190-191 °C, $[\alpha]_D^{20}$ +28.6 (*c* 1.0, CH_2Cl_2), HPLC analysis: >99% ee [Daicel CHIRALPAK IA column, 20 °C, 254 nm hexane/*i*-PrOH = 70:30, 1.0 mL/min, 254 nm, 17.5 min (major), 24.0 min (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.5 Hz, 1H), 7.76 – 7.57 (m, 3H), 7.39 (d, *J* = 2.2 Hz, 1H), 7.37 – 7.19 (m, 4H), 7.20 – 7.08 (m, 2H), 6.33 (dd, *J* = 3.3, 1.9 Hz, 1H), 6.16 (d, *J* = 3.2 Hz, 1H), 6.00 (d, *J* = 6.4 Hz, 1H), 3.82 (t, *J* = 6.4 Hz, 1H), 3.55 (dd, *J* = 14.4, 4.0 Hz, 1H), 3.25 (ddd, *J* = 10.4, 6.4, 4.0 Hz, 1H), 2.39 (dd, *J* = 14.4,

10.4 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.5, 150.5, 143.2, 138.6, 134.1, 133.0, 131.2, 130.5, 129.2, 128.7, 126.8, 121.8, 121.7, 110.5, 108.6, 103.7, 47.9, 35.1, 31.4. IR (KBr) ν 3064.0 cm^{-1}, 1716.1 cm^{-1}, 1341.3 cm^{-1}, 1174.5 cm^{-1}. FTMS (ESI) *m/z*: $[M+Na]^+$ Calc. for $C_{22}H_{17}O_4NNaS^+$, 414.0771, Found 414.0765.



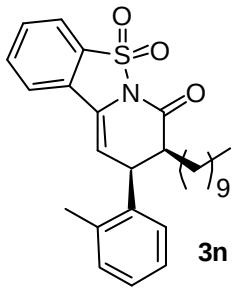
3l

(8S,9S)-8-benzyl-9-(thiophen-2-yl)-8,9-dihydro-7H-benzo[4,5]isothiazolo[2,3-a]pyridin-7-one 5,5-dioxide (3l). LZQ-670**#,** Yield: 105 mg (86%), white solid, Rf = 0.08 (ethyl acetate/petroleum ether, typically 1/5), mp 232-233 °C, $[\alpha]_D^{20}$ +29.2 (*c* 1.0, CH_2Cl_2), HPLC analysis: >99% ee [Daicel CHIRALPAK IA column, 20 ^{o}C, 254 nm hexane/*i*-PrOH = 65:35, 1.0 mL/min, 254 nm, 17.7 min (major), 29.3 min (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.87 (d, *J* = 7.5 Hz, 1H), 7.66 (pd, *J* = 8.5, 7.5, 4.0 Hz, 3H), 7.37 – 7.21 (m, 4H), 7.19 – 7.11 (m, 2H), 6.98 (dd, *J* = 5.2, 3.4 Hz, 1H), 6.87 (d, *J* = 3.4 Hz, 1H), 6.19 (d, *J* = 6.4 Hz, 1H), 4.04 (t, *J* = 6.4 Hz, 1H), 3.52 (dd, *J* = 14.5, 4.2 Hz, 1H), 3.36 (ddd, *J* = 10.3, 6.3, 4.2 Hz, 1H), 2.53 (dd, *J* = 14.5, 10.0 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.4, 139.7, 138.5, 134.2, 133.1, 131.2, 129.6, 129.2, 128.8, 127.5, 126.8, 126.6, 125.6, 121.9, 121.8, 107.1, 49.3, 36.8, 31.1. IR (KBr) ν 3064.8 cm^{-1}, 1715.6 cm^{-1}, 1342.4 cm^{-1}, 1173.2 cm^{-1}. FTMS (ESI) *m/z*: $[M+Na]^+$ Calc. for $C_{22}H_{17}O_3NNaS_2^+$, 430.0542, Found 430.0539.



3m

(8S,9S)-8-isopropyl-9-(o-tolyl)-8,9-dihydro-7H-benzo[4,5]isothiazolo[2,3-a]pyridin-7-one 5,5-dioxide (3m). LZQ-690#**,** Yield: 65 mg (60 %), white solid, Rf = 0.17 (ethyl acetate/petroleum ether, typically 1/5), mp 190-191 °C, $[\alpha]_D^{20}$ +14.6 (*c* 1.0, CH_2Cl_2), HPLC analysis: >99% ee [Daicel CHIRALPAK IA column, 20 ^oC, 254 nm hexane/*i*-PrOH = 80:20, 1.0 mL/min, 254 nm, 15.0 min (major), 16.3 min (minor)]. ^1H NMR (300 MHz, $CDCl_3$) δ 7.82 (d, *J* = 7.8 Hz, 1H), 7.74 – 7.67 (m, 2H), 7.59 (ddd, *J* = 8.2, 5.6, 2.9 Hz, 1H), 7.27 (dq, *J* = 4.8, 2.1 Hz, 1H), 7.20 (d, *J* = 3.3 Hz, 3H), 6.18 (d, *J* = 5.1 Hz, 1H), 4.42 (dd, *J* = 7.0, 4.9 Hz, 1H), 2.82 (t, *J* = 6.7 Hz, 1H), 2.42 (s, 3H), 2.04 (h, *J* = 6.7 Hz, 1H), 1.10 (d, *J* = 6.7 Hz, 3H), 0.76 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.6, 136.9, 135.3, 134.0, 132.7, 131.2, 130.9, 129.4, 127.7, 127.3, 127.1, 126.8, 121.7, 121.6, 106.5, 51.1, 38.7, 26.7, 21.8, 21.1, 19.6. IR (KBr) ν 3066.7 cm^{-1}, 1709.0cm^{-1}, 1371.7 cm^{-1}, 1174.4 cm^{-1}. FTMS (ESI) *m/z*: $[M+Na]^+$ Calc. for $C_{21}H_{21}O_3NNaS^+$, 390.1134, Found 390.1129.



3n

(8S,9S)-8-decyl-9-(o-tolyl)-8,9-dihydro-7H-benzo[4,5]isothiazolo[2,3-a]pyridin-7-

one 5,5-dioxide (3n). LZQ-689#**,** Yield: 114 mg (82%), white solid, Rf = 0.20 (ethyl acetate/petroleum ether, typically 1/5), mp 44-45 °C, $[\alpha]_D^{20}$ +11.9 (*c* 1.0, CH_2Cl_2), HPLC analysis: 99% ee [Daicel CHIRALPAK IA column, 20 ^oC, 254 nm hexane/*i*-PrOH = 70:30, 1.0 mL/min, 254 nm, 10.9 min (major), 16.0 min (minor)]. ^1H NMR (300 MHz, $CDCl_3$) δ 7.72 (d, *J* = 7.8 Hz, 1H), 7.65 – 7.57 (m, 2H), 7.51 (ddd, *J* = 8.2, 5.8, 2.6 Hz, 1H), 7.09 (dd, *J* = 5.4, 3.8 Hz, 4H), 6.06 (d, *J* = 5.1 Hz, 1H), 4.29 (dd, *J* = 6.9, 5.0 Hz, 1H), 2.88 (q, *J* = 6.7 Hz, 1H), 2.33 (s, 3H), 1.39 – 0.97 (m, 18H), 0.78 (t, *J* = 6.9 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.0, 136.5, 135.6, 134.1, 132.6, 131.0, 130.8, 129.2, 127.6, 127.6, 127.0, 126.9, 121.7, 121.6, 106.5, 45.7, 38.8, 31.9, 29.6, 29.5, 29.4, 29.3, 27.3, 25.7, 22.7, 19.7, 14.2. IR (KBr) ν 2924.2 cm^{-1}, 1712.7 cm^{-1}, 1343.2 cm^{-1}, 1174.9 cm^{-1}. HRMS (ESI) *m/z*: $[M+H]^+$ Calc. for $C_{30}H_{23}ClNO_2^+$, 464.1411, Found 464.1412.

1.3 X-ray crystal structure of 3a

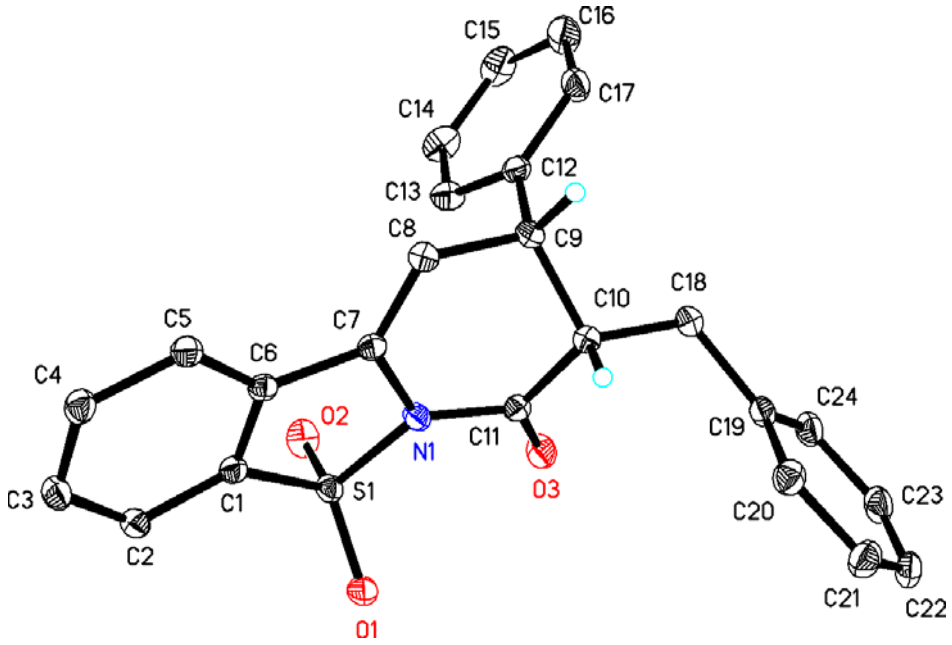


Figure S1 X-ray crystal structure of **3a**

The crystal was prepared from the solution of **3a** in dichloromethane/petroleum ether (1/10).

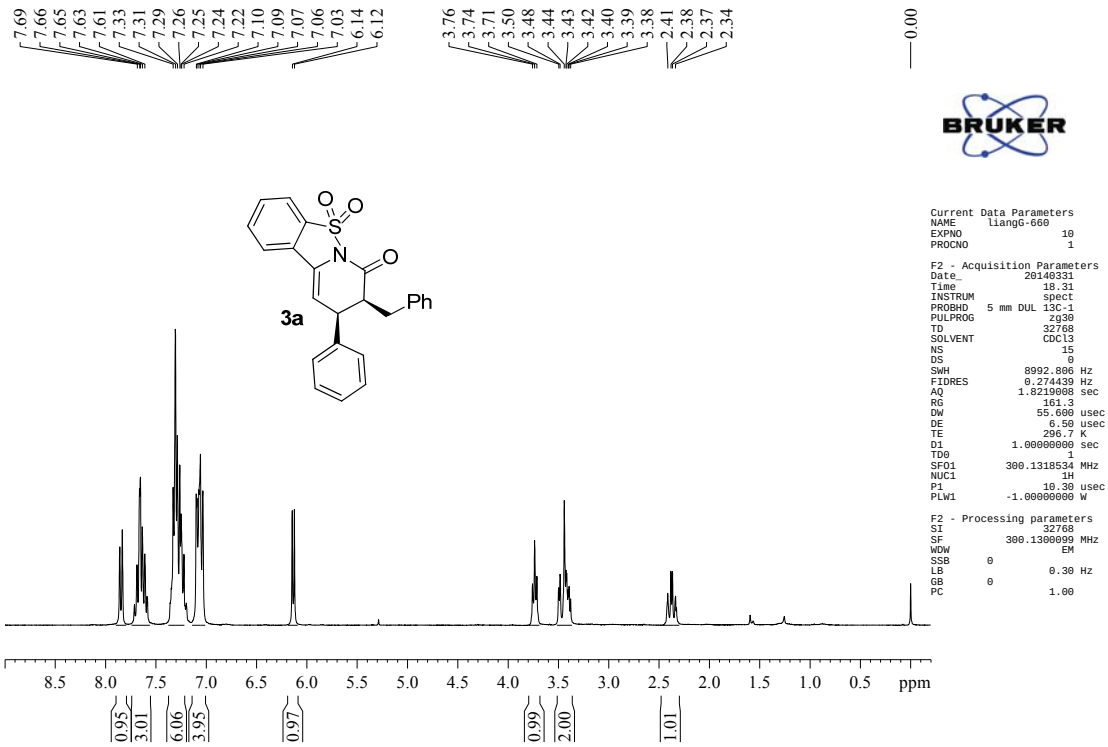
1.4 References

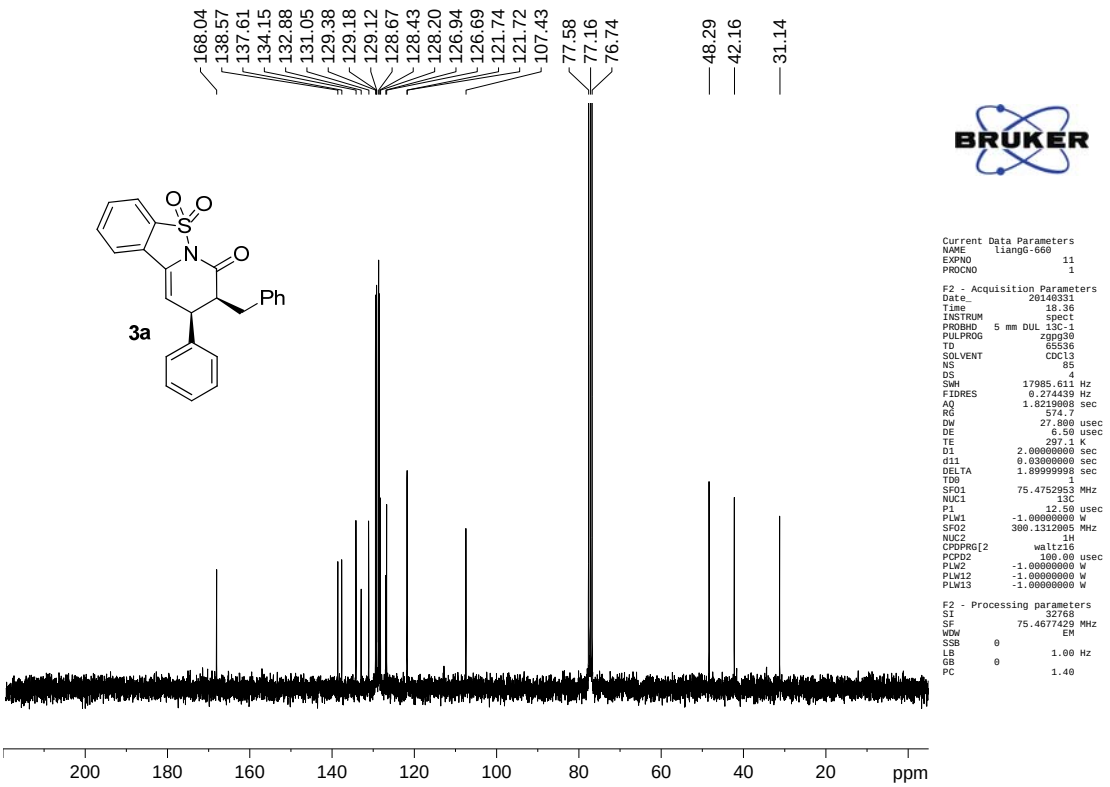
1 (a) Y.-R. Zhang, L. He, X. Wu, P.-L. Shao and S. Ye, *Org. Lett.*, 2008, **10**, 277; (b) H. Lv, Y.-R. Zhang, X.-L. Huang and S. Ye, *Adv. Synth. Catal.*, 2008, **350**, 2715; (c) L. He, Y.-R. Zhang, X.-L. Huang and S. Ye, *Synthesis*, 2008, 2825; (d) X. L. Huang, L. He, P. L. Shao and S. Ye, *Angew. Chem., Int. Ed.*, 2009, **48**, 192.

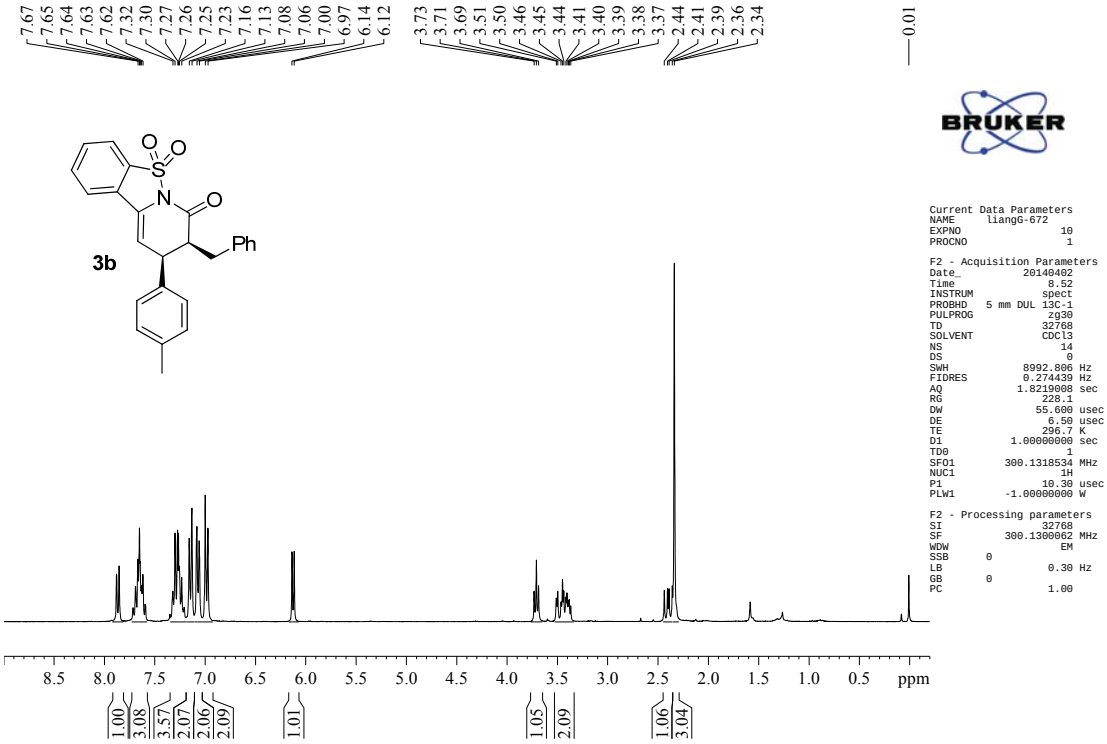
2 X. Feng, Z. Zhou, C. Ma, X. Yin, R. Li, L. Dong and Y.-C. Chen, *Angew. Chem., Int. Ed.*, 2013, **52**, 14173.

3 (a) M. He, B. J. Beahm and J. W. Bode, *Org. Lett.*, 2008, **10**, 3817; (b) T. Borg, J. Danielsson and P. Somfai, *Chem. Commun.*, 2010, **46**, 1281.

Part II NMR Spectra





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FIDRES          0.274439 Hz
AQ             1.8219008 sec
RG                 228.1
DW                55.600 usec
DE                 6.50 usec
TE                296.7 K
D1             1.00000000 sec
TD0                    1
SFO1         300.1318534 MHz
NUC1                  1H
P1                 10.30 usec
PLW1         -1.00000000 W

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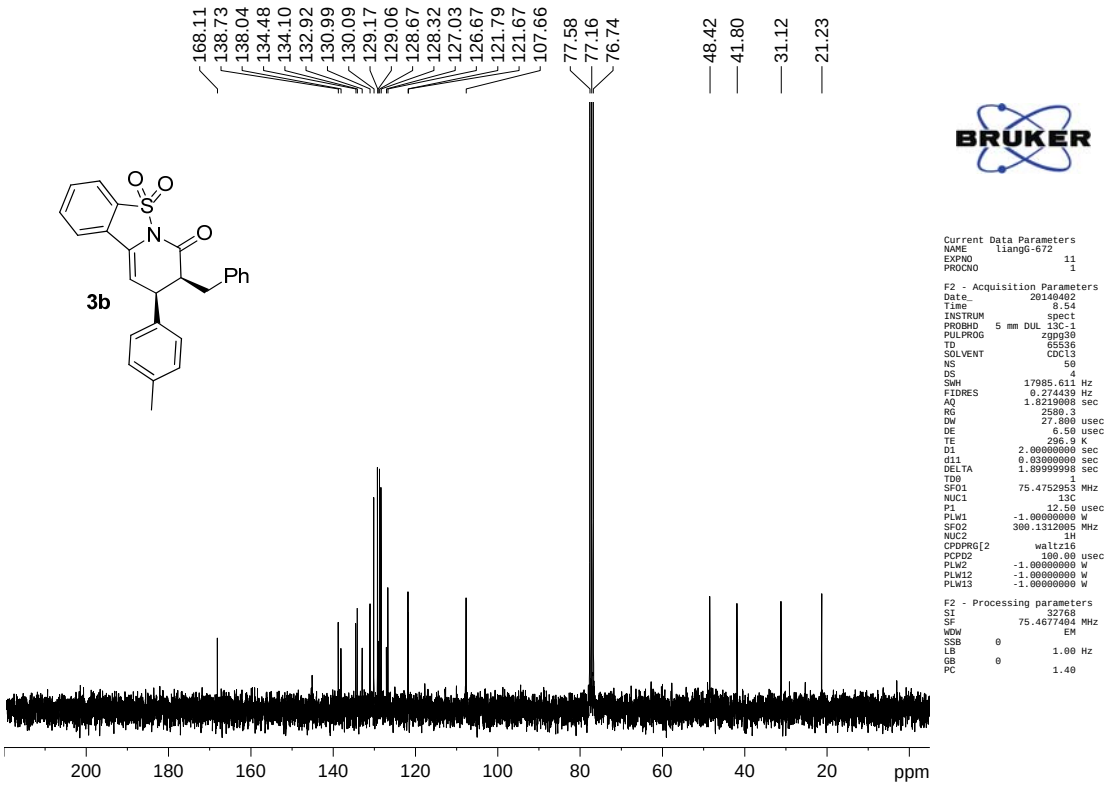


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PROCNO                 1

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SOLVENT            CDCl3
NS                    50
DS                     4
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FIDRES          0.274439 Hz
AQ             1.8219008 sec
RG                2580.3
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DE                 6.50 usec
TE                296.9 K
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d11            0.03000000 sec
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SFO1           75.4752953 MHz
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PLW1         -1.00000000 W
SFO2         300.1312005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2             100.00 usec
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PLW12        -1.00000000 W
PLW13        -1.00000000 W

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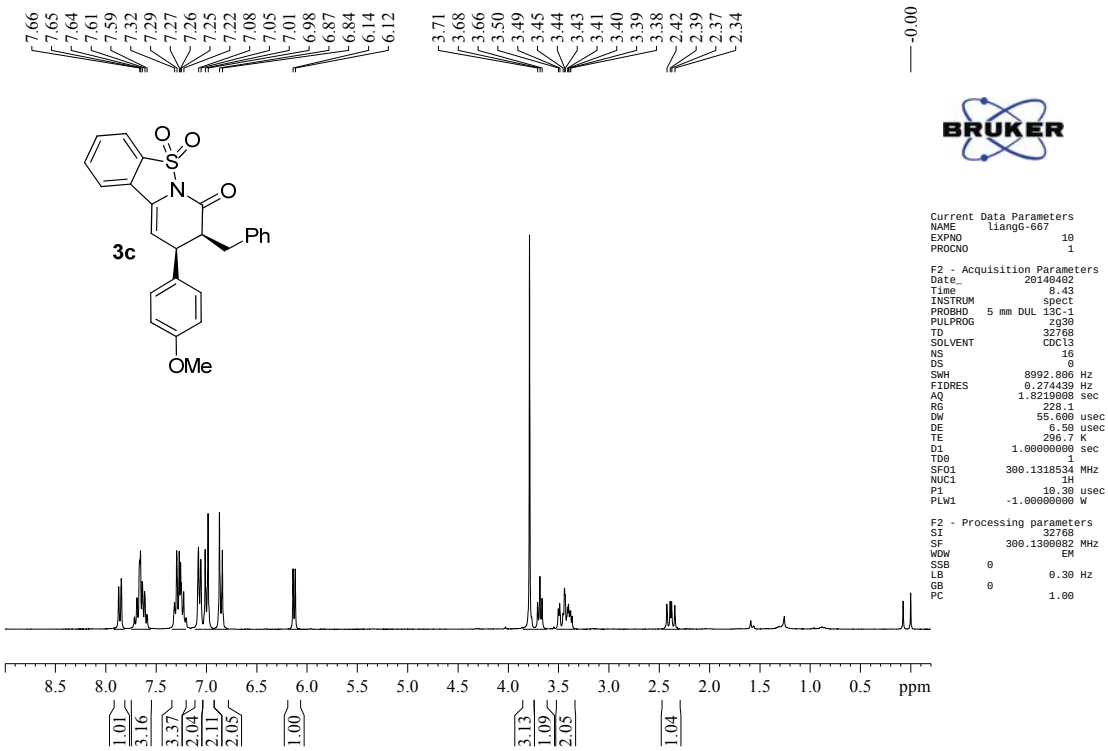


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PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              8992.806 Hz
FIDRES           0.274439 Hz
AQ               1.8219008 sec
RG                   228.1
DW                  55.600 usec
DE                    6.50 usec
TE                   296.7 K
D1            1.00000000 sec
TD0                     1
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NUC1                   1H
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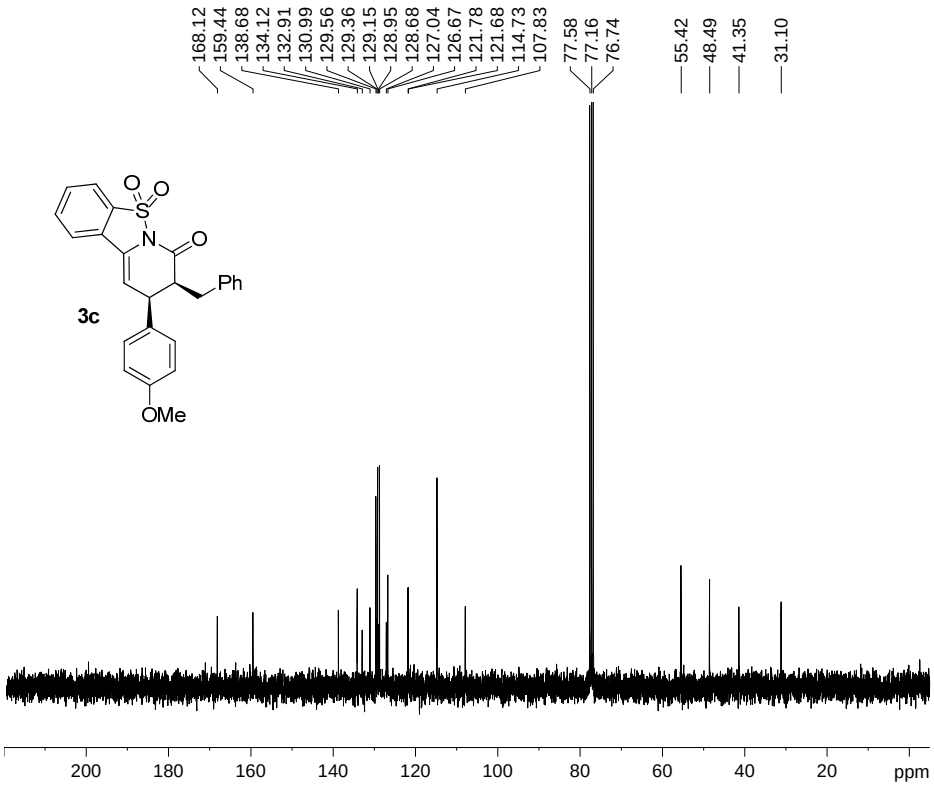


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FIDRES           0.274439 Hz
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DE                    6.50 usec
TE                   297.0 K
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d11           0.03000000 sec
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NUC1                  13C
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NUC2                   1H
CPDPRG[2          waltz16
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PLW12        -1.00000000 W
PLW13        -1.00000000 W

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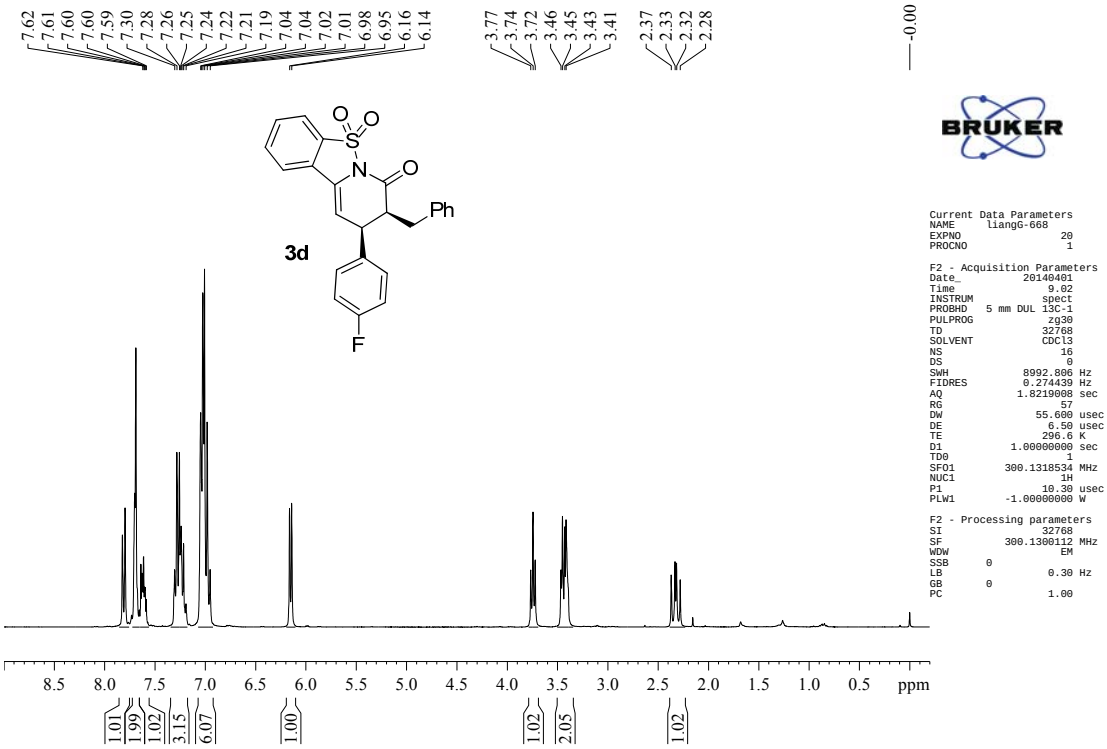


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PROCNO                1

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PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8992.806 Hz
FIDRES          0.274439 Hz
AQ             1.8219008 sec
RG                    57
DW                55.600 usec
DE                  6.50 usec
TE                 296.6 K
D1            1.00000000 sec
TD0                    1
SFO1          300.1318534 MHz
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PLW1         -1.00000000 W

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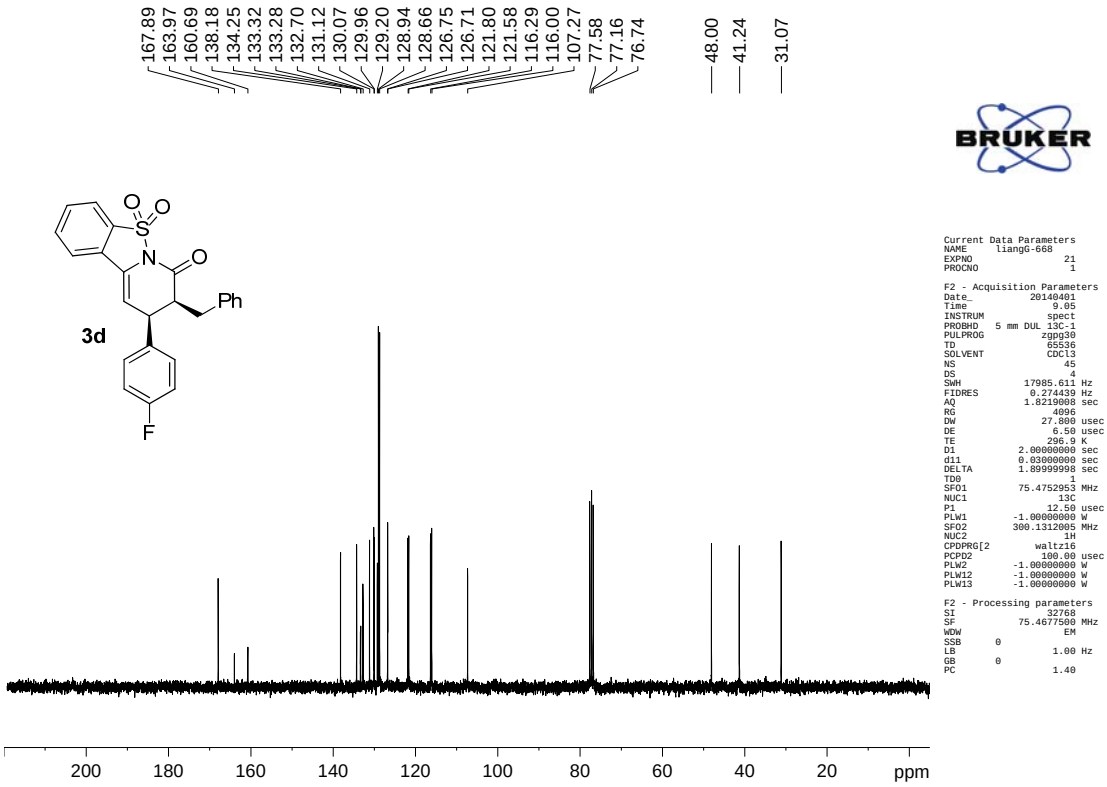


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PROCNO                1

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Time                9.05
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PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    45
DS                     4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ             1.8219008 sec
RG                  4096
DW                27.800 usec
DE                  6.50 usec
TE                 296.9 K
D1            2.00000000 sec
d11           0.03000000 sec
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TD0                    1
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NUC1                  13C
P1                 12.50 usec
PLW1         -1.00000000 W
SFO2          300.1312005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2             100.00 usec
PLW2         -1.00000000 W
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PLW13        -1.00000000 W

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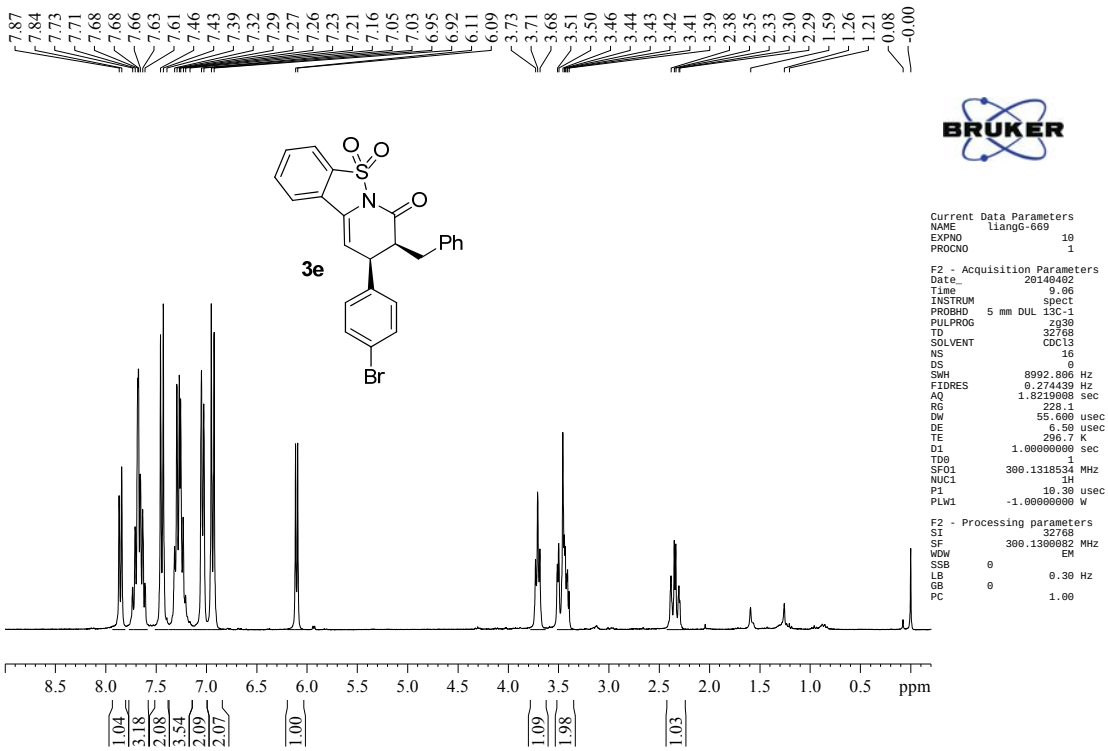


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RG                 228.1
DW                55.600 usec
DE                 6.50 usec
TE                296.7 K
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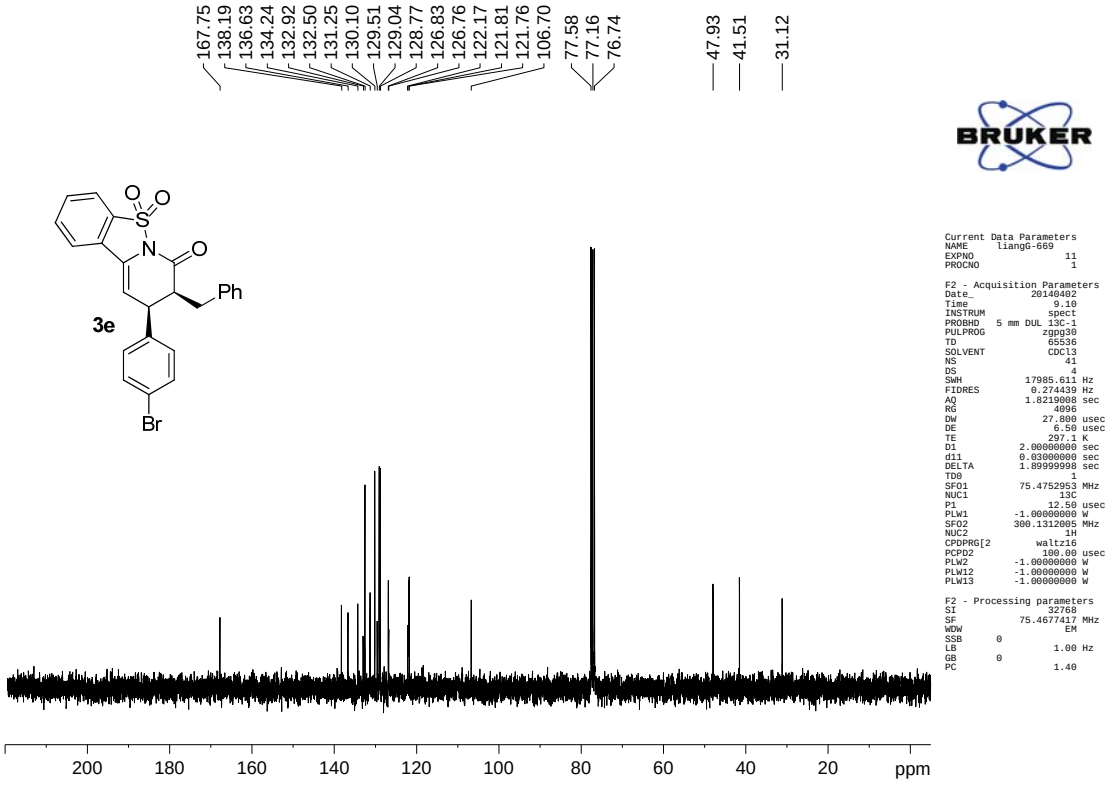


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PROCNO                 1

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DS                     4
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FIDRES          0.274439 Hz
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RG                 4096
DW                27.800 usec
DE                 6.50 usec
TE                297.1 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1
SFO1          75.4752953 MHz
NUC1                  13C
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SFO2         300.1312005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2             100.00 usec
PLW2         -1.00000000 W
PLW12        -1.00000000 W
PLW13        -1.00000000 W

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PC                 1.40
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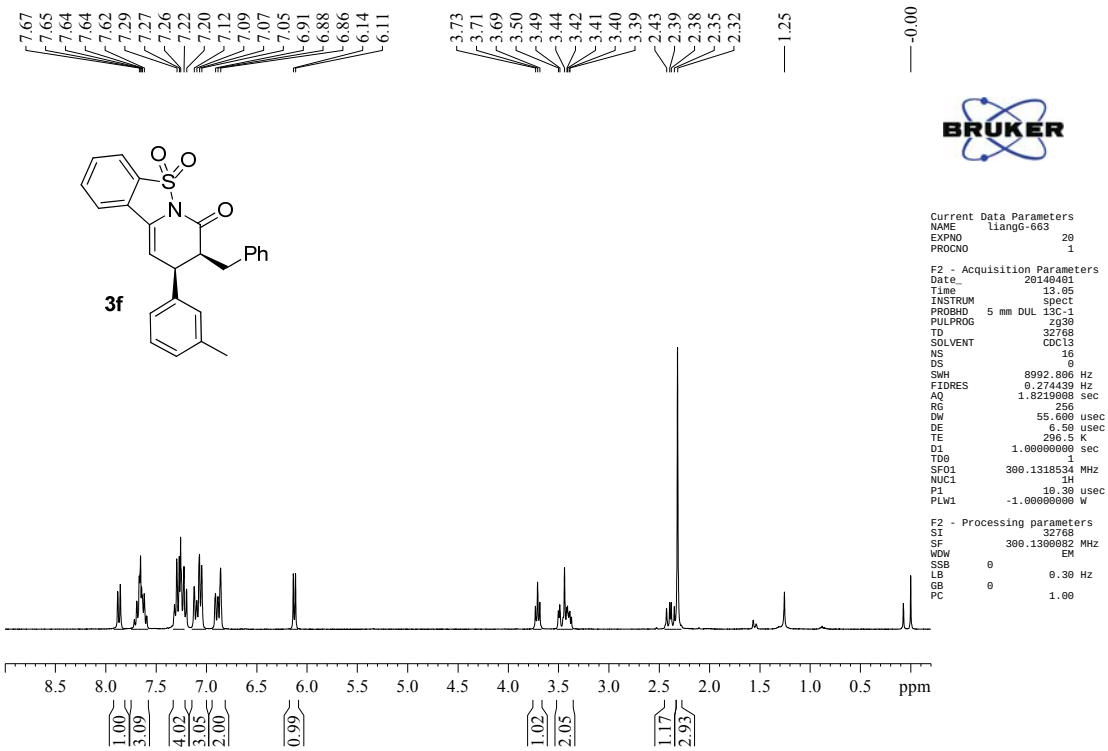
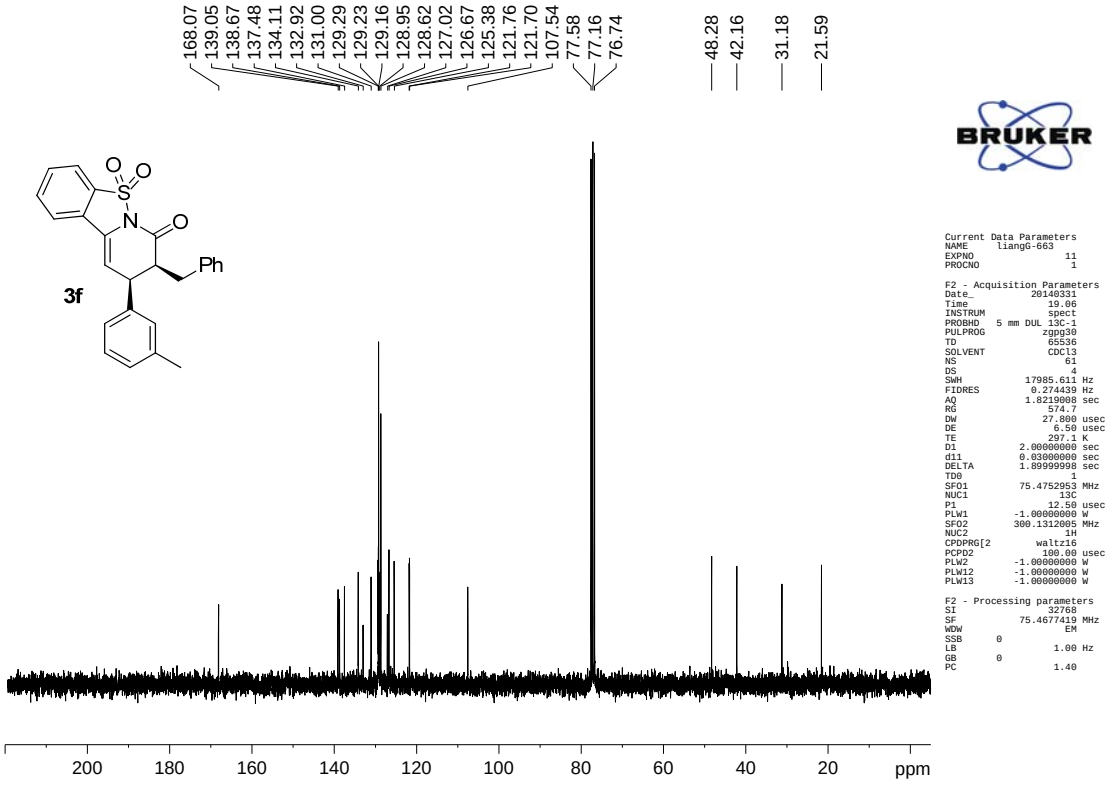
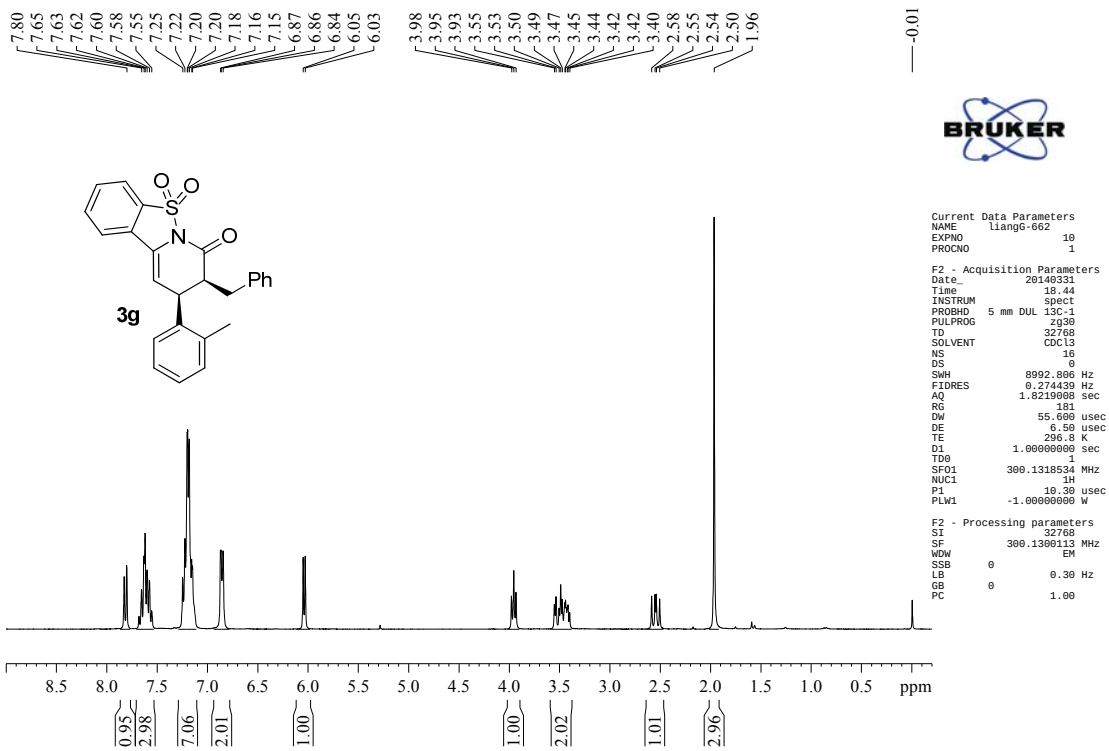
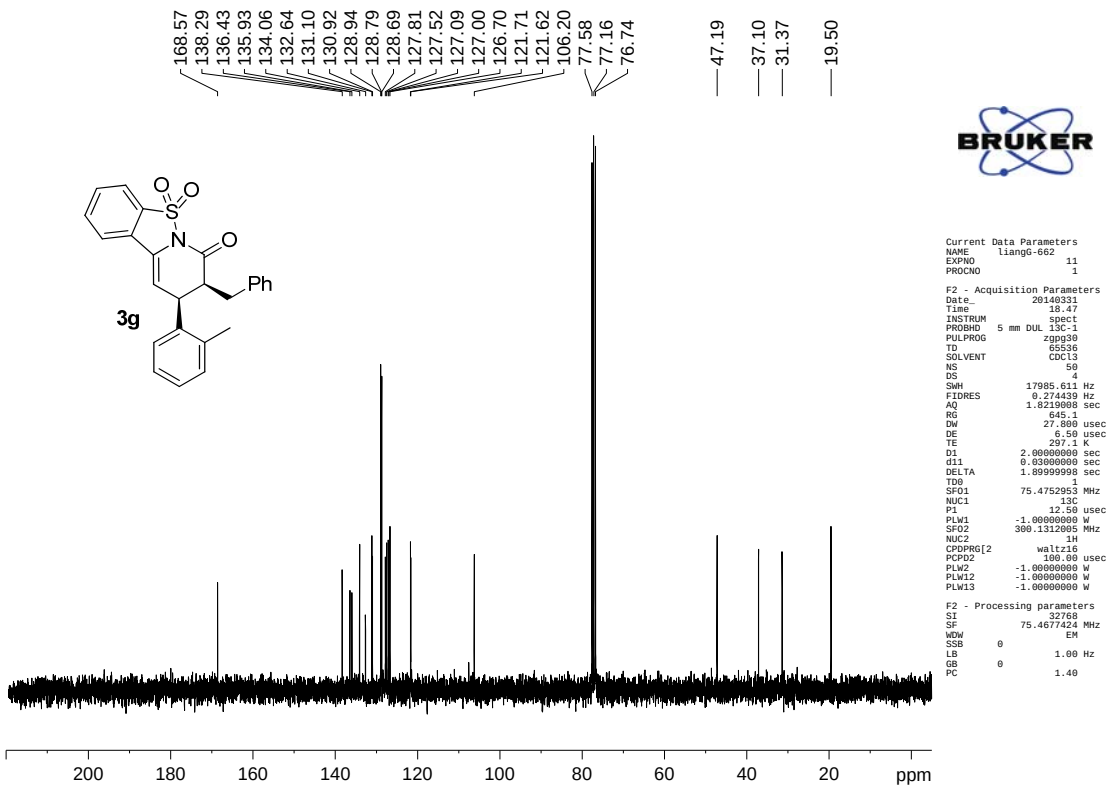
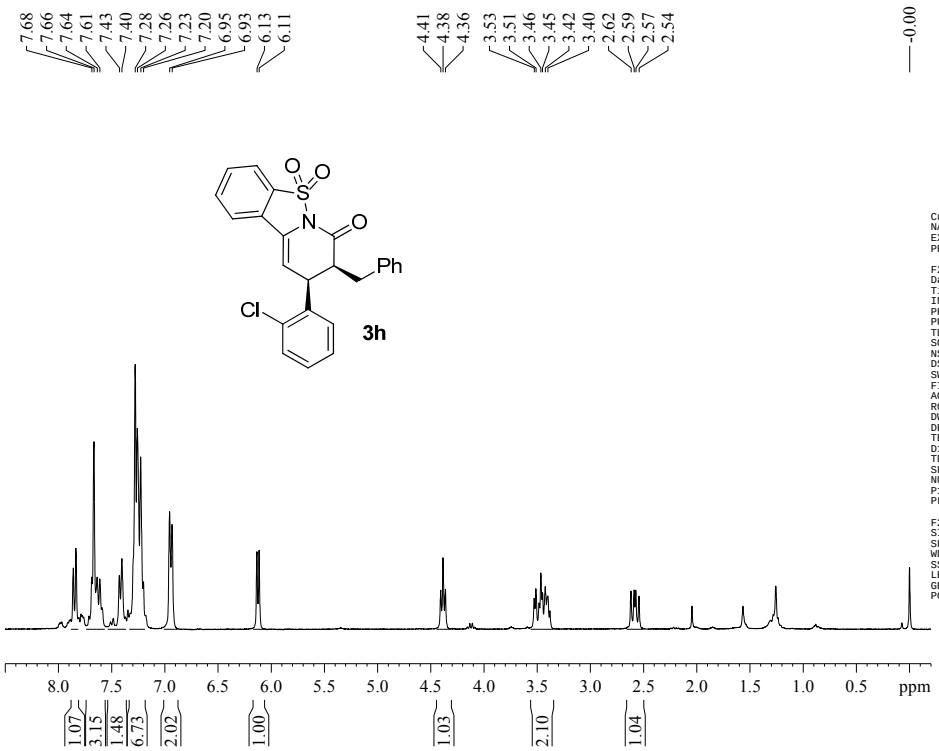

7.67 7.65 7.64 7.64 7.62 7.29 7.27 7.26 7.22 7.20 7.12 7.09 7.07 7.05 6.91 6.88 6.86 6.14 6.11
3.73 3.71 3.69 3.50 3.49 3.44 3.42 3.41 3.40 3.39 2.43 2.39 2.38 2.35 2.32
1.25
-0.00
3f
Ph
BRUKER
Current Data Parameters
NAME liangG-663
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140401
Time 13.05
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8992.806 Hz
FIDRES 0.274439 Hz
AQ 1.8219008 sec
RG 256
DW 55.600 usec
DE 6.50 usec
TE 296.5 K
D1 1.00000000 sec
TD0 1
SFO1 300.1318534 MHz
NUC1 1H
P1 10.30 usec
PLW1 -1.00000000 W
F2 - Processing parameters
SI 32768
SF 300.1300082 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00 3.09 4.02 3.05 2.00 0.99 1.02 2.05 1.17 2.93


168.07 139.05 138.67 137.48 134.11 132.92 131.00 129.29 129.23 129.16 128.95 128.62 127.02 126.67 125.38 121.76 121.70 107.54 77.58 77.16 76.74
48.28 42.16 31.18 21.59
3f
Ph
BRUKER
Current Data Parameters
NAME liangG-663
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140331
Time 19.06
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 61
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219008 sec
RG 574.7
DW 27.800 usec
DE 6.50 usec
TE 297.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 75.4752953 MHz
NUC1 13C
P1 12.50 usec
PLW1 -1.00000000 W
SFO2 300.1312005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 100.00 usec
PLW2 -1.00000000 W
PLW12 -1.00000000 W
PLW13 -1.00000000 W
F2 - Processing parameters
SI 32768
SF 75.4677419 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm


7.80 7.65 7.63 7.62 7.60 7.58 7.55 7.25 7.22 7.20 7.20 7.18 7.16 7.15 6.87 6.86 6.84 6.05 6.03
3.98 3.95 3.93 3.55 3.53 3.50 3.49 3.47 3.45 3.44 3.42 3.42 3.40 2.58 2.55 2.54 2.50 1.96
-0.01
3g
Ph
Current Data Parameters
NAME liangG-662
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140331
Time 18.44
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8992.806 Hz
FIDRES 0.274439 Hz
AQ 1.8219008 sec
RG 181
DW 55.600 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
TD0 1
SFO1 300.1318534 MHz
NUC1 1H
P1 10.30 usec
PLW1 -1.00000000 W
F2 - Processing parameters
SI 32768
SF 300.1300113 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.95 2.98 7.06 2.01 1.00 1.00 2.02 1.01 2.96

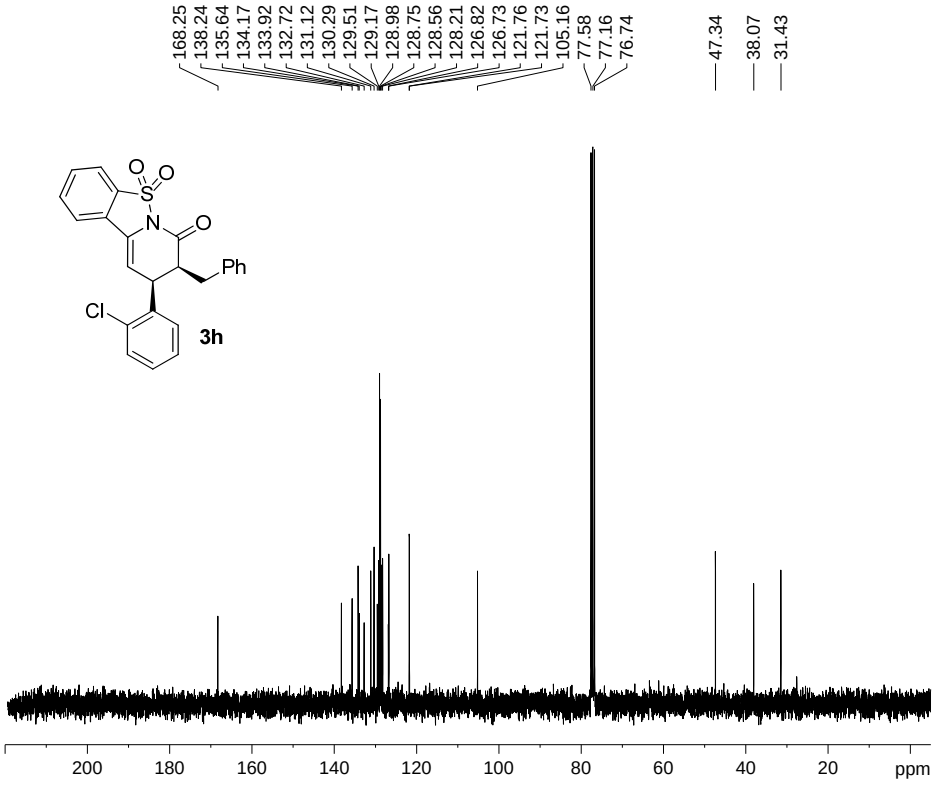

168.57 138.29 136.43 135.93 134.06 132.64 131.10 130.92 128.94 128.79 128.69 127.81 127.52 127.09 127.00 126.70 121.71 121.62 106.20 77.58 77.16 76.74
47.19 37.10 31.37 19.50
3g
Ph
Current Data Parameters
NAME liangG-662
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140331
Time 18.47
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 50
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219008 sec
RG 645.1
DW 27.800 usec
DE 6.50 usec
TE 297.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 75.4752953 MHz
NUC1 13C
P1 12.50 usec
PLW1 -1.00000000 W
SFO2 300.1312005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 100.00 usec
PLW2 -1.00000000 W
PLW12 -1.00000000 W
PLW13 -1.00000000 W
F2 - Processing parameters
SI 32768
SF 75.4677424 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm




```
Current Data Parameters
NAME      liangG-666
EXPNO              20
PROCNO              1

F2 - Acquisition Parameters
Date_        20140403
Time            21.07
INSTRUM         spect
PROBHD   5 mm DUL 13C-1
PULPROG         zg30
TD             32768
SOLVENT        CDCl3
NS                16
DS                 0
SWH          8992.806 Hz
FIDRES       0.274439 Hz
AQ          1.8219008 sec
RG              456.1
DW             55.600 usec
DE              6.50 usec
TE             296.6 K
D1         1.00000000 sec
TD0                1
SFO1       300.1318534 MHz
NUC1              1H
P1             10.30 usec
PLW1      -1.00000000 W

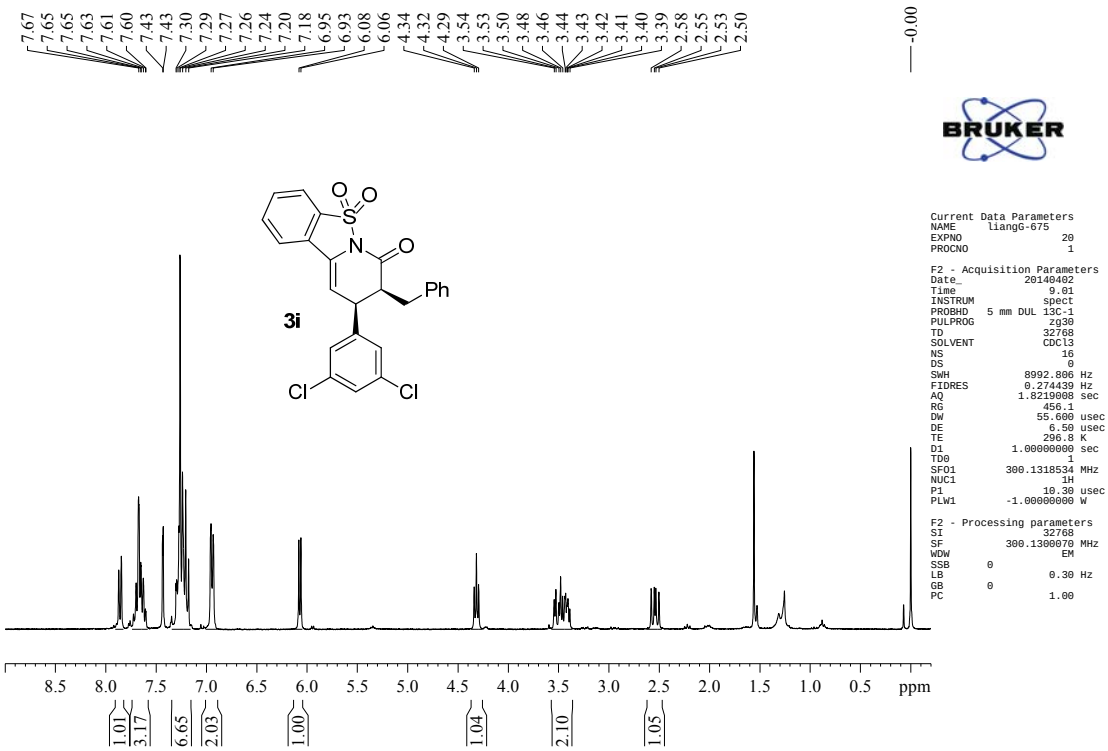
F2 - Processing parameters
SI             32768
SF       300.1300078 MHz
WDW              EM
SSB       0
LB            0.30 Hz
GB        0
PC            1.00
```

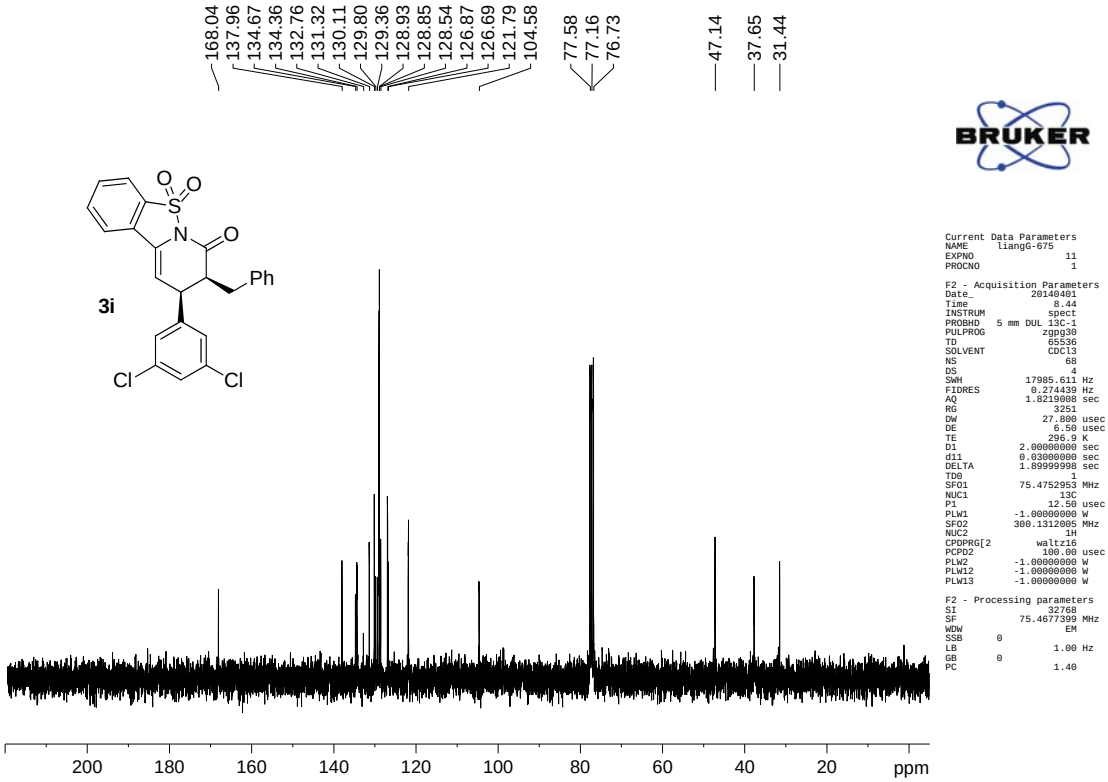


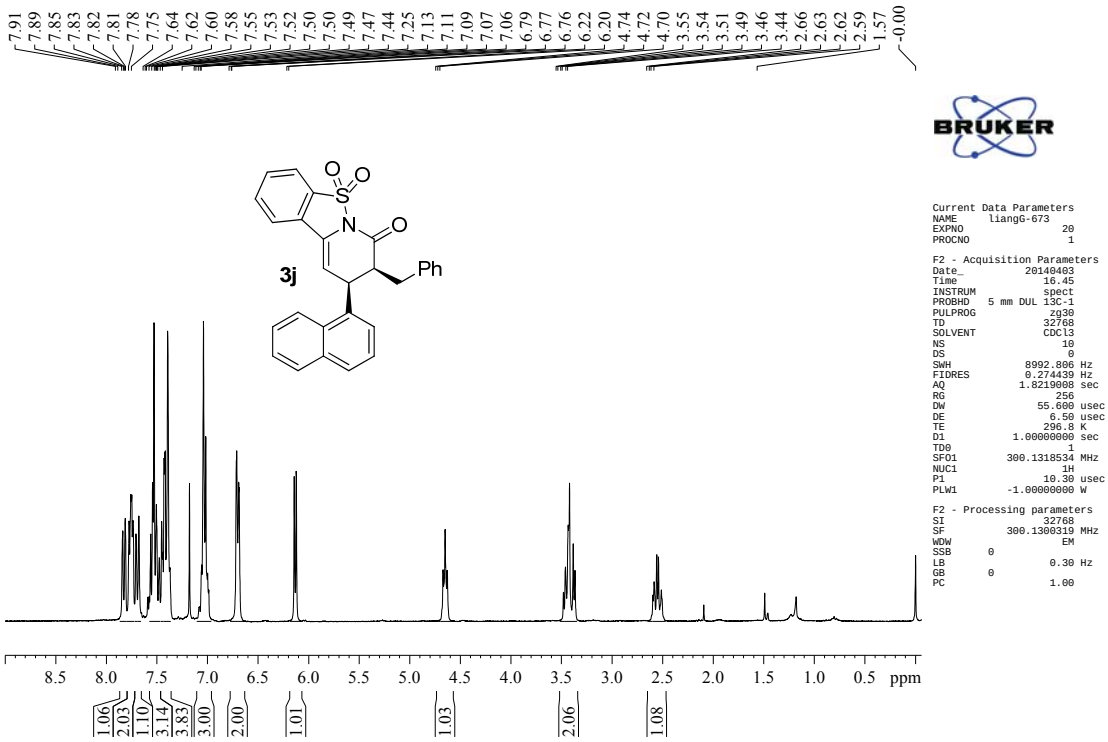

```
Current Data Parameters
NAME      liangG-666
EXPNO              11
PROCNO              1

F2 - Acquisition Parameters
Date_        20140331
Time            19.15
INSTRUM         spect
PROBHD   5 mm DUL 13C-1
PULPROG       zgpg30
TD             65536
SOLVENT        CDCl3
NS                55
DS                 4
SWH         17985.611 Hz
FIDRES       0.274439 Hz
AQ          1.8219008 sec
RG              2896.3
DW             27.800 usec
DE              6.50 usec
TE             297.1 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA      1.89999998 sec
TD0                1
SFO1       75.4752953 MHz
NUC1              13C
P1             12.50 usec
PLW1      -1.00000000 W
SFO2       300.1312005 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2         100.00 usec
PLW2      -1.00000000 W
PLW12     -1.00000000 W
PLW13     -1.00000000 W

F2 - Processing parameters
SI             32768
SF       75.4677421 MHz
WDW              EM
SSB       0
LB            1.00 Hz
GB        0
PC            1.40
```


7.67 7.65 7.65 7.63 7.61 7.60 7.43 7.43 7.30 7.29 7.27 7.26 7.24 7.20 7.18 6.95 6.93 6.08 6.06 4.34 4.32 4.29 3.54 3.53 3.50 3.48 3.46 3.44 3.43 3.42 3.41 3.40 3.39 2.58 2.55 2.53 2.50
-0.00
BRUKER
3i
Ph
Cl
Cl
Current Data Parameters
NAME liangG-675
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140402
Time 9.01
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8992.806 Hz
FIDRES 0.274439 Hz
AQ 1.8219008 sec
RG 456.1
DW 55.600 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
TD0 1
SFO1 300.1318534 MHz
NUC1 1H
P1 10.30 usec
PLW1 -1.00000000 W
F2 - Processing parameters
SI 32768
SF 300.1300070 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.01 3.17 6.65 2.03 1.00 1.04 2.10 1.05

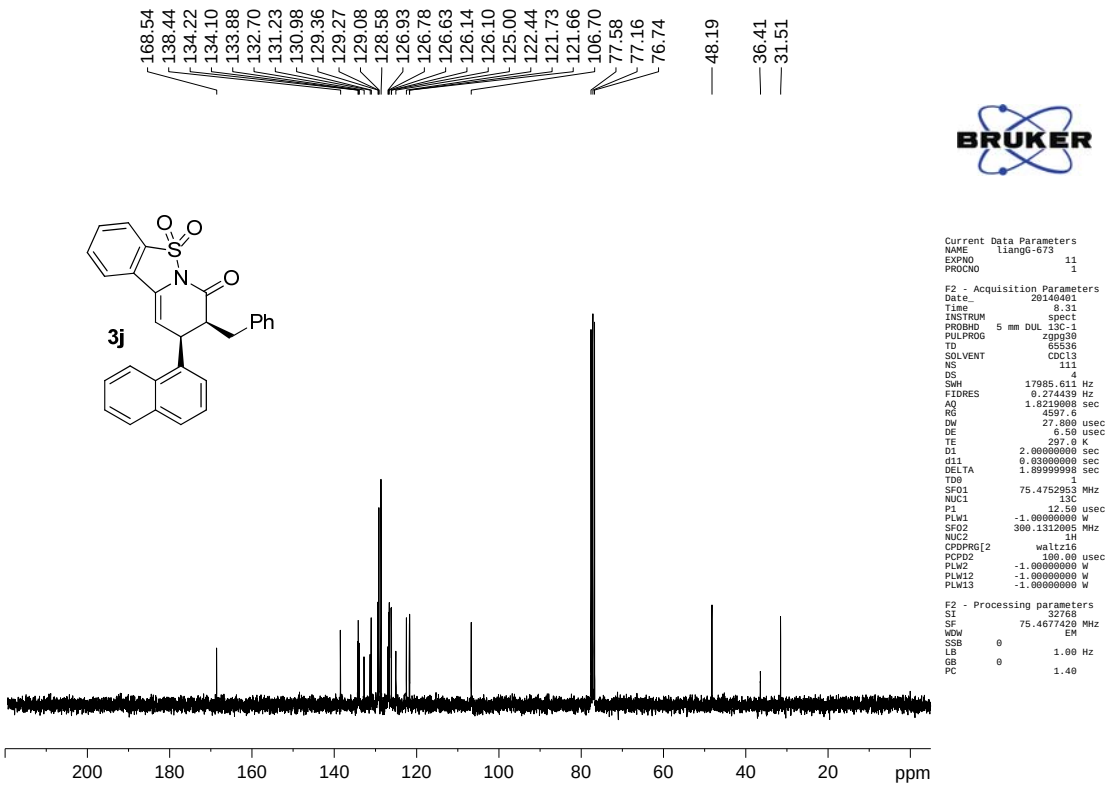

168.04 137.96 134.67 134.36 132.76 131.32 130.11 129.80 129.36 128.93 128.85 128.54 126.87 126.69 121.79 104.58 77.58 77.16 76.73 47.14 37.65 31.44
BRUKER
3i
Ph
Cl
Cl
Current Data Parameters
NAME liangG-675
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140401
Time 8.44
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 68
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219008 sec
RG 3251
DW 27.800 usec
DE 6.50 usec
TE 296.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 75.4752953 MHz
NUC1 13C
P1 12.50 usec
PLW1 -1.00000000 W
SFO2 300.1312005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 100.00 usec
PLW2 -1.00000000 W
PLW12 -1.00000000 W
PLW13 -1.00000000 W
F2 - Processing parameters
SI 32768
SF 75.4677399 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm




```
Current Data Parameters
NAME      liangG-673
EXPNO                 20
PROCNO                 1

F2 - Acquisition Parameters
Date_            20140403
Time                16.45
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    10
DS                     0
SWH             8992.806 Hz
FIDRES          0.274439 Hz
AQ             1.8219008 sec
RG                   256
DW                55.600 usec
DE                 6.50 usec
TE                296.8 K
D1            1.00000000 sec
TD0                    1
SFO1         300.1318534 MHz
NUC1                  1H
P1                 10.30 usec
PLW1         -1.00000000 W

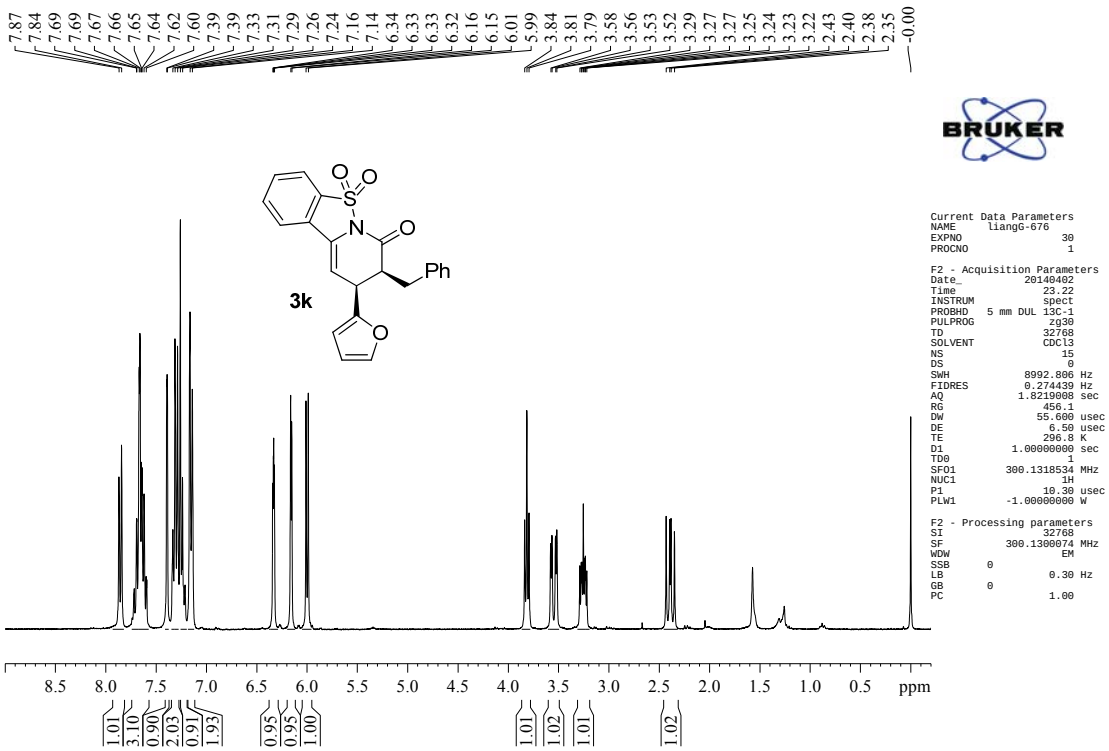
F2 - Processing parameters
SI                 32768
SF          300.1300319 MHz
WDW                   EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00
```

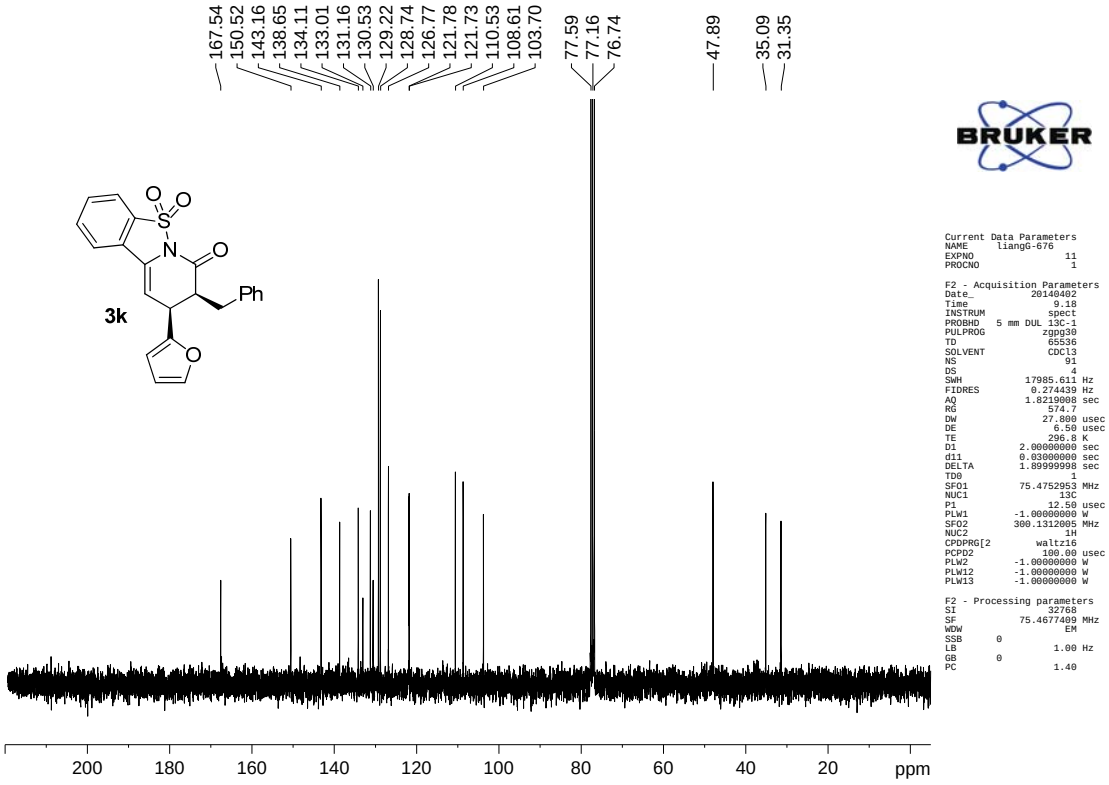


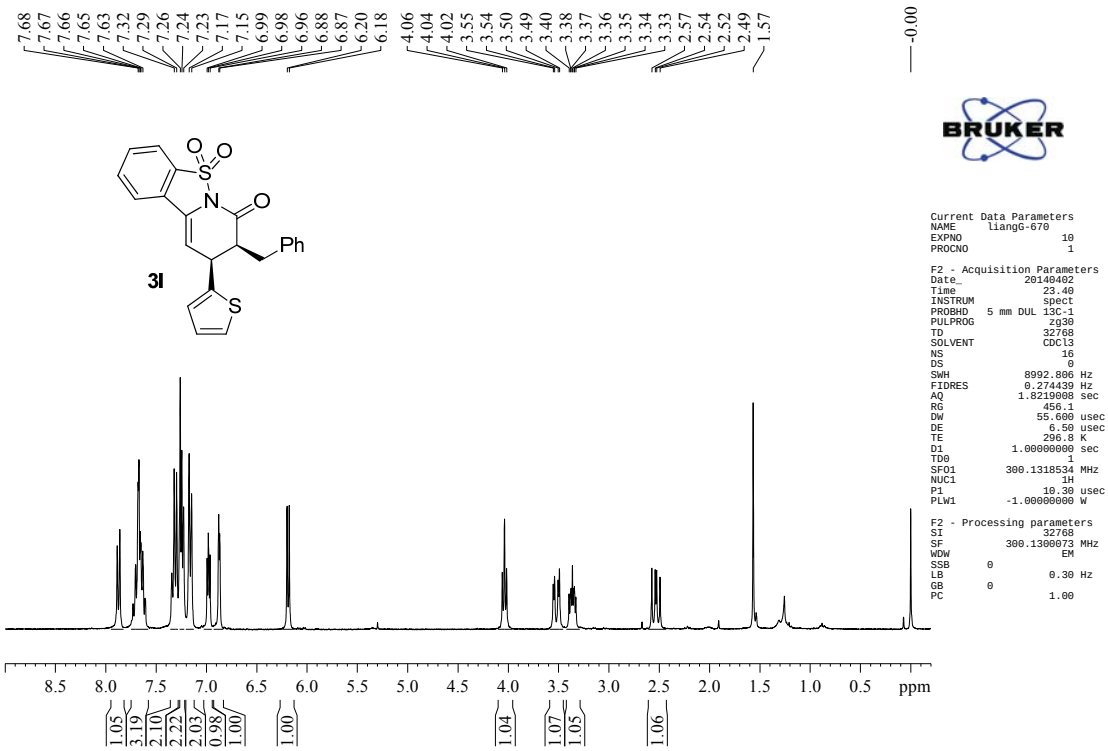

```
Current Data Parameters
NAME      liangG-673
EXPNO                 11
PROCNO                 1

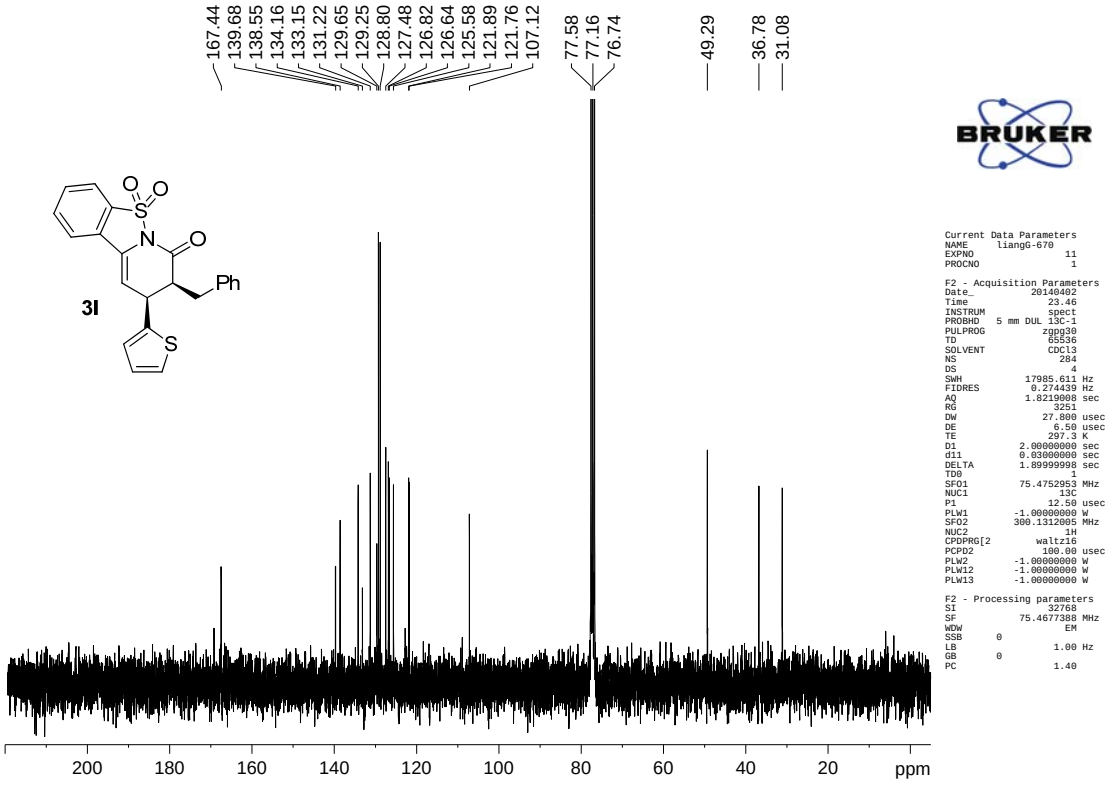
F2 - Acquisition Parameters
Date_            20140401
Time                 8.31
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   111
DS                     4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ             1.8219008 sec
RG                  4597.6
DW                 27.800 usec
DE                  6.50 usec
TE                 297.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1
SFO1          75.4752953 MHz
NUC1                  13C
P1                 12.50 usec
PLW1         -1.00000000 W
SFO2         300.1312005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2             100.00 usec
PLW2         -1.00000000 W
PLW12        -1.00000000 W
PLW13        -1.00000000 W

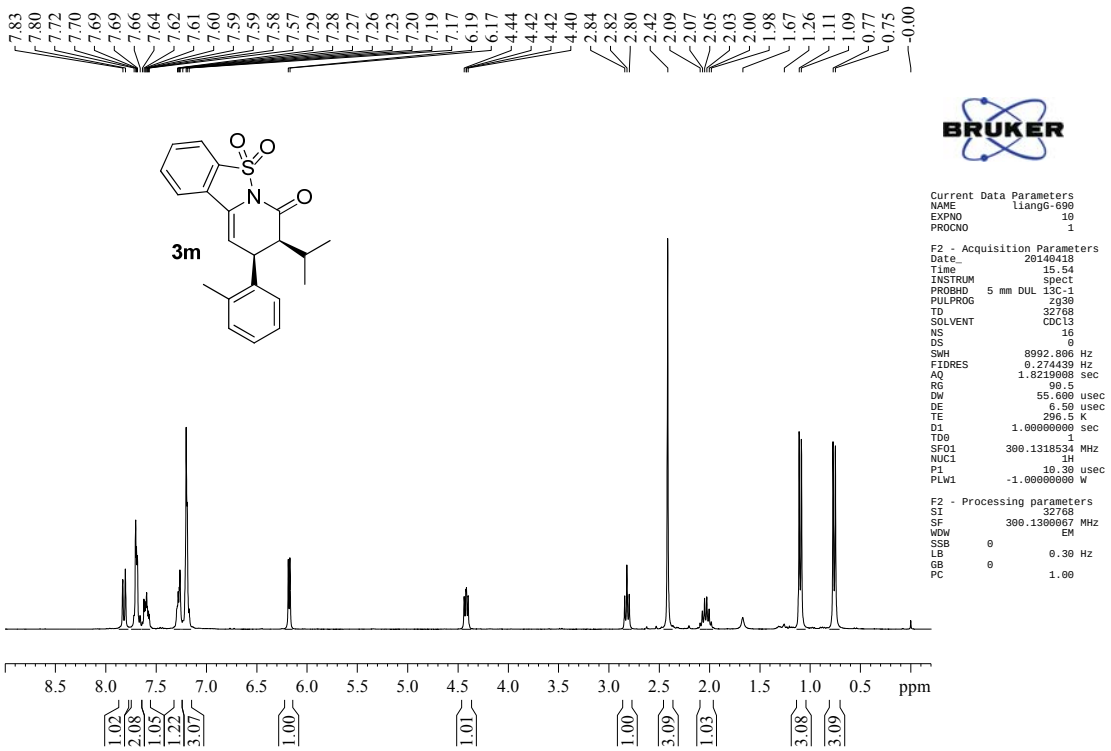
F2 - Processing parameters
SI                 32768
SF          75.4677420 MHz
WDW                   EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40
```


7.87 7.84 7.69 7.69 7.67 7.66 7.65 7.64 7.62 7.60 7.39 7.39 7.33 7.31 7.29 7.26 7.24 7.16 7.14 6.34 6.33 6.33 6.32 6.16 6.15 6.01 5.99 3.84 3.81 3.79 3.58 3.56 3.53 3.52 3.29 3.27 3.27 3.25 3.24 3.23 3.22 2.43 2.40 2.38 2.35 -0.00
BRUKER
3k
Ph
Current Data Parameters
NAME liangG-676
EXPNO 30
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140402
Time 23.22
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 15
DS 0
SWH 8992.806 Hz
FIDRES 0.274439 Hz
AQ 1.8219008 sec
RG 456.1
DW 55.600 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
TD0 1
SFO1 300.1318534 MHz
NUC1 1H
P1 10.30 usec
PLW1 -1.00000000 W
F2 - Processing parameters
SI 32768
SF 300.1300074 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.01 3.10 0.90 2.03 0.91 1.93 0.95 0.95 1.00 1.01 1.02 1.01 1.02


167.54 150.52 143.16 138.65 134.11 133.01 131.16 130.53 129.22 128.74 126.77 121.78 121.73 110.53 108.61 103.70 77.59 77.16 76.74 47.89 35.09 31.35
BRUKER
3k
Ph
Current Data Parameters
NAME liangG-676
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140402
Time 9.18
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 91
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219008 sec
RG 574.7
DW 27.800 usec
DE 6.50 usec
TE 296.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 75.4752953 MHz
NUC1 13C
P1 12.50 usec
PLW1 -1.00000000 W
SFO2 300.1312005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 100.00 usec
PLW2 -1.00000000 W
PLW12 -1.00000000 W
PLW13 -1.00000000 W
F2 - Processing parameters
SI 32768
SF 75.4677409 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm


7.68 7.67 7.66 7.65 7.63 7.32 7.29 7.26 7.24 7.23 7.17 7.15 6.99 6.98 6.96 6.88 6.87 6.20 6.18 4.06 4.04 4.02 3.55 3.54 3.50 3.49 3.40 3.38 3.37 3.36 3.35 3.34 3.33 2.57 2.54 2.52 2.49 1.57
-0.00
3l
Ph
Current Data Parameters
NAME liangG-670
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140402
Time 23.40
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8992.806 Hz
FIDRES 0.274439 Hz
AQ 1.8219008 sec
RG 456.1
DW 55.600 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
TD0 1
SFO1 300.1318534 MHz
NUC1 1H
P1 10.30 usec
PLW1 -1.00000000 W
F2 - Processing parameters
SI 32768
SF 300.1300073 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.05 3.19 2.10 2.22 2.03 0.98 1.00 1.00 1.04 1.07 1.05 1.06


167.44 139.68 138.55 134.16 133.15 131.22 129.65 129.25 128.80 127.48 126.82 126.64 125.58 121.89 121.76 107.12
77.58 77.16 76.74
49.29
36.78
31.08
3l
Ph
Current Data Parameters
NAME liangG-670
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140402
Time 23.46
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 284
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219008 sec
RG 3251
DW 27.800 usec
DE 6.50 usec
TE 297.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 75.4752953 MHz
NUC1 13C
P1 12.50 usec
PLW1 -1.00000000 W
SFO2 300.1312005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 100.00 usec
PLW2 -1.00000000 W
PLW12 -1.00000000 W
PLW13 -1.00000000 W
F2 - Processing parameters
SI 32768
SF 75.4677388 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm




```
Current Data Parameters
NAME          liangG-690
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140418
Time               15.54
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    16
DS                     0
SWH             8992.806 Hz
FIDRES          0.274439 Hz
AQ             1.8219008 sec
RG                  90.5
DW                55.600 usec
DE                 6.50 usec
TE                296.5 K
D1             1.00000000 sec
TD0                    1
SFO1         300.1318534 MHz
NUC1                  1H
P1                 10.30 usec
PLW1         -1.00000000 W

F2 - Processing parameters
SI                 32768
SF          300.1300067 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
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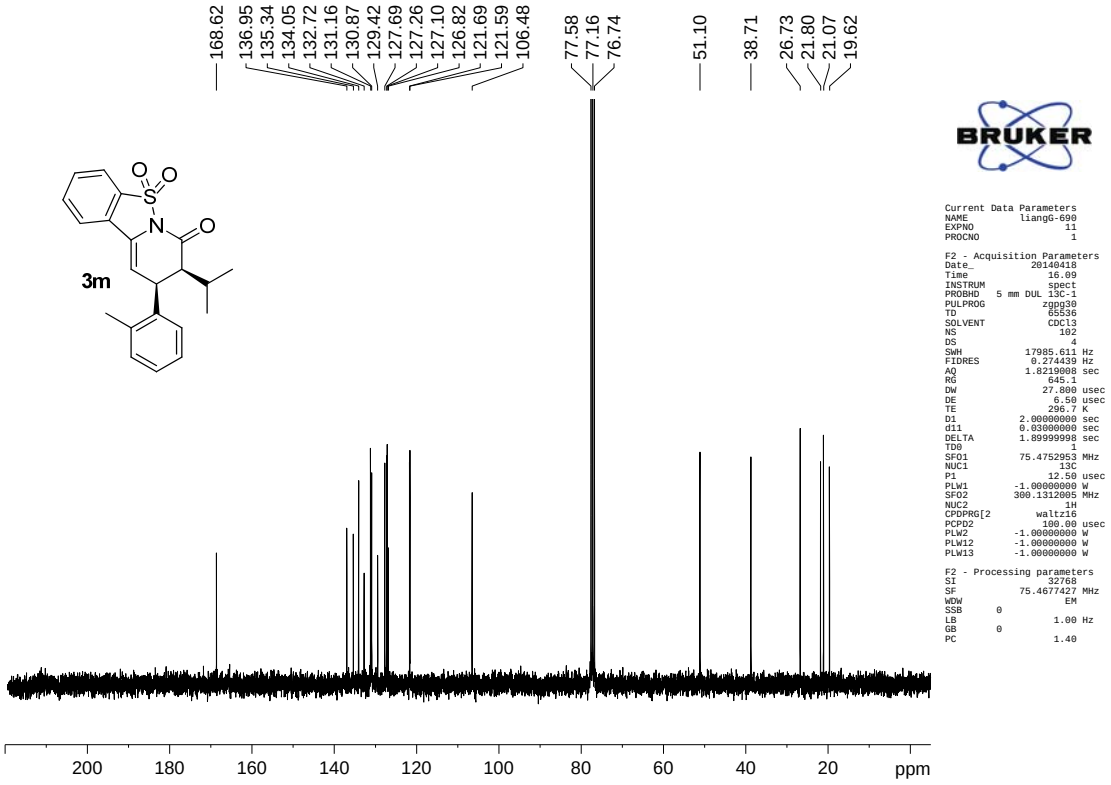


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Current Data Parameters
NAME          liangG-690
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140418
Time               16.09
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   102
DS                     4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ             1.8219008 sec
RG                 645.1
DW                27.800 usec
DE                 6.50 usec
TE                296.7 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1
SFO1          75.4752953 MHz
NUC1                  13C
P1                 12.50 usec
PLW1         -1.00000000 W
SFO2         300.1312005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2             100.00 usec
PLW2         -1.00000000 W
PLW12        -1.00000000 W
PLW13        -1.00000000 W

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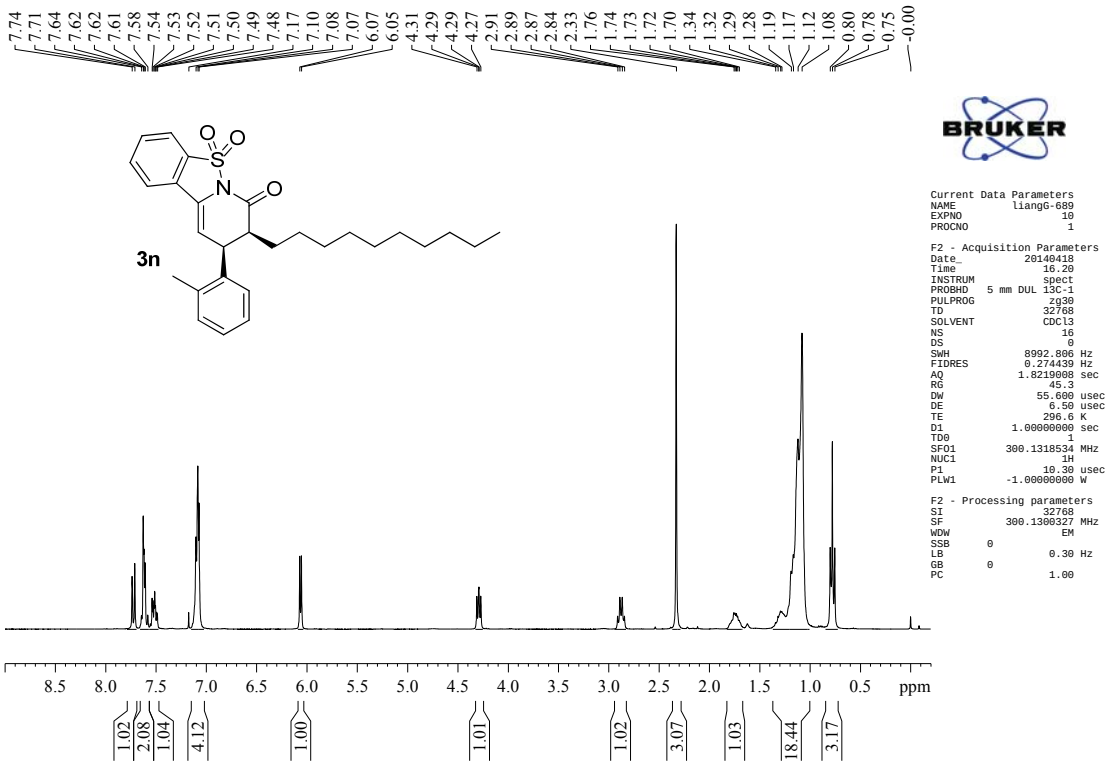
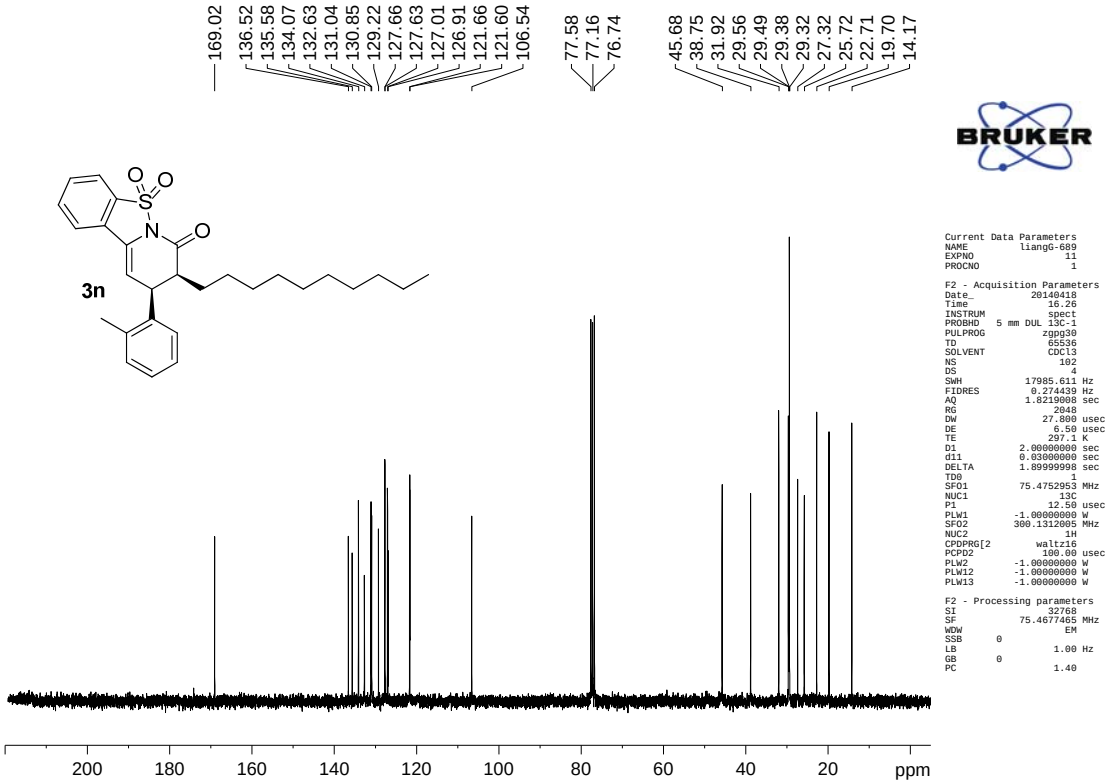
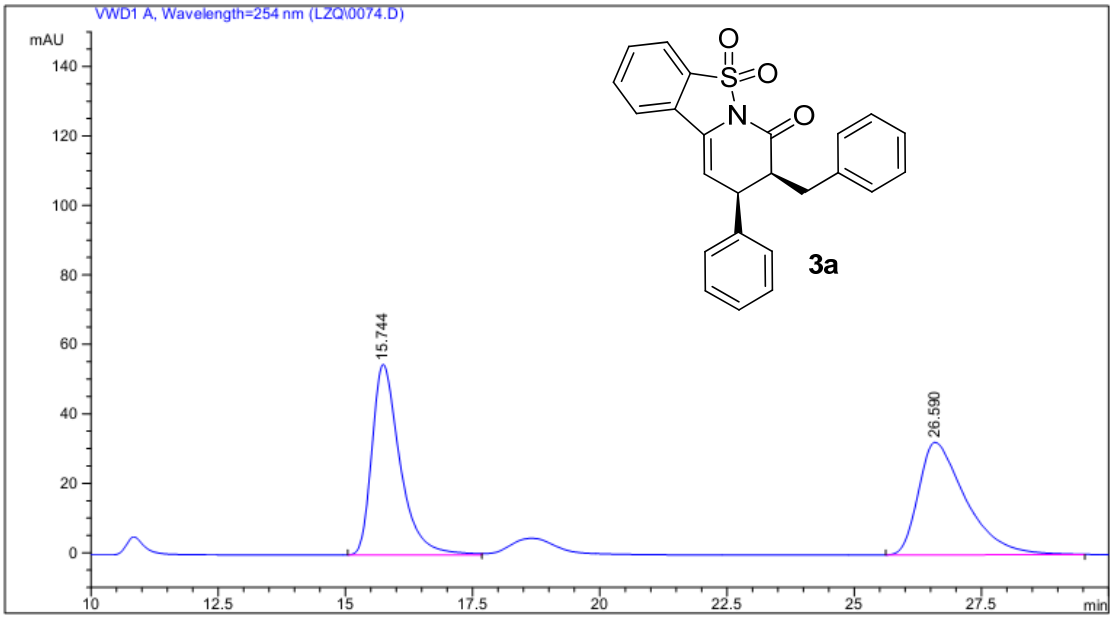

7.74 7.71 7.64 7.62 7.62 7.61 7.58 7.54 7.53 7.52 7.51 7.50 7.49 7.48 7.17 7.10 7.08 7.07 6.07 6.05 4.31 4.29 4.29 4.27 2.91 2.89 2.87 2.84 2.33 1.76 1.74 1.73 1.72 1.70 1.34 1.32 1.29 1.28 1.19 1.17 1.12 1.08 0.80 0.78 0.75 -0.00
3n
BRUKER
Current Data Parameters
NAME liangG-689
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140418
Time 16.20
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8992.806 Hz
FIDRES 0.274439 Hz
AQ 1.8219008 sec
RG 45.3
DW 55.600 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
SFO1 300.1318534 MHz
NUC1 1H
P1 10.30 usec
PLW1 -1.00000000 W
F2 - Processing parameters
SI 32768
SF 300.1300327 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.02 2.08 1.04 4.12 1.00 1.01 1.02 3.07 1.03 18.44 3.17

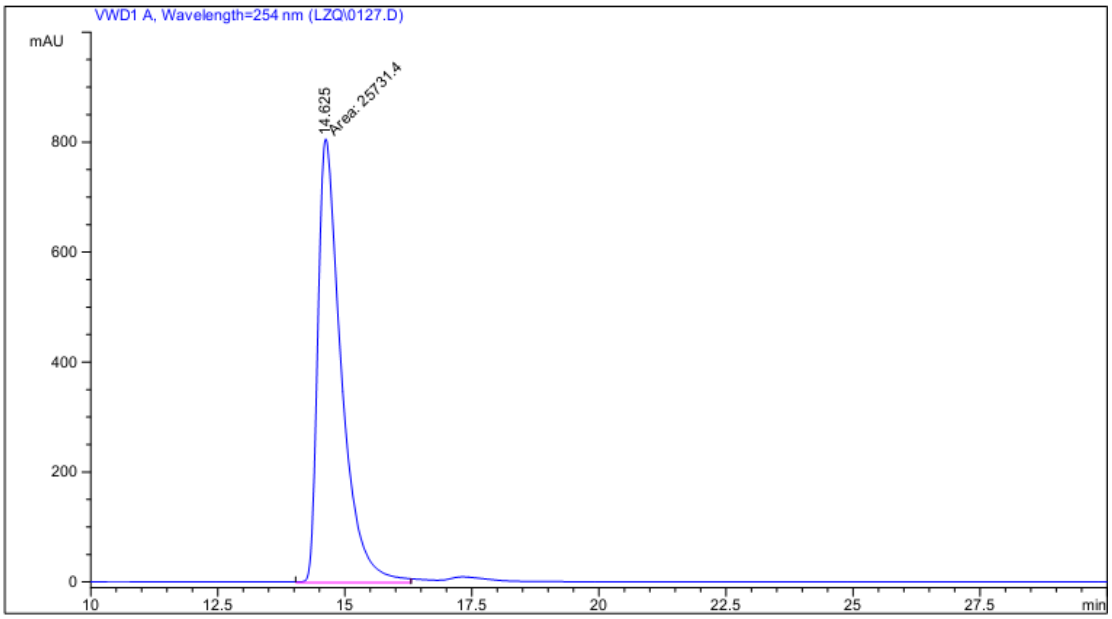

169.02 136.52 135.58 134.07 132.63 131.04 130.85 129.22 127.66 127.63 127.01 126.91 121.66 121.60 106.54 77.58 77.16 76.74 45.68 38.75 31.92 29.56 29.49 29.38 29.32 27.32 25.72 22.71 19.70 14.17
3n
BRUKER
Current Data Parameters
NAME liangG-689
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140418
Time 16.26
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 102
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219008 sec
RG 2048
DW 27.800 usec
DE 6.50 usec
TE 297.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 75.4752953 MHz
NUC1 13C
P1 12.50 usec
PLW1 -1.00000000 W
SFO2 300.1312005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 100.00 usec
PLW2 -1.00000000 W
PLW12 -1.00000000 W
PLW13 -1.00000000 W
F2 - Processing parameters
SI 32768
SF 75.4677465 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm

Part III HPLC Chromatography



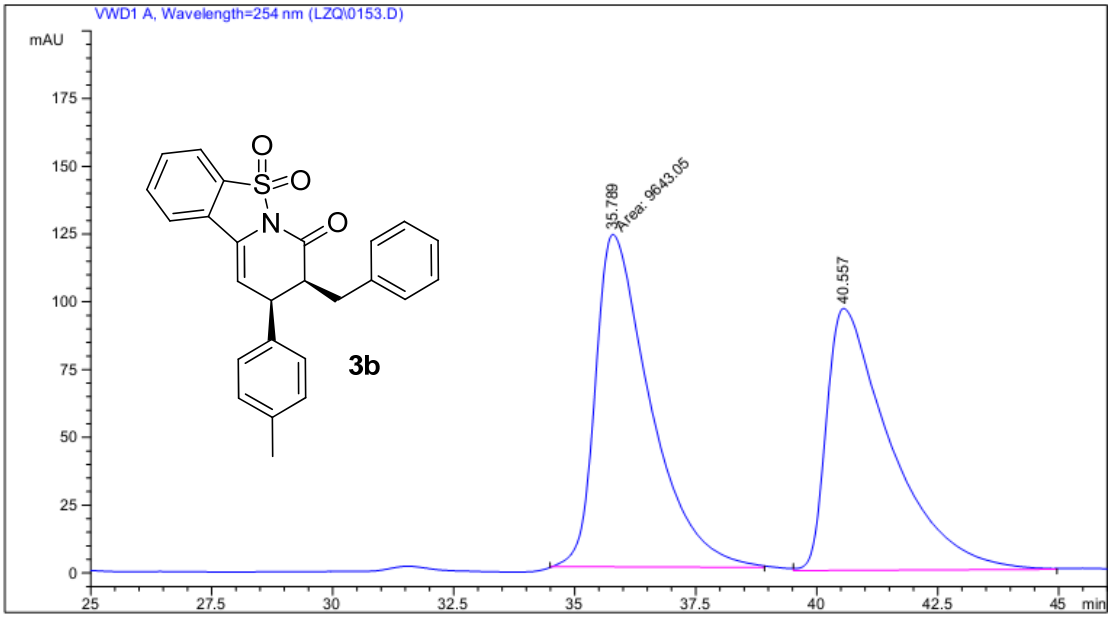
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	15.744	BV	0.5792	2107.87598	54.87305	50.1699
2	26.590	BB	0.9542	2093.60303	32.42423	49.8301
Totals :				4201.47900	87.29728	



Signal 1: VWD1 A, Wavelength=254 nm

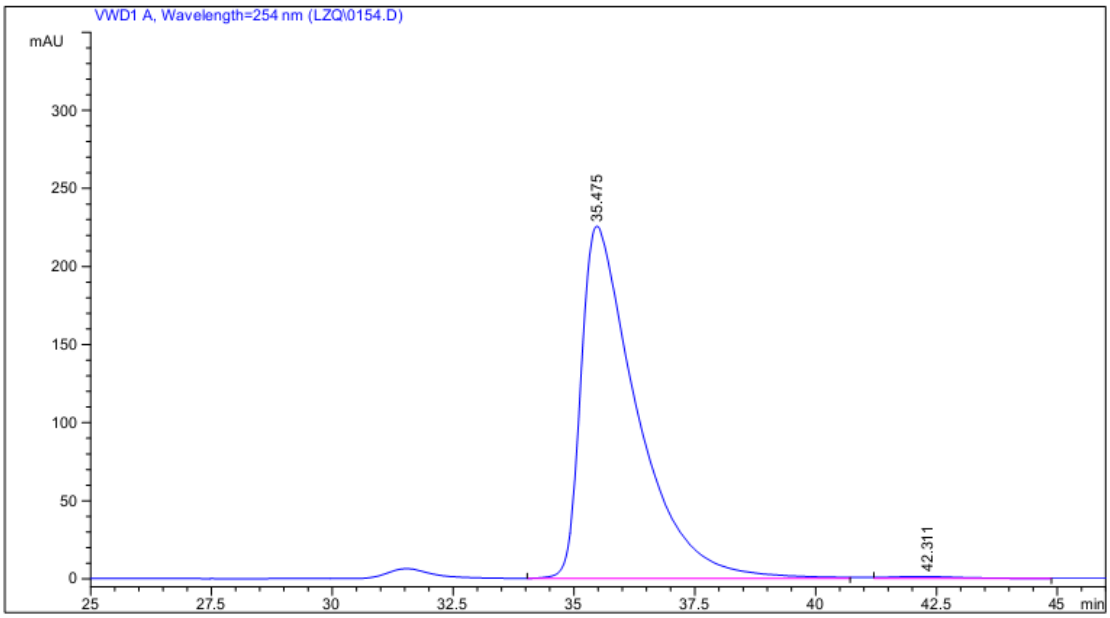
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.625	MM	0.5317	2.57314e4	806.55774	100.0000
Totals :				2.57314e4	806.55774	



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	35.789	MM	1.3107	9643.04980	122.61942	52.0715
2	40.557	VB	1.3219	8875.82129	96.64394	47.9285

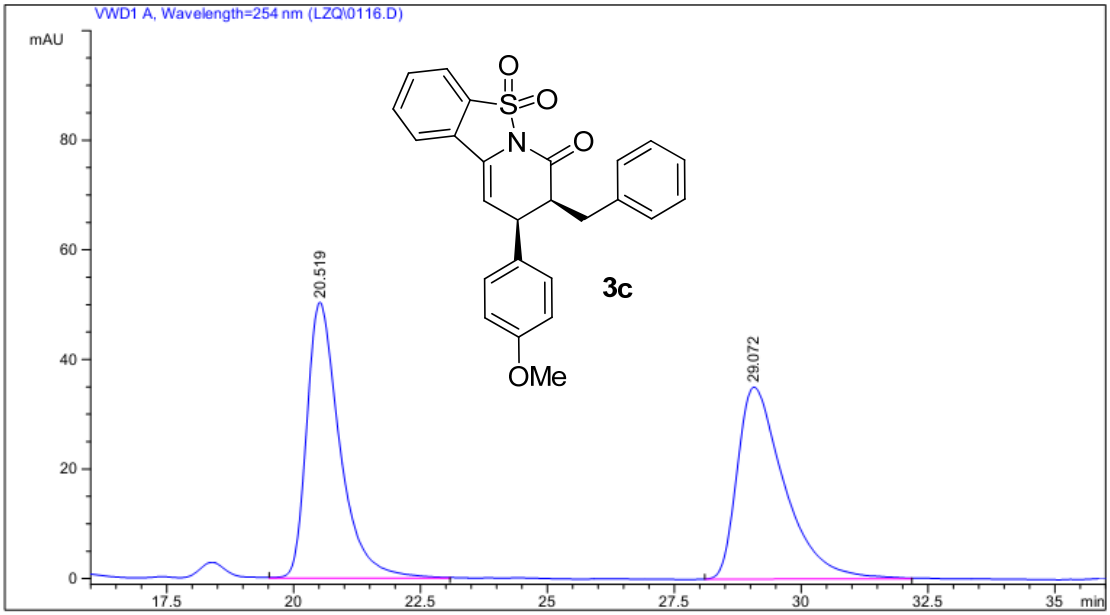
Totals : 1.85189e4 219.26336



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	35.475	BB	1.1345	1.81742e4	225.71002	99.2738
2	42.311	BB	1.2264	132.94359	1.32448	0.7262

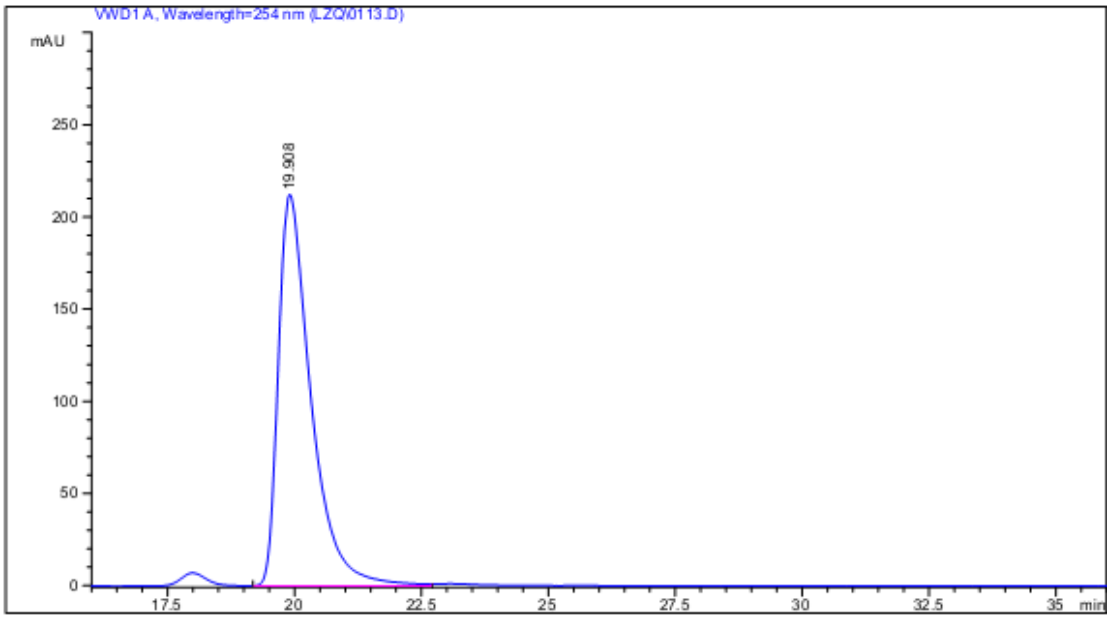
Totals : 1.83071e4 227.03450



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	20.519	VB	0.6845	2275.16724	50.34241	50.1402
2	29.072	BB	0.9518	2262.44458	35.01641	49.8598

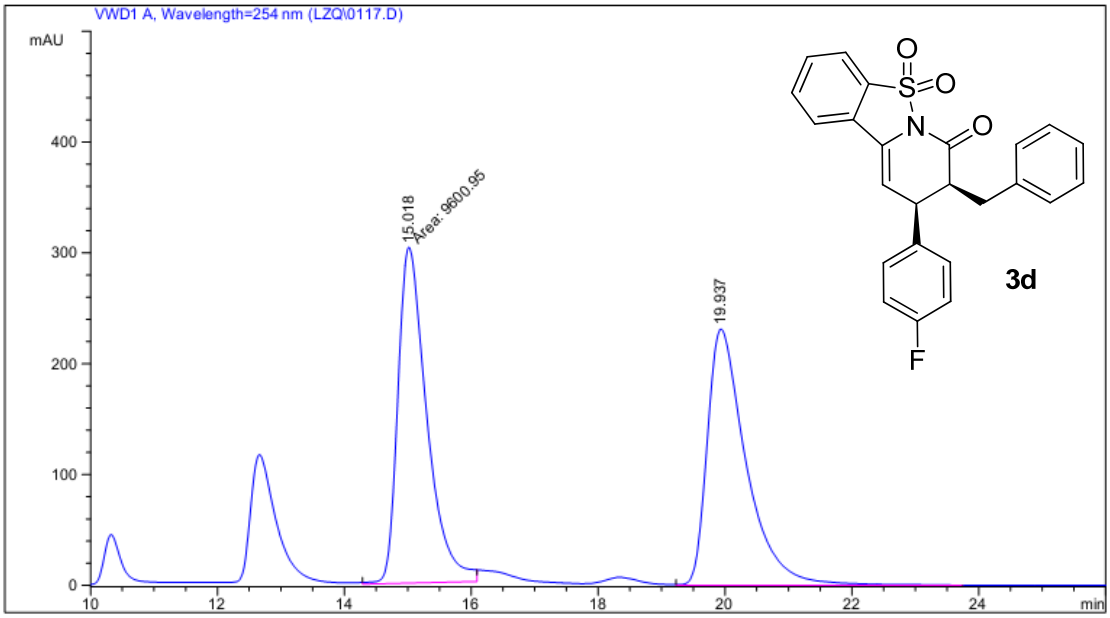
Totals : 4537.61182 85.35882



Signal 1: VWD1 A, Wavelength=254 nm

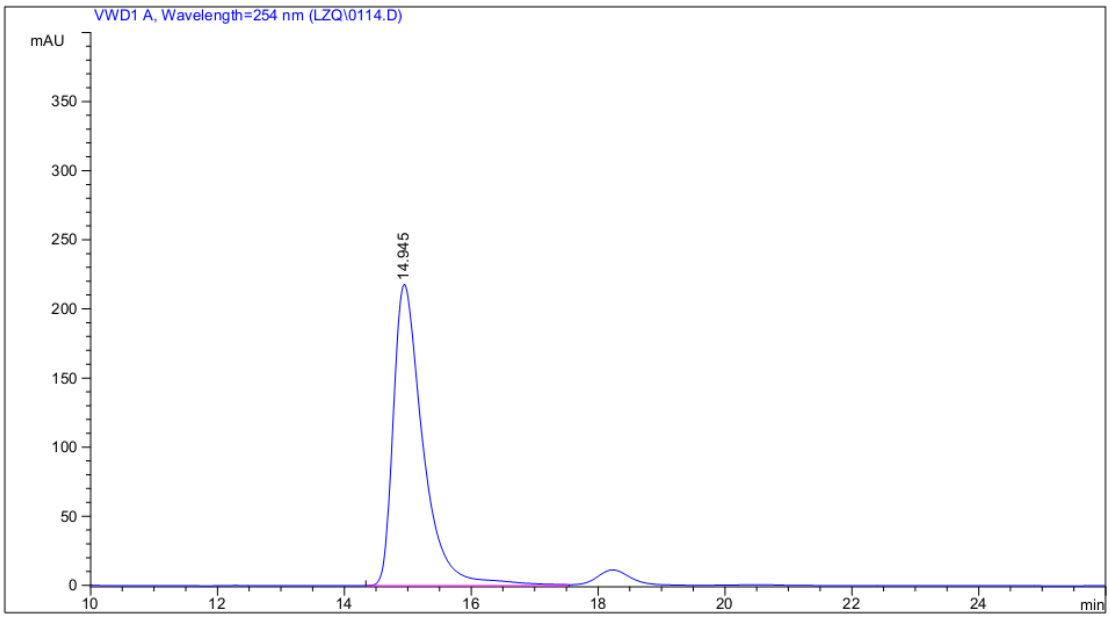
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	19.908	BB	0.6673	9498.10254	212.41368	100.0000

Totals : 9498.10254 212.41368



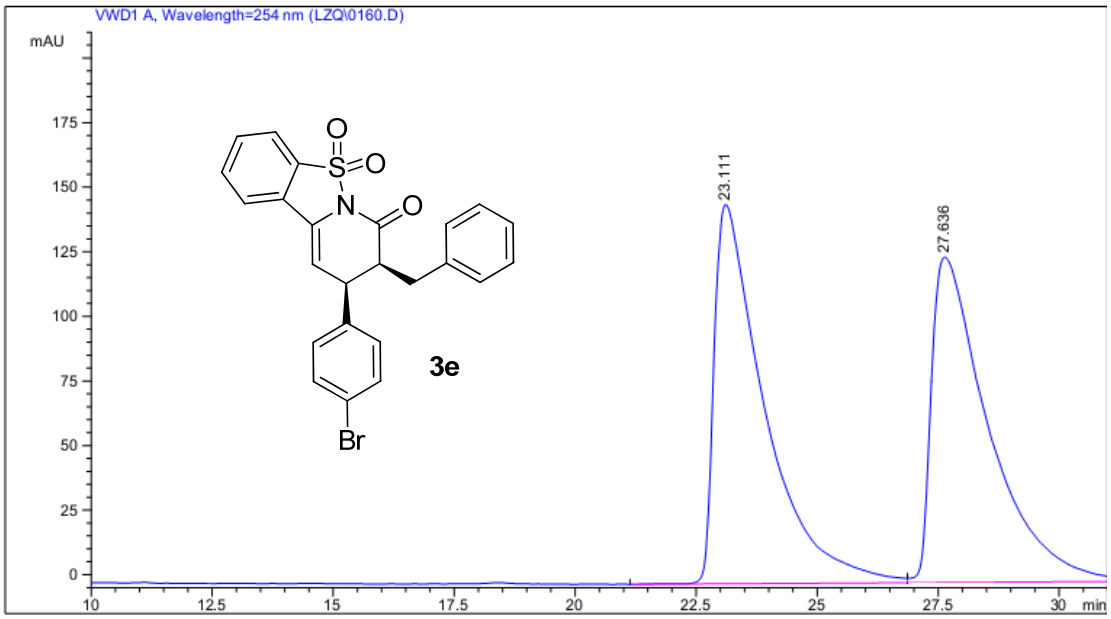
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	15.018	MM	0.5280	9600.95020	303.06305	49.3993
2	19.937	VB	0.6364	9834.46582	231.02711	50.6007
Totals :				1.94354e4	534.09016	



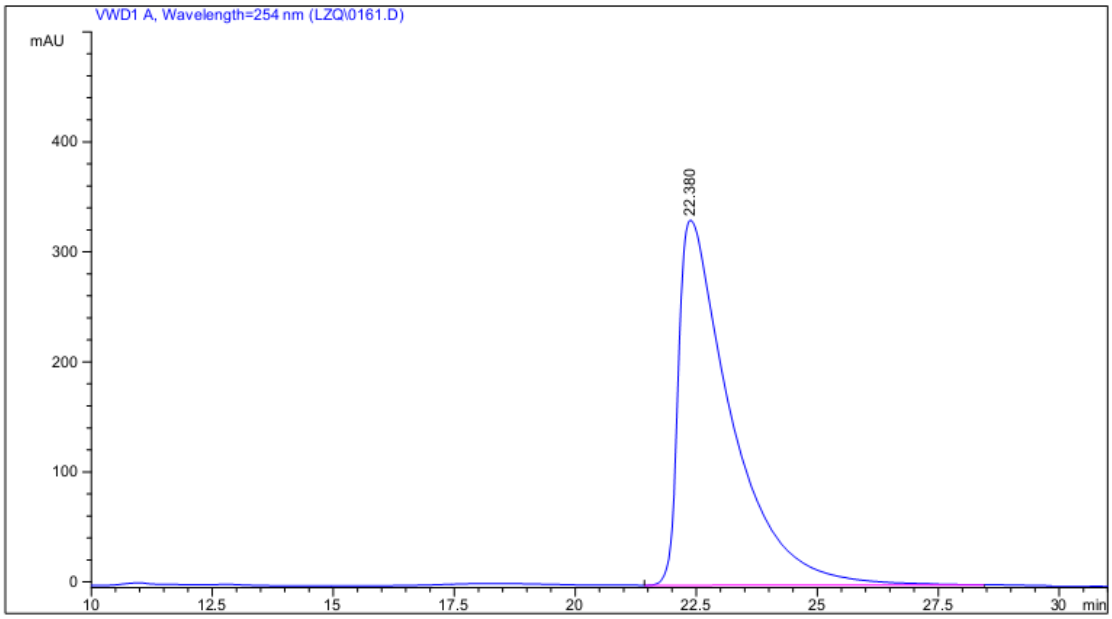
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.945	BV	0.4841	7087.44287	218.02707	100.0000
Totals :				7087.44287	218.02707	



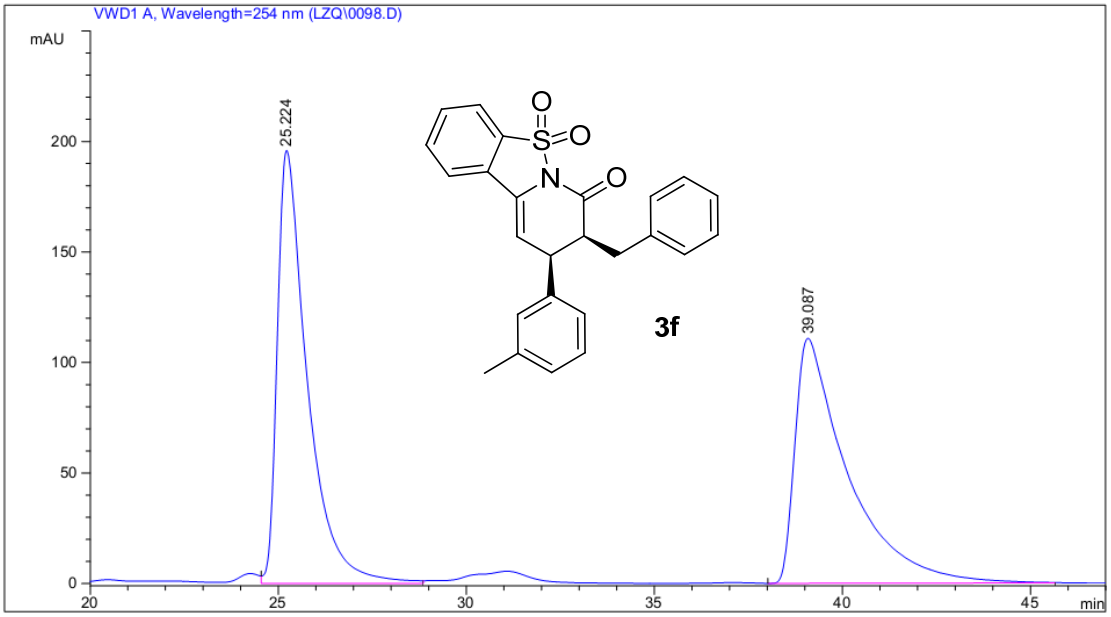
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	23.111	VV	0.9873	1.05964e4	146.87424	50.6862
2	27.636	VB	1.2057	1.03095e4	125.90478	49.3138
Totals :				2.09059e4	272.77901	



Signal 1: VWD1 A, Wavelength=254 nm

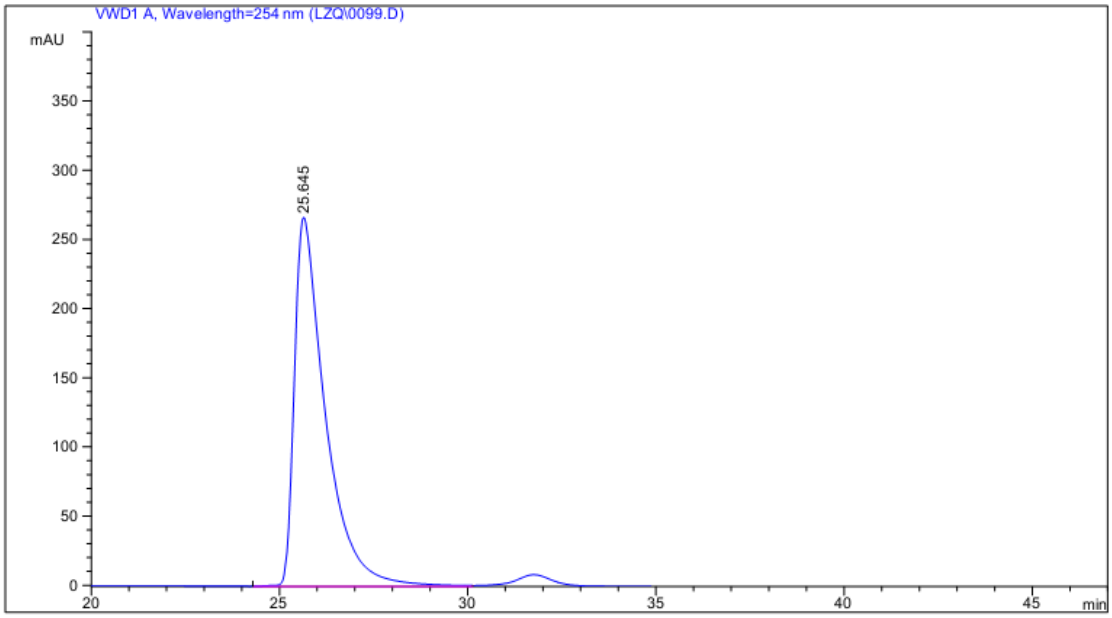
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	22.380	VB	1.0713	2.50878e4	331.82428	100.0000
Totals :				2.50878e4	331.82428	



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	25.224	VB	0.8120	1.08912e4	195.79839	50.2437
2	39.087	VB	1.3049	1.07856e4	110.76645	49.7563

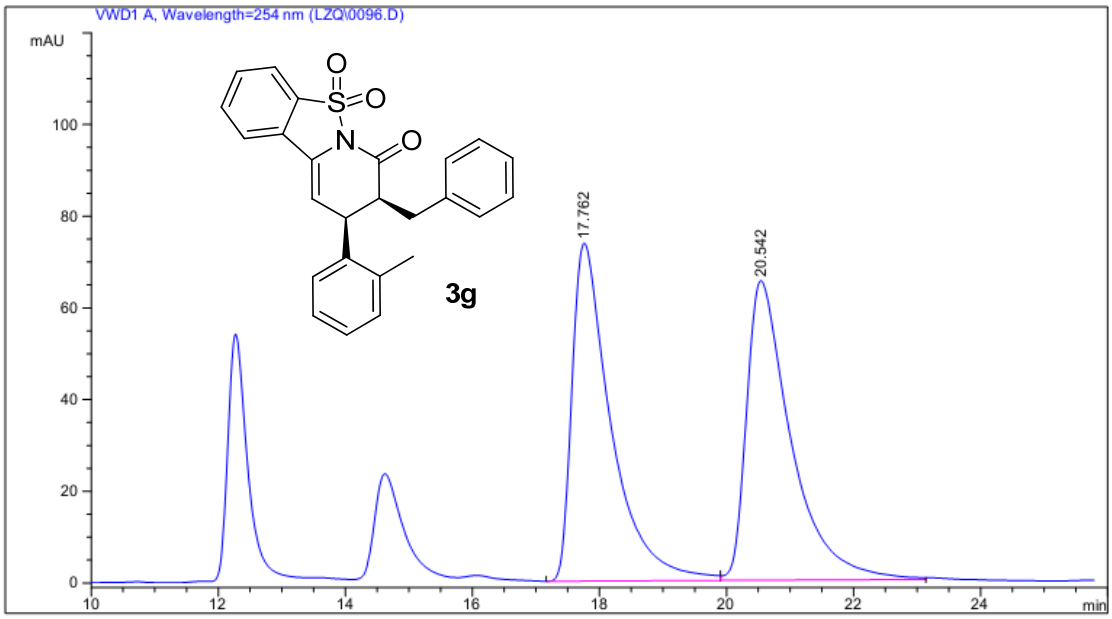
Totals : 2.16768e4 306.56483



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	25.645	BB	0.8243	1.52439e4	266.60263	100.0000

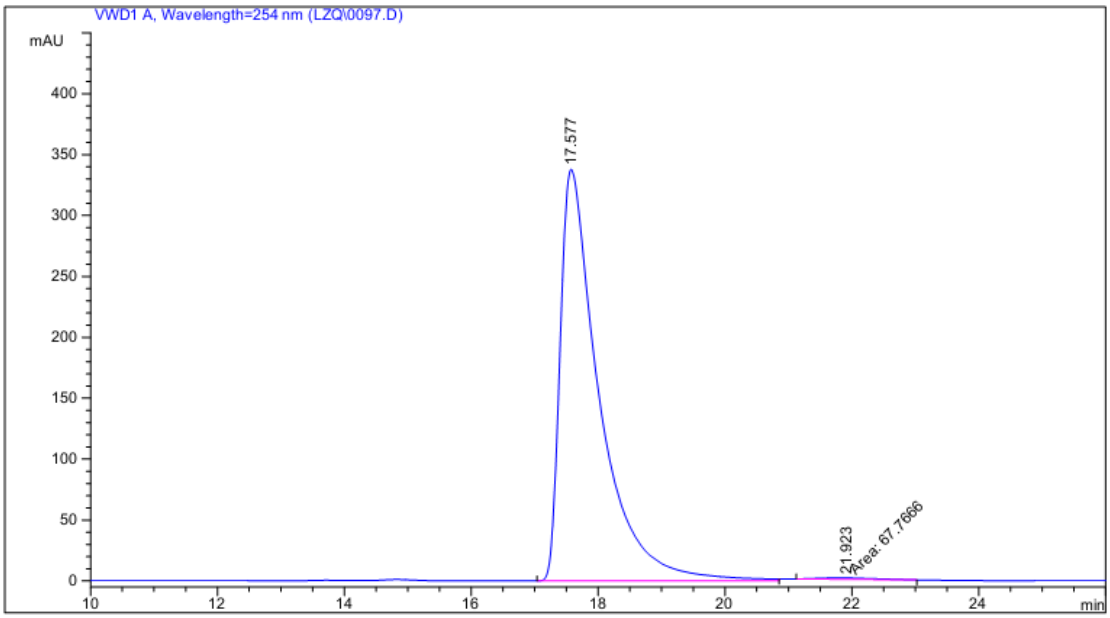
Totals : 1.52439e4 266.60263



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	17.762	VV	0.6191	3139.50928	73.72422	49.7494
2	20.542	VB	0.7120	3171.14331	65.32097	50.2506

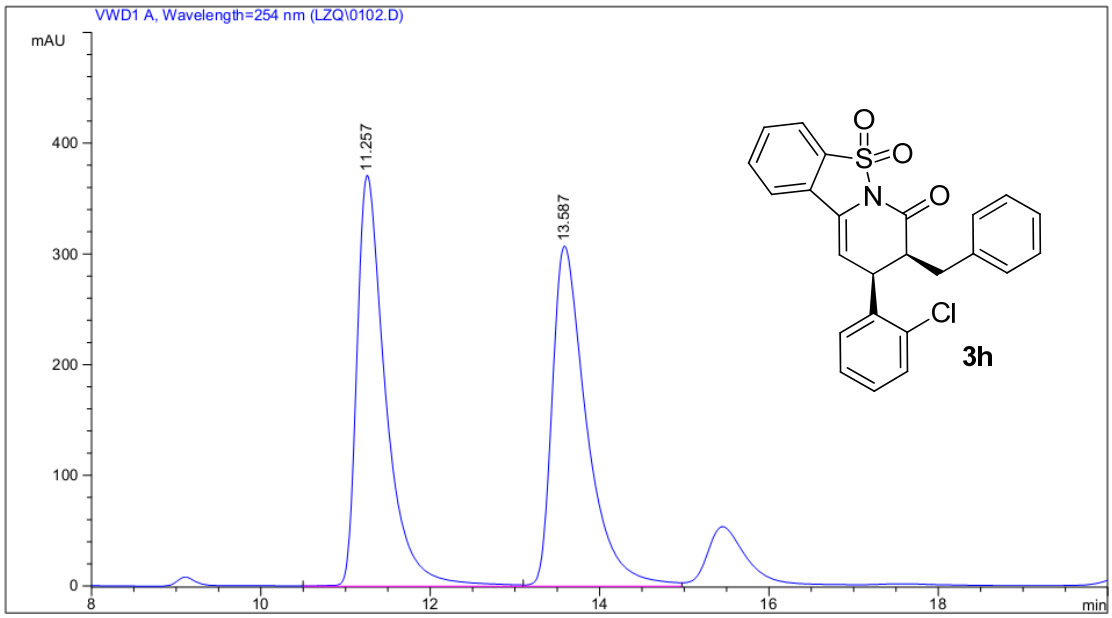
Totals : 6310.65259 139.04519



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	17.577	BV	0.6142	1.44128e4	337.84207	99.5320
2	21.923	MM	1.0763	67.76660	1.04934	0.4680

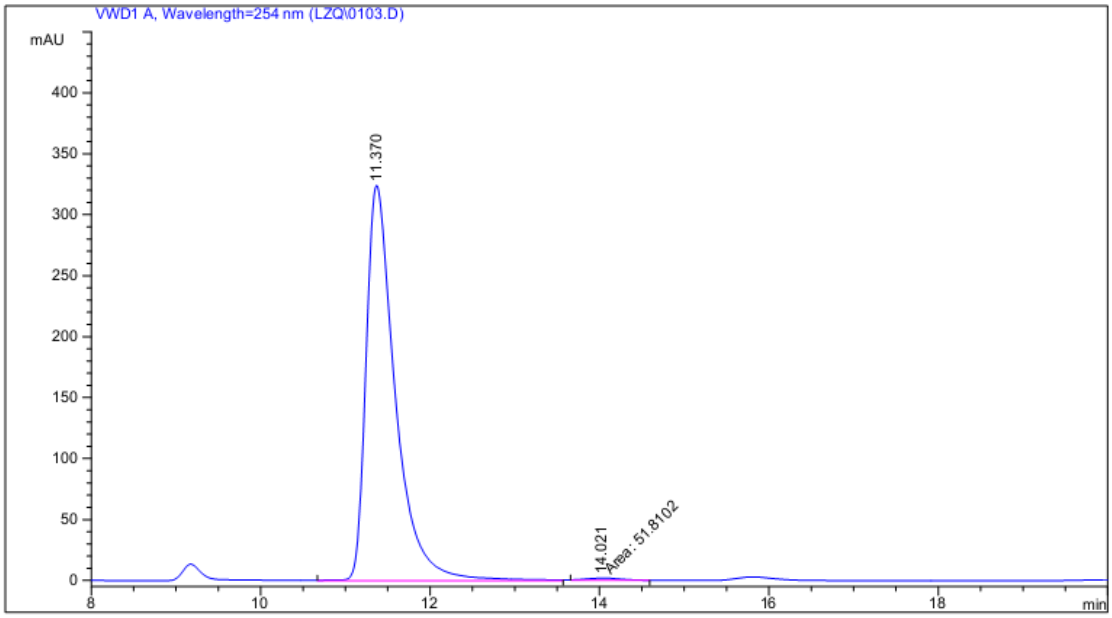
Totals : 1.44806e4 338.89141



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.257	VV	0.3500	8722.66699	371.04694	50.2107
2	13.587	VV	0.4187	8649.45020	307.28674	49.7893

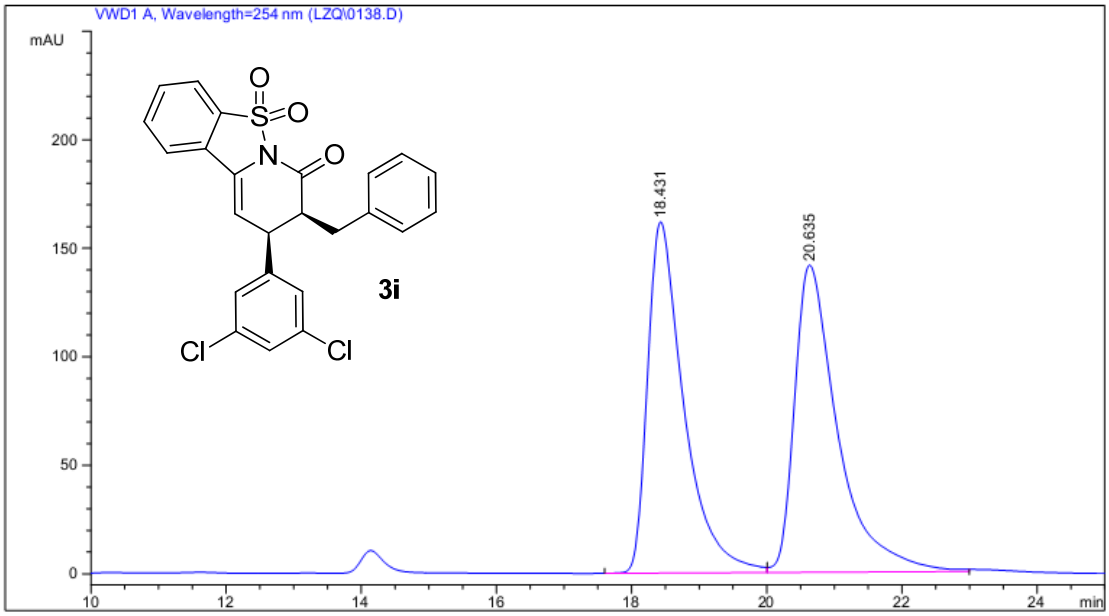
Totals : 1.73721e4 678.33368



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.370	BV	0.3579	7760.12988	324.20984	99.3368
2	14.021	MM	0.4578	51.81020	1.88614	0.6632

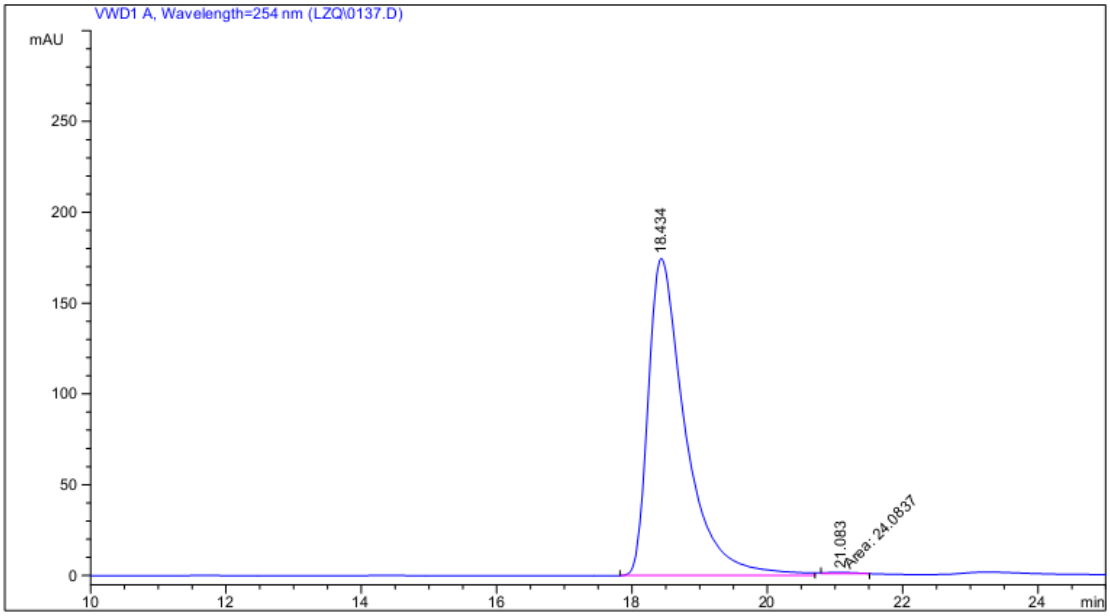
Totals : 7811.94008 326.09598



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	18.431	BV	0.5480	6024.44922	161.78488	49.0196
2	20.635	VB	0.6591	6265.43750	141.55469	50.9804

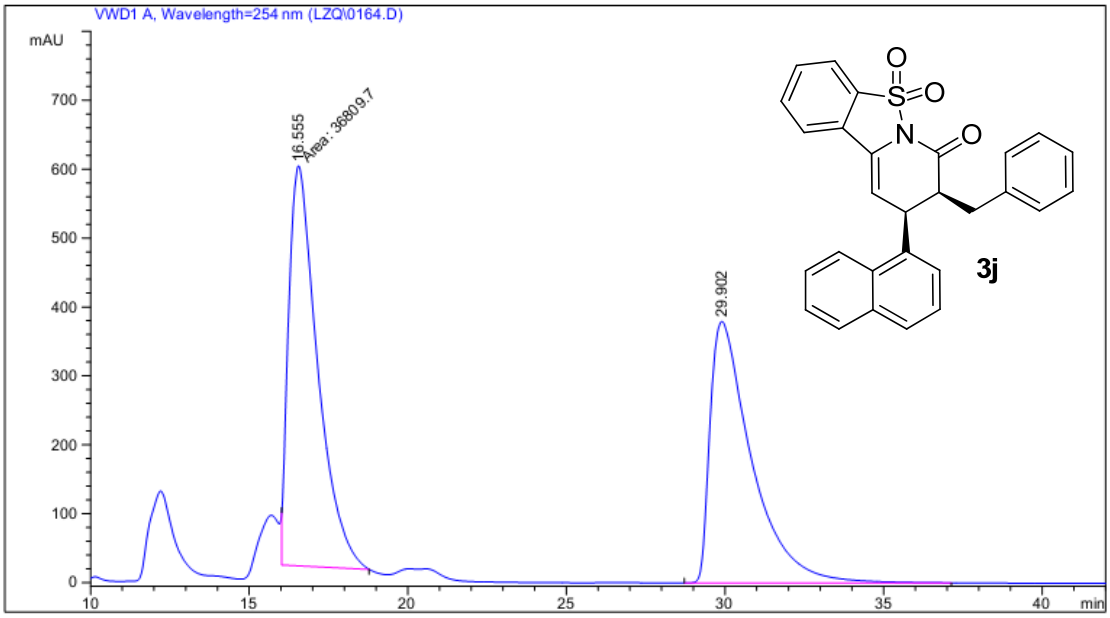
Totals : 1.22899e4 303.33957



Signal 1: VWD1 A, Wavelength=254 nm

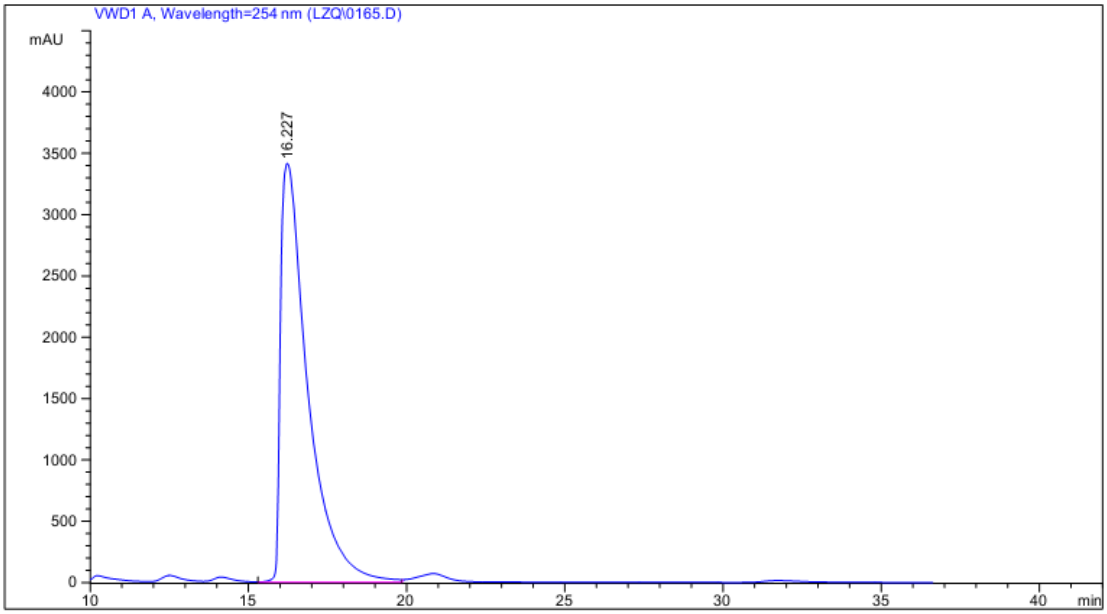
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	18.434	BV	0.5630	6635.36035	174.50510	99.6384
2	21.083	MM	0.5184	24.08370	7.74349e-1	0.3616

Totals : 6659.44405 175.27945



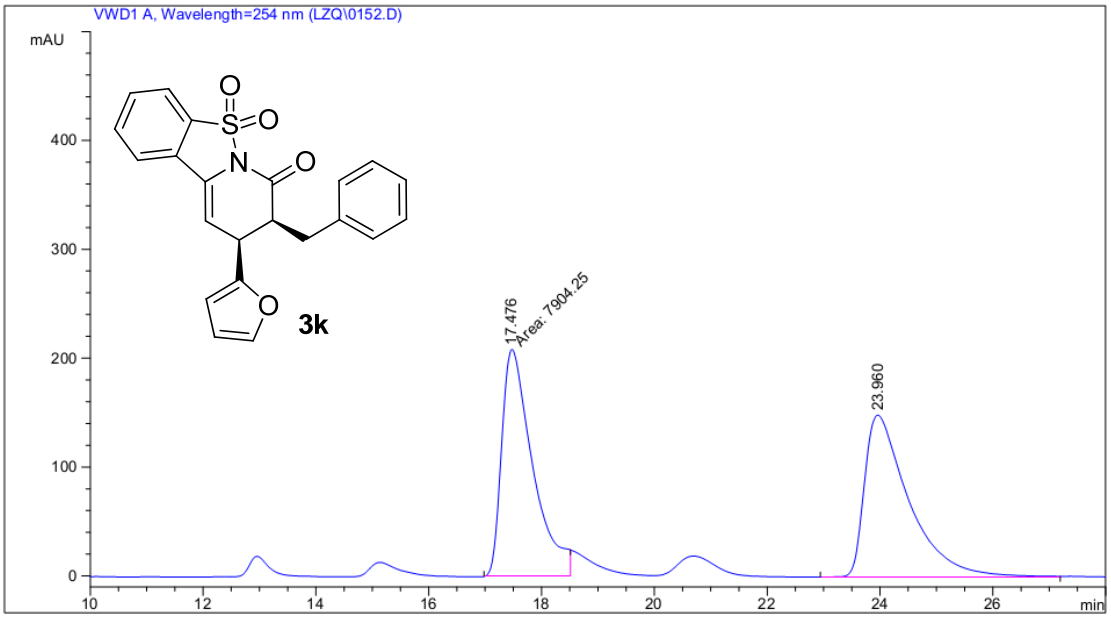
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	16.555	MM	1.0572	3.68097e4	580.30737	51.5687
2	29.902	BB	1.3309	3.45702e4	379.43445	48.4313
Totals :				7.13800e4	959.74182	



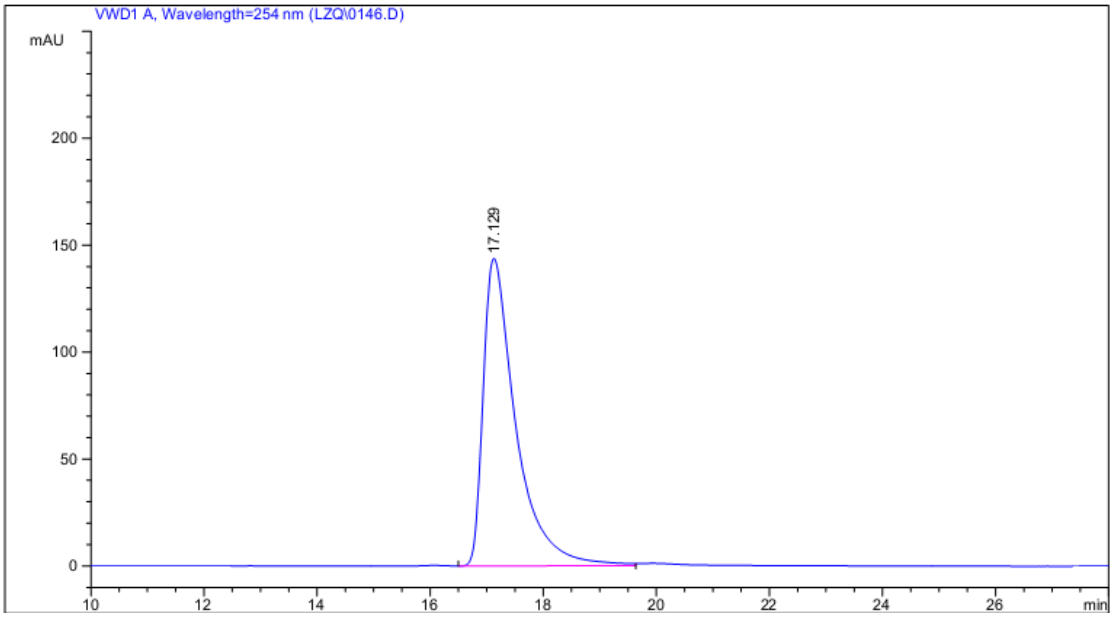
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	16.227	VV	0.8795	2.04755e5	3417.82568	100.0000
Totals :				2.04755e5	3417.82568	



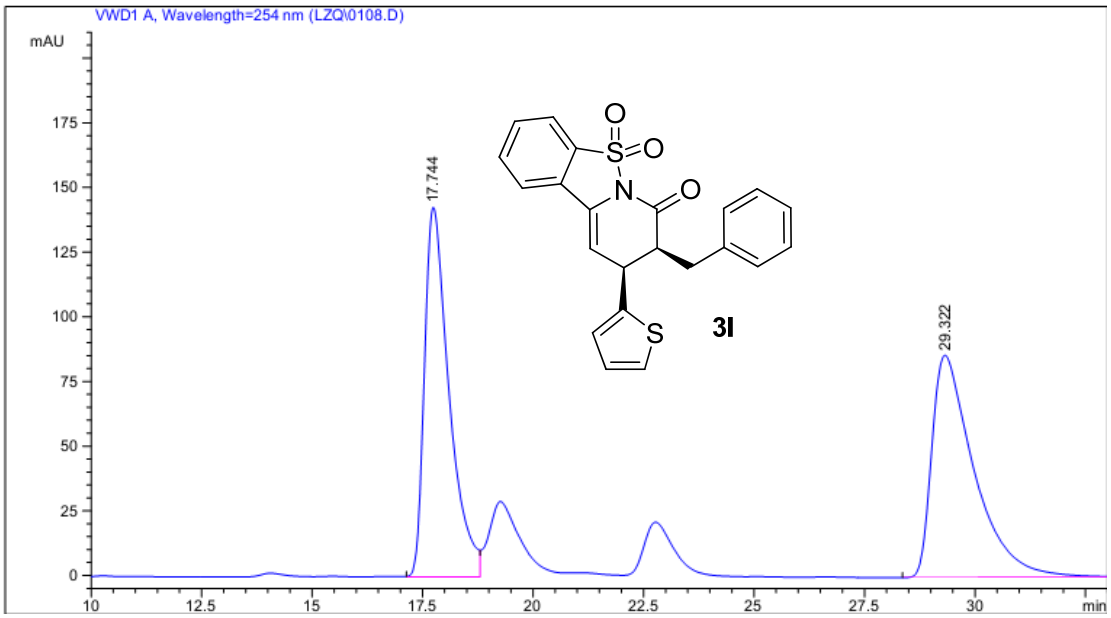
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	17.476	MM	0.6333	7904.25244	208.01161	49.3209
2	23.960	BB	0.8119	8121.93066	148.70094	50.6791
Totals :				1.60262e4	356.71255	



Signal 1: VWD1 A, Wavelength=254 nm

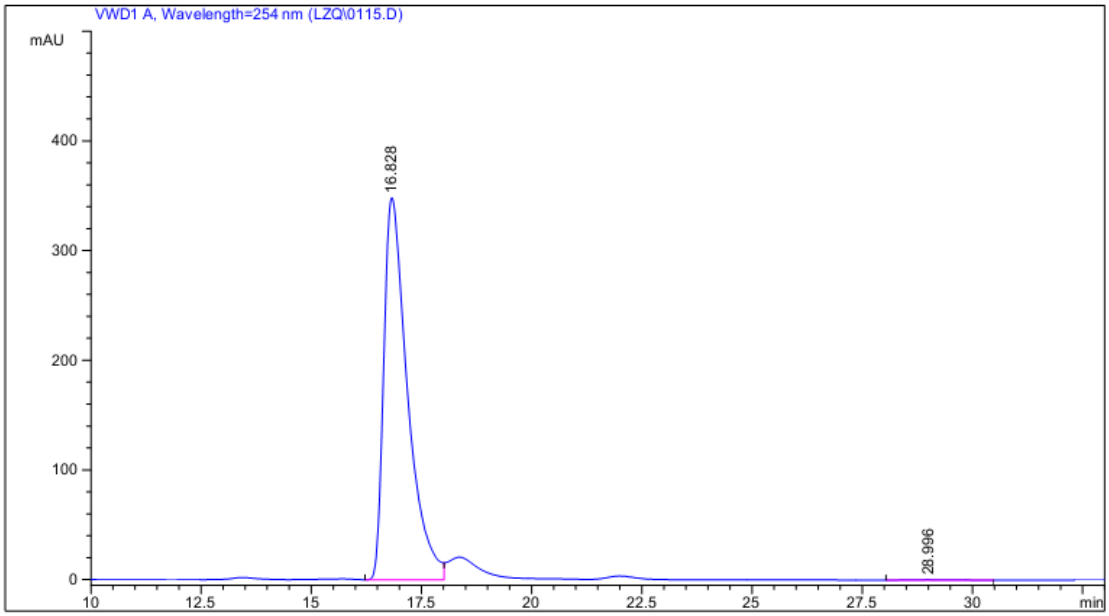
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	17.129	VV	0.5927	5762.56543	143.80203	100.0000
Totals :				5762.56543	143.80203	



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	17.744	BV	0.5737	5529.07422	142.88797	49.1887
2	29.322	BB	0.9675	5711.45557	85.94646	50.8113

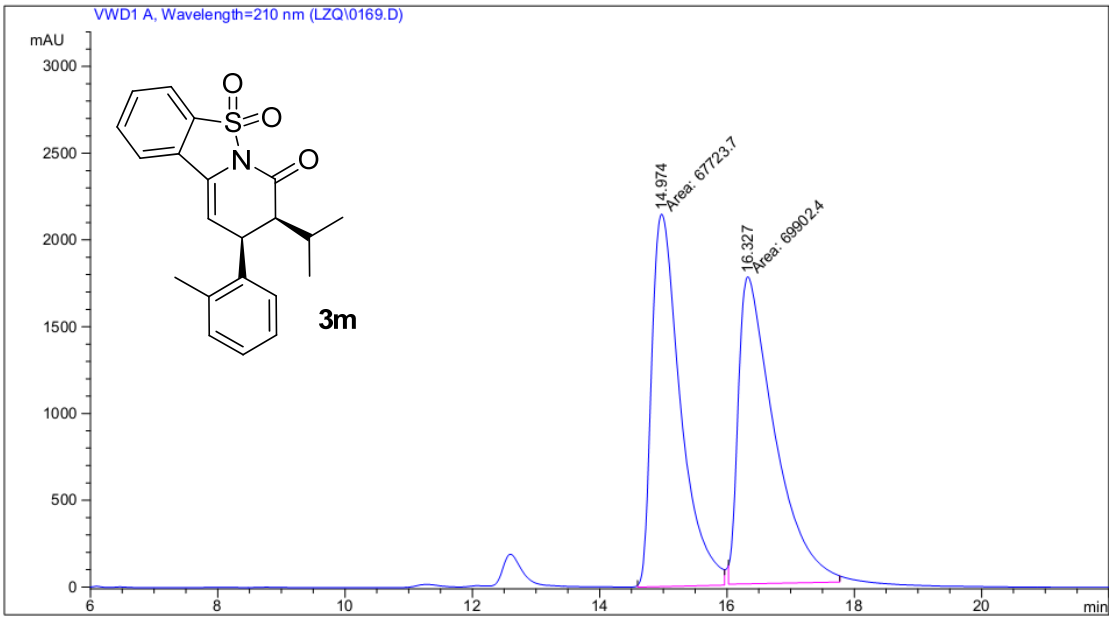
Totals : 1.12405e4 228.83443



Signal 1: VWD1 A, Wavelength=254 nm

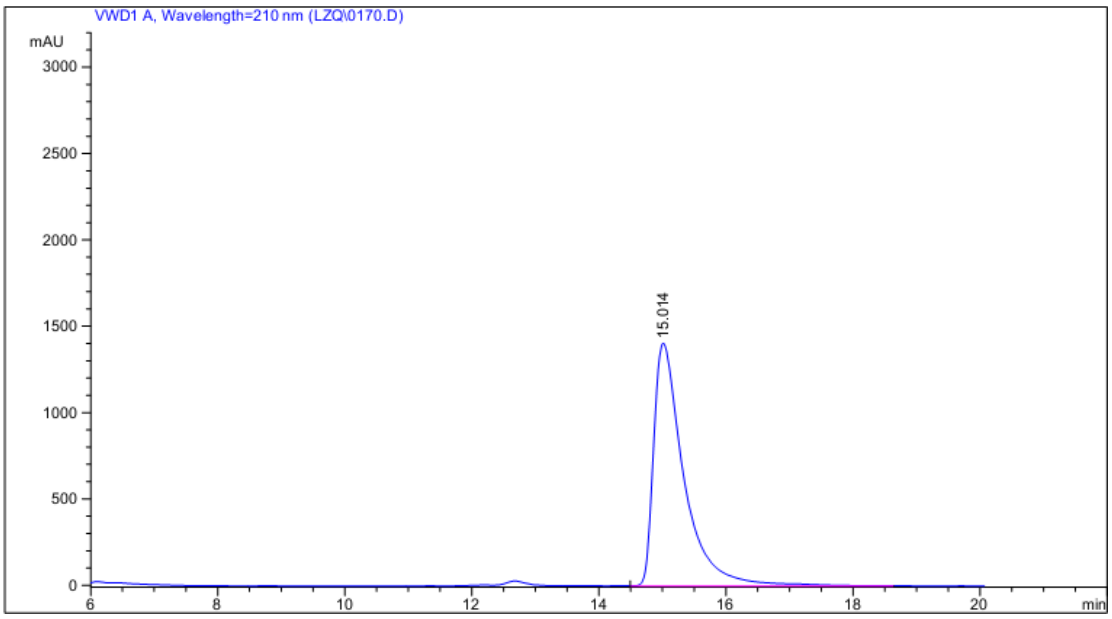
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	16.828	VV	0.5617	1.30366e4	348.43930	99.8224
2	28.996	BB	0.7636	23.19195	3.80436e-1	0.1776

Totals : 1.30598e4 348.81974



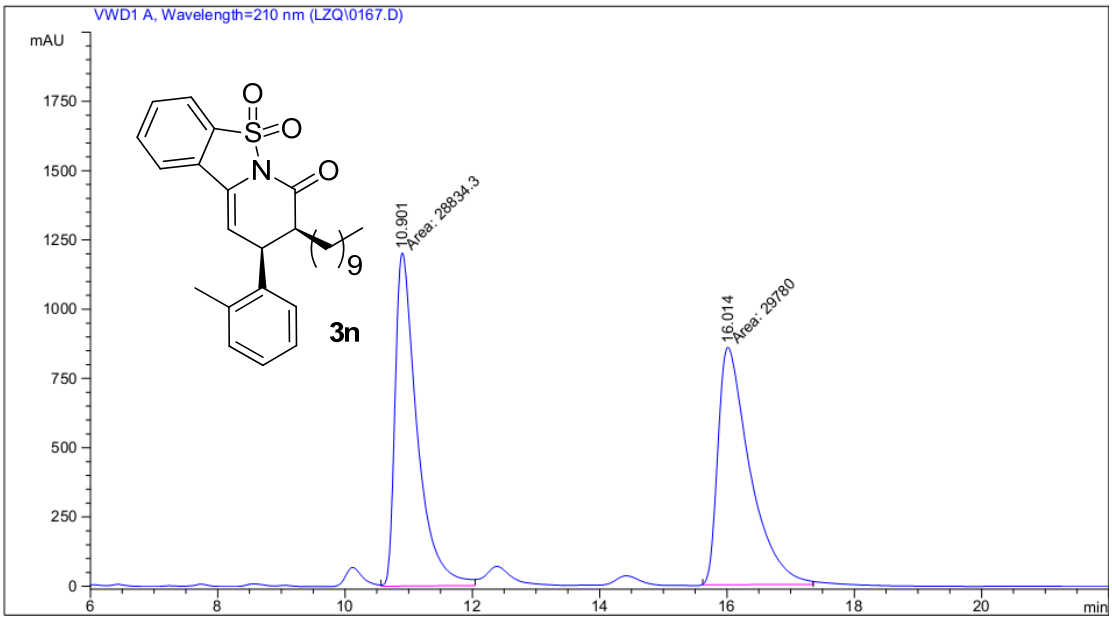
Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.974	MM	0.5258	6.77237e4	2146.66040	49.2085
2	16.327	MM	0.6585	6.99024e4	1769.27527	50.7915
Totals :				1.37626e5	3915.93567	



Signal 1: VWD1 A, Wavelength=210 nm

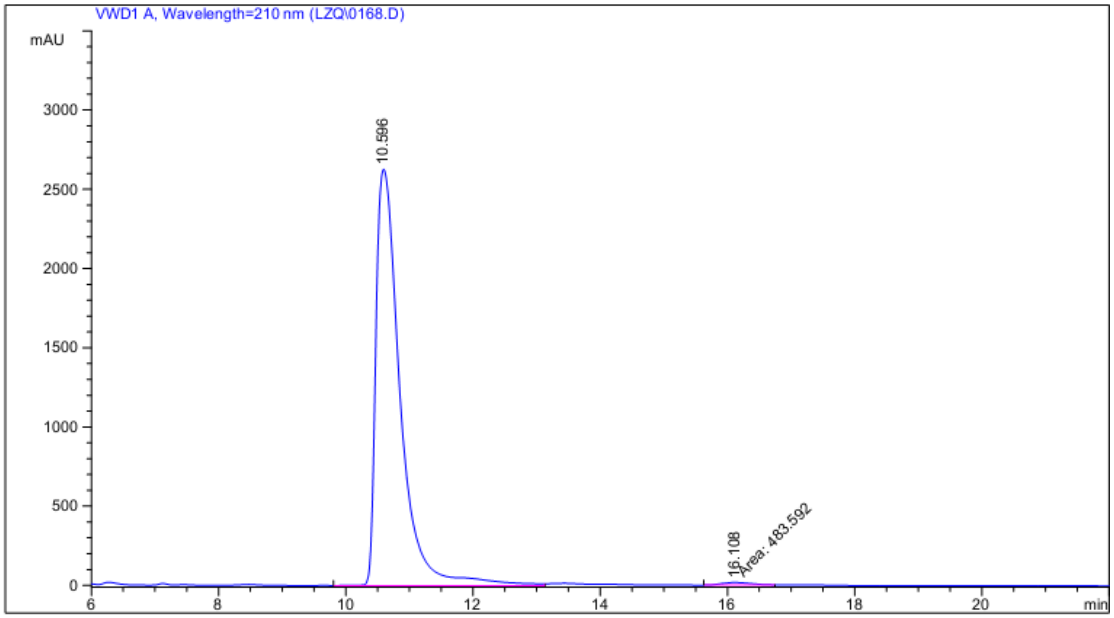
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	15.014	VV	0.4932	4.68427e4	1407.19080	100.0000
Totals :				4.68427e4	1407.19080	



Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.901	MM	0.3996	2.88343e4	1202.76257	49.1932
2	16.014	MM	0.5789	2.97800e4	857.36560	50.8068

Totals : 5.86143e4 2060.12817



Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.596	VV	0.4023	6.96179e4	2627.89233	99.3102
2	16.108	MM	0.5085	483.59198	15.85091	0.6898

Totals : 7.01015e4 2643.74324