Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Asymmetric synthesis of fluoroalkylated *N,O*-ketals via an organocatalytic dehydration/aminalization/aza-michael desymmetrization

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Supporting Information

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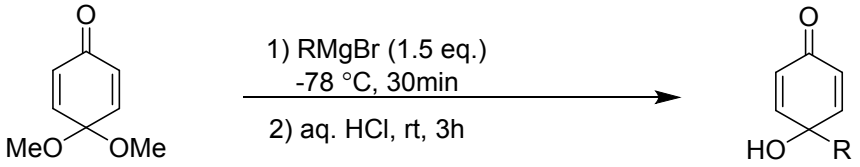
General Information

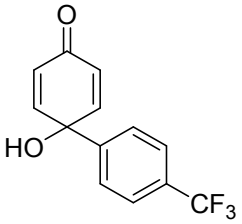
^{1}H NMR and ^{13}C NMR were recorded on a 400MHz Nuclear Magnetic Resonance Spectrometer (^{1}H NMR: 400MHz, ^{13}C NMR: 100MHz) using TMS as internal reference. The chemical shifts (δ) and coupling constants (J) were expressed in ppm and Hz, respectively. UV-Vis Spectrophotometry was carried out on infrared spectrometer. HPLC analysis was carried out on HPLC with a multiple wavelength detector by commercial chiral columns. Optical rotations were measured on a Polarimeter. HRMS (ESI) were recorded on a Q-TOF Premier. Commercially available compounds were used without further purification. Solvents were purified according to the standard procedures unless otherwise noted.

Catalysts **A-B** were prepared from corresponding amino acid[1], catalysts **C-J** were prepared from quinine[2]. Substrates **1**[1] and **2**[3] were synthetized according to literatures.

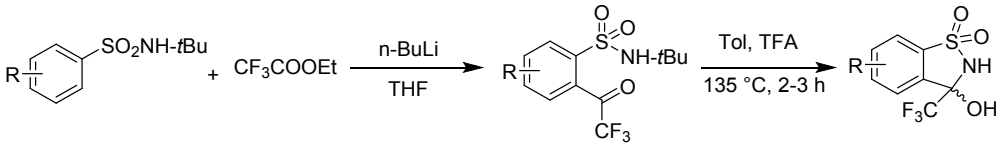
General procedure for the preparation of new dienones and fluoroalkylated hemiaminals



A solution of 4,4-dimethoxycyclohexa-2,5-dien-1-one (5.0 mmol) in THF (10 mL) was added to a solution of Grignard reagents (7.5 mmol) in THF (7.5 mL) at -78 °C. After 30 min, the reaction mixture was acidified by aq. HCl, then, reaction temperature was increased to room temperature. After 3 h, organic layer was extracted with EtOAc and dried in vacuo. The resulting crude product was purified by silica gel column chromatography followed by recrystallization to provide dienones as a solid.

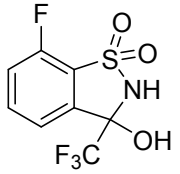


71% yield; white solid; m.p.128-130 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.77-7.75 (d, J = 8.0 Hz, 2H), 7.66-7.64 (d, J = 8.0 Hz, 2H), 6.96-6.93 (d, J =12.0 Hz, 2H), 6.80 (s, 1H), 6.22-6.19 (d, J =12.0 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 185.8, 152.3, 145.6, 129.3-128.4 (q, J = 30 Hz), 126.8, 126.6, 126.1, 123.3, 70.4; ^{19}F NMR (376 MHz, CDCl3) δ -61.0; HRMS (ESI) m/z calcd for $C_{13}H_9F_3NaO_2$ $[M+Na]^+$ 277.0452, found 277.0451.



n-Butyllithium (32 mmol) was added dropwise over 20 minutes period to a cold (0 °C), mechanically stirred solution of the aryl sulfonamide (15 mmol) in anhydrous tetrahydrofuran (100 ml) under a dry nitrogen atmosphere. After stirring an additional 25 min at 0 °C, a precipitate formed. The suspension was cooled further to -78 °C and ethyl trifluoroacetate (45 mmol) was added dropwise over 10 minutes. The resulting mixture was allowed to stir for 4 h at -30 °C and 2 h at ambient

temperature. The reaction was quenched with 5% HCl (40 ml) and extracted with ether (50×3 mL). The combined ether phase was washed with brine (200 mL), dried over anhydrous Na_2SO_4. The solvent was removed and the crude product was obtained which can be used directly in the next step or purified by column chromatography (DCM/PE = 1/3 - 1/1). To the crude product obtained above, TFA (75 mol) in toluene (20 mL) was added and the resulting mixture was stirred at 135 °C in sealed tube. After 2-3 h the solution was concentrated and the resultant solid was dissolved in CH_2Cl_2 (150 mL) and washed with $NaHCO_3$ saturated solution (50×2 mL) to remove traces of TFA. Then the organic phase was washed with brine and dried over Na_2SO_4. The solvent was removed and the obtained crude product was further purified by flash chromatography (PE/EA = 8/1 – 3/1).

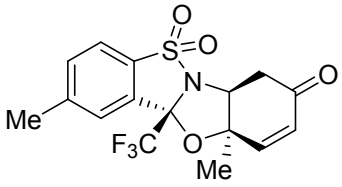


66% yield; white solid; m.p.120-121°C; ^{1}H NMR (400 MHz, acetone-d_6) δ 8.55 (br, 1H), 7.99-7.94 (m, 1H), 7.72-7.70 (d, J =8.0 Hz, 1H), 7.64-7.60 (m, 1H); ^{13}C NMR (100 MHz, acetone-d_6) δ 156.7-154.2 (d, J = 250.0 Hz), 137.1-137.0 (d, J = 10.0 Hz), 136.5, 127.3-119.3 (q, J = 266.7 Hz), 121.6, 119.5, 119.3, 85.6-84.6 (q, J = 33.3 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -82.6, -117.9; HRMS (ESI) m/z calcd for $C_8H_5NO_3F_4S$ $[M+H]^+$ 272.0005, found 271.9993.

General procedure

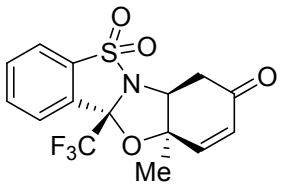
A mixture of dienones **2** (0.30 mmol), fluoroalkylated hemiaminals **1** (0.20 mmol), catalyst (0.040 mmol, chloroform (5.0 mL) in a sealed tube was heated at 50 °C (oil bath) for 72 h, cooled to room temperature, and purified by silica gel chromatography to give compound **3.**

Experimental data of the reaction



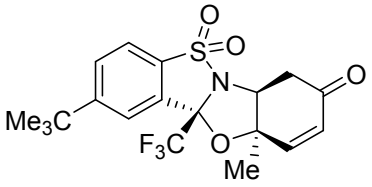
(6a*S*,10a*R*,11a*S*)-2,10a-dimethyl-11a-(trifluoromethyl)-6a,10a-dihydro-11a*H*-benzo[*d*]benzo[4,5]isothiazolo[3,2-*b*]oxazol-8(7*H*)-one 5,5-dioxide (**3a**)

The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid in 98% yield. m.p. 154-155°C; $[\alpha]_D^{20}$ 83.7 (*c* = 1.0, EtOAc, 92%*ee*); HPLC: Daicel Chiralpak OD-H, hexane: 2-propanol = 70: 30, flow rate = 0.9 mL/min, T = 30 °C, UV = 254 nm, t_R = 9.1 min (minor), t_R = 19.7 min (major). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70-7.68 (d, J = 8.0 Hz, 1H), 7.55-7.53 (d, J = 8.0 Hz, 1H), 7.50 (s, 1H), 6.70-6.67 (d, J = 12.0 Hz, 1H), 6.13-6.10 (d, J = 12.0 Hz, 1H), 4.26-4.24 (t, J = 4.0 Hz, 1H), 3.22-3.17 (dd, J = 16.0, 4.0 Hz, 1H), 2.78-2.73 (dd, J = 16.0, 4.0 Hz, 1H), 2.53 (s, 3H), 1.48 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.6, 145.9, 144.9, 133.2, 132.7, 132.3, 127.7, 125.3-116.8 (q, J = 283.3 Hz), 125.0, 120.5, 95.6-94.6 (q, J = 33.3 Hz), 82.4, 65.0, 38.1, 23.4, 20.8; ^{19}F NMR (376 MHz, $CDCl_3$) δ -78.1; HRMS (ESI) m/z calcd for $C_{16}H_{14}F_3NNaO_4S$ $[M+Na]^+$ 396.0493, found 396.0488.



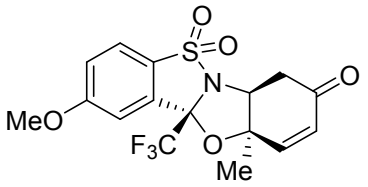
(6a*S*,10a*R*,11a*S*)-10a-methyl-11a-(trifluoromethyl)-6a,10a-dihydro-11a*H*-benzo[*d*]benzo[4,5]isothiazolo[3,2-*b*]oxazol-8(7*H*)-one 5,5-dioxide (**3b**)

The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid in 96% yield. m.p. 143-144°C; $[\alpha]_D^{20}$ 58.6 (*c* = 1.0, EtOAc, 90%*ee*); HPLC: Daicel Chiralpak AD-H, hexane: 2-propanol = 90:10, flow rate = 0.8 mL/min, T = 30 °C, UV = 254 nm, t_R = 9.5 min (minor), t_R = 10.8 min (major). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82-7.73 (m, 4H), 6.71-6.68 (d, *J* = 12.0 Hz, 1H), 6.12-6.09 (d, *J* = 12.0 Hz, 1H), 4.25 (s, 1H), 3.21-3.16 (dd, *J* = 16.0, 4.0 Hz, 1H), 2.79-2.73 (dd, *J* = 16.0, 4.0 Hz, 1H), 1.48(s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.5, 146.8, 136.0, 134.5, 133.9, 132.8, 128.9, 126.3-117.7 (q, *J* = 286.7 Hz), 125.9, 121.9, 96.8-95.7 (q, *J* = 36.7 Hz), 83.5, 66.1, 39.1, 24.4. ^{19}F NMR (376 MHz, $CDCl_3$) δ -78.0; HRMS (ESI) m/z calcd for $C_{15}H_{12}F_3NNaO_4S$ $[M+Na]^+$ 382.0337, found 382.0334.



(6a*S*,10a*R*,11a*S*)-2-(*tert*-butyl)-10a-methyl-11a-(trifluoromethyl)-6a,10a-dihydro-11a*H*-benzo[*d*]benzo[4,5]isothiazolo[3,2-*b*]oxazol-8(7*H*)-one 5,5-dioxide (**3c**)

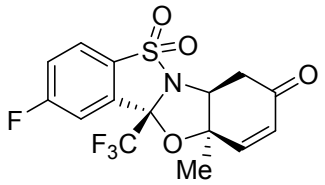
The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid in 92% yield. m.p. 202-203°C; $[\alpha]_D^{20}$ 63.8 (*c* = 1.0, EtOAc, 91%*ee*); HPLC: Daicel Chiralpak OD-H, hexane: 2-propanol = 70: 30, flow rate = 0.9 mL/min, T = 30 °C, UV = 254 nm, t_R = 7.3 min (minor), t_R = 24.8 min (major). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79-7.68 (m, 3H), 6.71-6.68 (d, *J* = 12.0 Hz, 1H), 6.13-6.10 (d, *J* = 12.0 Hz, 1H), 4.27-4.25 (t, *J* = 4.0 Hz, 1H), 3.23-3.17 (dd, *J* = 16.0, 4.0 Hz, 1H), 2.78-2.73 (dd, *J* = 16.0, 4.0 Hz, 1H), 1.49 (s, 3H), 1.39 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.6, 159.2, 147.0, 134.1, 133.2, 130.5, 128.8, 126.4-117.8 (q, *J* = 286.7 Hz), 122.3, 121.4, 96.5-95.8 (q, *J* = 35.0 Hz), 83.5, 66.0, 39.1, 35.7, 31.1, 24.4; ^{19}F NMR (376 MHz, $CDCl_3$) δ -77.9; HRMS (ESI) m/z calcd for $C_{19}H_{20}F_3NNaO_4S$ $[M+Na]^+$ 438.0963, found 438.0955.



(6a*R*,10a*R*,11a*S*)-2-methoxy-10a-methyl-11a-(trifluoromethyl)-6a,10a-dihydro-11a*H*-benzo[*d*]benzo[4,5]isothiazolo[3,2-*b*]oxazol-8(7*H*)-one 5,5-dioxide (**3d**)

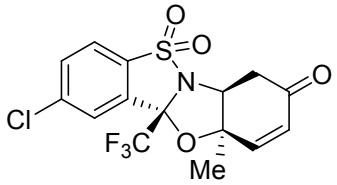
The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid in 93% yield. m.p. 207-208°C; $[\alpha]_D^{20}$ 74.2 (*c* = 1.0, EtOAc, 83%*ee*); HPLC: Daicel Chiralpak OD-H, hexane: 2-propanol = 70: 30, flow rate = 0.9 mL/min, T = 30 °C, UV = 254 nm, t_R = 13.7 min (minor), t_R = 26.3 min (major). ^{1}H NMR

(400 MHz, $CDCl_3$) δ 7.71-7.69 (d, J = 8.0 Hz, 1H), 7.23-7.11 (m, 2H), 6.69-6.66 (d, J = 12.0 Hz, 1H), 6.13-6.10 (d, J = 12.0 Hz, 1H), 4.27-4.25 (t, J = 4.0 Hz, 1H), 3.93(s, 3H), 3.22-3.17 (dd, J = 16.0, 4.0 Hz, 1H), 2.78-2.72 (dd, J = 16.0, 4.0 Hz, 1H), 1.49 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.6, 163.4, 145.7, 135.5, 127.8, 126.7, 122.4-119.6 (q, J = 280.0 Hz), 122.3, 118.7, 108.5, 94.6-94.3 (q, J = 30.0 Hz) 82.5, 65.0, 55.2, 38.1, 23.4, ^{19}F NMR (376 MHz, $CDCl_3$) δ -78.1; HRMS (ESI) m/z calcd for $C_{16}H_{14}F_3NNaO_5S$ $[M+Na]^+$ 412.0442, found 412.0440.



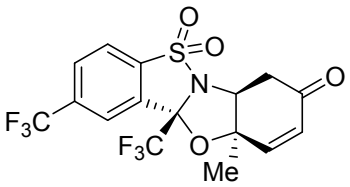
(6a*S*,10a*R*,11a*S*)-2-fluoro-10a-methyl-11a-(trifluoromethyl)-6a,10a-dihydro-11a*H*-benzo[*d*]benzo[4,5]isothiazolo[3,2-*b*]oxazol-8(7*H*)-one 5,5-dioxide (**3e**)

The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid in 85% yield. m.p. 174-175°C; $[\alpha]_D^{20}$ 53.3 (*c* = 1.0, EtOAc, 88%*ee*); HPLC: Daicel Chiralpak OD-H, hexane: 2-propanol = 70: 30, flow rate = 0.9 mL/min, T = 23 °C, UV = 254 nm, t_R = 10.7 min (minor), t_R = 17.6 min (major). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84-7.81 (m, 1H), 7.48-7.39 (m, 2H), 6.70-6.67 (d, J = 12.0 Hz, 1H), 6.14-6.11 (d, J = 12.0 Hz, 1H), 4.26-4.24 (t, J = 4.0 Hz, 1H), 3.21-3.16 (dd, J = 16.0, 4.0 Hz, 1H), 2.79-2.74 (dd, J = 16.0, 4.0 Hz, 1H), 1.52 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.3, 167.2-164.6 (d, J = 260.0 Hz), 146.6, 137.1-137.0 (d, J = 10.0 Hz), 132.0, 129.0, 126.1-117.5 (q, J = 286.7 Hz), 124.4-124.3 (d, J = 10.0 Hz), 121.1-120.8 (d, J = 30.0 Hz), 113.4-113.2 (d, J =20.0 Hz), 95.6-95.3(q, J = 30.0 Hz), 83.8, 66.4, 38.9, 24.3; ^{19}F NMR (376 MHz, $CDCl_3$) δ -77.9, -100.9 ; HRMS (ESI) m/z calcd for $C_{15}H_{11}F_4NNaO_4S$ $[M+Na]^+$ 400.0243, found 400.0244.



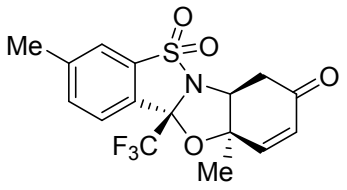
(6a*S*,10a*R*,11a*S*)-2-chloro-10a-methyl-11a-(trifluoromethyl)-6a,10a-dihydro-11a*H*-benzo[*d*]benzo[4,5]isothiazolo[3,2-*b*]oxazol-8(7*H*)-one 5,5-dioxide (**3f**)

The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid in 97% yield. m.p. 201-202°C; $[\alpha]_D^{20}$ 59.1 (*c* = 1.0, EtOAc, 84%*ee*); HPLC: Daicel Chiralpak OD-H, hexane: 2-propanol = 70: 30, flow rate = 0.9 mL/min, T = 30 °C, UV = 254 nm, t_R = 8.7 min (minor), t_R = 16.9 min (major). ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.24-8.22 (d, J = 8.0 Hz, 1H), 8.03-8.01 (d, J = 8.0 Hz, 1H), 7.95 (s, 1H), 6.89-6.86 (d, J = 12.0 Hz, 1H), 6.11-6.08 (d, J = 12.0 Hz, 1H), 4.36 (s, 1H), 3.24-3.18 (dd, J = 16.0, 4.0 Hz, 1H), 2.80-2.75 (dd, J = 16.0, 4.0 Hz, 1H), 1.57 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 194.0, 147.6, 140.2, 135.2, 134.1, 134.0, 128.0, 126.1-117.5 (q, J = 286.7 Hz), 125.7, 124.2, 95.3-94.2 (q, J = 36.7 Hz), 84.8, 66.3, 38.9, 22.6; ^{19}F NMR (376 MHz, DMSO-d_6) δ -76.9; HRMS (ESI) m/z calcd for $C_{15}H_{11}ClF_3NNaO_4S$ $[M+Na]^+$ 415.9947, found 415.9951.



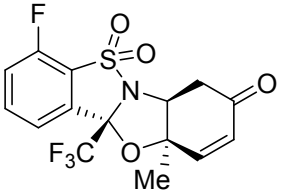
(6a*S*,10a*R*,11a*S*)-10a-methyl-2,11a-bis(trifluoromethyl)-6a,10a-dihydro-11a*H*-benzo[*d*]benzo[4,5]isothiazolo[3,2-*b*]oxazol-8(7*H*)-one 5,5-dioxide (**3g**)

The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid in 93% yield. m.p. 188-189°C; $[\alpha]_D^{20}$ 43.1 (*c* = 1.0, EtOAc, 90%*ee*); HPLC: Daicel Chiralpak OD-H, hexane: 2-propanol = 70: 30, flow rate = 0.9 mL/min, T = 30 °C, UV = 254 nm, t_R = 8.5 min (minor), t_R = 25.2 min (major). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03-7.94 (m, 3H), 6.70-6.67 (d, *J* = 12.0 Hz, 1H), 6.14-6.11 (d, *J* = 12.0 Hz, 1H), 4.24-4.22 (t, *J* = 4.0 Hz, 1H), 3.21-3.16 (dd, *J* = 16.0, 4.0 Hz, 1H), 2.79-2.73 (dd, *J* = 16.0, 4.0 Hz, 1H), 1.51 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 194.6, 148.1, 139.4, 136.1-135.1 (q, *J* = 33.3 Hz),134.8, 131.74, 131.70, 128.5, 124.7, 124.6-119.1 (q, *J* = 275.0 Hz), 123.3, 95.6-94.9 (q, *J* = 35.0 Hz), 85.5, 66.9, 39.4, 23.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ -61.4, -76.8; HRMS (ESI) m/z calcd for $C_{16}H_{11}F_6NNaO_4S$ $[M+Na]^+$ 450.0211, found 450.0216.



(6a*S*,10a*R*,11a*S*)-3,10a-dimethyl-11a-(trifluoromethyl)-6a,10a-dihydro-11a*H*-benzo[*d*]benzo[4,5]isothiazolo[3,2-*b*]oxazol-8(7*H*)-one 5,5-dioxide (**3h**)

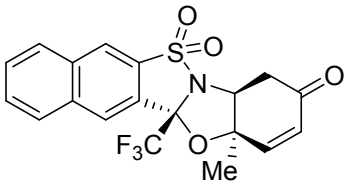
The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid in 88% yield. m.p. 157-158°C; $[\alpha]_D^{20}$ 75.3 (*c* = 1.0, EtOAc, 87%*ee*); HPLC: Daicel Chiralpak OD-H, hexane: 2-propanol = 70: 30, flow rate = 0.9 mL/min, T = 30 °C, UV = 254 nm, t_R = 8.9 min (minor), t_R = 14.6 min (major). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62-7.56 (m, 3H), 6.70-6.67 (d, *J* = 12.0 Hz, 1H), 6.11- 6.08 (d, *J* = 12.0 Hz, 1H), 4.25- 4.23 (t, *J* = 4.0 Hz, 1H), 3.19-3.13 (dd, *J* = 16.0, 4.0 Hz, 1H), 2.78-2.73 (dd, *J* = 16.0, 4.0 Hz, 1H), 2.51 (s, 3H), 1.47 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.3, 145.9, 143.1, 135.0, 134.5, 130.1, 127.7, 125.3-116.8 (q, *J* = 283.3 Hz), 124.5, 120.7, 95.7-94.6 (q, *J* = 36.7 Hz), 82.4, 65.0, 38.1, 23.4, 20.5; ^{19}F NMR (376 MHz, $CDCl_3$) δ -78.0; HRMS (ESI) m/z calcd for $C_{16}H_{14}F_3NNaO_4S$ $[M+Na]^+$ 396.0493, found 396.0496.



(6a*S*,10a*R*,11a*S*)-4-fluoro-10a-methyl-11a-(trifluoromethyl)-6a,10a-dihydro-11a*H*-benzo[*d*]benzo[4,5]isothiazolo[3,2-*b*]oxazol-8(7*H*)-one 5,5-dioxide (**3i**)

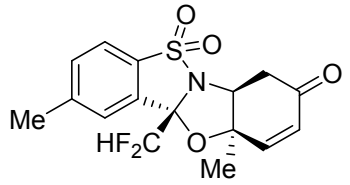
The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid in 83% yield. m.p. 166-167°C; $[\alpha]_D^{20}$ 48.4 (*c* = 1.0, EtOAc, 85%*ee*); HPLC: Daicel Chiralpak AD-H, hexane: 2-propanol = 90: 10, flow

rate = 1.0 mL/min, T = 30 °C, UV = 254 nm, t_R = 9.2 min (major), t_R = 10.0 min (minor). ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.05-8.00 (m, 1H), 7.84-7.80 (m, 1H), 7.71-7.69 (d, J = 8.0 Hz, 1H), 6.88-6.85 (d, J = 12.0 Hz, 1H), 6.11-6.08 (d, J = 12.0 Hz, 1H), 4.47 (s, 1H), 3.24-3.19 (dd, J = 16.0, 4.0 Hz, 1H), 2.78-2.74 (d, J = 16.0 Hz, 1H), 1.56 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 194.0, 156.3-153.8 (d, J = 300.0 Hz), 147.5, 139.0-138.9 (d, J = 10.0 Hz), 135.8, 128.1, 126.1-117.5 (q, J = 286.7 Hz), 122.9-122.7 (d, J = 20.0 Hz), 122.1, 120.5-120.3 (d, J = 20.0 Hz), 95.3-94.6 (d, J = 35.0 Hz), 84.9, 66.4, 38.9, 22.6; ^{19}F NMR (376 MHz, DMSO-d_6) δ -76.9, -115.9; HRMS (ESI) m/z calcd for $C_{15}H_{11}F_4NNaO_4S$ $[M+Na]^+$ 400.0243, found 400.0244.



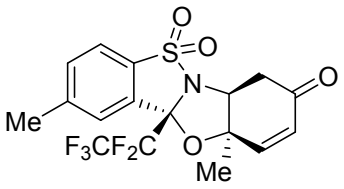
(4a*S*,12b*S*,13a*R*)-13a-methyl-12b-(trifluoromethyl)-4a,13a-dihydro-12b*H*-benzo[*d*]naphtho[2',3':4,5]isothiazolo[3,2-*b*]oxazol-3(4*H*)-one 6,6-dioxide (**3j**)

The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid in 92% yield. m.p. 136-137°C; $[\alpha]_D^{20}$ 74.9 (*c* = 1.0, EtOAc, 88%*ee*); HPLC: Daicel Chiralpak OD-H, hexane: 2-propanol = 70: 30, flow rate = 0.9 mL/min, T = 30 °C, UV = 254 nm, t_R = 13.1 min (minor), t_R = 21.1 min (major). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.34 (s, 1H), 8.22 (s, 1H), 8.07-8.03 (m, 2H), 7.77-7.70 (m, 2H), 6.74-6.71 (d, J = 12.0 Hz, 1H), 6.15-6.12 (d, J = 12.0 Hz, 1H), 4.32-4.30 (t, J = 4.0 Hz, 1H), 3.27-3.21 (dd, J = 16.0, 4.0 Hz, 1H), 2.82-2.76 (dd, J = 16.0, 4.0 Hz, 1H), 1.46 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.7, 145.8, 134.4, 133.1, 131.9, 128.8, 128.5, 128.4, 128.2, 128.1, 127.8, 125.4-116.9 (q, J = 283.3 Hz), 125.3, 121.8, 95.7-94.7 (q, J = 33.3 Hz), 82.4, 65.1, 38.2, 23.4; ^{19}F NMR (376 MHz, $CDCl_3$) δ -78.1; HRMS (ESI) m/z calcd for $C_{19}H_{14}F_3NNaO_4S$ $[M+Na]^+$ 432.0493, found 432.0493.



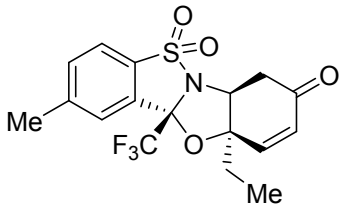
(6a*S*,10a*R*,11a*S*)-11a-(difluoromethyl)-2,10a-dimethyl-6a,10a-dihydro-11a*H*-benzo[*d*]benzo[4,5]isothiazolo[3,2-*b*]oxazol-8(7*H*)-one 5,5-dioxide (**3k**)

The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid in 90% yield. m.p. 198-199°C; $[\alpha]_D^{20}$ 54.6 (*c* = 1.0, EtOAc, 82%*ee*); HPLC: Daicel Chiralpak OD-H, hexane: 2-propanol = 70: 30, flow rate = 0.9 mL/min, T = 30 °C, UV = 254 nm, t_R = 12.7 min (minor), t_R = 17.5 min (major). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66-7.64 (d, J = 8.0 Hz, 1H), 7.52-7.49 (m, 2H), 6.75-6.72 (d, J = 12.0 Hz, 1H), 6.15-6.12 (d, J = 12.0 Hz, 1H), 6.02-5.75 (t, J = 54.0 Hz, 1H), 4.15 (s, 1H), 3.21-3.17 (d, J = 16.0 Hz, 1H), 2.75-2.70 (dd, J = 16.0, 4.0 Hz, 1H), 2.51 (m, 3H), 1.56 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.5, 147.7, 145.6, 134.3, 133.6, 133.3, 129.4, 126.5, 121.3, 115.0-110.0 (q, J = 250.0 Hz), 96.9-96.3 (q, J = 30 Hz), 82.9, 65.9, 38.8, 23.4, 21.9; ^{19}F NMR (376 MHz, $CDCl_3$) δ -126.3, -127.1, -128.4, -129.1; HRMS (ESI) m/z calcd for $C_{16}H_{15}F_2NNaO_4S$ $[M+Na]^+$ 378.0588, found 378.0591.



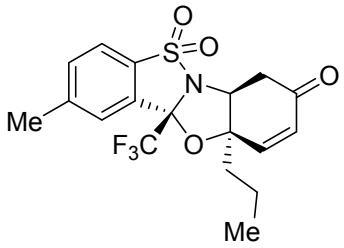
(6a*S*,10a*R*,11a*S*)-2,10a-dimethyl-11a-(perfluoroethyl)-6a,10a-dihydro-11a*H*-benzo[*d*]benzo[4,5]isothiazolo[3,2-*b*]oxazol-8(7*H*)-one 5,5-dioxide (**3l**)

The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid in 95% yield. m.p. 186-187°C; $[\alpha]_D^{20}$ 62.9 (*c* = 1.0, EtOAc, 81%*ee*); HPLC: Daicel Chiralpak OD-H, hexane: 2-propanol = 70: 30, flow rate = 1.0 mL/min, T = 30 °C, UV = 254 nm, t_R = 7.0 min (minor), t_R = 17.3 min (major). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71-7.69 (d, *J* = 8.0 Hz, 1H), 7.55-.53 (d, *J* = 8.0 Hz, 1H), 7.50 (s, 1H), 6.68-6.65 (d, *J* = 12.0 Hz, 1H), 6.12-6.09 (d, *J* = 12.0 Hz, 1H), 4.33-4.30 (t, *J* = 4.0 Hz, 1H), 3.18-3.13 (dd, *J* = 16.0, 4.0 Hz, 1H), 2.81-2.75 (dd, *J* = 16.0, 4.0 Hz, 1H), 2.53 (s, 3H), 1.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.0, 145.1, 144.6, 133.9, 132.6, 132.2, 127.8, 125.2, 121.7-113.3 (dt, *J* = 280.0, 35.0 Hz), 120.7, 113.3-107.6 (dt, *J* = 264.0, 35.0 Hz), 97.1-96.6 (q, *J* = 25.0 Hz), 82.2, 64.1, 38.4, 24.3, 20.9; ^{19}F NMR (376 MHz, $CDCl_3$) δ -77.9, -117.4, -118.2, -118.5, -119.2; HRMS (ESI) m/z calcd for $C_{17}H_{14}F_5NNaO_4S$ $[M+Na]^+$ 446.0461, found 446.0455.



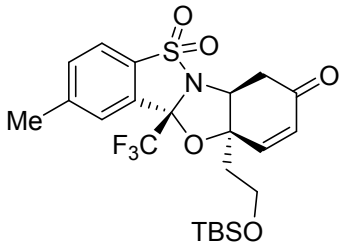
(6a*S*,10a*R*,11a*S*)-10a-ethyl-2-methyl-11a-(trifluoromethyl)-6a,10a-dihydro-11a*H*-benzo[*d*]benzo[4,5]isothiazolo[3,2-*b*]oxazol-8(7*H*)-one 5,5-dioxide (**3m**)

The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid in 87% yield. m.p. 145-146°C; $[\alpha]_D^{20}$ 82.1 (*c* = 1.0, EtOAc, 92%*ee*); HPLC: Daicel Chiralpak OD-H, hexane: 2-propanol = 70: 30, flow rate = 0.5 mL/min, T = 30 °C, UV = 254 nm, t_R = 11.3 min (minor), t_R = 12.5 min (major). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69-7.67 (d, *J* = 8.0 Hz, 1H), 7.55-7.51 (m, 2H), 6.70-6.68 (d, *J* = 8.0 Hz, 1H), 6.18-6.16 (d, *J* = 8.0 Hz, 1H), 4.32- 4.30 (t, *J* = 4.0 Hz, 1H), 3.18-3.13 (dd, *J* = 16.0, 4.0 Hz, 1H), 2.76-2.70 (dd, *J* = 16.0, 4.0 Hz, 1H), 2.53 (s, 3H), 1.80-1.69 (m, 2H), 0.91-0.87 (t, *J* = 8.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.9, 145.1, 144.9, 133.1, 132.7, 132.3, 128.7, 125.3-116.8 (q, *J* = 283.3 Hz), 124.9, 120.6, 95.6-94.6 (q, *J* = 33.3 Hz), 85.0, 62.7, 38.6, 29.4, 20.8, 6.8; ^{19}F NMR (376 MHz, $CDCl_3$) δ -78.1; HRMS (ESI) m/z calcd for $C_{17}H_{16}F_3NNaO_4S$ $[M+Na]^+$ 410.0650, found 410.0648.



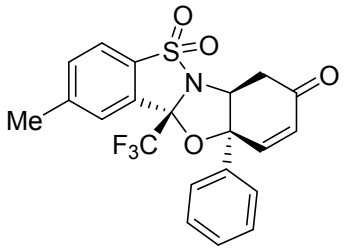
(6a*S*,10a*R*,11a*S*)-2-methyl-10a-propyl-11a-(trifluoromethyl)-6a,10a-dihydro-11a*H*-benzo[*d*]benzo[4,5]isothiazolo[3,2-*b*]oxazol-8(7*H*)-one 5,5-dioxide (**3n**)

The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid in 85% yield. m.p. 128-129°C; $[\alpha]_D^{20}$ 88.5 (*c* = 1.0, EtOAc, 87%*ee*); HPLC: Daicel Chiralpak OD-H, hexane: 2-propanol = 70: 30, flow rate = 0.7 mL/min, T = 30 °C, UV = 254 nm, t_R = 9.9 min (minor), t_R = 11.3 min (major). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69-7.67 (d, *J* = 8.0 Hz, 1H), 7.56-7.51 (m, 2H), 6.72-6.69 (d, *J* = 12.0 Hz, 1H), 6.17-6.14 (d, *J* = 12.0 Hz, 1H), 4.31-4.29 (t, *J* = 4.0 Hz, 1H), 3.19-3.14 (dd, *J* = 16.0, 4.0 Hz, 1H), 2.77-2.71 (dd, *J* = 16.0, 4.0 Hz, 1H), 2.54 (s, 3H), 1.77- 1.62 (m, 2H), 1.38-1.26 (m, 2H), 0.89-0.86 (t, *J* = 8.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.8, 145.4, 144.9, 133.1, 132.7, 132.4, 128.5, 125.3-116.8 (q, *J* = 283.3 Hz), 124.9, 120.5, 95.6-94.5 (q, *J* =36.7 Hz), 84.7, 63.3, 38.6, 38.5, 20.8, 15.9, 13.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ -78.0; HRMS (ESI) m/z calcd for $C_{18}H_{18}F_3NNaO_4S$ $[M+Na]^+$ 424.0806, found 424.0802.



(6a*S*,10a*R*,11a*S*)-10a-(2-((*tert*-butyldimethylsilyl)oxy)ethyl)-2-methyl-11a-(trifluoromethyl)-6a,10a-dihydro-11a*H*-benzo[*d*]benzo[4,5]isothiazolo[3,2-*b*]oxazol-8(7*H*)-one 5,5-dioxide (**3o**)

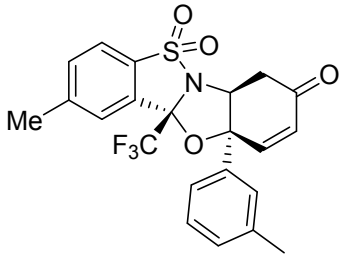
The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid in 85% yield. m.p. 103-104°C; $[\alpha]_D^{20}$ 71.4 (*c* =1.0, EtOAc, 85%*ee*); HPLC: Daicel Chiralpak OD-H, hexane: 2-propanol = 70: 30, flow rate = 0.7 mL/min, T = 30 °C, UV = 254 nm, t_R = 6.6 min (minor), t_R = 7.4 min (major). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68-7.66 (d, *J* = 8.0 Hz, 1H), 7.54-7.50 (m, 2H), 6.69-6.66 (d, *J* = 12.0 Hz, 1H), 6.16-6.13 (d, *J* = 12.0 Hz, 1H), 4.67-4.65 (t, *J* = 4.0 Hz, 1H), 3.64-3.60 (m, 2H), 3.19-3.14 (dd, *J* = 16.0, 4.0 Hz, 1H), 2.88-2.83 (dd, *J* = 16.0, 4.0 Hz, 1H), 2.53 (s, 3H), 1.97-1.91 (m, 2H), 0.79 (s, 9H), -0.07 (s, 3H), -0.16 (s,3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.2, 146.6, 145.8, 134.3, 133.7, 133.6, 129.4, 126.4-120.7 (q, *J* = 285.0 Hz), 125.8, 121.7, 96.0-95.3 (q, *J* = 35.0 Hz), 85.3, 64.2, 57.5, 39.4, 38.9, 25.7, 21.9, 18.0, -5.8, -5.9; ^{19}F NMR (376 MHz, $CDCl_3$) δ -77.9; HRMS (ESI) m/z calcd for $C_{23}H_{30}F_3NNaO_5SSi$ $[M+Na]^+$ 540.1464, found 540.1461.



(6a*S*,10a*S*,11a*R*)-2-methyl-10a-phenyl-11a-(trifluoromethyl)-6a,10a-dihydro-11a*H*-benzo[*d*]benzo[4,5]isothiazolo[3,2-*b*]oxazol-8(7*H*)-one 5,5-dioxide (**3p**)

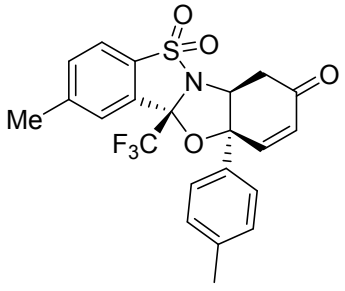
The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid in 93% yield. m.p. 177-178°C; $[\alpha]_D^{20}$

253.7 (*c* = 1.0, EtOAc, 90%*ee*); HPLC: Daicel Chiralpak OD-H, hexane: 2-propanol = 70: 30, flow rate = 0.5 mL/min, T = 30 °C, UV = 254 nm, t_R = 14.9 min (minor), t_R = 16.9 min (major). ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.02-8.00 (d, *J* = 8.0 Hz, 1H), 7.86 (s, 1H), 7.76-7.74 (d, *J* = 8.0 Hz, 1H), 7.41-7.33 (m, 5H), 6.98-6.95 (d, *J* = 12.0 Hz, 1H), 6.39-6.36 (d, *J* = 12.0 Hz, 1H), 4.32 (s, 1H), 3.14-3.08 (dd, *J* = 16.0, 4.0 Hz, 1H), 2.85-2.80 (dd, *J* = 16.0, 4.0 Hz, 1H), 2.56 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 194.3, 147.4, 145.6, 136.6, 135.1, 133.2, 133.1, 130.1, 129.8, 129.5, 126.2, 125.9, 123.9-121.1 (q, *J* = 280.0 Hz), 122.6, 96.4-96.0 (q, *J* = 40.0 Hz), 87.3, 68.2, 39.2, 21.7; ^{19}F NMR (376 MHz, DMSO-d_6) δ -77.0; HRMS (ESI) m/z calcd for $C_{21}H_{16}F_3NNaO_4S$ $[M+Na]^+$ 458.0650, found 458.0647.



(6a*S*,10a*S*,11a*S*)-2-methyl-10a-(*m*-tolyl)-11a-(trifluoromethyl)-6a,10a-dihydro-11a*H*-benzo[*d*]benzo[4,5]isothiazolo[3,2-*b*]oxazol-8(7*H*)-one 5,5-dioxide (**3q**)

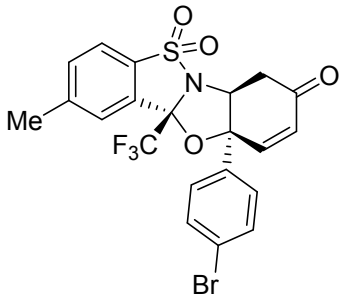
The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid in 82% yield. m.p. 171-172°C; $[\alpha]_D^{20}$ 207.2 (*c* = 1.0, EtOAc, 83%*ee*); HPLC: Daicel Chiralpak OD-H, hexane: 2-propanol = 70: 30, flow rate = 0.5 mL/min, T = 30 °C, UV = 254 nm, t_R = 16.2 min (minor), t_R = 17.4 min (major). ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.02-8.00 (d, *J* = 8.0 Hz, 1H), 7.89 (s, 1H), 7.76-7.74 (d, *J* = 8.0 Hz, 1H), 7.29-7.14 (m, 4H), 6.96-6.93 (d, *J* = 12.0 Hz, 1H), 6.38-6.35 (d, *J* = 12.0 Hz, 1H), 4.38 (s, 1H), 3.18-3.13 (dd, *J* = 16.0, 4.0 Hz, 1H), 2.86-2.81 (dd, *J* = 16.0, 4.0 Hz, 1H), 2.57 (s, 3H), 2.25 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 194.4, 147.3, 145.6, 138.8, 136.7, 135.1, 133.2, 133.1, 130.3, 129.9, 129.4, 126.8-118.2 (q, *J* = 286.7 Hz), 126.6, 126.3, 122.8, 122.6, 96.7-95.7 (q, *J* = 33.3 Hz), 87.2, 67.9, 39.4, 21.7, 21.4; ^{19}F NMR (376 MHz, DMSO-d_6) δ -77.1; HRMS (ESI) m/z calcd for $C_{22}H_{18}F_3NNaO_4S$ $[M+Na]^+$ 472.0806, found 472.0804.



(6a*S*,10a*S*,11a*S*)-2-methyl-10a-(*p*-tolyl)-11a-(trifluoromethyl)-6a,10a-dihydro-11a*H*-benzo[*d*]benzo[4,5]isothiazolo[3,2-*b*]oxazol-8(7*H*)-one 5,5-dioxide (**3r**)

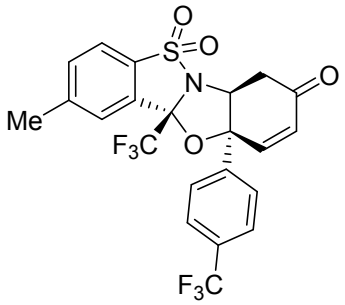
The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid in 91% yield. m.p. 168-169°C; $[\alpha]_D^{20}$ 221.2 (*c* = 1.0, EtOAc, 89%*ee*); HPLC: Daicel Chiralpak OD-H, hexane: 2-propanol = 70: 30, flow rate = 0.7 mL/min, T = 30 °C, UV = 254 nm, t_R = 9.5 min (minor), t_R = 10.8 min (major). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70-7.57 (m, 3H), 7.16-7.10 (m, 4H), 6.80-6.77 (d, *J* = 10.4 Hz, 1H), 6.39-6.37

(d, J = 10.4 Hz, 1H), 4.27 (m, 1H), 3.21- 3.15 (dd, J = 18.0, 4.4 Hz, 1H), 2.76-2.68 (dd, J = 18.0, 4.4 Hz, 1H), 2.60 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.7, 146.1, 145.4, 139.7, 134.0, 133.9, 133.6, 133.1, 130.0, 129.8, 126.4-117.8 (q, J = 285.5 Hz), 125.9, 125.3, 121.9, 96.5-95.8 (q, J = 35.1 Hz), 87.0, 68.4, 38.0, 22.0, 21.1; ^{19}F NMR (376 MHz, $CDCl_3$) δ -77.6; HRMS (ESI) m/z calcd for $C_{22}H_{18}F_3NNaO_4S$ $[M+Na]^+$ 472.0806, found 472.0804.



(6a*S*,10a*S*,11a*S*)-10a-(4-bromophenyl)-2-methyl-11a-(trifluoromethyl)-6a,10a-dihydro-11a*H*-benzo[*d*]benzo[4,5]isothiazolo[3,2-b]oxazol-8(7*H*)-one 5,5-dioxide (**3s**)

The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid in 86% yield. m.p. 187-188°C; $[\alpha]_D^{20}$ 208.6 (*c* = 1.0, EtOAc, 83%*ee*); HPLC: Daicel Chiralpak OJ-H, hexane: 2-propanol = 70: 30, flow rate = 0.5 mL/min, T = 30 °C, UV = 254 nm, t_R = 18.9 min (minor), t_R = 27.2 min (major). ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.02-8.00 (d, J = 8.0 Hz, 1H), 7.85 (s, 1H), 7.76-7.74 (d, J = 8.0 Hz, 1H), 7.58-7.56 (d, J = 8.0 Hz, 2H), 7.31-7.29 (d, J = 8.0 Hz, 2H), 6.96-6.93 (d, J = 12.0 Hz, 1H), 6.39-6.36 (d, J = 8.0 Hz, 1H), 4.39 (s, 1H), 3.15-3.10 (dd, J = 16.0, 4.0 Hz, 1H), 2.83-2.78 (dd, J = 16.0, 4.0 Hz, 1H), 2.56 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 194.3, 147.4, 145.0, 136.0, 135.2, 133.1, 133.0, 132.3, 130.3, 128.3, 126.7-121.0 (q, J = 285.0 Hz), 126.2, 123.2, 122.6, 96.7-95.7 (q, J = 33.3 Hz), 86.9, 67.7, 39.3, 21.8; ^{19}F NMR (376 MHz, DMSO-d_6) δ -77.6; HRMS (ESI) m/z calcd for $C_{21}H_{15}BrF_3NNaO_4S$ $[M+Na]^+$ 535.9755, found 535.9757.



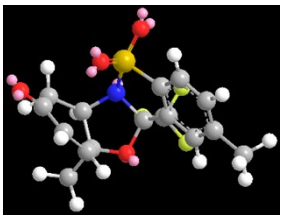
(6a*S*,10a*S*,11a*S*)-2-methyl-11a-(trifluoromethyl)-10a-(4-(trifluoromethyl)phenyl)-6a,10a-dihydro-11a*H*-benzo[*d*]benzo[4,5]isothiazolo[3,2-*b*]oxazol-8(7*H*)-one 5,5-dioxide (**3t**)

The title compound was prepared according to the general working procedure and purified by column chromatography to give the product as a white solid in 90% yield. m.p. 209-210°C; $[\alpha]_D^{20}$ 172.2 (*c* = 1.0, EtOAc, 80%*ee*); HPLC: Daicel Chiralpak OD-H, hexane: 2-propanol = 70: 30, flow rate = 0.5 mL/min, T = 30 °C, UV = 254 nm, t_R = 14.2 min (minor), t_R = 17.7 min (major). ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.01-7.99 (d, J = 8.0 Hz, 1H), 7.87 (s, 1H), 7.76-7.72 (m, 3H), 7.61-7.58 (d, J = 12.0 Hz, 2H), 6.99-6.96 (d, J = 12.0 Hz, 1H), 6.42-6.39 (d, J = 12.0 Hz, 1H), 4.46 (s, 1H), 3.20-3.15 (dd, J = 16.0, 4.0 Hz, 1H), 2.86-2.81 (dd, J = 16.0, 4.0 Hz, 1H), 2.57 (s, 3H); ^{13}C NMR (100

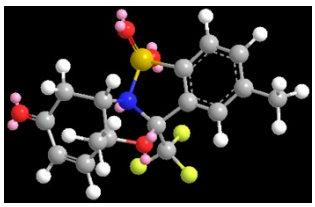
MHz, DMSO-d_6) δ 194.2, 147.4, 144.6, 141.2, 135.1, 133.1, 132.9, 130.6, 130.3-129.7 (q, J = 30 Hz), 128.3-120.2 (q, J = 270.0 Hz), 127.1, 126.33, 126.29, 126.25, 122.6, 96.9-95.8 (q, J = 35.0 Hz), 86.8, 67.6, 39.3, 21.7; ^{19}F NMR (376 MHz, DMSO-d_6) δ -61.5, -77.2; HRMS (ESI) m/z calcd for $C_{22}H_{15}F_6NNaO_4S$ $[M+Na]^+$ 526.0524, found 526.0532.

DFT calculations of diastereomers

Diastereoisomers **4** and **5** were chosen for DFT calculations. The relative Gibbs energies were finally reported in kJ/mol, and computed at the SMD ($CHCl_3$)/B3LYP/6-31g(d), showing that diastereoisomer **5** have a much lower energy than that of **4**. This could explain the excellent diastereoselectivity of the product **3**.



Diastereoisomer **4**



Diastereoisomer **5**

	Single point	Gibbs energy	Hatree	Kj/mol
4	-1671.381922	0.234535	-1671.147387	
5	-1671.393333	0.234202	-1671.159131	
4-5	0.00867	0.002879	0.0117442	7.369603

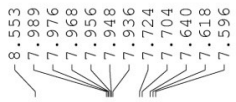
References

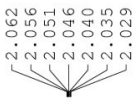
1.S. Zhang, L. Li, Y. H, Y. Li, Y. Yang, Z. Zha, Z. Wang, *Org. Lett.*, 2015, **17**, 5036.

2. K. L. Kimmel, M. T. Robak and J. A. Ellman, *J. Am. Chem. Soc*., 2009, **131**, 8754.

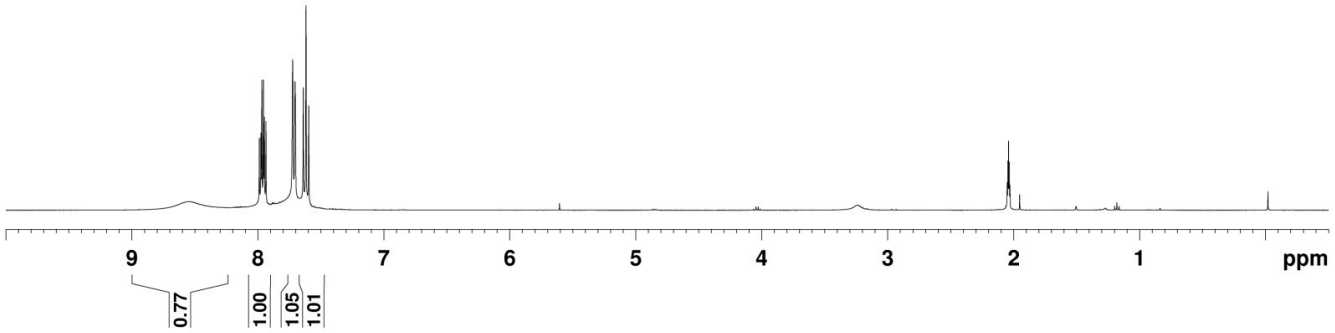
3. a) J. Zhang, J. Wu, Z. Yin, H. Zeng, K. Khanna, C. H, S. Zheng, *Org. Biomol. Chem.*, 2013, **11**, 2939. b) M. T. Corbett, J. S. Johnson, *Chem. Sci.*, 2013, **4**, 2828.

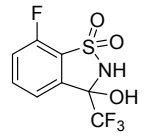
Copies of ^{1}H NMR, ^{19}F NMR and ^{13}C NMR Spectra



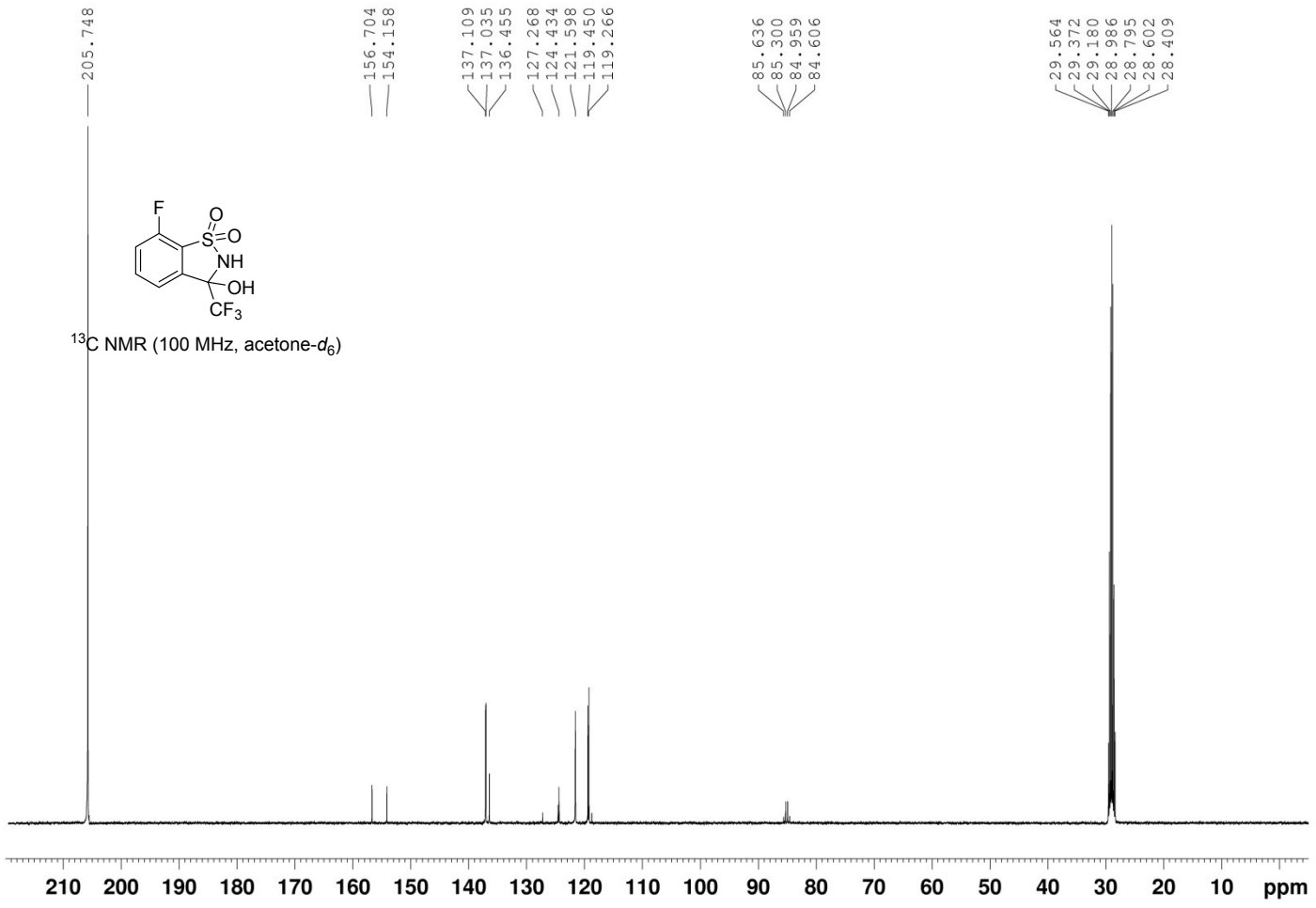


^{1}H NMR (400 MHz, acetone-d_6)

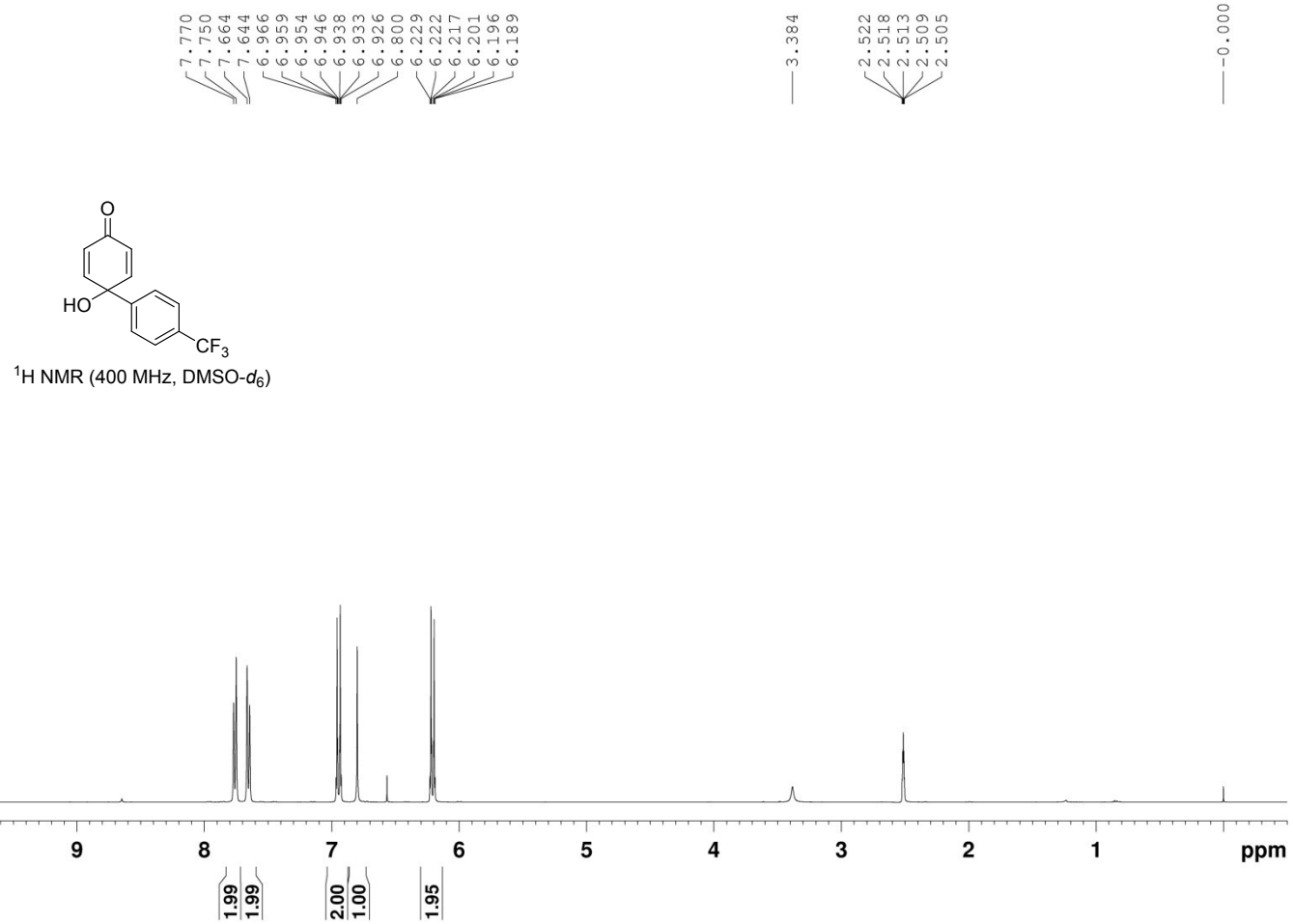




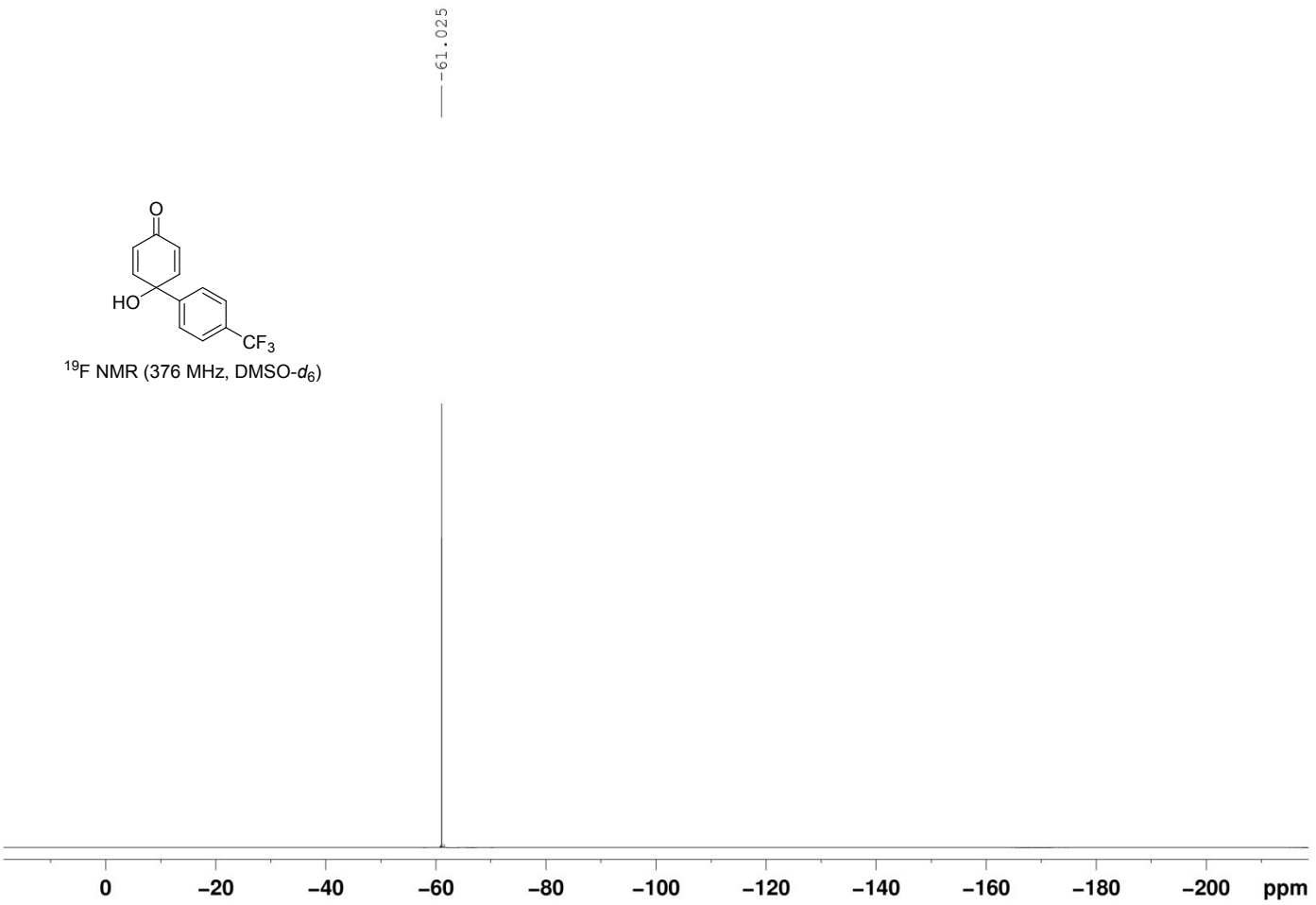
^{19}F NMR (376 MHz, acetone-d_6)



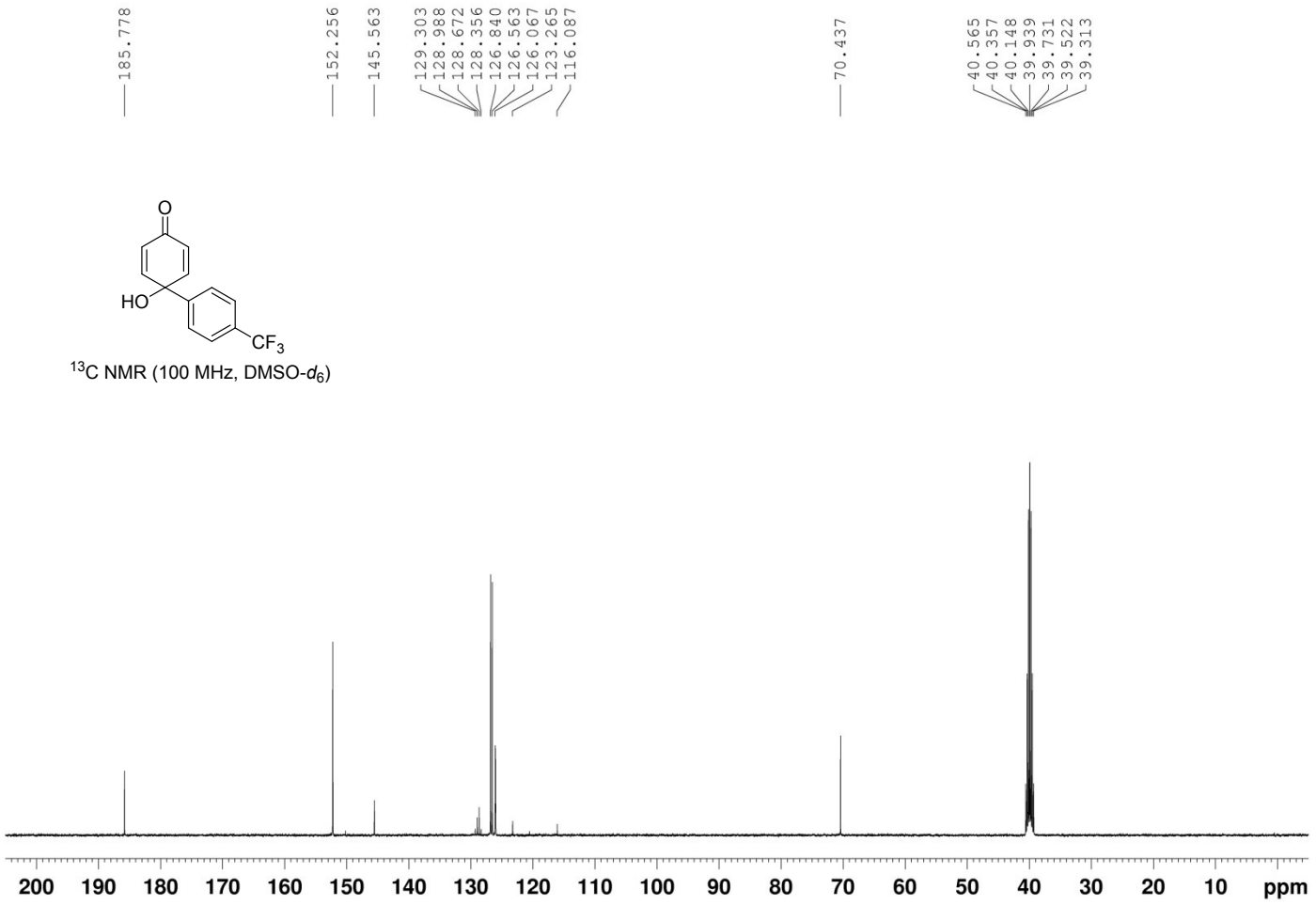
^{13}C NMR (100 MHz, acetone-d_6)



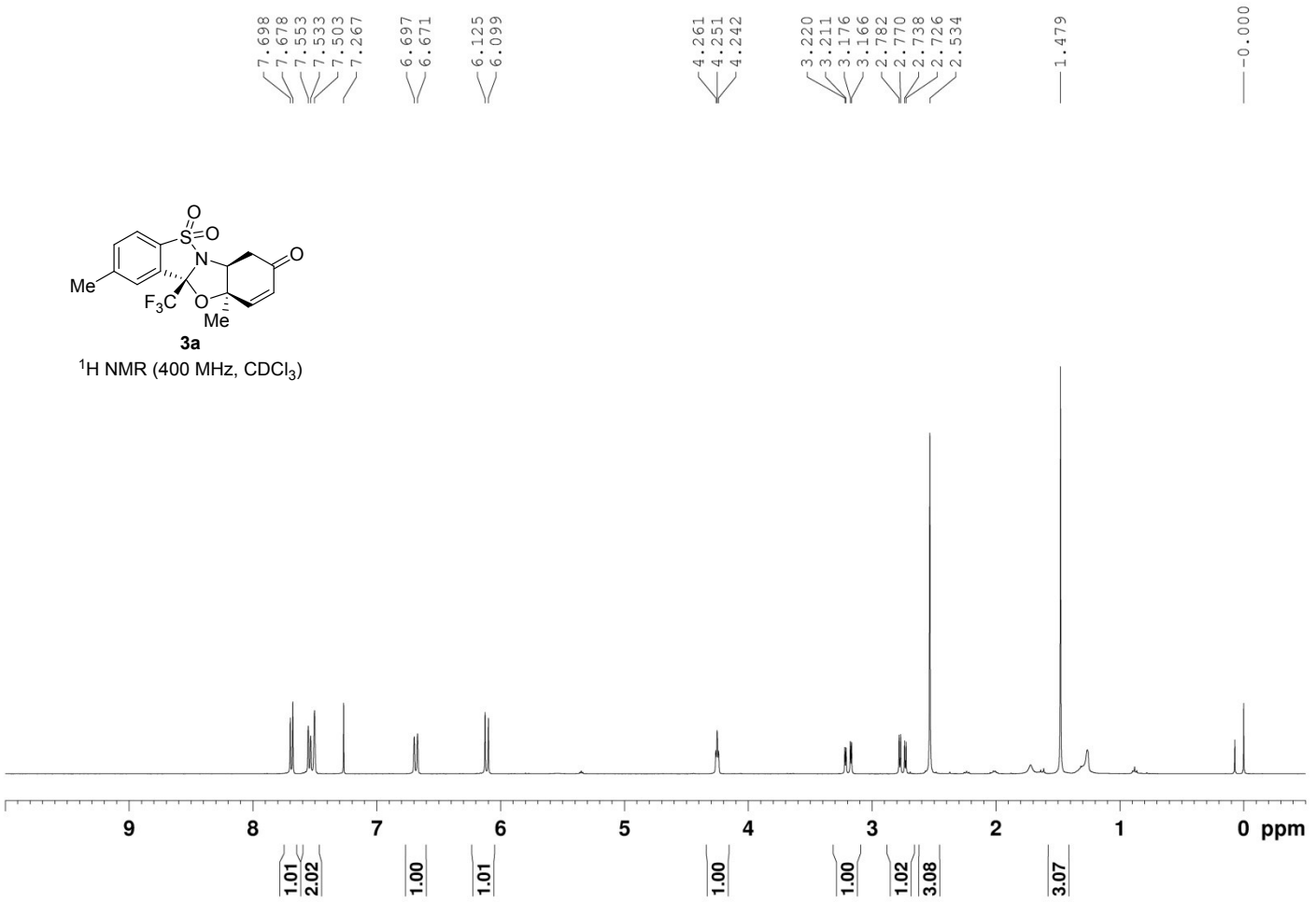
^{1}H NMR (400 MHz, DMSO-d_6)

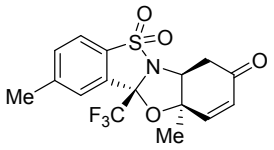


^{19}F NMR (376 MHz, DMSO-d_6)



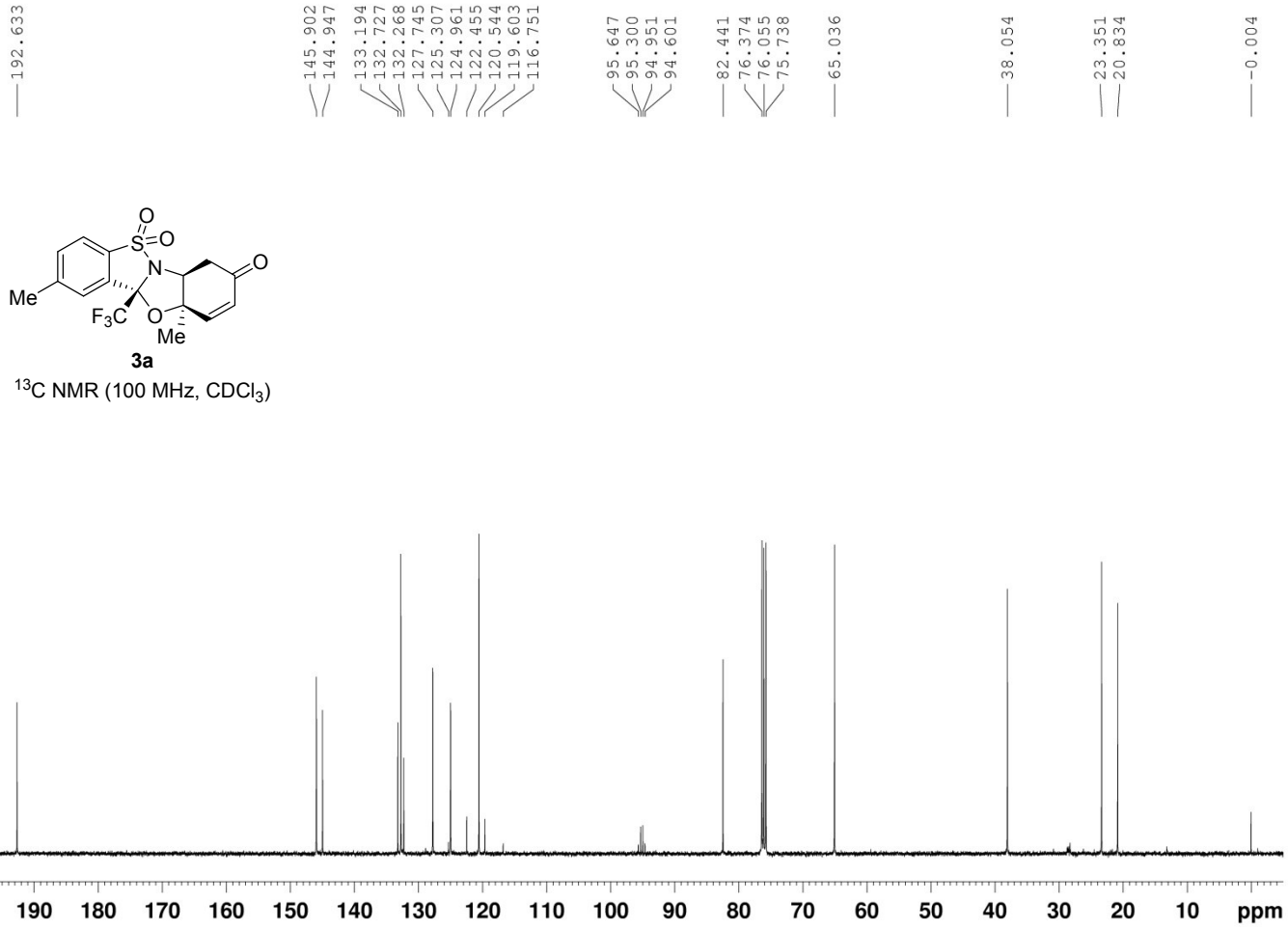
^{13}C NMR (100 MHz, DMSO-d_6)

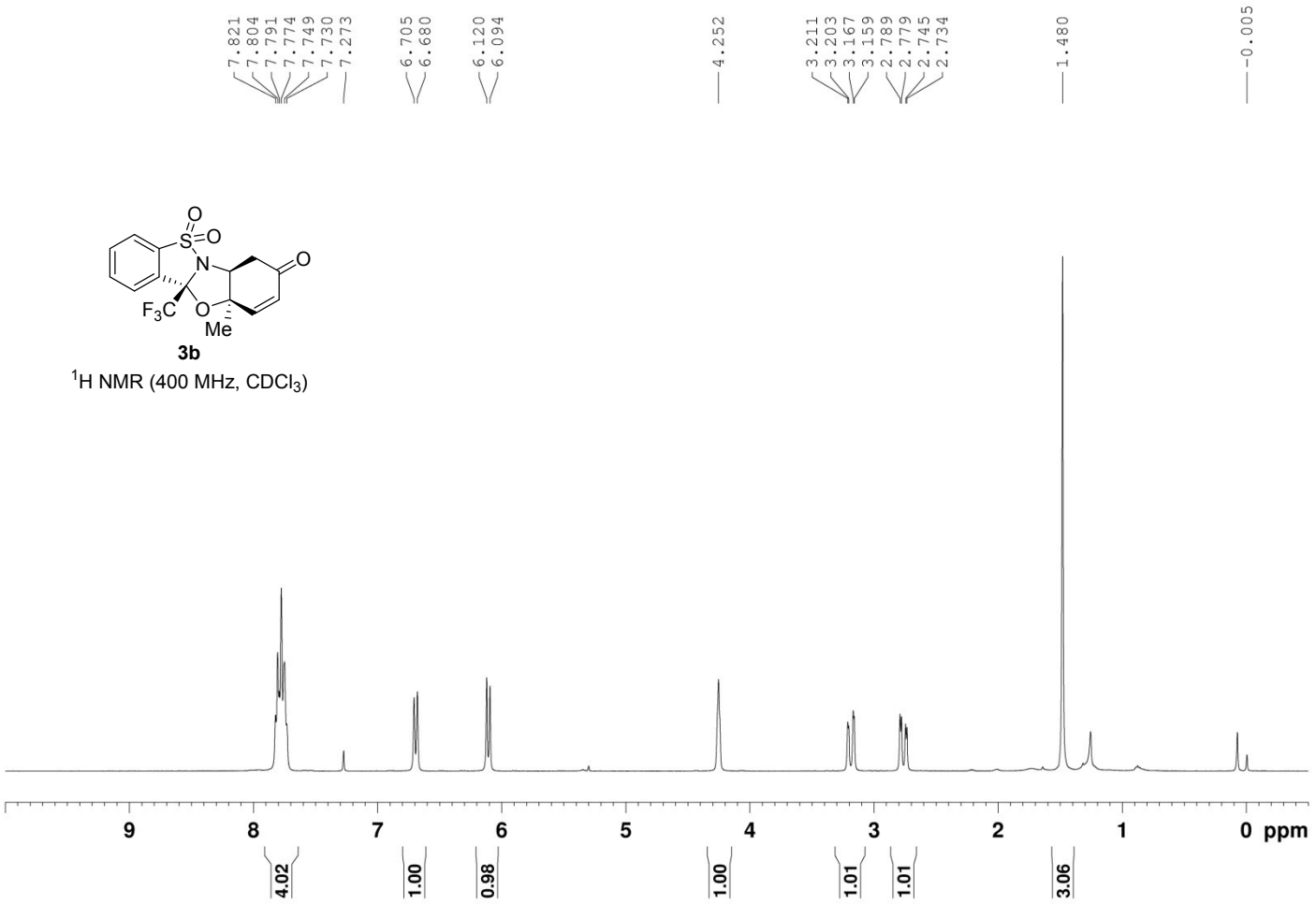

7.698
7.678
7.553
7.533
7.503
7.267
6.697
6.671
6.125
6.099
4.261
4.251
4.242
3.220
3.211
3.176
3.166
2.782
2.770
2.738
2.726
2.534
1.479
0.000
O
S
O
N
O
Me
F3C
O
Me
3a
1H NMR (400 MHz, CDCl3)
9
8
7
6
5
4
3
2
1
0 ppm
1.01
2.02
1.00
1.01
1.00
1.00
1.02
3.08
3.07



3a

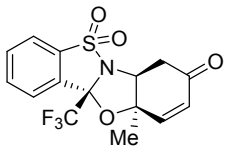
^{19}F NMR (376 MHz, $CDCl_3$)


192.633
145.902
144.947
133.194
132.727
132.268
127.745
125.307
124.961
122.455
120.544
119.603
116.751
95.647
95.300
94.951
94.601
82.441
76.374
76.055
75.738
65.036
38.054
23.351
20.834
0.004
Me
F3C
O
Me
3a
13C NMR (100 MHz, CDCl3)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm



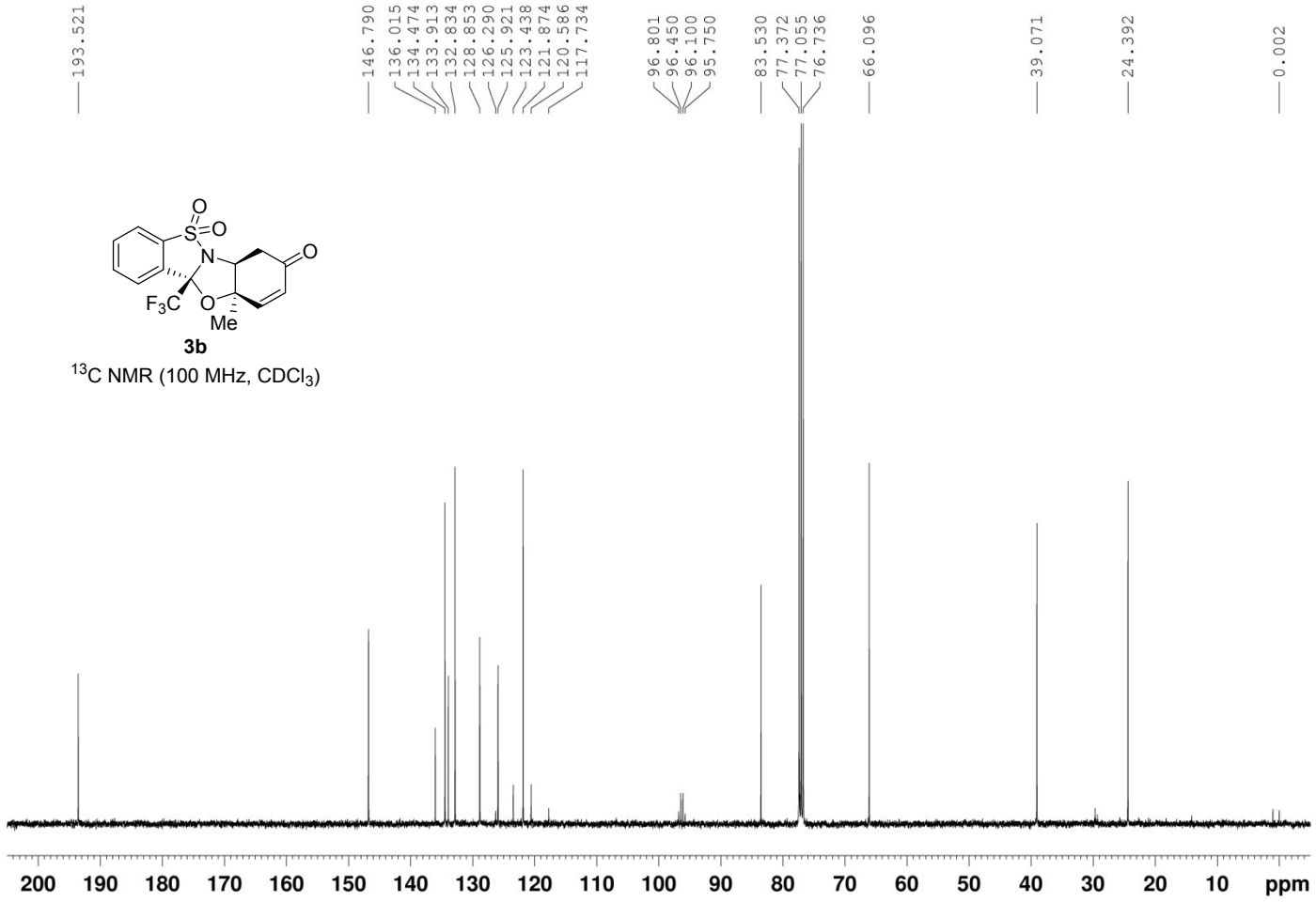
3b

^{1}H NMR (400 MHz, $CDCl_3$)



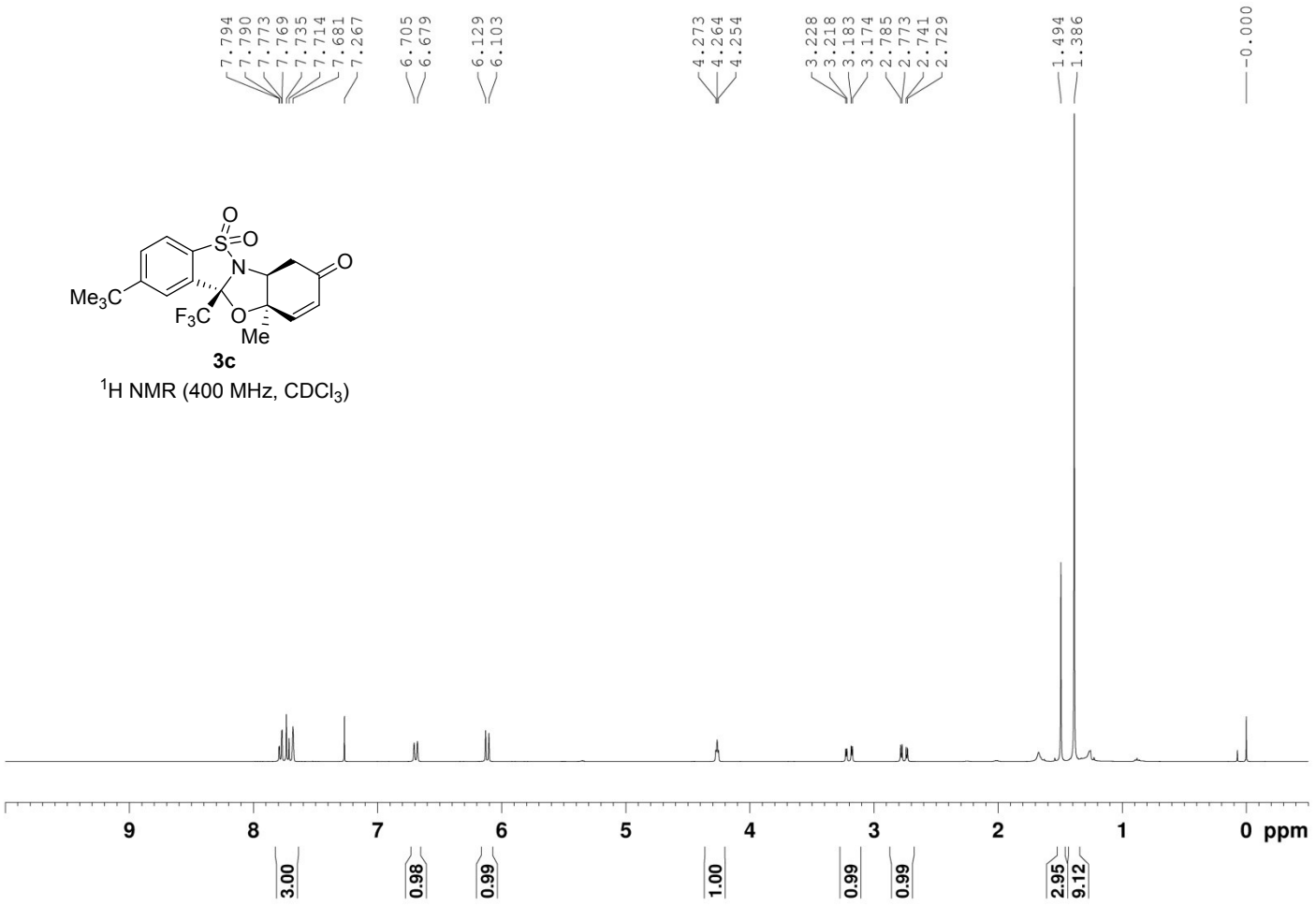
3b

^{19}F NMR (376 MHz, $CDCl_3$)



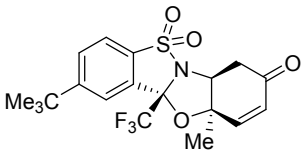
3b

^{13}C NMR (100 MHz, $CDCl_3$)



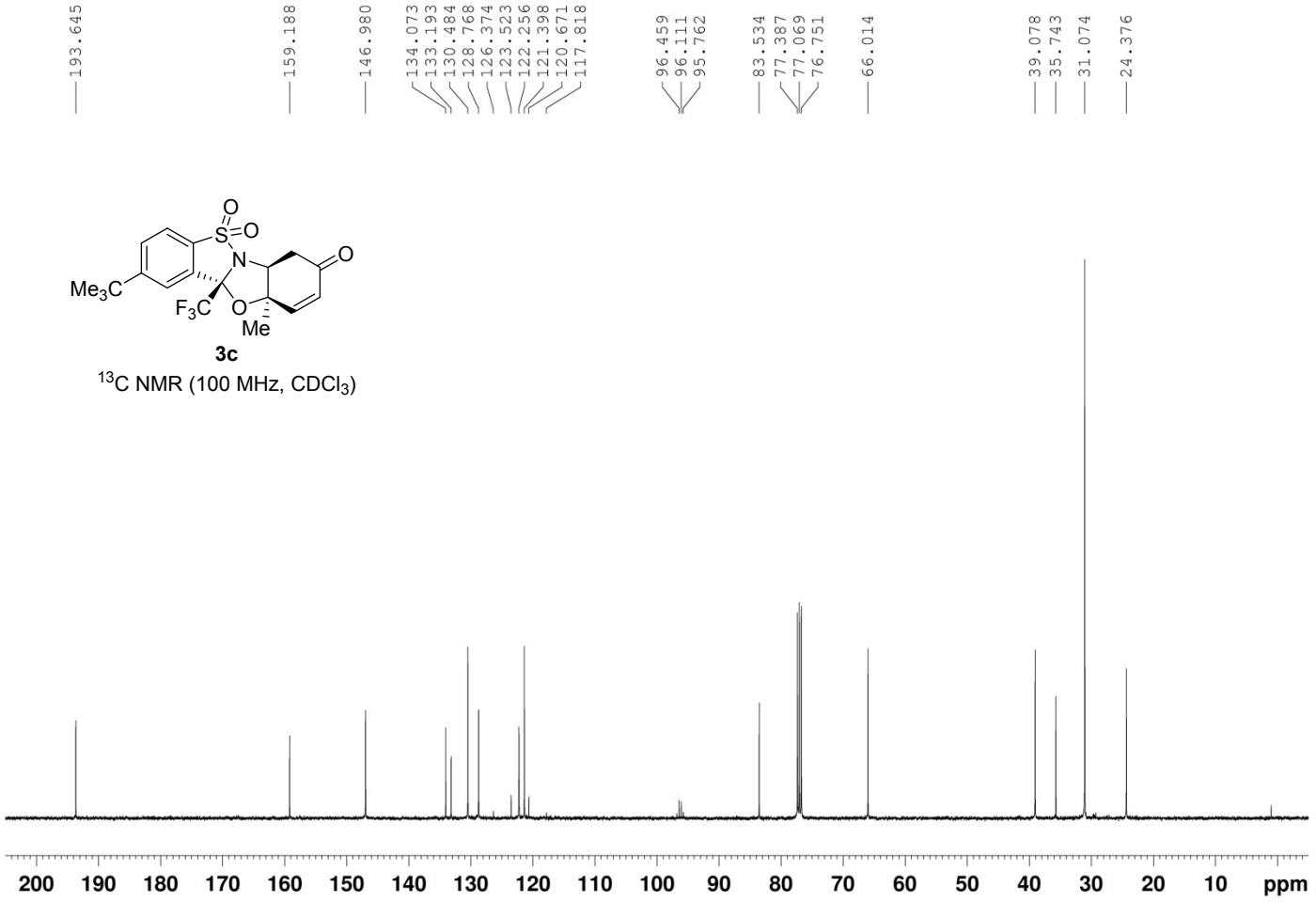
3c

^{1}H NMR (400 MHz, $CDCl_3$)



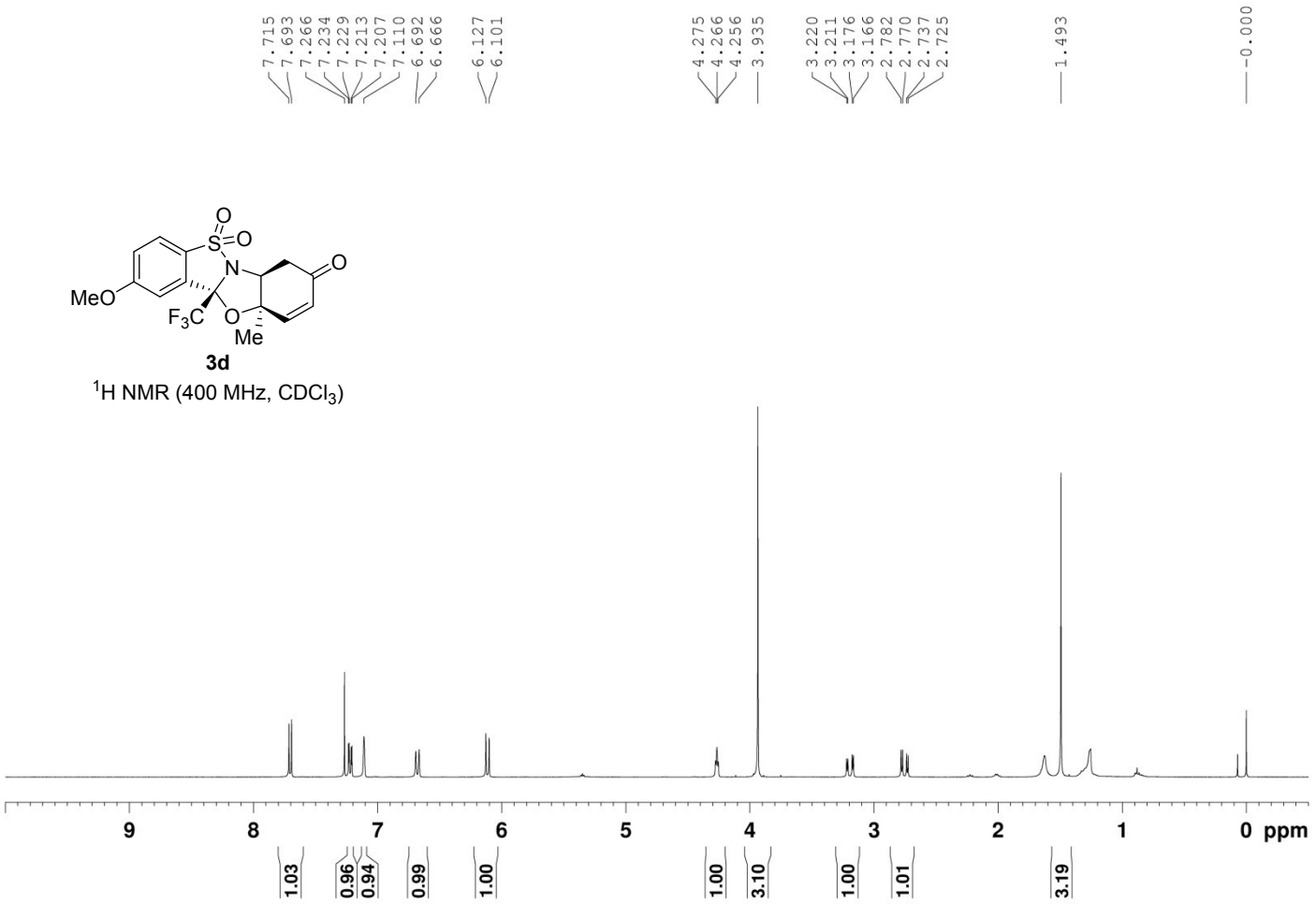
3c

^{19}F NMR (376 MHz, $CDCl_3$)



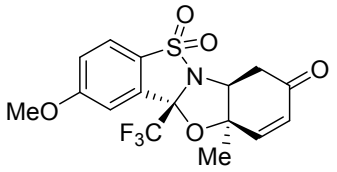
3c

^{13}C NMR (100 MHz, $CDCl_3$)



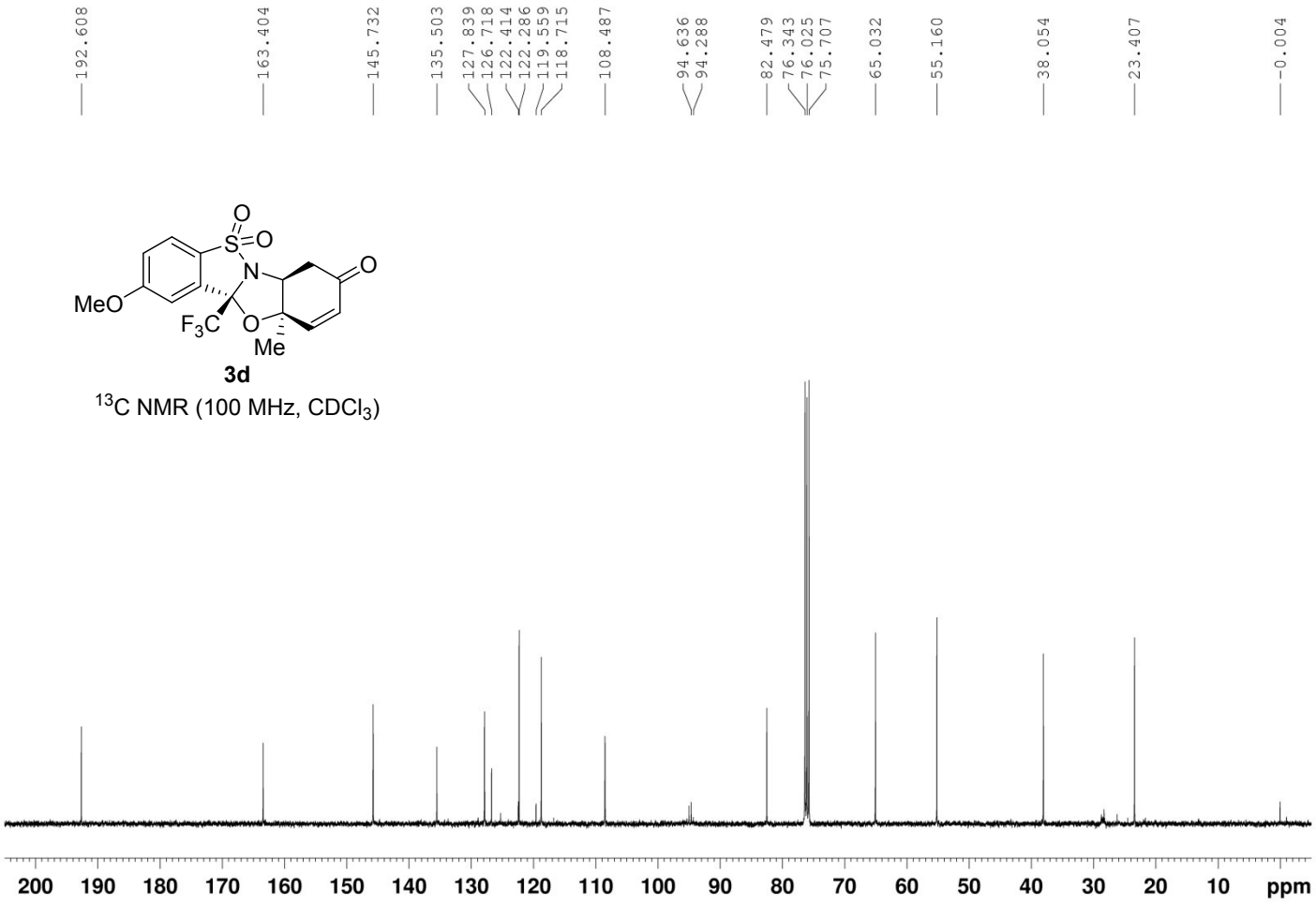
3d

^{1}H NMR (400 MHz, $CDCl_3$)



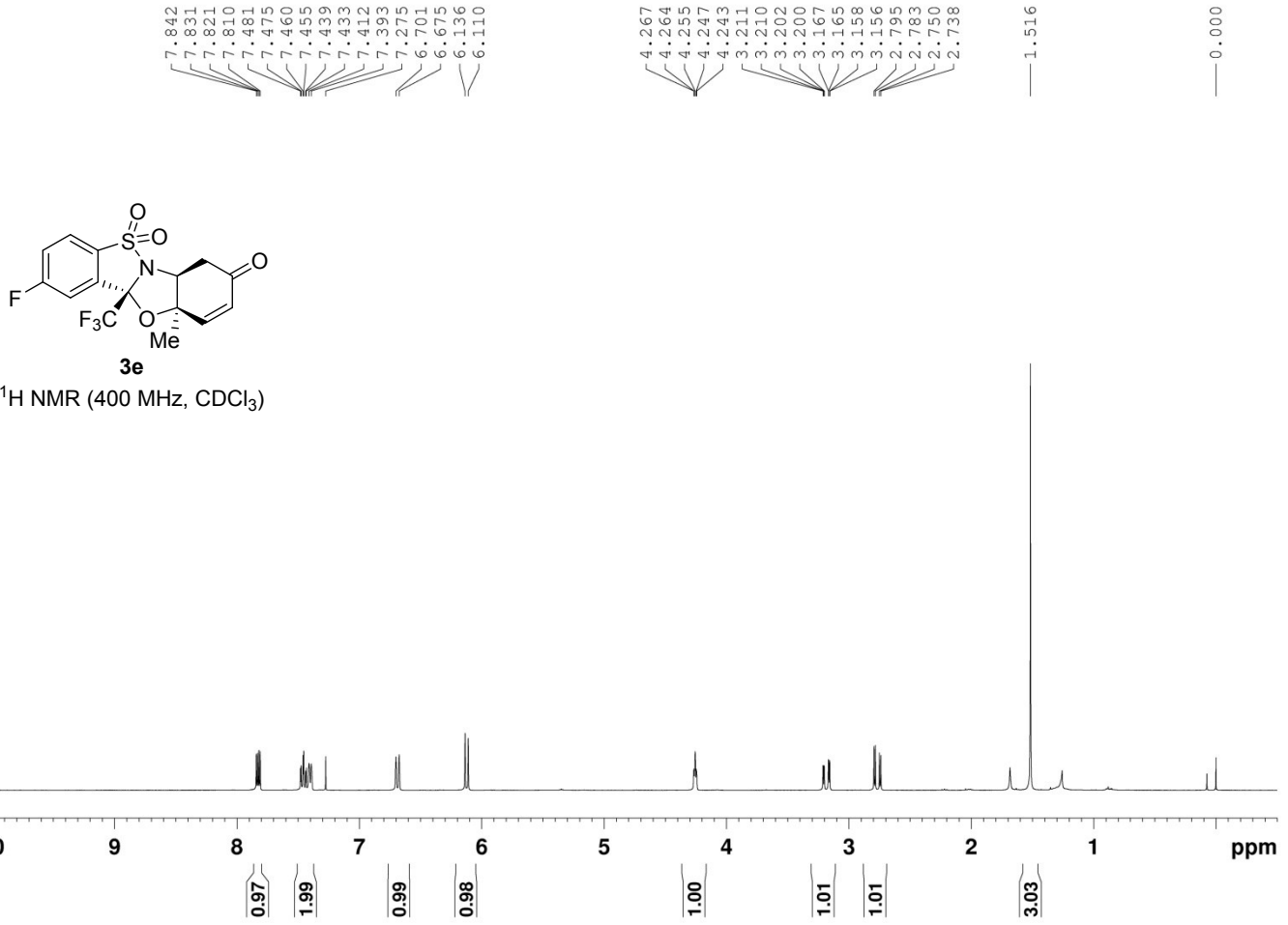
3d

^{19}F NMR (376 MHz, $CDCl_3$)



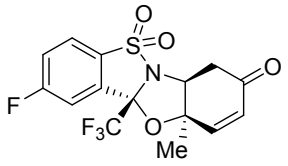
3d

^{13}C NMR (100 MHz, $CDCl_3$)



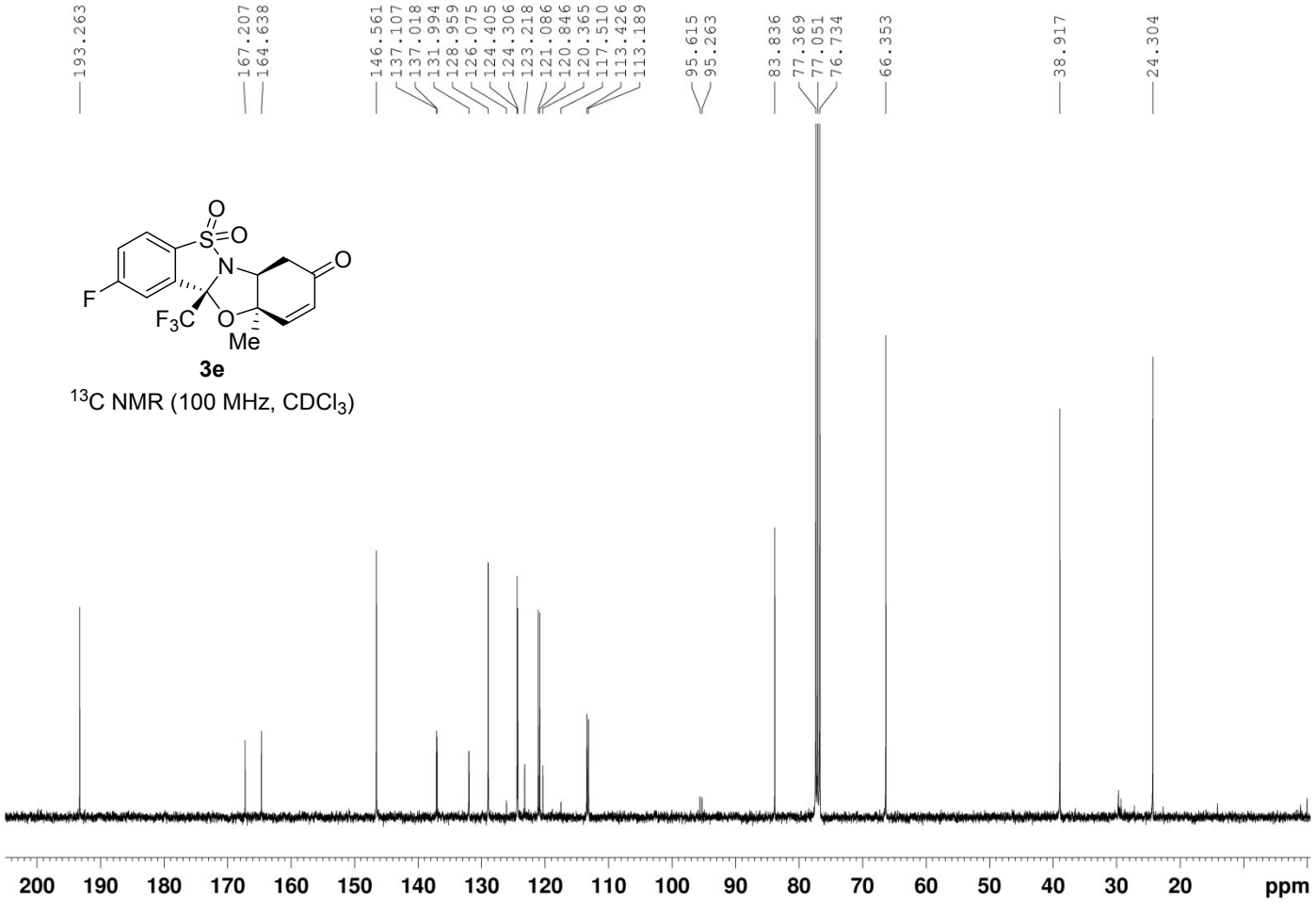
3e

^{1}H NMR (400 MHz, $CDCl_3$)



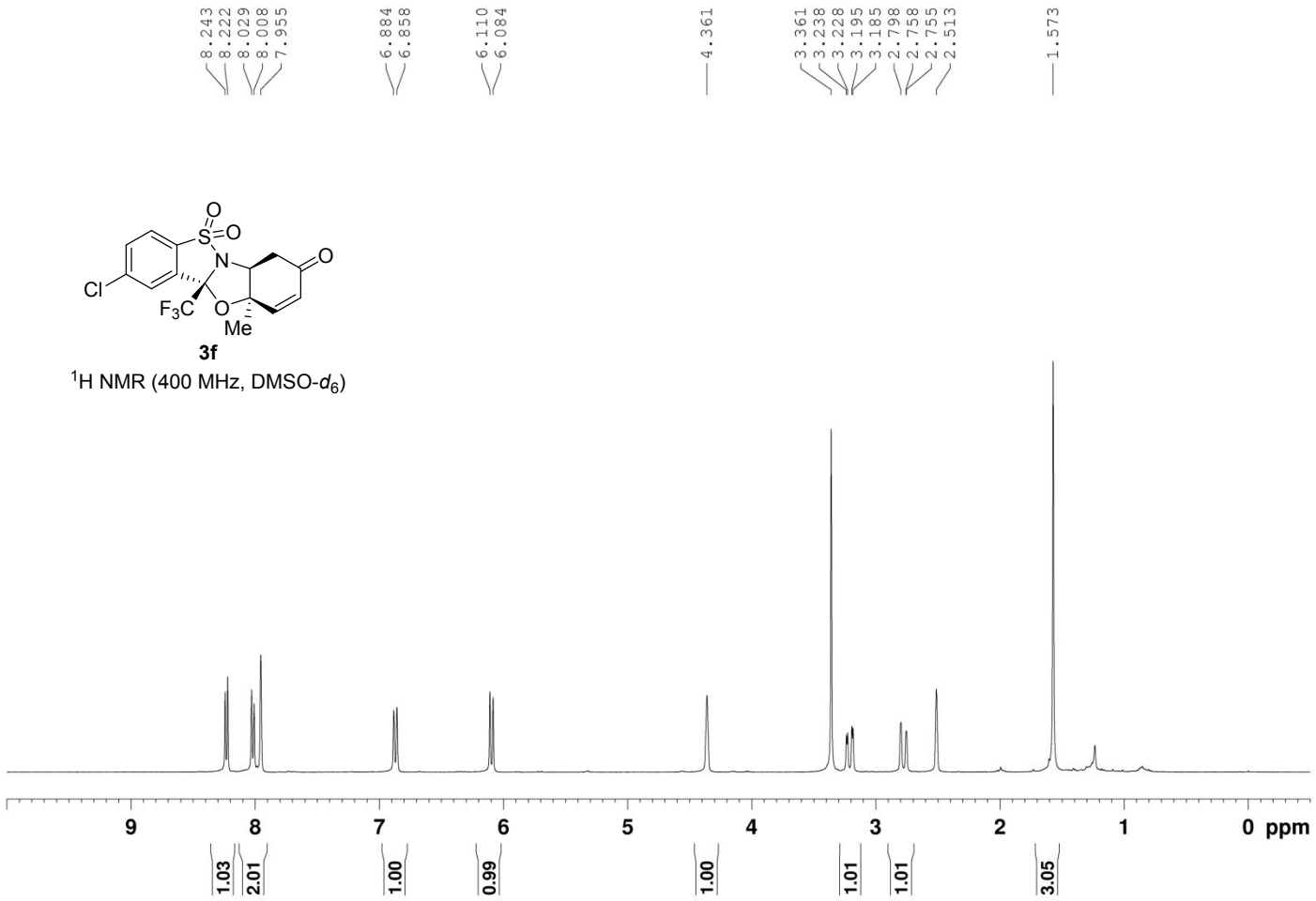
3e

^{19}F NMR (376 MHz, $CDCl_3$)



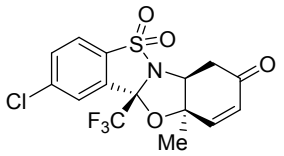
3e

^{13}C NMR (100 MHz, $CDCl_3$)



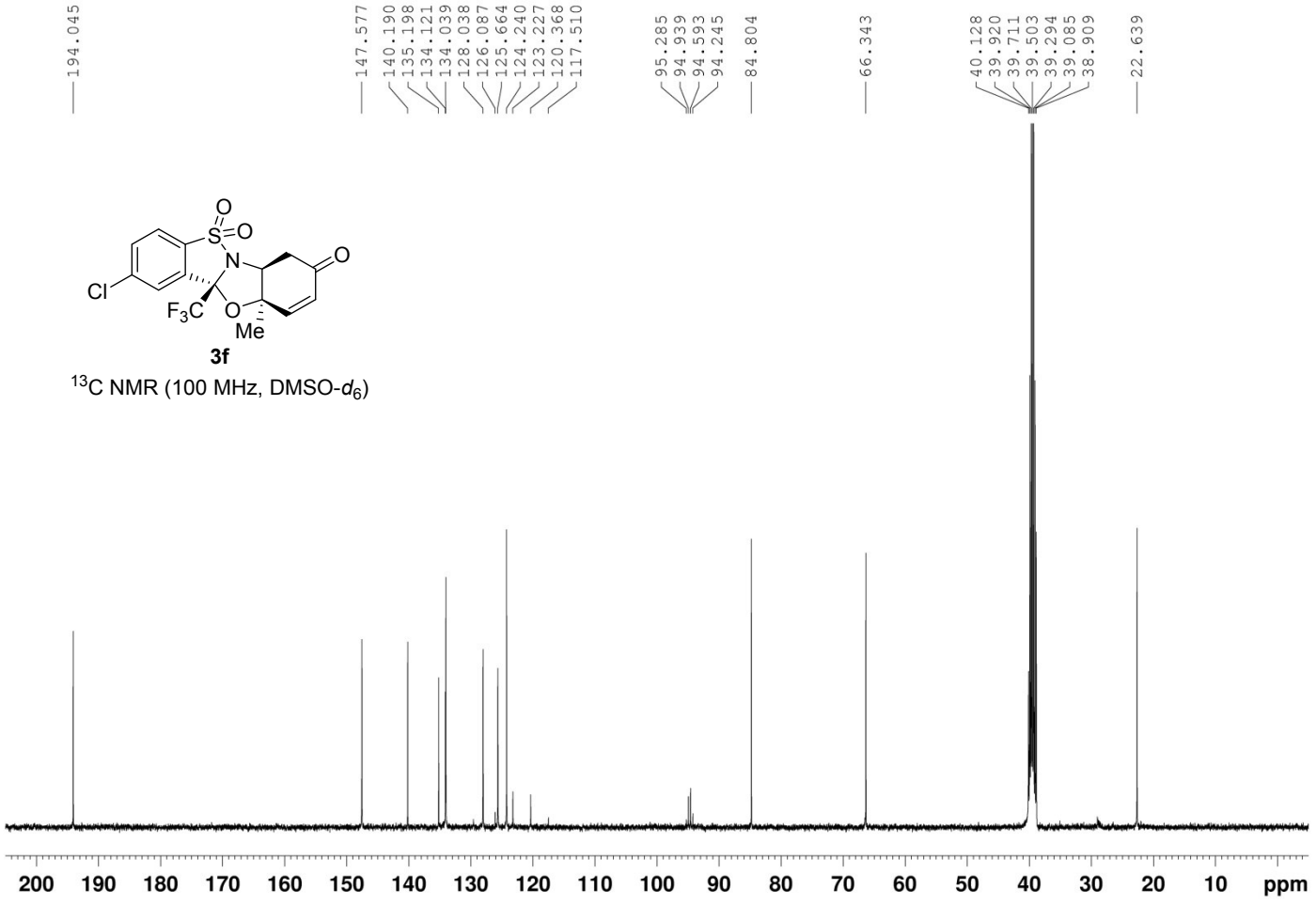
3f

^{1}H NMR (400 MHz, DMSO-d_6)



3f

^{19}F NMR (376 MHz, DMSO-d_6)



3f

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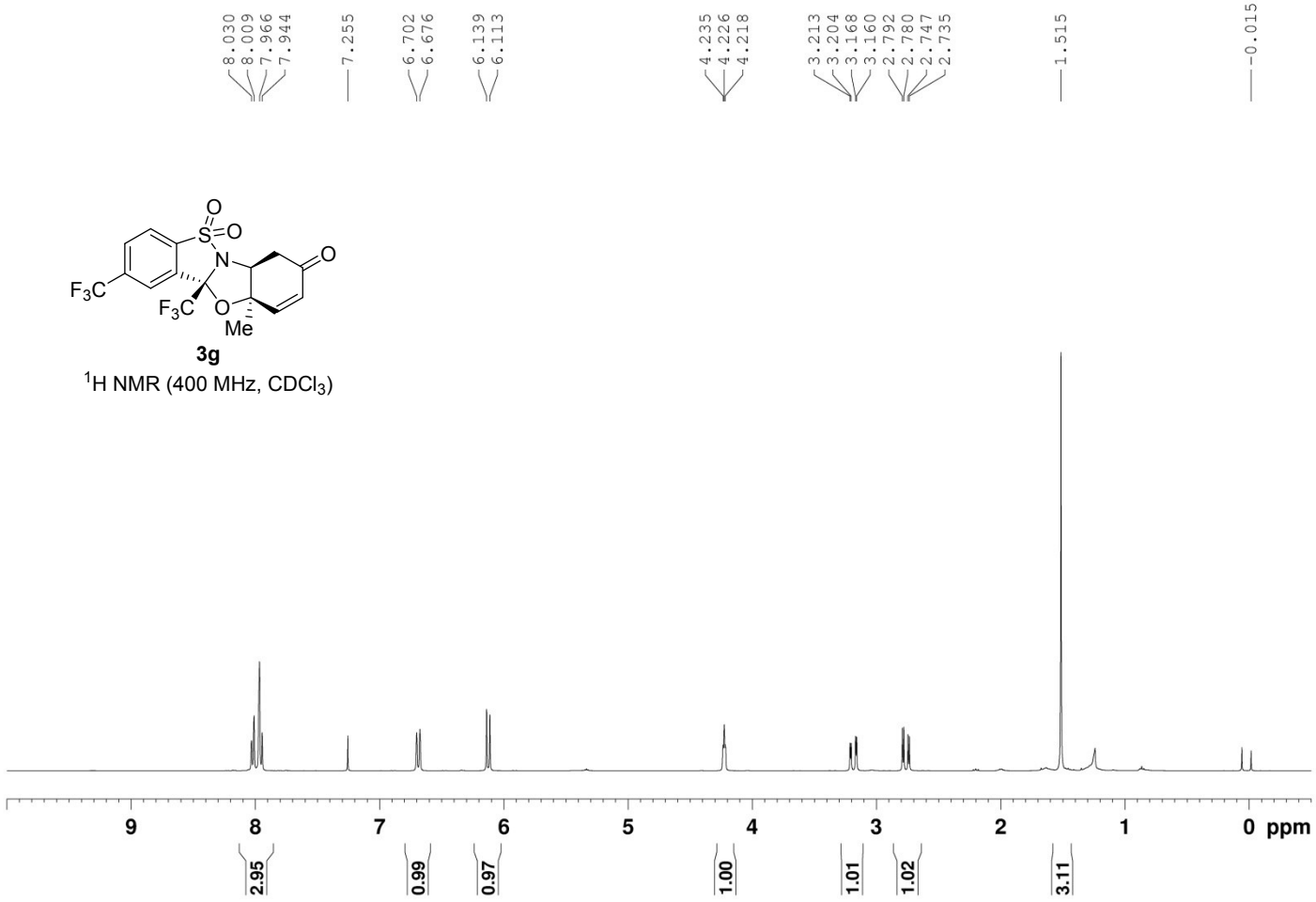

3g

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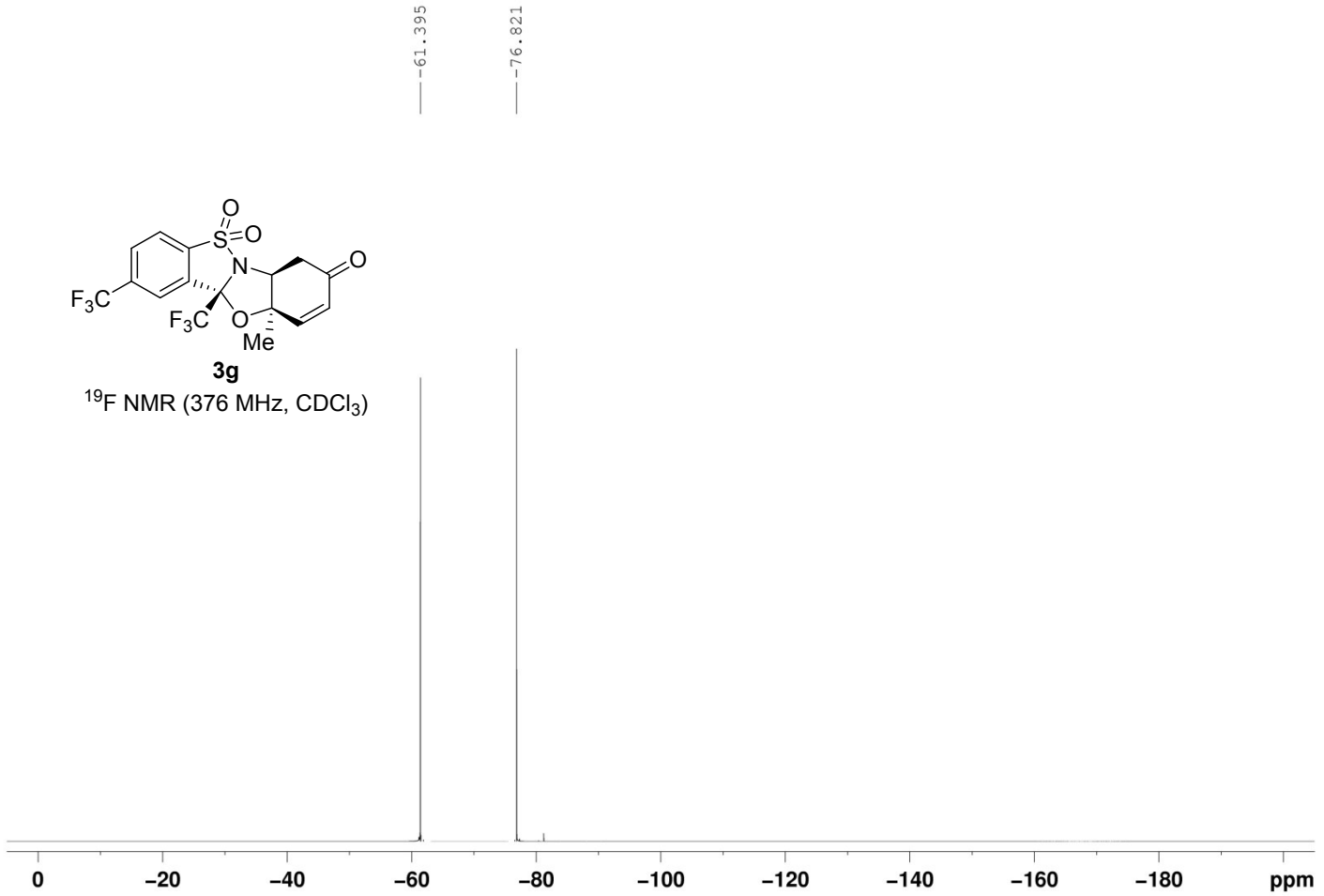

3g

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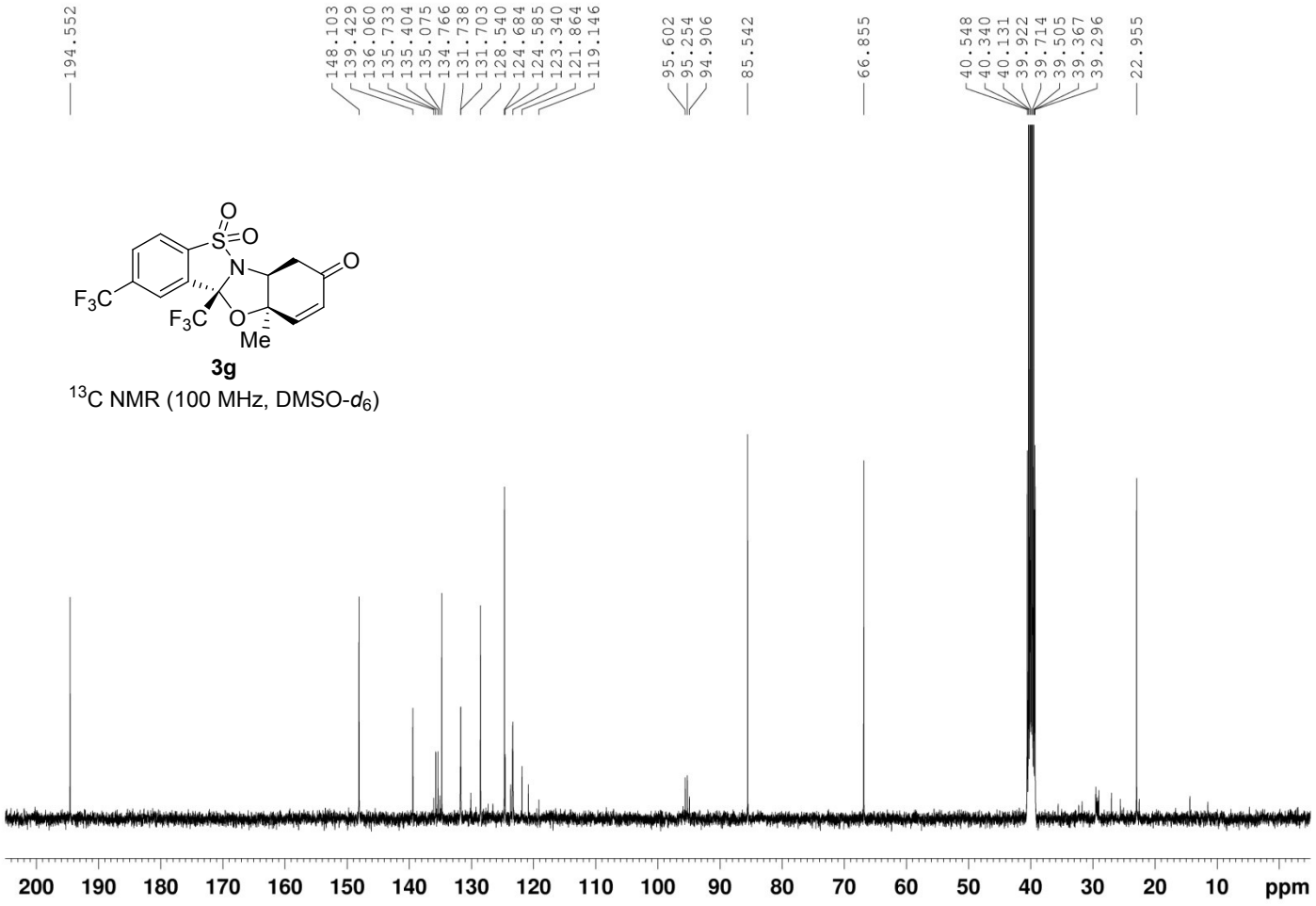

3g

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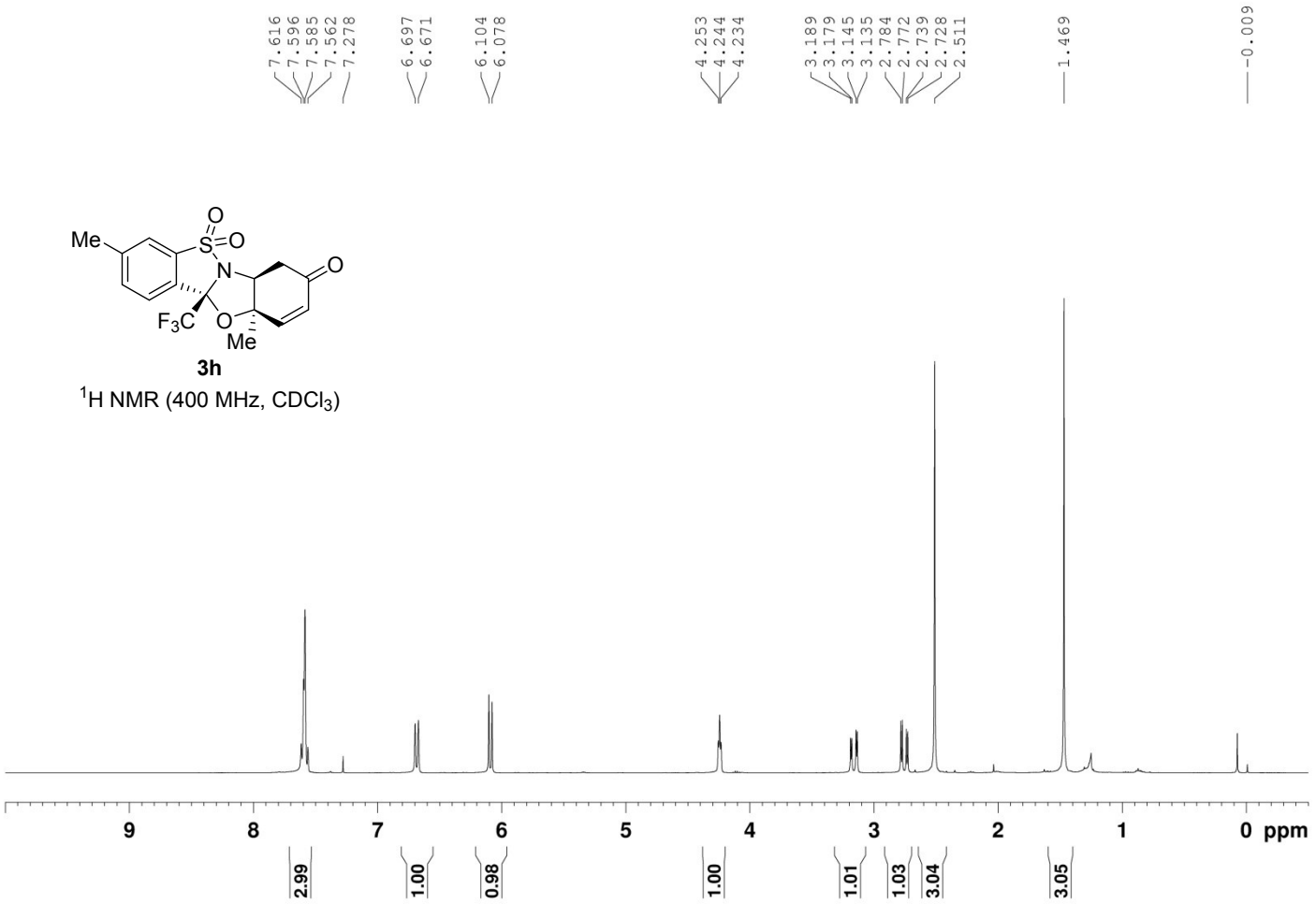
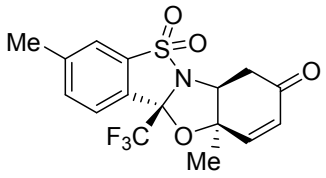


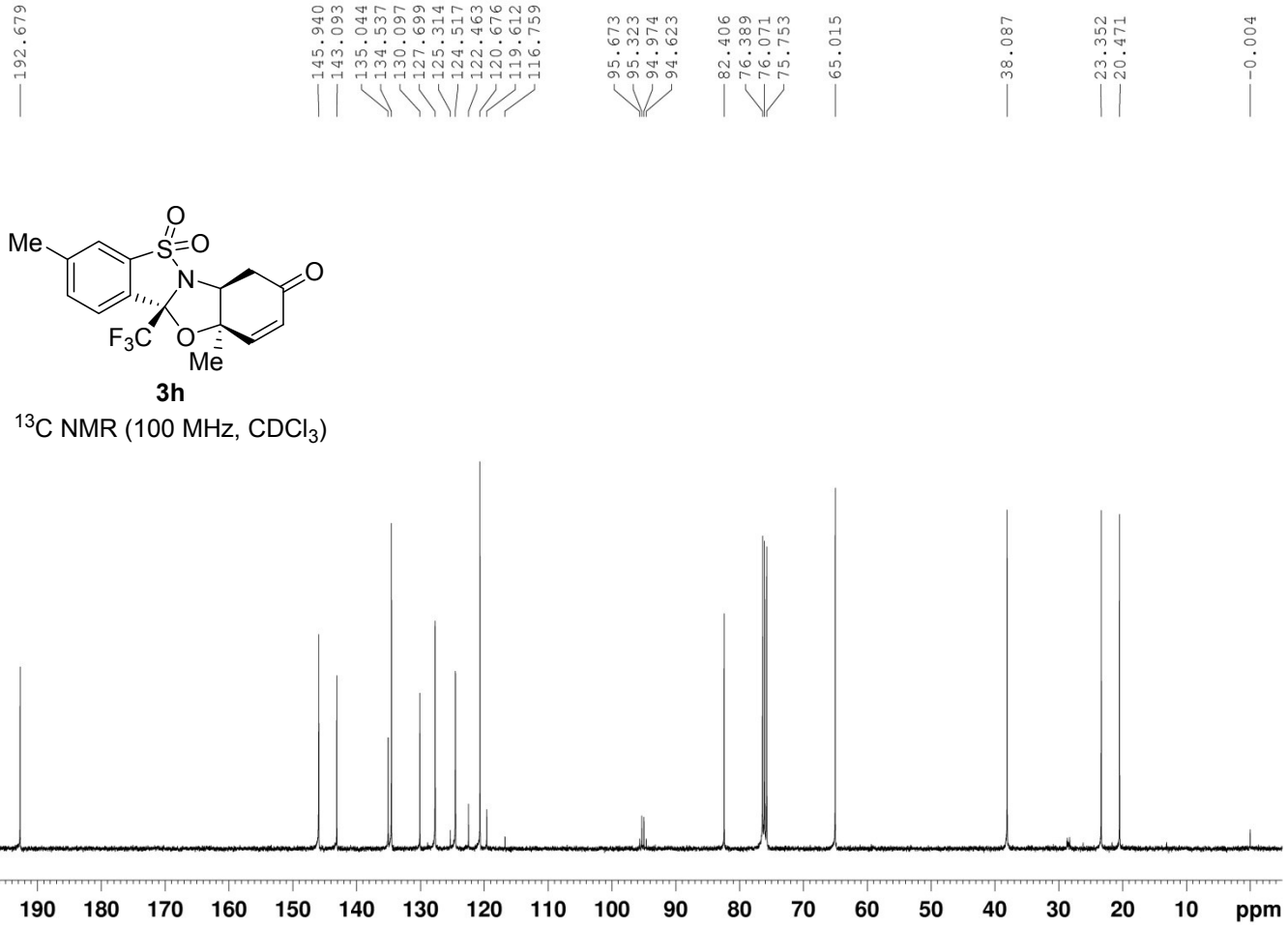
3h

^{1}H NMR (400 MHz, $CDCl_3$)



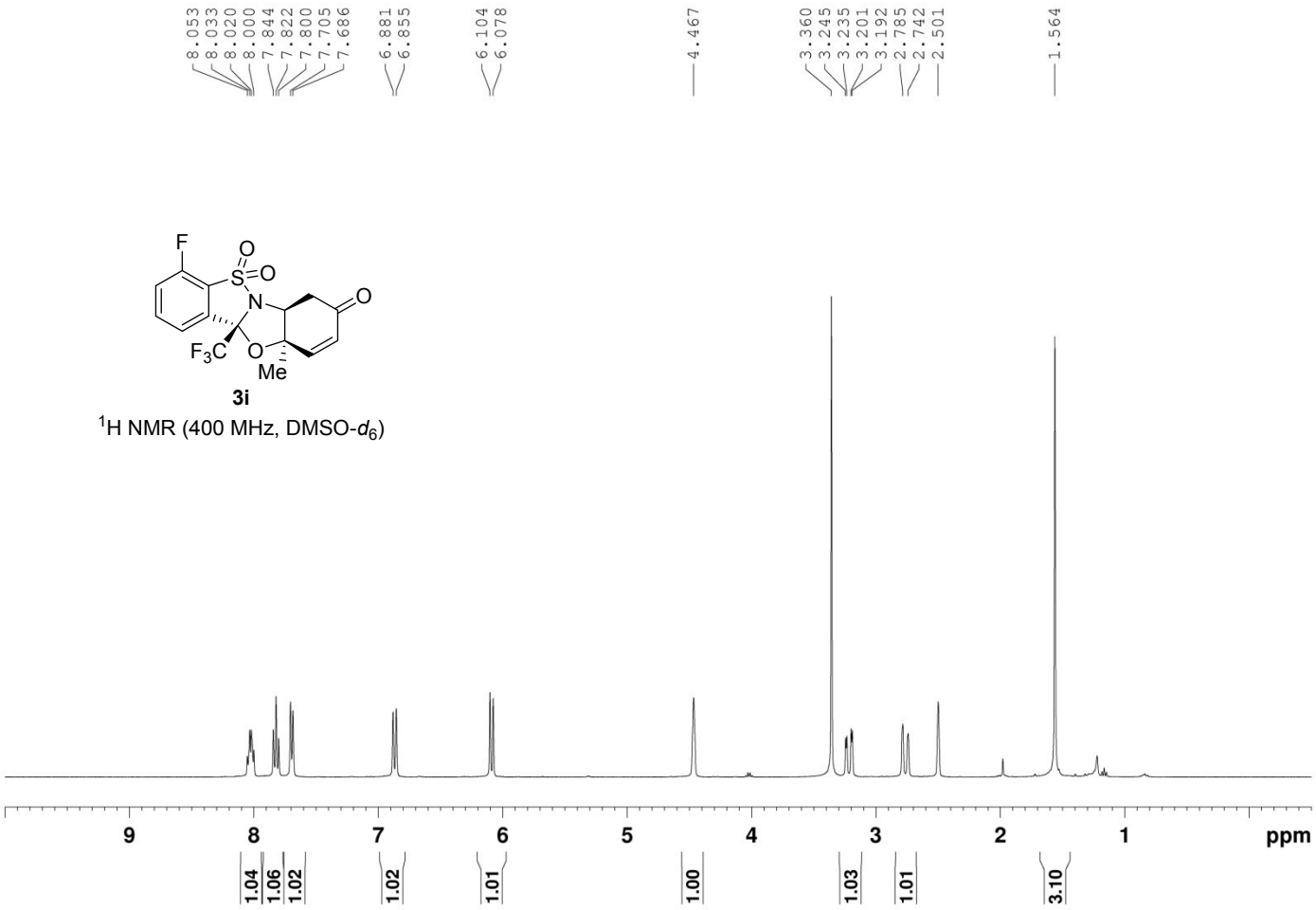
3h

^{19}F NMR (376 MHz, $CDCl_3$)



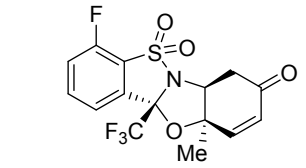
3h

^{13}C NMR (100 MHz, $CDCl_3$)



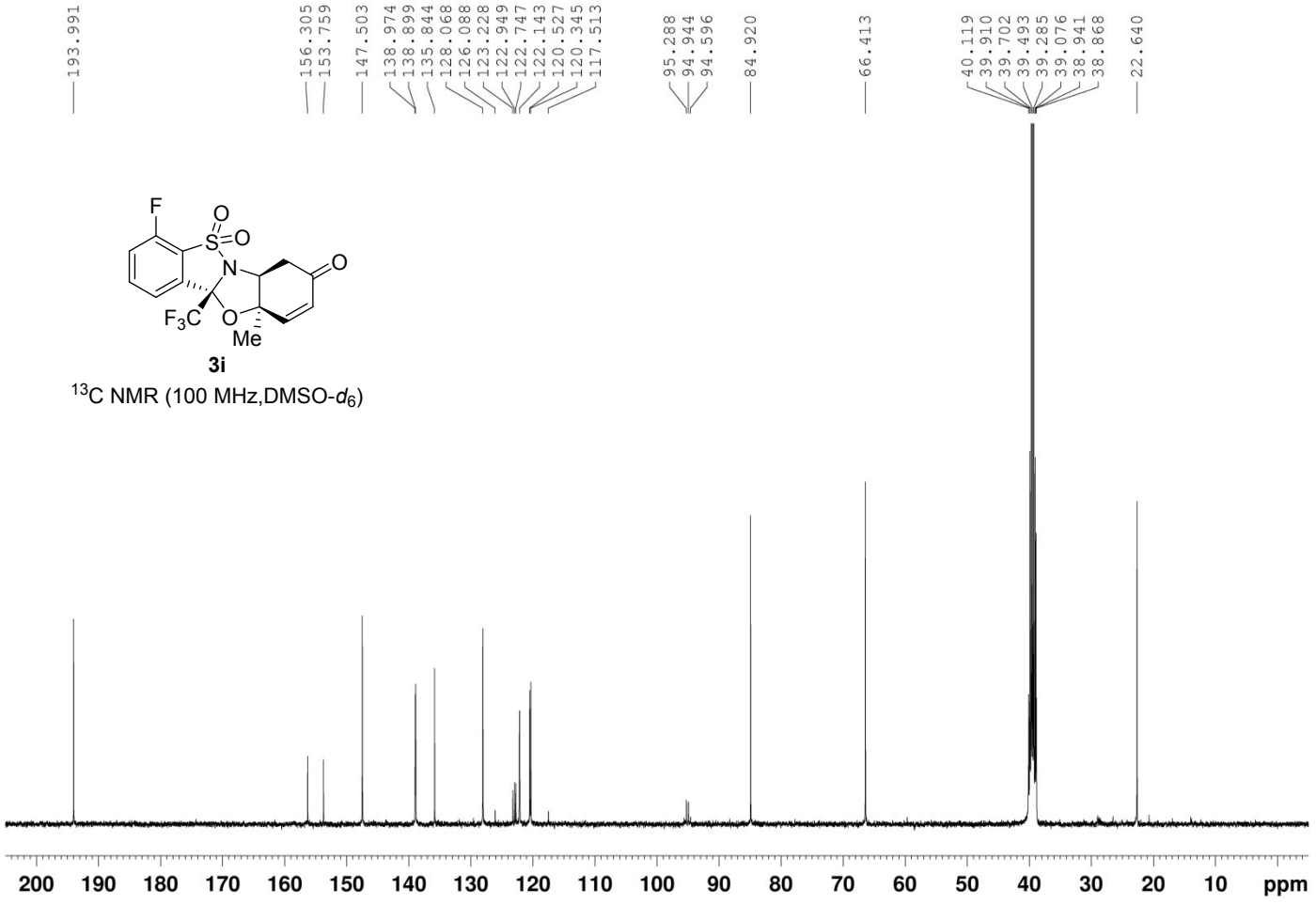
3i

^{1}H NMR (400 MHz, DMSO-d_6)



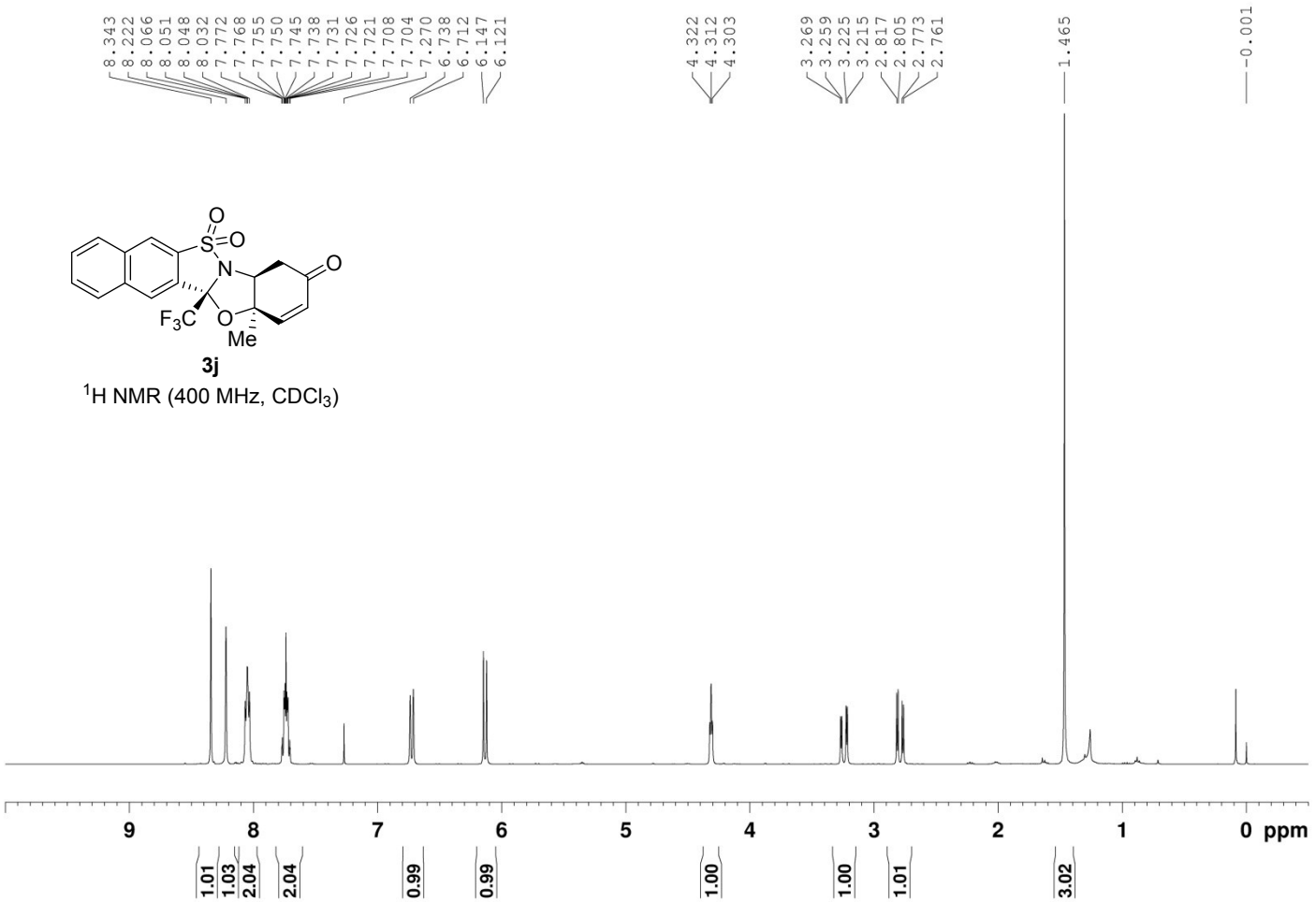
3i

^{19}F NMR (376 MHz, DMSO-d_6)



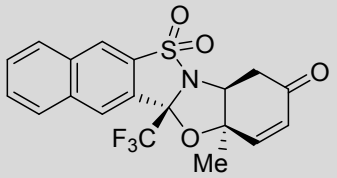
3i

^{13}C NMR (100 MHz,DMSO-d_6)



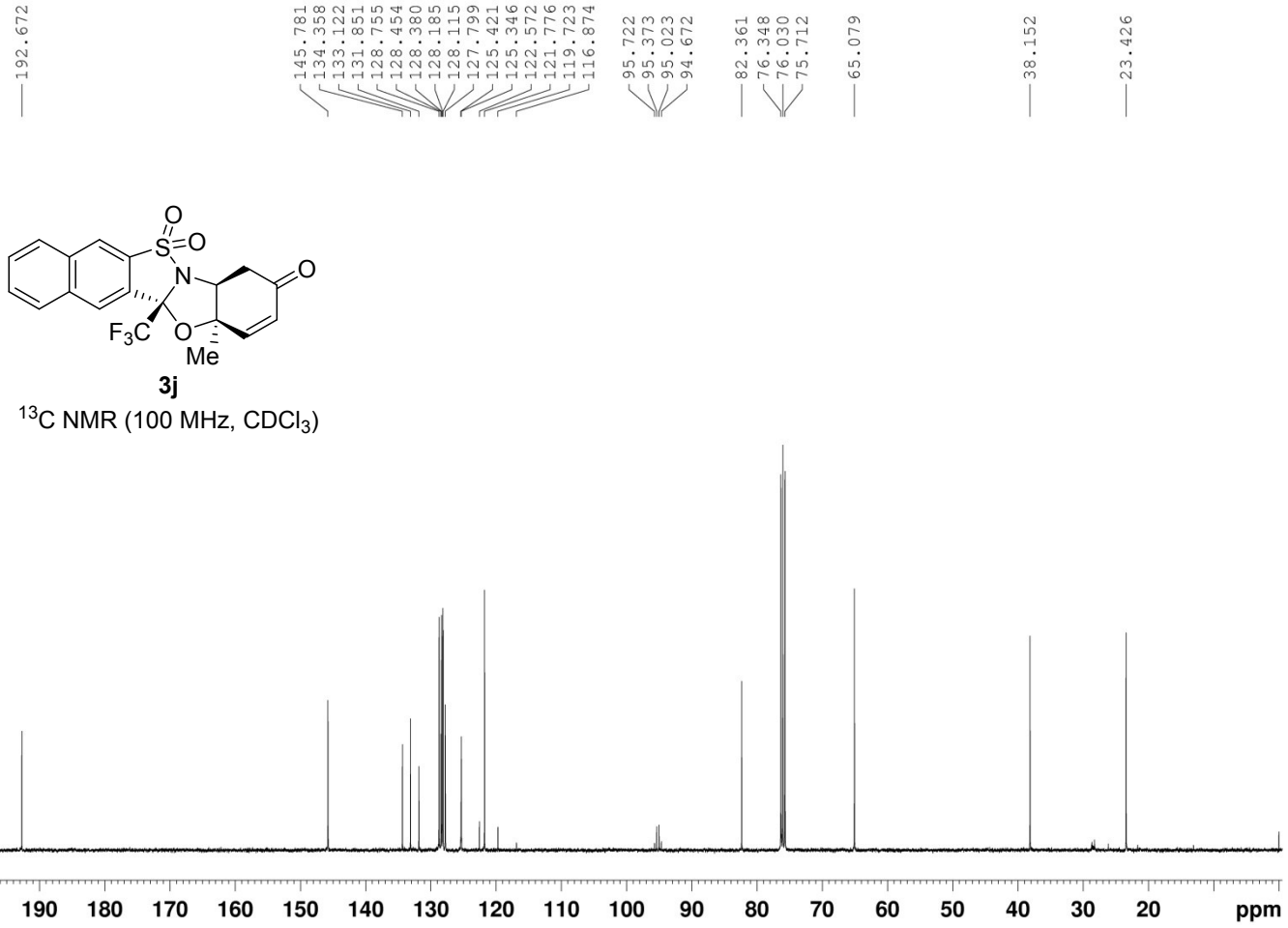
3j

^{1}H NMR (400 MHz, $CDCl_3$)



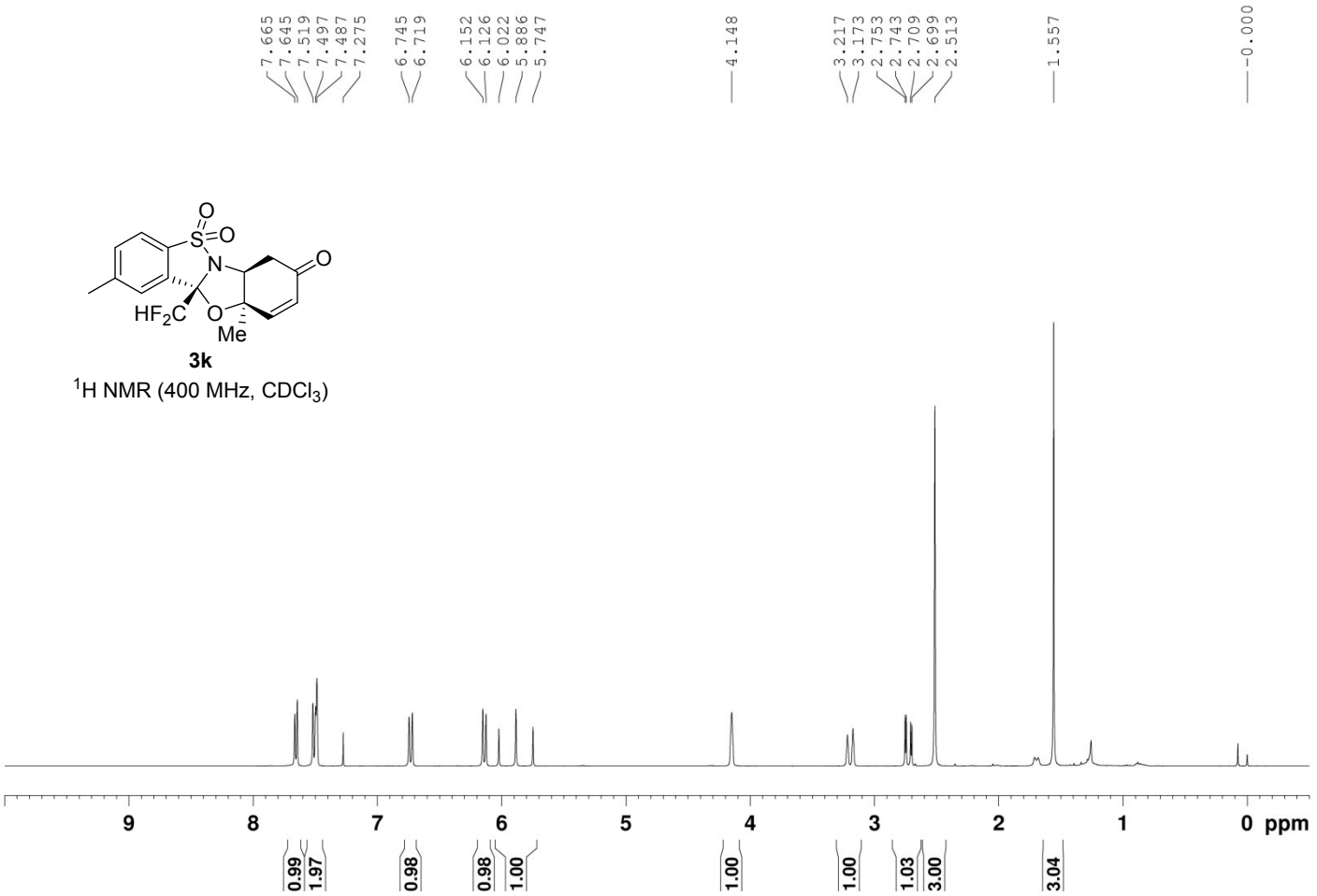
3j

^{19}F NMR (376 MHz, $CDCl_3$)



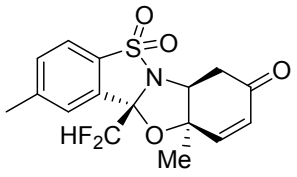
3j

^{13}C NMR (100 MHz, $CDCl_3$)



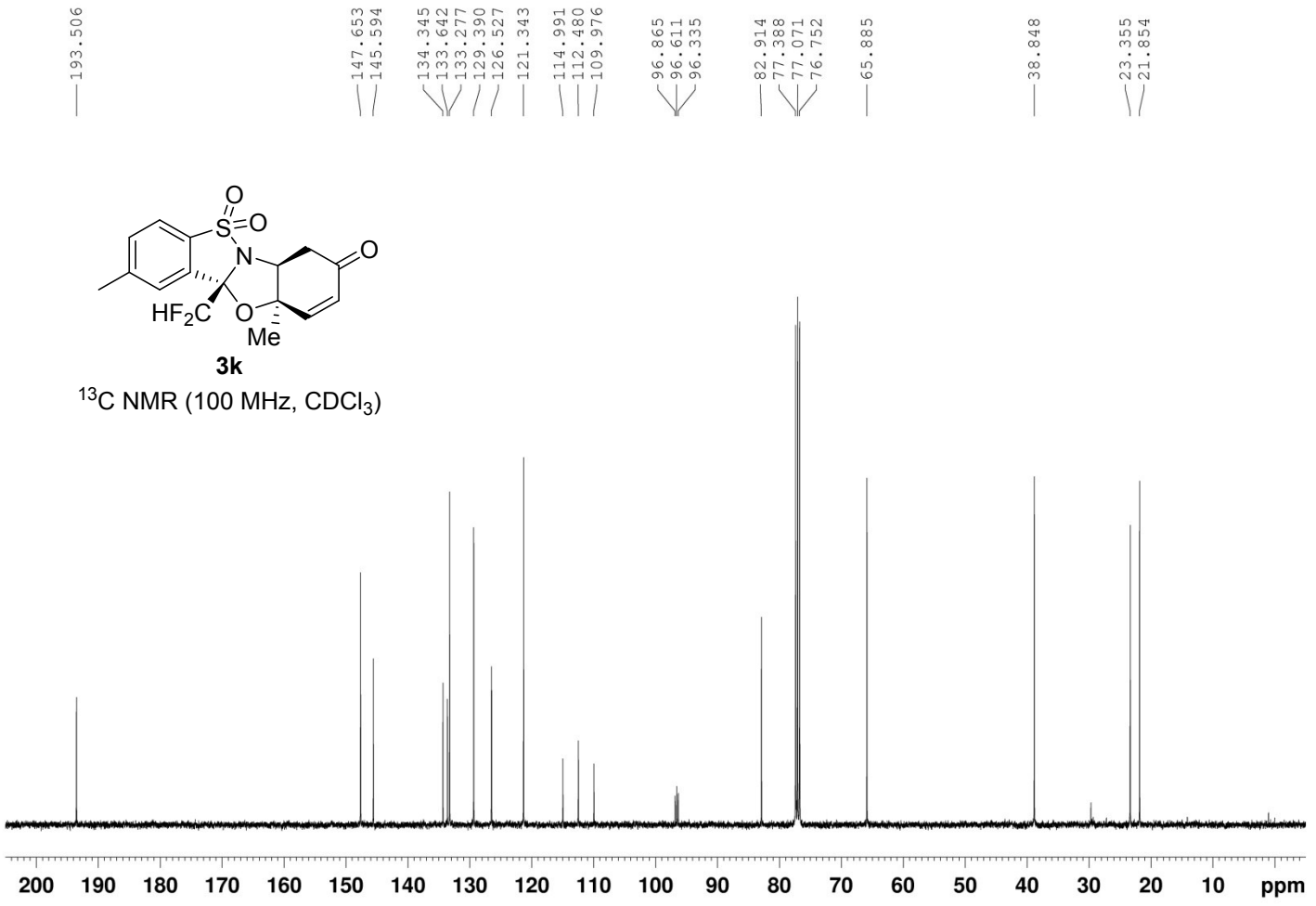
3k

^{1}H NMR (400 MHz, $CDCl_3$)



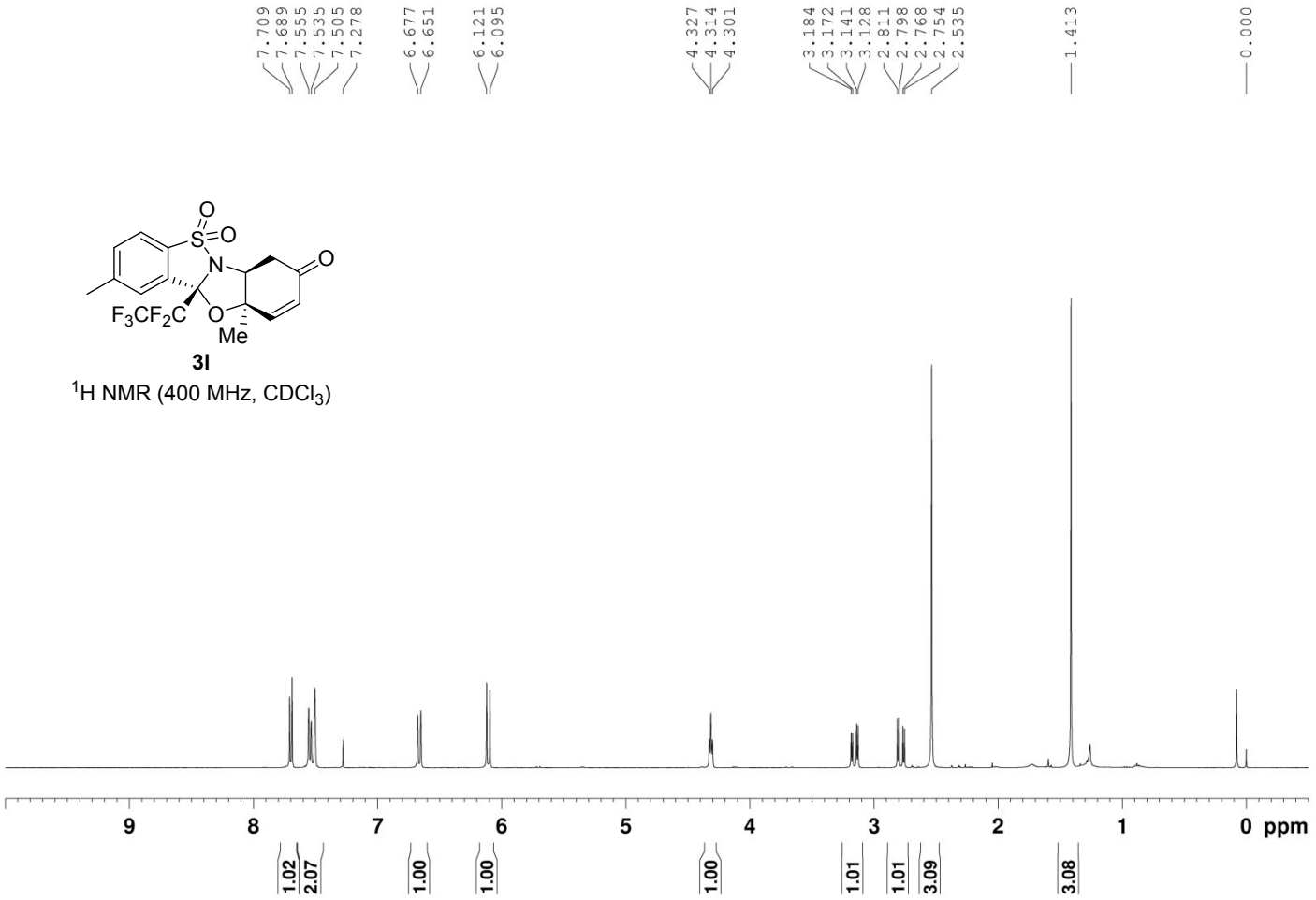
3k

^{19}F NMR (376 MHz, $CDCl_3$)



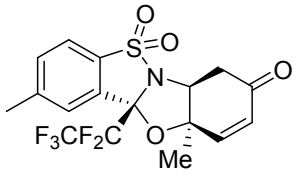
3k

^{13}C NMR (100 MHz, $CDCl_3$)



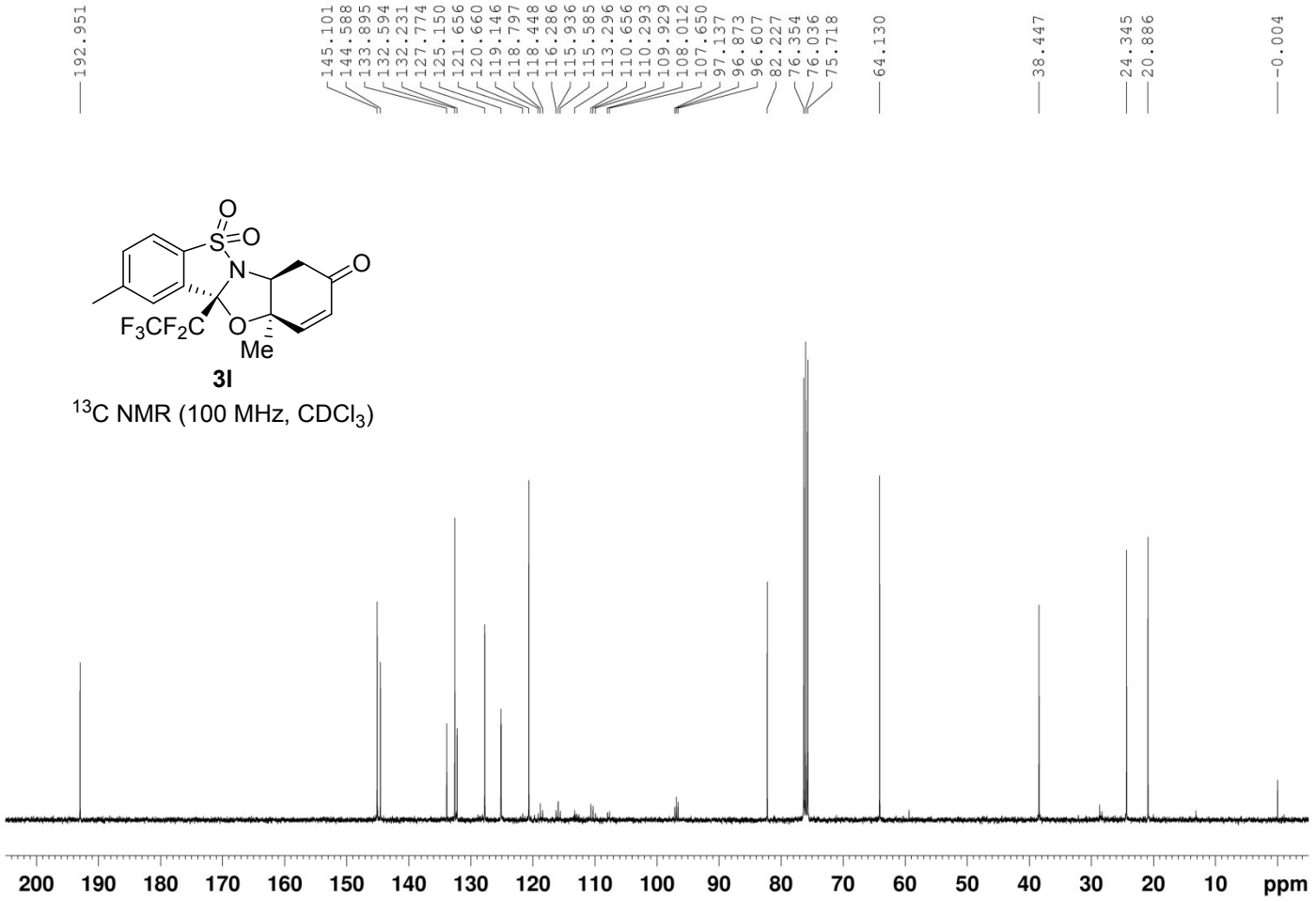
3l

^{1}H NMR (400 MHz, $CDCl_3$)



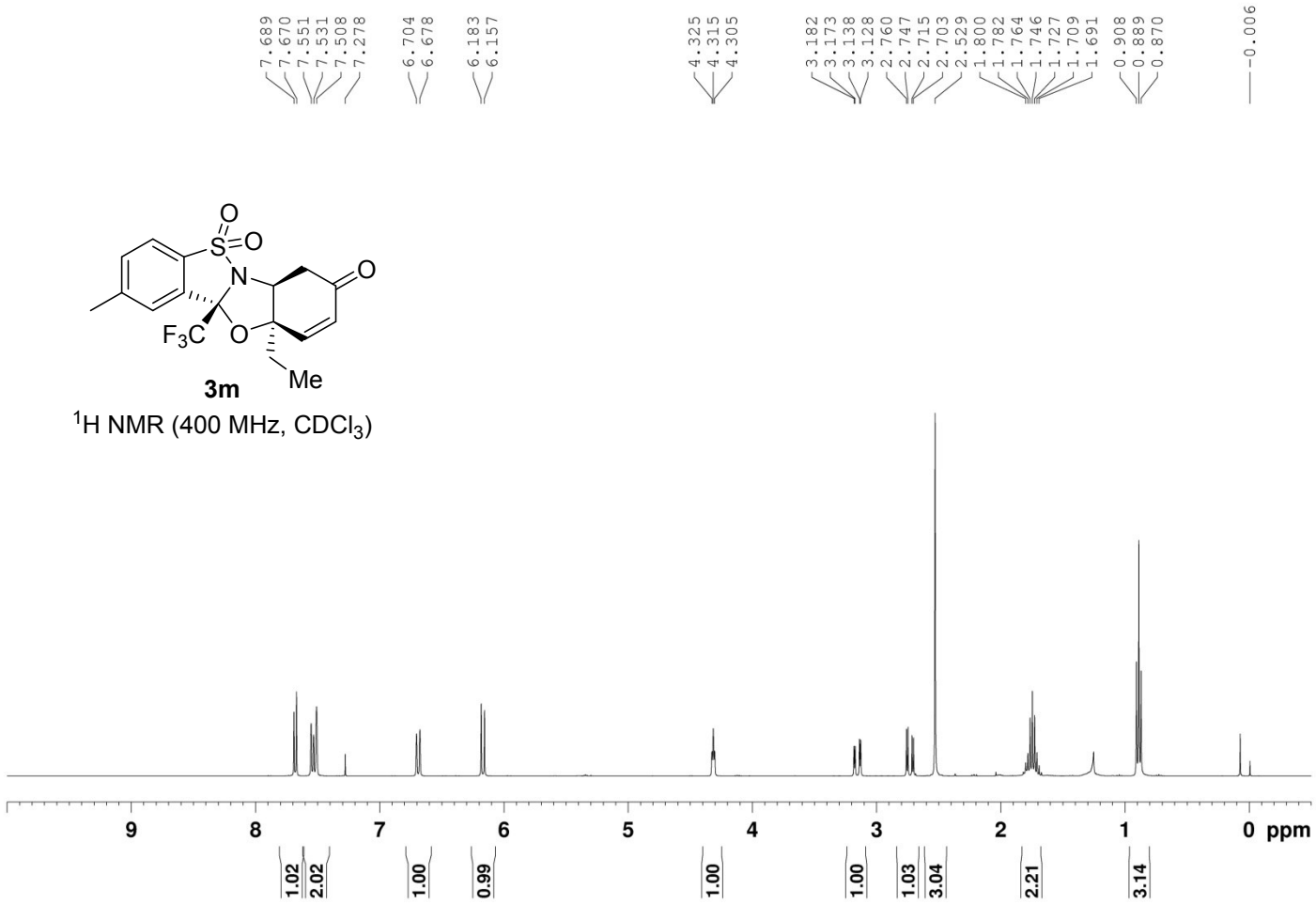
3l

^{19}F NMR (376 MHz, $CDCl_3$)



3l

^{13}C NMR (100 MHz, $CDCl_3$)


7.689
7.670
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6.678
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6.157
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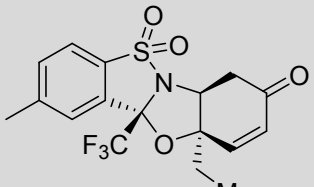
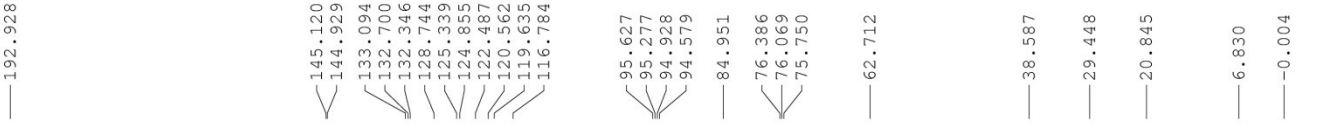
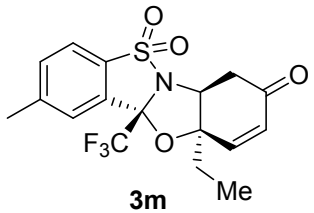
4.305
3.182
3.173
3.138
3.128
2.760
2.747
2.715
2.703
2.529
1.800
1.782
1.764
1.746
1.727
1.709
1.691
0.908
0.889
0.870
0.006
O
S
O
N
O
F3C
O
Me
3m
1H NMR (400 MHz, CDCl3)
9
8
7
6
5
4
3
2
1
0 ppm
1.02
2.02
1.00
0.99
1.00
1.00
1.03
3.04
2.21
3.14



3m

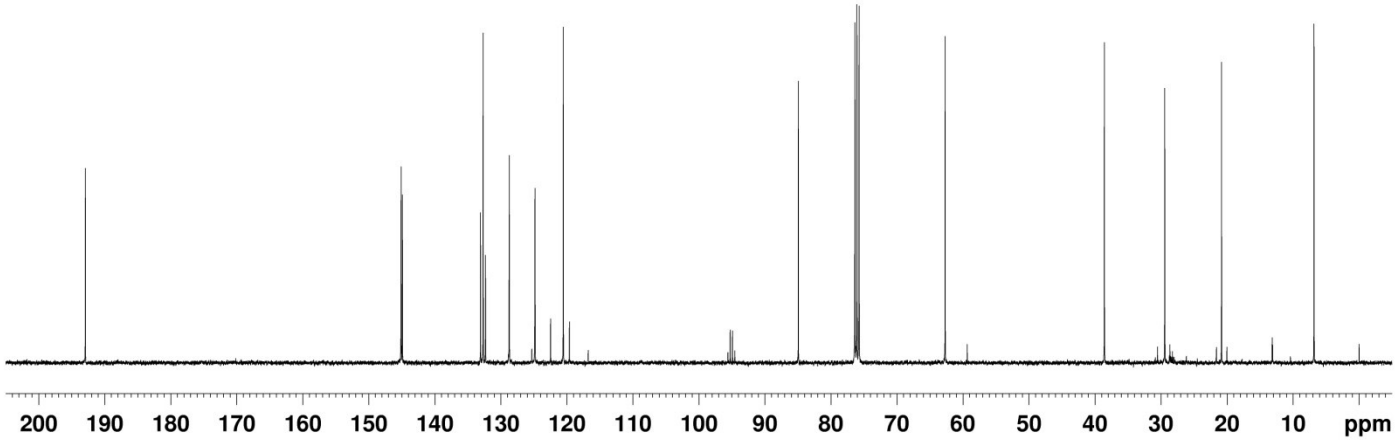
^{19}F NMR (376 MHz, $CDCl_3$)

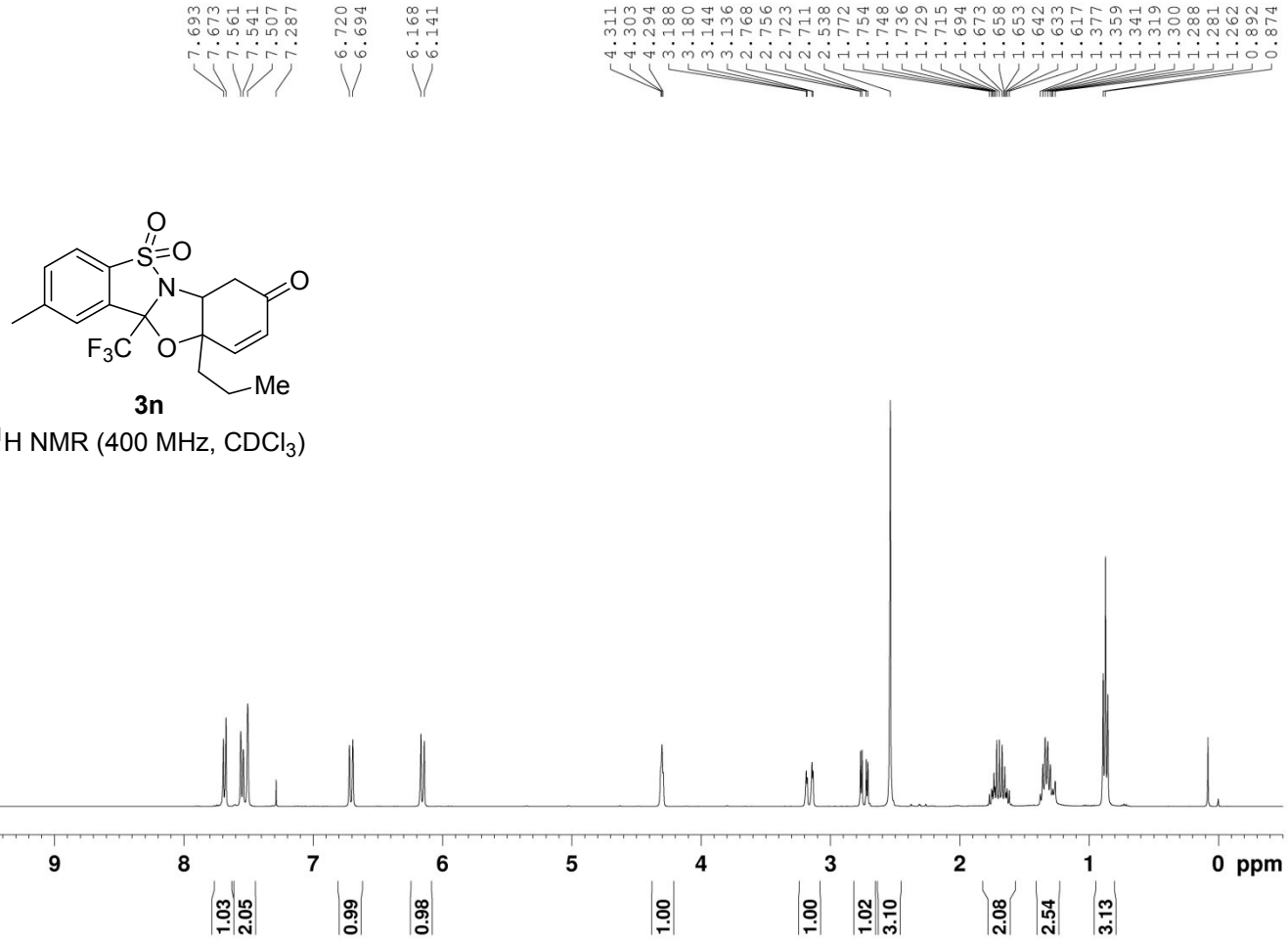




3m

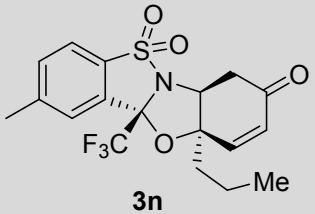
^{13}C NMR (100 MHz, $CDCl_3$)





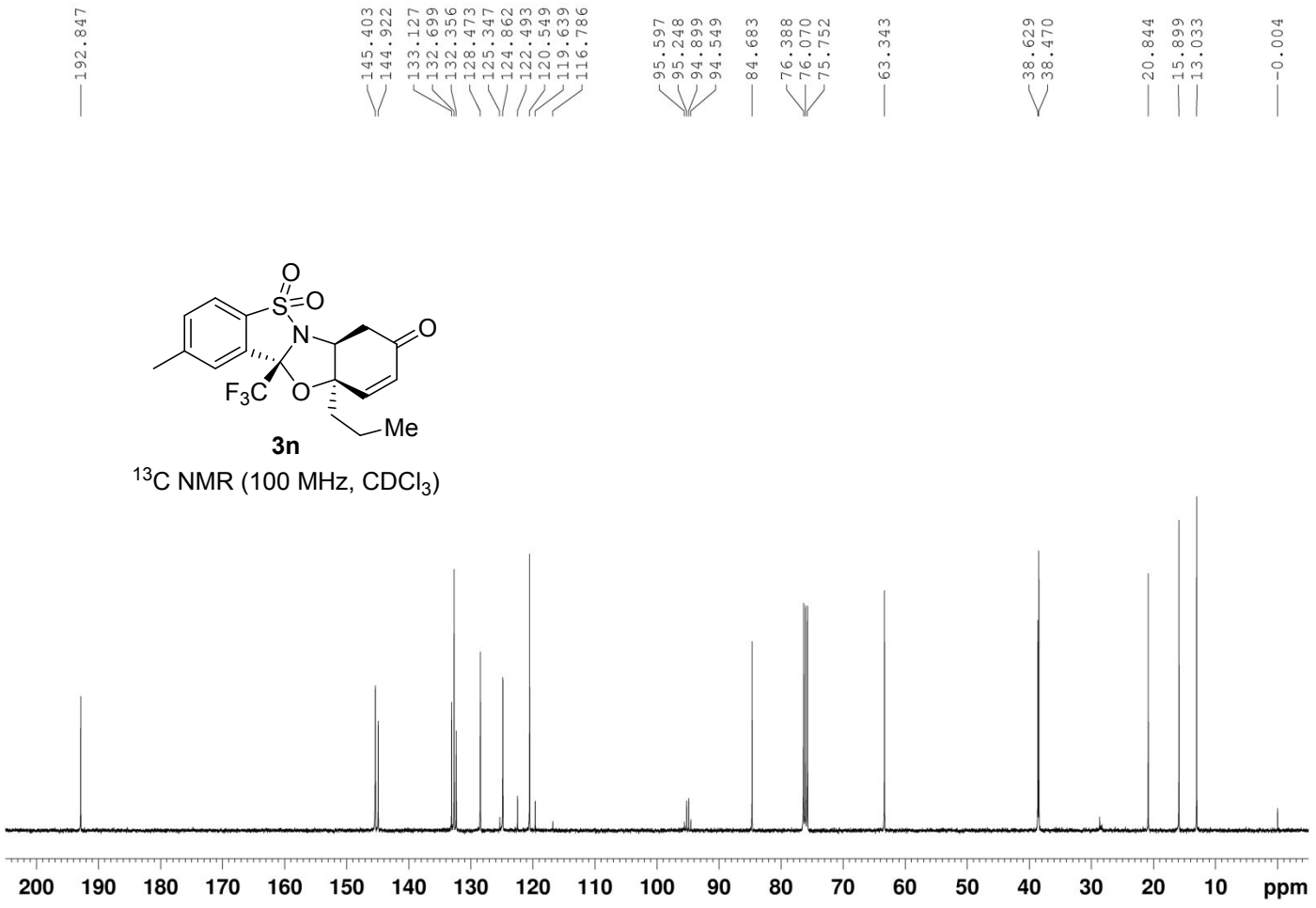
3n

^{1}H NMR (400 MHz, $CDCl_3$)



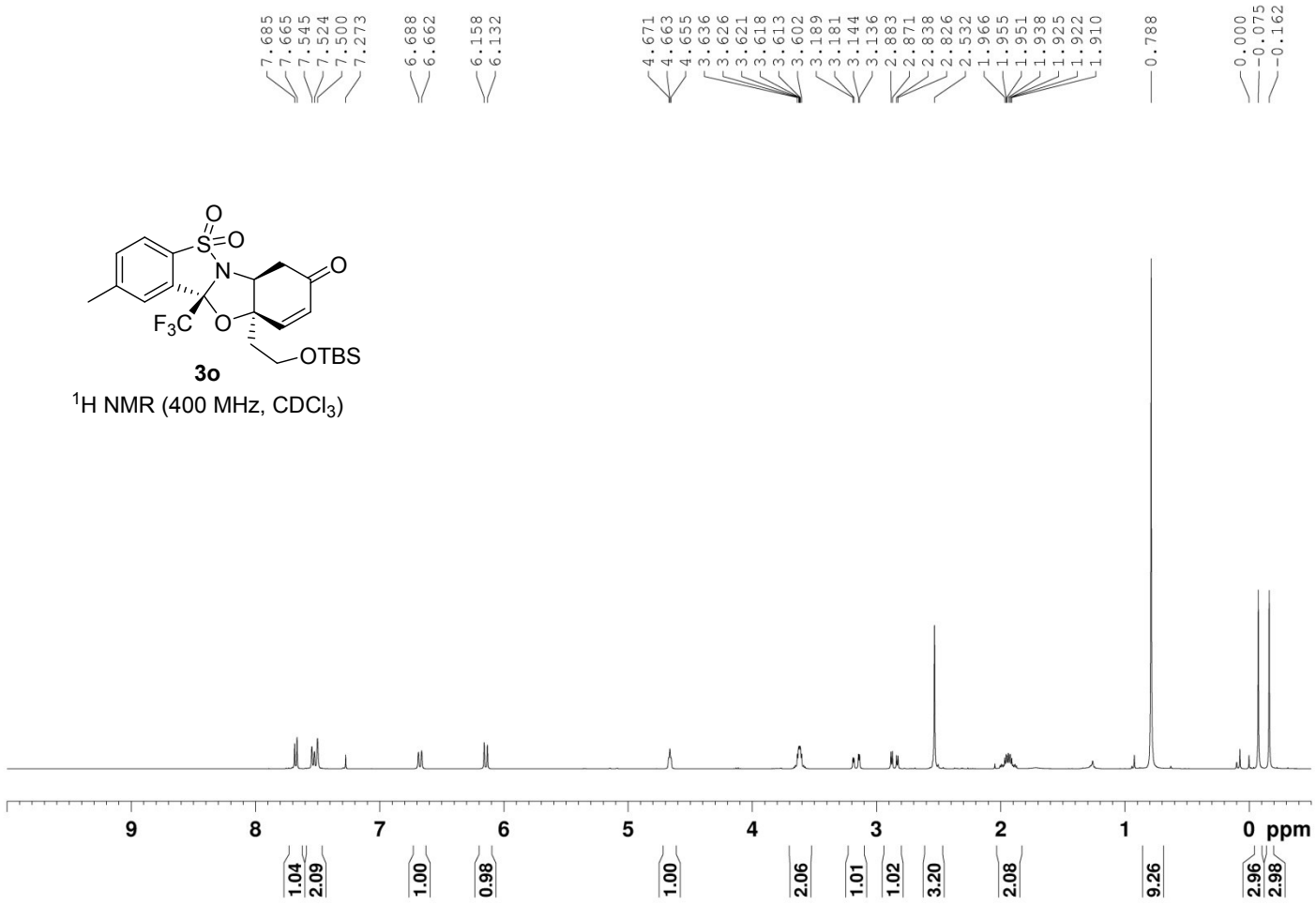
3n

^{19}F NMR (376 MHz, $CDCl_3$)



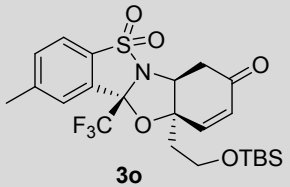
3n

^{13}C NMR (100 MHz, $CDCl_3$)



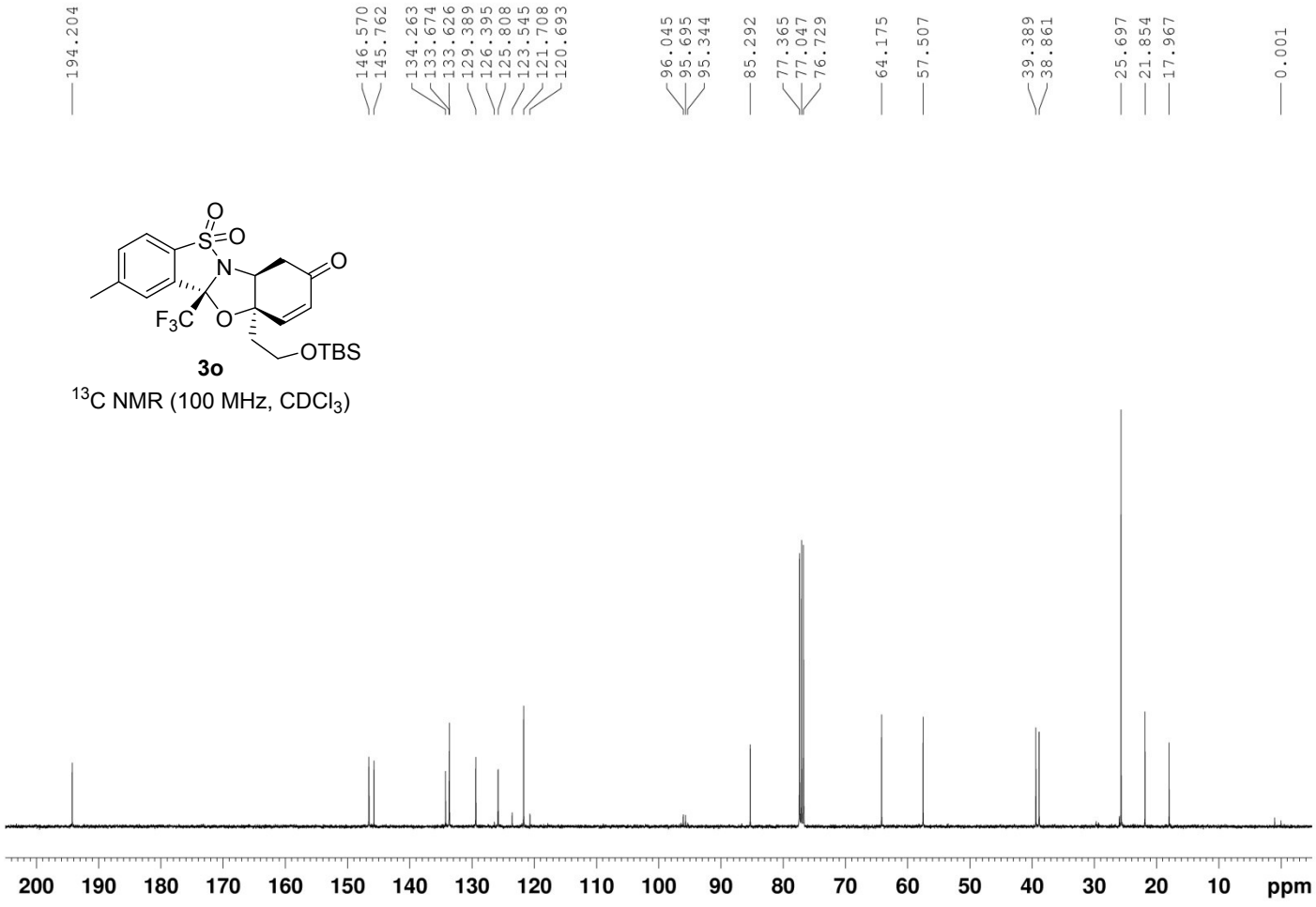
3o

^{1}H NMR (400 MHz, $CDCl_3$)



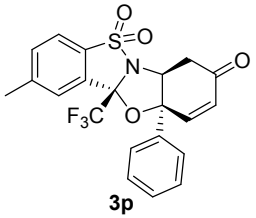
3o

^{19}F NMR (376 MHz, $CDCl_3$)



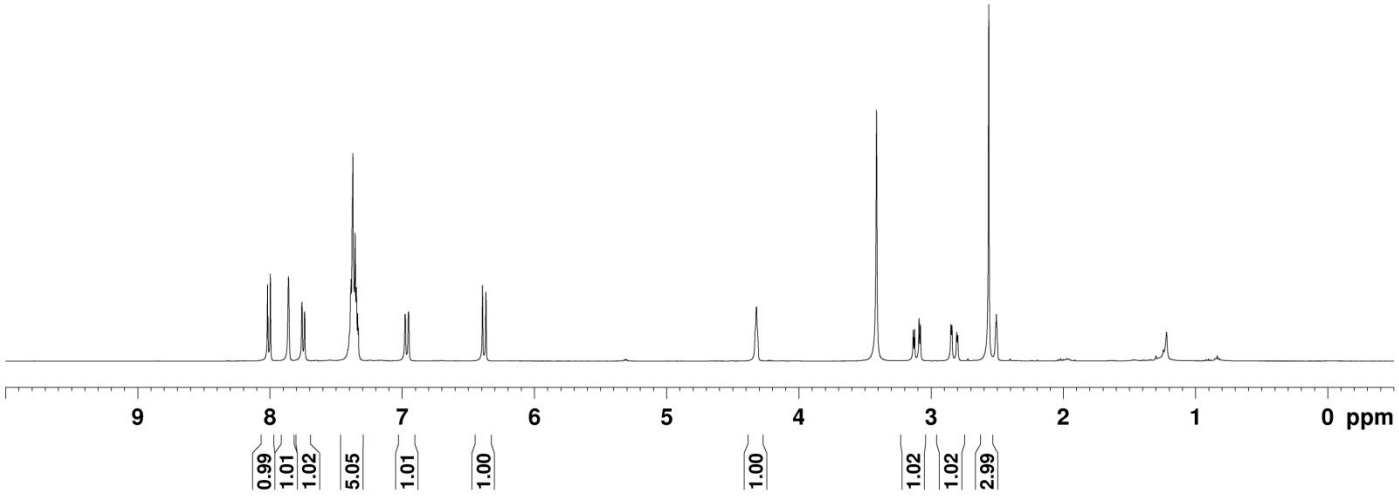
3o

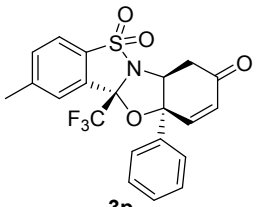
^{13}C NMR (100 MHz, $CDCl_3$)



3p

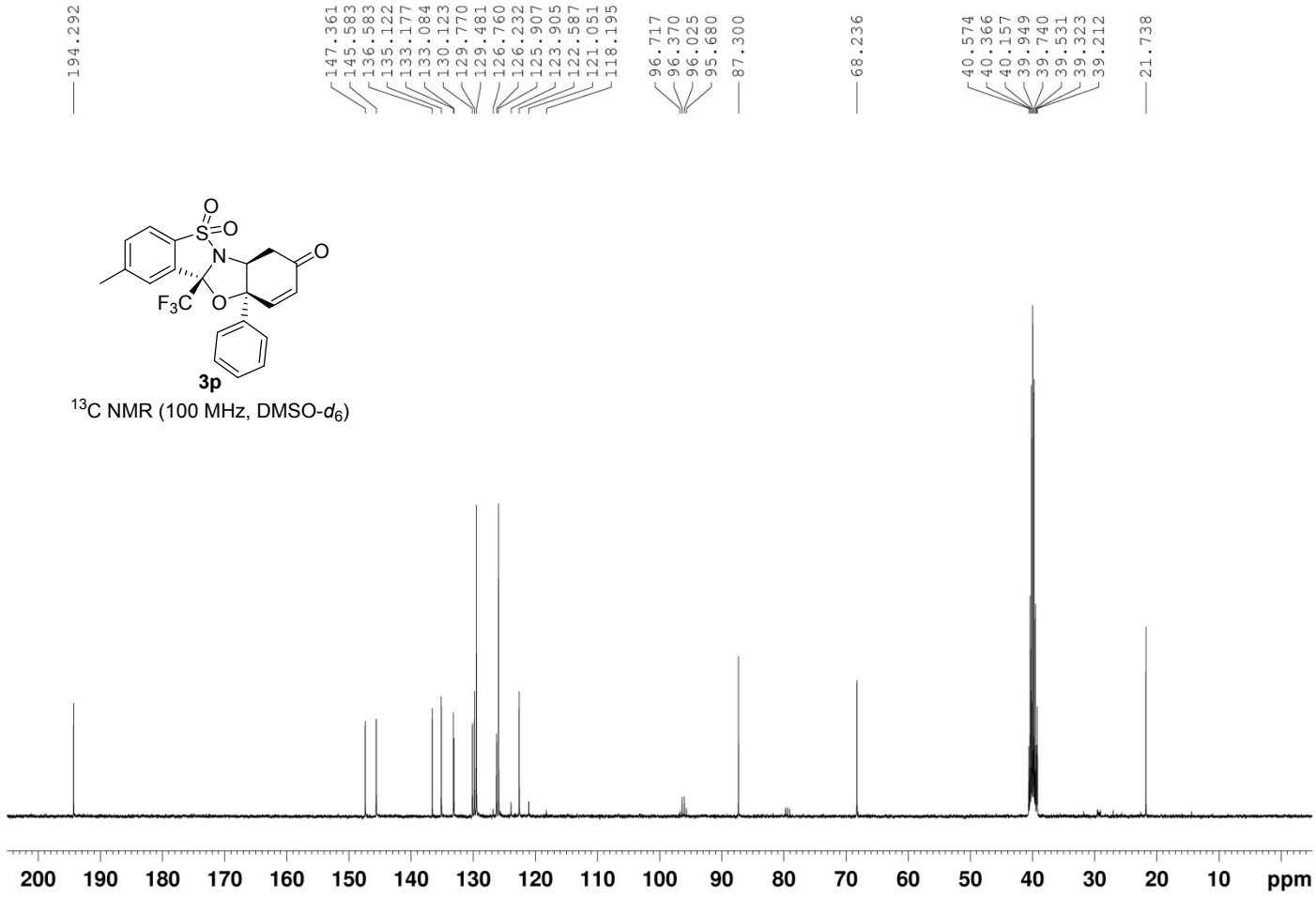
^{1}H NMR (400 MHz, DMSO-d_6)





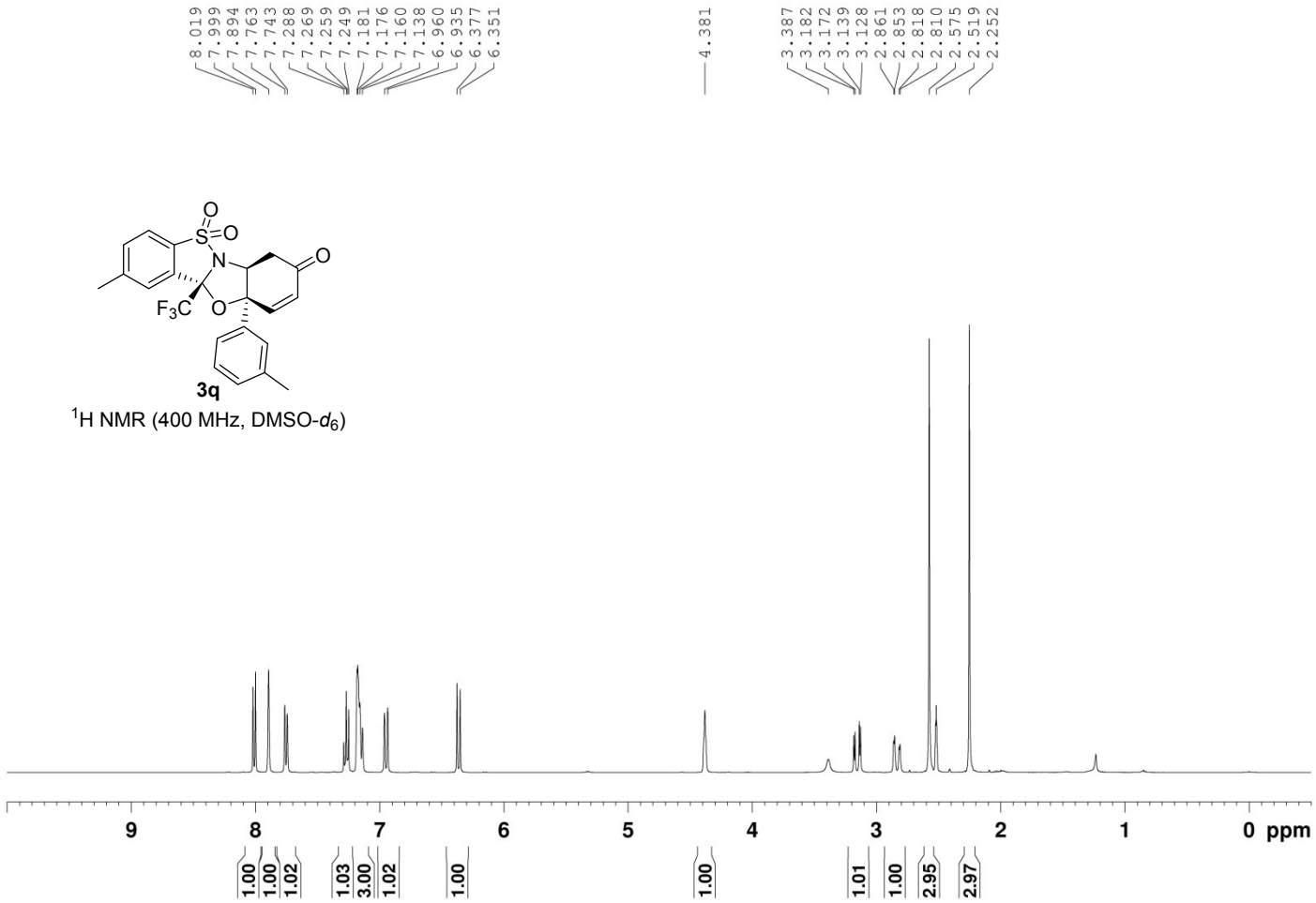
3p

^{19}F NMR (376 MHz, DMSO-d_6)



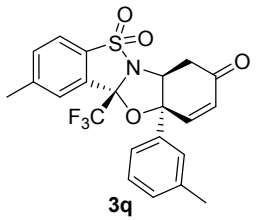
3p

^{13}C NMR (100 MHz, DMSO-d_6)



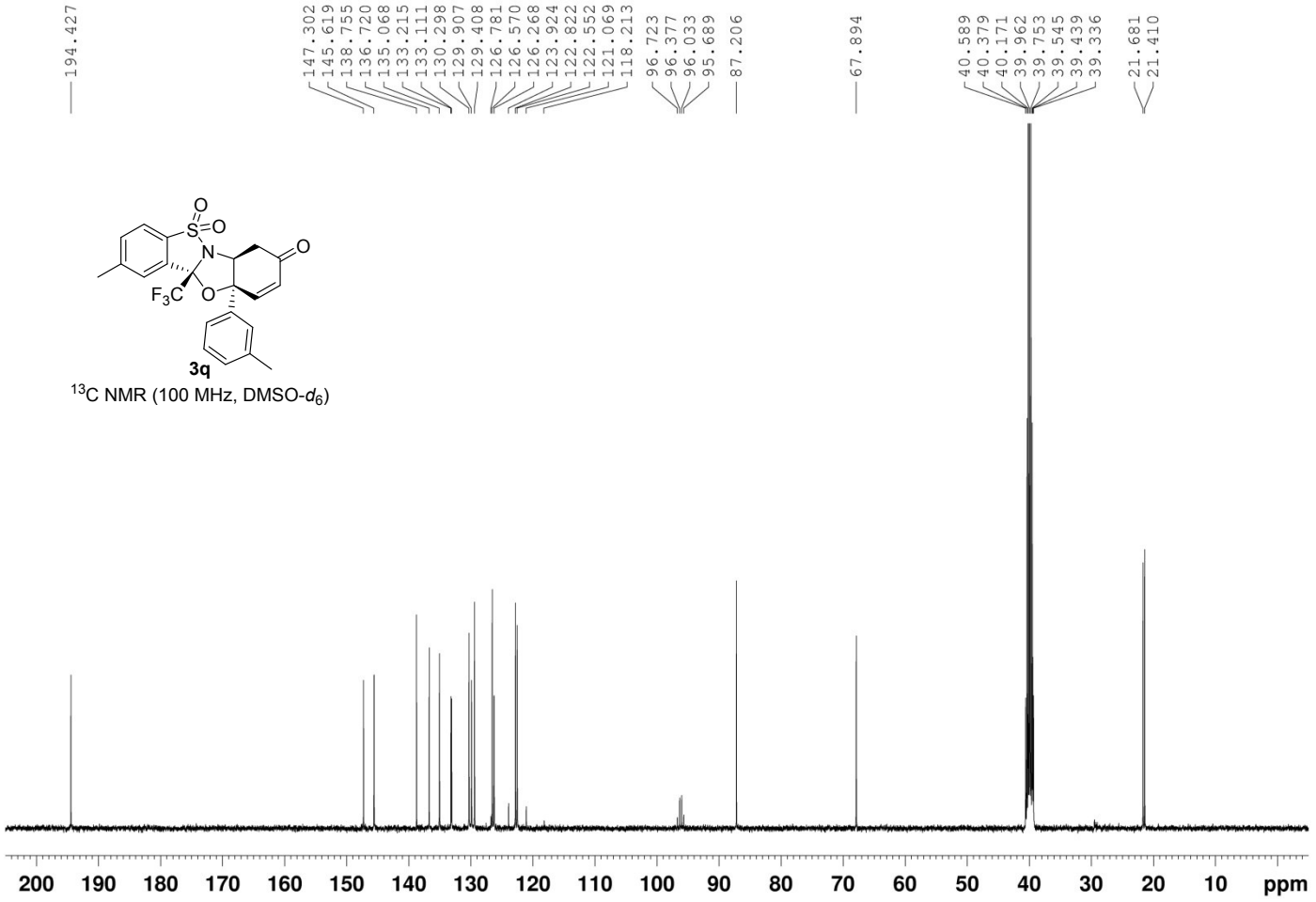
3q

^{1}H NMR (400 MHz, DMSO-d_6)



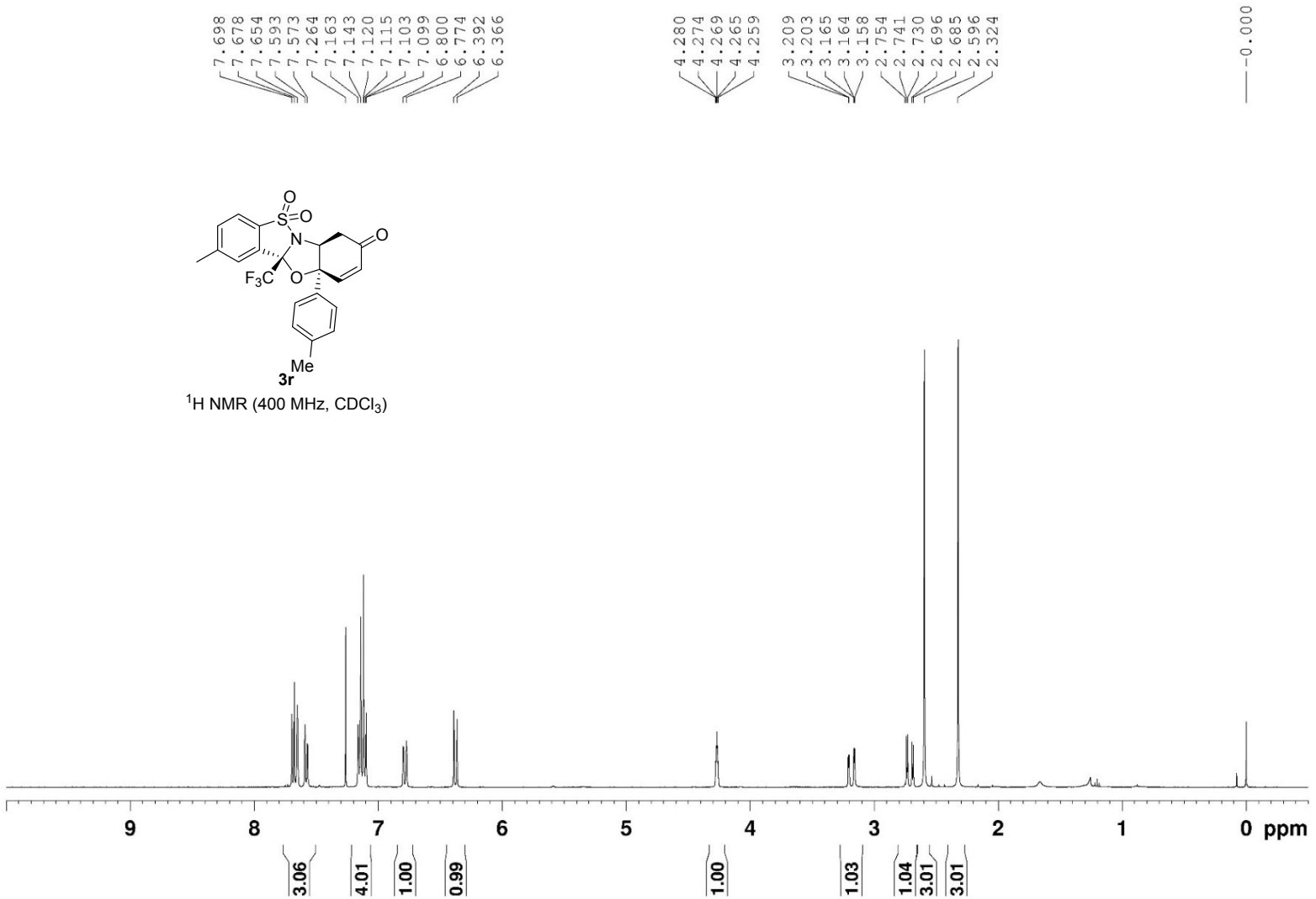
3q

^{19}F NMR (376 MHz, DMSO-d_6)



3q

^{13}C NMR (100 MHz, DMSO-d_6)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7.654
7.593
7.573
7.264
7.163
7.143
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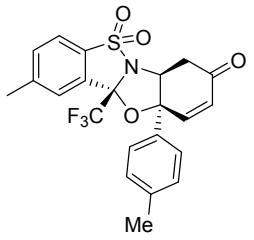
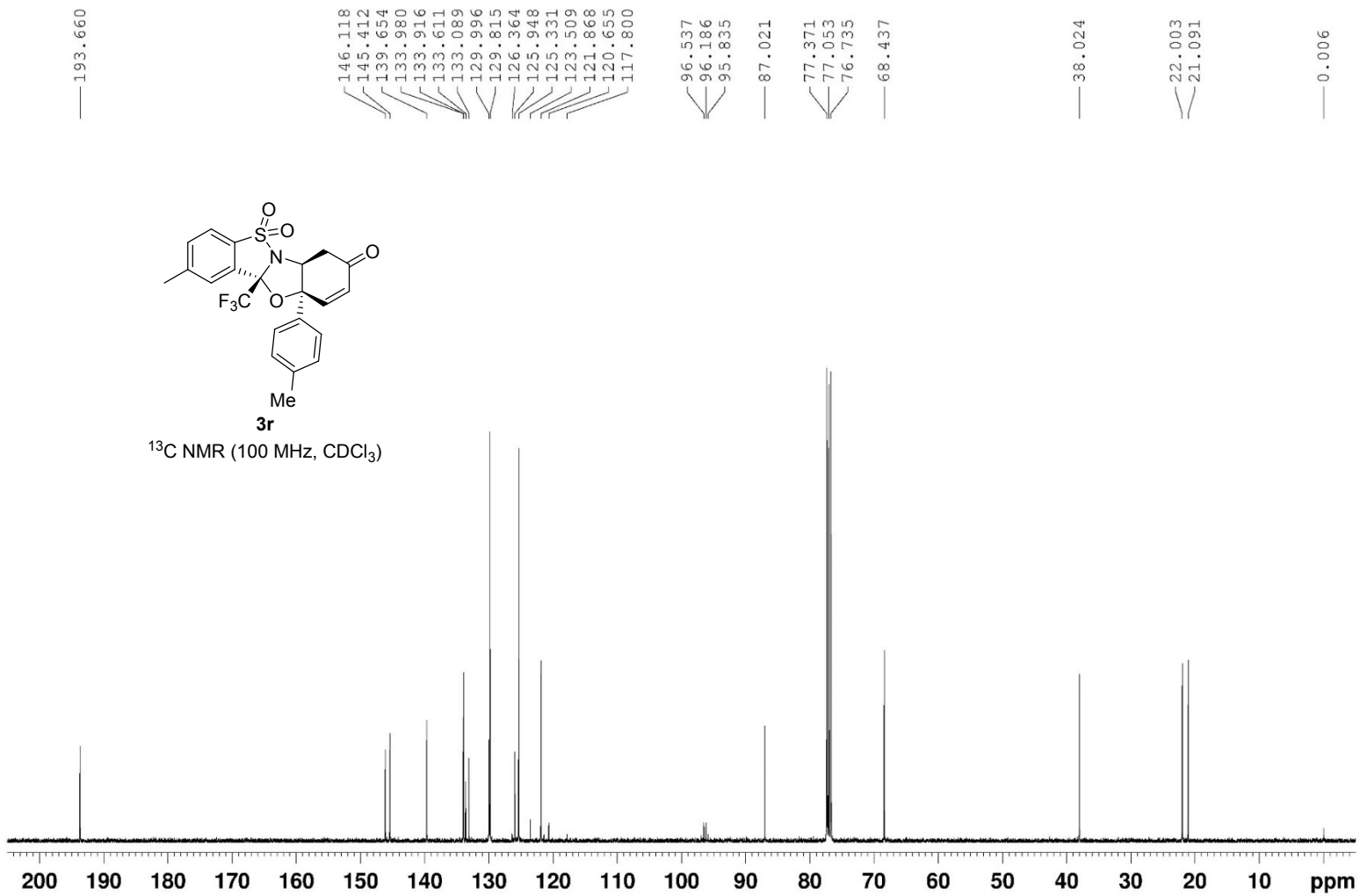
7.115
7.103
7.099
6.800
6.774
6.392
6.366
4.280
4.274
4.269
4.265
4.259
3.209
3.203
3.165
3.164
3.158
2.754
2.741
2.730
2.696
2.685
2.596
2.324
0.000
O
S
O
N
O
F3C
O
Me
3r
1H NMR (400 MHz, CDCl3)
9
8
7
6
5
4
3
2
1
0 ppm
3.06
4.01
1.00
0.99
1.00
1.03
1.04
3.01
3.01



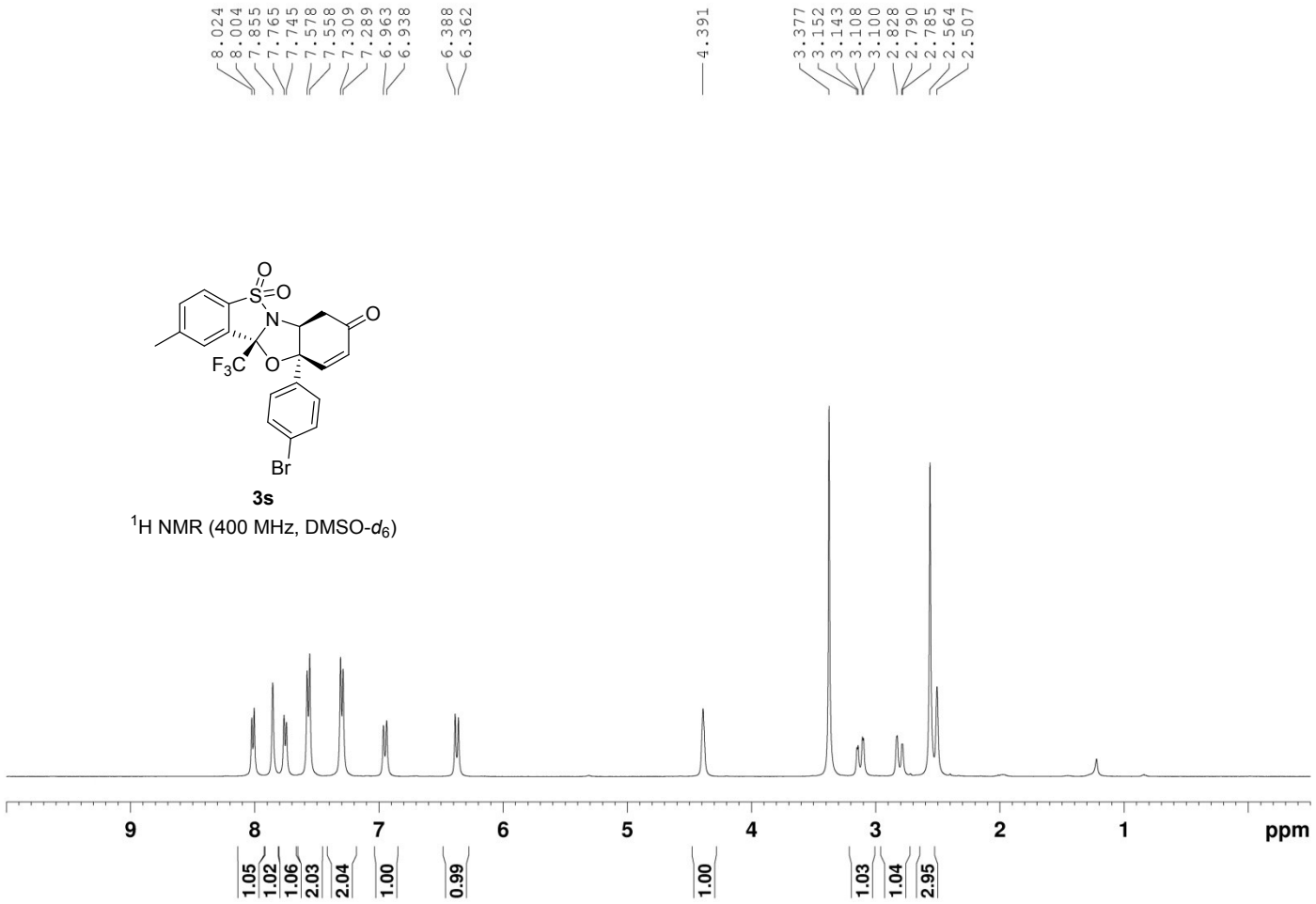
3r

^{19}F NMR (376 MHz, $CDCl_3$)



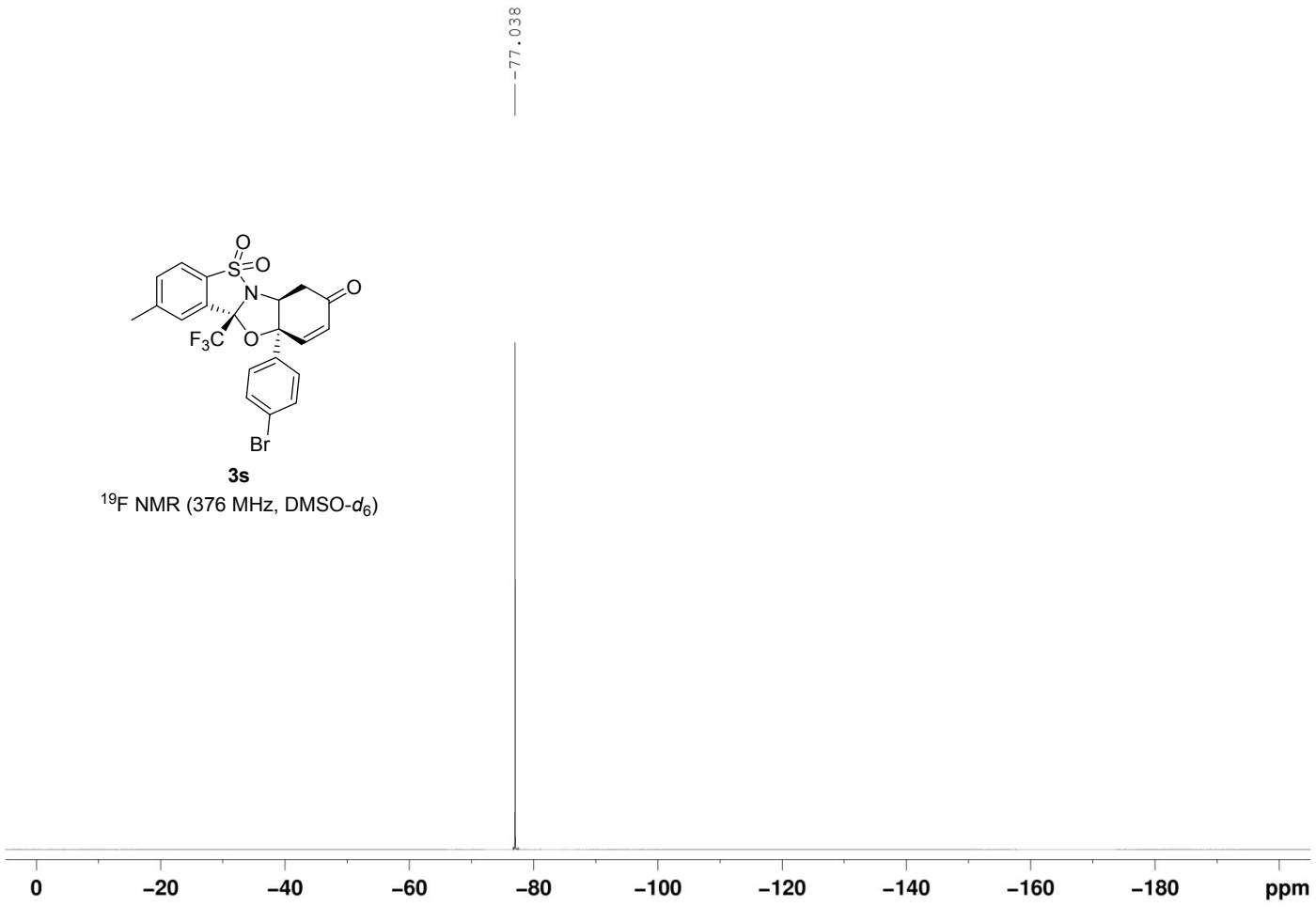
3r

^{13}C NMR (100 MHz, $CDCl_3$)



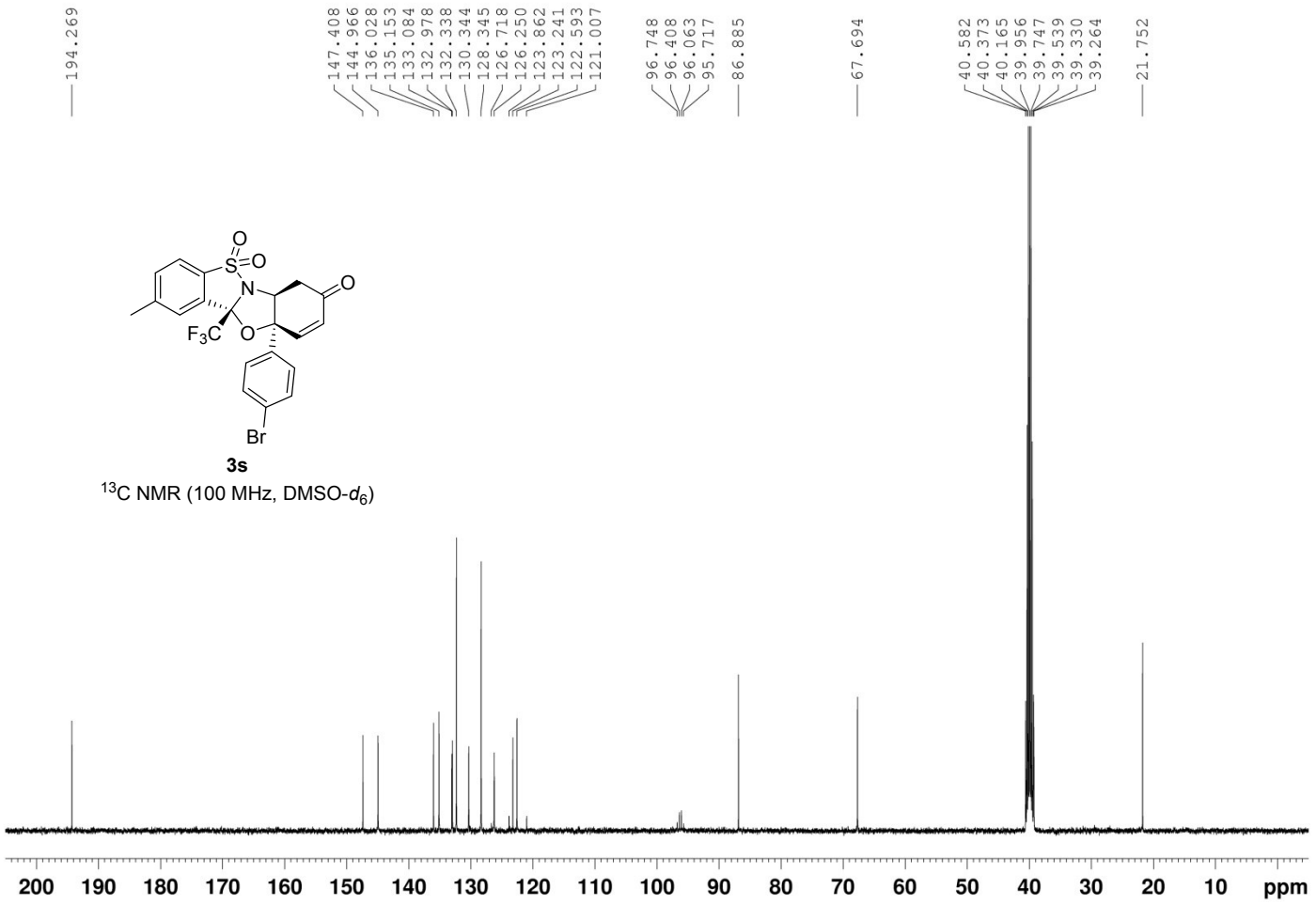
3s

^{1}H NMR (400 MHz, DMSO-d_6)



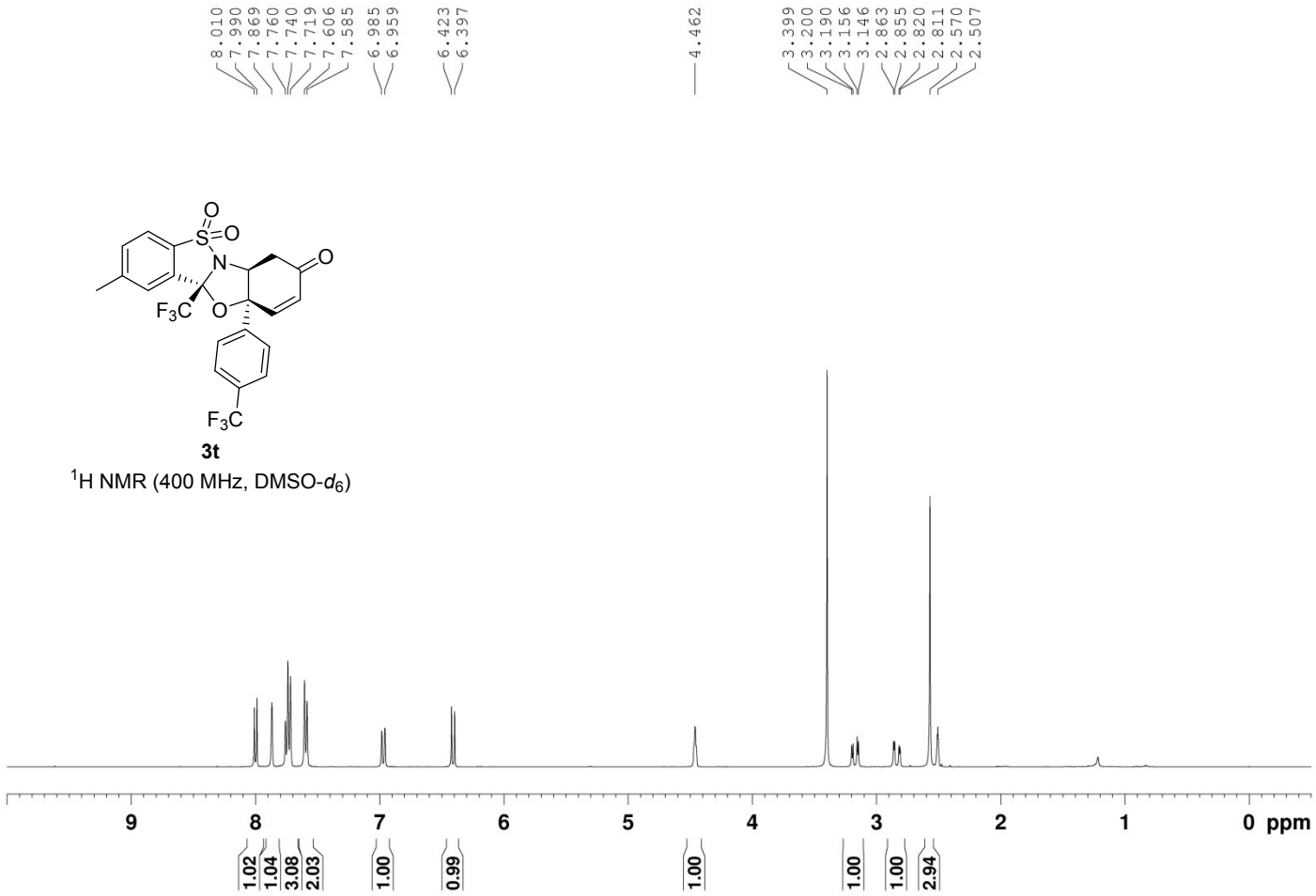
3s

^{19}F NMR (376 MHz, DMSO-d_6)



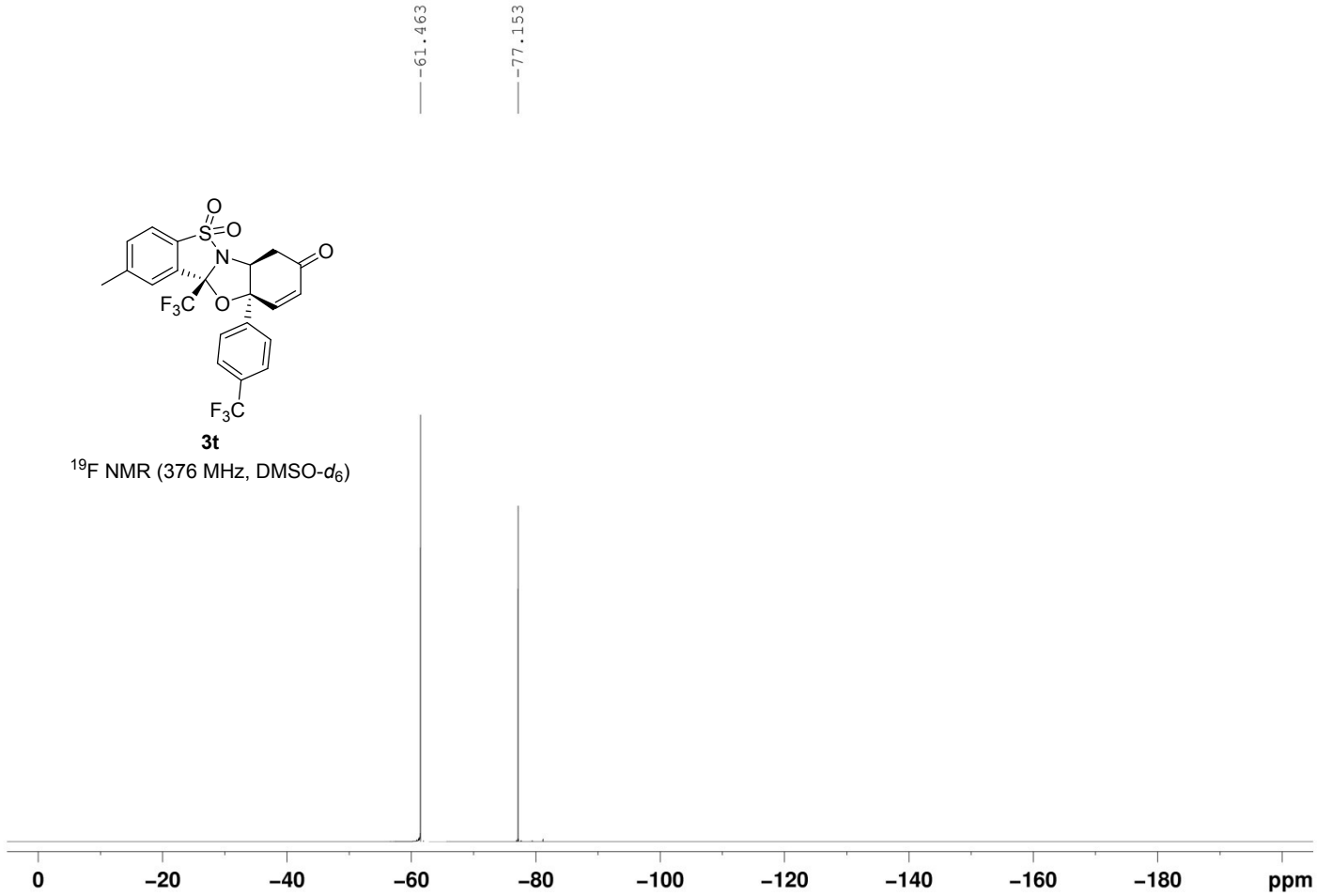
3s

^{13}C NMR (100 MHz, DMSO-d_6)



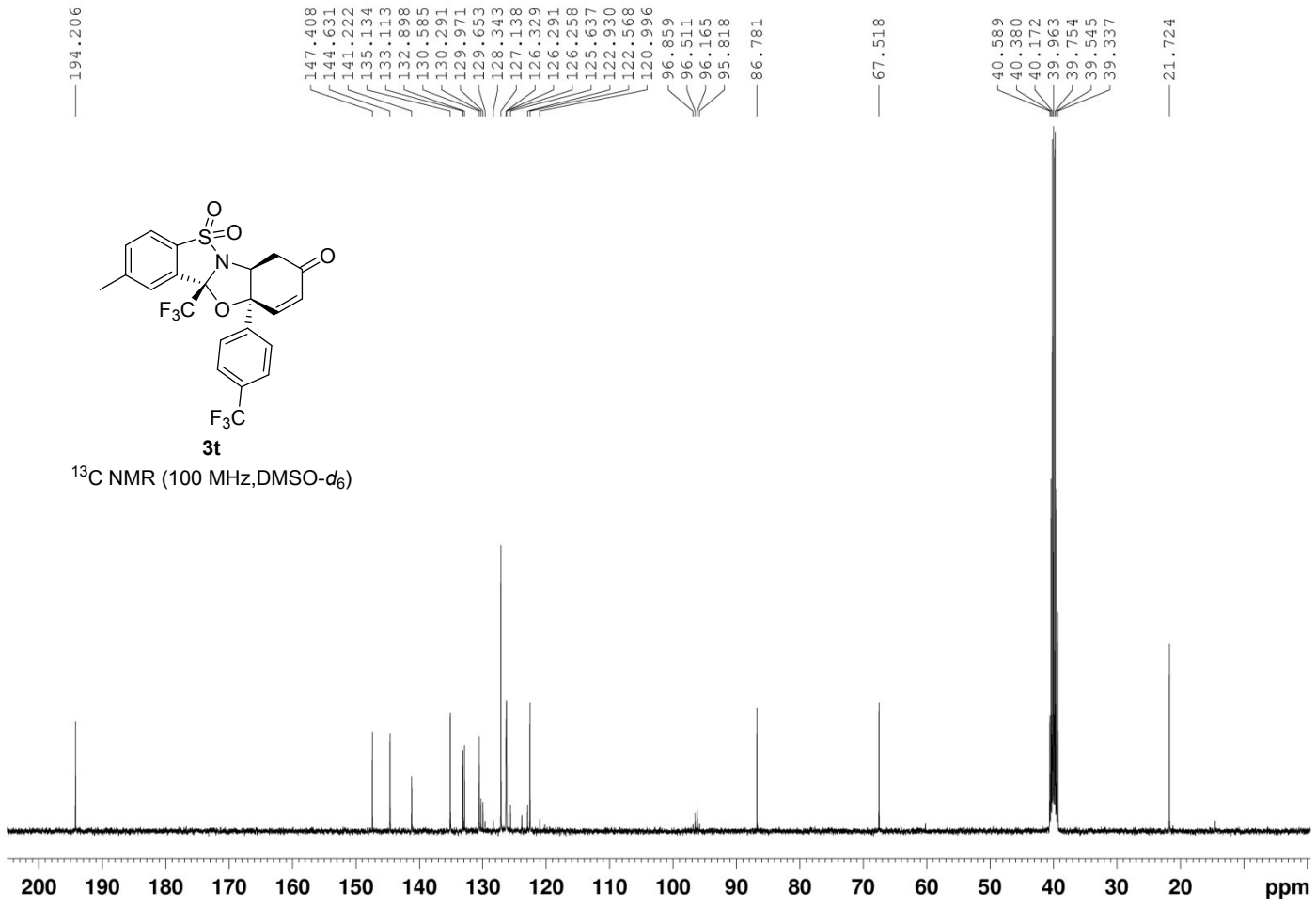
3t

^{1}H NMR (400 MHz, DMSO-d_6)



3t

^{19}F NMR (376 MHz, DMSO-d_6)

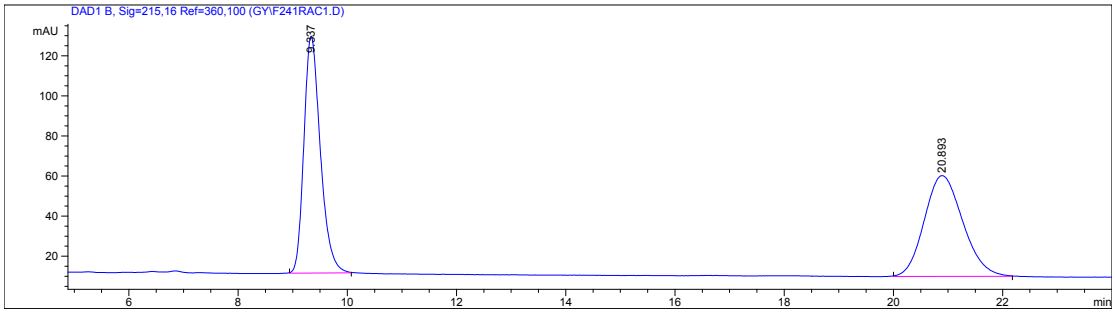


3t

^{13}C NMR (100 MHz,DMSO-d_6)

Copies of HPLC Traces

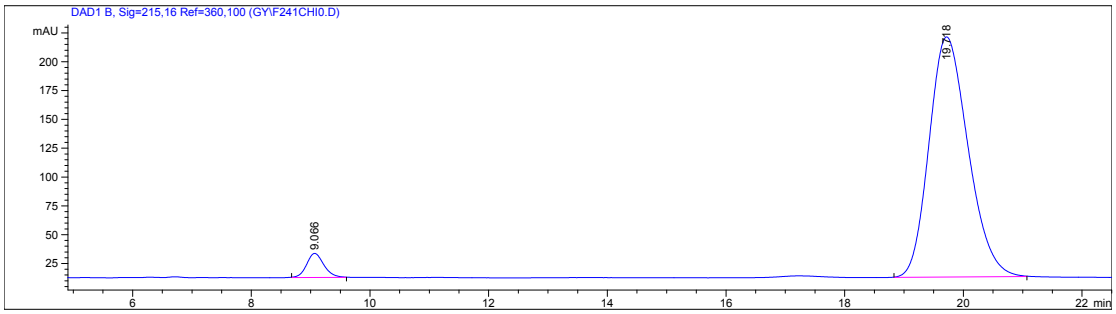
3a



Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.337	BB	0.3201	2474.32959	118.23615	50.1695
2	20.893	BB	0.7305	2457.60571	50.25473	49.8305
Totals :				4931.93530	168.49088	

Results obtained with enhanced integrator!

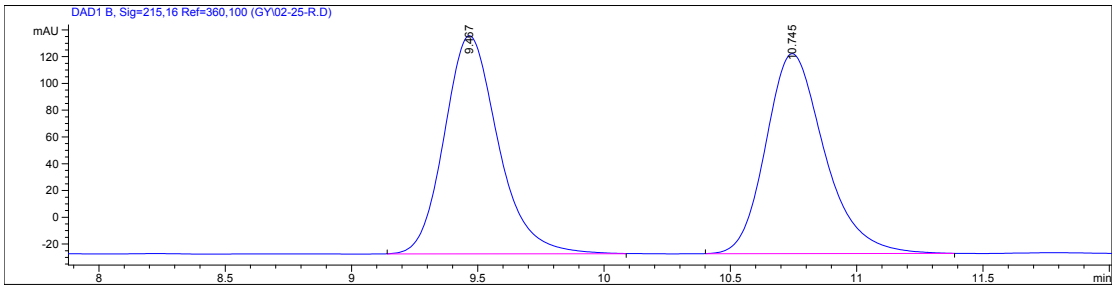


Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.066	BB	0.3136	422.69162	20.93247	4.2478
2	19.718	BB	0.7096	9528.15039	208.32077	95.7522
Totals :				9950.84201	229.25324	

Results obtained with enhanced integrator!

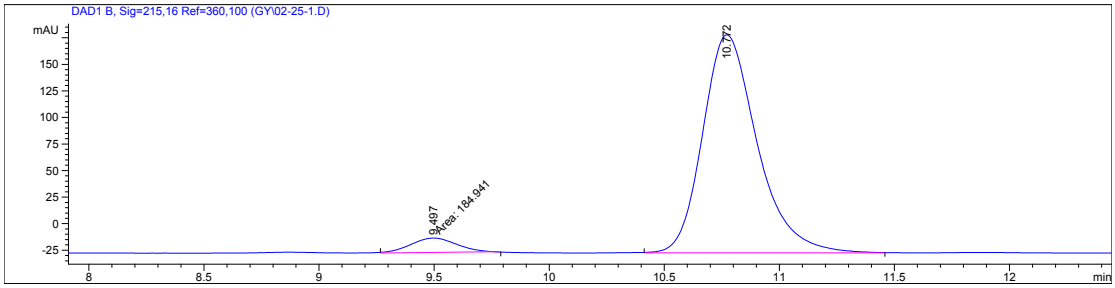
3b



Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.467	BB	0.2271	2418.93408	163.04536	50.0663
2	10.745	BB	0.2463	2412.52319	149.44856	49.9337
Totals :				4831.45728	312.49393	

Results obtained with enhanced integrator!

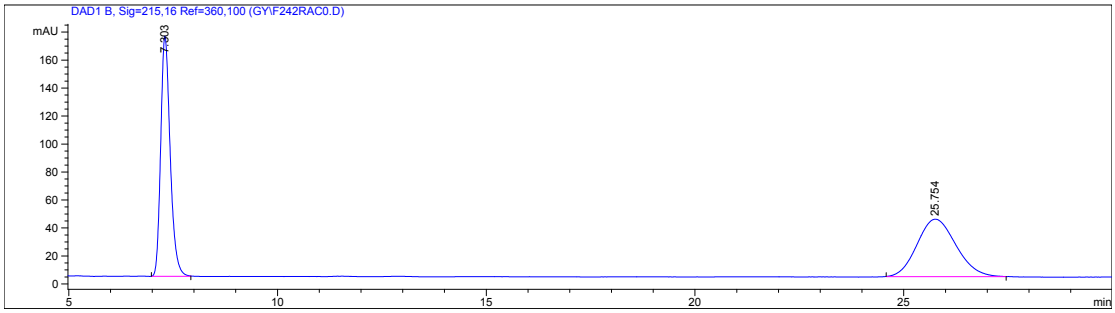


Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.497	MM	0.2285	184.94072	13.48864	5.1945
2	10.772	BB	0.2499	3375.38037	205.24947	94.8055
Totals :				3560.32109	218.73811	

Results obtained with enhanced integrator!

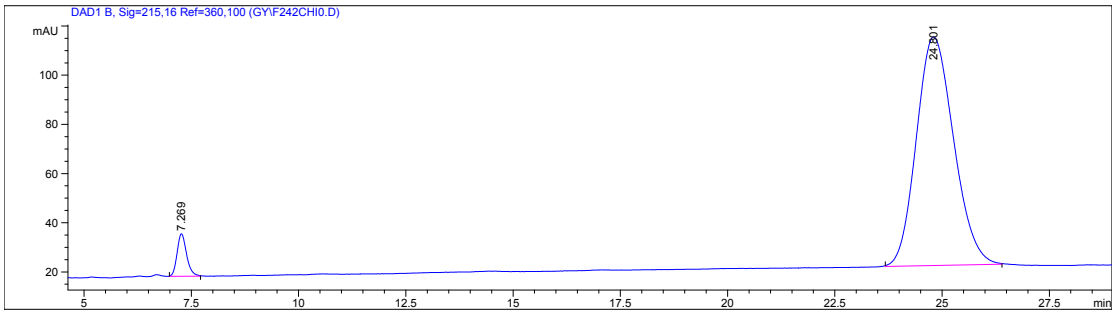
3c



Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.303	PB	0.2398	2704.30640	171.62347	50.3792
2	25.754	BB	0.8944	2663.60107	41.08314	49.6208
Totals :				5367.90747	212.70661	

Results obtained with enhanced integrator!

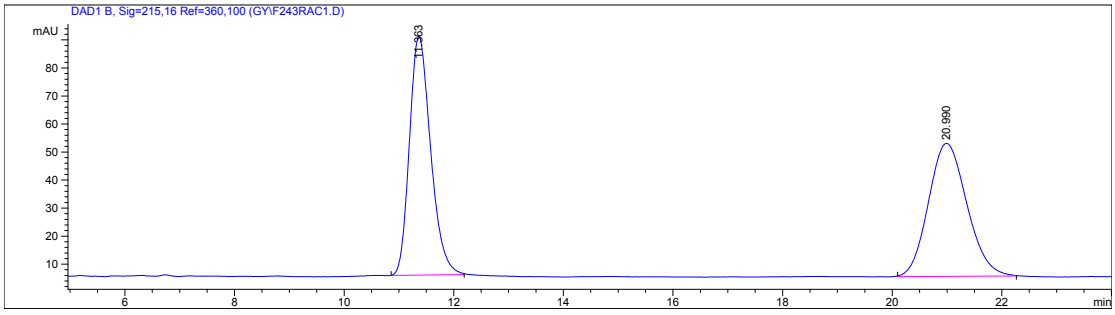


Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.269	BB	0.2354	265.94446	17.29000	4.5210
2	24.801	BB	0.9150	5616.48926	92.94878	95.4790
Totals :				5882.43372	110.23878	

Results obtained with enhanced integrator!

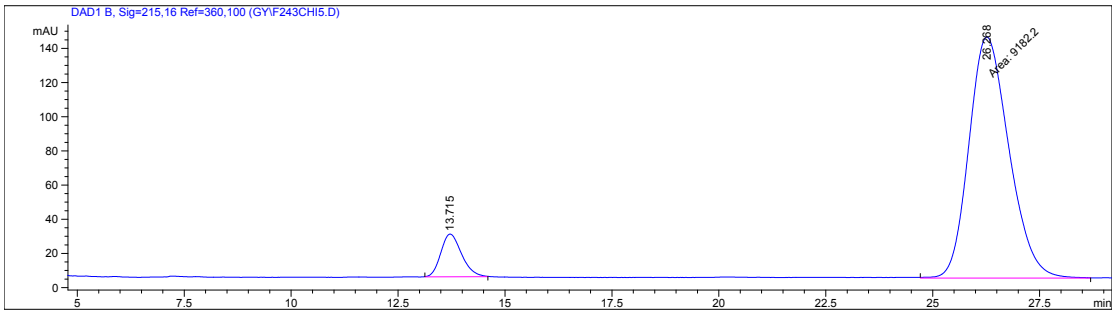
3d



Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.363	BB	0.4135	2290.09521	85.21412	49.8133
2	20.990	BB	0.7234	2307.26270	47.44514	50.1867
Totals :				4597.35791	132.65926	

Results obtained with enhanced integrator!

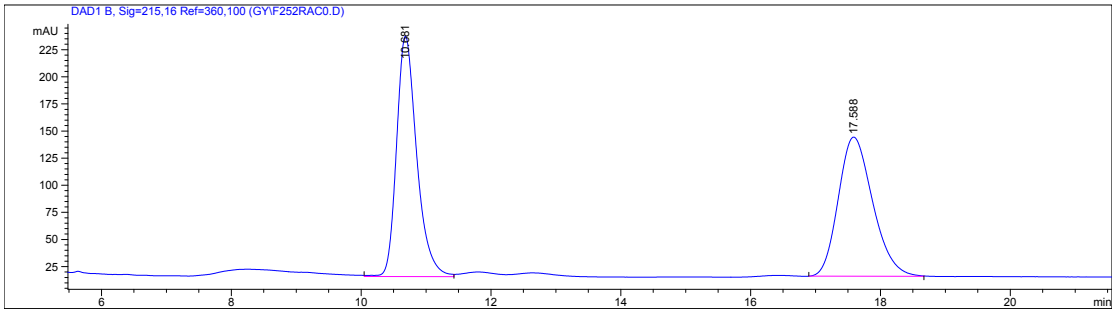


Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.715	BB	0.5131	846.65613	25.00082	8.4422
2	26.268	MM	1.0847	9182.19531	141.09076	91.5578
Totals :				1.00289e4	166.09158	

Results obtained with enhanced integrator!

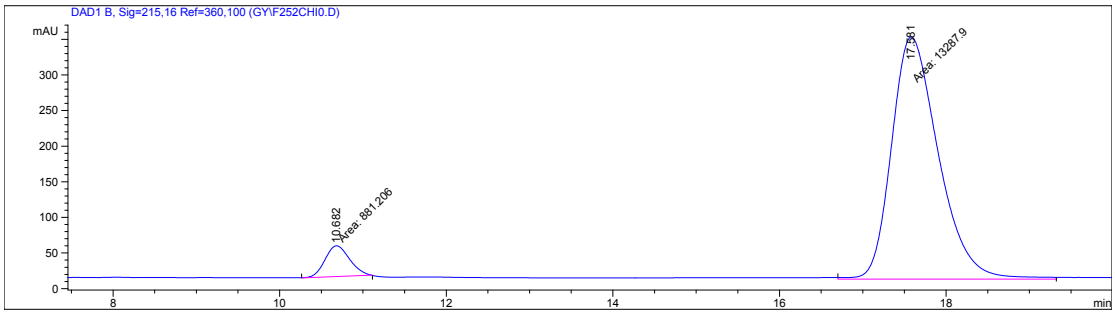
3e



Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.681	VB	0.3300	4786.33008	221.62508	50.3814
2	17.588	PB	0.5608	4713.86963	128.24417	49.6186
Totals :				9500.19971	349.86925	

Results obtained with enhanced integrator!

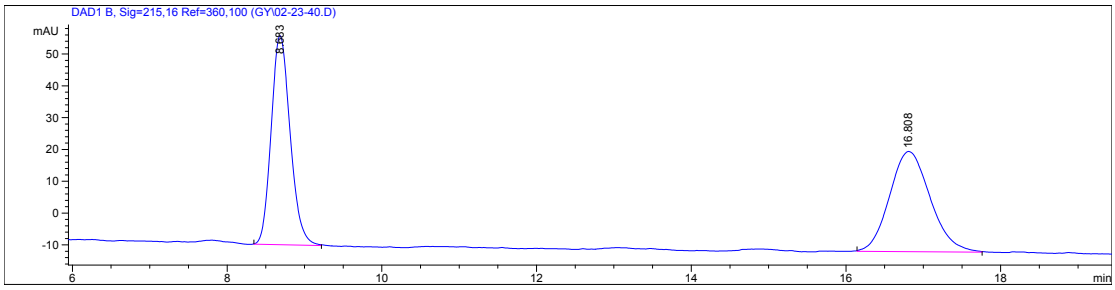


Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.682	MM	0.3403	881.20557	43.16413	6.2192
2	17.581	MM	0.6511	1.32879e4	340.12375	93.7808
Totals :				1.41691e4	383.28788	

Results obtained with enhanced integrator!

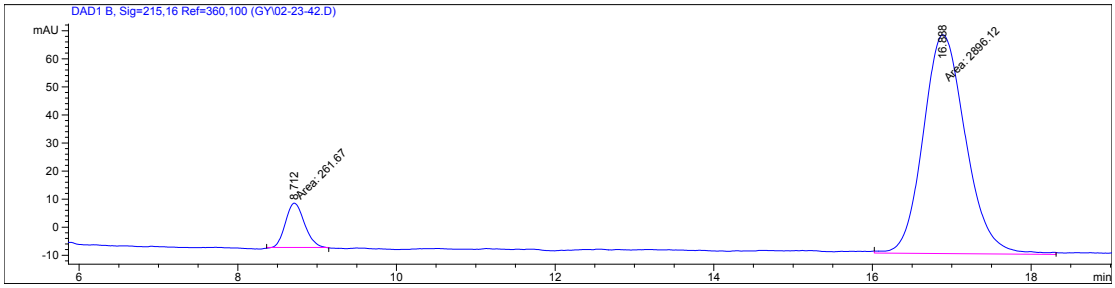
3f



Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.683	BB	0.2628	1119.59888	65.70511	49.3901
2	16.808	BB	0.5489	1147.25195	31.49907	50.6099
Totals :				2266.85083	97.20418	

Results obtained with enhanced integrator!

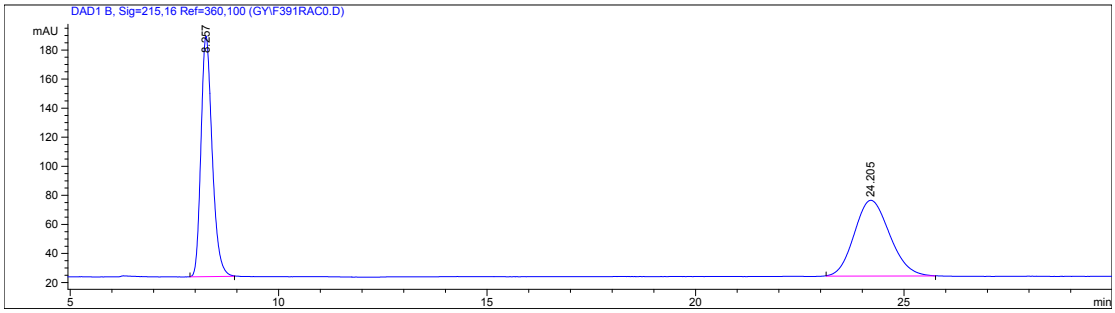


Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.712	MM	0.2764	261.67041	15.77636	8.2865
2	16.888	MM	0.6194	2896.11548	77.92461	91.7135
Totals :				3157.78589	93.70097	

Results obtained with enhanced integrator!

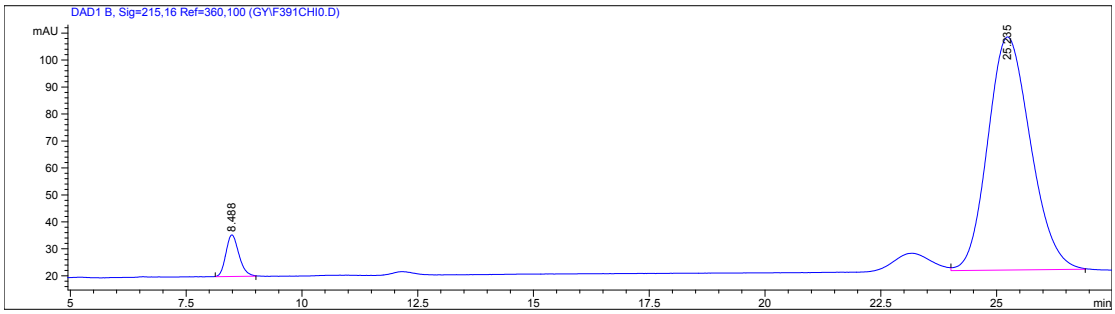
3g



Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.257	BB	0.2863	3096.47852	165.46751	50.1144
2	24.205	BB	0.8776	3082.34204	52.14561	49.8856
Totals :				6178.82056	217.61312	

Results obtained with enhanced integrator!

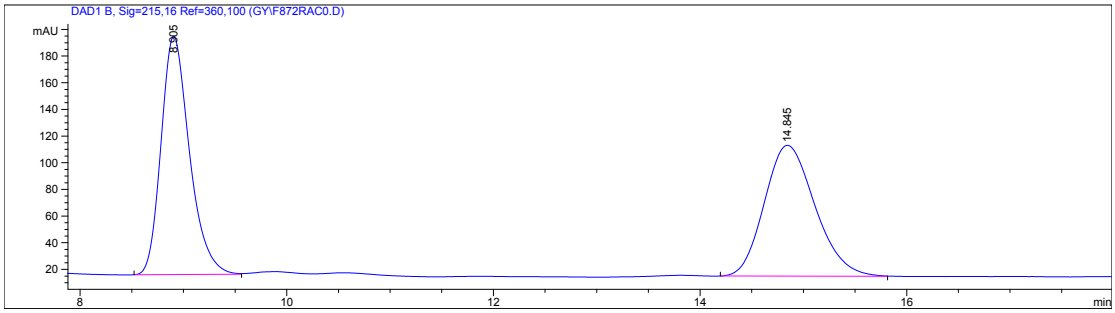


Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.488	BB	0.3001	301.20364	15.40321	5.1541
2	25.235	VB	0.9781	5542.71631	86.43806	94.8459
Totals :				5843.91995	101.84127	

Results obtained with enhanced integrator!

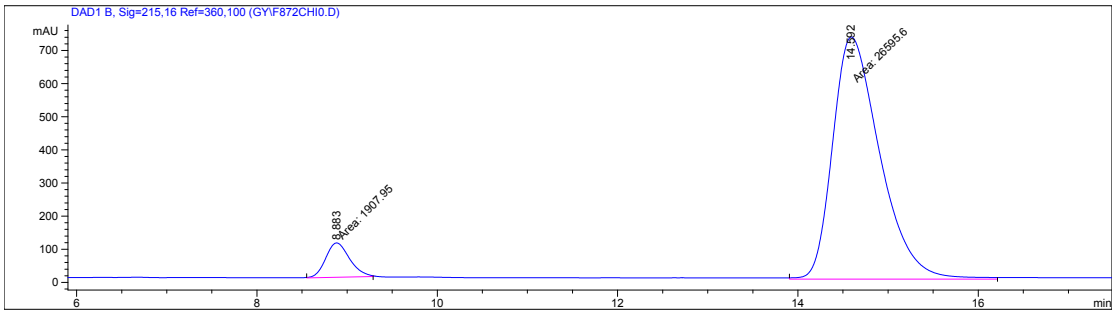
3h



Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.905	BB	0.2906	3412.56519	178.83540	50.5362
2	14.845	PB	0.5255	3340.14722	98.04029	49.4638
Totals :				6752.71240	276.87569	

Results obtained with enhanced integrator!

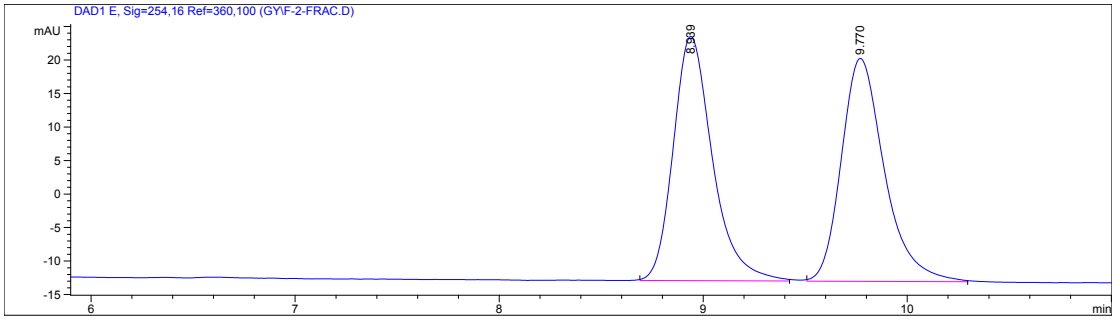


Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.883	MM	0.3067	1907.94775	103.68465	6.6937
2	14.592	MM	0.6061	2.65956e4	731.29083	93.3063
Totals :				2.85035e4	834.97549	

Results obtained with enhanced integrator!

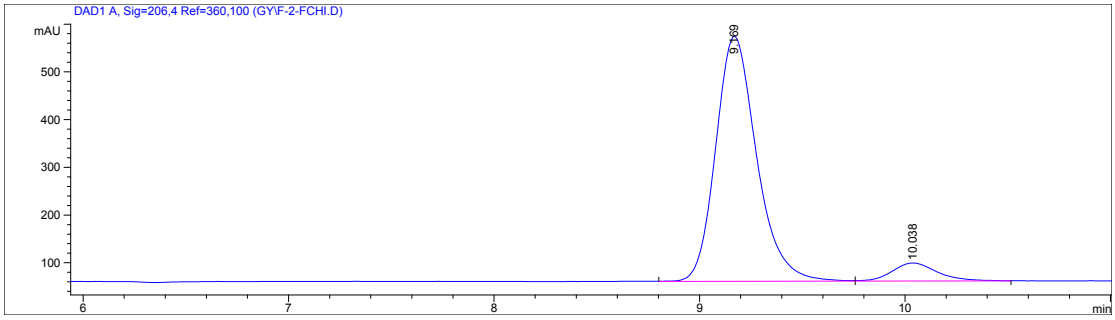
3i



Signal 5: DAD1 E, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.939	BB	0.2040	487.19089	36.44607	49.9668
2	9.770	BB	0.2231	487.83835	33.26895	50.0332
Totals :				975.02924	69.71502	

Results obtained with enhanced integrator!

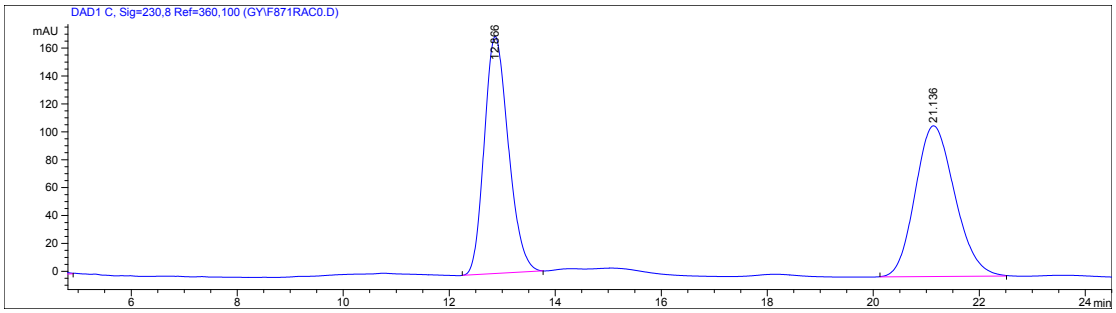


Signal 1: DAD1 A, Sig=206,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.169	BV	0.2114	7102.36328	513.52216	92.4946
2	10.038	VB	0.2355	576.31812	37.87993	7.5054
Totals :				7678.68140	551.40209	

Results obtained with enhanced integrator!

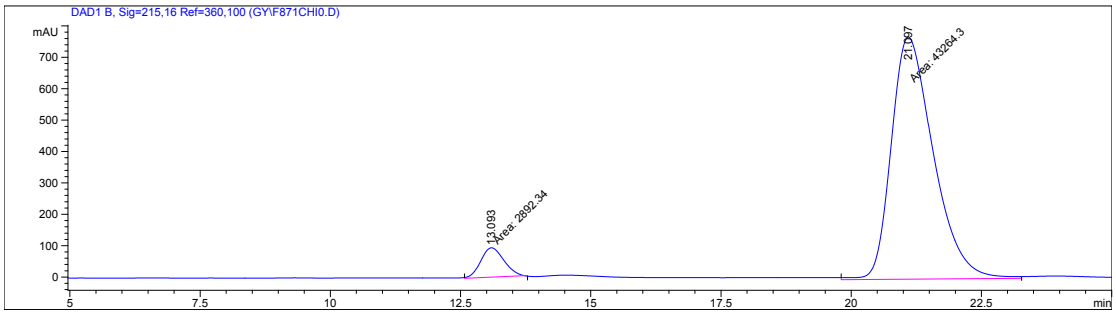
3j



Signal 3: DAD1 C, Sig=230,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.866	PP	0.4902	5363.74414	169.94264	48.9151
2	21.136	BB	0.8112	5601.66504	107.98713	51.0849
Totals :				1.09654e4	277.92977	

Results obtained with enhanced integrator!

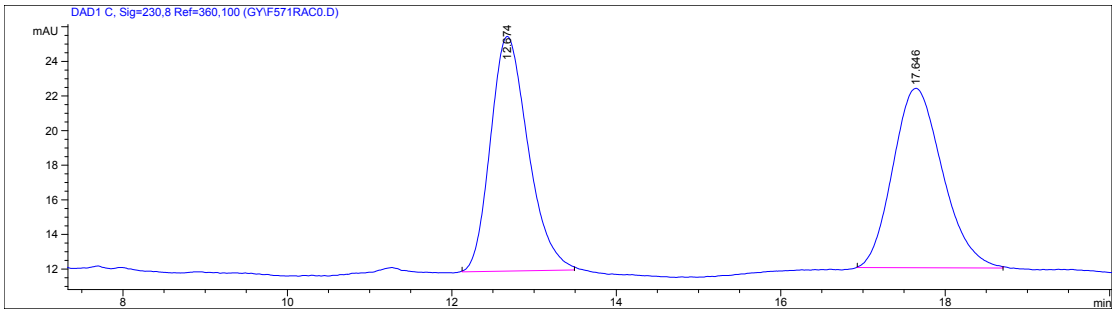


Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.093	MM	0.5156	2892.34058	93.50149	6.2664
2	21.097	MM	0.9354	4.32643e4	770.86383	93.7336
Totals :				4.61567e4	864.36532	

Results obtained with enhanced integrator!

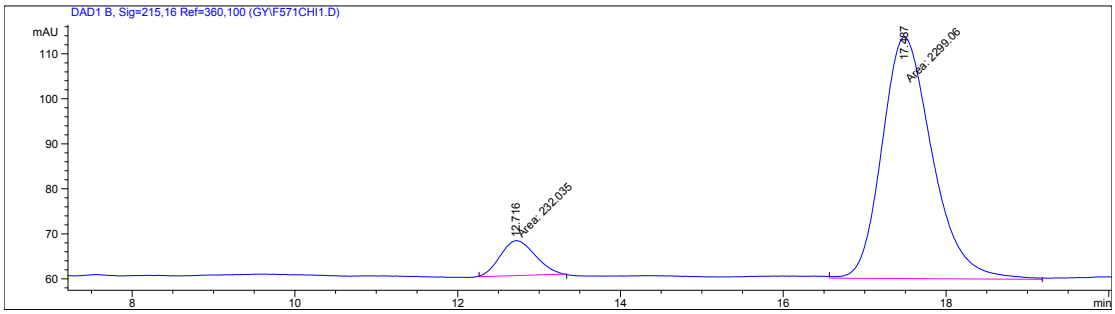
3k



Signal 3: DAD1 C, Sig=230,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.674	BB	0.4685	430.67780	13.56209	49.7892
2	17.646	BB	0.5908	434.32388	10.36702	50.2108
Totals :				865.00168	23.92911	

Results obtained with enhanced integrator!

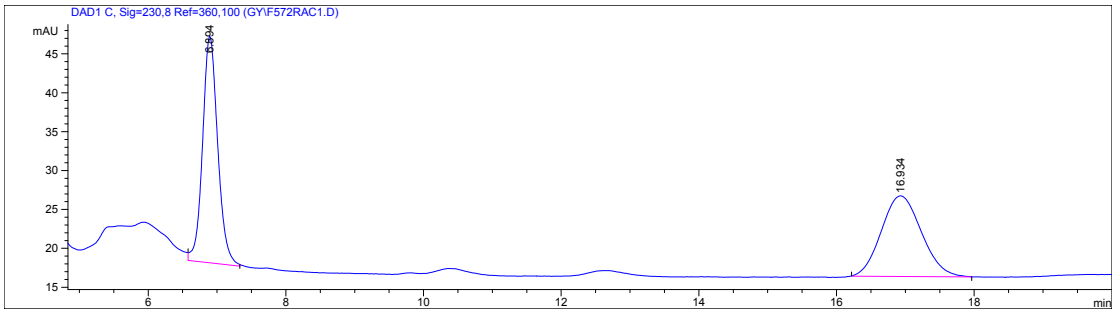


Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.716	MM	0.4989	232.03473	7.75102	9.1674
2	17.487	MM	0.7144	2299.06274	53.63423	90.8326
Totals :				2531.09747	61.38525	

Results obtained with enhanced integrator!

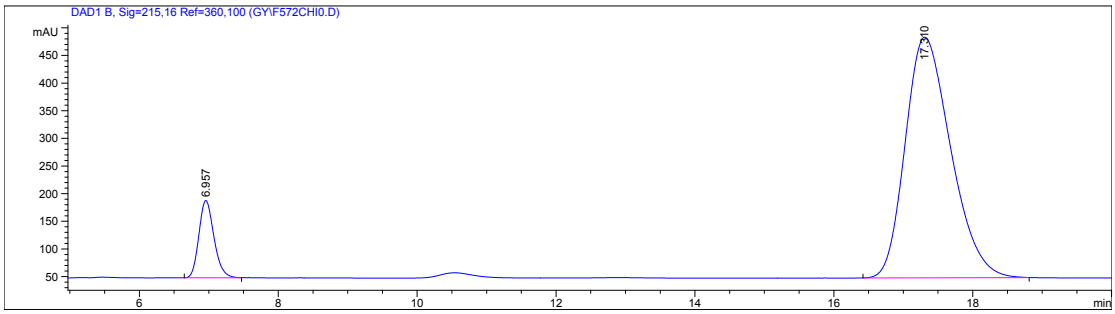
3l



Signal 3: DAD1 C, Sig=230,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.894	VB	0.2343	444.80011	29.10725	51.4135
2	16.934	BB	0.5971	420.34189	10.36584	48.5865
Totals :				865.14200	39.47309	

Results obtained with enhanced integrator!

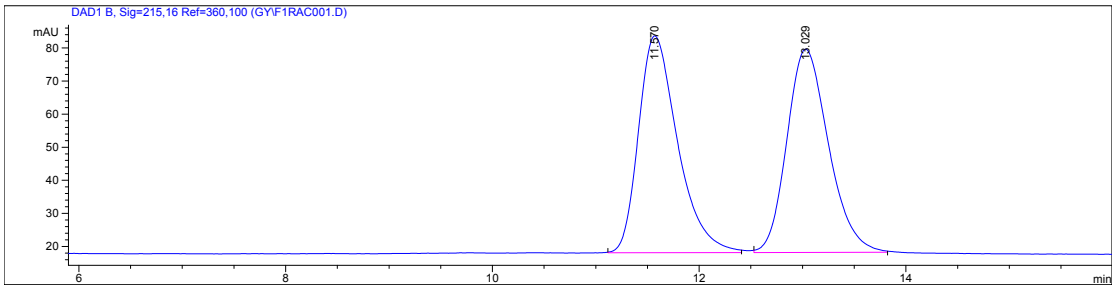


Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.957	PB	0.2316	2105.15405	139.82870	9.6093
2	17.310	BB	0.6986	1.98024e4	435.41336	90.3907
Totals :				2.19075e4	575.24207	

Results obtained with enhanced integrator!

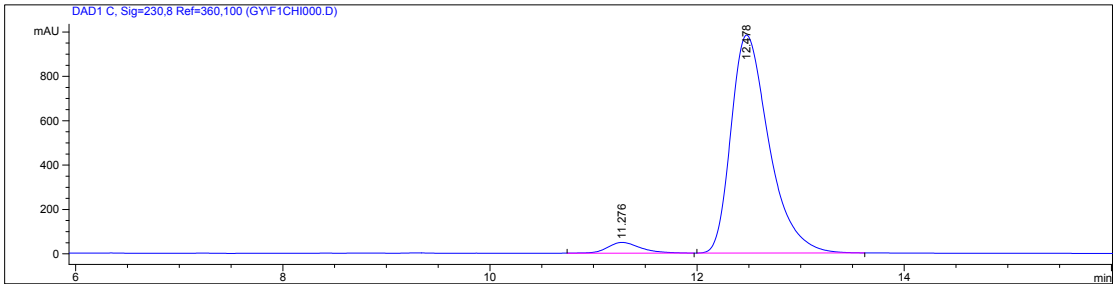
3m



Signal 3: DAD1 C, Sig=230,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.570	BB	0.3946	950.20587	36.63620	50.1199
2	13.029	BB	0.4246	945.66058	34.19651	49.8801
Totals :				1895.86646	70.83271	

Results obtained with enhanced integrator!

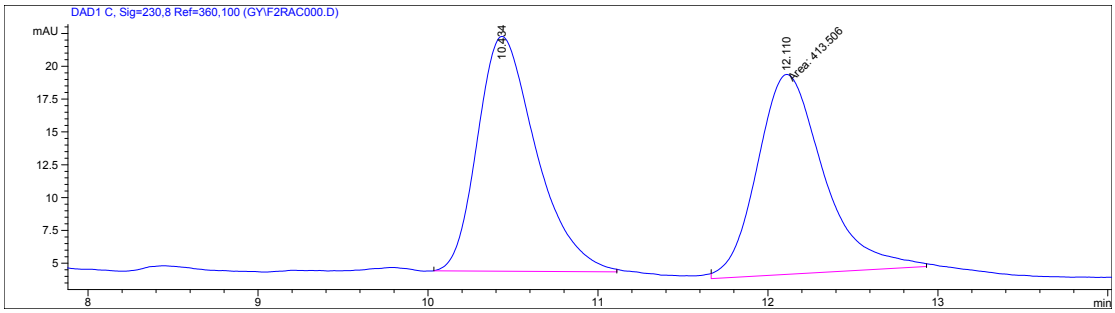


Signal 3: DAD1 C, Sig=230,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.276	BB	0.3354	1078.77905	48.51825	4.1368
2	12.478	BB	0.3888	2.49986e4	982.73853	95.8632
Totals :				2.60774e4	1031.25678	

Results obtained with enhanced integrator!

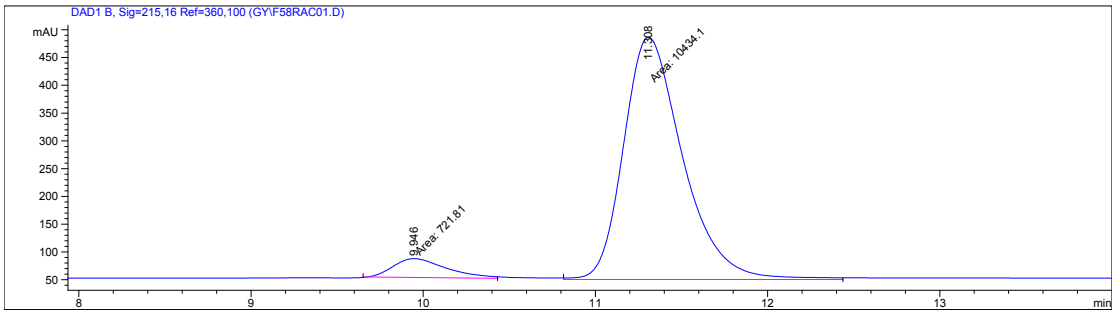
3n



Signal 3: DAD1 C, Sig=230,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.434	PB	0.3636	428.35342	17.87481	50.8818
2	12.110	MM	0.4527	413.50604	15.22388	49.1182
Totals :				841.85947	33.09869	

Results obtained with enhanced integrator!



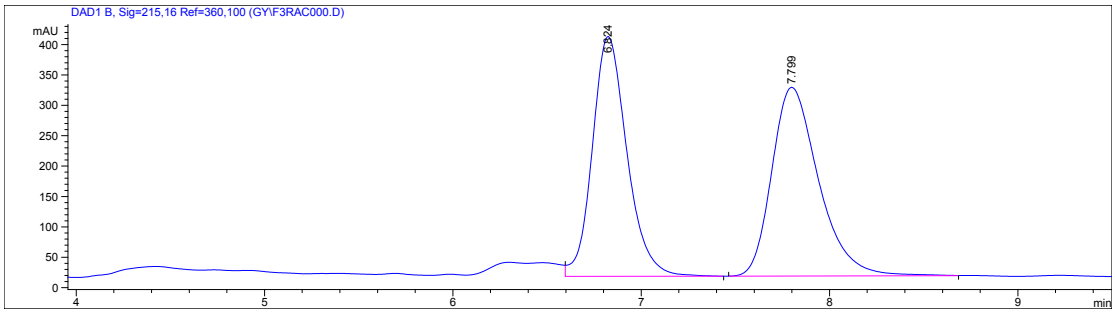
Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.946	MM	0.3559	721.80957	33.80137	6.4702
2	11.308	MM	0.3982	1.04341e4	436.66422	93.5298
Totals :				1.11559e4	470.46559	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=230,8 Ref=360,100

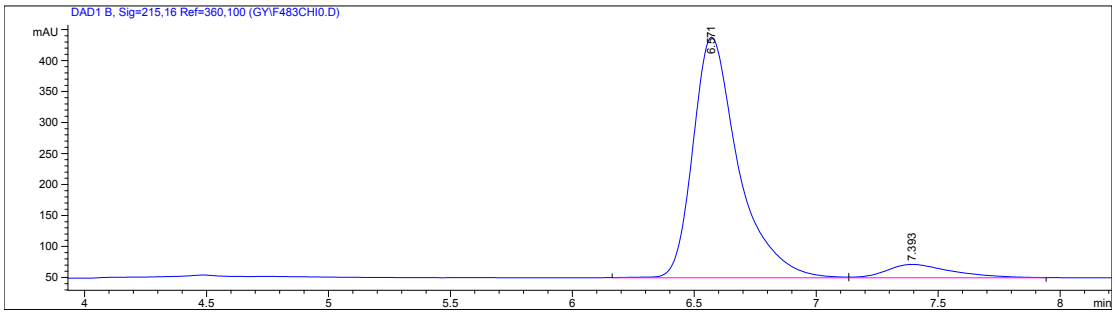
3o



Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.824	VP	0.1950	5042.59912	394.86038	48.4858
2	7.799	BB	0.2633	5357.55713	310.54477	51.5142
Totals :				1.04002e4	705.40515	

Results obtained with enhanced integrator!

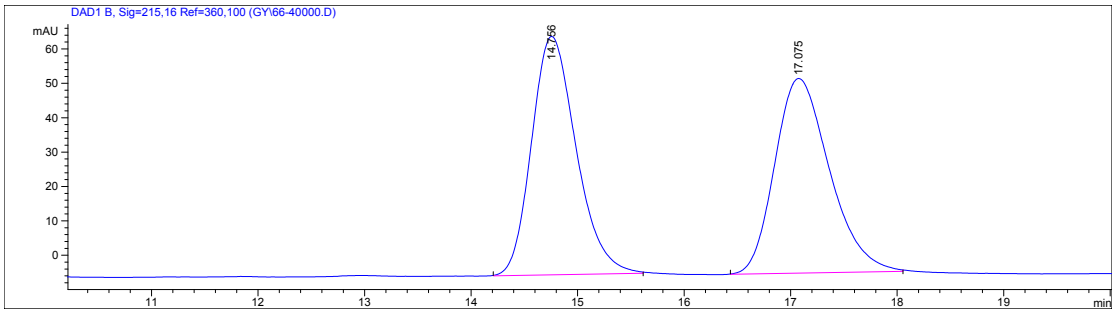


Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.571	BV	0.1925	5008.24316	388.28616	92.5948
2	7.393	VB	0.2725	400.53119	21.58668	7.4052
Totals :				5408.77435	409.87284	

Results obtained with enhanced integrator!

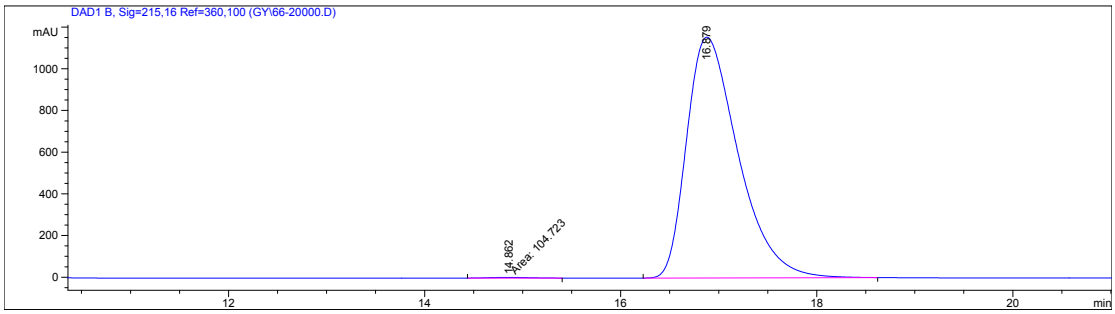
3p



Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.756	BB	0.4473	2016.13000	69.32650	49.9505
2	17.075	BB	0.5462	2020.12683	56.62203	50.0495
Totals :				4036.25684	125.94853	

Results obtained with enhanced integrator!

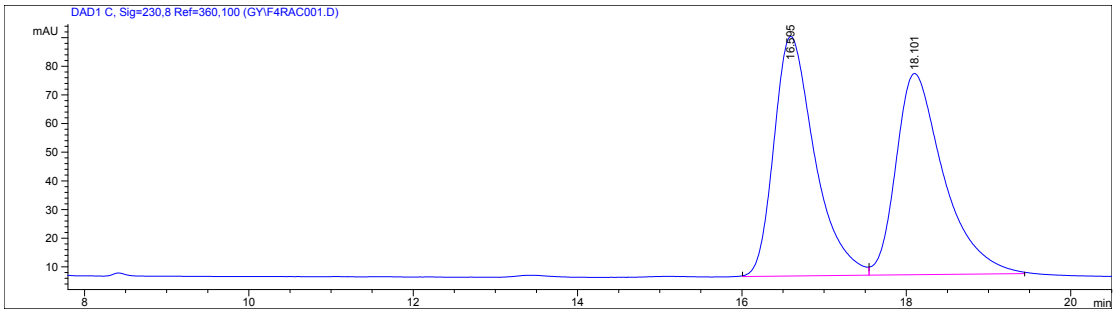


Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.862	MM	0.5026	104.72283	3.47242	0.2473
2	16.879	BB	0.5646	4.22500e4	1155.45154	99.7527
Totals :				4.23547e4	1158.92396	

Results obtained with enhanced integrator!

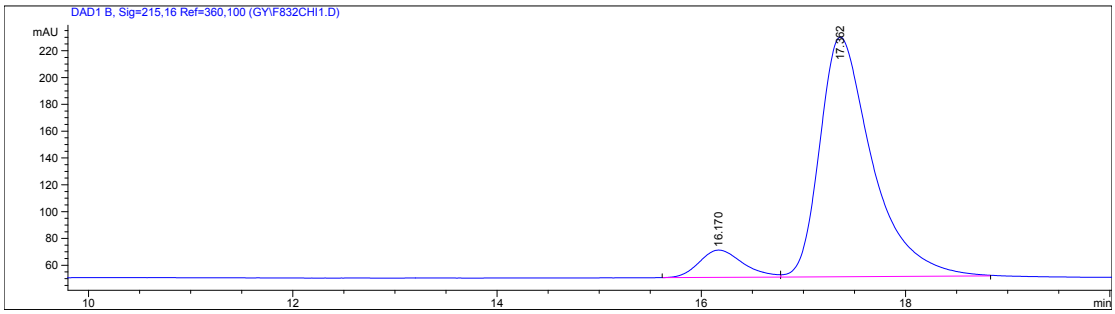
3q



Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.594	BV	0.5203	6230.10156	180.65445	49.9676
2	18.101	VB	0.6098	6238.18555	152.31424	50.0324
Totals :				1.24683e4	332.96869	

Results obtained with enhanced integrator!

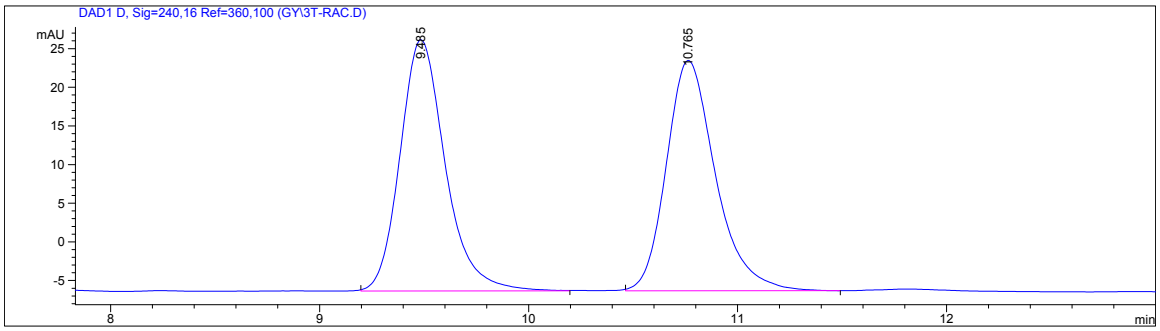


Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.170	BV	0.4479	602.53778	20.32400	8.6425
2	17.362	VB	0.5300	6369.28955	178.62598	91.3575
Totals :				6971.82733	198.94997	

Results obtained with enhanced integrator!

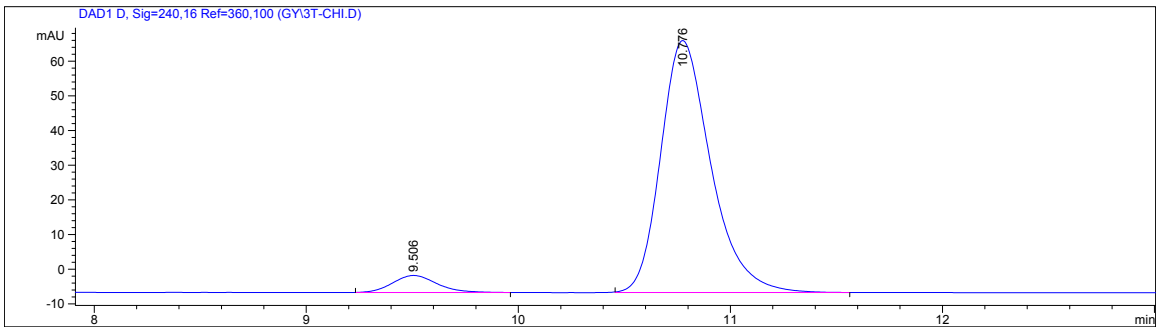
3r



Signal 4: DAD1 D, Sig=240,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.485	BB	0.2315	488.38916	32.47125	50.0727
2	10.765	BP	0.2508	486.97089	29.78315	49.9273
Totals :				975.36005	62.25440	

Results obtained with enhanced integrator!



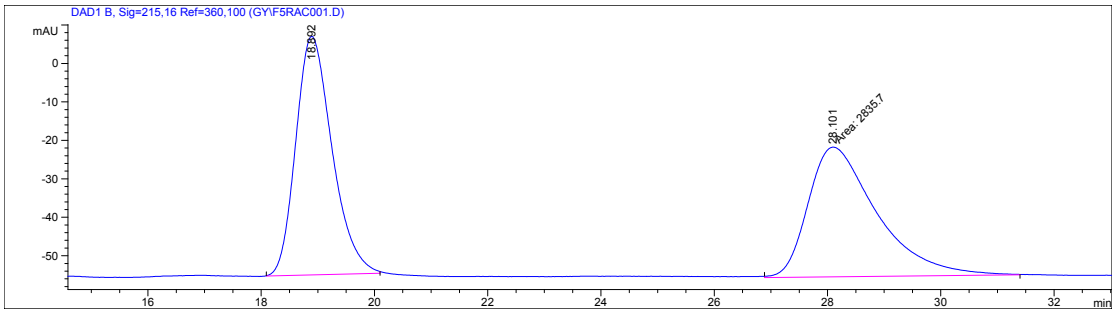
Signal 4: DAD1 D, Sig=240,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.506	BB	0.2266	71.85982	4.91526	5.6698
2	10.776	BB	0.2520	1195.54358	72.63984	94.3302
Totals :				1267.40340	77.55510	

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=254,16 Ref=360,100

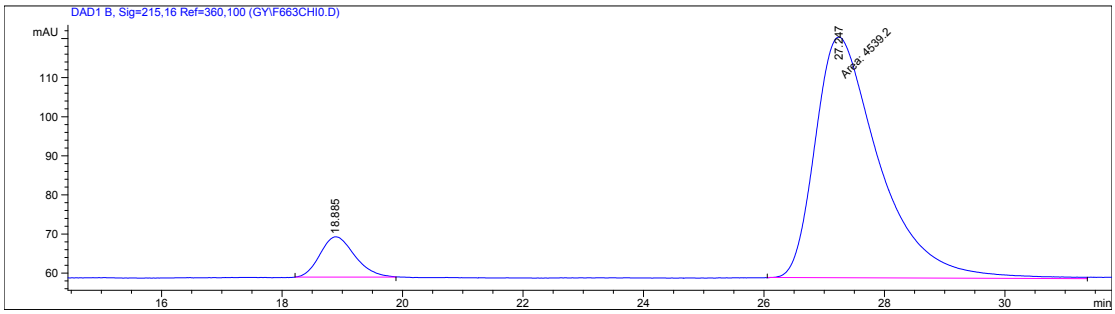
3s



Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.892	BB	0.6719	2762.99390	62.00513	49.3507
2	28.101	MM	1.4018	2835.69873	33.71495	50.6493
Totals :				5598.69263	95.72008	

Results obtained with enhanced integrator!

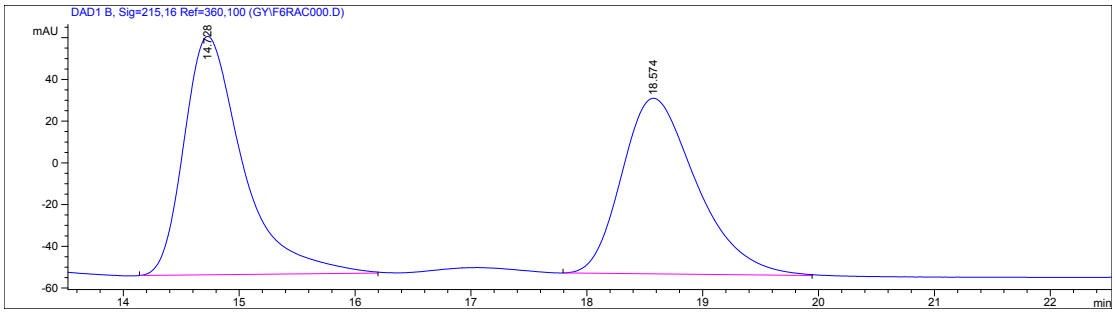


Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.885	BB	0.5660	413.49335	10.30754	8.3489
2	27.247	MM	1.2299	4539.20117	61.51377	91.6511
Totals :				4952.69452	71.82131	

Results obtained with enhanced integrator!

3t

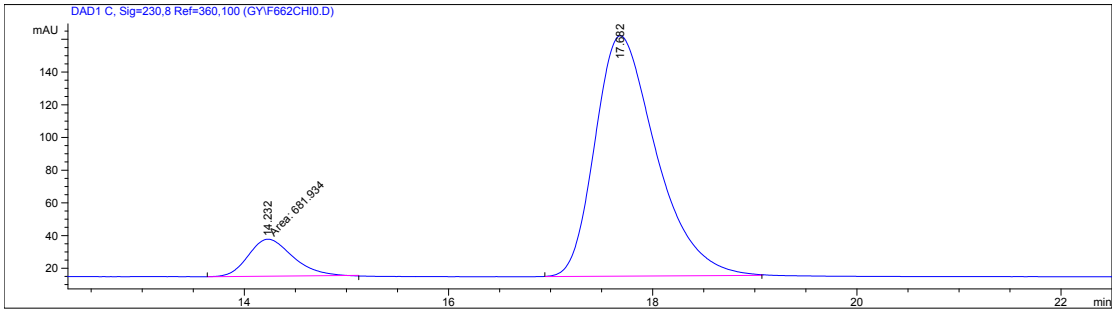


Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.728	PB	0.5283	4019.70166	114.29601	51.5931
2	18.574	PB	0.6867	3771.46582	84.17692	48.4069
Totals :				7791.16748	198.47292	

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=230,8 Ref=360,100

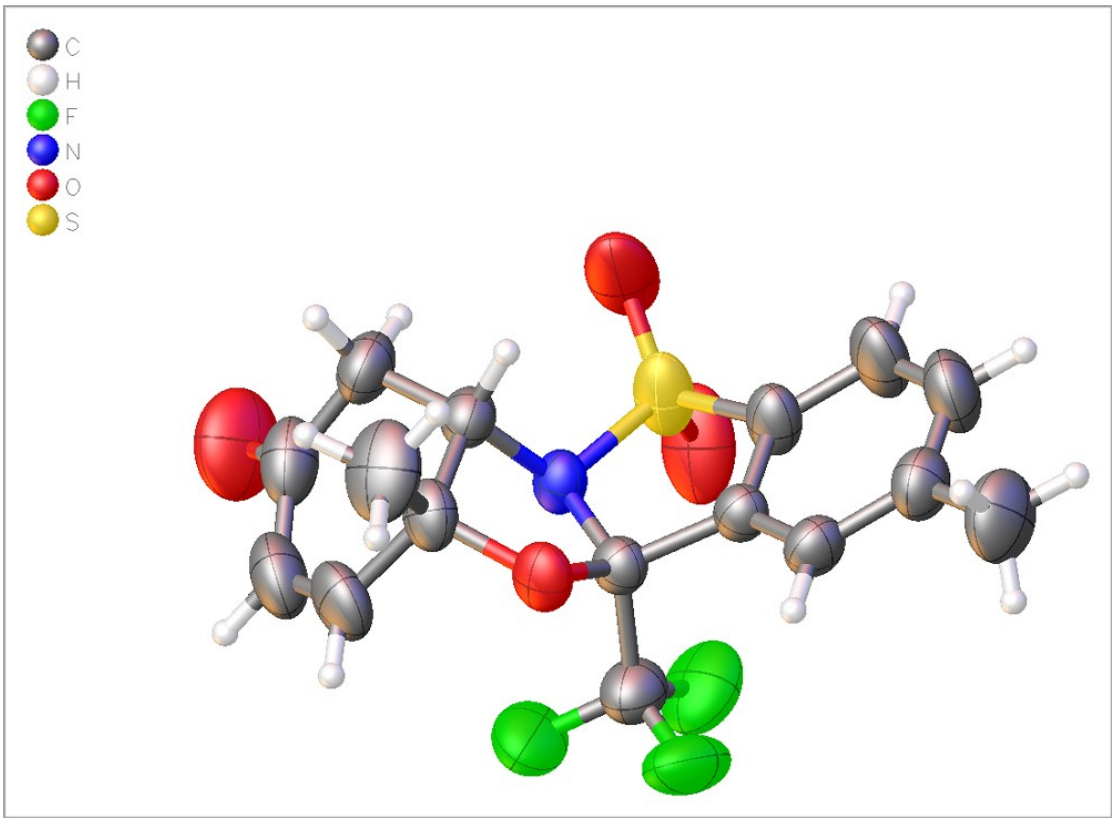


Signal 3: DAD1 C, Sig=230,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.232	MM	0.5029	681.93396	22.60186	10.0319
2	17.682	BB	0.6354	6115.72070	147.00592	89.9681
Totals :				6797.65466	169.60778	

Results obtained with enhanced integrator!

Crystal structure data of 3a



Crystal data and structure refinement for 3a

Empirical formula	$C_{16}H_{14}F_3NO_4S$
Formula weight	373.34
Temperature/K	291(2)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	7.91930(10)
b/Å	15.3744(2)
c/Å	16.4929(2)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	2008.08(4)
Z	4
ρ_{calc}g/cm^3	1.235
μ/mm^{-1}	1.853
F(000)	768.0
Crystal size/mm^3	0.250 × 0.240 × 0.210
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	7.862 to 142.202
Index ranges	$-9 \le h \le 4$, $-15 \le k \le 18$, $-19 \le l \le 19$

Reflections collected	6685
Independent reflections	3761 [R_{int} = 0.0198, R_{sigma} = 0.0201]
Data/restraints/parameters	3761/0/228
Goodness-of-fit on F^2	1.046
Final R indexes [I>=2σ (I)]	R_1 = 0.0401, wR_2 = 0.1113
Final R indexes [all data]	R_1 = 0.0407, wR_2 = 0.1125
Largest diff. peak/hole / e $Å^{-3}$	0.18/-0.33
Flack parameter	0.001(11)